INTERNATIONAL STANDARD

ISO 17531

First edition
2002-06-15

Corrected version
2003-06-15

Photography — Processing chemicals — Specifications for 4-(*N*-ethyl-*N*-2-methanesulfonylaminoethyl)-2-methylphenylenediamine sesquisulfate monohydrate

Photographie — Produits chimiques de traitement — Spécifications pour 4-(N-éthyle-N-2-sulfonylaminoéthyle de méthane)-2-phénylénédiamine de méthyle sesquisulfate monohydraté

Reference number
ISO 17531:2002(E)

ISO copyright office
Case postale 56 • CH-1211 Geneva 20
Tel. + 41 22 749 01 11
Fax + 41 22 749 09 47
E-mail copyright@iso.org
Web www.iso.org

Published in Switzerland

Contents

Page

Foreword iv

Introduction v

1 **Scope** 1

2 **Normative references** 1

3 **General** 2
3.1 **Physical properties** 2
3.2 **Hazardous properties** 2
3.3 **Handling and storage** 2

4 **Requirements** 2

5 **Reagents and glassware** 3

6 **Sampling** 3

7 **Test methods** 3
7.1 **Assay** 3
7.2 **Identity test** 4
7.3 **Residue after ignition** 5
7.4 **Heavy metals content** 5
7.5 **Iron content** 6
7.6 **Volatile matter** 6
7.7 **Appearance of solution** 6
7.8 **Photographic-use test** 6

Annex A (informative) **Preparation of standard ammonium cerium (IV) hexanitrate solution,** $c[(NH_4)_2Ce(NO_3)_6]$ = 0,05 mol/l (27,41 g/l) 8

Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established 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. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 3.

The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

Attention is drawn to the possibility that some of the elements of this International Standard may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 17531 was prepared by Technical Committee ISO/TC 42, *Photography*.

Annex A of this International Standard is for information only.

In this corrected version of ISO 17531, the following have been corrected:

— the lack of italicization of the symbol "N" in the main title and in a footnote;

— the definition of m in the equation of A.5;

— the non-italicization of the symbol for "concentration";

— an incorrect reference to another International Standard in the Foreword;

— some minor typographical errors.

Introduction

This International Standard is one of a series that establishes criteria of purity for chemicals used in processing photographic materials. General test methods and procedures cited in this International Standard are compiled in ISO 10349-1.

This International Standard is intended for use by individuals with a working knowledge of analytical techniques, which may not always be the case. Some of the procedures utilize caustic, toxic, or otherwise hazardous chemicals. Safe laboratory practice for the handling of chemicals requires the use of safety glasses or goggles and, in some cases, other protective apparel such as rubber gloves, face masks or aprons. Normal precautions for the safe performance of any chemical procedure shall be exercised at all times, but specific details have been provided for hazardous materials. Hazard warnings designated by a letter enclosed in angle brackets, ⟨ ⟩, are used as a reminder in those steps detailing handling operations and are defined in ISO10349-1. More detailed information regarding hazards, handling and use of these chemicals may be available from the manufacturer.

This International Standard provides chemical and physical requirements for the suitability of a photographic-grade chemical. The tests correlate with undesirable photographic effects. Purity requirements are set as low as possible consistent with these photographic effects. These criteria are considered to be the minimum requirements necessary to assure sufficient purity for use in photographic processing solutions; however, if the purity of a commonly available grade of chemical exceeds photographic processing requirements and if there is no economic penalty in its use, the purity requirements have been set to take advantage of the availability of the higher quality material.

Every effort has been made to keep the number of requirements to a minimum. Inert impurities are limited to amounts that will not unduly reduce the assay. All tests are performed on samples "as received" to reflect the condition of materials furnished for use. Although the ultimate criterion for suitability of such a chemical is its successful performance in an appropriate use test, the shorter, more economical test methods described in this International Standard are generally adequate.

Assay procedures have been included in all cases where a satisfactory method is available. An effective assay requirement serves not only as a safeguard of chemical purity, but also as a valuable complement to the identity test. Identity tests have been included whenever a possibility exists that another chemical or mixture of chemicals could pass the other tests.

All requirements listed in clause 4 are mandatory. The physical appearance of the material and any footnotes are for general information only and are not part of the requirements.

Efforts have been made to employ tests that are capable of being run in any normally equipped laboratory and, wherever possible, to avoid tests that require highly specialized equipment or techniques. Instrumental methods have been specified only as alternative methods or alone in those cases where no other satisfactory method is available.

Over the past several years, great improvements have been made in instrumentation for various analyses. Where such techniques have equivalent or greater precision, they may be used in place of the tests described in this International Standard. Correlation of such alternative procedures with the given method is the responsibility of the user. In case of disagreement in results, the method called for in the specification ought to prevail. Where a requirement states "to pass test", however, alternative methods are not to be used.

Photography — Processing chemicals — Specifications for 4-(*N*-ethyl-*N*-2-methanesulfonylaminoethyl)-2-methylphenylenediamine sesquisulfate monohydrate

1 Scope

This International Standard establishes criteria for the purity of photographic-grade 4-(*N*-ethyl-*N*-2-methanesulfonyl-aminoethyl)-2-methylphenylenediamine sesquisulfate monohydrate[1) 2)] and specifies the test methods to be used to determine the purity.

2 Normative references

The following normative documents contain provisions which, through reference in this text, constitute provisions of this International Standard. For dated references, subsequent amendments to, or revisions of, any of these publications do not apply. However, parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the normative documents indicated below. For undated references, the latest edition of the normative document referred to applies. Members of ISO and IEC maintain registers of currently valid International Standards.

ISO 565, *Test sieves — Metal wire cloth, perforated metal plate and electroformed sheet — Nominal sizes of openings*

ISO 10349-1, *Photography — Photographic-grade chemicals — Test methods — Part 1: General*

ISO 10349-4, *Photography — Photographic-grade chemicals — Test methods — Part 4: Determination of residue after ignition*

ISO 10349-5:1992, *Photography — Photographic-grade chemicals — Test methods — Part 5: Determination of heavy metals and iron content*

ISO 10349-8, *Photography — Photographic-grade chemicals — Test methods — Part 8: Determination of volatile matter*

---

1) Sold under such trade names as CD-3 (Eastman Chemicals), FCD-03 (Fuji), and Color Developer 3 (Merck). This information is given for the convenience of users of this International Standard and does not constitute an endorsement by ISO of these products.

2) Other names:
N-ethyl-*N*-(ß-methylsulfonamidoethyl)-3-methyl-p-phenylenediamine sesquisulfate monohydrate
4-amino-*N*-ethyl-*N*-(ß-methanesulfonamidoethyl)-m-toluidine sesquisulfate monohydrate
4-amino-*N*-(ß-methanesulfonamidoethyl)-3-methylaniline sesquisulfate monohydrate
N-(4-amino-*N*-ethyl-m-toluidine)-ethylmethanesulfonamide sesquisulfate monohydrate

3 General

3.1 Physical properties

4-(*N*-ethyl-*N*-2-methanesulfonylaminoethyl)-2-methylphenylenediamine sesquisulfate monohydrate ($C_{12}H_{21}N_3O_2S \cdot 3/2\ H_2SO_4 \cdot H_2O$) exists in the form of white to medium-tan or light-pink coloured granules or powder. It has a relative molecular mass of 436,52.

3.2 Hazardous properties

4-(*N*-ethyl-*N*-2-methanesulfonylaminoethyl)-2-methylphenylenediamine sesquisulfate monohydrate may cause skin irritation and allergic skin reaction. Also, this material is corrosive and should under no circumstances be swallowed. (DANGER: ⟨C⟩ ⟨S⟩)

NOTE Hazard warning codes are defined in ISO 10349-1.

3.3 Handling and storage

Handle this material carefully, avoid contact with skin and avoid breathing the dust. Store in tightly sealed containers and protect from light at room temperature.

4 Requirements

A summary of the requirements is shown in Table 1.

Table 1 — Summary of requirements

Test	Limit	Subclause	International Standard in which test method is given
Assay	Minimum: 99,0 % Maximum: 102,9 %	7.1	ISO 17531
Identity test Infrared spectrum	Equivalent to Figure 1	7.2	ISO 17531
Residue after ignition	Maximum: 0,10 %	7.3	ISO 10349-4
Heavy metals content (as Pb)	Maximum: 0,002 %	7.4	ISO 10349-5
Iron content (Fe)	Maximum: 0,002 %	7.5	ISO 10349-5
Volatile matter	Maximum: 0,60 %	7.6	ISO 10349-8
Appearance of solution	Clear and colourless or pale pink colour and free from insoluble matter	7.7	ISO 17531
Photographic-use test	To pass test	7.8	ISO 17531
NOTE % equals mass fraction.			

5 Reagents and glassware

All reagents, materials and glassware shall conform to the requirements specified in ISO 10349-1, unless otherwise noted. The hazard warning symbols, used as a reminder in those steps detailing handling operations, are defined in ISO 10349-1. These symbols are used to provide information to the user and are not meant to provide conformance with hazardous labelling requirements, as these vary from country to country.

6 Sampling

For details on sampling, see ISO 10349-1.

7 Test methods

7.1 Assay

7.1.1 Specification

Content of 4-(*N*-ethyl-*N*-2-methanesulfonylaminoethyl)-2-methylphenylenediamine sesquisulfate monohydrate shall be between 99,0 % and 102,9 %.

7.1.2 Reagents

7.1.2.1 Ammonium cerium (IV) hexanitrate, $c[(NH_4)_2Ce(NO_3)_6] = 0{,}050\ 0$ mol/l (27,41 g/l)

Commercially available analytical reagent solution is recommended. If the solution is to be prepared, see any quantitative analytical chemistry text.

NOTE A procedure for the preparation and standardization of this solution is given in annex A.

7.1.2.2 Ferrous sulfate, $c(FeSO_4) = 0{,}025$ mol/l (3,80 g/l)

Dissolve 4,25 g of $FeSO_4 \cdot H_2O$ in 1 litre of water. (Alternatively 5,60 g/l of $FeSO_4 \cdot 4H_2O$, 6,05 g of $FeSO_4 \cdot 5H_2O$ or 6,95 g of $FeSO_4 \cdot 7H_2O$ may be used.)

7.1.2.3 Ferroin solution

Dissolve 1,48 g of 1,10-phenanthroline in 100 ml of 0,025 mol/l ferrous sulfate (7.1.2.2).

7.1.2.4 Sulfuric acid, $c(H_2SO_4) = 0{,}5$ mol/l (DANGER:⟨C⟩)

NOTE This may be prepared from sulfuric acid, $\rho \sim 1{,}84$ g/l (DANGER: ⟨⟨C⟩⟩).

7.1.3 Apparatus

7.1.3.1 Potentiometric titrator, automatic titrator or equivalent, equipped with a 20 ml or 25 ml burette.

7.1.3.2 Indicator electrode, platinum electrode.

7.1.3.3 Reference electrode, double-junction reference electrode.

7.1.3.4 Stirring apparatus, magnetic or the equivalent.

7.1.4 Procedure

Weigh, to the nearest 0,000 1 g, a test portion of about 0,10 g into a 250 ml beaker. Add 60 ml of 0,5 mol/l sulfuric acid (7.1.2.4) and dissolve the sample. While starting to titrate, add 5 drops of ferroin solution (7.1.2.3). Set the solution on a potentiometric titrator (7.1.3.1). Titrate the solution with 0,05 mol/l ammonium cerium (IV) hexanitrate (7.1.2.1). Determine the endpoint in accordance with the instruction of the automatic titrator or a textbook for potentiometric titration.

7.1.5 Expression of results

The assay, expressed as a percentage by mass, for 4-(*N*-ethyl-*N*-2-methanesulfonylaminoethyl)-2-methyl-phenylenediamine sesquisulfate monohydrate ($C_{12}H_{21}N_3O_2S{\cdot}3/2\ H_2SO_4{\cdot}H_2O$), may be given automatically on the automatic titrator, or is given by

$$A = 21{,}83 \times \frac{cV}{m}$$

where

A is the assay, expressed as a percentage by mass, for 4-(*N*-ethyl-*N*-2-methanesulfonyl aminoethyl)-2-methylphenylenediamine sesquisulfate monohydrate ($C_{12}H_{21}N_3O_2S{\cdot}3/2\ H_2SO_4{\cdot}H_2O$);

c is the actual concentration, expressed in moles per litre, of the standard ammonium cerium (IV) hexanitrate solution $(NH_4)_2Ce(NO_3)_6$ (7.1.2.1);

V is the volume, expressed in millilitres, of the standard ammonium cerium (IV) hexanitrate solution (7.1.2.1) used to reach the titration endpoint;

M is the mass, expressed in grams, of the test portion;

21,83 is the conversion factor obtained from the mass of 4-(*N*-ethyl-*N*-2-methanesulfonyl-aminoethyl)-2-methylphenylenediamine sesquisulfate monohydrate equivalent to 1 mole of ammonium cerium (IV) hexanitrate (i.e. 436,52/2 = 218,3) × the conversion factor for millilitres to litres (i.e. 0,001) × 100 (for percentage).

The free base may be calculated by substituting the molecular weight of the free base (271,38) for the molecular weight of 4-(*N*-ethyl-*N*-2-methanesulfonylaminoethyl)-2-methylphenylenediamine sesquisulfate monohydrate (436,52).

7.2 Identity test

7.2.1 Infrared spectrum

7.2.1.1 Specification

The infrared absorption curve shall be essentially the same as that of the reference spectrum (Figure 1).

7.2.1.2 Apparatus

7.2.1.2.1 Test sieve, 63 µm aperture size, conforming to ISO 565.

7.2.1.2.2 Infrared spectrometer, equipped for the 2 µm to 16 µm region, and with accessory equipment for using potassium bromide plates or mineral oil mull.

7.2.1.3 Procedure

Grind about 10 mg of the sample to a homogeneous fine powder and prepare a 0,5 % (mass fraction) mixture of the sample in finely ground potassium bromide. Grind together thoroughly to pass through the test sieve (7.2.1.2.1). Prepare a pressed plate of the mixture containing 0,13 g to 0,16 g of the mixture per square centimetre. Record the infrared spectrum from 2 µm to 16 µm. Compare with the reference spectrum given in Figure 1.

NOTE As an alternative procedure, the sample may be ground and dispersed in mineral oil. It will then be necessary to take into account the absorption bands of the oil.

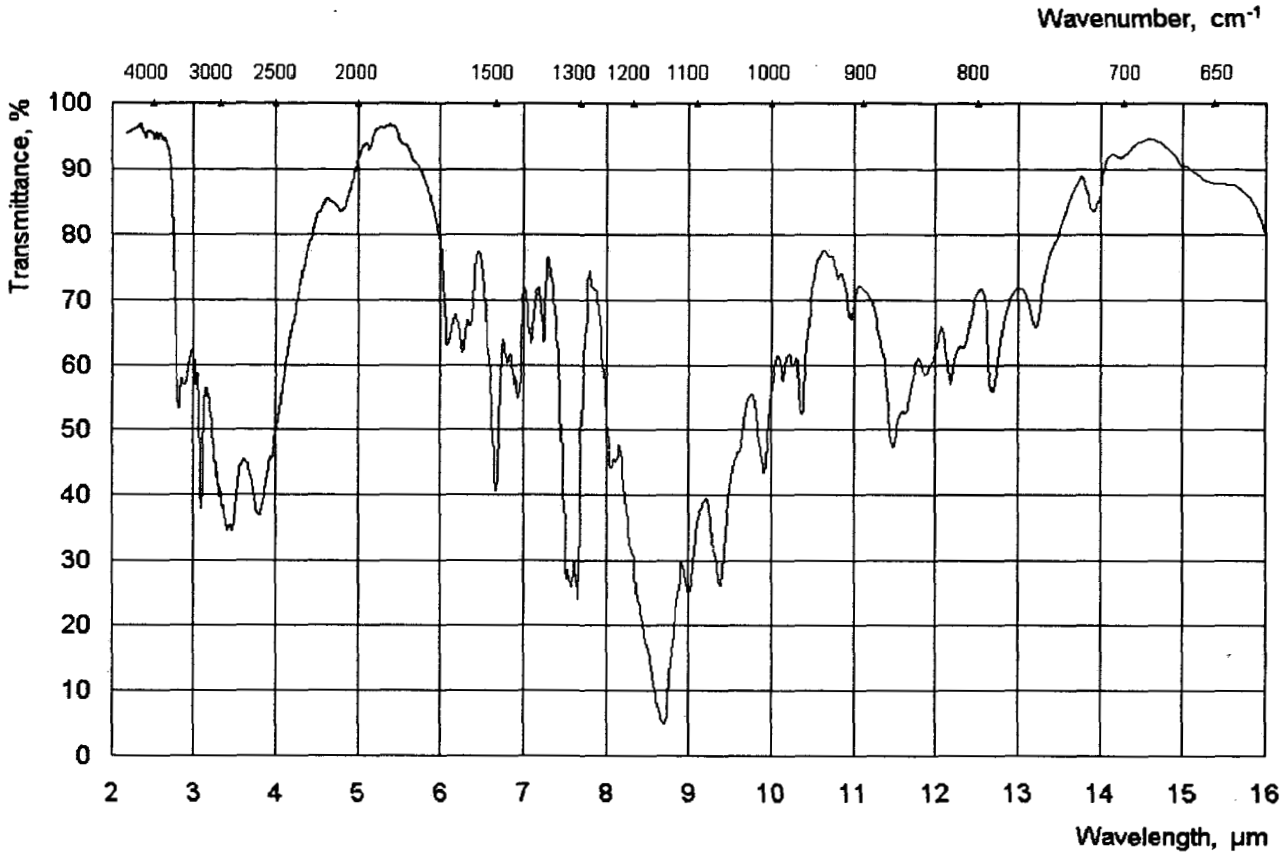

Figure 1 — Reference infrared spectrum of 4-(*N*-ethyl-*N*-2-methanesulfonylaminoethyl)-2-methylphenylenediamine sesquisulfate monohydrate (KBr plate)

7.3 Residue after ignition

7.3.1 Specification

Maximum residue after ignition shall be 0,10 %.

7.3.2 Procedure

Determine the percentage of the residue after ignition in accordance with ISO 10349-4. Use a test portion of about 5,0 g and ignite in a platinum crucible (600 °C ± 50 °C, 4 h, 0,000 1 g). Keep this residue for the heavy metals and iron content tests in 7.4 and 7.5.

7.4 Heavy metals content

7.4.1 Specification

Maximum content of heavy metals shall be 0,002 %.

7.4.2 Procedure

NOTE The standard for the iron test (7.5) is prepared in the same way as the heavy metals standard.

Determine the percentage of heavy metals in accordance with ISO 10349-5. Use a test portion of the residue after ignition (7.3) corresponding to 2 g of the sample prepared in accordance with 7.1 of ISO 10349-5:1992 (i.e. 10 ml of the 25 ml residue solution). Use 4 ml of the heavy metals standard prepared in accordance with 8.1.1 of ISO 10349-5:1992.

7.5 Iron content

7.5.1 Specification

Maximum content of iron shall be 0,002 %.

7.5.2 Procedure

Determine the percentage of iron in accordance with ISO 10349-5. Use a test portion of the residue after ignition (7.3) corresponding to 2 g of the sample prepared in accordance with 7.1 of ISO 10349-5:1992 (i.e. 10 ml of the 25 ml residue solution). Use 4 ml of the iron standard prepared in accordance with 8.1.1 of ISO 10349-5:1992.

7.6 Volatile matter

7.6.1 Specification

Maximum volatile matter shall be 0,60 %.

7.6.2 Procedure

Determine the percentage of volatile matter at 70 °C in accordance with ISO 10349-8. Use a test portion of 5 g, weighed to the nearest 0,00 1 g into a dry glass-stoppered weighing bottle (70 °C, 4 h, 0,000 1 g).

NOTE The notation system used for the drying process procedure is given in ISO 10349-1.

7.7 Appearance of solution

7.7.1 Specification

The solution shall be clear, colourless or a pale-pink colour, and free from insoluble matter.

7.7.2 Procedure

Dissolve a test portion of 10,0 g in 300 ml of water. Immediately observe the solution for colour and clarity.

7.8 Photographic-use test

7.8.1 Specification

To pass test.

7.8.2 Procedure

Since there is no chemical test currently available that can be used to successfully evaluate different batches of 4-(*N*-ethyl-*N*-2-methanesulfonylaminoethyl)-2-methylphenylenediamine sesquisulfate monohydrate, a photographic-use test shall be employed. Processing of colour negative and colour reversal films and colour papers has been found to be very useful for this purpose. The variability inherent in these colour processes or any process used for the photographic evaluation shall be established and well understood prior to the testing of the 4-(*N*-ethyl-*N*-2-methanesulfonylaminoethyl)-2-methylphenylenediamine sesquisulfate monohydrate samples. The sample is reported as satisfactory if it does not produce a photographic effect greater than the variability of the process or processes used in this test.

The processing solutions, photographic films, papers, processing procedures and temperatures used are to be the same as those regularly used for the colour negative and colour reversal films and colour paper processes selected for this test as per the manufacturer's instructions.

Annex A
(informative)

Preparation of standard ammonium cerium (IV) hexanitrate solution, $c[(NH_4)_2Ce(NO_3)_6]$ = 0,05 mol/l (27,41 g/l)

A.1 Reagents

A.1.1 Ammonium cerium (IV) hexanitrate, $(NH_4)_2Ce(NO_3)_6$

A.1.2 Sulfuric acid, H_2SO_4, $\rho \approx 1{,}84$ (DANGER: ⟨⟨C⟩⟩).

A.1.3 Sodium oxalate, $Na_2C_2O_4$, primary standard-grade.

A.1.4 Hydrochloric acid, HCl, $\rho \approx 1{,}18$ (DANGER:⟨C⟩⟨B⟩).

A.1.5 Ferrous sulfate, $c(FeSO_4)$ = 0,025 mol/l (3,80 g/l).

Dissolve 4,25 g of $FeSO_4 \cdot H_2O$ in 1 l of water. (Alternatively 5,60 g/l of $FeSO_4 \cdot 4H_2O$, 6,05 g of $FeSO_4 \cdot 5H_2O$ or 6,95 g of $FeSO_4 \cdot 7H_2O$ may be used.)

A.1.6 Ferroin solution

Dissolve 1,48 g of 1,10-phenanthroline in 100 ml of 0,025 mol/l ferrous sulfate (A.1.5).

A.2 Apparatus

A.2.1 Stirring apparatus, magnetic or the equivalent.

A.2.2 Oven, capable of maintaining a temperature between 70 °C and 150 °C to within 5 °C.

A.2.3 Desiccator, containing a suitable desiccant.

A.2.4 Hot plate.

A.2.5 Potentiometric titrator, automatic titrator or equivalent, equipped with a 20 ml or 25 ml burette.

A.2.6 Indicator electrode, platinum electrode.

A.2.7 Reference electrode, double-junction electrode.

A.2.8 Thermometer, 0 °C to 100 °C.

A.3 Preparation

Weigh about 30 g of ammonium cerium (IV) hexanitrate (A.1.1) and mix with 100 ml of water in a 1 l beaker with mechanical stirring (A.2.1). **Using extreme caution**, add 28 ml of sulfuric acid (A.1.2) (DANGER: ⟨⟨C⟩⟩). Stir for 2 min. Add consecutive 100 ml portions of water (no more than a total of 800 ml of water), with stirring, until all the ammonium cerium (IV) hexanitrate has dissolved. Place the beaker in a cooling bath and cool the solution to room

temperature. Transfer the solution in the 1 l beaker to a 1 l one-mark volumetric flask. Make up to the mark with water and mix well.

NOTE 1 Commercially prepared primary standard ammonium cerium (IV) hexanitrate solution may be used as an alternative to this preparation and the following standardization.

NOTE 2 To prepare 0,05 mol/l standard ammonium cerium (IV) hexanitrate solution by diluting 0,10 mol/l standard ammonium cerium (IV) hexanitrate solution, pipette 500 ml of 0,10 mol/l ammonium cerium (IV) hexanitrate solution into a 1 l one-mark volumetric flask, add 90 ml of 3 mol/l sulfuric acid and make up to the mark with water.

A.4 Standardization

Dry approximately 0,5 g of sodium oxalate (A.1.3) in an oven (A.2.2) at 105 °C for 2 h. Cool to room temperature in a desiccator (A.2.3). To a 250 ml beaker, add 100 ml of water. Weigh, to the nearest 0,000 1 g, 0,050 g of dried sodium oxalate. Transfer to the 250 ml beaker and stir to dissolve. Add 20 ml of hydrochloric acid (A.1.4) (DANGER: ⟨C⟩⟨B⟩) and stir the solution. Heat the solution to 70 °C ± 5 °C using a hot plate (A.2.4). Add 5 drops of ferroin solution (A.1.6). Set the solution on a potentiometric titrator (A.2.5). Allow the electrodes (A.2.6, A.2.7) to equilibrate for 5 min. Do not allow the solution to stand for longer than 5 min before the titration.

Titrate the solution with 0,05 mol/l ammonium cerium (IV) hexanitrate solution (A.3). Determine the endpoint in accordance with the instruction of the potentiometric titrator or a textbook for potentiometric titration.

A.5 Expression of results

The actual concentration, in moles per litre, of the ammonium cerium (IV) hexanitrate solution is given by

$$c = 14{,}93 \times \frac{m}{V}$$

where

c is the actual concentration, expressed in moles per litre, of the standard ammonium cerium (IV) hexanitrate solution;

m is the mass, expressed in grams, of the sodium oxalate;

V is the volume, expressed in millilitres, of the ammonium cerium (IV) hexanitrate solution;

14,93 is the conversion factor for the conversion of litres to millilitres (i.e. 1 000) divided by the equivalent weight of sodium oxalate (134,00/2).

ICS 37.040.30

Price based on 9 pages