

The Determination of Material Extractable by Carbon Tetrachloride and of certain Hydrocarbon Oil and Grease Components in Sewage Sludge 1978

Methods for the Examination of Waters and Associated Materials

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This booklet contains two methods with a common extraction procedure. Both methods are often carried out on the same sample and allowance has been made for this in the extraction procedure. The first method, which is solvent extraction gravimetric, determines the material extractable by carbon tetrachloride. The second method partially separates this extracted material by type of compound. This second method is chromatographic; such methods are very sensitive to minor physical and chemical variations in the quality of the materials and apparatus used, hence this method mentions the actual materials used for the evaluation tests. This in no way endorses these materials as superior to other similar materials. Equivalent materials are acceptable, though it must be understood that the performance characteristics may be different, and can vary with the batch of material used. It is left to the supervising analyst to evaluate and choose from the appropriate brands available.

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Warning to users

The analytical procedures given in this booklet should only be carried out by competent trained persons, with adequate supervision when necessary. Local Safety Regulations must be observed. Laboratory procedures should be carried out only in a properly equipped laboratory. Field operations should be conducted with due regard to possible local hazards, and portable safety equipment should be carried. Care should be taken against creating hazards for others. Lone working, whether in the laboratory or field, should be discouraged. Reagents of adequate purity must be used, along with properly maintained apparatus and equipment of correct specification. Specifications for reagents, apparatus and equipment are given in manufacturers' catalogues and various published standards. If contamination is suspected, reagent purity should be checked before use.

There are numerous handbooks on first aid and laboratory safety. One such publication is 'Code of Practice for Chemical Laboratories' issued by the Royal Institute of Chemistry, London. Another such publication, which includes biological hazards, is 'Safety in Biological Laboratories' (editors E Hartree and V Booth), Biochemical Society Special Publication No 5, The Biochemical Society, London.

Where the committee have considered that a special unusual hazard exists, attention has been drawn to this in the text so that additional care might be taken beyond that which should be exercised at all times when carrying out analytical procedures. It cannot be too strongly empha-

sised that prompt first aid, decontamination, or administration of the correct antidote can save life, but that incorrect treatment can make matters worse. It is suggested that both supervisors and operators be familiar with emergency procedures before starting even a slightly hazardous operation, and that doctors consulted after any accident involving chemical contamination, ingestion, or inhalation, be made familiar with the chemical nature of the injury, as some chemical injuries require specialist treatment not normally encountered by most doctors. Similar warning should be given if a biological or radiochemical injury is suspected. Some very unusual parasites, viruses and other micro-organisms are occasionally encountered in samples and when sampling in the field. In the latter case, all equipment including footwear should be disinfected by appropriate methods if contamination is suspected.

The best safeguard is a thorough consideration of hazards and the consequent safety precautions and remedies well in advance. Without intending to give a complete checklist, points that experience has shown are often forgotten include: laboratory tidiness, stray radiation leaks (including ultra violet), use of the correct protective clothing and goggles, removal of toxic fumes and wastes, containment in the event of breakage, access to taps, escape routes, and the accessibility of the correct and properly maintained first aid, fire-fighting and rescue equipment. If in doubt it is safer to assume that a hazard may exist and take reasonable precautions rather, than to assume that no hazard exists until proved otherwise.

About this series

This booklet is one of a series intended to provide recommended methods for the determination of water quality. In the past, the Department of the Environment and its predecessors, in collaboration with various learned societies, has issued volumes of methods for the analysis of water and sewage culminating in 'Analysis of Raw, Potable and Waste Waters'. These volumes inevitably, took some years to prepare, so that they were often partially out of date before they appeared in print. The present series will be published as individual methods, thus allowing for the replacement or addition of methods as quickly as possible without need of waiting for the next edition. The rate of publication will also be related to the urgency of requirement for that particular method, tentative methods being issued when necessary. The aim is to provide as complete and up to date a collection of methods and reviews as is practicable, which will, as far as possible, take into account the analytical facilities available in different parts of the Kingdom, and the quality criteria of interest to those responsible for the various aspects of the water cycle. Because both needs and equipment vary widely, where necessary, a selection of methods may be recommended for a single determinand. It will be the responsibility of the users – the senior analytical chemist, biologist, bacteriologist etc. to decide which of these methods to use for the determination in hand. Whilst attention of the user is drawn to any special known hazards which may occur with the use of any particular method, responsibility for proper supervision and the provision of safe working conditions must remain with the user.

The preparation of this series and its continuous revision is the responsibility of the Standing Committee of Analysts (to review Standard Methods for Quality Control of the Water Cycle). The Standing Committee of Analysts is one of the joint technical committees of the Department of the Environment and the National Water Council. It has nine Working Groups, each responsible for one section or aspect of water cycle quality analysis. They are as follows:

- 1.0 General principles of sampling and accuracy of results
- 2.0 Instrumentation and on-line analysis
- 3.0 Empirical and physical methods
- 4.0 Metals and metalloids
- 5.0 General non-metallic substances
- 6.0 Organic impurities
- 7.0 Biological methods
- 8.0 Sludge and other solids analysis
- 9.0 Radiochemical methods

The actual methods etc are produced by smaller panels of experts in the appropriate field, under the overall supervision of the appropriate working group and the main committee. The names of those associated with this method are listed inside the back cover.

Publication of new or revised methods will be notified to the technical press, whilst a list of Methods in Print is given in the current HMSO Sectional Publication List No 5, and the current status of publication and revision will be given in the biennial reports of the Standing Committee of Analysts.

TA DICK
Chairman

LR PITWELL
Secretary

20 July 1977

The Determination of Material Extractable by Carbon Tetrachloride and of Certain Hydrocarbon Oil and Grease Components in Sewage Sludge

Tentative Method (1978 version)

Introduction

These methods, like the majority of methods for hydrocarbons and greases, are empirical. The materials determined are decided by the procedure used.

The International Organization for Standardization (ISO) draft definition of oil and grease is as follows:

'Hydrocarbons, hydrocarbon derivatives and all liquid or unctuous substances that have boiling points of 90°C or above and are extractable from water using a specified solvent'.

Other similar definitions exist.

Because of the wide range of sources and properties of materials defined as oil, no one analytical method can be expected to determine all the substances so described. The analytical method in this document is for:

(i) material extractable by carbon tetrachloride under defined conditions.

The substances determined will include fats, all types of hydrocarbons and other materials that are retained in the extract under the conditions used for the evaporation of the solvent.

(ii) certain hydrocarbon oil components separated from the carbon tetrachloride extract by column chromatography.

These substances include the aliphatic, alicyclic and some mononuclear aromatic hydrocarbons in the carbon tetrachloride extract. More polar oil components which are present in materials such as residual fuel oils, bitumen and creosote are retained by the chromatographic column.

Because the test is empirical, it has been devised to give as meaningful results as possible.¹

1 Performance Characteristics of the Method

(For further information on the determination and definition of performance characteristics, see another publication in this series).

1.1 Determination of Material Extractable by Carbon Tetrachloride from Sewage Sludge.

1.1.1	Substance Determined	Those substances which are extracted from sewage sludge by carbon tetrachloride and remain after evaporation of the solvent at 105°C
1.1.2	Type of Sample	Sewage sludge.
1.1.3	Basis of Method	The sludge is extracted with carbon tetrachloride. The solvent is evaporated and the residue dried at 105°C. The material extracted is weighed.
1.1.4	Range of Application	Up to 1 gm in the sample.
1.1.5	Standard Deviation (a)	80 mg/l at a concentration of 511 mg/l.
1.1.6	Limit of Detection	Not determined.
1.1.7	Sources of Error	See section 3 below.

1.1.8	Time required for analysis (a)	Assuming a 16 hour extraction period, total time 18 hours, operator time for one determination 2 man hours. With multiple apparatus, one operator can perform 12 analyses per man day. If both determinations are carried out, the times required are similar to those given in Section 1.2.8.
1.2 Determination of Certain Hydrocarbon Oil and Grease Components in Sewage Sludge.		
1.2.1	Substance Determined	Those substances in the carbon tetrachloride extract which remain in the extract after passage through silica gel and Florisil columns i.e., aliphatic, alicyclic and some mono-aromatic hydrocarbons.
1.2.2	Type of Sample	Sewage sludge.
1.2.3	Basis of Method	A solution of the extract is passed through columns of Florisil, which removes polar materials, and through silica gel which removes most aromatic compounds. The solvent is then evaporated and the residue dried at 105°C. The residue is weighed.
1.2.4	Range of Application	Variable with the fat and aromatics content of the sludge but typically: Crude sewage sludge – up to 400 mg in the sample. Digested sludge – up to 1 g in the sample.
1.2.5	Standard Deviation (a)	18 mg/l at a concentration of 237 mg/l.
1.2.6	Limit of Detection	Not determined.
1.2.7	Sources of Error	See section 3 below.
1.2.8	Time required for analysis (a)	Assuming a 16 hour extraction period, total time 20 hours, operator time for one determination 4 man hours. With multiple apparatus, one operator can perform 6 analyses per man day. If both determinations are carried out, the times required are similar to those given above. It is unlikely that this determination will be performed on its own.
(a) Results obtained by Thames Water Authority on a sample of digested sludge (8 replicates in one batch).		

2 Principle

2.1 Determination of Material extractable by carbon tetrachloride from sewage sludge.

Sewage sludge is continuously extracted for at least 8 hrs using carbon tetrachloride. The solvent is removed from half the extract by evaporation, firstly in a rotary evaporator then using a steam bath. The residue is dried at 105°C and then weighed.

2.2 Determination of certain hydrocarbon oil and grease components in sewage sludge.

A portion of the second half of the carbon tetrachloride extract is passed through a

column containing Florisil, which removes polar materials, and silica gel, which removes most aromatic compounds. The solvent is removed from the eluate using firstly a rotary evaporator and then a steam bath. The residue is dried at 105°C and then weighed.

3 Sources of Error

3.1 Determination of material extractable by carbon tetrachloride from sewage sludge.

Provided the procedure is followed correctly, there are no serious sources of error.

3.2 Determination of certain hydrocarbon oil and grease components in sewage sludge.

The chromatographic columns described (see section 6.2) will retain 200 mg of polar materials (fats) and 200 mg of aromatics. If the columns are overloaded, polar and aromatic materials will pass into the eluate, giving a falsely high result.

4 Hazards

The procedure described uses carbon tetrachloride which is toxic. Avoid inhalation, skin contact and ingestion. Carbon tetrachloride can be an anaesthetic and repeated heavy exposure can cause liver damage.

5 Reagents

5.1 Except where otherwise stated, analytical reagent grade chemicals must be used. Reagents should be stored in glass bottles. All reagents except activated chromatograph packing materials, are stable for at least one month.

Reagents Common to both Methods

5.2 **Carbon tetrachloride**

5.3 **Water**

All water used must be distilled from an all glass or metal apparatus and stored in glass (contact with plastics and grease can cause high blanks).

5.4 **Sodium bicarbonate**

5.5 **Sodium sulphate**, anhydrous granular.

Determination of Material extractable by Carbon Tetrachloride.

No additional reagents are required.

Determination of Certain Hydrocarbon Oil and Grease Components.

5.6 **Florisil** 30/60 mesh (or equivalent)

Activate the required amount of Florisil by heating for 2 hours at $180 \pm 5^\circ\text{C}$ and cool in a desiccator. Store in a desiccator over silica gel. Prepare daily. (see also Section 6.2.3).

5.7 **Silica Gel** (*Davison 923* or equivalent)

Activate and store the required amount of silica gel in the same way as is given for Florisil in section 5.6. (See also Section 6.2.3).

5.8 **0.2% m/V n-hexadecane** solution in carbon tetrachloride.

Dissolve 200 ± 2 mg of n-hexadecane (better than 99% purity) in 100 ± 10 ml of carbon tetrachloride.

5.9 **0.2% m/V Glyceryl tristearate** solution in carbon tetrachloride.

Dissolve 200 ± 2 mg of glyceryl tristearate (better than 99% purity) in 100 ± 10 ml of carbon tetrachloride.

5.10 **0.2% m/V Anthracene** solution in carbon tetrachloride

Dissolve 200 ± 2 mg of anthracene (better than 99% purity) in 100 ± 10 ml of carbon tetrachloride.

5.19 **Cotton wool** – washed with carbon tetrachloride.

6 Apparatus

Greased joints and seals must not be used.

6.1 Glassware

6.1.1 *Extraction Apparatus* – 1 litre capacity continuous extraction apparatus for heavier than water solvents. Use with a 250 ml solvent flask (see fig. 1).

6.1.2 *Rotary Evaporator*

6.1.3 *Air Line* – air cleaned by passage through a molecular sieve and silica gel, the line terminating in a fine jet of glass or metal and controllable such that the air jet indents the surface of the solvent without splashing (typically 200 ml/min of air from a jet of internal diameter 0.5 mm at a distance of 2 cm from the liquid).

6.2 Preparation of Chromatography Columns

The packed column described below is suitable for removing up to 200 mg fatty material plus up to 200 mg of aromatic material. Prepare columns immediately prior to use.

6.2.1 Use glass columns of dimensions approximately 14 mm internal diameter and 750 mm long terminating in a tap. A 50 ml burette is suitable.

6.2.2 *Preparation*

Plug the column lightly with a small piece of cotton wool. Add 10.0 ± 0.5 g silica gel with

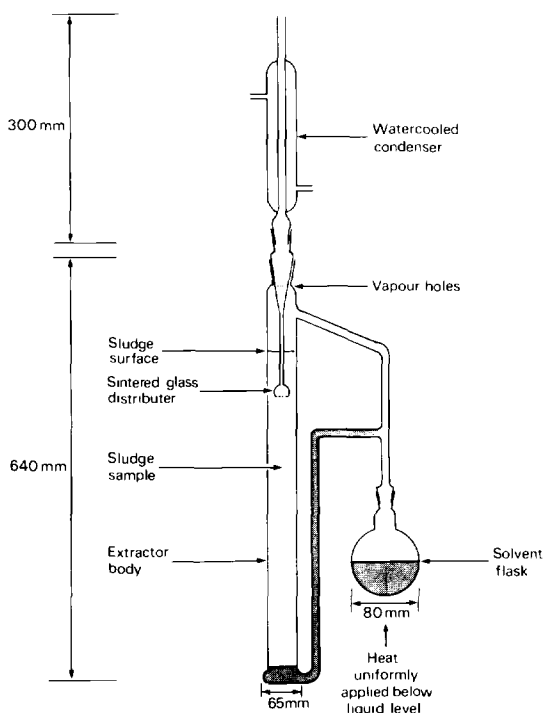


Figure 1. Continuous downward displacement Extraction Apparatus (Carbon tetrachloride layers indicated by shading)

tapping, followed by 9.0 ± 0.5 g Florisil with tapping and finally 2 ± 0.5 g anhydrous sodium sulphate.

6.2.3 *Checking Column Activity*

The activity of the silica gel and Florisil should be checked whenever new batches are used.

Use the solutions described in sections 5.8, 5.9 and 5.10 separately as samples in section 8.3. If the activities of the silica gel and Florisil are correct, more than 95% of the n-hexadecane will be recovered while more than 95% of the anthracene and glyceryl tristearate will be retained on the column. If satisfactory recoveries are not obtained the activation procedures must be modified as appropriate (see below).

As a guide, if the packing is too retentive, reduce the activation time or temperature; if the packing is not sufficiently retentive, increase either the activation time or the column packing length. For Florisil only, but not for silica gel, the activation temperature may be increased slightly.

7 Sample Collection and Preservation

Take as representative a sample of sludge as possible (see another booklet in this series⁽²⁾), *reference should also be made to the general booklet on sampling also published in this series.* Carbon tetrachloride extractable materials may degrade on storage giving lower results, but no quantitative information is available. The aliphatic, alicyclic and mononuclear aromatic hydrocarbons are unlikely to change on storage but no quantitative information is available. The analysis should be carried out as quickly as possible after sampling.

8 Analytical Procedure

READ SECTION 4 ON HAZARDS BEFORE STARTING THESE PROCEDURES

Step	Experimental Procedure	Notes
8.1	Extraction procedure common to both determinations	
8.1.1	<i>Extraction</i> Take a known volume (V_1 ml) of sewage sludge containing between 4 and 8 g dry matter. Add 10 ± 1 g of sodium bicarbonate and stir to dissolve. (notes a and b).	(a) The volume of sample may be varied outside the dry matter limits given if particularly high or low oil contents are expected. (b) If the results are to be reported on a dry weight basis it will be necessary to carry out a subsidiary determination of total solids on the bulk sample from which an aliquot is taken for extraction. (See another method in this series)
8.1.2	Place 200 ± 10 ml of carbon tetrachloride in the bottom of the extractor and 100 ± 5 ml in the solvent flask. Add the sample to the extractor and then add only sufficient distilled water to cause the solvent in the extractor body just to overflow into the solvent flask (note c). Extract for a minimum of 8 hours (note d), adjusting the rate of heating to give good droplet formation in the extractor.	(c) The total volume of aqueous phase in the extractor will vary with the apparatus but will be approximately 1 litre. The volume of solvent in the extractor flask will vary with the apparatus, but should be such that localised overheating is avoided. Other sizes of extraction apparatus may be suitable for the method. (d) A convenient period of extraction is overnight (approximately 16 hours).
8.1.3	<i>Drying</i> Cool the extract to below 25°C . Dry the extract in the solvent flask using sodium sulphate. Decant the extract through a filter funnel containing a cotton wool plug into a 500-ml measuring cylinder. Rinse the solvent flask twice with 10 ± 2 ml portions of carbon tetrachloride and pour through the filter into the measuring cylinder. Mix and note the volume of the combined extracts V_2 ml.	
8.1.4	If the determination of hydrocarbon in section 8.3 is to follow, divide the extract into two equal portions. Use one portion for step 8.2; use the other portion for step 8.3.	
8.2	Determination of Materials Extractable by Carbon Tetrachloride (note e)	
8.2.1	<i>Evaporation</i> Evaporate one portion of the extract from step 8.1.4 to 2 ± 1 ml using a rotary evaporator (notes e and f).	(e) If this determination is of no interest, proceed directly to Section 8.3. If only material extractable by carbon tetrachloride is to be determined, the whole extract may be used in Section 8.2, but the performance characteristics (Section 1.1) were obtained using a half portion of extract. If the whole extract is used, the calculation (step 8.2.6) must be modified accordingly. (f) A rotary evaporator has been found to be suitable and convenient. Other methods of removing solvent which avoid losses of extract by splashing or localised overheating may also be used.

Step	Experimental Procedure	Notes
8.2.2	Transfer the concentrated extract to a tared 25 ml beaker (W_1 mg). Rinse the evaporator flask with three 1.5 ± 0.2 ml portions of carbon tetrachloride and add the rinsings to the beaker.	
8.2.3	Place the beaker on a steam bath and cautiously evaporate off the carbon tetrachloride using the air line. This step must be performed in an efficient fume cupboard (notes g and h).	(g) Other means of heating the beaker may be used provided the heat source is below 100°C to avoid localised overheating. (h) Clean oil free nitrogen may be used in place of an air line. Air from compressors must be filtered (see section 6.1.3).
8.2.4	<i>Determination</i> Heat the beaker in an oven at $105 \pm 2^\circ\text{C}$ for 15 mins (note i). Allow to cool in a desiccator and reweigh (W_2 mg) (note j).	(i) Care should be taken to maintain the oven temperature during heating eg., do not open oven door. (j) When necessary the extract may be subject to further examination. This extract may no longer be completely soluble in CCl_4 due to oxidation of certain components during step 8.2.4.
8.2.5	<i>Blanks</i> A blank determination should be performed with each batch of samples using distilled water in place of samples, following the procedures given in Sections 8.1. and 8.2. The weight of material extracted by carbon tetrachloride (B_1 mg) should be noted.	
8.2.6	<i>Calculation</i> The material extractable by carbon tetrachloride (C_1 mg/l) in the sludge is calculated as follows:	
	$C_1 = \frac{2(W_2 - W_1 - B_1)}{V_1} \text{mg/l}$	
8.3	<i>Determination of Certain Hydrocarbon Oil and Grease Components in Sewage Sludge.</i>	
8.3.1	<i>Chromatography</i> Take an aliquot of the second portion of the extract solution (V_3 ml) obtained in step 8.1.4 such that it contains less than 200 mg of carbon tetrachloride extractable material. If necessary make up the aliquot to between 100 ml and 125 ml with carbon tetrachloride (note k).	(k) Step 8.3.1 ensures that the chromatography columns are not overloaded. With some samples this will result in weight differences which are too small to give adequate precision. In these cases and when the nature of the samples is known, larger aliquots or the whole second portion of the carbon tetrachloride extract may be used. Care must be taken to ensure that less than 200 mg fatty material and/or 200 mg aromatic material are applied to the column.
8.3.2	Wash a chromatography column (Section 6.2) with 20 ± 2 ml carbon tetrachloride, hold the meniscus just above the level of the sodium sulphate, and discard the washings. Add the extract aliquot to the column and allow to flow through at 1 to 2 drops per second until the meniscus just reaches the top of the sodium sulphate. Rinse the vessel containing the aliquot of the extract with 10 ± 1 ml of carbon tetrachloride. Add the rinsings to the column and allow to flow through as above. Repeat the rinsing twice more and combine all the eluates.	

Step	Experimental Procedure	Notes
8.3.3	<p><i>Evaporation</i></p> <p>Transfer the combined eluates to the flask of the rotary evaporator and evaporate as in Section 8.2.4 steps 1, 2 and 3. The weight of the total beaker in this instance being W_3 mg.</p>	
8.3.4	<p><i>Determination</i></p> <p>Heat the beaker in an oven at $105 \pm 2^\circ\text{C}$ for 15 mins (note i – section 8.2.4 above). Allow to cool in a desiccator and reweigh (W_4 mg) (note 1)</p>	(l) When necessary the extract may be subject to further examination.
8.3.5	<p><i>Blank</i></p> <p>A blank determination should be performed with each batch of samples using distilled water in place of samples, following the procedures given in Sections 8.1 and 8.3. The weight of the hydrocarbon oil components (B_2 mg) should be noted.</p>	
8.3.6	<p><i>Calculation</i></p> <p>The concentration of hydrocarbon oil components (C_2 mg/l) is calculated as follows:</p>	
	$C_2 = \frac{W_4 - W_3 - B_2}{V_1} \times \frac{V_2}{V_3} \text{mg/l}$	

9 Checking the Accuracy of Analytical Results

Once the methods have been put into normal routine operation many factors may subsequently adversely affect the accuracy of the analytical results. It is recommended that experimental tests to check certain sources of inaccuracy should be made regularly. Many types of tests are possible and they should be used as appropriate. As a minimum, however, it is suggested that at least one sample, of suitable concentration, in each batch of analyses be analysed in duplicate. The results obtained should then be plotted on a quality control chart which will facilitate detection of inadequate precision and allow the standard deviation of routine analytical results to be estimated.

10 References

- (1) The Use of Carbon Tetrachloride to extract oil and grease from aqueous samples. Appendix C, First Report (May 1973 – January 1977). Standing Committee of Analysts to Review Standard Methods for Quality Control of the Water Cycle. (STC Report No 7) Department of the Environment/National Water Council, London 1977.
- (2) The Sampling and Initial Preparation of Sewage and Waterworks' Sludges, Soils, Sediments and Plant Materials Prior to Analysis 1977. Methods for the Examination of Waters and Associated materials, HMSO, London, 1977.

11 Addresses for Correspondence

- 11.1 However thoroughly a method may be tested, there is always the possibility of a user discovering a hitherto unknown problem. Users with information on this method are requested to write to:

The Secretary
The DOE/NWC Standing Committee of Analysts
The Department of the Environment
2 Marsham Street
LONDON SW1P 3EB
England

- 11.2 At the present time, though based on work in several laboratories, thorough test

data is only available from one laboratory, hence the tentative status of the method. Additional test data would be welcomed. Results should be sent to:

The Secretary
Working Group 6
The DOE/NWC Standing Committee of Analysts
The Department of the Environment
2 Marsham Street
LONDON SW1P 3EB
England

Standing Committee of Analysts

Members of the Committee Responsible for this Method:

Mr BT Ashurst	³		
Dr GI Barrow	¹ (after December 1976)	Dr PJ Matthews	² (after November 1973), ^{3, 4}
Mr MJ Beckett	³ (from March–September 1978)	Mr JC McCullins	¹ (after September 1976)
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Mr WM Lewis	¹	Mr BT Whitham	¹ (after June 1975), ^{2, 4}
Mr PJ Long	¹ (after June 1975)	Mr AL Wilson	¹
		Dr R Wood	¹ (June 1975 to March 1978)

¹ Member of the Main Committee (from May 1973 unless stated)

² Member of the Oil and Pesticides Panel (October 1973 – November 1975 unless stated)

³ Member of Working Group 6 (from January 1976 unless stated)

⁴ Member of the Oils, Fats and Waxes Panel between December 1975 and June 1977 when this method was passed to Working Group 6 and the panel reconstituted

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