



Food and Agriculture Organization
of the United Nations

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FAO TENTATIVE SPECIFICATIONS FOR PLANT PROTECTION PRODUCTS

PROPINEB
zinc propylenebisdithiocarbamate

FOOD AND AGRICULTURE ORGANIZATION OF THE UNITED NATIONS
Rome, 1980

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FAO specifications are developed with the basic objective of promoting, as far as practicable, the manufacture, distribution and use of pesticides that meet basic quality requirements.

Compliance with the specifications does not constitute an endorsement or warranty of the fitness of a particular pesticide for a particular purpose, including its suitability for the control of any given pest, or its suitability for use in a particular area. Owing to the complexity of the problems involved, the suitability of pesticides for a particular purpose and the content of the labelling instructions must be decided at the national or provincial level.

Furthermore, pesticides which are manufactured to comply with these specifications are not exempted from any safety regulation or other legal or administrative provision applicable to their manufacture, sale, transportation, storage, handling, preparation and/or use.

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¹ This disclaimer applies to all specifications published by FAO.

INTRODUCTION TO FAO SPECIFICATIONS DEVELOPED UNDER THE OLD PROCEDURE

Between 1975 and 2000, FAO published booklets of specifications for technical materials and related formulations of plant protection products. Revisions of, and additions to, already published specifications will be issued when necessary. However, all changes and revisions of FAO specifications are now subject to the new procedure described in the *Manual on the development and use of FAO and WHO Specifications for Plant Protection Products*, FAO Plant Production and Protection Paper No. 173, Rome 2002 (*Revised First Edition* available only on the FAO home page of the Internet at: <http://www.fao.org/ag/agp/agpp/pesticid/>)

FAO specifications developed under the old procedure are based on the requirements defined in the Fourth Edition of the *Manual on the development and use of FAO specifications for plant protection products*, Plant Production and Protection Paper No. 128, Rome 1995.

This manual contained detailed definitions and other essential background information on basic procedures and technical principles adopted by the group on Pesticide Specifications of the FAO Panel of Experts on Pesticide Specifications, Registration Requirements, Application Standards and Prior Informed Consent, such as:

1. Categories of Specifications (Section 3.1 of the Manual)

FAO Tentative Specifications (Code 'S/T', formerly 'TS') are those which have been recommended by FAO as preliminary specifications and which are based on minimum requirements. The methods of analysis cited are normally supplied by the manufacturer or may already have been published or be the subject of collaborative work.

FAO Provisional Specifications [Code 'S/P', formerly ('S')] are those for which more evidence of the necessary parameters is available and where some collaborative study of the methods of analysis has been carried out.

FAO (full) Specifications (Code 'S/F', formerly 'S').

Specifications that have all necessary requirements together with CIPAC (full) methods, or other collaboratively studied (proven) methods.^{2,3}

Wherever possible, standards for apparatus and common names for pesticides are those approved by the International Organization for Standardization (ISO).

2. Expression of active ingredient content (Section 4.2.5 of the Manual)

- for solids, liquid technical materials, volatile liquids (of maximum boiling point 50°C) and viscous liquids (with minimum kinematic viscosity of $1 \times 10^3 \text{ m}^2/\text{s}$ at 20°C) the FAO Specification shall be based on expression of the content as g/kg;

- for all other liquids the active ingredient content of the product shall be declared in terms of g/kg *or* g/l at 20°C. If the customer requires both g/kg *and* g/l at 20°C, then in case of dispute the analytical results shall be calculated as g/kg.

3. Tolerance on content (Section 4.2.7 of the Manual)

A declared content of active ingredient must be included in all specifications, and one of the problems immediately arising is the level of tolerance acceptable about the nominal figure. The tolerance is influenced by (a) the reproducibility of the method of analysis, (b) the sampling error and (c) the manufacturing variance.

Allowable variations in analytical results (i.e. tolerances in content of active ingredient) with respect to specific pesticide consignments are intended to cover reasonable variations in the contents of active ingredients. For examples of such tolerances, see the table in Section 4.2.7 of the Manual.

4. Containers/packaging

FAO guidelines are in preparation.

Containers shall comply with pertinent national and international transport and safety regulations.

Technical materials, dustable powders and granules

Containers shall be suitable, clean, dry and as specified, and shall not adversely affect, or be affected by, the contents, but shall adequately protect them against external conditions.

Wettable powders

The product shall be packed in suitable, clean, dry containers as specified in the order. The container shall provide all necessary protection against compaction, atmospheric moisture, loss by vaporization and/or contamination to ensure that the product suffers no deterioration under normal transit and storage conditions.

The product shall be protected by an adequate moisture barrier. This may be a suitable bag of polyethylene or alternative means of giving equal or better protection.

Solutions and emulsifiable concentrates

Containers shall be lined, where necessary, with a suitable material, or the interior surfaces shall be treated to prevent corrosion and/or deterioration of the contents.

Additional information should be given in all specifications where particular pesticides present problems in packaging.

5. Biological information

Phytotoxicity

No test can be specified to cover the possible phytotoxicity of a formulation to all crops. When a crop is not mentioned in the instructions for use, purchasers should check with the supplier that the material is suitable, always provided that such a use is not restricted or legally forbidden.

Wetting of crops

The dilute spray should satisfactorily wet the leaves of the specified crops when used in accordance with the instructions. Test method MT 53.2, CIPAC F, p.162, may be useful.

¹ *Should national pesticide specifications developed from these approved FAO specifications deviate from them, the National Authority responsible for making such changes is requested to inform the FAO Plant Protection Service of the nature of, and the reasons for, the modifications.*

² *Methods of analysis and miscellaneous techniques referred to in these specifications have been developed and adopted by CIPAC (Collaborative International Pesticides Analytical Council Ltd.). See CIPAC Handbooks 1 (1970), 1A (1980), 1B (1983), 1C (1985), D (1988), E (1993), F (1995), G (1995), CIPAC Proceedings 1980 and 1981, obtainable from Black Bear Press Limited, King's Hedges Road, Cambridge CB4 2PQ, England. The page numbers of specific methods are given in parentheses in the specifications. Copies of methods not yet published can be obtained from the FAO Plant Protection Service.*

³ *Information on standard waters for laboratory evaluation of pesticidal formulations will be found in CIPAC Monograph 1, Standard Waters and an FAO Survey on Naturally Occurring Waters (1972), Black Bear Press Limited, King's Hedges Road, Cambridge CB4 2PQ, England.*

SUBMISSION OF DRAFT SPECIFICATIONS TO FAO

Any organization, commercial firm or interested individual is encouraged to submit relevant specifications, or proposals for revision of existing specifications, for pesticide products for consideration and possible adoption by FAO. Correspondence should be addressed to the Pesticide Management Group, Plant Production and Protection Division, FAO, Viale delle Terme di Caracalla, 00153 Rome, Italy.

General guidelines on preparing draft specifications are given in the *Manual on the development and use of FAO and WHO Specifications for Plant Protection Products*, FAO Plant Production and Protection Paper No. 173, Rome 2002 (Revised First Edition available only on the FAO home page of the Internet at: <http://www.fao.org/ag/agp/agpp/pesticid/>).

Specifications which are considered suitable for further processing are assigned priorities and circulated to appropriate organizations and specialists to comment. Comments, together with other relevant information, are then reviewed in detail by the Group on Specifications of the FAO Panel of Experts on Pesticide Specifications, Registration Requirements, Application Standards and Prior Informed Consent. The drafts are converted into FAO Provisional Specifications, or full FAO Specifications.

PROPINEB TECHNICAL

FAO Tentative Specification 177/1/ts/2 (177/TC/ts/2)

.1 DESCRIPTION

The material shall consist, essentially, of propineb, together with related manufacturing impurities and 6-10 percent of wetting and dispersing agents, as a cream-coloured powder. It shall be free from visible extraneous materials and added modifying agents, except those mentioned above.

.2 ACTIVE INGREDIENTS

.2.1 *Identity* (CIPAC H, p. 236)

Where the identity of the material is in doubt, it shall comply with the test.

.2.2 *Propineb Content* [(CIPAC H, p. 236)

The propineb content shall be declared. (Minimum declared: 80,0%). When the combined carbon disulphide content is determined and expressed as propineb (Note 1), the content obtained shall not differ from that declared by more than ± 3 percentage units.

.2.3 *Zinc* (CIPAC H, p. 236)

Minimum: 21.2%

of the propineb found under .2.2 (Note 2)

Maximum: 23.9%

.3 IMPURITIES (Note 3)

.3.1 *Arsenic*¹

Maximum: 25 ug/g

.3.2 *Water* [CIPAC 1; MT 30.2]

Maximum: 2.5%

Note 1 1.00% carbon disulphide = 1.90% propineb.

Note 2 On a result of 85% propineb the permitted zinc content would be 18.0 to 20.3%.

¹ The analytical method for determination of the relevant impurities is available from the Pesticide Management Group of the FAO Plant Protection Service or can be [downloaded here](#).

Note 3

During the manufacture of propineb technical a small quantity of propylanthiourea (PTU) is also produced. In good manufacturing practice this should not, at the time of manufacture exceed 0.5% of the propineb content found. During storage more PTU may be formed by decomposition. To minimize decomposition all products containing propineb should be stored under cool dry conditions.

PROPINEB DUSTS

FAO Tentative Specification 177/2/ts/2 (177/DP/ts/2)

.1 DESCRIPTION

The product shall consist of a homogeneous mixture containing propineb (complying with 177/1/ts/2) as the active ingredient, together with suitable carriers and any necessary formulants. It shall be a fine, free flowing, dustable material, free from visible extraneous materials and hard lumps.

.2 ACTIVE INGREDIENT

.2.1 Identity Tests (CIPAC H, p. 236)

Where the identity of the active ingredient is in doubt, it shall comply with the test.

.2.2 Propineb Content (CIPAC H, p. 236)

The propineb content shall be declared (Note 1). When the combined carbon disulphide content is determined and expressed as propineb, the content obtained shall not differ from that declared by more than $\pm 10\%$ of the declared content.

.2.3 Zinc [(CIPAC H, p. 236)

Minimum: 21.2%

of the propineb found under .2.2 (Note 2)

Maximum: 23.9%

.3 IMPURITIES (Note 3)

.3.1 Arsenic¹

Maximum: 20 $\mu\text{g/g}$

.3.2 Water [CIPAC 1; MT 30.2]

Maximum: 2.0%

.4 PHYSICAL PROPERTIES

¹ The analytical method for determination of the relevant impurities is available from the Pesticide Management Group of the FAO Plant Protection Service or can be [downloaded here](#).

.4.1 Dry Sieve Test [CIPAC 1; MT 59.1]

Maximum 2% retained on a 75 µm test sieve.

Note more than $(0.06 \times \underline{X})$ % of the sample used for the determination shall be present as propineb in the residue on the sieve, where X is the percentage propineb content declared under .2.2 (Note 4).

*.4.2 Flowability [CIPAC 1; (MT) 44]

Maximum flow number: 12

.5 STORAGE STABILITY

.5.1 Stability at 54°C [CIPAC 1; MT 46.1]

After storage at $54 \pm 2^\circ\text{C}$ for 14 days the product shall continue to comply with .2.2, (except that the permitted minimum content of propineb shall be 90% of that found under .2.2), and .4.1.

.6 CONTAINERS

They shall be suitable, clean, dry and as specified in the order and shall not affect, or be affected by the product but shall adequately protect it from external influences (Note 5). They shall comply with pertinent national and international transport and safety regulations.

.7 PHYSICAL PROPERTIES

*.7.1 Phytotoxicity

At the present stage of our knowledge, no test can be specified to cover phytotoxicity of formulations to crops.

When a crop is not mentioned in the instructions for use, purchasers should check with the supplier that the material is suitable, always provided that such a use is not restricted or legally forbidden.

Note 1 1.00% carbon disulfide = 1.90% propineb

Note 2 On a result of 20% propineb the maximum permitted zinc content would be 4.2 to 4.8%.

Note 3 During the manufacture of propineb technical a small quantity of propylenethiourea (PTU) is also produced. In good manufacturing practice this should not, at the time of manufacturer exceed 0.5% of the propineb content found. During storage more PTU may be formed by decomposition. To minimize decomposition all products containing propineb should be stored under cool, dry conditions.

Note 4 If the dust contains a declared content of 20% propineb and 20 g of sample is used in the test, then the amount of propineb in the residue on the sieve should not exceed 0.24 g i.e.

$$(0.06 \times X) \text{ weight of sample} / 100$$

Note 5 Propineb dusts will deteriorate in the presence of moisture

* For information

PROPINEB DISPERSIBLE POWDERS

FAO Tentative Specification 177/3/ts/2 (177/WP/ts/2)

.1 DESCRIPTION

The product shall consist of a homogeneous mixture containing propineb (complying with 177/1/ts/2) as the active ingredient, together with fillers and any necessary formulants. It shall be a fine powder, free from visible extraneous material and hard lumps.

.2 ACTIVE INGREDIENT

.2.1 Identity Tests (CIPAC H, p. 236)

Where the identity of the active ingredient is in doubt it shall comply with the test.

.2.2 Propineb Content(CIPAC H, p. 236)

The propineb content shall be declared (Note 1). When the combined carbon disulphide content is determined, and expressed as propineb (Note 2), the content obtained shall not differ from that declared by more than ± 3 percentage units.

.2.3 Zinc (CIPAC H, p. 236)

Minimum: 21.2%

Maximum: 23.9 under .2.2 (Note 3).

.3 IMPURITIES (Note 4)

.3.1 Arsenic¹

Maximum: 0.3X $\mu\text{g/g}$, where X is the percentage propineb declared under .2.2 (Note 5).

.3.2 Water [CIPAC 1; MT 30.2]

Maximum: 2.5%

.4 PHYSICAL PROPERTIES

.4.1 Wet Sieve Test [CIPAC 1; MT 59.3]

Maximum: 2% retained on a 75 μm test sieve.

¹ The analytical method for determination of the relevant impurities is available from the Pesticide Management Group of the FAO Plant Protection Service or can be [downloaded here](#).

.4.2 Suspensibility [CIPAC 1; MT 15.1]

A minimum of 70% of the propineb content, declared under .2.2 shall be in suspension after 30 min in CIPAC Water A on the sample as received and not less than 60% in CIPAC Standard Water C after the stability test at 54°C.

Alternatively, if the buyer requires other CIPAC Standard Waters to be used, then this shall be specified when ordering.

.4.3 pH Range of 1% Aqueous Dispersion [CIPAC 1A; MT 75]

5.0 to 9.0

.4.4 Wettability of the Product [CIPAC 1; MT 53.3.1]

Shall be completely wetted in 2 min without swirling.

.4.5 Persistent Foam [CIPAC 1; MT 47]

Maximum: 25 ml after 1 min.

.5 STORAGE STABILITY

.5.1 Stability at 54°C [CIPAC 1; MT 46.1]

After storage at $54 \pm 2^\circ\text{C}$ for 14 days the product shall continue to comply with .2.2 (except that the minimum permitted propineb content shall be 95% of that found under .2.2), .4.1, .4.3 and .4.4.

.6 CONTAINERS

The product shall be packed in suitable, clean, dry containers as specified in the order. The container shall provide all necessary protection against compaction, atmospheric moisture, oxidation, loss by vaporization and/or contamination to ensure that the product suffers no deterioration under normal transit and storage conditions.

The product shall be protected by an adequate moisture barrier. This may be an inner bag of polyethylene (Note 6) or an alternative means of giving equal or better protection. Containers shall comply with pertinent national and international transport and safety regulations.

.7 BIOLOGICAL PROPERTIES

*.7.1 Phytotoxicity

At the present stage of our knowledge, no test can be specified to cover phytotoxicity of formulations to crops.

When a crop is not mentioned in the instructions for use, purchasers should check with the supplier that the material is suitable, always provided that such a use is not restricted or legally forbidden.

*7.2 Wetting of Crops [-/M/1.13]

The dilute spray shall satisfactorily wet the leaves of the specified crops when used in accordance with the instructions. The wetting test specified may be useful.

- Note 1 The propineb content may decrease at the rate of up to 0.5% of the active ingredient per months, e.g. a product containing 70% propineb when manufactured may assay 64.0% at the end of 12 months. In very bad storage conditions or when unsuitable containers have been used, the deterioration may be accelerated.
- Note 2 1.00% carbon disulphide = 1.90% propineb.
- Note 3 On a result of 70% propineb, the permitted zinc content would be 14.8 to 16.7%.
- Note 4 During the manufacture of propineb technical a small quantity of propylenethiourea (PTU) is also produced. In good manufacturing practice this should not, at the time of manufacturer exceed 0.5% of the propineb content found. During storage more PTU may be formed by decomposition. To minimize decomposition all products containing propineb should be stored under cool, dry conditions.
- Note 5 On a declared propineb content of 70% the maximum permitted arsenic content would be 0.3×70 or 21 $\mu\text{g/g}$.
- Note 6 Because of the variation in the nature and size of the container, its destination and other factors, it is not possible to specify the thickness of the polyethylene, but as a guideline for a container with 50 kg product, the inner liner should be at least 0.075 mm thick.

* For information.



2011-0161801-90 / MD-99-013372

ZF-D Analytik Leverkusen
 Building O 13
 D-51368 Leverkusen

Analytical Method
 2011-0161801-90E
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Organic and Inorganic Samples

Arsenic by Hydride Atomic Absorption Spectrometry (HAAS)

As

Arsenic

$M(\text{As}) = 74.92 \text{ g/mole}$

Additional needed method(s):

2085/164/020/16 2011-0340401-92E

Reason for issue:

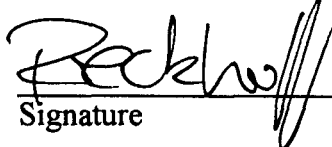
first issue

Replaces method no.:

2011-0141301-90D

Revised by:	Dr. Moehnle	orig.sign.	11.06.1997
	_____	_____	_____
	Name	Signature	Date

Approved by:	Dr. Ritter	orig.sign.	11.06.1997
	_____	_____	_____
	Name	Signature	Date

Translator:	Dr. Rieckhoff		30.06.97
	_____	_____	_____
	Name	Signature	Date

A prerequisite for the use of this analytical method is that the person carrying it out has appropriate training in chemical analysis, is familiar with the safe handling of chemicals and analytical apparatus, and has been informed about general laboratory guidelines relating to safety precautions and accident prevention and strictly observes these.

M-014036-01-2

1 Scope

This method is applicable for the determination of arsenic in aqueous samples or in samples which are soluble in water or acids.

The limit of detection (DL) is 100 µg/kg As or 100 µg/l As. The equation used to calculate DL is $DL = \bar{x}_m + 3 s$ where \bar{x}_m is the mean value of the blanks of the whole analytical procedure, and s is the standard deviation of 8 independent measurements after digestion using sulfuric acid/nitric acid due to a sample weight of 1,5 g or a sample volume of $V \text{ ml} = 1,5 \text{ ml}$.

2 Principles

The arsenic content is determined by hydride atomic absorption spectrometry using the standard addition method. Potassium iodide is added to the sample to reduce As(V) to As(III). In the case of chemical interferences with other ions pyridine-2-carbaldoxime is added.

In samples, prepared by the described procedure, arsenic is reduced to arsenic hydride using potassium borohydride. The arsenic hydride formed in the reaction solution is transferred by argon as carrier gas into a heated quartz glass tube where the dissociation and atomization take place. The signal formed on this occasion is recorded.

3 Chemicals

3.1 Water, deionized

3.2 Arsenic stock solution 1000 mg/l;
e.g. Baker chemicals, no: 6919

3.3 Preparation of the standard solution:
e.g. pipet 100 µl (200 µl) of the arsenic stock solution (3.2) into a 100 ml volumetric flask, add 100 µl nitric acid (3.4) and dilute to volume with water (3.1). This calibration solution contains $\beta(\text{arsenic calibration solution}) = C_0 \text{ mg/kg} = 1,0 \text{ mg/kg} (2,0 \text{ mg/kg})$.

3.4 Nitric acid, subboiling dist.;
w(nitric acid) approx. 65 %

3.5 Hydrochloric acid, subboiling dist.;
w(hydrochloric acid) approx. 35 %

- 3.5.1 Carrier solution for the flow injection analysis system:
Pipet 500 ml water (3.1) into a 1000 ml volumetric flask, add 100 ml hydrochloric acid (3.5) carefully and dilute to volume with water (3.1).
- 3.6 Sodium hydroxide, p. a.;
e.g. Merck, no. 6498
- 3.7 Pyridine-2-carbaldoxime, synthetic grade;
e.g. Fluka, no. 82760
- 3.7.1 Weigh 25 g of pyridine-2-carbaldoxime into a 1000 ml volumetric flask, dissolve the compound in 493 ml hydrochloric acid (3.5) and dilute to volume with water (3.1). The solution contains 2,5 % of pyridine-2-carbaldoxime.
- 3.8 Potassium borohydride;
e.g. Merck, no. 820747
- 3.8.1 Batch technique:
Weigh 30 g potassium borohydride and 10 g sodium hydroxide (4.7) into a 1000 ml volumetric flask, dissolve in water (3.1) and dilute to volume with water (3.1).
- 3.8.2 Flow injection analysis:
Weigh 7,5 g potassium borohydride and 10 g sodium hydroxide (4.7) into a 2500 ml volumetric flask, dissolve in water (3.1) and dilute to volume with water (3.1).
- 3.9 Potassium iodide, p. a.;
e.g. Merck, no. 5043
- 3.9.1 Weigh 10 g potassium iodide into a 100 ml volumetric flask, dissolve in water (3.1) and dilute to volume with water (3.1). The solution contains 10 % of potassium iodide.

4 Equipment

- 4.1 Atomic absorption spectrometer;
e.g. Perkin Elmer 4000 or 2100
- 4.2 Hydride generation for the batch technique;
e.g. Perkin Elmer MHS 20
Hydride generation for the flow injection analysis system;
e.g. Perkin Elmer FIAS 200
- 4.3 Recorder or printer for the documentation of the absorbance signal

- 4.4 Arsenic electrodeless discharge lamp (As EDL)
- 4.5 Different volumetric flasks
- 4.6 Reaction vessels suitable for the respective hydride system
- 4.7 Automatic pipettes
- 4.8 Quartz glass vessels for the wet chemical digestion

5 Instrument Conditions

- 5.1 Wavelength for arsenic:
193,7 nm at a characteristic concentration* of 0,074 ng.
*The characteristic concentration is the concentration of the element causing 1 % absorption (= 0,0044 absorbance).
- 5.2 Temperature of the quartz glass tube: 900 °C
- 5.3 Replicates: 3x
- 5.4 Further instrument conditions are based on the operating instructions and on the analytical method
(Perkin Elmer: 'Analytical methods using the mercury-/hydride system MHS', 310-A5M310/2.80, 092 317, order no. 310;
or
'FIAS 200 flow injection analysis system', software handbook B3503-A1.1-M1350/11.89, order no. B019-9138, hardware handbook B3501, order no. B050-1665).

6 Procedure

- 6.1 Preparation of the sample solution
 - 6.1.1 Aqueous samples which are soluble in diluted acids or water
On the basis of the occurrence of organic arsenic species the samples have to be mineralized by wet chemical digestion under reflux according to analytical method no. 2085/164/020/16.
Weigh 1,5 g (W g) of the solid sample into a quartz glass digestion vessel and record the weight to the nearest 0,1 mg or pipet 1,5 ml (V1 ml) of the liquid sample into a quartz glass digestion vessel using an automatic pipette. Afterwards mineralize the sample according to the analytical method mentioned above, transfer the sample solution by means of water to a

volumetric flask (V ml), dilute to volume with water and analyse the sample solution according to paragraph 4.

6.1.2 Samples which are insoluble in water and diluted acids

Weigh 1,5 g (W g) of the solid sample into a quartz glass digestion vessel and record the weight to the nearest 0,1 mg or pipet 1,5 ml (V1 ml) of the liquid sample into a quartz glass digestion vessel using an automatic pipette. Afterwards mineralize the sample according to the analytical method no. 2085/164/020/16, transfer the sample solution by means of water to a volumetric flask (V ml), dilute to volume with water and analyse the sample solution according to paragraph 4.

6.2 Measurement of the sample solution

Pipet 5 ml of the digestion solution into a reaction vessel (4.6), add 1 ml of the potassium iodide solution (3.9.1), 5 ml of pyridine-2-carbaldoxime solution (3.7.1) and dilute to a volume of 20 ml with water. The time of reaction is 15 min.

Performe the measurement according to paragraph 4. Analyse the total amount of the solution using the batch technique and e.g. 500 µl of the solution using the flow injection analysis system after adding the reduction solution (3.8.1 or 3.8.2) automatically.

The recorded instrument response of the sample solution prepared according paragraph 6.1.1 or 6.1.2 corresponds to absorbance A.

6.3 Preparation of the blank solution

Prepare the blank solution according to paragraph 6.1.1 or 6.1.2 but without the sample.

6.4 Measurement of the blank solution

The analysis of the blank solution is performend according paragraph 6.2. The recorded instrument response of the blank solution prepared according paragraph 6.3 corresponds to absorbance A (BS).

6.5 Calibration

The calibration is performed using the standard addition method.

Pipet 5 ml of the digestion solution (blank solution) into a reaction vessel (4.6), add 1 ml of the potassium iodide solution (3.9.1), 5 ml of pyridine-2-carbaldoxime solution (3.7.1), e.g. 20 µl (40 µl) of the arsenic calibration solution and dilute to a volume of 20 ml with water. The time of reaction is 15 min.

The spiked solution contains e.g. $\beta(\text{arsenic}) = C_0 \mu\text{g/l} = 1,0 \mu\text{g/l} (2 \mu\text{g/l})$.

6.6 Measurement of the calibration solution

The calibration solution (6.5) is analyzed according 3.8.1 or 3.8.2 after automatic addition of the reduction solution.

The recorded instrument response of the calibration solution prepared according paragraph 6.5 corresponds to the total absorbance $AK1 = A01 + A$ or $AK2 = A02 + A$ with the absorbance $A01$ for e.g. 1,0 µg/l As or $A02$ for e.g. 2,0 µg/l As and the absorbance $A(6.2)$ or $A(BS)$ (6.4).

7 Evaluation

Quantify the received peak high (paragraph 6.2, 6.4, 6.6) and record the high to the nearest 1 millimeter and determine the mean values of the replicates.

The measurement of the calibration solution using 2 µg/l arsenic (paragraph 6.6) is used to check the linear dependency between the concentration and absorbance which is necessary for the evaluation. In the case of linearity the mean value of the total absorbance $AK1 = A01 + A$ is used for evaluation.

The mean value of the replicates of the arsenic content of the blank solution has to be subtracted from the mean value of the replicates of the arsenic content of the sample solution (remark 9.2).

8 Calculations

Calculate the arsenic content in µg/kg in the sample as follows:

$$w(As) = \left[\frac{CO * V * A}{A01} - \frac{CO * V * A(BP)}{A01} \right] * \frac{f}{W} \text{ µg/kg}$$

Calculate the arsenic content in µg/l in the sample as follows:

$$\beta(As) = \left[\frac{CO * V * A}{A01} - \frac{CO * V * A(BP)}{A01} \right] * \frac{f}{W} \text{ µg/l}$$

w(As):	Arsenic content in the sample [$\mu\text{g}/\text{kg}$]
$\beta(\text{As})$:	Concentration of arsenic in the sample [$\mu\text{g}/\text{l}$]
C0:	Standard solution with 1 $\mu\text{g}/\text{l}$ As
A:	Absorbance of the sample solution (6.2)
A(BS):	Absorbance of the blank solution (6.4)
A01:	Absorbance of the standard solution with 1 $\mu\text{g}/\text{l}$ As according paragraph 6.6 (A01 = AK1-A)
W:	weight of sample [g] (paragraph 6.1.1 or 6.1.2)
V1:	Volume of the sample [ml] (paragraph 6.1.1 or 6.1.2)
V:	Final volume [ml] used for the measurement, e.g. V = 0,02 l according to paragraphs 6.1.1, 6.1.2, 6.2, 6.3 and 6.4.
f:	Dilution factor, e.g. f = 20 according to paragraphs 6.1.1, 6.1.2, 6.2, 6.3 and 6.4.

9 Remarks

- 9.1 The reduction solution 3.8.1 and 3.8.2 have to prepared freshly each day.
- 9.2 The evaluation and calculation of the concentration of the sample solution can also be performed using the computer programm (software) of the atomic absorption spectrometer.

10 Typical report (e.g. for Perkin Elmer 4000)

