PUB. NO. CP–P1–1973
PUB. NO. TR–P6–1973
JANUARY 25, 1973

NEMA

OFFICIAL STANDARDS PROPOSAL

PROPOSED AMERICAN NATIONAL STANDARD GUIDELINES FOR HANDLING AND DISPOSAL OF CAPACITOR– AND TRANSFORMER–GRADE ASKARELS CONTAINING POLYCHLORINATED BIPHENYLS, C107.1– ( )

DSW 010204

NATIONAL ELECTRICAL MANUFACTURERS ASSOCIATION 155 EAST 44TH STREET, NEW YORK, N. Y. 10017

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This NEMA Official Standards Proposal CP-P1-1973 (TR-P6-1973) will soon be submitted for review and approval to the C107 Committee on Use and Disposal of Askarel and Askarel Soaked Materials of the American National Standards Institute.

If you will fill out and return the postal card shown above, we will be glad to inform you of the final disposition of this NEMA Official Standards Proposal. There is no extra charge for this service. It would be appreciated if you will return one card from each copy of this publication which is in your possession. Each card should be filled out in full with your complete mailing address.

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PROPOSED AMERICAN NATIONAL STANDARD GUIDELINES FOR HANDLING AND DISPOSAL OF CAPACITOR- AND TRANSFORMER-GRADE ASKARELS CONTAINING POLYCHLORINATED BIPHENYLS, C107.1- ( )

(NEMA Official Standards Proposal 1-25-1973.)

This proposal was prepared by NEMA, in cooperation with the Capacitor and Transformer Working Groups of the American National Standards Institute Committee, C107, on Use and Disposal of Askarel and Askarel Soaked Materials.

These proposed standards will be periodically reviewed for any revisions necessary to keep them up to date with advancing technology. Proposed or recommended revisions should be submitted to:

Technical Director
Manager, Engineering and Safety Regulations Department
National Electrical Manufacturers Association
155 East 44th Street
New York, New York 10017

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GUIDELINES FOR HANDLING AND DISPOSAL OF CAPACITOR- AND TRANSFORMER-GRADE ASKARELS CONTAINING POLYCHLORINATED BIPHENYLS

SECTION 1

PREFACE

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OFFICIAL STANDARDS PROPOSAL

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SECTION 1
PREFACE

I. GENERAL

Askarels consisting of or containing polychlorinated bipheynls (PCB's) have been used in many applications for over forty years, but only recently was evidence discovered that PCB's are widely dispersed in the environment. Systematic investigations of the biological effects of PCB's have been undertaken within the past few years to establish the effects of specific formulations upon specific species. Some studies have shown that PCB's may be an environmental contaminant. Simultaneously, significant steps have been taken by U. S. Industry to limit further releases of PCB's to the environment.

PCB's have been used in three broad types of applications for the past forty years, as follows: (a) "open ended" applications, for example, in paints, specialty inks, paper coatings, plastics, etc., (b) "nominally closed" applications, for example, as the working fluid in hydraulic or heat transfer systems; and (c) "closed electrical system" applications, specifically as the insulating fluid in certain types of transformers and capacitors. The Monsanto Company is the sole U. S. producer of PCB's. It has discontinued supplying the material for all type (a) and (b) applications.

Evaluations of the alternatives and risk/benefits involved in the continued use of PCB's in closed electrical systems are summarized in the following.

II. BENEFITS

Askarel-filled transformers do not burn or sustain fire under conditions of internal electrical arcing.

Askarel-filled power and industrial capacitors are significantly smaller, more reliable, more durable, and safer than oil-filled capacitors. As a result, askarels have supplanted mineral oils in more than 90 percent of the power and industrial capacitors made today. Over the past few decades most of the equipment that incorporates such capacitors has been designed to take particular advantage of the size, safety and reliability benefits of askarel capacitors (e.g., many types are today less than 14 percent of the size of equivalent oil capacitors and have a life expectancy of ten to more than twenty years).

Various federal, state and local codes, therefore, require their continued use in or adjacent to public, commercial and industrial buildings which locations present the greatest potential danger to life and property.

III. RISKS

In the United States, medical records over a nearly forty-year period show that the only adverse health effects experienced by U. S. workers exposed to askarels, either during the manufacture of these liquids or of electrical equipment containing these liquids, have been limited to occasional cases of non-chronic chloracne or other temporary skin lesions or irritations.

Askarel-filled transformers and capacitors are delivered to customers as sealed units from which there is no escape of askarel under normal operation. While certain types of equipment failures can permit loss of some askarel to the environ-

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ment, transformer failures are limited to approximately 0.02 percent of the units in service per year. With respect to capacitors, such losses are limited to approximately 0.02 percent of the askarel put into service per year. In addition, limited amounts of PCB's can get into the environment during the manufacture, delivery, improper use, maintenance, repair and disposal of transformers and capacitors.

Specific control measures have been instituted by individual manufacturers and are supplemented and strengthened by national standards and procedures such as this Guide which provides information to prevent the inadvertent loss of PCB's to the environment at all stages from initial askarel manufacture through ultimate disposal.

IV. ALTERNATIVES

For technical and local and national code reasons, it would be impossible to replace most askarel-filled transformers now in service with oil-filled units of equivalent ratings, without major construction changes that would be required to compensate for the fire resistance of the askarel-filled units. For certain applications and locations, dry-type transformers may replace askarel-filled transformers.

For new installations, while many of the limitations noted above would still apply, building and installation design provisions could be made to accommodate the use of oil-filled, open dry-type, or sealed dry-type transformers provided necessary technical, code, physical size, and cost considerations are properly evaluated.

The principal alternative to askarels for capacitors is mineral oil, but such replacement would return capacitor technology to its pre-1932 level and would necessitate the redesign and replacement of such widely used equipment as fluorescent light fixtures and racks for power and induction heating capacitors, which could not now accommodate the increased size of oil capacitors while maintaining their present ratings.

The cost of askarel liquids is about five to ten times more than mineral oil. Thus, long before there were any environmental concerns about PCB's, there was a strong economic incentive to find other less expensive insulating liquids with the desirable characteristics of askarels. Since the 1930's at least ten major chemical or electrical companies have invested large amounts of time and money in this search, all with no success. While potential substitutes that are most costly than askarels have also received some consideration (e.g., fluorinated liquids), little is known about either their electrical performance or possible ill effects upon the environment. There are today no fluids that can be used as a direct replacement for askarels.

V. INTERDEPARTMENTAL TASK FORCE ON PCB'S

An in-depth study of PCB's has recently been completed by five Executive Branch Departments of the federal government. This Interdepartmental Task Force on PCB's issued their report entitled, "Polychlorinated Biphenyls and the Environment," in May, 1972. * See Section 1 Page 3 for the conclusion quoted from page 4 of this report.

* Distributed by the National Technical Information Service, U.S. Department of Commerce, Springfield, Virginia 22151. Price $6.00.

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"The use of PCB's should not be banned entirely. Their continued use for transformers and capacitors in the near future is considered necessary because of the significantly increased risk of fire and explosion and the disruption of electrical service which would result from a ban on PCB use. Also, continued use of PCB's in transformers and capacitors presents a minimal risk of environmental contamination. The Monsanto Company, the sole domestic producer, has reported voluntarily eliminating its distribution of PCB's to all except manufacturers of electrical transformers and capacitors."

Reference should be made to the Interdepartmental Task Force Report for additional information and conclusions.

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GUIDELINES FOR HANDLING AND DISPOSAL OF CAPACITOR- AND TRANSFORMER-GRADE ASKARELS CONTAINING POLYCHLORINATED BIPHENYLS

SECTION 2

CAPACITOR GUIDELINES

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SECTION 2
CAPACITOR GUIDELINES

I. INTRODUCTION

The environmental effects of askarels are under in-depth study by governmental and other agencies. Askarels have been considered relatively harmless to humans based on about forty years of safe industrial usage. There has been no known instance of human injury when they were used under the normally accepted precautions and conditions of handling in both manufacturing and user applications.

Traces of askarels are being found in the environment and in fish and birdlife. The long-term genetic and ecological effects are not yet completely understood. For these reasons, care should be taken to contain askarels and minimize their entry into the environment.

There are two general classes of askarels used by the electrical industry. The higher chlorinated grades are the more persistent in nature. Because of their high degree of nonflammability, they are used in transformers where personnel safety is of paramount importance.

Capacitor-grade askarel has a lower degree of chlorination (composed primarily of the 3-chlorine isomers of biphenyl) and a higher degree of biodegradability. Generally, it has not been found in animal life. It is used in capacitors where the extreme degree of nonflammability required in transformers is of less importance.

Although capacitor- and transformer-grade askarels both contain members of the PCB family, they do differ in composition, degree of biodegradability, persistence in nature, electrical stability, chemical stability, and degree of nonflammability (both are recognized as nonflammable). It is for these reasons that this section of the Guidelines is intended to apply to capacitor-grade askarel.

II. CAPACITOR-GRADE ASKAREL

In September of 1971, a new grade of capacitor impregnant, Aroclor 1016, was made available to the industry. This new grade contains a typical concentration of 0.4 percent by weight of the higher boiling homologs of the chlorinated biphenyls. * Aroclor 1016 replaces Aroclor 1242 which previously was the major capacitor impregnant and contained around 7 percent of the higher boiling homologs (the more persistent in nature). This 0.4 percent level of the higher boiling homologs should be the maximum concentration acceptable in any capacitor impregnant. Aroclor 1242 and 1254, previously used as impregnants, do not meet this requirement and should no longer be used in capacitors designed and manufactured for alternating-current applications.

Aroclor 1016 has the same UL nonflammability rating as Aroclor 1242. Its typical properties are given in Table 1. (See Section 2 Page 2.)

III. PLANT HOUSEKEEPING AND EMPLOYEE SAFETY

The following procedures and limits are intended to be minimum requirements to be met by manufacturers and users of capacitors containing askarel. Handling, control and disposal procedures are given, together with exposure limits and indicated antidotes and cleanup procedures.

A. Material

Askarel for use in capacitors should consist of homologs and isomers of chlorinated biphenyl with the concentation of the higher boiling homologs at about 0.4 percent. * Aroclor 1016 is considered to be the standard impregnant meeting these requirements.

* Monsanto Method T-02302.

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Table 1
Typical Properties of Aroclor 1016

Property and Method	Typical Values
Color (APHA)	40, max.
Condition	Clear
Specific Gravity at 25/15.5°C (ASTM D 1810)	1.362-1.372
Acidity (mg. KOH/g.) (ASTM D 974, D 664)	0.010 max.
Moisture	35 ppm, max.
Refractive Index at 25°C (ASTM D 1817)	1.6215-1.6235
Inorganic (Free) Chlorides (ASTM D 1821)	0.05 ppm, max.
Pour Point (ASTM D 97)	-14°C or lower
Dielectric Constant (ASTM D 924) (1000 Hz at 100°C)	4.70-4.90
Resistivity (ASTM D 1169) (500 VDC at 100°C, 0.1-inch Gap.)	500 x 10^9 ohm-cm, min.
Hydrolysis Stability Test (ASTM D 1820) (As Chlorides)	0.5 ppm, max.
Thermal Stability Test (ASTM D 1936) (As Chlorides)	0.4 ppm, max.
Distillation Range (ASTM D 20 Corrected)	
10 percent Distilled by Weight	323°C, min.
90 percent Distilled by Weight	356°C, max.
Higher Boiling Homologs (Monsanto Method T-02302)	0.4 percent, max.
Sulfates (ASTM D 117)	None
Dielectric Strength at 25°C (ASTM D 877)	35 kV, min.
Flash Point, Cleveland Open Cup (ASTM D 92)	338°F, min.
Fire Point	None to Boiling Point
Corrosion Test (6 hours at 210°C with bright aluminum foil)	
Change in Weight of Aluminum	0.0 percent
Viscosity at 100°F (SUS) (ASTM D 88)	71-81
Specific Heat at 25°C	0.30
Coefficient of Expansion (ASTM D 1903)	0.00068 cc/cc/°C
Fixed Chlorine (Carius)	41.3 ± 0.5 percent
Power Factor at 100°C 60 Hz (Monsanto Method T-2273)	
Bulk	1 percent, max.
Drums	4 percent, max.

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Commonly used solvents include benzene, kerosene, acetone, trichloroethane, trichloroethylene and perchloroethylene. Typical vapor pressure data for Aroclor 1016 are:

0°C	=	0.001 mm
25°C	=	0.006 mm
150°C	=	4.3 mm
200°C	=	29.0 mm

At 25°C and 760 mm Hg pressure, saturated air contains approximately 0.09 mg/liter.

[1 mg/liter = 90.0 ppm (v/v)]

[1 ppm (v/v) = 0.011 mg/liter]

B. Bulk Fluid Shipment, Receiving, and Transfer

Shipment of askarel from point of manufacture to point of receiving should be done in closed containers such as rail tank cars, truck tanks, marine or barge tanks, or sealed drums. Containers should be labeled as to contents and carry a label cautioning against loss of fluid to the open environment. Containers used to transport askarel should not be used for storage or to transport other material without being completely cleaned of all traces of askarel. (Cleaning procedures must take cognizance of precautions against excessive exposure and of the need for proper disposal of contaminated cleansing solvents and materials as set forth in Part V of this Section.) Transfer from shipping containers to processing systems should be through closed piping or tubing with appropriate valves, pumps, etc. Provision should be made for trapping and disposing of fluid lost by leakage or spills from the transfer system and from the storage containers.

Drums to be retired from use should be cleaned before crushing, delivery to scrap dealers, or other disposal. Contaminated cleaning fluids and materials should be disposed of as indicated in Part V.

C. General Safety Precautions

Although it is generally accepted that exposure to capacitor-grade askarel is not hazardous provided simple precautions are taken, exposure should still be avoided.

1. Vapors— The odor of askarel is noticeable well below the maximum air concentrations considered safe. Up to 1.0 milligram per cubic meter of air has been determined to be the maximum safe level of exposure during an 8-hour work day (Par. G, reference 8). The procedures for performing the necessary analyses are contained in Section 5, "A Tentative Procedure for the Determination of Airborne-Polychlorinated Biphenyls."

   Breathing vapor or fumes from heated askarel should be avoided. Provisions should be made for adequate ventilation and regulation of manufacturing operations to avoid open exposure to askarel (especially at 55°C or higher). The gases produced when askarel is decomposed by very high temperatures (such as that of an electric arc) in the presence of air or organic insulating materials contain a high percentage of hydrogen chloride, and small percentages of carbon dioxide, carbon monoxide and oxygen. Minute concentrations of this combination of gases are very unpleasant and irritating, thus giving ample warning of their presence. If exposure to high concentrations of askarel is necessary under emergency conditions, an approved gas mask or self-contained breathing apparatus should be worn. Such exposure should be under the surveillance of other personnel capable of rescue in case of an accident. If the odor of askarel is detected by the person wearing protective equipment, he should immediately go into fresh air. All gas masks, respirators and replacement parts should have Bureau of Mines approval and be maintained on a regular schedule in accordance with the manufacturer's recommendation.

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2. Liquid—Unlike insulating oil, there is virtually no fire hazard in handling askarel. A limited solvent action (similar to that for paint thinner) on the fats and oils of the skin with prolonged contact may lead to drying and chapping of the skin. As with insulating oil, some people are allergic to askarel and continued exposure may result in skin irritation. Both the liquid and vapor are moderately irritating to eye tissue.

Operating procedures should be such as to minimize or eliminate contact with askarels. Use of eye protection is recommended. The use of porous gloves which can absorb and retain askarels is to be avoided. Barrier creams* or resistant gloves† should be used if contact is unavoidable. Use of enclosed transfer and handling equipment, processing equipment, and mechanical washers reduces direct contact.

Medicinal washes or mild detergents followed by the application of cold cream will reduce the irritation resulting from the contact of an open cut or abrasion with askarel.

Safety glasses with side shields or a face shield should be worn when handling askarels. If liquid askarel contacts the eyes, the eyes should be irrigated immediately with large quantities of running water for 15 minutes and then examined by a physician. (A drop of castor oil has been found to reduce irritation.)

Persons developing a skin irritation or respiratory tract irritation while working with askarels should be placed under the supervision of a physician.

Ingestion or swallowing of askarels is not generally regarded as a problem of the industry. Should accidental ingestion occur, a physician should be consulted. Hands should be washed with warm water and soap before eating, drinking, smoking or using toilet facilities.

D. Manufacturing Housekeeping

Manufacturing equipment and operating procedures should safeguard against loss of askarels to the environment through proper containment and disposal procedures.

Enclosed systems of sealed piping, properly gasketed joints, valves, containers, and processing chambers should be used for any portion of the operation where askarel temperatures may exceed 55°C. Enclosure should preferably extend to all other portions of the system insofar as practicable.

Containment provisions should be established around all askarel processing areas to ensure against inadvertent loss to sewer systems by spillage, leakage, or other uncontrolled conditions or events.

Spills of askarel should be removed promptly by means of absorptive material, such as sawdust, or trapped and removed by pumping or other suitable means.

* For example, PLY No. 9 Gel (Milburn Co., Detroit, Michigan), or Kerodex #71 (Ayers Laboratories, P. O. Box 8236, Church Street Station, New York, N. Y. 10049), or the equivalent.

† Edmont, Solvit 5-352 (Mersick of Bridgeport, 22 Cross Street, Bridgeport, Conn. 06601), or the equivalent.

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Waste fluids containing askarel not suitable for reconditioning or reuse should be collected (by means of traps, drip pans, trays, etc.) from the various parts of the manufacturing and processing area (including washers or other cleaning devices). Disposal should be made in accordance with Part V of this section.

Wiper rags, clothing and other extraneous materials saturated with askarels should be collected within the containment area for properly controlled laundering or disposal (see Part V of this section).

E. Disposal of Askarel Wastes

Methods for disposal of liquids and saturated solids generated by the manufacturing operation should be in accordance with those outlined in Part V of this section and should include (but not be limited to) the following wastes:

1. Contaminated liquid askarel which is unsuitable for reclaiming as a dielectric fluid.
2. Liquid askarel from solvent operations or water/detergent type washers.
3. Saturated earth or other absorbent media from filtering operations.
4. Saturated sawdust or other absorptive materials from spills.
5. Saturated filters from vapor control devices and other filters.
6. Saturated wastes (paper, rags, etc.).
7. Saturated, spent gasket materials.
8. Askarel-contaminated vacuum pump oils.
9. Askarel-contaminated stream jet vacuum system condensates.

F. Miscellaneous Procedures

1. Spills by leakage from finished capacitors should be cleaned up promptly by means of absorbent media which should then be moved to containers provided for that purpose within the containment area and later disposed of properly.
2. Askarel wastes should never be disposed of down effluent drains or sewers. The utmost care must be exercised to prevent accidental loss by these avenues to the environment.
3. Capacitors failing tests or otherwise designated for disposal must be controlled and handled in accordance with the intent of the procedures above, finally being disposed of by one of the means outlined in Part V of this section.

G. References

1. Monsanto Industrial Chemicals Company: Technical Bulletin 0-FF/1R, Aroclor® Polychlorinated Polyphenyls (Biphenyls) (November 1971).
2. American Conference of Governmental Industrial Hygienists: Threshold Limit Values for 1964. AMA Archieves of Environmental Health 9:545 (1964).
3. Treon, J. F., F. P. Cleveland, J. Cappel, and R. W. Atchley: The Toxicity of the Vapors of Aroclor 1242 and Aroclor 1254. American Industrial Hygiene Association Quarterly 17:204 (1956).
4. Elkins, H. B.: The Chemistry of Industrial Toxicology. John Wiley and Sons, Inc., New York (1959).

5. Drinker, C.K., M.F. Warren, and G.A. Bennet: The Problem of Possible Systemic Effects from Certain Chlorinated Hydrocarbons. Journal of Industrial Hygiene and Toxicology 19:283 (1937).

6. Drinker, C.K.: Further Observations on the Possible Systemic Toxicity of Certain of the Chlorinated Hydrocarbons. Journal of Industrial Hygiene and Toxicology 21:155 (1939).

7. Greenburg, L., M.R. Mayers, and A.R. Smith: The Systemic Effects Resulting from Exposure to Certain Chlorinnated Hydrocarbons. Journal of Industrial Hygiene and Toxicology 21:29 (1939).

8. Hygienic Guide Series "Chlorodiphenyls" (January-February 1965) by the American Industrial Hygiene Association, 210 Haddon Avenue, Westmont, N.J. 08108.

IV. CONTROL OF WATER EFFLUENTS

The industry goal is to eliminate askarel in plant water effluent streams. However, it is recognized that existing drain systems in capacitor manufacturing plants are probably contaminated as a result of past practices, and askarel traces may continue to show up in effluent streams for some time. However, the level should continue to decrease with the proper containment of askarel wastes and no further discharges into drain systems. Other sections of this Guide provide that no askarel wastes of any kind be disposed of in any water effluent streams and that accidental spills be prevented from getting into such streams.

A. Concentration Limits

The 1972 EPA proposals are to keep PCB levels in rivers and lakes below 0.01 parts per billion. Plant effluent streams should be managed and controlled in a manner consistent with this goal.

B. Monitoring Streams

On a regular basis consistent with plant situations, all effluent streams should be analyzed. The procedures for performing the necessary analyses are contained in Section 5, Analysis of Water and Sediment for Polychlorinated Biphenyls. This procedure or its equivalent should be used.

C. Methods for Minimizing Effluent Stream Contamination

The ideal approach is to totally isolate all effluent streams that could be contaminated with askarels during manufacturing processes and prevent them from being discharged from the plant. Carbon adsorption, limestone beds, and solvent extraction are techniques which can be applied to reduce the askarel content of effluent streams. These techniques may be most useful in cleaning up water used in plant processing and to permit recycling.

V. SCRAP DISPOSAL PROCEDURES

The manufacture and use of capacitors involve processes which produce askarel-saturated solids and liquids containing or composed entirely of askarel, which should be disposed of as wastes. Specific sources of these materials appear throughout this document but may be placed into three categories:

1. Capacitor units impregnated with askarel, production and field rejects.

2. Manufacturing process liquid wastes containing askarel.

3. Solid waste purposely or accidently saturated with askarel.

Disposal should be done in a manner which is consistent with proper concern for the environment and which minimizes any release of askarels to the environment.

A. Disposal of Capacitor Units

Scrap capacitor units can be generated during manufacturing processes or during field service.

Production rejects are those capacitors which are rejected after the impregnation process in the course of production by the capacitor manufacturer. They may be rejected for mechanical or electrical reasons, or because of obsolescence.

Field rejects are those units which are rejected or which, for other reasons, are to be scrapped after shipment from the plant where they were manufactured.

1. Production Rejects—Rejected capacitors in capacitor manufacturing plants represent a concentration of askarel. It is important that their disposition be made in a manner consistent with proper concern for the environment. Therefore, capacitors should be disposed of only in supervised dry landfill sites which meet all applicable State requirements.

   Care should be exercised in insure that no loss of liquid will occur during transportation to the disposal site.

   Incineration of scrap capacitors in facilities designed to accept such solids should provide an alternative means of disposal as such services become available in the future.

2. Field Rejects—Small capacitors (defined as containing less than 2 pounds of askarel) are practically always used as components in other electrical or electromechanical equipment. Typical examples of large quantity usage of such capacitors are in fluorescent lamp ballasts and residential air conditioning equipment. Failure of such capacitors may result in scrapping of the device of which it is a part (as in a fluorescent ballast) or replacement and scrapping of the individual capacitor (as in a room air conditioner). However, the majority of such capacitors do not fail in service, but are scrapped as a result of wearing out or obsolescence of the devices in which the capacitors are used as components. Thus, the matter of disposal is characterized by a low concentration of small quantities of askarel throughout the country and, indeed, throughout the world. Fortunately, the nature of the devices and equipment in which such capacitors are used is such that they are normally disposed of in dry landfills as a matter of convenience. Since it is impractical at present to exercise any meaningful control over the disposition of the bulk of such devices and equipment, it is imperative that askarel used for impregnating small capacitors be limited to the recently introduced type which contains a typical concentration of 0.4 percent of the higher coiling homologs. *

   Large capacitors (those incorporating more than 2 pounds of askarel) should be disposed of according to the procedure for production rejects (see par. A.1).

* Monsanto Method T-02302.

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B. Disposal of Liquid Wastes

All waste askarel or liquid wastes containing askaral should be disposed of in accordance with one of the following procedures:

1. Incineration—Present knowledge indicates that proper incineration must involve a suitable balance between dwell time and temperature in the incinerator plus oxygen availability and, finally, suitable scrubbers to remove the HCl which will be formed; e.g., (1) 2-second dwell time at 2000°F and 3 percent excess oxygen in stack gas; (2) 1 1/2-second dwell time at 2700°F and 2 percent excess oxygen in stack gas.

   These facilities should meet the applicable requirements of the State in which they are located and should control effluents within the limits set forth in this document.

2. Toxic and Hazardous Waste Disposal Sites—Certain landfill sites have been classified by State and Federal Governments as suitable for the disposal of toxic and hazardous liquids. Where such approved sites exist, they may be used for the disposal of liquid wastes described in this document. (See Section 4.)

3. Packaging and Shipment—

   a. Transportation to the disposal facility should be in containers which will prevent leakage and accidental loss of askarel to the environment.

   b. Containers should be labeled as to contents and precautions relative to loss to the environment.

   c. Containers used for this purpose should not be used for any other materials or retired from service until they are completely cleaned. Any solvents used in cleaning these containers will be contaminated with askarel and should be disposed of according to the same procedures herein described.

C. Disposal of Solid Wastes
(See Par. A for Scrap Capacitors)

All solid wastes which have been saturated with askarel should be disposed of by the following procedure:

1. The saturated wastes should be placed into leak-proof containers and transported to a supervised dry landfill site meeting State requirements. Alternatively, they can be disposed of by incineration in State-approved facilities.

2. Solid absorbents used for spills can be disposed of uncontained in the supervised dry landfill site; transport to the site should be in closed containers. Alternatively, incineration can be used in accordance with par. B (See Section 4 for facilities).

VI. LABELING

Capacitor units vary greatly in size and in end use or application. Small capacitor units are frequently applied as a component of another piece of equipment such as a fluorescent lighting ballast, a roadway or area lighting luminaire, a motor, etc. In such applications a label on the capacitor unit referencing approved disposal procedure would not normally be visible when the piece of equipment is disposed of.

For the foregoing reasons the method of providing disposal instructions for small capacitors and large capacitors is treated separately.

1. Small Units (Incorporating Less Than 2 Pounds of Askarel)—Small capacitors are defined as those which contain askarel in quantities up to about two (2) pounds each and in which the free liquid does not exceed 0.4 pound. They are hermetically sealed in metallic cases. Such capacitors are applied as a component of a larger piece of equipment. Attaching a label to such equipment referencing this Guideline or describing disposal procedures would be of limited practical value.

2. Large Capacitor Units (Incorporating More Than 2 Pounds of Askarel)—The capacitor manufacturer should affix a label in a conspicuous place, referencing this document or describing disposal procedures consistent with it. This label should contain, as a minimum, the following information:

CAUTION

THIS CAPACITOR CONTAINS A POLYCHLORINATED BIPHENYL (PCB). TO AVOID POSSIBLE ENVIRONMENTAL CONTAMINATION, IT SHOULD BE DISPOSED OF ONLY IN SUPERVISED DRY LANDFILL AREAS MEETING STATE REQUIREMENTS OR IN INCINERATION FACILITIES DESIGNED FOR DISPOSAL OF PCB'S.

SEE AMERICAN NATIONAL STANDARDS INSTITUTE GUIDELINES C107.1- ( ) FOR FURTHER INFORMATION. (WHEN APPROVED, COPIES WILL BE AVAILABLE FROM ANSI, 1430 BROADWAY, NEW YORK, NEW YORK 10018.)

GUIDELINES FOR HANDLING AND DISPOSAL OF CAPACITOR- AND TRANSFORMER-GRADE ASKARELS CONTAINING POLYCHLORINATED BIPHENYLS

SECTION 3

TRANSFORMER GUIDELINES

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SECTION 3
TRANSFORMER GUIDELINES

I. GENERAL GUIDELINES

A. Definitions—Types of Transformer Askarels

The term askarel generally describes a broad class of nonflammable synthetic chlorinated hydrocarbon insulating liquids widely used in transformers, reactors, and accessory equipment operated at power frequencies. Askarels of various compositional types are in use [for the general properties and types, see ASTM Specification for Chlorinated Aromatic Hydrocarbons (Askarels) for Transformers, D 2283]. Under arcing conditions the gases produced, while consisting of predominantly noncombustible hydrogen chloride, can contain varying amounts of combustible gases depending upon the askarel type.

The following trademarks are used by electrical manufacturers to designate the askarels used in their products:

Asbestol
Chlorextol
Inerteen
No-Flamol
Pyranol
Saf-T-Kuhl

B. Safety Precautions

Based on about forty years of safe industrial usage, askarels have been considered as relatively harmless materials to humans. There has been no known instance of human injury when askarels are used under the normally prescribed conditions of precaution and handling.

Although it has been generally thought that exposure to askarels is not hazardous provided simple precautions are taken, exposure should still be avoided and minimized.

1. Vapors—The odor of askarel is noticeable well below the maximum safe air concentrations. Depending upon the composition of the askarel used, from 0.5 to 1.0 milligram per cubic meter of air has been determined to be the upper safe level of exposure during an 8-hour work day. (See American Industrial Hygiene Association Hygienic Guide Series—January/February, 1965.) The procedures for performing the necessary analyses are contained in Section 5, "A Tentative Procedure for the Determination of Airborne-Polychlorinated Biphenyls." This procedure or its equivalent shall be used.

   Breathing vapor or fumes from heated askarels should be avoided. High concentrations of vapors can cause irritation of the eyes, nose, throat and upper respiratory tract. Provisions shall be made for adequate ventilation and regulation of manufacturing operations to avoid open exposure of hot askarels (55°C or higher). The gases produced when askarel is decomposed by very high temperatures (such as that of an electric arc) in the presence of air or organic insulating materials contain a high percentage of hydrogen chloride, and small percentages of other gases. Minute concentrations of this combination of gases are very unpleasant and irritating, thus giving ample warning of their presence. If exposure to high concentrations of askarels or its arced products is necessary under emergency conditions, an approved gas mask of the organic canister-type or self-contained breathing apparatus must be worn. Such exposure should be under the surveillance of other personnel capable of rescue in case of accident. If the odor of askarel or its arced products is detected by the person wearing protective

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equipment, he should immediately go into fresh air. All gas masks, respirators and replacement parts should have Bureau of Mines approval and be maintained on a regular schedule in accordance with the manufacturer's recommendation.

2. Liquid—Unlike mineral insulating oil, there is no fire hazard in handling askarels. A limited solvent action (similar to that for paint thinner) on the fats and oils of the skin with prolonged contact may lead to drying and chapping of the skin. As with insulating oil, some people are allergic to askarel and continued exposure may result in skin irritation. Both the liquid and vapor are moderately irritating to eye tissue.

Operating procedures should require avoidance of contact with any askarels. The use of porous gloves which can absorb and retain askarels is to be avoided. Resistant gloves and aprons of the neoprene, polyethylene, viton type should be used if contact is unavoidable. In case of spillage on the clothing, the clothing should be removed as soon as practical, the skin washed and the clothing laundered.

Medicinal washes or mild detergents followed by the application of cold cream will reduce the irritation resulting from the contact of an open cut or abrasion with askarel.

Safety glasses with side shields or a face shield should be worn when handling askarels. Eyes which have been exposed to liquid askarel should be irrigated immediately with large quantities of running water for 15 minutes and then examined by a physician if the irritation persists. (A drop of castor oil has been found to reduce irritation.)

Persons developing a skin irritation or respiratory tract irritation while working with askarels should be placed under supervision of a physician.

Ingestion or swallowing of askarels is not generally regarded as a problem of the industry. Should accidental ingestion occur, a physician should be consulted. Hands should be washed with warm water and soap before eating, drinking, smoking or using toilet facilities.

C. Transport Container Marking

Any container such as tank cars, tank trucks, drums, cans, etc., used to transport transformer askarels, new or used, should be labeled with the following:

CAUTION

THIS PRODUCT CONTAINS POLYCHLORINATED BIPHENYLS (PCB'S). CARE SHOULD BE TAKEN TO PREVENT ENTRY INTO THE ENVIRONMENT THROUGH SPILLS, LEAKAGE, USE, VAPORIZATION, OR DISPOSAL OF LIQUID OR CONTAINERS. AVOID PROLONGED BREATHING OF VAPORS OR MISTS. AVOID CONTACT WITH EYES OR PROLONGED CONTACT WITH SKIN. IF SKIN CONTACT OCCURS, REMOVE BY WASHING WITH SOAP AND WATER. FOLLOWING EYE CONTACT, FLUSH WITH WATER. IN CASE OF SPILLAGE ONTO CLOTHING, THE CLOTHING SHOULD BE REMOVED AS SOON AS PRACTICAL, SKIN WASHED, AND CLOTHING LAUNDERED.

D. Receiving, Handling and Storage of Askarels

Askarels are shipped in tank cars, tank trucks, steel drums, metal cans and test sample containers. When received, all containers should be inspected for leaks.

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1. Storage Tanks—Storage tanks should be erected so that inspection can be made for leaks or spills. Construction should be such that inadvertent leakage or spills are prevented from reaching streams and sanitary or storm sewers.

2. Tank Cars and Tank Trucks—All bulk shipment equipment should be inspected for leaks immediately upon receipt. Drain pans must be provided to prevent spillage from unloading hoses and connections. Askarel liquid collected in drain pans should be placed in "SCRAP ASKAREL" drums for disposition.

3. Steel Drums, Cans, and Test Sample Containers—On delivery, all such shipments should be carefully inspected for leaks. The containers should be stored indoors in an area especially selected for this purpose. A curb should enclose the area to provide a basin for containing the askarel from one or more containers should the containers be damaged. The area must not have a drain which is connected to a sanitary or storm sewer.

   If an indoor storage area is not possible, the containers should be stored under a lean-to.

E. Water Effluents—Sampling, Concentration Limits and Analytical Procedures

The industry goal is to eliminate askarel in plant water effluent streams. However, it is recognized that existing drain systems from manufacturing plants, repair shops and installation sites may be contaminated as a result of past practices. Other sections of this Guide provide that no askarel wastes of any kind be disposed of in any water effluent streams and that accidental spills be prevented from getting into such streams.

1. Concentration Limits—The 1972 EPA proposals are to keep PCB levels in rivers and lakes below 0.01 parts per billion. Plant effluent streams should be managed and controlled in a manner consistent with this goal.

2. Monitoring Streams—All plant effluent streams should be monitored on a regular basis, consistent with plant situations. The procedures for performing the necessary analyses are contained in Section 5, "Analysis of Water and Sediment for Polychlorinated Biphenyls." This procedure or its equivalent shall be used.

3. Methods for Minimizing Effluent Stream Contamination—The ideal approach is to totally isolate all effluent streams that could be contaminated with askarels during manufacturing processes and prevent them from being discharged from the plant.

F. Disposal Procedures and Services

1. Sources of Materials Requiring Special Handling and Disposal Procedures—Liquids containing PCB's and solids containing or contaminated with PCB's may be obtained from any of the following sources: transport containers, transformer manufacturing processes, in-test failures, liquids contaminated beyond reclamation, in-service transformer leaks and failures, askarel-filled transformers scrapped for any reason, etc.

2. Classification for Disposal of Materials Containing PCB's—In general, there are three types of materials requiring disposal: liquids, burnable solid materials containing PCB's and nonburnable solid materials contaminated with PCB's.

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a. Liquids. Liquids containing PCB's requiring disposal by high-temperature incineration may consist of the following:

(1) PCB's contaminated with mineral oil.

(2) Mineral oil contaminated with PCB's.

(3) Nonreclaimable contaminated transformer askarels, arced askarels, askarels from manufacturing spills, and sump accumulation, etc., rich in PCB's, and askarels from holding basins, drip and drain pans, washings, sample jars and containers, etc.

b. Burnable Solid Waste Material Containing PCB's. These materials can be disposed of by high-temperature incineration and consist of cellulosic materials, rags, pressboard, wood, sawdust, fuller's earth in bulk or in cloth bags, blotter papers, nitrile or cork gaskets, etc.

c. Nonburnable Solid Waste Materials Containing or Contaminated With PCB's. These materials may consist of coil structures, steel, copper, aluminum filter units of the steel mesh construction type, askarel drums, cans, etc.

Materials of this nature should be allowed to drain with the liquid collected in drip pans, etc. Further removal of adhering PCB's can be accomplished by washing or solvent extraction with kerosene or other approved washing liquids such as perchloroethylene or trichloroethylene. Accumulated liquids can be disposed of as indicated in par. F.2.a. Solid materials may be handled as normal scrap.

3. Shipment of Scrap Liquids for Disposal— All liquid scrap material should be placed in appropriate metal transport drums, properly labeled, for shipment to a company offering an acceptable disposal service.

4. Shipment of Burnable Solid Waste Material Containing PCB's for Disposal— Material of this type should be placed in open head drums with suitable closures and with the drum properly labeled for shipment to a company offering an acceptable disposal service.

5. Liquid and Solid Waste Disposal Service Organizations—

a. General. Disposal of askarels and askarel-soaked materials should be accomplished by means in which there is no significant release of askarel to the environment. At present, disposal is accomplished by carefully-controlled incineration of liquids and soaked software, and by controlled landfill burial of apparatus and other hardware from which askarel has been previously drained and washed.

Present knowledge indicates that proper incineration must involve a suitable balance between dwell time and temperature in the incinerator plus oxygen availability and, finally, suitable scrubbers to remove the HCl which will be formed; e.g., (1) 2-second dwell time at 2000°F and 3 percent excess oxygen in stack gas; (2) 1 1/2-second dwell time at 2700°F and 2 percent excess oxygen in stack gas.

These facilities should meet the applicable requirements of the State in which they are located and should control effluents within the limits set forth in this document.

Controlled landfill or deep-well disposal can be used where permitted by Federal, State and local regulations.

b. Costs. In addition to the normal costs of collecting scrap liquids and solids for disposal, additional costs borne by the owner of such scrap include shipping containers, cost of transport to the disposal service organization, and a disposal fee usually based upon a per gallon or per pound charge.

c. Disposal Services. Organizations offering disposal services are listed in Section 4, including their location, facilities available, types of material handled, and disposal procedures used. Specific shipping directions, disposal procedures and costs should be obtained from the organization.

II. SPECIFIC GUIDELINES

A. Transformer Manufacturers, Service Shops, etc.—Housekeeping

It is necessary to assume that in filling equipment with askarel and during further handling of this equipment an askarel spill may occur. Therefore, it is necessary to provide facilities and a procedure for clean-up to prevent contamination.

1. Askarel Filling Area—

a. The location of the askarel filling area should be adjacent to the test area and final shipping area to minimize the danger of damage of units during handling.

b. The main manufacturing area for filling equipment with askarel should be provided with impervious surface floors or suitable basins so constructed that any inadvertent leakage or spills are prevented from reaching streams, sanitary, or storm sewers. All askarel-handling equipment such as pumps, hoses, etc., shall be of the askarel-resistant type.

c. Drip pans shall be provided for hose connections and filling valves.

2. Special Containers for Scrap Materials—

a. Drums labeled "SCRAP ASKAREL" should be available for handling all spilled and waste askarel from sumps, failed units, drip pans, sample jars, etc.

b. Open-head drums with suitable closures and labeled "SCRAP BURNABLE ASKAREL WASTE" should be available for handling contaminated cellulose insulation, rags, paper pressboard, wood, gaskets, sawdust, etc.

c. Separate containers for handling steel, copper, and aluminum, each adequately marked, shall be provided for the components of contaminated core and coil assemblies. These containers are required for the various materials when repairing or scrapping assemblies.

d. Containers for supplies of material for absorbing small askarel spills or clean-up of larger spills should be provided.

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3. Conditioning of Askarels—

   a. Askarel Conditioning Equipment. The conditioning unit should be located either in the storage tank area or the main transformer manufacturing area for filling with askarel.

   b. Fuller's Earth. Conditioning of new askarel or recycled askarel requires fuller's earth treatment. The spent fuller's earth in cartridges or bags, when replaced, should be allowed to thoroughly drain over drip pans to remove as much liquid askarel as possible. The cartridge units of steel mesh construction should be placed in the "STEEL CONTAMINATED WITH ASKAREL" container for disposition. Cloth bags filled with fuller's earth should be placed in the "SCRAP BURNABLE ASKAREL WASTE" container for disposition.

4. Teardown of Units for Repair or Scrap—

   a. Drain all askarel from the unit either into a holding tank for reuse or into the drum labeled "SCRAP ASKAREL" for disposition, and then allow sufficient time for all of the askarel to drain from the core and coils.

   b. Remove the core and coil assembly from the transformer. Sufficient absorbent material should be placed on the floor to absorb any askarel fluid that still drips from the transformer.

   c. Place all materials in the appropriate salvage containers during the dismantling for later disposition.

   d. All used materials, including rags, sawdust, tape, etc., regardless of quantity, shall be put into the appropriate containers for disposition.

B. Transformer Labeling

1. New Transformers—All new transformers that contain PCB's shall have a label of adequate durability permanently and prominently attached to the tank by the manufacturer, giving adequate warning and instructions. A suggested label includes the following:

CAUTION

THE INSULATING LIQUID IN THIS TRANSFORMER CONTAINS POLYCHLORINATED BIPHENYLS (PCB'S). CARE SHOULD BE TAKEN TO PREVENT ENTRY INTO THE ENVIRONMENT. IN CASE OF MALFUNCTTION OR LEAKS, CONSULT THE INSTRUCTION MANUAL OR THE MANUFACTURER.

2. In-service Transformers—The transformer manufacturer should make available suitable labels with a similar warning as shown in par. B.1 for use on existing transformers.

C. Transformer Users—Shipping, Installation, Maintenance, Sampling, Apparatus Disposal

1. General—Askarel-filled transformers are delivered to customers as sealed units from which there is no escape of askarel under normal operation. While certain types of equipment failures can permit loss of some askarel to the environment, such cases are extremely rare.

2. Transportation and Receiving—Immediately upon receipt of the equipment and following any transportation or handling accident which could affect the integrity of the tank, bush-

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ings or radiators, the transportation vehicle, tank and fittings should be examined for any leakage or spillage that may have occurred in shipping. If leakage is evident, the cause should be corrected and the spillage soaked up with absorbent materials such as sawdust, followed by a clean-up of the affected area with rags soaked with kerosene or other approved solvent such as perchloroethylene or trichloroethylene. All materials used should be collected for proper disposition as described in Section 3 Part I, par. F, "Disposal Procedures and Services."

3. Installation and Periodic Inspection—Following installation, the unit should again be inspected for any damage or leakage. It is recommended that periodic in-service inspections be made for any leaks.

4. Filling, Filtering, or Drying Askarel—Most askarel units are shipped with the proper amount of askarel but if it becomes necessary to top off a unit, the manufacturer's instructions should be followed.

   If it is necessary to dry or fuller's-earth-treat an askarel unit, the manufacturer's instructions should be followed. When filtering or conditioning askarel, all of the precautions previously described for drip pans, proper disposal of filter media, etc., apply.

5. Sampling—It is common practice to sample askarel from a transformer for periodic maintenance testing. As previously described, such samples should be taken in a manner to avoid any contamination of the environment. Washings should be collected for proper disposal. Field and laboratory test samples, washings, etc., should also be collected for proper disposal.

6. Transformer Disposal—The ultimate disposal of an askarel-filled transformer may be accomplished in either of two ways:

   a. Complete drainage and dismantling with the proper disposal of the askarel and askarel-soaked components as described earlier in Section 3, Part I, par. F, "Disposal Procedures and Services."

   b. Disposition of askarel transformers by means of junk or scrap dealers should be avoided unless a transformer is first drained, followed by soaking the interior with a suitable solvent. Accumulated liquids and washings are to be disposed of as described earlier.

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GUIDELINES FOR HANDLING AND DISPOSAL OF CAPACITOR- AND TRANSFORMER-GRADE ASKARELS CONTAINING POLYCHLORINATED BIPHENYLS

SECTION 4

DISPOSAL SERVICES

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SECTION 4
DISPOSAL SERVICES

In addition to the supervised dry landfill sites which may be used for the disposal of askarel-containing scrap, the following additional known facilities and services have been established, and others may be available.

Chem-Trol Polution Services, Inc.,
4818 Lake Avenue
Blasdell, New York 14219
Phone 716-826-5850

This organization has facilities and services capable of handling:

1. Liquids—Askarels alone or mixed with solvents or oils. Disposal by high temperature incineration.

2. Solids (Software)—Askarel-soaked compounds, rags cartons, absorbing earths, etc. Disposal by incineration or scientific landfill.

3. Solids (Hardware)—Capacitors, transformer tanks, cores, askarel-soaked metals. Disposal by scientific landfill. Has solvent extraction capability.

Monsanto Company
800 No. Lindbergh Boulevard
St. Louis, Missouri 63166
Phone 314-694-3352

This organization has facilities and services capable of handling askarel liquids alone or mixed with other oils or solvents by high-temperature incineration. Liquid is pumped through a gun with atomizing steam into incinerator. Temperatures are maintained at 2000-2500°F with auxiliary natural gas. Exit gases are quenched to 180°F by contact with water. Gas is then passed through a high-energy venturi scrubber for removal of particulates. Before exhausting to air (110°F), gases are passed through a packed column scrubber to remove HCl.

Nuclear Engineering Company

Eastern Division
P. O. Box 146
Morehead, Kentucky 40351
Phone 606-784-6611

Disposal Division
Sheffield, Illinois
Phone 815-454-2624

This organization provides containerization, transportation and disposal services of all liquids and solids (including hardware). Disposal is in controlled chemical and scientific landfill area. AEC licensed for radioactive waste disposal. Has two West Coast locations in states of Washington and California in addition to above.

Rollins-Purle, Inc.
Box 3349
Wilmington, Delaware 19899
Phone 302-478-5150

This organization has facilities and services capable of handling:

1. Liquids—Askarel alone or mixed with solvents or oils. Disposal is by high-temperature incineration.

2. Solids (Software)—Askarel-soaked compounds, rags, cartons, absorbing earths, etc. Disposal is by incineration at combustion temperatures up to 2500°F. Incineration gases are scrubbed, and entrained solids removed before exhausting to air.

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Disposal facilities are available at the following locations:

Logan:
Rollins-Purle, Inc.
Route 322
Logan Township
Bridgeport, New Jersey 08014

Baton Rouge:
Rollins-Purle, Inc.
Scenic Highway & W. Cheatham Lane
Scotlandville
East Baton Rouge Parish
Louisiana 70807

Houston:
Rollins-Purle, Inc.
Tidal Road & Highway 134
Deer Park, Texas 77536

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GUIDELINES FOR HANDLING AND DISPOSAL OF CAPACITOR- AND TRANSFORMER-GRADE ASKARELS CONTAINING POLYCHLORINATED BIPHENYLS

SECTION 5

APPENDIX

ANALYTICAL PROCEDURES AND LABORATORY SERVICE ORGANIZATIONS

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SECTION 5
APPENDIX

ANALYTICAL PROCEDURES AND LABORATORY SERVICE ORGANIZATIONS

Analytical procedures for determining PCB's in air, water, and sediments are given in Parts I and II of Section 5.

The following laboratories are representative of those offering services for PCB analyses:

Carus Chemical Corp.
1375 Eighth Street
LaSalle, Illinois 61301
Phone 815-223-1500

Limnetics, Inc. (Subsidiary of Carus Corp.)
6132 West Fond du Lac Avenue
Milwaukee, Wisconsin 53218
Phone 414-461-9500

Gollob Analytical Service Corp.
47 Industrial Road
Berkeley Heights, New Jersey 07922
Phone 201-464-3331

I. A TENTATIVE PROCEDURE FOR THE DETERMINATION OF AIRBORNE-POLYCHLORINATED BIPHENYLS

A. Scope

This procedure is based on techniques used by the Monsanto Industrial Chemical Company for the isolation and determination of polychlorinated biphenyls (PCB's) in water, soil/sediment, and biological materials.

Absolute confirmation of PCB structures is not obtained with this method. Where needed, additional structure proof should be obtained using techniques such as mass spectrometry to further identify GC fractions.

B. Principle

Airborne PCB's are absorbed in toluene by drawing the air through one or more fritted bubblers or impingers in cylinders filled with toluene. After sampling a suitable amount of air, the scrubbing solvent is diluted or concentrated, and interfering components, if present, are removed by chemical treatment and column absorption chromatography. The amount and type of PCB's present is determined by electron capture gas chromatography (EC/GC).

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C. Reagents

Hexane	Nanograde, Mallinckrodt Chemical Works, Catalog No. 4159.
Toluene	Nanograde, Mallinckrodt Chemical Works, Catalog No. 8092.
Sodium sulfate	Anhydrous, granular: AR grade, Mallinckrodt Chemical Works, Catalog No. 8042. Heat at 400°C for 1 hour prior to use.
Alumina adsorption	(For chromatographic analysis) 80/200 mesh, Fisher Scientific Company, Catalog No. A540. Heat at 400°C for a minimum period of 4 hours and deactivate with 5% (w/w) distilled water. Alumina column preparation: Fill a chromatographic column with hexane up to the point where the reservoir joins the column and push a glass wool plug to the bottom with a glass rod. In a 50 ml beaker measure 35 ml of deactivated alumina (∿ 30g), and pour this slowly into the column. Tap or vibrate the column to settle the alumina and top the alumina with 2-3 cm of anhydrous sodium sulfate. Wash the column with 50-100 ml of hexane prior to the addition of the sample.
Distilled water	Extracted with hexane to remove hexane soluble electron capturing impurities.
Sulfuric acid	Analytical Reagent Grade, SG = 1.84.
Potassium hydroxide	Analytical Reagent Grade.
Ethanol	Formula 2B.
2.5% (w/v) alcoholic potassium hydroxide	Dissolve ∿ 12.5 grams of AR grade KOH in 500 ml of ethanol.
9/1 (v/v) sulfuric acid-water	Carefully add 270 ml of AR grade sulfuric acid to 30 ml of distilled water in a 500 ml iced beaker.
PCB Standards	Aroclor 1016, 1221, 1242, 1248, 1254 and 1260. See Fig. 5-3 through 5-9 (pages 17 to 23).

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D. Apparatus

1. Gas scrubbing bottles, high form, ground glass joint, fritted coarse discs.
2. Separatory funnels equipped with ground glass stoppers and Teflon stopcocks: 125, 250, 500, 1000 and 2000 ml capacities.
3. Kunderna-Danish evaporative concentrators, 500 ml capacity equipped with 3-ball Snyder columns and graduated 5 ml capacity vials: Ace Glassware Company, Catalog No. 6707.
4. Chromatographic columns, glass, 10" x 20 mm (OD) with a 5" x 50 mm (OD) reservoir at the top, equipped with Teflon stopcocks.
5. Flat-bottomed boiling flasks, 125 ml capacity: Ace Glassware Company, Catalog No. 6896, Code - 04.
6. Liebig condenser, 200 mm in length: Ace Glassware Company, Catalog No. 5915, Code - 12.
7. Hot plates, Corning PC-100; Fisher Scientific Company.
8. Water bath, Thelco, Precision Scientific, Model No. 84, Fisher Scientific Company.
9. 10 μl Hamilton syringes, Catalog No. 701N.
10. Dry test meter or wet test gas meter.
11. Laboratory vacuum pump.
12. Rotating vacuum evaporator.
13. Usual laboratory glassware.

E. Sampling (See Fig. 5-1)

The air to be sampled for airborne PCB's is drawn through a gas scrubber(s) and a dry test meter using a laboratory vacuum pump. The sampling flow rate is controlled by bleeding in air via a needle valve located between the pump and the meter. At the end of the sampling period, the metered gas volumes are corrected for temperature and pressure to cubic meters at 25°C and 760 mm Hg.

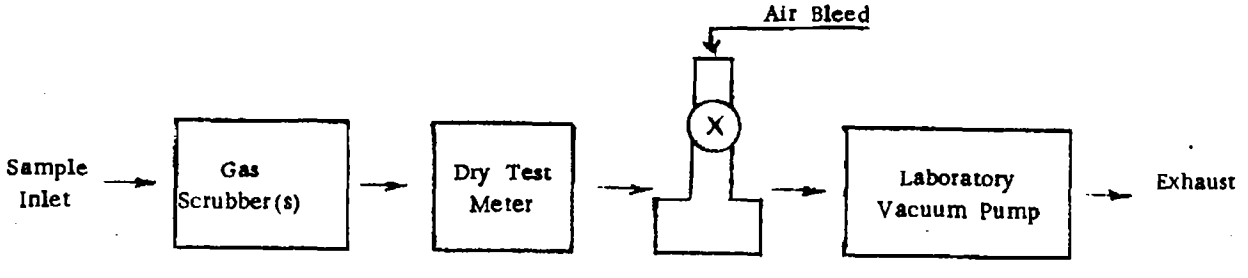

Fig. 5-1
SAMPLING TRAIN

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It is important to note that neither the capacity nor the efficiency of the gas scrubber(s) for removal of airborne PCB's have been experimentally evaluated. For this reason, it is best to minimize the sampling flow rate and maximize the sampling time period to obtain measurable amounts of PBC's. When high flow rates must be employed or a larger capacity may be needed, it is recommended that several gas scrubbers be used in tandem.

It is cautioned that, until the efficiency and capacity of the toluene gas scrubber has been experimentally established, this procedure should be only used to measure relative PCB levels sampled under equivalent conditions.

F. Procedure

1. After scrubbing the desired amount of air, record the metered volume, pressure, and temperature.

2. Quantitatively transfer the scrubbing solvent to a round-bottomed flask and reduce the volume to approximately 2 ml by rotary vacuum evaporation.

3. Quantitatively transfer the concentrate to a 30-ml beaker with the aid of several small portions of toluene.

4. Inject a fraction of a μ l of the concentrate into the gas chromatograph to check for interferences and determine the approximate level of PCB's present. If no interferences are present, dilute or concentrate the sample to a known volume, as determined by the electron capture chromatogram, and proceed with the gas chromatographic analysis.

5. If interferences are present, proceed with the chemical treatment and column chromatographic clean-up procedures.

6. Transfer the concentrate to a 125-ml extraction flask with the aid of several small portions of solvent.

7. Evaporate the concentrate just to dryness with a gentle stream of dry filtered air and add 25 ml of 2.5% alcholic potassium hydroxide.

8. Add a boiling chip, put a water condenser in place, and allow the solution to reflux for 45 minutes.

9. After cooling, transfer the solution to a 250-ml separatory funnel with the aid of 25 ml of distilled water.

10. Rinse the extraction flask with 25 ml of hexane and add it to the separatory funnel.

11. Stopper the separatory funnel and shake vigorously for at least 1 minute. Allow the layers to separate and transfer the lower aqueous phase to a second separator funnel.

12. Extract the saponification solution with a second 25-ml portion of hexane. After the layers have separated, add the first hexane extract to the second separatory funnel and transfer the aqueous alcohol layer to the original separatory funnel.

13. Repeat the extraction with a third 25-ml portion of hexane. Discard the saponification solution and combine the hexane extracts.

14. Carefully add 25 ml of the sulfuric acid solution (9:1 concentrated sulfuric acid/water) to the hexane extracts.

15. Stopper the separatory funnel and shake vigorously for at least 1 minute. Allow the layers to separate and discard the lower aqueous acid layer. Repeat this step until the acid layer is colorless.

16. Wash the hexane with a 25-ml portion of water. Discard the water wash.

17. Filter the hexane extract through a 4" funnel plugged with glass wool which is covered with a layer of sodium sulfate into a Kunderna-Danish evaporative concentrator.

18. Add a small boiling chip, put the Snyder column in place, and reduce the hexane volume to less than 5 ml by heating the apparatus in a 80-90°C water bath.

19. After cooling, remove the 5-ml graduated tube and transfer the hexane extract to an alumina adsorption column, washing it in with several 5-ml portions of hexane.

20. Carefully add 100 ml of hexane to the column reservoir and collect the total eluent in either a 250-ml volumetric flask or a Kunderna-Danish evaporative concentrator.

21. If the column eluent is collected in a volumetric flask, dilute to volume with hexane, and proceed with the gas chromatographic analysis.

22. If the column eluent is collected in a Kunderna-Danish evaporative concentrator, reduce solvent volume, cool, dilute to volume, and proceed with gas chromatographic analysis.

G. Electron Capture Gas Chromatographic Procedure

Instrument	F & M 402 Biomedial Gas Chromatograph
Detector	High Temperature Ni^{63} Electron Capture Cell 6 mm x 6' Glass Column, 4% XE-60 on 80/100 mesh Chromosorb W, HP, AW-DMCS
Column Temperature	160-190°C
Detector Temperature	300°C
Injection Port Temperature ..	195-215°C
Pulse	150
Flow Rates	Helium Carrier ∿ 60 ml/min Argon-Methane Purge ∿ 120 ml/min

Using EC/GC as the determinative step, inject in duplicate 1-10 μ l of each solution into the chromatograph. By comparison with standard solutions injected, in duplicate, under the same operating conditions, determine the amount and type of Aroclor using the individual or total peak height and area methods.

H. Sample Concentration

Concentration of sample extracts is necessary, prior to clean up by chromatographic or chemical means, to reduce sample size and increase sensitivity. The preferred method of concentrating allows minimum loss through volatilization or chemical decomposition and requires a minimum time. The three methods of solvent volume reduction most commonly used are evaporation by exposure to a stream of air, evaporation employing a Kunderna-Danish evaporative concentrator equipped with a Synder column, and evaporation under reduced pressure. All three techniques have been used without encountering any significant losses from volatization or chemical alternation.

DSW 010239

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I. Column Adsorption Chromatography and Chemical Clean Up

Silica gel, Florisil and alumina deactivated with 0, 1.0, 1.5, 2.0 and 5 percent water were investigated as adsorbants for the elimination of interferences. Alumina (5% water) was found to be more effective and reproducible than either silica gel or Florisil. The activity of alumina varies with age and lot; therefore, 5% water was added to the alumina, after heating for a minimum of 4 hours at 400°C, to insure a reproducible activity.

Saponification and subsequent extraction of the sample with sulfuric acid is an effective way to remove a number of chlorinated hydrocarbon interferences as well as other matrix interferences. PCB's are not affected.

J. Column Performance

Column performance is the key to effective gas chromatographic analysis and, as such, the choice of column materials is particularly important. Ideally, the support employed should be inert, mechanically strong, and of high surface area. Chromosorb W,* HP, AW-DMCS fulfills these requirements and is recommended for this work.

A variety of polar and non-polar liquid phases have been investigated. The following columns have been found to provide adequate separation, etc., for use in PCB analysis by electron capture: 4% (w/w) DC-200, SF-96, OV-17, SE-30, SE-54, XE-60, Apiezon L, and 6% QF-1. DC-200 and XE-60 or QF-1 have been found to be the most suitable of these liquid phases.

Another important consideration when working with an extremely sensitive detector and consequently low levels of materials is column conditioning. With polar phases such as XE-60 and QF-1, operating a new column overnight at a temperature 25-50°C higher than that to be used during analysis results in a more stable column. A no-flow conditioning technique is employed to condition non-polar columns. The column is purged with carrier gas, heated for 30 minutes at an elevated temperature without carrier flow and then cooled to room temperature. At the end of this cycle, the carrier flow is resumed and the conditioning is completed as in the case of the polar liquid phase. Two precautions: during conditioning, the column should not be connected to the detector, and one should not exceed the maximum safe temperature of the liquid phase.

Since all liquid substrates bleed to one degree or another and columns eventually degrade, all new columns should be characterized with two column performance indicators—the number of theoretical plates (N) and a tailing factor (T). p, p'-DDT is employed to check these parameters because it is known to degrade on "poor" columns. In this manner, one can determine if the performance of a new column is satisfactory and when the column performance begins to fall off. A column is considered good if the number of theoretical plates per foot is on the order of 400-500 with tailing factors of 1.0-1.3. Calculation of these parameters is shown in Fig. 5-2, page 8. Additionally, there should be no significant extraneous peaks upon injection of a pure p, p'-DDT standard.

Other chromatographic conditions that can be adjusted are column temperature and flow rates. Although resolution of a mixture increases with decreasing temperature, a temperature should be chosen that allows the elution of all components within a convenient time period. The flow rates shown are optimum for a given instrument, column and detector system. These should be adjusted if better results can be achieved.

DSW 010240

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Two gas chromatographic systems have been used for PCB analysis—F & M Model 402 and 5750. Any system of instrument and column suitable for chlorinated pesticides is satisfactory for PCB analysis. The use of the high temperature Ni^{63} electron capture cell is highly recommended. The ability to operate at higher temperatures prevents maintenance problems due to contamination from high boiling components. Glass columns should also be employed.

K. Detection and Measurement

Quantitative determinations employing the electron capture detector are non-stoichiometric measments made by comparing peak heights or areas for known concentrations with those for unknown compositions. Three variations of the peak height or area quantification procedures have been employed:

Case I — EC gas chromatogram of PCB unknown unchanged with respect to standard PCB with no evidence of interferences.

Case II — EC gas chromatogram of PCB unknown altered with respect to standard PCB with no evidence of interferences.

Case III— EC gas chromatogram of PCB unknown unchanged with respect to standard PCB with evidence of interference.

The amount of PCB's in Case I samples is determined by preparing a plot of the major peak height or area versus concentration. With Case III samples, a peak free from interference is used. When dominant interferences are present, one or more of the chemical clean-up procedures is employed.

In all cases, the response of the electron capture detector must be linear for quantitative analysis.

L. Contamination

In determining PCB's in biological materials by electron capture gas chromatography, laboratory sources of contamination can be a major problem. The samples and extracts should never be allowed to come in contact with materials other than glass, Teflon or metal. Laboratory glassware should be thoroughly washed with hot, soapy water, rinsed with distilled water, acetone, and then hexane. All equipment should also be rinsed again with hexane just prior to use, and blanks should be frequently carried through all steps of the procedures to insure against the possibility of contamination.

M. Sensitivity

Two parts per billion.
Absolute sensitivity—0.5×10^{-9} grams.
Volume injected—5 μ l.
Final volume of extract—5 ml.
Sample size—250 ml.

DSW 010241

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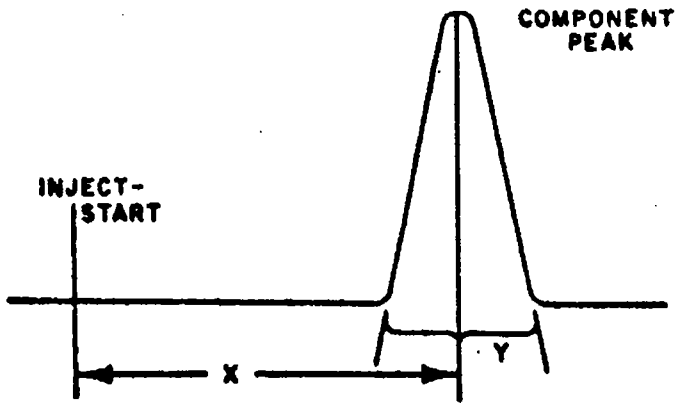
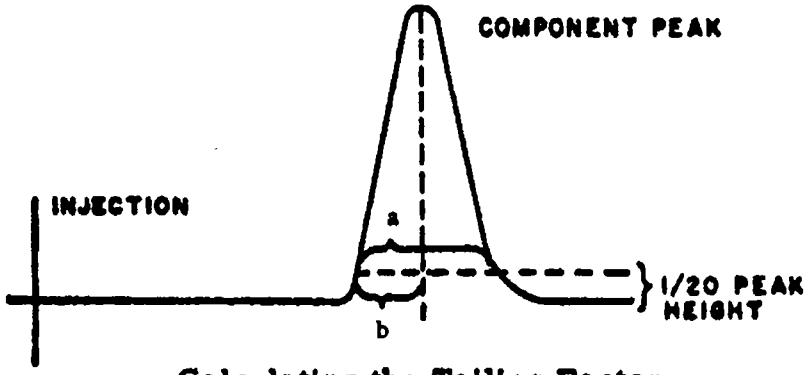
1. Theoretical Plates, N $N = 16 (x/y)^2$



Calculating Column Efficiency

2. Tailing, T $T = a/2b$



Calculating the Tailing Factor

Fig. 5-2
COLUMN PERFORMANCE INDICATORS

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11. ANALYSIS OF WATER AND SEDIMENT FOR POLYCHLORINATED BIPHENYLS

A. Scope

This methodology is used by the Monsanto Industrial Chemical Company for the determination of the amount and type of polychlorinated biphenyls (PCB's) in water and sediment samples. Absolute confirmation of PCB structures is not obtained with this method. Structure proof can be obtained using additional techniques such as mass spectrometry to further identify the GC fractions.

B. Principle

The PCB's in water and sediment samples are extracted into an organic solvent. Interfering components are then removed from the extracts by chemical treatment and column adsorption chromatography. The amount and type of PCB's present is determined by electron capture gas chromatography (EC/GC).

C. Reagents

Hexane.....	Nanograde, Mallinckrodt Chemical Works, Catalog No. 4159.
Acetonitrile.....	Nanograde, Mallinckrodt Chemical Works, Catalog No. 2442.
Sodium sulfate	Anhydrous, granular: AR grade Mallinckrodt Chemical Works, Catalog No. 8042. Heat at 400°C for 1 hour prior to use.
Alumina adsorption	(For chromatographic analysis) 80/200 mesh, Fisher Scientific Company, Catalog No. A540. Heat at 400°C for a minimum period of 4 hours and deactivate with 5% (w/w) distilled water.
	Alumina column preparation: Fill a chromatographic column with hexane up to the point where the reservoir joins the column and push a glass wool plug to the bottom with a glass rod. In a 50 ml beaker measure 35 ml of deactivated alumina ($\sim$ 30g). and pour this slowly into the column. Tap or vibrate the column to settle the alumina and top the alumina with 2-3 cm of anhydrous sodium sulfate. Wash the column with 50-100 ml of hexane prior to the addition of the sample.

(Continued)

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Distilled water	Extracted with hexane to remove hexane soluble electron capturing impurities.
Sulfuric acid	Analytical Reagent Grade, SG = 1.84.
Potassium hydroxide	Analytical Reagent Grade.
Ethanol	Formula 2B.
2.5% (w/v) alcoholic potassium hydroxide	Dissolve ~ 12.5 grams of AR grade KOH in 500 ml of ethanol.
9/1 (v/v) sulfuric acid-water	Carefully add 270 ml of AR grade sulfuric acid to 30 ml of distilled water in a 500 ml iced beaker.
PCB Standards	Aroclor 1242, 1248, 1254 and 1260. (See Fig. 5-4, page 18, and Fig. 5-6 through 5-9, pages 20 to 23.)

D. Apparatus

1. Separatory funnels equipped with ground glass stoppers and Teflon stopcocks: 125, 250, 500, 1000 and 2000 ml capacities.
2. Kunderna-Danish evaporative concentrators, 500 ml capacity equipped with 3-ball Snyder columns and graduated 5 ml capacity vials: Ace Glassware Company, Catalog No. 6707.
3. Chromatographic columns, glass, 10" x 20 mm (OD) with a 5" x 50 mm (OD) reservoir at the top, equipped with Teflon stopcocks.
4. Sintered glass filter funnels, 600 ml capacity, 90M.
5. Flat-bottomed boiling flasks, 125 ml capacity: Ace Glassware Company, Catalog No. 6896, Code - 04.
6. Liebig condenser, 200 mm in length: Ace Glassware Company, Catalog No. 5915, Code - 12.
7. Hot plates, Corning PC-100: Fisher Scientific Company.
8. Water bath, Thelco, Precision Scientific, Model No. 84, Fisher Scientific Company.
9. Reciprocating variable speed shaker, Eberbach Corporation, Fisher Scientific Company.
10. 10 μ l Hamilton syringes, Catalog No. 701N.
11. 32 oz all-glass mortars and pestles.
12. 8" x 12" x 2" (2 1/2 qt) Pyrex baking dishes.
13. U.S. Standard Sieve, No. 30, Fisher Scientific Company.
14. Usual laboratory glassware.

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E. Sampling

It is to be assumed that a rather wide variety of sampling techniques may be employed in collecting samples submitted for analysis. For this reason water and sediment samples should be treated as follows:

1. Water—Where possible the entire water sample, including the container in which it was collected, should be extracted with hexane. With larger samples, where this is not physically possible, the containers should be simply agitated and a 250 ml portion used for the analysis. (See par. F.)

2. Sediment—Any excess water should be decanted and the entire sediment transferred to a glass baking dish to air dry at room temperature. The dried material should be transferred from the dish into a mortar and ground. The ground sediment should be sieved, remixed, and a 250 g portion taken for analysis. (See par. G.)

F. Extraction of Water Samples

1. After agitating, transfer the entire aqueous sample or a 250 ml aliquot into a graduated glass cylinder. Record the volume of the sample and quantitatively transfer it to a separatory funnel with distilled water.

2. Rinse the graduated cylinder with two 50-ml portions of hexane and add each to the separatory funnel.

3. Stopper the separatory funnel and shake vigorously for at least 1 minute. Allow the layers to separate and transfer the lower aqueous phase to a second separatory funnel.

4. Extract the water sample a second time with a 50-ml portion of hexane. After the layers have separated, add the first hexane extract to the second separatory funnel and transfer the aqueous layer to the original separatory funnel.

5. Repeat the extraction with a third 50-ml portion of hexane. Discard the aqueous layer and combine the hexane extracts.

6. Filter the combined extracts through a 4" funnel plugged with glass wool which is covered with sodium sulfate. Collect the filtrate in a Kunderna-Danish evaporative concentrator, add a small boiling chip, put the Snyder column in place, and reduce the hexane volume to less than 5 ml by heating the apparatus in a 80-90°C water bath. (CAUTION: SOLVENT VAPORS MUST BE VENTED TO A HOOD.)

7. After cooling, remove the 5-ml graduated tube and transfer the hexane extract to an alumina adsorption column, washing it in with several 5-ml portions of hexane.

8. Carefully add 100 ml of hexane to the column reservoir and collect the total eluent in either a 250-ml volumetric flask or a Kunderna-Danish evaporative concentrator.

9. If the column eluent is collected in a volumetric flask, dilute to volume with hexane, and proceed with the gas chromatographic analysis.

10. If the column eluent is collected in a Kunderna-Danish evaporative concentrator, reduce solvent volume, cool, dilute to volume, and proceed with the gas chromatographic analysis.

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G. Extraction of Sediment and Soil Samples

1. Decant off any excess water and tranfer the entire sediment sample to a glass baking dish. Air dry at ambient temperature (heat should not be applied).

2. When dry, transfer the soil/sediment to a mortar and grind. Sieve the ground material through a No. 30 mesh sieve and weigh 250 g (to the nearest 0.01 g) into a 16-oz narrow neck screw cap (aluminum foil liner) glass bottle.

3. Moisten the soil with water (∿ 10 ml) and add 150 ml of acetonitrile. Cap the bottle tightly, and mechanically shake it for a minimum period of 1 hour.

4. Quantitatively transfer the acetonitrile extract into a 600-ml sintered glass filter funnel containing a 1/4" layer of anhydrous sodium sulfate. Collect the filtrate in a 600-ml beaker (vacuum filtration may be necessary).

5. After the acetonitrile has completely drained into the beaker, wash the bottle twice with 50-ml portions of acetonitrile, adding each wash to the funnel after the previous wash has completely percolated through the sediment.

6. Quantitatively transfer the extract to a Kunderna-Danish evaporative concentrator, add a small boiling chip, put the Snyder column in place, and reduce the solvent volume to less than 5 ml by heating the apparatus in a 80-90°C water bath. (CAUTION: SOLVENT VAPORS MUST BE VENTED INTO A HOOD.)

7. After cooling, remove the 5-ml graduated tube and transfer the concentrate of extracts to a 125-ml extraction flask with the aid of several small portions of solvent.

8. Evaporate the extract just to dryness with a gentle stream of dry filtered nitrogen and add 25 ml of 2.5% alcoholic potassium hydroxide.

9. Add a boiling chip, put a water condenser in place, and allow the solution to reflux for 45 minutes.

10. After cooling, transfer the solution to a 250-ml separatory funnel with the aid of 25 ml of distilled water.

11. Rinse the extraction flask with 25 ml of hexane and add it to the separatory funnel.

12. Stopper the separatory funnel and shake vigorously for at least 1 minute. Allow the layers to separate and transfer the lower aqueous phase to a second separatory funnel.

13. Extract the saponification solution with a second 25-ml portion of hexane. After the layers have separated, add the first hexane extract to the second separatory funnel and transfer the aqueous alcohol layer to the original separatory funnel.

14. Repeat the extraction with a third 25-ml portion of hexane. Discard the saponification solution and combine the hexane extracts.

15. Carefully add 25 ml of the sulfuric acid solution (9:1 concentrated sulfuric acid/water) to the hexane extracts.

16. Stopper the separatory funnel and shake vigorously for at least 1 minute. Allow the layers to separate and discard the lower aqueous acid layer. Repeat this step until the acid layer is colorless.

17. Wash the hexane with a 25-ml portion of water. Discard the water wash.

18. Filter the hexane extract through a 4" funnel plugged with glass wool which is covered with a layer of sodium sulfate into a Kunderna-Danish evaporative concentrator.

19. Add a small boiling chip, put the Snyder column in place, and reduce the hexane volume to less than 5 ml by heating the apparatus in a 80-90°C water bath.

20. After cooling, remove the 5-ml graduated tube and transfer the hexane extract to an alumina adsorption column, washing it in with several 5-ml portions of hexane.

21. Carefully add 100 ml of hexane to the column reservoir and collect the total eluent in either a 250-ml volumetric flask or a Kunderna-Danish evaporative concentrator.

22. If the column eluent is collected in a volumetric flask, dilute to volume with hexane, and proceed with the gas chromatographic analysis.

23. If the column eluent is collected in a Kunderna-Danish evaporative concentrator, reduce solvent volume, cool, dilute to volume, and proceed with gas chromatographic analysis.

H. Electron Capture Gas Chromatographic Procedure

Instrument	F & M 402 Biomedical Gas Chromatograph
Detector	High Temperature Ni^{63} Electron Capture Cell 6 mm x 6' Glass Column, 4% XE-60 on 80/100 mesh Chromosorb W, HP, AW-DMCS
Column Temperature.....	160°C
Detector Temperature.....	300°C
Injection Port Temperature ..	195°C
Pulse.....	150
Flow Rates	Helium Carrier ~ 60 ml/min Argon-Methane Purge ~ 120 ml/min

Using EC/GC as the determinative step, inject in duplicate 1-10 μl of each solution into the chromatograph. By comparison with standard solutions injected, in duplicate, under the same operating conditions, determine the amount and type of Aroclor using the individual or total peak height method.

The electron capture detector should also be used to guide the isolation procedures. Water and sediment extracts can be checked for the presence of PCB's and/or interferences by injection μl portions of the extracts at various points in the extraction and concentration schemes. In this manner, it can be determined if the sample needs to be concentrated or diluted and if the clean-up procedures should be employed.

I. Extraction

The extraction of PCB's from water, employing hexane as the extractant, has been found to be quantitative and sufficiently simple and rapid for use as a routine procedure.

The evaluation of this method was based on spiking water samples with standard acetone solutions of PCB's. The spiking method consisted of adding the PCB's in acetone (25-50 μl) to 500 ml of tap water

in a 32-oz narrow-neck screw cap jar. After thoroughly mixing the sample, duplicate 225-250 ml aliquots were taken and subjected to the proposed sample preparation and worked up as outlined. The results were quantified by preparing a calibration curve using standard hexane solutions of the PCB's used to spike the water samples. The major isomer peak height was used to construct the calibration plot.

The average recovery and deviation achieved substantiated the applicability of the method for the quantitative recovery and analysis of PCB's from water at the ppb-ppm level.

No PCB recovery experiments from spiked sediment and soil samples have been performed. Instead, several of the residual solids representative of some of the types of sediment or soil analyzed were re-extracted with hexane/acetone (40/60) in a soxhlet extractor to test for the efficiency of the acetonitrile extraction step. The hexane, after isolation by dilution with distilled water, was then carried through the purification steps. Recoveries by soxhlet extraction have indicated that the acetonitrile extraction of PCB's was essentially quantitative in the cases checked.

J. Sample Concentration

Concentration of sample extracts is necessary, prior to clean up by chromatographic or chemical means, to reduce sample size and increase sensitivity. The preferred method of concentrating allows minimum loss through volatilization or chemical decomposition and requires a minimum time. The three methods of solvent volume reduction most commonly used are evaporation by exposure to a stream of air, evaporation employing a Kunderna-Danish evaporative concentrator equipped with a Snyder column, and evaporation under reduced pressure. All three techniques have been used, and no significant losses from volatilization or chemical alternation have been encountered. However, the Kunderna-Danish evaporative concentrator and the stream-of-air methods are easier to use.

K. Column Adsorption Chromatography and Chemical Clean Up

Silica gel, Florisil and alumina deactivated with 0, 1.0, 1.5, 2.0 and 5 percent water were investigated as adsorbants for the elimination of interferences. Alumina (5% water) was found to be more effective and reproducible than either silica gel or Florisil. The activity of alumina varies with age and lot; therefore, 5% water is added to the alumina, after heating for a minimum of 4 hours at 400°C, to insure a reproducible activity.

Saponification and subsequent extraction of the sample with sulfuric acid is an effective way to remove a number of chlorinated hydrocarbon interferences as well as other matrix interferences. PCB's are not affected.

L. Column Performance

Column performance is the key to effective gas chromatographic analysis and, as such, the choice of column materials is particularly important. Ideally, the support employed should be inert, mechanically strong, and of high surface area. For these reasons, Chromosorb W, HP, AW-DMCS is recommended for this work.

A variety of polar and non-polar liquid phases have been investigated. The following columns have been found to provide adequate separation, etc., for use in PCB analysis by electron capture: 4% (w/w) DC-200, SF-96, OV-17, SE-30, SE-54, XE-60, Apiezon L, and 6% OF-1. DC-200 and XE-60 or QF-1 are the most suitable of these liquid phases.

Another important consideration when working with an extremely sensitive detector and consequently low levels of materials is column conditioning. With polar phases such as XE-60 and QF-1, operating a new column overnight at a temperature 25-50°C higher than that to be used during analysis results in a more stable column.

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A no-flow conditioning technique is employed to condition non-polar columns. The column is purged with carrier gas, heated for 30 minutes at an elevated temperature without carrier flow and then cooled to room temperature. At the end of this cycle, the carrier flow is resumed and the conditioning is completed as in the case of the polar liquid phase. Two precautions: during conditioning, the column should not be connected to the detector, and one should not exceed the maximum safe temperature of the liquid phase.

Since all liquid substrates bleed to one degree or another and columns eventually degrade, all new columns should be characterized with two column performance indicators—the number of theoretical plates (N) and a tailing factor (T). p,p'-DDT is employed to check these parameters because it is known to degrade on "poor" columns. In this manner, one can determine if the performance of a new column is satisfactory and when the column performance begins to fall off. A column is considered good if the number of theoretical plates per foot is on the order of 400-500 with tailing factors of 1.0-1.3. Calculation of these parameters is shown in Fig. 5-2, page 8. Additionally, there should be no significant extraneous peaks upon injection of a pure p,p'-DDT standard.

Other chromatographic conditions that can be adjusted are column temperature and flow rates. Although resolution of a mixture increases with decreasing temperature, a temperature should be chosen that allows the elution of all components within a convenient time period. The temperatures given are optimum for 42% chlorinated biphenyl; temperatures are increased when specifically analyzing for the higher chlorinated biphenyls, i.e., 54%, 60%, etc. The flow rates shown are optimum for a given instrument, column and detector system. These should be adjusted if better results can be achieved.

Two gas chromatographic systems have been used for PCB analysis—F & M Model 402 and 5750. Any system of instrument and column suitable for chlorinated pesticides is satisfactory for PCB analysis. The use of the high temperature Ni^{63} electron capture cell is highly recommended. The ability to operate at higher temperatures prevents maintenance problems due to contamination from high boiling components. Glass columns should also be employed.

M. Detection and Measurement

Quantitative determinations employing the electron capture detector are non-stoichiometric measurements made by comparing peak heights or areas for known concentrations with those for unknown compositions. Except for sharp peaks, peak area measurements are usually more reproducible than peak height measurements but are extremely time consuming unless a recording integrator is employed. However, peak height measurements are as accurate as disc integration of triangulation and, if the peak shape represents a gaussian curve, the height may be considered independent of the base. Consequently, peak height measurements were generally used. Three variations of the peak height quantification procedure were employed.

Case I — EC gas chromatogram of PCB unknown unchanged with respect to standard PCB with no evidence of interferences.

Case II — EC gas chromatogram of PCB unknown altered with respect to standard PCB with no evidence of interferences.

Case III— EC gas chromatogram of PCB unknown unchanged with respect to standard PCB with evidence of interference.

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The amount of PCB's in Case I samples is determined by preparing a plot of the major peak height versus concentration. For Case II, a plot is prepared of the total sum of all major peaks versus concentration. With Case III samples, a peak free from interference is used. When dominant interferences are present, one or more of the chemical clean-up procedures is employed.

In all cases, the response of the electron capture detector must be linear for quantitative analysis.

N. Contamination

In determining PCB's in water, soil and sediment by electron capture gas chromatography, laboratory sources of contamination can be a major problem. The samples and extracts should never be allowed to come in contact with materials other than glass, Teflon or metal. Laboratory glassware should be thoroughly washed with hot, soapy water, rinsed with distilled water, acetone, and then hexane. All equipment should also be rinsed again with hexane just prior to use, and blanks should be frequently carried through all steps of the procedures to insure against the possibility of contamination.

O. Sensitivity

Two parts per billion.
Absolute sensitivity—0.5×10^{-9} grams.
Volume injected—5 μl.
Final volume of extract--5 ml.
Sample size—250 ml.

AROCLOR 1016

H&P 402 Ni-63 EC
1/4" x 6' 4% XE-60 on 80/100
Chromosorb W HP
Col. Temp. - 200°C
Inj. Temp. - 220°C
Det. Temp. - 250°C
Carrier Gas - He 60 ml/min.
Purge Gas - 10% CH_4/Argon 120 ml/min.
Pulse Interval - 50 µsec
Amt. Injected - 5 µl
Std. Conc. - 1 µg/ml.
Range - 10
Attn. - 8

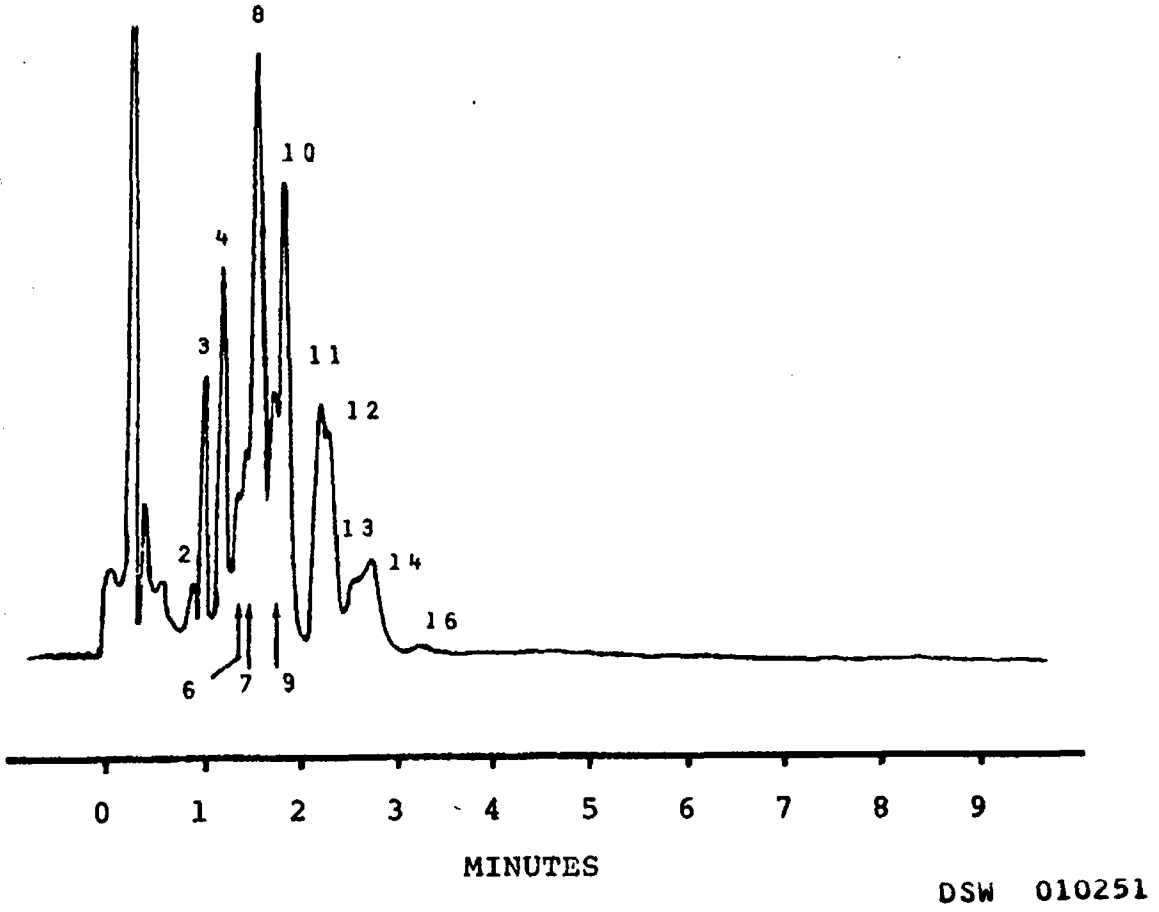

Fig. 5-3

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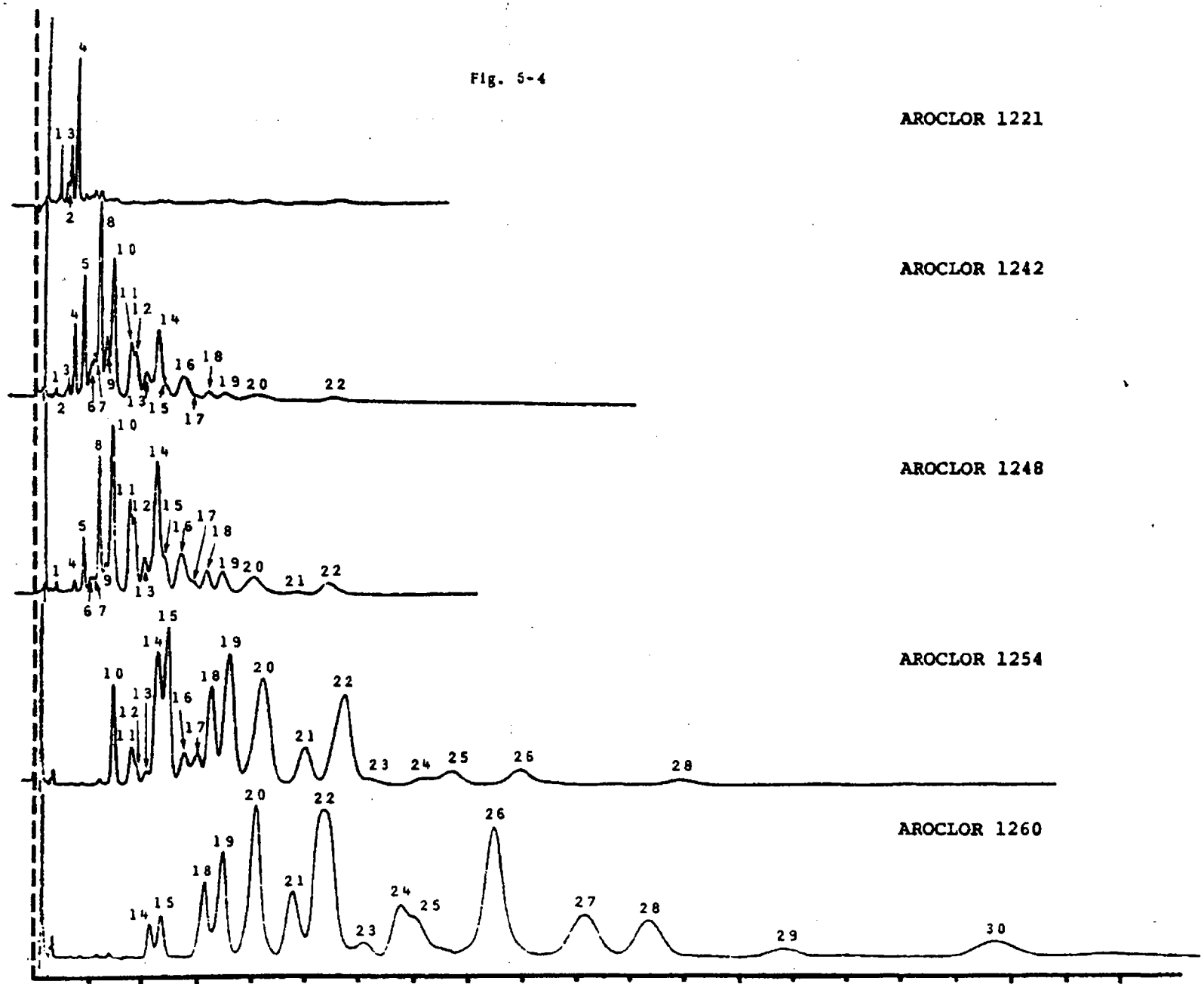
Fig. 5-4



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AROCLOR 1221

H&P 402 Ni-63 EC
1/4" x 6' 4% XE-60 on 80/100
Chromosorb W HP
Col. Temp. - 170°C
Inj Temp. - 220°C
Det. Temp. - 250°C
Carrier Gas - He 60 ml/min.
Purge Gas - 10% CH_4/Argon 120 ml/min.
Pulse Interval - 50 µsec
Amt. Injected - 5 µl
Std. Conc. - 1.48×10^{-6} g/ml
Range - 10
Attn. - 4

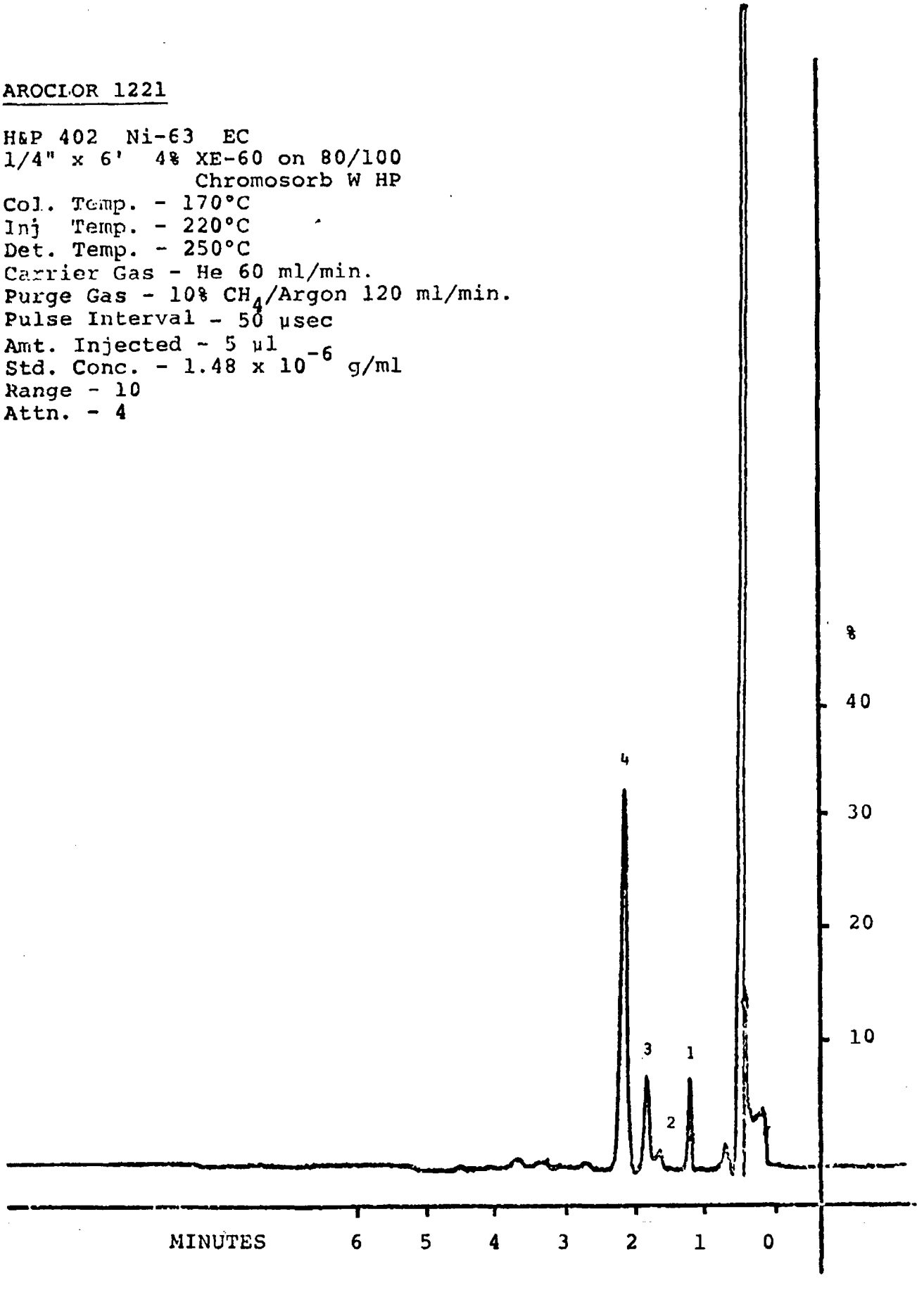

Fig. 5-5

STLCOPCB4001706

AROCLOR 1242

H&P 402 Ni-63 EC
1/4" x 6' 4% XE-60 on 80/100
Chromosorb W HP
Col. Temp. - 190°C
Inj. Temp. - 220°C
Det. Temp. - 250°C
Carrier Gas - He 60 ml/min.
Purge Gas - 10% CH_4/Argon 120 ml/min.
Pulse Interval - 50 µsec
Amt. Injected - 5 µl
Std. Conc. - 1.03 x 10^{-6} g/ml
Range - 10
Attn. - 8

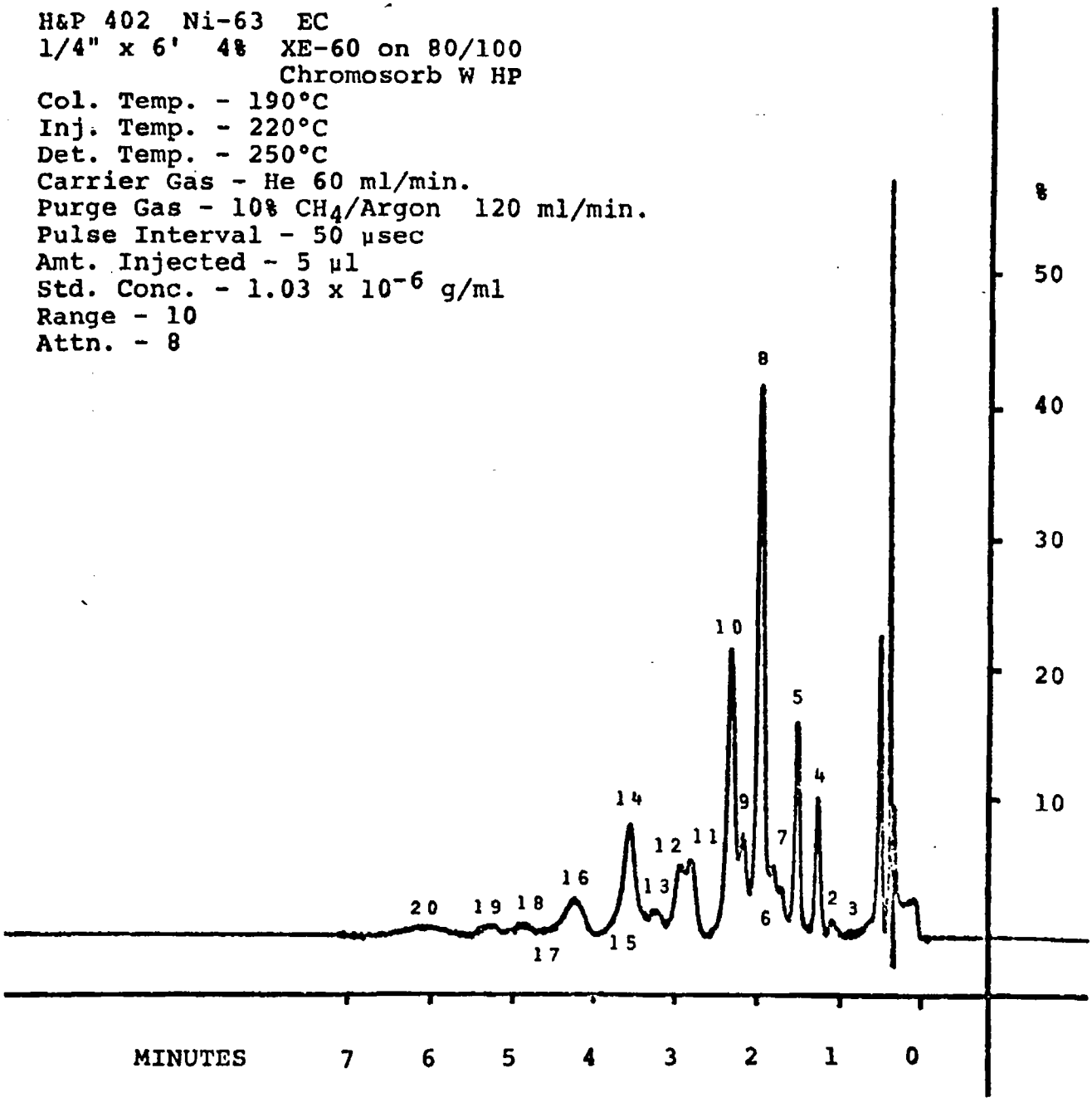

Fig. 5-6

DSW 010254

AROCLOR 1248

H&P 402 Ni-63 EC
1/4" x 6' 4% XE-60 on 80/100
Chromosorb W HP
Col. Temp. - 190°C
Inj. Temp. - 220°C
Det. Temp. - 250°C
Carrier Gas - He 60 ml/min.
Purge Gas - 10% CH_4/Argon 120 ml/min.
Pulse Interval - 50 µsec
Amt. Injected - 5 µl
Std. Conc. - 1.31×10^{-6} g/ml
Range - 10
Attn. - 4

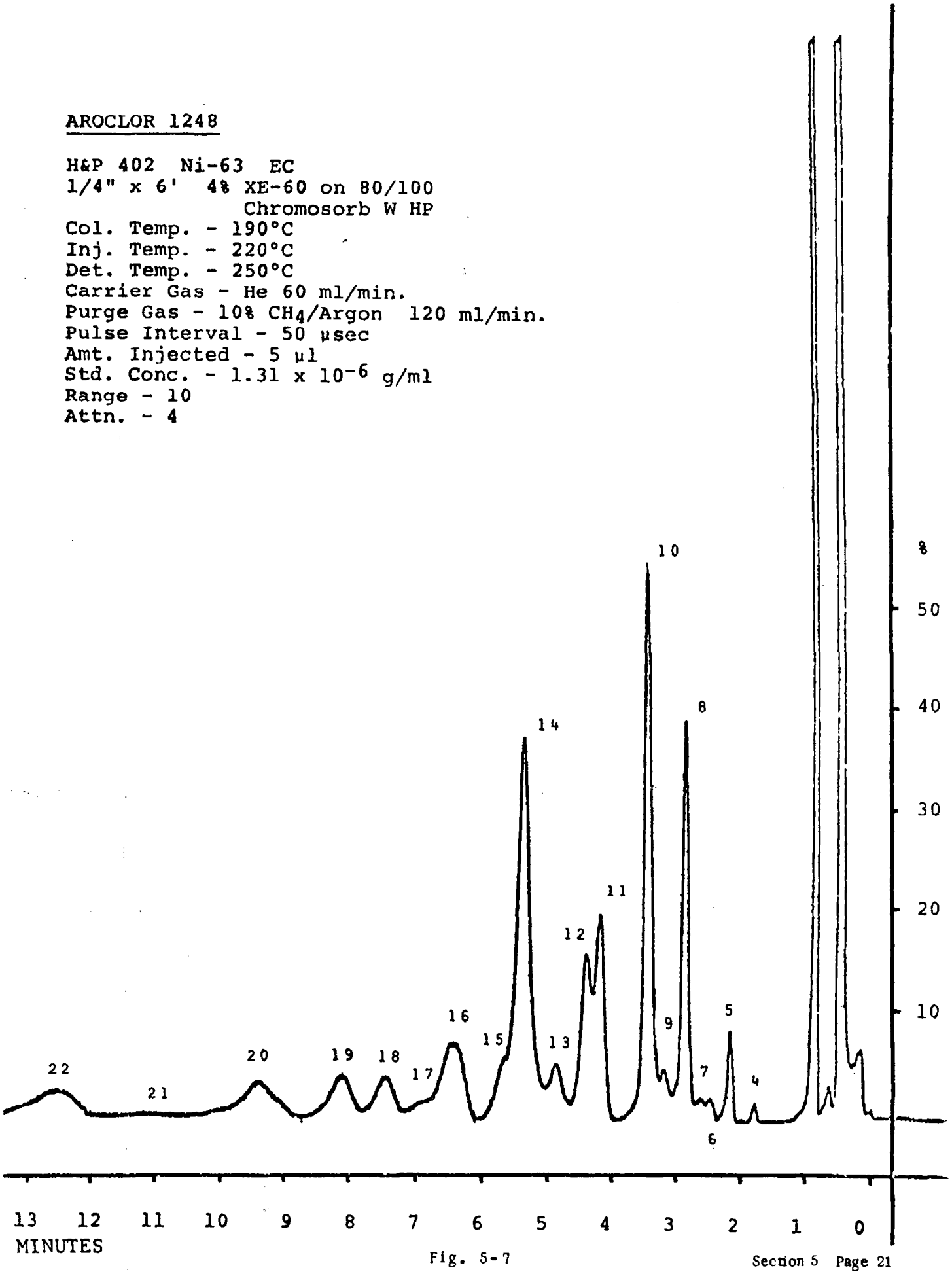

Fig. 5-7

DSW 010255

STLCOPCB4001708

AROCLOR 1254

H&P 402 Ni-63 EC
1/4" x 6' 4% XE-60 on 80/100
Chromosorb W HP
Col. Temp. - 205°C
Inj. Temp. - 220°C
Det. Temp. - 250°C
Carrier Gas - He 60 ml/min.
Purge Gas - 10% CH_4/Argon 120 ml/min.
Pulse Interval - 50 µsec
Amt. Injected - 5 µl
Std. Conc. - 1.02×10^{-6} g/ml
Range - 10
Attn. - 8

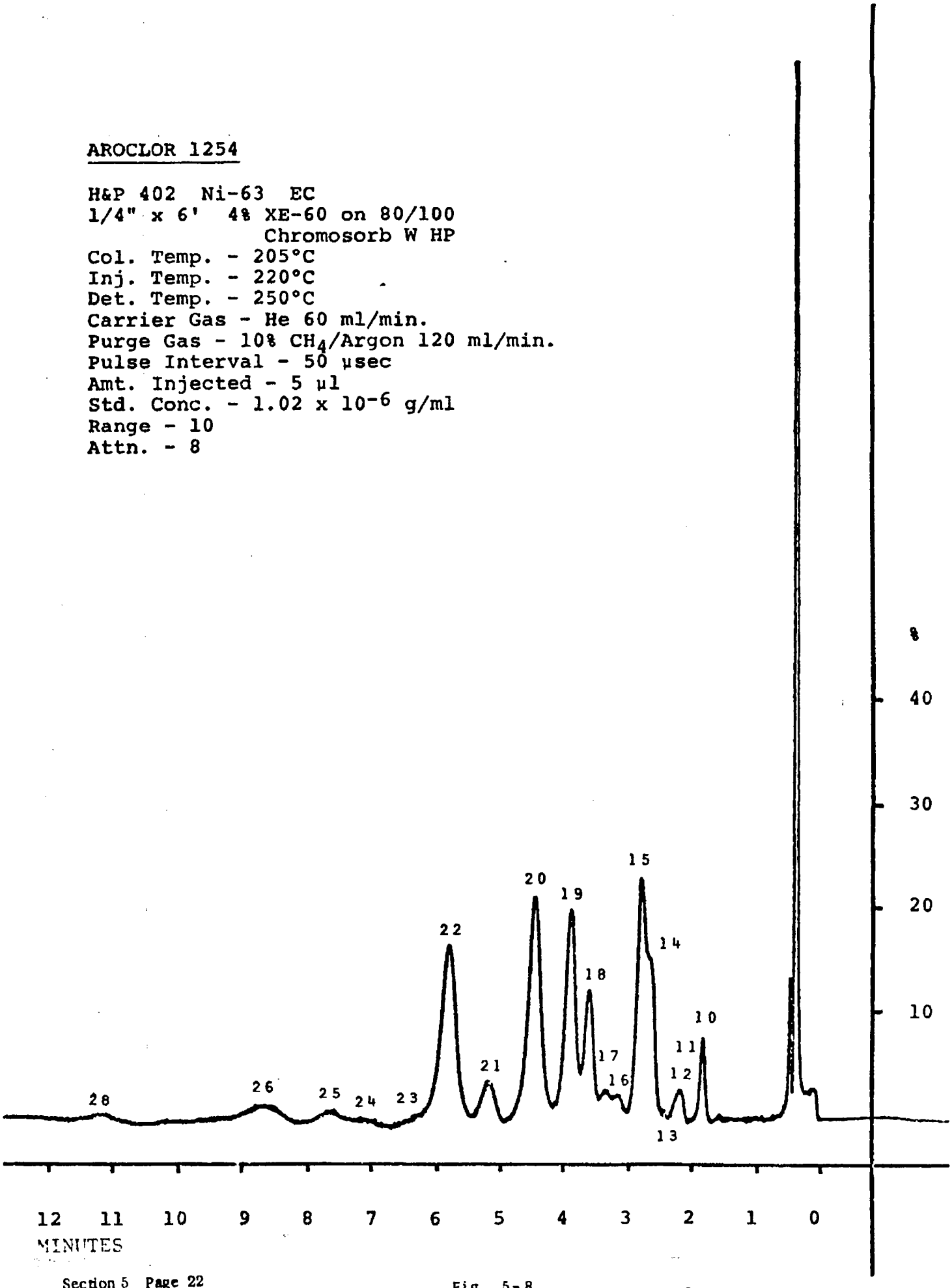

Fig. 5-8

DSW 010256

STLCOPCB4001709

AROCLOR 1260

H&P 402 Ni-63 EC
1/4" x 6' 4% XE-60 on 80/100
Chromosorb W HP
Col. Temp. - 220°C
Inj. Temp. - 220°C
Det. Temp. - 250°C
Carrier Gas - He 60 ml/min.
Purge Gas - 10% CH_5/Argon 120 ml/min.
Pulse Interval - 50 µsec
Amt. Injected - 5 µl
Std. Conc. - 0.98×10^{-6} g/ml
Range - 10
Attn. - 8

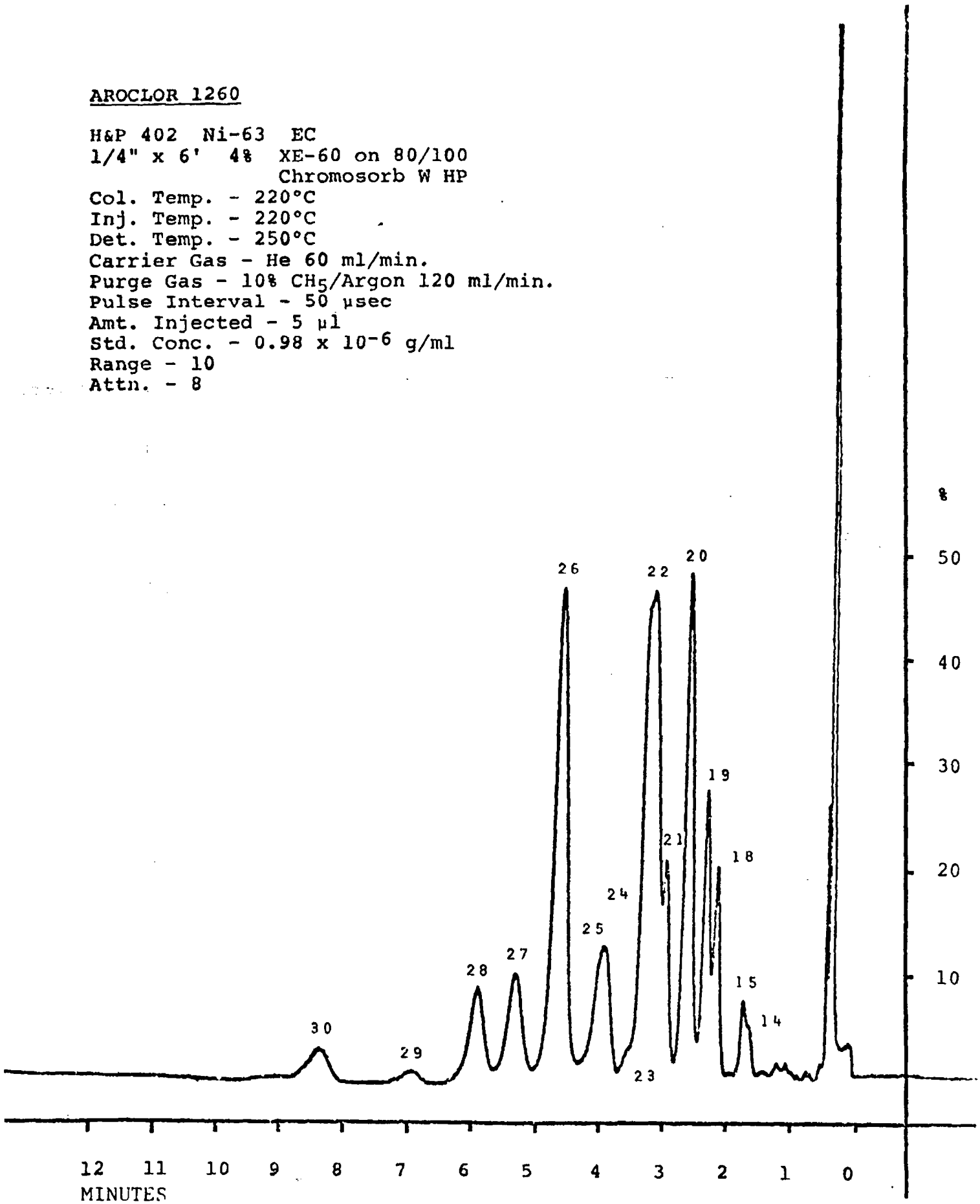

Fig. 5-9

DSW 010257

STLCOPCB4001710

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Colt Industries
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Transformer Division
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Dowzer Electric Inc. †
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DSW 010258

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† Member of Transformer Section.

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