BRITISH STANDARD

BS 5598-6: 1979
ISO 3363:1976

Methods of sampling and test for halogenated hydrocarbons —

Part 6: Determination of acidity of fluorochlorinated hydrocarbons

[ISO title: Fluorochlorinated hydrocarbons for industrial use — Determination of acidity — Titrimetric method]

UDC 661.723:547.411:543.062:543.241.5

Cooperating organizations

The Chemicals Standards Committee, under whose direction this British Standard was prepared, consists of representatives from the following Government departments and scientific and industrial organizations:

Association of Fatty Acid Distillers
British Tar Industry Association
Chemical Industries Association*
Chemical Society, Analytical Division
Department of Health and Social Security
Department of Industry: Laboratory of the Government Chemist
Fertiliser Manufacturers' Association Ltd.
Hydrocarbon Solvents Association
Ministry of Agriculture, Fisheries and Food
Ministry of Defence*
National Sulphuric Acid Association
Paintmakers' Association of Great Britain Ltd.
Royal Institute of Public Health and Hygiene
Soap and Detergent Industry Association
Standardization of Tar Products Tests Committee

The organizations marked with an asterisk in the above list, together with the following, were directly represented on the committee entrusted with the preparation of this British Standard:

British Plastics Federation
Fabric Car Research Association
Fire Extinguishing Trades Association
Institute of Metal Finishing
Institute of Refrigeration
Oil and Colour Chemists Association
Pharmaceutical Society of Great Britain
Royal Institute of Chemistry
Society of Chemical Industry
Individual companies

This British Standard, having been prepared under the direction of the Chemicals Standards Committee, was published under the authority of the Executive Board and came into effect on 31 January 1979

The following BSI references relate to the work on this standard:
Committee reference CIC/4
Draft for comment 74/53096

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Amendments issued since publication

Amd. No.	Date of issue	Comments

Contents

National foreword

This British Standard has been prepared under the direction of the Chemicals Standards Committee in order to provide methods of sampling and test for halogenated hydrocarbons.

For some years the United Kingdom has participated in the work of preparing methods of sampling and test applicable to halogenated hydrocarbons for industrial use, organized by Sub-committee 12, Halogenated hydrocarbons and amines, of Technical Committee 47, Chemistry, of the International Organization for Standardization (ISO). As international agreement is reached on the methods, it is proposed to publish them as Parts of this British Standard as follows:

— *Part 1: Sampling liquid products, identical with ISO 2209;*

— *Part 2: Sampling of liquefied gases, identical with ISO 3427;*

— *Part 3: Determination of residue on evaporation, identical with ISO 2210;*

— *Part 4: Determination of acidity — Titrimetric method, identical with ISO 1393;*

— *Part 5: Determination of cloud point, identical with ISO 1394;*

— *Part 6: Determination of acidity of fluorochlorinated hydrocarbons, identical with ISO 3363;*

— *Part 7: Methods of test for methyl chloride and ethyl chloride, identical with ISO 5781*[1)]*;*

— *Part 8: Determination of non-volatile residue in fluorinated hydrocarbons, identical with ISO 5789*[1)].

This Part is identical with ISO 3363 "*Fluorochlorinated hydrocarbons for industrial use — Determination of acidity — Titrimetric method*".

Terminology and conventions. The text of the International Standard has been approved as suitable for publication, without deviation, as a British Standard. Some terminology and certain conventions are not identical with those used in British Standards; attention is especially drawn to the following.

The comma has been used throughout as a decimal marker. In British Standards it is current practice to use a full point on the baseline as the decimal marker.

Where the words "International Standard" appear, referring to this standard, they should be interpreted as "British Standard".

Cross references

International Standard	Corresponding British Standard
ISO/R 1393[a]	BS 5598 *Methods of sampling and test for halogenated hydrocarbons* Part 4:1979 *Determination of acidity* (Identical)
ISO 2209:1973	BS 5598 *Methods of sampling and test for halogenated hydrocarbons* Part 1:1978 *Sampling of liquid products* (Identical)
ISO 3427:1976	BS 5598 *Methods of sampling and test for halogenated hydrocarbons* Part 2:1979 *Sampling of liquefied gases* (Identical)

[a] ISO/R 1393 has been superseded by ISO 1393:1977.

[1)] In course of preparation.

Additional information

WARNING NOTE. Care should be taken to avoid breathing the vapour of halogenated hydrocarbons. All operations likely to produce an appreciable concentration in the atmosphere should be conducted under a hood with an adequate exhaust.

This standard specifies a method of test only and should not be referred to as a specification defining limits of purity. Reference to the standard should be in the form of words indicating that the method of test used conforms to the requirements of BS 5598-6.

A British Standard does not purport to include all the necessary provisions of a contract. Users of British Standards are responsible for their correct application.

Compliance with a British Standard does not of itself confer immunity from legal obligations.

Summary of pages

This document comprises a front cover, an inside front cover, pages i to iv, pages 1 to 4, an inside back cover and a back cover.

This standard has been updated (see copyright date) and may have had amendments incorporated. This will be indicated in the amendment table on the inside front cover.

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1 Scope and field of application

This International Standard specifies a titrimetric method for the determination of the acidity of fluorochlorinated hydrocarbons for industrial use.

The method is applicable to products of which the acidity is in the range 2 to 200 μmol of univalent acid per kilogram.

Two procedures are given; one applicable to products that are liquid at ordinary temperatures (R 11, R 113, etc.), and the other to products that are gaseous at ordinary temperatures (R 12, R 22, R 114 etc.).

2 References

ISO/R 1393, *Liquid halogenated hydrocarbons for industrial use — Determination of acidity.*

ISO 2209, *Liquid halogenated hydrocarbons for industrial use — Sampling.*

ISO 3427, *Gaseous halogenated hydrocarbons (liquefied gases) — Taking of sample.*

3 Principle

Extraction of the acidity in the fluorochlorinated hydrocarbon by water.

Titration of the acidity of the aqueous phase with a standard volumetric sodium hydroxide solution in the presence of bromocresol green as indicator.

4 Sampling

The laboratory sample of liquid products shall be collected by the method specified in ISO 2209.

The laboratory sample of products which are gaseous at ordinary temperatures shall be collected as a liquefied gas in a stainless steel sample cylinder by the method specified in ISO 3427.

5 Reagents

During the analysis use only reagents of recognized analytical grade.

5.1 *Distilled water,* neutral to bromocresol green.

Add to distilled water, or water of equivalent purity, contained in a conical flask with a ground glass stopper, 1 % (*V*/*V*) of the bromocresol green solution (**5.3**) and neutralize with the sodium hydroxide solution (**5.2**) until the colour changes to clear blue.

5.2 *Sodium hydroxide,* 0,01 N standard volumetric solution.

Standardize this solution with 0,01 N standard volumetric hydrochloric acid solution under the conditions of the determination.

5.3 *Bromocresol green,* 1 g/l solution in 95 % (*V*/*V*) ethanol.

6 Apparatus

Ordinary laboratory apparatus and

6.1 *For products gaseous at ordinary temperatures*

See the Figure for the arrangement of apparatus.

6.1.1 *Needle valve,* of any convenient type, with screw-thread union for connection to the sample cylinder.

6.1.2 *Bubble indicator,* of glass, capacity about 15 ml.

NOTE The bubble indicator is used empty to check complete vaporization of the test portion.

6.1.3 *Three tail-form Drechsel-type gas washing bottles,* of capacity 100 ml, fitted with sintered glass disks.

6.2 *For both liquid and gaseous products*

6.2.1 *Microburette,* of capacity 2 ml, graduated in 0,01 ml.

7 Procedure

7.1 Liquid products

Use the method specified in ISO/R 1393 with the following modifications appropriate for the products concerned:

7.1.1 *Test portion*

Weigh, to the nearest 0,1 g, about 100 g of the laboratory sample.

7.1.2 *Determination*

In the case of R 11 (boiling point 24 °C), carry out the extraction with the water (**5.1**) previously cooled to 15 °C.

For all products, transfer the *whole* of the aqueous phase to a 250 ml conical flask and titrate.

7.2 Gaseous products

Weigh, to the nearest 1 g, the capped sample cylinder containing the laboratory sample, remove the cap and connect the needle valve (**6.1.1**) to the valve of inverted cylinder type (b) (ISO 3427), or to valve A of cylinder type (a) (ISO 3427) (see the Figure). Add 50 ml of the water (**5.1**) to each of the three gas washing bottles (**6.1.3**). Connect the outlet of the needle valve to the bubble indicator (**6.1.2**), connected in turn to the gas washing bottles connected in series as shown in the Figure.

Open the sample cylinder valve fully and open the needle valve to the extent required to give a steady vaporization rate of about 100 g of sample per hour.

NOTE 1 The required setting is judged by experience and observation of the bubble rate through the gas washing bottles.

NOTE 2 It may be necessary to warm the bubble indicator and cylinder valves by means of a warm air blower when analysing higher boiling products such as R 114.

After about 1 h, close first the cylinder valve and then the needle valve. Disconnect the needle valve and replace the cylinder cap. Allow the cylinder to warm up to room temperature, dry it if necessary, and reweigh it, to the nearest 1 g, under the same conditions as previously.

Mix the contents of the first two gas washing bottles; if the sample is acid, the solution will be coloured yellow. Titrate the acidity (if any) with the standard volumetric sodium hydroxide solution (**5.2**), using the microburette (**6.2.1**), until the appearance of a clear blue colour. The water in the third gas washing bottle should remain blue. If this is not the case, repeat the test.

8 Expression of results

8.1 The acidity, expressed in micromoles of univalent acid per kilogram, is given by the formula:

$$V \times 0{,}01 \times 1\,000 \times \frac{1\,000}{m} = \frac{10\,000 \times V}{m}$$

where

V is the volume, in millilitres, of the standard volumetric sodium hydroxide solution (**5.2**) used for the titration;

m is the mass, in grams, of the test portion (equal to the change in mass of the sample cylinder in the case of gaseous products).

8.2 The acidity, expressed in milligrams of hydrogen chloride (HCl) per kilogram, is given by the formula:

$$V \times \frac{365}{m}$$

where V and m have the same meaning as in **8.1**.

8.3 The acid number, expressed in milligrams of potassium hydroxide (KOH) per gram, is given by the formula:

$$\frac{56{,}1 \times V \times 0{,}01}{m} = \frac{0{,}561 \times V}{m}$$

where V and m have the same meaning as in **8.1**.

8.4 Express the result with a precision corresponding to the accuracy of weighing and titration.

NOTE If the concentration of the standard volumetric solution used is not exactly as specified in the list of reagents, an appropriate correction should be made.

9 Test report

The test report shall include the following particulars:

a) the reference of the method used;

b) the results and the method of expression used;

c) any unusual features noted during the determination;

d) any operation not included in this International Standard or in the International Standards to which reference is made, or regarded as optional.

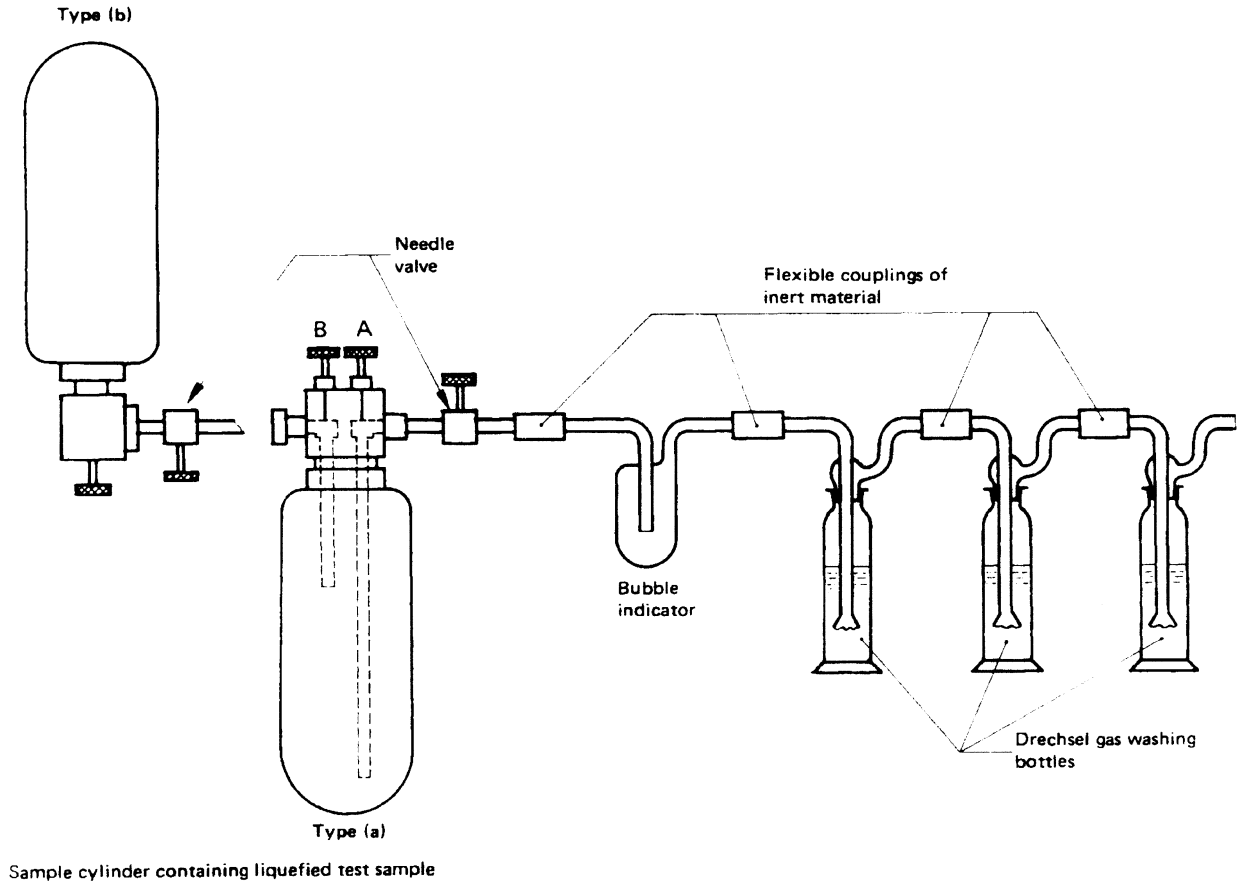

Figure — Apparatus for absorption of acidity for products gaseous at ordinary temperatures

Annex Other ISO publications relating to halogenated hydrocarbons for industrial use

ISO/R 1393, *Determination of acidity [liquids].*

ISO/R 1394, *Determination of cloud point [liquids].*

ISO 2209, *Sampling [liquids].*

ISO 2210, *Determination of residue on evaporation [liquids].*

ISO 3427, *Taking of a sample [liquefied gases].*

Publications referred to

See national foreword

BSI — British Standards Institution

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W4 4AL