BS EN ISO 17075-1:2017

BSI Standards Publication

Leather — Chemical determination of chromium(VI) content in leather

Part 1: Colorimetric method (ISO 17075-1:2017)

National foreword

This British Standard is the UK implementation of EN ISO 17075-1:2017. Together with BS EN ISO 17075-2:2017, it supersedes BS EN ISO 17075:2007 which is withdrawn.

The UK participation in its preparation was entrusted to Technical Committee TCI/69, Footwear, leather and coated fabrics.

A list of organizations represented on this committee can be obtained on request to its secretary.

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Cuir - Détermination chimique de la teneur en chrome(VI) du cuir - Partie 1: Méthode colorimétrique (ISO 17075-1:2017)

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European foreword

This document (EN ISO 17075-1:2017) has been prepared by Technical Committee CEN/TC 289 “Leather” the secretariat of which is held by UNI, in collaboration with Technical Committee IULTCS “International Union of Leather Technologists and Chemists Societies”.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by August 2017, and conflicting national standards shall be withdrawn at the latest by August 2017.

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Contents

Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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For an explanation on the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT) see the following URL: www.iso.org/iso/foreword.html

ISO 17075-1 was prepared by the Chemical Test Commission of the International Union of Leather Technologists and Chemists Societies (IUC Commission, IULTCS) in collaboration with the European Committee for Standardization (CEN) Technical Committee CEN/TC 289, *Leather*, the secretariat of which is held by UNI, in accordance with the Agreement on technical cooperation between ISO and CEN (Vienna Agreement).

IULTCS, originally formed in 1897, is a world-wide organization of professional leather societies to further the advancement of leather science and technology. IULTCS has three Commissions, which are responsible for establishing international methods for the sampling and testing of leather. ISO recognizes IULTCS as an international standardizing body for the preparation of test methods for leather.

This first edition of ISO 17075-1, together with ISO 17075-2, cancels and replaces ISO 17075:2007, which has been technically revised.

The main changes compared to ISO 17075:2007 are as follows:

- the sample preparation has been revised;
- mechanical shaking in 7.1, 7.2 and 7.3 has been revised.

A list of all parts in the ISO 17075 series can be found on the ISO website.

Leather — Chemical determination of chromium(VI) content in leather —

Part 1:
Colorimetric method

1 Scope

This document specifies a method for determining chromium(VI) in solutions leached from leather under defined conditions. The method described is suitable to quantify the chromium(VI) content in leathers down to 3 mg/kg.

This document is applicable to all leather types.

The results obtained from this method are strictly dependent on the extraction conditions. Results obtained by using other extraction procedures (extraction solution, pH, extraction time, etc.) are not comparable with the results produced by the procedure described in this document.

If a leather sample is tested with both this document and ISO 17075-2, the results obtained with ISO 17075-2 are considered as the reference. The advantage of the method described in ISO 17075-2 is that there are no interferences from the colour of the extract. Nevertheless, interlaboratory trials do not show significant differences (see Annex C) and the results are comparable between both methods.

2 Normative references

The following documents are 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.

ISO 2418, *Leather — Chemical, physical and mechanical and fastness tests — Sampling location*

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

ISO 4044:2017, *Leather — Chemical tests — Preparation of chemical test samples*

ISO 4684, *Leather — Chemical tests — Determination of volatile matter*

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

— IEC Electropedia: available at http://www.electropedia.org/

3.1
chromium(VI) content
amount of chromium(VI) in leather determined after extraction with an aqueous salt solution at pH 7,0 to 8,0

Note 1 to entry: The chromium(VI) content is reported as chromium(VI) in milligrams per kilogram (mg/kg), expressed as the dry mass of the sample.

4 Principle

Extractable chromium(VI) is leached from the sample in phosphate buffer at pH 7,0 to 8,0 and co-extracted coloured substances which influence the detection are removed by solid phase extraction if necessary. The chromium(VI) in solution oxidizes 1,5-diphenylcarbazide to 1,5-diphenylcarbazone to give a red/violet complex with chromium which can be quantified photometrically at 540 nm.

5 Chemicals

All reagents used shall have at least analytical grade purity.

5.1 Extraction solution

Dissolve 22,8 g dipotassium hydrogenphosphate, $K_2HPO_4 \cdot 3H_2O$, in 1 000 ml water, adjusted to pH 8,0 ± 0,1 with phosphoric acid (5.3). Degas this solution with either argon or nitrogen (5.6) or ultrasonic bath.

Standard practice is to make up a fresh solution each day. However, the solution can be kept for up to one week in a refrigerator at (4 ± 3) °C but shall be warmed to room temperature and degassed prior to use.

5.2 Diphenylcarbazide (DPC) solution

Dissolve 1,0 g 1,5-diphenylcarbazide, $CO(NHNHC_6H_5)_2$, in 100 ml acetone, $(CH_3)_2CO$, and acidified with one drop of glacial acetic acid, CH_3COOH.

The solution should be kept in a brown glass bottle. The shelf life is up to 14 days at 4 °C.

5.3 Phosphoric acid solution

700 ml *o*-phosphoric acid, ρ = 1,71 g/ml, made up to 1 000 ml with deionised water (5.7).

First add approximately 200 ml of deionised water (5.7) to a 1 000 ml volumetric flask, then add the 700 ml of *o*-phosphoric acid and dilute to the mark with deionised water.

5.4 Chromium(VI) stock solution

Dissolve 2,829 g potassium dichromate ($K_2Cr_2O_7$) (5.8) in water in a volumetric flask and make up to 1 000 ml with water. One millilitre of this solution contains 1 mg of chromium.

A stock solution at this concentration level of hexavalent chromium is an alternative available commercially.

5.5 Chromium(VI) standard solution

Pipette 1 ml of solution (5.4) into a 1 000 ml volumetric flask and make up to the mark with extraction solution (5.1). One millilitre of this solution contains 1 µg of chromium.

The solution can be kept for up to one week in a refrigerator at (4 ± 3) °C but shall be warmed to room temperature prior to use.

A stock solution at this concentration level of hexavalent chromium is an alternative available commercially.

5.6 Argon or nitrogen, oxygen-free

Preference should be given to argon as an inert gas instead of nitrogen because argon has a higher specific mass than air.

5.7 **Distilled** or **deionised water**, Grade 3 quality as specified in ISO 3696.

5.8 **Potassium dichromate** ($K_2Cr_2O_7$), dried for (16 ± 2) h at (102 ± 2) °C.

5.9 **Methanol**, HPLC grade.

6 Apparatus and materials

Usual laboratory equipment and, in particular, the following.

6.1 **Suitable mechanical orbital shaker**, (100 ± 10) min^{-1}.

6.2 **Conical flask**, of capacity 250 ml, with stopper.

6.3 **Aeration tube** and **flow meter**, suitable for a flow rate of (50 ± 10) ml/min.

6.4 **Membrane filter**, 0,45 µm pore size [polytetrafluoroethylene (PTFE) or polyamide 66].

6.5 **Volumetric flasks**, of capacity 25 ml, 100 ml and 1 000 ml.

6.6 **Pipettes**, various nominal volumes.

6.7 **Spectrophotometer** or **filterphotometer**, wavelength 540 nm.

6.8 **Photometric cell**, quartz, 40 mm length or any other suitable cell length.

6.9 **Glass** or **polypropylene cartridges filled with a suitable solid phase extraction (SPE) material**, e.g. reversed phase (RP) C18 or a suitable activated magnesium silicate, see Annex A for examples of SPE materials.

6.10 **Solid phase extraction (SPE) system**, with vacuum device or solvent-resistant medical syringe.

6.11 **Sharp cutting tool** or **blade**, suitable for cutting the leather into 3 mm to 5 mm pieces.

6.12 **Analytical balance**, capable of weighing to the nearest 0,1 mg.

7 Procedure

7.1 Sampling and preparation of samples

If possible, sample in accordance with ISO 2418. If sampling in accordance with ISO 2418 is not possible (e.g. leathers from finished products like shoes, garments), details about sampling shall be given in the test report.

Prepare the leather sample by cutting (6.11) into small pieces according to the method specified in ISO 4044:2017, 6.3.

7.2 Preparation of analytical solution

Weigh (6.12) approximately (2 ± 0,1) g of leather pieces to the nearest 0,001 g. Pipette 100 ml of degassed solution (5.1) into a 250 ml conical flask (6.2). Displace oxygen by passing oxygen-free argon (or nitrogen) (5.6) into the flask for 5 min with a volume flow of (50 ± 10) ml/min. Remove the aeration tube (6.3), add the leather pieces and close the flask with a stopper. Record the extract volume as V_0.

Shake the conical flask with the leather pieces for 3 h ± 5 min on a mechanical orbital shaker (6.1) at (100 ± 10) min^{-1} to extract the chromium(VI).

Shake the flask in a smooth circular movement to keep the leather pieces from adhering to the wall of the flask and avoid shaking faster than specified.

Immediately after completing 3 h of extraction, filter the content of the conical flask through a membrane filter into a glass or plastic vessel with lid. Check the pH of the solution. The pH of the solution shall be between 7,0 and 8,0. If the pH of the solution is not within this range, start the complete procedure again.

Consider using a smaller sample mass if the pH is not between 7,0 and 8,0. In this case, the quantification limit will be increased.

7.3 Determination of chromium(VI) in the solution obtained from the extraction procedure

If the leather sample is coloured, it is likely some coloured substances, e.g. dyes, will have been co-extracted. These can influence the detection of chromium(VI). The extracted colorants can be removed by passing the extraction solution (7.2) through a cartridge containing a suitable solid phase extraction material (6.9), see Annex A for examples.

Pre-treat the SPE cartridges (6.9) in the following way:

a) flush the cartridge (6.9) first with 5 ml methanol (5.9),

b) afterwards with 5 ml distilled water (5.7) and

c) directly afterwards with 10 ml of extraction solution (5.1).

Do not dry the cartridges (6.9) during or after the pre-treatment.

From the solution obtained in 7.2, take 10 ml (V_1) and transfer this quantitatively through the cartridge (6.9) on an SPE system with a vacuum device or syringe (6.10). Collect the eluate in a 25 ml volumetric flask (6.5). Flush the cartridge with 10 ml extraction solution (5.1) into the 25 ml flask. Make up the flask to volume (V_2) with extraction solution (5.1). Mark this solution as S_1.

Pipette (6.6) 10 ml (V_3) of solution S_1 into a 25 ml volumetric flask. Dilute the solution to 3/4 of the flask's volume with extraction solution (5.1). Add 0,5 ml of phosphoric acid solution (5.3) and afterwards 0,5 ml of diphenylcarbazide solution (5.2). Make up the flask to volume (V_4) with extraction solution (5.1) and mix well.

Let stand for at least 15 min ± 5 min. Measure the absorbance of the solution at 540 nm in a 40 mm cell (6.8) against the blank solution (7.4). Record the absorbance obtained as A_1.

For each run, pipette another 10 ml aliquot of solution S_1 into a 25 ml volumetric flask and treat it as described above, but without the addition of the diphenylcarbazide solution (5.2). Measure the absorbance of this solution in the same way as before and record it as A_2.

7.4 Blank solution

Fill a 25 ml volumetric flask three quarters full with extraction solution (5.1), add 0,5 ml of phosphoric acid (5.3) and 0,5 ml of diphenylcarbazide solution (5.2) and make up to the mark with extraction solution (5.1) and mix well. Prepare this solution daily and store it in the dark. Treat the blank solution in the same way as the analytical solution, excluding the solid phase extraction.

7.5 Calibration

Prepare calibrating solutions from the standard solution (5.5). The chromium concentration in these solutions should cover the expected range of measurements.

Prepare the calibration solutions in 25 ml volumetric flasks (6.5).

Plot a suitable calibration curve by using at least six standards, within the range 0,5 ml to 15 ml of standard solution (5.5). Pipette the given volumes of standard solution (5.5) into 25 ml volumetric flasks. Add 0,5 ml of phosphoric acid (5.3) and 0,5 ml diphenylcarbazide solution (5.2) to each flask. Make up to volume with extraction solution (5.1), mix well and allow to stand for (15 ± 5) min. Measure the absorbance of the solutions in the same photometric cell (6.8) as the samples at 540 nm against the blank obtained in 7.4.

Plot the chromium(VI) concentrations in micrograms per millilitre (µg/ml) against the absorbance measured. Plot the chromium(VI) concentration on the x-axis and the absorbance on the y-axis.

In interlaboratory tests, the 40 mm cell (6.8) proved to be most suitable. The standard solutions described above are intended for analysis using a 40 mm cell. In some cases, however, it may be suitable to use higher or lower cell path length. Care shall be taken to ensure that the calibration range used is within the linear measuring range of the spectrophotometer.

7.6 Determination of the recovery rate

7.6.1 Influence of the matrix

The determination of the recovery rate is important to provide information about possible matrix effects which can influence the results.

Spike a 10 ml aliquot of the solution obtained in 7.2 with a suitable volume of chromium(VI) solution to increase the chromium concentration by up to 10 mg/kg. Select the concentration of the spiking solution in that way that the final volume of the spiked solution is maximum 11 ml. Treat this solution in the same way as the sample (recording the absorbance as $A_{1\mathrm{S}}$ and $A_{2\mathrm{S}}$). (See 7.3.)

The absorbance of the solution shall be within the range of the calibration curve, otherwise repeat the procedure using a smaller aliquot. The recovery rate shall be greater than 80 %.

NOTE 1 If the added chromium(VI) is not detected, this is an indication that the leather contains reducing agents. In some cases, if the recovery rate according to 7.6.2 is greater than 90 %, and after intensive considerations, this leads to the conclusion that this leather has no chromium(VI) content (below detection limit).

NOTE 2 The recovery rate is an indicator of whether the procedure works or whether matrix effects are affecting the results. Normally, the recovery rate is greater than 80 %.

7.6.2 Influence of the SPE material (6.9)

Pipette a volume of solution (5.5) which corresponds to the chromium(VI) content of the leather into a 100 ml volumetric flask and make up to volume with extraction solution (5.1).

Treat this solution in the same way as the leather extract. Determine the content in this solution in the same way as that of the leather extract and compare with the calculated content. In cases where no chromium(VI) was detected in the leather sample, the concentration of the solution shall be 6 µg/100 ml. The recovery rate shall be greater than 90 %. If the recovery rate is equal to or lower than 90 %, the SPE material is not suitable for this procedure and shall be substituted.

8 Calculation and expression of results

8.1 Calculation of chromium(VI) content

$$w_{\mathrm{Cr(VI)}} = \frac{(A_1 - A_2) \cdot V_0 \cdot V_4 \cdot V_2}{V_1 \cdot V_3 \cdot m \cdot F} \tag{1}$$

where

$w_{\mathrm{Cr(VI)}}$ is the mass fraction, expressed in milligrams per kilogram (mg/kg), of extractable chromium(VI) in leather;

A_1 is the absorbance of sample solution with DPC;

A_2 is the absorbance of sample solution without DPC;

F is the gradient of calibration curve (y/x), expressed in millilitres per microgram (ml/µg);

m is the mass of the leather sample taken, expressed in grams (g);

V_0 is the extract volume of the initial sample, expressed in millilitres (ml);

V_1 is the aliquot taken from the extract volume of the initial sample, expressed in millilitres (ml);

V_2 is the total eluate (S_1) volume, after passage through the SPE column, to which the aliquot V_1 was made up, expressed in millilitres (ml);

V_3 is the aliquot taken from solution S_1, expressed in millilitres (ml);

V_4 is the final make-up volume of the aliquot from S_1, expressed in millilitres (ml).

Result based on dry matter:

$$w_{\mathrm{Cr(VI)\text{-}dry}} = w_{\mathrm{Cr(VI)}} \cdot D \tag{2}$$

where D is the factor for conversion to dry matter:

$$D = \frac{100}{100 - w} \tag{3}$$

where w is the mass fraction of the volatile matter determined using ISO 4684, expressed as a percentage.

8.2 Recovery rate (according to 7.6.1)

$$\eta = \frac{(A_{1\mathrm{s}} - A_{2\mathrm{s}}) - (A_1 - A_2)}{\rho \cdot F} \cdot 100 \tag{4}$$

where

η is the recovery rate, expressed in percent (%);

ρ is the mass concentration of chromium(VI) spiked, expressed in micrograms per millilitre (µg/ml);

F is the gradient of calibration curve, expressed in millilitres per microgram (ml/µg);

$A_{1\mathrm{s}}$ is the absorbance of solution after adding chromium(VI) and DPC;

A_{2s} is the absorbance of solution after adding chromium(VI), but without adding DPC;

A_1 is the absorbance of sample solution with DPC;

A_2 is the absorbance of sample solution without DPC.

8.3 Expression of results

The chromium(VI) content is given in milligrams per kilogram (mg/kg) rounded to the nearest 0,1 mg. The content is based on dry matter. The volatile matter, determined according to ISO 4684, is given in percent (%) rounded to the nearest 0,1 %.

The extraction matrix for leather is complex (for example due to coloration) and results below 3 mg/kg show a large variation and have limited reliability; therefore the limit of quantification should be considered 3 mg/kg.

In the case of levels of chromium(VI) being detected above 3 mg/kg, the UV/VIS spectrum of the test solution should be compared with a standard solution (5.5) to determine whether the positive result is due to interfering substances.

9 Test report

The test report shall include the following information:

a) the chromium(VI) content(s) obtained from 8.1 to the nearest 0,1 mg/kg;

b) a reference to this document, i.e. ISO 17075-1:2017;

c) a description of the sample tested and details about sampling (7.1), if necessary;

d) the cell length used, if not 40 mm;

e) the volatile matter of the leather in percent (%) to the nearest 0,1 %;

f) the recovery rate in percent (%) if lower than 80 % or higher than 120 %;

g) details of any deviations from the procedure.

Annex A
(informative)

Solid phase extraction (SPE) material

If coloured substances, e.g. dyes, are co-extracted in the extraction process in 7.2, it is necessary to remove these potentially interfering substances from the extraction solution.

Experience has shown that various types of solid phase extraction materials packed in small columns can efficiently remove the coloured substances.

The amount of extracted coloured substances from leather can differ depending on the dye application and fixation process. It is important that the co-extracted dye is removed by the SPE column material.

Examples of suitable SPE materials[1]:

— in interlaboratory trials, cartridges with 1 g RP (C18) material and Dionex cartridges (Dionex OnGuard® RP) have been tested;

— other suitable cartridges are supplied by Waters (WATERS Sep-Pak® Plus tC18);

— also suitable SPE material is a cartridge with activated magnesium silicate, e.g. Florisil®.

In order to remove the coloured substances, in some cases it is advisable to use more than 1 g of SPE material or other phases. In any case, the recovery rate has to be tested very carefully.

Activated charcoal proved to be unsuitable for the decolourization of the extracts.

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[1] Dionex OnGuard® RP, WATERS Sep-Pak® Plus tC18 and Florisil® are examples of suitable products available commercially. This information is given for the convenience of the users of this document and does not constitute an endorsement by ISO of these products.

Annex B
(informative)

Accuracy

Results obtained from two separate interlaboratory trials carried out in 2007 are illustrated in Tables B.1 and B.2.

Table B.1 — Precision data from trial 1 for two types of leathers

Values in milligrams per kilogram (mg/kg)

Chromium(VI) content [a]	Repeatability [b]	Reproducibility [b]
7,04	1,58	3,22
7,83	1,11	3,54

[a] Mean values.

[b] *P* 95 %, factor 2,8.

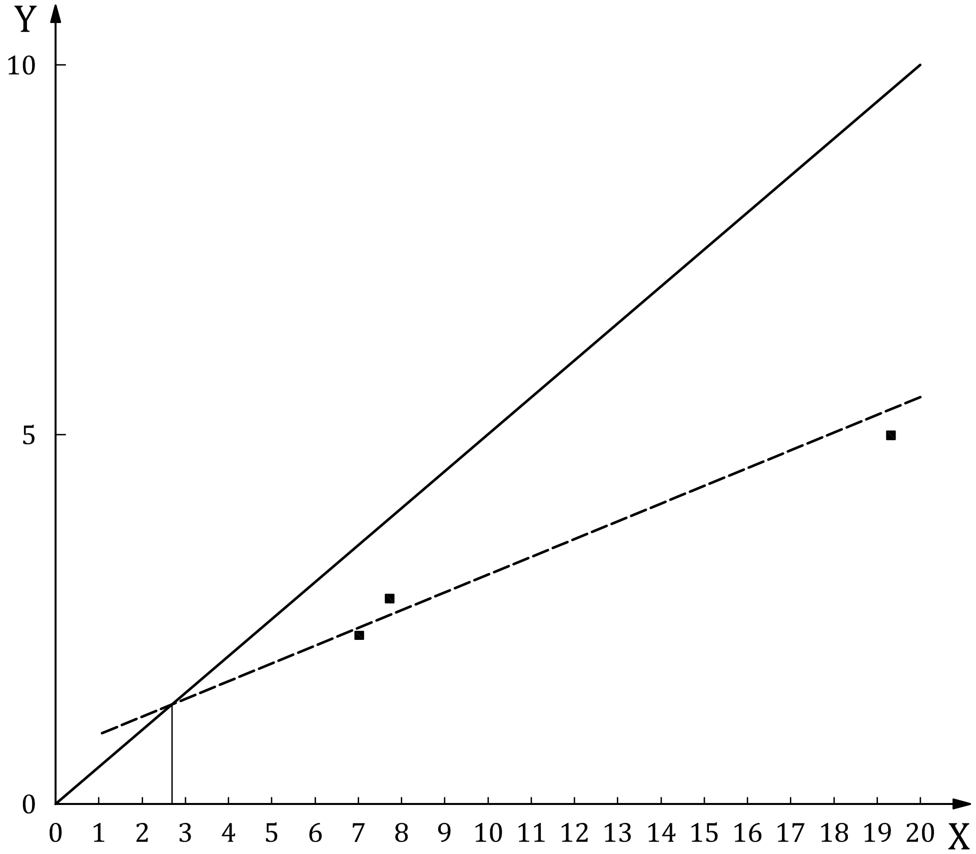
Table B.2 — Results of trial 2 for one type of leather

Laboratory	Chromium(VI) content mg/kg			Mean value mg/kg
1001a	19,40	18,70	19,3	19,13
1002a	20,60	21,30	21,7	21,20
1006a	21,80	22,20	21,8	21,93
1007a	19,00	18,00	21,0	19,33
1008a	25,00	24,30	24,9	24,73
1009a	19,20	17,80	18,3	18,43
1010a	16,80	13,60	13,7	14,70
501a	20,90	20,60	18,1	19,87
504a	22,00	20,00	19,00	20,33
505a	15,20	14,70	16,2	15,37
507a	24,10	23,40	25,7	24,40
508a	20,60	19,50	21,1	20,40
512a	18,70	15,40	16,5	16,87
2892a	18,40	19,00	—	18,70
2893a	16,40	16,40	—	16,40
2894a	19,40	19,30	18,9	19,20

Mean value: 19,4 mg/kg

Uncertainty: 5,0 mg/kg

Taking into account the data displayed in Figure B.1, the limit of quantification of the method is defined as 3 mg/kg for the mean value.



Key

X mean values of the interlaboratory tests (2003 to 2005)

Y measurements uncertainties associated to the mean value

Figure B.1 — Graphical determination of the quantification limit of the Cr(VI) test method

Annex C
(informative)

Comparison between the colorimetric method (ISO 17075-1) and the ionic chromatography method (ISO 17075-2)

The data in Table C.1 have been obtained in an interlaboratory trial (September 2015) with 13 participating laboratories. One cut leather sample with an unknown amount of chromium(VI) was analysed.

Table C.1 — Interlaboratory results from a leather sample using the colorimetric method (ISO 17075-1) and the ionic chromatography method (ISO 17075-2)

Colorimetric method (ISO 17075-1)		**Ionic chromatography method** (ISO 17075-2)	
Mean	Standard deviation	Mean	Standard deviation
mg/kg		mg/kg	
3,71	0,93	2,56	1,17

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