IN THE UNITED STATES DISTRICT COURT
FOR THE DISTRICT OF NEVADA

| | | |
|---|---|---|
| NEVADA POWER COMPANY,<br>A NEVADA CORPORATION,<br>PLAINTIFF, | : | |
| VS. | : | CV-S-89-555-LDG-LRL |
| MONSANTO COMPANY, A FOREIGN CORPORATION;<br>GENERAL ELECTRIC COMPANY,<br>A FOREIGN CORPORATION;<br>WESTINGHOUSE ELECTRIC CORPORATION, A FOREIGN CORPORATION; AND DOES I XXV, INCLUSIVE,<br>DEFENDENTS. | : | |

**EXHIBITS 19 - 37**
TO THE DEPOSITION OF ROBERT EMMET KELLY, M.D.
VOLUME I
TAKEN ON FEBRUARY 15, 1994



*MARTIN & ASSOCIATES*
*CERTIFIED COURT REPORTERS*
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# Aroclor 1260

(Aroclor Reg. U. S. Pat. Off.)

**CAUTION! AVOID PROLONGED AND REPEATED CONTACT WITH SKIN.**

**AVOID PROLONGED BREATHING OF VAPOR AND DUST.**

**MANUFACTURED BY MONSANTO CHEMICAL COMPANY • ST. LOUIS 4, MO.**

1060-290—7-54—53

NEV 011312

Monsanto

# TRANSFORMER PYRANOL* A-13-B 3B

(REPLACES PYRANOL 1467 and PYRANOL 1470)

## Caution! Contains chlorinated hydrocarbons

**Avoid prolonged breathing of vapors or mists.**

**Avoid contact with eyes or prolonged contact with skin.**

**If skin contact occurs, remove by washing with soap and water. Following eye contact flush with water.**

**If clothing becomes soaked with fluid, launder before wearing again.**

MADE FOR GENERAL ELECTRIC COMPANY

*Trademark of GENERAL ELECTRIC COMPANY

LOT NO. _____

**54 U.S. GALLONS** **NET WT. 675 LBS.**

MONSANTO CHEMICAL COMPANY ST. LOUIS, MISSOURI, U.S.A. 810.97-300.02/53

Monsanto

# INERTEEN* PPO

NEV 011262

## CAUTION!

**CONTAINS CHLORINATED HYDROCARBONS.**

**Avoid prolonged breathing of vapors or mists.**

**Avoid contact with eyes or prolonged contact with skin.**

**If skin contact occurs, remove by washing with soap and water. Following eye contact flush with water.**

**If clothing becomes soaked with fluid, launder before wearing again.**

Made for WESTINGHOUSE ELECTRIC CORPORATION.

*Trademark of WESTINGHOUSE ELECTRIC CORPORATION.

This product contains polychlorinated biphenyls, which some studies have shown may be an environmental contaminant. Extreme care should be taken to prevent any entry into the environment through spills, leakage, use, disposal, vaporization or otherwise.



LOT NO. _____ PACKER

| | | | | | | |
|---|---|---|---|---|---|---|
| **NET** | LEGAL **674** LBS. | **305.72** KILOS | TARE | 48 LBS. | 21.77 KILOS |
| | | | GROSS | 722 LBS. | 327.49 KILOS |

MONSANTO COMPANY, ST. LOUIS, MISSOURI, U.S.A. 810.92-200.0 8/53

# THE PROPER HANDLING OF AROCLORS AND THEIR MIXTURES IN THE ELECTRICAL INDUSTRY

Monsanto Chemical Co.
Organic Div. Sales Dept.
800 N. Lindbergh Blvd.
St. Louis 66, Mo.

P. G. BENIGNUS
Revised
January 1960

INDEX

Attachments:

Drawing No. 31-20847, The Tank Car
Drawing No. 31-20848, Dome Detail
Drawing No. 9C-8170-5, The Horizontal Storage Tank
Drawing No. D-13362, The Vertical Storage Tank
Drawing No. 9C-8248, The Breather
Drawing No. 9C-8278, The Varec Gauge
Drawing No. 9C-8178, The Unloading Platform

# THE PROPER HANDLING OF AROCLORS* AND THEIR MIXTURES IN THE ELECTRICAL INDUSTRY

## INTRODUCTION

Monsanto's Aroclors*, especially the chlorinated biphenyls including types 1242, 1248, 1254 and 1260, used alone or in combination with chlorinated benzenes, are commonly used dielectric materials of the askarel[1] class.

Askarel is a generic name referring to liquid dielectrics derived from halogenated aromatic hydrocarbons possessing excellent chemical and dielectric stability and fire-resistance over the temperature ranges and operating conditions required of transformers and capacitors in the electrical industry.

The properties of Aroclors and their mixtures, used as dielectrics are described in detail in Chapter 7 entitled, "Typical Properties". These dielectrics are manufactured under very carefully-controlled conditions in order to meet the strict and exacting electrical requirements and properties.

The electrical industry's use of these fluids has been largely in accordance with the General Electric Company's patents and developments.

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*Aroclors - Monsanto's chlorinated biphenyls and chlorinated polyphenyls, Registered U.S. Patent Office.

[1]F. M. Clark, "Electrical Insulation", Chem. Engg. News 25, 2977 (1947).

Resulting from the wide use of these materials in the industry, trade names have been established to identify them by different manufacturers of electrical equipment. Listed alphabetically the trade names include, "Chlorextol," Allis Chalmers; "Diaclor," Sangamo Electric; "Dykanol," Cornell Dubilier; "Elemex," Line Materials; "Hyvol," Aerovox; "Inerteen," Westinghouse Electric; "Noflamol," Wagner Electric; and Pyranol," General Electric Company.

The purpose of this bulletin is to assist the industry with the proper and safe handling of these dielectric materials in their operations.

## CHAPTER 1

## PROCEDURE FOR UNLOADING TANKCARS OF AROCLORS AND AROCLOR MIXTURES

### A. Description of the Cars

Aroclor and mixtures of Aroclors with chlorinated benzenes are shipped by Monsanto in two types of insulated tankcars- both of which are either aluminum lined or zinc-tin metallized. One type of car has heating coils inside of the tank and these are in direct contact with the product. The other, a more widely used type of car, is a double-shell tank with heating coils between the inner and outer shells. The steam coil connections are at the bottom of the car. Both types of tankcars are tested for 60 pounds pressure and their steam coils are tested for 200 pounds gauge pressure.

The cars are top-unloaded by displacement with dry air containing 10 mg. $H_2O$/cu. ft. maximum.

There are two or three connections on the tankcar dome depending on the type of car. Where three connections exist, one is a two inch diameter unloading line which extends to the bottom of the car, the second is a one inch diameter air inlet connection and the third is a two inch diameter pressure safety vent, which is a thin lead disc adjusted to release any pressure in excess of 60 pounds gauge. This safety vent is hooded for protection against dust, dirt or accidental bumping.

Where only two connections exist on the dome, one is the two inch diameter unloading line and the other is the safety vent. On these cars, it is necessary to remove the safety vent and introduce the displacement air through that connection.

* * * *

While the tankcar drawing gives much detail, we have been asked about the following points not given in the drawing:

1. The distance from the rail track to the top of the dome of the cars is variable. It is 13 feet and 4 inches for the 7,000 gallon cars and ranges from 11 feet to 14 feet for the 8,000 gallon cars.

2. The steam connections are located under the center of the cars.

3. The steam pipe connection is usually a two inch pipe, but on some cars the pipe size is 1-1/4 inches.

4. American Standard taper pipe threads are used.

* * * *

Drawing No. 31-20848 shows in detail the dome of a tankcar with three connections. "A" is the two inch unloading line which extends to a small sump at the bottom of the car. "B" is the one inch air inlet connection. "C" is the hooded safety vent. The car dome cover with fitted bolts is shown in the center. It is fitted with an aluminum envelope Goetze gasket. This drawing also shows a bottom opening in the car. This can be opened only from the inside of the car and its purpose is for cleaning operations. It has no use at all in unloading the car.

Drawing No. 31-20847 shows the overall dimensions of the 8,000 gallon Aroclor tankcar.

B. Procedure for Unloading the Cars

The car should be spotted at an unloading dock similar to the one shown by Drawing No. 9C-8178. The car must be level and the brakes set properly. "STOP - TANKCAR CONNECTED" signs should be placed fore and aft the car to warn switching crews.

If it is raning or snowing or the humidity is extremely high, it is not advisable to open the car. In case the car must be sampled and opened during bad weather, a canvas canopy must be placed over the dome of the car. It is preferable to unload the cars under roof or inside the factory. Unless it is absolutely necessary because of following described situations the dome cover should not be opened until ready for sampling. The dome cover is sealed with a standard railroad wire and seal, and Monsanto should be notified if this seal is found broken upon receipt of the car.

The first step in unloading is to inspect the dome and clean around the dome cover to remove all loose dirt, water or snow. Wiping rags and a brush should be used to clean before the dome cover and connections are opened.

Then, the screwed hood over the air-inlet valve should be removed and this valve opened fully and left open while heating the car. A Weston or metal encased thermometer should be inserted through this air-inlet valve opening and the temperature of the interior of the car determined.

If the car temperature is below the caution temperature shown in the following Table 1, it will be necessary to take the special step of inserting a "hair-pin" heating coil through the dome of the car to preclude rupturing the tankcar seams during the heating period.

TABLE I

| Product | ASTM Pour Point °C. | Temperature °C. Below which Caution Must Be Used in Heating |
|---|---|---|
| Aroclor 1260 | + 30 | + 40 |
| Aroclor 1254 | + 10 | + 20 |
| Aroclor 1248 | - 7 | + 5 |
| Aroclor 1242 | - 19 | - 10 |
| Interteen PPO | Pre-heating is not required* | |
| Pyranol 1467 | Pre-heating is not required* | |
| Pyranol 1470 | Pre-heating is not required* | |
| Pyranol 1481 | Pre-heating is not required* | |

*Except if the material has cooled below -10°C. and crystals of scavenger have separated. Then, the material should be heated to 70°C. (158°F.) until complete solution has been accomplished.

If the dome of the car is to be opened for the pre-heating operation, it is necessary that it be covered with a clean canvas. Extreme care must be taken to avoid getting dirt or moisture into the car.

It is due to the relatively high viscosity of some of the Aroclors at low temperatures that it becomes necessary to form a column of molten material from top to bottom of the car, in the center, to prevent hydraulic pressure build-up which may rupture the tank shell if there is too rapid localized heating when employing the main steam coils.

When such pre-heating is required, a satisfactory vent hole can be made by inserting a "hair-pin" coil (1/2 inch diameter brass, galvanized, or stainless steel pipe) into the open dome of the car and introducing steam through the coil until there is a column of fluid Aroclor from top to bottom.

After the vent hole is melted through the material to the bottom of the car, the "hair-pin" coil should be removed and the dome cover replaced and bolted.

Steam is then introduced into the main coils. It is recommended that the steam pressure be limited to 100 pounds gauge pressure, particularly when the coils are in direct contact with the Aroclor. The steam coil outlet should be trapped or throttled with a valve.

It will require eight to twenty hours to bring the material to pumping temperature - depending upon weather conditions. It is essential that the air inlet valve be open during the heating period in order to vent the tank.

Some calculations have been made to indicate the heat requirements for an Aroclor car. Data for an 8,000 gallon car of Aroclor 1254 are:

Specific Gravity = 1.5

Specific Heat = 0.26 Btu/lb./°F.

Heat requirement for heating Aroclor from 30°C. (86°F.) to 110°C. (230°F.) is:

8000 x 1.5 x 8.33 x 0.26 x (230-86) = 3,774,000 Btu.

For heating from 30°C. (86°F.) to only 75°C. (167°F.), the heat required is 2,110,000 Btu.

A nine horse power boiler operating at 80 psig produces 263 lbs./hr. of steam with no reused condensate. Returning condensate at 200°F. will increase the steam output to 296 lbs./hr. at 80 psig.

In the first case, heating to 110°C., the over-all heat transfer co-efficient is assumed to be too low to utilize the 100% capacity of the boiler. A value of 1500 for UA with an average $\Delta$ T of 133°F. indicates that the useable steam is 202,000 Btu/hr. or 226 lbs./hr. steam at 80 psig.

In the second case the $\Delta$ T is lower and the entire output of the boiler is useable. Table II sums up the approximate time calculated to heat Aroclor 1254 in an 8,000 gallon car.

TABLE II

Nine HP Boiler

| | Lbs. Steam/Hr. | 30-110°C. | 30-75°C. |
|---|---|---|---|
| 100% cap. (no reused condensate) | 263 | -- | 9 hrs. |
| 75% cap. (no reused condensate) | -- | 18 hrs. (86% cap) | 12 hrs. |
| 100% cap. (condensate @ 200°F.) | 296 | -- | 8 hrs. |
| 75% cap. (condensate @ 200°F.) | -- | 18 hrs. (76% cap) | 11 hrs. |

Calculations on a five horse power boiler give heating times of the following order:

Five HP Boiler

| | Lbs. Steam/Hr. | 30-110°C. | 30-75°C. |
|---|---|---|---|
| 100% cap. (no reused condensate) | 146 | 28 hrs. | 16 hrs. |
| 100% cap. (condensate @ 200°F.) | 164 | 25 hrs. | 14 hrs. |

Aroclor cars can be heated by steam (80-100 psig) to the proper handling temperatures in a reasonable time by using a boiler source capable of producing 200,000 to 300,000 Btu/hr. The times given here are approximate and will act as a guide until experience shows the exact time for this operation.

The proper handling temperature for the various fluids is given in the following Table III, which indicates corresponding viscosity values:

TABLE III

| Product | Handling and Pumping Temperature °C. | Approximate Viscosity, S.U.S. |
|---|---|---|
| Aroclor 1260 | 95 - 130 | 100 - 43 |
| Aroclor 1254 | 75 - 110 | 100 - 42 |
| Aroclor 1248 | 50 - 85 | 100 - 40 |
| Aroclor 1242 | 35 - 75 | 100 - 40 |
| Pyranol 1481 | 30 - 75 | 100 - 40 |
| Pyranol 1467 | 20 - 55* | 100 - 40 |
| Pyranol 1470 | 15 - 45* | 100 - 40 |
| Inerteen PPO | 20 - 55 | 100 - 40 |

*If any of the scavenger is out of solution, then the material must be heated at 70°C. (158°F.) until it has dissolved.

Selection of pumping temperatures for any dielectric not shown on this list or which may be developed in the future should be based on a viscosity of about 100 Saybolt Universal Seconds for average pumping and about 40 S.U.S. for fast pumping.

When the material has been heated to pumping temperature, a one-half inch diameter pipe "cross" arrangement containing a pressure gauge, air inlet, pressure relief valve to relieve at 30 Psig, and vent connections, should be connected to the dome air inlet pipe. Then the unloading line should be connected.

Rust free and clean galvanized piping or stainless steel pipe should be used for the unloading line.

(At this point a sample is taken as described in Chapter 4,) Dry* air is then introduced into the tankcar and pressure built up to 15 pounds gauge. The two inch valve cock on the stand pipe is opened and the discharge pipe observed to be sure the liquid is being unloaded. To protect the seams in the tankcar, the pressure must not exceed 30 pounds. The car will begin to unload at about 12 pounds pressure.

When the car is empty, the air pressure will drop off rapidly and air will blow out of the vent on the receiving tank. The air flow may be stopped at this point and the tankcar pressure released through the vent valve on the "cross" arrangement After inspecting the car to be sure that it has been completely unloaded, all connections and dome cover should be closed tightly. It is essential that the empty tankcar be sealed immediately after the car is unloaded in order to keep the car filled with dry air during return shipment.

*It is essential that the displacement air used for unloading be dried thoroughly by some dehumidfying unit such as soda lime, activated alumina or similar dehydrating agent drying unit. It may be necessary to recharge the dehumidifying unit each time that a car is unloaded. For unloading a tankcar of Aroclor within three hours, 15 standard cubic feet a minute of air at 15 pounds per square inch gauge pressure and a -100°F. dew point should be supplied. If the dry air unit is to be used only for unloading tankcars, a small single tower dryer unit containing a self-contained reactivating heater is suggested. Two manufacturers of air dryers of this type are: C. M. Kemp Mfg. Co., 405 E. Oliver Street, Baltimore 2, Maryland and Pittsburgh Lectrodryer Corporation, Foot of 32nd Street, Pittsburgh, Pennsylvania.

As a final step, it is desired that a standard railroad wire seal be inserted through the slotted bolts of the car fittings. Steam should be released from the car coils and all condensate removed from the coils by blowing with air with the steam trap by-passed. All connections must be replaced as received. Adequate care should be taken in preparing and sealing the car for return shipment.

Unloading with dry air as described is the preferred and recommended procedure because it is done with the car dome closed which avoids contamination.

Dry nitrogen may be used instead of dry air. If nitrogen is used it is essential to notify us (the supplier) so that we can take required safety precautions relative to replacing the nitrogen with air in the returned car prior to sending our men into it for cleaning.

If the car is unloaded by pumping out of the top, which required opening the dome, it is most desirable that the car be set inside of a building. If this cannot be done, then a canopy or roof should be provided over the car dome, and the unloading should be done when the weather is clear.

A clean centrifugal pump with minimum capacity of 40 gpm. is suggested. It will be necessary to prime the pump and only clean askarel should be used to do this. Another method for priming the pump is to use a Penberthy steam jet, No. 22A available from Penberthy Injector Co., 1242 Holden Ave., Detroit 2, Michigan. (Further pump detail is given on page 17).

C. Drum Packaging

Drum packaging is made with new and carefully inspected 55-gallon drums. These steel drums are lined with a specially selected baked phenolic coating. An example is NESCO No. 3 lining offered by the National Enameling and Stamping Company, Long Island, New York. Contents of the drums should not be heated by direct application of flame or strip heaters. Radiant heat from steam coils or hot air in a heated room is to be preferred. The screw plug in the drum head is fitted with a metal cap as a safe guard against tampering.

The drums should be stored indoors. If outdoor storage cannot be avoided, the drums should be placed in a horizontal position and covered with a tarpaulin.

## CHAPTER 2

## STORAGE TANKS

### A. General Description

The storage tanks should be a minimum of 10,000 gallons and preferably 12,000 to 15,000 gallons capacity to accommodate the normal 8,000 gallon tankcars.

It is preferable to locate the tanks above ground where they are easily accessible for any changes or repairs. Underground location presents difficulty in this respect.

Especially in cold climates, it is preferable to locate the tanks inside of a building. The tanks should be located conveniently with reference to the tankcar unloading facilities and the area where the dielectric is used.

Although Aroclors are non-corrosive to metals, corrosion or rusting of iron and steel equipment (by oxidation) may occur resulting in contamination of the products. The resistance of Aroclors to materials of construction is given in Monsanto Technical Bulletin, OP-115, entitled, "The Aroclors", Page 6.

Stainless steel tanks are very satisfactory but relatively expensive. Stainless steel pipe is relatively difficult to fabricate and it is difficult to make tight leak-proof connections.

Storage tanks may be of steel construction if properly metallized with zinc-tin or aluminum on the interior surfaces coming in contact with the Aroclors.

The metallizing should be done according to the following procedure:

1. Clean an area of the surface by sand blasting, or a similar method to give a perfectly clean and roughened surface. The area cleaned should not be greater than can be completely metallized within a few hours after cleaning.

2. If zinc-tin metallizing is used, a coating of zinc 0.005 inches thick should be sprayed on to the cleaned surface. This is followed immediately by a coating of tin 0.007 inches thick.

3. Aluminum metallized surfacing should be about 0.01 inch thick.

The detailed procedure for metallizing and cleaning is outlined as follows:

a) Sand blast. b) Coat with iron, 0.005 inches thick. (The purpose of this coating is to provide a rougher and better bond for the finish coat of aluminum or the zinc-tin combination.) c) Apply the selected finish coat. d) Fill the tank with tap water and warm it with steam. (If an open steam line is used, do not allow the steam to impinge directly onto the metallized surface of the tank.) e) Drain the tank. f) Fill with cold water and drain. g) Wipe dry and clean with clean diaper cloth or other fabric relatively free of lint. h) Heat the tank to at least 100°C. (212°F.) to expell moist air. It would be beneficial to heat the tank, allow it to cool and pull dry air through it using a dehumidfying breather in the air line, heat again etc., until the tank is full of comparatively dry air. i) Spray about 100 gallons of new, electrical grade Aroclor (not high in viscosity) or electrical grade trichlorobenzene onto the inner walls of the tank, washing the walls thoroughly (avoid breathing any fumes). j) Attach the circulating pump, the lines used, and

the filter press fitted with dry paper and circulate the fluid through the system and the tank. Install new dry filter paper several times in the press during this drying and cleaning operation. Discard the dielectric fluid used for cleaning.

k) Partially fill the tank with new Aroclor dielectric and analyze it electrically and chemically to determine whether it meets specifications. If all tests are met, then fill the tank with the dielectric.

The tanks should be insulated using, preferably, glass foam beads as supplied by Dow-Corning or Libby-Owens-Ford. The suggested thickness of the glass insulation is one inch minimum to two inches maximum. The glass insulation may be covered with tar material commonly used for weather-proofing. Another type of insulation which may be used instead of the glass is 85% Magnesia-Wool which should be covered also with the weather-proofing tar. The advantage of the glass insulation is that it is not moisture sensitive as is the case with Magnesia-Wool.

If the storage tank is located outdoors, it is best that the insulation be covered with riveted or bolted tin sheeting painted with aluminum paint. This type of metal surface weathers well and is cleaned easily. All piping must be galvanized and screwed fittings must be back brazed to assure tightness. All handling pipe lines must be traced with steam lines and insulation applied over the two lines in order to keep the handling lines and the material up to the desired pumping temperature. Usually a one-fourth inch copper steam line running parallel with the handling line will suffice.

Under very severe conditions of low temperatures, it would be desirable to wind the steam line around the handling line about two turns to the foot.

The tanks must be provided with ample pressurized heating coil surface to supply sufficient heat to the material to bring it to the proper temperature for pumping and handling as indicated in Table III. Heating coils should be either metallized steel, or preferably steel coils which have been galvanized after fabrication. It is recommended that the steam pressure on all heating coils should not exceed 100 pounds per square inch gauge; lower pressures may be used where practicable. It is essential that all steam coils be completely free from even minute leaks since this will introduce water into the product.

The steam coils may be introduced as "hair-pin" coils through a manhole at the side and bottom of the tank, or as is most often done, inserted through the manhole at the top of the tank and then located near the bottom.

External heating coils located in the jacket of the tank may be used but this construction is more expensive and less efficient than the internal coils.

The storage tanks may be fitted with a stirrer, either through the top, side, or bottom of the tank and the propeller blade should be located near the bottom of the tank. However, insertion of a stirrer through the side offers possible source of a leak and since these materials are homogeneous, it is not essential to provide such agitation for the purpose of mixing.

Adequate circulation can also be accomplished by using a centrifugal type pump. Gear pumps or other equipment where wear or chipping of metal parts may introduce contamination should not be used. The pumps must be of the type designed to handle hot oil. All wetted pump parts should be either stainless steel or bronze. The centrifugal pumps must be provided with a deep stuffing box and proper packing used, as described in Chapter 3. As examples of pumps found completely satisfactory for this service, reference is made to Worthington Worthite pumps. Blackmer pumps, Dean Bros., Peerless and Dayton Dowd Type C pumps for handling hot oil.

Also, a very satisfactory arrangement for mixing or circulating and pumping the fluid from the storage tank is to use a vertical sump pump such as a Taber pump.

All storage tanks must be amply provided with a dehumidifying breather such as soda lime, activated alumina, etc., units. This is essential to prevent moist air from coming in contact with the dielectric. A moisture content above 35 ppm adversely affects the electrical resistivity of these products. Provision should be made to preclude possible leakage of the drier material back into the storage tank and the drier should be inspected periodically to make sure that it is open and not plugged.

B. Detailed Description

Drawing No. 9C-8170 shows the detailed construction of a horizontal 15,000 gallon storage tank for Aroclor and its mixtures which has been found completely satisfactory.

The various nozzles on this tank are used as follows, considering them in order from left to right on the drawing:

| | |
|---|---|
| 3" nozzle | Inlet for recirculation |
| 24" nozzle | For future agitator if required (not used) |
| 3" nozzle | For soda lime or calcium chloride breather connection. |
| 36" manhole | For inspection, etc. The float guage is located in the center of this manhole. |
| 3" nozzle | Not used. |
| 24" nozzle | For future agitator if required (not used) |
| 3" nozzle | Not used. |
| 3" nozzle | Filling inlet connection. |
| 18" x 26" Oval nozzle | For sump pump |
| 3" nozzle | For thermometer well (see detail) |

The two 24" nozzles were originally installed for installation of agitators, if required. However, it has been found unnecessary to use agitators in the storage tanks, and these nozzles could be omitted.

It has been found that circulation of the fluid by the sump pump, and into the nozzle at the opposite end of the tank, for several hours gives satisfactory blending of the tank contents.

For pumping the dielectric from the storage tank, a Taber Pump Company all bronze 2-1/2" x 2" vertical sump pump with monel shaft has been found to be quite satisfactory for the application.

The liquid level gauge used in the storage must be gas-tight. The storage tanks are equipped with Vapor Recovery Systems Co.'s "Vareo", gas-tight, automatic tank gauge as shown by drawing No. 9C-8278.

The storage tanks should be provided with an operating platform suitable to the customer's conditions of operation.

The dehumidifying units used as breathers on the storage tanks can be constructed as shown by Drawing No. 9C-8248. The upper portion of the chamber is charged with anhydrous soda lime or another drying agent. Periodic inspection of the drying agent will show the formation of a cake of damp material on top about 1 to 1-1/2 inches deep. This cake should be removed and fresh material recharged. Any suitable construction similar to that shown on Drawing No. 9C-8248 may be used for the breather units.

Drawing No. D-13362 shows design detail of a 15,000 gallon vertical storage tank. The vertical type tank would seem especially desirable when insufficient space is available to accomodate the horizontal type tank.

## CHAPTER 3

## GASKETING AND PUMP PACKING

Aroclors and their mixtures soften and swell natural rubber and many of the synthetic "rubber" materials. Such material not recommended for use include, Hycar P, Koroseal, Perbunan, Neoprene, etc. These materials are known sources of contamination.

SUGGESTED TYPES OF PACKING AND GASKETING MATERIALS INCLUDE:

1. For Welded Flanged Pipe Connections: Garlock Packing Co., No. 901 or No. 7021, 1/8 inch asbestos fiber sheet. A ring of thin aluminum drawn tightly at the flange connections may be used satisfactorily also.

2. For Pumps: Garlock No. 234, No. 431, and Cheveron No. 7050-C are satisfactory packings. Likewise, Durametallic's spiral asbestos fiber may be used. Johns-Manville and others have comparable packing materials.

3. For Valves: Garlock No. 117 braided packing or its equivalent is suggested.

4. Other Resistant Materials: It is indicated that duPont's Teflon, poly tetrafluoroethylene is not attacked by hot (130°C.) Aroclor and is to be recommended as a gasket material. Dow-Corning's Silastic, Silicone 180, is very resistant to Aroclor and is suggested for gasket purposes.

5. In some cases cork impregnated under pressure with Chrysler's Cycloweld 55-9 or Armstrong Cork Co.'s 1162-J and cured at 170°C. may be used as a gasket material. These are baked phenolic type coatings.

6. Pipe Thread Compounds: When necessary to use pipe thread compounds, the following should be satisfactory if care is taken to prevent the pipe compound from getting on the inside of the pipe.

   (a) Plastic Lead Seal - manufactured by Durametallic Corporation

   (b) Ordinary white lead

Usually it is not necessary to use pipe thread compounds since all screwed pipe fittings should be sealed by back brazing.

7. All new lines and fittings should be cleaned thoroughly by steaming (for two hours) and dried with air or heat.

GASKETS FOR ASKAREL CAPACITORS AND TRANSFORMERS

1. For small capacitors requiring ring seals on the terminals, properly selected Silastic (silicone) tubing is cut to make the ring gasket.

2. The most effective and trouble free seal for transformer lids or covers is to weld the cover onto the transformer shell. To remove the welded cover a weld cutting tool or bar is used.

3. Cork - Nitrile rubber composition gaskets are sometimes used to combine the desired flow limiting property of cork with the resiliency of nitrile rubber. Only fine grained cork should be used in making this composition gasket. Special gasket cementing compounds such as GE's No. 1276 or No. 880 are used to coat the gaskets to further seal them against the transformer fluid and to accomplish firm bonding to the metal surface.

4. Nitrile rubber gaskets are also used and require no adhesive to make a liquid-tight seal. Exposure of nitrile rubber gaskets to transformer askarel should be kept at a minimum and the gasket should not be compressed beyond 2/3 of the original thickness.

   After long time exposure to transformer askarel fluid or its vapor, nitrile rubber is measurably deteriorated. While gaskets made of silicone or Teflon are not attacked, these materials are relatively expensive for use in large sizes. Accordingly, for the most efficient performance it is suggested that welded covers be used on askarel transformers.

5. Instruments, such as temperature gauges, etc. may be attached to the transformer using flange connection to pipe located below the liquid level of the fluid in the transformer. Such flange connections are usually not large in diameter. Accordingly, it seems practical to use Teflon or Silastic gaskets to make these seals, especially since it is known that askarel can migrate thru cork or composition cork gaskets used under the liquid level.

Another satisfactory approach used is to machine the flange surfaces of this type connection. Then a Spiritallic gasket made of stainless steel ring with asbestos inter liner for resiliency can be used satisfactorily.

6. Screwed pipe fittings on askarel transformers require thorough cleaning of the threads to remove oil, grease and dirt. Then the threads are coated with a compound such as GE's No. 880 and then tightened.

## CHAPTER 4

## SAMPLING METHODS

1.) The ASTM Standard Method for sampling electrical insulating oils is described in ASTM Designation: D923-49. This describes glass and metal thiefs for sampling drums, cans, and tankcars. A specially designed thief or bomb for sampling tankcars is described, also. A very good instrument of this type is the stainless-steel Bacon Bomb Thief with which samples of the liquid can be drawn from any level of the tankcar.

The ASTM procedure describes sample containers, their cleaning and storage.

Under general precautions, the ASTM mentions that, "Samples of the fluid shall not be taken until the oil is at least as warm as the surrounding air, because cold oil may condense enough moisture from a humid atmosphere to affect seriously its insulation properties. (In the case of tankcar lots, on some occasions there may be no choice, as it may be necessary to procure samples from a tankcar when the temperature is not above the surrounding air. On such occasions, the temperatu of oil and air also the humidity if possible, should be noted in the report of test results.) It is undesirable to do any sampling when the relative humidity of the atmosphere exceeds 75 percent, and samples shall never be taken in the rain".

Several electrical manufacturers using Aroclor dielectrics, switch the tankcars directly into the plant building or under roof before sampling and unloading.

These precautions in handling are taken to avoid any contamination of the fluids which are manufactured under very strict specifications. For example, the specification for ionizable chlorides allows no detectable amount, (less than 0.10 parts per million). Moisture may not exceed 20 to 35 parts per million.

Tankcars are cleaned and prepared under close inspection before they are filled. When filled, and analysis shows the material in the car to be satisfactory, the car is then sealed with a standard railroad wire and seal inserted through the slots of the dome fittings.

Likewise, after the tankcars have been unloaded in the industry, it is requested that the dome fittings should be sealed with a railroad wire and seal.

2.) Monsanto Methods used for sampling tankcars differ somewhat from the ASTM procedure. The modified techniques are used because of their greater simplicity and they have been entirely satisfactory as employed over many years.

A sample is never taken when it is raining or snowing, or when there is any chance of contaminated atmosphere moving in the direction of the car. However, in case of an emergency during inclement weather, a canopy is placed over

the car dome before sampling.

A satisfactory sample bottle is a five pint, round amber glass, packer type container fitted with a 38 millimeter Bakelite screw cap with an aluminum or tin cup liner. Bottles of this description can be purchased from the Northwestern Bottle Company, 3144 North Broadway, St. Louis, Missouri according to their No. A-7253.

Only new bottles and caps are used. When a shipment of bottles is received, the bottles are capped immediately and stored in their receiving cartons. Prior to use, the exterior of the bottles is wiped with a clean cloth.

The simplest sampling device used is a clean stainless steel or aluminum dipper. However, this is not a generally preferred device because it permits sampling the car from the near surface, only.

The sampling device commonly used consists of a stainless steel tube, seven feet long with one end bent into a hook, to serve as a handle and on the other end there is a stainless steel bucket with a perforated bottom. This bucket is about 5-1/4 inches inside diameter and 6 inches deep to hold the five pint bottle.

This bottle is held firmly by a stainless steel collar made to slide along the shaft of the sampling device. This collar has a clamp attachment for fixing it tightly into place where desired around the neck of the bottle.

When a car is to be sampled, a new sample bottle is clamped firmly in the bucket.

All dirt is brushed and wiped away from the car dome area using a clean rag.

The car dome is opened and the cap is then removed from the sample bottle.

The sampling device is inserted into the car so that the neck of the bottle is at least twelve to eighteen inches below the surface of the fluid. The sample taken is discarded as its purpose is to rinse the bottle. A portion of the sample is used to rinse the interior of the bottle cap.

This procedure is repeated until a minimum of three rinses has been made; each time the sample taken is not put back into the car. These rinses should be discarded.

Then the sample is taken and the cap of the bottle is screwed down tightly.

When the sample has been obtained, the car dome is replaced. immediately.

The exterior of the sample bottle is wiped with a clean cloth and when returned to the laboratory it is further cleaned with a cloth dampened with pure trichlorobenzene. If the sample is to be shipped, the cap is taped with Scotch Tape.

The sampling device is also cleaned with pure trichlorobenzene and is stored in a dust free, air conditioned room.

3.) Drum Sampling: A glass thief, thoroughly cleaned with pure trichlorobenzene and dried is used to sample drums.

## CHAPTER 5

## LABORATORY ANALYSIS AND PROCEDURE FOR TREATING AROCLORS AND THEIR MIXTURES WITH EARTH

A sample of the Aroclor or Aroclor mixture taken from the tankcar, or drums, as described in Chapter 4, is analyzed in the laboratory to determine its quality in accordance with the property values given in Chapter 7.

For capacitor use, usually the important properties tested are resistivity, power factor, chlorides, and moisture. For transformer use dielectric strength, resistivity, moisture and chlorides are the important properties.

If the sample is out of line with the shipping specifications, it is indicated that the sample has become contaminated. In this case, another sample is to be taken and the properties redetermined. If still out of specifications, the sample should be given treatment with earth.

Treatment of the dielectric with conditioned Attapulgus earth will bring the electrical properties to the maximum attainable values. While there is complete agreement on the benefits derived from treating with conditioned earth, there is difference of opinion on details of the method, arising from factors such as the following. No doubt, there are differences in the absorbent power of various types of diatomaceous earth with respect to removing moisture, impurities and additives such as stabilizers or scavengers from the dielectric

materials. The size of the earth particles, temperature and conditions of activation of the earth, the concentrations used and the temperature, the degree of agitation and time interval at which the dielectric is given earth treatment are all possible variables which are still being studied in various laboratories.

The method used by Monsanto for treating the fluid with activated earth in the laboratory is:

The absorbent is minus 200 mesh Attapulgus* earth activated just prior to use by heating in shallow trays for four hours at 400°C. (752°F.) or for at least twelve hours at 250°C. (482°F.).

At least one quart of the dielectric sample is placed into a clean two liter Pyrex beaker or three necked flask. The beaker or flask should be cleaned just prior to use in a manner similar to the procedure described in Chapter 6, Method No. 11,751, "Procedure for Cleaning of Electrodes, G.E. Cell and Accessories".

The flask or beaker is fitted with a glass or stainless steel agitator. Heat is applied using either a hot plate or a Glas-Col mantle and is controlled by a thermostat such as a Fenwal thermo switch with a stainless steel sheath.

About 0.1 to 0.2% of the activated earth, based on the weight of the liquid is added.

*Attapulgus Division, Minerals & Chemicals Corp. of America, 210 West Washington Square, Philadelphia 5, Pennsylvania

The more viscous dielectrics such as Aroclors 1248 and 1254 are heated at about 70° to 80°C. (158° - 176°F.) and the less viscous materials such as Aroclor 1242 and Pyranols 1481, 1467, and 1470 are heated at about 50° to 60°C. (122° to 140°F.).

After heating and stirring the sample for about four hours, it is filtered using a clean Pyrex glass suction flask and a Buchner funnel fitted with a Whatman No. 1 or No. 3 filter paper. This apparatus and the bottle into which the treated sample of dielectric is transferred should have been cleaned in a manner similar to the cleaning procedure described in Chapter 6.

The earth treated and "up-graded" sample is then ready for final analysis of its electrical properties.

## CHAPTER 6

## TEST PROCEDURES

### A. General Information

The Monsanto test methods described here with the special equipment used are some of the control tests employed to maintain the quality of Aroclors for dielectric use. They are suggested as a guide for test work needed to indicate the quality of the dielectrics used in the manufacture of electrical goods.

The most significant electrical tests made on Aroclors for capacitors are:

1. Dielectric constant.
2. Power Factor.
3. Resistivity.

For transformer use the most significant electrical tests of Aroclor mixtures are:

1. Dielectric Strength
2. Resistivity.

Other than electrical tests, significant measurements of quality include, moisture, chlorides, thermal and chemical stability.

The following terms are defined:

Dielectric Constant:

The dielectric constant (sometimes called specific inductive capacity) of any substance is equal to the ratio of the capacitance of a condenser when that substance is used as the dielectr: to the capacitance when there is a vacuum between the conductors (for all practical purposes air at ordinary pressures may be used instead of a vacuum).

Dielectric Strength:

Dielectric strength is the rupturing strength of an insulating material when subjected to voltage stress under specific conditions and expressed in kilovolts. Breakdown varies with the shape of the electrodes and does not increase directly in proportion to the thickness of the dielectric.

Power Factor:

The power factor of a dielectric is the ratio of the energy loss in the dielectric to the "apparent power" in the dielectric.

Resistivity:

Resistivity is electrical resistance offered to the passage of a steady current. The volume resistivity in ohms-centimeter of an oil is the ratio of the d-c potential gradient in volts per centimeter paralleling the current flow within the sample, to the current density in amperes per square centimeter at a given instant of time and under prescribed conditions. Volume resistivity is expressed in ohm-cm.

The analytical procedures described in detail include:

1. METHOD NO. 11,751, "PROCEDURE FOR CLEANING OF ELECTRODES, G.E. CELL AND ACCESSORIES."
2. METHOD NO. 11,608, "DIELECTRIC CONSTANT AND POWER FACTOR."
3. METHOD NO. 11,605, "DIELECTRIC STRENGTH."
4. METHOD NO. 11,607, "RESISTIVITY."
5. METHOD NO. 10,126, "CORROSION AND CHEMICAL STABILITY."
6. METHOD NO. 10,118, "INORGANIC CHLORIDES."
7. METHOD NO. 10,087, "ACID NUMBER."
8. METHOD (MODIFIED) NO. 10,620, "MOISTURE (WATER)."

9. Hydrolysis Stability Test.

10. Thermal Stability Test.

B. Detailed Instruction and Testing Methods.

1. METHOD NO. 11,751, "PROCEDURE FOR CLEANING OF ELECTRODES, G.E."

Cell and Accessories.

a. The Electrode Cleaning Procedure:

1) Place the electrodes in hot electrical grade Trichlorobenzene for ten minutes.

2) Wash with unheated TCB.

3) Rinse twice with methanol and twice with tap water.

4) Place the electrodes in hot 10% Tri Sodium Phosphate solution. Soak and heat for ten minutes.

5) Wash thoroughly with tap water.

CAUTION: After Step 5 -- DO NOT TOUCH THE ELECTRODES WITH HANDS!

6) Wash with distilled water twice.

7) Dry in drying oven for at least two hours at 120°C.

b. The G.E. Cell Cleaning:

1) Reclean the cell before use, when more than 8 hours have elapsed since the previous cleaning.

2) Follow the procedure for the electrodes starting at Step 4.

c. Cleaning of the Accessories:

1) Apply the same cleaning procedure as given for the electrodes (Steps 1 to 7) to prepare the glass spacer and beaker for next test.

2) Clean the thermometer in the same manner as the electrodes, except for Step 7.

3) Place the wet thermometer (after Step 6) directly in position in the temperature Heating Unit (Modified Fisher Isotemp Oven) and allow to dry.

2. METHOD NO. 11,608. "DIELECTRIC CONSTANT AND POWER FACTOR."

I. Apparatus

A. Oscilloscope: Heathkit Model O-6.

B. Constant Temperature Heating Unit: Fisher Isotemp oven. Model 13-245A, modified to include inter-wall connectors.

C. A. C. Generator: General Radio Type 1302-A

D. Amplifier and Null Detector: General Radio type 1231-B with type 1261-A power supply.

E. Capacitance Bridge: General Radio Co. Capacitance Bridge type 716-C.

F. Test cells: G. E. type, concentric cylinder electrodes Catalog #1,559,663. (G.E., Pittsfield, Mass. Transformer Lab.)

G. Class B driver transformer: This is used for 60 cycle measurements to excite the bridge directly from the domestic power line. It has 50 volts output with a 4800 ohm resistor in service.

H. Tuned Circuit Filters: General Radio Type 1231-P2 (400 and 1000 cycle) and 1231-P3 (60 cycle). These filters aid in obtaining a more accurate frequency for the measurements by removing harmonics, noise, hum, etc.

II. Adjustment of Controls on Electrical Appartus

A. On Panel No. 1 (Top Panel, Amplifier and Null Detector)

a. Turn the 4-way (main power) switch on the upper right hand side to the #3 position to determine the Dielectric Constant at 1000 cycles. Turn this switch to the #2 position for measurements at 60 cycles.

b. Allow the equipment to warm up 10 minutes.

c. Turn "GAIN CONTROL" to 6.

d. Depress "INPUT 0.03V." button.

B. On Panel No. 2 (Oscilloscope)

a. Turn "INTEN." to about the 12 o'clock position.

CAUTION: Do not allow a high intensity spot to remain stationary on the screen for any length of time.

b. Using "HOR. POSITION" and "VERT. POSITION" controls center the image on the screen.

c. Adjust "FOCUS" for sharp line.

d. Turn "FREQ. SELECTOR" to LOOKC.

e. Turn "FREQ. VERNIER" to 80.

f. Turn "VERTICAL GAIN" to 5.

g. Turn "VERTICAL INPUT" to "10 VOLT MAX.".

h. Turn "HORIZONTAL GAIN" to about 20.

i. Turn "SYNCHORONIZING" to ± 20.

j. Turn "SYN." to "EXT. SYN."

k. Turn "GEN." to "SWEEP GEN."

C. On Panel No. 3 (Capacitance Bridge)

a. Turn "RANGE SELECTOR" switch to "100 C" for 60 cycle measurements and to "1 KC" for 1000 cycle measurements.

b. Turn "METHOD SWITCH" to direct.

c. Turn "DISSIPATION FACTOR" selector switch to "0".

D. On Panel No. 4 (oscillator)

a. Disregard this panel for measurements at 60 cycles.

b. On 1000 cycle measurements, depress the No. 10 "MULTIPLY BY" button.

c. Set "FREQUENCY DIAL" to 100.

d. Turn "OUTPUT" dial so that pointer is at the end of the arrow.

e. Depress the "UNBAL. 5000 OHMS" button.

When all of the above adjustments are made, the electrical apparatus is ready for measurement of Dielectric Constant and Power Factor.

III. Determination of Constants for the Apparatus

1. Carefully assemble the cell which has been cleaned and dried within the last 8 hours. Refer to Method No. 11,751 for the procedure to use in cleaning the cells.

2. Place the cell assembly in the Fisher oven which has been adjusted to 25°C.

3. Connect the back wire inside the oven to the lead on the inner cylinder of the cell and the front wire to the lead on the outer cylinder of the cell.

4. Connect the cable from the capacitance bridge to the terminals on top of the oven so that the inner wire of the cable goes to the back terminal and the outside mesh casing of the cable (the ground) goes to the front terminal.

5. Remove the thermometer from the top of the oven before going on with the test. This is important.

6. Make all adjustments on the electrical apparatus as directed in Part I of this method.

7. Balance the bridge by rotating the "CAPACITANCE" and "DISSIPATION FACTOR" dials on Panel No. 3 until the wide vertical band on the oscilliscope is adjusted to a minimum width.

8. Record the sum of the readings on the "CAPACITANCE" dial and vernier and call this value A.

9. Remove the beaker containing the cell from the oven and fill it with C.P. benzene to a level 0.737 inches (ca. 3/4 inch) above the top of the concentric cylinders of the cell.

10. Adjust the temperature of the benzene to 25°C. while stirring with a thermometer.

11. Replace the cell in the oven (at 25°C.) and make the same electrical connections as in Steps 3 and 4. DO NOT interchange connections.

12. Balance the bridge again as in Step 7.

13. Record the sum of the readings on the "CAPACITANCE" dial and vernier and call this value B.

14. Calculate the cell constant by the following equation:

$$\text{Cell Constant, } K = \frac{B}{2.27 - 1.0} - A$$ (this is usually around 70 mmfd.)

15. Remove the cell from the oven and balance the bridge as in Step 7 with the "CAPACITANCE" and "DISSIPATION" "FACTOR" dials.

16. Record the sum of the readings on the "CAPACITANCE" dial and vernier and call this value F. (capacitance of connecting cable.)

17. Calculate the CELL LEAD CAPACITANCE by the following equation.

CELL LEAD CAPACITANCE, G = A - F - K (this is usually around 3 mmfd.)

WHERE:

A = CAPACITANCE OF ENTIRE SYSTEM IN AIR (SYSTEM CONSTANT)

G = CAPACITANCE OF THE CELL LEADS (CELL LEAD CONSTANT)

F = CAPACITANCE OF CABLE AND WIRES WHICH CONNECT THE CELL AND CELL LEADS TO THE BRIDGE. (CONNECTOR CONSTANT)

K = CAPACITANCE OF THE CELL ALONE (THE CELL CONSTANT)

Tabulate the system Constant (A), the Cell Lead Constant (G), the Connector Constant (F), and the Cell Constant (K) on a piece of stiff paper and post them near the instrument where they can be easily referred to for comparison and calculations.

These constants must be checked at least once every three months and in all cases where the Dielectric Constant and/or Power Factor are out of specification.

## IV. Measurement of Dielectric Constant and Power Factor on Aroclors, Pyranols, Inerteens, and Tri-Tetrachlorobenzene Blends.

### A. Test Run on Cell to Determine whether it is Clean and properly Aligned.

1. Carefully assemble a cell which has been cleaned and dried within the past 8 hours.

NOTE: Refer to method No. 11,751 for procedure to use in cleaning cells.

2. Adjust the oven control to hold at a temperature of 100°C. for all materials except Tri-Tetra Blends. If a Tri-Tetra blend is to be tested, adjust the oven to hold a temperature of 25°C.

3. Place the empty cell assembly in the oven and connect the back wire inside the oven to the lead on the inner cylinder of the cell, and connect the other (front) wire to the lead on the outer cylinder.

4. Connect the cables from the capacitance bridge to the terminals on top of the oven so that the inner wire of the cable goes to the back terminal and the outside metal casing (ground) goes to the front terminal.

5. Allow 15 minutes for the cell to reach temperature equilibrium inside the oven.

6. Remove the thermometer from the top of the oven before taking any measurements on the bridge. This is important.

7. Make all the adjustments on the electrical apparatus as directed in Part I of this method.

8. Balance the bridge by rotating the "CAPACITANCE" and "DISSIPATION FACTOR" dials on Panel No. 3 until the wide vertical band on the oscilloscope screen is adjusted to a minimum width.

9. Record the sum of the readings on the "CAPACITANCE" dial and vernier and compare this value with the SYSTEM CONSTANT determined in Part II of this method.

IMPORTANT: If the value obtained in Step 9 does not agree with the System Constant A (Part II) within 5 uuf, the cell must be re-cleaned, re-dried, re-assembled, and the test run for the System Constant must be repeated.

NOTE: Although the above test run must be made prior to each analysis, the value obtained in Step 9 is not to be used in calculations but is to be used only as a check on the cleanliness and alignment of the cell.

B. Procedure for Testing Materials

10. Remove the cell from the oven and fill the beaker with the material to be tested to a level 0.737 inches (ca 3/4 inch) above the cylinders of the cell.

11. Adjust the temperature of the sample to 100°C. (use hot plate) for all materials except Tri-Tetra blends. For Tri-Tetra blends, adjust the temperature of the sample to 25°C. using an ice-water bath if necessary.

NOTE: Stir sample continuously with a thermometer while adjusting the temperature.

12. Place the cell and sample in the oven and make the same connections from the cell to the bridge as in Steps 3 and 4. DO NOT interchange connections.

13. Allow 15 minutes for the cell to reach temperature equilibrium inside the oven.

14. Remove thermometer from the oven before taking a measurement. This is important.

15. Make the adjustment of controls on the electrical apparatus as directed in Part I of this method.

16. Balance the bridge by rotating the "CAPACITANCE" and "DISSIPATION FACTOR" dials on Panel No. 3 until the wide vertical band on the oscilloscope screen is adjusted to a minimum width.

17. Record the sum of the readings on the "CAPACITANCE" dial and vernier, and call this value X.

18. Record the sum of the readings on the "DISSIPATION FACTOR" dial and switch. Call this value D.

Calculations:

$$\text{Dielectric Constant} = \frac{X - F - G}{K}$$

Where:

X = Capacitance reading from Step 17.
F = Connector Constant (Determined in Part II)
G = Cell Lead Constant (Determined in Part II)
K = Cell Constant (Determined in Part II)

$$\% \text{ Power Factor} = \frac{f \times D}{f_o}$$

Where:

$f$ = Test Frequency (60 cycles or 1000 cycles)
$f_o$ = Frequency of "Range Selector" on Panel No.3
$D$ = Dissipation Factor reading from Step 18.

NOTE: When D (dissipation factor) is less than 0.1, the dissipation and power factors differ by less than 0.0005. Therefore, for our measurements, power factors and dissipation factors are equal.

Precision: (Reference: General Radio Manual for Model 716-C Capacitance Bridge)

a. Capacitance readings are precise to ± 2 mmfd. x multiplier reading (± 0.2% of full scale for each range) when the dissipation factor is less than 0.01.

b. Dissipation Factor (Power Factor) readings are precise to ± 0.0005 or ± 2% of the dial reading whichever is larger, for values less than 0.1 for D (Dissipation Factor).

3. METHOD NO. 11,605, "DIELECTRIC STRENGTH."

a. Apparatus and General Information

The electrical equipment necessary to provide high voltage to permit the determination of dielectric strength of liquid dielectric at commercial power frequencies is basically quite simple.

The equipment assembled in the laboratory consists of a high voltage transformer of good design and with a current capacity of 2.43 KVA and with equipment for control of the voltage and a means of measuring the voltage and to provide safety for the operator.

It is enclosed in a steel gray crackle finished cabinet measuring 42" high, 22" wide, 17" deep and set on truck casters for easy mobility.

Protective equipment incorporated in this apparatus prevents the application of high voltage unless all safeguards are complied with. The door on rear of cabinet must be closed. The cover over the oil must be all the way down and the voltage control must be at 0 position. Failure to comply with these requirements will prevent any action when the red button is depressed.

The test cup: Transformers, Voltmeters, and Accessories

The askarel testing cup type No. 224809 supplied by General Electric Company is mounted on the top rear of the cabinet. It is protected by a heavy plastic cover, hinged at the rear for accessibility to the receptical.

It is equipped with safety contactor so placed that the circuit energizing the high voltage contactor cannot be completed unless the protective cover is completely lowered and in place. It is impossible for the operator or anyone else to touch the testing cup when high voltage is applied.

The High Voltage Transformer manufactured by the Kelly-Koett Manufacturing Co. is of the closed core, oil immersed, shell type design. Rate @ 81,000 volt @ 40 millampers. It was recovered from a used X-ray machine purchased quite inexpensively

An auto transformer from the same X-ray machine is connected so as to limit the out-put voltage of the high voltage secondary to 50,000 volts.

The primary of the auto transformer is connected to the secondary of a 2-1/2 KVA powerstat variable auto transformer supplied by the Superior Electric Company.

Power to the powerstat is controlled by a 4 contact 30 amp. solenoid circuit breaker.

The voltmeter mounted on top near front edge is connected across the powerstat secondary and is calibrated to read directly in Kilovolts in the range of 0 - 50 K.V.

The overload circuit breaker consists of a small relay connected between one side of the high voltage transformer secondary center tap and ground. It is adjusted to break contact on a current drain of about 50 milliampers. The circuit for the coil of the solenoid circuit breaker is wired through the contacts of this relay.

Safety and Operating Controls

1) Door interlock switch located on rear door.

2) Test cup cover interlock switch.

3) Powerstat switch mounted on rear of unit arranged so that high voltage contactor cannot be closed unless powerstat is at zero position.

4) Main power switch on front panel.

5) Powerstat voltage control on front panel.

6) High voltage contactor push button on front panel (red).

7) Signal lamps mounted on top around voltmeter. Purpose and operation described in method of use.

b. Procedure

1) Ascertain that the temperature of the material under test is 25 (± 0.5)°C.

NOTE: Testing at other temperature is likely to give variable results which may be misleading.

2) Shake the sample container so as to thoroughly mix the askarel before filling the test cup.

NOTE: This operation is especially important with used Aroclor as the impurities may settle to the bottom and the test may be misleading.

3) Rinse the testing cup three times with small portions of the sample to be tested.

4) Immediately after final rinse, fill the cup to a height of not less than 20 mm. (0.787 in.) above the top of the electrodes.

5) Rock the cup a few times in order that any entrapped air may escape. Close cover over oil test cup.

6) Allow to stand 3 minutes. CAUTION: THIS IS IMPORTANT.

7. Turn main toggle switch on front panel to "ON" (or up) position. Both green and amber pilot lights on the top at either side of the voltmeter will now glow.

NOTE: The green signal light is connected across the 115 volt in-put and denotes that line voltage has been applied to operating control circuit.

Amber light is connected in series with sensitive switch located under high voltage contactors and connected to its armature. It indicates that high voltage contactor is in its rest position and away from contacts energizing auto transformer.

8) Turn voltage control (large knob on front) to extreme counter-clockwise position.

9) Depress red button on front. This energizes high voltage transformer and circuit breaker and is indicated by amber light going out and the red light directly over voltmeter will light.

10) Watch the voltmeter and, while holding the button "IN", turn the voltage control at such speed that will cause voltage as indicated on voltmeter to rise at a rate of 3 K.V. per second.

11) Note and Record the voltmeter reading at breakdown.

12) Repeat the test until two successive breakdowns occur on each of two fillings of the test cup which do not differ by more than 10%.

Report the average value of these two readings (Step 12) as the Dielectric Strength. If the limit of the instrument is reached before breakdown, report the Dielectric Strength as 50 K.V. at 25°C.

Cleaning of the test cup:

After the test is completed, drain the cup.

Flush the cup with benzene.

Then fill with Aroclor 1248 and let stand,

until the next analysis.

NOTE: An exception, when samples of oil from the plant are brought in for test, the cup must be thoroughly cleaned with benzene and carbon tetrachloride before and after running the test

The electrodes:

The testing cup has two electrodes. Both electrodes are movable and have twenty threads to the inch with index notches on both the electrodes and the lock nuts.

To set the Gap:

Arrange one of the electrodes and the lock nuts with the index marks in line. Move the other electrode until it comes in firm contact with the first electrode and lock it.

Now unscrew the electrode with the index marks in line (Step 1) two complete turns and lock it.

This will leave a gap of 0.1 inch between faces of the electrodes.

Cleaning of the electrodes and the test cup free of carbon coating:

The following ASTM method of cleaning shall be followed when it is apparent from visual inspection that the electrode discs of the cup are coated with carbon.

Wipe clean with dry calendered tissue paper the electrodes and the test cup.

The instrument is equipped with a 115 volt AC power supply which supplies all operating voltages for the bridge indicating circuits and in addition supplies 500 V DC for application to the material under test. The instrument is completely enclosed in a waxed finish shielded oak cabinet measuring 8-1/2" wide, 22-1/2" long and 8" high.
Approximate weight--26 pounds.

Test Electrodes:

Two concentric nickel cylinders with feet, obtained from General Electric Company. The inner electrode has outside diameter of 2.8" and a height of 3.25" with area of 184 sq. cm. The outer electrode has an inside diameter of 3" and a height of 3.25" with area of 198 sq. cm. The distance between electrodes is, therefore, 0.1" or 0.254 cm.
By theory, electrode constant (K) area/length is 191/0.254 or 752 where average area is 191 sq. cm.
Also K = 36 x 10" x C (farads with air as dielectric) or 11.29 x C (mmfd. with air as dielectric).

Glass Plate:

Pyrex about 3-1/2" diameter with concentric grooves to assist in spacing electrodes. Obtained from General Electric Company.

Heating Unit:

Assembled in the laboratory and is the same unit

described in Dielectric Constant Apparatus (see Method No. 11,608; Equipment).

b. Procedure:

1) Assemble the test cell. Place the recently cleaned (within the last 8 hours--see Method No. 11,751, Step 9) electrodes in an 800 ml. beaker.

2) Measure the capacitance of the test cell ($C_c$) according to Method No. 11,608 (Dielectric Constant and Power Factor measurements.)

3) Fill the cell assembly until the liquid level is 3/4 inch above the top of the electrodes.

4) Heat the assembly on the hot plate to 100 ($\pm$ 0.5)°C.

5) Place the assembly inside of the testing oven.

6) Attach top lead (+) on the megohm bridge to inner electrode.

7) Attach other lead to outer electrode.

8) Throw the three switches at the top of the megohm bridge to "ON" position.

9) Allow 10 minutes for assembly to reach temperature equilibrium inside the oven.

DANGER: Make sure control knob is in "CHECK" position. Otherwise, painful shock will result if leads are touched.

10) Bring the galvanometer pointer to zero by turning the "ZERO ADJUST" knob in the direction in which the pointer of the galvanometer should move.

11) Turn the control knob to "CHARGE" position for 30 seconds.

12) Turn the control knob to "OPERATE" position and return the galvanometer pointer to zero by adjustment of the "MULTIPLY BY" switch and the megohm dial.

13) Read after 30 seconds.

Calculation:

Resistivity* - Megohm dial reading (Step 12) x "Multiply By" reading (Step 11) x capacitance of cell (Step 2) in mmfd. x 11.29 x 0.001.

Report the result in units of 109 ohm-cm.

*Values of resistivity are qualified by designation of temperature and voltage. These are for this test, 100°C and 500 volts DC.

NOTE: It is important that the product under test, electrodes, and beaker be at uniform temperature for this determination. Temperature variations in different parts of the sample will cause the galvanometer zero to change constantly and give misleading results.

CAUTION: Inasmuch as measurements must be made at a potential of 500 volts DC a shock hazard exists in the handling of this appartus. With the control knob in the charge and operate position full voltage of the bridge (500 volts) is applied to the positive and low terminals and through the test leads to the electrodes. Do not attempt to handle the electrodes of the test leads unless the control knob is in the "CHECK" position. Possible penalty for failure to observe this precaution -- Painful Shock.

5. METHOD NO. 10,126 "CORROSION AND CHEMICAL STABILITY."

   a. Apparatus: G.E. Corrosion Apparatus consists of the following:

      1) A Corrosion Flask - It is a 300-ml. Pyrex flask

with a ground glass 24/40 joint equipped with a 12-inch straight tube as an air cooled condenser. The air-condenser is painted on the outside with aluminum.

2) The Corrosion Apparatus: A transite box 32" long x 8" wide x 5" deep. The top of the box represents a split transite board with 5 holes cut to fit the flasks.

The box is heated by two 500-watt, 15 volt G.E. Strip heaters with off-set terminals at one end (23.5" overall length).

The heating length of the heating element is covered by a copper strip 19-1/2" long x 4" wide x 1/4" thick.

The temperature is controlled by an automatic thermostat with temperature setting indicator.

b. Procedure:

1) Roll a rectangular (2" x 4") piece of aluminum foil so that it will pass through a ground glass 24/40 joint of the corrosion test flask.

CAUTION: Be careful that after rolling the specimen does not touch itself at any point.

2) Wash the aluminum foil (Step 1) scrupulously with acetone, distilled water, acetone, benzene, and chloride-free ether.

3) Then place the foil on a clean watch-glass and dry in an oven at 110°C. for 30 min. After cleaning handle the specimen with tongs or forceps only.

4) Weigh accurately on an analytical balance the specimen (Step 3) at room temperature.

5) Drop the weighed aluminum foil into the chloride-free corrosion flask of the "G.E. Corrosion Apparatus." Rinse out flask with sample and rinse end of condenser with sample.

6) Add 200 ml. of the product under test to the aluminum foil (Step 4 and 5).

7) Set the corrosion flask in the corrosion test apparatus.

8) Attach a 12-inch straight-tube air-cooled condenser, the outside of which is painted with aluminum.

9) Cover the exposed part of the flask with aluminum foil.

10) Heat the flask for 6 (± 0.1) hours at 210 (± 5)°C.

The temperature of the liquid in the test flask is measured indirectly using a thermometer inserted through a cork stopper and into similar liquid contained in an identical flask seated adjacent to the test flask on the heating chamber.

11) At the end of the heating period, detach condenser from the flask before removing it from the hot plate.

12) Remove the flask from the hot plate and cover all of the flask with aluminum foil (when the flask is not on the hot plate.)

13) Without removing the aluminum foil covering of the flask, analyze the product (Step 6) remaining in the corrosion apparatus for:

a) Appearance, Color, and Condition.
b) Inorganic (Free) Chlorides--Apply Method No.10,118
c) Acidity (Acid Number) - Follow Method No. 10,087

14) With a pair of clean, straight nichrome tongs, remove the aluminum foil specimen (Step 5), wash thoroughly, dry and weigh accurately on an analytical balance in the same manner as before (Steps 2, 3 and 4).

Report the corrosion as loss or gain in weight to the nearest 0.0001 g. and the Chemical Stability, as indicated by the analysis of the products "After Corrosion Test", in the same way as reported for the original (as received) material.

6. METHOD NO. 10,118, "INORGANIC CHLORIDES."

a. Preparation of Standards:

1) Make a primary standard of 100.0 ppm by weighing 0.1648 g. C.P. NaCl into a chloride-free 1 liter volumetric flask. Dilute to the mark and mix thoroughly. Make a 10.0 ppm standard by diluting 100 ml. of the primary standard to 1 liter, and mixing well. For every 0.1 ppm standard, dilute to 10 ml. of the 10 ppm standard to one liter and

mix well. A 0.1 ppm beam is considered the very faintest beam perceptible to the eye between 15-45 seconds after adding the $AgNO_3$ solution. If the beam intensity is not visible at all, or if easily visible (too strong), discard the solutions and make new standards.

2) Weigh 20.0 g. C.P. $AgNO_3$ into a chloride free dark bottle. Add 20 ml. C.P. $HNO_3$ (chloride-free). Dilute to 200 ml. with water.

3) All solutions should be freshly prepared every two weeks and stored in glass-stoppered Pyrex bottles.

b. Light Source:

Employ the 2 battery Penlite flashlight, having a 3-4 mm. light aperture. New batteries must be used frequently in order to perceive beams properly.

c. Procedure:

1) Thoroughly rinse two separatory funnels with chloride-free water three or four times. Then take an aliquot from each funnel in a test tube which has been rinsed with chloride-free water. Test these aliquots for Tyndall beams by adding 3-5 drops of $AgNO_3$ and allowing 45 sec. for full beam to evolve. Absolutely no dust or chloride beam should be present. (If beam is present, rinse all equipment with 1:1 $HNO_3$ and repeat Step 1).

2) When funnels are beam-free, drain out all the water except 50 ml. in one and 25 ml. in the other. Heat the water in both funnels to boiling. (Hold stopper while heating as steam may cause stopper to fail.)

3) Transfer 50 ml. of the sample from the sample bottle at a temperature of 95-100°C. into the separatory funnel containing the 50 ml. of boiling water. (As a precautionary measure, pour some of the sample from the sample bottle into a waste beaker before adding the 50 ml. to the funnel.)

4) Stopper the funnel and shake vigorously for at least 1 minute, venting frequently through the stopcock. (Care must be exercised at all times to touch neither the lower part of the funnel stem nor the ground part of the stopcock.)

5) Allow the layers to separate and drain off the sample into the second funnel containing the 25 ml. of boiling water. (As before, drain off a few ml. of the sample into a waste beaker before draining the sample into the second separatory funnel.) It may be necessary to heat the sample when transferring the sample to the second funnel; e.g., Aroclor 1260.

6) Repeat step 4 and allow the layers to separate. Then drain off the sample into a waste beaker.

7) Combine both water extracts in one funnel and shake thoroughly.

8) Take approximately a 10 ml. aliquot of the water extract out through the bottom of the funnel into a 3/4" x 6" test tube. Again, first allow a few ml. to drain out before taking the aliquot. (The test tube used should be rinsed with chloride-free water several times before using.)

9) Add approximately an equal portion of chloride-free ether. (The ether is tested by shaking a portion of it with chloride-free water and testing for Tyndall beam at the end of 45 sec. If beam is present, wash ether several times with chloride-free water until washings show no beam after adding 3-5 drops $AgNO_3$).

10) Shake the ether-water mixture until the emulsion in the sample disappears and the water layer is completely beam free before adding $AgNO_3$. If emulsion is difficult to break, add sample dropwise through the ether and then shake.

11) Add 3-5 drops of 10% $AgNO_3$ solution and test for chloride beam for 45 sec. exactly. If no beam is present at the end of 45 seconds, report as $<0.1$ ppm. The very faintest of beams is considered 0.1 ppm. If beam is stronger it will be necessary to compare with standards of 0.15, 0.20 up to 1.0 ppm, adding the 3-5 drops of $AgNO_3$ and comparing at the end of 45 sec.

The method is precise to the nearest 0.1 ppm.

Report results to the nearest 0.1 ppm.

7. METHOD NO. 10,087, "ACID NUMBER."

a. Reagents:

1) Nitration grade benzol.

2) Anhydrous methanol.

3) A saturated solution of phenol red (phenol sulfonphthalein) in methanol (approx. 0.1%).

4) A 0.01 N solution of KOH in methanol.

b. Procedure:

1) Place 100 ml. of benzol, 100 ml. of methanol and 0.5 ml. (pipette) of phenol red indicator into one of two clean dry 500 ml. Erlenmeyer flasks.

2) Neutralize carefully with the 0.01 N KOH (to be first definite pink color.)

3) Pour the mixture back and forth between the two flasks several times. If the solution is still neutral, divide it equally between the two flasks, if not, repeat steps 2 and 3.

4) Weigh (± 0.05 g.) into one of the flasks a 75.0 ± 5.0 g. sample and titrate with the 0.01 N, KOH until the sample matches the blank.

c. Calculations:

$$\text{Acid No. (mg. KOH/gram sample)} = \frac{\text{ml. 0.01 N KOH} \times 0.56}{\text{Sample Weight}}$$

Report the results to the nearest 0.001 if they are below 0.1, otherwise to the nearest 0.01.

The method is precise to ± 0.002 for acid numbers below 0.01 and to ± 0.01 in the range of 0.1 to 0.01.

NOTE 1: To convert mg. KOH/gram to mg. NaOH/gram, multiply by 0.715.

8. METHOD MODIFIED NO. 10,620, "MOISTURE (WATER)".

a. Introductory Comment:

The Karl Fischer Reagent titration method used in the analytical laboratory involves use of an analytical balance to weigh accurately about one drop of water used in preparing the standard. Since an analytical balance may not be available, the method has been modified and uses a purchased standard water solution as described below. Also, in the laboratory a "Dead Stop" potentiometric method for determining

the end point is often used. However, as this equipment may not be available, the procedure described below uses the visual indicator change for determining the end point.

b. Apparatus and Reagents:

1) Karl Fischer Burett, Automatic Pyrex No. 5750, 25 ml. capacity. Ace Glass Company, Vineland, New Jersey.

2) Water Standard in Methanol. No. SO-W-2 (1 ml. = 1 mg. $H_2O$) Fisher Scientific Company, 2800 Jefferson Ave., St. Louis, Missouri.

3) Karl Fischer Reagent Solution No. SO-K-2, Fisher Scientific Company.

c. Standardization of Karl Fischer Reagent

Into a 500 ml. clean, dry Erlenmeyer flask, place about 100 ml. "Anhydrous" methanol (commercially available, 99.95%). Add Karl Fischer reagent to this blank until the first color change from lemon yellow. It is not necessary to read the burette at this point. Carefully pipette 50 ml. of standard water solution into the blanked methanol. Refill the Karl Fischer burette. Titrate the solution with gentle swirling to mix, until the same color is obtained as was obtained for the blank. Now read the burette.

Moisture value of K.F. reagent in terms of grams $H_2O$ per ml. =

$$\frac{(\text{Ml. Standard } H_2O \text{ solution}) \times (\text{Moisture value of standard water solution in gm. per ml. stated on label.})}{\text{ml. Karl Fischer Reagent}}$$

d. Solvent Mixture:

Since the solubility of the different askarels varies, the following solvent mixtures are suggested:

| Material | Anhydrous Benzene | Anhydrous Methanol |
|---|---|---|
| Pyranol 1478 | 0 ml. | 300 ml. |
| Pyranol 1488 | 100 ml. | 200 ml. |
| Pyranol 1467 | 100 ml. | 200 ml. |
| Pyranol 1481 | 100 ml. | 200 ml. |
| Pyranol 1495 | 100 ml. | 200 ml. |
| All Aroclors | 110 ml. | 190 ml. |
| Inerteen PPO | 100 ml. | 200 ml. |

e. Procedure, "Visual End Point."

1) Place 100-300 ml. of dry solvent mixture (c) in a dry 500 ml. ground glass-stoppered Erlenmeyer flask.

2) Titrate the solvent with K.F. reagent to the visual endpoint, i.e., the first change from the yellow to reddish orange that persists for 30 seconds. Refill the burette.

3) Using a beam balance, weigh to the nearest 0.1 gram by difference, a sample containing 0.03 to 0.06 grams $H_2O$ into the flask.

4) Stopper and shake until the sample is in solution.

5) Titrate the solution with K.F. reagent to the endpoint described in Step 2. Record the volume of K.F. reagent used.

Calculation:

$$\% H_2O = \frac{\text{ml. of K.F. reagent} \times H_2O \text{ factor} \times 100}{\text{Sample Weight}}$$

References:
Mitchell, J. and Smith, D.M., Chemical Analysis, Vol. 5, Aquametry, Interscience Publishers, Inc., New York, (1948)

## 9. "HYDROLYSIS STABILITY TEST FOR ARCOLOR"

### Purpose

To quantitatively determine the presence of unstable chlorine compounds in chlorinated biphenyls (askarels).

### Principle

The method is based upon the hydrolysis of unstable chlorine compounds in askarels by methanolic sodium hydroxide. The resulting chloride ion is determined potentiometrically by titration with silver nitrate solution in an essentially non-aqueous medium. The measured chloride ion, reported as parts per million in the askarel sample, is indicative of the relative stability of the askarel in a dielectric system.

### Reagents

1. Methanol (chloride free) - 5 liters of methanol are refluxed with 0.5g Analytical Reagent Grade $AgNO_3$ for 1/2 hour. The methanol is then distilled from the $AgNO_3$, discarding the first 100 ml. to flush the apparatus. 90% of the charge is distilled from the flask and the contents of the flask are discarded. The methanol should be checked to assure purity by titration. The chloride ion concentration should be less than 0.01 ml. of 0.005N $AgNO_3$ per 100 ml. of methanol.

2. Sodium Hydroxide Reagent - Analytical Reagent Grade NaOH (may be obtained from Mallinckrodt Chemical Company) is used to prepare this reagent. A 0.1N solution is prepared by dissolving 4.0 grams of analytical reagent grade NaOH in 1 liter of chloride free methanol.

3. Sulfuric acid - Prepared by a 50:50 volumetric dilution of Analytical Reagent Grade concentrated sulfuric acid (can be obtained from Mallinckrodt Chemical Company) with chloride free (deionized or distilled) water. The acid is always poured into the water with constant stirring to prevent any dangerous build-up of heat.

4. 0.005N $AgNO_3$ and 0.0025N $AgNO_3$ - Prepared by dilution of an ampoule of concentrated aqueous $AgNO_3$. These ampoules can be obtained from Anachemica Chemical Limited, Champlain, New York. This reagent may also be prepared by dissolving 0.8495 g. of Analytical Reagent Grade $AgNO_3$ crystals (may be obtained from Mallinckrodt Chemical Company) in one liter of chloride free water containing 3.0 ml. of concentrated nitric acid. This solution should be standardized against a pure chloride standard. A sodium chloride crystal such as used in infrared spectrometer cells is a good source of pure NaCl. The $AgNO_3$ solutions should be checked (at least monthly) to assure a consistent reagent.

5. Acetone (chloride free) - Prepared by distillation from $AgNO_3$ as described above, for methanol and should also be checked by potentiometric titration to assure optimum purity. Normally a chloride content of less than 0.01 ml. of 0.0025N $AgNO_3$ per 100 ml. is derived by this method.

6. Benzene - Analytical Reagent Grade benzene should be used. This material is normally chloride free but should be checked by potentiometric titration to be certain. Analytical Reagent Grade benzene may be obtained from Mallinckrodt Chemical Co.

Equipment

1. 200 ml. tall form beaker (Berzelius type).

2. Magnetic stirrer - A suitable magnetic stirrer with ring stand base can be obtained from Fisher Scientific Co. Cat. #14-511-1. This stirrer has a built-in rheostat and should be set at full speed and operated through a variac to adjust its speed. This will prevent heating of the stirrer during the stirring operation.

3. Teflon magnetic stirring bar - The bar should be cylindrical in shape and of one piece molded construction. one inch long may be obtained from Fisher Scientific Co., Cat. #9-311-9.

4. Microburet graduated in 0.01 ml. divisions - A suitable buret may be obtained from Scientific Glass Apparatus Co., Inc. Bloomfield, New Jersey Cat. #JM-570.

5. Silver electrode - The Beckman silver billet electrode Cat #39261 is the preferred type.

6. Glass electrode - A standard glass electrode such as Beckman electrode Cat. #40498.

7. pH meter suitable for use with glass electrode - A model GS Beckman pH meter can be used. This instrument has the expanded scale and provides greatest sensitivity to incremental emf changes. A somewhat less sensitive but, nonetheless, useable meter such as Beckman "Zeromatic" or the Leeds Northrup line operated pH meter can be used.

8. Water bath - An individual glass water bath 150mm in diameter 75mm high and containing 600 ml. of water heated to 40°C. $\pm$ 1°C. is used. This glass water bath can be obtained from Corning Glass Co., Corning, N.Y. Cat. #3140.

9. Usual laboratory glassware - 25 ml. pipette, buret or pipette graduated to deliver 0.5 ml., wash bottles for pure acetone methanol, water and a sturdy ringstand.

Procedure for 1242 Aroclor (1499 Pyranol)

1. Twenty five grams of askarel is weighed into a tared 200 ml. beaker to the nearest 0.01 gram on a suitable balance.

2. The magnetic stirring bar is then added to the beaker containing the sample (without the bar touching the operators' hands).

3. Twenty five ml. 0.1N NaOH (methanolic) is added by means of a 25 ml. pipette and the beaker is covered with a watch glass.

4. The sample beaker is immersed to a depth of 1 1/4 inches in the 40° ± 1°C. water bath on a magnetic stirrer and clamped securely to a firm support. The sample is stirred at as fast a speed as possible, without pronounced splashing, for 1 hour. The water bath is not heated. No effort is made to maintain the temperature at 40°C., and it will drift toward equilibrium with room temperature.

5. After the 1 hour stir, the sample beaker is removed from the bath, 0.5 ml. of dilute sulfuric acid is added to the sample by means of a suitable pipette or buret. 125 ml. of chloride free acetone is then added (a graduated cylinder is suitable for this purpose).

6. The sample is then titrated with 0.005N $AgNO_3$ solution using the silver-glass electrode system.

   Normal samples of askarel require extremely small amounts of $AgNO_3$, for this reason the titration is run using 0.01 ml. additions and allowing sufficient time for equilibrium to be established before recording the emf change. If a change of less than 1mv per 0.01 ml. addition is observed for 3 or 4 .01 ml. increments, larger additions of $AgNO_3$ may be used for instance .05 ml. until such a change is observed. The additions then are reduced to 0.01 ml. again to complete the titration. The endpoint normally is defined by two 50mv changes. A normal titration would yield the following typical data.

| MV | dMV* | ML | dML | dMV/dML X $10^{-2}$ |
|---|---|---|---|---|
| 400 | 0 | .06 | 0 | 0 |
| 392 | 8 | .07 | .01 | 8 |
| 352 | 8 | .08 | .01 | 8 |
| 341 | 11 | .09 | .01 | 11 |
| 321 | 20 | .10 | .01 | 20 |
| 271 | 50 | .11 | .01 | 50 |
| 221 | 50 | .12 | .01 | 50 |
| 201 | 20 | .13 | .01 | 20 |
| 285 | 16 | .14 | .01 | 16 |

* Using the GS pH meter the change is measured in 0.2mv units and hence the meter changes observed would be 5 times this value (i.e. 25 units for 5mv).

To calculate the change per 0.01 ml. observed, the mv change is divided by the volume of $AgNO_3$. By plotting dmv/dml vs. ml., the endpoint may be found to the nearest 0.001 ml. This gives a sensitivity of ± 0.007ppm - to define the endpoint to ± .01 ml. no plotting is necessary and a sensitivity of ± 0.07ppm is assumed.

7. A reagent blank is run exactly as above omitting the askarel sample.

Calculations

Subtract the reagent blank from the total volume of $AgNO_3$ and for the sample then:

$$\text{Reactive Chlorine (ppm)} = \frac{\text{Net Volume } AgNO_3 \times \text{Normality } AgNO_3 \times 35.46 \times 10^3}{\text{weight}}$$

Procedure for 1254 and 1260 Aroclors (more viscous askarels)

The procedure is followed exactly as above except that under Procedure, Step 2, 5 ml. of benzene is immediately added to the askarel sample and stirring bar. The sample is heated until it dissolves in the benzene and cooled to room temperature before proceeding to Step 3.

The benzene, of course, should be included in the reagent blank determination.

Procedure for Micro Test

The dechlorination test may also be run on 5 gram samples of askarels with a reduction in sensitivity. It is run exactly as the 25 gram test above except that the amount of reagents then used are 5.01 ml. NaOH (0.1N methanolic), 0.1 ml. $H_2SO_4$ for acidification, and 50 ml. of acetone to dilute the sample. 0.0025N $AgNO_3$ is used to titrate this size sample. The sensitivity is then ± 0.18ppm rather than ± 0.07ppm given by the 25 g sample (without plotting the endpoint).

General Comments

A rapid titration can be made to the nearest 0.1 ml. using the normal potential at the equivalence point or use can be made of an automatic titrator for routine control procedures. The sensitivity in either case should be within ± 0.7ppm of the value obtained by more refined techniques with 0.005N $AgNO_3$ and a 25 gram sample.

The usual analytical precautions should be exercised in using this test method to prevent cross contamination from other sources of halogen in the laboratory. This means that all glassware, apparatus, and the area in which this test is run should be analytically clean.

10. "THERMAL STABILITY METHOD FOR AROCLORS"

Scope

This method measures the thermal stability (chloride content) of chlorinated biphenyls used primarily as dielectrics. It is used for determining the quality of finished Aroclors.

Principle

Certain impurities if present in chlorinated biphenyls will break down at elevated temperatures with the liberation of HCl. The volatile HCl is swept out of the sample with air, absorbed in water and titrated with silver nitrate solution. The results are expressed as parts per million chloride obtained during a 16 hour thermal stability test period.

Reagents

1. Acetone. No special grade is required. It must contain no titratable chlorides.

2. 1% $HNO_3$. Dilute 1 ml. concentrated $HNO_3$ to 100 ml.

3. 0.005 N$AgNO_3$. 0.8495g to 1000 ml. 5 ml. 0.1 N$AgNO_3$ (if available) to 100 ml.

Apparatus

1. Pressure Regulator. Moore - Model 40 - 2 - 0-50" Water. Moore Products Co. H & Lycoming St., Philadelphia 24, Pennsylvania.

2. Thermoregulator. Cenco - 99015 - (Central Scientific Co.)

3. Relay. Ebert Microlay SPST Std. Type. Ebert Electronics Corp., Queens Village, N.Y. (Any sensitive, reliable relay can be used).

4. Stirring Motor. Bodine NSI-13 B-2224 1/40 HP. 1725 R.P.M.

5. Bath Fluid. Dow Corning 550. 5 gallons

6. Leeds & Northrup ac. operated pH Meter - Cat. No. 7664

7. Silver wire electrode

8. Mercurous Sulfate Reference Electrode. Modified L & N calomel reference electrode prepared as follows. Dismantle the internal element from the salt bridge tube of a standard L & N calomel reference electrode. Discard the saturated KCl solution from the tube and clean out the mercurous

chloride and mercury from the internal element. Clean parts thoroughly. Add sufficient new clean mercury to the internal element to make contact with electrode wire and repack chamber of internal element with mercurous sulfate moistened with 0.5M potassium sulfate. Seal the chamber with non-absorbant cotton. Fill the salt bridge tube with 0.5M potassium sulfate and reassemble units.

9. Burette. 1.0 ml. microburette - Koch - Fisher Scientific Co. - 20-110.

10. Magnetic Stirrer and glass covered stirring bar.

11. Glass Apparatus for Samples in Bath. See attached diagram.

12. Capillaries. Glass capillaries approximately 0.2 mm in diameter and cut to a length that permits a flow of 45 ml. per min. of air.

13. Variac. 2 KVA.

14. Air Supply. Air under 40 lb. pressure is available in our laboratories. This air is purified by passing through a scrubber bottle containing 40% NaOH, an empty bottle which serves as a safety, a second bottle containing conc. $H_2SO_4$ and a trap immersed in dry ice and acetone. The purified air is connected to a glass manifold having one connection for each sample. Capillaries of the appropriate length are connected between the manifold and the outlet for each sample. In this manner a constant flow of air can be obtained on all samples by applying a constant pressure to the manifold.

15. Heating Bath. A stainless steel bath constructed according to the specifications given in the attached diagram is used. The bath is heated by applying 85 Volts to 3-500 Watt G.E. strip heaters bolted to the bottom of the bath. One 500 Watt Immersion heater is connected to the thermostat. Dow Corning 550 silicone is used as the bath liquid. The temperature is maintained at 210° ± 0.2°C. The bath should be placed in hood and the tests carried out in total darkness. Two 3 X 5" stainless steel plates not shown in the sketch are placed on top of the straightening vanes in the bottom of the bath. This provides better stirring to the ends of the bath. Twelve samples can be run in the bath at one time.

Procedure

Weigh a 290 ± 1 g. sample of Aroclor into the 300 ml. Erlenmeyer flask. Insert the gas inlet tube and position the flask in the 210° ± 0.5°C. bath. The bath fluid level should be approximately one inch below the bottom of the ground glass joint on the flask. Place 10 ml. distilled water in the Volhard flask absorber and attach to the receiver tube from the Erlenmeyer flask. Connect the purified air supply from the capillary to the inlet down tube

in the flask. Bubble air through the sample for 16 hours at the rate of 35 to 45 ml. per minute. (The apparatus and/or sample must be kept in the dark during the 16 hour period). Transfer the water from the absorber to a 100 ml. beaker using approximately 50 ml. acetone, and 2 drops 1% $HNO_3$ solution and titrate with 0.005N $AgNO_3$ solution using a magnetic stirrer. The titration is stopped at 75 mv. which represents the point of maximum potential change and the titration endpoint. Silver wire and mercurous sulfate electrodes (Ag-Hg, $Hg_2SO_4$, 0.5M $K_2SO_4$ system) are used for the titration.

Calculations

(Total ml. 0.005 $NAgNO_3$ used) (0.61) = ppm chloride

1 ml. 0.005 $NAgNO_3$ is equivalent to 0.0001773g. chloride or 0.61 ppm.

Precision and Reliability

The precision of the test (standard deviation) is 0.02 ppm at the 0.5 ppm chloride level and 0.06 ppm at the 2.8 ppm level. The method as written does not necessarily quantitatively measure the total unstable chlorides present. For screening purposes a total chloride figure is not necessary. Experience has shown that there is good correlation between the chloride figures obtained by this 16 hour thermal stability test and the quality of chlorinated biphenyls.

Discussion

The air supply can be checked for chloride contamination by passing the air through an empty sample flask immersed in the bath. Not more than 0.03 ml. 0.005 $NAgNO_3$ should be required to give the endpoint. The air supply can be checked for ammonia by measuring the pH of the absorber solution or titrating with 0.01N HCl. The pH should be between 6 and 7. Compressed cylinder air available for breathing purposes can perhaps be used without any purification. Experience has shown that nitrogen gives low chloride figures. This indicates that air is a necessary part of this test and that nitrogen cannot be used as a substitute. Gum rubber tubing is used in making all connections. The apparatus is cleaned with acetone.

## CHAPTER 7

## TYPICAL PROPERTIES

The 1200 series members of the Aroclor family are chlorinated biphenyls, and are made by chlorinating biphenyl to approximately the percentage of chlorine, by weight, indicated by the last two digits of the serial number. For example, Aroclor 1254 is approximately 54% chlorine on a weight basis. Accordingly, these Aroclors are not single or simple compounds. They are a mixture of isomeric compounds composed predominately of the chemical compound indicated below as being their approximate equivalent:

| | |
|---|---|
| Aroclor 1242 | Trichlorobiphenyl |
| Aroclor 1248 | Tetrachlorobiphenyl |
| Aroclor 1254 | Pentachlorobiphenyl |
| Aroclor 1260 | Hexachlorobiphenyl |

For transformer use and some capacitor use where lower viscosity is required for better low temperature operation than offered by the above Aroclors, these products are mixed with pour point depressants, particularly trichlorobenzene resulting in various General Electric Company Pyranols described briefly as follows:

| | |
|---|---|
| Transformer Pyranol 1467* | 60% of Aroclor 1260<br>40% of Elec. Grade Trichlorobenzene<br>0.125% of Tin Tetraphenyl |
| Transformer Pyranol 1470* | 45% of Aroclor 1260<br>55% of Elec. Grade Trichloro-Tetrachlorobenzene Mixture<br>0.125% of Tin Tetraphenyl |

*Use of tin tetraphenyl scavenger is subject to G.E. patents and license: Royalty arrangements should be checked before using Pyranol 1467. Questions about license concerning the use of anthraquinone stabilizer for DC capacitors should be referred to Western Electric, 195 Broadway, N.Y.C.

| | |
|---|---|
| Transformer Inerteen PPO | 60% of Aroclor 1260<br>40% of Elec. Grade Trichlorobenzene<br>0.20% Phenoxypropene oxide |
| Capacitor Pyranol 1481 | 75% of Aroclor 1254<br>25% of Elec. Grade Trichlorobenzene |

Detailed properties of all of these products are given in the following property lists.

## AROCLOR 1232

| PROPERTY | TYPICAL |
|---|---|
| Visc. @ 37.8°C. (ASTM D88) | 44 - 51 |
| Specific Gravity @ 25/15.5°C. (ASTM D287) | 1.270 - 1.280 |
| Color, APHA | 50 Max. |
| Condition | Clear |
| Acidity, mg. KOH/g. | 0.014 Max. |
| Pour Point, °C. (ASTM D97) | -30 or lower |
| Inorganic Chlorides, ppm. | 0.10 max. |
| Refractive Index @ 25°C. | 1.6200 - 1.6220 |
| Distillation Range (ASTM D20) Corrected for stem and barometic pressure | 10% - 293°C. min.<br>50% - 310 - 320°C.<br>90% - 360°C. max. |
| Corrosion | After heating with aluminum for six hours at 210°C. ± 10°C. the aluminum must not be corroded either on visual or weight inspection and the Aroclor 1242 should meet the following specs: |
| | Color, APHA 100 max.<br>Acidity, mg.KOH/g. 0.014 max.<br>Inorg. Chlorides, ppm. 0.10 max.<br>Condition Clear |
| Water Content, ppm | 35 max. |
| Resistivity 100°C., 500 volts DC @ 0.1"gap | 500 x $10^9$ Ohm-cm., min. |
| Dielectric constant 100°C. @ 1000 cycles (ASTM D924) | 4.3 - 4.5 |
| Sulfates (ASTM-D117-31)* | None |
| Fixed Chlorine content (Carius)* | 31.5 - 32.5 |
| Dielectric Strength (KV) (ASTM D877)* | 35 min. |
| Hydrolysis Stability Test Chlorides, ppm. | 3.0 (tentative) max. |
| Thermal Stability Test Chlorides, ppm. | 0.5 (tentative) max. |

*Not determined unless by special request.

## AROCLOR 1242

| PROPERTY | TYPICAL |
|---|---|
| Visc. at 27.8°C. (ASTM D88) | 82 - 92 seconds Saybolt Univer. |
| Specific Gravity at 25/15.5°C. (ASTM D287) | 1.381 - 1.392 |
| Color, APHA | 50 max. |
| Condition | Clear |
| Acidity, mg. KOH/g. | 0.01 max. |
| Pour Pt., °C. (ASTM D97) | -14 or lower |
| Inorganic Chlorides, ppm. | No detectable amount |
| Refractive Index at 25° C. | 1.6240 - 1.6260 |
| Distillation Range (ASTM D20) Corrected for stem and barometric pressure | 10% 325°C. min.<br>90% 360°C. max. |
| Corrosion | After heating with aluminum for six hours at 210°C ± 10°C, the aluminum must not be corroded either on visual or weight inspection and the Aroclor 1242 should meet the following specs: |
| | Color, APHA 60 max.<br>Acidity, mg.KOH/g. 0.01 max.<br>Inorg.Chlorides,ppm no detectable amount<br>Condition Clear |
| Water Content, ppm | 35 max. |
| Resistivity 100°C. 500 volts DC at 0.1" gap | 500 x $10^9$ ohm-cm., min. |
| Dielectric Constant 100°C. at 1000 cycles (ASTM D924) | 4.7 - 4.9 |
| Flash Point Cleve. Open Cup* | 170 - 200°C. |
| Fire Point °C.* | None to boiling point |
| Sulfates (ASTM-D117-31)* | None |
| Fixed chlorine content (Carius)* | 43 ± 0.5% |
| Specific Heat at 25°C.* | 0.29 |
| Evaporation at 100°C for 6 hrs.* | 0.4% max. |
| Dielectric Strength (KV) (ASTM D877)* | 35 Min. |

*Not determined unless by special request.

| | |
|---|---|
| Hydrolysis Stability Test chlorides, ppm | 1.0 (tentative) max. |
| Thermal Stability Test chlorides, ppm | 0.40 (tentative) max. |

## AROCLOR 1248

| PROPERTY | TYPICAL |
|---|---|
| Visc. at 54.4°C. (ASTM D-88) | 73 80, Sec. Saybold Universal |
| Spec. Gravity at 65/15.5°C. (ASTM D-287) | 1.405 - 1.415 |
| Color, APHA | 100 Max. |
| Condition | Clear |
| Acidity, mg. KOH/g. | 0.01 max. |
| Pour Point °C. (ASTM D-97) | -7 |
| Refrac. Index at 20°C. | 1.6285 - 1.6305 |
| Dist. Range (ASTM D-20) | First drop 310°C. min.<br>10% - 345°C. Min.<br>90% - 385°C. Max. |
| Water Content, ppm. | 35 |
| Resist. 100°C. 500 v D.C. at 0.1" gap | 500 x $10^9$ Ohm-cm., min. |
| Dielectric constant, 100°C. 1000 cycle | 4.6 |
| Dielectric Strength 25°C.* | 35 KV min. |
| Flash point, (C.O.C.)* | 193°C. |
| Fixed Chlorine (Carius)* | 47.5 - 48.5% |
| Specific heat at 25°C.* | 0.27 |
| Inorganic chlorides, ppm. | 0.10 max. |

*Not determined unless by special request.

| | |
|---|---|
| Hydrolysis Stability Test chlorides, ppm. | 3.0 (tentative) max. |
| Thermal Stability Test chlorides, ppm. | 0.5 (tentative) max. |

## AROCLOR 1254

| PROPERTY | TYPICAL |
|---|---|
| Visc. at 98.9°C. (ASTM D88) | 44 - 48 sec. Saybolt Univer. |
| Specific Gravity at 65/15.5°C. (ASTM D287) | 1.495 - 1.505 |
| Color, APHA | 100 max. |
| Condition | Clear |
| Acidity, mg.KOH/g. | 0.01 max. |
| Pour Pt. °C. (ASTM D97) | 7 - 12 |
| Inorganic Chlorides, ppm. | No detectable amount |
| Refractive Index at 25°C. | 1.6370 - 1.6390 |
| Distillation Range (ASTM D20) Corrected for stem and Barometric Pressure | 10% 366 - 378°C.<br>50% 371 - 383°C.<br>90% 379 - 394°C. |
| Corrosion | After heating with aluminum for 6 hours at 210°C. plus or minus 10°C. the aluminum must not be corroded either on visual or weight inspection and the Aroclor 1254 should meet the following specs:<br>Color, APHA — 150 max.<br>Acidity, mg.KOH/g. — 0.01 max.<br>Free Chlorides, ppm. — No detectable amount<br>Condition — Clear |
| Water Content, ppm. | 35 max. |
| Resistivity 100°C., 500 v D.C. at 0.1" gap | $500 \times 10^9$ ohm-cm., min. |
| Dielectric Constant, 100°C. 1000 cycles | 4.15 - 4.35 |
| Dielectric Strength, 25°C.* | 35 KV, min. |
| Burn Point (ASTM D92)* | Higher than 350°C. |
| Sulfates (ASTM D-117-31)* | None |
| Fixed Chlorine Content (Carius)* | 55 + 0.5% |
| Evaporation at 100°C. for 6 hrs.* | 0.4% max. |
| Stability* | There shall be no liberation of chlorine or chlorides when the material is heated at 100°C. in glass vessels in contact with air for periods of at least one month. |
| Ageing Characteristics* | No loss in resistivity over original value on heating in air for 96 hrs. at 100°C. |
| Specific Heat at 25°C.* | 0.26 |

*Not determined unless by special request.

| | |
|---|---|
| Hydrolysis Stability Test Chlorides, ppm. | 3.0 (tentative)max. |
| Thermal Stability Test Chlorides, ppm. | 0.5 (tentative)max. |

## AROCLOR 1260

| PROPERTY | TYPICAL |
|---|---|
| Visc. at 98.9°C. (ASTM D88) | 72 - 78 Sec. Saybolt Univ. |
| Specific Gravity at 90°C./15.5°C. (ASTM D287) | 1.555 - 1.566 |
| Color, APHA | 150 max. |
| Condition | Clear |
| Acidity, mg.KOH/g. | 0.01 max. |
| Pour Pt., °C.(ASTM D97) | 25 - 34 |
| Inorganic chlorides, ppm. | No detectable amount |
| Refractive Index, 25°C. | 1.6455 - 1.6470 |
| Distillation Range (ASTM D20) Corrected for stem and barometric pressure. | 10% 385 - 398°C.<br>50% 390 - 404°C.<br>90% 400 - 420°C. |
| Corrosion | After heating with aluminum for 6 hrs. at 210°C. ± 10°C. the aluminum must not be corroded either on visual or weight inspection and the Aroclor 1260 should meet the following specs: |
| | Color, APHA — 150 max.<br>Free Chlorides,ppm. — No detectable amount.<br>Acidity,mg.KOH/g. — 0.01 max.<br>Condition — Clear |
| Water content, ppm. | 35 max. |
| Resistivity,100°C. 500 volts at 0.1" gap | 500 x $10^9$ ohm-cm., min. |
| Dielectric Strength 50°C.* | 30 KV., min. |
| Dielectric Strength 100°C.* | 30 KV., min. |
| Dielectric Constant 100°C. at 1000 cycles* | 3.6 - 3.8 |
| Burn Pt. (ASTM D92)* | Higher than 350°C. |
| Sulfates (ASTM D117-31)* | None |
| Fixed chlorine content (Carius)* | 60 + 0.5% |
| Evaporation at 100°C. for 6 hrs.* | 0.2% max. |
| Stability* | There shall be no liberation of chlorine or chlorides when the material is heated at 100°C. in a glass vessel in contact with air for periods of at least one month. |
| Specific Heat at 25°C.* | 0.23 |

*Not determined unless by special request.

| | |
|---|---|
| Hydrolysis Stability Test chlorides,ppm. | 3.0 (tentative) max. |
| Thermal Stability Test chlorides, ppm. | 0.7 (tentative) max. |

## PYRANOL 1481

| PROPERTIES | TYPICAL |
|---|---|
| Viscosity at 37.8°C. | 70 - 82 sec. Saybolt Univ. |
| Spec. Gravity at 15.5/15.5°C. | 1.525 - 1.535 |
| Color, APHA | 150 max. |
| Condition | Clear |
| Acidity, mg. KOH/g. | 0.01 max. |
| Pour Pt., °C. | -15 or lower |
| Inorganic Chlorides, ppm. | 0.10 max. |
| Refractive Index at 25°C. | 1.6205 - 1.6215 |
| Distillation Range | |
| Corrected for stem and barometric pressure. | |
| First drop | 205°C. min. |
| 25% max. | Below 270°C. |
| 90% | 380 - 395°C. |
| Corrosion Test | |
| Change in Weight | 0.0% |
| Color, APHA | 200 max. |
| Acidity, after test, mg.KOH/g. | 0.01 max. |
| Free Chlorides, ppm. | 0.10 max. |
| Condition after test | Clear |
| Water Content, ppm. | 35 max. |
| Resistivity at 100°C 500 volts, DC, 0.1"gap | 100 x $10^9$ ohm-cm.,min. |
| Dielectric Constant (100°C., 1000 cycles) | 4.1 - 4.6 |
| Hydrolysis Stability Test chlorides, ppm. | 3.0 (tentative) max. |
| Thermal Stability Test chlorides, ppm. | 0.5 (tentative) max. |

# PYRANOL 1488

| PROPERTIES | TYPICAL |
|---|---|
| Visc. at 37.8°C. | 54 - 2 Sec. Saybolt Univ. |
| Spec. Grav. at 15.5/15.5°C. | 1.560 - 1.568 |
| Color, APHA | 150 max. |
| Acidity (Mg KOH/g) | .014 max. |
| Water, ppm. | 35 max. |
| Condition | Clear |
| Refrac. Index at 25°C. | 1.6137 - 1.6147 |
| Free Chloride, ppm. | 0.10 max. |
| Pour Point, °C. | Lower than -32°C. |
| Resis. at 100°C., 500 v D.C. 1" gap | $100 \times 10^9$ ohm-cm min. |
| Dielectric Strength (25°C.) | Over 35 KV |
| Corrosion: Loss of Aluminum | None. Heating with aluminum for 6 hrs. at 200-220°C. The Pyranol after heating should meet the following specs: |
| | Color, APHA — 200 max.<br>Acidity (MgKOH/g) — .014 max.<br>Free Chlorides ppm — .10 max.<br>Condition — Clear |
| Dielectric Constant at 1000 cycles at 100°C.* | 3.7 - 4.0 |
| Distilling Range (corrected)* | |
| 1st drop | 200°C. min. |
| Below 270°C. | 40% max. |
| 90% point | 295 - 415°C. |
| Burn Point (ASTM D-92)* | None up to boiling point |
| Fixed Chlorines* | 59.1% min. |
| Arc Formed Gases* (Oxygen-free liquid at 25°C.) | Less than 1.0% total combustible gases including carbon monoxide, hydrogen, and volatile hydrocarbons. |

*Not determined unless by special request.

## PYRANOL 1467

| PROPERTIES | TYPICAL |
|---|---|
| Visc. at 37.8°C., (ASTM D88) | 54 ± 2 sec. Saybolt Univ. |
| Specific Gravity at 15.5/15.5°C. (ASTM D-287) | 1.560 - 1.568 |
| Color, APHA | 150 max. |
| Condition | Clear |
| Acidity, mg. KOH/g. | 0.01 max. |
| Pour Point, °C. (ASTM D-97) | -32°C. or lower |
| Inorganic Chlorides, ppm. | 0.10 max. |
| Refractive Index at 25°C. | 1.6137 - 1.6147 |
| Distillation Range (ASTM D20) Corrected for stem and barometric pressure | 1st drop - 200°C. min.<br>Below 270°C. - 40% max.<br>90% - 395 - 415°C. |
| Corrosion | After heating with aluminum for 6 hrs. at 200-220°C., the aluminum must not be corroded either on visual or weight inspection and the Pyranol should meet the following specs: |

| | |
|---|---|
| Color, APHA | 200 max. |
| Acidity, mg.KOH/g. | 0.01 max. |
| Inorganic Chlorides ppm. | 5 max. |
| Condition | Clear |

| | |
|---|---|
| Water Content, ppm. | 30 max. |
| Resistivity, 100°C. 500 volts, 0.1" gap | 100 x $10^9$ ohm-cm., min. |
| Dielectric Strength, 25°C. | 35 KV., min. |
| Dielectric Constant, 100°C. 1000 cycles* | 3.7 - 4.0 |
| Tin Tetraphenyl* | 0.125% ± 0.01% by weight |
| Burn Point, (ASTM D92)* | None up to Boiling Point |
| Fixed Chlorine* | 59.1% min. |
| Arc Formed Gases* (Oxygen Free Liquid at 25°C.) | Less than 1.0% Total combustible gases including carbon monoxide, hydrogen and volatile hydrocarbons. |

*Not determined unless by special request.

# PYRANOL 1470

| PROPERTIES | TYPICAL |
|---|---|
| Visc. at 37.8°C. (ASTM D88) | 41-45 Sec. Saybolt Univ. |
| Spec. Gravity at 15.5/15.5°C., (ASTM D287) | 1.563 - 1.571 |
| Color, APHA | 150 max. |
| Condition | Clear |
| Acidity, mg. KOH/g. | 0.01 max. |
| Pour Pt., °C., (ASTM D97) | -44°C., or lower |
| Inorganic Chlorides, ppm. | 0.10 max. |
| Refractive Index at 25°C. | 1.6075 - 1.6085 |
| Distillation Range (ASTM D20) Corrected for stem and barometric pressure | |
| First drop | 210°C., min. |
| 35% | 240 - 256°C. |
| 55% | 290 - 330°C. |
| 65% | 385 - 400°C. |
| 95% | 395 - 415°C. |
| Corrosion | After heating with aluminum for 6 hrs. at 200-220°C., the aluminum must not be corroded either on visual or weight inspection and the Pyranol should meet the following specs: |
| | Color, APHA: 200 max.<br>Acidity, mg.KOH/g.: 0.01 max.<br>Inorg.Chlorides,ppm: 5 max.<br>Condition: Clear |
| Water Content, ppm. | 30 max. |
| Resistivity, 100°C., 500 v., 0.1"gap | 100 x $10^9$ ohm-cm., min. |
| Dielectric Strength, 25°C. | 35 KV., min. |
| Dielectric Constant, 100°C., 1000 cycles* | 3.8 - 4.3 |
| Tin Tetraphenyl* | 0.125% ± 0.01% by weight |
| Durn Point, (ASTM D92)* | None up to Boiling Point |
| Fixed Chlorine* | 60.5 ± 0.5 |
| Arc Formed Gases* (Oxygen Free Liquid at 25°C.) | Total combustible gases including carbon monoxide, hydrogen and volatile hydrocarbons. |
| Electrical Stability* | After heating for 96 hrs. at 100°C. in a closed container, the resistivity should not decrease more than 10%. |

*Not determined unless by special request.

## INERTEEN PPO

| PROPERTIES | TYPICAL |
|---|---|
| Visc. @ 37.8°C., (ASTM D88) | 54± 2 sec. Saybolt Universal |
| Specific Gravity @ 15.5/15.5°C. (ASTM D-287) | 1.560 - 1.568 |
| Color APHA | 150 max. |
| Condition | Clear |
| Acidity, mg. KOH/g. | 0.014 max. |
| Pour point, °C. (ASTM D-97) | -32°C. or lower |
| Inorganic Chlorides, ppm. | 0.10 max. |
| Refractive Index @ 25°C. | 1.6137 - 1.6147 |
| Distillation range (ASTM D20) Corrected for stem and barometic pressure | First drop - 200°C. min.<br>Below 270°C. - 40% max.<br>90% - 395 - 415°C. |
| Corrosion | After heating with aluminum for 6 hours at 200 - 220°C. the aluminum must not be corroded either on visual or weight inspection and the askarel should meet the following specs: |
| | Color, APHA 200 max.<br>Acidity, mg.KOH/g. 0.014 max.<br>Inorganic Chlorides, ppm. 2 max.<br>Condition Clear |
| Water content, ppm. | 30 max. |
| Resist., 100°C. 500 Volts, 0.1" gap | 100 x $10^9$ Ohm-cm., min. |
| Dielectric Strength, 25°C. | 35 KV., min. |
| Dielectric constant, 100°C. 1000 cycles* | 3.7 - 4.0 |
| Phenoxy Propene Oxide or Glycidyl Phenyl Ether | 0.18% - 0.22% by weight |
| Burn point, (ASTM D92)* | None up to boiling point |
| Fixed Chlorine* | 59.1% min. |
| Arc formed gases* (Oxygen free liquid @ 25°C.) | Less than 1.0% Total combustible gases including carbon monoxide, hydrogen and volatile hydrocarbons. |

*Not determined unless by special request.

## CHAPTER 8

## QUALITY REQUIREMENTS OF AROCLORS PRIOR TO USE IN THE ELECTRICAL INDUSTRY

### Quality as Supplied to the Electrical Industry

Aroclors and their mixtures supplied to the electrical industry must meet the strict requirements specified by the industry and given in the specifications shown in Chapter 7. The electrical qualities, such as resistivity and power factor of the materials, as supplied, are not the maximum attainable. It is impractical for the manufacturer to furnish these dielectrics to the customer at the maximum attainable qualities because even with careful packaging, sampling, shipping, and handling, these fluids may pick up traces of contaminants from "clean" tank cars, drums, pipe lines, pumps, etc. However, as supplied in accordance with the specifications, the fluids must respond readily to "up-grading" by earth treatment to arrive at the desired maximum refinement required for use by the electrical industry.

### Typical Electrical Quality of Aroclors Used in the Industry

#### Capacitor Impregnation

Table IV indicates the desirable minimum resistivity values of Aroclor dielectrics immediately after earth refinement by the user when ready to impregnate capacitors. These values are compared with the similar values of the material after the capacitor impregnation has been completed in a relatively clean system.

TABLE IV

| Dielectric | Volume Resistivity Ohm-cm at 100°C. and 500 volts DC. Prior to impregnation | After impregnation |
|---|---|---|
| Aroclor 1254 | 2,500 x $10^9$ | 800 x $10^9$ |
| Aroclor 1242 | 1,500 x $10^9$ | 600 x $10^9$ |
| Pyranol 1481 | 600 x $10^9$ | 400 x $10^9$ |

The power factor of earth refined Aroclor prior to capacitor impregnation should not exceed 0.1 percent at 100°C. and 1000 cycles.

Transformer Filling

The minimum resistivity of transformer askarel as specified for supply to the electrical industry is 100 x $10^9$ Ohm-cm. at 100°C., 500 volts and 0.1 inch gap. While power factor is not part of the suppliers' official specification, this value for freshly made transformer askarel ranges from 0.1 to 0.3 percent at 100°C. and 1000 cycles. This would be approximately 0.05 percent at 20°C. and 60 cycles.

In order to arrive at higher and yet practical dielectric values the transformer manufacturer needs to earth refine the fluids immediately prior to using.

It is reasonable to strive for a volume resistivity value around 1,500 x $10^9$ Ohm-cm. at 100°C. and power factor values of about 0.05 percent at 20°C. and 60 cycles or 2 percent at 100°C. and 60 cycles.

Table V compares resistivity readings with the corresponding power factor values obtained on the given samples of typical transformer askarel.

TABLE V

| Volume Resistivity $10^9$ Ohm-cm. at 100°C. | Power Factor 60 cy. 100°C. | 60 cy. 20°C. |
|---|---|---|
| 1,500 | 2% | 0.05% |
| 500 | 5% | 0.1% |
| 100 | 15% | 0.7% |
| 60-70 | 20-25% | 2.0% |

When adequately earth refined to give a resistivity in the range of 500 to 1500 x $10^9$ Ohm-cm. at 100°C., sample of such transformer askarel taken after filling a newly constructed and relatively clean transformer should have a resistivity of at least 200 x $10^9$ Ohm-cm. at 100°C. and a corresponding power factor less than 12 percent at 100°C. and 60 cycles.

## CHAPTER 9

## EARTH REFINEMENT OF AROCLORS TO ARRIVE AT THE DESIRED ELECTRICAL QUALITIES

### Earth Treatment in the Laboratory Preparatory to Analysis

In Chapter 5 on Page 27 the laboratory procedure for preparing the test sample using 0.1 to 0.2 percent of activated earth is given. It is also stated that the absorbent is minus 200 mesh Attapulgus earth activated just prior to use by heating in shallow trays for four hours at 400°C. (752°F.) or for at least 12 hours at 250°C. (482°F.)

### Earth Treatment by the Plant Manufacturing the Askarel

Earth refinement in the plant is essentially the same as used when preparing the laboratory sample. The same amount of freshly conditioned earth (0.1 percent to 0.2 percent by weight) is added to the askarel and the mixture is agitated thoroughly and heated for about four hours.

The more viscous dielectrics, such as Aroclor 1248 and Aroclor 1254 are heated at about 70 to 80°C. (158° to 176°F.) and the less viscous materials such as Aroclor 1242 and Pyranols 1481, 1467 and 1470, or Inerteen PPO are heated at about 50° to 60°C. (122° to 140°F.)

After about four hours contact the earth is removed from the dielectric fluids using a Sparkler or Sweetland or a comparable filter press, previously fitted with filter paper liners such as supplied by Carl Schleicher & Schuel Company, Inc., Keene, New Hampshire. The paper is usually 25 mils thick

and must be dried at 100°C. to remove moisture prior to use in the filter press.

Earth Refinement by the User

Capacitor Manufacturers: Capacitor manufacturers usually use the same procedure for earth refining as employed by the manufacturer of the askarels. Because this method employs loose earth which can be thoroughly mixed into the askarel, it is believed to be the most efficient and is certainly known to give very good results. However, towers (cylinders) filled with relatively coarse earth through which the dielectric fluids are pumped and recirculated have also been used by capacitor manufacturers.

Transformer Manufacturers: The large manufacturers of askarel transformers usually use the same type of procedure for refinement with loose earth as employed by the manufacturer of the askarel dielectrics. Handling relatively large amounts of the askarel transformer fluid justifies installing the tanks and filter presses required. This earth refining equipment is usually supplemented with portable cartridge type filters or a small portable platen frame type filter press. This latter equipment is then used when newly made askarel transformers are filled with the fluid and it is necessary to clean the transformer and the fluid by draining out the fluid pumping it through the filter press or cartridges containing the earth and recirculating until the desired electrical properties are attained.

Likewise, smaller manufacturers of askarel transformers can use most conveniently the portable cartridge or platen frame type filters.

The Effect of Earth Refinement on Removal of Tin Tetraphenyl Scavengers from the Transformer Askarel: As indicated above, normally transformer askarels respond readily to up-grading by the use of 0.1 to 0.2 percent by weight of earth based on the total weight of the fluid. However, if the fluids are unusually contaminated, larger amounts of earth are required to up-grade the dielectrics. This raises question about the selective adsorption of the scavengers by the earth treatment. It is indicated that to selectively adsorb significant amounts of the scavengers, repeated treatment with 1 percent or more of earth is required, as shown in the following table:

TABLE VI

REMOVAL OF TIN-TETRAPHENYL BY REPEATED TREATMENT OF ASKAREL WITH ONE PERCENT OF EARTH

| | | Tin-tetraphenyl Content | | |
|---|---|---|---|---|
| Sample | No. | Original | After 4 treatments at 90°C. | After 7 treatments at 90°C. |
| Pyranol 1470 | 1 | 0.119% | 0.018% | 0.006% |
| | | | After 4 treatments at 60°C. | After 6 treatments at 60°C. |
| Pyranol 1470 | 2 | 0.107% | 0.021% | 0.003% |

## CHAPTER 10

## CONTAMINATION

Askarels as supplied by the manufacturer respond readily to earth refining resulting in a very high order of dielectric properties. For example, it is possible to attain volume resistivity values up to 20,000 or 30,000 x $10^9$ Ohm-Cm. at 100°C., 500 volts, 0.1 inch gap and power factor values no more than 0.05% at 100°C. and 1000 cycles.

Except for very special situations, it is not practical to refine these dielectrics to this extent. In commercial use, transfer of the fluids from one clean container to another which may result in contacting traces of conducting impurities does not allow maintaining such a high order of dielectric properties.

Referring to electrical values, this accounts for the more practical order of specification values as indicated in Chapter 7 and to which the electric industry has committed the supplier of the dielectrics. This also accounts for the desirability and need of the user of askarel dielectrics to earth refine immediately prior to use in order to arrive at the maximum and yet practical dielectric values for his given purpose. Such quality values relative to askarels for capacitors and transformer work were indicated in the preceding Chapter 8.

### AVOIDANCE OF CONTAMINATING ASKAREL CAPACITORS

It is necessary, practical and economical that all steps possible be taken to avoid contaminating influences in the manufacture of askarel capacitors.

Sometimes capacitor manufacturers strive to attain the very high order of dielectric qualities possible for askarel as mentioned above. Since it is very difficult, if not almost impossible to maintain such a high order, usually capacitor manufacturers comply with the more practical schedule attainable by normal earth refining practices as shown in Table IV Chapter 8.

Equal care must be exercised in selecting, conditioning and handling the other construction materials of the askarel capacitor. For example, the water used in manufacturing capacitor tissue is either distilled or deionized. Quality control of the capacitor paper requires chemical tests to characterize the fiber and its purity. Physical and electrical tests to determine moisture and power factor are essential. Acceptable dielectric loss values of the dry and unimpregnated paper do not suffice for judging quality because in some instances after impregnation with good quality askarel, higher dielectric losses increasing rapidly with temperature may be obtained.

The aluminum foil used must be extremely pure and free from residual traces of rolling oils or compounds. Accordingly, the term usually applied to the foil is "dry" foil. Similar care and purity requirements apply to the aluminum tabs used.

The paper and aluminum foil is wound to form the core in an air conditioned room and often the machine operators are required to wear cotton gloves to prevent oil from the operators skin contaminating the cores.

The steel cans or turn plate capacitor cans require thorough cleansing and degreasing with perchloroethylene of required purity and free of any objectionable stabilizing agents. Similarly, the

capacitor impregnating equipment and chambers must be kept clean. To facilitate maintenance of cleanliness, sometimes stainless steel construction is used. However, ordinary steel equipment is common and when "conditioned", that is to say, coated with a thin film of clean Aroclor, this type of construction material is entirely satisfactory. To condition a new plant or clean an old one, askarel is circulated through the system, then purified by earth refining and recirculated. This process is repeated until all contaminating influences have been removed.

Moisture, probably the most obvious contaminent in askarel impregnated capacitors, increases the dielectric loss under AC voltage and decreases the capacitor life. Therefore, very efficient vacuum, as low as 5 or 10 microns, and heat carefully controlled up to 130°C. are employed to expel the moisture from the capacitor cores prior to impregnation. Also to avoid moisture entering the askarel during storage, it is common practice to warm the dielectric in the storage tank to about 50°C. in the presence of mild vacuum.

Traces of any substances soluble in askarel and capable of ionization will have a marked adversed effect on the dielectric loss of the askarel or the finished capacitor. Therefore, much care is required to avoid contamination with solder flux. For example, the use of rosin core solder is known to cause contamination. When rim sealing compounds are used in the lids of small capacitors, there must be assurance that the catalyst or other ingredients used in such materials do not cause contamination. Improperly selected pipe sealing compounds used on the threads on the fittings for sight glasses and instruments on the storage and impregnating equipment are known to have caused contamination.

* * * * * * *

All these factors about contamination must be kept in mind when impregnating capacitors, especially by the chamber method but also by the manifold method.

The moisture content of the paper used in the capacitor cores may easily introduce several gallons of water into the average impregnating chamber. This water is removed from the capacitors prior to impregnation -- usually by heating the chamber to 130°C. in the presence of efficient vacuum, 100 microns or less.

If the chamber is not made of the preferred materials of construction, slight corrosion (iron rusting) may occur and the film of askarel on the interior surface of the tank may become contaminated and introduce traces of impurities into the clean dielectric fluid entering the chamber for impregnating the capacitors. In fact, because of this possibility of contamination, in some operations the capacitors are conditioned and dried in a separate oven or chamber. Then when thoroughly dried, they are then transferred into a second chamber used only for impregnation.

In the case of relatively large sized capacitors, such as power factor correction units, a manifold with branches may be used to handle each unit individually rather than by the batch -- chamber method.

The unimpregnated capacitors are placed into an oven and vacuum is applied to the individual units attached to the manifold. After the capacitors have been evacuated and dried, the askarel is introduced through the manifold and its branches. Care must be taken that moist air or contaminants do not collect in the branches through which the dielectric is introduced.

* * * * * * *

Care must be taken to avoid contamination with any kind of grease, oil, packing material and "rubber" gaskets used with the machinery, such as pumps, etc., connected with the handling and impregnating facilities.

It is not practical to discuss all possible sources of contamination and it should suffice to say that the manufacturers of askarel capacitors need to and do exercise all known precautions to avoid contamination and should evaluate and life test representative units before supplying the finished merchandise.

## AVOIDANCE OF CONTAMINATING ASKAREL TRANSFORMERS

Obviously, in the manufacture of askarel transformers it is impractical and impossible to employ purification or refinements as required, for example, in the production of askarel power factor correction capacitors.

In a transformer, heat from the dielectric losses of "slightly" contaminated askarel is negligible compared with heat generated by the transformer core. While power factor and resistivity of the transformer fluid are important, they are not as critical as is the case when similar askarels are used in capacitors.

However, this does not excuse the askarel transformer manufacturer from striving to meet the practical quality requirements as given in Chapter 8. In order to meet these requirements, it is necessary to earth refine the transformer askarel immediately prior to filling the unit. After the initial fill, the fluid should be withdrawn from the transformer, circulated through an earthen filter, then pumped back into the transformer and recirculated through the filter until both the fluid and transformer are clean and show the desired power factor and resistivity values.

If such normal earth refinement fails to give the desired results, it will be necessary to study the quality of the materials of construction and look for all possible sources of contamination in the transformer and handling equipment.

The characteristic high dielectric strength of transformer askarel is not a good criterion of purity because with the exception of being adversely affected by moisture, it is not impaired irrespective of the contamination of soluble products which give increased power factor. F. M. Clark of General Electric Company tabulated the following values of transformer askarel selected from hundreds of askarel samples taken from commercially operating transformers to show lack of reduction of dielectric strength with marked increase of power factor values.

TABLE VII

DIELECTRIC STRENGTH AND POWER FACTORS OF ASKAREL IN USED TRANSFORMERS

| Sample | Power Factor, 60 cy. at 25°C., per cent | Dielectric Strength at 25°C. KV |
|---|---|---|
| No. 1 | 0.1 | 38 |
| No. 2 | 0.5 | 35 |
| No. 3 | 5 | 45 |
| No. 4 | 15 | 39 |
| No. 5 | 30 | 43 |

However, the need for care and proper selection of transformer construction materials is emphasized in the following tabulation which shows the marked increase of power factor resulting from contamination of the askarel with synthetic rubber materials and varnished cloth, as compared with acceptable materials of construction given in Table VIII.

TABLE VIII

POWER FACTOR CONTAMINATION PRODUCED BY TRANSFORMER MATERIALS AGED IN ASKAREL AT 100°C. FOR 96 HOURS

| Material | Askarel Power Factor, Percent | Dielectric Strength, KV |
|---|---|---|
| None | 1.0 | 35 |
| Black varnished cloth | 85.0 | 42 |
| Copper | 1.5 | 40 |
| Press Board | 2.0 | 37 |
| Manila Paper | 1.5 | 39 |
| Phenolic resin | 1.6 | 41 |
| Shellac | 6.0 | 36 |
| Iron | 5.0 | 39 |
| Synthetic rubber | 70.0 | 39 |

Contaminants which cause a high power factor will of course also impair resistivity values.

The following Table IX shows the degree to which acceptable and good commonly used materials of construction lower resistivity from the original high values possessed by the askarel.

TABLE IX

EFFECT OF COMMONLY USED INSULATION MATERIALS ON THE RESISTIVITY OF TRANSFORMER ASKAREL

| Sample | Volume Resistivity x $10^9$ Ohm-cm. at 100°C. |
|---|---|
| 1. Freshly made askarel before heat aging | 2,000 |
| 2. Same as 1, after heat aging | 1,900 |
| 3. After heat aging with the following materials added | |
| a - Phenolic resin tap changer material | 1,200 |
| b - Paper | 750 |
| c - Grade A press board (tan) | 500 |

| | |
|---|---|
| d - Grade A press board (gray) | 500 |
| e - Grade A press board, laminated strip | 400 |
| f - Cotton wrapping | 300 |
| g - Glyptal 1276 cement, cured 48 hours at 110°C. | 100 |

The procedure used to evaluate materials of construction is simple and should be employed by all makers of askarel transformers.

One inch square samples of the surface of the construction materials are immersed in one liter of good quality transformer askarel and heated for 96 hours at 100°C. The increase in power factor and reduction of resistivity of the fluid after such exposure are compared with the values of the original fluid heated similarly but in the absence of the construction materials.

The following Table X gives such a comparison and illustrates the unacceptable properties of the varnished cambric and black binding tape.

TABLE X

| PROPERTY | FRESHLY MADE TRANSFORMER ASKAREL | SAME FLUID EXPOSED TO FIBER BOARD | SAME FLUID EXPOSED TO VARNISHED CAMBRIC OR BLACK BINDING TAPE |
|---|---|---|---|
| PF, 100°C., 1 KC | 0.2% | 0.45% | 3% |
| Resistivity, 100°C. Ohm-cm. x $10^9$ | 2,500 | 436 | 18 |
| Dielectric Strength at 25°C. | 45 KV | 45 KV | 45 KV |

When the askarel fluid contaminated with varnished cambric or the black binding tape was treated for one hour at 75°C. with one percent by weight of attapulgus earth, the power factor and resistivit values were restored to those of the original fluid.

While there seem to be no reports of askarel transformers failing in service as a result of contamination from the use of questionable materials of construction, as discussed above, their unwise use is readily detectable and leads to embarrassing question about impairment of the transformer's life.

Such a case is illustrated by an askarel transformer giving a megger reading reading as low as 3, after two years service life. The transformer had not been subjected to arcing and the dielectric strength of the askarel remained above 35 KV at 25°C., or well within the specification of new askarel. Maintenance of high dielectric strength in the presence of contamination appears to account for the transformer not failing.

The following Table XI compares the properties of freshly made askarel with the similar values of the fluid taken from the transformer after two years use and also with the same contaminated fluid following refinement by earth treatment.

TABLE XI

COMPARISON OF PROPERTIES OF CONTAMINATED TRANSFORMER ASKAREL BEFORE AND AFTER EARTH REFINEMENT

| PROPERTY | TYPICAL OF NEW ASKAREL | SAMPLE FROM TRANSFORMER USED 2 YEARS | SAME SAMPLE AFTER EARTH TREATMENT 1 HOUR WITH 1% OF EARTH |
|---|---|---|---|
| PF., 100°C. 60 Cycles | 1 to 2% | 150%(dissipation factor) | 1% |
| PF., 100°C. 1 Kc. | 0.2% | 15% | 0.1% |
| Resist. 100°C. Ohm-cm. x $10^9$ | 500 to 1500 | 6 | 2600 |
| Dielectric Strength 25°C. | 45 | 40 | 45 |
| Moisture ppm. | 25 | 80 | 20 |
| Acidity mg. KOH/g. | 0.01 | 0.02 | 0.005 |
| Color APHA | 70 | 1000 | 275 |

In the above case it was determined that a varnished insulating material used in the transformer was the source of contamination Since this continuously dissolved in the askarel, obviously, removal of the fluid, followed by earth refinement and refilling the transformer accomplished nothing. The situation pertained at the time of the initial fill persisted until the deleterious component was removed.

Newly built or rebuilt transformers using acceptable materials of construction often contain undeterminable traces of impurities and "dirt" which effect power factor and resistivity, but can be removed by repeated flushing with clean askarel. The following Table XII compares the properties of the askarel sampled after the initial fill with similar values after earth refinement and "soaking" or flushing the transformer, twice with good askarel. After the "dirt" or contamination was removed from the transformer the askarel fluid remained in good condition in the unit as shown in Table XII.

TABLE XII

| PROPERTY | SAMPLE AS RECEIVED | AFTER TREATMENT |
|---|---|---|
| Resistivity @ 100°C. | $30 \times 10^9$ | $2600 \times 10^9$ |
| Dielectric constant @ 100°C. | 3.9 | 3.9 |
| Power Factor @ 100°C., 1000 Cycles | 2% | 0.27% |
| Moisture | 60 ppm. | 25 ppm. |

Moisture solubility in askarel at 25°C. is about 110 parts per million and the specification for new askarel allows a maximum of 30 parts per million of water.

Essentially, moisture-free askarel was found by Clark to have a dielectric strength of 70 KV. With increased amounts of dissolved

water the dielectric strength gradually decreased and appeared to level off at a value of about 38 KV when the water content reached 80 parts per million.

Water exceeding the solubility limit in askarel has a marked adverse affect on the power factor and resistivity of the dielectric fluid, which may, however, maintain its high breakdown strength even though water accumulates as a separate phase on the surface.

Undissolved moisture can be removed readily from transformer askarel by warming the fluid to 70°C. and blowing with dry nitrogen or by treatment with dry, conditioned earth and filtering through a press fitted with dry filter papers or through an earthen cartridge type filter.

As indicated previously, moisture must be kept out of askarel transformers by using adequate gaskets, as described in Chapter 3, and preferably sealing the device with dry nitrogen over the askarel.

Mineral oil is soluble in these fire-resistant transformer askarels and is regarded to be a contaminant. Petroleum hydrocarbons cannot be removed from askarels and the permissable amount may not exceed 2 percent by volume lest the fire-resistant values of the askarel is impaired beyond acceptable limits.

Possible contaminants in transformer manufacture include welding and solder fluxes, oils and greases, bituminous materials, pipe thread lubricants, and contamination from bushing and pot head compounds. Paint or varnish coatings must not touch the interior of the transformer shell. Adhesives or coatings applied to gaskets must not touch the interior of the transformer.

All natural or synthetic rubber plastics or polymeric materials, resins, varnishes and lacquers and adhesives must be regarded as contaminants unless included in the very few acceptable classes and proved suitable by actual testing.

It is disappointing to find an askarel transformer manufacturer exercising precautions against contamination, and employing earth refinement, and yet inadvertently using a neoprene or other objectionable hose line to transfer the fluid economically!! If flexible hose needs be used, it should be a flexible stainless steel type or a "rubber" hose lined with Teflon.

When using any of the following suggested suitable materials of construction, it is prudent to employ appropriate control evaluation tests to be certain that the given material within a class regarded as acceptable will meet the requirements from a physical and electrical standpoint.

TABLE XIII

ACCEPTABLE MATERIAL FOR CONSTRUCTION OF ASKAREL TRANSFORMERS

Structural Materials and Fillings

Metals - Commonly used metals including steel, copper, aluminum, tin and brass are suitable if clean.

Wood - Suitable if dry and free of natural gums and resins.

Paper - Suitable

Press Board - Suitable

Cotton - Suitable

Asbestos - Suitable

Glass - Suitable

Ceramics - Suitable

Phenol-formaldehyde resins - Suitable if adequately cured.

Melamine-formaldehyde resins - Suitable if adequately cured.

Cellulose acetate - Suitable

Cellulose tri-acetate - Suitable

Cork - Suitable

Gasketing Materials and Adhesives

Metals - (As above)

Teflon - Suitable

Silastic (silicone) - Suitable if adequately cured.

Polyurethane - Suitable if adequately cured.

Cork (fine grain and bonded with phenolic resin) - Suitable, but susceptible to penetration by askarel.

Nitrile rubber - Sometimes used for gaskets - but susceptible to attack by askarel.

Cork Nitrile rubber - Often used

Dewaxed Orange Shellac - Suitable

Epoxy - Suitable if adequately cured.

Isocyanate - Suitable if adequately cured.

Tapes and Wire

Insulation

Cotton - Suitable

Paper - Suitable

Asbestos - Suitable

Glass - Suitable

Rayon - Suitable

Cellulose acetate - Suitable

Teflon - Suitable

Silicone - Suitable

Surface Coating for Transformer Exterior

Baked Phenol-Formaldehyde - Suitable

Baked Melamine-Formaldehyde - Suitable

Baked Epoxies - Suitable

Polyurethane Coatings - Suitable

Surface Coating for Interior of Transformer Shell to Prevent Rusting in Storage

25 parts Aroclor 5460 dissolved in 75 parts lacquer thinner.

The need for clean shop practice and avoidance of contaminating influences when building askarel transformers is emphasized by the following information submitted by a highly qualified manufacturer to indicate the condition of askarel sampled from normally operating apparatus after a number of years service. Fluid sampled from the top and bottom of at least 25 askarel transformers operatin satisfactorily in different part of the country was analyzed, comprehensively. The following general conclusions were drawn:

1. The residual condition of the askarel samples for the most part was satisfactory, but in a few instances excessive dirt or sediment was noted.

2. Moisture content ranged from 19 to 61 parts per million with most samples between 25 and 35 ppm. This reflects a very good degree of dryness.

3. Acidity values were all below 0.01 mg. NaOH/g., which corresponds with the level of freshly made askarel.

4. In most cases the free chlorides did not exceed 0.1 ppm., the specification limit of new askarel. The highest reading was 0.15 ppm.

5. Dielectric strength values ranged from a maximum of 46 KV to a minimum of 28 KV which values are considred satisfactory.

6. Volume resistivity at 100°C. ranged from 20 to 75 x $10^9$ Ohm-cm. which is considered to be satisfactory and may be compared with the specification for new askarel at 100 x $10^9$ Ohm-cm., minimum.

7. Power factor values at 100°C. and 60 cycles ranged from 19 to 75 percent with most samples below 60 percent.

From the above data considered typical and satisfactory relative to all of these askarel transformers operating in a normal manner, it is seen that under satisfactory service life the resistivity of the askarel will decrease and the power factor will increase, as indicated, from similar values of fresh askarel.

These observations emphasize the need for askarel transformer makers to:

1. Earth refine the askarel immediately prior to use in order to arrive at the highest practical electrical values from the fluid.
2. Avoid construction materials which are a source of contaminat:
3. In using acceptable and satisfactorily tested construction materials - to flush the transformer to remove traces of contaminating influences and "dirt".

## CHAPTER 11

## REWORKING CONTAMINATED TRANSFORMER ASKAREL

### Normal Conditions

Askarel contaminated during manufacture of the transformer or after years of normal service life should respond very readily to refinement by treatment with a few tenths of a percent of dry Fullers earth, or Attapulgus clay. This was discussed in detail in previous chapters.

About askarel transformers, after years of normal service life and having continued satisfactory performance, question (difficult to answer) arises as to how high may be the power factor. Also, how low may be the volume resistivity. The case histories given in the preceding chapter seem helpful in arriving at an answer.

Doble Engineering suggests to their clients that, "When used askarel is found to have a power factor of 2.0 percent or more, the cause of high power factor should be determined." (This refers to power factor measured at 20°C. and 60 cycles.) Doble qualifies this suggestion stating, "If the high power factor is caused by water or other conducting matter, free chlorides or high neutralization number, the askarel is probably an operating hazard." Also, "If the high power factor is not due to these causes, it is probably not an operating hazard except that when the power factor is quite high, it may result in excessive heating of the device in which it is used."

Since heat resulting from power factor increase of the askarel in most any commercial transformer is negligible compared with heat generated by the core of the transformer, this consideration does not appear important.

If the suggestion were limited to a 2 percent power factor, it would appear low and probably subject to considerable objection. However, when stated as 2 percent or more, the intention and purpose of this suggestion justifies earnest consideration, although in absence of more knowledge, it appears that conclusive answer to this question is not at hand.

However, it should be apparent that when the power factor of the askarel is 2, 5, or 7 percent or higher at 20°C. and 60 cycles, and volume resistivity at 100°C. is 20 x $10^9$ Ohm-cm. or lower, contamination is present. As it is likely that such a condition can be rectified by simple earth treatment and filtration, there should be little question about desirability of doing this purification work to assure the best possible performance of the transformer.

Arced Conditions

It is fortunate that there seem to be very few cases of significantly arced transformer askarel, as it is difficult to estimate the possible success of reclaiming the fluid. Particularly, it is not easy to lower the free and also the after corrosion chloride levels within the extremely low specification limits for new askarel.

Treatment with dry earth as usually used to refine contaminated askarel, probably will not rectify arced askarel. Special refinement, including hydrolysis of the spent scavenger material, water extraction of excess chlorides, treatment with wet earth, special drying and finally treatment with dry earth is required. The following example is considered typical:

During routine testing of a transformer filled with askarel, a short occurred in the winding, and the arc resulted in formation of easily seen carbon particles in the fluid. The following shows the ineffectiveness of dry earth treatment and the need for wet or water treatment to reclaim a sample of this material in the laboratory.

Passing the damaged fluid through filter paper failed to remove the carbon. The carbon was removed by filtering through paper fitted with a one-half inch pad of Attapulgus earth. At this stage, analysis indicated the following pertinent properties as compared with the specification limits:

PROPERTIES OF TRANSFORMER ASKARELS

| Property | Specification | Sample |
|---|---|---|
| Inorganic chlorides | 0.01 ppm. max. | 0.25 ppm. |
| Acidity, mg. KOH/g. | 0.010 max. | 0.004 |
| Moisture | 30 ppm. max. | 75 ppm. |

Then 0.2 percent by weight of Attapulgus earth was added and the mixture held at 90°C. and agitated for two hours and filtered. This reduced the water to 15 ppm., but the chlorides remained at 0.25 ppm.

Another portion of the original sample was treated with 0.5 percent of earth, held at 50°C. and agitated for two hours and filtered. This lower temperature treatment reduced the water to only 60 ppm. and the chlorides remained at 0.25 ppm.

At this stage, the electrical properties were determined and found to be well within specification limits.

| Property | Specification | Sample |
|---|---|---|
| Resistivity @ 100°C. x $10^9$ Ohm-cm. | 100 | 576 |
| Power Factor @ 100°C. and 1,000 Cycles | ---- | 0.18% |
| Dielectric Constant @ 100°C. | 3.8 - 4.3 | 4.2 |

The above reclaiming tests were repeated using "wet" earth which contained 15 percent moisture. Again, the chlorides remained at the original level of 0.25 ppm., well out of specification.

Then a sample of the askarel was extracted with water, using 20 percent based on the weight of the dielectric fluid. This reduced the chlorides to 0.15 ppm., and a second aqueous extraction using 10 percent of water was required to reduce the chlorides to no detectable amount. The excess moisture was then removed by blowing with dry air and finally filtering through dry earth and filter paper.

Although transformer askarel is several times as expensive as mineral oil, the material is not a high cost item. Therefore, the economies of undertaking work as described above must be weighed against the cost of new askarel.

In any event a fair estimate of the reclaiming cost plus packaging, shipment to location for the work, the cost of several analyses involved, then repackaging in new containers and cost of return freight will indicate at least 50 percent of the cost of new askarel. Accordingly, usually the most practical expedient is to purchase new askarel to replace the arced material.

## CHAPTER 12

## DERMATOLOGY AND TOXICOLOGY

### Skin Exposure

Aroclors, or askarels, accidentally spilled on the skin do not cause an acute toxicity hazard, nor will they cause serious irritation. The materials should be washed from the skin with soap and water. Prolonged skin contact should be avoided. If work clothes become impregnated with these fluids, they should be removed and washed.

When sampling tank cars, canvas gloves and safety glasses, or goggles, should be worn. No special clothing is required, but the workers' garments should be laundered at least weekly and changed, if Aroclors or askarels are spilled on the clothes.

If accidental burns occur from contact with hot askarels, the burn should be treated the same as any ordinary burn. Aroclor, or askarel, adhering to the burned area need not be removed immediately unless treatment of the burn demands it in which case, soap and water, or repeated washings with a vegetable oil should be used. Accidental contact with the eyes results in painful irritation, but not permanent damage to the tissues or the sight. In event of such contact, the eyes should be flushed with a large amount of water for at least 15 minutes. The patient should then be referred to a physician who will treat with an ointment to soothe the eye.

Exposure to Vapors

Vapors from hot Aroclor, or askarel, have a degree of toxicity and should not be inhaled over a prolonged period of time. Experimental work on animals indicates that the maximum safe concentrations of vapors in work rooms is in the range of 0.5 to 1.0 mg. per cubic meter of air. Harmful amounts of the materials are readily detectable by odor and irritation to the eyes. Usually, people can detect concentrations of askarels in the amount of 1.0 mg. per cubic meter of air, which is the level regarded as the safe work room limit for an 8 hour day exposure.

Capacitor impregnations may be done at temperatures as high as 266°F. (130°C.). Following impregnation and draining the chamber, exhaust ventilation should be applied to the chamber to prevent askarel vapors entering the work room. Also, when opening a heated capacitor impregnating chamber, the workmen should wear a respirator during this short interval of exposure.

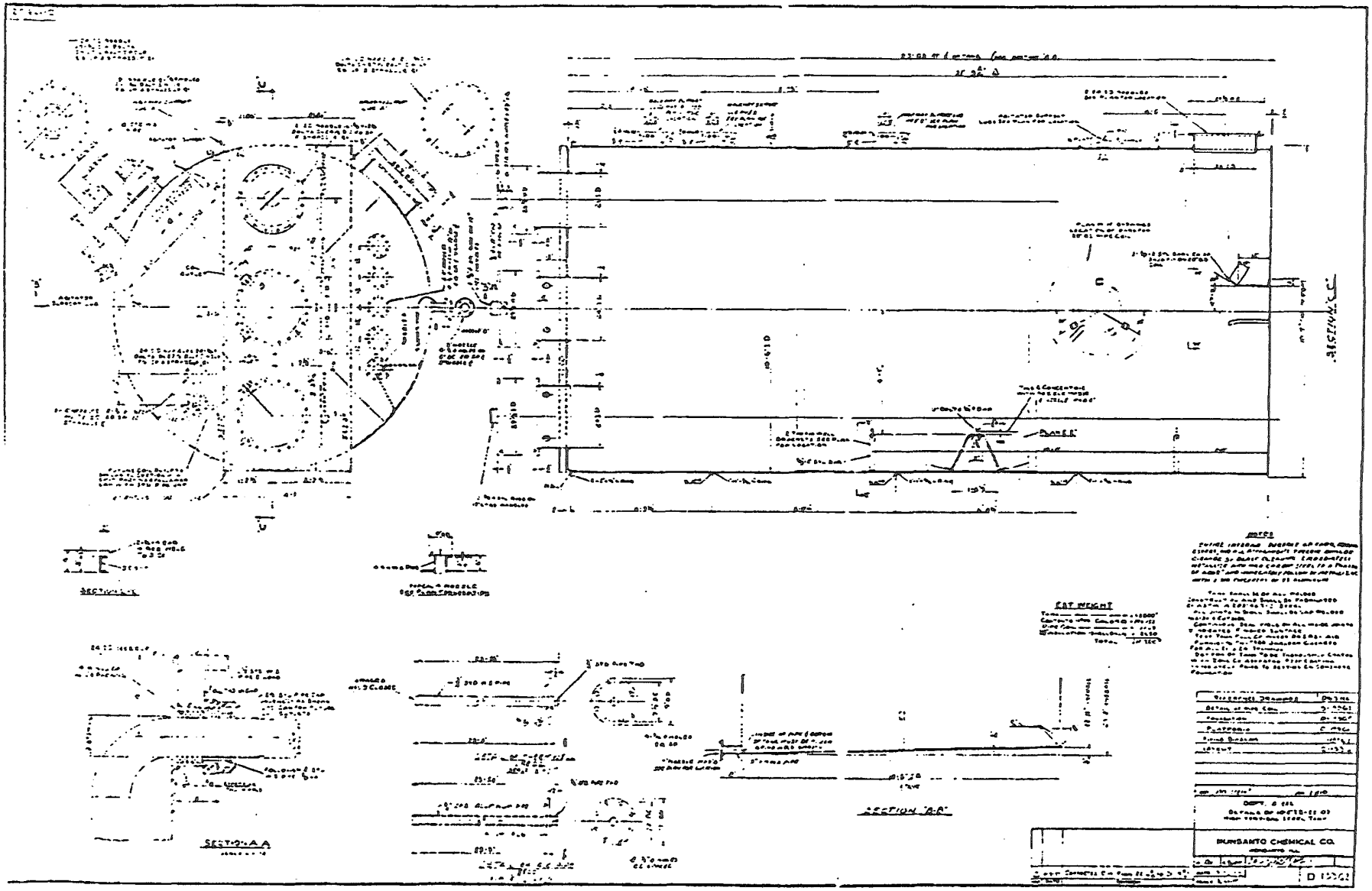
If transformer askarels are used at temperatures above 125° to 150°F. to fill an open transformer, exhaust ventilation should be provided in the immediate area.

The many years of satisfactory and safe use of Aroclors, or askarels, by the electrical industry for impregnating capacitors and filling transformers has demonstrated the industry's ability to handle these fluids without hazard to the workmen.

It is both simple and in line with "good housekeeping" and personal cleanliness to exercise the suggested precautions in all cases.

Vapors from a Severely Arced Askarel Transformer

Experimental data indicate that when askarel is decomposed by an electric arc, insignificant amounts of chlorine and phosgene gas are liberated. The gas is almost entirely hydrogen chloride, which is readily detectable by its odor and its irritating characteristics in even small amounts. Thereby, adequate warning of its presence is provided and significant amounts of fumes would be likely to cause a hazard only in a closed area. Individuals would not voluntarily expose themselves to serious toxic levels of the hydrogen chloride gas fumes.

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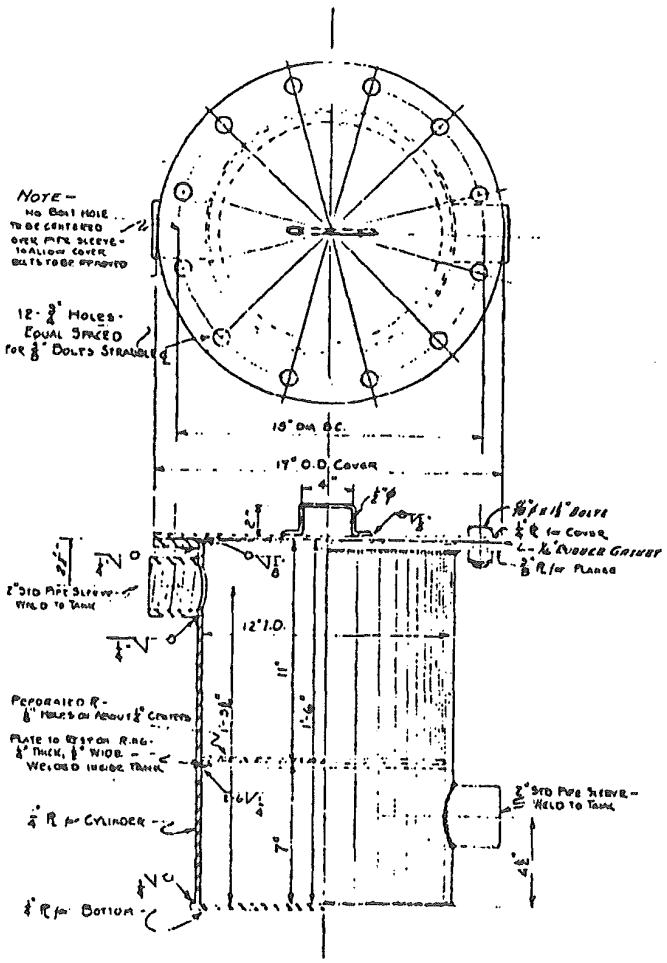


VENT TANK - MARK 9248-1

SCALE - 3" = 1'-0"

REFERENCE DRAWINGS -
PIPE LAYOUT -
STORAGE TANK - SC 8170
BILL OF MATERIAL - 9BM-88
PLATFORM FOR VENT TANK - SC-8235

**Monsanto Chemical Company**
PHOSPHATE DIVISION
ANNISTON, ALABAMA

| REVISIONS | | |
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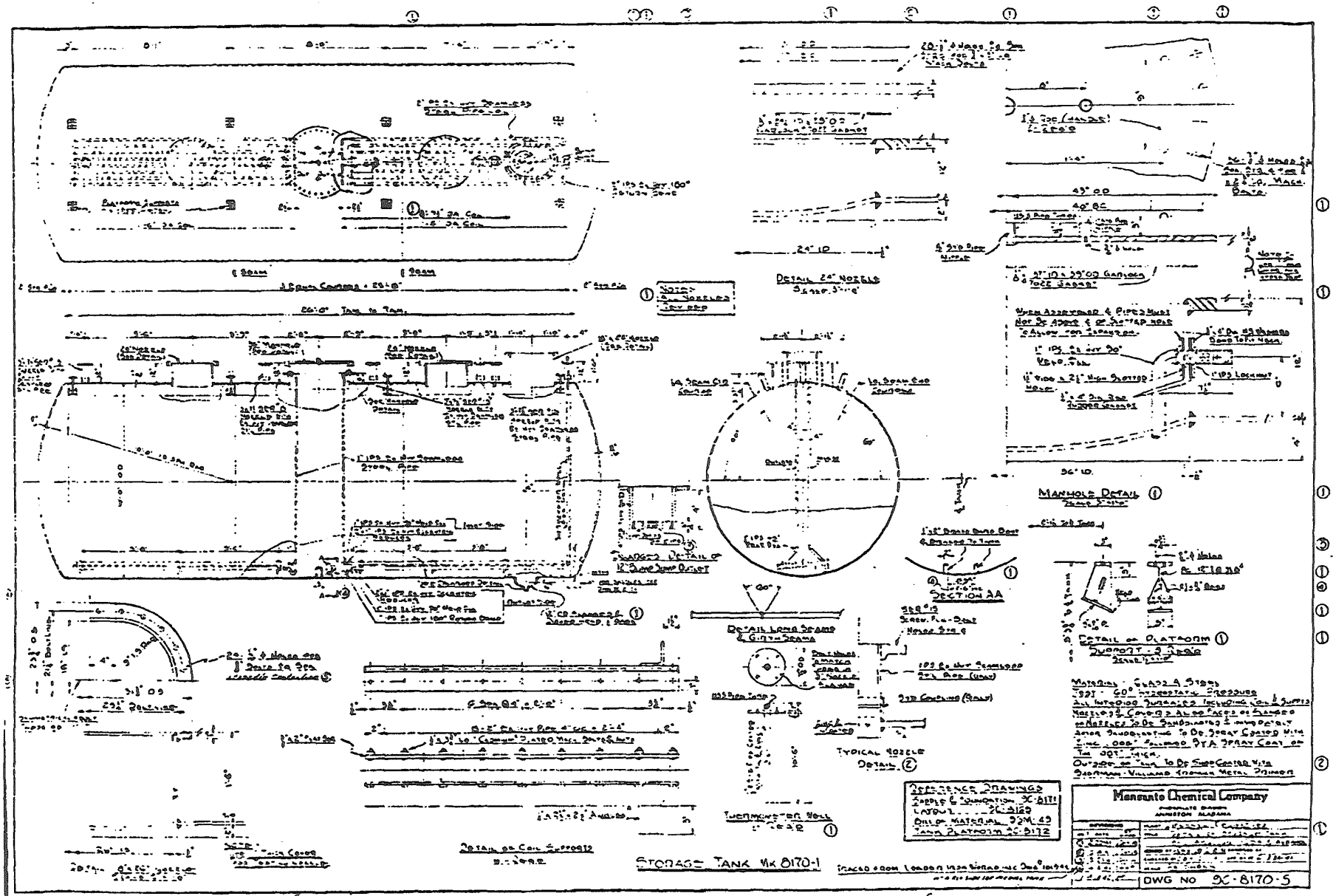
| NO. | DATE | BY WHOM |
| | | |

PLANT ANNISTON
TITLE DIPHENYL DERIVATIVES - VENT TANK & - FOR 260 STORAGE TANK
DRAWN BY A.P.S. DESIGNED BY J.A.P.
CHECKED BY J.W.N. 9-23-41 APP. BY WTD 9-23-41
SCALE 3" = 1'-0"
DWG. No. SC-9248

NEV 007429

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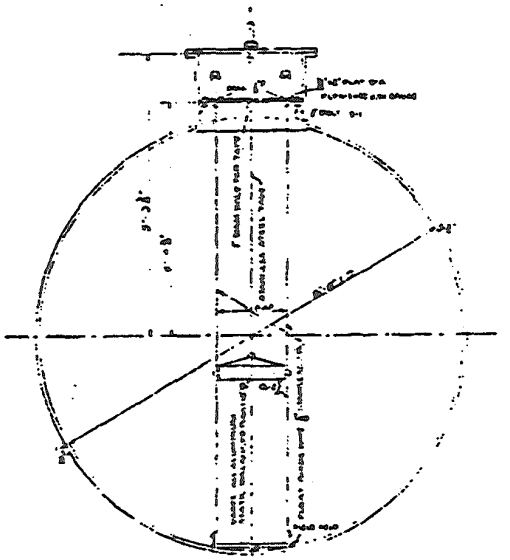
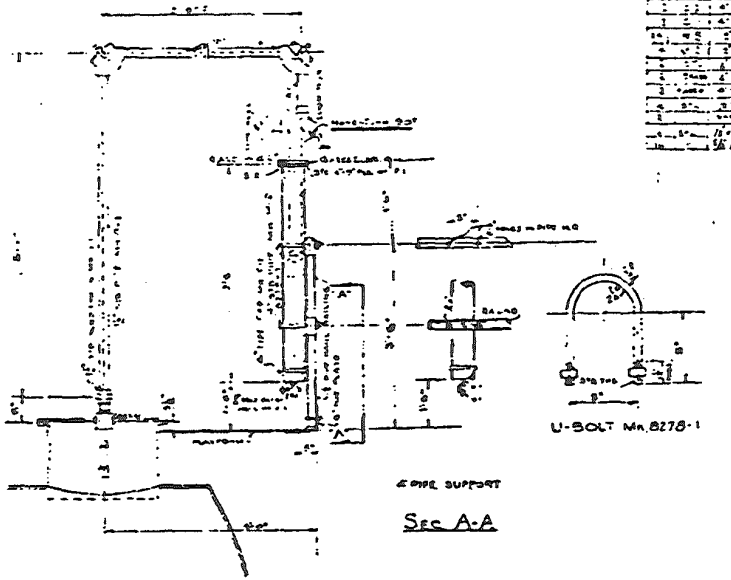


END ELEVATION



SIDE ELEVATION

SEC A-A

U-BOLT Mn.8278-1

BILL OF MATERIAL

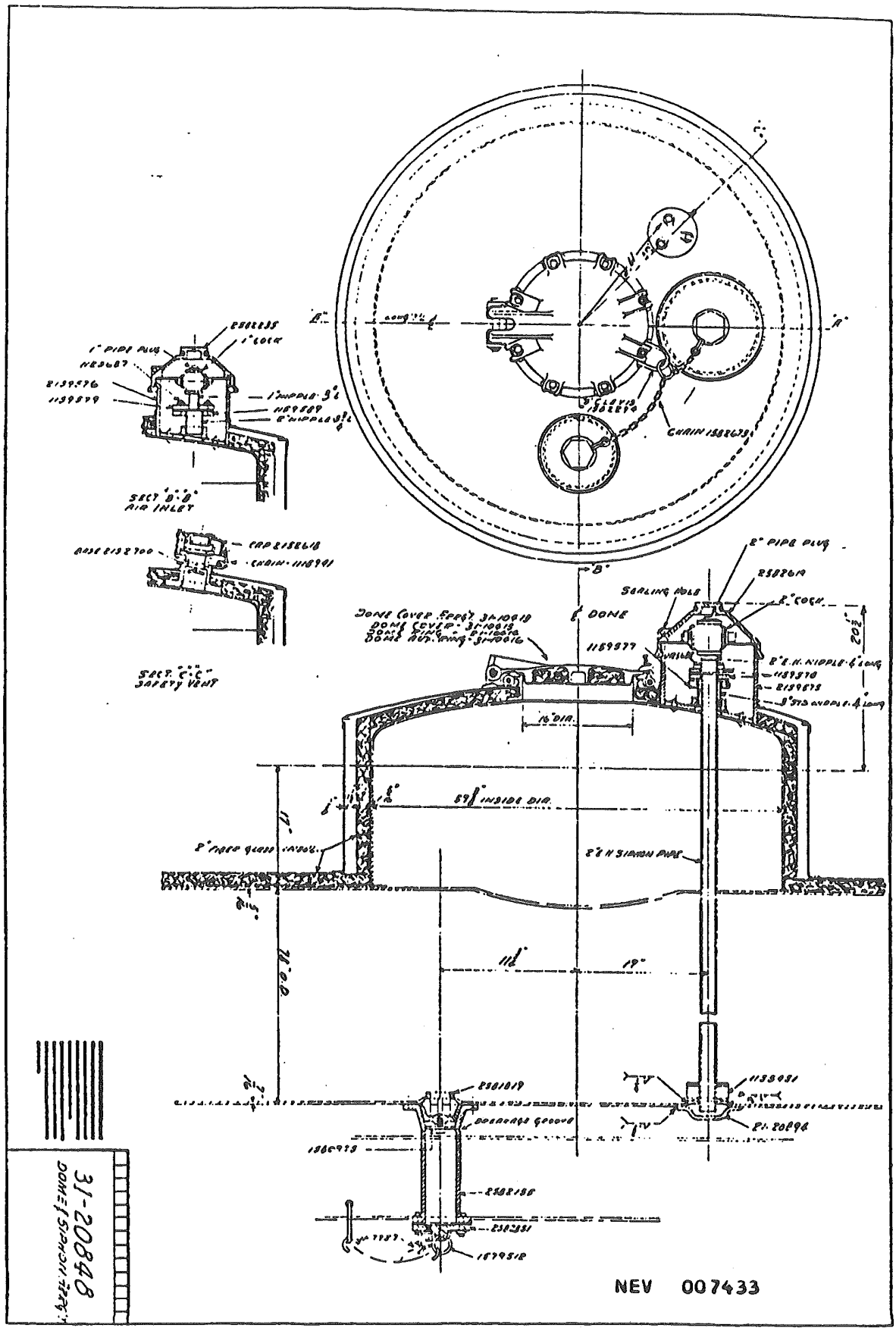
REFERENCE DRAWINGS

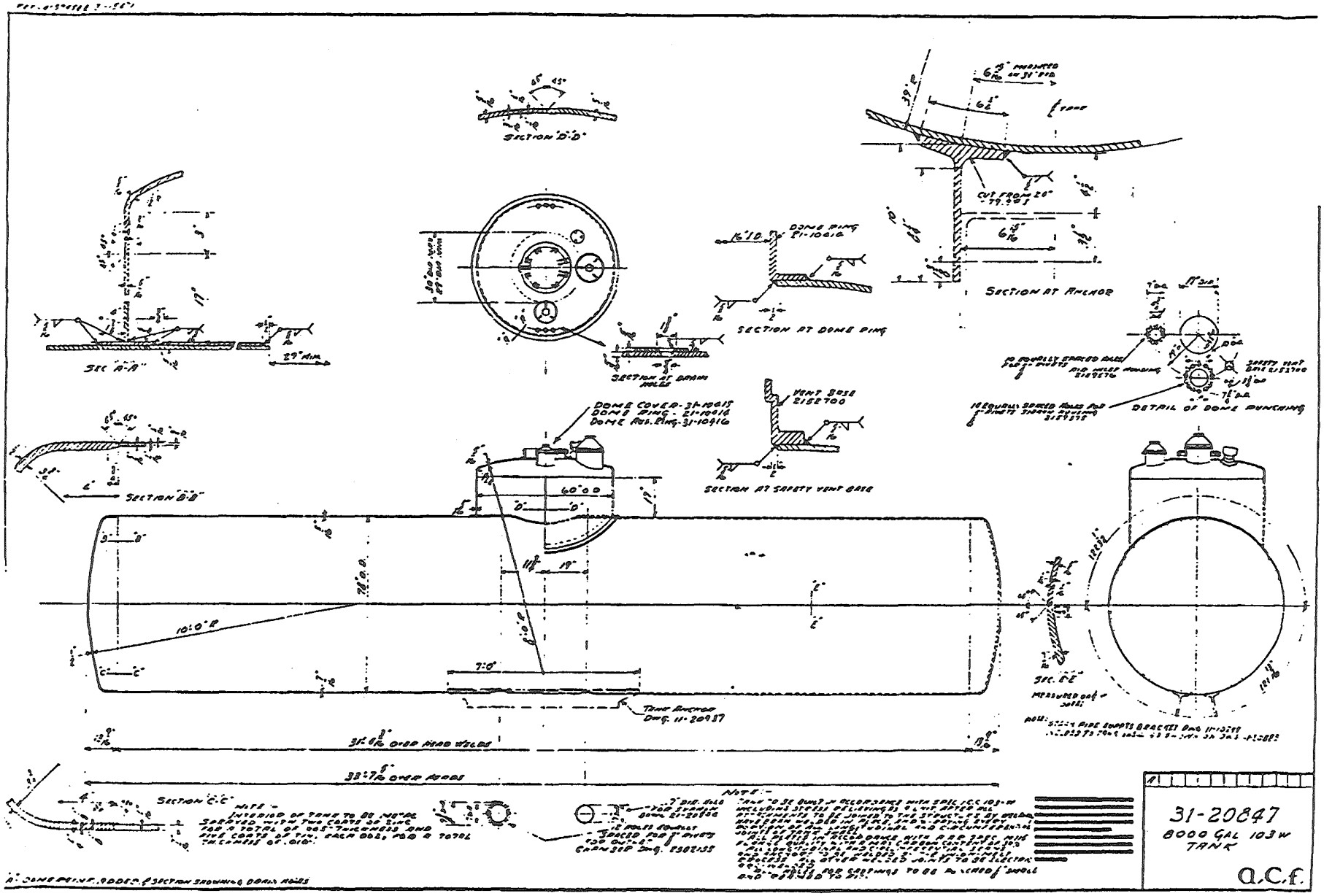
Monsanto Chemical Company

DWG. NO. 9C-8278

NEV 007431

NEV 007432






NEV 007434

Monsanto

# Monsanto ASKAREL

# Inspection and Maintenance Guide

Monsanto

# ASKAREL

## Inspection and Maintenance Guide

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## SECTION C



### Appendix

## SECTION A
## TRANSFORMER ASKAREL

### I. INTRODUCTION:

This manual describes the operating characteristics of transformer *askarel liquid* insulation and how it differs from mineral oil.

The information given is based on facts gathered by Monsanto over 30 years as producer of askarel, plus knowledge gained from the experience of transformer manufacturers and users. This guide is provided to outline the very simple maintenance required for askarel fluid in "modern" transformers and to offer suggestions for the sealing and maintaining of askarel in old units. By following this guide, users will obtain maximum service from askarel insulation with a reasonable minimum of maintenance. If questions arise relating to the designing and building of transformers — these should be referred to regular transformer suppliers.

Monsanto gratefully acknowledges the assistance, guidance, and the contributions of certain data by the following:

Frank M. Clark.....General Electric
Edward L. Raab.....General Electric
James G. Ford.....Westinghouse Electric
George Shombert, Jr.....Allis Chalmers

### II. HISTORY OF TRADE NAME TYPES

"Askarel" is the generic name for non-combustible (fire-resistant) liquid insulation. In this respect, the insulation is completely different from ordinary transformer mineral oils. Transformer askarel is marketed by Monsanto. Whatever the trademarked brand, the askarel contains Monsanto's Aroclor (chlorobiphenyl) . . . one of the best liquid insulations developed by science. This inert compound is chemically stable, fire-resistant, heat stable, non-corrosive, and has high dielectric strength under the operating conditions encountered in transformers.

Askarel liquid insulation is made by thinning Aroclor 1260 with trichlorobenzene or tri-, tetrachlorobenzene mix. The first transformer askarel was made in 1932, in accordance with General Electric Company's patents, and was trademarked Pyranol 1488. Westinghouse, too, offered this askarel insulation in 1936 under their trademark Inerteen.

In the mid-1940's, as shown in the following table, tin tetraphenyl was added to General Electric's Pyranol to scavenge hydrogen chloride. Shortly thereafter, Westinghouse added phenoxy propene oxide to Inerteen for the same purpose. In 1963, General Electric replaced tin tetraphenyl with a diepoxide scavenger. This new formula, which replaces previous Pyranols, is called Transformer Pyranol A13B3B. *Thus, today the two commercial types of formulations are General Electric's Transformer Pyranol A13B3B type and Westinghouse Inerteen PPO (7336-9) type.*

The composition changes made in transformer askarel formulations are shown in Table I.

Table I

THE COMPOSITION OF TRANSFORMER ASKARELS

| | Pyranol | | | | | Inerteen PPO |
|---|---|---|---|---|---|---|
| | 1488 | 1467 | 1470 | A13B3B | Inerteen | (7336-9) |
| Year Introduced..... | 1932 | 1944 | 1952 | 1963 | 1936 | 1945 |
| Ingredients: | | | | | | |
| Aroclor 1260..... | 60 | 60 | 45 | 45 | 60 | 60 |
| Trichlorobenzene..... | 40 | 40 | 40 | 40 | 40 | 40 |
| Tetrachlorobenzene..... | | | 15 | 15 | | |
| Tin tetraphenyl..... | | 0.125 | 0.125 | | | |
| Phenoxy propene oxide..... | | | | | | 0.20 |
| Diepoxide..... | | | | 0.125 | | |

## OTHER BRAND NAMES

Various electrical equipment manufacturers use other trade-marked names for askarel liquid insulation, such as Chlorextol (Allis-Chalmers); Noflamol (Wagner Electric); Saf-T-Kuhl (Kuhlman Electric). These askarel insulating liquids are one or the other of the two standard formulations. Still other manufacturers, who designate their insulation only by its generic name, askarel, assign it a number or code. By this number, Monsanto knows whether to furnish Pyranol A13B3B or Inerteen PPO (7336-9) type formulation to the user.

## III. OFFICIAL TRANSFORMER ASKAREL SHIPPING SPECIFICATIONS

The official shipping specifications for the two modern transformer askarel formulations are shown in Table II.

Table II

## OFFICIAL TRANSFORMER ASKAREL SHIPPING SPECIFICATIONS

| Typical Properties | General Electric Co. Transformer Pyranol A13B3B | Westinghouse Transformer Inerteen PPO (7336-9) |
|---|---|---|
| Color, APHA | 150 max. | 150 max. |
| Condition | Clear | Clear |
| Water content, ppm (ASTM D1533-60) | 30 max. | 30 max. |
| Acidity, mg. KOH/g. (ASTM D974-55) | 0.014 max. | 0.014 max. |
| Dielectric Strength, 25°C., 0.1 inch gap (ASTM D877-49) | 35 KV, min. | 35 KV, min. |
| Dielectric Constant, 100°C., 1 KC (ASTM D924-49) | 3.8 to 4.3 | 3.7 to 4.0 |
| Volume Resistivity, 100°C., 500 volts DC 0.1 inch gap, $10^9$ ohm-cm. | 100 | 100 |
| Inorganic chlorides, ppm. (ASTM D1821 and G.E. Method E4C41B) | 0.10 max. | 0.10 max. |
| Refractive index, 25°C. (ASTM D901-56) | 1.6075 to 1.6085 | 1.6137 to 1.6147 |
| Viscosity at 37.8°C. (ASTM D88-56) Saybolt Universal Seconds | 41 to 45 | 54 ± 2 |
| Pour point °C. (ASTM D-97-57) | —44 or lower | —32 or lower |
| Specific gravity 15.5/15.5°C. (ASTM D287) | 1.560 to 1.571 | 1.560 to 1.568 |
| Burn point (ASTM D92) | None to boiling | None to boiling |
| Distillation range (ASTM D20-56) corrected for stem and barometric pressure | 1st drop 210°C. min.<br>35% 240 to 256<br>55% 290 to 330<br>65% 385 to 400<br>95% 395 to 415 | 1st drop 200°C. min.<br>40% below 270°C.<br>90% 395 to 415°C. |
| Fixed chlorine | 60.5 ± 5% | 59.1% min. |
| Corrosion test | After heating with aluminum for 6 hrs. at 200 to 220°C., the aluminum must not be corroded on either visual or weight inspection and the askarel should meet the following specifications: | |
| Color, APHA | 200 max. | 200 max. |
| Acidity, mg. KOH/g. | 0.014 max. | 0.014 max. |
| Inorganic chlorides ppm. | 2.0 (tent.) | 2.0 max. |
| Condition | Clear | Clear |
| Scavenger content | 0.115 to 0.135% Diepoxide | 0.18 to 0.22% phenoxy propene oxide |
| Arc formed gases | Less than 1.0% of total combustible gases including carbon monoxide, hydrogen and volatile hydrocarbons. | |
| Coefficient of Thermal Expansion (ASTM D1903), CC./CC./°C. | 0.0007 | 0.0007 |

The above two formulas are used in modern transformers. There are a number of units in service dating back to 1933 with askarels of somewhat different compositions. These will be compatible with the above modern formulations.

## IV. INTERCHANGEABILITY AND STABILITY

### A. Interchangeability:

The two general types of askarel insulation shown in Table II can be either mixed or interchanged and there will be no difference in the operation of the transformer. However, askarel insulation *must never be mixed* with mineral oil.

Over 2% mineral oil in askarel begins to lower its fire resistance. Further, materials of construction in the transformer that are compatible with askarel may not be compatible with oil and vice-versa. For example: a significant amount of askarel in a transformer built for mineral oil will attack gaskets, adhesives, core bindings, impregnating varnishes, etc.

### B. Stability:

Askarel liquid insulation is highly pure, fire-proof liquid made under close chemical control. It does not vary in composition like the commercial range of mineral oils.

Askarel does not deteriorate when exposed to air, heat, hot metal; it does not break down over long use to form conducting or corrosive chemicals; it does not oxidize or sludge. Askarel will remain perfectly stable year after year unless broken down by exposure to severe arcing.

The only real "enemy" of askarel is *contamination by water*. Keeping askarel water-free will insure long-time service.

Askarel is heavier than water. If water gets into askarel insulation, only a tiny amount (125 ppm) dissolves — the rest floats on top.

## V. DIRECTIONS FOR HANDLING

### A. Keep Dry:

In handling, storing, sampling, inspecting askarel — and in operating askarel transformers — take every precaution to guard the askarel insulation from exposure to high humidity and moisture contamination. Keep 5, 30, or 55 gallon drums of askarel dry; lay stored drums on their sides with the bung at the highest point from floor to keep water off the drum head (which can be sucked into the askarel by the drum "breathing"). This precaution is not necessary when drums are stored indoors, which is the preferred way of storing.

### B. Use Ordinary Personal Precautions:

Transformer askarel has been made, handled, and used for over 30 years without causing toxic or other ill effects. It can be handled with only minor precautions. If accidentally spilled on hands, no serious skin irritation will occur. However, liquid askarel has a solvent action (similar to paint thinner) on the fats and oils of the skin and prolonged contact may lead to drying and chapping of the skin.

In case of contact, wash off the skin with soap and water; remove and dry clean saturated clothing. Clean up spills with rags, sawdust or absorbent clay. Eye contact may result in painful irritation but no permanent damage to tissues. If askarel gets in the eyes, flush with large amounts of water. As with all eye first-aid, refer to a physician. To relieve irritation, physicians have used a 1% Pontocaine as well as opthalmic cortisone acetate solution, or castor oil.

Infrequent exposure to askarel vapors will not cause ill effects. However, prolonged exposure to high vapor concentrations should be avoided. If hot askarel must be handled in a closed or confined area, provide the area with ordinary exhaust ventilation — or — wear an organic cartridge respirator approved by the U. S. Bureau of Mines.

## VI. EXPECTED SERVICE LIFE

Properly designed and installed askarel transformers will give trouble-free service for a minimum of 30 years. Since their introduction in 1932, the manufacturers report finding the over-all failure rate to be less than 0.5% for all units under test and service conditions. The Edison Electric Institute's report (1956-1958) on their member utilities publishes the failure rate for askarel transformers as 0.13 per hundred banks per year. Even the rare reports of failure are invariably found to be due to improper sealing that allows moisture to enter.

## VII. DIELECTRIC STRENGTH — MOISTURE RELATIONSHIP

The dielectric strength of askarel is highly sensitive to excess moisture; not sensitive to ordinary dissolved contaminants. While the dielectric strength can also be lowered by severe arcing, askarel turns noticeably black or has particles of sooty carbon floating in it if arcing has occurred. Then the transformer should be repaired and the askarel replaced.

New askarel has a minimum dielectric strength of 35 KV at 25°; a maximum moisture content of 30 ppm. If the dielectric strength is checked periodically and decreases significantly — this indicates moisture pick-up, arcing, or both. When the dielectric strength has dropped to 26 KV or less, an analysis for water is necessary. If water is found in excess of 100 ppm at 25° C., its source should be located and corrections made.

When the moisture content approaches 125 ppm (saturation level), the dielectric strength of askarel drops below the value required for efficient insulating. The moisture content should not be allowed to rise over 70 ppm; it should be held as near as possible to 30 ppm. Maintaining a low moisture level will assure high dielectric strength and top operating efficiency.

Table III shows the relationship of dielectric strength vs. moisture and Table IV indicates the approximate water solubility limits in askarel and mineral oil.

Table III

RELATION OF "BREAKDOWN STRENGTH" TO AMOUNT OF DISSOLVED WATER IN ASKAREL AND MINERAL OIL

| Water Content (PPM) | Breakdown Voltage (ASTM) Askarel | Mineral Oil |
|---|---|---|
| 0 | 70 KV | 50 KV |
| 20 | 55 | 39 |
| 40 | 47 | 30 |
| 60 | 40 | 26 |
| 80 | 38 | 22 |
| 110 | 10 | 5 |

Table IV

APPROXIMATE SOLUBILITY OF WATER IN TRANSFORMER ASKAREL AND MINERAL OIL

| Temperature °C. | °F. | Amount of Water (PPM) Dissolved Askarel | Mineral Oil |
|---|---|---|---|
| —30 | —22 | 8 | 8 |
| —20 | — 4 | 16 | 10 |
| —10 | 14 | 28 | 13 |
| 0 | 32 | 41 | 20 |
| 10 | 50 | 65 | 33 |
| 20 | 68 | 94 | 58 |
| 30 | 86 | 128 | 85 |
| 40 | 104 | 170 | 130 |

### TURBIDITY

. . . may be the visual sign of undissolved water, or may indicate contamination from core materials, dirt, or deteriorating construction materials. Cloudiness may also result from cold precipitation of tin tetraphenyl "scavenger" that was used in the earlier Pyranols. This scavenger begins to come out of solution around 15°F. above zero. To redissolve it requires heating to 150 - 200°F. and agitation.

High dielectric strength will quickly indicate that any turbidity present is not moisture; that the insulating efficiency of the askarel is still excellent. However, if the dielectric strength is below 26 KV, moisture should be determined, using the Karl Fischer method (ASTM D1533-60).

*The dielectric strength test for askarel serves primarily as an indicator for moisture. It is by far the most important maintenance test for transformer askarel.*

## VIII. TYPICAL VALUES FOUND IN ASKAREL FLUID UNDER VARIOUS USE CONDITIONS

Table V gives the entire spectrum of properties that are typical for freshly-made transformer askarel as it goes through the various normal and abnormal conditions of use.

The data in Table V are in terms of *only* the askarel *fluid* and do not refer to insulation resistance or power factor measurements on the over-all transformer insulation.

Several utilities studying the power factor values of the over-all transformer insulation system indicate that the unit power factor of a new askarel transformer should range from 1% *to not over* 5%. This would generally correspond with the askarel *fluid* properties as given under heading "B".

Table V

TYPICAL VALUES FOUND IN ASKAREL FLUID UNDER VARIOUS CONDITIONS OF USE

| | A | B | C | D | E | F |
|---|---|---|---|---|---|---|
| | New askarel | Askarel in properly* built new or rebuilt units prior to use. | After normal operation; a survey of 50 units, most 2-5 yrs. old; some 10. | Non-arced but slightly contaminated askarel in improperly* built units, new or slightly used. | Same askarel as in Column D — after refining. | Heavily arced askarel due to neglect and internal electrical malfunction. |
| **Inspection Check Points** | | | | | | |
| Color | Light straw | Light straw | Light straw | Can have foreign shades, blue, green, red cast. | Foreign shades remain showing extraction of oil soluble color. | Black |
| Clarity | Clear, free from particles | Clear, practically free from particles | Clear, trace of particles | Clear, slight amount of particles | Clear, free from particles | Contains carbon particles |
| Moisture 25°C. | 30 ppm | 30 ppm | 10-70 ppm all dissolved water | 20-100 ppm all dissolved water | 30 ppm | Near 125ppm saturation level — plus possible undissolved water |
| Dielectric Strength 25°C., 0.1" gap | 35 KV minimum | 35-48 KV | 35-45 KV | 30-42 KV | 40-45 KV | 5-15 KV |
| Volume Resistivity 100°C., 500 Volts, DC., 0.1" gap | 100 x $10^9$ ohm-cm. minimum usually 500-1,500 | 40 x $10^9$ ohm-cm. minimum usually at least 100 x $10^9$ | 15-400 x $10^9$ ohm-cm. | about 5 x $10^9$ ohm-cm. | at least 1,500 x $10^9$ ohm-cm. | about 3 x $10^9$ ohm-cm. |
| Power Factor: | | | | | | |
| 100°C. 60 cycles | 2-5% | 10-25% | 10 to near 80% | 30 to 100% | 2-5% | 100% |
| 25°C. 60 cycles | 0.05-0.1% | 0.5-2% | 0.5 to near 10% | about 7 to over 15% | 0.1-0.2% | at least 25% |

**A properly built transformer is defined as one in which the materials of construction are chemically and electrically compatible with askarel.*

Starting ideally with situation "*B*", *it is normal to expect the power factor of the fluid in a satisfactorily operating askarel transformer to increase as shown under heading* "*C*". The "over-all" power factor of the *transformer* in normal service is expected to rise, but preferably not much beyond 5%. In this normal situation, the moisture level of the askarel fluid and its dielectric strength will be satisfactory, as shown in *heading* "*C*".

Occasionally the power factor of a non-arced, satisfactorily-operating askarel transformer is found to be relatively high, i.e., 15%. *In this case, the power factor of the askarel fluid will also be high*, perhaps as high as 50% at 20°C. and 60 cycles.

It is not good practice merely to note that high power factor of the askarel fluid is to be expected. The important step is to check dielectric strength and note whether there has been a downward trend. A downward trend in dielectric strength very likely indicates moisture entrance and should be confirmed by a Karl Fischer test for water.

The importance of any abnormalities in dielectric strength and moisture content of the askarel fluid cannot be overemphasized! Where abnormal values for the dielectric strength or moisture content occur, the power factor of the transformer can be expected to be abnormally high; the expected high power factor of the askarel fluid will induce this.

The indication that a high power factor on the transformer and on the fluid is due to contamination can be verified by earth refining the askarel fluid and noting whether after refining the test results correspond with heading "*E*".

Assuming no mechanical defect or arcing, if the power factor of a *new askarel transformer* is relatively high, the power factor of the *fluid* will also be high, giving the situation under heading "*D*". This reflects contamination that should have been removed by the manufacturer of the transformer.

In absence of mechanical defect, where there are no abnormal losses in dielectric strength, and no moisture pick-up in non-arced askarel — the use history shows that a relatively high power factor for the askarel fluid (as compared with mineral oil) is to be expected. Normally, high power factor is due to contamination and not deterioration of the askarel operation.

As shown by F. M. Clark in his book, "*Insulating Materials For Design and Engineering Practice*," John Wiley & Sons, N. Y., 1962 — activated alumina can be used in the circulating system to bring the power factor down and keep it as low as possible.

## IX. CHECK POINTS FOR MAINTAINING ASKAREL INSULATION

### A. General Considerations:

Modern askarel transformers with welded construction or silicone gaskets for hand hole-cover, switch and terminal compartment covers, with properly constructed bushings require little or no maintenance. With properly constructed transformers, annual or semi-annual visual inspection and dielectric strength test of the askarel fluid should suffice for routine maintenance checking over many years of service.

However, many askarel units were installed in the early 1930's — before the development of some of the better modern gasketing materials and before improved designs were developed for sealing out moisture. Such early units should be, and can be, modernized. Leaky or deteriorated gaskets should be replaced. If the askarel has become contaminated, it should be reconditioned. At the same time, a general clean-up of the unit and possible refinishing may be desirable.

If it is not convenient to take an old transformer out of service for general repairs, leaky gaskets can be sealed temporarily by painting over the leaky area with epoxy cement.

A survey of users indicates a good number of early-built askarel transformers (over 20 years old) are kept in continuous service in critical installations by the following steps (instead of modernization).

The operating units are equipped with compound pressure gauges for reading pressure above and below atmospheric. Positive pressure is maintained on the shell by introducing nitrogen at 2 to 3 pounds above atomspheric. Regular workmen in the area daily record the temperature and pressure. If a sudend pressure drop is noted more nitrogen is introduced and the gaskets are checked for leaks with soap solution. Leaks are sealed by applying epoxy cement.

### B. Modern Sealing Procedures:

Transformer purchasers should specify the following modern techniques for sealing:

1. **Welding Construction:** Covers, radiator connections, switch and terminal housings, instrument connections, etc. should be welded.

2. **Bushing Connections:** The most satisfactory bushings are the type with rolled-on flanges and two ring seals rolled into a depression in the porcelain — sealed with silicone rubber rings held under compression. Metal-to-glass or metal-to-porcelain sealed bushings are also satisfactory.

   If for any reason the above type bushings cannot be used then use a porcelain or glass bushing with a silicone or Viton gasket retained in a groove. The gasket can be either rectangular or circular cross section, usually ¼ inch thick.

3. **Small Size Connections:** When not possible to weld, small size connection seals should be made with Flexitallic stainless steel rings. The surfaces must be machined and parallel. The filler between the steel laminations of the Flexitallic ring should be either silicone or Viton.

4. **Gaskets For Hand-Hole Covers:** Modern design specifies silicone gaskets. Such gaskets must be retained in a groove. The groove preferably is machined into the flange or cover. However, it can also be formed by welding concentric steel strips to the flange or the cover.

   Generally the gaskets should be 5/16 to ½ inch thick for covers, depending on the depth of the groove. A rectangular section is usually used.

   The silicone material should be Dow-Corning No. 50 Silastic or equivalent. This is a low compression set material. For best sealing 20 to 25% compression is recommended, with ample clearance in the groove to allow for this compression.

   No cement is required. With reasonable care the gasket is removable without damage and is reusable.

   Silastic 50 is slightly swelled by askarel which contributes to the tightness of the seal. It is not deteriorated by askarel fluid or vapors. It resists weathering and it is thermally stable and flexible at all operating temperatures. It is an excellent moisture barrier.

Notes: a. Dow-Corning, Midland, Michigan will supply a list of Silastic 50 gasket fabricators to all transformer manufacturers or users. They will also furnish technical data.

b. Any user of Pyranol transformers, made by General Electric Company at Rome, Ga., will receive prompt and generous help for converting to the modern silicone or Viton gaskets by contacting the General Electric Company's Service Engineering Department at Rome, Ga.

See Appendix K: Seals, Properties and Procurements.

**C. The Older Sealing Arrangements:**

The older type gaskets consist of either cork or cork-nitrile rubber combinations or straight nitrile rubber.

1. **Cork-Nitrile Combinations:** Covers for the main tank, hand-holes, switch and terminal chambers, relief diaphrams etc. are held in place by studs welded to the flange or by bolts. The gaskets are cut with openings and placed over the bolts. Often Shellac (Westinghouse Style No. 1150419, or General Electric Company's Glyptal 1276) is used to cement the cork to the flanges.

   The following is recommended for sealing with the cork — nitrile rubber combination:

   Use Armstrong NC-757 cork-nitrile material or equivalent. The gasket can be cut from a single sheet or by scarfing strips of the material. A convenient method for joining strips is to make a Keystone Type joint. For this, Westinghouse, Sharon, Pa., offers their gasket cutter Style No. 328 B614 G01, (about $15).

   The joints should be cemented and the gasket also cemented to the flange, using one of the above cements. Excess cement should not be allowed to reach the interior of the transformer.

After installation and bolting, the outside edge of the gasket should be coated thoroughly with epoxy cement to increase weather resistance.

This epoxy cement is a paste to which a curing catalyst is added immediately before use. Typical are:

a. Epoxy Patch Kit #1-C
Hysol Corporation, Olean, N. Y.

b. Scotchcast Resin #4
Minnesota Mining and Manufacturing Co.
St. Paul, Minnesota

c. Adhesive A-1 and Activator Type B
Armstrong Products Company
Argonne Rd., Warsaw, Ind.

d. Adhesive 9860-1, Synthetics Organic Company, Cleveland, Ohio, used with activator diethylene triamine (Carbide and Carbon Chem. Co.)

2. **Straight Nitrile Rubber:** When straight nitrile rubber was originally used, invariably the gasket was recessed in a groove. This was to prevent gasket flow and to protect the material against excessive compression. Although this type seal was not cemented, the nitrile rubber gasket is not reuseable.

Since grooves or stops have already been provided for the nitrile rubber seal, Silastic 50 can be easily substituted and is recommended. This conforms with modern practice.

## X. PERIODIC FLUID INSPECTION AND WHAT CHECKPOINTS MEAN

On a regular schedule — at six, nine, or twelve month intervals — make a simple visual inspection of your askarel insulation and run a dielectric strength check.

**A. Visual Inspection:**

Askarel is a clear, faint-yellow liquid. After long-term use this color may gradually intensify to light brown. The fluid should remain clear and free from turbidity or cloudiness.

Any color change — such as to a green, red or blue cast — indicates extraction of impurities from the solid insulation. If a distinct foreign color pick-up is noted, check the complete range of electrical characteristics and notify the transformer maker. The electrical characteristics may be found to be unimpaired. Color change alone (except for black) is not a danger signal since the contamination is not likely to impair the dielectric strength.

**B. Dielectric Strength:**

If the dielectric strength has decreased significantly from the last inspection, or if it has gradually decreased below 26 KV range (at 25°C.) — RUN A CHECK FOR MOISTURE. Use ASTM D901, D877 (Karl Fischer Method).

The dielectric strength of askarel is the major indicator to the operating efficiency of your liquid insulation and of the askarel transformer itself. Besides the "visual" inspection tests, dielectric strength is the only test necessary to run on a routine basis. Well-sealed askarel transformers have service records of 25 to 30 years on the original askarel. New askarel has a minimum dielectric strength of 35 KV at 35°C., a maximum moisture content of 30 ppm.

## XI. INSPECTION CHECK LIST

1. If askarel is clear, even though darkened to light brown; has no sediment or turbidity; has dielectric strength over 26 KV . . . give it the inspection "OK".
2. If askarel is clear; has foreign color of blue, green, red . . . it is "extracting color" from internal materials. This is not, of itself, an operating hazard when the dielectric strength stays over 26 KV and moisture remains low. However, this rare occurrence calls for checking into condition of the interior construction and consulting the transformer maker.
3. If askarel is clear; but dielectric strength drops to 22 or lower KV, and moisture rises over 80 ppm . . . the askarel is ready for simple "refining". If the moisture is near the saturation level (about 125 ppm), a thorough inspection should be made for water droplets in the transformer tank, and even for "globules" or water floating on the askarel surface. If found, the transformer manufacturer should be consulted for reconditioning both the transformer and the fluid.
4. If askarel is dark brown to black; if black particles of carbon are seen; and dielectric strength is low . . . the askarel has been broken down by arcing. It cannot be refined and should be discarded.

If any of these four simple inspection tests appear out of the ordinary, or the relationship between appearance and test values is abnormal — contact your transformer supplier for a complete analysis.

Whenever a sample is to be shipped to Monsanto, please follow the directions shown under: "SAMPLING ASKAREL".

## XII. CONTAMINATION IN TRANSFORMERS

Unlike mineral oil which can oxidize, sludge and deteriorate — askarel breaks down in transformer use *only when strongly arced.*

Thus, while askarel does not decompose in normal use — it can be contaminated more readily than the relatively non-polar mineral oil.

For example the following Table VI shows how a small amount of synthetic rubber or a bit of varnished cloth markedly increases the power factor of the askarel. Please note, however, that *such minor contamination has no adverse effect on the dielectric strength.* As explained, moisture entrance through faulty seals seems the only contaminant in normal use that lowers the dielectric strength of transformer askarel.

Table VI

EFFECT OF COMMON INSULATION MATERIALS ON POWER FACTOR AND DIELECTRIC STRENGTH (HEAT AGED 96 HOURS IN ASKAREL AT 100°C.)

| Material Immersed | Askarel After Exposure: Power Factor, Percent at 60 cyc., 100°C. | Askarel After Exposure: Dielectric Strength 25°C. |
|---|---|---|
| None (control) | 1.0 | 35 KV |
| Black varnished cloth | 85.0 | 42 |
| Copper | 1.5 | 40 |
| Pressboard | 2.0 | 37 |
| Manila paper | 1.5 | 39 |
| Phenol formaldehyde resins | 1.6 | 41 |
| Shellac | 6.0 | 36 |
| Iron | 5.0 | 39 |
| Synthetic rubber | 70.0 | 39 |

Similarly, trace contaminants from commonly used construction materials can lower the volume - resistivity of askarel, without affecting its dielectric strength. Table VII shows this.

Table VII

EFFECT OF COMMON INSULATION MATERIALS ON VOLUME RESISTIVITY OF ASKAREL

| Sample | Volume Resistivity x $10^9$ ohm-cm (at 100°C., 500 Volts DC., 0.1" gap) |
|---|---|
| 1. New askarel before heat aging | 2,000 |
| 2. New askarel after heat aging 96 hours at 100°C. | 1,900 |
| 3. After heat aging with 1 sq. inch specimens of: | |
| a. Phenolic resin tap changer material | 1,200 |
| b. Paper | 750 |
| c. Grade A press board (tan) | 500 |
| d. Grade A press board (gray) | 500 |
| e. Grade A press board, laminated strip | 400 |
| f. Cotton wrapping | 300 |
| g. Glyptal 1276 cement, cured 48 hrs. at 110°C. | 100 |

While trace contamination easily lowers volume resistivity from high levels, it is important as previously noted in Table V Case F, that heavy contamination (as when arced) does not lower the resistivity below the order of $10^9$ ohm-cm. at 100°C.

The different behavior of askarel vs. mineral oil in these respects can be summarized as follows:

High power factor and low volume resistivity in transformer mineral oils are commonly regarded as "danger signals" that the oil has deteriorated and broken down chemically.

This is NOT TRUE of askarel liquid insulation, unless the dielectric strength is low.

## XIII. REFINING ASKAREL FOR RE-USE

### A. Filtering Through Dry Blotter Paper to Remove Moisture and Extraneous Particles:

Most operators prefer portable refining apparatus, such as a plate press fitted with a dolly, (available from Sparkler, Mundelein, Ill., Westinghouse Maintenance and Repair Dept., Chicago, or General Electric Co., Pittsfield, Mass.); or the earthen cartridge filter type, (available from Industrial Filter Corp., Lebanon, Ind.). Filter paper liners for the plate press are available from Carl Schleicher and Schuell Company, Keane, New Hampshire and manufacturers of filter presses listed above.

The filter paper must be dried immediately before use. For best results, spread the paper for maximum surface exposure in a hot air circulating oven and heat it for 4 to 6 hours at 110°C.

If possible do not take the transformer out of service until ready to filter the fluid. This will keep the transformer coils relatively hot and dry. Processing the fluid should start immediately after de-energizing the unit.

Circulate the askarel hot (but not over 55-60°C.) through the filter fitted with the dry paper liners.

After filtration the dielectric strength of the askarel should be 35 KV minimum.

1. Precautions: Filtering should not be done when the relative humidity exceeds 75%.
2. Any flexible hoses and gaskets on the refining equipment should be lined with or made of materials that will not be softened by contact with askarel fluid. (Silicone or Teflon-lined, or flexible metal materials are suitable.)

**Table VIII**

**GUIDE TO RATE OF DISSOLVED WATER REMOVAL BY FILTERING ASKAREL THROUGH A PAPER PRESS**

| Passes Through Paper Press | Water In Askarel PPM |
|---|---|
| 0 | 115 |
| 1 | 35 |
| 2 | 22 |
| 3 | 18 |
| 4 | 12 |
| 5 | 10 |
| 6 | 10 |

**B. Earth Treatment for Maximum Improvement of Power Factor and Volume-Resistivity:**

**1. Procedure:**

(The askarel liquid should be relatively dry prior to the following earth filtration.)

Use finely divided Attapulgus clay or Fuller's earth (dried and activated by heating for 12 hours at 300-350°F. immediately prior to use) as a coating on the filter paper surface. The amount of earth used should be 0.1 to 0.2 per cent by weight on the weight of the askarel to be treated. (Askarel weighs about 13 pounds per gallon).

To deposit the earth evenly, stir one-third of the earth with a small portion of askarel in a clean container. Pump the mixture through the filter and follow with two more one-third portions. Then circulate askarel taken from near the top of the transformer, pass it hot (not over 55-60°C.) through the earth coated filter and feed back through the bottom transformer outlet. Continue circulation until the fluid is clear and test shows that the electrical properties are fully restored.

**2. Effect of Earth on Removal of Scavengers:**

Only slight and insignificant loss (by selective absorption) of tintetraphenyl and epoxides occurs when askarel is refined by treatment with 0.1 to 0.2 per cent by weight of earth. To remove significant amounts of the scavengers requires repetitious treatment with much larger amounts of earth.

**3. Table IX — Approximate Relationship Between Power Factor, Volume-Resistivity and Dielectric Strength of Transformer Askarel:**

| Power Factor (60 cyc.) | | Volume Resistivity x $10^9$ ohm-cm. (at 100°C., 500 Volts DC., 0.1" gap) | Dielectric Strength 25°C., 0.1" gap |
|---|---|---|---|
| 100°C. | 25°C. | | |
| 2% | 0.05% | 1500 | 35 KV |
| 5% | 0.1% | 500 | 35 |
| 15% | 0.7% | 100 | 35 |
| 20-25% | 2.0% | 60-70 | 35 |
| 40-50% | — | 25 | 35 |

If it is desired (although these factors are not generally considered important for askarel transformers), to keep the power factor as low as possible and the resistivity as high as possible, hang a container of anhydrous alumina or activated clay in the circulating system of the transformer.

## XIV. CLEANING ARCED TRANSFORMERS

If a unit has arced so that the askarel is no longer fit for use, a thorough cleaning of the unit is necessary before refilling with new askarel insulation and returning it to service*. Follow this procedure:

A. Drain out all dark, carbon-contaminated askarel. (Discard by dumping or burying where it will not contaminate a water supply.)

B. Carefully brush carbon deposits from internal parts and insulation, using a soft bristle brush making sure that insulation is not damaged.

C. Flush thoroughly using new askarel (*not* an oil, *not* a cleaning solvent).

D. Flush a second time with fresh askarel; drain; then fill to the proper level with new askarel.

E. Energize transformer to warm the fluid for 24 to 48 hours; then circulate the askarel through a filter, returning it to the unit filtered and ready for use.

* This assumes that the cause of arcing has been established and corrections made. When severe arcing occurs, major repairs are usually necessary and the unit rebuilt. This procedure can be applied for flushing out repaired units.

## XV. SAMPLING ASKAREL

Take a sample as close to the top of the liquid surface as possible. (Many large askarel transformers have a built-in sampling tube near the surface for convenient sampling). Then, to make sure that your SAMPLE truly represents your askarel insulation, take another sample from the bottom. If additional sampling tube connections are contrived on the valves for easier sampling, make the tubes of clean glass, stainless steel, aluminum or tin for rigid types; and silicone or Teflon tubing for flexible types.

Use NEW containers for the askarel sample. A new and thoroughly pre-dried small-mouth quart glass bottle fitted with a Bakelite screw cap fitted with an aluminum or tin bottle cap liner is recommended for "quick on-the-site testing." (If complete analysis is to be made, a 5-pint size sample is required).

Be sure that the new bottle does not stand open to collect dust or moisture. Rinse the sample bottle and cap lining two or three times with askarel from the transformer; then fill it. If the sample will be tested promptly, a clear glass bottle can be used. If sample is to be stored indefinitely, use an amber glass bottle or wrap clear glass with aluminum foil.

A. Select a dry day. Do not sample insulation on a warm, moist day when humidity exceeds 75%, and . . .

B. Make sure that the askarel is at least as warm as the surrounding air. (Cold liquids can condense moisture from humid air.)

C. When sampling askarel from transformers, it is best to take the sample when the unit is warm and operating at average or maximum load. Especially as a check on moisture (as reflected by a dielectric strength test), sampling the warm askarel more truly represents its condition during operation.

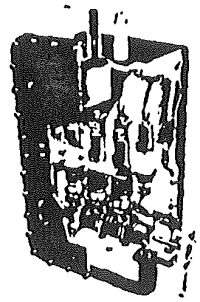
   Experience shows that water will migrate from a transformer's solid insulation to the askarel liquid and vice versa, depending on temperature. Therefore, when the transformer is hot, the moisture is most likely to be found in the liquid. This accounts largely for periodic variations in dielectric strength. For example: a relatively high dielectric strength may be found during winter months and a relatively low dielectric strength during the summer months on samples taken from the same unit.

## SECTION B

## ASKAREL FILLED SWITCHES AND TERMINAL CHAMBERS

### I. INTRODUCTION:

High voltage leads are usually connected to askarel or mineral oil-filled network transformers and power centers through terminal chambers and switches. In some cases terminal chambers are not used, and the high voltage leads are connected directly to the switch terminals. They may be filled with either askarel or mineral oil. Switches are usually rotary or drum type fitted with a revolving block and porcelain unit as the principal element arranged for three-phase service. (A cutaway view of a typical terminal chamber-switch combination is shown for reference.)



Askarel transformers with attached switches have been in use for about 30 years. When they were first introduced, the availability of insulating and gasketing materials was rather limited and even the best materials at the time had no service history. As a result, inadequate gasketing materials such as cork, nitrile rubber, and nitrile rubber-and-cork particles were used. While satisfactory for a limited period of time, these materials cannot be depended on for the expected long life of the equipment.

The terminal chamber is usually above or below the switch compartment and separated by a steel wall through which the bushings are inserted. When bushings are properly selected and correctly installed, there is no leakage from one compartment to the other. With poor bushing seals, and the terminal chamber above the switch, potting compounds or cable oil can seep into the askarel. When the terminal chamber is below the switch, askarel can drain into the terminal chamber.

### II. SOURCES OF CONTAMINATION

There are three possible sources of contamination for askarel in switches and terminal chambers; they rank in this order of frequency: (1) water entering through poor gaskets; (2) decomposition products from arcing when switch is used to break magnetizing current; (3) entrance of pothead or cable compounds through leaky bushing seals.

Unlike an askarel transformer where the amount of contaminant is likely to be very small (probably only trace amounts) in relation to the volume of askarel fluid — in switches or terminal chambers with faulty seals, the amount of contamination can be relatively large.

Experience has shown that, based on the number of installed askarel-switch units, the percentage of failures is extremely small. When investigated, it has been found that most failures originate in the switch chamber. Water is the chief source of contamination. However, heavy contamination of askarel with petrolatum and asphalt material, due to leakage, have caused a few failures.

Petrolatum is used frequently for filling terminal chambers. When either cable oil or petrolatum seeps into askarel, no great harm results. The fire resistance will be somewhat decreased and power factor of the askarel will increase with an accompanying drop in resistivity. While highly undesirable, it is doubtful that failure of the unit results. Where asphaltic compounds are used in place of petrolatum, the danger is increased somewhat because asphaltic contamination may cause excessively high dielectric losses in the askarel.

When the terminal chamber is below the switch chamber, the potting compound can be contaminated by askarel if the bushing seals are leaky. This is undesirable because the askarel will increase the power factor and conductivity of the potting compound or cable oil and develop heat from dielectric loss. If this mixture is drawn into the cable insulation, a cable failure is likely. This again emphasizes the importance of tight bushing assemblies.

## III. SEALING SWITCHES AND TERMINAL CHAMBERS

Proper bushing construction, use of Silastic seals and welding wherever possible is highly desirable (as covered in Section A). Where an elastomeric seal is to be used in contact with both askarel and petroleum oil, DuPont's Viton is suggested.

For new equipment the user should specify these modern sealing arrangements to keep out contaminants and minimize maintenance.

## IV. MAINTENANCE FOR ASKAREL FILLED SWITCHES

A. Switches used for grounding after power source has been de-energized will not undergo arcing.

B. Switches interrupting magnetizing current will be subject to arcing; the amount of decomposition will depend on power interrupted, time and frequency of operation. As a general rule, the liquid should be checked after 5 to 10 operations.

1. On newly installed switches, check the askarel at 3, 6 and 12 month intervals; if found satisfactory, check once annually thereafter. With proper attention to the gasketing of covers and bushings, experience will probably indicate that less frequent inspection is warranted.

2. Check askarel for:

   a. Dielectric strength (ASTM D877): It should be 26 KV minimum. If dielectric strength is low, confirm presence of water by Karl Fischer method ASTM D-1533. Filter to remove moisture. Dielectric strength should then be 30 KV minimum.

   b. Presence of carbon from arcing: Fluid should be relatively free of carbon. If badly arced and very black, replace fluid. If only minute amounts of carbon are present, filtration is recommended. Check power factor of liquid (should not be over 5% at 25°C. and 60 cycles). Flush out switch chamber with several gallons of fresh askarel before refilling.

3. If there is discoloration, high power factor, detectable change in specific gravity or refractive index, or if fluid flashes below 250°F., there is a possibility of seepage of potting compound into the switch compartment. In this case, correct any leaky bushing seals with proper replacements and fill with new askarel.

4. If terminal chamber is below switch, check potting compound for presence of askarel (can usually be detected by odor or by an increase in specific gravity). If askarel is present, correct any leaky bushing seals with proper replacements, and renew compounds.

5. Examine cover gaskets visually. Deterioration can be detected by swelling and cracking of the exposed edge. In cases of severe deterioration, liquid seepage is usually present.

6. Check for leakage at packing gland of switching shaft. If leaking, repack with a Silastic ring type gasket.

## SECTION C

## ANALYTICAL SERVICES ON TRANSFORMER ASKAREL AVAILABLE FROM MONSANTO

Transformer users not wishing to make their own fluid analyses can obtain the service from Monsanto. Simply contact Monsanto and specify what analyses are wanted. You will be sent the proper-sized sample container filled with fresh askarel. When you receive this, empty it and carefully take your sample (following the procedure for sampling in this guide). Send the container to Monsanto's laboratory. Charges listed include sample container, shipping, handling and laboratory costs.

### Types of Analyses Available

**Analysis 1)**

ROUTINE MAINTENANCE CHECK

Total Charge: $20.00

To determine the general condition of the fluid and find whether further analysis is necessary. (one-quart sample required)

**Properties Tested**
Color and Condition
Dielectric Strength
Moisture

You will be notified of the results of this test. If further testing is indicated, and you want a complete analysis, you will be sent a five-pint sample container. This sample will be used for the following series of tests:

**Analysis 2)**

COMPLETE ANALYSIS

Total Charge: $50.00

(a) To determine the extent of fluid contamination, (b) earth refinement to determine what degree of restoration of electrical and insulating properties is possible, (c) check test to see how the fluid re ponded to earth treatment.

a) Complete Analysis: to determine the extent of contamination

**Properties Tested**
Color and Condition
Specific Gravity
Refractive Index
Water
Free Chlorides
Acidity
Dielectric Strength
Power Factor, Dielectric Constant, and Resistivity

b) Earth Refinement Response:

Consists of treatment for 2.5 hours at 50-60°C. with 0.1 to 0.2 percent by weight of properly conditioned Attapulgus clay and then filtration through dry filter paper.

c) Analysis After Laboratory Earth Refinement:

**Properties Tested**
Refractive Index
Water
Free Chlorides
Acidity
Dielectric Strength
Power Factor, Dielectric Constant, and Resistivity

You will be notified of the results of this test series on your sample. Then after refining your entire transformer fluid fill you can check on the results by requesting the following analysis:

**Analysis 3)**

ANALYSIS AFTER EARTH REFINEMENT

Total Charge: $30.00 (This charge will not apply when analyses 1 and 2 have already been made.)

To determine whether the entire lot of the askarel fill responded to the same extent as the laboratory sample. (five-pint sample required)

**Properties Tested**
Color and Condition
Refractive Index
Water
Free Chlorides
Acidity
Dielectric Strength
Power Factor, Dielectric Constant, and Resistivity

To arrange for the tests described above write to the following address:

Paul G. Benignus
Monsanto Chemical Company
800 North Lindbergh Blvd.
St. Louis 66, Missouri

Samples to be tested should be clearly marked for identification and sent directly to:

Monsanto Chemical Company
W. G. Krummrich Laboratory
Monsanto, Illinois
Attention: R. Kuster

## APPENDIX A — Askarel Stability and Composition of Arc Formed Gas:

Askarel insulation is one of the most inert, chemically-stable heat resistant, non-corrosive liquids known. It will not break down, oxidize or sludge when exposed to air and high temperatures, 150°C. or even somewhat higher. Arcing, however, will break down the compound to liberate some hydrogen chloride and small amounts of carbon.

APPROXIMATE COMPOSITION
ARC FORMED GAS FROM TRANSFORMER ASKAREL

| Gas | Amount |
|---|---|
| carbon monoxide | 0.3 per cent |
| carbon dioxide | 0.3 |
| oxygen | 0.6 |
| inert gases | 1.5 |
| hydrogen chloride | 97.3 |

(note the absence of phosgene)

This arc-formed gas is non-flammable and non-combustible. These requirements must be met in accordance with the Underwriters' Laboratory for permission to use the term askarel.

Mineral oil evolves combustible hydrogen and hydrocarbon gases.

For all practical purposes, the amount of gas liberated from mineral oil or askarel under a given set of conditions is about 100 cubic centimeters per kilowatt-second.

## APPENDIX B

SOLUBILITY OF GAS IN
TRANSFORMER ASKAREL

| | Percent of Gas By Volume Corrected to: | | | |
|---|---|---|---|---|
| | 25°C. 760 mm | | 0°C. 760 mm | |
| | 25°C. | 100°C. | 25°C. | 100°C. |
| Carbon dioxide | 71% | 47% | — | — |
| Air | 5.7 | 4.9 | 5.8 | 5.0 |
| Nitrogen | 6.0 | 4.8 | 5.5 | 4.4 |
| Hydrogen chloride[1] | 37.8 | 50.9 | — | — |

1) In absence of scavenger

## APPENDIX C

APPROXIMATE VAPOR PRESSURE vs. TEMPERATURE FOR TRANSFORMER ASKAREL

| Temperature °C | Inerteen PPO, 7336-9 | Transformer Pyranol A13B3B |
|---|---|---|
| 40 | 1 mm Hg. | 0.9 mm Hg. |
| 60 | 5 | 3.5 |
| 80 | 9 | 8.3 |
| 100 | 30 | 18 |
| 120 | 60 | 32 |
| 140 | 90 | 53 |

## APPENDIX D

EFFECT OF TEMPERATURE ON DIELECTRIC STRENGTH OF ASKAREL

| Temperature °C. | Dielectric Strength |
|---|---|
| —60 | 67 KV |
| —40 | 63 |
| —20 | 57 |
| 0 | 55 |
| 20 | 50 |
| 40 | 50 |
| 60 | 48 |
| 80 | 45 |

## APPENDIX E

COMPARISON QF THE APPROXIMATE VISCOSITY IN SAYBOLT UNIVERSAL SECONDS OF TRANSFORMER ASKARELS AND MINERAL OIL

| Temp. °C. | Transformer Pyranol A13B3B | 10-C Mineral Oil | Inerteen PPO, 7336-9 |
|---|---|---|---|
| —20 | 1,000 | 1,000 | 2,800 |
| 0 | 100 | 150 | 195 |
| 20 | 70 | 85 | 85 |
| 40 | 45 | 49 | 50 |
| 60 | 39 | 40 | 40 |
| 80 | 34 | 34 | 36 |
| 100 | 30 | 30 | 33 |

## APPENDIX F

THE DENSITY OF INERTEEN PPO
7336-9 AND TRANSFORMER PYRANOL A13B3B
Approx. Density gm/cc.

| Temperature °C. | Inerteen PPO, 7336-9 | Transformer Pyranol A13B3B |
|---|---|---|
| 0 | 1.574 | 1.577 |
| 20 | 1.552 | 1.555 |
| 40 | 1.529 | 1.532 |
| 60 | 1.507 | 1.510 |
| 80 | 1.485 | 1.488 |

## APPENDIX G

The thermal conductivity values of transformer Pyranol A13B3B at 27°C. and 58°C. are 26.2 and 25.8 x $10^{-5}$ calories centimeters$^{-1}$, degrees centrigrade$^{-1}$, second$^{-1}$, respectively. Or, approximately 0.06 BTU per (hr.) (sq. ft.) (°F.) per foot. This same approximation applies to Inerteen PPO (7336-9).

## APPENDIX H

### Heat Capacity

Over the temperature range of 25° to 125°C. the specific heat of transformer askarel is close to 0.30 calories per gram per degree.

## APPENDIX I

### Coefficient of Expansion

The average coefficient of expansion of transformer askarel over the temperature range 20 to 100°C. is 0.0007 cc/cc/°C. One gallon would increase to 1.056 gallons on heating from 20 to 100°C.

## APPENDIX J

### Fire-Resistance

To use the generic name "askarel," the fluids must be approved by the Underwriters' Laboratories as possessing adequate fire-resistance and freedom from forming explosive gases when arced. These liquids do not have a burn point (ASTM D92-33) (Cleveland open cup method) up to about 205°C., at which temperatures they begin to boil. The significance of this is that they do not burn or support combustion under conditions encountered in transformer operation. Thus the danger of secondary explosion (or fire) is eliminated.

APPENDIX K

**Seals, Properties and Procurement**

Dow Corning Corporation, Midland, Michigan with District's at Atlanta, Boston, Chicago, Cleveland, Dallas, Los Angeles, New York City, Washington, D. C. and Toronto has available Bulletin 09-019, August 1962 entitled "Silastic Design Data". This lists the gasket fabricators throughout the country from whom the "Silastic 50" gasketing can be purchased in sheet, extrusions or molded shapes.

Generally Silastic 50 sheet goods are stocked by local die cutters, hence, could be generally purchased locally. Usually, small quantities of gaskets are die cut. If larger quantities are needed, tools are made of the same type used to cut other elastomers.

Where the gasket is extruded for fitting into a machined groove or between gasket stops, Dow-Corning advises use of a scarved joint. This joint is then cemented using Dow Corning's Silastic 140 (clear) or their RTV 731 (white) materials, which air cure.

Dow Corning points out that the local "rubber" fabricators purchase the Silastic 50 in billet form. This is worked on a roll mill in preparation for sheeting or extrusion. Then to obtain the desired physical properties the fabricator must oven cure the Silastic 50 for 24 hours at 480°F.

SPECIFICATIONS*

| | |
|---|---|
| | Color.....White |
| | Specific Gravity at 77°F. .....1.20 ± 0.02 |
| ASTM D676 — | Hardness, Shore A. Scale.....45 to 60 |
| ASTM D412 — | Tensile Strength, psi. min.....800 |
| ASTM D412 — | Elongation, percent, min.....250 |
| ASTM D395 — | Compression Set after 22 hours at 300°F., percent, max..... 30 |

* All physical properties measured on 0.075 inch thick samples molded 5 minutes at 240°F., and oven cured 24 hours at 480°F.

General Electric Company, Redmond Circle, Rome, Ga., uses silicone or DuPont's Viton wherever it is not possible or desirable to weld. For some small seals, where good matching surfaces are provided, Flexitallic stainless steel rings are used.

By contacting the Engineering Services Department of General Electric Company, Redmond Circle, Rome, Ga., users of Pyranol transformers made prior to development of these modern seals will obtain prompt assistance for conversion.

Users of askarel transformers made by other manufacturers should contact the original transformer manufacturer, or Monsanto for assistance in converting to these modern seals.

## NOTES

NOTES


MONSANTO
FUNCTIONAL FLUIDS DEPT.

800 N. LINDBERGH BLVD.
ST. LOUIS, MISSOURI 63166

The information herein regarding obtaining optimum results from askarel fluids in your transformer has been accumulated by Monsanto for over 30 years from the experience of makers and users of askarel transformers and it is believed will be helpful.

Nothing herein shall be construed as applying to other than askarel insulation. Data and maintenance suggestions herein do not apply to the other components of the transformer. All operating and maintenance suggestions recommended by the manufacturer of the transformer should also be carefully followed.

Because these maintenance directions apply only to the askarel insulation, Monsanto disclaims any liability for damage to property or injury to persons arising from transformer operation.

NEV 007576

Litho in U S A

bcc: Mr. Howard Bergen - CO
Mr. Bill Maddox - NY
Mr. Fred Sturm - NY

March 8, 1961

Mr. J. C. Sullivan
O. E. Linck Company
Clifton, New Jersey

Dear Mr. Sullivan:

Mr. Howard Bergen of our company has asked that we answer your request for oral toxicity information on Aroclor products. We have acute oral data on Aroclor 1242 and Aroclor 1254. These data are 4.15 ml/kg for Aroclor 1242 and 3.10 ml/kg for Aroclor 1254, both in rats.

I do not have chronic oral toxicity information on these products since we do not consider them as advisable for use in food products. The really significant problem in handling Aroclors comes from the possibility of dermatitis through repeated skin contact and the possibility of systemic effects through repeated inhalation of vapors in excessive concentrations.

As you probably know, the American Conference of Governmental Industrial Hygienists has established a Hygienic Standard or Maximum Allowable Concentration (MAC) for an eight-hour working day for two of the Aroclor products. These data are for Aroclor 1242, 1 milligram per cubic meter and for Aroclor 1254, 0.5 milligram per cubic meter.

We suggest to customers who handle these products that care be taken to avoid extensive exposure to the unprotected skin and to excessive vapors. Monsanto and its customers have handled many millions of pounds of these products with no difficulty, keeping these simple handling precautions in mind.

If we can be of any further help, please let us know.

Sincerely,

R. Emmet Kelly, M. D.
Medical Director

JTG
REK:pjk



NEV 161141

EXHIBIT 6D

# MONSANTO CHEMICAL COMPANY

ST. LOUIS 4, MISSOURI

Monsanto

Interrogatory #4

July 25, 1956

Mr. H. W. Speicher, Administrator
Industrial Hygiene
Westinghouse Electric Corporation
East Pittsburgh, Pennsylvania

Dear Mr. Speicher:

Thank you for your letter of July 19 requesting toxicity information on several of our Aroclors and atmospheric sampling methods.

I am sending under separate cover two detailed reports published by personnel at the Kettering Laboratories entitled, "The Toxicity of the Vapors of Aroclors 1242 and 1254." The data contained herein were condensed and presented by Dr. Treon at the AIHA meeting in Philadelphia this year. It was published in the form presented in the June, 1956 issue of the "AIHA Quarterly," a copy of which I am sure you must have. I am asking Dr. Treon, by a copy of this letter, to send you a reprint of his publication if such is available at the present time.

You will note that Dr. Treon has proposed that the hygienic standard for Aroclor 1242 be tentatively established at 2 milligrams per cubic meter and that the standard for 1254 be established at 1 milligram per cubic meter. I am sorry that we do not have similar data on Aroclor 1260. However, we have no particular reason to expect that a 60 per cent chlorinated biphenyl offers any greater hazard than a 54 per cent chlorinated biphenyl.

You will note further that Dr. Treon's work discusses a modified Willson hydrocarbon apparatus used in the sampling of his test atmospheres. In a limited amount of sampling in our own plants we have used the Willson equipment as shipped to us. I have enclosed a description of the operating procedure prepared by one of our research groups which is somewhat more detailed than that provided with the instrument.

You have previously seen our complete reports on the decomposition of Pydraul when allowed to drip or flow onto a heated metal Inconel surface. The data in those reports have convinced us that there is no greater acute hazard should Pydraul accidentally contact metals at the temperatures investigated than there would be with other industrial hydraulic fluids. I do not have any data to support any conclusions as to what might occur if the Pydraul were to be used

DEPOSITION EXHIBIT 25 2-15-94 PENGAD-Bayonne, N. J.

NPC00035799

as a hydraulic fluid in making magnesium die castings. We do have a report that one plant handling large quantities of molten magnesium purposely sprayed Pydraul into the molten material primarily to see if there would be a great deal of spattering of the magnesium. The company reported to us that their tests convinced them that Pydraul could be used. Whether or not they are using Pydraul, I cannot say.

I note in rereading your letter that I have not answered your question relative to skin effects following Aroclor contact. We recommend that repeated and prolonged skin contact with any of the Aroclors be avoided. We have patch tasted several of them with negative results. In addition, reported cases of skin irritation have been surprisingly few in view of the heavy tonnage quantities of Aroclor manufactured and handled in the last twenty years. We believe, however, that care in handling and good personal hygiene is necessary to avoid irritation.

As with many chlorinated hydrocarbons, exposure to sufficient quantities over a long enough period of time could conceivably result in chloracne. In this respect, however, the Aroclors are not the potent chloracenegens, such as chlorinated naphthalenes. To our knowledge, there have never been any cases of chloracne in the electrical industry's use of these products.

If I can be of any further assistance, please let me know.

Very truly yours,

Elmer P. Wheeler
Assistant Director
Medical Department

EPW:SMB

Enclosure

cc Dr. Joseph F. Treon
The Kettering Laboratory

Mr. Paul G. Benignus
Monsanto

NPC00035800

cc Paul Benignus-GO

October 29, 1959

AIR MAIL

Mr. H. Wilbur Speicher
Administrator, Industrial Hygiene
Westinghouse Electric Corporation
East Pittsburgh, Pennsylvania

Dear Wilbur:

Thank you for your letter of October 21 inquiring about the safe use of trichlorodiphenyl and a mixture of 40% trichlorobenzene and 60% hexachlorodiphenyl.

We market trichlorodiphenyl as "Aroclor 1242". The combination of hexachlorodiphenyl and 40% trichlorobenzene is known as "Inerteen PPO". The diphenyl in this combination is our "Aroclor 1260".

The physical and chemical properties of the Aroclors mentioned are shown on the table on pages 4 and 5 of the enclosed Aroclor bulletin. Enclosed also is a reprint indicating the results of chronic toxicity studies with Aroclors 1242 and 1254. The following comments answering your questions serially are based on information contained in these bulletins.

I believe prolonged and repeated skin contact with any of the Aroclors should be avoided for two reasons. In the first place, the less chlorinated products are liquids and are excellent solvents for oils and fats in the skin as well as other organic materials. Secondly, it is possible that prolonged or repeated skin contact could lead to chloracne. I know of only two cases where such experience has developed during the long history of production and use of the Aroclors. In one case, an Aroclor was being used as a heat transfer medium in a system that allowed vapors to escape when the material was heated to 600°F. Several workmen developed "blackheads" which were found by an industrial physician but which in his words were so insignificant that the men were not aware of them nor would a general practitioner notice them. This indicates to me, however,

that sufficient exposure, whether by inhalation of vapors or skin contact, can result in chloracne which I think we must assume could be an indication of more serious systemic injury if the exposure was allowed to continue.

A second case of mild chloracne about which we have any knowledge resulted from workmen dipping their hands in the liquid Aroclor as if it were mineral oil or vegetable oil. In addition, their clothing sooner or later became impregnated with the material and thus exposure was magnified.

You asked at what temperature local exhaust ventilation should be provided. Although the distillation range for Aroclor 1242 is 325-360°C. and 385-420°C. for Aroclor 1260, I feel that good practice dictates that local exhaust ventilation be provided when any of the Aroclors are heated above 150°F. in open tanks or in any systems where the material is not in a completely enclosed system (vapor-proof tanks, kettles, pumps, piping, etc.). This may be ultra conservative because as the distillation ranges and vapor pressure curves (page 12 of the Aroclor bulletin) indicate, the loss of vapors at 150°F. should not be significant except in any work area where general ventilation or air changes do not exist.

Your next question related again to possible skin and systemic effects and I believe it is answered above.

In connection with the 40% trichlorobenzene-60% Aroclor 1260 combination, I believe the thoughts expressed above also apply. I understand that the trichlorobenzene which is used is a mixture of the 1,2,3-, 1,2,4-, and 1,3,5-isomers which will begin to distill at 205°C. Again, I believe this indicates a low vapor pressure for the solvent at room temperatures. The only reference we have to trichlorobenzene toxicity is contained in the Public Health Service Publication No. 414 entitled "The Halogenated Hydrocarbons Toxicity and Potential Dangers" by Dr. W. F. von Oettingen, U.S. Government Printing Office, 1955 (pages 297-298). Dr. von Oettingen refers to some work done in 1937 which indicated that the trichlorobenzene was less toxic than mono or dichlorobenzene. In my opinion, a reasonable MAC for this material would be 100 parts per million.

The enclosed publication on the toxicity of Aroclor 1242 and 1254 suggests an MAC for 1242 of 2 milligrams per cubic meter which is twice that recommended by the ACGIH for a chlorinated diphenyl of unstated chlorine content. In the case of 1254, the publication suggests that the MAC of 1 milligram per cubic meter "recommended tentatively for safe industrial practice by the American Conference of Governmental Industrial Hygienists" is reasonable. On the other hand, reference to Henry Smyth's Cummings Memorial Lecture of 1956 in which he discussed the basis for the then existing "hygienic standards" indicates that

Henry agrees that MACs of 1 mg/cu.m. for Arcolor 1242 and 0.5 mg/cu.m. for Arcolor 1254 are more realistic. In the absence of any chronic toxicity data on Arcolor 1260, we suggest an MAC of 0.5 mg/cu.m., the same as in the case of 1254.

I have been in one plant where these materials were used for capacitors and transformers. The care and handling and the provisions of exhaust ventilation were not in accordance with the thoughts expressed above. The only reported difficulty was some mild eye irritation when impregnation cabinets where the items were heated to 110-120°C. released vapors of trichlorobenzene and the Arcolor into the workroom when the cabinet doors were opened. In my mind, such cabinets should be ventilated to remove the vapors rather than allow them to be released into the working atmosphere.

I have been told that your company has had some 20-25 years' experience with the products discussed above. It was suggested that Mr. James B. Ford, manager of transformer manufacturing engineering at your Sharon Plant would be a source of information regarding experience with Inerteen PFO. Similarly, Mr. R. E. Marbury of your Bloomington, Indiana Plant has been associated with the use of Arcolor 1242. Perhaps you have already discussed potential exposures with these gentlemen. If so, I would be interested in learning of their practical experience with these products.

I look forward to seeing you in Pittsburgh next week.

Sincerely,

Elmer P. Wheeler
Assistant Director
Medical Department

EPW:ch
Enclosures 2

bcc: Paul Benignus

April 26, 1966

Mr. Alvin W. Crow
Quarterman Maintenance
Public Works Department
U. S. Naval Station
Kodiak, Alaska

Dear Mr. Crow:

Your letter of April 15th has been referred to me for reply.

I have enclosed a booklet which describes fire resistant transformer fluids which have the generic name "Askarel." You will note on page 2 and page 3 of the enclosed Askarel Guide that there are several commercial or trademark types of formulations, some of which are described in tables 1 and 2.

Chemically these transformer fluids are mixtures of chlorinated diphenyl and trichloro and/or tetrachlorobenzene.

The history of the manufacturing, handling and use of these products dates back approximately 30 years. In that time there have been no cases of serious illness or toxic effects when simple precautions to avoid exposures have been followed. In the case of chlorinated diphenyls, a Hygienic Guide has been published by the American Industrial Hygiene Association for the materials containing 42% and 54% chlorine. The diphenyl in Askarel is diphenyl chlorinated to 60%. The toxicity characteristics then are believed to be similar to the 54% chlorinated material. The hazard (as differentiated from toxicity) is less in handling the 60% material since it is less volatile than the materials with lower degrees of chlorination.

The vapors from transformer Askarel are somewhat toxic and should not be breathed continuously over a prolonged period of time. With the fluid at room temperature we would expect that there would be some odor of the fluid. The level or concentration which is barely detectable by smell should not be hazardous. On the other hand, if the fluid is heated and vapors which are liberated are allowed to accumulate in an



NEV 161229

unventilated room or area, the concentration could become significant. In general, people can detect a concentration in the amount of 0.5 to 1.0 mg per cubic meter of air which is regarded as the safe workroom limit for an 8-hour daily exposure (5 days a week, week in and week out).

Transformer Askarel accidentally spilled on the skin does not present an acute toxicity hazard nor will it cause serious irritation. It should be removed from the skin by washing with soap and water, however, because repeated and prolonged exposure may lead to drying and chapping of the skin because of the "solvent action" of the fluid -- which might be compared to the drying of the skin from using paint remover or many other solvents in a careless fashion.

Vapors released in high concentrations when the fluid is heated will cause eye irritation. It is pointed out on page 5 of the enclosed Askarel Guide that typical opthalmic solutions or ointments for relieving pain in the eyes may be used after flushing the eyes with large amounts of water.

Long range accumulative effects from exposure to Askarel fluids would not be expected unless workmen were exposed to high levels of vapors a number of hours a day, day in and day out or allowed the material to remain on the skin for prolonged periods of time and at frequent and repeated intervals. Excessive vapor inhallation or skin contact can lead to an effect on the liver as is the case with many chlorinated hydrocarbons. We have never heard of such effects in workers installing, repairing or servicing transformers, however. If exhaust ventilation cannot be provided to remove the vapors released by leaks, the workmen should be provided with respiratory protection equipment if repairs and servicing are required in an area where the vapors are irritating the eyes and nose or throat.

If I can be of any further assistance, please do not hesitate to let me know.

Very truly yours,

R. Emmet Kelly, M.D.
Medical Director

REK:mjb

Encl. Askarel booklet
Hygienic Guide "Chlorodiphenyls"

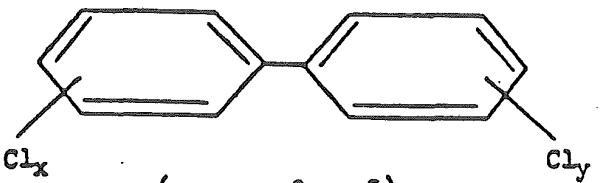
NEV 161230

Jan Feb 1965

# HYGIENIC GUIDE SERIES

## Chlorodiphenyls

### (Containing 42% and 54% Chlorine)




$(x + y = 3 \text{ or } 5)$

### Significant Physical Properties[1]

The chlorodiphenyls are light straw-colored mobile (42% chlorinated) and viscous (54% chlorinated) liquids with typical chlorinated aromatic odors. These compounds are chlorinated to specific weights of chlorine. Chlorodiphenyl (42%) contains 42.0 = 0.5 chlorine, an amount corresponding to three chlorine atoms in unassigned positions. Chlorodiphenyl (54%) contains 54.3% chlorine corresponding to five chlorine atoms in unassigned positions. Both compounds are insoluble in water, but soluble in benzene, kerosene, acetone, amyl alcohol, ether and chloroform.

| | *Chlorodiphenyl (42%)* | *Chlorodiphenyl (54%)* |
|---|---|---|
| Molecular weight: | 247.53 | 326.45 |
| Flash point (Cleveland O. C.): | 176°-180°C (319°-356°F) | None |
| Distillation range: | 325°-366°C | 363°-390°C |
| Specific gravity: | 1.381-1.392 (25°/15.5°C) | 1.495-1.505 (65°/15.5°C) |
| Vapor pressure: | | |
| 0°C | 0.001 mm | 0.00006 mm |
| 25°C | 0.006 mm (est) | 0.00054 mm (est) |
| 150°C | 4.3 mm | 1.4 mm |
| 200°C | 29.0 mm | 9.0 mm |
| At 25°C and 760 mm Hg: | | |
| Saturated air contains | 0.080 mg/liter | 0.009 mg/liter |
| 1 mg/liter = | 98.8 ppm | 74.9 ppm |
| 1 ppm = | 0.010 mg/liter | 0.013 mg/liter |

### 1. Hygienic Standards

A. RECOMMENDED MAXIMAL ATMOSPHERIC CONCENTRATIONS (8 hours): One milligram chlorodiphenyl (42%) per cubic meter of air and 0.5 milligram chlorodiphenyl (54%) per cubic meter of air.[2] This is based on the results of chronic animal inhalation studies.[3]

B. SHORT EXPOSURE TOLERANCE: Ten mg of a diphenyl of unspecified chlorine content per cubic meter of air has been reported as unbearably irritating.[4]

C. ATMOSPHERIC CONCENTRATION IMMEDIATELY HAZARDOUS TO LIFE: Not known for man. Irritation of the eyes, nose and throat at levels which have not caused acute illness preclude the likelihood of voluntary exposure to immediately hazardous concentrations.

The Committee wishes to acknowledge the preparation of the medical information section of this Guide by the Industrial Hygiene and Clinical Toxicology Committee of I. M. A., and to acknowledge also the assistance of Elmer P. Wheeler and Jack T. Garrett in the writing of this Guide.

## II. Toxic Properties

A. INHALATION: Experiments with chlorodiphenyl (42% chlorine) were run at concentrations of 8.6 μg/liter and at 6.83 μg/liter of air. Cats, mice, rabbits and rats were unaffected by the higher level while guinea pigs showed poor growth after seventeen 7-hour/day exposures over 24 days. Eighty-four 7-hour/day exposures at the lower concentration had essentially no effect on similar species of animals.[3]

In the case of chlorodiphenyl (54% chlorine) eighty-three 7-hour/day exposures over 121 days to a concentration of 5.4 μg/liter resulted in injury to liver cells and increased liver weights in rats. At a concentration of 1.5 μg/liter for one hundred and fifty 7-hour/day exposures over 213 days, the rats showed distinct microscopic changes in the liver.[3]

The literature contains many references to the potential toxic effects of chlorinated diphenyls in man and animals. The early work included investigations of chlorinated naphthalenes, chlorinated diphenyls, chlorinated diphenyl oxide and various mixtures of these. An early report indicated serious toxic effects from chlorodiphenyl chlorinated to the extent of 65%.[5] A later report by the principal author properly identified the earlier sample as a mixture of chlorinated diphenyl and chlorinated diphenyl benzene.[6] However, only a few authors of subsequent papers, bulletins or textbooks have noted this correction and the original data are cited repeatedly as relating to chlorinated diphenyl alone.

B. SKIN CONTACT: Both compounds are readily absorbed through the clipped, intact skin of rabbits. The minimum lethal dose when the undiluted materials were applied to the covered clipped, intact skin of rabbits for 24 hours was approximately 1.0 gm/kg for the 42% chlorinated and 1.5 gm/kg for the 54% chlorinated.[7]

Local action on the skin is similar to that of common organic solvents where contact leads to removal of natural fats and oils with subsequent drying and cracking of the skin.

Human cases of chloracne have not been reported from the use of these two specific chlorodiphenyls. The potential undoubtedly exists because of cases which were reported from the use of chlorodiphenyls with a higher chlorine content and from mixtures with other chlorinated aromatic compounds.[3,4,8]

C. EYE CONTACT: The liquid products and their vapors are moderately irritating to eye tissues.

D. INGESTION: The acute oral toxicities of the undiluted compounds are not great as evidenced by oral $LD_{50}$'s in rats of approximately 8.65 gm (7.61 to 9.78 gm) per kilogram for the 42% chlorinated and 11.9 gm (10.48 to 13.45 gm) per kilogram for the 54% chlorinated.[7] Central atrophy of the liver appears to be the chief toxic effect.

## III. Industrial Hygiene Practice

A. INDUSTRIAL USES: The chlorodiphenyls are used as dielectrics for condensers, capacitors and transformers, plasticizers in synthetic resins, in emulsion adhesives, as nonflammable hydraulic fluids and as transfer media.

B. EVALUATION OF EXPOSURES:

1. *Air sampling and analysis:*
   a. Direct field methods: None
   b. Laboratory methods:
      (1) Collect in secondary butyl alcohol in fritted bubbler or two large impingers in series; concentrate sample and complete analysis by one of various methods for chlorinated hydrocarbons.[4]
      (2) Sample air through combustion furnace, collect chloride ion from decomposed chlorodiphenyl in suitable alkaline solution and determine chloride ion concentration.[3]

C. HAZARDS AND THEIR RECOMMENDED CONTROL:

1. *Inhalation:* Absorption is chiefly by inhalation. Concentrations in the workroom atmosphere should be maintained below the recommended levels. Where the chlorinated diphenyls are used at room tempera-

tures, the hazard of inhalation is considered slight or absent. When these materials are subjected to elevated temperatures, the process either should be completely enclosed or other adequate mechanical exhaust ventilation must be provided to reduce concentrations to safe levels. In heat transfer media applications, the system must be designed and constructed so that it is leakproof. Special gasket materials and pump seals are available to prevent leakage. The reservoir tank should be airtight except for a vent to the outdoors. In the event of spills or leaks of hot fluids, use chemical cartridge respirators or gas masks approved by the U. S. Bureau of Mines for protection against organic vapors. These will provide good protection up to the concentrations shown on the approval labels and the odor will give ample warning if it comes through the device.

2. *Skin contact:* Operations and handling procedures should be such as to avoid the possibility of prolonged or repeated skin contact. Contaminated clothing must be laundered before reuse.
3. *Eye contact:* Eye protection should be used where there is a possibility of liquid splashes.
4. *Ingestion:* Ingestion of these materials is not a problem in industry.
5. *Fire and explosion:* The chlorodiphenyls are fire-resistant or essentially nonflammable liquids. When exposed to flame or hot surfaces they may decompose to form CO, $CO_2$, HCl, phenolics and aldehydes depending on the temperature, availability of oxygen, area of the heated surface, rate of application of the liquid to the surface and other variables.

## IV. Medical Information

A. EMERGENCY TREATMENT: Skin surfaces exposed to chlorodiphenyls should be thoroughly washed with soap and water at once. If clothing has been contaminated it should be removed promptly.

If exposure to a high vapor concentration occurs, as in the case of spills at elevated temperatures, the patient should be moved from exposure and kept at rest until seen by a physician. Oxygen should be administered if breathing is difficult.

Eyes contaminated with chlorodiphenyl should be irrigated with water for at least 15 minutes and the patient should be seen by a physician. Depending on the amount of inflammation and pain present, drugs like Cortisporin® ointment and topical Pontocaine® may be indicated.

B. SPECIAL PROCEDURES: Persons who are regularly or repeatedly exposed to chlorodiphenyls should be examined periodically to detect early evidence of skin irritation and/or liver damage. Persons with known liver disease should not be exposed to repeated contact with the chlorodiphenyls.

## V. References


1. Monsanto Company, Organic Chemicals Division: *Tech. Bulletin #PL-306, Aroclor Plasticizers* (Dec. 1960).
2. American Conference of Governmental Industrial Hygienists: Threshold Limit Values for 1964. *AMA Arch. Environ. Health 9:* 545 (1964).
3. Treon, J. F., F. P. Cleveland, J. Cappel, and R. W. Atchley: The Toxicity of the Vapors of Aroclor 1242 and Aroclor 1254. *Amer. Ind. Hyg. Assoc. Quart. 17:* 204 (1956).
4. Elkins, H. B.: *The Chemistry of Industrial Toxicology.* John Wiley and Sons, Inc., New York (1959).
5. Drinker, C. K., M. F. Warren, and G. A. Bennet: The Problem of Possible Systemic Effects from Certain Chlorinated Hydrocarbons. *J. Ind. Hyg. Toxicol. 19:* 283 (1937).
6. Drinker, C. K.: Further Observations on the Possible Systemic Toxicity of Certain of the Chlorinated Hydrocarbons. *J. Ind. Hyg. Toxicol. 21:* 155 (1939).
7. Wheeler, E. P.: Personal Communication, Monsanto Company, St. Louis, Missouri 63166.
8. Greenburg, L., M. R. Mayers, and A. R. Smith: The Systemic Effects Resulting from Exposure to Certain Chlorinated Hydrocarbons. *J. Ind. Hyg. Toxicol. 21:* 29 (1939).

## HYGIENIC GUIDES COMMITTEE

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Hygienic Guide sheets may be obtained from the American Industrial Hygiene Association, 14125 Prevost, Detroit 27, Michigan, at 25 cents each for two-page reprints; 50 cents each for four-page reprints. All orders for less than $2.00 must be prepaid. Discount of 20% on orders of five or more Guides; 40% discount on orders of 100 or more Guides. Special loose-leaf binders for the Guides may also be ordered from the Association office for $1.25 each.

# Monsanto

Monsanto Company
800 N. Lindbergh Boulevard
St. Louis, Missouri 63166
Phone: (314) 694-1000

## TOXICITY AND SAFE HANDLING OF ASKAREL

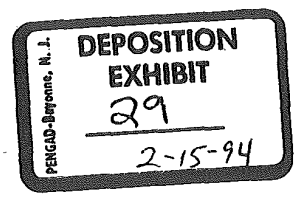
On the basis of animal toxicity studies, the Askarels may be considered only slightly toxic from the standpoint of accidental ingestion or a single accidental massive skin exposure. Similarly, single exposures to high concentrations of vapors (when heated sufficiently to volatilize) or high concentrations of decomposition products (if the fluid is accidentally discharged into the fire chamber) are not serious hazards because the irritating character of such concentrations preclude voluntary exposure.

Repeated or prolonged skin exposure should be avoided since the Askarel fluids act as solvents for fats and oils of the skin. Removal of these natural, protective barriers can lead to drying and chapping such as occurs with exposures to paint thinners. More important, the fluid may be absorbed if it is allowed to remain on the unbroken skin. For these reasons, we recommend that the skin be washed with soap and water if there is contact. A skin burn resulting from accidental contact with hot fluid should be treated in the normal manner for any thermal burn due to hot oils.

Because of low vapor pressure, there is no significant vapor inhalation hazard when an Askarel fluid is at room temperature. For example, there is no vapor exposure problem while transferring the fluid from its shipping container to the using equipment. On the other hand, the vapors emitted by Askarels heated to elevated temperatures are injurious on prolonged or repeated exposure. The "Threshold Limit Value" or atmospheric vapor concentration which cannot be safely exceeded (on a daily basis) is 0.5 - 1.0 milligrams of Askarel vapor per cubic meter of air in the workroom.

R. Emmet Kelly, M. D.
Medical Director

March 4, 1971




NEV 135516

Monsanto
EUROPE S.A.

FROM : Brussels, Belgium
(NAME & LOCATION)

DATE : February 22, 1967

SUBJECT :

REFERENCE : DW

TO : Dr. R. Emmet Kelly
St. Louis

cc : J.R. Buchanan, St Louis
J.E. Filer, Brussels
D.V. Hardy, MCL
Eugene Wilde, St Louis

Dear Dr. Kelly,

I should like to refer to your letter dated the 10th of February and our telephone discussion of yesterday's date.

I enclose a copy of Sören Jensen's original paper which in fact was presented in English - Dr. Jensen apologises for the typing etc. but this copy is taken from his original paper which has not yet been re-typed and tidied up.

I asked our agent in Sweden to dig out the various pieces of information that you required and I attach a copy of his reply dated February 17th which I think fully covers the point raised in your letter.

If there are any small points outstanding, please let me know and I will try to get you further information.

I am sending the only copy of Mr. Jensen's paper to you and therefore if any of the other recipients of this letter needs to have access, they can take a copy from yourself.

I would however emphasize the post script on page 3 of Ola Palm's letter in which he states Mr. Jensen has consented to send us the full copy of his paper on the understanding that this is used only internally within the Monsanto organization.

Best wishes.

David Wood
D. Wood

PENCAD-Bayonne, N. J.
DEPOSITION EXHIBIT
30
2-15-94

NEV 023647

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Mr Chairman, Ladies and gentlemen.
In honor to our Brittish guest I will try to hold this lecture in English.

As the title of this lecture states, I am today going to tell about the discovery of some hitherto unobserved chlorinated hydrocarbons having up to eight chlorine in the molecule and found in residue analysis. The chemical name of polychlorinated bifenyls ( In the following called PCB). To get familiar with PCB I will start with the chemistry and toxicologi.

Chemistry

Th

The main characteristic of PCB is 1. Their very high stability. As an example they can be boiled with nitrin acid without being destroyed. 2. They are hardly metabolised in living organism. 3. if more than 4 chlorine are prebsent they are non inflamable. It is clear that these three characteristics does it easy to understand that when they have entered the living organism. the will have a low persistence . But it is difficult to explain how they find their way into the living organism. One thing seems to be clear, they don't come from agricultural use, but from a technical one and most probable it comes to the nature via wastes that are tried to be burnt up, because then we have them at once in the air, because of their non inflamability.

Toxicologi

NEV 106549

The PCB were introduced in 1929 and as early as 1936 Jones and Aldon reported that 23 out of 24 men employed in manufacturing of PCB suffered from an acne form eruption of the skin. Acne did not appear until 6 to 8 months after the material was first used. In 1937 Drinker reported that rats exposed to chlorinated biphenyls in concentration of approximately 1 $mg/m^3$ for 16 hours a day for 6 weeks showdd damage of the livor. After that time the allowed concentration of PCB in air is 0,5 $mg/m^3$. (For DDt the same value is 0.5 - 1 $mg/m^3$). The same authors finished their experiments in 1938, and related that these compounds have an injourious effect, manifested solely in the liver. Chlorinated bipehnyls appeared to be the most injourious chlorinated compounds of all tested.

Greenburg, Mayer and Smith 1939 reported that PCB and polychlorinated naphtalenes are blamed for the death of three young workers, and that pregnant women and persons who have at any time had any liver diseases are particularly susceptible.

Wedel, Haller and Benton gave 1942 animals PCB including administration by inhalation, ingestion and skin absorbtion. Histological examination of the viscera showed important toxic effect only in the skin and liver, and the degeneration effects in the liver are essentially the same whatever was the method for the administration. Paribok (1955/ found as an occupational poison in the electrical industry, mixed tetra and penta chlorobiphenyl causes folliculitis, comedo, pyodermia and other skin affections, and that its principal toxic effect is fatty degeneration of the liver.

Miller (1944) injected 69 mg PCB (4 and 5 chlorine) subcontaneously in 32 guinea pigs. Eight to ten days after injection, fat droplets were noted in the liver cells, and after 16 days they were present in moderate or very large numbers. Rabbits and rats were also tested in this investigation, as well as the PCB was administated both continously, subcuntinously or ingested in the food. In the feeding experiment 8 guinea pigs received 2 doses of 69 mg of the chlorinated biphenyl 1 week apart. Death occurred in 11 to 29 days.

Finally Mc Laughlin 1964 reported a method to test the chemical toxicity and teratogenic effect by injection into the yolk sac of fertile eggs prior to incubation. PCB was found between the eight compounds among 100 tested having the highest order of toxicity. No hatch was found at a level of 25 mg pp egg. At a level of 10 mg per egg, one chick hatched out of 20 injected eggs, but died 2 days later. Some embryos which were examined after they died, showed weak deformities (often a short upper brak) and growth retardation. Lead acetate resulted as an example in no hatch at a level of 1 mg per egg. Autopsy of the dead embyos have showed extensive brain damage. Mercuric chloride showed no hatch even at a level of 0,5 mg per egg.

As the analytical chemistry is a pronounced service science I have been in contact with many scientists from other fields during the work with residue analysis, and I have always found this contact very stimulating for my own work. This co-operation often demands that we are talking the same scientific language. Because of this need I will today try to give a lecture in low level analytical chemistry for biologists, illustrated by the residue analysis of polychlorinated biphenyls.

The lecture will be divided in the following three sub-divisions:

1. Chemistry of PCB and their toxicology.
2. Analytical methods for residue analysis and proof of structures.
3. Behaviour of PCB in nature, differencies in metabolising rate of the PCB components, potensation in an ecological serie, concentration levels and examples of samples which have been proved to contain PCB.

A residue analysis can be divided in:

1. Extraction of the pesticides from the biological material, followed by a careful cleaning-up to take away interferring substances, most often fats.
2. Identification analysis by mean of gas chromatography. Thin-layer chromatography and mass spectrometry.
3. Quantitative analysis.

At an ecological laboratory in Riksmuséet in Stockholm 1-2 g of a sample is cut out of the biological material and transferred into a weighed and carefully cleaned test tube, and stored at -20° until analysis. Smaller samples have been used, min. 5 mg of body fat, and with dry materials such as hair, feathers, pine needles 100 mg are sufficient to reach the desired 10 ng/g level in residue analysis. In cases of water proofs 1 l. is used for reaching the 10 pg/g. level.

B.1(homog) In order to facilitate complete extraction of the fatty materials from the biological sample, the doulbe amount of finely powdered anhydrous magnesium sulphate is added to the sampling tube, and the whole is homogenised with an insertable homogeniser. The resulting powder is transferred into a special Soxhlet extractor. After 4 hours of extraction the solvent is evaporated, leaving the fat in a small weighed test tube at the bottom Sox.-tube) of the extractor. This fat is dissolved in methylene chloride in such a way that 100 ul (0,1 ml) contain 20 mg of fat.

The 100 ul solution is now transferred to a little object glass, 3 x 7 cm, covered with a silicagel layer 1 mm thick, in order to form a line 0,7 cm from one end of the slide. Inserting this thin-layer plate into a vessel the bottom of which is covered by a few mm of methylene chloride, the solvent will be sucked up in the dry layer of silicagel, and at least reach the upper end of the plate. The fact is that the fat has a greater affinity to the powder on the plate than the chlorinated hydrocarbons have. - and we get a separation. The fat being more polar than the chlorinated hydrocarbons will never go longer than 2 cm before the

solvent reaches the upper part of the glass.

7 ELUTION tube

The front of the fat appears quite visible against a lamp, and with the aid of a razor blade the zone above the fat is transferred to the elution tube and the chlorinated biocides absorbed on the powder can now be eluted by one ml of ether. The concentration is sufficient for detection of the chlorinated hydrocarbons down to the $10^{-12}$ g level.

The next step in the analytical procedure concerns the separation of the

5 GC

different chlorinated hydrocarbons that the sample may contain. As a matter of fact, this is a troublesome task. It is easy to estimate what is not present, but more difficult to say exactly one is present. We suffer from the negative demonstration, as will be shown later.

At first a few words about the separation of the components present in the sample and their visualisation.

6 GC

The separation is accomplished by mean of a gas chromatograph fitted to a detector that transfers its impulse to a recorder.

The system is shortly described:

A spirally formed glass tube with an inner diameter of 2 mm and about 2 m in length is filled up by a support, covered with an thin layer of an oil. The tube is heated in the chromatograph to about 200°. Through the tube a stream of nitrogen continously follows. When about 10 ul (1/100 of 1 ml) of the purified sample is injected into the tube, the components of the sample will be evaporized and go forward through the column with the gas stream. As the constituents have different affinity to the column filling they will pass the column with different speed and it will take different time for them to reach the detector at the other end of the glass tube. If the temperature and the nitrogen flow are held constant this time, the retention time, has a specificvalue for a certain compound. This is true, but unfortunately it is also a fact that two components can have the same retention time. This is one of the bigger problems in gas chromatographic analysis of unknown samples, as will soon be obvious.

To make it possible to estimate the retention time it is necessary to visualize the chlorinated hydrocarbons. For that purpose more or less specific detectors are used. The detector most often used in pesticide analysis is the so called electron capture detector, which can detect down to one picogram (= $10^{-12}$ g of lindan). Unfortunately this detector is not specific for chlorine, bu gives answer also for oxygencontaining compounds. The response here is much lower but can be counterbalanced if the concentration of the oxygen containing is much higher.

The principle for the electron capture detector is shortly:

At the end of the gas chromatographic tube is placed a little tube containing a foil made of titanium tritide. This is ap-radiant. The p-

particles are reacting with the nitrogen molecules coming from the column. Then we get $+ N_2 \rightarrow e^- + N_2^+$. Over the detector we have a tension of 90 volt and by mean of the electrons we will get a constant electrical current over the detector. This standing current is transferred to a one-mV recorder as a constant baseline. When now a chlorinated hydrocarbon leaves the column this compound has a high affinity to the electrons and this means that the amount of electrons will diminish, and they will diminish proportionally to the amount of chlorine. The electrical current will also diminish and this is noted as a peak on the recorder. The area of the peak will be proportional to the amount of substance in the sample. By mean of a standard injection it is now possible to compare the retention time and the area of an unknown component with the retention time and area of the known standard. As said before this detector is not specific for chlrine but anyhow very useful, because of its high sensitivity. The system described has, as we have seen, two disadvantages:

1. Two different compounds can have the same retention time and be detected as one peak.

2. A registrated peak does not need to be chlorinated , because the detector is not specific.

If the sample is injected in two different columns with different chemical properties we have increased the chance for a good separation. If two compounds have the same retention time on one column they may not have it on another. When a result seems doubtful, - if the compound being responsible for a certain peak contains chlorine or not - it is possible to concentrate the sample and analyse it on a less sensitive detector such as the microcloumetric one, which is specific for chlorine. The compound is burned in a furnace and teh generated chlorine titrated directly.

As is seen from the two last mentioned possibilities it is anyhow possible to get a rather high degree of certainty in residue analysis, but it is a rather time-consumeing work.When using this method just described, we very often found that many chromatograms from residue analysis of most carefully purified samples still contain a large number of peaks. Many of these have retention times that do not agree with any known chlorinated pesticides, or their metabolites. This chromatogram can serve as an example. It was obtained by residue analysis of a sea-eagle found dead in the archipelago of Stockholm. In the range of the known peaks, there are so many unidentified that there also must be an obvious risk of the known peaks to be covered by unknown ones.

If this remark is found true, the reported results of many previous quan-

titabive analysis must be brought into question. In the present investigation it is shown that most of the unknown peak of chromatograms at residue analysis of chlorinated pesticides are due to polychlorinated biphenyls.

I will show a chromatogram of human fat analysed on a so called SF 96 column, the most often used type in pesticide analyses. Early retention times were in agreement with DDE, DDTop and DDTpp. Next slide shows the same sample analysed on a QF-1 column. Now the former 2 DDT peaks have divided into 4 peaks, and two of them are still in agreement with DDTpp and op., the two new were unknown.

Logically, these unknown components were at first thought to be metabolites of the insecticides. Against that spoke that neigther treatment nor concentrated sulfuric acid in other. This treatment made it rather sure that the compounds did not contain oxygen. In Sweden residues of organic mercury have been investigated rather intensively in the Swedish fauna. As these compounds give very high responses to the electron capture detector it was also investigated if the unknown peaks could have a mercuric origin.

It was found that the water-ecological series had high residues of both mercury (Westermark, Johnels) and the unknown ones, when the same individuals were analysed. Anyhow, the pheasant suffering most from mercury poisoning only contained low levels of electron capturing compounds and these belonged
to the normal insectisides. Therefore the unknown could hardly be mercurials or metabolites of them.

As the eagle sample giving the chromatogram shown in fig. 10. could be estimated to contain DDT and DDE up to 15 g/kg in extractable fat, the amount of unknown compounds also were suggested to be in the same range, and tehn sufficiently high to do a run on the combined gas chromatograph - mass spectrometer. If this could be done successfully it would be possible to get very important informations about the chemical nature of the unknown, for ex. the molekular weight numbers of chlorine etc. This method is up to now the method giving the highest degree of certainty in the low level analytical chemistry, amounts of 100 ng substance being enough.

As this method for identification of totally unknown residues surely will be very important in the future (when f.ex. a biologist has found that fishes in a river die) it may be possible by mean of this method to find out exactly what compounds are responsible for the death.
For this reason, I will go into some details with this method.
In the actual case we took the extract from 20 mg eagle and concentrated

it as much as possible and made an injection on the gas chromatograph combined with the mass spectrometer. The result was the chromatogram shown on the next slide. Every time the recordor showed that a compound is leaving the column, the effluent is led to the mass spectrometer. Now just a few words about the mass spec.

The molecules leaving the column are bomded with electrons at E. We have now got the molecule positive charged, but with the same mass as before. This $M^+$ is accelerated in a vacuum and will then get a kinetic energi.

where is the speed. Next comes the magnetic field that tries to bend the direction of the molecule. This will be big for a small molecule and less for

If we have a sieve in the other end we can directly read the molecular weight. Added to this parent molecule $M^+$ we will also get addition informations, because of the fact that $M^+$ may not be stbble, a part of them will be broken down before they reach the sieve in the other end.

F.ex. $M^+_{DDT}$ $M^+_{DDT} - CCl_3$

Mass spectrograms from the different unknown peaks in the eagle sample as shown. The mass numbers equal to the molecular weights of the unknowns could be read to 426,392, 358, 324. Astonishingly, the molecular differences were constantly 34 mass units. This difference shows a familarity in origin of the unknown. Now the fact is that chlorine exsists as a mixture of two isotopes with atom weights 35 and 37 in proportion 75:25. If the molecule has one chlorine, this will give two molecule peaks, one for $Cl_{35}$ and one for $Cl_{37}$. If there are two chlorine we have the possibility of one with only $Cl_{35}$, one with both $Cl_{35}$ and 37 and one with 2 $Cl_{37}$ and therefore

The relation of the peaks found on the different mass spec were:

| | | | | |
|---|---|---|---|---|
| Molecular weight | 324 | 358 | 392 | 426 |
| Chlorine content | 5 | 6 | 7 | 8 |

An explanation of the familiarity of the compounds can be given if one substance is built from the former by substituting a hydrogen with chlorine

$$RH + Cl_2 \rightarrow RCl + HCl$$
$$M^+ + 34$$

Then it is possible to calculate the molecular weight of the parent hydrocarbon PHC.

$M_{PHC} = M - x M_{Cl} + x M_H$ , where M is the molecular weight of the component having x chlorine atoms. F.ex. for m = 426 and 8 Cl we will get $M_{PHC}$ = 426 - 280 + 8 = 154 and equal with the other molekyls.

The most probable formula with carbon and hydrogen giving this molecular weight is $C_{12} H_{10}$ and this can only be satisfied when the parent-hydrocarbon is biphenyl, and the unknown being polychlorinated biphenyls.

This explanation was later fully verified by injection of a synthetic PBC on the mass spec.

Furthermore extensive gas chromatographic investigations proved that the PBC standard gave peaks with the same retention time as the unknown peaks from the sea eagle.

With the method just described I suppose that we have a new possibility to study the residues in the air because the pine needles can allways be . We have had great difficult; in quantifying the PCB, but when getting a little more time it will be possible. We have done a few calculations on a few species, and I suppose they are right within a factor 2. We have found the residue to be from

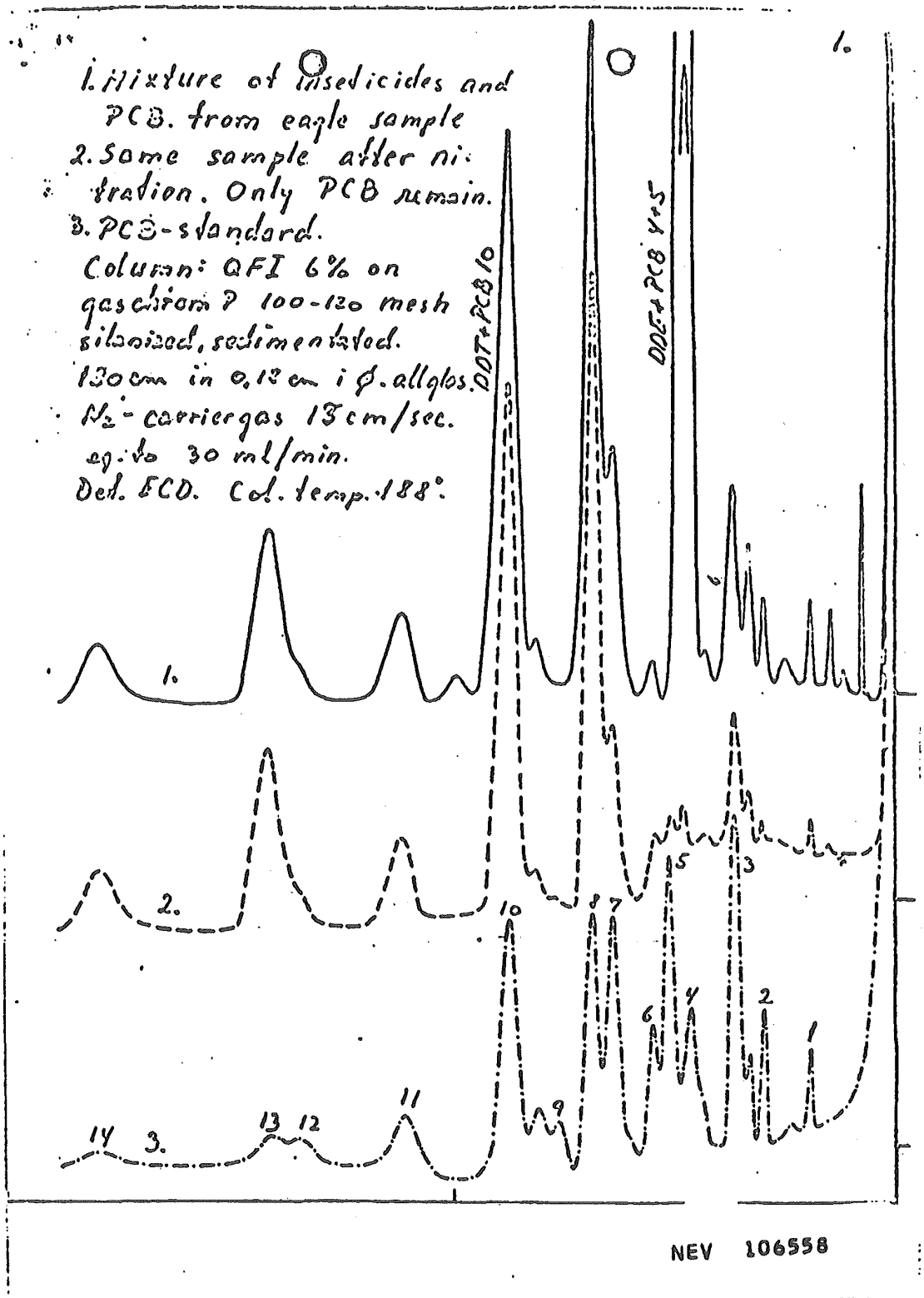
It has been my statement here to-day to present this method for studies of defiling of the nature, and with this method a new type of defiling agents has been found to be present in nature, and a few experiment have shown where they may be found.

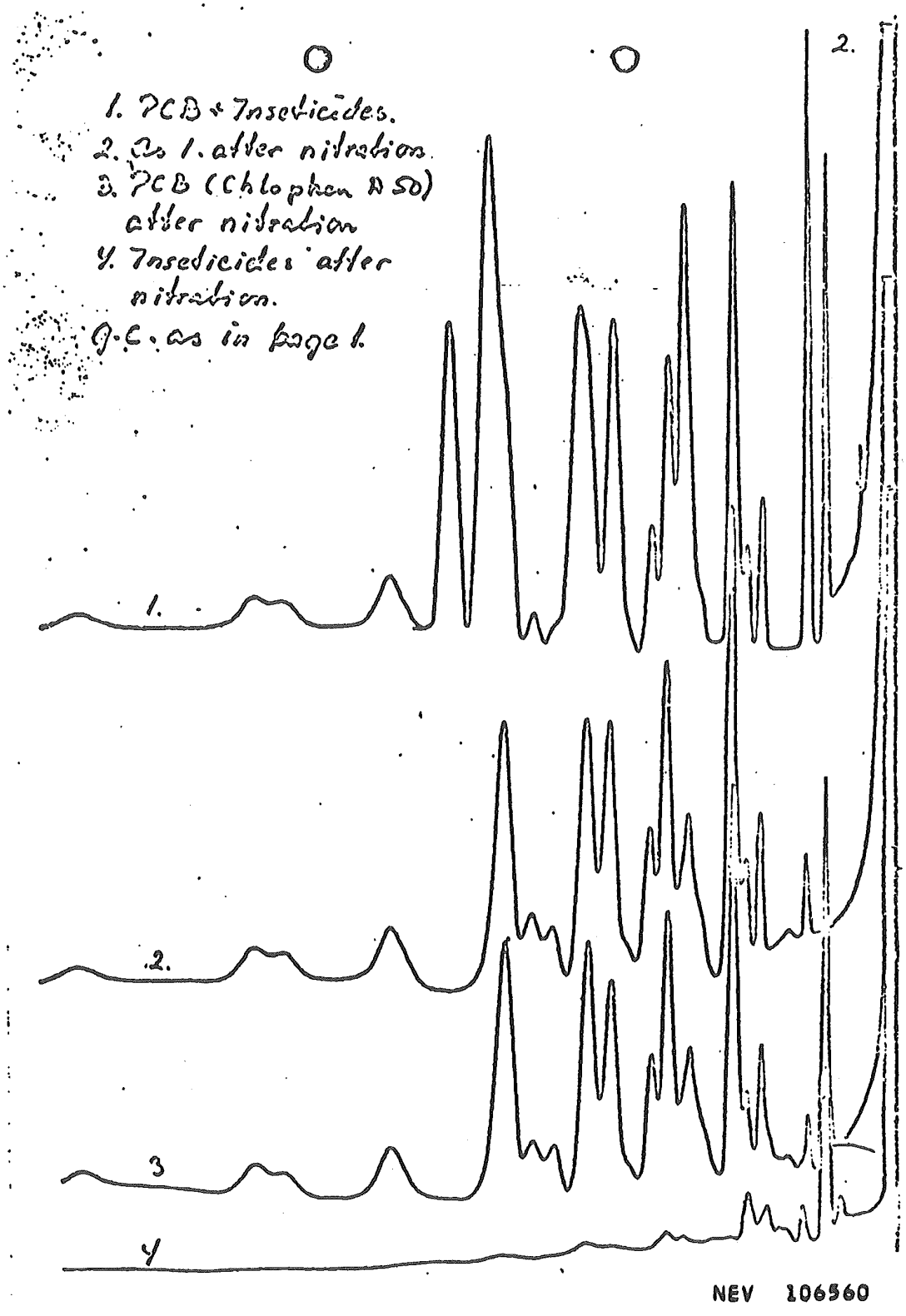
Now this method is going to be used in the first hand to estimate how the situation is in nature as a whole, and in the other hand to find the leaks throug which they find its way to nature. Some maybe are present here today to get news about the leaks, and to them I want to say come back in a year.

NEV 106556

So much I think I can say again that the PCB hardly can come from agriculture. As support for this suggestion I can say that we have found PCB in eagle feathers from Riksmuseet from 1944, where hardly any chlorinated pesticides were used in agricultrue. One more thing that I find important to say is that in contrast to the mercury problem this does not seem to be a pure Swedish problem. I have just studied chromatograms taken from London air, and they clearly contain PCB, and dr. Holden has told me that he also find them in his fishsamples. But finally in waiting at more results I should like to point out one morething. It is proved that PCB comes to nature, we dont know now where they are used, but they are very persistant to chemicals and to fire. I think the poison jury should try to state that a content of PCB shall always be found in an open declaration.

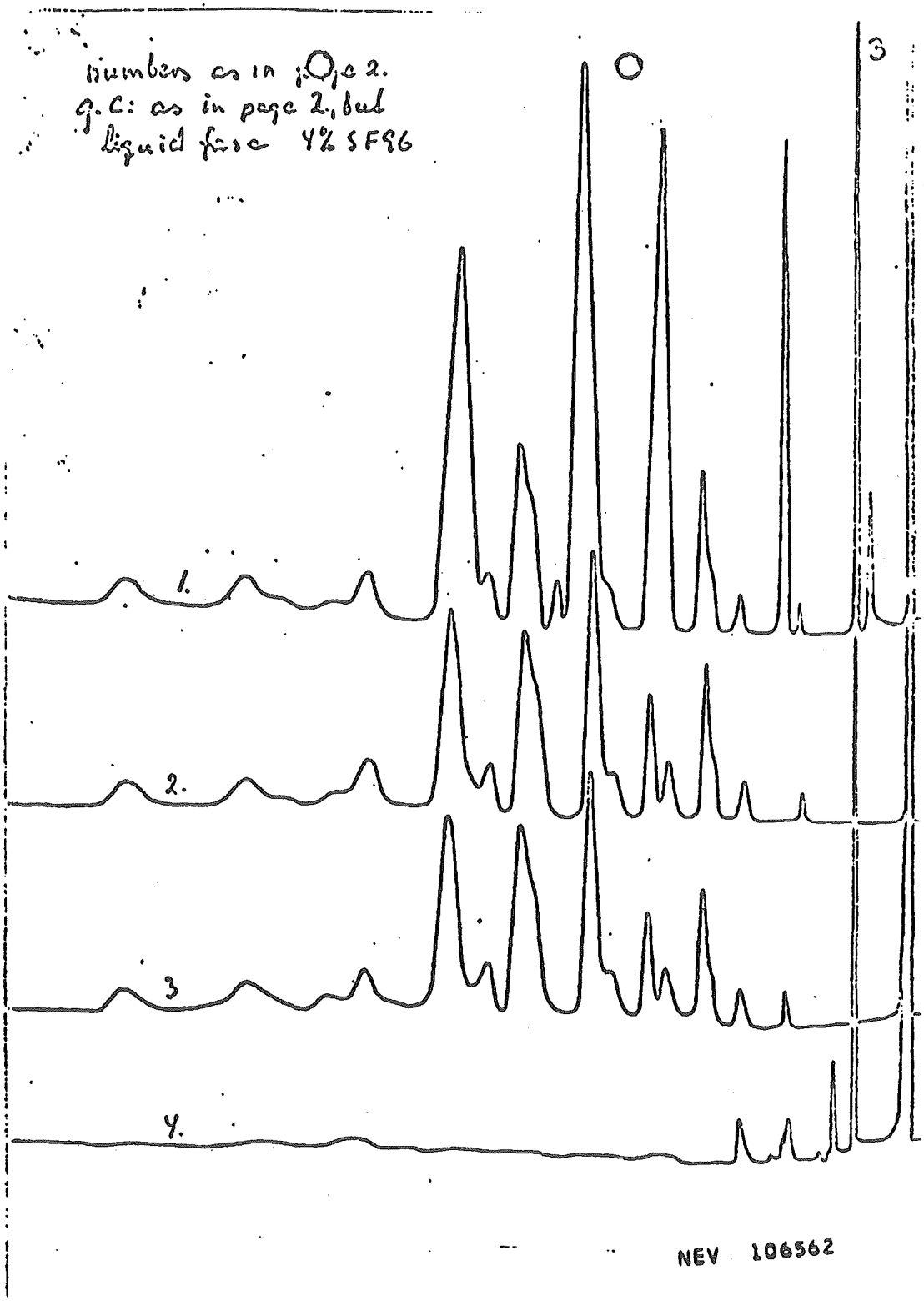
NEV 106557






NEV 106560

NEV 106561

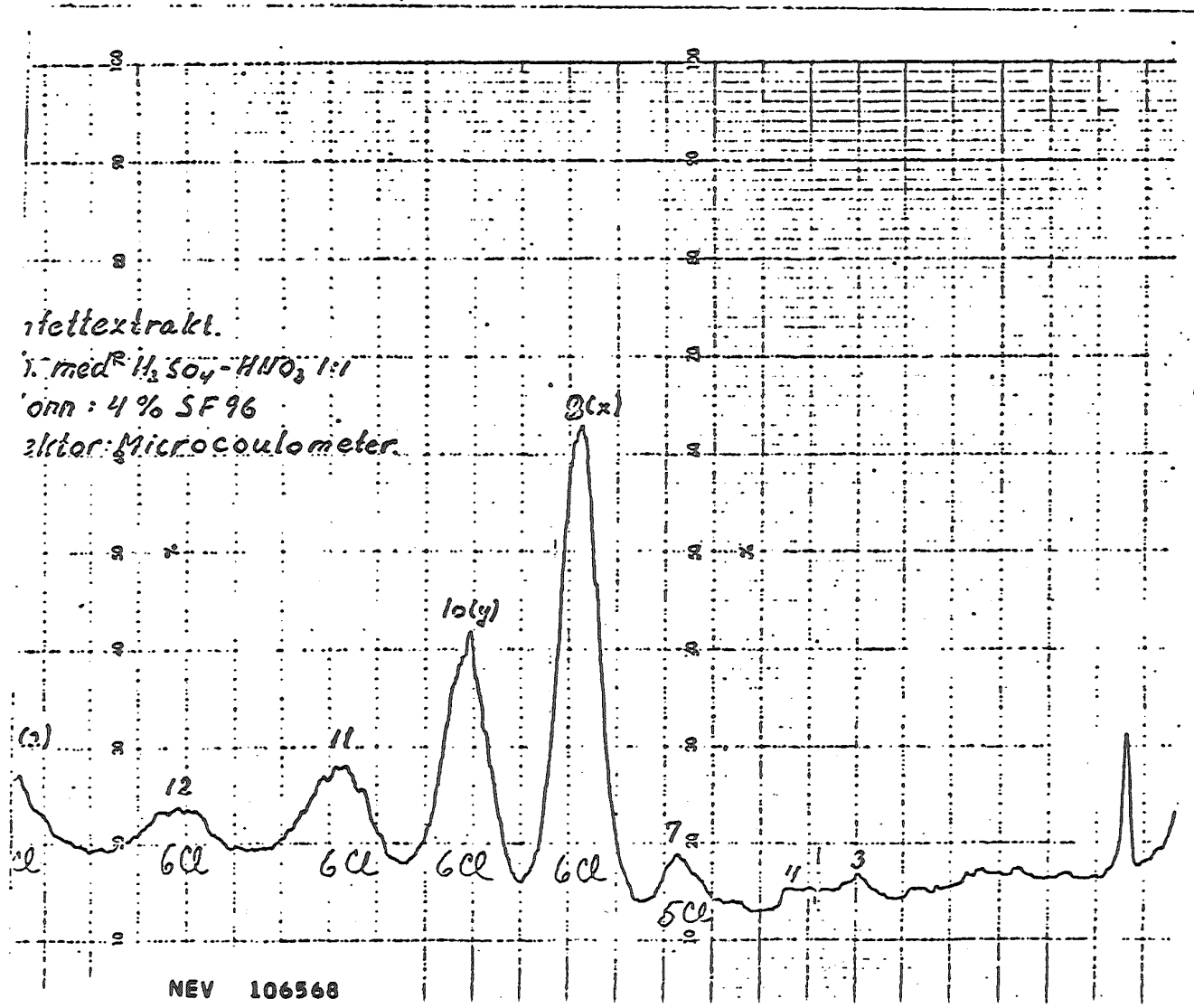



NEV 106562

Fig 3. GC-MS chromatogram from eagle sample. Peak numbers represent nine pcbnis at scanning.

NEV 106564

Column: 4% SF96
EC Detector.
NEV 106566
Eagle
PCB



Clophen A30
Top run 7



NEV 106569

Clophen 1150

Top or B

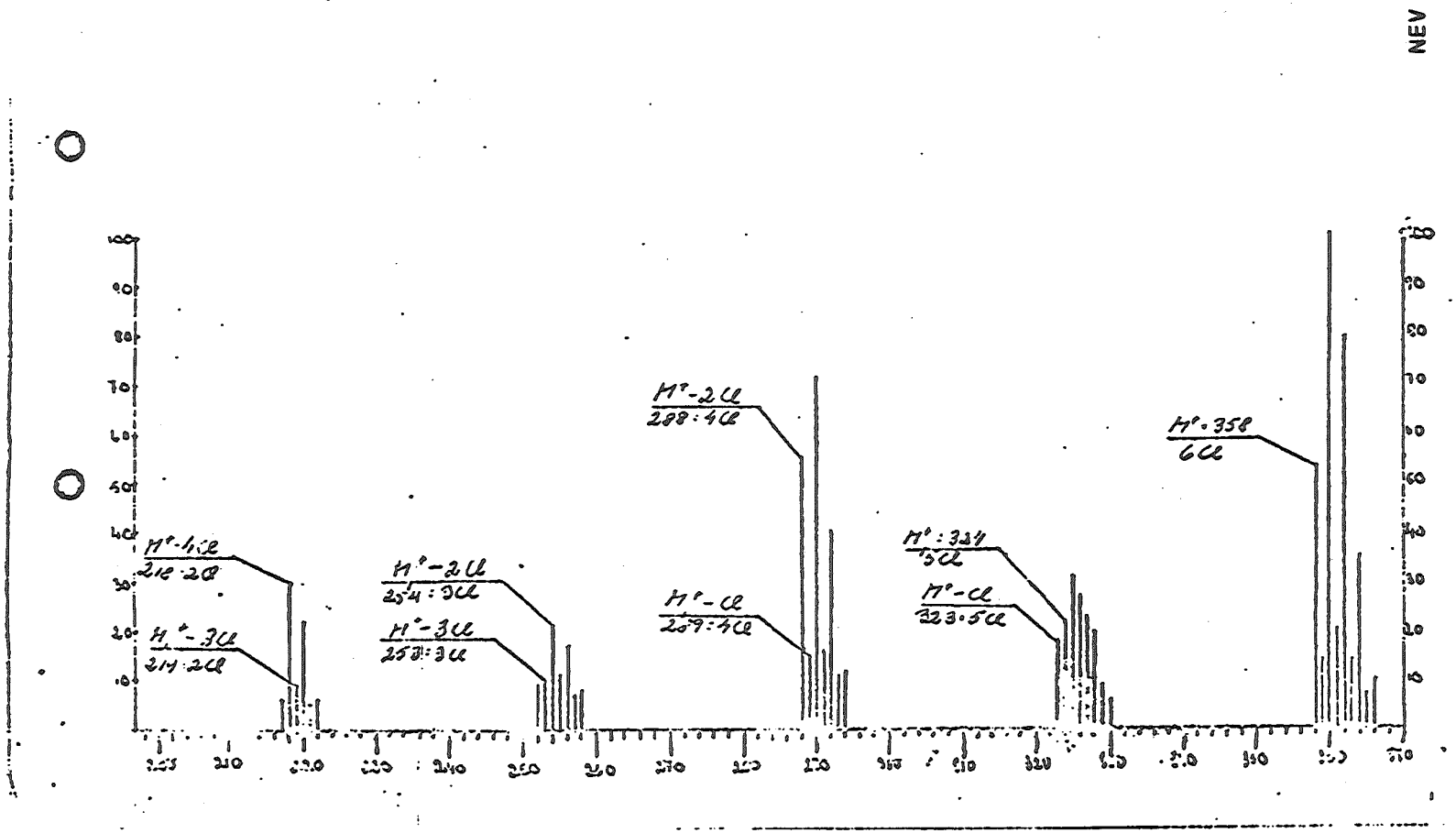


NEV 106570

NEV 106571

Clophen A50
topp nr. 11.

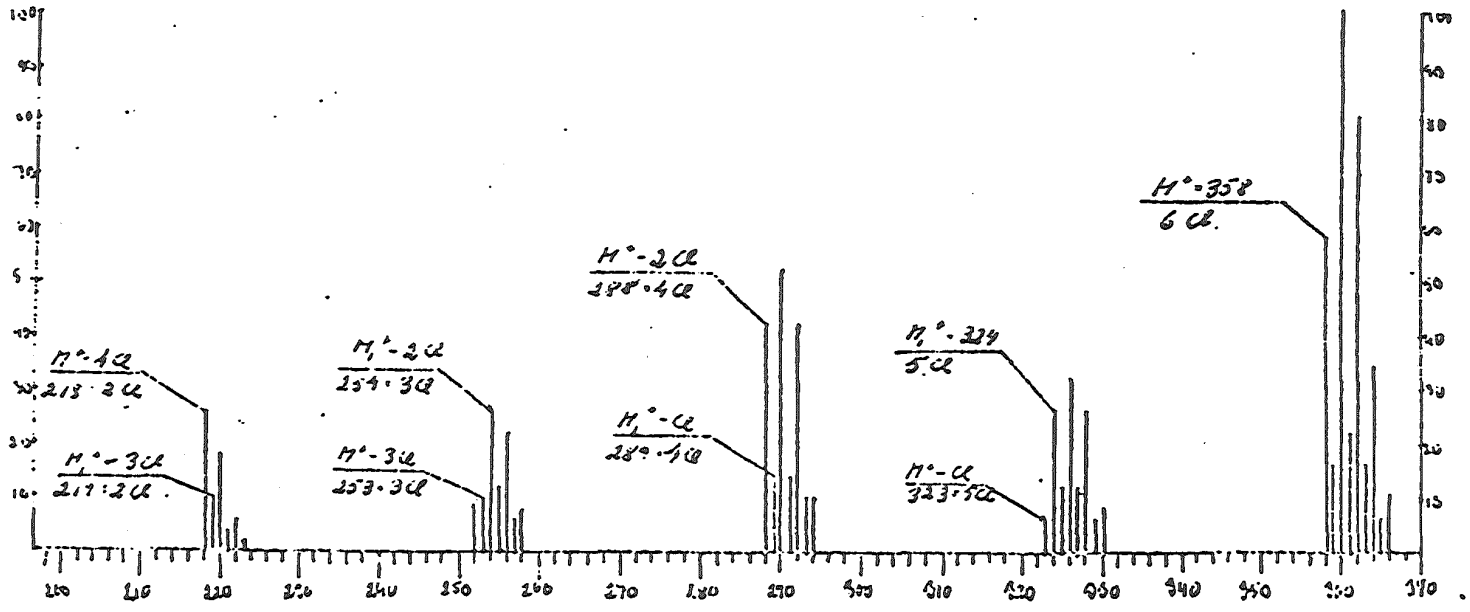
NEV 106572



Clophen A50

Topp nr 12

NEV 106573



Monsanto

FROM (NAME & LOCATION) E. P. Wheeler - St. Louis

DATE September 11, 1968 FF

SUBJECT Proposed Protocols for Aroclor Toxicity Studies at Industrial Bio-Test Lab.

REFERENCE

cc: Dr. R. E. Kelly
Dr. Scott Tucker
Mr. R. L. Keller
Mr. Cumming Paton —

TO : Dr. W. R. Richard

Attached is a copy of a letter from Industrial Bio-Test and proposed protocols for Aroclor toxicity studies. This arrives just as I am leaving for two weeks.

I will take this material with me and if possible send you my comments before the end of next week. If it appears desirable to have a session with Dr. Kelly and Bill Hunt to discuss this matter before I return, I am sure Dr. Kelly will be happy to see you.

EPW

Elmer P. Wheeler

EPW:cjs

81-85-1-9-272-3030



NEV 167288

IN 10 REV 11-65

SEP 11 1968

Industrial BIO-TEST Laboratories, Inc.
1810 FRONTAGE ROAD
NORTHBROOK, ILLINOIS 60062
AREA CODE 312
TELEPHONE 272-9030

September 9, 1968

Mr. Elmer P. Wheeler
Monsanto Company
800 N. Lindbergh Blvd.
St. Louis, Missouri 63166

Dear Mr. Wheeler:

I am submitting a collection of protocols for studies with Aroclors. The studies proposed, with prices, are as follows:

I. Rat Tissue Study

| | |
|---|---|
| Aroclor I | 2 levels |
| Aroclor II | 2 levels |
| Aroclor III | 2 levels |
| Toxaphene | 2 levels |
| Controls | |
| Total | $2,700 |

Additional reference materials could be included for $600 per material (2 levels).

This price is exclusive of analyses. If you choose for us to do all or part of the analytical work, we could work on a cost plus basis or we could provide you with a quotation after we have run through the method.

We found no really useful information in our scan of the literature on the metabolism of chlorinated pesticides. I feel that we can get much information from analysis for tissue residues and from study of urine and feces collected from treated animals. It is not possible to quote for such studies in the absence of fore-knowledge of findings. This could be set up on a cost plus basis with provision to review after the expenditure of a defined sum. One should be able to at least define the problem on a budget of $5,000.

NEV 162002

II. Chicken Toxicity, Reproduction and Meat and Egg Residue Study

| | |
|---|---|
| Three Aroclors with common controls Exclusive of analytical costs | $15,000 |

III. Mallard Duck Study--

| | |
|---|---|
| Three Aroclors with common controls Exclusive of analytical costs | $5,000 |

IV. 3-Generation Reproduction Study in Rats

| | |
|---|---|
| Three Aroclors with common controls | $35,000 |

V. 2-Year Chronic Toxicity Study in Rats

| | |
|---|---|
| Three Aroclors with common controls | $57,500 |

VI. 2-Year Chronic Toxicity Study in Dogs

| | |
|---|---|
| Three Aroclors with common controls | $85,000 |

VII. Subacute Fish Toxicity Study

We have not done such a study but we have built one apparatus which allows investigation of as many as five concentrations of one material plus controls simultaneously for extended periods of time, starting with fingerlings and continuing through a reproduction cycle if this is desired.

If one wanted only to measure the accumulation of test materials over a 90-day period, the cost, using 5 concentrations, would be approximately $1,500 per species for one material, exclusive of analyses. Extending the study through a reproduction cycle would cost an additional $3,500.

In any of these studies, material (urine, feces or tissues) could be retained for analysis to determine species differences relative to accumulation and metabolism.

NEV 162003

The cumulative cost of the defined studies, exclusive of analytical costs and fish studies, amounts to $200,200.

As Dr. Calandra has told you, billings could be arranged to accommodate to your budgetary needs.

Your comments on this program will be welcome and should further personal discussions seem desirable, we will be glad to meet with you either here or in St. Louis.

Sincerely yours,

Otis E. Fancher

Otis E. Fancher, Ph.D.
Director

OEF:DMS
enc.

NEV 162004

*Industrial* BIO-TEST *Laboratories, Inc.*
1810 FRONTAGE ROAD
NORTHBROOK, ILLINOIS 60062
AREA CODE 312
TELEPHONE 272-9030

# PROTOCOLS FOR

# MONSANTO COMPANY

# AROCLOR STUDIES

NEV 143472

# PROTOCOL FOR

# MONSANTO COMPANY

# A TISSUE RESIDUE STUDY IN ALBINO RATS

# WITH AROCLORS

I. Outline of Experiment

A. Type and Length: Tissue Residue Study; duration 30 days

B. Animal Species Tested: Male Albino Rats (approximately 100 g)

C. Organization of Groups:

| Group | Number of Animals Male | Female | Dietary Concentration in ppm |
|---|---|---|---|
| Control | 15 | 15 | None |
| I | 15 | 15 | Low dose level |
| II | 15 | 15 | High dose level |

D. Test Material Administration

Diets will be prepared fresh weekly by mixing the calculated quantity of test material with a standard pulverized rat ration. Food will be allowed ad libitum.

II. Parameters to be Studied

A. Feed Consumption

Feed consumption will be determined weekly for each animal.

B. Reactions

Observations for untoward behavioral reactions will be made.

C. Gross Pathology

Upon sacrifice, organs and tissues will be examined grossly for pathologic alterations.

III. Sample Collection

At the end of 30 days all animals will be sacrificed and samples of muscle, fat, liver and kidney will be taken from each animal. Samples of each tissue from each animal will be frozen and will either be shipped to Monsanto for analysis or analyses will be done by Bio-Test.

OEF:DMS
9-9-68

PROTOCOL FOR

MONSANTO COMPANY

CHICKEN TOXICITY, REPRODUCTION AND RESIDUE STUDY

ON AROCLOR

The object of the study will be to investigate the general toxic effects of the test material on young adult male and female chickens with special emphasis upon hatchability of eggs and viability of offspring. In addition, samples of meat and eggs will be analyzed for test material residues (either by Monsanto or by Bio-Test).

Sixteen male and 80 female 12-week old white Leghorn chickens will be selected for the study. These animals will be equally divided into one control and three test groups. The three test groups will correspond to graded dietary levels of the test material. Dosing will begin immediately and will continue through the egg collection period.

The chickens in the control and test groups will be weighed weekly and observations for mortality, reactions and general food disappearance will be made periodically during the study.

As each group begins laying (at 17 to 18 weeks of age), the eggs will be collected daily and egg weights recorded. When the egg weights are consistently between 50 and 60 grams, and at least 20 per cent of the hens are laying, the daily egg production will be collected and placed in an incubator tray. Egg collection will be continued for 28 days from collection

Day One or until at least 100 eggs per group are available for hatching.

During the storage period (not to exceed five days for any group of eggs), the eggs will be turned twice daily. The eggs will be placed in an incubator maintained at 99° to 100°F with a wet bulb reading of 85°F.

The egg production for an additional week will be collected for analysis. Total egg production and egg weights will be recorded.

At the end of the egg collection period, one-half of the chickens in each group will be sacrificed, defeathered and quick-frozen. The carcasses and the eggs retained for analysis will be shipped to Monsanto or will be retained for analysis at Bio-Test.

The remaining chickens will be placed on a 30-day recovery regimen during which time the test material will be removed from the test diets. Eggs will be collected once each week. At the end of this period, the remaining chickens will be sacrificed and handled in the same manner as those at the end of the test feeding period. The carcasses and eggs will again be retained for analysis.

The chicks from the first generation will be observed for 30 days post-hatching.

RJP:DMS
9-9-68

NEV 143476

# PROTOCOL FOR

# MONSANTO COMPANY

# DUCK TOXICITY AND RESIDUE STUDY

# ON AROCLOR

The object of the study will be to investigate the general toxic effects of the test material on young adult male and female mallard ducks and to determine residue levels in specific organs and whole carcass.

Twenty male and twenty female young adult mallard ducks will be selected for the study. These animals will be equally divided into one control and three test groups. The three test groups will correspond to graded dietary levels of the test material.

The ducks in the control and test groups will be weighed weekly and observations for mortality, reactions and general food disappearance will be made periodically during the study.

At the end of a 30-day feeding period, one-half of the animals in each group will be sacrificed, defeathered and subjected to a gross pathological examination. The liver, kidneys and samples of skeletal muscle and fat will be removed from each bird and packaged separately. These tissues will be quick-frozen and retained for analysis for the test material

The remaining ducks will be placed on a 30-day recovery regimen during which time the test material will be removed from the test diets.

At the end of this period, the remaining ducks will be sacrificed and handled in the same manner as those at the end of the test feeding period.

RJP:DMS
9-9-68

NEV 143478

# PROTOCOL FOR

# MONSANTO COMPANY

# THREE-GENERATION REPRODUCTION STUDY OF AROCLOR

# IN ALBINO RATS

## I. General

A three-generation reproduction study will be conducted with Sprague-Dawley derived albino rats. A total of 96 weanlings (32 males and 64 females) will be equally subdivided into three test groups and one control group. The three test groups will correspond to the feeding of three graded dietary levels of the test material.

## II. First Generation

### A. Parental Animals

#### 1. Diets and Feeding

All diets will be prepared in the central diet room of the laboratories. The basic ration with which all diets are prepared is a standard, pulverized rat stock diet*. The diets for any given test group will be prepared by adding a calculated weight of the test material to a pre-weighed portion of the stock diet.

The amount of food allotted per week to each rat is to be sufficient for ad libitum feeding. However, checks will be made daily to ensure that the food jars are not empty.

NEV 143479

* Purina Rat Chow, Ralston Purina Company, St. Louis, Missouri.

2. Body Weights and Weight Gains

The body weight of each rat in every group will be determined and recorded initially, and weekly thereafter until the animals are 100 days old.

3. Mortality, Reactions and General Observations

Mortality and abnormal behavioral reactions will be recorded daily. The animals also will be observed for fertility, length of gestation and lactation performance.

4. Mating Procedure

When the animals reach 100 days of age, each male will be randomly mated with two females from the same group. After successful copulation, determined by the presence of a copulation plug or blood in the vagina, the male will be removed from the females and returned to his original cage. Any male failing to copulate within two estrus cycles of the female will be replaced by another male of the same group. However, no more than two males will be used per female during a given breeding cycle. Records will be kept of the number of successful copulations observed, the number of estrus cycles required to obtain a mating, and the number of resulting pregnancies. These data will be utilized to calculate mating and fertility indices.

The F1a litters obtained will be weaned at 21 days postpartum. The females will then be given a 10-day rest period and again mated, the above procedure being repeated to obtain F1b weanlings.

5. Pathologic Studies

After the second litter has been weaned (following approximately 33 weeks on test), 8 male and 8 female animals from each group will be sacrificed and gross pathologic observations will be made. Organ weights will be taken on the liver, kidneys, spleen, gonads, heart, brain and any organs which appear abnormal. A complete set of tissues will be removed and fixed in a ten per cent formalin solution.

In addition, microscopic examinations will be conducted upon five males and five females from both the control and the high test groups. If abnormalities are noted, the affected organs of the lower test group animals will also be examined.

The following tissues and organs will be examined:

| Tissues and Organs Examined Grossly | Tissues and Organs Examined Microscopically |
|---|---|
| Heart | Heart (right and left ventricles) |
| Trachea | Trachea |
| Lungs | Lungs |
| Liver | Liver |
| Pancreas | Pancreas |
| Esophagus | Esophagus |
| Stomach | Stomach (cardiac, fundic and pyloric regions) |
| Intestinal tract | Small intestine (duodenum, jejunum and ileum) |
| Spleen | Caecum |
| Lymph nodes | Colon |
| Kidneys | Spleen |
| Urinary bladder | Lymph node (cervical and mesenteric) |
| Testes | Kidney |
| Ovaries | Urinary bladder |
| Prostate | Testis |
| Seminal vesicles | Ovary |
| Uterus | Prostate |
| Pituitary gland | Seminal vesicles |
| Adrenal glands | Uterus |
| Salivary glands | Pituitary gland |
| Thyroid glands | Adrenal gland |
| Skeletal muscle | Salivary gland (submaxillary) |
| Bone | Thyroid gland |
| Peripheral nerve | Parathyroid gland |
| Eyes | Bone marrow (sternum and femur) |
| Brain | Peripheral nerve (sciatic) |
| Thymus | Brain (cerebrum, cerebellum and pons) |
| Optic nerves | Spinal cord (three levels) |
| Aorta | |
| Spinal cord | |

Post-mortem animals will be examined in the same manner but organ weights will not be recorded.

Throughout gross and microscopic examination, particular attention will be paid to the reproductive organs.

NEV 143482

B. Progeny

1. Body Weight Data

Pups will be weighed at weaning.

2. Mortality, Reactions and General Observations

All pups will be examined for physical abnormalities at birth and the number of viable and stillborn members of each litter recorded. Records of survival at periodic intervals during the lactation period will be maintained and a final examination for physical abnormalities will be made at the weaning of each litter.

3. Survival Data

Survival indices will be calculated for various points during the lactation period. On the fifth day of lactation, litters of greater than 10 pups will be reduced to that number.

4. Pathologic Studies

A gross internal pathologic examination will be made upon any pup appearing abnormal. No such examinations will be made upon progeny which appear normal.

III. Second and Third Generations

The procedures followed for the second and third generations will be identical to those described for the first generation except that the parental animals for the F2 generation in each group will be selected from F1b weanlings of that group. Parents for the F3 generation will be selected from F2b litters.

In addition, ten male and ten female pups from the F3b litters of each test group and the control group will be sacrificed at weaning (21 days post-partum) and subjected to complete gross and microscopic examination.

IV. Reports

A complete formal report will be submitted upon completion of each generation.

MLK:PSH 9-5-68

NEV 143484

PROTOCOL FOR

MONSANTO COMPANY

TWO-YEAR CHRONIC ORAL TOXICITY
OF
AROCLOR

IN ALBINO RATS

I. Outline of Investigation

A. Type and Length: Two-Year Chronic Oral Toxicity

B. Animal Species Tested: Charles River Strain Albino Rats

C. Organization:

| Group | Number of Animals Male | Female | Dietary Level of Test Material (ppm) |
|---|---|---|---|
| Control | 30 | 30 | None Administered |
| Test I | 30 | 30 | Low Level |
| Test II | 30 | 30 | Middle Level |
| Test III | 30 | 30 | High Level |

D. Means of Administration: Voluntary oral ingestion; ad libitum feeding of diets containing various levels of test material.

II. Parameters To Be Investigated

NEV 143485

A. Body Weights

All animals will be weighed weekly during the first three months of the study and monthly for the remainder of the two-year test period.

B. Food Consumption

Weekly food consumption will be recorded for the first three months of the investigation. Thereafter, monthly spot checks will be made.

C. Mortality and Reactions

Checks for mortality and untoward behavioral reactions will be conducted daily.

D. Hematologic Studies, Clinical Blood Chemistry Studies and Urine Analyses

The studies listed below will be conducted on five rats of each sex from the Control Group and Test Group III at the beginning of the study and after 3, 6, 9, 12, 18 and 24 months of feeding:

1. Hematology: Hematocrit value, hemoglobin concentration, erythrocyte count and total and differential leukocyte counts.
2. Clinical Blood Chemistry: Blood urea nitrogen concentration (BUN), serum alkaline phosphatase activity (SAP), blood glucose concentration and serum glutamic-pyruvic transaminase activity (SGPT).
3. Urine Analyses: Albumin concentration, glucose concentration, microscopic elements examination, pH determination and specific gravity.

The above listed studies will be conducted on animals in the lower dose groups if significant findings appear among high dose animals.

E. Pathologic Studies

1. Gross

Complete gross autopsies will be conducted upon all post-mortem animals unless precluded by post-mortem autolysis, all animals sacrificed in extremis, five males and five females from each group sacrificed after three months of testing and upon all animals surviving 24 months of testing. At the time of each autopsy, representative tissues and organs will be taken and fixed in 10 per cent formalin solution.

Absolute organ weights will be recorded and organ - body weight ratios and organ - brain weight ratios will be calculated and subjected to statistical analysis, viz. Analysis of Variance and/or "t"-tests. The following organs will be included: liver, kidneys, spleen, gonads, heart, brain and any other organ appearing abnormal upon gross examination.

2. Microscopic

Microscopic examinations will be conducted upon tissues and organs taken from selected animals sacrificed in extremis, and five males and five females from the control and highest dietary dose groups sacrificed after three months and 24 months of testing. The tissues and organs to be examined will include: heart, liver, lung, pancreas, stomach (cardia, fundus and pylorus), small intestine (duodenum, jejunum and ileum), caecum, colon, spleen, lymph node, kidney, urinary bladder, testes, ovary, prostate, uterus, pituitary gland, adrenal gland, salivary

gland (submaxillary), thyroid gland, parathyroid gland, skeletal muscle, bone marrow, peripheral nerve, trachea, spinal cord, eye, optic nerve and brain (cerebrum, cerebellum and pons).

Any significant tissue or organ changes at the high dose level would also be examined at the lower levels.

F. Reports

Interim status reports will be submitted after 3, 12 and 18 months of testing. These reports will contain pertinent data collected to that date.

At the conclusion of the study, three copies of a final formal report will be submitted.

NEV 143488

MLK:PSH 9-5-68

# PROTOCOL FOR

# MONSANTO COMPANY

# TWO-YEAR CHRONIC ORAL TOXICITY OF AROCLOR

# IN BEAGLE DOGS

## I. Outline of Experiment

A. Type and Length: Two-Year Chronic Oral Toxicity

B. Animal Species Tested: Pure-Bred Beagle Dogs

C. Test Material: Aroclor

D. Organization: Three Test Groups and One Control Group

1. Number of Dogs/Group: Eight (four males and four females)

2. Dose Levels: Control Group - None
Test Group I - Low level
Test Group II - Middle level
Test Group III - High level

3. Means of Administration: Voluntary oral ingestion; ad libitum feeding of test material in diet.

## II. Parameters To Be Investigated

A. Body Weights

All animals will be weighed weekly.

B. Food Consumption

Weekly food consumption will be recorded.

C. Mortality and Reactions

Mortality and untoward behavioral reactions will be recorded daily.

D. Hematologic Studies, Blood Chemistry Studies and Urine Analyses

The studies listed below will be conducted upon all dogs prior to the inception of the test, after 1, 3, 6, 12 and 18 months of testing and just prior to its conclusion.

1. Hematology: Hematocrit, hemoglobin, erythrocyte count, and both total and differential leukocyte counts.

2. Blood Chemistry: Blood urea nitrogen, blood glucose, serum alkaline phosphatase, serum glutamic-pyruvic transaminase and serum glutamic-oxalacetic transaminase.

3. Urine Analyses: Albumin, glucose, pH, microscopic elements and specific gravity.

E. Pathologic Studies

1. Gross Pathologic Studies

Complete gross autopsies will be conducted upon animals sacrificed in extremis and upon all post-mortem animals during the investigation. At the conclusion of the 24-month study, all surviving dogs will be sacrificed and examined. At the time of each sacrifice, a complete set of tissues will be removed from each dog and preserved in ten per cent formalin solution for histopathologic examination. Also at the time of each sacrifice, the weight of the liver, kidneys, spleen, gonads, thyroid, adrenals, pituitary and heart will be recorded.

2. Histopathologic Studies NEV 143490

Histopathologic examinations will be conducted on tissues taken from animals sacrificed in extremis, from post-mortem animals

and from all animals sacrificed after 24 months of feeding. The tissues to be included in the examinations are as follows: lung, heart, liver, pancreas, mesenteric lymph node, stomach (cardia, fundus and pylorus), small intestine (duodenum, jejunum and ileum), colon, caecum, kidney, adrenal gland, urinary bladder, testes, ovaries, prostate, thyroid gland, submaxillary salivary gland, parathyroid gland, uterus, brain (cerebrum, cerebellum and pons), bone and bone marrow (femur), and skeletal muscle (gastrocnemius).

F. Reports

Interim status reports will be submitted after 3, 12 and 18 months of testing. These reports will contain pertinent data collected to that date.

At the conclusion of the study, three copies of a final formal report will be submitted.

MLK:PSH 9-5-68 NEV 143491

12/20/68

December 20, 1968

Dr. Joseph C. Calandra
Industrial Bio-Test Laboratories, Inc.
1810 Frontage Road
Northbrook, Illinois 60062

Dear Joe:

FF This letter authorizes the initiation of two of the proposed Aroclor studies as follows:

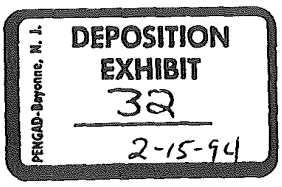
Project No. BTL - Aroclor I-Fowl - Gross Toxicity

Ia - Aroclor 1242
Ib - Aroclor 1254
Ic - Aroclor 1260
Id - Aroclor 5460
Ie - Toxaphene
If - DDT
Ig - Chlorinated naphthalene

The protocol for these studies is to be consistent with our discussions when you were in St. Louis and I would appreciate receiving a note from you outlining what you plan to do with an approximate cost.

Project No. BTL - Aroclor II - 30 Day Rat Tissue Study

IIa - Aroclor 1242
IIb - Aroclor 1254
IIc - Aroclor 1260
IId - Aroclor 5460
IIe - Toxaphene
IIf - DDT
IIg - Chlorinated naphthalene




NEV 008162

These studies are to be carried out as proposed in your protocol submitted with the letter of Dr. Fancher dated September 9, 1968. That letter indicates a cost of $2,700 for three of the Aroclors, Toxaphene and controls. Further it indicates that additional materials could be included for $600 per material. Since we are adding additional Aroclor, DDT and chlorinated naphthalene I assume that the rat tissue study will cost approximately $4,500.

We are obtaining samples of several chlorinated naphthalenes and will provide you the material to be investigated.

Do you have a readily available source of DDT or shall I get this for you through our Agricultural Chemical Group?

I have enclosed a copy of my summary of our meeting when you were down here (as you and I discussed it). There is also a copy of Bill Richard's version.

I hope you have licked the flu bug and will have a happy holiday.

Sincerely,

Elmer P. Wheeler
Manager, Environmental Health

EPW:cjs

Enclosure

cc: W. R. Richard
R. E. Keller/Scott Tucker
C. Paton
W. K. Johnson
R. E. Kelly/W. H. Hunt

NEV 008163

April 8, 1970

Herbert Blumenthal, Ph.D.
Chief, Petitions Review Branch
Bureau of Foods, Pesticides and
Product Safety
U. S. Food and Drug Administration
200 C Street, Southwest
Washington, D. C. 20204

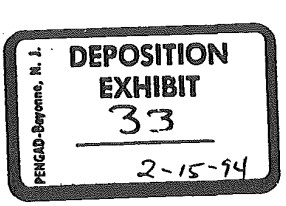
Dear Herb:

Confirming our telephone conversation I have enclosed Xerox copies of the status reports for the chronic toxicity studies for our Aroclors at Industrial Bio-Test, Inc.

Specifically, three of our polychlorinated biphenyls are under investigation. These are Aroclors 1242 (biphenyl chlorinated to the extent of 42%), 1254 (biphenyl chlorinated to the extent of 54%), and 1260 (biphenyl chlorinated to the extent of 60%). Studies include two year oral administration to rats, two year oral feeding to dogs and a three generation rat reproduction study.

As indicated in the enclosures, the levels of administration are 1, 10 and 100 ppm for each of the compounds. Further, the status reports for the chronic dog studies represent data at the end of nine months.

The enclosed copies on the rat studies show data at the end of six months administration. As we mentioned on the telephone, we added 300 rats to the chronic studies after the original two year project had gotten underway to allow a sufficient number of animals for sacrifices at three, six and twelve months. Histopathology has been completed on the sacrificed animals and Dr. Calandra has reported verbally that there are no positive pathological findings.



The rat reproduction studies are into the second generation. At this point Dr. Calandra has indicated that there definitely has been no effect with any of the compounds at the 10 ppm dietary level. There is some equivocal possible effect in the animals at the 100 ppm level with Aroclor 1254.

If you have any specific questions after reviewing the enclosed status summaries, please feel free to call Dr. Calandra or Dr. Fancher directly.

Best personal regards.

Sincerely,

R. Emmet Kelly, M. D.
Medical Director

REK:ju

c.c. Dr. Joseph C. Calandra
Dr. Otis E. Fancher
b.c. W. B. Papageorge, G. W. Ingle
Enclosures

NEV 000630

Monsanto

Monsanto Company
800 N Lindbergh Boulevard
St. Louis, Missouri 63166
Phone: (314) 694-1000

# THREE-GENERATION REPRODUCTION STUDY

# IN ALBINO RATS WITH:

# AROCLOR 1242

# AROCLOR 1254

# AROCLOR 1260

## RESULTS OF THE FIRST GENERATION

September 4, 1970

Submitted by:

R. Emmet Kelly, M. D.
Medical Director

Industrial BIO-TEST Laboratories, Inc.
1810 FRONTAGE ROAD
NORTHBROOK, ILLINOIS 60062

AREA CODE 312
TELEPHONE 272-9

REPORT TO

MONSANTO COMPANY

# THREE-GENERATION REPRODUCTION STUDY IN ALBINO RATS - AROCLOR 1242

RESULTS OF THE FIRST GENERATION

SEPTEMBER 4, 1970

IBT NO. P8797

Industrial BIO-TEST Laboratories, Inc.
1810 FRONTAGE ROAD
NORTHBROOK, ILLINOIS 60062

September 4, 1970

Mr. Elmer Wheeler
Monsanto Company
Medical Department
800 N. Lindbergh Boulevard
St. Louis, Missouri 63166

Dear Mr. Wheeler:

Re: IBT No. P7277 - Three-Generation Reproduction Study in Albino Rats - Aroclor 1242

We are submitting herewith our laboratory report dated September 4, 1970, prepared in connection with the above study.

This report presents the results of the first generation.

Very truly yours,

J. C. Calandra
President

JCC/kjl

NPC00009505

Institute BIO-TEST Laboratories, Inc.

REPORT TO

MONSANTO COMPANY

THREE-GENERATION REPRODUCTION STUDY
IN ALBINO RATS - AROCLOR 1242

RESULTS OF THE FIRST GENERATION

SEPTEMBER 4, 1970

IBT NO. P7297

1. Outline of Study

A. Type and Length: Three-generation reproduction study

B. Animal Species Tested: Charles River albino rats

C. Material Tested: Aroclor 1242, Lot No. AK-255

D. Starting Date: May 8, 1969

E. Status of this Report: Completion of the first generation (Fo parents - F1a and F1b progeny)

F. Organization: A structural outline of the experiment is given in Table 1.

NPC00009506

TABLE I

TEST MATERIAL: Aroclor 1242

Outline of Experiment

Fo Generation

| Group | Dietary Level (ppm) | Number of Animals Male | Female |
|---|---|---|---|
| C | None | 8 | 16 |
| T-I | 1 | 8 | 16 |
| T-II | 10 | 8 | 16 |
| T-III | 100 | 8 | 16 |

C. Means of Administration: Voluntary oral ingestion.

II. Summary

The first generation of a three-generation reproduction study of Aroclor 1242, Lot No. AK-255, at dietary levels of 1, 10 and 100 parts per million has been completed. The following data were obtained during the first generation:

A. Progeny (F1a and F1b)

The numbers of pups delivered and weaned by treated females were generally as large or larger than those from the control group. The survival indices of progeny from treated females were higher than the indices for controls. The lactation indices of the T-III group, but not the T-I or T-II groups, were slightly lower than these indices for the control group. All progeny body weights were normal and there were no unusual reactions noted among any offspring.

B. Parental Animals (Fo)

The body weights of the treated males and females were normal and compared favorably with those of the controls.

One T-III male died during the pre-mating period; one T-I female died while lactating for her second litter; one control female died during parturition of her first litter.

The data obtained from gross pathologic examinations of these animals revealed no significant differences between test and control rats. Organ weight data, the results of the histopathologic studies confirmed the absence of any pathologic changes which could be related to the ingestion of Aroclor 1242.

Mating indices, fertility indices, incidences of pregnancy and parturition for treated animals were normal and compared favorably to those of control animals.

Respectfully submitted,

INDUSTRIAL BIO-TEST LABORATORIES, INC.

Report prepared by:

James B. Plank
Group Leader
Rat Toxicology Department

Report approved by:

M. L. Knuckles, Ph.D.
Manager, Toxicology

Otis E. Fancher, Ph. D.
Scientific Director

September 4, 1970

rh:kjl:psh

NPC00009509

4. Reactions

No untoward behavioral reactions were observed among either test or control progeny.

*Industrial* BIO-TEST *Laboratories, Inc.*
1810 FRONTAGE ROAD
NORTHBROOK, ILLINOIS 60062

AREA CODE 312
TELEPHONE 272-9030

# REPORT TO

# MONSANTO COMPANY

# THREE-GENERATION REPRODUCTION STUDY IN ALBINO RATS - AROCLOR 1254

## RESULTS OF THE FIRST GENERATION

SEPTEMBER 4, 1970

IBT NO. P7297

Industrial BIO-TEST Laboratories, Inc.
1810 FRONTAGE ROAD
NORTHBROOK, ILLINOIS 60062

September 4, 1970

Mr. Elmer Wheeler
Monsanto Company
Medical Department
800 N. Lindbergh Boulevard
St. Louis, Missouri 63166

Dear Mr. Wheeler:

Re: IBT No. P7297 - Three-Generation Reproduction Study in Albino Rats - Aroclor 1254

We are submitting herewith our laboratory report dated September 4, 1970, prepared in connection with the above study.

This report presents the results of the first generation.

Very truly yours,

J. C. Calandra
President

JC/kjl

NPC00009512

BIO-TEST Laboratories, Inc.

REPORT TO

MONSANTO COMPANY

THREE-GENERATION REPRODUCTION STUDY
IN ALBINO RATS - AROCLOR 1254

RESULTS OF THE FIRST GENERATION

SEPTEMBER 4, 1970

IBT NO. P7297

Outline of Study

A. Type and Length: Three-generation reproduction study

B. Animal Species Tested: Charles River albino rats

C. Material Tested: Arocior 1254, Lot No. AK-38

D. Starting Date: May 8, 1969

E. Status of this Report: Completion of the first generation (Fo parents - F1a and F1b progeny)

F. Organization: A structural outline of the experiment is given in Table I.

NPC00009513

TABLE I

TEST MATERIAL: Aroclor 1254

Outline of Experiment

Fo Generation

| roup | Dietary Level (ppm) | Number of Animals Male | Female |
|---|---|---|---|
| | None | 8 | 16 |
| -I | 1 | 8 | 16 |
| -II | 10 | 8 | 16 |
| -III | 100 | 8 | 16 |

C. Means of Administration: Voluntary oral ingestion.

II. Summary

The first generation of a three-generation reproduction study of Aroclor 1254, Lot No. AK-38, at dietary levels of 1, 10 and 100 ppm has been completed. The following data were obtained during the first generation:

A. Progeny

The data from progeny of females fed either 1 or 10 ppm were normal in all respects.

Although no adverse findings were noted in the first litter of females fed 100 ppm, the number of pups delivered and weaned for the second litter was significantly lower than normal. The survival of pups that were delivered was also significantly lower than normal. In view of these findings, the females fed 100 ppm were remated for a third litter. The data from this litter substantiated the findings noted in the second litter.

B. Parental Animals

Females fed 100 ppm gained slightly less weight that did controls. Males fed 100 ppm and all rats fed either 1 or 10 ppm exhibited weight gains which were not different from control gains. There were no deaths which could be related to the ingestion of Aroclor 1254, and no unusual behavioral reactions were noted.

Gross pathologic examinations revealed no significant differences between test and control rats.

Statistical analysis upon organ weight data revealed several significant differences. Significantly higher liver to body weight ratios were recorded for all rats fed 100 ppm, however the absolute weights were significantly higher only in males. Males fed 1 or 10 ppm exhibited significantly lower liver weights and liver to body weight ratios. Elevation of thyroid weights was noted for both males and females fed 100 ppm.

Histopathologic examination of tissues and organs revealed nephritis in three of five males. No other significant differences were noted between test and control animals.

The mating index for the third litters of rats fed 100 ppm was somewhat low and, in the second matings of these animals, three females cannibalized their litters. All other mating indices, fertility indices, incidences of pregnancy and parturition and mean gestation times were normal.

Respectfully submitted,

INDUSTRIAL BIO-TEST LABORATORIES, INC.

Report prepared by: James B. Plank

James B. Plank
Group Leader
Rat Toxicity Department

Report approved by:

M. L. Kopinger, Ph.D.
Manager, Toxicology

NPC00009516

4. Reactions

No untoward behavioral reactions were observed among either test or control progeny.

Industrial BIO-TEST Laboratories, Inc.
1810 FRONTAGE ROAD,
NORTHBROOK, ILLINOIS 60062

# REPORT TO

# MONSANTO COMPANY

# THREE-GENERATION REPRODUCTION STUDY IN ALBINO RATS - AROCLOR 1260

## RESULTS OF THE FIRST GENERATION

SEPTEMBER 4, 1970

IBT NO. P7297

*Industrial* BIO-TEST *Laboratories, Inc.*
1810 FRONTAGE ROAD
NORTHBROOK, ILLINOIS 60062

September 4, 1970

Imer Wheeler
nto Company
d Department
Lindbergh Boulevard
is, Missouri 63166

Ir. Wheeler:

Re: IBT No. P7297 - Three-Generation Reproduction
Study in Albino Rats - Aroclor 1260

We are submitting herewith our laboratory report dated

ber 4, 1970, prepared in connection with the above study.

This report presents the results of the first generation.

Very truly yours,

J. C. Calandra
President

NPC00009519

IO-TEST Laboratories, Inc.

REPORT TO

MONSANTO COMPANY

THREE-GENERATION REPRODUCTION STUDY
IN ALBINO RATS - AROCLOR 1260

RESULTS OF THE FIRST GENERATION

SEPTEMBER 4, 1970

IBT NO. P7297

Outline of Study

A. Type and Length: Three-generation reproduction study

B. Animal Species Tested: Charles River albino rats

C. Material Tested: Aroclor 1260, Lot No. AK-3

D. Starting Date: May 8, 1969

E. Status of This Report: Completion of the first generation (Fo parents - F1a and F1b progeny)

F. Organization: A structural outline of the experiment is given in Table I.

NPC00009520

TABLE I

TEST MATERIAL: Aroclor 1260

Outline of Experiment

Fo Generation

| Dietary Level (ppm) | Number of Animals Male | Female |
|---|---|---|
| None | 8 | 16 |
| 1 | 8 | 16 |
| 10 | 8 | 16 |
| 100 | 8 | 16 |

Means of Administration: Voluntary oral ingestion.

NPC00009521

Summary

The results of the first generation of a three-generation reproduc-
study conducted on albino rats fed Aroclor 1260, Lot No. AK-3, at
ry levels of 1, 10 and 100 parts per million are presented below.

\. Progeny (F1a and F1b)

The number of pups delivered and weaned by treated females
ssentially the same as controls. In the second (F1b) litters,
es fed 100 ppm delivered slightly more stillborn pups than did
ols. Survival indices of progeny from treated females were not
ent from the control indices. The body weights of the weanlings
treated females were normal, and there were no unusual reactions
among any offspring.

. Parental Animals (Fo)

The body weights of the males and females receiving Aroclor
n their diet were about the same as those of the controls. One
l female died while giving birth during her first pregnancy.
were no untoward behavioral reactions noted.

Gross autopsy conducted upon males and females in all groups
ed no observable changes between test and control rats. The
eights and ratios of males fed 1 or 10 ppm were lower than con-
while the liver weights and ratios of males fed 100 ppm were
r than controls. Histopathologic examinations revealed no

anges in the tissues and organs which could be attributed to the inges-
n of Aroclor 1260.

The mating indices for both litters of rats fed 100 ppm were
ver than control. However, the index for the second (F1b) litter is
thin the normal range of the rat strain used. All other mating indices,
rtility indices, incidences of pregnancy and parturition and gestation
nes for treated animals compared favorably to the controls.

Respectfully submitted,

INDUSTRIAL BIO-TEST LABORATORIES, INC.

port prepared by: James B. Plank
James B. Plank
Group Leader
Rat Toxicity Department

port approved by: M. L. Keplinger
M. L. Keplinger, Ph.D.
Manager, Toxicology

Otis E. Fancher
Otis E. Fancher, Ph.D.
Scientific Director

tember 4, 1970

NPC00009523

:pah

3. Reactions

No untoward behavioral reactions were noted among the al animals in any group.

4. Autopsy Findings

a. Gross Autopsy Findings

At the time of sacrifice, gross autopsy revealed no ences between test and control animals.

b. Organ Weights and Ratios

Mean organ weights, organ to body weight ratios and to brain weight ratios are assembled in Tables VI through XIII.

4. Reactions

No untoward behavioral reactions were observed among either test or control progeny.

*Industrial* BIO-TEST *Laboratories, Inc.*
1810 FRONTAGE ROAD
NORTHBROOK, ILLINOIS 60062

November 12, 1971

II-3
BTL - 11-69
L

Mr. Elmer P. Wheeler
Monsanto Company
800 North Lindbergh Boulevard
St. Louis, Missouri 63166

Dear Mr. Wheeler:

Re: IBT No. B7298 - Two-Year Chronic Oral
Toxicity Study with Aroclor 1260 in Albino Rats

We are submitting herewith our laboratory report dated November 12, 1971, prepared in connection with the above study.

Very truly yours,

J. C. Calandra
President

JCC/kjl

PROJECT NO: B7298-71-69
REPORT FILE

*Industrial* BIO-TEST *Laboratories, Inc.*
1810 FRONTAGE ROAD
NORTHBROOK, ILLINOIS 60062

REPORT TO

MONSANTO COMPANY

# TWO-YEAR CHRONIC ORAL TOXICITY WITH AROCLOR 1260 IN ALBINO RATS

NOVEMBER 12, 1971

IBT NO. B7298

REPORT TO

MONSANTO COMPANY

# TWO-YEAR CHRONIC ORAL TOXICITY WITH AROCLOR 1260 IN ALBINO RATS

NOVEMBER 12, 1971

IBT NO. B7298

## I. Introduction

At the request of Monsanto Company, a two-year chronic oral toxicity study was conducted using albino rats to determine the potential toxicity of Aroclor 1260, Lot No. AK-3. The following report presents the results of this investigation.

## II. Summary

A two-year chronic toxicity study was conducted using albino rats fed diets containing 1 (T-I), 10 (T-II), or 100 (T-III) ppm Aroclor 1260.

Food consumption and body weight gains were not altered at any level of Aroclor 1260 fed. The number of animals dying and the time at which the deaths occurred did not vary among the test and control groups.

Hematological and clinical blood chemistry studies conducted after 3, 6, 9, 12, 18, or 24 months did not reveal any affects related to Aroclor 1260. Urine analyses failed to reveal any differences between test and control animals.

At sacrifice after 3, 6, or 12 months on test organ weights, organ to body weight and organ to brain weight ratios disclosed several randomly occurring intergroup differences. The lack of any consistent dose related response and the absence of any deleterious histopathologic change confirm that these differences were not related to the ingestion of Aroclor 1260.

At the final sacrifice after 24 months on test, the liver weights and liver to body weight or brain weight ratios were significantly elevated in the rats from the T-III group. Histologic examination of the livers from the T-III group revealed several animals with vacuolar change. This lesion is morphologically indicative of fatty degeneration. Specific fat stains confirmed the presence of fat in these vacuoles. Focal hypertrophy and focal hyperplasia were also found in the livers from animals fed Aroclor 1260.

Hyperplasia of the urinary bladder was found in an animal from the control group but not in any of the test animals.

The incidences and types of all tumors were about the same in all groups, including the control group, and are considered normal for rats of this age.

Respectfully submitted,

INDUSTRIAL BIO-TEST LABORATORIES, INC.

Report prepared by: Philip S. Smith

Philip Smith, B. S.
Assistant Toxicologist
Rat Toxicity

Report approved by: James B. Plank

James B. Plank
Senior Group Leader
Rat Toxicity

Paul L. Wright

Paul L. Wright, Ph. D.
Section Head, Toxicology

M. L. Kepfinger

M. L. Kepfinger, Ph. D.
Manager, Toxicology

November 12, 1971

lam:sjn

## III. Procedure

### A. Experimental Animals

The animals employed in the test were Charles River strain* albino rats. Four hundred rats (200 males and 200 females) were selected for the experiment, ear-punched with the animal number assigned and housed individually in standard wire-bottomed steel rat cages. Each cage bore a color-coded card identifying the rat with respect to project number, dose level assignment, individual animal number and sex.

### B. Organization of Groups

A structural outline of the experiment is given in Table I.

TABLE I

TEST MATERIAL: Aroclor 1260

Two-Year Chronic Oral Toxicity Study - Albino Rats

Outline of Experiment

| Group | Number of Animals Male | Female | Dietary Level (ppm) |
|---|---|---|---|
| Control | 50 | 50 | None Administered |
| T-I | 50 | 50 | 1 |
| T-II | 50 | 50 | 10 |
| T-III | 50 | 50 | 100 |

* Charles River Breeding Laboratories, North Wilmington, Mass.

C. Diets, Feeding and Food Consumption

All diets were prepared in the central diet room of this laboratory. The basic ration from which all diets were constructed was a standard, pulverized stock rat ration*. The diet for any given test group was prepared by blending the calculated amount of Aroclor 1260 with a pre-weighed portion of the stock ration in a Hobart mixer.

Fresh diets were prepared each week and every rat was offered an amount of food sufficient for ad libitum feeding.

Food consumption was recorded for five rats of each sex in every group weekly for the first three months and monthly for the next nine months. Thereafter, periodic spot checks were made.

D. Body Weights and Weight Gains

Initially, the body weight of each rat in every group was determined and recorded. Thereafter, individual weighings were made weekly for the first 13 weeks and monthly thereafter until the conclusion of the investigation.

E. Mortality and Reactions

Checks for mortality and abnormal behavioral reactions were made daily throughout the investigation.

F. Hematologic Studies and Urine Analyses

Blood studies, including determinations of hemoglobin concentrations, hemotocrit value, erythrocyte count and both total and differential

* Purina Rat Chow, Ralston Purina Co., St. Louis, Mo.

leukocyte counts, were conducted upon five males and five females from both the control and 100 ppm groups.

Urine analyses for the presence of glucose, albumin, microscopic elements, and determinations of pH and specific gravity were conducted upon urine samples at the same time intervals and from the same animals as those employed for the blood studies.

These studies were conducted after 3, 6, 9, 12, 18, and 24 months of feeding.

G. Clinical Blood Chemistry Studies

Determinations of blood urea nitrogen concentration (BUN), serum alkaline phosphatase activity (SAP), fasted blood glucose concentration and serum glutamic-pyruvic transaminase activity (SGPT) were conducted after 3, 6, 9, 12, 18, and 24 months of feeding upon the same rats as were the routine blood studies.

H. Pathologic Studies

When an animal succumbed during the test period, a gross autopsy was performed. In those cases when postmortem autolysis was not advanced, representative tissues were taken and preserved in ten percent formalin solution for possible future histopathologic study.

After 3, 6, and 12 months of testing, five animals of each sex from each group were sacrificed and subjected to complete gross pathologic examinations.

Absolute organ weights were recorded and organ to body weight and organ to brain weight ratios computed. The following organs were included: liver, kidneys, spleen, gonads, heart and brain. These data were then subjected to statistical analysis. An Analysis of Variance was conducted first and significant effects disclosed by that treatment were further studied by "t"-tests.

Complete microscopic examinations were conducted upon the tissues and organs from all control and 100 ppm group rats sacrificed after 3, 6, or 12 months of testing. The following tissues and organs were included in the examinations: heart, trachea, lungs, liver, pancreas, esophagus, stomach, small intestine (duodenum, jejunum and ileum), caecum, colon, spleen, lymph nodes, kidneys, urinary bladder, gonads, prostate gland, seminal vesicles, uterus, pituitary gland, adrenal glands, salivary glands, thyroid gland, parathyroid glands, skeletal muscle, sternum, bone, peripheral nerve, spinal cord and brain (cerebrum, cerebellum and pons).

The aforementioned tissues were prepared and stained with Hematoxylin-Eosin stain.

After two years of feeding, all surviving animals were sacrificed and subjected to complete gross pathologic examination.

The absolute weights of the liver, kidneys, spleen, gonads, heart and brain were recorded immediately after sacrifice.

Microscopic examinations were conducted upon tissues and organs from selected animals dying during the experiment and upon all animals from the final sacrifice.

Tissues and organs examined were the same as those previously mentioned.

I. Tumor Incidence and Classification

A tabulation of the incidence of tumor formation in each group was made at the conclusion of the investigation. In addition, tumor data for individual animals including location, weight, size and pathologic classification were recorded.

## IV. Results

### A. Body Weights and Weight Gains

The male and female body weight data are assembled in Table II. The data in this table also include the 3, 12 and 24 month total weight gains.

There were no significant effects due to ingestion of the compound

TABLE II

TEST MATERIAL: Aroclor 1260

Two-Year Chronic Oral Toxicity Study - Albino Rats

Body Weight and Total Weight Gain Data

Summary of Mean Values

| Dietary Level of Aroclor 1260 (ppm) | Sex | Body Weight (grams) Week: 0 | 1 | 2 | 3 | 4 | 5 | 6 | 7 | 8 | 9 | 10 | 11 | 12 | 13 | 90-Day Total Weight Gain (grams/rat) |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Control | M | 133 | 203 | 245 | 313 | 362 | 373 | 391 | 409 | 424 | 440 | 440 | 449 | 467 | 474 | 341 |
| | F | 136 | 178 | 190 | 219 | 244 | 254 | 259 | 265 | 270 | 276 | 281 | 278 | 290 | 288 | 152 |
| 1 | M | 133 | 192 | 250 | 322 | 361 | 374 | 394 | 416 | 428 | 448 | 450 | 474 | 479 | 490 | 357 |
| | F | 136 | 167 | 189 | 218 | 241 | 252 | 256 | 261 | 268 | 273 | 277 | 281 | 290 | 288 | 152 |
| 10 | M | 133 | 204 | 245 | 317 | 368 | 382 | 400 | 417 | 436 | 448 | 461 | 467 | 475 | 481 | 348 |
| | F | 136 | 174 | 190 | 217 | 242 | 254 | 260 | 263 | 268 | 274 | 281 | 279 | 285 | 288 | 152 |
| 100 | M | 133 | 208 | 247 | 319 | 363 | 375 | 394 | 425 | 437 | 451 | 460 | 479 | 486 | 500 | 367 |
| | F | 136 | 173 | 188 | 222 | 240 | 253 | 258 | 262 | 265 | 267 | 270 | 275 | 275 | 280 | 144 |

TABLE II continued

TEST MATERIAL: Aroclor 1260

Two-Year Chronic Oral Toxicity Study - Albino Rats

Body Weight and Total Weight Gain Data

Summary of Mean Values

| Dietary Level of Aroclor 1260 (ppm) | Sex | Body Weight (grams) Month: 4 | 5 | 6 | 7 | 8 | 9 | 10 | 11 | 12 | 12-Month Total Weight Gain (grams/rat) |
|---|---|---|---|---|---|---|---|---|---|---|---|
| Control | M | 498 | 533 | 565 | 593 | 622 | 642 | 667 | 688 | 701 | 568 |
| | F | 297 | 310 | 322 | 333 | 345 | 359 | 374 | 383 | 390 | 254 |
| 1 | M | 516 | 547 | 578 | 600 | 628 | 643 | 668 | 688 | 699 | 566 |
| | F | 298 | 307 | 314 | 321 | 333 | 340 | 353 | 367 | 379 | 243 |
| 10 | M | 496 | 526 | 551 | 581 | 606 | 633 | 660 | 685 | 709 | 576 |
| | F | 291 | 306 | 314 | 331 | 340 | 358 | 370 | 381 | 388 | 252 |
| 100 | M | 522 | 555 | 580 | 607 | 633 | 650 | 681 | 707 | 717 | 584 |
| | F | 289 | 302 | 307 | 314 | 326 | 333 | 342 | 347 | 363 | 227 |

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TABLE II continued

TEST MATERIAL: Aroclor 1260

Two-Year Chronic Oral Toxicity Study - Albino Rats

Body Weight and Total Weight Gain Data

Summary of Mean Values

| Dietary Level of Aroclor 1260 (ppm) | Sex | Body Weight (grams) Month: 13 | 14 | 15 | 16 | 17 | 18 | 19 | 20 | 21 | 22 | 23 | 24 | 24-Month Total Weight Gain (grams/rat) |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Control | M | 728 | 746 | 759 | 768 | 769 | 767 | 760 | 758 | 739 | 726 | 687 | 642 | 509 |
| | F | 426 | 444 | 466 | 481 | 500 | 515 | 533 | 547 | 551 | 550 | 564 | 551 | 415 |
| 1 | M | 721 | 740 | 760 | 768 | 776 | 774 | 762 | 753 | 733 | 718 | 715 | 654 | 521 |
| | F | 390 | 427 | 466 | 433 | 445 | 476 | 481 | 493 | 514 | 530 | 484 | 525 | 385 |
| 10 | M | 728 | 745 | 776 | 761 | 768 | 780 | 774 | 761 | 735 | 715 | 683 | 645 | 512 |
| | F | 416 | 434 | 482 | 449 | 473 | 508 | 519 | 530 | 536 | 542 | 536 | 584 | 448 |
| 100 | M | 746 | 760 | 772 | 775 | 774 | 769 | 760 | 750 | 733 | 714 | 711 | 641 | 508 |
| | F | 380 | 386 | 417 | 414 | 435 | 453 | 471 | 481 | 500 | 507 | 485 | 537 | 401 |

NEV 010003

B. Food Consumption

The data obtained from the food consumption measurements conducted during the first 12 months of feeding are summarized in Table III.

These data revealed no significant difference between test and control rats. The periodic checks conducted during the remainder of the investigation also revealed no outstanding differences between test and control animals.

TABLE III

TEST MATERIAL: Aroclor 1260

Two-Year Chronic Oral Toxicity Study - Albino Rats

Food Consumption Data

Summary of Mean Values

| Dietary Level of Aroclor 1260 (ppm) | Sex | Food Consumption (grams/rat/seven days) Week: 1 | 2 | 3 | 4 | 5 | 6 | 7 | 8 | 9 | 10 | 11 | 12 | 13 | Average Food Consumption (grams/rat/seven days) |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Control | M | 94 | 160 | 175 | 163 | 137 | 137 | 225 | 197 | 204 | 188 | 191 | 194 | 196 | 174 |
| | F | 73 | 140 | 144 | 107 | 104 | 133 | 208 | 154 | 165 | 151 | 146 | 150 | 151 | 140 |
| 1 | M | 118 | 154 | 151 | 169 | 201 | 116 | 200 | 198 | 201 | 193 | 189 | 187 | 194 | 175 |
| | F | 95 | 118 | 97 | 139 | 160 | 107 | 162 | 139 | 165 | 149 | 150 | 143 | 147 | 136 |
| 10 | M | 106 | 146 | 144 | 142 | 190 | 136 | 196 | 214 | 214 | 192 | 191 | 199 | 200 | 175 |
| | F | 94 | 124 | 128 | 124 | 179 | 90 | 190 | 167 | 155 | 151 | 150 | 149 | 152 | 143 |
| 100 | M | 121 | 150 | 156 | 117 | 187 | 124 | 121 | 190 | 196 | 186 | 184 | 199 | 189 | 163 |
| | F | 89 | 93 | 123 | 151 | 151 | 101 | 193 | 136 | 140 | 144 | 144 | 147 | 146 | 135 |

TABLE III continued

TEST MATERIAL: Aroclor 1260

Two-Year Chronic Oral Toxicity Study - Albino Rats

Food Consumption Data

Summary of Mean Values

| Dietary Level of Aroclor 1260 (ppm) | Sex | Food Consumption (grams/rat/seven days) Month: 4 | 5 | 6 | 7 | 8 | 9 | 10 | 11 | 12 | Average Food Consumption (grams/rat/seven days) |
|---|---|---|---|---|---|---|---|---|---|---|---|
| Control | M | 196 | 189 | 168 | 147 | 163 | 184 | 179 | 170 | 187 | 176 |
| | F | 148 | 154 | 136 | 121 | 138 | 152 | 160 | 141 | 127 | 142 |
| 1 | M | 196 | 200 | 189 | 192 | 179 | 181 | 182 | 172 | 184 | 186 |
| | F | 148 | 163 | 141 | 163 | 160 | 153 | 157 | 143 | 132 | 151 |
| 10 | M | 186 | 191 | 188 | 193 | 197 | 197 | 168 | 164 | 196 | 186 |
| | F | 142 | 138 | 145 | 144 | 144 | 151 | 136 | 139 | 141 | 142 |
| 100 | M | 179 | 170 | 180 | 188 | 186 | 166 | 181 | 188 | 180 | 180 |
| | F | 156 | 127 | 147 | 144 | 134 | 132 | 162 | 149 | 139 | 143 |

C. Mortality and Reactions

A frequency distribution of natural deaths occurring during the investigation appears in Table IV.

No untoward behavioral reactions were noted among any of the animals employed in the investigation.

TABLE IV

TEST MATERIAL: Aroclor 1260

Two-Year Chronic Oral Toxicity Study - Albino Rats

Mortality Data

Frequency Distribution of Deaths

| Dietary Level (ppm) | Sex | Frequency Distribution of Deaths Months: 1-3 | 4-6 | 7-9 | 10-12 | 13-15 | 16-18 | 19-21 | 22-24 | Total Dead Number Tested* |
|---|---|---|---|---|---|---|---|---|---|---|
| Control | M | 0 | 0 | 0 | 2 | 1 | 7 | 6 | 9 | 25/35 |
| | F | 0 | 0 | 0 | 1 | 3 | 5 | 7 | 3 | 19/35 |
| 1 | M | 0 | 0 | 1 | 1 | 4 | 7 | 5 | 8 | 26/35 |
| | F | 0 | 0 | 0 | 2 | 3 | 3 | 6 | 5 | 19/35 |
| 10 | M | 1 | 1 | 0 | 2 | 4 | 7 | 8 | 6 | 29/35 |
| | F | 0 | 0 | 0 | 3 | 2 | 4 | 5 | 3 | 17/35 |
| 100 | M | 1 | 0 | 0 | 2 | 2 | 8 | 6 | 9 | 29/35 |
| | F | 0 | 2 | 2 | 0 | 3 | 5 | 7 | 4 | 23/35 |

* Does not include the five animals of each sex from every group sacrificed for interim pathologic studies.

D. Hematologic and Clinical Blood Chemistry Studies

Mean male and female hematologic and clinical blood chemistry data are summarized in Tables V through IX.

Values for all parameters investigated were within the normal range for the albino rat. No significant differences between test and control values were observed.

TABLE V

TEST MATERIAL: Aroclor 1260

Two-Year Chronic Oral Toxicity Study - Albino Rats

Hematologic Data

Summary of Mean Values

| Dietary Level (ppm) | Sex | Total Leukocyte Count (thousands/mm$^3$) Month: 3 | 6 | 9 | 12 | 18 | 24 | Erythrocyte Count (millions/mm$^3$) Month: 3 | 6 | 9 | 12 | 18 | 24 |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Control | M | 14.0 | 15.4 | 10.1 | 11.1 | 9.9 | 11.2 | 8.1 | 8.2 | 8.7 | 8.8 | 7.6 | 6.5 |
| | F | 9.1 | 9.4 | 6.7 | 7.6 | 8.8 | 7.6 | 7.5 | 7.7 | 7.3 | 7.8 | 7.7 | 6.8 |
| 100 | M | 10.8 | 13.1 | 7.9 | 12.8 | 8.6 | 8.4 | 7.5 | 8.1 | 8.0 | 7.6 | 8.0 | 7.6 |
| | F | 13.2 | 8.2 | 7.9 | 6.5 | 7.8 | 7.4 | 7.3 | 7.5 | 7.3 | 7.5 | 7.4 | 6.4 |

TABLE VI

TEST MATERIAL: Aroclor 1260

Two-Year Chronic Oral Toxicity Study - Albino Rats

Hematologic Data

Summary of Mean Values

| Dietary Level (ppm) | Sex | Hemoglobin Concentration (g/100 ml) Month: 3 | 6 | 9 | 12 | 18 | 24 | Hematocrit Value (%) Month: 3 | 6 | 9 | 12 | 18 | 24 |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Control | M | 17.1 | 17.7 | 15.7 | 17.6 | 16.1 | 13.6 | 51 | 49 | 48 | 50 | 41 | 35 |
| | F | 16.9 | 17.4 | 14.1 | 16.7 | 16.4 | 14.6 | 50 | 47 | 43 | 46 | 42 | 38 |
| 100 | M | 16.1 | 17.2 | 14.0 | 15.3 | 15.9 | 14.5 | 49 | 47 | 43 | 43 | 41 | 38 |
| | F | 16.3 | 17.0 | 13.7 | 15.6 | 15.3 | 12.9 | 48 | 47 | 41 | 44 | 39 | 32 |

TABLE VII

TEST MATERIAL: Aroclor 1260

Two-Year Chronic Oral Toxicity Study - Albino Rats

Hematologic Data

Summary of Mean Values

| Dietary Level (ppm) | Sex | Differential Leukocyte Count (Number of Cells per Hundred) Lymphocytes Month: 3 | 6 | 9 | 12 | 18 | 24 | Neutrophils Month: 3 | 6 | 9 | 12 | 18 | 24 |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Control | M | 82.0 | 86.6 | 80.4 | 78.8 | 79.0 | 65.4 | 16.0 | 11.4 | 18.6 | 20.6 | 20.0 | 32.2 |
| | F | 83.0 | 86.6 | 86.8 | 89.0 | 70.2 | 61.4 | 15.4 | 11.6 | 11.8 | 10.0 | 29.2 | 36.0 |
| 100 | M | 85.8 | 85.4 | 83.4 | 72.4 | 73.8 | 66.6 | 12.6 | 13.8 | 15.8 | 22.6 | 24.6 | 31.6 |
| | F | 92.0 | 89.6 | 78.8 | 82.2 | 80.6 | 65.6 | 7.6 | 10.0 | 19.4 | 17.0 | 17.4 | 34.2 |

TABLE VII continued

TEST MATERIAL: Aroclor 1260

Two-Year Chronic Oral Toxicity Study - Albino Rats

Hematologic Data

Summary of Mean Values

| Dietary Level (ppm) | Sex | Differential Leukocyte Count (Number of Cells per Hundred) Monocytes Month: 3 | 6 | 9 | 12 | 18 | 24 | Eosinophils Month: 3 | 6 | 9 | 12 | 18 | 24 |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Control | M | 0.6 | 0.0 | 0.0 | 0.4 | 0.2 | 1.2 | 1.4 | 2.0 | 1.0 | 0.2 | 0.8 | 1.6 |
| | F | 0.6 | 0.0 | 0.0 | 0.0 | 0.2 | 1.0 | 1.0 | 1.8 | 1.4 | 0.2 | 0.6 | 1.6 |
| 100 | M | 0.0 | 0.0 | 0.0 | 0.0 | 0.4 | 0.8 | 1.4 | 0.8 | 0.8 | 1.0 | 1.2 | 1.0 |
| | F | 0.0 | 0.0 | 0.0 | 0.0 | 0.4 | 0.2 | 0.4 | 0.4 | 0.8 | 0.8 | 1.6 | 0.0 |

TABLE VII continued

TEST MATERIAL: Aroclor 1260

Two-Year Chronic Oral Toxicity Study - Albino Rats

Hematologic Data

Summary of Mean Values

| Dietary Level (ppm) | Sex | Differential Leukocyte Count (Number of Cells per Hundred) Basophils Month: 3 | 6 | 9 | 12 | 18 | 24 |
|---|---|---|---|---|---|---|---|
| Control | M | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 |
| | F | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 |
| 100 | M | 0.2 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 |
| | F | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 |

TABLE VIII

TEST MATERIAL: Aroclor 1260

Two-Year Chronic Oral Toxicity Study - Albino Rats

Clinical Blood Chemistry Data

Summary of Mean Values

| Dietary Level (ppm) | Sex | Serum Glutamic-Pyruvic Transaminase Activity (Dade Units) Month: 3 | 6 | 9 | 12 | 18 | 24 | Blood Urea Nitrogen Concentration (mgs %) Month: 3 | 6 | 9 | 12 | 18 | 24 |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Control | M | 38 | 48 | 45 | 45 | 42 | 36 | 17 | 15 | 12 | 10 | 14 | 15 |
| | F | 33 | 39 | 38 | 42 | 32 | 30 | 14 | 16 | 15 | 9 | 13 | 11 |
| 100 | M | 33 | 39 | 42 | 43 | 31 | 36 | 14 | 15 | 17 | 16 | 16 | 15 |
| | F | 29 | 37 | 50 | 40 | 32 | 33 | 15 | 15 | 14 | 13 | 12 | 14 |

TABLE IX

TEST MATERIAL: Aroclor 1260

Two-Year Chronic Oral Toxicity Study - Albino Rats

Clinical Blood Chemistry Data

Summary of Mean Values

| Dietary Level (ppm) | Sex | Fasted Blood Glucose Concentration (mgs%) Month: 3 | 6 | 9 | 12 | 18 | 24 | Serum Alkaline Phosphatase Activity (King-Armstrong Units) Month: 3 | 6 | 9 | 12 | 18 | 24 |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Control | M | 128 | 123 | 89 | 88 | 138 | 99 | 28.6 | 24.7 | 21.5 | 20.3 | 16.2 | 22.3 |
| | F | 130 | 118 | 115 | 92 | 134 | 109 | 18.9 | 17.8 | 10.6 | 11.9 | 16.8 | 20.2 |
| 100 | M | 118 | 118 | 116 | 135 | 146 | 98 | 37.8 | 21.8 | 21.9 | 23.9 | 11.9 | 16.1 |
| | F | 117 | 116 | 115 | 138 | 146 | 94 | 12.0 | 12.1 | 7.6 | 11.7 | 7.8 | 12.4 |

E. Urine Analyses

Mean male and female urine analyses data are summarized in Tables X through XII.

Determinations for glucose concentration were negative for all rats at every interval of examination.

No differences were noted between the urine from control and test animals at the intervals of examination.

# TABLE X

## TEST MATERIAL: Aroclor 1260

## Two-Year Chronic Oral Toxicity Study - Albino Rats

## Urine Analyses Data

## Summary of Mean Values

| Dietary Level (ppm) | Sex | Glucose Month: 3 | 6 | 9 | 12 | 18 | 24 | Albumin Month: 3 | 6 | 9 | 12 | 18 | 24 |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Control | M | n | n | n | n | n | n | n | n | t | t | +2 | +3 |
| | F | n | n | n | n | n | n | n | n | n | n | t | +2 |
| 100 | M | n | n | n | n | n | n | n | t | t | t | +1 | +3 |
| | F | n | n | n | n | n | n | n | n | n | n | +1 | +4 |

Key:
n = negative
t = trace; less than 30 mg/100 ml urine
+1 = 30 to 100 mg/100 ml urine
+2 = 100 to 300 mg/100 ml urine
+3 = 300 to 500 mg/100 ml urine
+4 = more than 500 mg/100 ml urine

TABLE XI

TEST MATERIAL: Aroclor 1260

Two-Year Chronic Oral Toxicity Study - Albino Rats

Urine Analyses Data

Summary of Mean Values

| Dietary Level (ppm) | Sex | Microscopic Elements Month: 3 | 6 | 9 | 12 | 18 | 24 | pH Month: 3 | 6 | 9 | 12 | 18 | 24 |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Control | M | N | N | N | N | N | +1 | 7 | 7 | 6 | 7 | 7 | 6 |
| | F | N | N | N | N | t | t | 8 | 8 | 7 | 8 | 6 | 6 |
| 100 | M | N | N | N | N | N | t | 7 | 7 | 6 | 7 | 6 | 6 |
| | F | N | N | N | N | t | t | 7 | 8 | 6 | 7 | 6 | 6 |

Key:
N= Normal
t = trace; less than 5 per low power field
+1 = 5 to 10 per low power field
+2 = 10 to 15 per low power field
+3 = more than 15 per low power field

TABLE XII

TEST MATERIAL: Aroclor 1260

Two-Year Chronic Oral Toxicity Study - Albino Rats

Urine Analyses Data

Summary of Mean Values

| Dietary Level (ppm) | Sex | Specific Gravity Month: 3 | 6 | 9 | 12 | 18 | 24 |
|---|---|---|---|---|---|---|---|
| Control | M | 1.029 | 1.022 | 1.033 | 1.027 | 1.042 | 1.051 |
| | F | 1.020 | 1.019 | 1.025 | 1.024 | 1.032 | 1.039 |
| 100 | M | 1.021 | 1.025 | 1.031 | 1.030 | 1.043 | 1.039 |
| | F | 1.016 | 1.023 | 1.035 | 1.029 | 1.028 | 1.045 |

F. Pathologic Studies

1. Three-Month Sacrifice

a. Gross Pathologic Findings

No outstanding differences were noted between test and control rats upon gross pathological examination.

b. Organ Weight and Organ to Body and Organ to Brain Weight Ratio Data

The results of the statistical analyses conducted on absolute organ weights, organ to body weight and organ to brain weight ratios are summarized in Tables XIII through XVIII.

Significant differences between a test group and the control group are designated by asterisks following the test values.

Organ weights, organ to body weight and organ to brain weight ratios disclosed several randomly occurring intergroup differences. The lack of any consistent dose related response and the absence of any deleterious histopathologic change confirm that these differences were not related to the ingestion of Aroclor 1260.

TABLE XIII

TEST MATERIAL: Aroclor 1260

Two-Year Chronic Oral Toxicity Study - Albino Rats

Organ Weight and Ratio Data

Three-Month Sacrifice

Summary of Mean Values

Organ: Liver

| Dietary Level of Aroclor 1260 (ppm) | Organ Weight (g) | | Organ/Body Weight Ratio (g/100 g) | | Organ/Brain Weight Ratio (g/g) | |
|---|---|---|---|---|---|---|
| | Males | Females | Males | Females | Males | Females |
| Control | 21.4 | 13.0 | 4.13 | 4.16 | 10.3 | 6.82 |
| 1 | 20.4 | 13.2 | 3.90 | 4.59 | 9.98 | 6.86 |
| 10 | 24.0** | 12.2 | 4.48 | 3.61 | 11.8 | 6.38 |
| 100 | 29.5** | 14.5 | 5.73** | 4.58 | 14.4** | 7.72 |

** Statistically significant difference at the 99 percent confidence level.

TABLE XIV

TEST MATERIAL: Aroclor 1260

Two-Year Chronic Oral Toxicity Study - Albino Rats

Organ Weight and Ratio Data

Three-Month Sacrifice

Summary of Mean Values

Organ: Kidneys

| Dietary Level of Aroclor 1260 (ppm) | Organ Weight (g) | | Organ/Body Weight Ratio (g/100 g) | | Organ/Brain Weight Ratio (g/g) | |
|---|---|---|---|---|---|---|
| | Males | Females | Males | Females | Males | Females |
| Control | 4.03 | 2.49 | 0.782 | 0.802 | 1.95 | 1.31 |
| 1 | 4.37 | 2.62 | 0.838 | 0.911 | 2.14 | 1.36 |
| 10 | 4.00 | 2.61 | 0.747 | 0.782 | 1.96 | 1.37 |
| 100 | 4.00 | 2.52 | 0.779 | 0.793 | 1.96 | 1.35 |

TABLE XV

TEST MATERIAL: Aroclor 1260

Two-Year Chronic Oral Toxicity Study - Albino Rats

Organ Weight and Ratio Data

Three-Month Sacrifice

Summary of Mean Values

Organ: Spleen

| Dietary Level of Aroclor 1260 (ppm) | Organ Weight (g) | | Organ/Body Weight Ratio (g/100 g) | | Organ/Brain Weight Ratio (g/g) | |
|---|---|---|---|---|---|---|
| | Males | Females | Males | Females | Males | Females |
| Control | 0.766 | 0.578 | 0.150 | 0.184 | 0.371 | 0.302 |
| 1 | 0.846 | 0.516 | 0.161 | 0.180 | 0.412 | 0.269 |
| 10 | 0.838 | 0.632 | 0.156 | 0.189 | 0.411 | 0.331 |
| 100 | 0.718 | 0.556 | 0.140 | 0.177 | 0.352 | 0.297 |

TABLE XVI

TEST MATERIAL: Aroclor 1260

Two-Year Chronic Oral Toxicity Study - Albino Rats

Organ Weight and Ratio Data

Three-Month Sacrifice

Summary of Mean Values

Organ: Gonads

| Dietary Level of Aroclor 1260 (ppm) | Organ Weight (g) | | Organ/Body Weight Ratio (g/100 g) | | Organ/Brain Weight Ratio (g/g) | |
|---|---|---|---|---|---|---|
| | Males | Females | Males | Females | Males | Females |
| Control | 3.60 | 0.114 | 0.698 | 0.0370 | 1.74 | 0.0599 |
| 1 | 3.67 | 0.128 | 0.704 | 0.0450 | 1.80 | 0.0666 |
| 10 | 3.83 | 0.121 | 0.715 | 0.0368 | 1.89 | 0.0636 |
| 100 | 3.71 | 0.138 | 0.725 | 0.0429 | 1.81 | 0.0728 |

TABLE XVII

TEST MATERIAL: Aroclor 1260

Two-Year Chronic Oral Toxicity Study - Albino Rats

Organ Weight and Ratio Data

Three-Month Sacrifice

Summary of Mean Values

Organ: Heart

| Dietary Level of Aroclor 1260 (ppm) | Organ Weight (g) | | Organ/Body Weight Ratio (g/100 g) | | Organ/Brain Weight Ratio (g/g) | |
|---|---|---|---|---|---|---|
| | Males | Females | Males | Females | Males | Females |
| Control | 1.59 | 1.05 | 0.307 | 0.339 | 0.767 | 0.551 |
| 1 | 1.57 | 1.01 | 0.300 | 0.353 | 0.765 | 0.526 |
| 10 | 1.58 | 1.04 | 0.296 | 0.311 | 0.781 | 0.546 |
| 100 | 1.52 | 0.946 | 0.298 | 0.297 | 0.743 | 0.505 |

TABLE XVIII

TEST MATERIAL: Aroclor 1260

Two-Year Chronic Oral Toxicity Study - Albino Rats

Organ Weight and Ratio Data

Three-Month Sacrifice

Summary of Mean Values

Organ: Brain

| Dietary Level of Aroclor 1260 (ppm) | Organ Weight (g) | | Organ/Body Weight Ratio (g/100 g) | | Final Body Weights of Sacrificed Rats (g) | |
|---|---|---|---|---|---|---|
| | Males | Females | Males | Females | Males | Females |
| Control | 2.07 | 1.91 | 0.402 | 0.616 | 518 | 314 |
| 1 | 2.05 | 1.92 | 0.392 | 0.672 | 523 | 287 |
| 10 | 2.04 | 1.91 | 0.380 | 0.572 | 536 | 337 |
| 100 | 2.05 | 1.89 | 0.400 | 0.593 | 517 | 321 |

c. Histopathologic Findings

Histopathologic examination of tissues and organs taken from all animals from both the control and 100 ppm groups was conducted.

Tables XIX and XX list all histopathologic changes noted.

All of the lesions noted in the microscopic examination of tissues were those of spontaneous disease and are not unusual for the albino rat. The most frequent findings were lesions in the trachea and lungs, indicating chronic murine pneumonia. These occurred in the control as well as the rats fed Aroclor 1260.

TABLE XIX

TEST MATERIAL: Aroclor 1260

Two-Year Chronic Oral Toxicity Study - Albino Rats

Histopathologic Changes

Three-Month Sacrifice

Group: Control

| Number of Animals | Organ Examined | Findings | Incidence | Average Grade |
|---|---|---|---|---|
| 5 Males | Trachea | Tracheitis | 1 | 1.0 |
| | Lung | Chronic respiratory disease | 1 | 1.0 |
| | Urinary bladder | Hyperplasia | 3 | 1.0 |
| 5 Females | Lung | Chronic respiratory disease | 1 | 1.0 |

All other tissues and organs were normal histologically.

Grading System

0.5 = minimal
1.0 = slight
2.0 = mild
3.0 = moderate
4.0 = severe
5.0 = extreme

TABLE XX

TEST MATERIAL: Aroclor 1260

Two-Year Chronic Oral Toxicity Study - Albino Rats

Histopathologic Changes

Three-Month Sacrifice

Group: 100 ppm

| Number of Animals | Organ Examined | Findings | Incidence | Average Grade |
|---|---|---|---|---|
| 5 Males | Trachea | Tracheitis | 4 | 1.0 |
| | Lung | Chronic respiratory disease | 2 | 1.0 |
| 5 Females | Trachea | Tracheitis | 3 | 1.0 |
| | Lung | Chronic respiratory disease | 2 | 1.0 |
| | Kidney | Focal lymphoid infiltration | 1 | 1.0 |

All other tissues and organs were normal histologically.

Grading System

0.5 = minimal
1.0 = slight
2.0 = mild
3.0 = moderate
4.0 = severe
5.0 = extreme

2. Six-Month Sacrifice

a. Gross Pathologic Findings

No outstanding differences were noted between test and control animals.

b. Organ Weight and Organ to Body and Organ to Brain Weight Ratio Data

The results of the statistical analyses conducted on absolute organ weights, organ to body weight and organ to brain weight ratios are summarized in Tables XXI through XXVI.

Organ weights, organ to body weight and organ to brain weight ratios disclosed several randomly occurring intergroup differences. The lack of any consistent dose related response and the absence of any deleterious histopathologic change confirm that these differences were not related to the ingestion of Aroclor 1260.

TABLE XXI

TEST MATERIAL: Aroclor 1260

Two-Year Chronic Oral Toxicity Study - Albino Rats

Organ Weight and Ratio Data

Six-Month Sacrifice

Summary of Mean Values

Organ: Liver

| Dietary Level of Aroclor 1260 (ppm) | Organ Weight (g) | | Organ/Body Weight Ratio (g/100 g) | | Organ/Brain Weight Ratio (g/g) | |
|---|---|---|---|---|---|---|
| | Males | Females | Males | Females | Males | Females |
| Control | 22.9 | 13.1 | 3.74 | 3.77 | 11.7 | 6.71 |
| 1 | 14.8** | 15.1 | 2.92** | 3.74 | 7.05** | 7.86 |
| 10 | 19.0* | 10.5** | 3.31* | 3.08* | 9.45* | 6.07 |
| 100 | 24.4 | 11.6 | 4.69* | 4.03 | 12.3 | 6.17 |

* Statistically significant difference at the 95 percent confidence level.
** Statistically significant difference at the 99 percent confidence level.

TABLE XXII

TEST MATERIAL: Aroclor 1260

Two-Year Chronic Oral Toxicity Study - Albino Rats

Organ Weight and Ratio Data

Six-Month Sacrifice

Summary of Mean Values

Organ: Kidneys

| Dietary Level of Aroclor 1260 (ppm) | Organ Weight (g) | | Organ/Body Weight Ratio (g/100 g) | | Organ/Brain Weight Ratio (g/g) | |
|---|---|---|---|---|---|---|
| | Males | Females | Males | Females | Males | Females |
| Control | 4.04 | 2.79 | 0.664 | 0.806 | 2.06 | 1.43 |
| 1 | 3.64 | 2.73 | 0.716 | 0.674* | 1.73 | 1.42 |
| 10 | 3.85 | 2.35 | 0.670 | 0.684* | 1.92 | 1.36 |
| 100 | 3.92 | 2.35 | 0.753 | 0.814 | 1.97 | 1.25 |

* Statistically significant difference at the 95 percent confidence level.

TABLE XXIII

TEST MATERIAL: Aroclor 1260

Two-Year Chronic Oral Toxicity Study - Albino Rats

Organ Weight and Ratio Data

Six-Month Sacrifice

Summary of Mean Values

Organ: Spleen

| Dietary Level of Aroclor 1260 (ppm) | Organ Weight (g) | | Organ/Body Weight Ratio (g/100 g) | | Organ/Brain Weight Ratio (g/g) | |
|---|---|---|---|---|---|---|
| | Males | Females | Males | Females | Males | Females |
| Control | 0.752 | 0.684 | 0.122 | 0.196 | 0.384 | 0.350 |
| 1 | 0.754 | 0.908 | 0.148* | 0.220 | 0.359 | 0.472 |
| 10 | 0.836 | 0.566* | 0.146* | 0.167 | 0.416 | 0.327 |
| 100 | 0.636 | 0.443* | 0.122 | 0.151 | 0.320 | 0.236* |

* Statistically significant difference at the 95 percent confidence level.

TABLE XXIV

TEST MATERIAL: Aroclor 1260

Two-Year Chronic Oral Toxicity Study - Albino Rats

Organ Weight and Ratio Data

Six-Month Sacrifice

Summary of Mean Values

Organ: Gonads

| Dietary Level of Aroclor 1260 (ppm) | Organ Weight (g) | | Organ/Body Weight Ratio (g/100 g) | | Organ/Brain Weight Ratio (g/g) | |
|---|---|---|---|---|---|---|
| | Males | Females | Males | Females | Males | Females |
| Control | 3.55 | 0.0960 | 0.593 | 0.0279 | 1.81 | 0.0492 |
| 1 | 3.23 | 0.120* | 0.636 | 0.0297 | 1.54 | 0.0625* |
| 10 | 3.56 | 0.0968 | 0.620 | 0.0287 | 1.77 | 0.0560 |
| 100 | 3.53 | 0.0710* | 0.686 | 0.0250 | 1.77 | 0.0378* |

* Statistically significant difference at the 95 percent confidence level.

TABLE XXV

TEST MATERIAL: Aroclor 1260

Two-Year Chronic Oral Toxicity Study - Albino Rats

Organ Weight and Ratio Data

Six-Month Sacrifice

Summary of Mean Values

Organ: Heart

| Dietary Level of Aroclor 1260 (ppm) | Organ Weight (g) | | Organ/Body Weight Ratio (g/100 g) | | Organ/Brain Weight Ratio (g/g) | |
|---|---|---|---|---|---|---|
| | Males | Females | Males | Females | Males | Females |
| Control | 1.70 | 1.05 | 0.282 | 0.302 | 0.867 | 0.538 |
| 1 | 1.45 | 1.12 | 0.286 | 0.277 | 0.690 | 0.583 |
| 10 | 1.63 | 1.05 | 0.283 | 0.307 | 0.810 | 0.606 |
| 100 | 1.49 | 1.06 | 0.286 | 0.368** | 0.749 | 0.564 |

** Statistically significant difference at the 99 percent confidence level.

TABLE XXVI

TEST MATERIAL: Aroclor 1260

Two-Year Chronic Oral Toxicity Study - Albino Rats

Organ Weight and Ratio Data

Six-Month Sacrifice

Summary of Mean Values

Organ: Brain

| Dietary Level of Aroclor 1260 (ppm) | Organ Weight (g) | | Organ/Body Weight Ratio (g/100 g) | | Final Body Weights of Sacrificed Rats (g) | |
|---|---|---|---|---|---|---|
| | Males | Females | Males | Females | Males | Females |
| Control | 1.96 | 1.95 | 0.320 | 0.560 | 613 | 348 |
| 1 | 2.10 | 1.92 | 0.412* | 0.474* | 509 | 405 |
| 10 | 2.01 | 1.73 | 0.349 | 0.504 | 575 | 343 |
| 100 | 1.99 | 1.88 | 0.382 | 0.650* | 522 | 289 |

* Statistically significant difference at the 95 percent confidence level.

c. Histopathologic Findings

Histopathologic examination of tissues and organs taken from all animals from both the control and 100 ppm groups was conducted.

Tables XXVII and XXVIII list all histopathologic changes noted.

All of the lesions noted in the microscopic examination of tissues were those of spontaneous disease and are not unusual for the albino rat. The most frequent findings were lesions in the trachea and lungs, indicating chronic murine pneumonia. These occurred in the control as well as the rats fed Aroclor 1260.

TABLE XXVII

TEST MATERIAL: Aroclor 1260

Two-Year Chronic Oral Toxicity Study - Albino Rats

Histopathologic Changes

Six-Month Sacrifice

Group: Control

| Number of Animals | Organ Examined | Findings | Incidence | Average Grade |
|---|---|---|---|---|
| 5 Males | Trachea | Tracheitis | 2 | 1.5 |
| | | Focal tracheitis | 2 | 1.0 |
| | Lung | Focal pneumonitis | 1 | 0.5 |
| | | Hyperemia | 2 | 1.0 |
| | Liver | Focal pericholangitis | 1 | 1.0 |
| | Skeletal muscle | Focal inflammation | 1 | 1.0 |
| 5 Females | Trachea | Focal tracheitis | 1 | 1.0 |
| | Lung | Hyperemia | 2 | 2.5 |
| | Liver | Hyperemia | 1 | 1.0 |
| | | Pericholangitis | 1 | 0.5 |
| | Kidney | Focal inflammation | 1 | 1.0 |

All other tissues and organs were normal histologically.

Grading System

0.5 = minimal
1.0 = slight
2.0 = mild
3.0 = moderate
4.0 = severe
5.0 = extreme

TABLE XXVIII

TEST MATERIAL: Aroclor 1260

Two-Year Chronic Oral Toxicity Study - Albino Rats

Histopathologic Changes

Six-Month Sacrifice

Group: 100 ppm

| Number of Animals | Organ Examined | Findings | Incidence | Average Grade |
|---|---|---|---|---|
| 5 Males | Trachea | Tracheitis | 2 | 2.0 |
| | | Focal tracheitis | 1 | 0.5 |
| | Lung | Focal pneumonitis | 4 | 1.0 |
| | | Hyperemia | 4 | 1.5 |
| | Liver | Hyperemia | 1 | 1.0 |
| | | Focal pericholangitis | 1 | 0.5 |
| 5 Females | Trachea | Focal tracheitis | 2 | 0.5 |
| | Lung | Focal pneumonitis | 2 | 1.0 |
| | | Hyperemia | 1 | 3.0 |
| | Liver | Hyperemia | 2 | 1.5 |
| | | Focal pericholangitis | 1 | 0.5 |
| | Kidney | Focal nephritis | 3 | 0.5 |

All other tissues and organs were normal histologically.

Grading System

0.5 = minimal
1.0 = slight
2.0 = mild
3.0 = moderate
4.0 = severe
5.0 = extreme

3. Twelve-Month Sacrifice

a. Gross Pathologic Findings

Gross pathologic findings among test animals were not significantly different from those noted in control animals.

b. Organ Weight and Ratio Data

The mean organ weight and ratio data collected at the twelve month sacrifice are presented in Tables XXIX through XXXIV.

Statistically significant differences are designated by asterisks following the test group value.

Organ weights, organ to body weight and organ to brain weight ratios disclosed several randomly occurring intergroup differences. The lack of any consistent dose related response and the absence of any deleterious histopathologic change confirm that these differences were not related to the ingestion of Aroclor 1260.

TABLE XXIX

TEST MATERIAL: Aroclor 1260

Two-Year Chronic Oral Toxicity Study - Albino Rats

Twelve-Month Sacrifice

Organ Weight and Ratio Data

Summary of Mean Values

Organ: Liver

| Dietary Level (ppm) | Organ Weight (g) | | Organ/Body Weight Ratio (g/100 g) | | Organ/Brain Weight Ratio (g/g) | |
|---|---|---|---|---|---|---|
| | Males | Females | Males | Females | Males | Females |
| Control | 22.1 | 15.5 | 3.79 | 3.52 | 10.8 | 7.72 |
| 1 | 17.1* | 10.7* | 2.79** | 3.01* | 9.23 | 5.76 |
| 10 | 20.0 | 13.6 | 3.30 | 3.54 | 10.8 | 7.28 |
| 100 | 28.8* | 14.1 | 4.26 | 3.79 | 13.4* | 8.52 |

* Statistically significant difference at the 95 percent confidence level.
** Statistically significant difference at the 99 percent confidence level.

TABLE XXX

TEST MATERIAL: Aroclor 1260

Two-Year Chronic Oral Toxicity Study - Albino Rats

Twelve-Month Sacrifice

Organ Weight and Ratio Data

Summary of Mean Values

Organ: Kidneys

| Dietary Level (ppm) | Organ Weight (g) | | Organ/Body Weight Ratio (g/100 g) | | Organ/Brain Weight Ratio (g/g) | |
|---|---|---|---|---|---|---|
| | Males | Females | Males | Females | Males | Females |
| Control | 4.04 | 3.19 | 0.692 | 0.738 | 1.97 | 1.58 |
| 1 | 3.84 | 2.58** | 0.625 | 0.724 | 2.06 | 1.39 |
| 10 | 4.00 | 2.78* | 0.660 | 0.736 | 2.16 | 1.48 |
| 100 | 5.11* | 2.81 | 0.758 | 0.755 | 2.38* | 1.69 |

* Statistically significant difference at the 95 percent confidence level.
** Statistically significant difference at the 99 percent confidence level.

TABLE XXXI

TEST MATERIAL: Aroclor 1260

Two-Year Chronic Oral Toxicity Study - Albino Rats

Twelve-Month Sacrifice

Organ Weight and Ratio Data

Summary of Mean Values

Organ: Spleen

| Dietary Level (ppm) | Organ Weight (g) | | Organ/Body Weight Ratio (g/100 g) | | Organ/Brain Weight Ratio (g/g) | |
|---|---|---|---|---|---|---|
| | Males | Females | Males | Females | Males | Females |
| Control | 0.778 | 0.796 | 0.133 | 0.183 | 0.380 | 0.396 |
| 1 | 0.756 | 0.460* | 0.122 | 0.129* | 0.408 | 0.247* |
| 10 | 0.836 | 0.578 | 0.139 | 0.150 | 0.457 | 0.308 |
| 100 | 0.785 | 0.474* | 0.116 | 0.127* | 0.365 | 0.284 |

* Statistically significant difference at the 95 percent confidence level.

TABLE XXXII

TEST MATERIAL: Aroclor 1260

Two-Year Chronic Oral Toxicity Study - Albino Rats

Twelve-Month Sacrifice

Organ Weight and Ratio Data

Summary of Mean Values

Organ: Gonads

| Dietary Level (ppm) | Organ Weight (g) | | Organ/Body Weight Ratio (g/100 g) | | Organ/Brain Weight Ratio (g/g) | |
|---|---|---|---|---|---|---|
| | Males | Females | Males | Females | Males | Females |
| Control | 3.46 | 0.116 | 0.596 | 0.0275 | 1.70 | 0.0574 |
| 1 | 3.38 | 0.103 | 0.551 | 0.0287 | 1.82 | 0.0552 |
| 10 | 3.51 | 0.112 | 0.578 | 0.0294 | 1.89 | 0.0584 |
| 100 | 4.03** | 0.088* | 0.598 | 0.0238 | 1.88 | 0.0527 |

* Statistically significant difference at the 95 percent confidence level.
** Statistically significant difference at the 99 percent confidence level.

TABLE XXXIII

TEST MATERIAL: Aroclor 1260

Two-Year Chronic Oral Toxicity Study - Albino Rats

Twelve-Month Sacrifice

Organ Weight and Ratio Data

Summary of Mean Values

Organ: Heart

| Dietary Level (ppm) | Organ Weight (g) | | Organ/Body Weight Ratio (g/100 g) | | Organ/Brain Weight Ratio (g/g) | |
|---|---|---|---|---|---|---|
| | Males | Females | Males | Females | Males | Females |
| Control | 1.71 | 1.23 | 0.293 | 0.287 | 0.835 | 0.611 |
| 1 | 1.68 | 1.07 | 0.273 | 0.301 | 0.902 | 0.575 |
| 10 | 1.59 | 1.16 | 0.261 | 0.307 | 0.845 | 0.620 |
| 100 | 1.86 | 1.27 | 0.275 | 0.340 | 0.868 | 0.760 |

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TABLE XXXIV

TEST MATERIAL: Aroclor 1260

Two-Year Chronic Oral Toxicity Study - Albino Rats

Organ Weight and Ratio Data

Twelve-Month Sacrifice

Summary of Mean Values

Organ: Brain

| Dietary Level (ppm) | Organ Weight (g) | | Organ/Body Weight Ratio (g/100 g) | | Final Body Weight of Sacrificed Rats (grams) | |
|---|---|---|---|---|---|---|
| | Males | Females | Males | Females | Males | Females |
| Control | 2.04 | 2.02 | 0.349 | 0.460 | 584 | 439 |
| 1 | 1.87 | 1.86 | 0.304 | 0.522 | 616 | 356 |
| 10 | 1.88 | 1.90 | 0.310 | 0.493 | 607 | 385 |
| 100 | 2.15 | 1.67** | 0.318 | 0.449 | 675 | 372 |

** Statistically significant difference at the 99 percent confidence level.

c. Histopathologic Findings

Histopathologic examination of tissues taken from each rat sacrificed from the control and 100 ppm groups after twelve months of testing was conducted.

Tables XXXV and XXXVI list all hisopathologic changes noted.

All of the lesions noted in the microscopic examination of tissues were those of spontaneous disease and are not unusual for the albino rat. The most frequent findings were lesions in the trachea and lungs, indicating chronic murine pneumonia. These occurred in the control as well as the rats fed Aroclor 1260.

# TABLE XXXV

## TEST MATERIAL: Aroclor 1260

## Two-Year Chronic Oral Toxicity Study - Albino Rats

## Histopathologic Changes

## Twelve-Month Sacrifice

## Group: Control

| Number of Animals | Organ Examined | Findings | Incidence | Average Grade |
|---|---|---|---|---|
| 5 Males | Trachea | Tracheitis | 1 | 1.0 |
| | Lung | Chronic respiratory disease | 2 | 1.0 |
| | Urinary Bladder | Calculus | 1 | 1.0 |
| 5 Females | Trachea | Tracheitis | 1 | 1.0 |
| | Lung | Chronic respiratory disease | 1 | 1.0 |
| | Colon | Parasites | 1 | 1.0 |

All other tissues and organs were normal histologically.

Grading System

0.5 = minimal
1.0 = slight
2.0 = mild
3.0 = moderate
4.0 = severe
5.0 = extreme

TABLE XXXVI

TEST MATERIAL: Aroclor 1260

Two-Year Chronic Oral Toxicity Study - Albino Rats

Histopathologic Changes

Twelve-Month Sacrifice

Group: 100 ppm

| Number of Animals | Organ Examined | Findings | Incidence | Average Grade |
|---|---|---|---|---|
| 5 Males | Trachea | Tracheitis | 1 | 1.0 |
| | Lung | Chronic respiratory disease | 3 | 1.0 |
| | Kidney | Focal lymphoid infiltration | 1 | 1.0 |
| | Liver | Vacuolar change of random cells | 1 | 1.0 |
| 5 Females | Trachea | Tracheitis | 1 | 1.0 |
| | Lung | Chronic respiratory disease | 1 | 1.0 |

All other tissues and organs were normal histologically.

Grading System

0.5 = minimal
1.0 = slight
2.0 = mild
3.0 = moderate
4.0 = severe
5.0 = extreme

4. Final Sacrifice

a. Gross Pathologic Findings

Gross pathologic findings among test animals were not significantly different from those noted in control animals.

b. Organ Weight and Ratio Data

The mean organ weight and ratio data collected at the final sacrifice are presented in Tables XXXVII through XLII. Statistically significant differences are designated by asterisks following the test group value.

Absolute liver weights and liver to body weight or liver to brain weight ratios were significantly elevated in those rats fed 100 ppm Aroclor 1260. Other organ weights, organ to body weight and organ to brain weight ratios disclosed several randomly occurring intergroup differences. The lack of any consistent dose related response and the absence of any deleterious histopathologic change confirm that these differences were not related to the ingestion of Aroclor 1260

TABLE XXXVII

TEST MATERIAL: Aroclor 1260

Two-Year Chronic Oral Toxicity Study - Albino Rats

Organ Weight and Ratio Data

Summary of Mean Values

Final Sacrifice

Organ: Liver

| Dietary Level (ppm) | Organ Weight (g) | | Organ/Body Weight Ratio (g/100 g) | | Organ/Brain Weight Ratio (g/g) | |
|---|---|---|---|---|---|---|
| | Males | Females | Males | Females | Males | Females |
| Control | 22.0 | 16.3 | 3.47 | 3.05 | 10.10 | 8.42 |
| 1 | 22.0 | 16.6 | 3.40 | 3.35 | 9.93 | 8.62 |
| 10 | 24.1 | 18.1 | 2.87* | 3.19 | 11.20 | 9.26 |
| 100 | 29.9** | 22.7** | 4.67** | 4.42** | 13.40* | 11.60* |

* Statistically significant difference at the 95 percent confidence level.
** Statistically significant difference at the 99 percent confidence level.

TABLE XXXIX

TEST MATERIAL: Aroclor 1260

Two-Year Chronic Oral Toxicity Study - Albino Rats

Organ Weight and Ratio Data

Summary of Mean Values

Final Sacrifice

Organ: Spleen

| Dietary Level (ppm) | Organ Weight (g) | | Organ/Body Weight Ratio (g/100 g) | | Organ/Brain Weight Ratio (g/g) | |
|---|---|---|---|---|---|---|
| | Males | Females | Males | Females | Males | Females |
| Control | 1.15 | 0.96 | 0.182 | 0.181 | 0.520 | 0.492 |
| 1 | 1.17 | 0.72 | 0.178 | 0.148 | 0.529 | 0.370 |
| 10 | 1.09 | 0.83 | 0.131 | 0.142 | 0.509 | 0.428 |
| 100 | 0.89 | 0.75 | 0.138 | 0.146 | 0.400 | 0.379 |

TABLE XL

TEST MATERIAL: Aroclor 1260

Two-Year Chronic Oral Toxicity Study - Albino Rats

Organ Weight and Ratio Data

Summary of Mean Values

Final Sacrifice

Organ: Gonads

| Dietary Level (ppm) | Organ Weight (g) | | Organ/Body Weight Ratio (g/100 g) | | Organ/Brain Weight Ratio (g/g) | |
|---|---|---|---|---|---|---|
| | Males | Females | Males | Females | Males | Females |
| Control | 2.52 | 0.123 | 0.397 | 0.0222 | 1.15 | 0.0628 |
| 1 | 3.56* | 0.140 | 0.550* | 0.0317 | 1.61* | 0.0718 |
| 10 | 3.36* | 0.177 | 0.400 | 0.0338 | 1.56* | 0.0900 |
| 100 | 2.97 | 0.113 | 0.464 | 0.0229 | 1.33 | 0.0584 |

* Statistically significant difference at the 95 percent confidence level.

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TABLE XLI

TEST MATERIAL: Aroclor 1260

Two-Year Chronic Oral Toxicity Study - Albino Rats

Organ Weight and Ratio Data

Summary of Mean Values

Final Sacrifice

Organ: Heart

| Dietary Level (ppm) | Organ Weight (g) | | Organ/Body Weight Ratio (g/100 g) | | Organ/Brain Weight Ratio (g/g) | |
|---|---|---|---|---|---|---|
| | Males | Females | Males | Females | Males | Females |
| Control | 1.87 | 1.54 | 0.294 | 0.288 | 0.860 | 0.795 |
| 1 | 1.98 | 1.45 | 0.304 | 0.294 | 0.893 | 0.747 |
| 10 | 1.90 | 1.58 | 0.225** | 0.279 | 0.884 | 0.809 |
| 100 | 2.00 | 1.49 | 0.313 | 0.288 | 0.904 | 0.764 |

** Statistically significant difference at the 99 percent confidence level.

NEV 010055

TABLE XLII

TEST MATERIAL: Aroclor 1260

Two-Year Chronic Oral Toxicity Study - Albino Rats

Organ Weight and Ratio Data

Summary of Mean Values

Final Sacrifice

Organ: Brain

| Dietary Level (ppm) | Organ Weight (g) | | Organ/Body Weight Ratio (g/100 g) | | Final Body Weight of Sacrificed Animals (grams) | |
|---|---|---|---|---|---|---|
| | Males | Females | Males | Female | Males | Females |
| Control | 2.18 | 1.95 | 0.342 | 0.362 | 642 | 551 |
| 1 | 2.21 | 1.94 | 0.343 | 0.407 | 654 | 525 |
| 10 | 2.15 | 1.96 | 0.255** | 0.347 | 845** | 584 |
| 100 | 2.22 | 1.95 | 0.346 | 0.378 | 641 | 644 |

** Statistically significant difference at the 99 percent confidence level.

c. Histopathologic Findings

Histopathologic examination of tissues and organs taken from all animals sacrificed after 24 months on test was conducted.

Tables XLIII through XLVI list all histopathologic changes noted. Table XLVII contains the results of the histopathologic examination of the urinary bladders.

Significant liver injury was found in animals fed 100 ppm Aroclor 1260 (T-III). The compound associated lesions consisted of vacuolar change, focal hypertrophy and focal hyperplasia. In addition, there were lesions of inflammation, necrosis, fibrosis and minor degeneration in this group which, although seen in the controls and lower test groups, were more severe and frequent in the T-III group.

The vacuolar change was not seen in the control animals but was observed occasionally in livers from T-I (1 ppm) and T-II (10 ppm) animals. It was frequent in the T-III animals. This lesion is morphologically indicative of fatty degeneration. Formalin-frozen sections of livers from represenative animals which displayed vacuolar changes were stained with Oil Red 0 to reveal the presence of fat. The vacuolar lesion in the cytoplasm of these cells was positively identified as fat.

The hypertrophic change found in the liver was focal and often limited to the central lobular area where groups of cells were swollen to two or three times their normal size with clear pink homogenous cytoplasm. The hyperplasia was associated with the same cells and appeared to be an

extension of the hypertrophic lesion. The hyperplastic cells were also usually hypertrophic. The most severe examples of hyperplasia appeared as nodular growths with limited compression of the surrounding normal hepatic tissue.

The minor lesions of degeneration, hepatitis, ductule cell proliferation, necrosis and focal lymphoid infiltration seen in the control animals and the T-I and T-II groups are lesions of spontaneous disease and are not related to the Aroclor 1260. This level of liver disease is not unusual in old animals.

Hyperplasia of the urinary bladder was found in an animal from the control group. This finding was not present in any of the rats fed Aroclor 1260. All of the other lesions in other tissues found in these animals are related to spontaneous disease and they are not unusual for old rats.

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TABLE XLIII

TEST MATERIAL: Aroclor 1260

Two-Year Chronic Oral Toxicity Study - Albino Rats

Histopathologic Changes

Final Sacrifice

Group: Control

| Number of Animals | Organ Examined | Findings | Incidence | Average Grade |
|---|---|---|---|---|
| 11 Males | Heart | Chronic focal myocarditis | 3 | 1.0 |
| | Trachea | Tracheitis | 3 | 1.0 |
| | Lung | Chronic respiratory disease | 9 | 1.0 |
| | Liver | Focal degeneration | 1 | 1.0 |
| | | Ductile hyperplasia | 2 | 1.0 |
| | Stomach | Chronic gastritis | 1 | 1.0 |
| | Spleen | Hemosiderosis | 3 | 1.0 |
| | Kidney | Chronic nephritis | 8 | 1.0 |
| | Testes | Testicular degeneration | 2 | 1.0 |
| | | Periarteritis | 2 | 1.0 |
| | Prostate | Inflammation | 1 | 1.0 |
| | Adrenal Gland | Telangiectasis | 1 | 1.0 |
| | | Nodular hyperplasia | 1 | 1.0 |
| | | Focal fatty change | 1 | 1.0 |
| | Brain | Focal encephalitis | 1 | 1.0 |
| | Eye | Inflammation | 2 | 1.0 |

TABLE XLIII continued

TEST MATERIAL: Aroclor 1260

Two-Year Chronic Oral Toxicity Study - Albino Rats

Histopathologic Changes

Final Sacrifice

Group: Control

| Number of Animals | Organ Examined | Findings | Incidence | Average Grade |
|---|---|---|---|---|
| 14 Females | Heart | Chronic focal myocarditis | 7 | 2.0 |
| | Trachea | Tracheitis | 9 | 1.0 |
| | Lung | Chronic respiratory disease | 13 | 1.0 |
| | Liver | Focal lymphoid infiltration | 3 | 1.0 |
| | | Ductile hyperplasia | 2 | 1.0 |
| | | Focal hepatitis | 1 | 1.0 |
| | | Non-lipid vacuolar change | 2 | 0.5 |
| | Stomach | Chronic gastritis | 3 | 1.0 |
| | | Acanthosis - squamous epithelium | 5 | 1.0 |
| | Colon | Parasites | 1 | 1.0 |
| | Spleen | Hemosiderosis | 4 | 1.0 |
| | Uterus | Metritis | 3 | 2.0 |
| | Kidney | Chronic nephritis | 6 | 1.0 |
| | | Focal lymphoid infiltration | 1 | 1.0 |
| | Ovary | Cyst | 1 | 1.0 |
| | Adrenal Gland | Telangiectasis | 9 | 1.0 |
| | | Nodular hyperplasia | 4 | 1.0 |
| | Salivary Gland | Chronic fibrosis and atrophy | 1 | 1.0 |

All other tissues and organs were normal histologically.

Grading System

0.5 = minimal 2.0 = mild 4.0 = severe

TABLE XLIV

TEST MATERIAL: Aroclor 1260

Two-Year Chronic Oral Toxicity Study - Albino Rats

Histopathologic Changes

Final Sacrifice

Group: 1 ppm

| Number of Animals | Organ Examined | Findings | Incidence | Average Grade |
|---|---|---|---|---|
| 11 Males | Heart | Chronic focal myocarditis | 4 | 1.0 |
| | Trachea | Tracheitis | 7 | 1.0 |
| | Lung | Chronic respiratory disease | 10 | 2.0 |
| | Liver | Focal hepatitis | 1 | 1.0 |
| | | Focal scarring | 1 | 1.0 |
| | | Hemosiderosis | 1 | 1.0 |
| | Pancreas | Chronic focal pancreatitis | 2 | 1.0 |
| | Stomach | Acute focal gastritis | 1 | 3.0 |
| | Spleen | Hemosiderosis | 1 | 1.0 |
| | Lymph Node | Congestion | 1 | 1.0 |
| | Kidney | Chronic nephritis | 7 | 2.0 |
| | Testes | Testicular degeneration | 1 | 2.0 |
| | | Periarteritis | 2 | 1.0 |
| | Prostate | Inflammation | 4 | 2.0 |
| | Seminal Vesicle | Inflammation | 1 | 2.0 |

TABLE XLIV continued

TEST MATERIAL: Aroclor 1260

Two-Year Chronic Oral Toxicity Study - Albino Rats

Histopathologic Changes

Final Sacrifice

Group: 1 ppm

| Number of Animals | Organ Examined | Findings | Incidence | Average Grade |
|---|---|---|---|---|
| 11 Males | Adrenal Gland | Nodular hyperoplasia | 2 | 1.0 |
| | | Focal fatty change | 2 | 1.0 |
| 14 Females | Heart | Chronic focal myocarditis | 3 | 1.0 |
| | | Chronic focal myocarditis | 1 | 3.0 |
| | Trachea | Tracheitis | 9 | 1.0 |
| | Lung | Chronic respiratory disease | 13 | 1.0 |
| | | Focal fibrosis | 1 | 1.0 |
| | Liver | Focal lymphoid infiltration | 1 | 1.0 |
| | | Ductile hyperplasia | 1 | 0.5 |
| | | Congestion | 1 | 1.0 |
| | | Vacuolar change (fatty) | 1 | 2.0 |
| | Stomach | Chronic gastritis | 1 | 1.0 |
| | | Acanthosis | 2 | 1.0 |

TABLE XLIV continued

TEST MATERIAL: Aroclor 1260

Two-Year Chronic Oral Toxicity Study - Albino Rats

Histopathologic Changes

Final Sacrifice

Group: 1 ppm

| Number of Animals | Organ Examined | Findings | Incidence | Average Grade |
|---|---|---|---|---|
| 14 Females | Stomach | Ulceration and acanthosis | 1 | 1.0 |
| | Colon | Parasites | 1 | 1.0 |
| | Spleen | Hemosiderosis | 11 | 1.0 |
| | | Hyperplasia | 1 | 1.0 |
| | Lymph Node | Congestion | 1 | 1.0 |
| | Kidney | Chronic nephritis | 3 | 2.0 |
| | | Vacuolar change (fatty) | 1 | 1.0 |
| | Uterus | Metritis | 4 | 1.0 |
| | Adrenal Gland | Telangiectasis | 9 | 1.0 |
| | | Nodular hyperplasia | 3 | 1.0 |
| | | Congestion | 1 | 1.0 |

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All other tissues and organs were normal histologically.

Grading System

0.5 = minimal
1.0 = slight
2.0 = mild
3.0 = moderate
4.0 = severe
5.0 = extreme

TABLE XLV

TEST MATERIAL: Aroclor 1260

Two-Year Chronic Oral Toxicity Study - Albino Rats

Histopathologic Changes

Final Sacrifice

Group: 10 ppm

| Number of Animals | Organ Examined | Findings | Incidence | Average Grade |
|---|---|---|---|---|
| 7 Males | Heart | Chronic focal myocarditis | 3 | 1.0 |
| | Trachea | Tracheitis | 2 | 1.0 |
| | Lung | Chronic respiratory disease | 5 | 1.0 |
| | | Suppurative pneumonia | 1 | 3.0 |
| | Liver | Vacuolar change (fatty) | 2 | 1.0 |
| | Stomach | Chronic gastritis | 1 | 1.0 |
| | | Acanthosis | 2 | 1.0 |
| | Spleen | Hemosiderosis | 3 | 1.0 |
| | Kidney | Chronic nephritis | 6 | 1.0 |
| | Prostate | Inflammation | 2 | 1.0 |
| | Adrenal Gland | Nodular hyperplasia | 1 | 1.0 |
| 14 Females | Heart | Chronic focal myocarditis | 3 | 1.0 |
| | Trachea | Thracheitits | 8 | 1.0 |
| | Lung | Chronic respiratory disease | 14 | 1.0 |

TABLE XLV continued

TEST MATERIAL: Aroclor 1260

Two-Year Chronic Oral Toxicity Study - Albino Rats

Histopathologic Changes

Final Sacrifice

Group: 10 ppm

| Number of Animals | Organ Examined | Findings | Incidence | Average Grade |
|---|---|---|---|---|
| 14 Females | Liver | Vacuolar change (fatty) | 1 | 1.0 |
| | | Focal hyperplasia | 1 | 1.0 |
| | Pancreas | Chronic focal pancreatitis | 1 | 3.0 |
| | | Periarteritis | 1 | 3.0 |
| | Stomach | Acanthosis | 1 | 1.0 |
| | Spleen | Hemosiderosis | 12 | 1.0 |
| | Kidney | Chronic nephritis | 10 | 1.0 |
| | Uterus | Metritis | 7 | 2.0 |
| | Pituitary | Congestion | 1 | 1.0 |
| | Adrenal Gland | Telangiectasis | 7 | 1.0 |
| | | Nodular hyperplasia | 1 | 1.0 |
| | | Congestion | 1 | 1.0 |

All other tissues and organs were normal histologically.

Grading System

0.5 = minimal
1.0 = slight
2.0 = mild
3.0 = moderate
4.0 = severe
5.0 = extreme

TABLE XLVI

TEST MATERIAL: Aroclor 1260

Two-Year Chronic Oral Toxicity Study - Albino Rats

Histopathologic Changes

Final Sacrifice

Group: 100 ppm

| Number of Animals | Organ Examined | Findings | Incidence | Average Grade |
|---|---|---|---|---|
| 10 Males | Heart | Chronic focal myocarditis | 5 | 1.0 |
| | Trachea | Tracheitis | 8 | 1.0 |
| | Lung | Chronic respiratory disease | 9 | 2.0 |
| | | Congestion | 1 | 1.0 |
| | Liver | Vacuolar change (fatty) | 2 | 2.0 |
| | | Focal degeneration | 1 | 1.0 |
| | | Congestion | 3 | 2.0 |
| | | Centrolobular hypertrophy | 2 | 1.0 |
| | | Telangiectasis | 1 | 1.0 |
| | Pancreas | Chronic focal pancreatitis | 2 | 1.0 |
| | Stomach | Inflammation | 2 | 2.0 |
| | | Acanthosis | 2 | 1.0 |
| | Spleen | Hemosiderosis | 7 | 1.0 |
| | Kidney | Focal lymphoid infiltration | 1 | 1.0 |
| | | Calcified concretions in pelvis | 1 | 1.0 |
| | | | | 1.0 |

TABLE XLVI continued

TEST MATERIAL: Aroclor 1260

Two-Year Chronic Oral Toxicity Study - Albino Rats

Histopathologic Changes

Final Sacrifice

Group: 100 ppm

| Number of Animals | Organ Examined | Findings | Incidence | Average Grade |
|---|---|---|---|---|
| 10 Males | Kidney (continued) | Calcified concretions in pelvis | 1 | 1.0 |
| | Testes | Aspermia | 1 | 1.0 |
| | Prostate | Inflammation | 1 | 1.0 |
| | Thymus | Cyst | 1 | 1.0 |
| | Adrenal Gland | Telangiectasis | 3 | 1.0 |
| | | Nodular hyperplasia | 1 | 1.0 |
| 15 Females | Heart | Chronic focal myocarditis | 5 | 1.0 |
| | Trachea | Tracheitis | 8 | 1.0 |
| | Lung | Chronic respiratory disease | 11 | 1.0 |
| | | Congestion | 1 | 1.0 |
| | | Edema | 1 | 1.0 |
| | Liver | Vacuolar change (fatty) | 5 | 1.0 |

TABLE XLVI continued

TEST MATERIAL: Aroclor 1260

Two-Year Chronic Oral Toxicity Study - Albino Rats

Histopathologic Changes

Final Sacrifice

Group: 100 ppm

| Number of Animals | Organ Examined | Findings | Incidence | Average Grade |
|---|---|---|---|---|
| 15 Females | Liver (continued) | Focal hyperplasia | 4 | 1.0 |
| | | Chronic inflammation of liver capsule | 1 | 1.0 |
| | | Centrolobular hypertrophy | 5 | 1.0 |
| | | Necrosis | 2 | 2.0 |
| | | Ductile cell hyperplasia, portal fibrosis and widespread eosinophil inclusions in hepatocyte cytoplasm | 1 | 1.0 |
| | Pancreas | Chronic focal pancreatitis and periarteritis | 1 | 3.0 |
| | Spleen | Hemosiderosis | 9 | 2.0 |
| | Kidney | Chronic nephritis | 8 | 1.0 |
| | Uterus | Metritis | 7 | 1.0 |
| | Pituitary | Congestion | 1 | 1.0 |
| | Adrenal Gland | Telangiectasis | 6 | 1.0 |
| | | Nodular hyperplasia | 1 | 1.0 |
| | | Congestion | 1 | 1.0 |
| | Parathyroid | Hyperplasia | 1 | 1.0 |

All other tissues and organs were normal histologically.

Grading System

TABLE XLVII

TEST MATERIAL: Aroclor 1260

Two-Year Chronic Oral Toxicity Study - Albino Rats

Histopathologic Changes of the Urinary Bladder

Final Sacrifice

| Group | Sex | Number Examined | Findings* | Incidence | Average Grade |
|---|---|---|---|---|---|
| C | M | 11 | Proteinaceous plug (34) | 1 | - |
| | F | 14 | Focal epithelial hyperplasia (62) | 1 | 1.0 |
| T-I | M | 10 | Proteinaceous plug (86, 105, and 107) | 3 | - |
| (1 ppm) | F | 11 | None | - | - |
| T-II | M | 7 | Proteinaceous plug (187) | 1 | - |
| (10 ppm) | F | 14 | None | - | - |
| T-III | M | 10 | Proteinaceous plug (271) | 1 | - |
| (100 ppm) | F | 14 | None | - | - |

* Individual animal numbers are shown in parentheses.

Grading System

1.0 = minimal or trace
2.0 = mild
3.0 = moderate
4.0 = severe
5.0 = extreme

NEV 010069

G. Detailed Description of Tumor Findings

Tumor data for individual animals including location, weight and pathologic classification are presented in Tables XLVIII through LI.

None of these tumors could be related to the ingestion of Aroclor 1260 and are considered normal for a random population of rats this age.

TABLE XLVIII

TEST MATERIAL: Aroclor 1260

Two-Year Chronic Oral Toxicity Study - Albino Rats

Description of Tumor Findings

Group: Control

| Animal Number and Sex | Location | Weight (grams) | Size (cm) | Classification | Months on Test |
|---|---|---|---|---|---|
| 1-M | Abdomen | - | - | Mammary fibroadenoma | 24 |
| 2-M | Pancreas | 11.76 | - | Adenocarcinoma | 24 |
| 46-F | Abdomen | 64.98<br>35.09 | 8 x 1 x 2<br>5 x 2 x 2 | Mammary fibroadenoma<br>Mammary fibroadenoma | 24 |
| 47-F | Lymph node | - | - | Reticulum cell sarcoma | 24 |
| 48-F | Abdomen | - | 2 x 2 x 1 | Mammary fibroadenoma | 24 |
| 50-F | Abdomen | - | 2 x 2 x 3 | Mammary fibroadenoma | 7 |
| 52-F | Abdomen | - | - | Mammary fibroadenoma | 24 |
| 59-F | Abdomen<br>Adrenal gland | -<br>- | -<br>- | Mammary fibroadenoma<br>Pheochromocytoma | 24 |
| 64-F | Abdomen | 88.92 | 13 x 11 x 4 | Fibroadenoma | 24 |
| 66-F | Abdomen | 230.00. | 13 x 11 x 4 | Mammary adenoma | 24 |

NEV 010071

TABLE XLIX

TEST MATERIAL: Aroclor 1260

Two-Year Chronic Oral Toxicity Study - Albino Rats

Description of Tumor Findings

Group: 1 ppm

| Animal Number and Sex | Location | Weight (grams) | Size (cm) | Classification | Months on Test |
|---|---|---|---|---|---|
| 99-M | Pituitary | - | - | Chromophobe adenoma | 20 |
| 127-F | Pituitary | - | - | Chromophobe adenoma | 24 |
| | Abdomen | 25.00 | 6 x 3 x 1 | Mammary fibroadenoma | |
| 128-F | Pituitary | - | - | Chromophobe adenoma | 21 |
| | Abdomen | 60.80 | 5 x 6 x 2 | Mammary fibroadenoma | |
| 138-F | Abdomen | 20.07 | - | Mammary fibroadenoma | 24 |
| | Pituitary | - | - | Chromophobe adenoma | |
| 141-F | Ovary | - | - | Adenoma | 24 |
| 151-F | Abdomen | 14.89 | - | Mammary fibroadenoma | 24 |
| 152-F | Abdomen | 540.00 | 13 x 15 x 2 | Mammary fibroadenoma | 22 |

NEV 010072

TABLE L

TEST MATERIAL: Aroclor 1260

Two-Year Chronic Oral Toxicity Study - Albino Rats

Description of Tumor Findings

Group: 10 ppm

| Animal Number and Sex | Location | Weight (grams) | Size (cm) | Classification | Months on Test |
|---|---|---|---|---|---|
| 205-F | Abdomen | 190.00 | 9 x 5 x 4 | Mammary fibroadenoma | 24 |
| 208-F | Pituitary | - | - | Chromophobe adenoma | 24 |
| 213-F | Abdomen | - | - | Mammary fibroadenoma | 24 |
| 216-F | Right shoulder | 138.00 | - | Subcutaneous tumor | 23 |
| 220-F | Abdomen | 9.57 | - | Mammary fibroadenoma | 24 |
| | Pituitary | - | - | Chomophobe adenoma | |
| 221-F | Submaxillary region | 14.44 | 2 x 3 x 1 | Fibroadenoma | 24 |
| | Abdomen | 29.0 | 3 x 5 x 2 | Mammary fibroadenoma | |

NEV 010073

TABLE L continued

TEST MATERIAL: Aroclor 1260

Two-Year Chronic Oral Toxicity Study - Albino Rats

Description of Tumor Findings

Group: 10 ppm

| Animal Number and Sex | Location | Weight (grams) | Size (cm) | Classification | Months on Test |
|---|---|---|---|---|---|
| 22-F | Left femoral region | 56.00 | 6 x 8 x 2 | ✓ Subcutaneous tumor | 6 |
| 233-F | Pituitary | - | - | ✓ Chromophobe adenoma | 24 |
| 234-F | Abdomen | 60.82 | 6 x 4 x 3 | ✓ Mammary fibroadenoma | 24 |
| | Pancreas | - | - | ✓ Islet cell adenoma | |

TABLE LI

TEST MATERIAL: Aroclor 1260

Two-Year Chronic Oral Toxicity Study - Albino Rats

Description of Tumor Findings

Group: 100 ppm

| Animal Number and Sex | Location | Weight (grams) | Size (cm) | Classification | Months on Test |
|---|---|---|---|---|---|
| 244-M | Pancreas | - | - | Adenoma | 24 |
| 264-M | Pancreas | - | - | Islet cell adenoma | 21 |
| 281-F | Abdomen | - | - | Mammary fibroadenoma | 24 |
| 293-F | Liver | 35.04 | 6 x 4 x 2 | Lymphosarcoma | 23 |
| 294-F | Abdominal cavity | 50.75 | 6 x 4 x 3 | Fibroma | 24 |
| 301-F | Liver | - | - | Lymphosarcoma | 23 |

TABLE LI continued

TEST MATERIAL: Aroclor 1260

Two-Year Chronic Oral Toxicity Study - Albino Rats

Description of Tumor Findings

Group: 100 ppm

| Animal Number and Sex | Location | Weight (grams) | Size (cm) | Classification | Months on Test |
|---|---|---|---|---|---|
| 302-F | Lymph node | - | - | Lymphosarcoma | 24 |
| 308-F | Pituitary | - | - | Chromophobe adenoma | 24 |
| 311-F | Abdomen | 14.00<br>- | 4 x 3 x 2<br>1 x 2 x 1 | Mammary fibroadenoma<br>Mammary fibroadenoma | 24 |
| 312-M | Pituitary | - | - | Chromophobe adenoma | 24 |
| 314-F | Abdomen | 26.00 | 5 x 4 x 3 | Mammary fibroadenoma | 24 |

NEV 010076

E. P. Wheeler - St. Louis

October 21, 1968

Polychlorinated Biphenyls
in the Environment

W. R. Richard
WRICH

R. E. Kelly, M. D./ W. H. Hunt
Scott Tucker - Queen [illegible]
R. A. Keller - RKELL
C. Payton - CPAYT
H. Bergen - HBERG
W. K. Johnson - WJOHN

Attached is a Xerox copy of a technical paper which Scott Tucker and I picked up in Washington recently. This was provided us by Donald A. Spencer of the National Agricultural Chemicals Association. Mr. Spencer requested that the paper be held "confidential" until such time as it may be published. Spencer indicated that if this paper were distributed one of his principal "sources" would refuse to give him prepublication information in the future.

Risebrough's presentation (the attached) was made at a meeting of 20 to 30 toxicologists held at the University of Rochester in June. The meeting was billed as "The First Annual Conference on Toxicology" and was underwritten presumably by the AEC which has had contracts at Rochester for many years. Attendance was by invitation only.

The meeting dealt "exclusively" with pesticides with about one-third of the papers relating to mercury. As far as Spencer knows the papers, including the attached, will undoubtedly be printed as proceedings of the conference although I suspect individual authors were given permission to publish elsewhere.

In a few words, Risebrough has found PCBs along with chlorinated pesticides in a number of species of fish and birds along the California coast as well as in waters off Baja California and Central America. He further reports PCB in fish from the Channel Islands and Puget Sound. No PCB was detected in the liver of tuna taken in the Galapagos Archipelago. Scott Tucker is going to scrutinize the analytical aspects and particularly the validity of some of the assumptions made by the author.

Elmer P. Wheeler

cs

Attachment

MINUTES OF MEETING OF THE CORPORATE DEVELOPMENT COMMITTEE

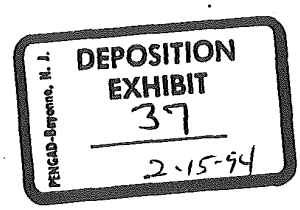
November 17, 1969

Present: Messrs.
E. J. Bock, Chairman
H. H. Bible
J. R. Eck
J. L. Gillis
E. J. Putzell
C. H. Sommer
J. N. Ehlers, Secretary

ORGANIC DIVISION, LAW AND MEDICAL DEPARTMENTS -- REPORT ON POLY-CHLORINATED BIPHENYLS

Present: Messrs. C. J. Smith, J. Mason, T. K. Smith, H. S. Bergen, J. E. Springgate, R. E. Kelly, E. P. Wheeler, Rodney Harris, Jr., D. W. Miller, W. C. Robinson

Monsanto's worldwide Aroclor business amounts to 104 M lbs./yr., 70M used in functional fluids and 34 M in plasticizers. This represents $22 M in sales. Products range from monochlorobiphenyl



to decachlorobiphenyl, terphenyls, and chlorinated terphenyls. Production locations are at Anniston, Alabama, Sauget, Illinois, Newport, U.K. and Yokkaichi, Japan.

Environmental Aspects - E. P. Wheeler

The 5 and 6 chlorinated biphenyls (Aroclor 1254 and 1260) have been found at limited locations in water, in birds and some forms of aquatic life. Recent indications are that such biphenyls may affect reproduction of fowl life and may be toxic to shrimp. These products are not toxic from the acute standpoint to man or fish but there is some evidence of ecological buildup in certain water deposits, in fish and ultimately in bird life.

- Aroclors 1248 and lower in number are believed to be biodegradable but this has not been conclusively established as yet.

Plan of Action - H. S. Bergen and J. E. Springgate

The availability of alternate products to satisfy customer requirements was reviewed. Main problems are that no replacement product is available for capacitors and replacement products for other uses pose a pollution problem.

In plasticizer uses, evidence is not available as to whether Aroclors escape from end products, either through leeching or by dispersal in burning.

The recommended plan of action is to establish a tailored program for each business group and each customer market situation to assure that the loss of PCB's in the environment, if any, is minimal.

NEV 176945

1. Appoint a Project Manager - responsible for the overall management of the Aroclor pollution problem. He would be assisted by a Task Force from members of each business group plus Medical, Law, Engineering and Manufacturing.

2. Notify all Aroclor customers of PCB problem.

3. Reduce and effectively control PCB effluents from Monsanto plants.

4. Educate customers on need to reduce and effectively control PCB effluents at their plants.

5. Develop and implement new packaging systems for Aroclor 1254/1260.

6. Introduce to market, replacement products for Aroclor 1254/1260.

7. Continue and expand biodegradation test program with Aroclor series, particularly 1242, 1248 and 1254.

8. Continue toxicological test program.

9. Accelerate present analytical test program.

10. Determine feasibility and cost of eliminating 5/6 $Cl_2$ in Aroclors 1242 and 1248.

11. Study incineration products.

12. Develop business plan to offer:

    - Monsanto Fluid Reclamation and Recovery with Enviro Chem. (Reclamation already under way at Findett.)

Cost of this program is estimated at approximately $400M SARE and $700M capital to change equipment.

Conclusions: In light of the recent and developing evidence of a possible threat to certain species of bird and aquatic life, we should plan to discontinue the manufacture of Aroclors 1254 and 1260. The Division is instructed to develop a program to discontinue these products and report this to the Committee.

The status of Aroclor 1242 should continue to be tested to determine whether it contributes to this problem. Other products which might be involved should also be examined.
(Excerpt to Messrs. H. L. Minckler, Rodney Harris, Jr., R. E. Kelly, T. K. Smith.)

NEV 176946

Springate — X

PCB PRESENTATION

Nov 17, 1969

TO

CORPORATE DEVELOPMENT COMMITTEE

I. INTRODUCTION:

We are here today to acquaint you with the PCB (Aroclor) pollution problem and to secure your guidance and approval on a recommended plan of action.

Certain PCB's have recently been identified by various scientists along with DDT in fish, birds, and other wildlife.

From the standpoint of reproduction, the PCB's are highly toxic to birds. In a few moments, Elmer Wheeler will describe the problem in detail.

Our objective is to describe for you the basic problems, the issues involved, review alternative courses of action, and suggest an action plan program for your approval.

This is a serious matter, not only from the pollution viewpoint, but also because of the $22 M worldwide customer business involved with resultant gross profits of $10 M and a net investment of approximately $9 M. In addition, there could be possible adverse legal and public relations problems leveled against Monsanto.

Our Agenda will be as follows:

NEV 176947

# PCB AGENDA REVIEW

I. INTRODUCTION

II. THE PROBLEM

- DEVELOPMENTS INCRIMINATING PCB's
- COMPLEXITY OF IDENTIFICATION
- NATURE OF
- SERIOUSNESS

III. LAW DEPARTMENT VIEWPOINT AND RECOMMENDATIONS

IV. EFFECT ON MONSANTO AND ALTERNATIVES

V. FUNCTIONAL FLUID BUSINESS GROUP DISCUSSION

- MARKETS, USES
- SOURCES OF POLLUTION
- CUSTOMER EFFECT

VI. PLASTICIZER BUSINESS GROUP DISCUSSION

- MARKETS, USES
- SOURCES OF POLLUTION

VII. RECOMMENDED ACTION PLAN

VIII. SUMMARY

NEV 176948

By way of introduction, the Organic Division and the Medical Department has been actively engaged for the last 18 months in developing facts and knowledge on this subject by personal visits to Universities and Industrial test laboratories, other worldwide producers, and other industrial collaborators, as well as keeping abreast of all literature and news sources on the subject as well as funding a toxicological and analytical test program in excess of $100 M. We established an Ad Hoc Committee of both Business Groups and Medical which recently issued a report - much of which will be discussed today. We have learned a lot, but there is much yet to learn as you will hear.

What are PCB's? They are polychlorinated biphenyls - better known to us as Aroclors. The next slide will quickly re-familiarize you with our Aroclor business.

## MONSANTO WORLDWIDE AROCLOR BUSINESS

| | | |
|---|---|---|
| POUNDS/YEAR | 104 M | (70 M in Functional Fluids<br>34 M in Plasticizers) |
| SALES/YEAR | $22 M | ($16 M in Functional Fluids<br>$ 6 M in Plasticizers) |
| GROSS PROFIT/YEAR | $10.0 M | ($7.5 M in Functional Fluids<br>$2.5 M in Plasticizers) |
| GROSS INVESTMENT | $13 M | ($8.8 M net investment) |
| ROI | 10.5% | |
| WORLDWIDE M/I | 62% | |
| MONSANTO PRODUCTION LOCATIONS: | USA (2 plants, Anniston, Alabama<br>Sauget, Illinois) | |
| | UK (Newport) | |
| | JAPAN (Yokkaichi) | |
| OTHER PRODUCERS: | Bayer, Prodelec, Caffaro, Flick, Kanegahuchi, and several Eastern European producers (all ex-USA) | |

NEV 176950

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## THE AROCLOR PRODUCT LINE

| CHEMICAL NAME | TRADE NAME | NATURE OF MATERIAL |
|---|---|---|
| MONOCHLOROBIPHENYL | AROCLOR 1221 | THIN LIQUID |
| DICHLOROBIPHENYL | AROCLOR 1232 | ↓ |
| TRICHLOROBIPHENYL | AROCLOR 1242 | OILY LIQUID |
| TETRACHLOROBIPHENYL | AROCLOR 1248 | ↓ |
| PENTACHLOROBIPHENYL | AROCLOR 1254 | HEAVY MOLASSES |
| HEXACHLOROBIPHENYL | AROCLOR 1260 | THICK TAR |
| HEPTACHLOROBIPHENYL | AROCLOR 1262 | ↓ |
| OCTACHLOROBIPHENYL | AROCLOR 1268 | ↓ |
| DECACHLOROBIPHENYL | AROCLOR 1270 | SOLID |
| TERPHENYLS | SANTOWAX | ↓ |
| CHLORINATED TERPHENYL | AROCLOR 5460 | SOLID |

NEV 176951

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There are theoretically 210 different isomers of chlorinated biphenyls.

Monsanto entered the Aroclor market in 1930 by acquiring Swan Chemical Company. The first load of Aroclor went out of Anniston, Alabama to General Electric in 1931. Since then, the market has grown to one of Monsanto's most profitable franchises. This franchise is now being threatened by [illegible] recently found pollution problems which Elmer Wheeler will now discuss.

II. The Problem (Wheeler) - see attached Appendix A

III. Law Department Viewpoint and Recommendations (French)

IV. Effect on Monsanto and Our Alternative Courses of Action

As discussed, Aroclors 1254 and 1260 -- the 5 and 6 Cl ringed biphenyls are the ones most seriously involved in the pollution problem. Both Plasticizers and Fluids Groups are involved as shown:

NEV 176952

## AROCLOR SALES

(M POUNDS)

| | FLUIDS | PLASTICIZERS | TOTAL |
|---|---|---|---|
| AROCLOR 1254 | 1.45 | 5.4 | 6.85 |
| AROCLOR 1260 & ABOVE | 3.7 | 1.7 | 5.4 |
| | 5.15 | 7.1 | 12.25 |

NEV 176953

We considered 4 alternative courses of action;

(Slide)

Alternative 1: Do nothing was considered unacceptable from a legal, moral, and customer, public relations & company policy viewpoint. This is also the quickest route to being forced out of business.

Alternative 2: Go out of total Aroclor business was considered unacceptable from a Divisional viewpoint, but from a Corporate viewpoint may be necessary. ~~Only you can make that decision.~~ All Aroclor products are not serious pollutants - many degrade; there is too much customer/market need and selfishly too much Monsanto profit to go out. To go out would require a write off of Aroclor net investment of $7 MM (10¢/share) or if biphenyl included $8.8 MM (12¢/share). In addition, inventory disposition, continuing cost of utilities, and back-up capital and serious manpower & resources reallocation at Anniston.

Alternative 3: Go out of Aroclor 1254 and 1260. This was seriously considered and may eventually occur by our actions and customer actions, nevertheless, we feel that segments of this business are defensible or are so "confined" in use that specific plans of action are called for this portion. Our reasons for eliminating this alternative will become clearer as we outline our action plans.

NEV 176954

## ALTERNATIVE COURSES OF ACTION

1. DO NOTHING - JUST REACT TO LEGISLATION AND EMOTION.
2. GO OUT OF TOTAL AROCLOR BUSINESS.
3. GO OUT OF AROCLOR 1254 AND 1260 PRODUCTION
4. DEVELOP SPECIFIC ACTION PLANS "TAILORED" TO EACH BUSINESS GROUP AND EACH CUSTOMER/MARKET SITUATION TO "CLEAN UP" THE MESS.



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Alternative 4: Develop specific action plans tailored to each Business Group and each customer/market situation, - was the alternative selected at this point of time and based on our knowledge from a Divisional viewpoint as making Monsanto act in the most positive, responsible way to society and our customers, as well as our interests.

However, because of the magnitude and seriousness of this problem and its total implications for Corporate Monsanto, the final decision on this matter must be made by the CDC.

V. Functional Fluids Business Group Discussion:

Aroclors are used widely in 3 of our 4 market areas in the Fluids Group:

## FLUIDS USE OF AROCLORS BY MARKET AREA

| AROCLOR PRODUCT | DOMESTIC MARKET AREA | | | TOTAL |
|---|---|---|---|---|
| | INDUSTRIAL | HEAT TRANSFER | ELECTRICAL | |
| 1242 | 4.1 | 1.1 | 36 | 41.2 |
| 1248 | 1.2 | 1.0 | - | 2.2 |
| 1254 | - | 0.1 | 0.8 | 0.9 |
| 1260 & Above | 0.6 | - | 3.5 | 4.1 |
| | 5.9 | 2.2 | 40.3 | 48.4 |

NEV 176957

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## SOURCES OF FLUIDS POLLUTION

| APPLICATION | INTENSITY OF POLLUTION | |
|---|---|---|
| INDUSTRIAL FLUIDS | GREATEST | (DIRECT) |
| DIELECTRICS | ↓ | (INDIRECT CONTAINED) |
| HEAT TRANSFER | ↓ | (INDIRECT CONTAINED) |
| PRODUCING PLANTS | LEAST | (DIRECT) |

NEV 176958

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# FLUIDS CUSTOMER ALTERNATIVES

| AREA OF APPLICATION | PRODUCT OF CHOICE | CUSTOMER OPTIONS |
|---|---|---|
| Industrial Fluids | Pydraul 312/F-9/ A-200/Phosphate Esters/ Water Glycol | Customer could get along without us, but Pydraul 312 favored. $H_2O$ Glycol has some pollution problem Phosphate ester route ok at present. |
| Transformer | Air/Oil/Aroclor/Gas | Could drop Aroclor at sacrifice of safety, cost or size of equipment or noise level. |
| Capacitors | Aroclors | No immediate replacement available. Longer term - oil at expense of size and cost of efficiency and redesign of equipment. |
| Heat Transfer | Therminol | No option for FR liquid market. Other system possibility. |
| | Oil/Dowtherm/T66<br>T55<br>T77<br>T88 | Liquid systems favored. T66 and T55 increasing rapidly in use. Oil also a pollution problem. |

NEV 176959

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Customer Choices & Alternatives & Penalties:

Summarizing, some of our customers have no immediate alternative, some could change only at sacrifices of safety, or cost or various technical factors. Only in the Industrial field could the customer make an immediate conversion.

PCB Threat to Functional Fluids Business and Profit:

## FLUIDS BUSINESS THREATENED

## (1970 BUDGET)

| PROBLEM | SALES | GROSS PROFIT |
|---|---|---|
| 1. Confined to A-1254/ 1260 only. | $ 3.0 M | $1.36 M |
| 2. Spreads to A-1242 and 1248 | | |
| First to: a) Industrial Fluids | $ 4.0 M | $1.6 M |
| Then to: b) Dielectric Fluids | $ 8.0 M | $3.8 M |
| Then to: c) Heat Transfer | $ 1.0 M | $ .6 M |
| | $16.0 M | $7.36 M |

— Turn over to Jim Springer

NEV 176961

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15 +1

VI

## PLASTICIZERS
## (WORLD-WIDE)

| | ALL AROCLORS | AROCLOR 1254/1260 TYPE |
|---|---|---|
| 1969 SALES, DOLLARS | $ 6.0 M | $1.7 M (28%) |
| POUNDS | 34.0 M | 9.5 M (28%) |
| 1969 GROSS PROFIT | $ 2.5 M | $0.8 M (32%) |

NEV 176962

COMMENTS: DISTINCTIONS FROM F.F.

1. Large number of direct U.S. customers - 570.
2. Customers are small: 23 direct customers - 47% A-1254/1260 sales.
3. 50% domestic A-1254/1260 sales through distributors - difficult to police.

NEV 176963

| MARKETS | 1968 SALES | MAJOR AROCLOR USED |
|---|---|---|
| Carbonless Carbon Paper | 8.8 M lb. | Aroclor 1242 |
| Hot Melt Adhesives | 5.7 M lb. | Aroclor 5460 |
| Swimming Pool Paints | 1.7 M lb. | Aroclor 1254 ✓<br>Aroclor 5460) |
| Protective Coatings | 5.3 M lb. | Aroclor 1254 ✓<br>Aroclor 5460) |
| Emulsion Adhesives | 1.5 M lb. | Aroclor 1254)<br>Aroclor 1260 ✓ |
| Sealants | 3.0 M lb. | Aroclor 1254)<br>Aroclor 1262) |
| Wax Modification | 2.0 M lb. | Aroclor 1254 ✓<br>Aroclor 5460) |
| Miscellaneous | 5.0 M lb. | Aroclor 1[illegible] ✓<br>Aroclor 12[illegible]) |

COMMENTS: 1. [illegible] major customer (85% of Aroclor 1242 sold).

2. 35% of domestic Aroclors sold through distributors.

NEV 176964

## POSSIBLE CONTAMINATION SOURCES

## (PLASTICIZERS)

| DEGREE OF CONTAMINATION | MARKET | APPLICATION | SOURCE | IS A-1254 /1260 USED? |
|---|---|---|---|---|
| Most | Coatings | Marine Paints) Water tank linings ) | Leaching | Yes |
| | Coatings | Swimming Pool Paints | Leaching | Yes |
| | Carbonless Carbon Paper | - | Vaporization | No |
| | Wax Modification | - | Vaporization | Yes |
| | Emulsion Adhesives | - | Contact with product via packaging. Incineration. | Yes |
| | Hot Melt Adhesives | - | Contact with product via packaging. Incineration. | No |
| Least | Sealants | Automotive Construction joint sealants | Long-term leaching | Yes |

NOTES: 1. Unlike fluids, Aroclor plasticizers are combined into plastics to produce the final product - therefore, far less mobile.

2. Problems such as wastes from our manufacturing plants, processors plants and and leasing of drums common to both groups.

3. Aroclor protective coatings are not considered a high [illegible]

4. The [illegible] plant processing or [illegible]

NEV 176965

## PLASTICIZER BUSINESS THREATENED

| PROBLEM | SALES RETAINED* | $ G.P. RETAINED (LOST) |
|---|---|---|
| 1. Confined to A-1254/1260 type only. | $4.3 M | $1.7 M (-$0.8 M) |
| 2. Spreads to all chlorinated biphenyls. | $2.0 | $0.6 M (-$1.9 M) |
| 3. Spreads to all PCB's and all chlorinated terphenyls | 0.0 | 0.0 (-$2.5 M) |

*Based on 1969 prospects.

NOTE: Plasticizers sell Aroclor 1262/4465 which are very close to A-1254/1260 and these have been included in A-1254/1260.

NEV 176966

## RECOMMENDED ACTION PLAN

THE JOINT ACTION PLAN DEVELOPED BY THE FUNCTIONAL FLUIDS AND PLASTICIZER BUSINESS GROUPS, AND THE MEDICAL AND LAW DEPARTMENTS IS AS FOLLOWS:

1. Appoint a Project Manager - responsible for the overall management of the Aroclor pollution problem. He would be assisted by a Task Force from members of each Business Group plus Medical, Law, Engineering and Manufacturing.

2. Notify all Aroclor customers of PCB problem and relabel containers - within 60 days.

3. Clean up Monsanto plants' effluents within 12 months.

4. Develop and implement new packaging systems for Aroclor 1254/1260 - within 6 months.

5. Educate customers on need for clean-up at their plants - within 4 months.

6. Introduct to market, replacement products for Aroclor 1254/1260 - beginning 1/1/70 (Fluids), 4/1/70 (Plasticizers).

## RECOMMENDED ACTION PLAN

7. Continue and expand biodegratation test program with Aroclor series, particularly 1242, 1248 and 1254.

8. Continue toxicological test program.

9. Accelerate present analytical test program.

10. Determine feasibility and cost of eliminating 5/6 $Cl_2$ in Aroclors 1242 and 1248. (3/70)

11. Study incineration products. (3/70)

12. Develop business plan to offer:

    Monsanto Fluid Reclamation and Recovery with Enviro Chem (4/70). (Reclamation already underway at Findett.)

NEV 176968

S

WHAT COULD WE EXPECT FROM THIS PROGRAM?

Through this action program, Monsanto would expect to:

1. Retain or convert a good portion of our business and profits:

| PROBLEMS | CONVERT OR RETAIN | $\overline{M}$ SALES OUT OF PRESENT | ODDS OF SUCCESS |
|---|---|---|---|
| a. Confined to A-1254/ 1260. | $20.3 $\overline{M}$ | $22 $\overline{M}$ | 70% |
| b. Spreads to A-1248 and 1242. | $10 $\overline{M}$ | $22 $\overline{M}$ | 60% |

2. Gain further valuable knowledge and time to:
   a. Learn more facts.
   b. Protect our position.
   c. Make further decisions regarding our program.
   d. Contribute to overall pollution knowledge.

3. Clean up the major contributing PCB pollution factors.

4. Minimize customer complaints and hardships.

NEV 176969

The Program Would:

1. Cost some money.

   Est. SARE - $400-500 M

   Est. Capital - $700 M

   $1.1 M - 1.2 M

2. Expose us to continued adverse publicity and possible law suits.

3. Cause some customer discontent - but much less than an abrupt termination of production.

NEV 176970

SUMMARY

In summary, the PCB pollution problem is a very serious one. It is a worldwide ecological problem. At present the most serious offenders are the 5 and 6 chlorine containing products (Aroclors 1254 and 1260). There are some indications other members are biodegradable to varying degrees. Currently, much scientific testing is underway and more information will be forthcoming. Monsanto must act in a positive cooperative fashion. A plan of action has been discussed which we feel indicates responsible action. Our stakes are large and because of the many possible effects on the Corporation -- your guidance and approval of the suggested action program is requested.

Thank you for your consideration. May we answer any questions?

A

GENTLEMEN:

MY PARTICIPATION IN THIS PRESENTATION WILL BE A BRIEF REVIEW OF THE DEVELOPMENTS WHICH INCRIMINATE THE POLY-CHLORINATED BIPHENYLS INCLUDING OUR AROCLORS AS NEARLY WORLD-WIDE ENVIRONMENTAL CONTAMINANTS, EXAMPLES ARE THE ALMOST COMPLETE DISAPPEARANCE OF THE PEREGRINE FALCON ALONG THE NORTHEAST COAST OF THE UNITED STATES; DEPLETION IN POPULATION OF THIS SPECIES IN CALIFORNIA; AND THE REPORTED EFFECT ON THE BROWN PELICAN IN CALIFORNIA.

3) PCB'S ARE PARTICULARLY TOXIC TO SHRIMP.

AT PENSACOLA, THERE WAS A LOSS OF 1 TO 3 GALLONS PER DAY OF AROCLOR 1254 INTO THE ESCAMBIA RIVER FROM OUR NYLON PLANT. THE RESULTANT CONCENTRATIONS OF PCB ONE-QUARTER MILE BELOW THE PLANT WERE ABOVE THE LEVELS NEEDED TO KILL JUVENILE SHRIMP IN LABORATORY TESTS.

4) THERE ARE STILL MANY UNKNOWNS BUT SPECULATION AS TO THE ROLE OF PCB'S IS RAMPANT.

ALTHOUGH SOME OF THE REPORTS OF ANALYTICAL IDENTIFICATION may not be valid ARE NOT CERTAIN, THE MEDICAL DEPARTMENT--AFTER 18 MONTHS OF INVESTIGATION IN CONJUNCTION WITH THE ORGANIC DIVISION--IS CERTAIN THAT THE PCB'S CAN BE RESPONSIBLE FOR ECOLOGICAL DAMAGE. THERE IS NO SCIENTIFIC CONSENSUS AS TO THE DEGREE OF PCB INVOLVEMENT BUT THESE COMPOUNDS CANNOT ESCAPE A SIGNIFICANT SHARE OF THE BLAME FOR DAMAGE.

NEV 176972

C

5) SPECULATION IS LEADING TO CONCERN WHICH MAY GROW TO ALARM.

THE MEDICAL DEPARTMENT BELIEVES THAT THE MYRIAD OF INVESTIGATORS PRESENTLY WORKING ON PCB'S IN GOVERNMENT AND UNIVERSITY LABORATORIES IS CERTAIN TO PROMOTE A MORE PROMINENT ROLE FOR PCB DAMAGE, LEADING AT BEST TO EXTREMELY DAMAGING PUBLICITY AND, AT WORST, LEADING TO COMPLETE BANNING OF THESE PRODUCTS.

NEV 176973

(1)

CERTAINLY ALL OF YOU ARE AWARE OF THE PRESENT CONCERN ABOUT THE RESIDUES OF DDT, ITS METABOLITES AND OTHER CHLORINATED HYDROCARBON PESTICIDES THROUGHOUT THE WORLD. SINCE WORLD WAR II AND IN THE LAST DECADE IN PARTICULAR, THERE HAS BEEN A SAMPLING NETWORK WHICH HAS IDENTIFIED DDT AND ITS METABOLITES IN PRACTICALLY EVERY LIVING ORGANISM AND IN THE AIR, WATER AND SOIL ACROSS THE FACE OF THE GLOBE. IT WOULD NOT BE UNEXPECTED TO FIND THAT EACH OF US IN THIS ROOM HAS 5 TO 10 ppm OF DDT IN OUR FATTY TISSUES.

AS ANALYTICAL TECHNIQUES HAVE IMPROVED AND INCLUDED VERY SENSITIVE GAS CHROMATOGRAPHY METHODS, INTERFERING SUBSTANCES HAVE APPEARED WHICH RAISE QUESTIONS AS TO THE VALIDITY OF PRESENT AND EARLIER DETERMINATIONS OF DDT MATERIALS.

THIS DIAGRAM INDICATES THE GAS CHROMATOGRAMS OF SEVERAL OF OUR AROCLORS. AT THE BOTTOM THE PEAKS FOR VARIOUS HYDROCARBON PESTICIDES ARE SHOWN. IT IS OBVIOUS THAT SOME OF THE PREDOMINANT PEAKS IN THE AROCLORS FALL IN THE SAME POSITION IN TERMS OF RETENTION TIME AS DDT

NEV 176974

AND ITS METABOLITES. PLEASE NOTICE THAT THIS IS PARTICULARLY TRUE FOR AROCLORS 1254 AND 1260 AND IT IS THESE TWO PARTICULAR POLYCHLORINATED BIPHENYLS, WHETHER MANUFACTURED BY MONSANTO OR OUR COMPETITORS, THAT ARE THE PRODUCTS WHICH ARE MOST CONSISTENTLY IDENTIFIED ( PENTA ~~TO OCTA~~ and hexa CHLORO). LET ME EMPHASIZE THIS POINT BECAUSE IT HAS A PARTICULAR BEARING ON THE FUTURE MANUFACTURE, SALES AND USE OF THE WHOLE PRODUCT LINE.

WHERE HAVE THE PCB'S BEEN FOUND?

THIS NEXT TRANSPARENCY INDICATES LOCATIONS WHERE THESE MATERIALS HAVE BEEN REPORTED. THE AMOUNTS FOUND VARY FROM PARTS PER TRILLION IN SOME WATER SAMPLES TO HUNDREDS OF PARTS PER MILLION IN FISH AND SOME FISH-EATING BIRDS.

LET ME DIGRESS A MOMENT TO EQUATE THESE UNITS OF PPM, PPB AND PPT TO SOMETHING THAT WE ALL RECOGNIZE (SHOW TRANSPARENCY ON COMPARISON OF PPM TO FIFTHS).

RETURNING TO THE REPORTED FINDINGS OF THE PCB'S, THE VALUES THAT HAVE BEEN REPORTED IN SWEDEN VARY FROM 10 PPB IN FISH, BIRDS AND EGGS TO 20,000 ppm IN ONE OR TWO SINGLE SPECIMENS. IN THE LATTER CASE, THESE HAVE BEEN DEAD BIRDS AND THERE IS SOME QUESTION AS TO THE VALIDITY OF THE RESULTS. IN REGARD TO CHILDREN"S HAIR, DR. JENSEN HAS POINTED OUT THAT HIS FIVE MONTH OLD CHILD HAD MORE PCB'S IN THE HAIR THAN HIS THREE AND SIX YEAR OLD CHILDREN AND HE POSTULATED THAT THIS WAS DUE TO THE PCB BEING IN MOTHER'S MILK. THE SAME LEVELS OF PCB'S HAVE BEEN FOUND IN FISH, BIRDS, AND EGGS IN

GREAT BRITAIN AND THE NETHERLANDS. IN THE UNITED STATES AS WE HAVE INDICATED, THE PCB'S HAVE BEEN FOUND IN IN-LAND WATERS. THIS IS QUITE DIFFERENT UP TO THIS POINT AS REGARDS THEIR DISTRIBUTION IN GREAT BRITAIN, IN THE NETHERLANDS AND IN THE SCANDANAVIAN COUNTRIES. PERHAPS I SHOULD REMIND YOU THAT MONSANTO IS THE SOLE PRODUCER AND SUPPLIER OF POLYCHLORINATED BIPHENYLS IN THE UNITED STATES AND GREAT BRITAIN. THIS IS IMPORTANT SINCE ANY PUBLIC OR GOVERNMENTAL AGENCY ACTIVITY WHICH MAY LEAD TO THE RESTRICTION OF USE OF POLYCHLORINATED BIPHENYLS MAKES MONSANTO PARTICULARLY VULNERABLE FROM A PUBLIC RELATIONS STANDPOINT IN THE UNITED STATES, GREAT BRITAIN AND PROBABLY CANADA.

IN TERMS OF TOXIC OR HARMFUL EFFECTS FROM THE PRESENCE OF PCB'S, WE CAN MAKES THESE STATEMENTS SUPPORTED BY A GROWING AMOUNT OF TOXICOLOGICAL RESEARCH DATA:

1. THE AROCLOR'S ARE NOT HIGHLY TOXIC FROM AN ACUTE STANDPOINT TO MAN, ANIMALS, BIRDS OR FISH. FOR EXAMPLE, THEY ARE NOT NEARLY AS TOXIC AS DDT AND CERTAINLY MUCH LESS TOXIC THAN DIELDRIN AND ALDRIN.
2. FROM A CHRONIC TOXICITY STANDPOINT, THE PCB'S MAY BE CONSIDERED "MODERATELY TOXIC" TO MAN, ANIMALS AND FISH. FROM THE STANDPOINT OF REPRODUCTION, THE PCB'S ARE HIGHLY TOXIC TO BIRDS. THIS CONCLUSION IS BASED ON ON-GOING RESEARCH AT OUR CONSULTING LABORATORY IN CHICAGO WHERE WHITE LEGHORN CHICKENS--A RELATIVELY RESISTANT MEMBER OF THE BIRD SPECIES--ARE LAYING EGGS AFTER BEING

FED A DIET OF 10 PPM WHICH FAIL TO HATCH. AT 100 PPM THE EGGS HAVE GREATLY REDUCED EGG SHELL THICKNESS AS WELL.

IN THE AQUATIC ENVIRONMENT, SHRIMP APPEAR TO BE PARTICULARLY SENSITIVE TO THE PCB'S. IN A TEST CONDUCTED AT THE BUREAU OF COMMERCIAL FISHERIES LABORATORY, GULFSTREAM, FLORIDA, 5 PPB CAUSED THE DEATH OF 18 OUT OF 25 JUVENILE SHRIMP IN 18 DAYS.

ALTHOUGH AT THIS POINT THE PCB'S ARE NOT BEING TOUTED AS SERIOUS TOXICANTS, THERE HAVE BEEN COMMENTS IN EACH OF THE PUBLICATIONS WHICH HAVE APPEARED WHICH HAVE IMPLIED--IF NOT STATED DIRECTLY--THAT THESE MATERIALS ARE "HIGHLY TOXIC".

THE FUTURE OF THESE MATERIALS IS THREATENED MORE BY THE POTENTIAL EFFECT ON SOME FORMS OF WILDLIFE RATHER THAN POTENTIAL TOXIC EFFECTS AS WE USUALLY THINK OF THEM IN RELATION TO HUMAN OR ANIMAL FOODS. IN THE ENVIRONMENT WE ARE FACED WITH THIS CYCLE--FISH IN WATER CONTAINING PPB OF PCB CONCENTRATE THE MATERIAL TO PPM. THE SWEDES SAY THAT LARGER FISH EATING SUCH SMALLER ONES SHOW A 10 FOLD INCREASE IN THE AMOUNT OF PCB IN THEIR TISSUES. BIRDS THEN EATING THESE FISH SHOW 100 FOLD CONCENTRATION OF PCB FROM THAT IN THE FISH WHICH THEY HAVE EATEN. IT IS POSTULATED AS IN THE CASE OF DDT THAT THIS CONCENTRATION LEADS TO EGGS WITH LITTLE OR NOT SHELL THICKNESS AND WITH DECREASED OR NO REPRODUCTION.

HOW DO THE PCB'S GET INTO THE ENVIRONMENT?

OUR EFFORTS TO DATE HAVE NOT BEEN COMPLETELY SUCCESSFUL IN PIN-POINTING SOURCES OF THE PCB'S. AS MR. BERGEN AND MR. SPRINGATE WILL DISCUSS SHORTLY, WE DO HAVE SOME IDEAS CONCERNING SPECIFIC PRODUCT USES.

WE CAN, HOWEVER, MENTION THREE INSTANCES WHICH WILL GIVE SOME IMPRESSION AS TO SOURCES SUCH AS OUR MANUFACTURING PLANTS, A MONSANTO "CUSTOMER" PLANT, AND EARLY RESULTS OF WIDE-SPREAD SAMPLING IN LAKE MICHIGAN (SHOW TRANSPARENCY OF SEVERN RIVER, PENSACOLA AND LAKE MICHIGAN). THIS IS AN INDICATION OF THE PCB CONCENTRATION IN MUD NEAR THE OUTFALL OF OUR NEWPORT PLANT. YOU WILL NOTE THE SCALE IN MILES AT THE BOTTOM AND THE DISTRIBUTION OF PCB ALONG THE ESTUARY OF THE SEVERN RIVER.

SIMILAR FINDINGS COULD BE SHOWN FOR THE STREAMS BELOW OUR ANNISTON AND KRUMMRICH PLANTS.

AN INDICATION OF "CUSTOMER" USAGE AS A SOURCE IS INDICATED IN THIS SLIDE. THIS IS A MONSANTO CUSTOMER PLANT WHERE THE PENSACOLA INSTALLATION WAS USING FIRE RESISTANT AIR COMPRESSOR LUBRICANT CONTAINING AROCLOR 1254. IT WAS REPORTED THAT ONE-FOURTH MILE BELOW OUR PLANT OUTFALL, 40 PPB OF PCB WAS PRESENT. AT THE BRIDGE AT HIGHWAY 90, 0.5 PPB WAS REPORTED. THE STATE AND UNIVERSITY IN DOING FURTHER SAMPLING REACHED IN BEHIND THE SKIMMERS ON THE OUTFALL OF OUR PLANT AND FOUND 240 PPB.

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AS A FURTHER EXAMPLE OF THE POSSIBLE SOURCES FROM OUR CUSTOMER PLANTS, THIS TRANSPARENCY SHOWS EARLY REPORTS OF SAMPLES FROM LAKE MICHIGAN. WATER SAMPLES HAVE SHOWN PARTS PER TRILLION TO PARTS PER BILLION OF THE HIGHER CHLORINATED BIPHENYLS. WHEN WE BROUGHT THIS BACK TO OUR MARKETING GROUPS, THEY QUICKLY POINTED OUT THAT ONE COULD ALMOST PIN-POINT OUR CUSTOMER USAGE ALONG THE SHORES OF LAKE MICHIGAN.

THESE THREE INSTANCES REFLECT WHAT MIGHT BE TERMED DIRECT CONTAMINATION. AS YOU WILL HEAR, THE POSSIBLE SOURCES OF INDIRECT CONTAMINATION MIGHT BE RELATED TO EVERY SINGLE USE OF OUR PRODUCTS WHETHER THE USES BE IN ELECTRICAL APPLICATIONS, OTHER INDUSTRIAL FLUIDS, OR PLASTICIZER USAGE.

THE SCIENTISTS WHO HAVE PUBLISHED THEIR FINDING OF THE PCB'S IN NATURE HAVE IN EVERY INSTANCE MENTIONED THE POSSIBLE SOURCES AS THE LIQUID MATERIALS AS INDUSTRIAL POLLUTANTS AND THE PLASTICIZER USES WHERE IN THE MANUFACTURE OF A PLASTIC MATERIAL SOME VAPORS ARE LOST TO THE ATMOSPHERE AND ULTIMATELY END UP IN THE OCEAN AS CHEMICAL FALL-OUT. The PCB's ~~WE~~ HAVE THE HONOR IF IT BE SUCH OF HAVING THIS QUOTED IN THIS NEW BOOK ON CHEMICAL FALL-OUT. Scientists ~~THEY~~ HAVE POSTULATED ALSO THAT SINCE THE PCB'S ARE VIRTUALLY INDESTRUCTIBLE, EVERY POUND EVER MANUFACTURED ULTIMATELY HAS ESCAPED INTO THE ENVIRONMENT AND REMAINS THERE.

AS FAR AS FUTURE ACTION IS CONCERNED, THE MEDICAL DEPARTMENT PROPOSES TO CONTINUE A PROGRAM, AND I MIGHT ADD--AN EXPANDED

PROGRAM--WHICH WE HOPE WILL PERMIT THE CONTINUED USE OF AROCLORS 1254 AND 1260 IN THOSE APPLICATIONS WHERE ESCAPE INTO THE ENVIRONMENT CAN BE POLICED AND PREVENTED.

SECONDLY, WE HOPE TO DEVELOP DATA THAT WILL SHOW THAT THE LOWER CHLORINATED BIPHENYLS, THAT IS--AROCLOR 1242 AND THOSE LOWER IN CHLORINATION, ARE DEGRADED IN THE ENVIRONMENT AND THUS DO NOT PRESENT A THREAT TO WILDLIFE.

THERE IS SOME INDICATION FROM WORK UNDERWAY IN RUABON THAT AROCLOR 1242 WILL INDEED DEGRADE BIOLOGICALLY. THERE IS A PUBLISHED REPORT FROM THE UNIVERSITY OF UTRECHT WHERE 1242 FED TO QUAIL SHOWED VIRTUALLY COMPLETE DEGRADATION. AS I HAVE MENTIONED EARLIER, EXCEPT FOR ONE OR TWO INSTANCES AROCLOR 1242 HAS NOT BEEN IDENTIFIED ALONG WITH THE PESTICIDE RESIDUES. THIS IN ITSELF SUGGESTS THAT THIS MATERIAL IS DEGRADED.

WE WOULD HOPE THEN THAT OUR EFFORTS WILL PROTECT THE CONTINUED SALE AND USE OF AROCLORS 1242, WHICH AS MR. BERGEN AND MR. SPRINGATE WILL INDICATE; MAKES UP A MAJOR PORTION OF OUR AROCLOR BUSINESS.

WE WOULD BE LESS THAN HONEST, HOWEVER, IF WE DID NOT POINT OUT THAT THE SCIENTIFIC DATA TO BE DEVELOPED FROM OUR OWN RESEARCH AS WELL AS BY ANY NUMBER OF THE 50 GOVERNMENTAL OR UNIVERSITY LABORATORIES THAT HAVE REQUESTED SAMPLES OF AROCLORS MAY NOT BE FAVORABLE REGARDING AROCLOR 1242.

IN SUMMARY, THE MEDICAL DEPARTMENT FEELS THAT LONG-RANGE, THERE MAY BE LESS THAN A 50% CHANCE OF SUCCESS IN PROTECTING

THE MARKETS FOR THE POLYCHLORINATED BIPHENYLS. WE FEEL THE ODDS DEPEND ON AT LEAST FIVE FACTORS BEYOND OUR CONTROL:

1. WE MAY FIND THAT THE PREVENTION OF ESCAPE OF THESE MATERIALS TO THE ENVIRONMENT IS IMPOSSIBLE--EXCEPT IN VERY LIMITED APPLICATIONS.
2. ALTHOUGH, AS I HAVE INDICATED EARLIER, THERE IS SOME EVIDENCE THAT THE LOWER CHLORINATED MATERIALS SUCH AS AROCLOR 1242 ARE BIODEGRADABLE, FURTHER RESERACH SPONSORED BY MONSANTO OR OTHERS, MAY PROVE THAT THIS IS NOT THE CASE.
3. WE CANNOT CONTROL THE EFFORTS OF THE CHLORINATED PESTICIDE MANUFACTURES WHO--IN DEFENSE OF THEIR PRODUCTS, DDT AND THE OTHERS--HAVE BEGUN TO EMPHASIZE IN GOVERNMENT HEARINGS AND SCIENTIFIC SEMINARS THAT THE PAST AND CURRENT RESIDUE ANALYSIS MAY BE UNDULY ALARMING BECAUSE OF THE PCB INTERFERENCE.
4. IT WILL BE DIFFICULT--IF NOT IMPOSSIBLE--TO COUNTERACT THE EFFORTS OF OUR COMPETITORS IN THE FUNCTIONAL FLUID APPLICATIONS WHO HAVE BEGUN TO BROADCAST THE POTENTIAL PCB PROBLEM--NOT ONLY TO OUR CUSTOMERS BUT TO REGULATORY AGENCIES SUCH AS THE MICHIGAN DEPT. OF NATURAL RESOURCES, AND
5. WE MAY FIND IT IMPOSSIBLE TO COUNTERACT THE EFFORTS OF THOSE SCIENTISTS AND PSEUDO-SCIENTISTS NOW INVOLVED IN THE U. S. AND EUROPEAN WILDLIFE CONSERVATION EFFORT WHO--TO PUT IT MILDLY--DO NOT ALWAYS REACT RESPONSIBY.

NEV 176981

HAVE ASKED US FOR SAMPLES MAY NOT BE FAVORABLE TO AROCLOR 1242.

AS YOU CAN IMAGINE, WE IN THE MEDICAL DEPARTMENT HAVE BEEN CONCERNED DURING THESE MANY MONTHS ABOUT THE POSSIBILITY OF LEGAL AND FINANCIAL LIABILITY WHICH MAY FACE MONSANTO IN THIS SITUATION. Dennis Miller ~~BOB FRENCH~~ IS NEXT ON THE PROGRAM TO DISCUSS THIS ASPECT OF THE PROBLEM.

# F A C T S

1) THE 5 AND 6 CHLORINATED BIPHENYLS--OUR AROCLORS 1254 AND 1260--ARE PRESENT IN THE ENVIRONMENT, IN BIRDS, FISH, AND OTHER AQUATIC LIFE.

2) THESE, IN PART OR SOLELY, ARE AFFECTING REPRODUCTION OF SOME SPECIES OF BIRDS.

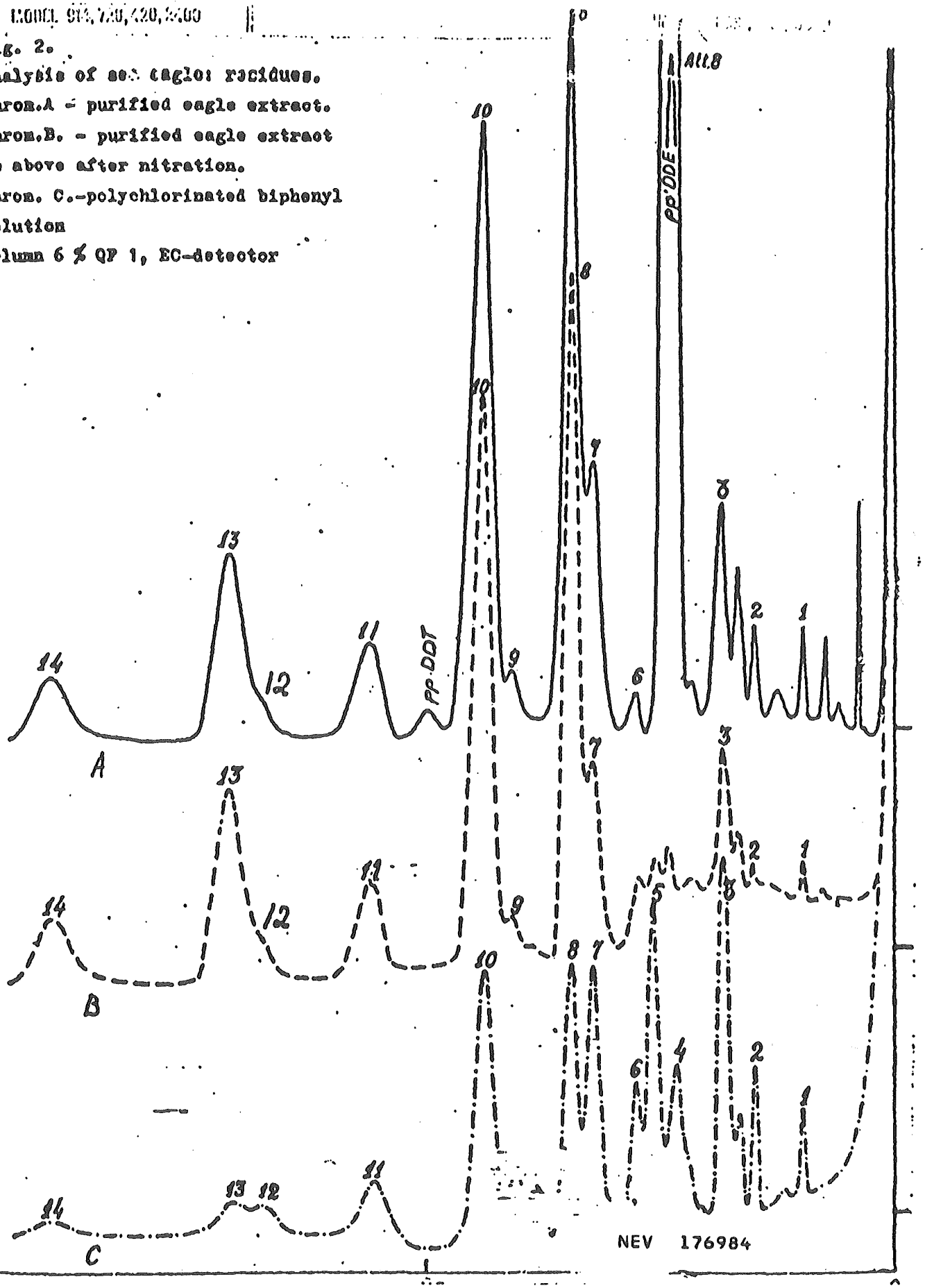
3) PCB'S ARE PARTICULARLY TOXIC TO SHRIMP.

4) THERE ARE STILL MANY UNKNOWNS BUT SPECULATION AS TO THE ROLE OF PCB'S IS RAMPANT.

5) SPECULATION IS LEADING TO CONCERN WHICH MAY GROW TO ALARM.

NEV 176983

Fig. 2.
Analysis of sea eagle residues.
Chrom.A = purified eagle extract.
Chrom.B. = purified eagle extract as above after nitration.
Chrom. C.-polychlorinated biphenyl solution
Column 6 % QF 1, EC-detector



1 PART PER MILLION --- 1 DROP IN 90 FIFTHS

1 PART PER BILLION --- 1 DROP IN 90,000 FIFTHS

OR --- 1 OUNCE IN 1,000 TANK CARS

NEV 176985

# I N D E X

THE WITNESS: ROBERT EMMET KELLY, M.D.
VOLUME III

Index (Continued)

# I N D E X

THE WITNESS: ROBERT EMMET KELLY, M.D.
VOLUME IV

May 25, 1934

REPORT OF DR. FREDERICK B. FLINN OF PATCH TESTS MADE ON MATERIAL RECEIVED FROM SWANN RESEARCH, INC.

The object of this investigation was to determine whether or not the various chlorinated diphenyl compounds submitted or some impurities contained therein might be the causative agent producing the dermatitis which had developed among some of the workmen in the plant.

Large white rabbits were used in making the patch tests to determine the action of the various materials submitted on the skin. The procedure was to shave the animal 36 hours before the patch was applied so that there would be no broken skin to interfere with or to aggravate the test. The material being tested was mixed with ethyl alcohol, either getting a solution or a suspension for the purpose of diluting the material. The patch was left on for various periods during the tests - some for 24 hours, others for 48 hours. The patch was then removed and the exposed area watched for two weeks to be sure that the test was negative. This was done to avoid the chance of not observing any latent or delayed action such as do sometimes take place in making patch tests. For our own information the materials which were negative were applied in a concentrated form in duplicate tests to see if a dermatitis might not develop under these circumstances.

Eight independent tests were made with each material and were made on different days with freshly prepared solutions to be sure that no error had crept in. Controlled tests were made each time with the alcohol and gauzes used to determine whether they might not be the causative agent causing the observed lesions.

Given below is Dr. Flinn's report, showing designations given the various materials by Swann Research followed by Dr. Flinn's designation and report of each specific material:

Aroclor 1268 (Lot S, made before 8/1/33).

Aroclor 1268, Lot S:- All eight tests were negative. The two tests with concentrated material were also negative. An intradermal test was negative.

Aroclor 1268 (Lot Notebook No. 176, page 121; prepared by Harris of 94.5% diphenyl and 4.5% styrene and styrene high boiler:

Aroclor 1268, Lot Notebook 176, page 121, was negative to all tests including the concentrated and intradermal.

Kelly
EXHIBIT NO. 38
2-17-94 ICR
GBRN002055

Aroclor Special (Lot Notebook No. 192, page 9; prepared by Hubbard, of 75% diphenyl and 25% styrene and styrene high boiler). (Liquid Aroclor, specific gravity = 1.55).

Aroclor Special Notebook No. 192, page 9:- This chemical gave a positive test with each of the eight applications. It also gave a positive reaction with the intradermal test. The type of dermatitis was mild.

Aroclor 1248 (Lot 9, made 5/30/36):

Aroclor 1248, Lot 9: All eight tests gave a positive reaction. The intradermal test was also positive. The reaction was mild.

Aroclor 1248 (Lot 5, made 5/2/35):

Aroclor 1248, Lot 5 All eight tests gave a positive reaction with this compound. The intradermal test was also positive. I feel that the dermatitis produced by this material was milder than the two previous compounds which were also found to be positive. In several tests it did not make its appearance for 24 hours after the patch had been removed.

Aroclor 1269 (Lot 3, Dry Ground; made 6/22/38):

Aroclor 1269, Lot 3, ground dry: All eight tests were negative. The same is true of the two tests made with the concentrated material. The intradermal test gave negative reaction.

Aroclor 1269 (Lot Notebook No. 176, page 121; prepared by Harris, of 95.5% diphenyl and 4.5% styrene and styrene high boiler - dry ground):

Aroclor 1269. Lot Notebook 176, page 121, dry ground: All eight tests were negative. The concentrated tests were also negative. The intradermal test gave negative reaction.

Aroclor 1269 (Lote Notebook No. 176, page 121; same as immediately above but wet ground)

Aroclor 1269, Lot Notebook 176, page 121, wet ground: All eight tests were negative. The two tests made with concentrated material were also negative. The intradermal test gave a negative reaction.

Aroclor 1269, Lot 3, Notebook No. 192, page 10 (dry ground in pebble mill):

Aroclor 1269, Lot 3, Notebook No. 192, page 10: The eight patch tests were negative. The tests made with concentrated material were also negative. The intradermal tests gave a negative reaction.

Halowax #1000:

Halowax 1000: This compound gave a positive test in all of the eight tests in which it was applied. The reaction was different from that of the three Arochlors giving a positive reaction. They were mild, but this material gave an ulcerative area.

Halowax #1001:

Halowax 1001: All tests with this compound, both concentrated and dilute, were negative.

Halowax #1004:

Halowax 1004: All tests with this material, both concentrated and dilute, were negative.

Styrene Dichloride (Notebook No. 192, page 16):

Styrene Dichloride: Notebook 192, page 16: Every test made with this material was positive. It was noticed that the rabbit pulled away when this chemical was applied even in the dilute form. This also gave an ulcerative lesion.

Arochlor 1248 Special (Batch 8, Sample 04 - made 10/4/30):

Arochlor 1248 Special Batch 8-0-4. Made 10/4/30: This Arochlor gave a very slight reaction with the skin but one must say that it is positive.

Arochlor 1260 (Lot 32, made 6/23/31):

Arochlor 1260. Lot 32. Made 6/23/31): All eight tests were negative. No intradermal or concentrated tests were made.

Arochlor 1262 (Batch 8A, Sample J.L-6, Made 7/8/30):

Arochlor 1262. Batch 8A. Made 7/8/30: All tests were negative.

Chlor Ethyl Benzene (Notebook No. 192, page 21):

Chlor ethyl benzene. Notebook 192, page 21: Every test made with this material gave a positive test. The lesion was of an ulcerative type.

Chlorinated Styrene (Notebook No. 192, page 19 - 5/16/34):

Chlorinated Styrene. Notebook 192, page 19. 5/16/34: Every test made with this compound gave an ulcerative lesion.

Dr. Flinn's comments are copied verbatim, as follows:

"Comments:

One is impressed with the fact that each of the Aroclors giving a positive reaction were of a fluid nature. Attempts were made to expose the animals to vapors but observations made us conclude that the only difference was that the animal would be exposed to the hot material and it was well known that the reaction where such an exposure is given is more severe.

One cannot but feel that any styrene compound which may be found to be present as an impurity is the cause of your trouble. I was rather surprised that more of the compounds submitted did not show or give reactions with the skin. It has been shown in some investigations that chlorine did not produce a dermatitis when metallic electrodes were used but did if the metallic electrodes were replaced by carbon electrodes. The theory was advanced that some organic chlorine compounds were produced in the latter case.

I would suggest that you study your ventilation system in places where fumes are given off. That means be provided for the men to take a bath with soap and water if they come in contact with the type of material found to be positive. By this I mean if a leak or spillage occurs. The immediate bathing under these circumstances should be insisted on. I found that where I had gotten the material on my hands if I washed them thoroughly no trouble arose.

(Signed) Frederick B. Flinn

(2)

Medical Research Project No. MR-46.

THE TOXICITY AND POTENTIAL DANGERS OF INERTEEN

Submitted by

W. F. von Oettingen, M.D., Ph.D.

Director

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Wilmington, Delaware.

## The Toxicity and Potential Dangers of Inerteen and Related Substances, Including a Method of Analysis for Halogenated Hydrocarbons in Air.

W. F. von Oettingen, M.D., Ph.D.

The toxicological experiments on this problem were carried out by Dr. J. H. Foulger and Dr. A. J. Fleming of the Departmen of Toxicology and the pathological studies were made by Dr. W. Read, Jr. of the Department of Pathology of the Haskell Laborat

The material was investigated with oral administration to rats, application to the skin of rats, and in inhalation experi ments. The latter were planned in such a way as to approach as closely as possible the actual conditions encountered in the us of this material in transformers, in order to evaluate the maxi hazard which may be encountered in its use for this purpose. F comparison, additional tests were made regards the toxic effect from inhalation of vapors from Pyranol and Transformer Oil (We inghouse). In addition, a method was worked out for the deter mination of Inerteen in air.

### Effect of Oral Administration.

In order to determine the minimal fatal concentration of Inerteen for rats, 100 animals were given by stomach tube sing doses of Inerteen, as indicated in the table.

| Dose cc./kgm. | No. of animals treated | No. of dead | Time of death | Per cent mortality |
|---|---|---|---|---|
| 1.0-1.49 | 26 | 5 | 4 ✠ within 3 days | 19.2 |
| 1.5-1.99 | 24 | 9 | 6 ✠ " 2 " | 37.5 |
| 2.0-2.49 | 10 | 3 | 3 ✠ " 2 " | 30.0 |
| 2.5-2.99 | 20 | 10 | 9 ✠ " 2 " | 50.0 |
| 3.0-3.49 | 20 | 15 | 15 ✠ " 3 " | 75.0 |

In these, as in subsequent experiments, all rats showed signs of discomfort immediately following the administration, later they became depressed and refused, frequently, water and food; within a few hours they became weak and stuporous, so that they were finally unable to rise. When this stage was reached they invariably died. One of these rats was found to suffer from albuminuria, one developed tremors of the head and fore-limbs, and one showed severe hemorrhages from the intestinal tract. The table indicates that with oral administration the minimal fatal dose of Inertech for rats, which kills 75 per cent of the animals within 5 days, is 8.0 to 2.49 cc. per kilogram body weight.

Upon pathological examination of 20 of these animals, those that had died spontaneously showed congestion of the lungs, which was combined with edema and hemorrhage in several instances. Ten of the animals had mottled gray or yellow livers and in three instances there was a diffuse pallor with moderate yellowness. In 5 out of 6 animals studied microscopically the liver showed marked and rather extensive hyaline necrosis in the central part of the lobules; the liver cells in this area revealed fatty degeneration of the cytoplasm; and in 3 animals that died 52 and 54 hours after the treatment these cells contained increased numbers of mitotic figures. In the majority of the animals the mucosa of the intestine was congested and the fecal material in the intestines contained old blood; and in one instance fresh blood was found in the lower third of the ileum, lower colon,

and sigmoid. Microscopically the stomach of two animals presented typical hemorrhagic erosions of the mucosa; another showed hemorrhagic areas in the mucosa; and in the remaining animals postmortem changes did not allow an exact investigation

In order to follow up the toxic effect of Inerteen on the liver, a group of 30 white rats was treated in the same way wit single oral doses of 1.15 to 1.20 cc. per kilogram body weight. Six of these were killed 3, 8, and 11 days, respectively, after the administration.

Upon pathological examination it was found that two of the 6 animals killed after 3 days had bloody materials in their stomachs and in 50 per cent of these animals the liver was slightly yellow in color and the liver cells were large and con tained numerous mitoses and occasionally there was necrosis of cells immediately about the central veins. Animals killed aft 8 days presented a slight enlargement of the liver with a ques tionable yellowish color, the liver cells were large, those ar the central veins were dark, and occasionally there was some fatty degeneration of these cells. Rats killed after 11 days showed no important gross pathology. Microscopically, all sec tions showed a slight swelling of the liver cells with occasio vacuolization of the cytoplasm. No other organs showed pathol changes referable to the exposure.

In order to study the effect of repeated oral administrat of Inerteen, 3 groups of 10 rats each were treated in the foll ing way:

The first group received from 0.85 to 0.86 cc. per kilo for 3 consecutive days. None of them died and 5 were killed after the 3rd treatment for pathological examination. The second group was fed doses from 0.83 to 0.88 cc. per kilo. Two died following the 2nd dose and 5 were killed for autopsy after the 3rd administration. In the third group the doses ranged from 1.23 to 1.78 cc. per kilo. One rat died after the 1st, three after the 2nd, and four after the 3rd treatment, and the remaining animals were killed four days after the 3rd treatment.

Upon pathological examination the livers of the rats of the first group appeared somewhat lighter than usual and one showed large yellow areas; there was a diffuse mild swelling of the liver cells with some polychromasia; occasionally the cells show some vacuolar changes of the cytoplasm, but in general these changes were not very severe. In 4 rats of the third group which died after 3 treatments the stomachs were dilated with food and in two of these there was also some old blood present. Microscopically, one rat had small inflammatory ulcers in the fore stomach and another showed hemorrhagic erosions of the pylorus. All showed some hyperemia of the liver and in one rat this was of mottled appearance. Microscopically two rats showed swelling of the liver cells with fatty degeneration of those in the central parts of the liver lobules. Two others presented central necrosis of the liver cells with hyperplasia of the endothelial cells in these areas, one showing in addition, numerous mitotic figures

the liver cells. Of 2 rats which were killed 4 days following the 3rd administration, one had a small amount of blood in the gastric content. The livers of both animals appeared moderately enlarged and microscopically there was a generalized swelling of the liver cells with some polychromasia. One rat suffered from a fairly general vacuolar degeneration of the liver cells and another presented vacuolar changes in the central part of the lobules.

In order to compare the toxic effects of Inerteen with trichlorobenzene and ethyl-tetra-penta-chlorobenzene, a group of 10 rats was given daily doses of trichlorobenzene ranging from 0.36 to 0.58 cc. per kilo body weight. One died after the 1st, four after the 2nd and four after the 3rd administration, the remaining rat was killed 4 days following the 3rd and last treatment.

It appears, therefore, that daily doses of 0.36 to 0.58 cc. per kilo of trichlorobenzene killed 80 per cent of the animals with 3 doses, whereas it was found that doses of 0.65 to 1.04 cc per kilo of ethyl-tetra-penta-chlorobenzene, given in the same way, killed only 10 per cent, and Inerteen in doses of 1.42 to 1.78 cc. per kilo, given in the same way, killed 80 per cent of the rats.

Upon pathological examination, animals which were treated with trichlorobenzene and which died spontaneously showed a passive congestion of the lungs and the liver; the stomachs were distended with food; in four some blood was present in the

gastric contents and in one instance there was a haemorrhagic erosion of the mucosa. In two rats the liver showed irregular mottling. Liver sections of all animals showed necrosis of the liver cells around the central veins and those in the outer parts showed vacuolar degeneration. In rats which died 3 to 4 days after the first treatment there was some endothelial hyperplasia in the areas of necrotic liver cells. One animal presented an increase in the number of mitotic figures in the liver cells.

Of the 7 rats treated with ethyl-tetra-penta-chlorobenzene, the one which died spontaneously after the 3rd dose showed congestion and haemorrhages of the lungs, generalised passive congestion of the spleen, bone marrow and other viscera, distended stomach, and moderate yellow mottling of the liver, the liver cells being mildly swollen and showing fine vacuolar degeneration The animals which were killed showed only a questionable enlargement of the liver and kidney and a slight swelling of the liver cells with occasional vacuolar degeneration, which changes were extremely mild.

It appears, therefore, that Inerteen is considerably less toxic than trichlorobenzene, the minimal fatal dose with oral administration, killing 70 per cent of the animals, being around 2.0 cc. per kilo rat; whereas ethyl-tetra-penta-chlorobenzene does not appear to be nearly as toxic with oral administration. The pathological changes observed after oral administration of Inerteen appear to be fairly consistent and obvious. Large doses

which are fatal in one or two days, cause terminal passive congestion of the viscera, extensive degeneration of the liver with necrosis around the veins, and locally, ulceration of the gastric mucosa. Thereas sublethal doses cause within three days gastric irritation and hepatic degeneration with signs of regeneration, the latter is decidedly more marked after eight days, and after eleven days no definite liver pathology may be present. It should also be pointed out that single large doses appear to be more injurious than such doses given in fractions.

Effect of Application to the Skin.

Ten white rats were treated daily with an application of about 0.5 cc. of Inerteen to the shaved skin of the back, precautions being taken to prevent absorption through the gastro-intestinal tract by licking and by inhalation. All rats lost weight during the experiment but they showed no toxic signs aside from a slight restlessness at the beginning of the experiment. At the site of the administration the skin became dry, thickened and scaled without any fissures being formed. Similar reactions were observed on the human skin especially on the hands, cheeks and forehead. One of the rats died after 11, and three after 13 treatments, the remaining being killed 24 hours after the 18th application.

Upon pathological examination the liver of all animals was found to be dark gray brown in color with mottling on the

surface. In those rats which died spontaneously there was fatty degeneration of the liver cells with necrosis of those around the central veins and in animals killed at the end of the experiment there was a mild swelling of the liver cells with occasional vacuolar degeneration in some. Microscopically the skin at the site of application of 4 rats showed hyperkeratinization with occasional blister formation, one animal showed perifolliculitis, one hyperplasia of the epithelium rather than hyperkeratinization, and in 3 rats there was a mild fibroblasti proliferation with some round cell infiltration in the underlyi connective tissue.

It appears, therefore, that the contact of Inerteen with the skin does not only cause local reactions such as dryness, thickening and scaling of the skin, but also systemic effects as indicated by the injurious effect on the liver.

Effect of Inhalation of Vapors.

In evaluating the toxic effects of inhalation of vapors liberated from Inerteen at elevated temperatures, attempts were made to reproduce conditions more severe than would be met in the overheating of a transformer. In these experiments rats were exposed in a limited space of about 1 cubic foot capacity to the vapors of the boiling material which entered directly into the chamber through a hole in the bottom. In each experiment 4 rats were exposed on 58 to 66 occasions for 30 minutes, twice daily, to vapors from Inerteen, Pyranol, or

Transformer Oil (Westinghouse). During this time they showed irritation of the skin, which appeared to be most marked on those parts of the body not protected by hair, and which persisted for some time after the exposure. Pyranol appeared to produce the most effect in this respect, followed by Inerteen and Transformer Oil, in the order given. In opposition to the 8 rats exposed to Pyranol and Transformer Oil, 3 out of 6 rats exposed to Inerteen lost weight, but no other toxic signs were observed. It is quite possible that rats exposed in this way absorbed some toxic material through the gastro-intestinal tract by licking the fur during or after the exposure since the material had a tendency to condense on the walls of the chamber and on the surface of the animals. The maximum concentration of Inerteen to which these rats were exposed was determined as about 0.5 m.g. per liter of air.

Upon pathological examination of rats exposed in this way on 14, 35 and 66 occasions to Inerteen, no definite pathological signs could be observed in the liver or other organs which could be referred to the exposure.

Two rats treated with 41 exposures to the vapors of Pyranol showed no definite cellular changes in the liver although in one animal there was an increased number of mitotic figures in the liver cells.

Two rats exposed on 44 occasions to the vapors of Transformer Oil (Westinghouse) showed no definite pathological changes of the internal organs.

In order to study the effects of continued exposure, 10 rats were exposed 8 hours daily on 5 days a week for 20 days to concentrations of 0.05 mg. to 0.09 mg. of Inerteen per liter of air. They showed no toxic nor pathological signs referable to the exposure; there were no definite changes of the red blood corpuscles or the hemoglobin. The animals were killed 2 days after 20 exposures.

It appears, therefore, that neither frequently repeated short exposures for 20 minutes each to concentrations of 0.5 mg. of Inerteen per liter air, nor continued exposure to concentrations of 0.05 to 0.09 mg. per liter air cause a definite toxic effect on the liver.

Determination of Concentrations of Inerteen in the Air.

In order to determine the concentration of Inerteen vapors in the air, the following nephelometric method was worked out, after several other procedures had been considered.

A definite volume of air to be tested is drawn at a rate of about 1 liter every 4 minutes through a test tube containing 5 cc. of ethyl alcohol, immersed in dry ice-acetone mixture in order to prevent the evaporation of the alcohol. At the end of the sampling 1 cc. of the alcohol was mixed with 10 cc. of distilled water and the resulting turbidity was measured with a photo electric cell, using a sodium vapor lamp as the source of light.

The turbidity of unknown samples was compared to the

These experiments show that the concentrations of Inerteen in air decrease rapidly with the distance from the source of their production.

In order to study variations of the concentrations over a period of time and with different temperatures, a current of 10 liters per minute of air was passed over the surface of Inerteen of about 10 square inches, contained in a flask and thence into a bell jar. Samples were taken from the jar at various intervals after the flow was started and with various temperature The following concentrations were determined:

| Time of sampling in hours | Concentration of trichlorobenzene in mg. per liter | Temperature of trichlorobenzene in flask °C. |
|---|---|---|
| | Trichlorobenzene | |
| ½ | 0.29 | 23 |
| 2 | 0.51 | 23 |
| 6 | 0.60 | 23 |
| 1 | 1.68 | 50 |
| 4 | 1.70 | 50 |
| 1 | 1.80 | 90 |
| | Inerteen | |
| 6 | 0.58 | 23 |

This tabulation shows that the determinations made at 23°C. and with the 6 hour sample were almost the same for trichlorobenzene (0.60 mg. per liter) and Inerteen (0.58 mg. per liter). This suggests the possibility that the bulk of the vapors liberated from Inerteen at this temperature may be trichlorobenzene; so that the hazards from inhalation of Inerteen vapors are identical with those from the inhalation of

trichlorobenzene. In order to confirm this, 885 cc. of Inerteen were submitted to fractional distillation and the following results were obtained:

| Fraction | Temperature of distillation °C. | Volume collected cc. | Specific Gravity of fraction | Boiling Point °C. |
|---|---|---|---|---|
| 1 | 240 | 35 | 1.469 | 208-219 |
| 2 | 240-254 | 45 | 1.478 | 228 |
| 3 | 254-280 | 45 | 1.493 | 240 |
| 4 | 280-300 | 80 | 1.577 | 269 |
| Residue | above 300 | 180 | 1.592 | - |

Since the specific gravity of trichlorobenzene is 1.466 and the boiling point 208°C.-219°C., it is evident that up to 240°C. the vapors consist exclusively of trichlorobenzene and that for this reason the above statement is justified, namely, that for most conditions the hazards from vapors of Inerteen are identical with those from trichlorobenzene.

## Résumé.

The experiments discussed in this report show that with oral administration Inerteen is considerably less toxic than trichlorobenzene, the minimal fatal dose killing from 70 to 75 per cent of the rats being around 3.0 cc. per kilogram body weight and 1.4 to 1.8 per kilogram body weight, respectively. Fatal doses will cause passive congestion of all viscera, extensive degeneration of the liver, and ulceration of the gastric mucosa. With sublethal doses these effects are less severe and there is evidence that they will heal when no further exposure occurs.

Continued application of Inerteen to the skin of rats and humans causes dryness of the skin, thickening and scaling; and the observations made in rats indicate that sufficient quantities may be absorbed through the skin to produce injurious effects on the liver.

Repeated inhalation for short periods of concentrations of 0.5 mg. of Inerteen vapors per liter of air, or repeated inhalation for 8 hours of concentrations of 0.05 to 0.09 mg. per liter of air causes no definite injurious effects in rats. Vapors liberated from boiling Pyranol, Inerteen, and Transformer Oil (Westinghouse) are irritant and the irritant action decreases from Pyranol to Transformer Oil (Westinghouse), Inerteen being intermediate in this respect.

It has been shown that vapors liberated from Inerteen under conditions approaching those as might be encountered in transformers consist mainly of trichlorobenzene.

A simple method for the determination of Inerteen (trichlorobenzene) vapors in air is given. If 10 liters of air are drawn at a rate of about 1 liter every 2 minutes through a test tube containing 1 cc. of ethyl alcohol, cooled in dry ice or in an ice salt mixture, and the alcohol is then poured carefully on the surface of a few cubic centimeters of water in a test tube, a ring of turbidity is produced if the concentration of Inerteen (trichlorobenzene) is over 0.03 mg. per liter of air. If under these conditions a white ring is produced,

the air tested should be regarded with suspicion and should be improved by more effective ventilation.

It was found that the concentration of Inertem (trichlorobenzene) in a closed space 14 inches above the surface of boiling Inertem is 4.8 mg. per liter and in an open space 5 inches above the surface of boiling Inertem is 0.56 mg. per liter, and when the temperature in a transformer, standing in a closed room (vault), rises to 90°C. the air may contain 1.8 mg per liter of air.

From the animal experiments reported, it appears that concentrations below 0.05 mg. per liter of air will cause no toxic signs or symptoms even with continued exposure.

Regards preventive measures, the following may be suggeste

It appears to be of paramount importance that the contamination of the air be reduced to concentrations below 0.05 mg. pe liter of air by adequate forced ventilation at the site of the production of such vapors.

Greatest personal hygiene is not less important. Contamination of the skin should be avoided by wearing proper protectiv garments such as gloves, caps, and coveralls. In case of accide or with exposure to higher concentrations of and above 0.2 mg. p liter, respirators or open air masks should be worn. The skin should be kept immaculately clean and ointments such as lanolin or Aquafor should be applied to the skin after washing the hands

In view of the hepatotoxic action of this compound, individuals suffering from injuries of the liver, syphilis, and hear

diseases should be excluded from operations in which Inerteen is handled.

Workers handling Inerteen or with frequent exposure to its vapors should have periodic examinations, special attention should be paid to their nutritional condition, and the condition of the liver may be checked by determining the icteric index in the blood, and the urobilin and urobilinogen excretion in the urine.

Regards the treatment of toxic effects produced by exposur to Inerteen, it appears advisable to increase the resistance of the liver by carbohydrate diet; administration of glucose and insulin may be advisable, and duodenal lavage with magnesium sulfate may be tried. Local irritation of the skin produced by Inerteen should be treated with bland ointments after the toxic material has been removed by intensive washing and scrubbing. In case Inerteen has been taken by mouth, the toxic material should be removed from the stomach by emetics such as soap water or mustard water, and this should be followed by gastric lavage with a suspension of activated charcoal in water and saline cathartics such as Epsom salts.

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# THE TOXICOLOGY OF INERTEEN AND RELATED SUBSTANCES INCLUDING A METHOD OF ANALYSIS FOR HALOGENATED HYDROCARBONS IN THE AIR.

A. J. Fleming, M. D.

The Toxicology of Inerteen was investigated by oral administration, skin application, and inhalation experiments in rats. In the latter an attempt was made to investigate the substance in a way relative to its use in transformers and to determine the maximum hazard that might arise should the substance be used for that purpose.

The minimum fatal dose was determined by oral administration and the effect of repeated oral doses was studied and compared with the effect of similar doses of trichlorobenzene and ethyl-~~tetra~~-penta-chlorobenzene.

A method of air analysis for the halogenated hydrocarbons was devised and air analyses were made to determine the concentrations of Inerteen in the air near open and closed containers when the substance was heated to various temperatures and when samples were taken at various distances from the source. A statement of the problems incident to the toxicology of Inerteen and the answers to these problems will be found in the conclusions and summary at the end of the paper.

## Inerteen

### Oral Administration:

(1). Single Doses.

The substance was given in single graded doses to several groups of rats to determine the minimum fatal dose.

TABLE I

MINIMUM FATAL DOSE OF INKHTEEN WITH SINGLE ORAL ADMINISTRATION

| Dose gm./kilo | No. of Animals Treated | No. of Deaths | Time of Deaths | Percent Mortality |
|---|---|---|---|---|
| 1.00 - 1.49 | 26 | 5 | 4 died within 3 days | 19.2 |
| 1.50 - 1.99 | 24 | 9 | 6 " " 3 " | 37.5 |
| 2.00 - 2.49 | 10 | 3 | 3 " " 3 " | 30.0 |
| 2.50 - 2.99 | 20 | 10 | 9 " " 3 " | 50.0 |
| 3.00 - 3.49 | 20 | 15 | 15 " " 3 " | 75.0 |

The results are conveniently tabulated in table 1.

Table 1

As noted, a dose of 8 to 8.5 cc. per kilogram gave a 75 per cent mortality. Twenty-two of this group of animals were autopsied. Another group of 30 rats was given single oral doses ranging from 1.15 to 1.20 cc. per kilogram. Six of these were killed 3 days after treatment and examined by the pathology department for possible liver damage. Six were killed 8 days after treatment and examined for possible kidney change and 6 were killed 11 days after treatment, and examined for possible brain damage.

(2) Repeated Doses

Thirty rats were divided into groups of ten. The first group received from 0.35 to 0.56 cc. per kilogram for 3 treatments. None died and 5 were killed for autopsy within 24 hours after the 3rd treatment. In the second group, the dosage ranged from 0.63 to 0.88 cc. per kilogram. Two died after the second treatment and 5 were killed for autopsy 24 hours after the last treatment. In the third group the dosage ranged from 1.23 to 1.78 cc. per kilo. One died after 1 treatment, 3 died after 2 treatments, and 4 died after the 3rd treatment. All animals in this last group were autopsied within 24 hrs. of the last treatment.

In general all rats behaved the same following oral administration of Inerteen. They proceeded at once to eat as if to allay the discomfort caused by the substance. Following this they became depressed and would not eat and often would not

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drink. Weakness and stupor generally supervened within a few hours until the animals could no longer rise for food and water. Liquids were taken, however, if administrated and for a few minutes following administration the animals perked up a bit. The improvement was only temporary and the outcome, when they had thus far progressed, was invariably fatal. One animal showed an albumenuria (tested post mortem) and one developed tremors of the head and front limbs. One had severe hemorrhages from the rectum and the autopsy indicated the bleeding was from the lower end of the small bowel.

Comparison of Inerteen with Trichlorobenzene and Ethyl-tetra-penta-chlorobenzene

A group of 10 animals was given repeated doses of trichlorobenzene ranging from 0.36 to 0.58 cc. per kilogram. One died after the 1st treatment, 4 died after the 2nd, and 4 died after the 3rd. The remaining animal was killed 4 days after the 3rd and last treatment. All animals were autopsied. Similar groups were treated with Inerteen and ethyl-tetra-penta chlorobenzene and the following noted:

Daily doses of 1.23 to 1.76 cc. per kilogram of Inerteen killed 80 per cent of the animals, when 3 doses were given, while daily doses of 0.36 to 0.58 cc. per kilogram of trichlorobenzene killed 90 per cent of the animals with 3 doses On the other hand daily doses of ethyl-tetra-penta-chlorobenzen ranging from 0.62 to 1.04 cc. per kilogram killed 1 rat in a group of 10 with 3 doses.

Conclusion: C0000454

The minimum fatal dose of Inerteen appears to be

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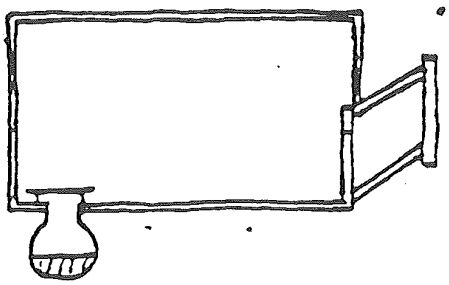
around 8.0 cc. per kilogram. Repeated doses of Inerteen were fatal in concentrations between 0.86 and 1.78 cc. per kilogram, while repeated doses of trichlorobenzene were fatal in concentrations between 0.86 and 0.58 cc. per kilogram. Ethyl-tetrapenta-chlorobenzene does not appear to be nearly so toxic by oral administration.

Skin Exposure:

Half a cubic centimeter of Inerteen was applied daily to the shaved backs of 10 rats by means of a small glass cup filled with a piece of cotton wool soaked in the substance. The whole was strapped to the animals with adhesive tape. One rat died after 11 treatments. Three died after 13 treatments and the remaining animals were killed for autopsy 24 hours after the 15th treatment. All rats lost weight during the experiment, but outside of a slight restlessness at the beginning of the experiment showed no other signs. The Inerteen acted locally on the skin, producing an excessive dryness with subsequent thickening and scaling without any fissures being formed. The same effects were noted on the human skin with respect to the drying and slight scaling and were most notable on the hands, cheeks and forehead.

Inhalation

An attempt was made to reproduce conditions more severe than would be met in the overheating of a transformer and exposing rats to these conditions for 20 minutes twice a day.



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The aforegoing diagram represents a box of about 1 cubic foot capacity, in one corner of which is the inlet fro a glass flask containing the substance being investigated. In ortoon, Pyranol or transformer oil (Westinghouse) were placed in the flask and brought to a boil. Two rats were then placed in the box and allowed to remain for 30 minutes, during which time a generalized itching of the skin appeared which was most marked in the parts of the body not protected by hair. The itchiness persisted for sometime after the animals were remove from the box. Pyranol seemed to produce the most effect in this respect with Inerteen and Transformer oil following in the order named. Three out of 6 of the Inerteen treated rats lost weight, while the Pyranol and Transformer oil treated rats gain weight (2 rats only in each of the last two groups). It is quite possible that these animals ingested a considerable amoun of these substances by licking their fur after removal from the box. No marked change in respirations were noted, although the animals once or twice breathed faster than normal and occasionally the extremities were slightly blue. At autopsy no gross pathology was noted. The maximum concentration of Inerteen to which these rats were exposed was about 0.5 mg. per liter. The box was aired between readings, but not washed out.

With Inerteen two rats received 28 treatments, two received 35 treatments and two received 66 treatments. With Pyranol two rats received 42 treatments and with Transformer Oil two rats received 44 treatments.

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A group of ten rats was exposed for 20 days, eight hours per day to concentrations of Inerteen in the air ranging from 0.03 to 0.09 mg. per liter. All rats were killed for patology within two days following the 20th days of treatment. Eight of this group gained weight, one lost weight and one showed no change. Five showed a slight drop in the red blood count, three showed an increased red blood count and two showed no change. The changes in the hemoglobin paralleled the change in the red blood count.

Air Analysis for Halogenated Hydrogenated Hydrocarbons.

Considerable difficulty was experienced in obtaining a satisfactory method of air analysis for these substances and a nephelometric method was finally adopted. In this, a definite volume of the air to be tested was drawn at a rate of about 1 liter every four minutes through 5 cc. of ethyl alcohol cooled in a dry ice-acetone mixture. The latter being necessary to prevent evaporation of the alcohol. One cubic centimeter of the alcohol was then mixed with 10 cc. of distilled water and the resultant turbidity measured with a photo electric cell, using a sodium vapour lamp as a source of light. The turbidity of unknown samples was compared to the turbidity produced by known standards made up of trichlorobenzene and ethyl alcohol. This method gave a satisfactory range of readings for concentrations varying from 0.30 mg. per liter to 0.03 mg. per liter which, when the original dilutions were taken into account, was sufficient to detect as low as 0.03 mg. per liter of Inerteen in the air tested. The method is not specific for the various members of the halogenated hydrocarbons.

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Sampling at Various Distances from a Source of Inerteen.

The effect of varying the distance at which samples were taken from a closed space over boiling Inerteen was determined with the following results:

| Distance Inches | Concentration Mg. per liter |
|---|---|
| 14 | 4.50 |
| 20" | 1.38 |
| 28" | 1.14 |
| 36" | 1.14 |

Similarly the effect of varying the distance at which samples were taken over an open beaker of boiling Inerteen was determined with the following results:

| Distance Inches | Concentration Mg. per liter |
|---|---|
| 5 | 0.56 |
| 10 | 0.48 |
| 15 | 0.24 |
| 20 | 0.24 |

The concentrations fall off rapidly and if any cooling surface is above the Inerteen and no air currents are blowing over the surface of the Inerteen, there is much less chance of any appreciable amount escaping into the atmosphere as from a container like a transformer.

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Sampling at Various Time Intervals and Temperatures.

Air at 10 liters per minute was passed over a surface of Inerteen (about 10 sq. inches) contained in a flask and thence into a bell jar. Samples were taken from the bell

3000020

jar at various intervals after the air flow was started and under various conditions of temperature.

| | Time of Sampling Hours | Concentration of Trichlorobenzene Mg. per Liter | Temperature of Trichlorobenzene °C. |
|---|---|---|---|
| At 23°C. | 1/2 hour | 0.89 | 23 |
| | 2 " | 0.51 | 23 |
| | 6 " | 0.60 | 23 |
| At 50°C. | 1 " | 1.68 | 50 |
| | 4 " | 1.70 | 50 |
| At 90°C. | 1 " | 1.90 | 90 |
| Inerteen at 23°C. | 6 " | 0.58 | 23 |

From the foregoing it will be noted that the 6 hour sample with trichlorobenzene at 23°C. gives almost the same concentration (0.60 mg. per liter) as did the six hour sample with Inerteen at 23°C. (0.58 mg. per liter). This suggests the possibility that the bulk of the stuff coming off the Inerteen at that temperature may be trichlorobenzene and that the hazards o Inerteen may thus be the hazards of trichlorobenzene. Fractional distillation of about 325 cc. of Inerteen was carried out in a distilling flask with a 300°C. thermometer reaching to the level of the side arm. The distilling flask was attached to a Liebig condenser and a collecting flask. Samples were collecte over temperature ranges from 240°C. to 300°C. (The temperature recorded being that of the vapors around the thermometer bulb. The distance between the bulb and the surface of the liquid was about 6 inches. Although the liquid boiled vigorously several minutes elapsed before the vapor reached the bulb). The boil-

ing point and specific gravity was measured for each fraction collected.

| Fraction No. | Temperature of Condensing Liquid °C. | Boiling Point Fraction °C. | Spec. Grav. | Vol. cc. |
|---|---|---|---|---|
| 1 | 240 | 208 - 219 | 1.469 | 35 |
| 2 | 240-254 | 228 | 1.478 | 45 |
| 3 | 254-280 | 240 | 1.493 | 43 |
| 4 | 280-300 | 269 | 1.577 | 80 |
| Residue | Above 300 | ---- | 1.592 | 180 |

Fraction #1 was probably trichlorobenzene (Specific gravity 1.466, boiling point 208 - 219°C.).

Conclusions and Summary:

In investigating the toxicology of Inerteen answers to the following questions were sought and are here given as far as possible.

(1). What is the composition of the vapors liberate from Inerteen at a maximum working temperature of 90°C.,

Trichlorobenzene only.

(2). What is the maximum concentration of this fraction to which workers might possibly be exposed,

(a) In a closed space 14" over boiling Inertee 4.5 mg. per liter.

(b) In an open space 5" over boiling Inerteen 0.56 mg. per liter.

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(3). What might be the maximum concentration in a room where transformers are placed when the temperature of the transformers are 90°C. and the air has a chance to get satu-

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1.9 mg. per liter.

(4). What is a safe concentration as shown by animal experiments. Considering the hazard is due, under ordinary working conditions to trichlorobenzene alone,

(a). From trichlorobenzene data previously obtained, .058 mg. per liter.

(b). From Inerteen data - .048 mg.per liter

The last figure has been verified by exposing rats for 8 hours per day to such concentrations over a period of weeks.

(5). Is there a simple test that will detect concentrations of Inerteen below .048 mg. per liter,

Yes. If 10 liters of air are drawn at a rate of about one liter every 8 minutes through 1 cc. of ethyl alcohol (cooled in dry ice or an ice salt mixture) and the alcohol poured over the surface of a few cc. of distilled water in a test tube a ring of turbidity will be produced if the concentration is over 0.03 mg. per liter. If a ring is produced, the air tested should be regarded with suspicion and increased ventilation provided.

(6). What is the minimum fatal dose orally? 3 to 3.5 cc. per kilogram.

(7). Is there any skin hazard? Constant exposure makes the skin excessively dry, and long continued exposure

may render persons exposed susceptible to those dermatoses which are prone to arise in excessively dry skins.

Allen J. Fleming MD
J. H. Long

8/28/36
AF
JC

300032

# The Effect of Inertoen and Several Related Substances Upon the White Rat.

W. T. Read, Jr., M.D.

Inertoen, a mixture of trichlorobenzene, ethyl-tetra-penta-chlorobenzene and chlorinated diphenyl, was administered to white rats orally, by skin application, and by inhalation. In addition several animals were exposed to trichlorobenzene and ethyl tetra-penta-chlorobenzene separately.

Two rats were employed to study the effects of inhalation of a compound known as Pyranol, and two other animals were exposed to vapors of Westinghouse Transformer Oil.

## Inertoen

### Oral Administration.

Twenty-four white rats received single doses from 1.21 to 3.44 cc. per kilogram of body weight. Following treatment death occurred in 20 to 54 hours. One died immediately following treatment, the material having been partly introduced into the lungs.

### Gross Pathology.

Lungs: Most of the animals that died show congestion with or without edema and hemorrhage.

Liver: Ten rats have mottled gray or yellow livers. Three animals show a diffuse pallor with moderate yellowness.

Stomach: In 17 rats the stomach contains blood. Nine of these present grossly visible erosions of the pylorus.

Intestine: In the majority of the animals the fecal material contains old blood. In one animal killed 54 hours after treatment the lower third of the ileum contains bloody material. There is congestion of the mucosa. The lower colon and sigmoid also contain bloody material.

Microscopic Pathology.

Six rats are examined histologically.

Liver: Five show marked and rather extensive hyalin necrosis in the central part of the lobules. The liver cells about these areas present fatty degeneration of the cytoplasm. In two animals living 52 and 64 hours after treatment there are increased numbers of mitotic figures.

Stomach: Two animals present typical hemorrhagic erosions of the mucosa, another has hemorrhagic areas in the mucosa. The rest of the animals show post mortem change which partially masks preceding pathology.

The remainder of the viscera presents no changes that are thought to be of importance.

Eighteen rats were given a dose of 1.15 to 1.20 cc. per kilogram body weight. Six of these animals were killed 3 days after treatment. Six animals were killed 8 days after treatment Six animals were killed 11 days after treatment.

Gross Pathology.

Stomach: In 2 of the 6 animals killed after 3 days there is bloody material in the stomach.

Liver: In half of the animals killed after 3 days the liver are slightly pale yellow in color. Those killed after 8 days present slight enlargement of the liver with a questionable yellowish color.

Rats killed after 11 days present no important pathology.

Microscopic Pathology.

Liver: Exposure 3 days. The liver cells are large with numerous mitoses. Occasionally there is necrosis of cells immediately about the central veins.

Exposure 8 days. The liver cells are large. Those about the central veins are dark. Only occasional fatty degeneration is seen.

Exposure 11 days. All of the sections show slight swelling of liver cells with very occasional vacuolizati of liver cell cytoplasm.

None of the other viscera present pathology of importance to the experiment.

Repeated Oral Treatment.

Twenty-one rats received multiple treatments by mouth. The amount of a single dose varied from 0.40 to 1.75 cc. per kilogram body weight. The animals studied histologically were:

Group 1. 4 rats, 1.24-1.75 cc. per kilogram. Died after 2 daily treatments.

Group 2. 4 rats, 1.5-1.55 cc. per kilogram. Killed 4 days after 3 daily treatments.

Group 3. 5 rats, 0.4-0.48 cc. per kilogram. Killed 4 days after 3 daily treatments.

Gross Pathology.

Group 1. Stomach: All are dilated with food. In 2 there is some old blood present in the content.

Liver: All present some hyperemia. In 1 the surface has a mottled appearance.

Group 2. Stomach: One animal has a small amount of blood in the gastric content.

Liver: Both present moderate enlargement.

Group 3. Liver: The livers appear somewhat lighter than usual. In one there are large yellow areas.

Microscopic Pathology.

Group 1. Stomach: One rat has small inflammatory ulcers of the fore-stomach, another presents hemorrhagic erosions of the pylorus.

Liver: Two animals show swelling of liver cells with fatty degeneration of cells in the centr part of the liver lobules. Two animals prese central necrosis of liver cells with hyperpla of the endothelial cells in these areas. In of these animals numerous mitotic figures are found in liver cells.

Group 2. Liver: There is a generalized swelling of liver cells with some polychromasia. In one rat th is a fairly generalized vacuolar degeneration liver cells. The other presents vacuolar cha in the central part of the lobules.

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Group 3. Liver: There is a diffuse, mild swelling of liver cells with some unevenness in staining ability. Occasional liver cells present vacuolar changes in the cytoplasm.

Pathology of all of the animals is not severe.

Comment.

The pathology resulting from oral ingestion of Inerteen is fairly consistent and obvious.

Large doses which are lethal in one or two days cause a terminal passive congestion of viscera, extensive degeneration of the liver with necrosis about the central veins and local ulcerations of the gastric mucosa.

If sub-lethal doses are given and the animals sacrificed at different time intervals, the resulting pathology is:

3 days - gastric irritation, hepatic degeneration and active regeneration.

6 days - mild degenerative changes in the liver with foci of regenerated liver cells.

11 days - indefinite liver pathology.

In the group of animals given repeated oral treatments, those that died present gastric ulceration and hepatic degeneration similar to those receiving a single lethal dose.

Two rats surviving 5 successive treatments show similar changes but to a less severe degree.

The group of rats receiving 5 daily doses of Inerteen, the total of which amounted to lethal or sub-lethal level, presents mild to moderate degenerative changes in the liver. C0000566

Other visceral pathology has not been included because it is considered to be coincidental in character. It may be men-

Skin Application.

Seven rats received about 0.5 cc. of Inertec daily to the shaved skin of the back. This was protected by a glass ring and adhesive tape to prevent licking. One animal died after 11 treatments, the rest were killed after 18 applications.

Gross Pathology.

Liver: All of the livers are dark gray brown in color with mottling of the surface.

Skin: No gross change is observed except in the animal that died. Here the area of application is soft and necrotic in appearance. This animal also presents pulmonary congestion.

Microscopic Pathology.

The animal that died presents:

Liver: Fatty degeneration of liver cells with necrosis of those about the central veins.

Skin: Hyperkeratinization with blister formation and leucocytic infiltration. There are mild inflammatory changes in the underlying connective tissue.

In the six animals killed there is:

Liver: Mild swelling of the liver cells in all of the animals with occasional vacuolar degeneration in some of the liver cells.

Skin: There is hyperkeratinization in 4 rats with occasional blister formation. Hair follicles are increased in number in three and decreased in one. One animal also presents peri-folliculitis. One of the animals has a hyperplasia of the epithelium rather than hyperkeratinization. In 3 rats there is mild fibroblastic proliferation with some round cell infiltration in the underlying connective tissue.

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There is no question that skin application produces some absorption of the material. In one animal this was sufficient to produce death. Since there is injury to the skin the degree of absorption may well depend on the amount of dam[illegible]

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Inhalation.

Group 1. Two rats were given maximum concentrations of vapors of Inerteen (about 0.4 mg. per liter) with exposure twice daily for 20 minutes each. The animals were killed 8 days after 14 treatments.

Group 2. Two rats were given vapors containing about 0.4 mg. per liter. This was given twice daily for 35 treatments. One died, the other animal was killed.

Group 3. Two rats received two daily exposures for 20 minutes each of vapors containing about 0.4 mg. per liter. These animals were killed after 68 treatments.

Group 4. Ten rats received 8 hour exposures 5 days a week of vapors containing an average of 0.057 and 0.061 mg. per liter. These animals were killed after 20 treatments.

Gross Pathology.

The animal that died in Group 2 presents a coincidental infection.

In the rest of the animals there are tape worm infestations of the liver and two rats have pulmonary infections.

No pathology of importance is seen.

Microscopic Pathology.

Group 1. No striking pathology. One animal presents bronchitis.

Group 2. In the animal that died the bone marrow, liver and spleen suggest coincidental infection.

Group 3. The liver shows questionable swelling of cells. Fat stains are entirely negative for degenerative changes.

Group 4. Lung: Bronchitis or bronchopneumonia or both are found in 4 animals.
Spleen: Five rats present fairly marked erythrophagia.
Bone Marrow: In those animals that have pulmonar infections there is hyperplasia of cells of the granular series.

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## Comment

While the incidence of lung infections is high in the rats exposed to Inerteen by inhalation of vapors, it cannot be said that this is directly due to Inerteen. There is no definite liver damage and no other pathology that is of any significance.

## Summary

Oral administration of Inerteen may produce local gastric ulceration and hepatic degeneration particularly of the central part of the lobules. Generalised passive congestion is produced as a terminal event in lethal cases.

Following single oral treatments there is evidence of hepatic regeneration in about 2 to 3 days which becomes complete in about 10 days to 2 weeks.

Skin application results in injury to the skin and absorption of Inerteen, the amount of absorption presumably depending on the amount of cutaneous damage.

Inhalation of Inerteen produces no definite pathology of the viscera.

## Trichlorobenzene

Nine rats received daily oral treatments with trichlorobenzene, the individual dose varying from 0.36 to 0.59 cc. per kilogram body weight. Two rats died overnight after 1 treatment; two died 4 hours after the 2nd treatment; one died overnight after 3 treatments; one died 4 hours after 5 treatments;

CONFIDENTIAL

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two died overnight after 3 treatments; and one rat was killed 4 days after 3 treatments.

Gross Pathology.

In all animals that died there is passive congestion of the lungs and liver.

Stomach: In all animals the stomach is overdistended with food. In four there is some blood present in the gast content.

Liver: In addition to passive congestion two rats show an irregular yellow mottling.

Microscopic Pathology.

Liver: All sections reveal necrosis of liver cells about the central veins of the lobules, the liver cells in the outer part show vacuolar degeneration. If death occurred early hyperemia is also severe. When the animal died 3 or 4 days after the first treatment, endothelial hyperplasia is seen in the areas of necrotic liver cells. One animal presents increase in number of mitotic figures in liver cells.

Stomach: In one animal there is hemorrhagic erosion of the mucosa.

Ethyl-tetra-penta-chlorobenzene

Seven rats received daily doses from 0.62 to 0.94 cc. per kilogram body weight of ethyl-tetra-penta-chlorobenzene. One rat receiving the highest dose died after the 3rd treatment. Six rats were killed 4 days after 3 treatments.

Gross Pathology.

The animal that died reveals:

Liver: Moderate yellow mottling.

Stomach: Dilated with food.

Lungs: Congestion and hemorrhage.

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The six rats killed present questionable enlargement of kidney and liver.

Microscopic Pathology.

In the rat that died there is mild swelling of liver cells with fine vacuolar degeneration. Generalised passive congestion of lung, spleen, bone marrow and other viscera.

In the animals that were killed there is:

Liver: Slight swelling of liver cells with occasional vacuolar degeneration. The changes are extremely mild.

Comment

The hepatic changes following trichlorobenzene are identical with those seen after Inert.en. Ethyl-tetra-penta-chlorobenzene produced relatively little morphological change in the seven animals examined. The one animal that died presents mainly generalized visceral passive congestion with only mild degenerative changes in the liver.

Pyranol

Two rats were exposed to vapors from distillation of Pyranol. Exposure was given twice a day for 20 minutes. Forty-one treatments were given.

Gross Pathology.

No definite pathology is seen.

Microscopic Pathology.

In one animal there are increased numbers of mitotic figures in the liver. The liver, however, presents no definite cellular change.

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## Transformer Oil

Two rats were exposed to vapors of distilling Transformer Oil. This was given 20 minutes, twice daily for 44 treatments.

Gross Pathology.

No definite pathology is seen.

Microscopic Pathology.

Lung: In one animal several of the bronchioles contain macrophages filled with eosin staining granules or small refractile droplets.

No striking pathology is observed in either animal.

William T. Read Jr.

5.28.38
4*

EW

February 14, 1950

Dr. Louis W. Spolyar, Director
Division of Industrial Hygiene
Indiana State Board of Health
1098 West Michigan Street
Indianapolis 7, Indiana

Dear Dr. Spolyar:

I enclose an application bulletin on our Aroclors. On page 19, there is a summary of almost all our toxicology information on this compound.

If the case you refer to is in Brazil, Indiana, our company has had some contact with the problem. This particular installation used a temporary heat transfer system, and thus did not make the installation air tight. This is contrary to our expressed instructions when Aroclor is to be used at elevated temperatures. Upon hearing of the illness, one of our development engineers went to the plant and gave his recommendations, and then I called the plant physician to try to obtain some idea of what the illnesses were. As far as I could determine, two men suffered from gastrointestinal upset. I suspected the possibility that the Aroclor fumes might have caused liver damage, but was unable to obtain this information over the phone. I was also unable to contact the employee's physician.

The toxicology of Aroclors is somewhat confused. The experimental work was done by Dr. Drinker at Harvard about 12 years ago, and was done in connection with chlorinated naphthylene, chlorinated diphenyl, and chlorinated diphenyl high boiler. Both of these last two are Aroclors. In the particular work at Harvard, Dr. Drinker found that Aroclor 1263, which means diphenyl chlorinated to 68%, was of low toxicity. The confusion existed in his findings that Aroclor 1254, which is the diphenyl chlorinated to only 54%, was considerably more toxic on inhalation. We did not supply him with this material, and I was never convinced that some error might not have been made in the sample.

At any rate, we have advised protection against all Aroclor fumes when an elevated temperature is used. I will appreciate it if you will let me know the result of your investigation, if one is to be made.

Very truly yours

R. Emmet Kelly, M.D
Medical Director

RFK:rg

B CC: Mr. Paul Benignus
St. Louis

Kelley
EXHIBIT NO. 42
2-17-94 ICR

4453

900973

# STATE OF INDIANA

ADDRESS REPLY TO:
L. E. BUSKEY, M. D.
SECRETARY AND EXECUTIVE OFFICER
INDIANA STATE BOARD OF HEALTH
1330 WEST MICHIGAN STREET
INDIANAPOLIS 7, INDIANA

STATE BOARD OF HEALTH

February 28, 1950

Dr. R. Emmet Kelly
Medical Director
Monsanto Chemical Company
St. Louis 4, Missouri

Dear Doctor Kelly:

Thank you very much for your very informative letter of February 14, relative to the use of Aroclors in industry. The plant under question is the Brazil plant mentioned in your letter.

The chief complaints were irritation of the upper respiratory tract plus possible liver damage. At the time of our study Aroclor was circulated in galvanized pipes and most pipes were leaking. It was suggested that new pipes be installed and local exhaust employed.

Plant repiped with copper tubing and installed a local exhaust system over extruder rolls. With this improvement very little Aroclor vapors were escaping. This probably will solve their problem. Further the plant is considering building an enclosure around the extruder and rolls so that this equipment will be both enclosed and exhausted.

Thanks again for your help.

Very truly yours,

Louis W. Spolyar

L. W. Spolyar, M. D., Director
Division of Industrial Hygiene
Indiana State Board of Health

LWS:pm

PLAINTIFF'S EXHIBIT 8

PUBLIC HEALTH IS "THE ART AND SCIENCE OF PREVENTING DISEASE, PROLONGING LIFE AND PROMOTING PHYSICAL AND MENTAL EFFICIENCY THROUGH ORGANIZED COMMUNITY EFFORT." C.-E. A.

Tox - Aroclors

St. Louis

December 12, 1966

AROCLOR SWEDEN

A. Arpino - BRUSSELS
G. R. Buchanan - CBUCH
D.V.N. Hardy - LONDON
R. A. Steenrod - RSTEE

Mr. D. Wood
BRUSSELS

I do not believe that we can glibly accept Aroclors as a synonym for polychlorinated phenols that were discussed at the meeting of scientists at the Wenner-Gren Centre in Stockholm on November 27.

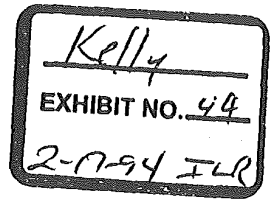
There are polychlorinated phenols which presumably could include derivitives from, or impurities in pentachlorophenol and, especially, 2,4-D and 2,4,5-T. These compounds would be much more liable to appear in salmon, pike, and sea eagles than any derived from Aroclors.

There are many chlorinated polyphenyls that can be formed during the manufacture of 2,4,5-T and probably pentachlorophenol, as well. Our only problem is whether or not we want to bring these facts up and have our herbicide program receive another black eye. This, I will have to leave to your judgment.

I think the question here is primarily an analytical problem. How can we find out what product Mr. Jensen is talking about? Can we compare these chromatographic peeks that Mr. Jensen is describing with anything found in Aroclors, pentachlorophenol, 2,4,5-T, etc? I admit I am out of my depth here but I think another compound is indicted rather than Aroclor.

R. Emmet Kelly, M. D.

REK/ln




NEV 023924

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Brussels, Belgium

1st December, 1966

Aroclor Sweden

Dr. gh

C. L. Reichers & St. Louis

THIS COPY FOR

A. Arpino - Brussels
Dr. Ernest Kelly - St. Louis
D.V.H. Hardy - London
R.A. Stenrod - St. Louis

Dear George,

I attach a copy of a letter received from Ole Palm in Stockholm. I have sent copies of this letter also to the appropriate departments within our own organisation. In consideration of the importance we are placing on development of the Swedish market for Aroclor over the next five years, we would be grateful if you could arrange for this information to be considered by the appropriate departments in St. Louis and their comments transmitted to us as soon as possible.

Based on the recommendations made by our medical departments we shall have to decide whether to arrange for publication of data in Sweden or not.

In relation to the specific problem mentioned of disposal of used materials, we would be interested to learn how this problem is handled in America. In the U.K. many companies have been burying material in drums, material in the drums having been absorbed into vermiculite or some similar porous material. Has any entirely safe method been developed for the disposal of waste Aroclor ?

Best wishes.

GB.

pp D. Med.

P.S. Thinking of the total quantities of Aroclor used in Sweden compared with the much larger volumes of Pentachlorophenol and Sodium Pentachlorophenate, for water treatment in the paper industry and mycelin control in the timber industry. Is it likely that the chlorinated phenols show similar chromatographic traces to the chlorinated bi-phenols?

Kelly
EXHIBIT NO. 45
2-17-94 ICR

184
STR 017390

cc: Mr. D.S. Cameron - ME

# RISING & STRAND

AKTIEBOLAG

OP/BO

TELEFON: 34 01 33 TELEGRAM: HOWARD TELEX: 1634
POSTGIRO: 3 44 43 BANKGIRO: 73-6446

STOCKHOLM 70
SVEAVÄGEN 67

November 28, 19[illegible]

Monsanto Europe
BRUSSELS 3
Belgium

For the attention of Mr. D. WOOD

Dear David,

re: ARCCLORS

As mentioned, there has been some publicity in Sweden concerning investigations made at the Institution of Analytical Chemistry at the Stockholm University. These have revealed that a group of products called Polychlorinated Bi-Phenols - PCB for short - accumulated in certain organs of animals. They are said to be related to DDT and equally poisonous.

The findings were discussed at a meeting of scientists at the Wenner-Gren Centre in Stockholm on November 22. Below please find a translation of an article in the Swedish daily paper "Dagens Nyheter":-

"It is found in salmon and in pike. It is found in sea eagle living on fish. It is found on the surface of the needles of the fir trees, that is in the air. It is found in the hair of a five months baby...

The scientists working with biocides have for a long time seen this something as unknown "peeks" on their gas chromatographs and at a meeting at the Wenner-Gren Center, Research Assistant Sören Jensen of the Institution for Analytical Chemistry at the Stockholm University could reveal the identity of these peeks. It has been found that they consist of a group of poisons, Polychlorinated Biphenols (for short PCB) which are closely related to, and equally poisonous as, DDT.

PCB is broken down considerably slower than DDT and gives rise to damage of liver and skin. PCB is not

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STR 017391

used as a herbicide. It is not manufactured in Sweden but is supposed to used by the industry to quite some extent. No special industry can so far be accused of being the source of contamination.

Research Asst. S. Jensen has tested 200 fishes and a number of birds. He has taken several samples of air and has reached the conclusion that PCB is equally common in Nature as chlorinated hydrocarbons of the type of DDT, DDE, and Lindane. Even fish in Laddjojaure in Laplard contain PCB. Mr. Jensen has also found that PCB does not appear in animals living on a vegetarian diet, such as the elk.

In the course of his work, Mr. Jensen has not found anything indicating that the source of contamination comes from agricultural additives. It is, however, obvious already now that PCB is most frequently found in organisms living in water or feeding from water animals. In all examined pikes PCB was found. In a sea eagle found dead outside Stockholm it was found that the liver contained 30 mg of mercury per kilo, 76 mg DDT and considerably more PCB. The exact figure has not yet been determined.

PCB is found in water and in air, and not only in the Swedish air, but also in e.g. London air. Mr. Jensen has not yet been in London for sampling but could identify the poison by studying a gas chromatogram of air published in a British technical journal.

Mr. Jensen har also examined the hair of his family and himself and has found PCB on all samples. Most PCB was found in the hair of his wife but most sensational was that the girl aged 5 months had more PCB in her hair than her brothers and sisters of 3 and 6 years. Probably the girl had got the poison via the mother's milk.

In the State Museum Mr. Jensen has examined the whole collection of sea eagles dating back to 1880. By testing it could be established that PCB was present only in birds from 1944 and thereafter while birds collected before 1944 were quite free from PCB.

The use of PCB in Sweden is not established in detail. According to American sources these types of products are used in the manufacture of a variety of heat-resistant materials. They are used for electrical insulation, for fire-proof heat transport in hydraulic oils, in lubricating oils used at high

temperature and pressure, in paints and as pigments in various plastics. PCB is not imported only as such. It is also part of several finished products. Nothing is known as to the way in which it reaches the water and the air. According to Mr. Jensen, products containing PCB should have this openly declared.

PCB is equally harmful whether absorbed via the skin, through the food, or by inhalation. In contact with the skin it can cause oedema. For DDT the highest permissible concentration in the air has been set at 0.5 - 1 mg/cu.metre. For PCB it has been mentioned to be 0.5 mg/cu.metre. Mr. Jensen will now try to get more complete analytical material. He hopes eventually to be able to disclose the source of the contamination and will also increase his cooperation with toxicologists and geneologists."

Another daily paper, Svenska Dagbladet, has a similar article. Here it is mentioned that Mr. Jensen, working under Laborator (Professor) Gunnar Widmark, has disclosed facts which will have far-reaching importance since the findings have proved a new source of pollution of the nature.

One of the participants at the meeting was Dr. A.V. Holden of Scotland, scientific contact man between the twelve O.E.C.D. countries, who has established coordinated analysis of chemicals used and found in nature.

I suppose there is no doubt that what has been termed Polychlorinated Biphenyls is equal to Aroclor. There is also no doubt that the published facts will cause considerable unrest in several quarters. We probably will have to have Aroclor registered with the Swedish Board of Poisonous Substances and the industry will have to be particularly careful in handling the material. The problem in some cases of course may be the disposal of used material. I understand that there hardly exists a convenient method of destroying Aroclor and that possibly burying unuseable material may be the only answer.

We shall be glad to hear from you soon.

Yours sincerely,

RISING & STRAND [illegible]
HENRY STRAND

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STR 017393

Monsanto

D.V.N. Hardy, London

12th January, 1967

Aroclor - Sweden

DVNH/ARW

10

P.G. Benignus, St. Louis
G.R. Buchanan, St. Louis
D.S. Cameron, Brussels
Dr. R. Emmet Kelly, St. Louis
G.R. Graham, New York
R.A. Steenrod, St. Louis
D. Wood, Brussels
J.A. Evans, London
R.A. Baxter, Ruabon

| FILE | |
|---|---|
| Destroy | |
| JTG | |
| WHH | ✓ |
| MNJ | ✓ |
| REK | ✓ |
| RAM | |
| EPW | ✓ |

JAN 16 1967

On 2nd January 1967 Mr. A. Richardson of Shell Chemicals' Tunstall Laboratory, Sittingbourne, Kent talked with me over the telephone concerning the Swedish Press report relating to the identification of "Polychlorinated Biphenols" as trace contaminant in sea birds, fish etc. Richardson has been working for some years on the similar problem with insecticides such as DDT, which are known to have wide distribution in trace quantities. He had already found that the chlorine-containing residue contained substances more stable than DDT. and just as Sören Jensen reports he has obtained spectrographic evidence that these are very similar if not identical with Aroclors. He has obtained samples of Aroclors 1242, 1254, 1262 and 5460 from us, and would now like to have small samples of any chemically pure Aroclor constituents which we may be able to supply. Milligram quantities would suffice for his purpose.

Mr. Richardson was quite sure that the compounds reported to be "polychlorinated biphenols" are really meant to be polychlorinated biphenyls and as support he has sent me a copy of the synopsis of a paper entitled "Pesticide Analysis: Presence of Polychlorinated Biphenyls at Residue Analysis of Biological Samples" by Sören Jensen and Gunner Widmark (photocopy attached).

I discussed with Richardson the soundness of Jensen's claims, and was assured that his work and findings are sound. Jensen is on the staff of the Institute of Analytical Chemistry, University of Stockholm. A note on the staff and work of the Institute is attached. From this you will see that Jensen is wholly concerned with the analysis of chlorinated pesticides and with the work of stations for routine analysis.

I would be glad if Dr. Baxter and Dr. Buchanan would arrange to send me milligram samples of any pure Aroclor constituents that may be available at Ruabon and St. Louis respectively.

Kelly
EXHIBIT NO. 46
2-17-94 ILR

D.V.N. HARDY

NEV 022115

732170

M : Brussels, Belgium
ME & LOCATION)

: 26th January, 1967

T : SWEDEN, AROCLOR

RENCE : DW:gb

: G. R. Buchanan - St. Louis

cc P. G. Benignus - St. Louis
D. S. Cameron - Brussels
Dr. D. V. N. Hardy - London
Dr. R. Emmet Kelly - St. Louis
R.A. Steenrod - St. Louis

3

We recently sent you a translation of a Swedish newspaper article referring to the identification in nature of polychlorinated biphenols. Because some of the uses claimed for the materials fell in line with the uses of our own chlorinated diphenyls, we made a point, during our recent visit, to Sweden, of visiting the research institute involved and discussing their particular programme of work.

To eliminate any earlier confusion that there may have been, I should like to emphasise that there is no doubt that the chemical which is the subject of the investigation and the news release, is chlorinated diphenyl i.e. Aroclor.

The company that supplied the mass spectrometer which was used in the research programme, in fact have recently put out a press release on this work. Although I am horrified by some of the headlines in this press release, it does basically describe the research programme carried out in Sweden, and describes in clear terms how chlorinated diphenyls were identified. I therefore, enclose a copy for your files.

Jensens only aim in life as an analytical chemist, was to identify the substances found in his research work on the occurrence of insecticides in nature. The unfortunate aspect of the situation is the comments which have been added to Jensens work. He showed what was present and unqualified people have made statements as to the possible effect of what he has found.

Summarising the publication position, there were original articles covering the Stockholm conference in the Swedish daily press, and these reports were picked up also by the Danish press. You will have seen from D.V.N. Hardy's memo of the 12th January that it has also been picked up by the Shell Chemicals Laboratory in Kent, U.K. Jensen also divulged that he had been contacted by the Swedish American press agency who intended to include information about this research work in their monthly review "The Swedish American Journal". There is additionally the press review issued by LKB Productor AB which I imagine to have been sent to a number of technical journals.

Kelly
EXHIBIT NO. 47
2-18-94 TLR

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732211

Effect in Sweden

This matter was raised with us by every capacitor manufacturer in Sweden that we visited. Fortunately there has not been too much adverse comment as yet from plant workers since they have not associated the polychlorinated biphenols mentioned in the article with Aroclor or Pyralene used in the Swedish factories. Jensen, however, stated that he had been approached personally by several workers associated with chlorinated diphenyls for non-electrical uses and these workers were quite worried as to the possible effect on their health.

Future Research

Arrangements are being made in Sweden for this work to be taken over by one of the medical institutes who hope to study the toxicology of the poloychlorinated diphenyl residues at the levels of concentration found by Jensen. Additionally a geographical survey is to be carried out to try and determine where the highest concentrations of residue are appearing and, if possible, to detect from this the method of escape so that more security precautions can be taken. We were asked by Jensen if it was possible for Monsanto to supply any samples of the pure isomers of chlorinated diphenyl since his work indicated at the moment that the lower chlorinated isomers are fairly easily metabolised, and the potentially more dangerous constituents are the more highly chlorinated members. If he can get hold of pure isomers, he would like to carry out some work on comparitive rates of metabolisation.

Jensen is forwarding me copies of his mass spectrographs and details of sample preparation so that we have all the details of his research work.

The point that I have made to Jensen is the need for care in any further publication of his work which is made. He accepts that the toxicology of chlorinated diphenyls should only be discussed with detailed information about exposure concentrations and exposure times and that generalised statements out of context can only arouse undue public concern. If any technical journal takes up the press release from the LKB Productor Company, there is little that Monsanto could, or should do in the way of publishing rebuttals. We do not want, personally as Monsanto to get too involved in this question. I am hopeful that we

might persuade Jensen himself to write a letter defining the true extent of his own research work and placing his results in their proper perspective. It would certainly be helpful in gaining his further support if we were able to make available to him any small quantities of pure isomers.

Since we are not alone in supplying polychlorinated diphenyls to the Scandinavian market, I have drawn this matter to the attention of the other askarel manufacturers in Europe.

As you will see from the press release one of the major points that is made is the difficulty in disposing of waste chlorinated diphenyls and again I must mention that constructive recommendations for the safe disposal of our materials would be most helpful.

D. Wood.

2/13/67

Gene Wilde - General Offices

February 13, 1967

R. M. Parks
W. F. Waychoff
C. R. Buchanan
→ IPG Research - R. E. Keller

EVIL PUBLICITY ON
CHLORINATED BIPHENYLS

R. Ernst Kelly, M. D.

Here is a brief summary of what we discussed during our meeting February 8 in your office.

We reviewed the information we have received so far on the adverse publicity in Europe on polychlorinated biphenyls. For the record, we have received the following published information.

Letter from Dunmar Widmark of the Institute of Analytical Chemistry, University of Stockholm, dated December 29, about his Mr. Jensen defining some of the unknowns in his analytical work as polychlorinated biphenyls. Attached to this letter was a report on the presence of polychlorinated biphenyls at residue analysis of biological samples written by Mr. Jensen and Dunmar Widmark.

Dave Wood of our Brussels office sent us the LKB press release of January 10, 1967, about the Swedish success in detecting the polychlorinated biphenyl.

A memo written by Dave Wood dated January 26 summarizing his visit with Jensen.

The January 30, 1967, issue of Chemical Engineering, page 52, makes comment about the work on polychlorinated biphenyls in Sweden. This was the first published information in the U. S.

December 15, 1966, the New Scientist Magazine, carried an article about a new chemical hazard, also reporting the work that had been done by the University of Stockholm.

Essentially, all of these reports covered the same items.

Due to the importance of the Aroclor products to the Organic Division, we decided on the following plan of action. Since all of the action required falls within the realm of your authority, you accepted the responsibility to follow through on the following:

Kelly
EXHIBIT NO. 48
2-18-94

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1. Prepare a statement or letter for use by Marketing with customers who inquire about this publicity.

2. You will talk to Don Forrestal on preparing a press release and hold it until such a time that we feel as though it should be released.

3. Give some thought to how we would approach this problem on a toxicological and pharmacological basis.

4. Talk to the NCR people after we define the person to contact.

5. Arrange for contact with Bayer and Prodelec.

Two questions that kept coming back to our minds during the meeting were that in all of the propaganda published, there has been nothing about the levels that have been found particularly in the air and no one has defined anything about what level would be considered harmful. Bob Keller brought out the fact that there must have been other products found in this complete analysis and he would be interested in knowing what other things were found.

All of these actions need immediate attention with the exception, of course, of the gathering of the toxicological and pharmacological information.

Please do let me know if there is anything I can do or any way I can help in getting these actions started and getting our information together so that we can make sure our Aroclor business is not affected by this evil publicity.

Cass Wilde

/bjk

From MONSANTO CHEMICAL COMPANY
At St. Louis, Mo.

Mr. H.K. Nason-M.O.
Mr. R.E. Soden-Nitr
Mr. L.C. Weger-Nitr

Date June 12, 1956

To Dr. R. Emmet Kelly
At Medical Dept.

Reference Nitro

Subject CHLORACNE CASES AT BADISCHEN ANILIN DUE TO TRICHLORPHENOL

Persons Present: Dr. H. Oettel
Dr. H. T. Hofmann
A. Palm (Ph.D.)
W. Soenksen (Plant Supt., part-time)

On the 17th of November, 1953, Badische was producing a batch of trichlorphenol from tetrachlorbenzene when the process exceeded contro pressure and temperatures similar to our incident at Nitro. No one wa injured at the time. Within one week, as clean-up was being carried out, the first cases of chloracne developed. Fifteen or sixteen cases (6 serious) developed within the next 1-2 months and additional cases showed up during the next 10-12 months until there was a total of 50-60 cases.

I did not see our most severe Nitro cases nor have I seen photographs of these cases. The photographs of the worst Badische cases show - horrible skin eruptions with nearly blister-like welts and some ulcera tion where infection ensued. Areas involved included the face, neck, arms, and upper half of the body. It is my impression that their severe cases were much worse than ours. In addition to the skin manifestations, their men reported all the additional symptoms as experiences in our workers, i.e, fatigue, vertigo, loss of libido, painful joints, etc.

Kelly EXHIBIT NO. 99 2-18-94

About ten days following the incident, and after initial clean-up, Dr. Oettel was asked to expose animals to the workroom atmosphere. Rabbits (in open wire cages) were placed in the operating area for 24-48 hours. There were no obvious symptoms which developed in the animals until one week after exposure - when they died. Autopsy showed liver necrosis. Oettel thought there might be virus infection or some other cause for death until he exposed additional animals in the department, others in cages suspended inside the "decontaminated" autoclave, and some in the adjacent department. All died within 1-2 weeks following exposure. Subsequently, animals placed in the cages which had previously been in the department died of liver necrosis.

A thorough systematic investigation has isolated impurities in the trichlorphenol process (or residues) which will cause the same effects in rabbits. Liver necrosis will develop in rabbits at the following doses of the indicated materials:

15-20 mg. of pentachlor naphthalene
1 mg. of chlorinated diphenyl oxide
0.1 mg. of residue from trichlorphenol distillation
0.01 mg. of residue fraction from trichlorphenol (above 230°C)

CONFIDENTIAL

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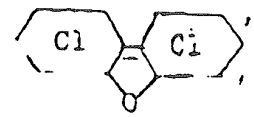
Dr. Oettel believes that the most potent chloracnogen is a compound somewhat similar to chlorinated diphenyl oxide, 

, but probably with additional oxygen atoms in the molecules. He has discussed with Don Irish at Dow who either reached the same conclusion independently or, in addition to the potential of chlorinated diphenyl oxide, influenced Oettel's thinking. Oettel believes, further, that this impurity can be a result in the production of any chlorinated phenol and is probably responsible for any chloracne which has been due allegedly to chloronaphthalenes, pentachlorophenol, chlorinated biphenyl etc.

Dr. Oettel has no faith in any animal skin tests for isolating chloracnogens. He was very interested in Kettering's work and was not aware of the publication referred to in Dr. Suskind's first report and which describes the cyclic skin development in new-born rats. (Reference: Parnell, J.P.: Postnatal Development and Functional Histology of the Sebaceous Glands in the Rat, Am. J. Anat., 85:41, 1949). He is convinced that the Bromsulphalein test reported in the attached report is significant. In this regard, Badische routinely uses this test on each batch of trichlorphenol which they now purchase from Bayer, and refuses to accept material which fails to pass the animal tests. (I also learned at Bayer that they have experienced chloracne during the production of trichlorphenol but "have now licked the problem" according to Dr. Hansen (chemist - Research Director At Elberfeld).

Badische has been able to reproduce in the laboratory the conditions which lead to the incident such as theirs and ours at Nitro and was quite surprised that we had not been able to do so. I was not given the Nitro process information, i.e., temperatures and pressures, but if I had had this information I am sure that I could have obtained Badische's. One of their chemists, a Dr. Palm, sat in on our discussions and was prepared to go into details. He did mention that "with 3 mols of trichlorphenol, methyl alcohol, and alkali, and a temperature of 180°C" the process gets out of control. His remarks are in quotes because he does not speak English and I'm not certain of his remarks.

Dr. Oettel would be very happy to receive samples of any of our materials for investigation. He would like particularly:

(1) Samples of any of the materials involved in our 1949 incident including tetrachlorbenzene, trichlorphenol, or Na salt and any residues from the autoclave or material cleaned from the equipment and structural members.

(2) Trichlorphenol (or Na salt) from regular production.

(3) Any samples from raw materials, intermediates, and final product which may have been involved in our cases which developed during our normal 2,4,5-T production in the years following the initial incident.

CONFIDENTIAL

C15580

He would be happy to exchange freely any and all information with us and Kettering. It was my suggestion that this be direct with Dr. Suskind to eliminate "third parties." He would routinely send carbons of any correspondence to us and we could request Dr. Suskind to do likewise. It is my opinion that this might save unnecessary duplication of effort and expense. In addition, Kettering might obtain valuable information to further their investigation involving human volunteers.

I left with Dr. Oettel copies of the following:

(1) The five (5) reports from the Industrial Hygiene Foundation which discuss their rabbit ear tests.

(2) Kettering's report - "Clinical and Environmental Survey at Nitro, 1953."

(3) Kettering's report - "Environmental Survey Carried Out In Building 30, Monsanto Chemical Company at Nitro, February 2, 1956."

We reviewed thoroughly Mr. Weger's excellent "History of Chloracne" which he sent to me with covering memo dated May 15, 1956, and which included descriptions of Kettering's reports on their human and animal research. I did not give Dr. Oettel a copy of this account because I did not have permission to do so. As a result of my visit, it may be desirable to edit this report somewhat and add further process information before forwarding this to Badische.

Any specific recommendations are implied in the above narrative account of my visit to Badische and must be based on an agreement that full and complete exchange of information with Badische is desirable and possible.

EPW

Elmer P. Wheeler

EPW:dh

Attachment

C15581

*This document has been retyped for clarity.*

*From* MONSANTO CHEMICAL COMPANY
*Att* St. Louis, Mo.

Mr. H.K. Nason-M.O.
Mr. R.E. Soden-Nitro
Mr. L.C. Weger-Nitro

*Date* June 12, 1956

*To* Dr. R. Emmet Kelly *Reference*

*At* Medical Dept. *Subject* CHLORACNE CASES AT BADISCHEN ANILIN DUE TO TRICHLORPHENOL

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EXHIBIT NO. 150 2-18-94

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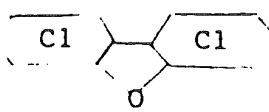
Dr. Oettel believes that the most potent chloracnogen is a compound somewhat similar to chlorinated diphenyl oxide,  but,



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