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МЕЖДУНАРОДНАЯ ОРГАНИЗАЦИЯ ПО СТАНДАРТИЗАЦИИ

Photography — Processing waste — Determination of ammoniacal nitrogen content — Microdiffusion method

Photographie — Effluents de traitements — Détermination de la teneur en azote ammoniacal — Méthode par microdiffusion

Reference number
ISO 6853 : 1987 (E)

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.

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. They are approved in accordance with ISO procedures requiring at least 75 % approval by the member bodies voting.

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

Users should note that all International Standards undergo revision from time to time and that any reference made herein to any other International Standard implies its latest edition, unless otherwise stated.

Photography — Processing waste — Determination of ammoniacal nitrogen content — Microdiffusion method

1 Scope

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

2 Field of application

The method is applicable for the determination of ammonia content of typical photoprocessing wastes in the range 10 to 200 mg/l of NH_3 or 8 to 160 mg/l of nitrogen. Other volatile amines are determined as ammonia, but the concentrations of these in photoprocessing waste are usually very low. Total amino nitrogen is determined by the microdiffusion Kjeldahl method.

3 References

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

ISO 5667, *Water quality — Sampling —*

Part 1: Guidance on the design of sampling programmes.

Part 2: Guidance on sampling techniques.

Part 3: Guidance on the preservation and handling of samples.

ISO 6851, *Photography — Processing waste — Determination of total amino nitrogen — Microdiffusion Kjeldahl method.*

4 Principle

The ammonia is liberated from the sample by treatment with potassium metaborate and is absorbed into boric acid. The ammonia absorbed is then determined by titration with standard sulfuric acid. The liberation and absorption is carried out in a microdiffusion cell. This 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, to ensure a room temperature distillation in a reasonable time. Scale-up of this determination or alteration of the sample-to-buffer ratio should be avoided as these modifications change the rate of ammonia diffusion.

5 Reactions

$$NH_4X + OH^- = NH_3 + H_2O + X^-$$

6 Reagents

Reagents shall be handled in conformity to health and safety precautions as shown on containers or as given in other sources of such information. The discharge of reagents shall conform to applicable environmental regulations.

WARNING

— **Potassium hydroxide, sodium hydroxide and sulfuric acid are corrosive and cause burns. Avoid contact with eyes, skin and clothing.**

— **Methanol is flammable. Keep away from heat, sparks and open flame. Use adequate ventilation. Harmful if swallowed.**

Reagents used in the tests shall be certified reagent grade chemicals or chemicals of a purity acceptable for the analysis. The acids and ammonia solution referred to shall be undiluted unless dilution is specified. Dilution is specified in terms of amount-of-substance concentration when standardization is required.

Distilled water, or water otherwise produced of at least equal purity, shall be used whenever water is required.

6.1 Potassium tetraborate solution, 514 g/l.

Weigh 673 ± 0,1 g of potassium tetraborate ($K_2B_4O_7 \cdot 4H_2O$) and dissolve in 550 ml of water in a 1 000 ml beaker. Then weigh 247 ± 0,1 g of potassium hydroxide (KOH) 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 1 000 ml 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, so that the flask must be shaken vigorously before each use.

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

6.2 Boric acid absorbent solution.

Add about 800 ml of water to a 1 000 ml volumetric flask. Stir using a magnetic stirrer and add 2 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. Add 6 ± 0,1 g of boric acid (H_3BO_3), keeping the contents of the flask stirred until all the constituents are dissolved. Dilute to within about 15 ml of the mark and mix. Place 1,5 ml of this solution in the centre of a microdiffusion cell and observe the colour.

If the colour in the cell is pink, add just sufficient 0,1 mol/l sodium hydroxide solution to the solution in the 1 000 ml flask to obtain a neutral colour when 1 ml is viewed in the microdiffusion cell.

Check that excess sodium hydroxide is absent by adding 0,10 ml of 0,002 50 mol/l sulfuric acid (6.3) to 1 ml of solution, at which point a pink colour should be produced. Note that the solution in the 1 000 ml flask will appear red, even when 1 ml in the microdiffusion cell looks neutral.

6.3 Sulfuric acid, $c(H_2SO_4) = 0{,}002\ 50$ mol/l.

Pipette 50,0 ml of standard volumetric 0,050 00 mol/l sulfuric acid into a 1 000 ml volumetric flask and dilute to volume with water.

6.4 Cleaning solutions for microdiffusion cells.

6.4.1 Cleaning solution A

Add to a 2 000 ml beaker, from a graduated measuring cylinder, about 750 ml of water and about 750 ml of 0,5 mol/l sulfuric acid and stir. Continue stirring and add 2 to 3 ml of household liquid dishwashing detergent and 1 ml of methyl red indicator solution.

6.4.2 Cleaning solution B

Add to a 2 000 ml about 1 500 ml of water and 2 to 3 ml of the detergent and mix. Add 10 ml of 1,0 mol/l sodium hydroxide and then add enough methyl red indicator solution, while stirring, to produce a yellow colour.

6.4.3 Cleaning solution C

Add to a 2 000 ml beaker about 1 500 ml of water and about 5 ml of the detergent and stir. Continue to stir and add about 10 ml of 0,05 mol/l sulfuric acid and enough methyl red indicator to produce a pink colour.

6.5 Standard nitrogen samples (to check ammonia liberation technique).

Dry ammonium chloride for 2 h in an oven at 100 °C and allow to cool in a desiccator before weighing. Weigh 3,819 ± 0,001 g of this ammonium chloride and quantitatively transfer to a 1 000 ml volumetric flask. Dissolve in water and dilute to volume. This stock solution is equivalent to 0,071 4 mol of nitrogen per litre (1,00 mg/ml) and is stable for at least 3 months.

Pipette 10 ml of this stock solution into a 100 ml volumetric flask and dilute to the mark with water. This standard solution is equivalent to 0,007 14 mol of nitrogen per litre (100 mg/l).

In a similar manner, prepare a standard solution equivalent to 0,001 428 mol of nitrogen per litre (200 mg/l), by pipetting 2,00 ml of the stock solution into a 100 ml volumetric flask and dilute to the mark with water.

7 Apparatus

Ordinary laboratory apparatus and

7.1 Microdiffusion cell, 83 mm, Obrink modification.

7.2 One-mark pipette, 0,500 ml capacity conforming to ISO 648.

7.3 Syringe pipette, 1,00 ml capacity.

7.4 Syringe micro-burette.

7.5 Syringe, to deliver 1,50 ml.

7.6 Polytetrafluoroethylene-coated magnetic bar, 7 mm × ø2 mm.

8 Procedure

Samples shall be taken and prepared in accordance with ISO 5667.

8.1 Cleaning microdiffusion cells and covers

Soak the cells and covers in the cleaning solution A (6.4.1) for 1 h; then remove and soak in the cleaning solution B (6.4.2) for 1 h; then remove and soak in the cleaning solution C (6.4.3) for 1 h.

Remove the cells and covers, shake them as dry as possible and invert them to dry on a clean towel.

Do not touch the insides of the clean cells and covers.

If the cleaned cells and covers have not been washed on the day they are to be used, rinse them with distilled water and set them on a clean towel to dry before using.

8.2 Liberation of ammonia from samples

Using the syringe (7.5), add 1,50 ml of the boric acid absorbent (6.2) to the centre of the microdiffusion cell (7.1). Using the 1,00 ml syringe pipette (7.3), add 1,00 ml of the 2,2 mol/l potassium tetraborate (6.1) to the outer sealing chamber and 1,00 ml to the sample chamber (i.e. the second chamber from the outside). The tetraborate solution should be vigorously shaken before sampling in order to distribute the NPPO. Great care shall be taken not to splash any tetraborate into the centre chamber. If this occurs, a green coloration will be produced,

and the sample shall be discarded. The tetraborate should be deposited on only one part of the sample chamber. Leave enough space for the sample to be added without mixing until the cell is sealed.

Using the calibrated 0,500 ml one-mark pipette (7.2), carefully add 0,500 ml of the sample to the empty side of the sample chamber. Cover the cell immediately and rotate the cover to spread the tetraborate and form a good seal. Using a rotary tipping motion, mix the sample with the tetraborate for 30 s, making certain that the sample and absorbent chambers are fully covered but that the contents of the two separate chambers do not mix. Leave the cell to stand for 2 h. Longer standing times will not introduce any error.

8.3 Titration

Remove the lid carefully from the cell and place the cell on a magnetic stirrer. Place the magnetic bar (7.6) in the centre chamber without splashing. Using the syringe micro-burette (7.4), titrate the solution in the centre chamber with the 0,002 50 mol/l sulfuric acid (6.3) to the first pink colour. Keep the burette tip immersed while titrating. Stir for about 15 s. If the colour fades to light green, continue to titrate until the pink colour remains for at least 15 s. Let the volume used for titration be V_1.

8.4 Blank reading and a calibration curve check

Concurrently with the sample to be analysed, run procedures 8.1, 8.2 and 8.3 on 0,500 ml of distilled water, on 0,500 ml of the 0,007 14 mol/l standard nitrogen sample and on 0,500 ml of the 0,001 428 mol/l standard nitrogen sample. Let the volume used for the titration on the blank be V_2; on the 0,007 14 mol/l (100 mg of nitrogen per litre) V_3 and on the 0,001 428 mol/l (200 mg of nitrogen per litre) V_4.

9 Expression of results

9.1 Method of calculation

Total nitrogen (as N), in milligrams per litre, is given by the formula

$$\frac{72{,}6\,(V_1 - V_2)}{V_s}$$

where

V_1 is the volume, in millilitres, used for the titration of the sample;

V_2 is the volume, in millilitres, used for the titration of the water blank;

V_s is the volume, in millilitres, of the sample taken for diffusion.

If V_s is 0,500 ml, the formula becomes

$$145\,(V_1 - V_2)$$

As a check on the precision, perform similar calculations on the results $(V_3 - V_2)$ and $(V_4 - V_2)$ for the two standard nitrogen samples. These should be carried out for each set of effluent samples or at least daily. The conformance of these results to levels as prepared indicates the probable reliability of the results on unknown samples.

9.2 Precision

The 95 % confidence limits for a single determination are expected to be between 4 and 8 mg of nitrogen per litre. The stoichiometric yield by the microdiffusion method is about 96 %.

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