INTERNATIONAL STANDARD

ISO 3617

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Photography — Processing chemicals — Specifications for sodium hydroxide

Photographie — Produits chimiques de traitement — Spécifications pour l'hydroxyde de sodium

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established 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. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

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 3617 was prepared by Technical Committee ISO/TC 42, *Photography*.

This second edition cancels and replaces the first edition (ISO 3617:1976), which has been technically revised.

Annex A of this International Standard is for information only.

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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, 5, 6 and 7 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 sodium hydroxide

1 Scope

This International Standard establishes criteria for the purity of photographic-grade sodium hydroxide (DANGER: <<C>>)[1] and specifies the tests to be used to determine the purity.

2 Normative references

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

ISO 10349-5:1992, *Photography — Photographic-grade chemicals — Test methods — Part 5: Determination of heavy metals and iron content.*

ISO 10349-6:1992, *Photography — Photographic-grade chemicals — Test methods — Part 6: Determination of halide content.*

ISO 10349-7:1992, *Photography — Photographic-grade chemicals — Test methods — Part 7: Determination of alkalinity or acidity.*

3 General

3.1 Physical properties

Sodium hydroxide, NaOH, exists in the form of white sticks, pellets, flakes, granules or powder. It has a relative molecular mass of 40,00.

3.2 Hazardous properties

Sodium hydroxide is corrosive (DANGER:<<C>>). Avoid contact with eyes, skin and clothing. Avoid breathing dust. Refer to the manufacturer's Material Safety Data Sheet (MSDS) for additional information.

3.3 Handling and storage

Sodium hydroxide shall be stored in a properly labelled and tightly sealed plastic container. Sodium hydroxide readily absorbs moisture with the liberation of heat and reacts violently with acids.

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.

7 Test methods

7.1 Assay

7.1.1 Specification

Content of NaOH shall be 95,0 % (m/m) min.

1) Hazard warning codes are defined in ISO 10349-1:1992, clause 4.

Table 1 — Summary of requirements

Test	Limit	Subclause	International Standard in which test method is given
Assay (as NaOH)	95,0 % (*m/m*) min.	7.1	ISO 3617
Heavy metals (as Pb)	0,003 % (*m/m*) max.	7.2	ISO 10349-5
Iron (Fe)	0,002 % (*m/m*) max.	7.3	ISO 10349-5
Halides (as Cl^-)	0,3 % (*m/m*) max.	7.4	ISO 10349-6
Carbonate (as Na_2CO_3)	2,5 % (*m/m*) max.	7.5	ISO 3617
Calcium and magnesium (as Mg)	0,06 % (*m/m*) max.	7.6	ISO 3617
Appearance of solution	Clear and free from insoluble matter except for a slight flocculence	7.7	ISO 3617
NOTE — *m/m* = mass/mass			

7.1.2 Reagents

7.1.2.1 Hydrochloric acid, HCl, standard volumetric solution of 1,0 mol/l (36,46 g/l)[2) 3)].

7.1.2.2 Phenolphthalein indicator, 5 g/l.

Dissolve 0,5 g of phenolphthalein in 50 ml of methanol or ethanol, then dilute to 100 ml with water. Filter if necessary.

7.1.2.3 Carbon-dioxide-free water.

Prepare carbon-dioxide-free water in accordance with ISO 10349-7.

7.1.2.4 Sodium hydroxide, NaOH, standard volumetric solution of 0,10 mol/l (4,0 g/l)[2) 4)].

Use analytical grade sodium hydroxide for this solution, not the material under test.

7.1.2.5 Barium chloride, $BaCl_2$, neutral solution, 100 g/l.

Dissolve 100 g of barium chloride dihydrate ($BaCl_2 \cdot 2H_2O$) in 1 litre of carbon-dioxide-free water (7.1.2.3). Check that the solution is neutral to the phenolphthalein indicator (7.1.2.2). If not, adjust with a few drops of sodium hydroxide (7.1.2.4).

7.1.3 Apparatus

7.1.3.1 One-mark volumetric flask, of 500 ml capacity.

7.1.3.2 Burette, of 50 ml capacity.

7.1.3.3 Pipette, of 50 ml capacity.

7.1.4 Procedure

Weigh, to the nearest 0,01 g, a test portion of 19 g to 21 g and transfer it to a beaker containing 250 ml of carbon-dioxide-free water (7.1.2.3). Allow to cool to room temperature. Analytically transfer the contents to a 500 ml volumetric flask (7.1.3.1) and then dilute to the mark with the water (7.1.2.3). Transfer a 50 ml aliquot of the sample solution, using the pipette (7.1.3.3), to a 500 ml glass stoppered conical flask and dilute to about 200 ml with the water (7.1.2.3). Add 5 ml of barium chloride (7.1.2.5). Stopper the flask and shake. Allow the mixture to stand for 5 min. Add three drops of phenolphthalein indicator (7.1.2.2) and titrate with hydrochloric acid (7.1.2.1) to the first disappearance of the pink colour. Retain this solution for use in the carbonate test (7.5).

7.1.5 Expression of results

The assay, expressed as a percentage by mass of NaOH, is given by

$$40{,}00 \cdot c \cdot V/m$$

where

c is the actual concentration, in moles per litre, of the hydrochloric acid (7.1.2.1);

V is the volume, in millilitres, of the hydrochloric acid used to reach the titration endpoint (7.1.4);

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

2) Commercially available analysed reagent is recommended. If solutions are to be prepared, see any quantitative analytical chemistry text.

3) This solution can be prepared from concentrated hydrochloric acid, $\rho \approx 1{,}18$ g/ml (DANGER: <C><B>).

4) This solution can be prepared from solid sodium hydroxide (DANGER: <<C>>).

40,00 is the conversion factor obtained from the mass of sodium hydroxide equivalent to 1 mole of hydrochloric acid (i.e. 40,0) × the conversion factor for millilitres to litres (i.e. 0,001) × the sampling factor (i.e. 10) × 100 (for percentage).

7.2 Heavy metals content

7.2.1 Specification

Maximum content of heavy metals shall be 0,003 % (*m/m*).

7.2.2 Procedure

NOTE 1 The standard for the iron test (7.3) 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 0,90 g to 1,10 g prepared in accordance with ISO 10349-5:1992, 7.3. Use 3 ml of the heavy metals standard prepared in accordance with ISO 10349-5: 1992, 8.1.2.

7.3 Iron content

7.3.1 Specification

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

7.3.2 Procedure

Determine the percentage of iron in accordance with ISO 10349-5. Use a test portion of 0,90 g to 1,10 g of the sample prepared in accordance with ISO 10349-5: 1992, 7.3. Use 2 ml of the iron standard prepared in accordance with ISO 10349-5:1992, 8.1.2.

7.4 Halides content (as Cl^-)

7.4.1 Specification

Maximum content of halides shall be 0,3 % (*m/m*) as Cl^-.

7.4.2 Procedure

Determine the percentage of halides (expressed as Cl^-) in accordance with ISO 10349-6. Use a 10 ml aliquot of the solution and 15 ml of the Halide A standard.

7.5 Carbonate content (as Na_2CO_3)

7.5.1 Specification

Maximum content of carbonate shall be 2,5 % (*m/m*), as Na_2CO_3.

7.5.2 Reagents

7.5.2.1 Hydrochloric acid, HCl, standard volumetric solution of 0,1 mol/l, (3,646 g/l)[2)5)].

7.5.2.2 Methyl orange indicator.

Dissolve 0,1 g of methyl orange indicator in 250 ml of water.

7.5.3 Apparatus

7.5.3.1 Burette, of 50 ml capacity.

7.5.4 Procedure

Add two drops of methyl orange indicator (7.5.2.2) to the solution from the assay titration in 7.1.4 and continue the titration with hydrochloric acid (7.5.2.1) to a permanent pink colour. The additional acid used represents carbonate.

7.5.5 Expression of results

The carbonate content, as Na_2CO_3, expressed as a percentage by mass of NaOH, is given by

$$52{,}99 \cdot c' \cdot V' / m'$$

where

c' is the actual concentration, in moles per litre, of the hydrochloric acid (7.5.2.1);

V' is the volume, in millilitres, of the hydrochloric acid used to reach the titration endpoint (7.5.4);

m' is the mass, in grams, of the test portion;

52,99 is the conversion factor obtained from the mass of sodium carbonate equivalent to 1 mole of hydrochloric acid (i.e. 52,99) × the conversion factor for millilitres to litres (i.e. 0,001) × the sampling factor (i.e. 10) × 100 (for percentage).

5) This can be prepared by diluting 100 ml of hydrochloric acid (7.1.2.1) to 1 litre with carbon-dioxide-free water.

7.6 Calcium and magnesium content (as Mg)

7.6.1 Specification

Maximum calcium and magnesium content shall be 0,06 % (*m*/*m*) as Mg.

7.6.2 Reagents

7.6.2.1 Ammonium hydroxide, NH_4OH, $\rho \approx 0{,}91$ g/ml (DANGER: <C><B>).

7.6.2.2 Buffer solution, pH 9,5 to 10,0.

Dissolve 54 g of ammonium chloride (NH_4Cl) in 200 ml of water. Add 350 ml of ammonium hydroxide (7.6.2.1) (<C><B>) and dilute to 1 litre.

7.6.2.3 EDTA solution, standard volumetric solution of 0,01 mol/l (3,36 g/l of the disodium salt, $C_{10}H_{14}Na_2N_2O_8$)[2) 6)].

7.6.2.4 Hydrochloric acid, HCl, $\rho \approx 1{,}18$ g/ml (DANGER: <C><B>).

7.6.2.5 Magnesium standard solution (1 ml contains 1 mg Mg).

Dissolve 10,141 g of magnesium sulfate heptahydrate ($MgSO_4 \cdot 7H_2O$) in water containing 1 ml of hydrochloric acid (7.6.2.4) (<C><B>) in a 1 litre volumetric flask (7.6.3.1). Dilute to the mark and mix.

7.6.2.6 Mordant black indicator.

Grind 0,25 g of Mordant black 11[7)] with 25 g of sodium chloride (NaCl) in a mortar.

7.6.3 Apparatus

7.6.3.1 One-mark volumetric flask, of 1 litre capacity.

7.6.3.2 Burette, of 50 ml capacity.

7.6.4 Procedure

Weigh, to the nearest 0,01 g, a test portion of about 2 g and dissolve it in 75 ml of water. Using litmus paper, neutralize the solution with hydrochloric acid (7.6.2.4) (<C><B>), boil for 5 min and cool. Prepare a ten-fold dilution (1 + 9) of a portion of the magnesium standard solution (7.6.2.5) and add 1 ml of this diluted solution, followed by 5 ml of the buffer solution (7.6.2.2) and 0,1 g of the Mordant black 11 indicator (7.6.2.6). Titrate with the EDTA solution (7.6.2.3) to the colour change from violet-red to blue. Carry out a similar titration on a blank solution which is treated the same way except for the addition of the test portion.

7.6.5 Expression of results

The calcium and magnesium content, as Mg, expressed as a percentage by mass of the NaOH, is given by

$$0{,}024\ 3(V_1 - V_2)/m$$

where

V_1 is the volume, in millilitres, of the EDTA solution (7.6.2.3) used to reach the titration endpoint of the test solution;

V_2 is the volume, in millilitres, of the EDTA solution (7.6.2.3) used to reach the titration endpoint of the blank;

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

0,024 3 is the conversion factor obtained from the mass of magnesium equivalent to 1 mole of EDTA (i.e. 24,3) × the concentration of the EDTA solution (i.e. 0,01) × the conversion factor for millilitres to litres (i.e. 0,001) × 100 (for percentage).

7.7 Appearance of solution

7.7.1 Specification

The solution shall be clear and free from insoluble matter except for a slight flocculence.

7.7.2 Procedure

Dissolve a test portion of 10,0 g in 50 ml of water and dilute to 100 ml with water. Observe the solution for colour and clarity.

6) A procedure for the preparation and standardization of EDTA solution is given in annex A.

7) Listed in colour index as C.I. 14645. Chrome Fast Black CAT, KIT*TS, Eriochrome Black DW, T and TDW, and Potting Black C are examples of suitable products available commercially. This information is given for the convenience of users of this International Standard and does not constitute an endorsement by ISO of these products.

Annex A
(informative)

Preparation of EDTA solution: Standard volumetric solution of 0,01 mol/l (3,36 g/l)

A.1 Reagents

A.1.1 Ethylenediaminetetraacetic acid (EDTA) dihydrate, disodium salt, $C_{10}H_{14}N_2Na_2O_8{\cdot}2H_2O$.

NOTE 2 The relative molecular mass for the dihydrate salt is 372,23. The relative molecular mass for the non-hydrated salt is 336,20.

A.1.2 Calcium carbonate, $CaCO_3$, chelometric standard grade material[8].

A.1.3 Hydrochloric acid, HCl (1 + 3)[9].

A.1.4 Sodium hydroxide, NaOH, standard volumetric solution of 1 mol/l (40,0 g/l)[10].

Use analytical grade sodium hydroxide for this solution, not the material under test.

A.1.5 Hydroxynaphthol blue indicator.

A.2 Apparatus

A.2.1 Burette, of 50 ml capacity.

A.2.2 Pipette, of 2 ml capacity.

A.3 Procedure

Dissolve 20 g of disodium EDTA dihydrate (A.1.1) in water and dilute to 1 litre.

Weigh, to the nearest 0,000 1 g, about 0,050 g of the calcium carbonate (A.1.2). Transfer the calcium carbonate to a 400 ml beaker and add 10 ml of water. Cover the beaker with a watch glass and add 2 ml of hydrochloric acid (A.1.3) from a pipette (A.2.2) inserted between the watch glass and the lip of the beaker. Swirl the beaker to assist dissolution of the calcium carbonate. Wash down the sides of the beaker, the watch glass and the outside of the pipette and dilute the solution to about 100 ml with water. While stirring with a magnetic stirrer, add about 40 ml of the prepared EDTA solution using the burette (A.2.1), followed by 15 ml of sodium hydroxide (A.1.4) and 0,300 g of hydroxynaphthol blue indicator (A.1.5). Continue the titration with the EDTA solution to the endpoint when a blue colour appears.

A.4 Expression of results

The actual concentration of the EDTA solution, c, in moles per litre, is given by the formula

$$c = m/(0{,}100\ 09 \cdot V)$$

where

m is the mass, in grams, of the calcium carbonate (A.1.2);

V is the volume, in millilitres, of the prepared EDTA solution (A.1.1);

0,100 09 is the relative molecular mass of calcium carbonate (i.e. 100,09) × the conversion factor for millilitres to litres (i.e. 0,001).

Adjust the concentration of the EDTA solution to exactly 0,01 mol/l by diluting with water. The volume of water required, in millilitres, V_W, is given by the formula

$$V_W = (c \cdot S/0{,}01) - S$$

where

c is the actual concentration of the prepared EDTA solution, in moles per litre;

S is the volume, in millilitres, of the prepared EDTA solution to be diluted.

8) It is recommended that the calcium carbonate be dried to constant mass in a low-temperature oven and maintained in a desiccator to prevent absorption of water.

9) This solution can be prepared from concentrated hydrochloric acid, $\rho \approx 1{,}18$ g/ml (DANGER: <C><B>).

10) This solution can be prepared from solid sodium hydroxide (DANGER: <<C>>).