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Photographic grade sodium carbonate, monohydrate — Specification

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FOREWORD

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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.

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It has been approved by the Member Bodies of the following countries :

Australia	Italy	Spain
Austria	Japan	Switzerland
Belgium	Mexico	Turkey
Canada	Poland	United Kingdom
France	Romania	U.S.A.
Germany	South Africa, Rep. of	U.S.S.R.

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Photographic grade sodium carbonate, monohydrate — 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 sodium carbonate, monohydrate.

2 CHARACTERISTICS

Sodium carbonate, monohydrate, is in the form of white crystalline granules, of chemical formula $Na_2CO_3.H_2O$ and relative molar mass 124,0.

3 REQUIREMENTS

3.1 Assay

The assay shall be not less than 98,5 % (*m/m*), expressed as $Na_2CO_3.H_2O$, when determined by the method described in 4.1.

3.2 Appearance of solution

An aqueous solution shall be clear and free from sediment, other than a slight flocculence, when prepared and examined by the method described in 4.2.

3.3 Total halides content

The total halides content, expressed as sodium chloride (NaCl), shall be not greater than 0,5 % (*m/m*).

Conformity with this requirement shall be determined by the limit test described in 4.3, when the turbidity produced in the test solution shall be not greater than that produced in the control solution.

3.4 Bicarbonate content

The bicarbonate content, expressed as sodium bicarbonate, ($NaHCO_3$), shall be not greater than 0,6 % (*m/m*), when determined by the method described in 4.4.

3.5 Free alkali content

The free alkali content, expressed as sodium hydroxide (NaOH), shall be not greater than 0,17 % (*m/m*), when determined by the method described in 4.5.

3.6 Heavy metals content

The heavy metals content, expressed as lead (Pb), shall be not greater than 10 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.

3.7 Iron content

The iron content, expressed as iron (Fe), shall be not greater than 20 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.

3.8 Reaction to ammoniacal silver nitrate solution

The colour or turbidity produced in the test solution by ammoniacal silver nitrate solution shall be not greater than that produced in the control solution by ammonia solution, when examined by the method described in 4.8.

4 TEST METHODS

Reagents used in making 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 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 wherever water is required.

4.1 Assay

4.1.1 *Reagents*

4.1.1.1 Hydrochloric acid, 1 M standard volumetric solution.

4.1.1.2 Methyl orange indicator solution, 0,4 g/l.

4.1.2 *Apparatus*

Ordinary laboratory apparatus and

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

4.1.3 *Procedure*

Weigh, to the nearest 0,001 g, a test portion of about 2 g of the sample in a glass-stoppered weighing bottle. Wash into a 250 ml conical flask with about 50 ml of water, add 2 drops of methyl orange indicator solution (4.1.1.2) and titrate with the hydrochloric acid solution (4.1.1.1) until the colour changes.

1) 1 mol/l = 1 kmol/m^3 = 1 mol/dm^3 = 1 M

4.1.4 *Calculation*

The assay, expressed as a percentage by mass of sodium carbonate, monohydrate ($Na_2CO_3.H_2O$), is given by the formula

$$\frac{6{,}20\ VT}{m}$$

where

V is the volume, in millilitres, of the hydrochloric acid solution (4.1.1.1) used for the titration;

T is the exact molarity of the hydrochloric acid solution (4.1.1.1);

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

4.2 Appearance of solution test

Prepare a 235 g/l solution of the sample in water and examine for clarity and sediment.

4.3 Limit test for halides

4.3.1 *Reagents*

4.3.1.1 **Nitric acid** solution, dilute (1 + 9).

4.3.1.2 **Silver nitrate** solution, 100 g/l.

4.3.1.3 **Chloride,** standard solution.

Dissolve 20 mg of sodium chloride in 1 000 ml of water.

4.3.2 *Apparatus*

Ordinary laboratory apparatus and

4.3.2.1 **One-mark volumetric flask,** 100 ml capacity, conforming to class A of ISO 1042.

4.3.2.2 **Two matched Nessler cylinders,** 50 ml capacity.

4.3.3 *Procedure*

Weigh, to the nearest 0,1 g, a test portion of 1 g of the sample, dissolve in water and make up to 100 ml in the volumetric flask (4.3.2.1). Take a 2 ml aliquot and add 10 ml of the nitric acid solution (4.3.1.1), then add 1 ml of the silver nitrate solution (4.3.1.2), dilute to 50 ml and mix well. Treat 5 ml of the standard chloride solution (4.3.1.3) in a similar manner, and compare, in the Nessler cylinders (4.3.2.2), the turbidities in the test and control solutions.

4.4 Determination of bicarbonate content

4.4.1 *Reagents*

4.4.1.1 **Sodium hydroxide,** approximately 0,1 M standard volumetric solution.

4.4.1.2 **Barium chloride,** 1 M neutral solution.

Dissolve 244 g of barium chloride dihydrate ($BaCl_2.2H_2O$) in 1 000 ml of carbon dioxide-free water. Check that the solution is neutral to phenolphthalein indicator (4.4.1.3). If not, adjust with a few drops of the 0,1 M sodium hydroxide solution (4.4.1.1).

4.4.1.3 **Phenolphthalein indicator,** ethanol/water solution, 5 g/l.

Dissolve 5 g of phenolphthalein in 500 ml of ethanol and add 500 ml of water, with constant stirring. Filter if necessary.

4.4.2 *Apparatus*

Ordinary laboratory apparatus and

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

4.4.3 *Procedure*

Weigh, to the nearest 0,1 g, a test portion of 2 g of the sample into a 125 ml conical flask. Add 30 ml of freshly boiled water, stopper and dissolve the sample. Then add 25 ml of the barium chloride solution (4.4.1.2), 10 drops of the phenolphthalein indicator solution (4.4.1.3), stopper and swirl. If the solution has a pink colour, proceed as stated in 4.5.3. If colourless, titrate with the sodium hydroxide solution (4.4.1.1) until the first appearance of a faint pink colour which persists for 30 s.

4.4.4 *Calculation*

The bicarbonate content, expressed as a percentage by mass of sodium bicarbonate ($NaHCO_3$), is given by the formula

$$4{,}2\ VT$$

where

V is the volume, in millilitres, of the sodium hydroxide solution (4.4.1.1) used for the titration;

T is the exact molarity of the sodium hydroxide solution (4.4.1.1).

4.5 Determination of free alkali content

4.5.1 *Reagent*

4.5.1.1 **Hydrochloric acid,** approximately 0,1 M standard volumetric solution.

4.5.2 *Apparatus*

Ordinary laboratory apparatus and

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

4.5.3 *Procedure*

If the solution has a pink colour after the addition of the indicator under 4.4.3, titrate with the hydrochloric acid solution (4.5.1.1) until the pink colour is discharged. If the solution was not initially pink, then the free alkali is zero.

4.5.4 *Calculation*

The free alkali content, expressed as a percentage by mass of sodium hydroxide (NaOH), is given by the formula

$$2\,VT$$

where

V is the volume, in millilitres, of the hydrochloric acid solution (4.5.1.1) used for the titration;

T is the exact molarity of the hydrochloric acid solution (4.5.1.1).

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.

4.6.2 *Apparatus*

Ordinary laboratory apparatus and

4.6.2.1 Two matched Nessler cylinders, 50 ml capacity.

4.6.3 *Procedure*

Weigh, to the nearest 0,1 g, a test portion of about 5 g of the sample and dissolve in 15 ml of water. Also take 5 ml of the standard heavy metals solution (4.6.1.4) and treat this and the test solution in the following manner. Add 10 ml of the hydrochloric acid solution (4.6.1.1) and evaporate to dryness on a steam-bath. Take up the residue in 5 ml of the dilute hydrochloric acid solution (4.6.1.2) and then add 25 ml of water.

To each, add 1 drop of the *p*-nitrophenol indicator solution (4.6.1.6) and then add the ammonia solution (4.6.1.3), drop by drop, until the solutions turn yellow. Add the dilute hydrochloric acid solution (4.6.1.2), drop by drop, until the solutions become colourless and add 0,5 ml in excess. Dilute each to 50 ml with water.

Transfer 20 ml aliquots of each solution to separate Nessler cylinders (4.6.2.1), retaining the balance of the test solution for the iron test under 4.7.3. Finally, 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 under 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 solution and dilute to 1 000 ml with water. Adjust the final pH of the solution to 5,0 ± 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 volumes 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 way as the standard heavy metals solution under 4.6.3 down to the first dilution to 50 ml. Transfer 20 ml of this solution to one Nessler cylinder (4.7.2.1) and 20 ml of the sample solution (retained from 4.6.3) to the other Nessler cylinder. Add 5 ml of the 1,10-phenanthroline reagent solution (4.7.1.3) to each, mix well and allow to stand for 10 min. Then dilute each to 50 ml and mix well.

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

4.8 Reaction to ammoniacal silver nitrate test

4.8.1 *Reagent*

4.8.1.1 Silver nitrate, ammoniacal solution.

Immediately before use mix equal volumes of ammonia solution, ρ approximately 0,910 g/ml, and 100 g/l silver nitrate solution.

4.8.2 *Apparatus*

Ordinary laboratory apparatus and

4.8.2.1 Two matched Nessler cylinders, 50 ml capacity.

4.8.3 *Procedure*

Weigh, to the nearest 0,1 g, a test portion of about 2 g of the laboratory sample and dissolve in 40 ml of water. Divide this volume equally between the two Nessler cylinders (4.8.2.1). To one, the test solution, add 10 ml of the freshly prepared ammoniacal silver nitrate solution (4.8.1.1) and mix well. To the other, the control solution, add 5 ml of ammonia solution, ρ approximately 0,910 g/ml, and 5 ml of water and mix well. Allow each to stand for 2 min.

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

CAUTION : Dispose of all test solutions and rinse apparatus used immediately. Explosive compounds may be formed on standing.