
INTERNATIONAL STANDARD



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Photographic grade *p*-methylaminophenol sulphate — Specification

Sulfate de p-méthylaminophénol de qualité photographique — Spécifications

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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 now in the process of being transformed into International Standards. As part of this process, Technical Committee ISO/TC 42 has reviewed ISO Recommendation R 422 and found it technically suitable for transformation. International Standard ISO 422 therefore replaces ISO Recommendation R 422-1965.

ISO Recommendation R 422 was approved by the Member Bodies of the following countries :

Belgium	Italy	Switzerland
Brazil	Japan	United Kingdom
Canada	Netherlands	U.S.A.
Chile	New Zealand	U.S.S.R.
France	Romania	
Germany	Sweden	

No Member Body expressed disapproval of the Recommendation.

No Member Body disapproved the transformation of ISO/R 422 into an International Standard.

Photographic grade *p*-methylaminophenol sulphate — Specification

0 INTRODUCTION

This International Standard is one of a series of specifications for photographic grade chemicals which are commonly used in the processing of sensitized photographic materials. These specifications have been prepared to establish criteria of purity which will provide a practical and economical grade and prevent possible faulty processing which might be caused by chemicals of inferior quality, and to furnish manufacturers, suppliers, and processors with reliable and readily available specifications for photographic chemicals of satisfactory quality.

Photographic grade chemicals are those which meet the requirements specified in the appropriate International Standards. These specifications set out purity standards and state the limiting concentrations and test methods for certain inert or photographically harmful impurities that may be present.

Originally these specifications were based on known requirements for black-and-white photographic processing, but increased attention has been paid to the requirements of colour processing. Experience to date indicates that chemicals meeting these specifications are satisfactory for colour processes in general use.

0.1 Specification requirements

These specifications set out chemical and physical requirements. While it is recognized that the ultimate criterion of the quality of a photographic chemical is its successful performance in a photographic test, present knowledge indicates that, from a practical standpoint, chemical and physical methods of testing are generally adequate. The photographic industry has accumulated a comprehensive collection of such chemical tests for impurities. These tests, which correlate with objectionable photographic effects, have been drawn upon in the formulation of these specifications. Chemical tests are generally more sensitive, less variable, and less costly than photographic tests.

Purity requirements have been set as low as possible, consistent with the objectives mentioned. If, however, 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 higher quality materials.

Every effort has been made to keep the number of requirements in each specification to a minimum. The requirements generally include only those photographically harmful impurities which, through experience, are likely to be present. Inert impurities are limited to amounts which will not unduly reduce the assay.

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. All assays are intended to be made on undried samples in view of the fact that photographic processing chemicals are normally used "as received".

Identity tests have been included in the specifications wherever a possibility exists that another chemical or a mixture of chemicals could pass the other tests.

All requirements listed in clause 3 of each specification are mandatory. The physical appearance of the material and any footnotes are for general information only and are not part of the requirements.

0.2 Selection of test methods

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.

While the test methods set out in the specifications are recommended, the use of other equally reliable methods is allowed. 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.

0.3 Reagents

An effort has been made to minimize the number of reagents employed in this series of specifications. The methods of preparation and of standardization have been included in all cases where these are not common, or where a preferred method is desirable.

Details of reagent preparation and standardization are included in each specification in which the reagent is called for so that each specification shall be self-sufficient.

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies the purity requirements of, and test methods for, photographic grade *p*-methylaminophenol sulphate. These test methods are generally adequate to ensure satisfactory material but under certain conditions some samples may give high fog. In such cases a photographic test should be made under the conditions of intended use.

2 CHARACTERISTICS

p-Methylaminophenol sulphate is in the form of white crystalline powder, of chemical formula $\text{HOC}_6\text{H}_4\text{NHCH}_3 \cdot 1/2\text{H}_2\text{SO}_4$ and relative molar mass 172,2.

3 REQUIREMENTS

3.1 Assay

3.1.1 The assay shall be not less than 99,0 % (*m/m*) and not greater than 102,0 % (*m/m*), expressed as $\text{HOC}_6\text{H}_4\text{NHCH}_3 \cdot 1/2\text{H}_2\text{SO}_4$, when determined by the cerate titration method described in 4.1.

3.1.2 The assay shall be not less than 97,5 % (*m/m*), expressed as $\text{HOC}_6\text{H}_4\text{NHCH}_3 \cdot 1/2\text{H}_2\text{SO}_4$, when determined by the alkali titration method described in 4.2.

3.2 Appearance of solution

An acidified aqueous solution shall be clear with not more than a slight yellow colour when prepared and examined by the procedure described in 4.3.

3.3 Volatile matter at 105 °C

The volatile matter at 105 °C shall be not greater than 0,3 % (*m/m*), when determined by the method described in 4.4.

3.4 Residue after ignition

The residue after ignition shall be not greater than 0,10 % (*m/m*), when determined by the method described in 4.5.

3.5 Heavy metals content

The heavy metals content, expressed as lead (Pb), shall be not greater than 20 mg/kg.

Conformity with this requirement shall be determined by the limit test described in 4.6, when the colour produced in the test solution shall be not greater than that produced in the control solution.

3.6 Iron content

The iron content, expressed as iron (Fe), shall be not greater than 50 mg/kg.

Conformity with this requirement shall be determined by the limit test described in 4.7, when the colour produced in the test solution shall be not greater than that produced in the control solution.

3.7 *p*-Aminophenol content

The *p*-aminophenol content shall be not greater than 2,5 % (*m/m*).

Conformity with this requirement shall be determined by the limit test described in 4.8, when the solution shall have a net absorbance, measured at 312 nm in a 10 mm quartz cell, not greater than 0,775.

3.8 *p*-Amino-*N,N*-dimethylaniline sulphate content

The *p*-amino-*N,N*-dimethylaniline sulphate content shall be not greater than that permitted by the limit test described in 4.9, when no pink colour shall be produced in the test solution.

3.9 Solubility in ethyl ether

The solubility in ethyl ether shall be not greater than 0,2 % (*m/m*), when determined by the method described in 4.10.

3.10 Identity

3.10.1 Melting point

The melting point of the derivative shall be not lower than 130 °C and not higher than 135 °C, when determined by the method described in 4.11.1.

3.10.2 Mixed melting point

The melting point of the mixture of sample and standard shall be not lower than the melting point of either the sample or the standard, when determined by the method described in 4.11.1.

3.10.3 Infra-red spectrum

The infra-red absorption curve shall be essentially the same as that of the reference spectrum (see the figure), when determined by the method described in 4.11.2.

This requirement is an optional identity requirement supplementary to those of 3.10.1 and 3.10.2.

4 TEST METHODS

Reagents used in the tests shall be recognized reagent grade chemicals normally used for careful analytical work. In all the directions the acids and ammonia solution referred to shall be of full strength unless dilution is specified. Dilution is specified in terms of molar concentration (molarity)¹⁾ when standardization of the reagent is required. When

1) 1 mol/l = 1 kmol/m³ = 1 mol/dm³ = 1 M

dilution is indicated as (1 + x), it means that 1 volume of the reagent or strong solution is added to x volumes of distilled water.

Distilled water, or water otherwise produced of at least equal purity, shall be used whenever water is required.

4.1 Assay – Cerate titration method

4.1.1 Reagents

4.1.1.1 Arsenic trioxide (As_2O_3), primary standard.

4.1.1.2 Osmium tetroxide solution.

Dissolve 0,25 g of osmium tetroxide in 100 ml of the 0,05 M sulphuric acid solution (4.1.1.6).

CAUTION – Osmium tetroxide is poisonous – avoid contact.

4.1.1.3 Sulphuric acid solution, ρ approximately 1,84 g/ml.

4.1.1.4 Sulphuric acid solution, dilute (1 + 5).

4.1.1.5 Sodium hydroxide solution, 100 g/l.

4.1.1.6 Sulphuric acid, 0,05 M standard volumetric solution.

4.1.1.7 Cerate, 0,1 M standard volumetric solution

4.1.1.7.1 PREPARATION

Weigh 50 to 54 g of ammonium cerium(IV) hexanitrate $[(\text{NH}_4)_2\text{Ce}(\text{NO}_3)_6]$ and mix with 27 ml of the sulphuric acid solution (4.1.1.3) in a 600 ml beaker, with mechanical stirring. Using extreme caution, add water in 100 ml portions¹⁾ with mechanical stirring, allowing 2 to 3 min

between each portion. Continue the addition of water until the cerate is completely dissolved. Dilute to 1 000 ml with water and mix well

NOTE – A primary standard cerium(IV) compound may be used as an alternative to standardization with arsenic trioxide as described in 4.1.1.7.2.

4.1.1.7.2 STANDARDIZATION

Weigh, to the nearest 0,000 1 g, approximately 0,2 g of primary standard dry arsenic trioxide (4.1.1.1) on the watch glass (4.1.2.1). Transfer the watch glass and contents to a 250 ml wide-mouth conical flask. Add 15 ml of the sodium hydroxide solution (4.1.1.5) and warm the mixture gently. When dissolution is complete, cool to 20 ± 5 °C and add 25 ml of the sulphuric acid solution (4.1.1.4). Dilute to 100 ml with water, add 3 drops (approximately 0,15 ml) of the osmium tetroxide solution (4.1.1.2) and 1 drop of the ferroin indicator solution (4.1.1.8).

Titrate this solution with the cerate solution (4.1.1.7) until the reddish-orange colour changes to colourless or very pale blue. A sluggish end-point indicates insufficient osmium tetroxide; up to 10 to 15 drops (approximately 0,7 ml) may be required as the solution ages.

4.1.1.7.3 CALCULATION

The molarity, T , of the cerate solution is given by the formula

$$T = \frac{20,22 m}{V}$$

where

m is the mass, in grams, of the arsenic trioxide (4.1.1.1);

V is the volume, in millilitres, of the cerate solution (4.1.1.7) used for the standardization.

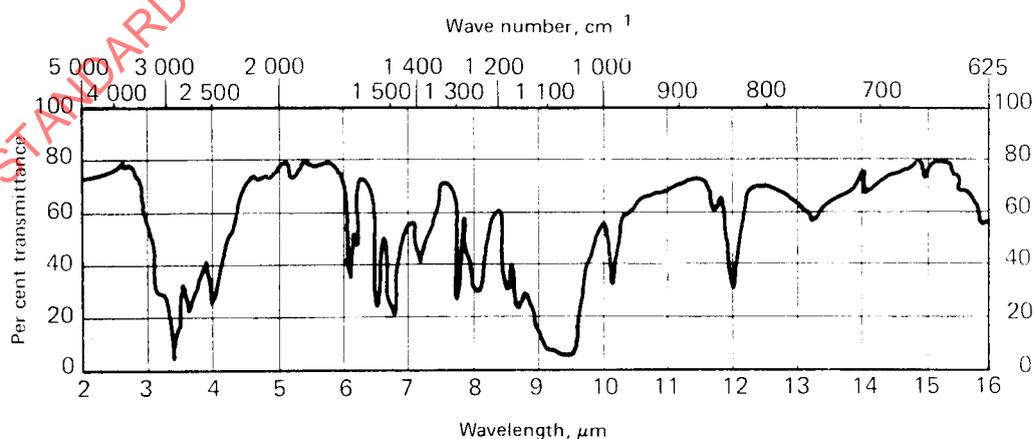


FIGURE – Reference infra-red spectrum of *p*-methylaminophenol sulphate (KBr plate)

1) 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 the possible splattering due to heat of evolution.

4.1.1.8 Ferroin [tris(1,10-phenanthroline)iron(2+) ion] indicator solution.

4.1.2 Apparatus

Ordinary laboratory apparatus and

4.1.2.1 Watch glass, 25 mm diameter.

4.1.2.2 Burette, 50 ml capacity, conforming to class A of ISO/R 385.

4.1.3 Procedure

Weigh, to the nearest 0,000 1 g, a test portion of about 0,25 g of the laboratory sample on the watch glass (4.1.2.1). Transfer the watch glass and test portion to a 250 ml wide-mouth conical flask containing 100 ml of water and 10 ml of the standard volumetric sulphuric acid solution (4.1.1.6). Dissolve the test portion, add 3 drops of the ferroin indicator solution (4.1.1.8) and titrate with the standard volumetric cerate solution (4.1.1.7) to a light-green colour which persists for 15 s.

4.1.4 Calculation

The assay, by cerate titration, expressed as a percentage by mass of *p*-methylaminophenol sulphate ($\text{HOC}_6\text{H}_4\text{NHCH}_3 \cdot 1/2\text{H}_2\text{SO}_4$), is given by the formula

$$\frac{8,61 V T}{m}$$

where

V is the volume, in millilitres, of the cerate solution (4.1.1.7) used for the titration;

T is the exact molarity of the cerate solution (4.1.1.7);

m is the mass, in grams, of the test portion.

NOTE – This titration also includes oxidizable material other than *p*-methylaminophenol sulphate.

4.2 Assay – Alkali titration method

4.2.1 Reagents

4.2.1.1 Acetone.

4.2.1.2 Sodium hydroxide, 0,1 M standard volumetric solution.

4.2.2 Apparatus

Ordinary laboratory apparatus and

4.2.2.1 Burette, 50 ml capacity, conforming to class A of ISO/R 385.

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

4.2.3 Procedure

Weigh, to the nearest 0,000 1 g, a test portion of about 0,6 g of the laboratory sample and dissolve in 50 ml of water. Add 90 ml of the acetone (4.2.1.1) and titrate potentiometrically with the standard volumetric sodium hydroxide solution (4.2.1.2). Plot the titration curve and determine the end-point.

4.2.4 Calculation

The assay, by alkali titration, expressed as a percentage by mass of *p*-methylaminophenol sulphate ($\text{HOC}_6\text{H}_4\text{NHCH}_3 \cdot 1/2\text{H}_2\text{SO}_4$), is given by the formula

$$\frac{17,22 V T}{m}$$

where

V is the volume, in millilitres, of the sodium hydroxide solution (4.2.1.2) used for the titration;

T is the exact molarity of the sodium hydroxide solution (4.2.1.2);

m is the mass, in grams, of the test portion.

4.3 Appearance of solution

4.3.1 Reagent

4.3.1.1 Hydrochloric acid solution, dilute (3 + 97).

4.3.2 Procedure

Prepare a 30 g/l solution of the laboratory sample in the hydrochloric acid solution (4.3.1.1) and examine for clarity and colour.

4.4 Determination of volatile matter at 105 °C

4.4.1 Apparatus

Ordinary laboratory apparatus and

4.4.1.1 Oven, capable of being controlled at 105 ± 5 °C.

4.4.2 Procedure

Weigh, to the nearest 0,001 g, a test portion of about 5 g of the laboratory sample into a previously weighed low-form, glass-stoppered weighing bottle. Remove the stopper and heat in the oven (4.4.1.1), controlled at 105 ± 5 °C, for 4 h. Cool in a desiccator, replace the stopper and weigh to the nearest 1 mg.

4.4.3 Calculation

The volatile matter at 105 °C is given, as a percentage by mass, by the formula

$$\frac{m_2 - m_3}{m_2 - m_1} \times 100$$

where

m_1 is the mass, in grams, of the weighing bottle;

m_2 is the mass, in grams, of the weighing bottle and test portion before heating;

m_3 is the mass, in grams, of the weighing bottle and test portion after heating.

4.5 Determination of residue after ignition

4.5.1 Apparatus

Ordinary laboratory apparatus and

4.5.1.1 Platinum crucible.

4.5.1.2 Muffle furnace, capable of being controlled at 600 ± 50 °C.

4.5.2 Procedure

Weigh, to the nearest 0,1 g, a test portion of about 5 g of the laboratory sample into the previously weighed platinum crucible (4.5.1.1), and heat carefully. Finally, ignite the residue in the furnace (4.5.1.2), controlled at 600 ± 50 °C, for 4 h. Cool in a desiccator and weigh the crucible and contents to the nearest 1 mg. Retain this residue for the heavy metals and iron limit tests under 4.6.3 and 4.7.3.

4.5.3 Calculation

The residue after ignition, expressed as a percentage by mass, is given by the formula

$$\frac{m_3 - m_1}{m_2 - m_1} \times 100$$

where

m_1 is the mass, in grams, of the crucible;

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

m_3 is the mass, in grams, of the crucible and residue.

4.6 Limit test for heavy metals

4.6.1 Reagents

4.6.1.1 Hydrochloric acid solution, ρ approximately 1,18 g/ml.

4.6.1.2 Hydrochloric acid solution, dilute (1 + 99).

4.6.1.3 Ammonia solution, dilute (1 + 9).

4.6.1.4 Heavy metals, standard solution.

Dissolve a soluble lead salt in water to give a solution containing 10 mg of lead per 1 000 ml.

4.6.1.5 Water, saturated at room temperature with hydrogen sulphide.

4.6.1.6 *p*-Nitrophenol indicator, solution, 2,5 g/l. g/l.

4.6.2 Apparatus

Ordinary laboratory apparatus and

4.6.2.1 Two matched Nessler cylinders, 50 ml capacity.

4.6.3 Procedure

Dissolve the residue from the ignition test under 4.5.2 in 0,5 ml of the hydrochloric acid solution (4.6.1.1), warming if necessary, and transfer the solution (with washing) to a beaker using about 40 ml of water. Also take 4 ml of the standard heavy metals solution (4.6.1.4) and treat this and the test solution in the following manner. Add 1 drop of the *p*-nitrophenol indicator solution (4.6.1.6) and then, drop by drop, the ammonia solution (4.6.1.3), until just yellow, followed by the hydrochloric acid solution (4.6.1.2), drop by drop until colourless again. Add 2,5 ml in excess.

Dilute the test solution to 50 ml with water and transfer a 20 ml aliquot to one of the Nessler cylinders (4.6.2.1), retaining the balance for the iron limit test under 4.7.3. Transfer the treated standard heavy metals solution to the other Nessler cylinder and treat both solutions in the following manner. Add 5 ml of the hydrogen sulphide water (4.6.1.5), dilute to 50 ml and mix well.

Compare, in the Nessler cylinders, the colours produced in the test and control solutions.

4.7 Limit test for iron

4.7.1 Reagents

As specified in 4.6.1 and

4.7.1.1 Acetate buffer solution, pH 5,0.

Dissolve 23 g of anhydrous sodium acetate in 58 ml of 2 M acetic acid and dilute to 1 000 ml with water. Adjust the final pH to $5,0 \pm 0,1$ with glacial acetic acid or 100 g/l sodium hydroxide solution.

4.7.1.2 Iron, standard solution.

Dissolve a soluble iron(III) salt in water to give a solution containing 10 mg of iron(III) per 1 000 ml.

4.7.1.3 1,10-Phenanthroline reagent solution.

Thoroughly mix equal parts of a 1 g/l aqueous solution of 1,10-phenanthroline, a 100 g/l aqueous solution of hydroxylammonium chloride and the acetate buffer solution (4.7.1.1).

4.7.2 Apparatus

Ordinary laboratory apparatus and

4.7.2.1 Two matched Nessler cylinders, 50 ml capacity.

4.7.3 Procedure

Take 10 ml of the standard iron solution (4.7.1.2) and treat in the same manner as the 4 ml of the standard heavy metals solution (4.6.1.4) as described in 4.6.3, as far as the addition of excess hydrochloric acid solution (4.6.1.2).

Transfer this treated iron solution to one of the Nessler cylinders (4.7.2.1) and 20 ml of the balance of the diluted test solution from 4.6.3 to the other Nessler cylinder and treat each solution in the following manner. Add 5 ml of the 1,10-phenanthroline reagent solution (4.7.1.3), mix, and allow to stand for 10 min. Dilute each to 50 ml and mix well.

Compare, in the Nessler cylinders, the colours produced in the test and control solutions.

4.8 Limit test for *p*-aminophenol

4.8.1 Reagents

4.8.1.1 Ethyl ether, water saturated, spectrographic grade.

Shake 100 ml of ethyl ether, spectrographic grade, with 10 ml of water. Using one of the separating funnels (4.8.2.2), draw off and discard the water.

4.8.1.2 Hydrochloric acid solution, dilute (1 + 9).

4.8.1.3 Sodium nitrite solution, 100 g/l.

4.8.2 Apparatus

4.8.2.1 Filtering funnel, 60 ml capacity, with medium porosity fritted disc.

4.8.2.2 Three separating funnels, 125 ml capacity.

4.8.2.3 Two volumetric flasks, 250 ml capacity, conforming to class A of ISO 1042.

4.8.2.4 Pipette, 10 ml capacity, conforming to class A of ISO/R 648.

4.8.2.5 Pipette, 25 ml capacity, conforming to class A of ISO/R 648.

4.8.2.6 Ultra-violet spectrophotometer, capable of operating below 350 nm.

4.8.3 Procedure

Weigh, to the nearest 0,05 g, a test portion of about 1,0 g of the laboratory sample and dissolve in 100 ml of the hydrochloric acid solution (4.8.1.2). Stir and add slowly 25 ml of the sodium nitrite solution (4.8.1.3). Continue stirring for about 1 min. Filter this mixture, using vacuum, through the filtering funnel (4.8.2.1), and, without washing, transfer the filtrate to one of the volumetric flasks (4.8.2.3) and dilute to the mark with water. Using the pipette

(4.8.2.5), transfer a 25 ml aliquot of this solution into one of the clean, dry, separating funnels (4.8.2.2). Extract it three times (using a clean and dry funnel for each extraction) with the ethyl ether (4.8.1.1). Discard the ether and retain the aqueous solution.

Using the pipette (4.8.2.4), transfer a 10 ml aliquot of the aqueous extract to the other volumetric flask, dilute to the mark and mix well.

Using water as a blank, determine the net absorbance in a 10 mm long quartz cell using the ultra-violet spectrophotometer (4.8.2.6).

4.9 Limit test for *p*-amino-*N,N*-dimethylaniline sulphate

4.9.1 Reagents

4.9.1.1 Ethyl ether.

4.9.1.2 Sodium hydroxide solution, 400 g/l.

4.9.1.3 Sulphuric acid solution, dilute (1 + 9).

4.9.2 Apparatus

Ordinary laboratory apparatus and

4.9.2.1 Separating funnel, 100 ml capacity.

4.9.2.2 Glass-stoppered test tube.

4.9.3 Procedure

Weigh, to the nearest 0,1 g, a test portion of about 2 g of the laboratory sample in a 50 ml beaker, add 10 ml of water and dissolve. Filter immediately, using a long-stem funnel, and collect the filtrate in the separating funnel (4.9.2.1). To the filtrate add 10 ml of the sodium hydroxide solution (4.9.1.2) and 10 ml of the ethyl ether (4.9.1.1). Shake the mixture, allow the two layers to separate and draw off and discard the aqueous layer. Rinse the sides of the funnel with approximately 10 ml of water without shaking and draw off and discard the water. Add 10 ml of the sulphuric acid solution (4.9.1.3) and shake the mixture. Draw off the acid layer into the glass-stoppered test tube (4.9.2.2). Shake the acid solution for 1 min and note the colour.

4.10 Solubility in ethyl ether

4.10.1 Reagent

4.10.1.1 Ethyl ether, anhydrous.

4.10.2 Apparatus

Ordinary laboratory apparatus and

4.10.2.1 Soxhlet extraction apparatus.

4.10.3 Procedure

Weigh, to the nearest 0,1 g, a test portion of about 5 g of the laboratory sample into the previously weighed extraction thimble of the Soxhlet extraction apparatus (4.10.2.1). Place the thimble and contents in the apparatus and extract with the ethyl ether (4.10.1.1) 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 1 mg.

4.10.4 Calculation

The solubility in ethyl ether is given, as a percentage by mass, by the formula

$$\frac{m_4 - m_3}{m_2 - m_1} \times 100$$

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 evaporation and drying.

4.11 Identity tests

4.11.1 Melting point tests

4.11.1.1 REAGENTS

4.11.1.1.1 Sulphuric acid solution, ρ approximately 1,84 g/ml.

4.11.1.1.2 Sodium nitrite solution, 100 g/l.

4.11.1.2 APPARATUS

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

4.11.1.3 PROCEDURE

Prepare nitroso derivatives of the laboratory sample and of

a sample known to be *p*-methylaminophenol sulphate by treating each in the following manner. Dissolve about 0,5 g in 10 ml of water and cool with ice to 5 to 10 °C. Add 5 drops of the sulphuric acid solution (4.11.1.1.1) and then add, drop by drop, 2 ml of the sodium nitrite solution (4.11.1.1.2). Filter, wash the precipitates with water and dry at 70 °C for 1 h.

Prepare three capillary tubes, containing :

- 1) the derivative of the sample to be tested;
- 2) the derivative of the sample known to be *p*-methylaminophenol sulphate;
- 3) a finely ground mixture of 1) and 2) mixed in the ratio 1 : 1.

Identify the tubes and attach them to the thermometer. Heat the apparatus (4.11.1.2) to about 110 °C, insert the thermometer with 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.

4.11.2 Infra-red identity test

4.11.2.1 APPARATUS

4.11.2.1.1 Test sieve, 63 μ m aperture size, conforming to ISO 565.

4.11.2.1.2 Infra-red spectrophotometer, equipped for the 2 to 16 μ m regions and accessory equipment for using potassium bromide plates or mineral oil mull.

4.11.2.2 PROCEDURE

Grind about 1 g of the laboratory 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 the test sieve (4.11.2.1.1). Prepare a pressed plate of the mixture containing 0,13 to 0,16 g of the mixture per square centimetre. Record the infra-red spectrum from 2 to 16 μ m. Compare with the reference spectrum given in the figure.

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.