Classical Methods for the Characterization of Oils, Fats and Waxes by Saponification, Hydroxyl, Iodine and Acid Values 1983

Methods for the Examination of Waters and Associated Materials

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Contents

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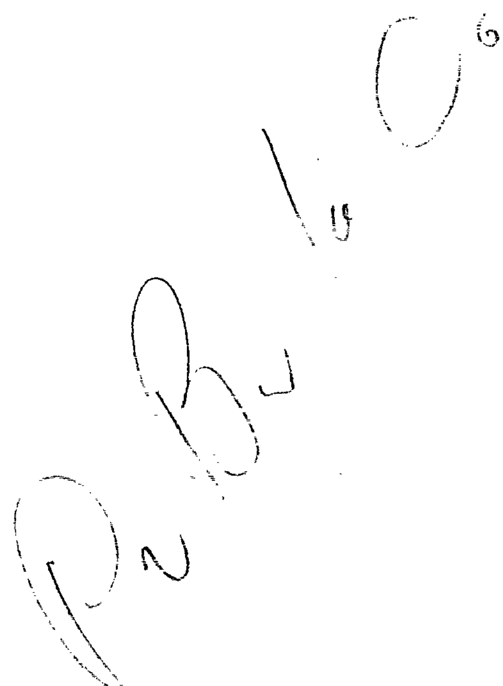

Warning to Users

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 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. 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 might 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.

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 to 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 nonmetallic 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

Classical Methods for the Characterization of Oils, Fats and Waxes by Saponification, Hydroxyl, Iodine and Acid Values

A Introduction

A.1 General Explanation

A.1.1 These methods form part of a continuing series which when complete can be used as part of a general co-ordinated scheme of analysis (see another publication in this series).

A.1.2 A sample of fat or oil may be characterized by gas chromatographic methods (see another publication in this series[7]) as being of animal or vegetable origin, but further complementary evidence might be required. It may also be classified as such a type of material by prior knowledge of the nature of the situation from which the sample was taken. The methods described in this publication provide the classical chemical information which may be used as the complementary evidence.

The methods included in this publication are:

B Saponification Value
C Hydroxyl Value
D Iodine Value
E Acid Value

In some publications the American description of Number rather than Value is used e.g. Saponification Number.

A.1.3 Table A1 (References 1 and 5) gives values for typical samples of a range of uncontaminated natural fats and oils etc. which could be found in environmental samples in practice. The values should be determined on samples under examination and then compared to the values given in Table A1. It must be recognized that because of the natural origin of the materials the exact size of the values for a particular oil or fat can vary slightly from batch to batch and the comparisons recommended above must be made carefully to account for these variations and for changes which might occur on storage or on exposure to environmental factors. As with other methods for characterization, the best comparisons are made between an environmental sample and samples from the suspected source.

Fresh reference materials should be analysed as appropriate as checks on the laboratory procedures.

A.2 Sampling and Sample Preservation

A.2.1 The same requirements apply to the four methods.

The sample should be prepared for analysis as follows:—
(a) to make it homogenous
(b) to separate substances insoluble in the fat
(c) to eliminate water

The procedures used for this purpose differ according to the physical state of the oil or fat to be analysed and if it has been isolated from the environmental sample by solvent extraction, it is likely that sample preparation as listed below will be unnecessary.

A.2.2 Liquid sample, clear and without sediment

The sample should be rendered as homogenous as possible by turning the container upside down several times.

A.2.3 Liquid sample, turbid or with sediment

Heat the sample in a beaker in an oven or water bath to a temperature of not more than 10°C above the melting point of the fat. If after heating and stirring, the sample is not

completely clear, filter the fat while maintaining its temperature at not more than 10°C above the melting point. Glass wool in a funnel is usually sufficient. The filtrate should be clear.

A.2.4 Solid samples

Melt the sample in a beaker by heating it in an oven or water bath to a temperature of not more than 10°C above the melting point of the fat. If the sample is turbid it must be filtered before analysis. Glass wool in a funnel is usually sufficient.

A.2.5 All samples must be dried before analysis using the following procedure:

Add anhydrous sodium sulphate, in the proportion of 1g to 2g per 10g fat sample maintained at 10°C above the melting point. Stir vigorously and filter through glass wool in a funnel, whilst maintaining the temperature at not more than 10°C above the melting point (to prevent solidification during filtration).

A.3 List of Related British Standards

There are a number of British Standards which may provide useful information. These are:

627 : 1965 Methods for sampling fats and fatty oils
628 : 1967 Crude Coconut Oil
629 : 1967 Crude Groundnut Oil (Arachis oil)
(630 : 1967 Edible Olive Oil — withdrawn 1977)
631 : 1967 Crude Rapeseed Oil
684 : 1958 Methods of analysis of oils and fats (being replaced)
Introduction : 1976
/1.1 1976 Density etc
/1.3 1976 Melting point (slip point)
/1.4 1976 Flow and drop points
/1.5 1976 Cloud Point
/1.6 1976 Titre
/1.7 1976 Surface-drying time
/1.8 1976 Smoke point
/1.9 1976 Water-insoluable solvents
/1.10 1976 Moisture and volatile matter
/1.11 1976 Penetration value
/1.12 1976 Dilation of fats
/1.13 1976 Cooling curve
/1.14 1976 Colour
/1.15 1978 Extinction in ultra-violet light
/1.16 1981 Water content — entrainment method (Dean and Stark)
/2.1 1976 Water by the Karl Fischer method
/2.2 1976 Ash
/2.3 1976 Impurities
/2.4 1976 Total Fatty matter
/2.5 1977 Dissolved soap (alkalinity)
/2.6 1977 Saponification value
/2.7 1977 Unsaponifiable matter
/2.8 1977 Total neutral oil
/2.9 1977 Hydroxyl value and acetyl value
/2.10 1976 Acidity, acid value and mineral acidity
/2.11 1976 Volatile acids
/2.13 1976 Iodine value
/2.14 1976 Peroxide value
/2.16 1976 Copper (colorimetric method)
/2.17 1976 Iron (colorimetric method)
/2.20 1977 Carotene in vegetable oils
/2.23 1977 Bömer value
/2.25 1979 Oxidative stability value
/2.29 1978 Cotton seed oil test
/2.30 1978 Sesame oil test
/2.31 1978 Arachis oil test
/2.34 1980 Preparation of methylesters of fatty acids
/2.35 1980 Gas Liquid chromatography of methylesters of fatty acids

B Determination of Saponification Value (SV)

B.0 Introduction

B.0.1 Saponification value is the number of mg potassium hydroxide which is required to saponify 1g of fat or oil (i.e. to neutralize the free fatty acids and to release and neutralize the fatty acids combined as glycerides). The determination is used by the food and chemical industries as part of the characterization of natural oils and fats.

B.0.2 However, it can be used for any material containing esters and acids and therefore may be used for the characterization of mineral oil and fat mixtures such as may be found in sewer blockages, or digester and primary sedimentation tank scums and sludges.

It can be seen from Table A1 that fats and oils likely to occur in sewage have saponification values of about 200. An approximate proportion of fat in sewer blockages and scums etc. may be deduced by dividing their saponification value by 200. The saponifiable and unsaponifiable fractions of the sample may be isolated by a modified saponification procedure and by a diethyl ether extraction as described in the Institute of Petroleum method IP 284/72 T (Reference 2).

B.1 Performance characteristics of the method

B.1.1	Parameter determined	Saponification value denotes the number of mg potassium hydroxide which is required to saponify lg of fat or oil i.e. to neutralize the free fatty acids and the fatty acids combined as glycerides.				
B.1.2	Type of sample	Animal and vegetable oils and fats. Modifications to procedures are given for application of the method to wool grease and its derivatives, waxes, oxidized and polymerized oils.				
B.1.3	Basis of the method	The saponification value is determined by completely saponifying the oil or fat with a known amount of potassium hydroxide, the excess of which is determined titrimetrically with 0.5M HCl.				
B.1.4	Standard deviation*	S_W	S_B	S_T	RS_T%	Mean 9 degrees of freedom
	Expected value					
	277–291**	3.5	0	3.5	1.25	279
	99–209	0.71	0	0.71	0.35	202
B.1.5	Limit of detection	Not applicable				
B.1.6	Sensitivity	0.05 ml 0.5 M KOH on a 4.000 g sample = SV of 0.35.				
B.1.7	Bias	None known.				
B.1.8	Interferences	Any acid or ester present but not associated directly with the fat or oil being analysed.				
B.1.9	Time required for analysis	1½ Hours. (operator time up to ½ hour including blank, excluding reagent proportion).				

* The results were obtained by G. Hindle, P. M. Hoyle and M. R. Wright of the Cambridge College of Art and Technology under contract to the DOE. Actual samples were used to obtain the results; the method may be evaluated in absolute terms using a pure sample of glyceryl tristearate.

** Supplied by Unilever plc.

B.2 Principle

The saponification value is related to the molecular weight of the fat and denotes the number of mg potassium hydroxide which is required to saponify lg of fat or oil i.e. to neutralize the free fatty acids and the fatty acids combined as glycerides. The saponification value is determined by completely saponifying the oil or fat with a known amount of potassium hydroxide, the excess of which is determined titrimetrically with 0.5M hydrochloric acid.

B.3 Field of application and interferences

This procedure is a general method for determination of the saponification value of oils and fats. Also included are modifications to the procedure for application of the method to wool grease and its derivatives, waxes, oxidized and polymerized oils. Any ester or acid will record a positive saponification value as defined by the method. However, for the purposes of characterizing fats and oils, if the sample of the fat or oil being analysed contains esters or acids which are not normally constituent parts, they are considered to be interferents.

B.4 Hazards

Care must be taken when handling acid solutions, particularly, concentrated hydrochloric acid, caustic solutions and flammable solvents.

B.5 Reagents

Except where otherwise stated analytical grade reagents should be used.

B.5.1 Water

Either distilled or deionized water can be used provided the blank value remains acceptable.

B.5.2 Phenolphthalein indicator

Dissolve 1g of phenolphthalein in 100 ml of 96% v/v ethanol to obtain a 1% m/v solution. In determinations giving dark coloured soap solutions, observation of the end point may be facilitated by:

(a) substitution of thymolphthalein or Alkali Blue 6B for phenolphthalein indicator solution, or
(b) addition of 1 mg of 0.1% w/v solution of methylene blue to each 100 ml of the phenolphthalein indicator solution.

B.5.3 0.5M Hydrochloric acid solution

Prepared and standardized accurately with sodium carbonate by a standard procedure (e.g. reference 3 or 4).

B.5.4 Ethanol (96% v/v)

Neutralized to a phenolphthalein end/point by dropwise addition of 0.5M hydrochloric acid solution or 0.5M aqueous potassium hydroxide solution.

B.5.5 Potassium Hydroxide ethanolic solution

0.5M solution in 96% v/v ethanol. A stable, colourless ethanolic potassium hydroxide solution may be prepared as follows:

Boil 1 litre 96% v/v ethanol under reflux with 8g potassium hydroxide and 5g aluminium grains or aluminium foil for 1 hour and then distil the ethanol. Dissolve by shaking the required amount of potassium hydroxide (most potassium carbonate may be removed immediately prior to solution by rinsing quickly with distilled water) in the distilled ethanol to give a solution containing 28 ± 1 g KOH/1.

Allow the solution to stand for up to a week during which time the remaining potassium carbonate settles. The supernatant liquid is poured off and is then ready for use. It does not become yellow on further standing or heating. The solution should be stored in a brown or yellow glass bottle fitted with a rubber or preferably polyethylene or polypropylene stopper.

There may be a need to prepare 1M or 2M potassium hydroxide ethanolic solution (see B7.1).

B.5.6 Reference materials

It may be useful as appropriate to check the procedure with fresh reference materials of the type likely to be found in the environment e.g. *cooking fat, cooking oil, linseed oil.* An absolute check for SV may be made using glyceryl tristearate using the procedure described in B7.

B.5.7 Boiling stones

B.6 Apparatus

B.6.1 250 ml conical flasks

Made of alkali-resistant glass with ground glass joint.

B.6.2. Air or water cooled reflux condensers

With ground glass joints to fit 250 ml flask, of sufficient length to retain refluxing ethanol.

B.6.3 Water or steam bath

B.6.4 Standard laboratory glassware

B.6.5 All glassware should be cleaned thoroughly before use and it is recommended that petroleum or silicone grease or PTFE sleeves or equivalent be used to lubricate joints.

B.7 Analytical procedure

Step	Procedure	Notes
B.7.1	Weigh about 4g accurately (± 0.001 g) of prepared sample (W_g) into a 250 ml conical flask. Add 50.0 ± 0.05 ml of 0.5M ethanolic hydroxide solution to the cold sample and a few boiling stones and attach a reflux condenser to the flask (Note a).	(a) The size of the determination can be scaled down for small samples. For wool grease or fat, or its derivatives, waxes, oxidized or polymerized oils, the strength of the ethanolic potassium hydroxide should be increased to 1M or 2M.
B.7.2	Heat the contents of the flask on the water or steam bath. When the ethanol boils shake the flask gently, if necessary, to ensure complete solution of the fat (Note b). Boil for at least 60 minutes after the fat has dissolved (Note c).	(b) Shaking before the ethanol boils causes loss of volatile esters. (c) For wool grease etc. (see above), the reflux time is 2 hours.
B.7.3	Rinse down the condenser with water and disconnect it. Add 1.0 ± 0.1 ml phenolphthalein indicator and slowly titrate the hot solution with 0.5M hydrochloric acid. Note the volume V_1ml (Note d).	(d) A pH electrode and meter may be used to end point pH 8.1. Care must be taken to avoid excessive contamination of the electrode and it must be cleaned between each determination.
B.7.4	Repeat steps B.7.1 to B.7.4 with a blank i.e. no sample taking care to ensure that the conditions of the procedure are replicated. Note the volume of hydrochloric acid V_2ml.	

Calculation of the result

B.7.5 Saponification Value = $56.1 \quad \frac{M(V_1 - V_2)}{Wg}$

where M is the molarity of the hydrochloric acid, W is the weight of sample in grams, V_1 and V_2 are titrant volumes and 56.1 is derived from the molecular weight of KOH (56.1).

C Determination of Hydroxyl (HV) and Acetyl Values (ACET.V)

C.0 Introduction

C.0.1 Hydroxyl value is the number of mg of potassium hydroxide equivalent to the hydroxyl content of 1 gram of fat or oil.

C.0.2 Acetyl value is the number of mg of potassium hydroxide equivalent to the acetic acid produced by the hydrolysis of 1 gram of fat or oil which has been acetylated.

C.0.3 The two values are interchangeable and are regarded as alternatives to each other. They were developed originally as aids in the characterization of natural fats and oils by the food and chemical industries.

C.1 Performance characteristics of the method

C.1.1	Characteristic determined	Hydroxyl value denotes the number of mg potassium hydroxide equivalent to the hydroxyl content in 1g fat or oil. Acetyl value denotes the number of mg potassium hydroxide equivalent to the acetic acid produced by the hydrolysis of 1g of fat which has been acetylated.				
C.1.2	Types of sample	Animal and vegetable oils and fats.				
C.1.3	Basis of the method	Acetylation of the hydroxyl groups with a known amount of acetic anhydride in pyridine. The excess acetic anhydride is measured titrimetrically (as acetic acid) using sodium hydroxide.				
C.1.4	Standard deviation*	S_W	S_B	S_T	$RS_T\%$	Mean (9 degrees of freedom)
	Expected value					
	261–263**	2.8	NS	3.6	1.37	262
	40	0.36	NS	0.44	1.09	40.3
		NS = Not Significant				
C.1.5	Limit of detection	Not applicable.				
C.1.6	Sensitivity	0.05 ml 0.325M NaOH on a 1.000g sample = HV of 0.91				
C.1.7	Bias	Not applicable.				
C.1.8	Interferences	Any material which will react with acetic anhydride but which is present but not directly part of the fat or oil being analysed.				
C.1.9	Time required for analysis	2½–3 hours (operator time up to 1½–2 hours including blanks etc. excluding reagent preparation).				

* The results were obtained by G. Hindle, P. M. Hoyle and M. R. Wright of the Cambridge College of Art and Technology under contract to the DOE. Actual samples were used to obtain the results, the method may be evaluated in absolute terms using a pure sample of ricinoleic acid.

** Supplied by Unilever plc.

C.2 Principle

The fat is acetylated with a measured quantity of acetic anhydride in pyridine, the excess acetic anhydride is decomposed by boiling water and the acetic acid formed is titrated with sodium hydroxide solution in ethanol.

A control test with the acetic anhydride and pyridine without the fat is carried out to determine the amount of acetic anhydride available for acetylation and a similar test is carried out with the fat omitting the acetic anhydride to determine the free fatty acids present.

C.3 Field of Application and Interferences

The method described is suitable for the determination of the hydroxyl value and the acetyl value of animal and vegetable oils and fats. It is essential that the sample should be dry as the presence of moisture is liable to lead to erroneous results.

This method for determining hydroxyl content does not differentiate between hydroxyl groups in the fatty acid chains and those present in mono- or diglycerides. Hence partially hydrolysed fats may show high hydroxyl values, without containing hydroxy acids. Any material reacting with acetic anhydride will record a positive hydroxyl or acetyl value as defined by the method.

However, for the purposes of characterizing fats and oils, if the sample contains materials which are not normally part of the fat or oil present they are considered to be interferents.

C.4 Hazards

Toxic vapours may be emitted during sample preparation and analysis. The complete analytical procedure should be conducted in a fume cupboard to prevent toxic pyridine and butan-l-ol vapours from entering the work areas. Always handle the chemicals with care.

C.5 Reagents

Except where otherwise stated analytical grade reagent chemicals are to be used. Reagents should be stored in glass bottles.

C.5.1 Pyridine

Dry the pyridine by allowing it to stand in contact with barium oxide for at least two days. Then distil the dry Pyridine and use the fraction which distils above 114°C.

C.5.2 Acetic Anhydride

C.5.3 Acetylating reagent

Mix carefully one volume of acetic anhydride with seven volumes of pyridine (some heat is generated). The solution must be stored in a brown, glass stoppered bottle. The acetylating agent must be kept free from moisture, carbon dioxide and other acidic vapours. If, owing to exposure to light, a slight discolouration occurs the solution may still be used.

C.5.4 0.325M Sodium hydroxide

Make up 20–30 ml aqueous sodium hydroxide solution (600 g/l) with 96% ethanol (v/v) to make one litre of a 0.3 – 0.35M solution. This solution must be filtered to remove any precipitated carbonate. The solution should be standardized on the day of use against a standard acid solution by a standard procedure (e.g. references 3 or 4) using the phenolphthalein indicator solution. If stored in a dark bottle at a temperature below 25°C the sodium hydroxide solution will remain colourless for a long time.

C.5.5 Butan-1-ol

C.5.6 Ethanol (96%, v/v) neutralized to a phenolphthalein end point.

C.5.7 Phenolphthalein Indicator

Dissolve 1g of phenolphthalein in 96% v/v ethanol to obtain a 1% m/v solution (reference 3). In determinations giving dark coloured soap solutions, observation of the end point may be facilitated by:

(a) substitution of thymolphthalein or Alkali Blue 6B for phenolphthalein indicator solution, or

(b) addition of 1 ml of 0.1% w/v solution of methylene blue to each 100 ml of the phenolphthalein indicator solution.

C.5.8 Boiling stones

C.5.9 Reference materials (see B.5.6)

An absolute check for HV may be made using ricinoleic acid.

C.6 Apparatus

C.6.1 150–200 ml round bottomed flasks with ground glass joints.

C.6. 2100 cm air condenesers with ground glass joints.

C.6.3 Oil bath or electric hotplate with temperature regulating equipment or electric isomantle.

C.6.4 20 ml burette

C.6.5 Standard laboratory glassware

C.6.6 All glassware should be thoroughly cleaned before use and it is recommended that petrolatum or silicone grease or PTFE sleeves or equivalent be used to lubricate joints.

C.7 Analytical Procedure

C.7.0 READ SECTION 4 ON HAZARDS BEFORE STARTING THIS PROCEDURE. All operations should be performed with care.

Step	Procedure	Notes
C.7.1	Weigh the appropriate amount of prepared fat or oil (see Table C1 below) accurately (± 0.001 g) into the round bottomed flask (W_g).	
C.7.2	Measure accurately (± 0.1 ml) at least 5.0 ml of acetylating reagent into the round bottomed flask from the 20 ml burette. It should be added quickly by drops without allowing time for each drop to drain completely (Note a).	(a) The acetylating agent should be in at least 80% excess of the volume required; it may be weighed instead of added by volume (i.e. 5.0 ± 0.1g).
C.7.3	Add one or two boiling stones and attach the air condenser to the flask (Note b). Mix the fat and reagent by gentle shaking, and boil gently for at least one hour on the oil bath or hotplate (Note c).	(b) PTFE sleeves are recommended. The condenser may be fitted with a drying tube. (c) The solvent vapour should reflux no higher than the bottom end of the condenser.
C.7.4	Cool the flask to about 50°C; add 5.0 ± 0.1 ml of water from the top of the condenser which is rotated to assist washing.	
C.7.5	Mix the solution with gentle shaking and then boil for 5 to 10 minutes in the oil bath or hotplate, shaking the flask two or three times during the period.	

Table C1

Type of oil or fat	Mass of test portion (g)
Fatty alcohol	0.5 –0.7
Castor Oil	1.0
Others	2.0 –3.0

Step	Procedure	Notes
C.7.6	Cool the flask and contents to room temperature and add 30 ± 1 ml of butan-l-ol.	
C.7.7	Detach to the condenser and wash the neck and mouth of the flask and the tip of the condenser with 20 ± 1 ml of butan-l-ol. If the contents are not homogeneous, add butan-l-ol until they become so.	
C.7.8	Add a few drops of phenophthalein indicator and titrate the solution with 0.325M sodium hydroxide solution. Note the volume used Vml (Note d).	(d) A pH electrode and meter may be used to end point pH 8.1. Care must be taken to avoid excessive contamination of the electrode and it must be cleaned between each determination.
C.7.9	Repeat steps C.7.2 – C.7.8 (i.e. omitting fat). Note the volume of sodium hydroxide solution used V_2 ml (Note d).	
C.7.10	Repeat step C.7.1	
C.7.11	Measure accurately (± 0.1 ml) at least 5.0 ml of pyridine into the round bottomed flask.	
C.7.12	Proceed as in steps C.7.3 to C.7.8. Note the volume of sodium hydroxide used V_3ml (Note e).	(e) This determines free fatty acids present; a slight increase in acetyl value has been found to occur with increasing free fatty acid content of fats.

Calculation of Result

C.7.13 Hydroxyl value (or acetyl value) =

$$56.10 \quad \frac{M\,(V_2 - V_1 - V_3)}{Wg}$$

Where M is the molarity of the sodium hydroxide solution. W is the weight of sample in grams V_1, V_2 and V_3 are titrant volumes and 56.10 is derived from the molecular weight of KOH (56.10).
The results should be expressed to the nearest 0.5.

D Determination of Iodine Value (IV)

D.0 Introduction

D.0.1 Iodine value is the amount of halogen expressed as grams iodine which reacts with unsaturated groups present in 100g fat under the conditions of the tests. The procedure is known as Wijs' Method.

D.0.2 The value was developed originally by the food and chemical industries as an aid in the characterization of natural fats and oils.

D.1 Performance characteristics

D.1.1	Characteristic determined	Iodine value denotes the amount of halogen, expressed as gm. iodine, which reacts with unsaturated groups in 100g of fat under the conditions of the test.
D.1.2	Types of sample	The method describes the procedure for oils, fats and fatty acids and includes a modification for determining the iodine value of oils having conjugated double bonds such as tung oil and dehydrated castor oil.
D.1.3	Basis of the method	The material under test is treated with an iodine monochloride solution. After addition of halogen, the excess of iodine monochloride is determined titrimetrically using thiosulphate solution.
D.1.4	Range of application	Up to 200 Iodine Value Units.

D.1.5 Standard deviation*

	S_W	S_B	S_T	$RS_T\%$	Mean 9 degrees of freedom
Expected value 95–110	0.71	NS	1.13	1.17	96.5
14– 18	0.41	0	0.41	2.68	15.4

NS = Not significant

D.1.6	Limit of detection	Not applicable.
D.1.7	Sensitivity	0.05 ml 0.1M sodium thiosulphate on a 1.000 g sample = IV of 0.06.
D.1.8	Bias	None known.
D.1.9	Interference	Materials with an affinity for halogens, moisture, and oxidizable materials which are present but which are not part of the fat or oil being analysed.
D.1.10	Time required for analysis	1–2 hours. (up to 1 hour operator time, including blanks but excluding reagent preparation).

* The results were obtained by G. Hindle, D. M. Hoyle and M. R. Wright of the Cambridge College of Art and Technology under contract to the DOE. Actual samples were used to obtain the results, the method may be evaluated in absolute terms using a pure sample of oleic acid.

** Supplied by Unilever plc.

D.2 Principle

The fat is dissolved in carbon tetrachloride (see D4) and allowed to react for a specified time with a solution of iodine monochloride. The quantity of the halogen which has reacted with the double bonds in the compounds present in the fat is then determined indirectly by adding potassium iodide solution and titrating the liberated iodine with sodium thiosulphate solution.

D.3 Field of Application and Interferences

The method is applicable to oils, fats and fatty acids. The procedure has to be modified slightly when determining the iodine value of oils having conjugated double bonds such as tung oil and dehydrated castor oil. Iodine values of fatty acids are to some extent empirical and therefore strict adherence to the reaction time is essential if reproducible results are to be obtained. In general, impurities with an affinity for halogens, moisture and oxidizable materials can interfere with the analysis.

D.4 Hazards

Carbon tetrachloride is a suspected carcinogen and wherever possible an alternative solvent should be used. In the case of iodine value, determination, trichlorethylene has been used as the substitute.

Care should also be exercised when handling the iodine trichloride and glacial acetic acid required in making up the Wijs' solution and when using Wijs' solution itself.

D.5 Reagents

Except where otherwise stated analytical grade reagents should be used.

D.5.1 Glacial Acetic Acid (17 M)

the acetic acid should be free from oxidizable matter (see D.5.2).

D.5.2 Carbon Tetrachloride

The absence of oxidizable matter should be checked for glacial acetic acid and carbon tetrachloride. This should be carried out by shaking 10 ± 0.1 ml of the reagent with 1 ± 0.1 ml of saturated aqueous potassium dichromate solution and 2 ± 0.1 ml of concentrated sulphuric acid (d_{20} 1.84). No green colouration should appear at room temperature.

D.5.3 Potassium Iodide

10% m/v aqueous solution free from iodine and iodate.

D.5.4 Wijs' Solution

If stored in a well-stoppered bottle away from light, the solution remains usable for several months.

It is available commercially or can be prepared as follows:

Weigh 9.0 ± 0.1g iodine trichloride into a brown glass bottle. Dissolve in a mixture of 700 $\pm$ 10 ml glacial acetic acid and 300 $\pm$ 10 ml carbon tetrachloride or alternative (see D4). Determine the halogen content by the following method. To 5.0 ± 0.05 ml of the solution add 5 ± 0.05 ml potassium iodide solution and 30.0 ± 0.5 ml water. Titrate with 0.1M sodium thiosulphate solution in the presence of a few drops of starch indicator solution. Add 10g powered iodine to the bulk of the reagent and dissolve by shaking. Redetermine the halogen content as described above. The titration should now equal one and a half that of the first determination. If this is not the case, add a small quantity of iodine until the halogen content slightly exceeds the limit of one and a half times. It is essential that no trace of iodine trichloride should remain as it would cause secondary reactions. Let the solution stand, then decant the clear liquid into a yellow or brown bottle.

D.5.5 0.1M Sodium Thiosulphate

Prepared and standardized with potassium iodate by a standard procedure (e.g. reference 3 or reference 8, adjusting the weights of reagents proportionately).

D.5.6 Starch Indicator

Stir 10g soluble starch and 10mg mercuric iodide in 30 ml water and pour the mixture into 1 litre boiling water. Keep the solution just at the boil for three minutes and cool. A starch solution so prepared remains fit for use for a considerable period.

D.5.7 Reference materials (see B.5.6). An absolute check for IV may be made using oleic acid.

D.6 Apparatus

D.6.1 300–500 ml conical flasks with ground glass stoppers; preferably standard iodine flasks with special wide lips at the neck to retain potassium iodide solution.

D.6.2 Standard laboratory glassware

D.6.3 All glassware should be cleaned thoroughly before use and it is recommended that grease of any description is not used to lubricate joints; PTFE sleeves may be used.

D.7 Analytical Procedure

Step	Procedure	Notes
D.7.1	Weigh the oil or fat accurately (± 0.001 g) (W_g). The mass recommended varies with the character of the fat, (see Table D1 below. (Note a).	(a) Reactable halogen must be at least 150% in excess of that consumed.
D.7.2	Transfer the glass weighing vessel to a conical flask. Add 15 ± 0.5 ml carbon tetrachloride and 25.0 ± 0.05 ml of Wijs' solution (Note b). Insert the stopper, shake gently and place the flask in the dark. If an iodine flask is used place 20.0 ± 0.5 ml 10% m/v potassium iodide solution in the special lip.	(b) Do not transfer by mouth. Wijs' reagent has a large coefficient of expansion. Make sure that the reagent is measured out at the same temperature throughout the procedure.
D.7.3	For fats with an IV below 150 allow the flask to stand for 60 minutes, those above 150 or polymerized or industrially oxidized fats, 120 minutes and tung oil or dehydrated castor oil 30 minutes (Note c).	(c) Unless it is known what the general nature of the sample is before the start of the determination, it may be necessary to do a second determination with appropriate conditions after the initial determination gives a general indication of IV.
D.7.4	Add 20.0 ± 0.5 ml of 10% m/v potassium iodide solution (Note d) and 150 ml water. Shake the contents gently.	(d) If the solution is already contained in the lip, release the stopper gently and allow it to run into the flask. Wash in with distilled water.
D.7.5	Titrate the liberated iodine with 0.1M sodium thiosulphate solution using the starch indicator. Note the volume used V_1 ml.	
D.7.6	Repeat steps D.7.2 – D.7.5 as a blank and note the volume of 0.1M sodium thiosulphate used V_2 ml.	

TABLE D1

Expected iodine value	Recommended mass (g)
5	3.0
5 – 20	1.0
21 – 50	0.4
51 –100	0.2
101 – 150	0.13
151 –200	0.10
Tung oil Dehydrated castor oil	0.115 – 0.125

$$IV = 12.69 \frac{M (V_2 - V_1)}{Wg}$$

Where M is the molarity of the sodium thiosulphate, W_g is the weight of sample, in grams V_1 and V_2 are titrant volumes and 12.69 is derived from the atomic weight of iodine (126.9).

E Determination of Acid Value (AV)

E.0 Introduction

E.0.1 Acid value is the number of mg of potassium hydroxide required to neutralize the free fatty acids present in 1g of fat or oil.

E.0.2 Natural oils and fats contain some free fatty acids but ageing and the effect of environmental factors causes the breakdown of the glyceride esters to release more fatty acids i.e. rancidity. The saponification value of the fat remains unchanged but the rancidity (i.e. acid value) can be used to make deductions about the history of the sample.

E.0.3 The determination was originally developed by the food and chemical industries as an aid in the identification of natural fats and oils but modifications have been made to determine the quantities of acids produced in oils during use under oxidizing conditions. The method published by the Institute of Petroleum (IP 177) differentiates between total and strong acids and also contains procedures for the determination of basic constituents (Reference 2).

E.1 Performance Characteristics

E.1.1	Characteristic determined	Acid value denotes the number of mg potassium hydroxide required to neutralize 1g of fat or oil.
E.1.2	Types of sample	Oils and fats (see E.1.4).
E.1.3	Basis of the method	The acid value is determined by titrating a solution of test sample to neutralization with 0.1N potassium hydroxide solution.
E.1.4	Range of application	Three methods are discussed: Method 1: is applicable to all fats other than oxidizable or polymerized fats, linseed oil, lanolin and wool fat. Method 2: is applicable to oxidizable or polymerized fats and to linseed oil, lanolin and wool fat. Method 3: is applicable to most animal and vegetable fats other than oxidizable or polymerized fats, linseed oil, lanolin and wool fat.
E.1.5	Standard deviation*	

Expected value 255–265**	Method	1	2	3
	S_w	1.79	0.86	1.25
	S_b	0	NS	NS
	S_t	1.79	1.14	1.65
	RS_t%	0.70	0.45	0.65
	Mean	258	256	253
	9 degrees of freedom			

Expected value 200–208**	Method	1	2	3
	S_w	1.97	0.79	2.11
	S_b	NS	NS	0
	S_t	2.22	0.98	2.11
	RS_t%	1.08	0.48	1.03
	Mean	205	205	204
	9 degrees of freedom			

E.1.6	Limit of detection	Not applicable.
E.1.7	Sensitivity	0.05 ml 0.1M KOH on a 1.000g sample = AV of 0.28.

E.1.8	Bias	None known.
E.1.9	Interferences	Any acid present but which is not directly part of the fat or oil being analysed.
E.1.10	Time required for analysis	15 minutes.

* The results were obtained by G. Hindle, P. M. Hoyle and M. R. Wright of the Cambridge College of Art and Technology under contract to the DOE. Actual samples were used to obtain the results, the method may be evaluated in absolute terms using a pure sample of lauric acid.
** Supplied by Unilever plc.

E.2 Principle

E.2.1 Method 1

A known mass of sample is dissolved in ethanol and titrated with aqueous potassium hydroxide.

E.2.2 Method 2

A known mass of sample is dissolved in an ethanol/toluene mixture and titrated with an ethanolic solution of potassium hydroxide.

E.2.3 Method 3

A known mass of sample is dissolved in an ethanol/diethyl ether mixture and titrated with an ethanolic solution of potassium hydroxide.

E.3 Field of Application and Interferences

E.3.1 Method 1 and Method 3

Applicable to all fats except oxidizable or polymerized fats, linseed oil, lanolin, or wool fat. The choice between the two methods depends on how easily the sample is dispersed in the conditions of Method 1. If dispersion is not achieved, Method 3 should be used.

E.3.2 Method 2

Applicable to oxidizable or polymerized fats linseed oil, lanolin and wool fat.

Note: In all methods, acids which are not normally part of the oil or fat present will give an abnormally high acid value. The onset of rancidity in an oil or fat increases the acid value and extra care has to be employed in interpretation of results. Used mineral lubricating oils and engineering oils can contain acid materials as the result of oxidation and degradation processes in the oil matrix and in additives which are present.

E.4 Hazards

Care must be taken when handling caustic solutions and inflammable solvents.

E.5 Reagents

Except where otherwise stated, analytical grade chemicals should be used.

E.5.1 Solvents

E.5.1.1 96% v/v ethanol

E.5.1.2 Ethanol-toluene1:1mixture: mix equal volumes of 96% v/v ethanol and toluene and neutralize with potassium hydroxide solution (E.5.2.2) using phenolphthalein indicator.

E.5.1.3 Ethanol-Diethyl ether 1:1 mixture: proceed as E.5.1.2.

E.5.2 Potassium hydroxide solutions

E.5.2.1 0.5M and 0.1M aqueous solutions prepared and standardized accurately with potassium hydrogen phthalate by a standard procedure (eg reference 3).

E.5.2.2 0.5M and 0.1M solution in 96% v/v ethanol prepared as follows:

Boil 1.0 litre of 96% (v/v) ethanol under reflux with 8.0g potassium hydroxide and 5g aluminium grains or foil for 1 hour and then distil the ethanol immediately. Dissolve by shaking the required amount of potassium hydroxide (most potassium carbonate may be removed, immediately prior to solution) in the ethanol distillate to give a solution of 28 ± 1g KOH/1 (for 0.5M) and 5.6 ± 0.1g KOH/1 (for 0.1M) by rinsing quickly with distilled water. Allow the solution to stand for several days. Decant the supernatant liquid from the settled potassium carbonate.

It is essential that the solution is standardized with standard hydrochloric acid immediately before use by a standard procedure (eg Reference 3 and 4). Use a solution prepared at least 5 days previously and decanted into a brown glass bottle with a rubber or preferably polyethylene or polypropylene stopper. The solution should be either colourless or pale straw yellow. Methanol may be used instead of ethanol.

E.5.3 Phenolphalein Indicator

1% m/v solution in 96% ethanol. In determinations giving dark coloured solution, observation of the end point may be facilitated by either substituting the indicator by thymolphthalein or alkali blue 6B for phenolphthalein indicator solution, or the addition of 1 ml of 0.1% w/v solution of methylene blue to each 100 ± 10 ml of phenolphthalein indicator solution before the titration.

E.5.4 Reference materials (see B.5.6)

An absolute check for AV may be made using lauric acid.

E.6 Apparatus

E.6.1 200–250 ml conical flasks.

E.6.2 50 ml burette graduated in 0.1 ml. For fats with a low acid value the use of a microburette is preferable.

E.6.3 Standard laboratory glassware.

E.6.4 Water or steam bath.

E.6.5 All glassware should be cleaned thoroughly before use and it is recommended that petrolatum or silicone grease or PTFE sleeves or equivalent may be used to lubricate joints.

E.7 Analytical Procedure

E.7.0 It is recommended that all determinations are duplicated.

Step	Procedure	Notes
	Method 1	
E.7.1	Weigh, accurately (± 0.005g) the appropriate amount (Wg) of sample (see Table E1) (Note a) and transfer quantitatively to a 200–250 ml conical flask.	(a) The mass of sample taken for analysis and the molarity of the titrant should be such that the titre does not exceed 10 ml.
	Table E1	
E.7.2	In another flask heat 50.0 ml of 95–96% v/v ethanol to boiling on a water or steam water. Whilst the temperature is still over 70°C neutralize with 0.1M potassium hydroxide using 0.5 ± 0.1 ml of phenolphthalein solution (Note b).	(b) More than 50 ml of solvent may be required for dark coloured samples.
E.7.3	Add the neutralised ethanol to the sample, bring to the boil and titrate immediately with 0.5M or 0.1M aqueous potassium hydroxide (Note c). Note the volume used V_1ml.	(c) The end point is reached when the addition of a single drop produces a slight but definite change persisting for at least 15 seconds. A pH electrode and meter may be used to end point pH 8.1. Care must be taken to avoid excessive contamination of the electrode and it must be cleaned between each determination.
	Calculation of Result	
E.7.4	Acid value $= \frac{56.1\ V_1\ M}{W}$	
	Where M is the molarity of the potassium hydroxide, V is the titrant volume in mls. W the weight of sample in grammes and 56.1 is derived from the molecular weight of KOH.	
	Method 2	
E.7.5	Weigh, accurately (± 0.005g), a suitable mass W_g of sample according to expected acidity. W_g will usually be 2–10g. Transfer quantitatively to a 200–250 ml conical flask.	
E.7.6	Add 50.0 ml of ethanol-toluene mixture and shake until solution is complete. (Note a).	
E.7.7	Titrate with 0.1 or 0.5M ethanolic potassium solution using 0.5 ± 0.1 ml phenolphthalein indicator. Note the volume used V_2ml (Notes b, c, d).	(d) If the solution becomes turbid during titration add more ethanol/toluene mixture until clarity is achieved.

Table E1		
Sample	Recommended mass (g)	Titrant molarity
Unrefined oil	20 – 50	0.1
Refined Oil	10	0.1
Acid oils/ fatty acids	Up to 4	0.5

Step	Procedure	Notes

Calculation of Result

E.7.8 Acid value $= \frac{56.1\ M\ V_2}{Wg}$

Where M is the molarity of the potassium hydroxide, W is the weight of sample in grammes, V is the volume of titrant and 56.1 is derived from the molecular weight of KOH.

Method 3

E.7.9 Weigh accurately (± 0.005g) a suitable mass Wg of sample according to expected acidity. Wg will usually be 5–10g. Transfer quantitatively to a 200–250 ml conical flask.

E.7.10 Add 150 ml of ethanol-diethyl ether mixture and 0.5 ± 0.1 ml of phenolphthalein indicator. (Note e)

(e) More than 150 ml may be needed for some samples in order to achieve a clear solution.

E.7.11 Titrate with shaking with 0.1M ethanolic potassium hydroxide (until the pink colour persists for at least 10 seconds. If the quantity of titrant exceeds 20.0 ml, the determination should be repeated with 0.5M ethanolic potassium hydroxide (Notes c and f). Note the volume of titrant used V_3ml.

(f) If the solution becomes turbid during titration, add more ethanol/diethyl ether mixture until clarity is achieved.

Calculation of Result

E.7.12 Acid Value $= \frac{56.1 \times M \times V_3}{Wg}$

Where M is the molarity of the potassium hydroxide, W is the weight of sample in grammes, V_3 is the volume of titrant and 56.1 is derived from the molecular weight of KOH (56.1).

Sources of error

Provided that the conditions of the methods are followed, then the sources of error are not of significance. By definition, the Value obtained in each case is that of the material submitted for analysis. However, if the natural oil or fat present is in a mixture then the other materials may contribute to the sample Values, thus obscuring the characterization of the natural oil or fat. Values of uncontaminated samples also vary slightly from batch to batch due to their natural origin. The points are discussed more fully in A2 and in each procedure.

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 (reference 6). However, it is most likely that these Values will only be measured occasionally and it is recommended that a reference oil/fat should be analysed at the same time as a check. If any doubts arise then a pure compound, as recommended in each procedure, should be analysed. If large numbers of samples are analysed regularly, it is suggested that, as a minimum, each batch should contain a reference material and from time to time a pure compound. The results obtained should then be plotted on a quality control chart, which will facilitate detection of inadequate accuracy and will also, allow the standard deviation of routine analytical results to be estimated.

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Some Typical Values of Oils and Fats Which may be Encountered

Oil/Fat	Saponification value	Hydroxyl value	Iodine value	Acid value
Tallow	195 – 200	–	45 – 57	2.6 – 2.8
Coconut	250 – 264	–	8 – 10	3.5 – 5.6
Sunflower	186 –189	–	130 – 144	1.2 – 5.2
Palm	190 –202	–	53 – 61	Wide range
Palm Kernel	245 – 255	–	12 – 20	Wide range
Soya	189 – 195	–	128 – 138	0.5 – 1.7
Castor	179 – 196	144– 150	81 – 88	192
Cotton Seed	194	–	107	0.1 – 2.9
Beef	196 – 200	–	40 – 50	–
Pork	196 – 202	–	52 – 70	1.9

Note: These vary with the source of the product

Address for Correspondence

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

The Secretary
The Standing Committee of Analysts
Department of the Environment
Romney House
43 Marsham Street
LONDON
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