INTERNATIONAL STANDARD

ISO 6853

Second edition
2001-12-15

Photography — Processing waste — Determination of ammoniacal nitrogen (microdiffusion method)

Photographie — Effluents de traitement — Détermination de l'azote ammoniacal (méthode par microdiffusion)

Reference number
ISO 6853:2001(E)

ISO copyright office
Case postale 56 • CH-1211 Geneva 20
Tel. + 41 22 749 01 11
Fax + 41 22 749 09 47
E-mail copyright@iso.ch
Web www.iso.ch

Printed in Switzerland

Contents

Page

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.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 3.

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.

Attention is drawn to the possibility that some of the elements of this International Standard may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

International Standard ISO 6853 was prepared by Technical Committee ISO/TC 42, *Photography*.

This second edition cancels and replaces the first edition (ISO 6853:1987), of which it constitutes a technical revision.

Introduction

This International Standard is one of a series devoted to the analysis of photographic wastes; it encompasses the field of analysis of the ammoniacal nitrogen content in a photographic effluent.

This International Standard is intended for use by individuals with a working knowledge of analytical techniques. Some of the procedures use caustic, toxic or otherwise hazardous chemicals. Safe laboratory practice for the handling of chemicals requires the use of safety glasses or goggles and, in some cases, other protective apparel such as rubber gloves, face masks or aprons. Normal precautions for the safe performance of any chemical procedure must be exercised at all times, but specific details have been provided for hazardous materials. Hazard warnings are designated by a letter enclosed in angle brackets "〈 〉." These are defined in clause 5 and then used throughout the text. More detailed information on hazards, handling and use of these chemicals may be available from the manufacturer.

Photographic laboratories can establish conformity to effluent regulations only by chemical analysis. If this cannot be done in-house, an outside laboratory should be used.

Photography — Processing waste — Determination of ammoniacal nitrogen (microdiffusion method)

1 Scope

This International Standard specifies a method for the determination of ammonia and other volatile amines that can be liberated from photographic processing wastes by strong alkali, the results being expressed in terms of nitrogen.

The method is applicable for the determination of the ammonia concentration of typical photoprocessing wastes in the range of 10 mg/l to 200 mg/l of ammonia or 8 mg/l to 160 mg/l of nitrogen. Other volatile amines are determined as ammonia, but their concentrations in photoprocessing wastes are usually very low.

2 Normative references

The following normative documents contain provisions which, through reference in this text, constitute provisions of this International Standard. For dated references, subsequent amendments to, or revisions of, any of these publications do not apply. However, parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the normative documents indicated below. For undated references, the latest edition of the normative document referred to applies. Members of ISO and IEC maintain registers of currently valid International Standards.

ISO 648:1977, *Laboratory glassware — One-mark pipettes*.

ISO 5667-1:1980, *Water quality — Sampling — Part 1: Guidance on the design of sampling programmes*.

ISO 5667-2:1991, *Water quality — Sampling — Part 2: Guidance on sampling techniques*.

ISO 5667-3:1994, *Water quality — Sampling — Part 3: Guidance on the preservation and handling of samples*.

ISO 6353-1:1982, *Reagents for chemical analysis — Part 1: General test methods*.

ISO 6353-2:1983, *Reagents for chemical analysis — Part 2: Specifications — First series*.

ISO 6353-3:1987, *Reagents for chemical analysis — Part 3: Specifications — Second series*.

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

3 Principle

Ammonia is liberated from the sample by treatment with potassium metaborate and is absorbed into boric acid. The absorbed ammonia is then determined by titration with standard sulfuric acid. The liberation and absorption are carried out in a microdiffusion cell, which is a small covered dish with concentric chambers for sample, sealant and absorbing solution. When the sample and the metaborate are mixed in the sample chamber, ammonia is evolved and absorbed into the boric acid chamber. The gaseous free path from sample to absorbent is short, in order to ensure a room temperature distillation in a reasonable time. Scale-up of this method or alteration of the sample to buffer ratio should be avoided, as these modifications change the rate of ammonia diffusion.

4 Reaction

$$NH_4X + OH^- \rightarrow NH_3 + H_2O + X^-$$

5 Safety and operational precautions

5.1 Hazard warnings

Some of the chemicals specified in the test procedures are caustic, toxic or otherwise hazardous. Safe laboratory practice for the handling of chemicals requires the use of safety glasses or goggles, and in some cases other protective apparel such as rubber gloves, face masks and aprons. Specific danger notices are given in the text for particularly dangerous materials, but normal precautions are required during the performance of any chemical procedure at all times.

The first time that a hazardous material is noted in the procedures, the hazard will be indicated by the word "**DANGER**" followed by a symbol consisting of angle brackets "〈 〉" containing a letter that designates the specific hazard. A double bracket "〈〈 〉〉" will be used for particularly perilous situations. In subsequent statements involving handling of these hazardous materials, only the hazard symbol consisting of the brackets and letter(s) will be displayed. Furthermore, for a given material, the hazard symbols will be used only once in a single paragraph.

Hazard warning symbols will not be used for common organic solvents when used in quantities of less than 1 litre, unless they are particularly hazardous.

Detailed warnings for handling chemicals and their diluted solutions are beyond the scope of this International Standard.

Employers shall provide training and health and safety information in conformance with legal requirements.

The hazard code system used in this International Standard is intended to provide information to the users and is not meant for compliance with any legal requirements for labelling, as these vary from country to country.

It is strongly recommended that anyone using these chemicals obtain pertinent information from the manufacturer about the hazards, handling, use and disposal of these chemicals.

5.2 Hazard information code system

〈B〉 Harmful if inhaled. Avoid breathing dust, vapour, mist or gas. Use only with adequate ventilation.

〈C〉 Harmful if contact occurs. Avoid contact with eyes, skin or clothing. Wash thoroughly after handling.

〈F〉 Will burn. Keep away from heat, sparks and open flame. Use with adequate ventilation.

〈O〉 Oxidizer. Contact with other material may cause fire. Do not store near combustible materials.

〈S〉 Harmful if swallowed. Wash thoroughly after handling. If swallowed, obtain medical attention immediately.

〈〈S〉〉 May be fatal if swallowed. If swallowed, obtain medical attention immediately.

5.3 Safety precautions

ALL PIPETTE OPERATIONS SHALL BE PERFORMED WITH A PIPETTE BULB OR PLUNGER PIPETTE. THIS IS A CRITICAL SAFETY WARNING!

Safety glasses shall be worn for all laboratory work.

6 Materials and reagents

6.1 General

6.1.1 Handling and labelling

Reagents shall be handled in conformity with health and safety precautions as shown on containers, or as given in other sources of such information. Proper labelling of prepared reagents includes the chemical name, date of preparation, expiration date, restandardization date, name of preparer, and adequate health and safety precautions. The discharge of reagents shall conform to applicable environmental regulations.

6.1.2 Purity

Reagents used in the test procedures shall be certified reagent-grade chemicals and shall meet appropriate standards, or be chemicals of a purity acceptable for the analysis. For details, see ISO 6353-1, ISO 6353-2 and ISO 6353-3.

6.1.3 Water

Whenever water is specified without other qualifiers in the test procedures, only distilled water or water of equivalent purity shall be used.

6.1.4 Strength of solutions

6.1.4.1 Acids and ammonium hydroxide are full strength unless otherwise specified.

6.1.4.2 When a standardized solution is required, its amount-of-substance-concentration is expressed in moles per litre. The number of significant figures to which the molarity is known shall be sufficient to ensure that the reagent does not limit the reliability of the test method.

6.1.4.3 When a standardized solution is not required, its concentration is expressed in grams per litre (g/l) to the appropriate number of significant figures.

6.1.4.4 When a solution is to be diluted, its dilution is indicated by (X + Y), meaning that X volumes of reagent, or concentrated solution, are to be diluted with Y volumes of water (6.1.3).

6.2 Reagents

6.2.1 **Potassium tetraborate solution**, 514 g/l.

Weigh 673 g ± 0,1 g of potassium tetraborate, $K_2B_4O_7 \cdot 4H_2O$, and dissolve it in 550 ml of water in a 1 litre beaker. Then weigh 247 g ± 0,1 g of potassium hydroxide, KOH (DANGER: ⟨⟨C⟩⟩), and dissolve it in the tetraborate solution. Boil on a hotplate for 5 min, cool and add 5 ml of a 10 % aqueous solution of nonylphenoxypoly (6–10) ethylene oxide, NPPO, or similar wetting agent[1)]. Transfer to a 1 litre volumetric flask, rinsing the beaker into the flask several times. When cool, dilute to volume and mix well. Note that the wetting agent will separate out on standing; therefore, the flask must be shaken vigorously before each use.

6.2.2 Boric acid absorbent solution

Add about 800 ml of water to a 1 litre volumetric flask. Stir, using a magnetic stirrer, and add 2 mg to 3 mg of xylene cyanole FF, weighed to the nearest 1 mg, followed by 0,5 ml of NPPO, followed by 5,0 ml of methyl red indicator solution prepared by dissolving 0,125 g of methyl red in 250 ml of methanol (DANGER: ⟨F⟩⟨S⟩). Add 6 g ± 0,1 g of boric acid, H_3BO_3, keeping the contents of the flask stirred until all the constituents are dissolved.

1) Non-ionic detergent with a hydrophilic lipophilic balance in the range of 13 to 14.