

DETERMINATION OF
POLYCHLORINATED
BIPHENYLS (PCBs) IN WASTE
OILS BY GAS
CHROMATOGRAPHY WITH
ELECTRON CAPTURE
DETECTOR

EPA VICTORIA METHOD NUMBER: 6013

GUIDELINES FOR ENVIRONMENTAL MANAGEMENT

**DETERMINATION OF POLYCHLORINATED BIPHENYLS (PCBs) IN
WASTE OILS BY GAS CHROMATOGRAPHY WITH ELECTRON
CAPTURE DETECTOR**

EPA Victoria Method Number: 6013

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1 BACKGROUND

Methods that have been developed for the analysis of polychlorinated biphenyls (PCBs) in used insulating oil are not usually applicable to the analysis of PCBs in waste oil. This method is to serve specifically as a method for the analysis of PCBs in waste oil. The method is applicable to the determination of commercial mixtures of PCBs in hydrocarbon based waste oils using gas chromatography (GC) with capillary columns. The method is based on US EPA methods SW846 8082A [1] and EPA-600/4/81-045 [2].

2 SCOPE

This method sets out a procedure for the determination of polychlorinated biphenyls (PCBs), as Aroclors[®], in extracts from waste oil by GC using capillary columns with electron capture detectors (ECD). The method is intended for use to determine whether waste oils contain PCBs at the regulatory decision level of 2mg/kg.

3 SUMMARY

The sample is diluted with solvent. The resulting solution is treated to remove interfering substances using a combination of acid cleanup and adsorption chromatography. A small volume of the resulting solution is injected into a capillary gas chromatographic column equipped with an electron capture detector and recorded as a chromatogram. The test method is made quantitative by comparing the sample chromatogram with a chromatogram of a known quantity of one or more standard Aroclors[®], obtained under the same conditions.

4 INTERFERENCES

Most waste oils will consist of a complex mixture of hydrocarbons such as n-alkanes, isoalkanes, naphthenes and paraffins. These matrix components will most likely interfere with the PCB determination and a sample preparation procedure, consisting of least an acid cleanup followed by adsorption chromatography, is required before analysis and quantification.

Sources of interference include:

4.1 Contaminated solvent, reagents or sample processing hardware.

4.2 Contaminated GC carrier gas, parts, column surfaces, or detector surfaces.

4.3 Compounds which are extracted from the sample matrix to which the detector responds, such as single-component chlorinated pesticides, including dichlorodiphenyltrichloroethane (DDT) analogues dichlorodiphenylethylene (DDE) and dichlorodiphenyldichloroethane (DDD).

4.4 Phthalate esters introduced during sample preparation. Phthalate esters are easily extracted from common flexible plastics. They can be minimised by avoiding contact between the samples and any plastic materials.

4.5 Cross-contamination of clean glassware. Glassware must be scrupulously cleaned by rinsing with the last solvent used, followed by detergent washing with hot water and rinses with tap water and organic free reagent water. Drain the glassware, and dry it in an oven at 130°C for several hours.

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4.6 Sulfur (S₈) (usually present in crude oils) can cause chromatographic interferences. An additional sulfur removal step may be required when using the cleanup steps described in this method. Refer to Appendix II for information on how to remove sulfur interferences.

The sensitivity of ECD detectors is reduced by mineral oils due to quenching of the detector response by high boiling hydrocarbons. The degree of error is matrix dependent and is not predictable for samples of unknown origin. Retention time shifts for individual congeners have also been found to occur in the presence of mineral oil.

All calibration solutions must contain PCB-free transformer oil at the same concentration as the concentration of waste oil that is expected in the sample solutions that are injected into the GC (i.e. PCB-free transformer oil must be diluted with the solvent to the same extent that the waste oil matrix is diluted)

5 APPARATUS

5.1 Adsorption Column:

400 mm long x 19 mm ID; with Pyrex[®] glass wool at the bottom and a teflon stopcock.

Fritted glass disks are difficult to decontaminate after highly contaminated extracts have been passed through. Columns without frits may be purchased.

Use a small pad of Pyrex[®] glass wool to retain adsorbent. Prewash the glass wool pad with 50 mL of acetone followed by 50 mL of elution solvent prior to packing the column with adsorbent.

5.2 Beakers.

Appropriate sizes.

5.3 Concentrator tube.

10mL graduated. Calibration must be checked. Ground glass stopper is used to prevent evaporation of solvent.

5.4 Erlenmeyer flasks.

Appropriate sizes.

5.5 Evaporative flask.

500mL. Attach to concentrator tube with springs.

5.6 Gas chromatograph.

An analytical system complete with gas chromatograph suitable for on-column and split-splitless injection and all required accessories including syringes, analytical columns, gases, electron capture detector (ECD) and recorder/integrator or data system.

The gas lines (carrier gas and make up gas) shall be fitted with water vapour and oxygen traps.

5.7 GC Columns.

Two columns are used, the DB-5 is used for primary analysis and the DB608 or DB1701 for confirmation. Columns of other dimensions may be used.

A fused-silica capillary column chemically bonded with DB-5 or equivalent.

Length	= 30m
Internal diameter	= 0.25 or 0.32mm
Film thickness	= 1µm

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A fused-silica capillary column chemically bonded with 35 percent phenyl methylpolysiloxane (DB608) or equivalent.

Length = 30m
Internal diameter = 0.25mm
Film thickness = 1µm

A fused-silica capillary column chemically bonded with 14percent cyanopropylmethylsiloxane (DB1701) or equivalent.

Length = 30m
Internal diameter = 0.53mm
Film thickness = 1µm

5.8 Kudeerna-Danish.

(K-D) evaporative concentrator apparatus.

5.9 Muffle Furnace

5.10 Reagent bottles.

Appropriate sizes.

5.11 Vacuum manifold.

Consisting of glass vacuum basin, collection rack and funnel, collection vials, replaceable stainless steel delivery tips, built in vacuum bleed valve and gauge. The system is connected to a vacuum pump or water aspirator through a vacuum trap made from a 500 mL sidearm flask fitted with a one-hole stopper and glass tubing.

5.12 Vials.

Glass of appropriate volume with teflon-lined screw-caps or crimp tops.

5.13 Vials.

With aluminium or polytetrafluoroethylene (PTFE)-lined caps and various pipettes and beakers for making dilutions.

5.14 Volumetric glassware.

Appropriate for making dilutions (tolerance better than $\pm 0.4\%$).

6 REAGENTS

All reagents and materials, including those for cleanup, shall be free from PCB contamination and compounds that respond to the ECD.

6.1 Concentrated sulfuric acid.

(96% to 98%) Analytical grade.

6.2 Florosil® cartridges.

40µm particles, 60Å pores. Single use one gram cartridges are used in this method but larger cartridges may be used. Recovery data must be developed for any size cartridges that are used.

6.3 Granular Florosil®

(PR grade or equivalent). Activate the Florosil® by heating in a glass container loosely covered with aluminium foil in an oven at 130°C overnight. Cool the Florosil® in a dessicator before use.

6.4 Insulating oil.

Fresh, unused and PCB free. A portion of the oil diluted in solvent must be analysed to determine if it is free of PCBs and compounds that interfere with

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PCBs. The oil may have to be subjected to a cleanup to remove interferences to PCBs. The method of cleanup is left to the discretion of the analyst. An example of a cleanup method is given in Appendix I.

6.5 Reagent water.

All references to water in this method refer to organic-free reagent water.

6.6 Silica cartridges.

40µm particles, 60Å pores. Single use one gram cartridges are used in this method but larger cartridges may be used. Recovery data must be developed for any size cartridges that are used.

6.7 Silica gel.

100/200 mesh (Davison Chemical grade 923 or equivalent). Before use, activate for at least 16 hours at 130°C in a shallow glass tray, loosely covered with foil. Deactivate it to 3.3% by adding 3.3 g of reagent water to 100 g of silica gel in a 500mL glass jar. Mix the contents thoroughly and allow to equilibrate for six hours. Store the deactivated silica gel in a sealed glass jar inside a dessicator.

6.8 Sodium sulfate.

(Granular, anhydrous), Na₂SO₄. Purify by heating at 400°C for four hours in a shallow tray, or by precleaning the sodium sulfate with dichloromethane. A method blank must be analysed in order to demonstrate that there is no interference from sodium sulfate.

6.9 Solvents.

Hexane or isooctane (2,2,4-trimethylpentane).

All solvents should be pesticide quality or equivalent, and each lot of solvent should be determined to be free of phthalates.

The choice of solvent is left to the analyst and, unless specified, any reference to solvent in this method means either hexane or iso-octane.

6.10 Stock standard solutions.

(1000mg/L) each of Aroclor[®] 1016, Aroclor[®] 1221, Aroclor[®] 1232, Aroclor[®] 1242, Aroclor[®] 1248, Aroclor[®] 1254, Aroclor[®] 1260. These may be prepared from pure standard materials or purchased as certified standard solutions.

Prepare stock standard solutions by accurately weighing 0.0100 g of pure compound. Dissolve the compound in solvent and dilute to volume in a 10mL volumetric flask.

Commercially prepared stock solutions may be used at any concentration if they are certified by the manufacturer or an independent source.

6.11 Calibration Solutions

A standard containing a mixture of Aroclor[®] 1016 and 1260 will include many of the peaks represented in the other five Aroclor[®] mixtures. As a result a multi-point initial calibration employing a mixture of Aroclors[®] 1016 and 1260 at five concentrations should be sufficient to demonstrate the linearity of the detector response without the necessity of performing multi-point initial calibrations for each of the possible Aroclors[®]. In addition such a mixture can be used to demonstrate by the absence of peaks

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that a sample does not contain peaks that represent either Aroclor® 1016 or Aroclors® 1260. This standard can also be used to determine the concentrations of either Aroclor® 1016 or Aroclor® 1260, should they be present in the sample.

The lowest concentration calibration standard establishes the method quantitation limit based on the final volume of extract (or sample). At least one of the calibration standards should correspond to a concentration below that necessary to meet the regulatory compliance limit of 2mg/kg.

All calibration solutions must contain PCB free insulating oil at the same concentration as the concentration of waste oil that is expected in the sample solutions that are injected into the GC.

6.11.1 Initial calibration solutions.

Prepare a minimum of five calibration standards containing equal concentrations (w/v) of both Aroclor® 1016 and Aroclor® 1260 by dilution of stock standard with solvent. The concentration should correspond to the expected range of concentrations found in real samples and should bracket the linear range of the detector.

6.11.2 Individual Aroclor® standard solutions.

Single standards of each of the other five Aroclors® (1221, 1232, 1242, 1248 and 1254) are necessary for pattern recognition. When employing the model of linear calibration through the origin, these standards are also used to determine a single-point calibration factor for each Aroclor®, assuming the Aroclor® 1016/1260 mixture has been used to demonstrate the linearity of the detector.

Prepare separate standards of Aroclors® 1221, 1232, 1242, 1248 and 1254. The concentrations of each Aroclor® should correspond to the decision level PCB concentration in waste oils of 2mg/kg.

Store the standard solutions in the dark at 4°C in glass containers sealed with a PTFE lid. When a batch of standards is prepared, it is recommended that aliquots of that batch be stored in individual small vials. All stock standards must be replaced after one year, or sooner if routine quality control checks indicate a problem. All other standard solutions must be replaced after one month, or sooner if routine quality control checks indicate a problem.

6.12 Surrogate standards

The performance of the method should be monitored using surrogate compounds. Surrogate standards should be added to all samples, method blanks, matrix spikes, and calibration standards.

Decachlorobiphenyl (C209) must be used as a surrogate, and is added to each sample prior to extraction. Prepare a solution of C209 in solvent. The recommended spiking solution concentration is 0.1 mg/L.

Prepare a minimum of five calibration standards containing C209 in solvent. The concentration should correspond to the expected range of concentrations added to samples and should bracket the linear range of the detector. Each surrogate standard solution must contain PCB free insulating oil at the same concentration as the concentration of waste oil that is expected in the sample solutions that are injected into the GC.

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6.13 Laboratory control standard.

Prepare a laboratory control standard (LCS) by spiking a PCB free oil typical of the matrix normally analysed such as used lubricating oil, with PCBs at a concentration 2.0 mg/kg. Use a PCB mixture typical of those normally found in samples, such as Aroclor® 1260.

7 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

Sample containers should have a volume of 20mL or more, and have PTFE lined screw caps.

7.1 Sample bottle preparation.

Wash all sample bottles and cap seals in detergent solution. Rinse first with tap water and then with distilled water. Allow the bottles and seals to drain and dry in a contaminant-free area. Then rinse the seals with hexane and allow to air dry.

Heat sample bottles to 400°C for 15 to 20 minutes or rinse with pesticide grade hexane and allow to air dry.

Store the clean bottles inverted or sealed until use.

7.2 Sample preservation.

The samples should be stored in a cool, dry, dark area until analysis. Storage times in excess of four weeks are not recommended for unknown or undefined sample matrices.

Extracts should be stored under refrigeration in the dark and should be analysed within 40 days of extraction.

7.3 Sample collection.

If practical, mix the waste oil in the sample source prior to sampling. Fill a large container, such as a 500mL beaker, from a representative portion of the sample source. Mix the oil in the 500mL beaker. Fill a minimum of two 20mL sample bottles approximately 80 percent full from the 500mL beaker.

8 CHROMATOGRAPHIC OPERATING CONDITIONS

The chromatographic operating conditions given here are to serve as a guide only. Variations between columns and GCs may necessitate alterations in the conditions described to produce suitable results.

8.1 DB5 or equivalent column.

Carrier gas (He or H ₂)	16 psi
Injector temperature	225°C
Detector temperature	300°C
Initial temperature	100°C, hold 2 minutes
Temperature program	100°C to 160°C at 15°C/min, followed by 160°C to 270°C at 5°C/min
Final temperature	270°C
Make up flow (N ₂)	60mL/min

8.2 DB608 or equivalent column.

Carrier gas (He or H ₂)	20 psi
Injector temperature	225°C
Detector temperature	300°C

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Initial temperature	160°C, hold 2 minutes
Temperature program	160°C to 290°C at 5°C/min
Final temperature	290°C, hold 1 min
Make up flow (N ₂)	60mL/min

Alternative clean-up methods to those specified here may be used with approval from EPA. When seeking approval from EPA, validation of the proposed method must be demonstrated before approval can be granted. The procedures that should be followed for method validation are available in NATA Technical Note No. 17, "Requirements for the Format and Content of Test Methods and Recommended Procedures for the Validation of Chemical Test Methods." [3]

9 PROCEDURE

9.1 Sample preparation

Weigh approximately 1g of the test sample to the nearest 0.001g into a 10mL volumetric flask. Add the surrogate standard to give a suitable concentration in the diluted sample. Make to volume with solvent. This solution is designated solution A.

An alternative approach is to add 1g of test sample into a disposable glass vial. Ten mL of solvent containing surrogate standard at a suitable concentration is then added to the vial. A final volume of 11mL can be assumed with reasonable accuracy. This approach enables the use of disposable glassware reducing the risk of cross contamination.

Any water present in the sample has to be removed which can be done by the addition of an appropriate quantity of sodium sulfate to Solution A.

9.2 Removal of interferences

Reference materials, field-contaminated samples, or spiked samples should be used to verify the applicability of the selected clean-up technique. When other materials are not available and spiked samples are used, the Aroclor® 1016/1260 mixture may be an appropriate choice for spiking.

9.2.1 Acid treatment.

Due to the complex nature of waste oils the procedures and information given here serve only as a guide. The analyst may need to conduct more extensive acid cleanup for more difficult samples.

Either of the two following acid cleanup procedures may be used as an initial treatment to remove interferences. Alternative acid cleanup methods may also be used.

9.2.1.1 Sulfuric Acid Treatment [4]

For some waste oils this procedure has been found to result in the formation of emulsions that have prevented further analysis. In such cases it is recommended that the silica gel/sulfuric acid procedure described in Section 9.2.1.2 is used.

Extended acid treatment may degrade some of the less chlorinated PCBs. Sulfuric acid should not be allowed to remain in contact with the extract at elevated temperatures for longer than necessary to complete the analysis.

Add a 5mL portion of solution A into a 40mL narrow mouth screw cap bottle. Add 2.5mL of concentrated sulfuric acid into the bottle. Seal the bottle with a

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Teflon lined screw cap and shake or vortex mix for one minute. Depending on the degree of contamination the colour of the acid layer will range from yellow to dark brown.

Allow the phases to separate (centrifuge if necessary), transfer the sample (upper phase) to a clean narrow mouth screw cap bottle. Some loss of extract may occur during the acid cleanup; however this should not change the concentration of the analytes in the sample. Do not bring up to original volume after clean-up as this will cause a dilution of the original concentration. Repeat the process until no change in colour of either layer is seen. This solution is designated solution B.

It may be necessary to wash the solvent layer with several portions of reagent water until the wash water is neutral to pH paper. This step can extend the life of the GC column and detector.

9.2.1.2 Silica Gel/Sulfuric Acid cleanup [5]

The silica gel/sulfuric acid procedure described here serves as a guide and must be verified before use. A recovery check must be performed using a waste oil spiked with a known concentration of Aroclor[®] 1016 and Aroclor[®] 1260. Commercial cartridges containing silica gel/sulfuric acid can also be used.

Weigh 28 ± 1 g chromatographic grade activated silica gel (particle size 100 – 200 μ m) and 22 ± 1 g of sulfuric acid (96 – 98%) into a 200mL Erlenmeyer flask. Shake until any lumps have disappeared. The mixture will heat up considerably. Store the mixture in a closed dessicator over P₂O₅. The silica gel/sulfuric acid mixture should be used within one week.

Depending on the mass of silica gel/sulfuric acid required greater or smaller quantities of silica gel and sulfuric acid can be used, provided the mass ratio of silica gel to sulfuric acid remains the same.

Put 4.0 ± 0.05 g of the silica gel/sulfuric acid mixture into a suitable size adsorption column. Add 1g of anhydrous sodium sulfate to the top of the silica gel/sulfuric acid.

The mass of silica gel/sulfuric acid mixture used will depend on the type of waste oil to be cleaned up. Four grams has been found to produce a satisfactory result for most waste oils. Greater or smaller quantities of silica gel/sulfuric acid can be used to meet the cleanup needs of the sample matrix.

Pre-elute the column three times with 5mL of hexane. Stop the hexane eluate flow just prior to the exposure of the sodium sulfate to air. Discard the eluate.

Transfer 1mL of solution A onto the column. Bring the solvent level to just above the sodium sulfate to distribute the sample evenly over the packing. Wait at least 5 minutes before elution.

Elute the column twice with 4mL aliquots of hexane. The elution must be carried out at a maximum flow rate of 2mL/min and the column eluted each time until the solvent level is just above the top of the adsorbent (except for the final elution).

Collect the eluates in a 15mL graduated centrifuge tube. Reduce the volume of the eluate to 1mL under nitrogen prior to further chromatographic cleanup. This solution is designated solution B.

The silica gel/sulfuric acid mixture can also be added directly to the top of adsorption columns containing other packings enabling acid cleanup

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and adsorption cleanup in a single step. If this is done the analyst must verify that the analytes of interest are being quantitatively recovered and that interferences are sufficiently removed before applying the method to actual samples.

9.3 Column adsorption chromatography cleanup

An acid cleanup alone does not usually remove all of the PCB interferences that occur in waste oil samples.

An additional cleanup involving the use of adsorption chromatography must also be used. Either or both of the chromatographic cleanup procedures described may be used.

The analyst may use other adsorption chromatography methods provided validation proves that interferences to PCBs are effectively removed.

The methods described here are examples of traditional column chromatography techniques and solid-phase cartridge extraction. Generally, the traditional column chromatography techniques use larger amounts of adsorbent and therefore, have a greater cleanup capacity.

A reagent blank should be prepared and analysed for PCBs prior to the use of these chromatographic cleanup methods. The level of interferences must be below the method detection limit before the method is performed on actual samples.

Phthalate ester contamination may be a problem with certain cartridges. The more inert the column and/or cartridge material (i.e., glass or teflon), the less problem with phthalates.

9.3.1 Standard column cleanup procedure [6, 2]

Variations between batches of silica or Florosil® may affect the volume of solvent needed to elute all of the PCBs from the adsorption column. For this reason, the volume of solvent required to completely elute all of the PCBs must be verified by the analyst.

	Silica Gel	Florosil®
M	3	20
V _i	10	70
V _{ii}	80	225
V _{iii}	0	25

Transfer **M** g of adsorption media into a 10mm ID glass chromatographic column and top it with 2 - 3cm of anhydrous sodium sulfate.

Add **V_i** mL of hexane to the top of the column to wet and rinse the sodium sulfate and adsorption media. Just prior to exposure of the sodium sulfate layer to air, stop the hexane eluate flow by closing the stopcock on the adsorption column. Discard the eluate.

Transfer 1mL of solution B onto the column. Rinse the extract vial twice with 1 -2mL of hexane and add each rinse to the column. Elute the column with **V_{ii}** mL of hexane at a rate of 5mL/min. Collect and discard the first **V_{iii}** mL of the eluate. Collect the rest of the eluate in a collection flask.

Remove the collection flask and reduce the volume of hexane to provide a concentration of PCB in the final extract that falls within the range of the calibration curve. This solution is designated solution C.

9.3.2 Cartridge cleanup procedure [6, 7]

Arrange the 1g silica or Florosil® cartridges on the manifold in the closed-valve position.

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Condition the cartridges by adding 4mL of hexane to each cartridge. Close the valves and allow the solvent to soak the entire sorbent bed for five minutes. Do not turn off the vacuum.

Slowly open the cartridge valves to allow hexane to pass through the cartridges. Close the cartridge valves when there is still at least 1mm of solvent above the sorbent bed. Do not allow the cartridges to become dry. If cartridges become dry, repeat the conditioning step.

Transfer 1mL of solution B onto the cartridge. Open the cartridge valve and allow the extract to pass through the cartridge bed at approximately 2mL/min.

When the entire extract has passed through the cartridges, but before the cartridge becomes dry, add an additional 0.5mL of hexane to the cartridge.

Add 5mL of hexane to the cartridge. Allow the solvent to soak the sorbent bed for one minute or less. Slowly open the cartridge valve and collect the eluate into the collection vial. Close the cartridge valve.

Adjust the final volume of the collected fraction to a known volume that will result in a PCB concentration appropriate for the analysis. This solution is designated Solution C.

9.4 Calibration

Prepare calibration standards as described in Sec. 6.11

9.4.1 Initial Calibration [1,8]

The initial calibration involves establishing the linearity of the calibration curve using the Aroclor®

1016/1260 standard solutions. Once linearity through the origin is established using the Aroclor 1016/1260 standard solutions, quantification of PCBs can be performed using one point calibration curves of the other Aroclor® standard solutions.

Set up the GC at the conditions specified. Optimise the instrumental conditions for resolution of the target compounds and sensitivity. A final temperature of 240-275°C may be required to elute C209. The use of injector pressure programming will improve the chromatography of late eluting peaks. Once established, the same operating conditions must be used for both calibrations and sample analysis.

A 2µL injection of each of the initial calibration solutions is recommended. Other injection volumes may be employed, provided that the analyst can demonstrate adequate sensitivity for the compounds of interest.

Record the peak area (or height) for each characteristic Aroclor® peak to be used for quantitation in the initial calibration solution. A minimum of three and preferably five peaks that are at least 25per cent of the area (or height) of the largest Aroclor® peak should be used in the calculation. At least one peak should be chosen that is unique to the Aroclor® that is to be used for quantification.

Example chromatograms obtained on a DB5 equivalent column are shown for each Aroclor® in Appendix III.

Calculate the calibration factor (CF) for each characteristic Aroclor® peak in each of the initial calibration standards using the equation below.

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$$CF = \frac{\text{Peak Area (or Height) of the congener in the Standard}}{\text{Total Mass of the Standard Injected (in nanograms)}}$$

Using the equation above, a calibration factor will be determined for each characteristic peak, using the total mass of the Aroclor[®] injected. These individual calibration factors are used to quantitate sample results by applying the factor for each individual peak to the area or height of that peak.

For a five point calibration, five sets of calibration factors will be generated for the initial calibration solutions, each set consisting of the calibration factors for each of the five (or more) peaks chosen for this mixture, for example, there will be at least 25 separate calibration factors for the mixture. The single standard for each of the other Aroclors[®] will generate at least three calibration factors, one for each selected peak.

The calibration factors from the initial calibration are used to evaluate the linearity of the initial calibration. This involves the calculation of the mean calibration factor (\overline{CF}), the standard deviation (SD), and the relative standard deviation (RSD) for each Aroclor[®] peak using the equations below.

$$\overline{CF} = \frac{\sum_{i=1}^n CF_i}{n}$$

$$SD = \sqrt{\frac{\sum_{i=1}^n (CF_i - \overline{CF})^2}{n - 1}}$$

$$RSD = \frac{SD}{\overline{CF}} \times 100$$

where n is the number of calibration standards and RSD is expressed as a percentage.

If the RSD of the calibration factors for each characteristic Aroclor[®] peak is less than or equal to 20% over the calibration range, then linearity through the origin may be assumed, and the average calibration factor may be used to determine sample concentrations.

If the RSD of the calibration factors is greater than 20 per cent over the calibration range then linearity through the origin cannot be assumed. In these cases a linear calibration using a least squares regression may be used for each characteristic Aroclor[®] peak. A five point calibration needs to be performed for each Aroclor[®] that is to be used for quantification purposes when a least squares regression is used.

The regression will produce the slope and intercept terms for a linear equation in the form:

$$y = ax + b$$

where:

y = Instrument response (peak area or height)

a = Slope of the line

x = Concentration of the calibration standard

b = Intercept

When using a regression the analyst must not force the line through the origin or include the origin as a data point, but have an intercepted line calculated

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from the five data points. The intercept value must be less than five percent of the response of the decision level standard to be used for quantitative purposes.

The regression calculation will generate a correlation coefficient (r) that is a measure of the 'goodness of fit' of the regression line to the data. To be used for quantitative purposes, r must be greater than or equal to 0.99.

9.5 Retention Time Windows [1,8]

Absolute retention times are generally used for compound identification. When absolute retention times are used, retention time windows are crucial to the identification of target compounds. Other approaches to the one described here may be used but they must be documented by the analyst.

Before establishing retention time windows, ensure that the chromatographic system is operating reliably and that the system has been optimised for PCBs.

Make five injections of each of the Aroclor[®] standards and surrogate over a 72 hour period.

Record the retention time (in minutes) for each of the three to five congeners that are to be used for quantification and surrogate to three decimal places (e.g. 0.007 min). Calculate the mean and standard deviation of the five absolute retention times for relevant congener and surrogate.

If the standard deviation of the retention times is 0.000 minutes, then use a default standard deviation of 0.01 minutes.

The width of the retention time windows for each congener and surrogate is defined as ± 3 times the

standard deviation of the mean absolute retention time established during the 72-hour period.

Establish the centre of the retention time window for each congener and surrogate by using the absolute retention time for each congener and surrogate from the calibration verification standard at the beginning of each analytical shift. For samples run during the same shift as an initial calibration, use retention time of the mid-point standard of the initial calibration.

Retention time windows must be calculated for each chromatographic column and instrument that the analysis is to be performed on. New retention time windows must be established when a new GC column is installed.

The surrogate retention time may be useful in tracking retention time shifts. Whenever the observed retention time of the surrogate is outside of the established retention time window, the analyst must determine the cause and correct the problem before continuing analyses.

9.6 Gas Chromatographic Analysis of Sample Extracts

The same GC operating conditions used for the initial calibration must be employed for the analysis of samples.

Calibration verification must be performed periodically during sample analysis. Further information on how to perform calibration verification is described in Section 11.2.

Inject a measured aliquot of Solution C into the GC. A 2 μ L aliquot is suggested, however the same injection volume must be used for both the

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calibration standards and the sample extracts.

Record the volume injected and the resulting peak size in area or height units.

9.6.1 Qualitative Identification

The identification of PCBs as Aroclor® is based on the agreement between the retention times of the peaks in the sample chromatogram with the retention time windows established through the analysis of the standards of the target analytes.

Tentative identification of an analyte occurs when a peak from a sample extract falls within the established retention time window for a specific target analyte. Confirmation is necessary when the sample composition is not well characterised. That is, if the Aroclor® present in the sample cannot be unambiguously identified or if suspect interference peaks appear in the chromatogram, confirmation on a second column is necessary.

Confirmation is performed using a second GC column of dissimilar stationary phase. The analyst should check the agreement between the quantitative results on both columns once the identification has been confirmed.

Retention time windows must have been established for the second GC column in order to be used for confirmation. In addition the analyst must demonstrate the sensitivity of the second column analysis. This demonstration must include the analysis of a standard of the target analyte at a concentration at least as low as the concentration estimated for the primary analysis. The standard may be either the individual Aroclor® or the Aroclor 1016/1260 mixture.

GC/MS confirmation may be used if the concentration is sufficient for detection by GC/MS. Refer to SW846 Method 8082A Sec. 7.10 [1] for further information.

9.6.2 Quantitation of PCBs

The quantitation of PCB residues as Aroclors® is accomplished by comparison of the sample chromatogram to that of the most similar Aroclor® standard. A choice must be made as to which Aroclor® is most similar to that of the residue and whether that standard is truly representative of the PCBs in the sample.

Use the Aroclor® 1221, 1232, 1242, 1248, and 1254 standards to determine the peak pattern of these Aroclors®. The patterns for Aroclor® 1016 and 1260 will be evident in the mixed calibration standards.

Once the Aroclor® pattern has been identified, compare the responses of three to five major peaks in the single point calibration standard for that Aroclor® with the peaks observed in the sample extract.

The amount of Aroclor® is calculated using the individual calibration factor for each of the three to five characteristic peaks. Those three to five concentrations are then averaged to determine the concentration of that Aroclor®.

If the chromatographic pattern contains mixed Aroclors®, compare the response of three to five major peaks in the single point calibration standard for each Aroclor® in the mixture with the peaks observed in the sample extract of each Aroclor®. It is essential that the peaks used for quantification are unique to the Aroclor® and do not overlap with any other Aroclor® peaks in the mixture.

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If the chromatographic pattern of the sample extract does not contain a recognisable Aroclor® pattern then measure the total area of the PCB pattern and quantitate on the basis of the Aroclor® standard that is most similar to the sample. Any peaks that are not identifiable on the basis of retention times should be subtracted from the total area. When quantitation is performed in this manner, the problems should be fully described for the data user and the specific procedures employed by the analyst should be thoroughly documented.

If the responses in the sample chromatogram exceed the calibration range of the system, dilute the extract and reanalyse. Dilute the extract using a solvent containing clean transformer oil at a concentration equivalent to that in the standards. Peak height measurements are recommended when overlapping peaks cause errors in area integration. If peak response is less than 2.5 times the baseline noise level, the validity of the quantitative result may be questionable. The analyst should consult with the source of the sample to determine whether further concentration of the sample is warranted.

Use the calibration standards analysed during the sequence to evaluate retention time stability. If any of the standards fall outside their daily retention time windows, the system is out of control. Determine the cause of the problem and correct it.

If compound identification or quantification is precluded due to interferences (for example broad, rounded peaks or ill-defined baselines are present), corrective action is warranted. Cleanup of the extract or replacement of the capillary column or detector may be necessary. The analyst may begin by

rerunning the sample on another instrument to determine if the problem results from analytical hardware of the sample matrix.

9.7 Calculation [8]

The concentration of each characteristic Aroclor® peak is calculated as follows:

$$\text{Concentration (mg/kg)} = \frac{A_s D}{CF V_i W_s}$$

where:

A_s = Area (or height) of the Aroclor® peak.

D = Total dilution of the sample (mL).

CF = Mean calibration factor from the initial calibration (area or height per ng).

V_i = Volume of the extract injected (μ L).

W_s = Weight of the sample diluted (g).

If a linear calibration that does not pass through the origin has been used, then the regression equation is rearranged to solve for x as follows:

$$x = \frac{y - b}{a}$$

where:

y = Instrument response (peak area or height)

a = Slope of the line

x = Concentration of the calibration standard

b = Intercept

When using this equation it is the analysts responsibility to ensure the calculation takes into account the weight of the original sample and the dilution factor.

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10 REPORTING RESULTS

- a** Report all data in mg/kg.
- b** Report the results obtained for each Aroclor[®] identified.
- c** If an Aroclor[®] was not detected report the result as non-detect quoting the detection limit in mg/kg.
- d.** If an Aroclor[®] result is below the reporting limit but greater than the detection limit report the result obtained noting that the result is below the reporting limit.
- e.** Add the results of all Aroclors[®] detected and report a total PCB concentration. A note should be included on the report that total PCB concentration refers to the sum of the Aroclors[®] analysed for.
- f.** If the sample extract contained an unrecognisable Aroclor[®] pattern report a total PCB concentration and what was used as the standard. For example 2.2mg/kg measured as Aroclor[®] 1242.
- g. If Aroclors[®] are not detected in the sample extract report non-detect for the total PCB result and quote a total detection limit. The total detection limit can be calculated by adding half the detection limits of all the Aroclors[®] used for quantification.**
- h** Report results with the associated uncertainty. The uncertainty should be calculated according to the ISO GUM method [9] or equivalent. For example 2.2 ± 0.3 mg/kg (95% confidence level).
- i** For results in the 1.7 to 2mg/kg range report the recovery data.

11 QUALITY CONTROL [1, 2, 8]

A formal quality control program is an integral part of this method. In addition before processing any samples the analyst should demonstrate through the analysis of a PCB-free oil sample, that all glassware and reagents are free of interferences.

Each time a set of samples is analysed or there is a change in reagents, a laboratory reagent blank should be processed as a safeguard against contamination.

11.1 Initial Generation of Accuracy and Precision Data

Before performing any analyses, the analyst must to generate accuracy and precision data with this method by analysing four separate samples of representative waste oil spiked with PCBs in the concentration range from 1 to 3mg/kg. A mixture of Aroclor[®] 1016 and 1260 can be used for this purpose.

An aliquot of the representative waste oil sample must be analysed prior to spiking to determine the PCB background level. The spike level must exceed twice the background level for the test to be valid.

Calculate the average per cent recovery (R) and the relative standard deviation (s) of the concentration found. Waste oil background corrections must be made before R calculations are performed.

The laboratory must develop and maintain separate accuracy statements of laboratory performance for waste oil samples. An accuracy statement for the method is defined as $R \pm s$. The accuracy statement should be developed by the analysis of four aliquots of waste oil followed by the calculation of R and s.

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Alternatively, the analyst may use four waste oil data points gathered through the requirements of continuing quality control. The accuracy statements should be updated regularly.

11.2 Calibration Verification

Verify calibration at least at the beginning and end of an analysis sequence. For sequences with more than 10 samples verify calibration once each 12 hour shift or every 10 samples whichever is more frequent. Calibration verification is done by injecting an Aroclor® 1016/1260 calibration standard. Calibration verification must be performed using the concentration standard corresponding to a PCB concentration in waste oil of 2mg/kg. The calibration verification process does not require analysis of the other Aroclor® standards used for pattern recognition, but the analyst may wish to include a standard for one of these Aroclors® after the 1016/1260 mixture used for calibration verification throughout the analytical sequence.

If a linear calibration curve through the origin has been used, calibration verification involves the calculation of the percent difference of the instrument response between initial calibration and each subsequent analysis of the verification standard.

$$\% \text{ Difference} = \frac{\overline{CF} - CF}{\overline{CF}} \times 100$$

If a linear calibration curve using the least square regression has been used, calibration verification involves the calculation of the percent drift of the instrument response between initial calibration and each subsequent analysis of the verification standard.

$$\% \text{ Drift} = \frac{\text{Calculated concentration} - \text{Theoretical concentration}}{\text{Theoretical concentration}} \times 100$$

If the percentage difference or percentage drift is less than ±15% then the initial calibration is considered valid and the analyst may continue to use the initial calibration values to quantitate samples.

If the percentage difference or percentage drift is greater than ±15% then the no sample analyses may take place until the calibration has been verified or a new initial calibration is performed.

When a calibration verification standard fails to meet the QC criteria, all samples that were injected after the last standard that meet the QC criteria must be evaluated to prevent misquantitations and possible false negative results, and re-injection of the sample extracts may be required.

If an analyte was not detected in the sample and the standard response is more than 15per cent below the initial calibration response, then re-injection is necessary. The purpose of this re-injection is to ensure that the analyte could be detected, if present, despite the change in the detector response, for example, to protect against a false negative result.

If an analyte was not detected in the sample and the standard response is greater than 15per cent above the initial calibration response, then re-injection is not necessary.

Sample injections may continue for as long as the calibration verification standards meet the instrument QC requirements. It is required that standards be analysed every 10 samples. The sequence ends when the full set of samples has

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been injected or when qualitative or quantitative QC criteria are met.

11.3 Matrix Spike, Surrogate Recoveries and Duplicates

At least 10per cent of the samples analysed must be analysed in duplicate. The duplicate can be an unspiked sample or a matrix spike duplicate. The decision on which to use must be based on knowledge of the sample batch and whether target analytes are expected to be present or not.

The laboratory is required to collect a portion of their samples in duplicate to monitor spike recoveries. If samples are not expected to contain target analytes the Aroclor® 1016/1260 mixture can be used for spiking. If specific Aroclors® are known to be present, the specific Aroclors® should be used for spiking. The frequency of spiked sample analysis

must be at least 10per cent of all samples or one sample per sequence, whichever is greater. One aliquot of sample must be spiked and analysed at a PCB concentration of 2mg/kg. The LCS recovery data may be used instead of the matrix spike recovery data if the matrix spike recovery is invalid due to high background levels

A laboratory control sample should be included with each analytical batch. When the results of the matrix spike analysis indicative a potential problem due to the sample matrix itself, the LCS results are used to verify that the laboratory can perform the analysis in a clean matrix.

The laboratory must also evaluate surrogate recovery data from individual samples versus the surrogate control limits.

Matrix spike and surrogate recovery is calculated as follows:

$$\text{Recovery (\%)} = \frac{\text{Conc. (or amount) found} - \text{Conc. (or amount) in unspiked sample}}{\text{Conc. (or amount) added}} \times 100$$

If recovery is not within the limits described in Sec 11.3.1, corrective action is necessary.

If no problem is found, the sample should be re-extracted and re-analysed. If, upon re-analysis the recovery is again not within limits, report the data as an 'estimated concentration.' If the recovery is within the limits in the re-analysis, provide the re-analysis data to the data user.

11.3.1 Performance criteria for matrix spike and surrogate recoveries.

Calculate the average percentage recovery (p) and the standard deviation (s) for the surrogate after the analysis of 15 to 20 field samples.

Calculate the upper and lower control limit for each matrix spike or surrogate compound:

$$\begin{aligned} \text{Upper control limit} &= p + 3s \\ \text{Lower control limit} &= p - 3s \end{aligned}$$

Calculate warning limits as:

$$\begin{aligned} \text{Upper warning limit} &= p + 2s \\ \text{Lower warning limit} &= p - 2s \end{aligned}$$

Any results outside the control limits require evaluation by the laboratory.

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APPENDIX I PREPARATION OF CLEAN TRANSFORMER OIL

Add a 15 mL portion of the clean transformer oil into a 40mL narrow mouth screw cap bottle. Add 15mL of concentrated sulfuric acid into the bottle. Seal the bottle with a Teflon lined screw cap and shake or vortex mix for one minute. Depending on the degree of contamination the colour of the acid layer will range from yellow to dark brown.

Allow the phases to separate (centrifuge if necessary), remove the lower acid layer. Repeat the process until no change in colour of either layer is seen.

Wash the oil layer with several 15mL portions of reagent water until the wash water is neutral to pH paper.

A check must be made before adding the cleaned transformer oil to the standard solutions to determine if there are any chromatographic peaks that will interfere with the PCB analysis.

APPENDIX II SULFUR CLEANUP METHODS

II.1 Copper Powder Procedure [10]

This technique requires the copper powder to be very reactive, as evidenced by a bright shiny appearance.

II.1.1 Apparatus and Materials

II.1.1.1 Mechanical shaker or mixer

Vortex Genie or equivalent.

II.1.1.2 Pipettes, disposable

Pasteur type.

II.1.1.3 12mL Centrifuge tubes calibrated

II.1.2 Reagents

II.1.2.1 Dilute Nitric acid, HNO₃ II.1.2.2 Organic free reagent water

II.1.2.3 Acetone

Pesticide quality or equivalent

II.1.2.4 Copper powder

(fine granular Mallinckrodt 4649 or equivalent)

Remove oxides by treating with dilute nitric acid, rinse with reagent water to remove all traces of acid, rinse with acetone and dry under a stream of nitrogen.

II.1.3 Procedure

Concentrate the sample extract to a known volume. Add approximately 2 g of cleaned copper powder to the centrifuge tube. Vigorously mix the extract and copper powder for at least 1 minute. Allow the phases to separate.

Separate the extract from the copper by drawing off the extract with a pipette and transfer to a clean vial for further cleanup or analysis.

II.2 Copper Strip Procedure

II.2.1 Reagents

II.2.1.1 Nitric acid, HNO₃ 20%

II.2.1.2 Organic free reagent water

II.2.1.3 Acetone

Pesticide quality or equivalent.

II.2.1.4 Copper foil

(Analar-BDH or equivalent) Cut foil into strips approximately 30 mm x 3 mm. React copper with 20% nitric acid solution briefly until gas evolution is

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established and the solution turns a pale blue. Remove from acid and quickly, rinse the foil with water, then acetone, then hexane. Use immediately.

II.2.2 Procedure

Concentrate the sample extract to a known volume. Add two copper strips to the extract and allow to react for 1 to 2 hours. If the strips are blackened during this time, add further strips and treat again, until no further blackening occurs. Remove the strips, rinsing with hexane and adjust the solution volume to the original volume with a stream of high purity nitrogen. Use the extract for further cleanup or analysis.

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APPENDIX III EXAMPLE AROCLOR® CHROMATOGRAMS

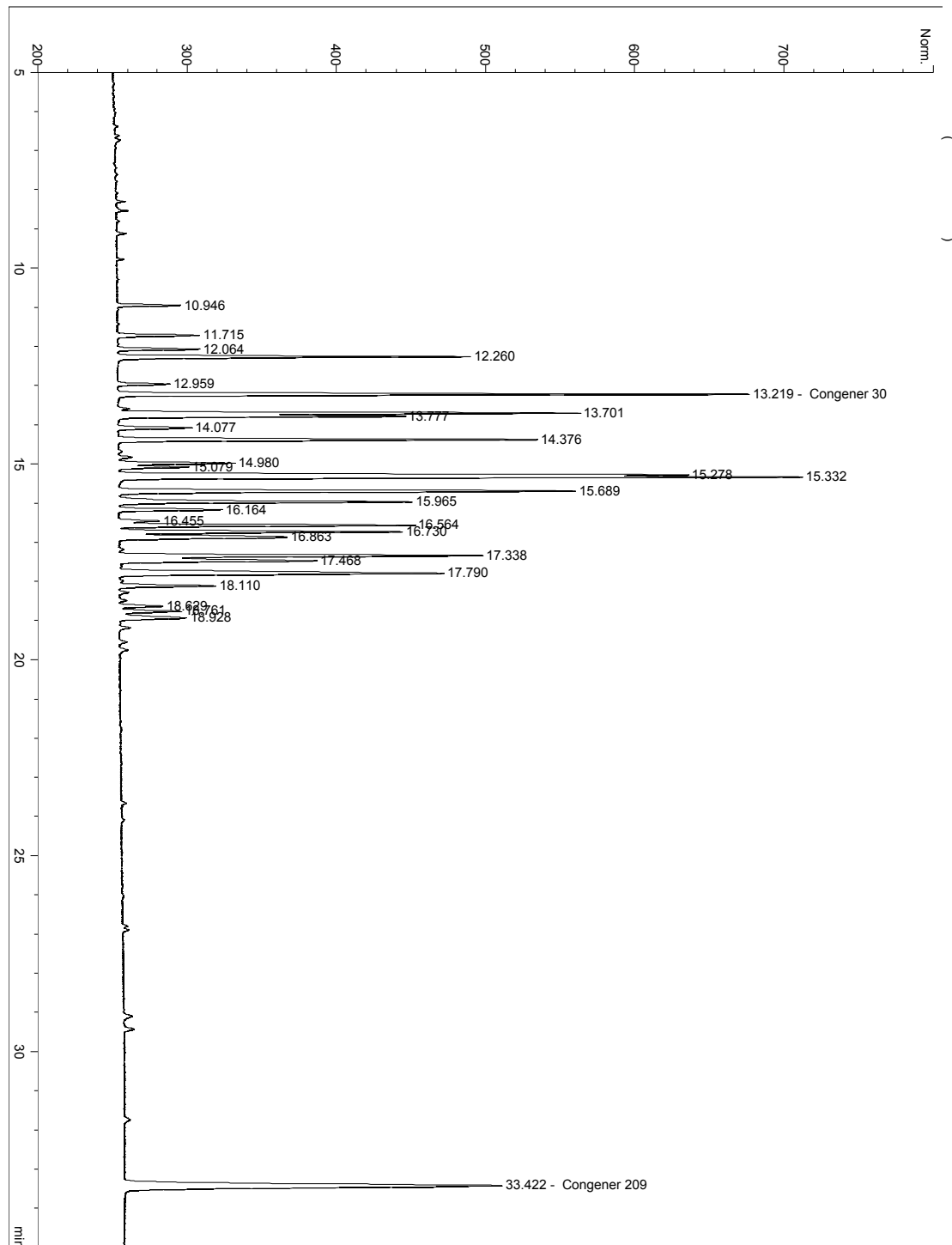


Figure III.1. Example GC/ECD chromatogram of Aroclor® 1016 analysed on a BP5 column (30 m x 0.32 mm, 0.5 µm film thickness). Temperature program: 100°C (2 min hold) to 160°C at 15°C/min, to 270°C at 5°C/min (10 min hold). Congener 30 and 209 added to standard as reference peaks.

DETERMINATION OF PCBs IN WASTE OILS BY GAS CHROMATOGRAPHY WITH ELECTRON CAPTURE DETECTOR

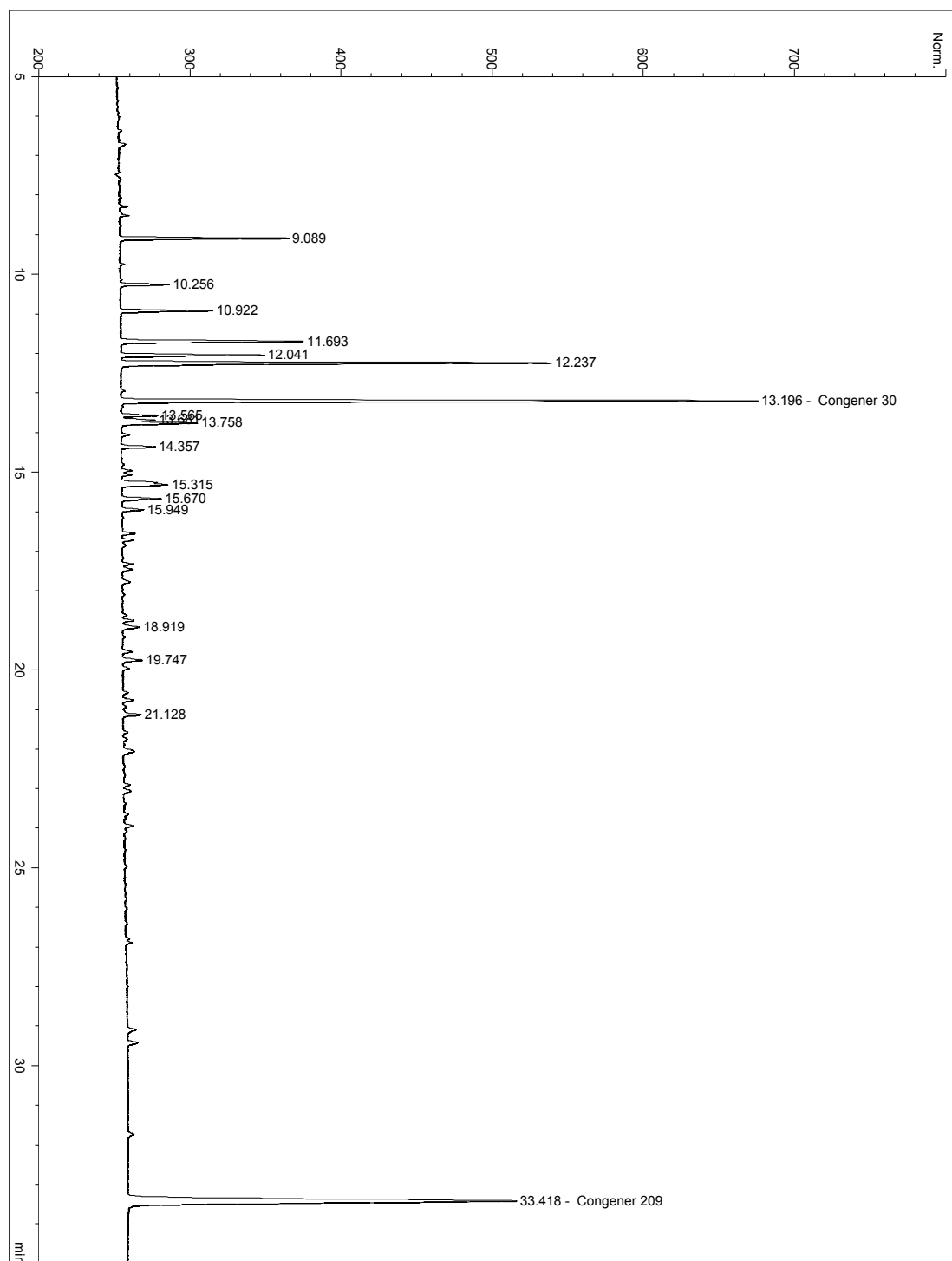


Figure III.2. Example GC/ECD chromatogram of Aroclor[®] 1221 analysed on a BP5 column (30 m x 0.32 mm, 0.5 µm film thickness). Temperature program: 100°C (2 min hold) to 160°C at 15°C/min, to 270°C at 5°C/min (10 min hold). Congener 30 and 209 added to standard as reference peaks.

DETERMINATION OF PCBs IN WASTE OILS BY GAS CHROMATOGRAPHY WITH ELECTRON CAPTURE DETECTOR

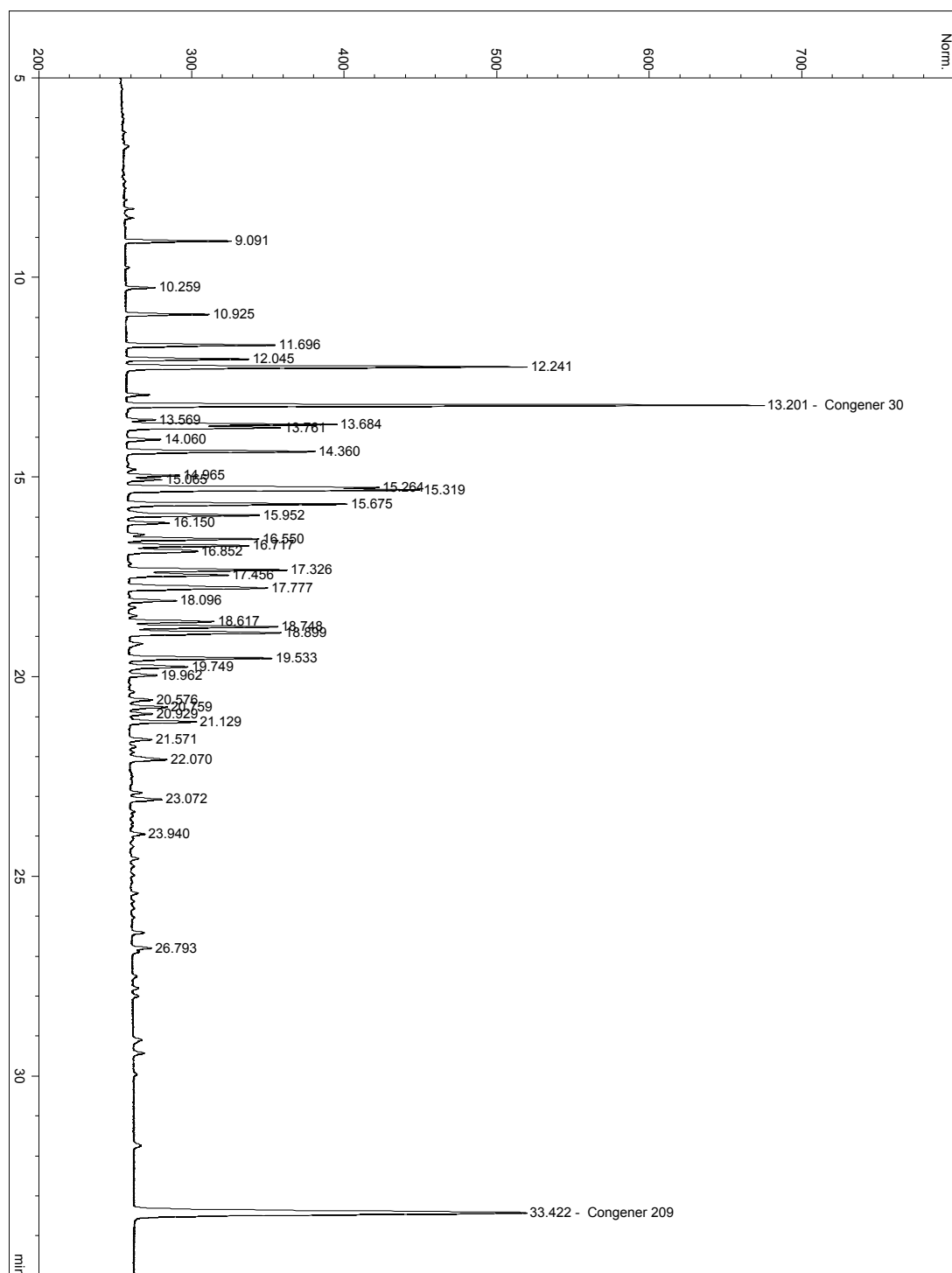


Figure III.3. Example GC/ECD chromatogram of Aroclor[®] 1232 analysed on a BP5 column (30 m x 0.32 mm, 0.5 µm film thickness). Temperature program: 100°C (2 min hold) to 160°C at 15°C/min, to 270°C at 5°C/min (10 min hold). Congener 30 and 209 added to standard as reference peaks.

DETERMINATION OF PCBs IN WASTE OILS BY GAS CHROMATOGRAPHY WITH ELECTRON CAPTURE DETECTOR

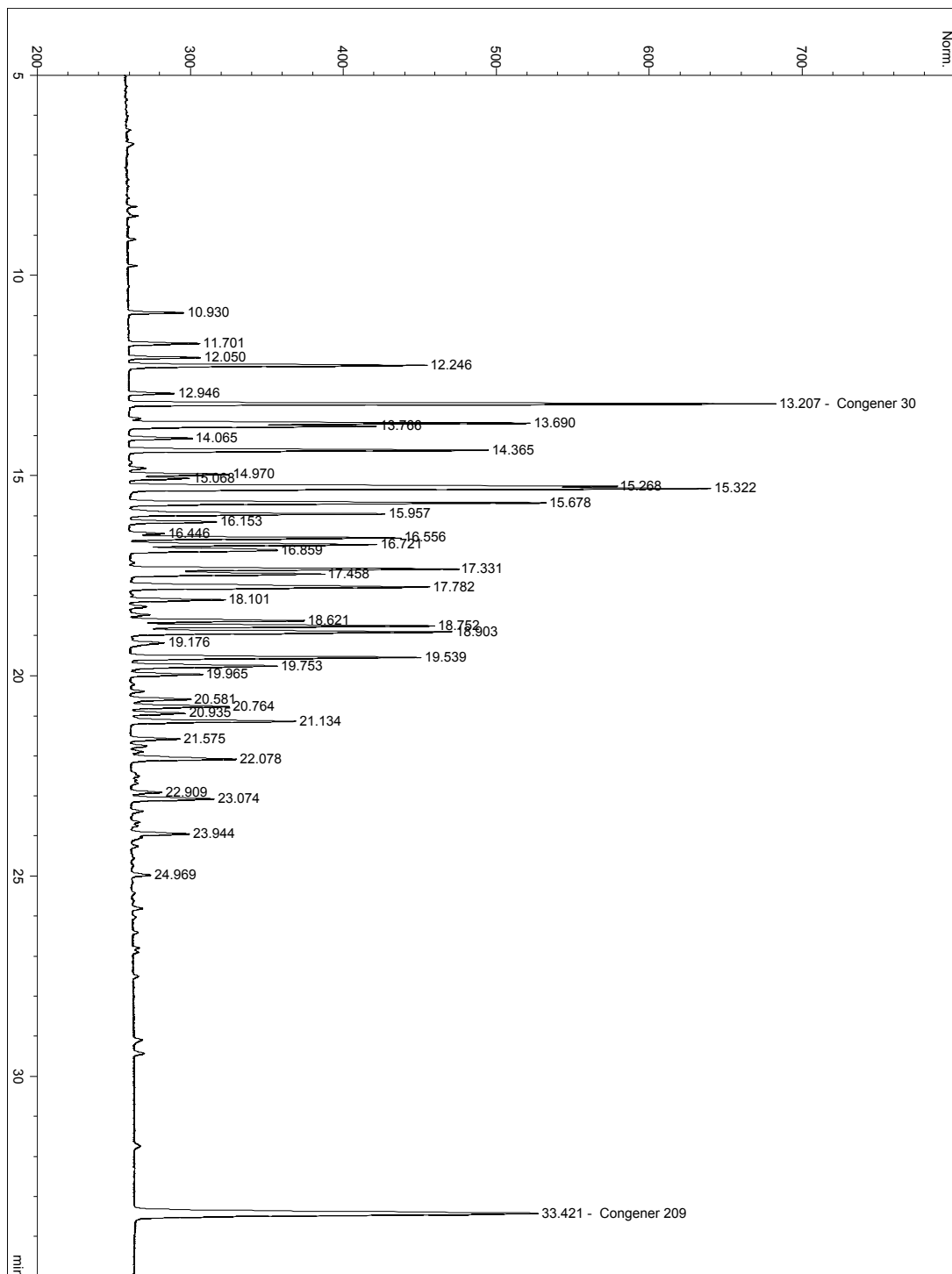


Figure III.4. Example GC/ECD chromatogram of Aroclor® 1242 analysed on a BP5 column (30 m x 0.32 mm, 0.5 µm film thickness). Temperature program: 100°C (2 min hold) to 160°C at 15°C/min, to 270°C at 5°C/min (10 min hold). Congener 30 and 209 added to standard as reference peaks.

DETERMINATION OF PCBs IN WASTE OILS BY GAS CHROMATOGRAPHY WITH ELECTRON CAPTURE DETECTOR

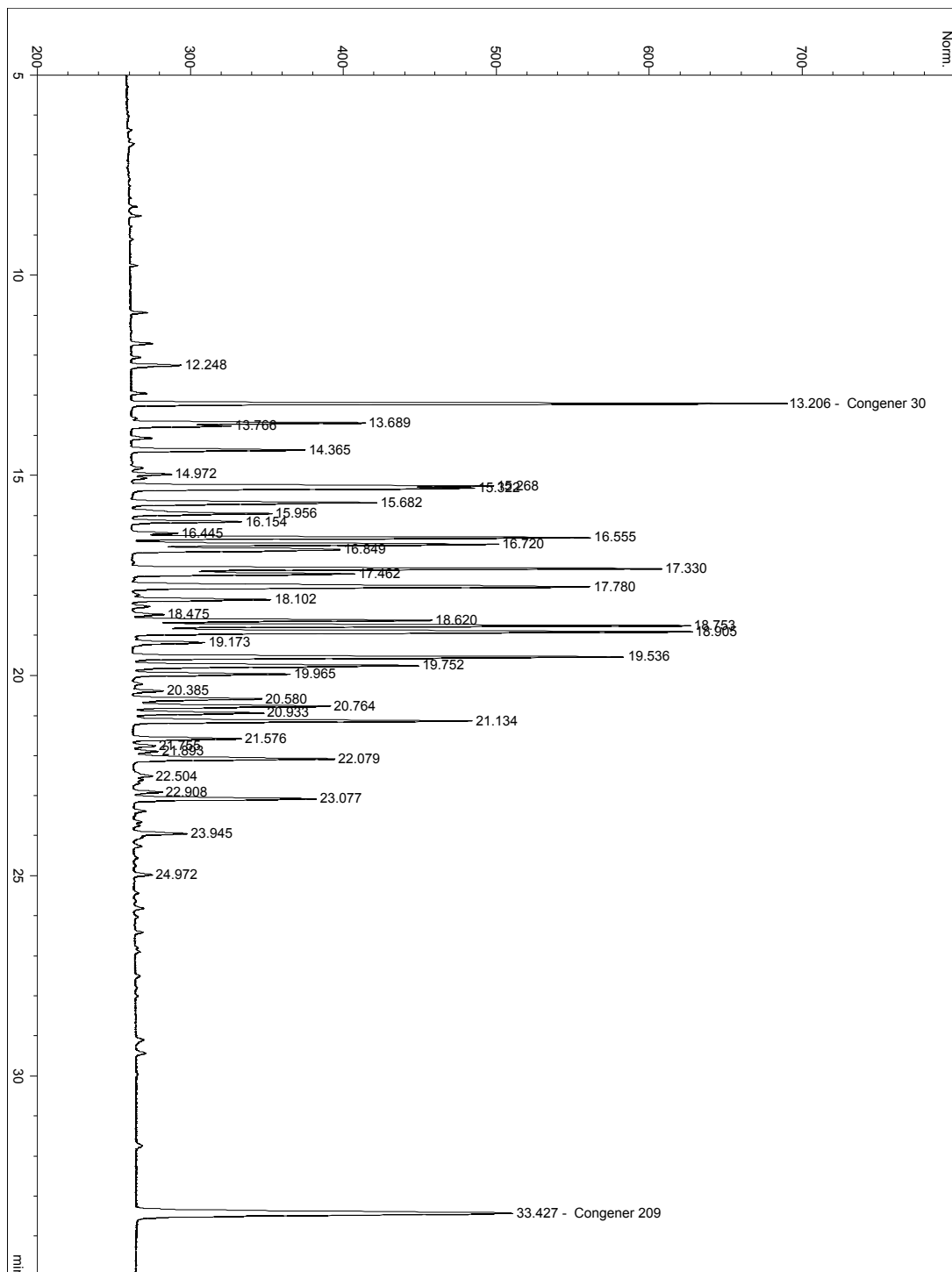


Figure III.5. Example GC/ECD chromatogram of Aroclor® 1248 analysed on a BP5 column (30 m x 0.32 mm, 0.5 µm film thickness). Temperature program: 100°C (2 min hold) to 160°C at 15°C/min, to 270°C at 5°C/min (10 min hold). Congener 30 and 209 added to standard as reference peaks.

DETERMINATION OF PCBs IN WASTE OILS BY GAS CHROMATOGRAPHY WITH ELECTRON CAPTURE DETECTOR

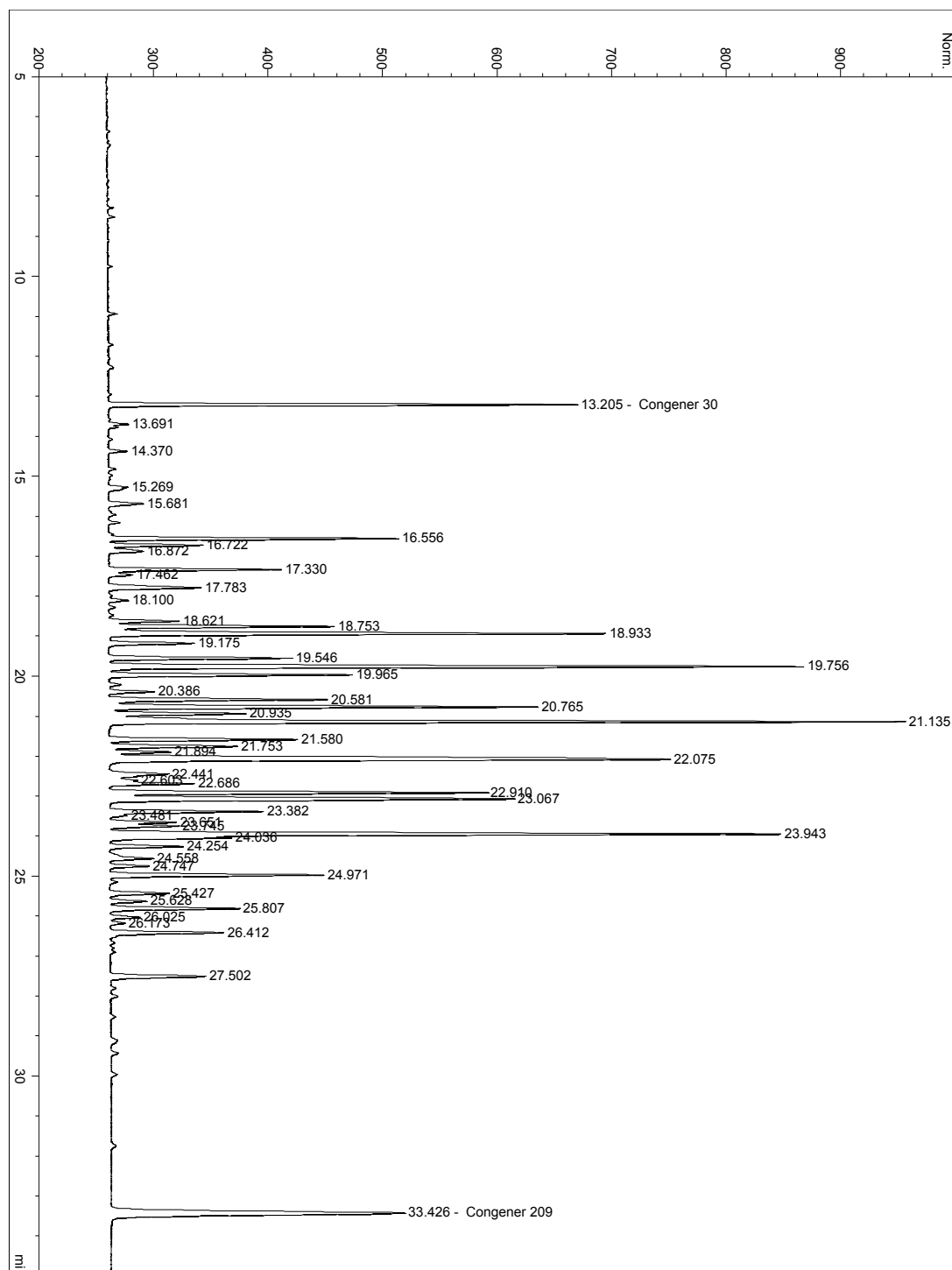


Figure III.6. Example GC/ECD chromatogram of Aroclor[®] 1254 analysed on a BP5 column (30 m x 0.32 mm, 0.5 μ m film thickness). Temperature program: 100°C (2 min hold) to 160°C at 15°C/min, to 270°C at 5°C/min (10 min hold). Congener 30 and 209 added to standard as reference peaks.

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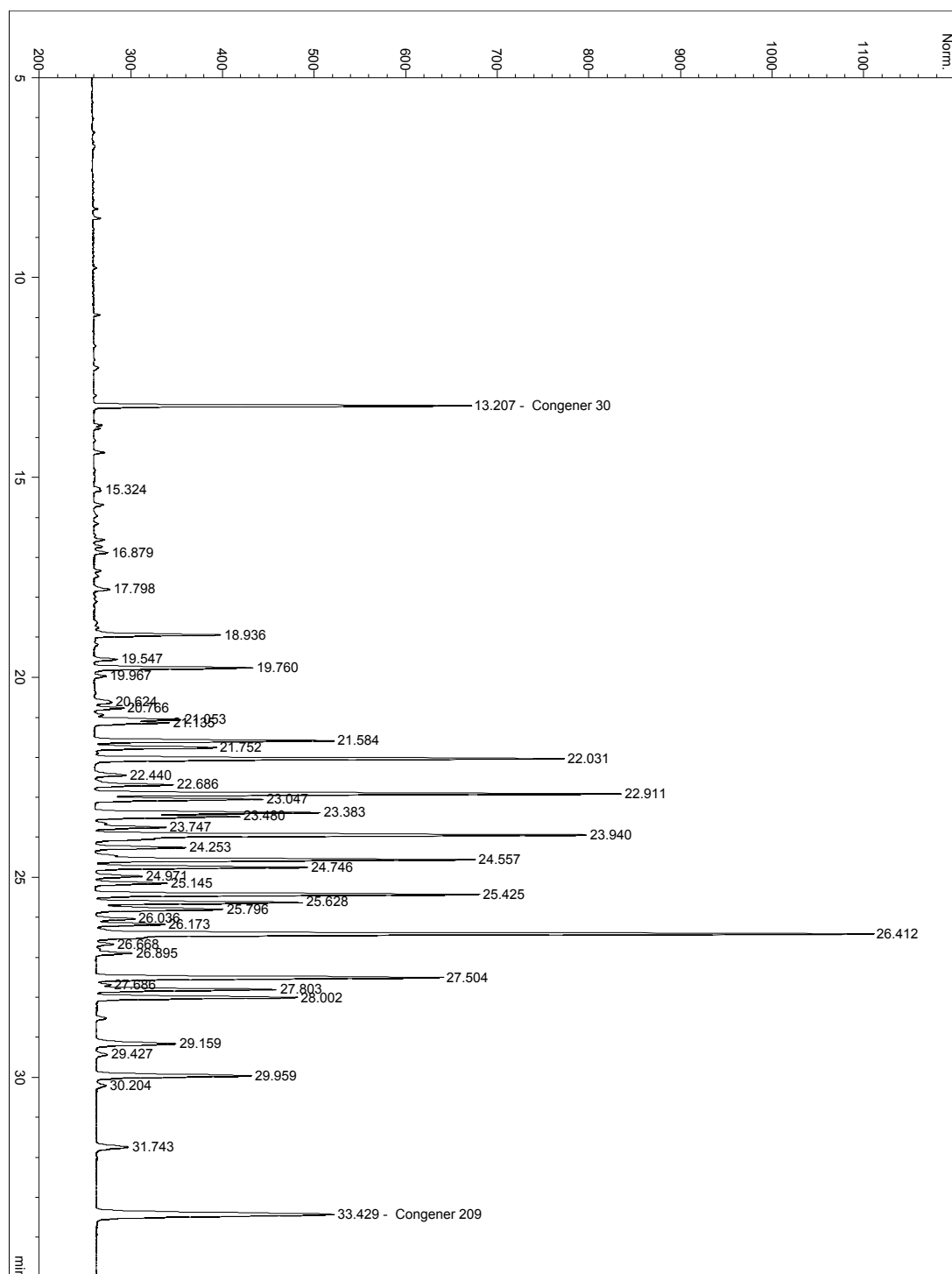


Figure III.7. Example GC/ECD chromatogram of Aroclor® 1260 analysed on a BP5 column (30m x 0.32mm, 0.5µm film thickness). Temperature program: 100°C (2 min hold) to 160°C at 15°C/min, to 270°C at 5°C/min (10 min hold). Congener 30 and 209 added to standard as reference peaks.

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APPENDIX IV REFERENCES

- [1] SW 846 Method 8082A, *Polychlorinated Biphenyls (PCBs) by Gas Chromatography*, United States Environmental Protection Agency, November 2000.
- [2] EPA-600/4-81-045, *The Determination of Polychlorinated Biphenyls in Transformer Fluid and Waste Oils*, United States Environmental Protection Agency, September 1982.
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- [5] AS 1767.2.7-1999, *Insulating Liquids Part 2: Test Methods, Method 2.7: Determination of PCB contamination in Insulating Liquids by Capillary Column Gas Chromatography – Identification of Congeners*, Standards Australia, 1999.
- [6] SW846 Method 3630C, *Silica Gel Cleanup*, United States Environmental Protection Agency, December 1996.
- [7] SW846 Method 3620B, *Florosil® Cleanup*, United States Environmental Protection Agency, December 1996.
- [8] SW846 Method 8000B, *Determinative Chromatographic Separations*, United States Environmental Protection Agency, December 1996.
- [9] *Guide to the Expression of Uncertainty in Measurement*. ISO, Geneva, 1993. (ISBN 92-67-10188-9)
- [10] SW846 Method 3660B, *Sulfur Cleanup*, United States Environmental Protection Agency.