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INTERNATIONAL STANDARD

**ISO 10348**

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1993-02-01

# Photography — Processing wastes — Determination of silver content

*Photographie — Effluents de traitement — Détermination de la teneur en argent*

Reference number
ISO 10348:1993(E)

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# Contents



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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 10348 was prepared by Technical Committee ISO/TC 42, *Photography*.

Annex A forms an integral part of this International Standard.



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## Introduction

This International Standard is one of a series devoted to the analysis of photographic wastes; it encompasses the field of analysis of silver in photographic effluents.

Analysis for silver in photographic products and effluents presents unique problems in sampling, handling and treatment. These problems are not always adequately covered in standard references. It is the purpose of this International Standard to provide methodology both for sample handling and for the analysis of silver in effluents. Some of the chemicals specified in the test procedures are caustic, toxic or otherwise hazardous. Specific warning, caution and danger notices are noted for particularly hazardous materials but normal precautions required during the performance of any chemical procedure shall be exercised at all times.

In the case of effluents, the photographic laboratory can best establish its conformity to regulations by appropriate chemical analysis. In some cases, in-house analyses will be possible; often the use of an outside laboratory will be required.

Silver in photographic processing wastes originates as a soluble thiosulfate or other complex. Other waste components, however, may interact, resulting in an unstable system containing varying amounts of soluble forms of silver. Waste samples to be analysed for heavy metals are normally stabilized by acidification with nitric acid. This treatment is unsatisfactory for silver in effluents containing photographic processing wastes. Since thiosulfate is unstable in acid solutions, the conventional treatment can actually promote the formation of insoluble silver. Cyanogen iodide (CNI) solution is the effective preservative for silver in these effluents.[1)]

This International Standard includes flame atomic absorption spectroscopy (AAS) and two potentiometric iodide titration (PT) methods of analysis. The method employed will dictate the way in which the sample is preserved and treated. Samples to be analysed by the AAS method are normally ready for analysis, once stabilized with CNI solution. The iodide titration methods, however, require a digestion to solubilize the silver and remove interfering species followed by a boiling step (for Digestion A) to concentrate the sample. The standard digestion methods for AAS, which recommend the use of hydrochloric acid, are not suitable for the preparation of samples for silver analysis.

1) Owerbach, D. The use of cyanogen iodide (CNI) as a stabilizing agent for silver in photographic processing effluents. *Journal of Applied Photographic Engineering,* **4**(1), pp. 2-24, 1978.



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# Photography — Processing wastes — Determination of silver content

## 1 Scope

This International Standard gives methods for determining the silver content in photographic effluents from photographic processing wastes. Sampling, sample preservation and analytical methodology are included.

Three analytical procedures are given with two supporting sample treatment methodologies:

a) a flame atomic absorption spectroscopy (AAS) method;

b) two potentiometric iodide titration (PT) methods.

The choice of treatment is dependent on the analysis method and form of sample. Where AAS is the chosen method for analysis, cyanogen iodide-treated or preserved samples may be analysed directly. For the PT method, two digestion procedures are given: Digestion A for effluents with low salt content, and Digestion B for samples with high solids content.

## 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 385-1:1984, *Laboratory glassware — Burettes — Part 1: General requirements.*

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

ISO 835-1:1981, *Laboratory glassware — Graduated pipettes — Part 1: General requirements.*

ISO 835-2:1981, *Laboratory glassware — Graduated pipettes — Part 2: Pipettes for which no waiting time is specified.*

ISO 835-3:1981, *Laboratory glassware — Graduated pipettes — Part 3: Pipettes for which a waiting time of 15 s is specified.*

ISO 835-4:1981, *Laboratory glassware — Graduated pipettes — Part 4: Blow-out pipettes.*

ISO 1042:1983, *Laboratory glassware — One-mark volumetric flasks.*

ISO 3696:1987, *Water for analytical laboratory use — Specification and test methods.*

ISO 4788:1980, *Laboratory glassware — Graduated measuring cylinders.*

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:—[2], *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.*

2) To be published. (Revision of ISO 5667-3:1985)



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## 3 Principle

### 3.1 Flame atomic absorption spectroscopy (AAS) methodology

A silver-containing lamp, when heated to excitation, generates a spectrum which includes the ultraviolet emission bands of the silver atom. The silver ions in a solution aspirated into a flame will absorb the silver emission bands when light from the lamp is passed through the flame, according to a Beer's law relationship:

$$c_{Ag} = k \cdot \log(\tau/\tau_0)$$

where

$c_{Ag}$ is the concentration of silver ions;

$\tau$ is the transmittance of light through the flame at the specified wavelength aspirated with the sample;

$\tau_0$ is the transmittance of light through the flame at the specified wavelength aspirated with a silver-free reference sample;

$k$ is a constant.

A calibration curve is normally generated to define the relationship specifically.

### 3.2 Potentiometric titration (PT) methodology

The titration of a solution containing silver ions with an iodide solution will form a precipitate of silver iodide in accordance with the reaction:

$$Ag^+ + I^- \rightarrow AgI(solid)$$

A silver electrode, either prepared from a silver billet (11.1.2) or an iodide-selective electrode, with an appropriate reference electrode will generate a potential in the presence of a solution containing silver ions in accordance with the Nernst equation:

$$E = E_0 + 0{,}023(RT/nF) \cdot \log(c_{Ag})$$

where

$E$ is the measured potential, in volts;

$E_0$ is the base potential, including the reference electrode contribution, in volts;

$R$ is the universal gas constant;

$T$ is the thermodynamic temperature;

$n$ is the number of electrons transferred;

$F$ is the Faraday constant;

$c_{Ag}$ is the concentration of silver ions.

In the presence of an excess of iodide ions, the silver ion concentration, $c_{Ag}$, is given by

$$c_{Ag} = K_{SP}/c_I$$

where

$K_{SP}$ is the solubility product for silver iodide;

$c_I$ is the concentration of iodide ions.

A sharp change in potential is obtained during the titration as the solution progresses from one with silver ions in excess to one with iodide ions in excess.

## 4 Reliability

The practical lower limits (approximate) for silver analyses are the following

| | |
|---|---|
| **Direct flame AAS** | 0,1 mg/l |
| **PT, Digestion A** (sample 500 ml; titrant 0,001 mol/l potassium iodide) | 0,2 mg/l |
| **PT, Digestion B** (sample 1,0 ml; titrant 0,001 mol/l potassium iodide) | 100 mg/l |

The $2\sigma$ confidence limits for Digestion A, as determined through inter-laboratory tests, are equal to $\pm$ 0,12 mg/l for samples containing 0,2 mg/l to 4,0 mg/l of silver in effluent samples. These results are based on seventeen data points obtained from six different laboratories[3]. This value is true for both potentiometric iodide titration methods using either the manual method (pH-meter) or the automatic titrimeter method with a titrant addition rate of 0,1 ml/min.

The $2\sigma$ confidence limits for the flame AAS method are equal to $\pm$ 0,007 mg/l for samples containing 0,02 mg/l to 0,05 mg/l of silver in photographic effluents, when directly aspirated after a five-fold sample volume reduction.

## 5 Safety and operational precautions

The test procedure for silver analysis given in this International Standard requires careful technique by an experienced operator. It requires care in sample storage and safe handling of both the sample and reagent solutions due to the hazards and/or toxicity of the cyanogen iodide stabilizing agent and other solutions. Due to the vigorous agitation and possible loss by splashing in the potentiometric iodide titration, careful monitoring of the procedure is required. The unique requirements and the large number of items

3) For an explanation on how the data were treated, see the *ASTM Handbook on Statistical Methods*. Copies are available from ASTM, 1916 Race Street, Philadelphia, PA 19103, USA.



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in separate categories result in the following 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, rubber gloves and other protective apparel such as face masks or aprons where appropriate. Specific danger notices are given in the text and footnotes 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 test procedure section, the hazard will be indicated by the word "DANGER" followed by a symbol consisting of angle brackets "< >" containing a letter which 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.

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 symbol 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 from the manufacturer pertinent information 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.

< 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.

< 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.

## 5.3 Safety precautions

**ALL PIPETTE OPERATIONS SHALL BE PERFORMED WITH A PIPETTE BULB OR PLUNGER PIPETTE. Failure to observe this warning notice can result in cyanide poisoning. THIS IS A CRITICAL SAFETY WARNING!**

**Digestion procedures shall be performed in a fume hood. Hydrogen cyanide or other toxic substances can be evolved.**

**Safety glasses shall be worn for all laboratory work.**

**Cyanogen iodide may be decomposed by treatment with sodium hypochlorite.**

## 5.4 Operational precautions

**5.4.1** The cyanogen iodide (CNI) silver solvent shall be added to the bulk sample a sufficient time before the test sample is taken, to ensure complete dissolution of occluded or precipitated silver. If a bulk sample is acidic, it shall be neutralized before CNI is added. The minimum reaction time after CNI addition is 1 h. CNI may be added at the bulk sampling time. The treated sample is stable indefinitely.

**5.4.2** Bulk samples containing large quantities of silver, to be dissolved by the addition of large volumes of CNI (up to 20 % addition), shall have the original bulk sample volumes recorded at the bulk sampling time before the addition of CNI. When tested, the dilution factor given in 8.2.3 shall be used with the AAS value to find the true concentration of the sample. The recommended reaction time is overnight for samples with large silver concentrations resulting from particulate matter. Representative samples, including solids and particulate matter, shall be taken for true silver values. Representative sampling shall also take into account adsorption of silver or precipitation of silver species on the container walls. Appropriate treatment of the container when removing the sample is necessary.

**5.4.3** Where CNI preservation is used, the sample container material is not of significant concern. In these cases, plastic containers are preferred to avoid breakage. In any event, sample containers shall be properly cleaned (see 5.4.5).



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**5.4.4** Analyses of samples for dissolved silver require immediate filtration after sampling and the use of support apparatus that will not affect the silver concentration after filtration. A 0,45 µm membrane filter medium with a stainless steel support is an acceptable system. Fritted glass and ceramic filter elements may absorb silver from the filtrate and are not recommended.

**5.4.5** All glassware and containers shall be cleaned by soaking with a 1 % to 2 % CNI solution or concentrated nitric acid for a minimum of 4 h to prevent absorbed silver from being released from the container walls and becoming part of the sample during digestion. The containers shall then be rinsed several times with distilled water.

**5.4.6** Digestions shall be carried to completion in order to eliminate interfering materials that react with the potassium iodide titrant.

**5.4.7** The titration rate with potassium iodide shall not exceed 0,1 ml/min until the end-point is approached. The final titration rate shall not exceed 0,05 ml/min. In cases of low silver concentrations and, therefore, slow chemical reaction rates, faster titration rates will result in apparent high values. These titration rates apply to automatic titration equipment as well.

**5.4.8** The specified silver electrode cleaning and coating procedure shall be performed to provide the correct coating depth of sulfide. Excess time in the sulfide reagent can overcoat the electrode, resulting in slow reaction and apparent high values.

**5.4.9** The digested sample volume, immediately before titration, should be 150 ml or less. A larger volume may degrade the potentiometric break required for determining the end-point of the titration, especially for samples low in silver.

**5.4.10** The sample shall be stirred vigorously during titration to assist reaction completion. Failure to do so can result in invalid values for the analysis.

**5.4.11** All containers shall be labelled and dated. Appropriate warning labels shall be affixed to the containers.

## 6 Reagents

**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 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.

**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. See ISO 6353-1, ISO 6353-2 and ISO 6353-3.

Whenever water is specified without other qualifiers in the test procedures, only distilled water or water of equal purity shall be used. See ISO 3696.

**Strength of solutions:** When a standardized solution is required, its concentration should be expressed in moles per litre. The number of significant figures to which the molar concentration is known should be sufficient to ensure that the reagent does not limit the reliability of the test method.

When a standardized solution is not required, its concentration should be expressed in grams per litre to the appropriate number of significant figures.

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

### 6.1 Reagents for atomic absorption spectroscopy (AAS)

**6.1.1 Cyanogen iodide solution (CNI)** (DANGER: <<S>> <B> <C>)[4].

**6.1.2 Silver (Ag) standard solutions**, 0,5 mg/l, 1,0 mg/l, 3,0 mg/l and 5,0 mg/l.[4]

### 6.2 Reagents for potentiometric titrations (PT)

**6.2.1 Acetic acid ($CH_3COOH$)**, glacial, $\rho \approx 1{,}05$ g/ml (DANGER: <C> <B>).

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

**6.2.3 Hydrogen peroxide ($H_2O_2$)**, 30 % (*m/m*) (approximately) (DANGER: <C> <B> <O>).

**6.2.4 Nitric acid ($HNO_3$)**, 70 % (*m/m*) (approximately) (DANGER: <C> <B> <O>).

**6.2.5 Potassium iodide standard solutions (KI)**, 0,1 mol/l, 0,01 mol/l and 0,001 mol/l.[4]

**6.2.6 Potassium nitrate solution ($KNO_3$)**, saturated.

Add 50 g of potassium nitrate to 100 ml of water. Stir for 5 min then warm to room temperature. Maintain

4) Procedures for the preparation of these solutions are given in annex A.



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an excess of undissolved potassium nitrate crystals in the reagent container.

**6.2.7 Silver nitrate ($AgNO_3$) standard solutions**, 0,100 mol/l, 0,010 mol/l and 0,001 mol/l.[4)]

**6.2.8 Sulfuric acid ($H_2SO_4$)**, $\rho \approx 1{,}84$ g/ml (DANGER: << C >>).

## 7 Glassware

All glassware subject to heating shall be of heat-resistant borosilicate glass.[5)]

Pipettes and other volumetric glassware shall meet the volume requirements of Class A glassware as specified in ISO 385-1, ISO 648, ISO 835-1, ISO 835-2, ISO 835-3, ISO 835-4, ISO 1042 and ISO 4788.

## 8 Sampling and sample pretreatment

This International Standard covers a choice of analytical methods and sample preparation procedures. In order to ensure that the analysis yields a meaningful result, it is necessary that the proper choices and decisions are made from among the options. It is, therefore, the intent of this clause to provide a systematic and rational approach to making the choices consistent with the material type and analysis technique for

a) representative sampling;

b) sample size determination;

c) sample preparation.

Table 1 provides an overview of the choices based on sample type treatment and analysis method. For AAS, any sample treatment is useable but CNI is preferred for simplicity where applicable. PT requires a digestion treatment, and the less vigorous Digestion A method is preferred where applicable. Samples with a high solids content require the more vigorous Digestion B method for PT and AAS when the CNI method is not suitable for the latter.

### 8.1 Sampling and preservation

It is necessary that the analysis be carried out on a representative sample and the sampling of a process effluent or a plant effluent can encompass many difficulties and due care shall be exercised. See especially ISO 5667-1, ISO 5667-2 and ISO 5667-3. Sampling shall be carried out in conformance with regulatory requirements. Sampling should be carried out under typical operating conditions and normally should be representative of the overall plant effluent. Daily samples that are truly representative of the effluents require sampling over 24 h and sampling that is proportional to flow rate. Samples taken during a sudden discharge or during another non-routine operation will not yield results representative of the normal operation.

The method of analysis and desired result will determine the need for sample preservation. It is generally recommended that all effluent samples intended for silver analysis be treated with CNI. It should be noted that samples treated with CNI, intended for shipment, may be in violation of transport regulations and, if so, the CNI treatment can be carried out after arrival. It is only necessary that the treatment be carried out at least 1 h before the analysis. Normal treatment with CNI for stabilization requires the addition of CNI at a rate of 1,0 ml of CNI solution per 100 ml of sample.

CNI is used to stabilize samples for AAS analysis. After CNI stabilization, such samples can normally be directly used for aspiration. CNI is used for PT samples to prevent loss of material by absorption to the container walls. Since PT analysis requires a digestive procedure, CNI stabilization is not required, provided that there is no loss of silver due to absorption.

**Table 1 — Operational flowchart**

| Sample type | Analysis method | Subclause | Treatment | Subclause |
|---|---|---|---|---|
| Low-salt effluents | AAS<br>AAS or PT<br>AAS or PT | 10.2<br>10.2 or 11.2<br>10.2 or 11.2 | CNI<br>Digestion A<br>Digestion B | 8.2.2 or 8.2.3<br>9.2<br>9.3 |
| High-solids effluents | AAS or PT | 10.2 or 11.2 | Digestion B | 9.3 |

5) Pyrex® is an example of suitable glassware available commercially. This information is given for the convenience of users of this International Standard and does not constitute an endorsement by ISO of this product.



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## 8.2 Sample size and pretreatment

The practical lower limits for the analysis techniques are 0,1 mg/l for AAS and 0,2 mg/l for PT (see 9.2.2, Digestion A). A minimum sample volume for AAS is 10 ml and this is equivalent to 0,001 mg of silver. PT requires a minimum titration equivalent to 1 ml of 0,001 mol/l potassium iodide solution. This is equivalent to 0,1 mg of silver; therefore, at 0,2 mg/l an initial sample of 500 ml is required for the titration of silver. The analytical techniques may be extended into lower ranges (0,1 mg/l to 0,2 mg/l) when trying to avoid the concentration step, or where the amount of sample is limited.

The procedures given in this International Standard are for a higher range of silver content. This range is considered to be better suited for both sample handling and analysis technique. In view of this, a dilution may be necessary at a subsequent stage, but the increase in convenience and reliability more than justify the extra step.

Most AAS instruments operate linearly in the range of 0,1 mg/l to 3,0 mg/l of silver. This International Standard provides appropriate dilutions to achieve this. Instruments operating in a different linear range require appropriate adjustment of the dilutions. For calculation purposes, 1,0 mg/l will be used as the aim point. A convenient aspiration volume is 50 ml (10 ml is minimum) and a 50 ml volume would thus contain about 0,05 mg of silver.

The analysis of silver by PT is conveniently and reliably carried out with a titration requiring 10 ml of 0,001 mol/l potassium iodide solution. This is equivalent to 1,08 mg of silver and, in order to obtain a relatively sharp end-point, the volume of the sample shall not exceed 150 ml.

Under certain conditions, it is possible to achieve these silver levels directly from the samples and the primary solution prepared is then suitable for use in the analysis procedure. This is particularly true for effluents when they are analysed by the AAS method. In these instances, an appropriate-size sample treated with CNI is then ready for direct AAS analysis.

Under some circumstances, a digestion procedure or concentration procedure followed by digestion is required. In these instances, the sample is taken to obtain approximately 0,1 g of silver and, in the case of materials with a high solids content, approximately 0,5 g of silver. The sample is treated and results in a solution that is approximately 100 mg/l or 500 mg/l for materials with a high solids content. These primary solutions are then appropriately diluted to obtain a solution of approximately 1,0 mg/l for AAS and 10 mg/l for PT, from which an aliquot is taken for the analysis.

It becomes apparent that the first step in the process requires that an estimate be made of the silver content of the sample. If the estimate is higher than the real value, the problem encountered will be of having less silver than is desired. This can result in values near or below the working limits and correction will require resampling and retreatment. On the other hand, if the estimate made is lower than the actual value, a solution would be obtained that is outside the working range of the analysis on the high side. In this case, a simple dilution would provide a solution that is in the working range of the procedure. For this reason, it is preferrable to bias the estimate of the silver content lower than expected.

Determination of the sample size according to the type of sample and its pretreatment is considered in 8.2.1. The actual analysis requires calibration measurements as well as sample measurements. The preparation of calibration samples is given in annex A.

### 8.2.1 Determination of sample size

The concentration of silver in effluents can be as high as 1 000 mg/l, but is usually below 10 mg/l. The test sample is taken in accordance with table 2, based on the estimate of the silver concentration.

**Table 2 — Test sample size for AAS or digestion procedure**

| Estimated Ag concentration mg/l | Test sample size, Digestion A ml | Dilution for AAS |
|---|---|---|
| 200 to 1 000 | 1,0 | 1,0:1 000 |
| 50 to 250 | 5,0 | 1,0:100 |
| 20 to 100 | 10,0 | 1,0:100 |
| 5 to 25 | 50,0 | 10,0:100 |
| 2 to 10 | 100,0 | 10,0:100 |
| 0,2 to 2,5 | 500,0 | No dilution |
| 0,1 to 0,2 | 1 000,0 | No dilution |
| Less than 0,1 | Concentrate sample | |

### 8.2.2 Sample pretreatment for AAS

Effluents containing 0,1 mg/l to 3,0 mg/l of silver can be analysed by AAS by direct analysis. Treat the bulk sample with CNI (DANGER: << S >> < B > < C >) at the proportion of 1 ml of CNI reagent for 100 ml of sample. Allow the treated solution to stand for at least 1 h and, after mixing, aspirate it directly into the spectrometer (see 10.2). Dilute samples, if necessary, to bring them into the 0,1 mg/l to 3,0 mg/l range. Effluent samples containing less than 0,1 mg/l require a concentration step such as that used in 9.2.1 and then a volume adjustment.

### 8.2.3 Sample pretreatment for AAS for samples with significant particulate matter

**8.2.3.1** Some effluents may contain significant amounts of particulate matter. Where AAS is available, these samples can be handled by a modified CNI procedure and the digestion procedure is avoided.



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Treat bulk samples having large amounts of particulate matter with 20 % CNI solution (<< S >> < B > < C >). The sampling procedure and formula correction are defined in 8.2.3.2. Specific volumes used, such as those shown in an example given in table 3, must be known to calculate the dilution factor.

**Table 3 — Example of pretreatment of sample**

| Step | Item | Volume ml |
|---|---|---|
| 1 | Bulk sample volume | 2 000 |
| 2 | CNI added; 20 % bulk sample | 400 |
| 3 | Total volume (step 1 + step 2) | 2 400 |
| 4 | Desired test sample volume (25 % of original bulk sample) | 500 |
| 5 | Volume added to graduated cylinder (25 % of step 3) | 600 |
| 6 | Total volume after filtration and rinsing | 750 |

**8.2.3.2** The following procedure shall be performed:

a) If the bulk sample is acidic, first neutralize it. Then mix CNI (<< S >> < B > < C >) into the sample. Allow it to react until clear or leave it overnight.

NOTE 1 Overnight treatment is sufficient to dissolve any silver but may not clear the solution.

b) Vigorously shake the sample or blend it and quickly pour into a graduated cylinder a volume equal to the desired test sample *plus* the added CNI percent volume for that sample (see table 3, step 5). Be sure to include particulate matter for a representative sample. Record the sample volume.

c) Suction-filter the test sample through sintered glass or a porcelain frit and rinse the filtering apparatus with CNI (<< S >> < B > < C >) and then with distilled water. Pour the filtrate into a graduated cylinder to obtain the volume of the solution (see table 3, step 6). Record the total volume of the solution.

Calculate the dilution factor, $K$, from the following equation:

$$K = \frac{V_6}{V_4}$$

where

$V_4$ is the desired volume of the test sample, in millilitres (see table 3, step 4);

$V_6$ is the total volume of solution after filtration, in millilitres (see table 3, step 6).

EXAMPLE

Referring to table 3, the following dilution factor is used:

$$K = \frac{750}{500} = 1{,}5$$

The dilution factor is required because of the addition of excess CNI required to solubilize the silver in the total bulk sample. The dilution factor shall be used with both AAS and potentiometric titration end-point values. The corrected equation using this dilution factor is as follows for AAS:

$$c_{Ag} = K \times c_{DR}$$

where

$c_{Ag}$ is the concentration of silver, in milligrams per litre of solution;

$K$ is the dilution factor;

$c_{DR}$ is the direct reading of the concentration from the spectrometer.

### 8.2.4 Sample pretreatment for PT analysis

This requires a preliminary digestion (see 9.2 for Digestion A or 9.3 for Digestion B). A concentration step (see 9.2.1) is used for low silver content samples that are also low in salt content.

## 9 Preparation of test sample

Three sample preparation methods are given. The first method (see 9.1) is the simplified method of treatment with CNI with various sample types and normally provides a sample suitable for direct analysis by AAS. The second and third methods are digestion techniques. The first of these (see 9.2) includes a concentration and then a digestion procedure suitable for dilute materials to be analysed by either technique (AAS or PT). The second digestion technique (see 9.3) is a more vigorous one and is necessary for effluents containing materials with a high solids content. If in doubt, the second digestion procedure shall be used.

### 9.1 Cyanogen iodide (CNI) treatment

For analysis using AAS, the CNI method of preservation normally eliminates the need for digestion pretreatment. One exception may be samples with very large quantities of particulate matter or colloidal suspension. In such cases the addition of more CNI (up to 20 % by volume) can be attempted (see 8.2.3) and the appropriate volume correction made.



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## 9.2 Concentration and digestion (Digestion A)

This technique is intended for effluents with low silver contents and low salt contents.

**WARNING — Carry out all evaporations in a fume cupboard (exhaust hood) with adequate ventilation. During the digestion steps, the dense white fumes ($SO_3$) given off are toxic, therefore avoid inhalation.**

### 9.2.1 Sample concentration

Add, using a graduated cylinder, 500 ml of the sample to a 1 000 ml graduated beaker containing 5 to 10 glass beads. Evaporate the sample on a hot-plate to about 50 ml in an exhaust hood. Remove the beaker from the hot-plate and allow the solution to cool for 5 min at room temperature.

### 9.2.2 Sample digestion (Digestion A)

**WARNING — Use a fume cupboard (exhaust hood) with a safety shield. Wear gloves, safety goggles and apron for this operation. Carry out the heating in a fume cupboard (exhaust hood) with adequate ventilation. During acid or peroxide addition, bumping may occur. The dense white fumes ($SO_3$) given off are toxic, therefore avoid inhalation.**

**9.2.2.1** To a 1 000 ml beaker containing the concentrated sample, very slowly add with stirring 5 ml of sulfuric acid (6.2.8) (<< C >>) from a graduated cylinder. Add, very slowly, using a graduated cylinder, 5 ml of nitric acid (6.2.4) (< C > < B > < O >) to the beaker. Then add, very slowly, using a graduated cylinder or a tip-up pipette, 4 ml of 30 % hydrogen peroxide (6.2.3) (< C > < B > < O >). Place the uncovered beaker on a hot-plate and heat until 2 min after the appearance of dense white fumes ($SO_3$). Remove the beaker from the hot-plate and allow the solution to cool for 5 min at room temperature.

**9.2.2.2** Repeat the procedure given in 9.2.2.1 twice more. On the second repeat, continue the concentration until the remaining volume is approximately 25 ml. Remove the beaker from the hot-plate and allow the solution to cool to room temperature.

### 9.2.3 Adjusting the pH of the digested sample

**9.2.3.1** Quantitatively transfer the contents of the 1 000 ml beaker into a 250 ml beaker using distilled water. The final volume should be approximately 100 ml. Add, very slowly, using a graduated cylinder, concentrated ammonium hydroxide (6.2.2) (< C > < B >) until the sample is just basic (red litmus paper turns blue). Approximately 25 ml to 40 ml of ammonium hydroxide will be required. Add, using a graduated cylinder, acetic acid (6.2.1) (< C > < B >) until the sample is just acidic (blue litmus paper turns red). Then add another 2 ml of acetic acid (6.2.1).

**9.2.3.2** This solution contains about 100 mg of silver (500 mg if from a high-solids source). Transfer the solution quantitatively to a 1 000 ml one-mark volumetric flask and make up to the mark with water.

**9.2.3.3** For AAS, proceed according to 10.2.3.2. For PT, proceed according to 11.2.

## 9.3 Vigorous digestion (Digestion B)

### 9.3.1 Concentration of solutions with high levels of solutes

Add the appropriate sample to a beaker rated at least twice the size of the sample. Add 5 to 10 glass beads and, in a fume cupboard (exhaust hood), evaporate the sample on a hot-plate to about 50 ml. Remove the beaker from the hot-plate and allow the solution to cool to room temperature.

### 9.3.2 Sample digestion (Kjeldahl)

**WARNING — Use a fume cupboard (exhaust hood) with a safety shield. Wear gloves, safety goggles and apron for this operation. Carry out the heating in a fume cupboard (exhaust hood) with adequate ventilation. During acid or peroxide addition, bumping may occur. Ensure that the neck of the flask is pointed toward the back of the hood. The dense white fumes ($SO_3$) given off are toxic, therefore avoid inhalation.**

**9.3.2.1** Set up a 100 ml Kjeldahl flask in a fume cupboard (exhaust hood). The neck of the flask should form an angle of about 30° to 45° with the supporting ringstand rod. Insert a piece of porous plate or a few glass beads to prevent bumping. Introduce the sample obtained into the Kjeldahl flask. Using a graduated cylinder, very slowly add 4 ml to 6 ml of sulfuric acid (6.2.8) (<< C >>) to the Kjeldahl flask. Add, very slowly, using a graduated cylinder, 4 ml to 6 ml of nitric acid (6.2.4) (< C > < B > < O >) to the Kjeldahl flask. Add 4 ml to 6 ml of 30 % hydrogen peroxide (6.2.3) (< C > < B > < O >), a drop at a time, swirling the flask to stir the contents. Heat the flask gently with a Bunsen burner or other suitable heating device to decompose and dissolve the sample. Continue to alternately add hydrogen peroxide (6.2.3) and to heat until the solution clears.

**9.3.2.2** Boil the solution until the solid matter has completely dissolved. The solution may be colourless or slightly yellow. Continue boiling until the dense white fumes have issued from the mouth of the flask for a period of approximately 30 s (these dense white fumes should not be confused with the water vapour that first appears). Continue to make hydrogen peroxide (6.2.3) (< C > < B > < O >) additions, alternately



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cooling before the addition and heating afterwards, until the solution clears.

#### 9.3.3 Adjusting the pH of the sample

**WARNING — Explosive boiling occurs if water is added too rapidly to concentrated sulfuric acid (<<C>>). Add the water in small increments, mixing well, and allow some cooling time between additions. Make certain the Kjeldahl flask is pointed away from people. Heat the solution to dissolve any crystallized salts, then cool.**

**9.3.3.1** Allow the Kjeldahl flask (see 9.3.2) to cool to room temperature. Slowly and carefully add, in small portions, 25 ml of water to the Kjeldahl flask. Drop a small piece of red litmus paper into the flask and cautiously add ammonium hydroxide (6.2.2) (<C> <B>) dropwise until the litmus paper turns blue (disregard any white or red-brown precipitate). Add acetic acid (6.2.1) (<C> <B>) dropwise until the litmus paper just turns red. Add an additional 2 ml of acetic acid (6.2.1) and swirl.

**9.3.3.2** This solution contains about 100 ml of silver (500 g if from a high-solids source). Transfer quantitatively to a 1 000 ml volumetric flask and dilute to the mark.

**9.3.3.3** For AAS, proceed according to 10.2.3.2. For PT, proceed according to 11.2.

## 10 Analysis by atomic absorption spectroscopy (AAS)

### 10.1 Special apparatus

**10.1.1 Atomic absorption spectrometer**, with the following characteristics.

a) Silver hollow cathode lamp.

b) Wavelength: 328,1 nm.

c) Fuel: acetylene.

d) Oxidant: air.

e) Type of flame: oxidizing.

### 10.2 Procedure

#### 10.2.1 General

Differences between the various makes and models of satisfactory atomic absorption spectrometers prevent the formation of detailed instructions applicable to every instrument. The analyst should follow the manufacturer's operating instructions for the particular instrument.

In general, after installing the silver hollow cathode lamp, the lamp should be allowed to warm up for a minimum of 15 min. During this period, align the spectrometer, position the monochromator at the correct wavelength, select the proper monochromator slit width, and adjust the hollow cathode current according to the manufacturer's recommendation. Subsequently, light the flame and regulate the flow of fuel and oxidant, adjust the burner and nebulizer flow rate for maximum percent absorption and stability, and balance the spectrometer.

Run a series of silver standards (6.1.2) containing CNI solution (6.1.1) and construct a calibration curve by plotting concentrations of the standards against the absorbance. For those instruments that read directly in concentration, set the curve corrector to read out the proper concentration. Aspirate the samples and determine the concentrations either directly or from the calibration curve. For best results, run silver standards, bracketing the sample range each time a series of samples is run.

#### 10.2.2 Calibration curve

Prepare the silver standards for AAS and, if the samples are CNI treated, then add CNI to the standards in the proportion of 1 ml per 100 ml. Use background correction if available. Run the standards and construct the calibration curve. Only use the straight-line portion of the curve. This will generally lie between 0,1 mg/l to 3,0 mg/l of silver.

#### 10.2.3 Final treatment of samples

Measure the samples and determine the silver level from the calibration curve. If the value does not fall on the straight line portion of the curve, concentrated solutions should be appropriately diluted and the diluted samples measured.

##### 10.2.3.1 CNI-treated samples

CNI-treated samples and appropriate dilutions are normally suitable for direct AAS analysis.

##### 10.2.3.2 Digested samples

Samples resulting from the digestion procedures (9.2 or 9.3) are targeted at 100 mg/l and therefore require dilution before analysis. The dilution is normally 100:1 (1,0 ml to 100 ml). In the case of high-solids samples (resins, sludges, residues, metals), the dilution is 500:1 (1,0 ml to 500 ml). The diluted samples are suitable for AAS analysis.

## 11 Analysis by potentiometric iodide titration (PT)

This method is more involved and time-consuming than the AAS method. It is used when AAS is not



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available or when a referee method is required. It covers silver contents as low as 0,1 mg/l and may be applied at higher contents by using a smaller sample, or at lower contents by using a larger sample. The principles and procedures for preservation with CNI solution are the same as those given in clause 9. Digestion, however, is required prior to potentiometric titration to remove interferences. A concentration step is used prior to carrying out Digestion A. A pH-meter can be used for titrating manually, but the addition of titrant shall be very slow, 0,1 ml/min and 0,05 ml/min near the end-point.

## 11.1 Special apparatus

**11.1.1 Potentiometric read-out device**, consisting of either of the following.

**11.1.1.1 pH-meter**, with 2 decimal capability.

**11.1.1.2 Potentiograph (automatic titrimeter)**, capable of titrating at 0,05 ml/min.

### 11.1.2 Silver sulfide electrode

Polish a silver billet or rod electrode with a suitable abrasive material, e.g. a non-woven fabric. Wipe it then dip it for approximately 15 s in nitric acid (< C > < B > < O >). Rinse with water and immerse in a sodium sulfide solution [1 % (*m/m*)] for 10 s to 15 s.

NOTE 2 The specified cleaning and coating procedure for the silver electrode should be performed to provide the correct coating depth. Excess time in the sulfide solution can overcoat the electrode, resulting in slow response and apparent high values.

### 11.1.3 Reference electrode

Any double-junction reference electrode with a non-halide outer solution (i.e. nitrate) is usable.

**11.1.4 Fume cupboard (exhaust hood)**, with positive exhaust fan.

### 11.1.5 Burette

**11.1.6 Stirring apparatus**, magnetic or the equivalent.

### 11.1.7 Laboratory glassware

Clean all laboratory glassware and containers by soaking with 1 % to 2 % CNI solution (6.1.1) (<< S >> < B > < C >) or nitric acid (6.2.4) (< C > < B > < O >) for a minimum of 4 h to prevent absorbed silver being released from the container walls and contributing to the sample during digestion. After treatment, rinse the containers several times with water.

## 11.2 Procedure

### 11.2.1 General

The electrode pair consists of the silver sulfide (11.1.2) and reference electrodes (11.1.3). Adjust the magnetic stirring unit (11.1.6) to stir the sample vigorously during the titration. The samples taken for digestion should have a silver content of 100 mg/l (500 mg/l for high-solids samples).

When using a potentiograph (11.1.1.2), use a maximum speed setting of 0,1 ml/min. Near the end-point, reduce the titrating speed to 0,05 ml/min.

### 11.2.2 Low-salt-content samples containing 0,2 mg/l to 4,0 mg/l of silver (Digestion A)

Titrate the sample potentiometrically, using 0,001 mol/l potassium iodide (6.2.5).

For titration, use 1 ml or more of titrant. A silver content of 0,2 mg/l is the lowest to be titrated under the above conditions when a 500 ml sample is taken initially. A content of 0,1 mg/l of silver is obtained by using a 1 000 ml sample.

### 11.2.3 Samples containing more than 4 mg/l of silver

If approximate silver contents are known, then the conversion factor given in table 4 can be used effectively. When in doubt, use the larger sample size.

NOTE 3 A 0,001 mol/l potassium iodide (KI) solution (6.2.5) is generally adequate for most of these kinds of samples. In cases where the sample has a higher content of silver than expected, the use of 0,001 mol/l KI will result in excessively long titrations. One suggestion is to record the 0,001 mol/l KI consumed to a point and then switch to 0,01 mol/l KI titrant. Take into account in the calculation both these titrant volumes (and molar concentrations).

### 11.2.4 Dilution factor

It is necessary to use the dilution factor given in 8.2.3 when large concentrations of CNI are required to elute silver from a sample containing large amounts of particulate matter.

## 11.3 Calculations for titration methods

Calculate the silver concentration, $c_{Ag}$, in milligrams per litre of solution, by using the following equation:

$$c_{Ag} = 1{,}079 \times 10^5 (c_T V_T / V_S)$$

where

$c_T$ is the exact concentration, in moles per litre, of the KI titrant;

$V_T$ is the volume, in millilitres, of KI titrant used;



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$V_S$ is the sample volume, in millilitres;

$1{,}079 \times 10^5$ is a combined conversion factor obtained from the mass of silver, in grams, equivalent to 1 mol of potassium iodide (i.e. 107,9) × the conversion factor for grams to milligrams (i.e. 1 000).

**Table 4 — Conversion factors**

| Sample size ml | Concentration of KI mol/l | Conversion factor mg/mol |
|---|---|---|
| 1 000 | 0,001 | 0,108 |
| 500 | 0,001 | 0,216 |
| 100 | 0,001 | 1,079 |
| 50,0 | 0,001 | 2,158 |
| 10,0 | 0,001 | 10,79 |
| 5,0 | 0,001 | 21,58 |
| 1,0 | 0,001 | 107,9 |



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# Annex A
(normative)

# Preparation of reagent solutions

## A.1 Preparation of cyanogen iodide solution (CNI) (DANGER: << S >> < B > < C >)

### A.1.1 Reagents

**A.1.1.1 Potassium cyanide (KCN)** (DANGER: << S >> < C >).

**A.1.1.2 Iodine ($I_2$)**

**A.1.1.3 Potassium iodide (KI)**

**A.1.1.4 Ammonium hydroxide ($NH_4OH$),** $\rho \approx 0{,}91$ g/ml, (DANGER: < C > < B >).

### A.1.2 Procedure

**A.1.2.1** Dissolve 40 g of potassium iodide (A.1.1.3) in 25 ml of water in a 100 ml one-mark volumetric flask. Add 12,7 g ± 0,1 g of iodine (A.1.1.2) to the flask. Mix by swirling until it is certain that the iodine has dissolved, and then make up to the mark with water. Store in a glass-stoppered brown bottle in the dark. Standardization is not required.

**A.1.2.2** Weigh out 6,5 g ± 0,1 g of potassium cyanide (A.1.1.1) (<< S >> < B >) and dissolve it in 50 ml of water. Transfer this solution (<< S >> < B >) to a 100 ml volumetric flask. Add, using a graduated cylinder, 4,0 ml of ammonium hydroxide (A.1.1.4) (< C > < B >) then 5,0 ml of 1,0 mol/l iodine solution (A.1.2.1) to the 100 ml one-mark volumetric flask. Make up to the mark with water, stopper the flask and invert several times to mix. Transfer the cyanogen iodide solution (<< S >> < B > < C >) to a brown bottle and store in a fume (exhaust) hood. Label and date the bottle(s). The reagent is usable until it develops a dark amber colour.

## A.2 Preparation of silver standard solutions (0,5 mg/l, 1,0 mg/l, 3,0 mg/l and 5,0 mg/l) for AAS method

### A.2.1 Reagents

**A.2.1.1 Silver nitrate ($AgNO_3$)** (DANGER: < C >).

**A.2.1.2 Ammonium hydroxide ($NH_4OH$),** $\rho \approx 0{,}91$ g/ml (< C > < B >).

**A.2.1.3 Cyanogen iodide solution (CNI)** (A.1) (<< S >> < B > < C >).

### A.2.2 Procedure

See 5.4.5 for cleaning of the containers.

#### A.2.2.1 Silver stock solution, 1,0 g/l

Weigh 1,572 g ± 0,001 g silver nitrate (A.2.1.1) (< C >) and transfer it to a 1 000 ml one-mark volumetric flask. Make up to the mark with water, stopper the flask and invert several times until well mixed. Store in a brown bottle.

NOTE 4 A 1,000 g/l silver stock solution is available commercially from manufacturers of atomic absorption spectrometers or laboratory suppliers.

#### A.2.2.2 Silver stock solution, 100 mg/l

Pipette 10,0 ml of 1,0 mg/l stock silver solution (A.2.2.1) into a 100 ml one-mark volumetric flask. Make up to the mark with water, stopper the flask and invert several times until well mixed. (It is assumed that the diluted solution is still sufficiently acidic to keep the silver in solution.)

#### A.2.2.3 Silver reference solutions

Label four 100 ml one-mark volumetric flasks as follows: 0,5 mg/l, 1,0 mg/l, 3,0 mg/l and 5,0 mg/l. Pipette the labelled amount (in millilitres) of the 100-mg/l silver stock solution (A.2.2.2) into the respective 100 ml volumetric flasks. Add 1 to 2 drops of ammonium hydroxide (A.2.1.2) (< C > < B >) to each of the flasks to neutralize the solution. Add 1 ml of CNI (A.1) (<< S >> < B > < C >) to each of the flasks and swirl. Make each flask up to the mark



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with water, stopper the flask and invert several times until well mixed.

## A.3 Preparation of silver nitrate standard solutions (0,100 mol/l, 0,010 mol/l and 0,001 mol/l) for PT methods

### A.3.1 Reagent

**A.3.1.1 Silver nitrate ($AgNO_3$)** (DANGER: < C >).

### A.3.2 Procedure

See operational precautions in 5.4.5 for cleaning of the containers.

NOTE 5 Reagent-grade silver nitrate is sufficiently pure to use as a primary standard in this method. The silver nitrate solutions prepared as described in A.3.2.1 to A.3.2.3 do not require standardization.

**A.3.2.1 Standard 0,100 mol/l silver nitrate solution** (16,989 g/l)

Dissolve 16,989 g of silver nitrate (A.3.1.1) (< C >) in 500 ml of water in a 1 000 ml one-mark volumetric flask. Make up to the mark with water and mix well. Store in a brown bottle.

**A.3.2.2 Standard 0,010 mol/l silver nitrate solution** (1,6989 g/l)

Pipette 25,0 ml of 0,100 mol/l silver nitrate solution (A.3.2.1) into a 250 ml one-mark volumetric flask. Make up to the mark with water and mix well. Store in a brown bottle.

**A.3.2.3 Standard 0,001 mol/l silver nitrate solution** (0,16989 g/l)

Pipette 10,0 ml of 0,100 mol/l silver nitrate solution (A.3.2.1) into a 1 000 ml one-mark volumetric flask. Make up to the mark with water and mix well. Store in a brown bottle.

## A.4 Preparation of potassium iodide standard solutions (0,1 mol/l, 0,01 mol/l and 0,001 mol/l)

### A.4.1 Reagents

**A.4.1.1 Potassium iodide (KI)**, reagent grade.

**A.4.1.2 Sulfuric acid ($H_2SO_4$)** (1 + 4).

Stir on a magnetic stirrer 800 ml of water in a 2 000 ml heat-resistant beaker. Slowly and carefully add 200 ml of sulfuric acid, $\rho \approx 1{,}84$ (DANGER: << C >>). Cool to room temperature, transfer to a storage bottle, label and date.

### A.4.2 Procedure

**A.4.2.1 Potassium iodide, 0,100 mol/l solution** (16,7 g/l)

Dissolve 16,7 g of potassium iodide (A.4.1.1) in water. Dilute to 1 000 ml and mix well.

**A.4.2.2 Potassium iodide, 0,010 mol/l solution** (1,67 g/l)

Dissolve 1,67 g of potassium iodide (A.4.1.1) in water. Dilute to 1 000 ml and mix well. Store in a brown bottle.

**A.4.2.3 Potassium iodide, 0,001 mol/l solution** (0,167 g/l)

Dilute 100 ml of 0,01 mol/l potassium iodide solution (A.4.2.2) to 1 000 ml with water and mix well. Prepare this solution within 24 h of use. Store in a brown bottle.

### A.4.3 Standardization of potassium iodide solutions

**A.4.3.1 Standardization of 0,100 mol/l potassium iodide solution** (16,7 g/l)

Pipette 25,0 ml of 0,100 mol/l potassium iodide solution (A.4.2.1) into a 150 ml beaker. Add 25 ml of water and 1 ml of sulfuric acid solution (1 + 4) (A.4.1.2) and titrate potentiometrically with standard 0,100 mol/l silver nitrate solution (A.3.2.1).

Calculate the concentration of the KI solution, $c_{KI}$, from

$$c_{KI} = V_{Ag} \times 0{,}004\ 00$$

where $V_{Ag}$ is the volume of silver nitrate solution, in millilitres, used in the titration.



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**A.4.3.2 Standardization of 0,010 mol/l potassium iodide solution** (1,67 g/l)

Pipette 25,0 ml of 0,010 mol/l potassium iodide solution (A.4.2.2) into a 150 ml beaker. Add 25 ml of water and 1 ml of sulfuric acid (1 + 4) (A.4.1.2) and titrate potentiometrically with standard 0,010 mol/l silver nitrate solution (A.3.2.2).

Calculate the concentration of the KI solution, $c'_{KI}$ from

$$c'_{KI} = V'_{Ag} \times 0{,}000\ 40$$

where $V'_{Ag}$ is the volume of silver nitrate solution, in millilitres, used in the titration.

**A.4.3.3 Standardization of 0,001 mol/l potassium iodide solution** (0,167 g/l)

Pipette 25,0 ml of 0,001 mol/l potassium iodide solution (A.4.2.3) into a 150 ml beaker. Add 25 ml of water and 1 ml of sulfuric acid (1 + 4) (A.4.1.2) and titrate potentiometrically with standard 0,001 mol/l silver nitrate solution (A.3.2.3).

Calculate the concentration of the KI solution, $c''_{Ag}$, from

$$c''_{KI} = V''_{Ag} \times 0{,}000\ 04$$

where $V''_{Ag}$ is the volume of silver nitrate solution, in millilitres, used in the titration.



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