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American National Standard

guidelines for handling and disposal of capacitor- and transformer-grade askarels containing polychlorinated biphenyls

C107.1-1974

american national standards institute, inc
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American National Standard Guidelines for Handling and Disposal of Capacitor- and Transformer-Grade Askarels Containing Polychlorinated Biphenyls

Secretariat

National Electrical Manufacturers Association

Approved January 9, 1974

American National Standards Institute, Inc

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American National Standard

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Foreword

(This Foreword is not a part of American National Standard Guidelines for Handling and Disposal of Capacitor- and Transformer-Grade Askarels Containing Polychlorinated Biphenyls, C107.1-1974.)

Recognizing that the polychlorinated biphenyls present in askarels were being identified as environmental pollutants and the urgent need for procedures and guides for their safe use and disposal, the Board of Directors of the Power Equipment Division of the National Electrical Manufacturers Association (NEMA) in June 1970 appointed a committee to review the general problem and propose recommendations. In September 1970, the NEMA board approved, as recommended by the committee, the sponsorship of an American National Standards Committee for the use and disposal of askarels in electrical equipment.

American National Standards Committee C107 was established in April 1971. NEMA was designated as the secretariat. An organizational meeting was held on September 14, 1971. Mr W. B. Papageorge, Monsanto Company, was appointed chairman. Committee members included representatives of capacitor and transformer manufacturers, utilities, governmental departments and agencies, and maintenance and disposal service companies. A steering committee and two subcommittees – one to develop guidelines for the capacitor industry, the other for the transformer industry – were established.

The scope and primary objective of the committee was designated as the development of: Procedures and guides for the safe use, maintenance, and disposal of askarel and askarel-soaked materials used in electrical equipment.

Additional objectives proposed were as follows:

(1) To serve as a source for technical information and advice for federal, state, and local authorities and for the information of all others concerned.

(2) To encourage the development of suitable disposal facilities and maintain a list of their capabilities and locations, for the information of all concerned.

(3) To serve as the advisory group for U.S. participation in the International Commission on Rules for the Approval of Electrical Equipment (CEE), the International Electrotechnical Commission (IEC), the Pan American Standards Commission (COPANT), the International Conference on Large High Tension Electric Systems (CIGRE), and other international organizations.

The final draft of the guidelines was completed in late November 1972. NEMA issued the guidelines as an Official Standards Proposal in January 1973. It was submitted to the American National Standards Institute for approval as an American National Standard and was approved by the Standards Institute on January 9, 1974.

Suggestions for improvement of this standard will be welcome. They should be sent to the American National Standards Institute, 1430 Broadway, New York, N.Y. 10018.

This standard was processed and approved for submittal to ANSI by American National Standards Committee on Use and Disposal of Askarel and Askarel-Soaked Materials in Electrical Equipment, C107. Committee approval of the standard does not necessarily imply that all committee members voted for its approval. At the time it approved this standard, the C107 Committee had the following members:

W. P. Papageorge, Chairman
A. M. Salazar, Secretary

Organization Represented	*Name of Representative*
Certified Ballast Manufacturers Association	N. R. Clark
	A. Poxefsky
Chem-Trol Pollution Services Inc	Louis E. Wagner
Doble Engineering Company	A. L. Rickley
Electrical Utilities Company	A. O. Hauser
	E. M. Moore (Alt)
Electric Light and Power Group	F. R. Lengefeld
	H. A. Onithi
	J. J. Cawley (Alt)

0071022

HARTOLDMON0030128

Organization Represented	*Name of Representative*
Electronic Industries Association	Arnold S. Doty
Environmental Protection Agency	Kenneth J. Hood
General Services Administration	Charles C. Travis
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National Bureau of Standards	Stanley P. Wasik
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Tennessee Valley Authority	William R. Nicholas
U.S. Department of Agriculture, Rural Electrification Administration	John Leutritz, Jr
U.S. Department of the Army	D. M. Crabtree

0071023

HARTOLDMON0030129

Contents

SECTION PAGE

1. Scope . . . 7

2. General Information . . . 7
   2.1 General . . . 7
   2.2 Benefits . . . 7
   2.3 Risks . . . 7
   2.4 Alternatives . . . 8
   2.5 Interdepartmental Task Force on PCBs . . . 8

3. Capacitor Guidelines . . . 8
   3.1 General . . . 8
   3.2 Capacitor-Grade Askarel . . . 9
   3.3 Plant Housekeeping and Employee Safety . . . 9
   3.4 Control of Water Effluents . . . 11
   3.5 Scrap-Disposal Procedures . . . 12
   3.6 Labeling . . . 13

4. Transformer Guidelines . . . 13
   4.1 General . . . 13
   4.2 Specific Guidelines . . . 15

5. References . . . 17
   5.1 References to the Text . . . 17
   5.2 General References . . . 17

6. Revision of American National Standards Referred to in This Document . . . 18

Table 1 Typical Properties of Aroclor 1016 . . . 9

Appendixes

Appendix A Disposal Services . . . 19

Appendix B Analytical Procedures and Laboratory Service Organizations . . . 20
   B1. General . . . 20
   B2. Laboratories . . . 20
   B3. An Analytical Procedure for the Determination of Airborne PCBs . . . 20
   B4. Analysis of Water and Sediment for PCBs . . . 31

Figures

Fig. B1 Aroclor 1016 Electron Capture Chromatogram . . . 21
Fig. B2 Comparison of Electron Capture Chromatograms for Aroclor 1221, 1242, 1248, 1254, and 1260 . . . 22
Fig. B3 Aroclor 1221 Electron Capture Chromatogram . . . 23
Fig. B4 Aroclor 1242 Electron Capture Chromatogram . . . 24
Fig. B5 Aroclor 1248 Electron Capture Chromatogram . . . 25
Fig. B6 Aroclor 1254 Electron Capture Chromatogram . . . 26
Fig. B7 Aroclor 1260 Electron Capture Chromatogram . . . 27
Fig. B8 Sampling Train . . . 28
Fig. B9 Calculating Column Efficiency . . . 30
Fig. B10 Calculating the Tailing Factor . . . 30

0071024

HARTOLDMON0030130

American National Standard Guidelines for Handling and Disposal of Capacitor- and Transformer-Grade Askarels Containing Polychlorinated Biphenyls

1. Scope

This standard establishes guidelines for the safe use, maintenance, and disposal of askarel and askarel-soaked material used in capacitors and transformers.

2. General Information

2.1 General. The term "askarel" generally describes a broad class of nonflammable synthetic chlorinated hydrocarbon insulating liquids widely used in capacitors, transformers, reactors, and accessory equipment operated at power frequencies.

Askarels consisting of or containing polychlorinated biphenyls (PCBs) have been used in many applications for more than 40 years, but only recently was evidence discovered that PCBs are widely dispersed in the environment. Systematic investigations of the biological effects of PCBs have been undertaken within the past few years to establish the effects of specific formulations upon specific species. Some studies have shown that PCBs may be an environmental contaminant. Simultaneously, significant steps have been taken by U.S. industry to limit further releases of PCBs to the environment.

PCBs have been used in three broad types of applications for the past 40 years, as follows:

(1) "Open-ended" applications; for example, in paints, specialty inks, paper coatings, plastics, etc

(2) "Nominally closed" applications; for example, as the working fluid in hydraulic or heat-transfer systems

(3) "Closed electrical system" applications, specifically as the insulating fluid in certain kinds of transformers and capacitors[1]

[1] The Monsanto Company is the sole U.S. producer of PCBs. It has discontinued supplying the material for all applications given in 2.1(1) and 2.1(2).

Evaluations of the benefits, risks, and alternatives involved in the continued use of PCBs in closed electrical systems are summarized in 2.2 through 2.4.

2.2 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% 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 (for example, many types are today less than 14% of the size of equivalent oil capacitors and have a life expectancy of 10 to more than 20 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.

2.3 Risks. In the United States, medical records over a nearly 40-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 nonchronic 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. Although certain types of equipment failures can permit loss of some askarel to the environment, transformer failures are limited to approximately 0.02% of the units in service per year. With respect to capacitors, such losses are limited to approximately 0.02% of the askarel put into service per year. In addition, limited amounts of PCBs can get into the environment during the manufacture, delivery, improper use, maintenance, repair, and disposal of transformers and capacitors.

0071025

HARTOLDMON0030131

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

2.4 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, although many of the foregoing limitations 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 that 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 PCBs, there was a strong economic incentive to find other, less expensive insulating liquids with the desirable characteristics of askarels. Since the 1930s at least ten major chemical or electrical companies have invested large amounts of time and money in this search, all with no success. Although potential substitutes that are more costly than askarels (such as fluorinated liquids) have also received some consideration, 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.

2.5 Interdepartmental Task Force on PCBs. An in-depth study of PCBs has recently been completed by five Executive Branch Departments of the federal government. This Interdepartmental Task Force on PCBs issued their report, entitled *Polychlorinated Biphenyls and the Environment,* in May 1972.[2] The following conclusion is quoted from page 4 of this report:

"The use of PCBs 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 PCBs 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 PCBs to all except manufacturers of electrical transformers and capacitors."

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

3. Capacitor Guidelines

3.1 General. 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 40 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 bird life. 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 Section 3 of this standard is intended to apply to capacitor-grade askarel.

[2] Available from the National Technical Information Service, U.S. Department of Commerce, Springfield, Va 22151.

0071026

HARTOLDMON0030132

Table 1
Typical Properties of Aroclor 1016

Property	Test Method (See Note)	Typical Values
Color	APHA*	40, max
Condition	–	Clear
Specific Gravity at 25/15.5°C	ANSI C59.68-1965 (R1973) [2]	1.362–1.372
Acidity (mg KOH/g)	ANSI Z11.131-1964 (R1974) [3] ANSI Z11.59-1958 (R1971) [4]	0.010, max
Moisture	–	35 ppm, max
Refractive index at 25°C	ASTM D 1817-66 (1972) [5]	1.6215–1.6235
Inorganic (free) chlorides	ANSI C59.55-1963 (R1973) [6]	0.05 ppm, max
Pour point	ANSI Z11.5-1966 (R1972) [7]	−14°C or lower
Dielectric constant (1000 Hz at 100°C)	ANSI C59.22-1967 (R1973) [8]	4.70–4.90
Resistivity (500 V dc at 100°C, 0.1-inch gap)	ANSI C59.51-1965 (R1973) [9]	500 × 10^9 Ω · cm, min
Hydrolysis stability test (as chlorides)	ANSI C59.106-1970 [10]	0.5 ppm, max
Thermal stability test (as chlorides)	ANSI C59.111-1970 [11]	0.4 ppm, max
Distillation range (corrected) 10% distilled by weight 90% distilled by weight	ANSI A37.9-1974 [12]	 323°C, min 356°C, max
Higher-Boiling Homologues	ASTM D 3303-74 [1]	0.4%, max
Sulfates	ANSI C59.2-1974 [13]	None
Dielectric strength at 25°C	ANSI C59.19-1968 (R1973) [14]	35 kV, min
Flash point, Cleveland open cup	ANSI Z11.6-1973 [15]	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%
Viscosity at 100°F (SUS)†	ANSI Z11.2-1956 (R1971) [16]	71–81
Specific heat at 25°C	–	0.30
Coefficient of expansion	ANSI C59.57-1963 (R1973) [17]	0.00068 $cm^3/cm^3/°C$
Fixed chlorine	Carius	41.3 ± 0.5%
Power factor at 100°C, 60 Hz Bulk Drums	Munch [18]	 1%, max 4%, max

NOTE: Numbers in brackets refer to correspondingly numbered test methods in 5.1, References to the Text.

*American Public Health Association.

†Saybolt Universal seconds.

3.2 Capacitor-Grade Askarel. In September 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% by weight of the higher-boiling homologues of the chlorinated biphenyls. (See ASTM D 3303-74 [1].)[3] Aroclor 1016 replaces Aroclor 1242, which previously was the major capacitor impregnant and contained around 7% of the higher-boiling homologues (the more persistent in nature). This 0.4% level of the higher-boiling homologues 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 Underwriters' Laboratories, Inc, nonflammability rating as Aroclor 1242. Its typical properties are given in Table 1.

3.3 Plant Housekeeping and Employee Safety. The procedures and limits given in 3.3 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

[3] Numbers in brackets refer to corresponding numbers in 5.1, References to the Text.

0071027

HARTOLDMON0030133

with exposure limits and indicated antidotes and clean-up procedures.

3.3.1 **Material.** Askarel for use in capacitors should consist of homologues and isomers of chlorinated biphenyl with the concentration of the higher-boiling homologues at about 0.4%. (See ASTM D 3303-74 [1].) Aroclor 1016 is considered to be the standard impregnant meeting these requirements.

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

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

At 25°C and 760 mmHg pressure, saturated air contains approximately 0.09 mg/l.

NOTE:
1 mg/l = 90.0 ppm (v/v)
1 ppm (v/v) = 0.011 mg/l

3.3.2 **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 3.5). 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 3.5.

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

3.3.3.1 **Vapors.** The odor of askarel is noticeable well below the maximum air concentrations considered safe. Up to 1.0 milligram per cubic metre of air has been determined to be the maximum safe level of exposure during an 8-hour workday. (See reference [19].) The procedure for performing the necessary analyses is contained in Section B3 of Appendix B. This procedure or its equivalent should be used.

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 temperatures of 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 effecting 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 U.S. Bureau of Mines approval and be maintained on a regular schedule in accordance with the manufacturer's recommendation.

3.3.3.2 **Liquid.** In contrast to the situation in which mineral insulating oils are handled, 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 that can absorb and retain askarels is to be avoided. Barrier creams[4] or resistant gloves[5] 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 aska-

[4] For example, PLY No. 9 Gel (Milburn Company, Detroit, Mich), or Kerodex No. 71 (Ayerst Laboratories, New York, N.Y.), or the equivalent.

[5] For example, Edmont – Solvit 5-352 (Mersick of Bridgeport, Bridgeport, Conn), or the equivalent.

0071028

HARTOLDMON0030134

rel 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.

3.3.4 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.

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 3.5.

Wiper rags, clothing, and other extraneous materials saturated with askarels should be collected within the containment area for properly controlled laundering or disposal (see 3.5).

3.3.5 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 3.5, and should include (but not be limited to) the following wastes:

(1) Contaminated liquid askarel that is unsuitable for reclaiming as a dielectric fluid

(2) Liquid askarel from solvent operations or water and 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

3.3.6 Miscellaneous Procedures. Other safety considerations include the following:

(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 given in 3.3.6(1) and 3.3.6(2), finally being disposed of by one of the means outlined in 3.5.

3.4 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 standard 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.

3.4.1 Concentration Limits. The 1972 Environmental Protection Agency proposals are to keep PCB levels in rivers and lakes below 0.01 part per billion. This is currently under review by the EPA and standards are expected to be promulgated in 1974. Plant effluent streams should be managed and controlled in a manner anticipating these government standards.

3.4.2 Monitoring Streams. On a regular basis consistent with plant situations, all effluent streams should be analyzed. The procedure for performing the necessary analyses is contained in Section B4 of Appendix B. This procedure or its equivalent should be used.

3.4.3 Methods for Minimizing Effluent Stream Contamination. The ideal approach is to isolate totally 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 tech-

0071029

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niques that 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.

3.5 Scrap-Disposal Procedures. The manufacture and use of capacitors involve processes that produce askarel-saturated solids and liquids containing or composed entirely of askarel, which should be disposed of as wastes. Specific sources of these materials are described throughout this standard. They 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 that is consistent with proper concern for the environment and minimizes any release of askarels to the environment.

3.5.1 Disposal of Capacitor Units. Scrap capacitor units can be generated during manufacturing processes or during field service.

Production rejects are those capacitors that 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 that are rejected or, for other reasons, are to be scrapped after shipment from the plant where they were manufactured.

3.5.1.1 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 that meet all applicable state requirements.

Care should be exercised to ensure 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.

3.5.1.2 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% of the higher-boiling homologues. (See ASTM D 3303-74 [1].)

Large capacitors (those incorporating more than 2 pounds of askarel) should be disposed of according to the procedure for production rejects (see 3.5.1.1).

3.5.2 Disposal of Liquid Wastes. All waste askarel or liquid wastes containing askarel should be disposed of in accordance with one of the procedures given in 3.5.2.1 through 3.5.2.3.

3.5.2.1 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 that will be formed; for example, 2-second dwell time at 2000°F and 3% excess oxygen in stack gas, or 1.5-second dwell time at 2700°F and 2% 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 standard.

3.5.2.2 Toxic and Hazardous Waste Disposal Sites. Certain landfill sites have been classified by state governments and the federal government 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 standard. (See Appendix A.)

3.5.2.3 Packaging and Shipment

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

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

3.5.2.3.3 Containers used for this purpose should not be used for any such materials or retired from service until they are completely cleaned. Any

0071030

HARTOLDMON0030136

solvents used in cleaning these containers will be contaminated with askarel and should be disposed of according to the same procedures described in 3.5.2.

3.5.3 Disposal of Solid Wastes. (See 3.5.1 for scrap capacitors.) All solid wastes that have been saturated with askarel should be disposed of by the following procedure:

(1) The saturated wastes should be placed into leakproof 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 3.5.2. (See Appendix A for a listing of facilities.)

3.6 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 methods of providing disposal instructions for small capacitors and large capacitors are treated separately in 3.6.1 and 3.6.2, respectively.

3.6.1 Small Units. Small capacitors are defined as those that contain askarel in quantities up to about 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 large piece of equipment. Attaching a label to such equipment referencing this standard or describing disposal procedures would be of limited practical value.

3.6.2 Large Units. Large capacitor units are defined as those that contain more than 2 pounds of askarel. The capacitor manufacturer should affix a label in a conspicuous place, referencing this standard 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 PCBs. See American National Standard C107.1-1974 for further information. Copies are available from American National Standards Institute, 1430 Broadway, New York, N.Y. 10018.

4. Transformer Guidelines

4.1 General

4.1.1 Types of Transformer Askarels.[6] Askarels of various compositional types are currently in use (for the general properties and types, see ASTM D 2283-73a [20]). Under arcing conditions, the gases produced, though predominantly consisting of noncombustible hydrogen chloride, can contain varying amounts of combustible gases depending upon the askarel type.

4.1.2 Safety Precautions. Based on about 40 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 that simple precautions are taken, exposure should still be avoided or minimized.

4.1.2.1 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 metre of air has been determined to be the upper safe level of exposure during an 8-hour workday. (See reference [19].) The procedure for performing the necessary analyses is contained in Section B3 of Appendix B. This procedure or its equivalent should 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 effecting rescue in case of accident. If the odor of askarel or its arced products is detected

[6] The following trademarks are among those employed by electrical manufacturers to designate the askarels used in their products: Asbestol, Chlorextol, Inerteen, No-Flamol, Pyranol, and Saf-T-Kuhl.

0071031

HARTOLDMON0030137

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

4.1.2.2 Liquid. In contrast to the situation in which mineral insulating oils are handled, 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 that can absorb and retain askarels is to be avoided. Resistant gloves and aprons of the neoprene, polyethylene, or fluoroelastomer[1] 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 that 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 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.

4.1.3 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 (PCBs). 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.

4.1.4 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.

4.1.4.1 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.

4.1.4.2 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 drums labeled "SCRAP ASKAREL" for disposition.

4.1.4.3 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 that 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.

4.1.5 Control of Water Effluents. 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 standard 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.

4.1.5.1 Concentration Limits. The 1972 Environmental Protection Agency proposals are to keep PCB levels in rivers and lakes below 0.01 part per billion. This is currently under review by the EPA and standards are expected to be promulgated in 1974. Plant effluent streams should be managed and controlled in a manner anticipating these government standards.

4.1.5.2 Monitoring Streams. All plant effluent streams should be monitored on a regular basis, consistent with plant situations. The procedure for performing the necessary analyses is contained in Section B4 of Appendix B. This procedure or its equivalent should be used.

[1] For example, Viton.

0071032

HARTOLDMON0030138

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

4.1.6 Disposal Procedures and Services

4.1.6.1 Sources of Materials Requiring Special Handling and Disposal Procedures. Liquids containing PCBs and solids containing or contaminated with PCBs 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.

4.1.6.2 Classification for Disposal of Materials Containing PCBs. In general, there are three types of materials requiring disposal: liquids, burnable solid materials containing PCBs, and nonburnable solid materials contaminated with PCBs.

4.1.6.2.1 Liquids. Liquids containing PCBs requiring disposal by high-temperature incineration may consist of the following:

(1) PCBs contaminated with mineral oil.

(2) Mineral oil contaminated with PCBs.

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

4.1.6.2.2 Burnable Solid Waste Materials Containing PCBs. 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.

4.1.6.2.3 Nonburnable Solid Waste Materials Containing, or Contaminated with, PCBs. 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 PCBs can be accomplished by washing or solvent extraction with kerosene or other approved washing liquids such as perchloroethylene or tricholoroethylene. Accumulated liquids can be disposed of as indicated in 4.1.6.2.1. Solid materials may be handled as normal scrap.

4.1.6.3 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.

4.1.6.4 Shipment of Burnable Solid Waste Material Containing PCBs 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.

4.1.6.5 Liquid and Solid Waste Disposal Service Organizations

4.1.6.5.1 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 incineration plus oxygen availability and, finally, suitable scrubbers to remove the HCl that will be formed; for example, 2-second dwell time at 2000°F and 3% excess oxygen in stack gas, or 1.5-second dwell time at 2700°F and 2% 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 standard.

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

4.1.6.5.2 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.

4.1.6.5.3 Disposal Services. Organizations offering disposal services are listed in Appendix A, 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.

4.2 Specific Guidelines

4.2.1 Plant 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 cleanup to prevent contamination.

4.2.1.1 Askarel Filling Area

4.2.1.1.1 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.

4.2.1.1.2 The main manufacturing area for filling equipment with askarel should be provided with impervious surface floors or suitable basins so con-

0071033

HARTOLDMON0030139

structed that any inadvertent leakage or spills are prevented from reaching streams, sanitary sewers, or storm sewers. All askarel-handling equipment, such as pumps, hoses, etc, shall be of the askarel-resistant type.

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

4.2.1.2 Special Containers for Scrap Materials

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

4.2.1.2.2 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.

4.2.1.2.3 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.

4.2.1.2.4 Containers for supplies of material for absorbing small askarel spills or cleanup of larger spills should be provided.

4.2.1.3 Conditioning of Askarels

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

4.2.1.3.2 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 drain thoroughly 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.

4.2.1.4 Teardown of Units for Repair or Scrap

4.2.1.4.1 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.

4.2.1.4.2 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.

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

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

4.2.2 Transformer Labeling

4.2.2.1 New Transformers. All new transformers that contain PCBs 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 (PCBs). Care should be taken to prevent entry into the environment. In case of malfunction or leaks, consult the instruction manual or the manufacturer.

4.2.2.2 In-Service Transformers. The transformer manufacturer should make available suitable labels with a similar warning as shown in 4.2.2.1 for use on existing transformers.

4.2.3 Information for Transformer Users

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

4.2.3.2 Transportation and Receiving. Immediately upon receipt of the equipment and following any transportation or handling accident that could affect the integrity of the tank, bushings, 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 cleanup 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 4.1.6.

4.2.3.3 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.

4.2.3.4 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 an askarel unit or to treat an askarel unit with fuller's earth, 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.

4.2.3.5 Sampling. It is common practice to sample askarel from a transformer for periodic maintenance testing. As previously described, such samples should be

0071034

HARTOLDMON0030140

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.

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

(1) Complete drainage and dismantling with the proper disposal of the askarel and askarel-soaked components as described in 4.1.6.

(2) Disposition of askarel transformers by means of junk or scrap dealers. This 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 in 4.1.6.

5. References

5.1 References to the Text

[1] Standard Method of Test for Rapid Gas Chromatographic Estimation of Higher Boiling Homologues of Chlorinated Biphenyls for Capacitor Askarels, ASTM D 3303-74

[2] American National Standard Method of Test for Specific Gravity of Askarels, C59.68-1965 (R1973) (ASTM D 1810-63 (1973))

[3] American National Standard Method of Test for Neutralization Number by Color-Indicator Titration, Z11.131-1964 (R1974) (ASTM D 974-64 (1973), IP 139/65)

[4] American National Standard Method of Test for Neutralization Number by Potentiometric Titration, Z11.59-1958 (R1971) (ASTM D 664-58 (1968), IP 177/64)

[5] Standard Method of Test for Density of Rubber Chemicals, ASTM D 1817-66 (1972)

[6] American National Standard Method of Test for Inorganic Chlorides in Askarels, C59.55-1963 (R1973) (ASTM D 1821-63 (1973))

[7] American National Standard Method of Test for Pour Point of Petroleum Oils, Z11.5-1966 (R1972) (ASTM D 97-66 (1971), IP 15/67)

[8] American National Standard Method of Test for Power Factor and Dielectric Constant of Electrical Insulating Liquids, C59.22-1967 (R1973) (ASTM D 924-65 (1973))

[9] American National Standard Method of Test for Specific Resistance (Resistivity) of Electrical Insulating Liquids, C59.51-1965 (R1973) (ASTM D 1169-64 (1973))

[10] American National Standard Method of Test for Hydrolyzable Chlorine Compounds in Chlorinated Aromatic Hydrocarbons (Askarels), C59.106-1970 (ASTM D 1820-65 (1971))

[11] American National Standard Method of Test for Thermal Stability of Chlorinated Aromatic Hydrocarbons (Askarels), C59.111-1970 (ASTM D 1936-64 (1971))

[12] American National Standard Method of Test for Distillation of Road Tars, A37.9-1974 (ASTM D20-72)

[13] American National Standard Methods of Testing Electrical Insulating Oils, C59.2-1974 (ASTM D 117-71 (1973))

[14] American National Standard Method of Test for Dielectric Breakdown Voltage of Insulating Liquids Using Disk Electrodes, C59.19-1968 (R1973) (ASTM D 877-67 (1971))

[15] American National Standard Method of Test for Flash and Fire Points by Cleveland Open Cup, Z11.6-1973 (ASTM D 92-72, IP 36/67)

[16] American National Standard Method of Test for Saybolt Viscosity, Z11.2-1956 (R1971) (ASTM D 88-56 (1973))

[17] American National Standard Method of Test for Coefficient of Thermal Expansion of Electrical Insulating Liquids of Petroleum Origin, and Askarels, C59.57-1963 (R1973) (ASTM D 1903-63 (1973))

[18] MUNCH, R. H. Measuring the dissipation factor, dielectric constant, and resistivity of liquids. *Insulation/Circuits*, vol 16, Mar 1970, pp 46–49.

[19] Chlorodiphenyls. *Hygienic Guide Series.* Westmont, N.J.: American Industrial Hygiene Association, Jan-Feb 1965.

[20] Standard Specification for Chlorinated Aromatic Hydrocarbons (Askarels) for Transformers, ASTM D 2283-73a

5.2 General References

Aroclor® Polychlorinated Polyphenyls (Biphenyls), Technical Bulletin O-FF/1R. St. Louis: Monsanto Industrial Chemicals Company, Nov 1971.

DRINKER, C. K. Further observations on the possible systemic toxicity of certain of the chlorinated hydrocarbons. *Journal of Industrial Hygiene and Toxicology,* vol 21, 1939, pp 155–159.

0071035

HARTOLDMON0030141

DRINKER, C. K.; WARREN, M. F.; and BENNET, G. A. The problem of possible systemic effects from certain chlorinated hydrocarbons. *Journal of Industrial Hygiene and Toxicology*, vol 19, 1937, pp 283-311.

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GREENBURG, L.; MAYERS, M. R.; and SMITH, A. R. The systemic effects resulting from exposure to certain chlorinated hydrocarbons. *Journal of Industrial Hygiene and Toxicology*, vol 21, 1939, pp 29-38.

Threshold Limit Values for Chemical Substances and Physical Agents in the Workroom Environment. Cincinnati: American Conference of Governmental Industrial Hygienists, 1973.

TREON, J. F.; CLEVELAND, F. P.; CAPPEL, J.; and ATCHLEY, R. W. The toxicity of the vapors of Aroclor 1242 and Aroclor 1254. *American Industrial Hygiene Association Quarterly*, vol 17, 1956, pp 204-213.

6. Revision of American National Standards Referred to in This Document

When the American National Standards referred to in this document are superseded by a revision approved by the American National Standards Institute, Inc, the revision shall apply.

0071036

HARTOLDMON0030142

Appendixes

(These Appendixes are not a part of American National Standard Guidelines for Handling and Disposal of Capacitor- and Transformer-Grade Askarels Containing Polychlorinated Biphenyls, C107.1-1974, but are included for information purposes only.)

Appendix A
Disposal Services

In addition to the supervised dry landfill sites that 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 Pollution Services, Inc
P.O. Box 200
1550 Balmer Road
Model City, N.Y. 14107
Phone: 716 754-8231

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 North Lindbergh Boulevard
St. Louis, Mo 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°F-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, Ky 40351
Phone: 606 784-6611

Nuclear Engineering Company
Disposal Division
Sheffield, Ill. 61361
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. Licensed by Atomic Energy Commission for radioactive waste disposal. The organization also has two West Coast locations, in the states of California and Washington.

Rollins Environmental Services, Inc
P.O. Box 2349
Wilmington, Del 19899
Phone: 302 658-8451

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 are removed before exhausting to air.

Rollins Environmental Services maintains disposal facilities in the following areas:

Philadelphia/Camden:
Rollins Environmental Services, Inc
Route 322
Logan Township
Bridgeport, N.J. 08014

* The listing given herein is representative of some of the sources that provide this service, and is not presumed to be complete. Any other organizations that wish to be listed should notify the Standards Institute so that they may be included in the next edition of this standard.

0071037

HARTOLDMON0030143

Baton Rouge:
Rollins Environmental Services, Inc
Scenic Highway & West Cheatham Lane
Scotlandville
East Baton Rouge Parish, La 70807

Houston:
Rollins Environmental Services, Inc
Tidal Road & Highway 134
Deer Park, Tex 77536

Appendix B
Analytical Procedures and Laboratory Service Organizations

B1. General

Analytical procedures for determining PCBs in air, water, and sediments are given in Sections B3 and B4.

B2. Laboratories

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

Carus Chemical Corporation
1375 Eighth Street
La Salle, Ill. 61301
Phone: 815 223-1500

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

Gollob Analytical Service Corporation
47 Industrial Road
Berkeley Heights, N.J. 07922
Phone: 201 464-3331

B3. An Analytical Procedure for the Determination of Airborne PCBs

B3.1 Scope. This procedure is based on techniques used by the Monsanto Industrial Chemical Company for the isolation and determination of PCBs 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 for further identification of gas chromatography fractions.

B3.2 Principle. Airborne PCBs are absorbed in toluene by drawing the air through one or more fritted bubblers or impingers in cylinders filled with toluene. After a suitable amount of air is sampled, 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 PCBs present are determined by electron capture gas chromatography (EC/GC).

B3.3 Reagents

(1) Hexane: Pesticide grade.

(2) Toluene: Pesticide grade.

(3) Sodium sulfate: Anhydrous, granular, analytical reagent (AR) grade. Heat at 400°C for 1 hour prior to use.

(4) Alumina adsorption: For chromatographic analysis, 80/200 mesh. Heat at 400°C for a minimum period of 4 hours and deactivate with 5% (w/w) distilled water.

For 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 (about 30 grams) of deactivated alumina, and pour this slowly into the column. Tap or vibrate the column to settle the alumina, and top the alumina with 2 to 3 cm of anhydrous sodium sulfate. Wash the column with 50 to 100 ml of hexane prior to the addition of the sample.

(5) Distilled water: Extracted with hexane to re-

*The listing given herein is representative of some of the sources that provide this service, and is not presumed to be complete. Any other organizations that wish to be listed should notify the Standards Institute so that they may be included in the next edition of this standard.

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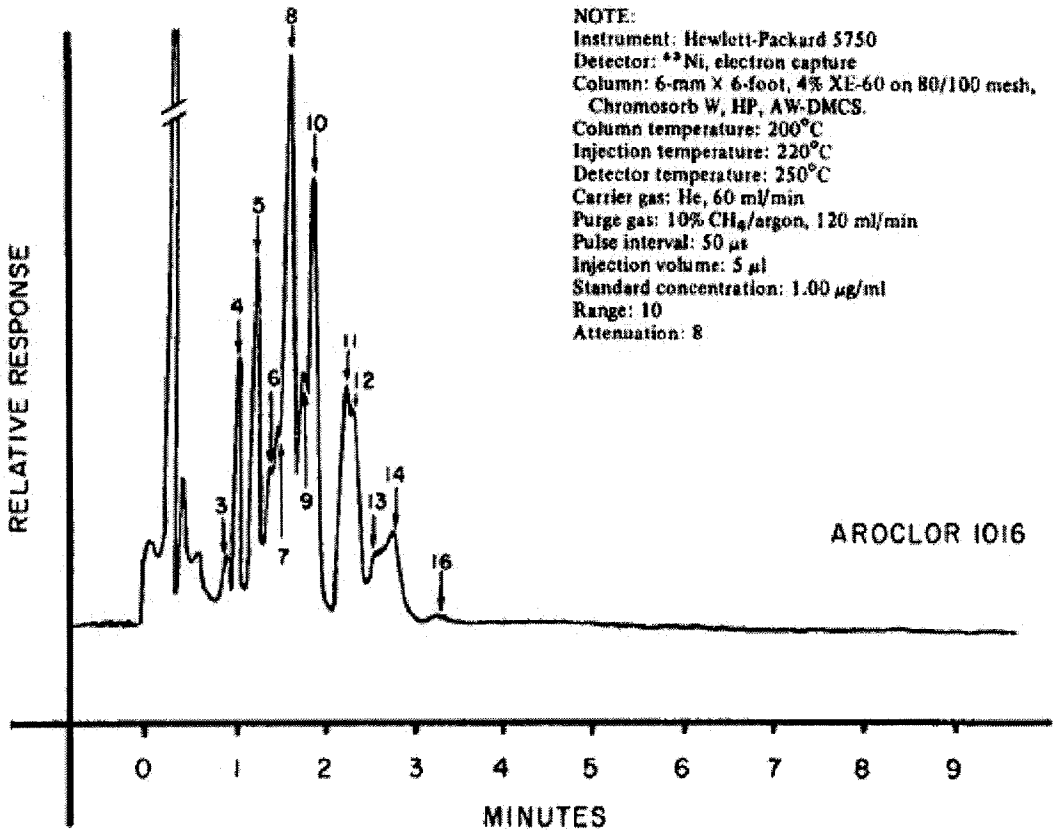

Fig. B1
Aroclor 1016 Electron Capture Chromatogram

move hexane-soluble electron capturing impurities.

(6) Sulfuric acid: AR grade, specific gravity = 1.84.

(7) Potassium hydroxide: AR grade.

(8) Ethanol: Formula 2B.

(9) Alcoholic potassium hydroxide, 2.5% (w/v): Dissolve about 12.5 grams of AR grade KOH in 500 ml of ethanol.

(10) Sulfuric acid–water, 9:1 (v/v): Carefully add 270 ml of AR grade sulfuric acid to 30 ml of distilled water in a 500-ml iced beaker.

(11) PCB standards: Aroclor 1016, 1221, 1242, 1248, 1254, and 1260. (See Fig. B1 through B7.)

B3.4 Apparatus

(1) Gas scrubbing bottles, high form, ground-glass joint, fritted coarse disks.

(2) Separatory funnels equipped with ground-glass stoppers and TFE-fluorocarbon stopcocks; capacities of 125, 250, 500, 1000, and 2000 ml.

(3) Kunderna-Danish evaporative concentrators, 500-ml capacity, equipped with three-ball Snyder columns and graduated 5-ml capacity vials.

(4) Chromatographic columns, glass, 10 inches X 20 mm (OD), with a 5-inch X 50-mm (OD) reservoir at the top, equipped with TFE-fluorocarbon stopcocks.

(5) Flat-bottomed boiling flasks, 125 ml capacity.

(6) Liebig condenser, 200 mm in length.

(7) Hot plates.

(8) Water bath.

(9) 10-μl syringes.

(10) Dry-test meter or wet-test gas meter.

(11) Laboratory vacuum pump.

(12) Rotating vacuum evaporator.

(13) Usual laboratory glassware.

B3.5 Sampling. The air to be sampled for airborne PCBs is drawn through a gas scrubber (or scrubbers) and a dry-test meter using a laboratory vacuum pump. See Fig. B8. The sampling flow rate is controlled by bleeding in air via a needle valve located between the

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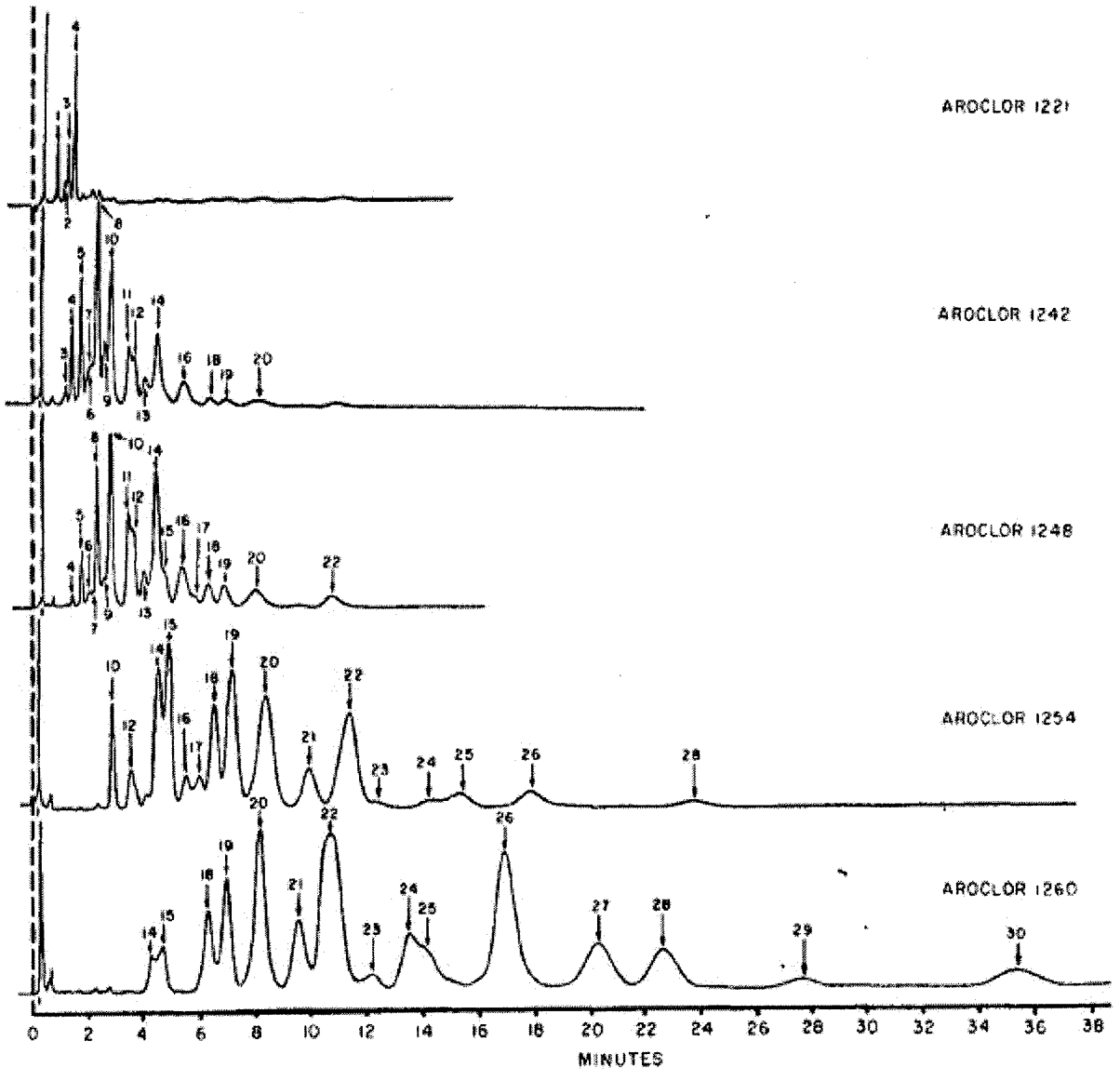

Fig. B2
Comparison of Electron Capture Chromatograms for Aroclor 1221, 1242, 1248, 1254, and 1260

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NOTE:
Instrument: Hewlett-Packard 5750
Detector: ^{63}Ni electron capture
Column: 6-mm × 6-foot, 4% XE-60 on 80/100 mesh, Chromosorb W, HP, AW-DMCS
Column temperature: 170°C
Injection temperature: 220°C
Detector temperature: 250°C
Carrier gas: He, 60 ml/min
Purge gas: 10% CH_4/argon, 120 ml/min
Pulse interval: 50 μs
Injection volume: 5 μl
Standard concentration: 1.48 μg/ml
Range: 10
Attenuation: 4

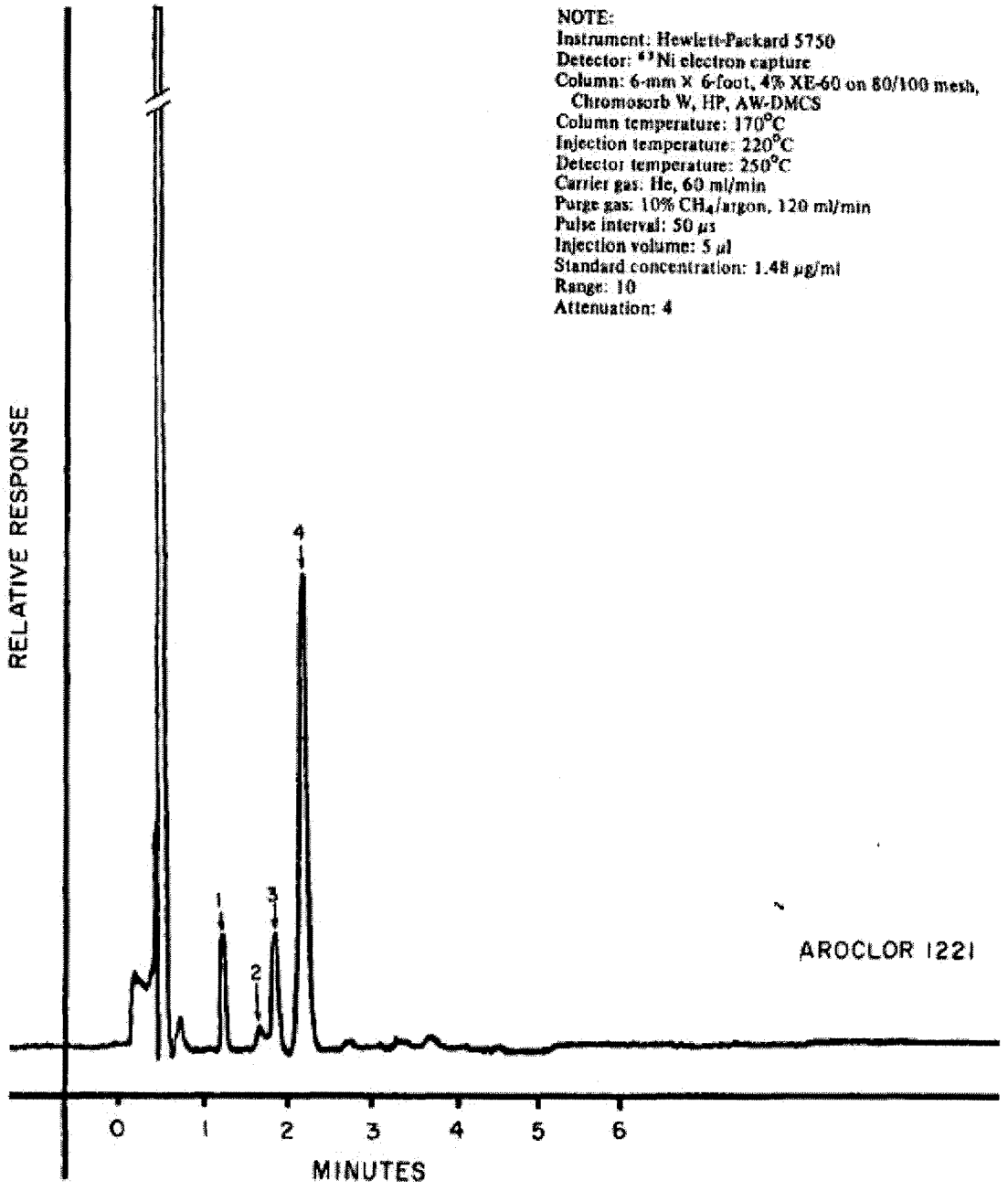

Fig. B3
Aroclor 1221 Electron Capture Chromatogram

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NOTE:
Instrument: Hewlett-Packard 5750
Detector: ^{63}Ni electron capture
Column: 6-mm X 6-foot, 4% XE-60 on 80/100 mesh, Chromosorb W, HP, AW-DMCS
Column temperature: 190°C
Injection temperature: 220°C
Detector temperature: 250°C
Carrier gas: He, 60 ml/min
Purge gas: 10% CH_4/argon, 120 ml/min
Pulse interval: 50 µs
Injection volume: 5 µl
Standard concentration: 1.03 µg/ml
Range: 10
Attenuation: 8

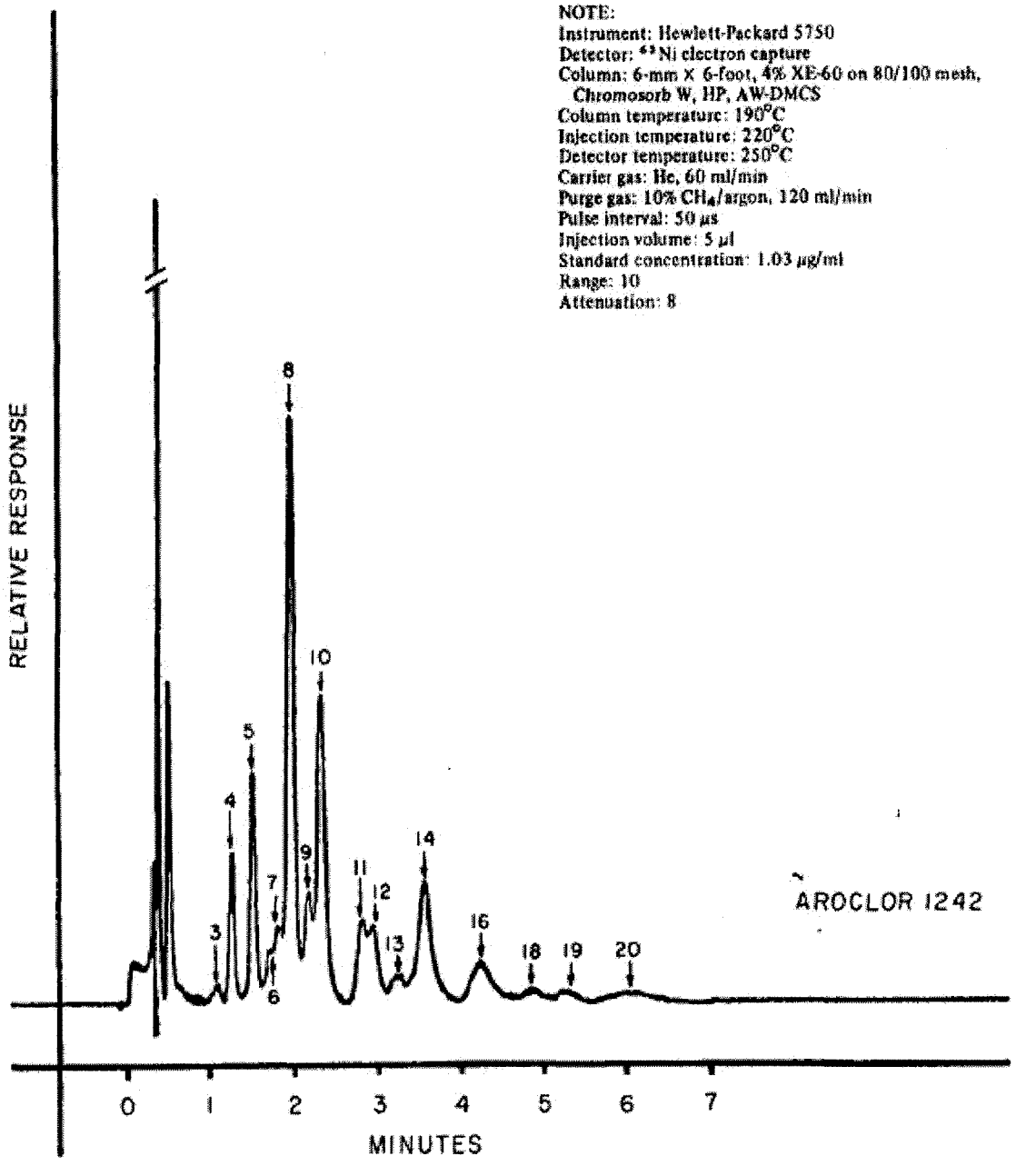

Fig. B4
Aroclor 1242 Electron Capture Chromatogram

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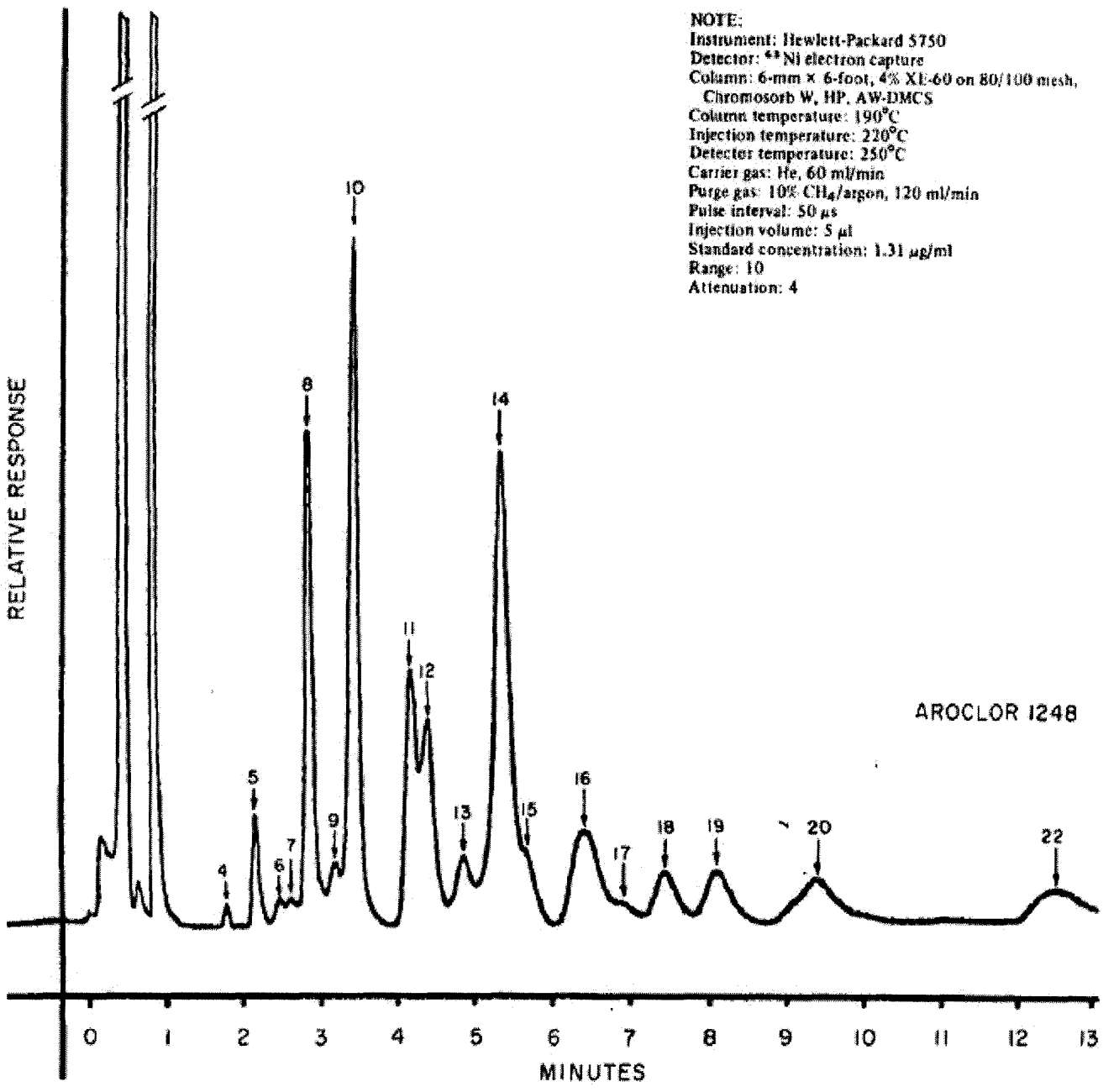

Fig. B5
Aroclor 1248 Electron Capture Chromatogram

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NOTE:
Instrument: Hewlett-Packard 5750
Detector: ^{63}Ni electron capture
Column: 6-mm × 6-foot, 4% XE-60 on 80/100 mesh, Chromosorb W, HP, AW-DMCS
Column temperature: 205°C
Injection temperature: 220°C
Detector temperature: 250°C
Carrier gas: He, 60 ml/min
Purge gas: 10% CH_4/argon, 120 ml/min
Pulse interval: 50 μs
Injection volume: 5 μl
Standard concentration: 1.02 μg/ml
Range: 10
Attenuation: 8

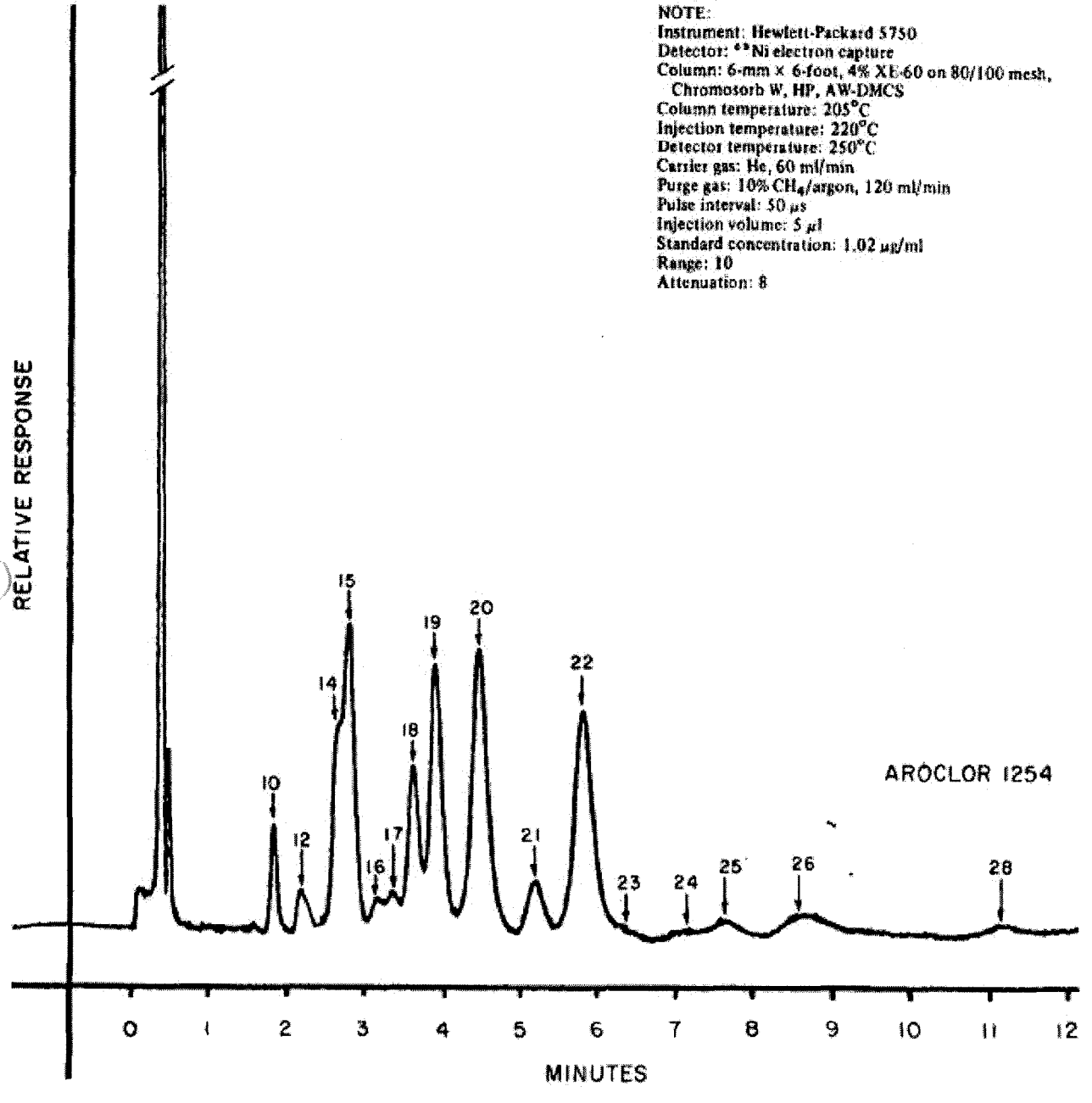

Fig. B6
Aroclor 1254 Electron Capture Chromatogram

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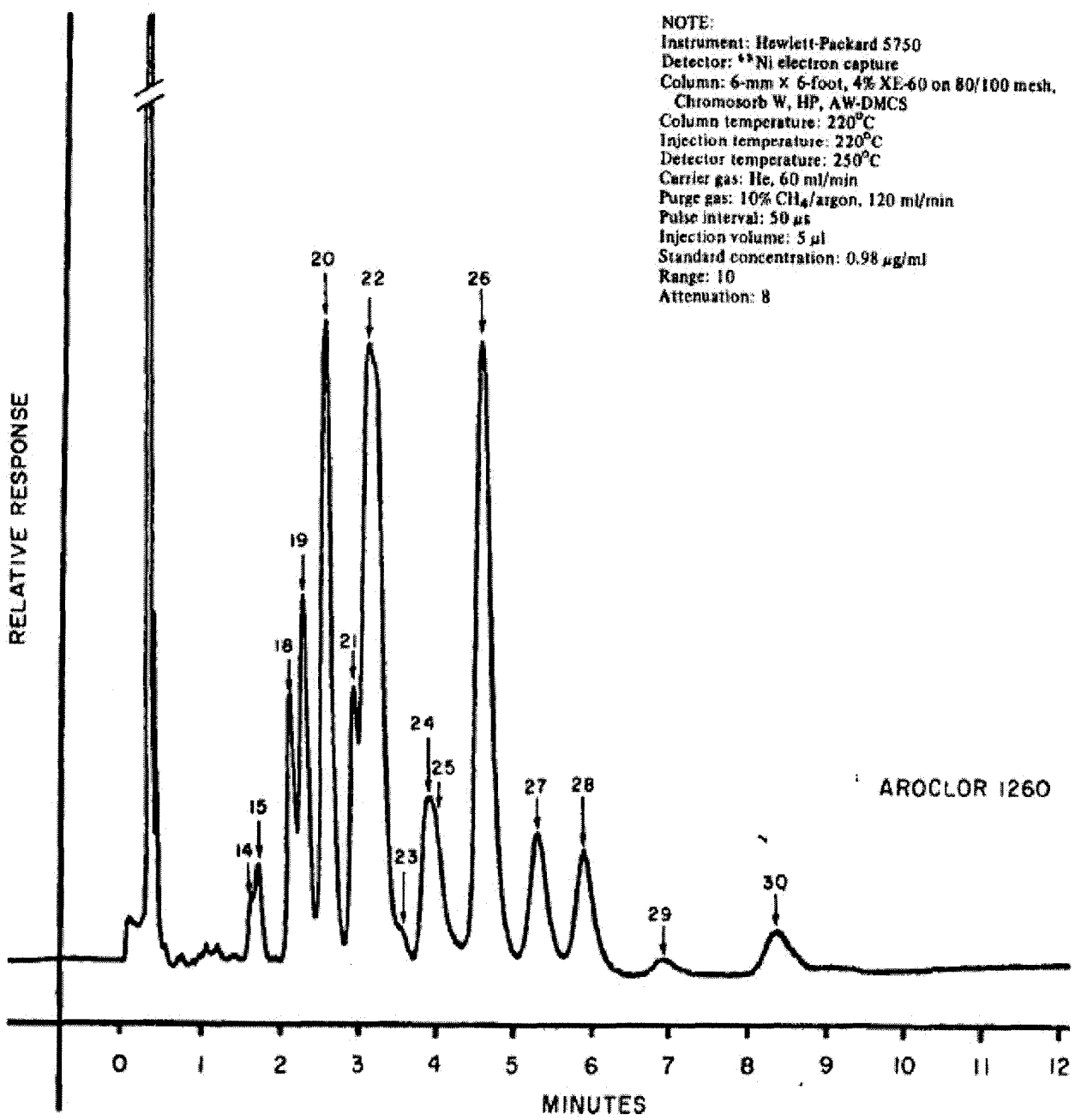

Fig. B7
Aroclor 1260 Electron Capture Chromatogram

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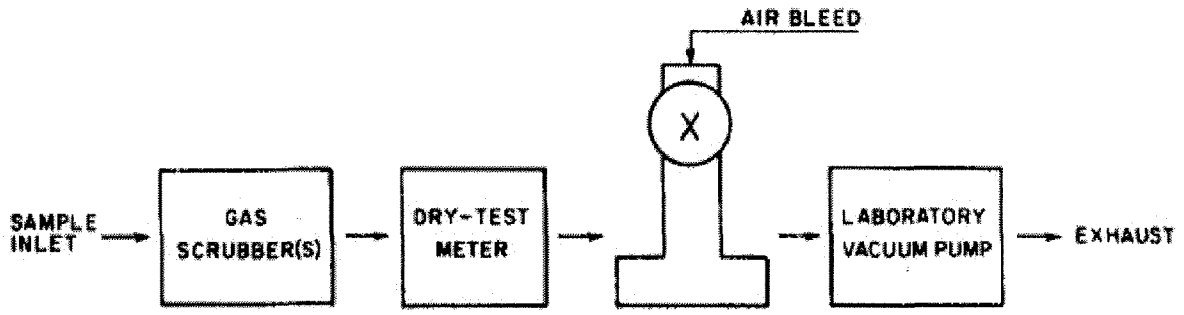

Fig. B8
Sampling Train

pump and the meter. At the end of the sampling period, the metered gas volumes are corrected for temperature and pressure to cubic metres at 25°C and 760 mmHg.

It is important to note that neither the capacity nor the efficiency of the gas scrubber(s) for removal of airborne PCBs 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 PBCs. 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 have been experimentally established, this procedure should be used only to measure relative PCB levels sampled under equivalent conditions.

B3.6 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 microlitre of the concentrate into the gas chromatograph to check for interferences and determine the approximate level of PCBs 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 cleanup 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% alcoholic 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 separatory 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-inch funnel, plugged with glass wool that 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

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than 5 ml by heating the apparatus in a 80°C to 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.

B3.7 Electron Capture Gas Chromatographic Procedure

(1) Instrument: Gas chromatograph (for example, Hewlett-Packard Model 5750, or the equivalent)

(2) Detector: High-temperature ^{63}Ni, electron capture cell

(3) Column: 6-mm X 6-foot glass column, 4% XE-60 on 80/100 mesh, Chromosorb W, HP, AW-DMCS

(4) Column temperature: 160°C to 190°C

(5) Injection port temperature: 195°C to 215°C

(6) Detector temperature: 300°C

(7) Pulse interval: 50 μs

(8) Flow rates: Helium carrier, approximately 60 ml/min, argon-methane purge, approximately 120 ml/min

Using EC/GC as the determinative step, inject, in duplicate, 1 to 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 and area methods.

B3.8 Sample Concentration. Concentration of sample extracts is necessary, prior to cleanup 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 without encountering any significant losses from volatization or chemical alternation. However, the Kunderna-Danish evaporative concentrator and the stream-of-air methods are easier to use.

B3.9 Column Adsorption Chromatography and Chemical Cleanup. Silica gel, a magnesia silica gel,[9] and alumina deactivated with 0%, 1.0%, 1.5%, 2.0%, and 5% 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 a magnesia silica gel.[9] 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 ensure 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. PCBs are not affected.

B3.10 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 nonpolar 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°C to 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 nonpolar 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 should be observed: during conditioning, the column should not be connected to the detector, and the maximum safe temperature of the liquid phase should not be exceeded.

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

[9] For example, Florisil.

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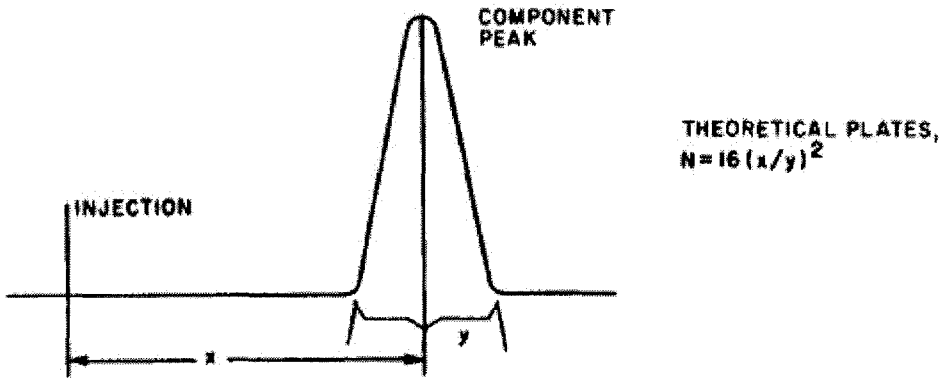

Fig. B9
Calculating Column Efficiency

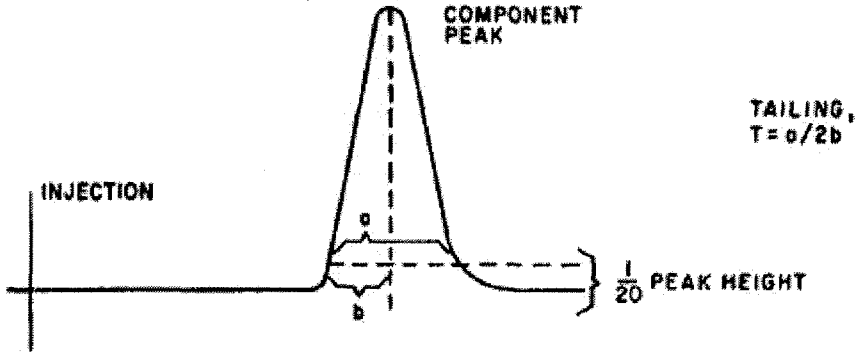

Fig. B10
Calculating the Tailing Factor

"poor" columns. In this manner, one can determine whether 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 of the order of 400 to 500, with tailing factors of 1.0 to 1.3. Calculation of these parameters is shown in Fig. B9 and B10. 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.

Any system of instrument and column suitable for chlorinated pesticide analysis is satisfactory for PCB analysis. The use of the high-temperature ^{63}Ni 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 also should be employed.

B3.11 Detection and Measurement. Quantitative determinations employing the electron capture detector are nonstoichiometric measurements 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

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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 PCBs 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 cleanup procedures is employed.

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

B3.12 Contamination. In determining PCBs 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, TFE-fluorocarbon, or metal. Laboratory glassware should be thoroughly washed with hot, soapy water, and 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 ensure against the possibility of contamination.

B3.13 Sensitivity

(1) Concentration: 2 ppb (parts per billion)
(2) Absolute sensitivity: 0.5×10^{-9} gram
(3) Volume injected: 5 µl
(4) Final volume of extract: 5 ml
(5) Sample size: 250 ml

B4. Analysis of Water and Sediment for PCBs

B4.1 Scope. This methodology is used by the Monsanto Industrial Chemical Company for the determination of the amount and type of PCBs 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 gas chromatography fractions.

B4.2 Principle. The PCBs 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 PCBs present are determined by electron capture gas chromatography (EC/GC).

B4.3 Reagents

(1) Hexane: Pesticide grade.
(2) Acetonitrile: Pesticide grade.
(3) Sodium sulfate: Anhydrous, granular, analytical reagent (AR) grade. Heat at 400°C for 1 hour prior to use.
(4) Alumina adsorption: For chromatographic analysis, 80/200 mesh. Heat at 400°C for a minimum of 4 hours and deactivate with 5% (w/w) distilled water.

For 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 (about 30 grams) of deactivated alumina, and pour this slowly into the column. Tap or vibrate the column to settle the alumina, and top the alumina with 2 to 3 cm of anhydrous sodium sulfate. Wash the column with 50 to 100 ml of hexane prior to the addition of the sample.

(5) Distilled water: Extracted with hexane to remove hexane-soluble electron capturing impurities.
(6) Sulfuric acid: AR grade, specific gravity = 1.84.
(7) Potassium hydroxide: AR grade.
(8) Ethanol: Formula 2B.
(9) Alcoholic potassium hydroxide, 2.5% (w/v): Dissolve about 12.5 grams of AR grade KOH in 500 ml of ethanol.
(10) Sulfuric acid–water, 9:1 (v/v): Carefully add 270 ml of AR grade sulfuric acid to 30 ml of distilled water in a 500-ml iced beaker.
(11) PCB standards: Aroclor 1242, 1248, 1254, and 1260. (See Fig. B2 and Fig. B4 through B7.)

B4.4 Apparatus

(1) Separatory funnels equipped with ground-glass stoppers and TFE-fluorocarbon stopcocks; capacities of 125, 250, 500, 1000, and 2000 ml.
(2) Kunderna-Danish evaporative concentrators, 500-ml capacity, equipped with three-ball Snyder columns and graduated 5-ml capacity vials.
(3) Chromatographic columns, glass, 10 inches × 20 mm (OD) with a 5-inch × 50-mm (OD) reservoir at the top, equipped with TFE-fluorocarbon stopcocks.
(4) Sintered glass filter funnels, 600-ml capacity, 90-mm disk diameter, medium porosity.
(5) Flat-bottomed boiling flasks, 125-ml capacity.
(6) Liebig condenser, 200 mm in length.
(7) Hot plates.
(8) Water bath.
(9) Reciprocating variable-speed shaker.
(10) 10-µl syringes.
(11) 32-oz all-glass mortars and pestles.
(12) 8-inch × 12-inch × 2-inch (2-1/2 qt) heat-resistant glass baking dishes.

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(13) U.S. Standard sieve, No. 30.
(14) Usual laboratory glassware.

B4.5 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 given in B4.5.1 and B4.5.2.

B4.5.1 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 B4.6.)

B4.5.2 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-gram portion taken for analysis. (See B4.7.)

B4.6 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-inch funnel, plugged with glass wool that 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 an 80°C to 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 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.

B4.7 Extraction of Sediment and Soil Samples

(1) Decant off any excess water and transfer the entire sediment sample to a glass baking dish. Air-dry at ambient temperature (heat should 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 grams (to the nearest 0.01 gram) into a 16-ounce narrow-neck screw-cap (aluminum-foil liner) glass bottle.

(3) Moisten the soil with about 10 ml of water 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 acetronitrile extract into a 600-ml sintered glass filter funnel containing a 1/4-inch layer of anhydrous sodium sulfate. Collect the filtrate in a 600-ml beaker (vacuum filtration may be necessary).

(5) After the acetronitrile 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 an 80°C to 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 it

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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-inch funnel, plugged with glass wool that 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 an 80°C to 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.

B4.8 Electron Capture Gas Chromatographic Procedure

(1) Instrument: Gas chromatograph (for example, Hewlett-Packard Model 5750, or the equivalent)

(2) Detector: High-temperature ^{63}Ni, electron capture cell

(3) Column: 6-mm X 6-foot glass column, 4% XE-60 on 80/100 mesh, Chromosorb W, HP, AW-DMCS

(4) Column temperature: 160°C

(5) Injection port temperature: 195°C

(6) Detector temperature: 300°C

(7) Pulse interval: 50 μs

(8) Flow rates: Helium carrier, approximately 60 ml/min; argon-methane purge, approximately 120 ml/min

Using EC/GC as the determinative step, inject, in duplicate, 1 to 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 and area methods.

The electron capture detector should also be used to guide the isolation procedures. Water and sediment extracts can be checked for the presence of PCBs or interferences, or both, by injection of microlitre portions of the extracts at various points in the extraction and concentration schemes. In this manner it can be determined whether the sample needs to be concentrated or diluted and whether the cleanup procedures should be employed.

B4.9 Extraction. The extraction of PCBs 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 PCBs. The spiking method consisted of adding the PCB in 25 to 50 μl of acetone to 500 ml of tap water in a 32-ounce narrow-neck screw-cap jar. After the sample was thoroughly mixed, duplicate 225- to 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 PCBs 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 PCBs 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 reextracted 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 PCBs was essentially quantitative in the cases checked.

B4.10 Sample Concentration. Concentration of sample extracts is necessary, prior to cleanup by chromato-

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graphic 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.

B4.11 Column Adsorption Chromatography and Chemical Cleanup. Silica gel, a magnesia silica gel,[9] and alumina deactivated with 0%, 1.0%, 1.5%, 2.0%, and 5% 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 a magnesia silica gel.[9] 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 ensure 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. PCBs are not affected.

B4.12 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 nonpolar 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 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°C to 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 nonpolar 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 should be observed: during conditioning, the column should not be connected to the detector, and the maximum safe temperature of the liquid phase should not be exceeded.

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 whether 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 of the order of 400 to 500, with tailing factors of 1.0 to 1.3. Calculation of these parameters is shown in Fig. B9 and B10. 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; that is, 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.

Any system of instrument and column suitable for chlorinated pesticide analysis is satisfactory for PCB analysis. The use of the high-temperature ^{63}Ni 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 also should be employed.

B4.13 Detection and Measurement. Quantitative determinations employing the electron capture detector are nonstoichiometric 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 disk integration of triangulation and, if the

*For example, Florisil.

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peak shape represents a Gaussian curve, the height may be considered independent of the base. Consequently, peak height measurements are generally used. 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 PCBs in Case I samples is determined by preparing a plot of the major peak height or area 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 cleanup procedures is employed.

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

B4.14 Contamination. In determining PCBs 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, TFE-fluorocarbon, or metal. Laboratory glassware should be thoroughly washed with hot, soapy water and 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 ensure against the possibility of contamination.

B4.15 Sensitivity

(1) Concentration: 2 ppb
(2) Absolute sensitivity: 0.5×10^{-9} gram
(3) Volume injected: 5 μl
(4) Final volume of extract: 5 ml
(5) Sample size: 250 ml

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American National Standards

The standard in this booklet is one of nearly 5,600 standards approved to date by the American National Standards Institute, formerly the USA Standards Institute.

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