INTERNATIONAL
STANDARD

ISO
422

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Photography — Processing chemicals — Specifications for *p*-methylaminophenol sulfate

Photographie — Produits chimiques de traitement — Spécifications pour le sulfate de p-*méthylaminophénol*

Reference number
ISO 422:1994(E)

Foreword

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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.

International Standard ISO 422 was prepared by Technical Committee ISO/TC 42, *Photography*.

This second edition cancels and replaces the first edition (ISO 422:1976) which has been technically revised.

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Introduction

0.1 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 parts 1, 4, 5 and 8 of ISO 10349.

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, rubber gloves and other protective apparel such as face masks or aprons where appropriate. Normal precautions required in the performance of any chemical procedure are to be exercised at all times but care has been taken to provide warnings 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 ISO 10349-1. More detailed information regarding hazards, handling and use of these chemicals may be available from the manufacturer.

0.2 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 the minimum requirements necessary to assure sufficient purity for use in photographic processing solutions, except that 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 which 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.

0.3 Efforts have been made to employ tests which are capable of being run in any normally equipped laboratory and, wherever possible, to avoid tests which 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 few 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 shall prevail. Where a requirement states "to pass test", however, alternative methods shall not be used.

Photography — Processing chemicals — Specifications for *p*-methylaminophenol sulfate

1 Scope

This International Standard establishes criteria for the purity of photographic-grade *p*-methylaminophenol sulfate and specifies the test methods to be used to determine the purity.

2 Normative references

The following International Standards contain provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards

ISO 565:1983, *Test sieves — Woven metal wire cloth, perforated plate and electroformed sheet — Nominal sizes of openings.*

ISO 10349-1:1992, *Photography — Photographic-grade chemicals — Test methods — Part 1: General.*

ISO 10349-4:1992, *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 10349-8:1992, *Photography — Photographic-grade chemicals — Test methods — Part 8: Determination of volatile matter.*

3 General

3.1 Physical properties

p-Methylaminophenol sulfate, $HOC_6H_4NHCH_3 \cdot \frac{1}{2}H_2SO_4$, exists in the form of a white crystalline powder. It has a relative molecular mass of 172,20.

3.2 Hazardous properties

p-Methylaminophenol sulfate is not hazardous when handled with normal precautions.

3.3 Handling and storage

p-Methylaminophenol sulfate shall be stored in a closed container at room temperature.

4 Requirements

A summary of the requirements is shown in table 1.

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

See ISO 10349-1.

Table 1 — Summary of requirements

Test	Limit	Subclause	International Standard in which test method is given
Assay			
cerate titration	99,0 % (*m*/*m*) min. 102,0 % (*m*/*m*) max.	7.1	ISO 422
or alkali titration	97,5 % (*m*/*m*) min.	7.2	ISO 422
Identity			
melting point of nitroso derivative	130 °C to 135 °C	7.3.1	ISO 422
mixed melting point of nitroso derivative	Not less than sample or standard	7.3.1	ISO 422
infrared spectrum (optional)	Equivalent to figure 1	7.3.2	ISO 422
Residue after ignition	0,10 % (*m*/*m*) max.	7.4	ISO 10349-4
Heavy metals (as Pb)	0,002 % (*m*/*m*) max.	7.5	ISO 10349-5
Iron (Fe)	0,005 % (*m*/*m*) max.	7.6	ISO 10349-5
Volatile matter	0,3 % (*m*/*m*) max.	7.7	ISO 10349-8
p-Aminophenol	2,5 % (*m*/*m*) max.	7.8	ISO 422
p-Amino-*N*,*N*-dimethylaniline sulfate	To pass test	7.9	ISO 422
Solubility in diethyl ether	0,2 % (*m*/*m*) max.	7.10	ISO 422
Appearance of solution	Clear and not more than slightly yellow	7.11	ISO 422

NOTE — *m*/*m* = mass/mass

7 Test methods

7.1 Assay – Cerate titration

7.1.1. Specification

Content of $HOC_6H_4NHCH_3 \cdot \frac{1}{2}H_2SO_4$ shall be between 99,0 % (*m*/*m*) min. and 102,0 % (*m*/*m*) max.

7.1.2 Reagents

7.1.2.1 Ammonium cerium(IV) hexanitrate, $(NH_4)_2Ce(NO_3)_6$, standard volumetric solution of 0,100 mol/l (54,82 g/l)[1] [2].

7.1.2.2 Iron(II) sulfate solution, 0,025 mol/l (3,80 g/l).

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

7.1.2.3 Ferroin indicator [tris(1,10-phenanthroline) iron(II) ion].

Dissolve 1,48 g of 1,10-phenanthroline in 100 ml of the iron(II) sulfate solution (7.1.2.2).

7.1.2.4 Sulfuric acid, H_2SO_4, 0,050 mol/l (4,9 g/l)[1] [3] [4].

7.1.3 Apparatus

7.1.3.1 Watch glass.

7.1.3.2 Burette, of 50 ml capacity.

7.1.4 Procedure

Weigh, to the nearest 0,000 1 g, a test portion of about 0,25 g on to a watch glass (7.1.3.1). Transfer the watch glass and test portion to a 250 ml wide-mouth conical flask containing 100 ml of water and

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[1] Commercially available analysed reagent solutions are recommended. If solutions are to be prepared, see any quantitative analytical chemistry text.

[2] A procedure for the preparation and standardization of this solution is given in annex A.

[3] This may be prepared from sulfuric acid, $\rho \approx 1,84$ g/l (DANGER:<<C>>).

[4] Hazard warning codes are defined in ISO 10349-1.

10 ml of the sulfuric acid (7.1.2.4). Dissolve the sample add 3 drops of the ferroin indicator (7.1.2.3) and titrate with the ammonium cerate solution (7.1.2.1) to a light green colour which persists for 15 s.

7.1.5 Expression of results

The assay, expressed as a percentage by mass, for *p*-methylaminophenol sulfate ($HOC_6H_4NHCH_3 \cdot ½H_2SO_4$), is given by

$$8{,}61 cV/m$$

where

c	is the actual concentration, expressed in moles per litre, of the ammonium cerate solution, $(NH_4)_2Ce(NO_3)_6$ (7.1.2.1);
V	is the volume, in millilitres, of the ammonium cerate solution (7.1.2.1) used to reach the titration endpoint;
m	is the mass, in grams, of the test portion;
8,61	is the conversion factor obtained from the mass of *p*-methylaminophenol sulfate equivalent to 1 mole of ammonium cerate (i.e. 86,1) × the conversion factor for millilitres to litres (i.e. 0,001) × 100 (for percentage).

7.2 Assay – Alkali titration

7.2.1 Specification

Content of $HOC_6H_4NHCH_3 \cdot ½H_2SO_4$ shall be 97,5 % (m/m) min.

7.2.2 Reagents

7.2.2.1 Acetone, $(CH_3)_2CO$.

7.2.2.2 Sodium hydroxide, NaOH, standard volumetric solution of 0,100 mol/l (4,00 g/l)[1].

7.2.3 Apparatus

7.2.3.1 Burette, of 50 ml capacity.

7.2.3.2 Electronic pH-meter, equipped with a glass electrode and standard reference electrode.

7.2.4 Procedure

Weigh, to the nearest 0,000 1 g, a test portion of about 0,6 g and dissolve it in 50 ml of water. Add 90 ml of the acetone (7.2.2.1) and titrate potentiometrically using the burette (7.2.3.1) and pH-meter (7.2.3.2) with the sodium hydroxide (7.2.2.2). Plot the titration curve and determine the endpoint.

7.2.5 Expression of results

The assay, expressed as a percentage by mass, for *p*-methylaminophenol sulfate ($HOC_6H_4NHCH_3 \cdot ½H_2SO_4$), is given by

$$17{,}22 c'V'/m'$$

where

c'	is the actual concentration, expressed in moles per litre, of the sodium hydroxide (7.2.2.2);
V'	is the volume, in millilitres, of the sodium hydroxide (7.2.2.2) used to reach the titration endpoint;
m'	is the mass, in grams, of the test portion;
17,22	is the conversion factor obtained from the mass of *p*-methylaminophenol sulfate equivalent to 1 mole of sodium hydroxide (i.e. 172,2) × the conversion factor for millilitres to litres (i.e. 0,001) × 100 (for percentage).

7.3 Identity tests

7.3.1 Melting point of nitroso derivative

7.3.1.1 Specifications

The melting point shall be from 130 °C to 135 °C.

A mixed melting point shall be from 130 °C to 135 °C and shall not be less than either the sample nitroso derivative or the standard nitroso derivative.

7.3.1.2 Reagents

7.3.1.2.1 Sulfuric acid, H_2SO_4, $\rho \approx 1{,}84$ g/l (DANGER:<<C>>)[4].

7.3.1.2.2 Sodium nitrite solution, $NaNO_2$, 100 g/l.

Dissolve 10 g of sodium nitrite in 100 ml of water.

7.3.1.3 Apparatus

7.3.1.3.1 Capillary-tube melting point apparatus, complete with thermometer for the range 100 °C to 200 °C.

7.3.1.4 Procedure

7.3.1.4.1 Preparation of nitroso derivatives

Prepare the nitroso derivatives of the test sample and a sample known to be *p*-methylaminophenol sulfate by treating each in the following manner. Dissolve about 0,5 g in 10 ml of water and cool with ice to 5 °C to 10 °C. Add 5 drops of sulfuric acid (7.3.1.2.1) (<<C>>) and then add, drop by drop, 2 ml of the sodium nitrite solution (7.3.1.2.2). Filter the precipitates, then wash with water and dry at 70 °C for 1 h.

7.3.1.4.2 Melting point determinations

Prepare three capillary tubes containing:

a) the derivative of the sample to be tested;

b) the derivative of the sample known to be *p*-methylaminophenol sulfate;

c) a finely ground mixture of a) and b) mixed in equal proportions.

Identify the tubes and attach them to the thermometer. Heat the apparatus (7.3.1.3.1) to about 110 °C. Insert the thermometer with the derivatives of the samples attached, and thereafter heat at a constant rate of 1 °C/min. Note the melting point of each derivative as indicated by the first appearance of liquefaction.

7.3.2 Infrared spectrum

7.3.2.1 Specification

The infrared absorption curve shall be essentially the same as that of the reference spectrum (figure 1). This optional recommendation is supplementary to the requirements of 7.3.1.

7.3.2.2 Apparatus

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

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

7.3.2.3 Procedure

Grind about 1 g of the sample to a homogeneous fine powder and prepare a 0,5 % (*m*/*m*) mixture of the sample in finely ground potassium bromide. Grind together thoroughly to pass through the test sieve (7.3.2.2.1). 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 1 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.

7.4 Residue after ignition

7.4.1 Specification

Maximum residue after ignition shall be 0,10 % (*m*/*m*).

7.4.2 Procedure

Determine the percentage 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)[5]. Retain this residue for the heavy metals and iron content tests in 7.5 and 7.6.

7.5 Heavy metals content

7.5.1 Specification

Maximum content of heavy metals shall be 0,002 % (*m*/*m*).

7.5.2 Procedure

NOTE 2 The standard for the iron test (7.6) 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 (7.4) corresponding to 2 g of the sample prepared in accordance with ISO 10349-5:1992, 7.1 (i.e. 10 ml of the 25 ml residue solution). Use 4 ml of heavy metals standard prepared in accordance with ISO 10349-5:1992, 8.1.1.

7.6 Iron content

7.6.1 Specification

Maximum content of iron shall be 0,005 % (*m*/*m*).

7.6.2 Procedure

Determine the percentage of iron in accordance with ISO 10349-5. Use a test portion of the residue after ignition (7.4) corresponding to 2 g of the sample prepared in accordance with ISO 10349-5:1992, 7.1 (i.e. 10 ml of the 25 ml residue solution). Use 10 ml of iron standard prepared in accordance with ISO 10349-5:1992, 8.1.1.

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

7.7 Volatile matter

7.7.1 Specification

Maximum volatile matter shall be 0,3 % (*m*/*m*).

7.7.2 Procedure

Determine the percentage of volatile matter at 105 °C in accordance with ISO 10349-8. Use a test portion of 10 g, weighed to the nearest 0,1 g into a dry glass-stoppered weighing bottle (105 °C, 1 h, 0,000 1 g)[5].

7.8 *p*-Aminophenol content

7.8.1 Specification

Maximum *p*-aminophenol content shall be 2,5 % (*m*/*m*).

7.8.2 Reagents

7.8.2.1 Diethyl ether, $(C_2H_5)_2O$, spectrographic grade (DANGER:<<F>>).

7.8.2.2 Diethyl ether, $(C_2H_5)_2O$, water satured (DANGER:<<F>>).

Shake 100 ml of diethyl ether (7.8.2.1) with 10 ml of water. Using one of the separating funnels (7.8.3.2), draw off and discard the water.

7.8.2.3 Hydrochloric acid, HCl (1 + 9)[6].

7.8.2.4 Sodium nitrite, $NaNO_2$, 100 g/l.

Dissolve 10 g of sodium nitrite in 100 ml of water.

7.8.3 Apparatus

7.8.3.1 Filtering funnel, of 60 ml capacity, with medium-porosity fritted disc.

7.8.3.2 Three separating funnels, of 125 ml capacity.

7.8.3.3 Two volumetric flasks, of 250 ml capacity.

7.8.3.4 Pipette, of 10 ml capacity.

7.8.3.5 Pipette, of 25 ml capacity.

7.8.3.6 Ultraviolet spectrometer, capable of operating below 350 nm.

7.8.4 Procedure

Weigh, to the nearest 0,05 g, a test portion of about 1,0 g and dissolve it in 100 ml of the hydrochloric acid (7.8.2.3). Stir and add slowly 25 ml of the sodium nitrite (7.8.2.4). Continue stirring for about 1 min. Filter the mixture, using vacuum, through the filtering funnel (7.8.3.1) and, without washing, transfer the filtrate to one of the volumetric flasks (7.8.3.3) and dilute to the mark with water. Using the pipette (7.8.3.5), transfer a 25 ml aliquot of this solution into one of the clean, dry separating funnels (7.8.3.2). Extract it three times (using a clean dry funnel for each extraction) with the water-saturated diethyl ether (7.8.2.2) (<<F>>). Discard the ether and retain the aqueous solution.

Using the pipette (7.8.3.4), transfer a 10 ml aliquot of the aqueous extract to the other volumetric flask (7.8.3.3), dilute to the mark with water and mix well.

Using water as a blank, determine the net absorbance in a 10 mm quartz cell using the ultraviolet spectrometer (7.8.3.6) at 312 nm.

The net absorbance shall not be greater than 0,775, which corresponds to a concentration of 2,5 % (*m*/*m*).

7.9 *p*-Amino-*N*,*N*-dimethylaniline sulfate content

7.9.1 Specification

To pass test.

7.9.2 Reagents

7.9.2.1 Diethyl ether, $(C_2H_5)_2O$, spectrographic grade (DANGER:<<F>>).

7.9.2.2 Sodium hydroxide, NaOH, 400 g/l (DANGER:<C>).

7.9.2.3 Sulfuric acid, H_2SO_4 (1 + 9)[3].

7.9.3 Apparatus

7.9.3.1 Separating funnel, of 100 ml capacity.

7.9.3.2 Glass-stoppered test tube.

7.9.4 Procedure

Weigh, to the nearest 0,1 g, a test portion of about 2,0 g in a 50 ml beaker. Add 10 ml water and dissolve.

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[6] This may be prepared from hydrochloric acid, $\rho \approx 1{,}18$ g/l (DANGER:<C><B>).

Filter immediately, using a long-stem funnel, and collect the filtrate in the separating funnel (7.9.3.1). To the filtrate add 10 ml of the sodium hydroxide (7.9.2.2) (<C>) and 10 ml of the diethyl ether (7.9.2.1) (<<F>>). Shake the mixture, allow the two layers to separate and draw off and discard the aqueous layer. Rinse the side of the funnel with approximately 10 ml of water without shaking and draw off and discard the water. Add 10 ml of the sulfuric acid (7.9.2.3) and shake the mixture. Draw off the acid layer into the glass-stoppered test tube (7.9.3.2). Shake the acid solution for 1 min and note the colour.

The absence of a pink colour provides conformance with the specification.

7.10 Solubility in diethyl ether

7.10.1. Specification

Maximum solubility in diethyl ether shall be 0,2 % (*m*/*m*).

7.10.2 Reagent

7.10.2.1 Diethyl ether, $(C_2H_5)_2O$, anhydrous (DANGER:<<F>>).

7.10.3 Apparatus

7.10.3.1 Soxhlet extraction apparatus

7.10.4 Procedure

Weigh, to the nearest 0,1 g, a test portion of about 5 g into the previously weighed extraction thimble of the Soxhlet extraction apparatus (7.10.3.1). Place the thimble and contents in the apparatus and extract with the diethyl ether (<<F>>) for 6 h. Transfer the extract into a dried, previously weighed 150 ml beaker and evaporate to dryness on a steam bath. Dry at 70 °C for 30 min, cool in a desiccator and weigh the beaker and contents to the nearest 0,001 g.

7.10.5 Expression of results

The solubility in diethyl ether, expressed as a percentage by mass, is given by

$$100\,(m_4 - m_3)/(m_2 - m_1)$$

where

m_1 is the mass, in grams, of the extraction thimble;

m_2 is the mass, in grams, of the extraction thimble and test portion;

m_3 is the mass, in grams, of the beaker;

m_4 is the mass, in grams, of the beaker and residue after drying.

7.11 Appearance of solution

7.11.1 Specification

The prepared solution shall be clear and not more than slightly yellow in colour.

7.11.2 Reagent

7.11.2.1 Hydrochloric acid, HCl (3 + 97)[6].

7.11.3 Procedure

Dissolve a test portion of 15,0 g in 500 ml of the hydrochloric acid (7.11.2.1). Observe the solution for colour and clarity.

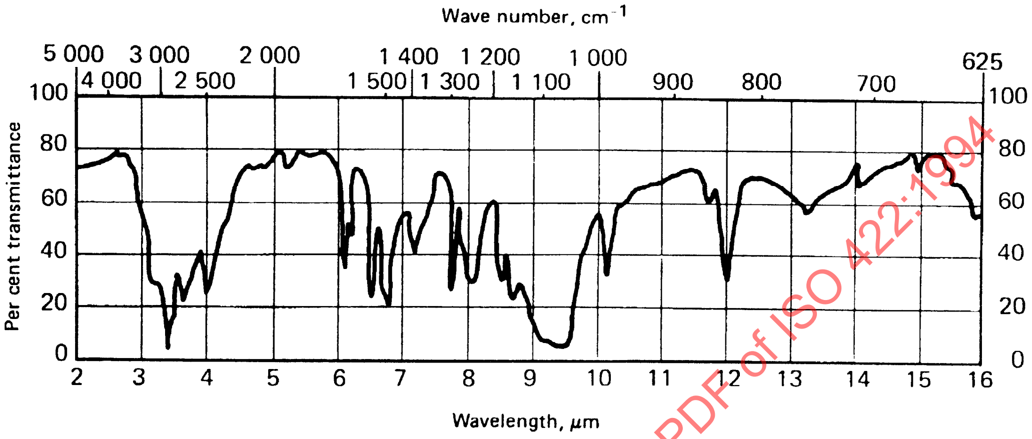

Figure 1 — Reference infrared spectrum of *p*-methylaminophenol sulfate (KBr plate)

Annex A

(informative)

Preparation of standard ammonium cerium(IV) hexanitrate solution, $c[(NH_4)_2Ce(NO_3)_6] = 0{,}100$ mol/l (54,82 g/l)

A.1 Reagents

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

A.1.2 Arsenic trioxide, As_2O_3, primary standard grade (DANGER:<<S>>).

A.1.3 Ferroin indicator [tris(1,10-phenanthroline) iron(II) ion]

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

A.1.4 Osmium tetroxide solution, OsO_4, (DANGER:<<S>> <<B>><C>).

Dissolve 0,25 g of osmium tetroxide (<<S>> <<B>><C>) in 100 ml of the 0,05 mol/l sulfuric acid (7.1.2.4).

A.1.5 Sodium hydroxide, NaOH, 100 g/l (DANGER:<C>).

A.1.6 Sulfuric acid, H_2SO_4, $\rho \approx 1{,}84$ g/l (DANGER:<<C>>).

A.1.7 Sulfuric acid, H_2SO_4 (1 + 5) (DANGER:<C>).

This may be prepared from sulfuric acid (A.1.6) (<<C>>).

A.2 Preparation

Weigh 50 g to 54 g of ammonium cerium(IV) hexanitrate (A.1.1) and mix with 27 ml of sulfuric acid (A.1.6) (<<C>>) in a 600 ml beaker with mechanical stirring. **Using extreme caution,** add water in 100 ml portions, with mechanical stirring, allowing 2 min to 3 min between each portion.

WARNING — The normal procedure for mixing acid and water is to add acid slowly to the water. The procedure being used here is the reverse of the normal. Therefore, extreme caution must be used to avoid possible splattering due to evolution of heat.

Continue the addition of water until the ammonium cerium(IV) hexanitrate is completely dissolved. Dilute to 1 litre with water and mix well.

NOTE 3 Commercially prepared primary standard cerium(IV) solutions may be used as an alternative to this preparation and the following standardization.

A.3 Standardization

Weigh, to the nearest 0,000 1 g, approximately 0,2 g of primary standard dry arsenic trioxide (A.1.2) (<<S>>) on a watch glass (7.1.3.1). Transfer the watch glass and contents to a 250 ml wide-mouth conical flask. Add 15 ml of sodium hydroxide (A.1.5) (<C>) and warm the mixture gently.

When dissolution is complete, cool to 20 °C ± 5 °C and add 25 ml of the dilute sulfuric acid (A.1.7) (<C>). Dilute to 100 ml with water, add 3 drops (approximately 0,15 ml) of the osmium tetroxide solution (A.1.4) (<<S>><<B>><C>) and 1 drop of the ferroin indicator (A.1.3).

Titrate this solution with the ammonium cerate solution (A.2) until the reddish orange colour changes to colourless or very pale blue. A sluggish endpoint indicates insufficient osmium tetroxide (A.1.4); up to 10 to 15 drops (approximately 0,7 ml) may be required as the solution ages.

A.4 Expression of results

The actual concentration, in moles per litre, of the ammonium cerate solution, $(NH_4)_2Ce(NO_3)_6$, is given by

$$20{,}22\ m/V$$

where

m is the mass, in grams, of the arsenic trioxide (A.1.2);

V is the volume, in millilitres, of the ammonium cerate solution (A.2) being standardized;

20,22 is the conversion factor for the conversion of litres to millilitres (i.e. 1 000) divided by the equivalent weight of arsenic trioxide (197,84/4).