INTERNATIONAL STANDARD ISO 3363

H-20-17

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION • МЕЖДУНАРОДНАЯ ОРГАНИЗАЦИЯ ПО СТАНДАРТИЗАЦИИ • ORGANISATION INTERNATIONALE DE NORMALISATION

Fluorochlorinated hydrocarbons for industrial use — Determination of acidity — Titrimetric method

Hydrocarbures fluorochlorés à usage industriel — Détermination de l'acidité — Méthode titrimétrique

First edition — 1976-09-01

UDC 661.723-13/-16 : 543.241.5 **Ref. No. ISO 3363-1976 (E)**

Descriptors : fluorochlorinated hydrocarbons, chemical analysis, determination of content, acidity, volumetric analysis.

Price based on 3 pages

FOREWORD

ISO (the International Organization for Standardization) is a worldwide federation of national standards institutes (ISO Member Bodies). The work of developing International Standards is carried out through ISO Technical Committees. Every Member Body interested in a subject for which a Technical Committee has been set up has the right to be represented on that Committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work.

Draft International Standards adopted by the Technical Committees are circulated to the Member Bodies for approval before their acceptance as International Standards by the ISO Council.

International Standard ISO 3363 was drawn up by Technical Committee ISO/TC 47, *Chemistry,* and circulated to the Member Bodies in February 1974.

It has been approved by the Member Bodies of the following countries :

Austria	Hungary	South Africa, Rep. of
Belgium	India	Spain
Bulgaria	Ireland	Switzerland
Chile	Israel	Thailand
Czechoslovakia	Italy	Turkey
Egypt, Arab Rep. of	Netherlands	United Kingdom
France	Portugal	U.S.S.R.
Germany	Romania	

No Member Body expressed disapproval of the document.

Printed in Switzerland

3363-76 4851903 0024799 8

Fluorochlorinated hydrocarbons for industrial use — Determination of acidity — Titrimetric method

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 tall-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.

NOTES

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

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.

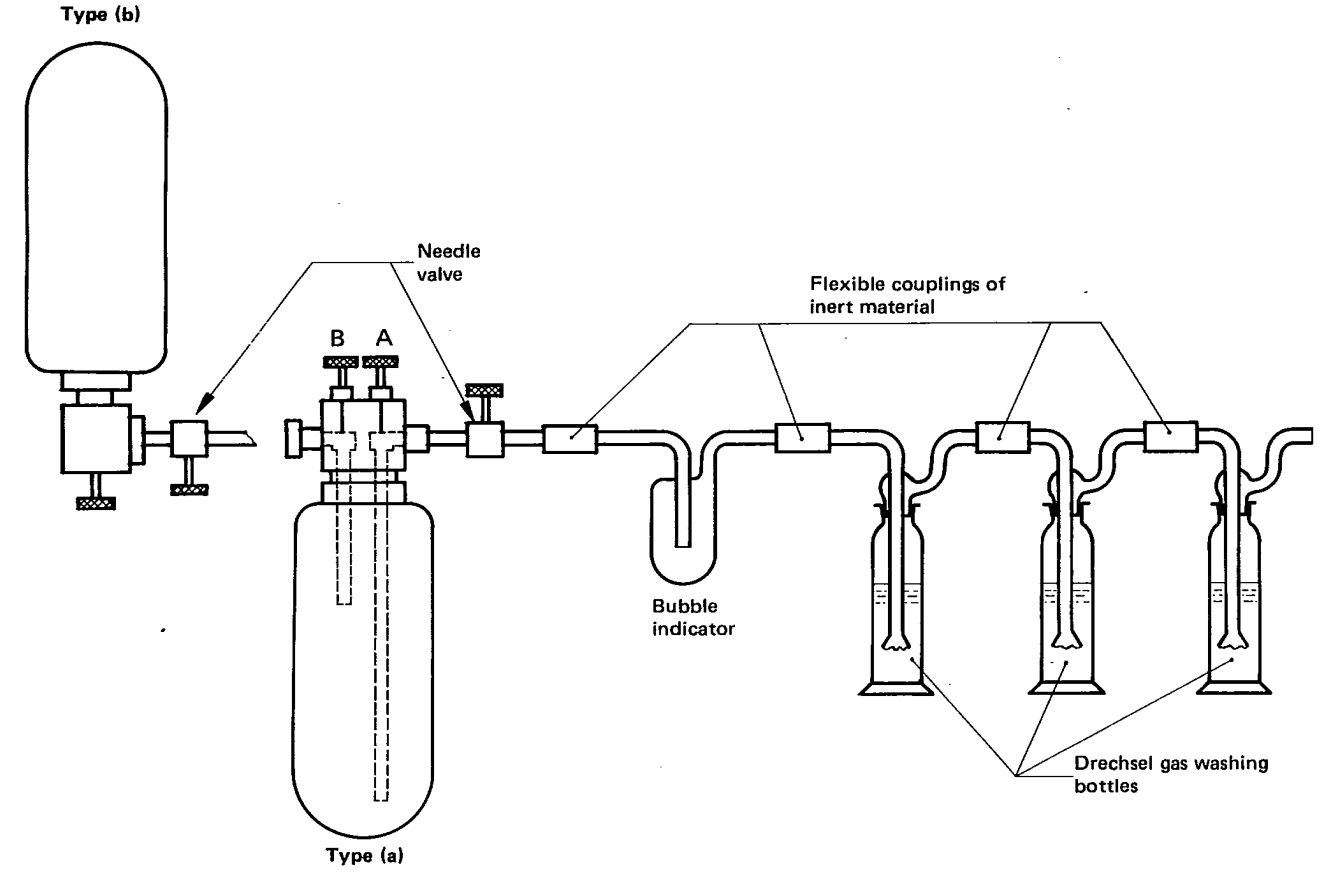

FIGURE — **Apparatus for absorption of acidity for products gaseous at ordinary temperatures**

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].