

Thin Layer Chromatographic Characterization of Oils, Fats, Waxes and Tars 1983

Methods for the Examination of Waters and Associated Materials

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Contents

Warning to users	2
About this series	3
About this method	4
1. Introduction	5
2. Principle	5
3. Interferences	6
4. Hazards	6
5. Reagents	6
6. Apparatus	7
7. Analytical Procedure	7
8. Characterization	9
Tables	10-11
9. References	12
Address for correspondence	13
Membership responsible for this method	14

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 properly equipped laboratories. 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 whether for one's self, one's colleagues in the laboratory, outsiders or subsequently for maintenance workers. 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 specifications. 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. Among such publications are: 'Code of Practice for Chemical Laboratories' and 'Hazards in the Chemical Laboratory' issued by the Royal Society of Chemistry, London; 'Safety in Biological Laboratories' (Editors Hartree and Booth), Biochemical Society Special Publication No 5, The Biochemical Society, London, which includes biological hazards; and 'The Prevention of Laboratory Acquired Infection', 'Public Health Laboratory Service Monograph 6', HMSO, 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 must be taken beyond that which should be exercised at all times when carrying out analytical procedures. It cannot be too strongly emphasized 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 radio-chemical 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. If calling an ambulance or sending someone to hospital after a known or suspected poisoning, mention this at the time of the call as such cases are usually sent to the nearest specially equipped hospital.

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 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. Hazardous reagents and solutions should always be stored in plain sight and below face level. Attention should also be given to potential vapour and fire risks. If in doubt, it is safer to assume that the hazard may exist and take reasonable precautions, rather than to assume that no hazard exists until proved otherwise.

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About this Series

This booklet is part of a series intended to provide both recommended methods for the determination of water quality, and in addition, short reviews of the more important analytical techniques of interest to the water and sewage industries. In the past, the Department of the Environment and its predecessors, in collaboration with various learned societies, have 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 the 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 a committee of the Department of the Environment set up in 1972. Currently it has seven 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
- 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
- 9.0 Radiochemical methods.

The actual methods and reviews 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.

Whilst an effort is made to prevent errors from occurring in the published text, a few errors have been found in booklets in this series. Correction notes and minor additions to published booklets not warranting a new booklet in this series will be issued periodically as the need arises. Should an error be found affecting the operation of a method, the true sense not being obvious, or an error in the printed text be discovered prior to sale, a separate correction note will be issued for inclusion in that booklet.

L R PITTWELL
Secretary

31 October 1983

About this Method

0.1 It is the long term objective of the Oils, Fats and Waxes Panel of the Standing Committee of Analysts to produce a comprehensive scheme of analysis for the characterization of oils, fats, waxes and tars which may be causing pollution of water.

0.2 A series of quantitative and qualitative methods is to be published which will eventually be co-ordinated into a General Scheme for the Characterization of Oil, Fat, Waxes or Tar Pollutants which will be published as a separate document (8). For methods already published in this series see refs. 9–12. See also refs. 13–19 for further related publications in this series.

0.3 When an oil, fat, wax or tar of unknown character has been isolated from a pollution sample it may be classified by thin layer chromatography (TLC). In addition, TLC

may be used to confirm the characterization of the sample obtained by gas chromatography, see ref. 10, or as an alternative to the technique for involatile samples.

0.4 Chromatographic 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 batch. It is left to the senior supervising analyst to evaluate and choose from the appropriate brands available. It should be noted that manufacturers may change their products, often without notice, and this may affect performance characteristics.

Thin Layer Chromatographic Characterization of Oils, Fats, Waxes and Tars

1 Introduction

Samples of oil, fat, wax and tar may be presented for thin layer chromatographic (TLC) analysis in the form of a solid (including coherent semi solids), liquid, or in a solution of suitable solvent, as derived from the General Analytical Scheme, see (8).

Classification may be obtained by subjecting the sample to successive TLC analysis with different systems and visualization under ultra violet light particularly at 350 nm and observation of the fluorescence. More precise characterization may be obtained by comparison of those features with chromatograms obtained from oils of known character and preferably from the same source as the pollutant. Mixtures can give ambiguous results and the technique is used to greatest effect in conjunction with others such as gas chromatography (see ref. 10). The method described is based on an Institute of Petroleum method IP 314T (1).

2 Principle

2.1 For the purpose of characterization of oil, fat, wax or tar samples by TLC, a scheme of successive classification is used whereby the materials are classified according to the TLC and fluorescence properties viz:

Class I

- (a) Petrolatums
- (b) Heavy lube oils — eg motor and gear oils

Class II

- (a) Heavy engineering oils — ie heavier oils used in industrial machinery, eg heavy non-soluble cutting oils and light lubricating oils
- (b) Light engineering oils — ie lighter oils used in industry, eg light non-soluble cutting oils and cable oils
- (c) Diesel fuels — eg gas and Derv oils
- (d) Solvent fuels — eg white spirit and kerosene

Class III

- (a) Coal tars — eg certain pitches and refined and crude tars
- (b) Coal tar oils — eg creosote, anthracene oil

Class IV

- (a) Lake asphalts
- (b) Fuel oils, heavier crude oils and tanker sludges
- (c) Bitumens
- (d) Lighter crude oil

Class V

- (a) Other fluorescing oils — eg methyl salicylate (oil of wintergreen)
- (b) Non-fluorescent or very weakly fluorescent — eg certain vegetable oils or saturated hydrocarbons (eg corn oil or liquid paraffin)

2.2 The sample is subjected to TLC in two stages. The plate from each stage is examined by ultra violet light at 350 nm and 254 nm, and the fluorescence noted. The results obtained from the first stage (Alumina (T), F₂₅₄ and acetone) determine the nature of the system used in the second stage. In addition, Silica Gel F₂₅₄ and Kieselguhr F₂₅₄ are also used in the second stage, in which solvents of differing polarities are used.

2.3 Classes I and II are characterized by the blue fluorescence at 350 nm of the polynuclear hydrocarbons in refined oil products, Class III is characterized by the varied fluorescence of a wide variety of compounds, Class IV is characterized by the presence of asphaltenes which fluoresce yellow. Class V represents other fluorescent materials and all non-fluorescent materials.

2.4 Generally, samples can be classified, and identified occasionally as individual materials, especially when reference samples of suspect substances are available. Often the separate components of complex mixtures cannot be classified accurately; however, the mixtures will have characteristics sufficient to make them distinguishable from other oils and as with other methods of oil analysis, this method is very effective when a sample of reference material can be taken from the same source as the pollutant sample.

3 Interferences

Any compound in a given sample with thin layer chromatographic and fluorescent characteristics the same as those of the oil, fat, wax or tar in the sample but which is not a component of these materials.

4 Hazards

The solvents used in the chromatography are flammable and care should be exercised to avoid sparks or naked flames during the preparation of the tanks and the procedure. The procedure should be carried out in a room with adequate ventilation. Exposure to ultra violet radiation can cause damage to eyes and if several determinations are carried out repetitively on a routine basis, it is advisable to wear suitable eye protection. Relevant safety regulations for the use of ultra violet radiation equipment should be observed. Some of the oils being examined or used as standards may be skin irritants or carcinogenic; suitable gloves should be worn and care exercised in using the oils. Some of the additional reagents in Section 8 can be absorbed through the skin and are toxic in large doses.

5 Reagents

All reagents should be chromatographic grade where available, but good laboratory grade will suffice for the solvents.

5.1 Solvents

Light petroleum 40–60°C

Acetone

Ethanol

Ethyl acetate

Chloroform

Toluene

n-Hexane

Iso-octane

5.2 Stationary Phases

Precoated glass plates may be purchased as 5 cm × 20 cm, 10 cm × 20 cm and 20 cm × 20 cm, 0.25 mm thickness plates.

5.2.1 *Aluminium oxide F₂₅₄ (Type T)*

5.2.2 *Silica gel F₂₅₄*

5.2.3 *Kieselguhr F₂₅₄*

5.3 Standards, commercially available unused products:

- Class I — Petrolatum, motor oil, gear oil
- Class IIa — Heavy engineering oil — (cutting oil, heavy non-soluble)
- Class IIb — Light engineering oil — (cable oil)
- Class IIc — Derv Oil
- Class IId — Kerosene
- Class IIIa — Crude coal tar
- Class IIIb — Creosote
- Class IVa — Trinidad lake asphalt
- Class IVb — Light fuel oil, medium fuel oil, heavy fuel oil, heavier crude oils
- Class IVc — Bitumen
- Class IVd — Lighter crude oils

These materials may be obtained by purchase from commercial sources or as appropriate, from the Laboratory of the Government Chemist (Oil, Identification Unit), the National Physical Laboratory, Teddington, Middlesex TW11 0LW and the Director and Secretary, British Tar Industry Association, 132/135 Sloane Street, London SW1X 9BB.

5.4 Additional Reagents

These vary with substance suspected, see Section 8.

6 Apparatus

6.1 Standard thin layer chromatographic tanks — to be used for the ascending technique. The dimensions will vary according to the size and number of chromatographic plates used.

6.2 Glass plates where necessary.

6.3 Capillary tubes — capable of delivering 3 mg or 3 μ l to a TLC plate.

6.4 UV lamp unit with 350 nm and 254 nm lamps.

7 Analytical Procedure

Step	Procedure	Notes
Stage 1		
7.1	Prepare and equilibrate a tank containing acetone to 1 cm depth (see refs. 3 and 19 and references therein).	(a) It is advisable to use an absorbent paper liner in the tank.
7.2	Activate an aluminium oxide F ₂₅₄ (type T) plate by placing it in an oven at 105°C for 30 minutes and then allowing it to cool in a desiccator containing a self-indicating silica gel desiccant.	
7.3	Mark a position on the edge of the layer 2 cm from one end. This gives a visual guide to produce an even start line (note b). Make a 0.5 cm score across the plate 5.0 cm from the start line.	(b) A faint pencil line may be drawn between the marks to aid 'spotting'.
7.4	A drop (approximately 3 μ l) of sample is placed at the start line (note c). Place similar drops of standards representative of each class onto the start line at approximately 1 cm intervals. Wide plates will be needed when several samples and standards are being examined. Stand the plates upright in the solvent in the tank (acetone for stage 1).	(c) The drop size of 3 μ l has been found to give a reasonable sized spot, but it is only an approximate size. If the sample or reference standard is too thick it may be diluted with chloroform until it is just thin enough to be 'spotted' or it may be warmed gently. If a chloroform solution is used, ensure that the 'spot' is thoroughly dry before proceeding. A template may be used.
7.5	Allow the solvent to ascend until it reaches the score at 5 cm height. (note d).	(d) This will take about 5 minutes.

Step	Procedure	Notes
7.6	Remove the plate from the tank and allow the solvent to evaporate preferably in a fume cupboard.	
7.7	Examine the plate under 350 nm and 254 nm UV light and note the details including the colour and shape of the spot. Measure the distance travelled by the mid-point of the spot from the start line to ± 0.05 cm. Calculate the R_f value (note d).	(e) $R_f = \frac{\text{Distance travelled by spot}}{\text{Distance travelled by solvent}}$
7.8	Classify the oil broadly according to Table I. Repeat the thin layer chromatography (steps 7.1–7.7) using different solid and mobile phases according to the initial classification as described in steps 7.9–7.12. Apply standards appropriate to the class of oil being examined.	
Stage 2 for Class I products		
7.9	Repeat steps 7.1–7.7 using a Silica gel F_{254} plate (scored to allow the solvent to travel 6.5 cms instead of 5.0 cms) and ethyl acetate.	
Stage 2 for Class II products		
7.10	Repeat steps 7.1–7.7 using Aluminium oxide F_{254} (type T) and acetone — ethanol 1:1 by volume (note f).	(f) Class IIa requires the use of specific systems, such as those described in ref 2.
Stage 2 for Class III products		
7.11	Prepare a 10% m/v or v/v solution of the sample in toluene. Place five 3 μ l spots alongside one another so as to form a band about 1.5 cm in length along the startline of an Aluminium oxide F_{254} (type T) plate. Allow the toluene to evaporate so as to leave the bands dry. Repeat steps 7.5–7.7 using light petroleum 40–60°C. In some instances more distinct results are obtained with n-hexane or iso-octane. The observations and calculations are made as described previously in step 7.7. See note g.	(g) Sample solutions of 15% to 20% in toluene, give optimum separations in some instances, but the solution strength should not exceed 20%. Table II gives examples of the characteristic bands which have been observed for certain materials. The most effective results are obtained when sample and reference materials are run simultaneously. Variations may occur for individual samples but the coloured bands are characteristic of coal tar and its products. Generally the products such as creosote have less bands than the tars (refs. 1 and 2).
Stage 2 for Class IV products		
7.12	Repeat 7.11 using Kieselguhr F_{254} acetone. The plate should remain for at least 2 minutes under the UV light at 350 nm before noting the characteristics of the fluorescence. Note the R_f value and colour of each band (note h and see step 7.7).	(h) Asphaltenes are responsible for the yellow fluorescence. The asphaltenes content increases as the oil becomes heavier and the intensity of yellow fluorescence increases; bitumens have the greatest intensity.
All Classes		
7.13	Classify the oil further using the results obtained from the secondary systems (see Table 1). Further TLC systems can be used to provide more detailed classification. Optimum systems can be developed for specific samples of oil by varying the nature of the solid and the polarity of the solvent. Examples of such systems have been reported (2) and some are described briefly in Section 8 on characterization.	

8 Characterization

8.1 Samples of oil pollutant may be characterized by comparison of fluorescent features of their thin layer chromatograms with those of typical known oils. Such characterization is achieved by the use of successive systems of solid phases and solvents in which the classification becomes increasingly precise. However, the chromatographic characteristics of oil and tar types vary widely and therefore the ability of the method to provide a precise characterization is often constrained by the number of typical oil samples available. Whilst the procedure described has been used successfully in a wide range of laboratories many of which had no previous experience of the techniques, the analyst is advised to gain experience in interpreting the chromatograms and to have access to a wide range of typical oils and tars, if fairly precise characterization is to be attempted.

8.2 The general appearance of the thin layer chromatograms under ultra violet light, including colour, and general shape and R_f values of the principal features are used to classify oils and tars. The technique is less useful for natural oils and fats or any oil with weak fluorescence but other thin layer chromatographic systems may be used (3). In the case of petroleum oils and coal tar products fairly precise classification can be obtained by the use of two stages of chromatography; further characterization within classes may be obtained by the use of specific thin layer chromatographic systems as described in refs. 2 and 3.

8.3 Oils isolated from pollution incidents can be mixtures or modified in nature due to the weathering and ageing processes (experience has shown that these latter phenomena have less influence on the accuracy of characterization than it does in gas chromatography). One of the most effective ways of using thin layer chromatograms is by comparison of the pollutant and suspected source irrespective of the exact characteristics of either, see General Analytical Scheme (8). Fuel oils may be differentiated from weathered crude oils using a system of silica gel (with fluorescent indicator) and toluene on an extract prepared by sequential extractions involving dimethylformamide and cyclohexane (6).

8.4 The complex nature of oils and tars, particularly if they are in admixture, as often happens in the case of pollutant samples, sometimes makes the characteristics of chromatograms diffuse, streaky and difficult to interpret. In such cases the sample can be subjected to treatment to resolve components prior to chromatography (2), (4). An example is the isolation of aromatic, hetero-cyclic and polar compounds (containing nitrogen, oxygen and sulphur) by glacial acetic acid/methanol extraction and subsequent chromatography of the extract (4). Characterization of the commercial brand of petroleum products may be achieved by thin layer chromatography of additives (5).

8.5 Whilst this technique is complementary to other available procedures, such as gas chromatography, it has the advantage of only requiring the simplest laboratory facilities and is usable in the field.

Table I Systematic Scheme for Differentiation of Oils with Fluorescence at 350 nm

System 1	Result	System 2	Result	Conclusion
	Class I $R_f = 0$ blue, streak to front	Silica gel (6.5 cm) and ethyl acetate	(a) $R_f = 0$ streak to front (b) $R_f = 0$ slightly diffuse streak to front	Petrolatums Motor and gear oils
	Class IIa $R_f 0 - 0.3$ strong blue streak to front	Use a TLC system specific for a given oil	Specific for the oil	Heavy engineering oils
	Class IIb, c, d $R_f 0.3$ blue, may be diffuse or very pale at high R_f values	Alumina T and 1:1 ethanol: acetone	(b) $R_f 0.4$ (c) Long pale blue spot between $R_f 0.3$ to 0.7 (d) Diffuse at solvent front, very pale blue. Dark at 254 nm	Light engineering oils Diesel fuel oils Solvent fuels
Alumina T and acetone	Class IIIa, b Orange or brown head at solvent front and brown streak	Band technique: Alumina T and light petroleum 40–60°C	(a) Multicoloured bands up to solvent front (b) Some of bands missing when compared to crude coal tar	Coal tars Coal tar oils
	Class IVa, b, c, d Multi-coloured, yellow, brown, blue streak and head	Band technique: Kieselguhr and acetone	(a) Very pale yellow brown head and band (b) Yellow-purple band ranging from pale to fairly strong sometimes pale blue head (c) Intense yellow band (d) Very pale yellow band, blue yellow head	Lake asphalts Fuel oils Heavier crude oils Tanker sludges Bitumens Lighter crude oils
	Class Va Fluorescing characteristics not listed above	Examine by specific TLC system or other techniques	Specific for the oil	Other non-petroleum fluorescing oils
	Class Vb Very weak or no fluorescence, some purple at 254 nm	eg saponification		Non-fluorescing, non-petroleum oils or liquid paraffin

Table II Examples of observations of coal-tar products

Band technique, activated Alumina T/light petroleum 40-60°C fluorescence at 350 nm

For reference only

High Temp (HT) Tar	Low Temp (LT) Tar		Anthracene Oil (from HT Tar)		Road Tar (from HT Tar)		Pitch (from HT Tar)		Creosote (from HT Tar)		Creosote (from LT Tar)	
	R	Colour	R	Colour	R	Colour	R	Colour	R	Colour	R	Colour
Purple	0.75	Purple	0.72							Purple	0.74	Purple
Light Blue	0.70	Light Blue	0.69							Light Blue	0.73	Light Blue
Yellow	0.63	Buff	0.65	Yellow	0.65	Green-Blue	0.75	Light Yellow	0.68	Blue-Grey	0.65	
Orange	0.57	Light Brown	0.59	Blue	0.52	Yellow	0.66	Yellow	0.63	Yellow	0.57	
Blue-Brown	0.49	Blue-Purple	0.50	Green-Blue	0.47	Orange	0.57	Brown	0.51	Blue-Purple	0.46	
Purple	0.39	Purple*	0.40	Grey Brown	0.21	Brown	0.50	Dark Brown	0.36	Purple	0.37	
Light Blue*	0.22	Light Blue*	0.20	Light Blue	0.15	Purple	0.37	Grey-Brown*	0.15	Grey-Brown	0.24	
Brown*	0.08	Blue-Brown	0.08	Light Blue	0.08	Grey-Brown*	0.16			Light Blue	0.17	
				Grey-Brown*	0.05					Grey-Brown*	0.05	

These examples were observed for samples examined by the British Carbonization Research Association.

*very broad.

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- (12) *Classical Methods for the Characterization of Oils, Fats and Waxes by Iodine, Hydroxyl, Saponification and Acid Values 1983*, HMSO, London, in this series.
- (13) *Determination of Very Low Concentrations of Hydrocarbons in Water 1984*. HMSO, London, to be published in this series.
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- (15) *The Determination of Material Extractable by Carbon Tetrachloride and of certain Hydrocarbon Oil and Grease Components in Sewage Sludge 1978*. HMSO, London, in this series.
- (16) *The Quantification of Natural Oils and Fats in Effluents* in preparation in this series.
- (17) *The Determination of Polynuclear Aromatic Hydrocarbons in Aqueous and Related Samples 1985*, to be published in this series.
- (18) *Gas Chromatography, an Essay Review 1981*, HMSO, London, in this series.
- (19) *High Performance Liquid Chromatography, Ion Chromatography, Thin Layer and Column Chromatography of Water Samples 1983*, HMSO, London, in this series.

**Address for
Correspondence**

However thoroughly a method may have been tested, there is always the possibility of a user discovering a hitherto unknown problem. In such event, please supply as much information as possible to:

The Secretary
The Standing Committee of Analysts
Department of the Environment
Romney House
43 Marsham Street
London SW1P 3PY
England

Standing Committee of Analysts

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