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Printing ink for food wrappers, packages and receptacles — Specification

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Foreword

Uganda National Bureau of Standards (UNBS) is a parastatal under the Ministry of Trade, Industry and Cooperatives established under Cap 327, of the Laws of Uganda, as amended. UNBS is mandated to co-ordinate the elaboration of standards and is

(a) a member of International Organisation for Standardisation (ISO) and

(b) a contact point for the WHO/FAO Codex Alimentarius Commission on Food Standards, and

(c) the National Enquiry Point on TBT Agreement of the World Trade Organisation (WTO).

The work of preparing Uganda Standards is carried out through Technical Committees. A Technical Committee is established to deliberate on standards in a given field or area and consists of key stakeholders including government, academia, consumer groups, private sector and other interested parties.

Draft Uganda Standards adopted by the Technical Committee are widely circulated to stakeholders and the general public for comments. The committee reviews the comments before recommending the draft standards for approval and declaration as Uganda Standards by the National Standards Council.

The committee responsible for this document is Technical Committee UNBS/TC 301, Chemistry.

Introduction

Food packaging is used to provide information to the final consumer and plays an important role in the presentation and advertising of foodstuffs. Some of this information is legally required, such as weight, vendor details, information about composition, presence of allergens and nutritional details, etc. In addition, printing is carried out for decorative and protective purposes. There are exceptional instances where printing inks are applied to the inner side of the packaging or on inserts, e.g. for promotional purposes, and intentionally have direct food contact. Direct food contact inks represent a special case and comprise less than 1% of food packaging applications.

Printing ink for food wrappers, packages and receptacles — Specification

1 Scope

This Draft Uganda Standard specifies the requirements, sampling and test methods for printing inks for food wrappers, packages and receptacles.

This Standard does not cover non pigment based printing inks such as dye-based and UV printing inks.

2 Normative references

The following referenced documents referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

US ISO 1524, Paints and varnishes — Determination of fineness of grind

US ISO 8124-3, Safety of toys — Part 3: Migration of certain elements (3rd Edition)

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at http://www.iso.org/obp

3.1
Food package
Any material for concealing food, additional to food wrappers

3.2
Food Wrapper
material used to conceal food with which the food is in direct contact.

3.3
pigment
coloured particulate matter suspended in the ink in a carrier liquid

3.4
soluble metal content of ink
metal content of the pigment that is soluble in a defined dilute acid plus the total metal content present in the liquid portion of the ink.

3.5
food receptacle
containment vessel (or container) that is used for storing, holding and carrying food (including water for domestic use).

4. Description of printing inks

The inks for food wrappers, packages and receptacles shall be of the following categories

4.1 solvent based printing inks- suitable for all the three food packaging materials

4.2 water based printing inks- suitable for food packages

5. Requirements

5.1 General Requirement

5.1.1 The printing ink for food wrappers, packages and receptacles shall be based on pigments that are finely dispersed in the ink.

5.1.2 The ink shall be suitable for the substrate it is printed on.

5.1.3 The ink shall contain a suitable solvent(s) that is able to dissolve the resins, be stable during printing and ensure good drying of the ink.

5.1.4 The ink shall not contain more than 10 parts by weights of additives- change to 5.1.4.

5.1.5 The ink shall meet the requirements for food safety

5.2 Specific requirements

The printing ink for food wrappers, packages and receptacles shall meet the specific requirements prescribed in table 1 when tested in accordance with the test methods specified therein

Table 1— Specific requirements for printing ink for food wrappers, packages and receptacles

Parameter	Requirements	Test method
Fineness of grind, µm, max	5	US ISO 1524
Solvent content, %, m/m	< 70	Annex A
Pigment content, % m/m, max	20	Annex B

5.3 Heavy metal contaminants

The printing ink for food wrappers, packages and receptacles shall comply with the limits for heavy metal contaminants given in Table 2 when tested in accordance with the test methods specified therein.

Table 2 — Limits for Heavy metal contaminants

S/No.	Parameter	Maximum limit ,mg/kg	Test method
i	Lead	250	Annex C
ii	Antimony	250	Annex D
iii	Barium	500	Annex E
iv	Cadmium	100	Annex F
v	Chromium	100	US ISO 8124-3
vi	Arsenic	100	

6 Packaging

The printing ink for food wrappers, packages and receptacles shall be packaged in suitable containers that permits the product's integrity.

7 Labelling

The inks shall be labelled with the following information:

a. Manufacturer's name and the recognized trade-mark, if any;

b. Name of product as printing ink

c. Type of ink, as solvent or water based

d. Lot number or batch number

e. Country of origin and

f. Physical address of the manufacturer

g. Date of manufacture

h. Expiry date

i. Instructions for use, storage and disposal marked on the container or enclosed in the pack;

j. The words, Suitable for use on food wrappers, packages and receptacles.

k. List of ingredients

Annex A
(normative)

Determination of solvent content

A.1 Requirements

A.1.1 Weighing scale

A.1.2 porcelain dish

A.1.3 Oven

A.2 Procedure

A.2.1 weigh a porcelain dish and record weight (w_0).

A.2.2 add 1.0 gms of ink and record as gross weight (w_1).

A.2.3 transfer the content (A.2.2) into the oven (100 degree) for 1 hour to evaporate off the solvent.

A.2.4 remove the porcelain dish from the oven and cool at room temperature for 30 minutes.

A.2.5 weigh the porcelain dish (A.2.4) and record the weight (w_2).

A.3 calculation

Solvent content, expressed in percent by mass, shall be calculated using the formula below

$(w_1 – w_0) – (w_2 - w_0) \times 100\%$

where

w_0 weight of porcelain dish;

w_1 weight of both porcelain dish and the ink;

w_2 weight of both porcelain dish and the ink, after heating in the oven.

Annex B
(normative)

Preparation of acid extracts from inks

B.1 Separation of the pigment from the ink

B.1.1 Preparation of the test sample

Condition the sample for 24 h at 25 ± 2 ^{0}C and relative humidity 65 ± 2 per cent. If any skin is present, remove it as far as possible. Thoroughly stir the sample and if necessary, pass it through a sieve of nominal aperture 150 µm to remove any remaining skin and other extraneous matter.

B.1.2 Reagents

Select a solvent that effects the optimum separation of the pigment. The solvent selected shall be recorded and subsequently reported. Examples of suitable solvents or solvent mixtures are as follows:

a) Toluene/ethanol (4:1 v/v),

b) Xylene/butan-1-o1 (9:1 v/v),

c) Toluene,

d) Butanone (methyl ethyl ketone).

B.1.3 Apparatus

Ordinary laboratory apparatus and glassware and in particular:

a) Suitable laboratory centrifuge, with tubes of inert material of capacity 50 mL or 100 mL, capable of imparting a relative centrifuge acceleration of 100 km s $^{-2}$

b) Air-ventilated oven, capable of being maintained at 105 ± 2 °C,

c) Stoppered glass container, of at least 2 L capacity.

B.1.4 Procedure

Weigh to the nearest 10 mg, a number of centrifuge tubes. Add 10 to 20 g of the prepared sample to each tube, taking care to avoid contamination of the walls and lip of the tube. Immediately weigh the tubes and contents to the nearest 10 mg. Approximately half fill the tubes with the selected solvent and stir thoroughly using a glass rod. Wash each glass rod thoroughly with the solvent, adding all washings to the appropriate tube. Balance the opposing centrifuge tubes to within 0.1 g by adding further solvent, taking care to ensure that an adequate working level is not exceeded. Centrifuge until there is a complete separation into a liquor and a pigment cake. Decant the supernatant liquor from all the tubes comprising a ‘set’ into the container. Add further solvent to each tube and mix thoroughly as specified above, taking care to disperse the pigment cake completely Repeat the centrifuging and transfer the liquor to the same container. Repeat the addition of solvent, centrifuging and transfer of liquor for further three times, taking special care, as before, to thoroughly disperse the pigment cake. As a final treatment for the pigment cake and to assist rapid drying, use acetone in place of the selected solvent. Add the acetone and mix, talking special care to disperse the whole pigment cake. Centrifuge and transfer the liquor as before to the container. Retain the container with the combined extracts for the procedure described later.

After ensuring that excess acetone has evaporated, place the centrifuge tubes in the oven, maintained at 105 ± 2 °C, for a minimum period of 3 h. Remove, transfer to a desiccator, allow to cool to ambient temperature and weigh each tube and contents to the nearest 10 mg. Return the tubes and contents to the oven for a minimum period of 1 h, allow to cool to ambient temperature in the desiccator and reweigh. Repeat the heating, cooling and weighing operations until the results of two consecutive weighing do not differ by more than 10 mg.

Calculate the pigment content of the ink as a percentage by mass of the ink sample.

At the end of the separation procedure, check that the dried pigment cake can be crumbled easily to indicate that the binder has been satisfactorily extracted. If the cake remains cohesive, repeat the whole procedure on the original ink using a more suitable solvent or solvent mixture.

B.1.5 Blank test solution

Prepare a mixture of solvent using the same proportions as used in the separation (B 1.4). Retain the mixture for use as the blank in the determinations described later.

B.1.6 Treatment of the separated pigment

Take all the dried pigment from the tubes in one set obtained by the procedure described in B.1.4. Carefully break up the pigment cake by placing it between two sheets of glazed paper and by the application of a minimum pressing or rolling action without and grinding effect disperse it so that all the pigment just passes through a 500 µm sieve. Ensure that all the pigment passes through the sieve and is collected.

B.1.7 Method for the acid extraction of soluble metals

B.1.7.1 Reagents

Only reagents of analytical grade and distilled water shall be used:

a) Dilute Hydrochloric acid , 0.07 M

b) Hydrochloric acid strong, dilute 1 part by volume of hydrochloric acid (S.G1.18) with 1 part by volume of water,

c) Ethanol, minimum 95 per cent (v/v).

B.1.7.2 Apparatus

Ordinary laboratory apparatus and glassware and in particular

a) Suitable mechanical stirrer (see Note in B.1.7.3),

b) pH meter and electrodes,

c) Membrane filter, pore diameter 0.15 pm or any other suitable filter capable of giving a clear filtrate,

d) Filtration apparatus, for the membrane filter,

e) Water-bath, capable of being maintained at 23 ± 2 °C.

B.1.7.3 Procedure

Carry out the extraction of the prepared pigment B.1.6 in duplicate. Protect the test portion from direct sunlight during the extraction and before the analysis.

Weigh 5.0 ± 0.01g of the sample into a clean, dry 150 mL beaker. Wet the test portion with 2 mL of the ethanol or the minimum larger quantity to wet the test portion, fit the stirrer and add 75 mL of the dilute

hydrochloric acid previously adjusted at 23 ±2 °C by means of the water-bath. Place the beaker in the water-bath and immediately commence stirring the mixture, insert the electrodes of the pH metre into the mixture and, if necessary, adjust the pH to that of the dilute hydrochloric acid, using the strong hydrochloric acid.

Continue stirring for 15 ± 1 min, checking that the temperature of the mixture is maintained at 23 ± 2 °C throughout the test period. Maintain the pH of the mixture by carefully adding the strong hydrochloric acid. At the end of the period of stirring, allow the mixture to stand for a further 5 ± 1 min at 23 ± 2 °C. Then decant the mixture through the membrane filter using the filtration apparatus and collect the filtrate obtained in the first 10 min (which should be a clear solution) in a suitable glass container. Immediately stopper the container.

Retain the filtered extract for the determination of the various ‘soluble" metals as described in the appropriate annexes of this standard.

Take appropriate aliquot portions for each determination.

Carry out the determination parts of this standard, of the ‘soluble’ metal content of the filtrate as soon as possible and within 4 h of the preparation of the extract.

NOTE: During the whole period of extract, the speed of the stirrer should be adjusted so that the pigment is kept in continuous suspension whilst taking care to avoid splashing.

B.1.8 Treatment of the extracted liquid portion

B.1.8.1 Reagents

Only reagents of analytical grade and distilled water shall be used.

a) Nitric acid — Dilute 1 part by volume of nitric acid, 65 % (m/m), (S.G=1.40) with 1 part by volume of water,

b) Dilute Hydrochloric acid, 0 07 M.

B.1.8.2 Apparatus

a) Water-bath,

b) Hot-plate,

c) Muffle furnace, capable of being maintained at 475 ± 25 °C,

d) One-mark volumetric flask of capacity 100 mL

B.1.8.3 Procedure

Carry out the following procedure in duplicate on the extracted liquid portion.

Carry out the same procedure on the blank test solution B.1.5 using the same quantities of all reagents.

Transfer the contents of the stoppered glass container, obtained according to B.1.4, into a suitable graduated vessel and dilute to a specific volume with the solvent or solvent mixture. Transfer an aliquot portion to a porcelain or silica dish of suitable capacity.

Evaporate the main portion of the solvent, using the water-bath.

Place the dish on the hot-plate and slowly increase the temperature in order to remove all residual solvent. Gradually increase the temperature of the hot-plate until the material begins to char. Then transfer the dish to the muffle furnace, maintained at 475 ± 25 °C, and ash. When the ashing is complete, remove the dish from the muffle furnace and allow it to cool to ambient temperature. Break up the ash into fine particles with a glass rod and leave the road in the dish during the following filtration step.

Taking care to avoid loss of material if the ash reacts vigorously, slowly add 10 mL of the nitric acid. Heat carefully on the hot-plate until 2 to 3 mL of solution remain. Add an additional 10 mL of the nitric acid and continue heating on the hot-plate until less than 5 mL of solution remain. Add 20 to 25 mL of water and filter the solution through a medium porosity filter paper into the 100 mL one-mark volumetric flask. If the filtrate is not clear, refilter through a fine porosity filter paper.

Wash the dish and the filter paper several times with water and add the washings to the flask. Dilute to the mark with the hydrochloric acid and mix well.

Retain the extract for the determination of the various "soluble" metals as described in the appropriate annexes of this standard. Take appropriate aliquot portions for each determination.

NOTE 1 If the ink sample contains cellulose nitrate, add 2 g liquid paraffin to the liquid portion of the ink before removing the volatile solvents.

NOTE 2 If desired, the bulk of the solvent may be removed by distillation from a flask. This latter method is preferred when, for example, the Solvents include butanol or xylene

NOTE 3 If the ink sample contains chlorinated compounds, acidic products will be formed during ashing. Therefore, before ashing, the residue obtained on evaporation to dryness of the extract of the liquid portion should be covered with anhydrous sodium carbonate to neutralize these products. Approximately 1 g of sodium carbonate should be added for each 1 g of residue. If it should be necessary to add a relatively large quantity of sodium carbonate, it is desirable to take an appropriate liquot portion of the combined extract.

The same procedure should also be applied to the blank solution.

B.1.8.4 Blank solution

Take 75 or 500 mL, as appropriate, of the dilute hydrochloric acid and if necessary add 2 mL of the ethanol. Retain this solution for the blank determinations on the pigment portion of the ink as described later in the standard.

Annex C
(normative)

Determination of lead content by AAS

C.1 Principle

Aspiration of the test solution into an acetylene/air flame. Measurement of the absorption of the selected spectral line, emitted by a lead hollow-cathode or lead discharge lamp, in the region of 283.3 nm.

C.2 Reagents and materials

Only analytical grade and distilled water shall be used:

a) Dilute Hydrochloric acid, 0.07 M,

b) Acetylene, commercial grade, in a steel cylinder,

c) Compressed air,

d) Lead, standard stock solution containing 1 g of Pb per litre.

Procedure

either,

Transfer the contents of an ampoule of standard lead solution containing exactly 1 g of Pb into a 1000 mL one-mark volumetric flask, dilute to the mark with dilute hydrochloric acid and mix well,

Or

Weigh, to the nearest 1 mg, 1.598 g of lead nitrate ($Pb(N0_3)_2$) (previously dried for 2 h at 105 °C), dissolve in dilute hydrochloric acid in a 1 000 mL one-mark volumetric flask, dilute to the mark with the same hydrochloric acid and mix well.

Note: 1 mL of this standard stock solution contains 1 mg of Pb per litre

e) Lead, standard solution containing 100 mg of Pb per litre.

Note: Prepare this solution on the day of use.

Pipette 100 mL of the standard stock solution into a 1 000 mL one-mark volumetric flask, dilute to the mark with dilute hydrochloric acid and mix well.

Note: 1 mL of this standard solution contains 100 µg of Pb.

C.3 Apparatus

Ordinary laboratory apparatus and

a) Flame atomic absorption spectrometer, suitable for measurements at a wavelength of 283.3 nm and fitted with a burner fed with acetylene and air,

b) Lead hollow-cathode lamp or lead discharge lamp,

c) Burette, of capacity 50 mL,

d) One-mark volumetric flasks, of capacity 100 mL

Concentration, dilute the test solution appropriately (dilution factor F) with a known volume of the hydrochloric acid (B.5.2.1, Table E below).

C.4 Procedure

C.4.1 Preparation of the calibration curve

C.4.1.1 Preparation of the standard matching solutions

Prepare these solutions on the day of use into a series of six 100 mL one-mark volumetric flasks. Introduce from the burette, respectively, the volumes of the standard lead solution given in Table C below, dilute each to the mark with the hydrochloric acid and mix well.

Table C — Concentration of lead in standard matching solution

Standard matching solution No.	Volume of the standard lead solution, mL	Corresponding concentration of lead in the standard matching solution, µg/mL
0[a]	0	0
1	2.5	2.5
2	5	5
3	10	10
4	20	20
5	30	30
[a]Blank matching solution		

C.4.1.2 Spectrometric measurements

Install the cadmium spectral source in the spectrometer and optimize the conditions for the determination of lead. Adjust the instrument in accordance with the manufacturer's instructions and adjust the monochromator to the region of 283.8 nm in order to obtain the maximum absorbance.

Adjust the flow of the acetylene and of the air according to the characteristics of the aspirator-burner, and ignite the flame. Set the scale expansion, if fitted, so that the standard matching solution No. 5 (see Table B) gives almost a full-scale deflection.

Aspirate into the flame each of the standard matching solutions in ascending order of concentration, and repeat with the standard matching solution No 4 to verify that the instrument has achieved stability. Aspirate water through the burner between each measurement, taking care to keep the rate of aspiration uniform

C.4.1.3 Calibration graph

Plot a graph having the masses, in micrograms, of Pb contained in 1 mL of the standard matching solutions as abscissae and the corresponding values of the absorbances, reduced by the reading for the blank matching solution, as ordinates.

C.4.2 Test solutions

C.4.2.1 Pigment portion of the liquid ink

Use the solution obtained by the procedure described in B.1.7.3.

C.4.2.2 Liquid portion of the ink

Use the solutions obtained by the procedure described in B.1.8.3.

C.5 Determination

Measure first the absorbance of the hydrochloric acid in the spectrometer after having adjusted it as described in C.4.1.2. Then measure the absorbance of each test solution three times and, afterwards, that of the hydrochloric acid again Finally, determine the absorbance of standard matching solution No. 4 in order to verify that the response of the apparatus has not changed. If the absorbance of a test solution is higher than that of the standard matching solution with the highest lead concentration, dilute the test solution appropriately (dilution factor F) with a known volume of the hydrochloric acid.

C.6 Expression of results

C.6.1 calculations

C.6.1.1 Pigment portion of the liquid ink

Calculate the mass of ‘soluble’ lead in the hydrochloric acid extract obtained by the method described in B.1.7.3 using the equation:

$m_o = [(a_1 – a_0) \div 10] \times V_1 \times F_1$

Where,

a_0, is the lead concentration, in micrograms per millilitre, of the blank test solution prepared by the method described in B.1.8.4;

a_1, is the lead concentration, in micrograms per millilitre, of the test solution obtained from the calibration graph;

F_1, is the dilution factor;

m_o, is the mass, in grams, of "soluble" lead in the hydrochloric acid extract;

V_1 is the volume, in mL, of the hydrochloric acid plus ethanol used for the extraction described in B.1.7.3 (assumed to be 77 mL or 502 mL respectively).

Calculate the ’soluble’ lead content of the pigment portion of the ink using the equation:

$^cPb_1 = m_o \times 10^2/m_1 \times p/10^2 = (m_o \times p)/m_1$

Where,

$^cPb_1$, is the ‘soluble’ lead content, of the pigment portion of the paint, expressed as a percentage by mass of the ink,

m_1, is the mass, in grams, of the test portion taken to prepare the solution described in B.1.7.3;

p, is the pigment content of the liquid ink, expressed as a percentage by mass, obtained by the method described in B.1.4.

C.6.1.2 Liquid portion of the ink

Calculate the mass of lead in the solution (extract), obtained by the method described in B.1.8 3

$M_2 = (b_1 – b_o)/10^6 \times V_2 \times F_2$

Where,

b_o, is the lead concentration, in micrograms per mL, of the blank test solution prepared by the method described in B.1.5;

b_1, is the lead concentration, in micrograms per mL, of the test solution obtained from the calibration graph;

F_2, is the dilution factor;

M_2, is the mass, in g, of lead in the liquid portion of the ink;

V_2, is the volume, in mL, of the solution obtained by the method described in B.1.8 3. (= 100 mL).

Calculate the lead content of the liquid portion of the ink using the equation.

$^cPb_2 = m_2/m_3 \times 10^2$

Where,

$^cPb_2$, is the lead content of the liquid portion of the ink, expressed as a percentage by mass of ink;

m_3, is the total mass, in grams of ink comprising a 'set' as described in C 4.

C.6.1.3 Liquid ink

Calculate the total "soluble" lead content of the liquid ink as the sum of the results obtained according to C.6.1.1 and C.6.1.2 thus,

$^cPb_3 = {}^cPb_1 + {}^cPb_2$

where,

$^cPb_3$, is the total 'soluble' lead content of the ink, expressed as a percentage by mass.

Annex D
(normative)

Determination of antimony by AAS

D.1 Principle

Aspiration of the test solution into an acetylene/air flame. Measurement of the absorption of the selected spectral line, emitted by an antimony hollow-cathode or antimony discharge lamp, in the region of 217.6 nm.

D.2 Reagents and materials

During the analysis, use only reagents of recognized analytical grade and distilled water.

a) Concentrated Hydrochloric acid, approximately 37% (v/v and S.G= 1.18),

b) Hydrochloric acid, c(HCL) — 1 M,

c) Compressed air,

d) Antimony, standard stock solution containing 1 g of Sb per litre,

Weigh, to the nearest 0.1 mg, 119.7 mg of dried antimony trioxide, dissolve in 40 mL of the hydrochloric acid in a 100 mL one-mark volumetric flask, dilute to the ark with water and mix well.

Note: 1 mL of this standard stock solution contains 1 mg of Sb.

e) Antimony, standard solution containing 100 mg of Sb per litre.

Note: Prepare this solution on the day of use.

Pipette 10 mL of the standard stock solution into a 100 mL one-mark volumetric flask, dilute to the mark with the hydrochloric acid and mix well.

Note: 1 mL of this standard solution contains 100 mg of Sb.

D.3 Apparatus

a) Flame atomic absorption spectrometer, suitable and, measurements at a wavelength of 217.6 nm and fitted with a burner fed with acetylene and air,

b) Antimony hollow-cathode lamp or antimony discharge lamp,

c) Burette, of capacity 50 mL,

d) One-mark volumetric flasks, of capacity 100 mL

D.4 Procedure

D.4.1 Preparation of the calibration graph

D.4.1.1 Preparation of the standard matching solution

Note: Prepare this solution on the day of use:

Into a series of five 100 mL one-mark volumetric flasks, introduce from the burette, respectively, the volumes of the standard antimony solution given in Table D below, dilute each to the mark with the hydrochloric acid and mix well.

Table D — Concentration of antimony in standard matching solution

Standard matching solution No.	Volume of the standard Antimony solution, mL	Corresponding concentration of Sb in the standard matching solution, µg/mL
0[a]	0	0
1	5	5
2	10	10
3	20	20
4	40	40
[a]Blank matching solution		

D.4.1.2 Spectrometric measurements

Install the antimony spectral in the spectrometer and optimize the conditions for the determination of antimony. Adjust the instrument in accordance with the manufacturer's instructions and adjust the monochromator to the region of 217.6 nm in order to obtain the maximum absorbance.

Adjust the flow of the acetylene and of the air according to the characteristics of the aspirator-burner, and ignite the flame. "Set the scale expansion, if fitted, so that the standard matching solution No. 4 (see Table D) gives almost a full-scale deflection.

Aspirate into the flame each of the standard matching solutions in ascending order of concentration, and repeat with the standard matching solution No 3 to verify that the instrument has achieved stability. Aspirate water through the burner between each measurement, taking care to keep the rate of aspiration uniform.

D.4.1.3 Calibration graph

Plot a graph having the masses, in micrograms, of Sb contained in 1 mL of the standard matching solutions as abscissae and the corresponding values of the absorbances, reduced by the reading for the blank matching solution, as ordinates.

D.4.2 Test solutions

D.4.2.1 Pigment portion of the liquid ink

Mix thoroughly 9 parts by volume of each of the solution obtained by the procedure described in B.1.7.3 with 1 part by volume of the hydrochloric acid

D.4.2.2 Liquid portion of the ink

Mix thoroughly 9 parts by volume of each of the solutions obtained by the procedure described in B.1.8.3 with 1 part by volume of the hydrochloric acid

D.5 Determination

Spectral interference occurs in the presence of lead, calcium or copper on the resonance line at 217.6 nm. In the presence of lead, use the antimony resonance line at 206.8 nm or 231.1 nm. In the presence of calcium, measure the absorbance at 217.0 nm and subtract the result from the absorbance at 217.6 nm. In the presence of copper, use the antimony resonance line at 231 1 nm.

Use a deuterium background corrector to correct for background absorption. Alternatively, the solutions can be reaspirated using a neighbouring non absorbing line for the background correction [see Note below).

NOTE Some hollow-cathode lamps for antimony have a non-absorbing line at 216.9 nm.

D.6 Expression of results

D.6.1 Calculations

D.6.1.1 Pigment portion of the liquid ink

Calculate the mass of "soluble" antimony in the hydrochloric acid extract obtained by the method described in B.1.7.3, using the equation:

$m_o = [(a_1 - a_0)/10^6] \times V_1 \times (10/9) \times F_1$

Where,

a_0, is the antimony concentration, in micrograms per millilitre, of the blank test solution prepared by the method described in B.1.8.4;

a_1, is the antimony concentration, in micrograms per millilitre, of the test solution obtained from the calibration graph;

F_1, is the dilution factor,

m_o, is the mass, in grams, of "soluble" antimony in the hydrochloric acid extract;

V_1, is the volume, in mL, of the hydrochloric acid plus ethanol used for the extraction described in B.1.7.3 (assumed to be 77 mL).

Calculate the 'soluble' antimony content of the pigment portion of the ink, using the equation:

$^cSb_1 = m_o \times 10^2/m_1 \times p/10^2 \times (m_o \times P)/m_1$

Where,

$^cSb_1$, is the soluble antimony content of the pigment portion of the ink, expressed as a percentage by mass of the ink

m_1, is the mass, in grams, of the test portion taken to prepare the solution described in B.1.7.3;

P, is the pigment content of the liquid ink, expressed as a percentage by mass.

D.6.1.2 Liquid portion of the ink

Calculate the mass of antimony in the solution (extract), obtained by the method described in B.1.8.3, using the equation:

$m_2 = (b_1 - b_o)/10^6 \times V_2 \times 10/9 \times F_2$

Where,

b_o, is the antimony concentration, in micrograms per mL, of the blank test solution prepared by the method described in B.1.5;

b_1, is the antimony concentration, in micrograms per mL, of the test solution obtained from the calibration graph;

F_2, is the dilution factor;

m_2, is the mass, in g, of antimony in the liquid portion of the ink,

V_2, is the volume, in mL, of the solution obtained by the method described in B.1.8.3 (=100 mL)

Calculate the antimony content of like liquid portion of the ink, using the equation:

$^cSb_2 = m_2/m_3 \times 10^2$

$^cSb_2$, is the antimony content of the liquid portion of the ink, expressed as a percentage by mass of the ink;

m_3, is the total mass, in grams of ink comprising a 'set' as described in B.1.4.

D.6.1.3 Liquid ink

Calculate the total ‘soluble’ antimony content of the liquid ink as the sum of the results obtained according to D.6.1.1 and D.6.1.2 thus,

$^cSb_3 = {}^cSb_1 + {}^cSb_2$

Where,

$^cSb_3$, is the total soluble antimony content of the ink expressed as a percentage by mass.

Annex E
(normative)

Determination of barium by FAES

E.1 Principle

Aspiration of the solution into a dinitrogen monoxide/acetylene flame. Measurement of the radiation emitted by barium at a wavelength of 553.5 nm. The ionization of the barium atoms in the flame is suppressed by addition of potassium chloride.

E.2 Reagents

During the analysis, use only reagents or recognized analytical grade and distilled water only

a) Potassium chloride, 50 g/L solution,

b) Hydrochloric acid, c (HCL) = 0 07 M,

c) Dinitrogen monoxide, commercial grade, in a steel cylinder,

d) Acetylene, commercial grade, in a steel cylinder,

e) Barium, standard stock solution containing 1 g of Ba per litre

Either,

Transfer the contents of an ampoule of standard barium solution containing exactly 1 g of Barium into a 1000 mL one-mark volumetric flask, dilute to the mark with the hydrochloric acid and mix well.

Or

Weigh, to the nearest 1 mg, 1.779 g of barium chloride dihydrate ($BaCl_2.2H_20$), dissolve in the hydrochloric acid in a 1000 mL one-mark volumetric flask, dilute to the mark with the same hydrochloric acid and mix well.

Note: 1 mL of this standard stock solution contains 1 mg of Ba.

f) Barium, standard solution containing 20 mg of Ba per litre.

Note: Prepare this solution on the day of use.

Pipette 20 mL of the standard stock solution into a 1000 mL one-mark volumetric flask, dilute to the mark with the hydrochloric acid and mix well.

Note: 1 mL of this standard solution contains 20 µg of Ba

E.3 Apparatus

For this test, the laboratory glassware shall be barium free.

Ordinary laboratory apparatus and

a) Flame atomic emission spectrometer, suitable for measurements at a wavelength of 553.5 nm and fitted with a burner fed with dinitrogen monoxide and acetylene,

b) Pipettes, of suitable volume,

c) Burettes, of capacity 10 and 50 mL,

d) One-mark volumetric flasks, of capacity 50 mL,

e) Recording apparatus, a compensating record is recommended.

E.4 Procedure

E.4.1 Preparation of the calibration graph

E.4.1.1 Preparation of the standard matching solutions

Prepare these solutions on the day of use into a series of six 50 mL one-mark volumetric flasks, introduce from the burette, respectively, the volumes of the standard barium solution given in Table E, add 5 mL of the potassium chloride solution, dilute each to the mark with the hydrochloric acid and mix well.

Table E — Concentration of barium in standard matching solution

Standard matching solution No.	Volume of the standard barium solution, mL	Corresponding concentration of barium in the standard matching solution, µg/mL
0[a]	0	0
1	2	0.8
2	5	2
3	10	4
4	20	8
5	40	16
[a]Blank matching solution		

E.4.1.2 Spectrometric measurements

Measure the emission of the standard matching solutions in the spectrometer, using the operating conditions specified by the manufacturers of the instrument.

In order to determine and correct for the constant background emission, due to the presence of calcium, the barium line or record the emission over a wavelength range from 553.0 nm to 554.0 nm.

E.4.1.3 Calibration graph

Plot a graph having the masses, in micrograms, Ba contained in 1 mL of the standard matching solutions as abscissae and the corresponding values of the emission, corrected for background emission, as ordinates.

E.5 Test Solution

E.5.1 Pigment portion of the liquid ink

Pipette a suitable volume (V_3) of each of the solutions obtained by the procedure described in solutions obtained by the procedure described in B.1.7.3 of 50 mL one-mark volumetric flasks so that the barium concentration of each test solution will be within the Calibration range. Add 5 mL of the potassium chloride solution, dilute to the mark with the hydrochloric acid and mix well

E.5.2 Liquid portion of the ink

Pipette a suitable volume (V_4) of each of the solutions obtained by the procedure described in B.1.8.3 of 50 mL one-mark volumetric flasks so that the barium concentration of each test solution will be within the calibration range. Add 5 ml. of the potassium chloride solution, dilute to the mark with the Hydrochloric acid and mix well.

E.6 Determination

Measure first the emission of the hydrochloric acid in the spectrometer after having adjusted it as described in E.4.1.2. Then measure the emission of each test solution E.5 three times and, afterwards, that of the hydrochloric acid again. Finally, re-determine the emission of the standard matching solution No. (E.4.1.1) in order to verify that the response of the apparatus has not changed. If the emission of a test solution is higher than that of the standard matching solution with the highest barium concentration, dilute the test solution approximately (dilution factor F1) with a known volume of the hydrochloric acid.

E.7 Expression of results

E.7.1 Calculations

E.7.1.1 Pigment portion of the liquid ink

Calculate the mass ‘soluble’ barium in the hydrochloric acid extract, obtained by the method described in B.1.7.3, using the equation:

$m_o = (a_1 – a_o)/10^6 (V_1/V_3) \times 50 F_1$

Where,

a_o, is the barium concentration, in micrograms per mL, of the bank test solution prepared by the method described in B.1.8.4;

a_1, is the barium concentration, in micrograms per mL, of the test solution obtained from the calibration;

F_1 is the dilution factor;

m_o, is the mass, in grams, of "soluble" barium in the hydrochloric acid extract;

V_1, is the volume, in mL, of the hydrochloric acid plus ethanol used for the extraction described in B.1.7.3 (assumed to be 77 mL);

V_3, is the volume, in mL, of the aliquot portion of the hydrochloric acid plus ethanol extract taken for the test (E.5.1).

Calculate the "soluble" barium content of the pigment portion of the ink, using the equation:

$^{c}Ba_1 = m_o \times 10^2/m_1 \times p/10^2$

Where,

$^{c}Ba_1$, is the 'soluble' barium content, of the pigment portion of the ink, expressed as a percentage by mass of the ink

m_1, is the mass, in grams, of the test portion taken to prepare the solution described in B.1.7.3;

P, is the pigment content of the liquid ink, expressed as a percentage by mass, obtained by the appropriate method described in B.1.4.

E.7.1.2 Liquid portion of the ink

Calculate the mass of barium in the solution (extract), obtained by the method described in B.1.8.3, using the equation:

$m_2 = (b_1 - b_o)/10^6 (V_2/V_4) \times 50 \times F_2$

Where,

b_o, is the barium concentration, in micrograms per mL, of the blank test solution prepared by the method described in B.1.5;

b_1 is the barium concentration, in micrograms per mL of the test solution obtained from the calibration graph;

F_2, is the dilution factor;

m_2, is the mass, in grams, of barium in the liquid portion of the ink;

V_2, is the volume, in mL, of the solution obtained by the method described in B.1.8.3 (= 100 mL);

V_4, is the volume, mL, of the aliquot portion of the solution taken for the test.

Calculate the barium content of the liquid portion of the ink, using the equation:

$^{C}Ba_2 = (m_2/m_3) \times 10^2$

Where,

$^{C}Ba_2$, is the barium content, of the liquid portion of the ink, expressed as a percentage by mass of the ink;

m_3, is the total mass, in g of ink comprising a 'set" as described in B.1.4.

E.7.1.3 Liquid ink

Calculate the total "soluble" barium content of the liquid ink as the sum of the results obtained according to E.7.1.1 and E.7.1.2 thus,

$^{c}Ba_3 = {}^{c}Ba_1 + {}^{c}Ba_2$

where,

$^{c}Ba_3$, is the total 'soluble" barium content of the ink expressed as a percentage by mass.

Annex F
(normative)

Determination of cadmium by AAS

F.1 Principle

Aspiration of the test solution into an acetylene/air flame. Measurement of the absorption of the selected spectral line emitted by a cadmium hollow-cathode or cadmium discharge lamp, in the region of 228. 8 nm.

F.2 Reagents

F.2.1 Hydrochloric acid, (HCL) = 0.07 M.

F.2.2 Acetylene, commercial grade, in a steel cylinder.

F.2.3 Compressed air.

F.2.4 Cadmium, standard stock solution containing 1 g of Cd per litre.

Either,

a) transfer the contents of an ampoule of standard cadmium solution containing 1 g of Cd into a 1 000 mL one-mark volumetric flask, dilute to the mark with the hydrochloric acid (F.2.1) and mix well;

b) weigh, to the nearest 1 mg, a mass of a water soluble cadmium salt of defined purity containing exactly 1 g of Cd, dissolve in the hydrochloric acid (F.2.1) in a 1 000 mL one-mark volumetric flask, dilute to the mark with the same hydrochloric acid and mix well.

Or

c) weigh, to the nearest 1 mg, exactly 1 g of cadmium metal dissolve it in the minimum of concentrated hydrochloric acid (e.g. approximately 1.18g/mL) in a 1000 mL one-mark with the hydrochloric acid (F.2.1) and mix well

Note: 1 mL of this standard stock solution contains 1mg of Cd.

F.2.5 Cadmium, standard solution containing 10 mg of Cd per litre.

Note: Prepare this solution on the day of use.

Pipette 10 mL of the standard stock solution (F.2.4) into a 1 000 mL one-mark volumetric flask, dilute to the mark with the hydrochloric acid (F.2.1) and mix well.

Note: 1 mL of this standard solution contains 10 µg of Cd.

F.3 Apparatus

Ordinary laboratory apparatus and

F.3.1 Flame atomic absorption spectrometer, suitable for measurement at a wavelength of 228.8 nm and fitted with a burner fed with acetylene and air.

F.3.2 Cadmium hollow-cathode lamp or cadmium discharge lamp.

F.3.3 Burette, of capacity 10 mL

F.3.4 One mark volumetric flasks, of capacity 100 mL

F.4 Procedure

F.4.1 Preparation of the calibration graph

F.4.1.1 Prepare these solutions on the day of use

Aspirate into the flame each of the standard matching solutions in ascending order of concentration, and repeat with the standard matching solution No. 3 to verify that the instrument has achieved stability. Aspirate water through the burner between each measurement, taking care to keep the rate of aspiration uniform.

F.4.1.2 Spectrometric measurements

Install the cadmium spectral source (F.3.2) in the spectrometer (F.3.1) and optimize the conditions for the determination of cadmium. Adjust the instrument in accordance with the manufacturer's instructions and adjust the monochromator to the region of 228.8 nm in order to obtain the maximum absorbance.

Adjust the flow of the acetylene (F.2.2) and of the air (F.2.3) according to the characteristics of the aspirator-burner, and ignite the flame. Set the scale expansion, if fitted, so that the standard matching solution No. 4 (see Table F) gives almost a full scale deflection.

F.4.2 Pigment portion of the liquid ink in powder form

Use the solutions obtained by the procedure described in B.1.8.3.

F.4.3 Determination

Measure first the absorption of the hydrochloric acid (F.2.1) in the spectrometer (F.3.1) after having adjusted it as described in (F.4.1.2). Then measure the absorbance of each test solution three times and, afterwards, that of the hydrochloric acid again. Finally, re-determine the absorbance of standard matching solution No.3 in order to verify that the response of the apparatus has not changed. If the absorbance of a test solution is higher than that of the standard matching solution with the highest cadmium, put it into a series of five 100 mL one mark volumetric flasks (F.3.4), introduce from the burette (F.3.3) respectively, the volumes of the standard cadmium solution (F.2.5) given in Table F below and mix well

Table F — Concentration of cadmium in standard matching solution

Standard matching solution No.	Volume of the standard cadmium solution (A.5.2.5), mL	Corresponding concentration of cadmium in the standard matching solution, µg/mL
0[a]	0	0
1	0.5	0.05
2	1	0.1
3	2	0.2
4	4	0.4
[a]Blank matching solution		

F.5 Expression of results

F.5.1 Calculations

F.5.1.1 Pigment portion of the liquid ink

Calculate the mass of 'soluble' cadmium in the hydrochloric acid extract obtained by the method described in B.1.7.3 using the equation:

$m_o = [(a_1 – a_o)/10^6] \times V_1 \times F_1$

Where,

a_o, is the cadmium concentration, in micrograms per millilitre, of the blank test solution prepared by the method described in B.1.8.4;

a_1, is the cadmium concentration, in micrograms per millilitre, of the test solution obtained from the calibration graph;

F_1, is the dilution factor referred to in F.4.3

m_o, is the mass, in grams, of 'soluble' cadmium in the hydrochloric acid extract;

V_1, is the volume, in mL, at the hydrochloric acid plus ethanol used for the extraction described in B.1.7.3 (assumed to be 77 mL or 502 mL respectively).

Calculate the 'soluble' cadmium content of the pigment portion of the ink using the equation:

$^cCd_1 = (m_o \times P)/m_1$

Where,

$^cCd_1$, is the 'soluble' cadmium content of the pigment portion of the ink, expressed as a percentage by mass of the ink;

m_1, is the mass, in grams of the test portion taken to prepare the solutions described in B.1.7.3;

P, is the pigment content of the liquid ink, expressed as a percentage by mass, obtained by the method described in B.1.4.

F.5.1.2 Liquid portion of the ink

Calculate the mass of cadmium in the solution (extract) obtained by the method described in (B.1.8.3).

$M_2 = [(b_1 – b_o)/10^6] \times V_2 \times F_2$

Where,

b_o, is the cadmium concentration, in micrograms per mL, of the blank test solution prepared by the method in B.1.5;

b_1, is the cadmium concentration, in micrograms per mL, of the test solution obtained from the calibration graph;

F_2 is the dilution factor referred to in F.4.3;

m_2, is the mass, in g of cadmium in the liquid portion of the ink;

V_2, is the volume, in mL, of the solution obtained by the method described in B.1.8.3 (= 100 mL).

Calculate the cadmium content of the liquid portion of the ink, using the equation,

$^{c}Cd_2 = (m_2/m_3) \times 10^2$

Where,

$^{c}Cd_2$, is the cadmium content, of the liquid portion of the ink, expressed as a percentage by mass of the ink,

m_3, is the total mass, in grams, of ink comprising a 'set' as described in B.1. 4.

F.5.1.3 Liquid ink

Calculate the total 'soluble' cadmium content of the liquid ink as the sum of the results obtained according to F.5.1.1 and F.5.1.2.

$^{c}Cd_3 = {}^{c}Cd_2 + {}^{c}Cd_1$

Where,

$^{c}Cd_3$, is the total 'soluble' cadmium content of the ink, expressed as a percentage by mass.

Bibliography

[1] KS 810:2013, Printing inks for use on food wrappers, packages and receptacles — Specification, 2nd edition

[2] International Life Sciences Institute (ILSI) Europe Report Series on Packaging Materials - Printing Inks for Food Packaging Composition and Properties of Printing Inks, December 2011

[3] IS 15495: 2020, Printing ink for food packaging — code of practice

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