UDC 77

Ref. No.: ISO / R 422 - 1965 (E)

ISO

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION

ISO RECOMMENDATION

R 422

SPECIFICATION FOR PHOTOGRAPHIC GRADE P-METHYLAMINOPHENOL SULPHATE

1st EDITION

March 1965

Printed in Switzerland

Also issued in French and Russian. Copies to be obtained through the national standards organizations.

BRIEF HISTORY

The ISO Recommendation R 422, *Specification for Photographic Grade P-Methylaminophenol Sulphate*, was drawn up by Technical Committee ISO/TC 42, *Photography*, the Secretariat of which is held by the American Standards Association, Inc. (ASA).

Work on this question by the Technical Committee began in 1956 and led, in 1959, to the adoption of a Draft ISO Recommendation.

In August 1961, this Draft ISO Recommendation (No. 425) was circulated to all the ISO Member Bodies for enquiry. It was approved, subject to a few modifications of an editorial nature, by the following Member Bodies:

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

No Member Body opposed the approval of the Draft.

The Draft ISO Recommendation was then submitted by correspondence to the ISO Council, which decided, in March 1965, to accept it as an ISO RECOMMENDATION.

ISO Recommendation R 422 March 1965

SPECIFICATION FOR PHOTOGRAPHIC GRADE P-METHYLAMINOPHENOL SULPHATE

1. SCOPE

This ISO Recommendation is one of a series to establish criteria of purity of chemicals suitable for processing photographic materials. A " photographic grade " chemical is one which meets purity requirements as described.

This specification states the purity requirements and the test methods for photographic grade p-methylaminophenol sulphate ($HOC_6H_4NHCH_3 \cdot \frac{1}{2} H_2SO_4$).

2. PHYSICAL APPEARANCE

p-methylaminophenol sulphate is in the form of white crystalline powder.

3. SUMMARY OF REQUIREMENTS

Assay: 99.0 per cent minimum, 102.0 per cent maximum by cerate titration; 97.5 per cent minimum by acid titration.

Volatile matter at 105 °C: 0.3 per cent maximum.

Ash: 0.10 per cent maximum.

Heavy metals (as Pb): 0.002 per cent maximum.

Iron (Fe): 0.005 per cent maximum.

p-aminophenol: 2.5 per cent maximum.

p-amino-NN-dimethylaniline (sulphate): To pass test.

Matter soluble in diethyl ether: 0.2 per cent maximum.

Solubility: To pass test.

Identity: To pass melting-point test. The infra-red identity test may be used as a supplemental method.

4. ASSAY

(by cerate titration: 99.0 per cent minimum, 102.0 per cent maximum)
(by acid titration: 97.5 per cent minimum)

4.1 Cerate titration

4.1.1 *Reagent: Standard cerate solution.* Mix 52 ± 2 g of ammonium ceric nitrate * with 27 ml of sulphuric acid in a 600 ml beaker, with mechanical stirring. Cautiously add distilled water in 100 ml portions, 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 litre with distilled water and mix well.

4.1.1.1 *Standardization of cerate solution.* Place about 0.2 g of primary standard dry arsenic trioxide on a 25 mm diameter watch glass and weigh accurately. Transfer the watch glass and contents to a 250 ml conical flask. Add 15 ml of 10 per cent sodium hydroxide solution and warm the mixture gently. When solution is complete, cool to room temperature and add 25 ml of dilute sulphuric acid (1 + 5). Dilute to 100 ml with distilled water. As catalyst, add 0.15 ml of 0.01 M osmium tetroxide (prepared by dissolving 0.25 g of osmium tetroxide in 100 ml of approximately 0.1 N sulphuric acid) and add 1 drop of ferroin indicator.

CAUTION: Osmium tetroxide is poisonous—avoid contact.

Titrate the arsenic trioxide solution with the cerate solution to be standardized, until the reddish-orange colour changes to colourless or very pale blue. A sluggish end-point indicates insufficient osmium tetroxide; up to 0.7 ml may be required as the solution ages.

$$\frac{\text{mass of } As_2O_3 \times 1000}{\text{millilitres of cerate solution} \times 49.45} = \text{normality of cerate solution.}$$

4.1.2 *Procedure.* Place about 0.25 g of the sample on a 25 mm diameter watch glass and weigh accurately. Transfer the watch glass and sample to a 250 ml conical flask containing 100 ml of distilled water and 10 ml of approximately 0.1 N sulphuric acid. Dissolve the sample, add 3 drops of ferroin indicator and titrate with the standard cerate solution to a light-green colour which persists for 15 seconds.

$$1 \text{ ml } 0.1 \text{ N cerate} = 0.00861 \text{ g } HOC_6H_4NHCH_3 \cdot \tfrac{1}{2}\, H_2SO_4$$

NOTE.—This titration includes oxidizable material other than p-methylaminophenol sulphate.

4.2 Acid titration

Take about 0.6 g of the sample, weigh accurately and dissolve in 50 ml of distilled water. Add 90 ml of neutral acetone and titrate potentiometrically with 0.1 N sodium hydroxide, using glass and calomel electrodes. Plot the titration curve and determine the end-point.

$$1 \text{ ml } 0.1 \text{ N NaOH} = 0.01722 \text{ g } HOC_6H_4NHCH_3 \cdot \tfrac{1}{2}\, H_2SO_4$$

5. VOLATILE MATTER AT 105°C

(0.3 per cent maximum)

Place 5.0 ± 0.1 g of the sample in a low-form glass-stoppered weighing bottle and weigh accurately. Dry at 105 °C for 4 hours. Cool in a desiccator and weigh. The loss in mass should be not more than 0.015 g.

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* Reagents used in making the tests should be recognized reagent grade chemicals normally used for careful analytical work. In all the directions, the acids and ammonium hydroxide referred to should be of ull strength, unless dilution is specified. Dilution is specified in terms of normality, when standardization of the reagent is required. When dilution is indicated as $(1+x)$, it means 1 volume of the reagent or strong solution diluted with x volumes of distilled water.

6. ASH

(0.10 per cent maximum)

Incinerate 5 ± 0.1 g of the sample in a tared platinum crucible and then ignite the residue at 600 ± 25 °C for 4 hours. Cool in a desiccator and weigh. The residue mass should be not more than 0.005 g.

NOTE.—Save the residue for the heavy metals and iron tests.

7. HEAVY METALS (as Pb)

(0.002 per cent maximum)

Prepare a 25 ml heavy metals test control containing 0.10 mg of lead ion and a 25 ml iron test control containing a soluble iron salt equivalent to 0.25 mg of iron (see section 8). Dissolve the residue from the ash test (see section 6) in 0.5 ml of hydrochloric acid, warming if necessary, and transfer the solution (with washing) to a 100 ml beaker. Treat both test controls and the sample solution in the same manner. Add 2 drops of p-nitrophenol indicator (0.25 per cent aqueous solution) and then add dilute ammonium hydroxide (1 + 9), dropwise, until the solution turns yellow. Add dilute hydrochloric acid (1 + 99), dropwise, until the solution becomes colourless, and then add 2.5 ml excess. Dilute to 50 ml with distilled water. To 20 ml aliquots of both the heavy metals test control and the sample solution (save the iron test control and the balance of the sample solution for the iron test in clause 8.2), add 5 ml of hydrogen sulphide water, dilute to 50 ml with distilled water and mix well. Any colour produced in the sample solution should be not stronger than that produced in the heavy metals test control. Use Nessler tubes for comparison.

8. IRON (Fe)

(0.005 per cent maximum)

8.1 Reagents

8.1.1 *pH 5 acetate buffer.* Add 23 g of anhydrous sodium acetate to 58 ml of 2 M acetic acid and dilute to 1 litre with distilled water. Adjust the final pH of the solution to 5.0 ± 0.1 with glacial acetic acid or 10 per cent sodium hydroxide solution.

8.1.2 *o-phenanthroline (1,10-phenanthroline) mixture.* Thoroughly mix equal parts of 0.1 per cent o-phenanthroline (aqueous solution), 10 per cent hydroxylamine hydrochloride solution and pH 5 acetate buffer.

8.2 Procedure

To 20 ml aliquots of both the iron test control and the sample solution as prepared in section 7, add 5 ml of the o-phenanthroline mixture, mix well and let stand for 10 min. Dilute to 50 ml with distilled water and mix well. Any colour produced in the sample solution should be not stronger than that produced in the iron test control. Use Nessler tubes for comparison.

9. PARA-AMINOPHENOL ($HOC_6H_4NH_2$)

(2.5 per cent maximum)

Take 1.0 ± 0.05 g of the sample and dissolve in 100 ml of dilute hydrochloric acid (1 + 9). Stir and add slowly 25 ml of 10 per cent sodium nitrite solution. Continue stirring for about 1 min. Filter through a 60 ml medium-speed, fritted-disc Buchner funnel and collect the filtrate, *without washing*, in a suitable vacuum flask. Transfer the filtrate to a 250 ml volumetric flask and dilute to volume with distilled water. Pipet a 25 ml aliquot into a clean, dry, 125 ml separatory funnel (use a clean and dry separatory funnel for each extraction) and extract it three times with 25 ml portions of water-saturated, specially purified (suitable for spectrophotometric use) diethyl ether. Dilute a 10 ml aliquot of the aqueous solution to 250 ml with distilled water in a volumetric flask and mix well. Determine the absorbance at 312 μm using an ultraviolet spectrophotometer. Use distilled water as a blank. The sample solution should have an absorbance value of not more than 0.775.

10. p-AMINO-NN-DIMETHYLANILINE (SULPHATE)

(to pass test)

Add 10 ml of distilled water to 2 g of the sample and mix. Filter immediately, using a long-stem funnel. Collect the filtrate in a 100 ml separatory funnel. To the filtrate, add 10 ml of 40 per cent sodium hydroxide solution and 10 ml of diethyl ether. Shake the mixture and allow the two layers to separate. Draw off and discard the aqueous layer. Rinse the sides of the funnel with approximately 10 ml of distilled water without shaking. Draw off and discard the water. Then add 10 ml of dilute sulphuric acid (1 + 9) and shake the mixture. Draw off the acid layer into a glass-stoppered test tube. Shake the acid solution for 1 min and note the colour. No pink colour should be formed.

11. MATTER SOLUBLE IN DIETHYL ETHER

(0.2 per cent maximum)

Weigh 5 ± 0.1 g of the sample into an extraction thimble. Place the thimble and contents in a Soxhlet extraction apparatus and extract with anhydrous diethyl ether for 6 hours. Transfer the extract into a dried and tared 150 ml beaker, evaporate to dryness on a steam bath and dry at 70 °C for 30 min. Cool in a desiccator and weigh. The residue mass should be not more than 0.01 g.

12. SOLUBILITY

(to pass test)

Three grams of the sample should dissolve in 3 ml of hydrochloric acid to form a clear solution with not more than a slight yellow colour.