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# INTERNATIONAL STANDARD



# 3943

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## Photographic grade sodium acetate, anhydrous — Specification

*Acétate de sodium anhydre de qualité photographique — Spécifications*

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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.

International Standard ISO 3943 was drawn up by Technical Committee ISO/TC 42, *Photography*, and was circulated to the Member Bodies in August 1975.

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

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

The Member Body of the following country expressed disapproval of the document on technical grounds :

Japan

# Photographic grade sodium acetate, anhydrous — 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 acetate, anhydrous.

## 2 CHARACTERISTICS

Sodium acetate, anhydrous, is in the form of a white powder or grey-white flakes, of chemical formula  $\text{NaC}_2\text{H}_3\text{O}_2$  and relative molar mass 82,0.

## 3 REQUIREMENTS

### 3.1 Assay

The assay shall be not less than 98,5 % (*m/m*), expressed as  $\text{NaC}_2\text{H}_3\text{O}_2$ , when determined by the method described in 4.1.

### 3.2 Appearance of solution

An aqueous solution shall be clear and colourless 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 ( $\text{NaCl}$ ), shall be not greater than 0,3 % (*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 Acidity

The acidity, expressed as acetic acid ( $\text{C}_2\text{H}_4\text{O}_2$ ), shall be not greater than 1,0 % (*m/m*), when determined by the method described in 4.4.

### 3.5 Free alkali content

The free alkali content, expressed as sodium hydroxide ( $\text{NaOH}$ ), shall be not greater than 0,04 % (*m/m*), when determined by the method described in 4.5.

### 3.6 Calcium, magnesium and other matter insoluble in ammonia solution

The matter insoluble in ammonia solution shall be not greater than 0,5 % (*m/m*), when determined by the method described in 4.6.

### 3.7 Heavy metals content

The heavy metals content, expressed as lead ( $\text{Pb}$ ), 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.

### 3.8 Iron content

The iron content, expressed as iron ( $\text{Fe}$ ), shall be not greater than 50 mg/kg.

Conformity with this requirement shall be determined by the limit test described in 4.8, when the colour produced in the test solution shall be not greater than that produced in the control solution.

## 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)<sup>1)</sup> 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 Acetous perchloric acid**, approximately 0,1 M standard volumetric solution.

Add 10 ml of 70-72 % (*m/m*) perchloric acid solution to approximately 900 ml of acetic acid in a 1 l flask with constant swirling.

**(CAUTION : Concentrated solutions of perchloric acid are powerful oxidants and explosions may be caused when placed in contact with some organic materials.)**

Add 25 ml of acetic anhydride and dilute to 1 000 ml with acetic acid. Allow to stand for 24 h. Standardize potentiometrically against a standard sample of potassium hydrogen phthalate using a glass electrode and a calomel electrode. (The temperatures of titrant during standardization and assay should be within 0,5 °C of each other.)

**4.1.1.2 Acetone**, dried over calcium chloride.

#### 4.1.2 Apparatus

Ordinary laboratory apparatus and

**4.1.2.1 Potentiometer/pH meter**, complete with glass and calomel electrodes.

1) 1 mol/l = 1 kmol/m<sup>3</sup> = 1 mol/dm<sup>3</sup> = 1 M

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

#### 4.1.3 Procedure

Weigh, to the nearest 0,001 g, a test portion of about 0,25 g of the sample. Transfer to a dry 250 ml beaker and add 10 ml of acetic acid. Dissolve the sample then add 90 ml of the dry acetone (4.1.1.2). Titrate potentiometrically with the 0,1 M acetous perchloric acid solution, in the same manner as in the standardization (4.1.1.1). In the region of the end-point, add titrant in 0,1 ml portions.

NOTE – Do not add any acetic acid for rinsing, etc.

#### 4.1.4 Calculation

The assay, expressed as a percentage by mass of sodium acetate, anhydrous ( $\text{NaC}_2\text{H}_3\text{O}_2$ ), is given by the formula

$$\frac{8,20 VT}{m}$$

where

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

$T$  is the exact molarity of the acetous perchloric acid solution (4.1.1.1);

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

#### 4.2 Appearance of solution test

Prepare a 100 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**, 100 g/l solution.

**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 10 ml aliquot and add

5 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 of the test and control solutions.

#### 4.4 Determination of acidity

##### 4.4.1 Reagents

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

**4.4.1.2 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, 3 g of the sample and dissolve in 50 ml of carbon dioxide-free water. Add 3 drops of the phenolphthalein indicator solution (4.4.1.2). If no pink colour is produced, titrate with the 1 M sodium hydroxide solution (4.4.1.1) until the appearance of a faint pink colour. If a pink colour was produced initially, pass directly to 4.5.

##### 4.4.4 Calculation

The acidity, expressed as a percentage by mass of acetic acid ( $\text{C}_2\text{H}_4\text{O}_2$ ), is given by the formula

$$\frac{6,0 VT}{m}$$

where

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

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

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

#### 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 in 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

$$\frac{4,0 VT}{m}$$

where

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

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

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

4.6 Determination of calcium, magnesium and other matter insoluble in ammonia solution

4.6.1 Reagents

4.6.1.1 Ammonium oxalate solution, 40 g/l.

4.6.1.2 Diammonium hydrogen orthophosphate solution, 100 g/l.

4.6.1.3 Ammonia solution, dilute (1 + 9).

4.6.1.4 Ammonia solution, dilute (1 + 39).

4.6.2 Apparatus

Ordinary laboratory apparatus and

4.6.2.1 Crucible.

4.6.2.2 Muffle furnace, capable of being controlled at  $600 \pm 50$  °C.

4.6.3 Procedure

Weigh, to the nearest 0,1 g, a test portion of about 10 g of the sample and dissolve in 75 ml of water. Add 10 ml of the ammonium oxalate solution (4.6.1.1) and 4 ml of the diammonium hydrogen orthophosphate solution (4.6.1.2) followed by 20 ml of the ammonia solution (4.6.1.3).

Allow to stand overnight. If any precipitate is formed, filter through an ashless filter paper and wash the precipitate with the ammonia solution (4.6.1.4). Transfer the filter paper with the precipitate to a weighed crucible (4.6.2.1), dry and ignite the residue in the furnace (4.6.2.2), controlled at  $600 \pm 50$  °C, for 4 h. Cool in a desiccator and weigh to the nearest 1 mg.

4.6.4 Calculation

Matter insoluble in ammonia solution is given, as a percentage by mass, by the formula

$$\frac{100 (m_2 - m_1)}{m_0}$$

where

*m*<sub>0</sub> is the mass, in grams, of the test portion;

*m*<sub>1</sub> is the mass, in grams, of the crucible;

*m*<sub>2</sub> is the mass, in grams, of the crucible plus residue.

4.7 Limit test for heavy metals

4.7.1 Reagents

4.7.1.1 Hydrochloric acid solution,  $\rho$  approximately 1,18 g/ml.

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

4.7.1.3 Ammonia solution, dilute (1 + 9).

4.7.1.4 Heavy metals, standard solution.

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

4.7.1.5 Water, saturated at room temperature with hydrogen sulphide.

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

4.7.2 Apparatus

Ordinary laboratory apparatus and

4.7.2.1 Two matched Nessler cylinders, 50 ml capacity.

4.7.3 Procedure

Weigh, to the nearest 0,1 g, a test portion of about 5 g of the sample and dissolve in 25 ml of water. Also take 25 ml of the standard heavy metals solution (4.7.1.4) and treat this and the test solution in the following manner. Add 15 ml of the hydrochloric acid solution (4.7.1.1) and evaporate to dryness on a steam-bath. Take up the residue in 25 ml of water. To each, add 2 drops of the *p*-nitrophenol indicator solution (4.7.1.6), and then add the ammonia solution (4.7.1.3) drop by drop until the solutions, turn yellow. Add the diluted hydrochloric acid

solution (4.7.1.2) until the solutions become colourless and add 2,5 ml excess. Warm for 10 min on a steam-bath, cool and dilute to 50 ml with water. Transfer 20 ml aliquots of each solution to separate Nessler cylinders (4.7.2.1), retaining the balance of the test solution for the iron test under 4.8.3. Finally, add 5 ml of the hydrogen sulphide water (4.7.1.5), dilute 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 iron

##### 4.8.1 Reagents

As specified under 4.7.1 and

##### 4.8.1.1 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  $5,0 \pm 0,1$  with glacial acetic acid or 100 g/l sodium hydroxide solution.

##### 4.8.1.2 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.

##### 4.8.1.3 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 (4.8.1.1).

##### 4.8.2 Apparatus

Ordinary laboratory apparatus and

##### 4.8.2.1 Two matched Nessler cylinders, 50 ml capacity.

##### 4.8.3 Procedure

Take 25 ml of the standard iron solution (4.8.1.2) and treat in the same way as the standard heavy metals solution under 4.7.3 down to the first dilution to 50 ml. Transfer 20 ml of this solution to one Nessler cylinder (4.8.2.1), and 20 ml of the sample solution (retained from 4.7.3) to the other Nessler cylinder. Add 5 ml of the 1,10-phenanthroline reagent solution (4.8.1.3) 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.

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