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Formic acid for industrial use - Methods of test -Part IV: Visual limit test for inorganic chlorides

Acide formique à usage industriel - Méthodes d'essai -Partie IV : Essai visuel limite de contrôle des chlorures minéraux

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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.

Prior to 1972, the results of the work of the technical committees were published as ISO Recommendations; these documents are in the process of being transformed into International Standards. As part of this process, Technical Committee ISO/TC 47, *Chemistry*, has reviewed ISO Recommendation R 731-1968 and found it technically suitable for transformation. ISO/R 731 has, however, been subdivised into seven parts. International Standard ISO 731/IV replaces clause 5 of ISO Recommendation R 731-1968, to which it is technically identical.

 $\ensuremath{\mathsf{ISO}}$ Recommendation R 731 had been approved by the member bodies of the following countries :

Austria India Romania South Africa, Rep. of Belgium Iran Israel Spain Bulgaria Chile Switzerland Italy Czechoslovakia Japan Turkey United Kingdom Egypt, Arab Rep. of Korea, Rep. of France Netherlands U.S.S.R. Germany New Zealand Yugoslavia Greece Poland Hungary Portugal

The member body of the following country had expressed disapproval of the Recommendation on technical grounds:

U.S.A.

The member body of the following country disapproved the transformation of the recommendation into an International Standard:

Netherlands

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Formic acid for industrial use — Methods of test — Part IV: Visual limit test for inorganic chlorides

1 SCOPE AND FIELD OF APPLICATION

This part of ISO 731 specifies a visual limit test for inorganic chlorides present in formic acid for industrial use.

The method is applicable to products having inorganic chlorides contents, expressed as chloride (Cl⁻), in the range 0,000 5 to 0,5 % (m/m). If the inorganic chlorides content lies outside this range, the range of applicability may be extended by increasing or reducing the mass of the test portion.

This document should be read in conjunction with part I (see the annex).

2 PRINCIPLE

Comparison of the turbidity, obtained by the addition of silver nitrate to a solution prepared from a test portion in the presence of nitric acid, with that similarly obtained from a standard chloride solution.

3 REAGENTS

During the analysis, use only reagents of recognized analytical grade and only distilled water or water of equivalent purity.

- 3.1 Nitric acid, approximately 5 N solution.
- 3.2 Silver nitrate, 50 g/l solution.
- **3.3 Chloride**, standard solution corresponding to 0,1 g of CI⁻ per litre.

Place 28,2 ml of 0,1 N standard volumetric hydrochloric acid solution in a 1 000 ml one-mark volumetric flask. Dilute to the mark and mix.

1 ml of this standard solution contains 0,000 1 g of Cl-.

4 APPARATUS

Ordinary laboratory apparatus and

4.1 Filter paper, chloride free.

4.2 Two matched Nessler cylinders, of capacity 100 ml.

5 PROCEDURE

5.1 Test portion

Weigh, to the nearest $0.5\,\mathrm{g}$, approximately $50\,\mathrm{g}$ of the laboratory sample.

5.2 Preparation of test solution

Transfer the test portion (5.1) to a 250 ml one-mark volumetric flask, dilute to the mark and mix thoroughly. If the solution is not clear, pass it through the filter paper (4.1). This should remove turbidity due to impurities such as aluminium. If there is turbidity in the filtrate due to contamination with wax, remove it by shaking with a suitable solvent, for example light petroleum.

5.3 Comparison

Place 1,0 ml of the standard chloride solution (3.3) in one of the Nessler cylinders (4.2), dilute to the mark, add 2 ml of the nitric acid solution (3.1) and mix.

For a sample required to contain not more than x % (m/m)

of inorganic chlorides expressed as CI⁻, transfer an aliquot portion numerically equal to $\left(\frac{0.05}{x}\right)^*$ millilitres of the test solution (5.2) to the other cylinder, dilute to the mark, add 2 ml of the nitric acid solution and mix.

Add 1 ml of the silver nitrate solution (3.2) to each Nessler cylinder and mix. Allow the cylinders to stand in the dark for 5 min and compare the turbidity produced by the aliquot portion of the test solution with that produced by

the standard chloride solution.

6 EXPRESSION OF RESULTS

If the turbidity produced from the test solution is less than that produced from the standard chloride solution, report that the sample contains less than x % (m/m) of inorganic chlorides expressed as Cl^- . Otherwise, report it as containing not less than x % (m/m).

^{*} If the mass of the test portion (5.1) was reduced or increased (see clause 1), adjust the numerator of this fraction accordingly.

ANNEX

ISO PUBLICATIONS RELATING TO FORMIC ACID FOR INDUSTRIAL USE

ISO 731/I -- General.

ISO 731/II — Determination of total acidity — Titrimetric method.

ISO 731/III — Determination of content of other acids — Potentiometric method.

ISO 731/IV — Visual limit test for inorganic chlorides.

ISO 731/V — Visual limit test for inorganic sulphates.

ISO 731/VI — Determination of iron content — 2,2'-Bipyridyl photometric method.

ISO 731/VII - Determination of low contents of other volatile acids - Titrimetric method after distillation.