



TO: Codex Contact Points
Contact Points of international organizations having observer status with Codex

FROM: Secretariat, Codex Alimentarius Commission,
Joint FAO/WHO Food Standards Programme

SUBJECT: Request for information relating to methods of analysis / examples of methods of analysis

DEADLINE: 31 March 2024

BACKGROUND

1. CCMAS42, when discussing endorsement of methods from several committees, took decisions as follows:

Review of methods of analysis for contaminants: performance criteria for lead and cadmium in foods

CCMAS agreed to:

- a. develop performance criteria for lead and cadmium in foods with the subsequent revocation of the *General Methods of Analysis for Contaminants* (CXS 228-2001) and methods in CXS 234 for lead and cadmium for commodities covered by the performance criteria¹. CAC46 adopted the performance criteria for inclusion in CXS 234 and revoked CXS 228-2001 and relevant methods in CXS234. The performance criteria have been reproduced in Appendix I to this CL.
- b. continue to review methods (see Appendix II to this CL) and other methods to identify examples of available methods that meet the performance criteria.

Note: CCMAS41 developed performance criteria for lead in butter, edible casein and whey powders (secondary milk products). The methods for lead in butter and edible casein were retained in CXS234 until review by CCMAS to determine if they meet the performance criteria. These methods have been included in Appendix II and the performance criteria in Appendix I.

Review of methods of analysis for irradiated foods in the *General Methods for the Detection of Irradiated Foods* (CXS 231-2001) and their incorporation into CXS 234

- a. CCMAS agreed that the methods from CXS 231 as recommended by CCFH53 (CCMAS42/CRD02, Appendix II) not be endorsed due to insufficient information available on the methods and their application and as a consequence to maintain the methods in CXS 231; and to gather additional information on the methods for further consideration.²
- b. The method proposals from CCFH53 are reproduced in Appendix III to this CL.

Performance criteria – sum of components: for methods to determine MLs for aflatoxins in certain cereals and cereal-based products including foods for infants and young children

- a. CCMAS endorsed the performance criteria (included in the sampling plans for total aflatoxins in certain cereals and cereal-based products including foods for infants and young children)³ which was adopted by CAC46. CCMAS43 will consider example methods that meet these performance criteria. The performance criteria have been reproduced in Appendix IV to this CL.

Note: the performance criteria are described in the sampling plans for total aflatoxins in certain cereals and cereal-based products including foods for infants and young children. The full sampling plan is available in Appendix II, [REP23/MAS](#).

¹ REP23/MAS, para. 11; REP23/CAC, para. 92

² REP23/MAS, para. 12

³ REP23/MAS, para. 34, REP23/CAC, para. 92

Methods of analysis for determination of moisture content in dried milk

- a. CCMAS recalled that CCMAS41 could not reach consensus on the method ISO 5537 | IDF 26 for determination of moisture content in dried milk. At CCMAS42 an alternative proposal was made together with performance criteria (see CX/MAS 23/42/3, Appendix II, Annex 2). The proposal was for CCMAS to endorse the methods as Type I for the determination of moisture in blend of skimmed milk and vegetable fat in powdered form, reduced fat blend of skimmed milk powder and vegetable fat in powdered form, dairy permeate powders, milk powders and cream powders and whey powders.
- b. The PWG on endorsement could not reach consensus on the aforementioned proposal but noting that the Procedural Manual did not preclude having a Type IV method when there was a Type I method identified for the same provision/commodity combination, this should be done on an exceptional basis only and should be fully justified.
- c. CCMAS agreed to endorse the method as Type IV for the matrices in REP23/MAS, Appendix II Part 1.6 except for dairy permeate and whey powders.⁴ CAC46 adopted the method⁵.
- d. CCMAS43 will consider the applicability of the method for dairy permeate and whey powders based on further information and data on applicability of this method to these matrices. The method is available in Appendix V to this CL.

REQUEST FOR COMMENTS

2. Codex Members and Observers are invited to submit:
 - a. Comments and information on i) the suitability of methods in Appendix II as example methods that can meet the performance criteria listed in Appendix I; and ii) other methods that can meet the performance criteria list in Appendix I.
 - b. Information on methods in CXS 231 (See Appendix III) to determine whether they are still in use and “fit for purpose” to allow endorsement and incorporation into CXS 234 and subsequent revocation of CXS 231.
 - c. Information on example methods that meet the performance criteria for methods for determination of MLs for aflatoxins in certain cereals and cereal-based products including foods for infants and young children. The performance criteria are provided in Appendix IV.
 - d. Information / data on the applicability of the method to determine moisture content in dried milk (Appendix V) for dairy permeate and whey powders.
 - e. Any other methods for provisions in Codex commodity standards developed by committees adjourned sine die for consideration / endorsement by the PWG on endorsement and CCMAS43⁶.

Note: methods of analysis for commodity standards being developed in active Codex committees should be submitted directly to those Codex committees.

3. Codex members and observers are invited to take into account where applicable the “[comprehensive guidance for the process of submission, consideration and endorsement of methods for inclusion in CXS 234](#)” when submitting information / proposals for methods of analysis.

GUIDANCE ON THE PROVISION OF COMMENTS

4. Comments should be submitted through the Codex Contact Points of Codex Members and Observers using the OCS.
5. Contact Points of Codex Members and Observers may login to the OCS and access the document open for comments by selecting “Enter” in the “My reviews” page, available after login to the system.
6. Guidance on the OCS comment categories and types can be found in the OCS [Frequently Asked Questions \(FAQs\)](#).
7. Other OCS resources, including the user manual and short guide, can be found at the following link: <http://www.fao.org/fao-who-codexalimentarius/resources/circular-letters/en/>.
8. For questions on the OCS, please contact Codex-OCS@fao.org.

⁴ Full discussion and rationale are provided for this decision in REP23/MAS, paras 35 -42;

⁵ REP23/CAC, para. 92

⁶ REP23/MAS, para. 63

Appendix I

Performance Criteria for Lead and Cadmium in Foods

Commodity	Provision	ML (mg/kg)	Method performance criteria						Principle
			Minimum applicable range (mg/kg)	Limit of Detection (LOD) (mg/kg)	Limit of Quantification (LOQ) (mg/kg)	Precision (RSDR) (%) No more than	Recovery (%)	Example of applicable methods that meet the criteria ⁷	
Natural mineral waters	lead	0.01	0.006 - 0.014	0.002	0.004	44	60-115%		
Infant formula, formula for special medical purposes intended for infants and follow-up formula	lead	0.01	0.006 - 0.014	0.002	0.004	44	60-115%		
Milk	lead	0.02	0.011 - 0.029	0.004	0.008	44	60-115%		
Secondary milk products	lead	0.02	0.011 - 0.029	0.004	0.008	44	60-115%		
Fruit juices, except juices exclusively from berries and other small fruits	lead	0.03	0.017 - 0.043	0.006	0.012	44	60-115%		
Fat spreads and blended spreads	lead	0.04	0.022 - 0.058	0.008	0.016	44	60-115%		
Grape juice	lead	0.04	0.022 - 0.058	0.008	0.016	44	60-115%		

⁷ Example methods will be reviewed by CCMAS43

Commodity	Provision	ML (mg/kg)	Method performance criteria						
			Minimum applicable range (mg/kg)	Limit of Detection (LOD) (mg/kg)	Limit of Quantification (LOQ) (mg/kg)	Precision (RSDR) (%) No more than	Recovery (%)	Example of applicable methods that meet the criteria ⁷	Principle
Canned chestnuts and canned chestnuts puree	lead	0.05	0.028 - 0.072	0.010	0.020	44	60-115%		
Fruit juices obtained exclusively from berries and other small fruits, except grape juice	lead	0.05	0.028 - 0.072	0.010	0.020	44	60-115%		
Fruiting vegetables, except fungi and mushrooms	lead	0.05	0.028 - 0.072	0.010	0.020	44	60-115%		
Preserved tomatoes	lead	0.05	0.028 - 0.072	0.010	0.020	44	60-115%		
Edible fats and oils	lead	0.08	0.045 - 0.115	0.016	0.032	44	60-115%		
Berries and other small fruits, except cranberry, currant, and elderberry	lead	0.1	0.03 - 0.17	0.01	0.02	44	80-110%		
Brassica vegetables, except kale and leafy Brassica vegetables	lead	0.1	0.03 - 0.17	0.01	0.02	44	80-110%		
Bulb vegetables	lead	0.1	0.03 - 0.17	0.01	0.02	44	80-110%		
Canned fruits	lead	0.1	0.03 - 0.17	0.01	0.02	44	80-110%		

Commodity	Provision	ML (mg/kg)	Method performance criteria						Principle
			Minimum applicable range (mg/kg)	Limit of Detection (LOD) (mg/kg)	Limit of Quantification (LOQ) (mg/kg)	Precision (RSDR) (%) No more than	Recovery (%)	Example of applicable methods that meet the criteria ⁷	
Canned vegetables	lead	0.1	0.03 - 0.17	0.01	0.02	44	80-110%		
Fruits, except cranberry, currants, and elderberry	lead	0.1	0.03 - 0.17	0.01	0.02	44	80-110%		
Legume vegetables	lead	0.1	0.03 - 0.17	0.01	0.02	44	80-110%		
Meat and fat of poultry	lead	0.1	0.03 - 0.17	0.01	0.02	44	80-110%		
Meat of cattle, pigs and sheep	lead	0.1	0.03 - 0.17	0.01	0.02	44	80-110%		
Pickled cucumbers (cucumber pickles)	lead	0.1	0.03 - 0.17	0.01	0.02	44	80-110%		
Poultry, edible offal of	lead	0.1	0.03 - 0.17	0.01	0.02	44	80-110%		
Pulses	lead	0.1	0.03 - 0.17	0.01	0.02	44	80-110%		
Root and tuber vegetables	lead	0.1	0.03 - 0.17	0.01	0.02	44	80-110%		
Wine from grapes harvested after July 2019	lead	0.1	0.03 - 0.17	0.01	0.02	44	80-110%		

Commodity	Provision	ML (mg/kg)	Method performance criteria						Principle
			Minimum applicable range (mg/kg)	Limit of Detection (LOD) (mg/kg)	Limit of Quantification (LOQ) (mg/kg)	Precision (RSDR) (%) No more than	Recovery (%)	Example of applicable methods that meet the criteria ⁷	
Fortified / Liqueur wine from grapes harvested after 2019	lead	0.15	0.05 - 0.25	0.015	0.03	43	80-110%		
Pig, edible offal of	lead	0.15	0.05 - 0.25	0.015	0.03	43	80-110%		
Cattle, edible offal of	lead	0.2	0.08 - 0.32	0.02	0.04	41	80-110%		
Cereal grains, except buckwheat, cañihua and quinoa	lead	0.2	0.08 - 0.32	0.02	0.04	41	80-110%		
Cranberry	lead	0.2	0.08 - 0.32	0.02	0.04	41	80-110%		
Currants	lead	0.2	0.08 - 0.32	0.02	0.04	41	80-110%		
Elderberry	lead	0.2	0.08 - 0.32	0.02	0.04	41	80-110%		
Wine (wine and fortified / liqueur wine) made from grapes harvested before July 2019	lead	0.2	0.08 - 0.32	0.02	0.04	41	80-110%		
Fish	lead	0.3	0.13 - 0.47	0.03	0.06	38	80-110%		

Commodity	Provision	ML (mg/kg)	Method performance criteria						
			Minimum applicable range (mg/kg)	Limit of Detection (LOD) (mg/kg)	Limit of Quantification (LOQ) (mg/kg)	Precision (RSDR) (%) No more than	Recovery (%)	Example of applicable methods that meet the criteria ⁷	Principle
Fresh farmed mushrooms (common mushrooms (<i>Agaricus bisporus</i>), shiitake mushrooms (<i>Lentinula edodes</i>), and oyster mushrooms (<i>Pleurotus ostreatus</i>))	lead	0.3	0.13 - 0.47	0.03	0.06	38	80-110%		
Leafy vegetables, except spinach	lead	0.3	0.13 - 0.47	0.03	0.06	38	80-110%		
Jams, jellies, and marmalades	lead	0.4	0.18 - 0.62	0.04	0.08	37	80-110%		
Mango chutney	lead	0.4	0.18 - 0.62	0.04	0.08	37	80-110%		
Table olives	lead	0.4	0.18 - 0.62	0.04	0.08	37	80-110%		
Salt, food grade	lead	1	0.5 - 1.5	0.1	0.2	32	80-110%		
Natural mineral waters	cadmium	0.003	0.0017 - 0.0043	0.0006	0.0012	44	40-120%		
Brassica vegetables, except Brassica leafy vegetables	cadmium	0.05	0.03 - 0.07	0.01	0.02	44	60-115%		

Commodity	Provision	ML (mg/kg)	Method performance criteria						
			Minimum applicable range (mg/kg)	Limit of Detection (LOD) (mg/kg)	Limit of Quantification (LOQ) (mg/kg)	Precision (RSDR) (%) No more than	Recovery (%)	Example of applicable methods that meet the criteria ⁷	Principle
Bulb vegetables	cadmium	0.05	0.03 - 0.07	0.01	0.02	44	60-115%		
Fruiting vegetables, except tomatoes and edible fungi	cadmium	0.05	0.03 - 0.07	0.01	0.02	44	60-115%		
Cereal grains, except buckwheat, cañihua, quinoa, wheat and rice	cadmium	0.1	0.03 - 0.17	0.01	0.02	44	80-110%		
Legume vegetables	cadmium	0.1	0.03 - 0.17	0.01	0.02	44	80-110%		
Pulses, except soya bean (dry)	cadmium	0.1	0.03 - 0.17	0.01	0.02	44	80-110%		
Root and tuber vegetables, except celeriac	cadmium	0.1	0.03 - 0.17	0.01	0.02	44	80-110%		
Stalk and stem vegetables	cadmium	0.1	0.03 - 0.17	0.01	0.02	44	80-110%		
Leafy vegetables	cadmium	0.2	0.08 - 0.32	0.02	0.04	41	80-110%		
Wheat (common wheat, durum wheat, spelt and emmer)	cadmium	0.2	0.08 - 0.32	0.02	0.04	41	80-110%		

Commodity	Provision	ML (mg/kg)	Method performance criteria						
			Minimum applicable range (mg/kg)	Limit of Detection (LOD) (mg/kg)	Limit of Quantification (LOQ) (mg/kg)	Precision (RSDR) (%) No more than	Recovery (%)	Example of applicable methods that meet the criteria ⁷	Principle
Chocolate containing or declaring < 30% total cocoa solids on a dry matter basis	cadmium	0.3	0.13 - 0.47	0.03	0.06	38	80-110%		
Rice, polished	cadmium	0.4	0.18 - 0.62	0.04	0.08	37	80-110%		
Salt, food grade	cadmium	0.5	0.23 - 0.77	0.05	0.10	36	80-110%		
Chocolate containing or declaring ≥30% to <50% total cocoa solids on a dry matter basis	cadmium	0.7	0.35 - 1.05	0.07	0.14	34	80-110%		
Chocolate containing or declaring ≥50% to <70% total cocoa solids on a dry matter basis, including sweet chocolate, Gianduja chocolate, semi – bitter table chocolate, Vermicelli chocolate / chocolate flakes, and bitter table chocolate	cadmium	0.8	0.40 - 1.20	0.08	0.16	33	80-110%		

Commodity	Provision	ML (mg/kg)	Method performance criteria						
			Minimum applicable range (mg/kg)	Limit of Detection (LOD) (mg/kg)	Limit of Quantification (LOQ) (mg/kg)	Precision (RSDR) (%) No more than	Recovery (%)	Example of applicable methods that meet the criteria ⁷	Principle
Chocolate containing or declaring ≥70% total cocoa solids on a dry matter basis, including sweet chocolate, Gianduja chocolate, semi – bitter table chocolate, Vermicelli chocolate / chocolate flakes, and bitter table	cadmium	0.9	0.46 - 1.34	0.09	0.18	33	80-110%		
Cephalopods	cadmium	2	1.1 - 2.9	0.2	0.4	29	80-110%		
Marine bivalve molluscs (clams, cockles and mussels), except oysters and scallops	cadmium	2	1.1 - 2.9	0.2	0.4	29	80-110%		

Performance criteria for lead in butter, edible casein and whey powders (developed by CCMAS41, adopted by CAC44 and included in CXS234)

Commodity	Provision	ML (mg/kg)	LOD (mg/kg)	LOQ (mg/kg)	RSDR (%)	Recovery	Minimum applicable range		Examples of applicable methods that meet the criteria	Principle
							Minimum	Maximum		
Butter, edible casein products and whey powders, (secondary milk products)	Lead	0.02	0.004	0.008	≤ 44	60-115%	0.011	0.029	-	-

Appendix II

Analytical methods for lead for review

(These methods will be removed from CXS 234 and transferred to the column of “example of applicable methods that meet the criteria”, if they meet the performance criteria as presented in Appendix I)

<i>Commodity</i>	<i>Provision</i>	<i>Method</i>	<i>Principle</i>	<i>Type</i>
Fats and Oils and Related Products				
Fats and Oils (all)	Lead	AOAC 994.02 / ISO 12193 / AOCS Ca 18c-91	Atomic absorption spectrophotometry (direct graphite furnace)	II
Named Vegetable Oils	Lead	AOAC 994.02 / ISO 12193 / AOCS Ca 18c-91	Atomic absorption spectrophotometry (direct graphite furnace)	II
Olive Oils and Olive Pomace Oils	Lead	AOAC 994.02 or ISO 12193 or AOCS Ca 18c-91	AAS	II
Butter	Lead	AOAC 972.25 (Codex general method)	Atomic absorption spectrophotometry	IV
Edible casein products	Lead	NMKL 139 (Codex general method) AOAC 999.11	Atomic absorption spectrophotometry	IV
Edible casein products	Lead	NMKL 161 / AOAC 999.10	Atomic absorption spectrophotometry	IV
Edible casein products	Lead	ISO/TS 6733 IDF/RM 133	Spectrophotometry (1,5-diphenylthiocarbazone)	IV
Processed Fruits and Vegetables				
Table olives	Lead	AOAC 999.11 NMKL 139 (Codex general method)	AAS (Flame absorption)	II
Miscellaneous Products				
Food grade salt	Lead	EuSalt/AS 015	ICP-OES	III
Food grade salt	Lead	EuSalt/AS 013	Atomic absorption spectrophotometry	IV

Appendix III

General Methods for the Detection of Irradiated Foods

(for review and possible inclusion in CXS 234)

(New texts added are shown in **bold/underlined** font. Texts proposed for deletion are shown in ~~strike through~~ (as proposed by CCFH53))

Commodity	Provision	Method	Principle	Type
Food containing fat (<u>e.g. raw meat and chicken, cheese, fruits</u>)	Detection of irradiated food - <u>Detection of radiation-induced hydrocarbons</u>	EN 1784 : 1996	Gas chromatographic analysis of hydrocarbons	Type II
Food containing fat (<u>e.g. raw meat and chicken, liquid whole egg</u>)	Detection of irradiated food - <u>Detection of radiation-induced 2-alkylcyclobutanones</u>	EN 1785 ¹ : 1996	Gas chromatographic/mass spectrometric analysis of 2-alkylcyclobutanones	Type III
Food containing bone	Detection of irradiated food - <u>Radiation induced Electron Spin Resonance (ESR) signal attributed to hydroxyapatite (principal component of bones)</u>	EN 1786: 1996	ESR spectroscopy	Type II
Food containing cellulose (<u>e.g. nuts and spices</u>)	Detection of irradiated food - <u>Radiation induced Electron Spin Resonance (ESR) signal attributed to crystalline cellulose</u>	EN 1787: 2000	ESR spectroscopy	Type II
Food containing silicate minerals (<u>e.g. herbs, spices, their mixtures and shrimps</u>)	Detection of irradiated food - <u>Thermoluminescence glow ratio used to indicate the irradiation treatment of the food</u>	EN 1788:2004	Thermoluminescence	Type II
Food containing silicate minerals (<u>e.g. shellfish, herbs, spices, seasonings</u>)	Detection of irradiated food - <u>Measurement of photostimulated luminescence intensity</u>	EN 13751 ² : 2002	Photostimulated luminescence	Type III
Food containing crystalline sugar (<u>e.g. dried fruits and raisins</u>)	Detection of irradiated food - <u>Radiation induced Electron Spin Resonance (ESR) signal attributed to crystalline sugar</u>	EN 13708:2004	ESR spectroscopy	Type II

Commodity	Provision	Method	Principle	Type
Herbs <u>and</u> spices and raw minced meat ³	Detection of irradiated food - <u>Difference between total microorganism count and viable microorganism count</u>	EN 13783:2004 NMKL 231 (2002)	Direct Epifluorescent Filter Technique/Aerobic Plate Count (DEFT/APC) (screening method)	Type III
Food containing DNA (e.g. food products, <u>both of animal and plant origin such as various meats, seeds, dried fruits and spices</u>)	Detection of irradiated food - <u>Detection of DNA fragmentation presumptive to irradiation treatment.</u>	EN 13784:2004	DNA comet assay (screening method)	Type III

Notes

¹ One Member noted that 2-alkylcyclobutanone was also present in some non-irradiated foods and hence EN1785 may need further consideration as a method for detection of irradiated foods.

² Consideration should be given to whether EN13751 should be specified as a screening method.

³ No information was found on validation of the method for this commodity.

Appendix IV

Performance criteria for methods for determination of MLs for aflatoxins in certain cereals and cereal-based products including foods for infants and young children

(Method criteria for total aflatoxins in cereals, considering AFB1: AFB2:AFG1:AFG2 of 1:1:1:1.)

Commodity	Analyte	ML (µg/kg)	LOD (µg/kg)	LOQ (µg/kg)	Precision (%)	Minimal applicable range (µg/kg)	Recovery (%)
Maize grain	AF B1+B2+G1+G2	15	≤ 3	≤ 6	<44	8.4 - 21.6	60-115
	AFB1	-	≤0.75	≤ 1.5	<44	2.1 – 5.4	40-120
	AFB2	-	≤0.75	≤1.5	<44	2.1 – 5.4	40-120
	AFG1	-	≤0.75	≤ 1.5	<44	2.1 – 5.4	40-120
	AFG2	-	≤0.75	≤ 1.5	<44	2.1 – 5.4	40-120
Maize flour, meal, semolina and flakes derived from maize; Sorghum grain; cereal-based foods for infants and young children for food aid programs	AF B1+B2+G1+G2	10	≤2	≤4	<44	5.6 - 14.4	60-115
	AFB1	-	≤0.5	≤1.0	<44	1.4 - 3.6	40-120
	AFB2	-	≤0.5	≤1.0	<44	1.4 - 3.6	40-120
	AFG1	-	≤0.5	≤1.0	<44	1.4 - 3.6	40-120
	AFG2	-	≤0.5	≤1.0	<44	1.4 - 3.6	40-120
Husked Rice	AF B1+B2+G1+G2	20	≤4	≤8	<44	11.2 - 28.8	60-115
	AFB1	-	≤1.0	≤2.0	<44	2.8 – 7.2	40-120
	AFB2	-	≤1.0	≤2.0	<44	2.8 – 7.2	40-120
	AFG1	-	≤1.0	≤2.0	<44	2.8 – 7.2	40-120
	AFG2	-	≤1.0	≤2.0	<44	2.8 – 7.2	40-120
Polished Rice; Cereal-based food for infants and young children	AF B1+B2+G1+G2	5	≤1	≤2	<44	2.8 - 7.2	40-120
	AFB1	-	≤0.25	≤0.5	<44	0.7 – 1.8	40-120
	AFB2	-	≤0.25	≤0.5	<44	0.7 – 1.8	40-120

	AFG1	-	≤ 0.25	≤ 0.5	< 44	0.7 – 1.8	40-120
	AFG2	-	≤ 0.25	≤ 0.5	< 44	0.7 – 1.8	40-120

Determination of Moisture Content in Dried Milk

(note: review applicability of this method for dairy permeate and whey powder)

Products	Parameter
Powdered milk, Powdered cream, and Blend of skimmed milk powder with vegetable fat	Moisture
<p>DESCRIPTION OF THE METHOD: DETERMINATION OF MOISTURE</p> <p>SCOPE</p> <p>This Standard specifies a method for the determination of moisture content for all types of powdered milk, powdered cream, and mixtures of powdered skimmed milk with vegetable fat.</p> <p>DEFINITION</p> <p>The content is the mass loss determined by the procedure specified in this Standard. It is expressed in percentage by mass g/100 g.</p> <p>PRINCIPLE</p> <p>A portion of the sample is dried in an oven set at (102 ± 2) °C until constant weight and weighed to determine the loss of mass.</p> <p>EQUIPMENT</p> <p>Common laboratory equipment and, in particular, the following.</p> <p>4.1 Analytical balance, capable of weighing with a precision of 1 mg, with a minimum resolution of 0.1 mg.</p> <p>4.2 Drying oven, with good ventilation, as far as possible with forced ventilation, capable of being thermostatically maintained at (102 ± 2) °C throughout the workspace, with a temperature controller.</p> <p>4.3 Desiccator, with freshly dried silica gel with hygrometric indicator or another effective desiccant.</p> <p>4.4 Flat-bottomed dishes, approximately 25 mm deep, approximately 50 mm in diameter, and made of an appropriate material (for example, glass, stainless steel, nickel, or aluminium), fitted with tight-fitting, removable lids easily.</p> <p>SAMPLING</p> <p>It is important that the laboratory receive a truly representative sample and that it has not been damaged or changed during transport or storage.</p> <p>Sampling is not part of the method specified in this Standard. A recommended sampling method is provided in ISO 707 IDF 50.</p>	

TEST SAMPLE PREPARATION

Transfer the entire sample to a dry, tightly closed container with a capacity of approximately twice the volume of the sample. Mix thoroughly by turning and shaking the container.

7. PROCEDURE**7.1 Preparation of the dish**

7.1.1 Heat the uncovered capsule and its lid (4.4) in the oven (4.2) controlled at $(102 \pm 2)^{\circ}\text{C}$, for 1 h.

7.1.2 Transfer the capped dish to the desiccator (4.3), allow it to cool to room temperature in the balance room, and weigh (4.1) to the nearest 0.1 mg.

7.2 Test sample

7.2.1 Place 1 - 1.5 g of the prepared test sample (6) in the dish, cover with the lid and weigh to the nearest 0.1 mg.

7.3 Determination

7.3.1 Uncover the capsule and place it together with the lid in the oven (4.2), controlled at $(102 \pm 2)^{\circ}\text{C}$ for 2 hrs.

7.3.2 Replace the cap, transfer the capped dish to the desiccator, allow to cool to balance room temperature, and weigh to the nearest 0.1 mg.

7.3.3 Uncover the capsule and heat again, along with its lid, on the oven for 1 h. Then repeat operation 7.3.2.

7.3.4 Repeat this process until the difference in mass between two successive weighings does not exceed 0.5 mg. Record the lowest mass.

CALCULATION AND EXPRESSION OF RESULTS**8.1 Calculation**

The moisture content in the sample, expressed in g/100 g, is equal to:

$$\text{moisture} = \frac{(m_1 - m_2)}{(m_1 - m_0)} \times 100$$

$$(m_1 - m_0)$$

where,

m_0 is the mass, in grams, of the dish and lid (7.1.2)

m_1 is the mass, in grams, of the dish, lid and test sample before drying (7.2.1)

m_2 is the mass, in grams, of the dish, lid and test sample after drying (7.3.4)

8.2 Expression of test results

Express the sample results to two decimal places.