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

MONS 077396

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LAB 106

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

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12/1/54

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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. Eng. News 25, 2977 (1947).

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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.

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 a Garlock 901 asbestos gasket or 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 over-all 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 raining 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

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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 1

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
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.

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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.

Due to the relatively high viscosity of some of the Aroclors at low temperatures, it is 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 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 in the case where 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

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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 coefficient is assumed to be too low to utilize the 100% capacity of the boiler. A value of 1500 for UA with an average ΔT of 134°F. indicates that the useable steam is 202,000 BTU/hr. or 226 lbs./hr. steam at 80 psig.

In the second case the ΔT is lower and the entire output of the boiler is useable. Table II sums up the approximate time calculated to heat Aroclor 1254.

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°	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 producting 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

*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.

(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. 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 bypassed. All connections must be replaced as received. Adequate care should be taken in preparing and sealing the car for return shipment.

*It is essential that the displacement air used for unloading be dried thoroughly by some dehumidifying unit such as soda lime, activated alumina or similar

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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 -10°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.

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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 dlimates, 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 O-P-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 sufaces

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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 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 dehumidifying 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. 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 weatherproofing. Another type of insulation which may be used instead of the glass is 85% Magnesia-Wool which should be covered also with the weatherproofing 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

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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.

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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, 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 gauge 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

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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 Co. 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 "Varec," 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 accommodate the horizontal type tank.

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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 attached 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 Corp.
   (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.

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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 temperature 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."

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At the plants of several electrical manufacturers using Aroclor dielectrics, the practice is to 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 more than 0.10 parts per million. Also, moisture may not exceed 30 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.

Drum packaging is made with new and carefully inspected drums, only. These drums are lined with specially selected baked phenolic coatings. An example is NESCO No. 3 lining offered by the National Enameling and Stamping Company, Long Island, New York. Contents of 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.

2.) Monsanto Methods used for sampling tankcars differ somewhat from the ASTM procedure. The modified techniques are

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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 5½ inches inside diameter and 6 inches deep to hold the five pint bottle.

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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.

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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.

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 and moisture. For transformer use dielectric strength, resistivity, moisture, and free 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 aborbent power of various types of diatomaceous earth with respect to removing moisture, impurities and additives such as stabilizers or scavengers from the dielectric

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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.

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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 tests of Aroclor mixtures are:

1. Dielectric Strength
2. Resistivity

In both cases a significant chemical test is corrosion chlorides.

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

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dielectric 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."

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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)."

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.

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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. Adjustment of Controls on Electrical Apparatus

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 @ 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 100KC.

e. Turn "FREQ. VERNIER" to 80.

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

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

h. Turn "HORIZONTAL GAIN" to about 20.

i. Turn "SYNCHRONIZING" 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.

II. 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:

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

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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 uuf.)

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.

III. 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.

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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 fifteen 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)

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

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 uuf 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).

IV. 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. 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.

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 consits 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 grey 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 O position. Failure to comply with these requirements will prevent any action when the red button is depressed.

The test cup: Transformers, Voltmeters, and Accessories

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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. Rated @ 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 ic connected to the secondary of a 2-1/2KVA powerstat variable auto transformer supplied by the Superior Electric Company.

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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.

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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 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:

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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.

CAUTION: It is important to avoid touching the electrodes with the finger or with portion of the tissue paper which has been in contact with hands.

Rinse the electrodes and cup with dry lead-free gasoline, Stoddard Solvent (or dry, waterwhite Kerosene) until they are entirely clean. Care

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should be taken not to touch the electrodes or the inside of the cup after cleaning so as to avoid possible contamination.

4. METHOD NO. 11,607, "RESISTIVITY".

a. Apparatus:

General Radio Company Megohm Bridge Type 544-B. This is a combination of Wheatstone bridge and vacuum tube voltmeter for indicating null. The direct measurement of resistance up to 1,000,000 megohms is made possible by the use of a vacuum tube detector which absorbs negligible amount of power.

The voltage applied to the unknown resistor is held approximately constant, regardless of the value of the unknown resistance. This condition is necessary to measure resistance properly.

The accuracy of the instrument in the range encountered in the measurement of Aroclor resistivity, 100 to 1000 megohm is $\pm$ 6%.

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.

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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 outside electrode has inside diameter of 3" and 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 191/9.254 or 752 where average area is 191 sq. cm.

Also K = 36 π x 10" x C (in farads with air as dielectric) or 11.29 x C (inuuf. 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

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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 one minute.

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.

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13) Read after one minute.

Calculation:

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

Report the result in units of 10^9 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 apparatus. With the control knob in the charge and operate position full voltage of the bridge (500 volts) is applied to the and low terminals and through the test leads to the electrodes. Do not attempt to handle the electrodes or the test leads unless the control knob is in the

"CHECK" position. Possible penalty for failure to observe this precaution -- Painfull 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 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

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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.

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

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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

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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 of 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. (This may be obtained from reserve samples of benzol which have been analyzed for Depts. 223 and 233.)

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. (This solution is prepared by the Service Section).

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 ($\pm$ 0.05 g.) into one of the flasks a 75.0 $\pm$ 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 $\pm$ 0.002 for acid numbers below

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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 laboraoory 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 Burette, 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 Avenue, 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. =

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

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 1488-1467	100 ml.	200 ml.
Pyranol 1481	100 ml.	200 ml.
Pyranol 1495	100 ml.	200 ml.
All Aroclors	110 ml.	190 ml.

e. Procedure, "Visual End Point".

1) Place 100-300 ml. of dry solvent mixture (c) in a dry 500 ml. ggs. 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).

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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 40% of Elec. Grade Trichlorobenzene 0.125% of Tin Tetraphenyl
Transformer Pyranol 1470*	45% of Aroclor 1260 55% of Elec. Grade Trichloro-Tetrachlorobenzene Mixture 0.125% of Tin Tetraphenyl

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

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Capacitor Pyranol 1481	75% of Aroclor 1254 25% of Elec. Grade Trichlorobenzene

Detailed properties of all of these products are given as follows:

AROCLOR 1242

PROPERTY	TYPICAL
Visc. @ 37.8°C. (ASTM D88)	82 - 92 seconds Saybolt Universal
Specific Gravity @ 25/15.5°C (ASTM D287)	1.381 - 1.392
Color, APHA	100 max.
Condition	Clear
Acidity, mg. KOH/g.	0.01 max.
Pour Pt., °C. (ASTM D97)	-14 or lower
Inorganic Chlorides, ppm.	0.10 max.
Refractive Index @ 25° C.	1.6245 - 1.6265
Distillation Range (ASTM D20) Corrected for stem and barometric pressure	10% 325°C. min. 90% 366°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 150 max. Acidity, mg.KOH/g. 0.01 max. Inorg.Chlorides,ppm 0.10 max. 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)	4.7 - 4.9
Flash Point Cleve. OpenCup*	160°C., min.
Fire Point °C.*	None to boiling point
Sulfates (ASTM-D117-31)*	None
Fixed chlorine content (Carius)*	41.5 - 42.5%
Specific Heat @ 25°C. *	0.29
Evaporation @ 100°C for 6 hrs.*	0.4% max.
Dielectric Strength (KV) (ASTM D877) *	35 Min.

*Not determined unless by special request.

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AROCLOR 1248

PROPERTY	TYPICAL
Visc. @ 54.4° C.(ASTM D-88)	73-80, sec. Saybolt Universal
Spec. Gravity @ 65/15.5°C. (ASTM D-287)	1.404 - 1.414
Color, APHA	100 max.
Condition	Clear
Acidity, mg. KOH/g.	0.01 max.
Pour Point °C. (ASTM D-97)	-7
Refractive Index @ 20°C.	1.630 - 1.631
Distillation Range (ASTM D-20)	343 - 373°C.
Water Content, ppm.	35
Resistivity 100°C. 500 volts D.C. @ 0.1" gap	500 x 10^9 ohm-cm, min.
Dielectric Constant, 100°C. 1000 cycle	4.6
Dielectric Strength 25°C *	4.6
Flash Point, (C.O.C.) *	193° C.
Fixed Chlorine (Carius)*	47.5 - 48.5%
Specific Heat @ 25° C.*	0.27

*Not determined unless by special request.

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AROCLOR 1254

PROPERTY	TYPICAL
Visc. @ 98.9°C. (ASTM D88)	44 - 48 secs. Saybolt Universal
Specific Gravity @ 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.	0.10 max.
Refractive Index @ 25°C.	1.6370 - 1.6390
Distillation Range (ASTM D20), Corrected for stem and Barometric pressure	10% 366 - 378°C 50% 372 - 383°C. 90% 383 - 396°C.
Corrosion	After heating with aluminum for 6 hours @ 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 specifications:
	Color, APHA 150 max. Acidity, mg.KOH/g. 0.01 max. Free Chlorides, ppm. 0.10 max. Condition Clear
Water Content, ppm.	35 max.
Resistivity 100°C., 500 v.D.C. @ 0.1" gap	500 x 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 @ 100°C. for 6 hrs.*	0.4% max.
Stability *	There shall be no liberation of chlorine or chlorides when the material is heated @ 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 @ 25°C.*	0.26

*Not determined unless by special request.

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AROCLOR 1260

PROPERTY	TYPICAL
Visc. @ 98.9°C.(ASTM D88)	72 - 78 seconds Saybolt Universal
Specific Gravity @ 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.	0.10 max.
Refractive Index, 25°C.	1.6455 - 1.6470
Distillation Range (ASTM D20) Corrected for stem and barometric pressure.	10% 385 - 398°C 50% 390 - 404°C. 90% 400 - 420°C.
Corrosion	After heating with aluminum for 6 hrs. @ 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. Free Chlorides, ppm. 0.10 max. Acidity, mg.KOH/g. 0.01 max. Condition Clear
Water content, ppm.	35 max.
Resistivity, 100°C. 500 volts @ 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. @ 1000 cycles*	3.6 - 3.8
Burn Pt. (ASTM D92)*	Higher than 350°C.
Sulfates (ASTM D117-31)*	None
Fixed chlorine content (Carius)*	50 ± 0.5%
Evaporation @ 100°C. for 6 hrs.*	0.2% max.
Stability *	There shall be no liberation of chlorine or chlorides when the material is heated @ 100°C. in a glass vessel in contact with air for periods of at least one month.
Specific Heat @ 25°C.*	0.23

*Not determined unless by special request.

PYRANOL 1481

PROPERTIES	TYPICAL
Viscosity @ 37.8°C.	70 - 82 secs. Saybolt Universal
Spec. Gravity @ 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 @ 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 @ 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

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PYRANOL 1467

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.	0.01 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)	1st drop - 200°C. min.
Corrected for stem and barometric pressure	Below 270°C. - 40% max.
	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 Specifications:
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 @ 25°C.)	Less than 1.0% Total combustible gases including carbon monoxide, hydrogen and volatile hydrocarbons.

*Not determined unless by special request.

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PYRANOL 1470

PROPERTIES	TYPICAL
Visc. @ 37.8°C. (ASTM D88)	41-45 Sec. Saybolt Universal
Spec. Gravity @ 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 @ 25°C.	1.6075 - 1.6085
Distillation Range (ASTM D20)	
Corrected for stem and barometric pressure	
First drop	210°C., min.
35%	238 - 256°C.
55%	275 - 345°C.
65%	380 - 400°C.
95%	390 - 415°C.
Corrosion	After heating with aluminum for 6 hrs. @ 200-220°C., the aluminum must not be corroded either on visual or weight inspection and the Pyranol should meet the following specifications:
Color, APHA	200 max.
Acidity, mg.KOH/g.	0.01 max.
Inorg. Chlorides, ppm.	5 max.
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
Burn Point, (ASTM D92)*	None up to Boiling Point
Fixed Chlorine*	60.5 ± 0.5
Arc Formed Gases* (Oxygen Free Liquid @ 25°C.)	Less than 1.0% Total combustible gases including carbon monoxide, hydrogen and volatile hydrocarbons.
Electrical Stability*	After heating for 96 hrs. @ 100°C in a closed container, the resistivity should not decrease more than 10%.

*Not determined unless by special request.

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CHAPTER 8

EARTH TREATMENT OF AROCLOR IN THE ELECTRICAL INDUSTRY PRIOR TO USE.

Aroclors and their mixtures supplied to the electrical industry must conform with most strict requirements specified by the industry as shown in Chapter 7. Since these products are sensitive to contamination from traces of impurities, they require careful handling in the industry when sampling or storing or using the materials to impregnate capacitors or fill transformers.

Common contaminants to be avoided include moisture, ionizable impurities, metallic impurities, such as rust or corrosion products from storage and handling equipment, and contaminants such as oils (mineral oils), lubricating oils or greases commonly used in equipment or other processes in the electrical industry.

Care must be taken to avoid contamination from contact with improper gaskets or packing materials, including natural and synthetic rubber and most plastic exclusive of certain silicones and Teflon; rosin solder fluxes, etc. may also introduce traces of deleterious impurities. The dielectrics are also susceptible to the harmful action of ultra-violet light and, therefore, exposure to direct sunlight should be avoided.

The adverse effect of ionizable impurities is especially pronounced in the low viscosity Aroclors such as Aroclor 1242

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or Aroclor mixtures such as Pyranol 1481. In the case of more viscous fluids, such as Aroclor 1254, the ionizable impurities are not so free to move about in the fluid and, consequently, the more viscous fluids can be handled with less care than required for the thinner fluids.

This is especially pertinent in capacitor manufacturing. When using the thinner type Aroclors to make capacitors that offer superior low temperature operation, the following types of trace contamination affecting the electrical properties of the dielectric have been experienced. Occasional droplets of perspiration from operators, when winding the capacitor cores, have fallen onto the paper causing contamination in the finished units. Small amounts of Glyptal resin and similar sealing compounds used to seal tiny leaks in the vacuum impregnating equipment or storage tanks have caused contamination. Also, small amounts of mineral oil entering the system have caused similar trouble. A small droplet of any of these materials in one or several liters of the thinner type fluids results in noticeable contamination, whereas it may not have as much adverse effect on the more viscous dielectrics.

To be certain that the fluids are free from traces of contamination, it is standard practice to treat them with conditioned diatomaceous earth and then to filter immediately before use. Treatment with this earth by removing trace contaminants results in "up-grading" the electrical values, e.g.,

the volume-resistivity may be brought up considerably beyond the specification minimum or the normal values of the dielectric as received. Likewise, improvement in power factor may be accomplished.

The earth treatment recommended for use by the electrical manufacturers is qualitatively the same as used in the production of the Aroclors and their preparation for shipment to the electrical industry. Treatment of the Aroclors with earth by the electrical manufacturers is a step required to assure that traces of impurity or contamination that may have been introduced during storage or handling in the electrical industry have been removed and that maximum electrical values have been attained immediately before the dielectric is introduced into capacitors or transformers.

It is impractical for the manufacturer to furnish these dielectrics to the customer at the maximum attainable electrical qualities because even with careful packaging, shipping, sampling and handling in the electrical industry, these fluids may easily pick up traces of contaminants from opened tankcars, drums, pipe line, pumps, etc. However, as supplied according to the specifications, the fluids are readily "up-graded" by the earth treatment.

The type of diatomaceous earth used is known as Fuller's Earth, supplied by the Floridin Earth Company, Warren, Pennsylvania, or the Attapulgus Division, Mineral & Chemicals Corp. of America, 210 West Washington Square, Philadelphia 5, Pa.,

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or their equivalent. Usually a minus 200 mesh size, regular volatile, is used and the quality should be specified as for use by the electrical industry. (Code 73122)

The earth as received needs to be conditioned and activated, after which it should be stored only a minimum length of time (about a day at the most) and in hermetically sealed containers, prior to use.

It is desirable that the earth be used immediately after it has been activated.

Activation should be done by heating the earth contained in stainless steel shallow trays for four hours at 400° C. in a muffle furnace. If a muffle furnace is not available or the capacity by this method may not be great enough, the earth can be activated by heating at least twelve hours in an electrically heated oven at 250° C. Another method used is to pan dry and activate by heating at 100° C. and using a strong vacuum, about 6 mm. of mercury. This latter method is used in connection with transformer work and the former methods are usually used for capacitor work.

In the place of pan drying, rotary driers may be used but their action should not be so severe as to break the earth particles to the extent of producing powder which is difficult to handle in the subsequent filtering operations.

It is the consensus that the earth should not be heated above 400° C. as this may cause collapse of the particles.

The amount of earth used is usually 0.1% to 0.3% based on the weight of the fluid. Larger amounts of earth can be used if the fluid is composed of only Aroclor or a mixture of Aroclor and chlorinated benzene or other pour point depressants. However, larger amounts of highly active earth, in the range of 2% or 3%, would be expected to selectively adsorb scavengers or stabilizers from the fluids containing these additives.

This is especially of concern in handling the transformer fluids which usually contain scavengers such as tin tetraphenyl. For transformer work it is suggested that the earth be conditioned and mildly activated by heating it at 100° C. under vacuum, about 6 mm. of mercury. Not more than about 0.1% of earth based on the total weight of the transformer fluid should be used.

For treatment with earth, the fluid is put into a suitable tank fitted with an agitator, heating coil, and a cover.

The proper amount of freshly conditioned earth is added and the mixture is agitated thoroughly and heated for about four hours.

The more viscous dielectrics such as Aroclors 1248 and 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 are heated at about 50° to 60° C. (122° to 140°F.).

After about four hours contact the material is filtered through a Sparkler or Sweetland or a comparable filter press previously fitted with filter paper liners such as supplied by

Carl Schleicher and Schuel Co., Inc., 116 West 14th Street, New York, N. Y. The paper is usually 25 mils thick and must be dried at 100° C. to remove moisture, prior to use in the filter press. The filtered dielectric material is then ready for impregnating capacitors or filling transformers.

In some cases, especially for AC capacitor work where the dielectric does not require addition of stabilizers and accordingly there is no danger of removing such additives by repeated earth treatment, good practice is to use continuous earth treating, circulation and filtration with constant reading of the resistivity of the filtered dielectric.

In Chapters 1 and 2 selection of proper materials of construction for the tankcars and storage tanks was discussed. It is equally important to select proper materials of construction for the processing tanks used in the capacitor and transformer industries and also for the impregnating chambers used in making capacitors.

It is preferable that this equipment be made of stainless steel or aluminum, or, if it is of steel construction, the interior should be either zinc-tin metallized or aluminum lined.

While iron or steel equipment is not regarded as desirable for handling the fluids, this type of construction is used in some of the plants. Here the possibilities of rusting or corrosion cannot be overlooked. If the equipment is kept free from water and if a film of clean dielectric adheres to the

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surface of the metal, e.g., when the tanks are empty, then satisfactory operation can be experienced.

However, a number of factors must be kept in mind and to cite an example, reference is made to the impregnation of capacitors by the chamber 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 about 130° C. under efficient vacuum, 100 microns or less. If the chamber is not made of the preferred materials of construction, slight corrosion may occur and the film of fluid on the interior surface of the tank may be contaminated and in turn introduce traces of impurities into the treated and clean dielectric subsequently introduced into the chamber for impregnating the capacitors. In fact, because of this possibility of contamination, in some operations the capacitors are conditioned and dried preparatory to impregnation in a separate oven or chamber and when thoroughly dried they are then transferred into a second chamber used for impregnation.

In the case of relatively large size capacitors, such as power factor correction units, a manifold pipe system 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.

When conditioned and dry, the dielectric is introduced into the unit through the manifold. Care must be taken that moist air or contaminants do not collect in the branch through which the dielectric is introduced.

In addition to removing moisture, another purpose of the heat and vacuum conditioning treatment given the capacitor units prior to impregnation is to remove traces of impurities such as residual solvent from the metal cleaning operations and from solder flux, etc.

The fluid is introduced hot into the evacuated capacitor units. The more viscous Aroclors, such as 1248 and 1254, are usually impregnated at 85° to 130° C. The less viscous and more volatile dielectrics such as Aroclor 1242 and mixtures of Aroclor with chlorinated benzene may be introduced best at lower temperatures -- about 50° C. optimum, 80° maximum.

Table IV indicates desirable minimum resistivity values of the dielectric materials when earth treated and ready for capacitor impregnation and similar values of the material following the process.

TABLE IV

Dielectric	Volume Resistivity Ohm-cm at 100° C. and 500 volts DC.	
	Prior to Impregnation	After Impregnation
Aroclor 1254	$2{,}500 \times 10^9$	500×10^9
Aroclor 1242	$1{,}500 \times 10^9$	500×10^9
Pyranol 1481	600×10^9	400×10^9

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In the case of new and freshly filled transformers using Aroclor dielectric mixtures, it seems reasonable that the power factor of a sample of the fluid drawn from the transformer should be in the range of about 5 to 12 percent, measured at 60 cycles and 100° C.

With field service of the transformer the power factor value is expected to increase somewhat. However, such a power factor increase alone does not seem to have harmful effect on the operation of the transformer.

A more usual measurement used for transformer fluid is volume-resistivity. This value of the new dielectric prior to filling the transformer should be about 500×10^9 ohm-cm at 1,000 cycles and 100° C. The similar value of the fluid taken from a new transformer should be at least, about, 50×10^9 ohm-cm and no lower than 25×10^9 ohm-cm.

With long field service volume-resistivity values decrease somewhat but seem to level off around 10×10^9 ohm-cm, with satisfactory transformer operation. However, if the volume-resistivity of the fluid falls below 10×10^9 ohm-cm, the matter should be looked into.

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CHAPTER 9

DERMATOLOGY AND TOXICOLOGY

Skin patch tests using Aroclor 1254 (biphenyl Chlorinated to the extent of 54% by weight) applied to gauze and placed in contact with the skin showed no primary irritancy or sensitization. The tests were conducted under competent medical supervision and the standard procedure recommended by Drs. Louis Schwartz and Samuel M. Peck, Reprint No. 2552, Public Health Reports, Vol. 59, No. 19 (April 28, 1944) was used.

If Aroclors are spilled on the skin, the skin should be washed in the usual manner with soap solutions. If accidental burns occur from contact with hot Aroclors, the burn should be treated the same as any ordinary burn. Aroclor adhering to the burned area need not be removed immediately unless treatment of the burn demands it, in which case use soap and water or repeated washings with a vegetable oil.

At ordinary temperatures Aroclors have not presented industrial toxicological problems.

If Aroclors are used at elevated temperatures in open systems, methods must be designed to exhaust any vapors arising. Experimental work on animals indicates that the maximum safe concentrations of vapors in workrooms is in the range of 0.5 to 1.0 milligram per cubic meter of air. This applies to all of the liquid Aroclors.

Laboratory technique merely requires keeping the hands

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free of the liquid and handling it under a well ventilated hood.

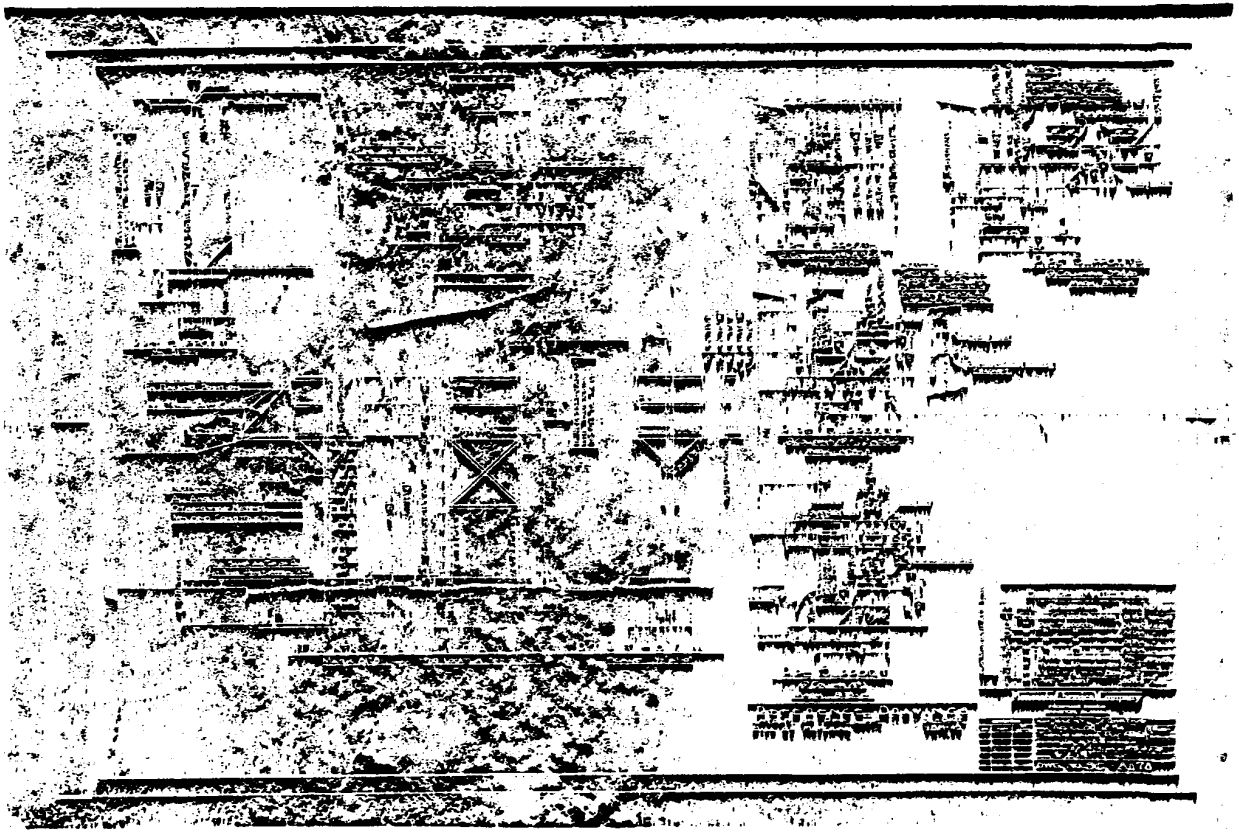
Localized or spot ventilation together with general workroom exhaust is recommended for plant operations.

When sampling tank cars, canvas gloves and safety glasses or goggles should be worn. No special clothing is required, but the worker's garments should be laundered at least weekly and changed in case Aroclor or Aroclor mixtures is spilled on the clothes accidentally.

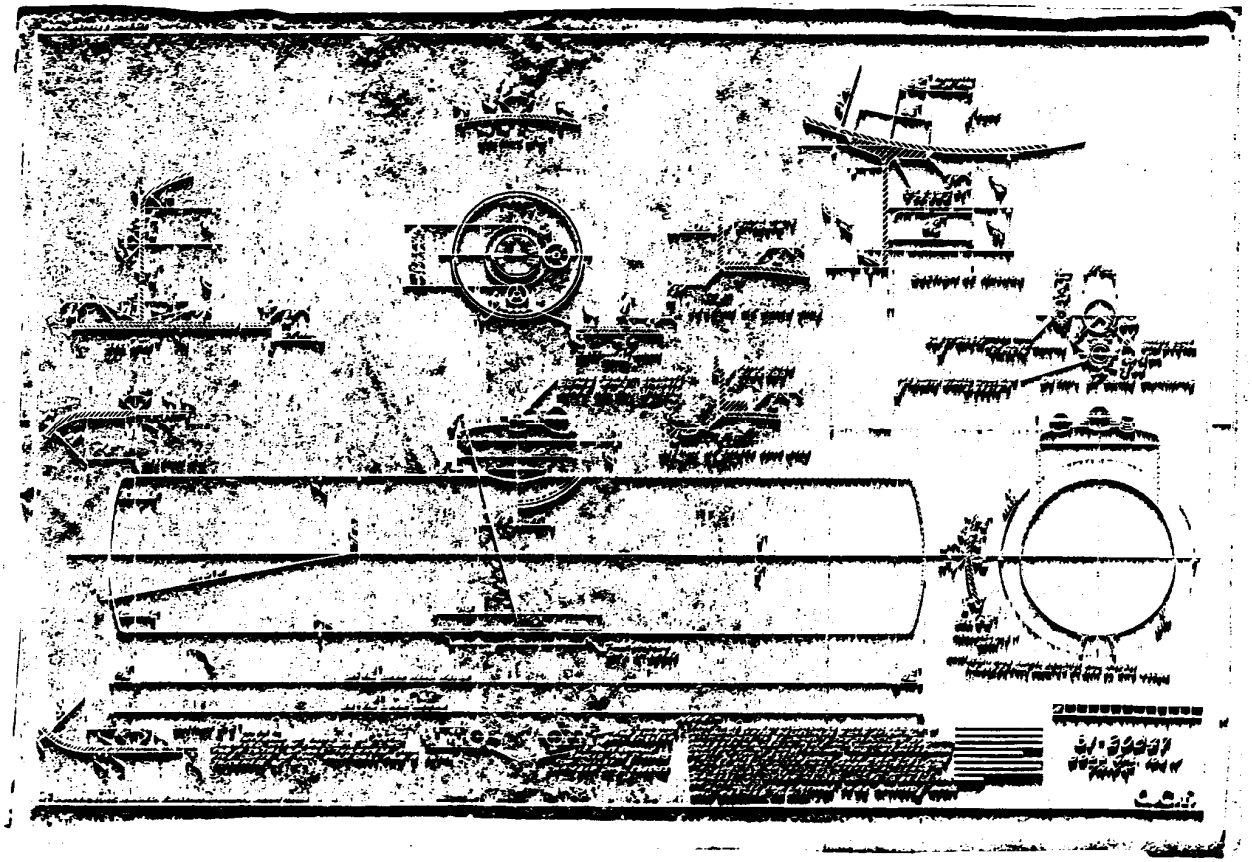
If workmen are exposed to Aroclor vapors at relatively high levels, as may be the case when opening a heated capacitor impregnating chamber, a respirator should be worn during these short intervals.

The many years of satisfactory and safe use of Aroclors and their mixtures with chlorinated benzenes in 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 a simple matter and in line with good "housekeeping" and personal cleanliness to exercize the suggested and required precautions in all cases.

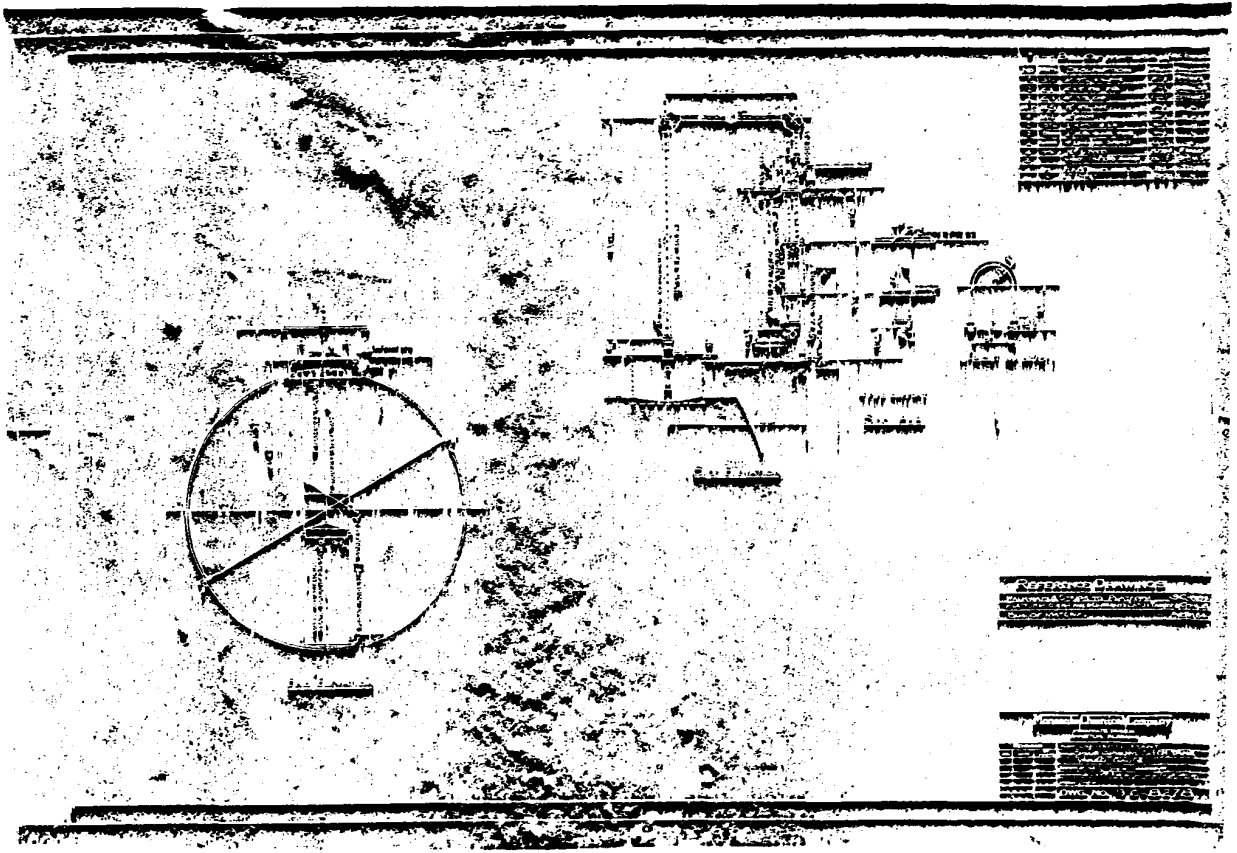
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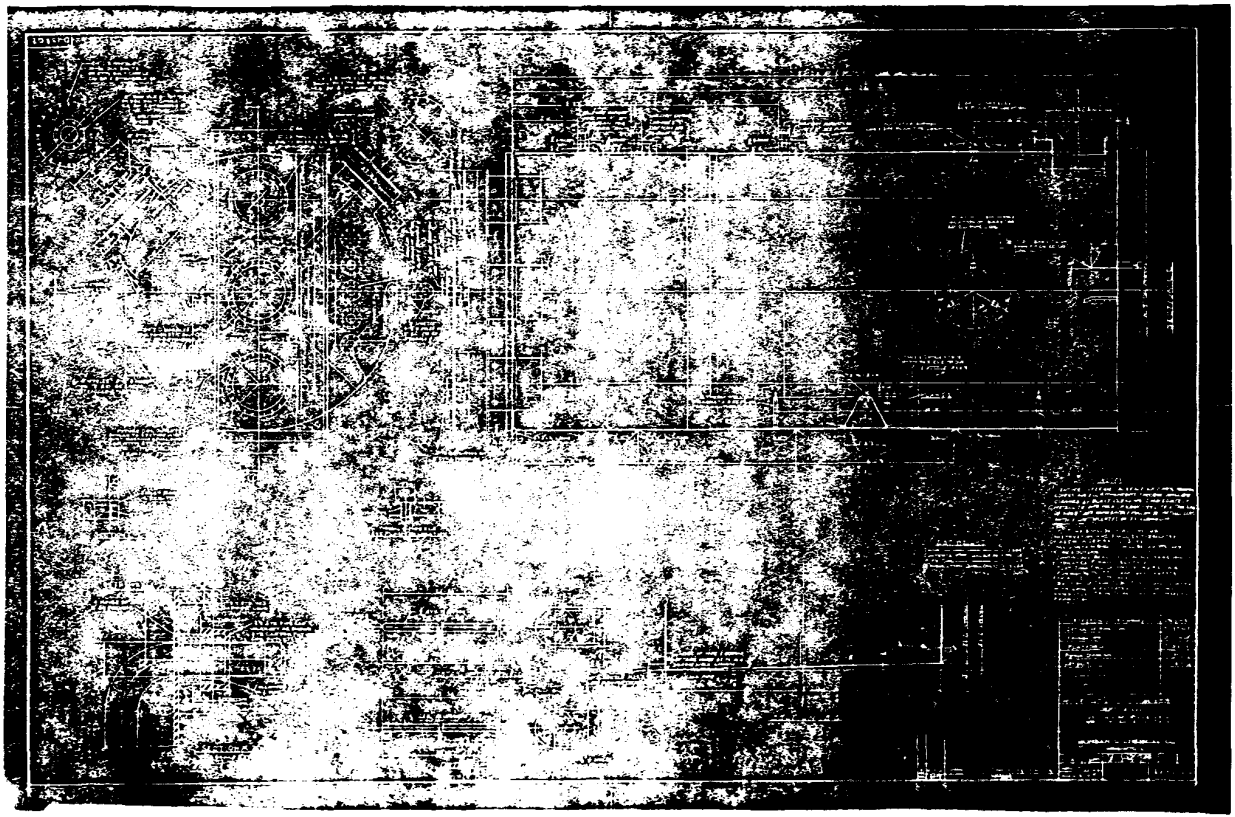
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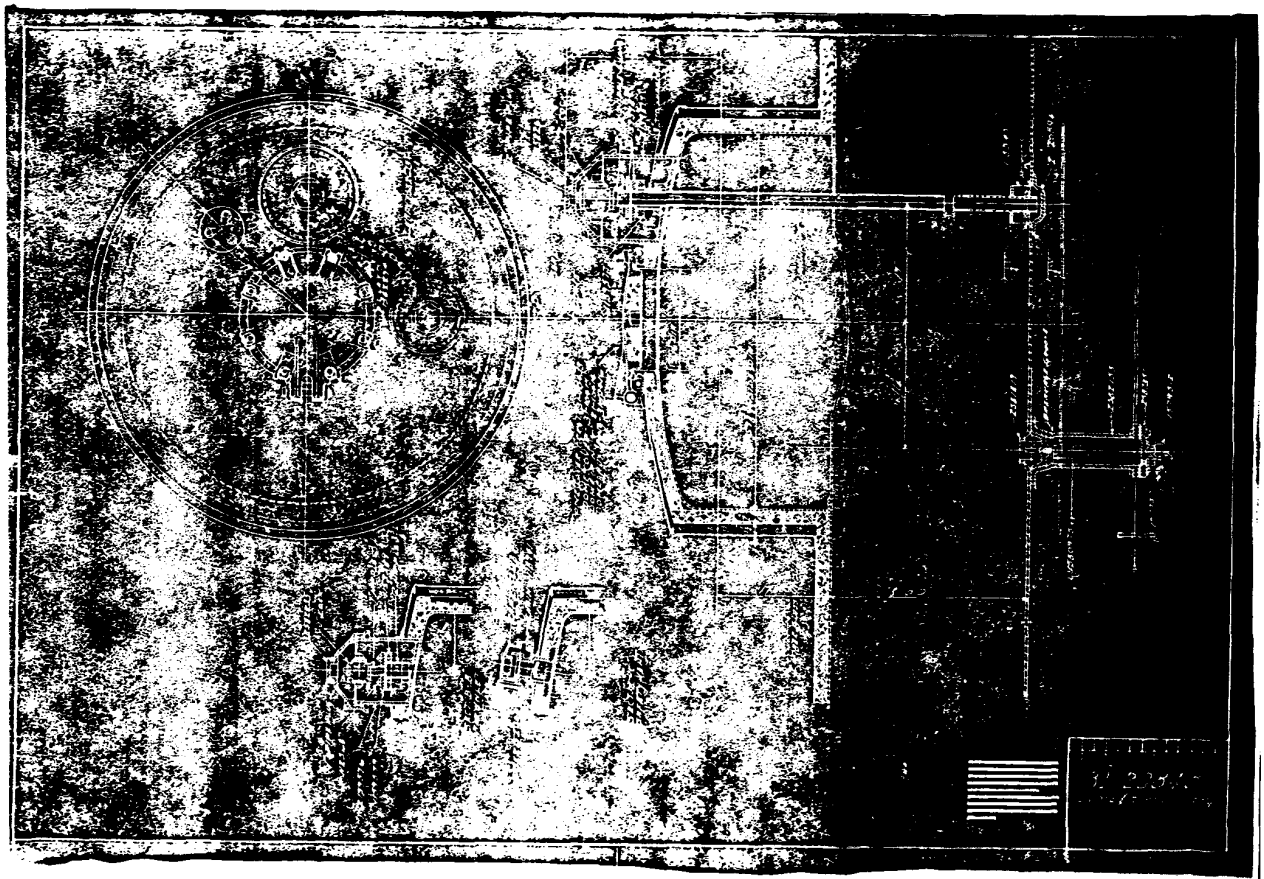
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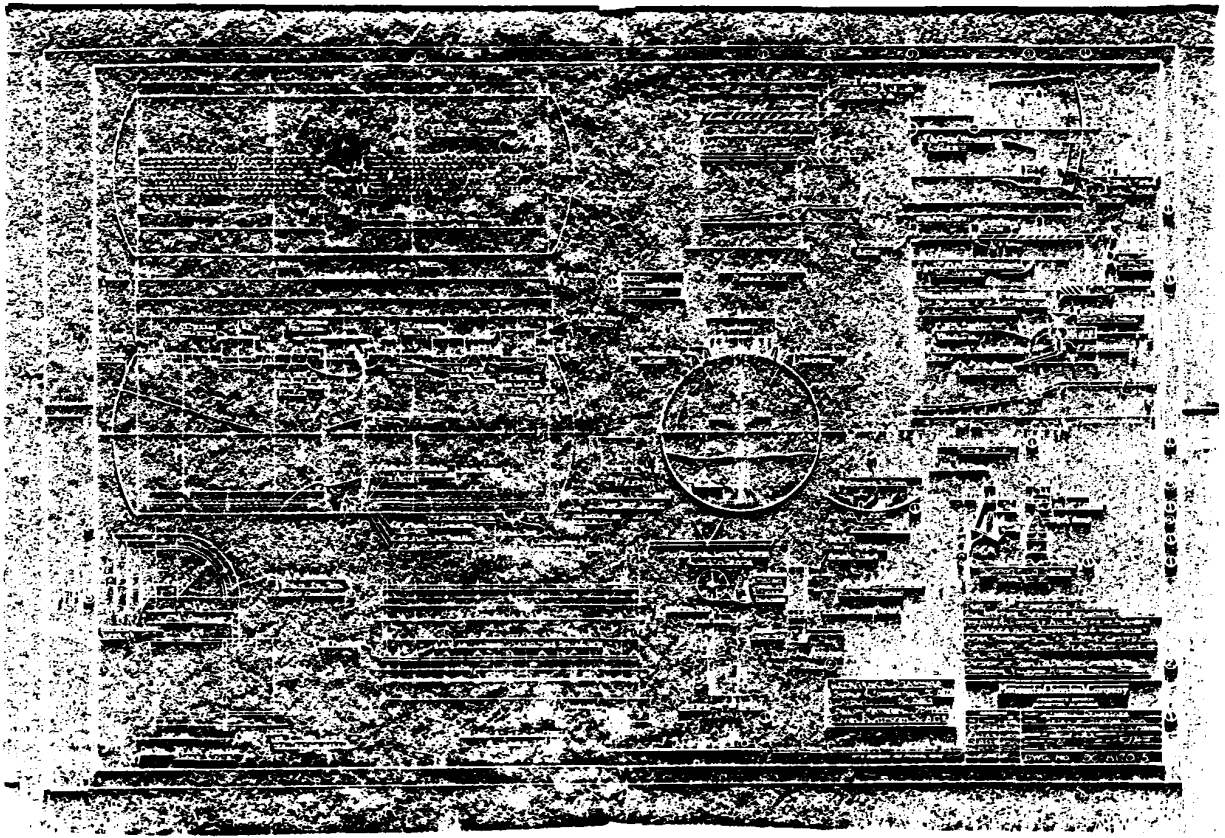
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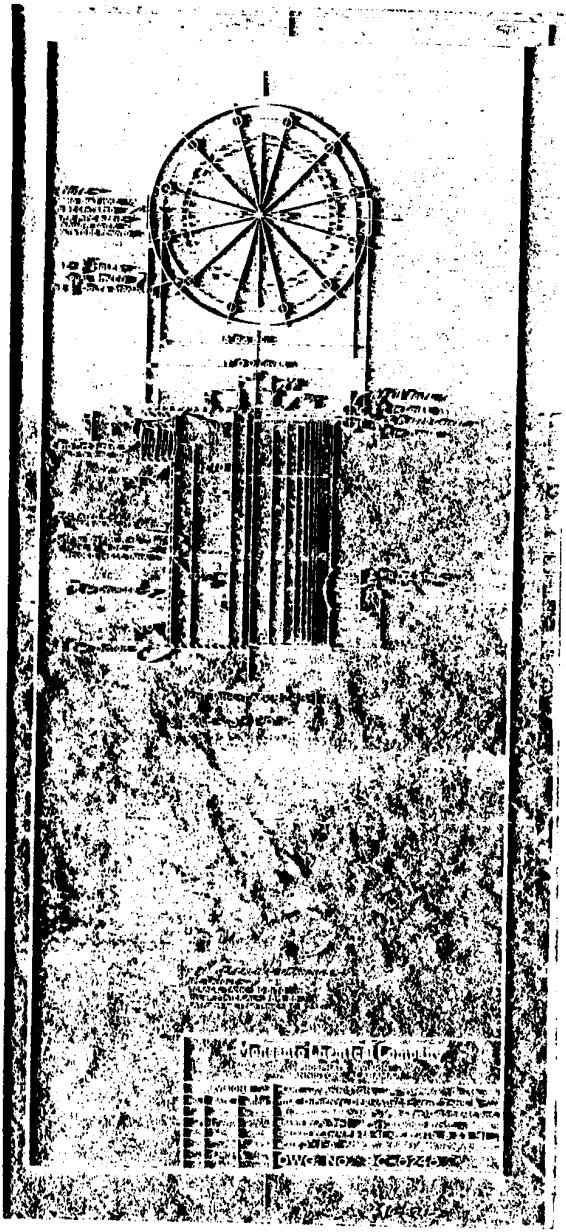
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