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Leather — Chemical determination of *N*-methyl-2-pyrrolidone (NMP) in leather

Cuir — Détermination de la teneur en N*-méthyl-2-pyrrolidone (NMP) dans le cuir*

Reference numbers
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Foreword

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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 WTO principles in the Technical Barriers to Trade (TBT) see the following URL: Foreword - Supplementary information

ISO 19070 was prepared by the Chemical Testing 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.

Introduction

Under the REACH regulation in the European Union (EU Regulation 1907/2006), *N*-methyl-2-pyrrolidone has been included in the candidate list of substances of very high concern (SVHC) and its use is restricted.

Accordingly, a new method is given for the determination of *N*-methyl-2-pyrrolidone in leather.

Leather — Chemical determination of *N*-methyl-2-pyrrolidone (NMP) in leather

1 Scope

This International Standard specifies a method to determine the amount of *N*-methyl-2-pyrrolidone (NMP) in leather and leather components.

This method may also be used for the determination of *N*-ethyl-2-pyrrolidone (NEP) in leather.

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. 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 4044, *Leather — Chemical tests — Preparation of chemical test samples*

3 Principle

The test sample is extracted using acetone at 50 °C in an ultrasonic bath for 1 h. An aliquot is then analysed using a gas chromatograph with a mass selective detector (GC-MS).

4 Reagents

All reagents shall be analytical grade.

4.1 **Acetone**, CAS[1)] No. 67-64-1.

4.2 ***N*-methyl-2-pyrrolidone**, at least 99,5 %, CAS No. 872-50-4.

5 Apparatus

Usual laboratory equipment and, in particular, the following.

5.1 **Analytical balance**, capable of reading to 0,000 1 g.

5.2 **Sealable jar**, approximately 20 ml, with lid (suitable for carrying out an acetone extraction at 50 °C).

5.3 **Ultrasonic bath** (temperature controlled).

5.4 **Micro-pipettes**, 50 µl and 100 µl.

5.5 **Pipettes**, 0,5 ml to 5 ml capacity.

5.6 **Volumetric flasks**, 100 ml.

1) CAS = Chemical Abstracts Service

5.7 Gas chromatograph, with mass selective detector (GC-MS).

5.8 PTFE-membrane filter, pore size 0,45 µm.

5.9 Sample vials, PTFE seal.

6 Procedure

6.1 Preparation of standard solutions

6.1.1 NMP stock solution (1 000 µg/ml)

Weigh accurately 0,1 g of *N*-methyl-2-pyrrolidone using the analytical balance (5.1), into a 100 ml volumetric flask (5.6) and fill up to the mark with acetone (4.1).

6.1.2 NMP Calibration solution

Four calibration solutions are used to establish the calibration curve. They are all prepared in 100 ml volumetric flasks, as specified in Table 1.

Table 1 — Preparation of calibration solutions

Calibration solution	Volume of the NMP stock solution (6.1.1)	Concentration of NMP in the calibration solution
1	0,1 ml	1 µg/ml
2	0,5 ml	5 µg/ml
3	1 ml	10 µg/ml
4	5 ml	50 µg/ml
Filled up to the mark with acetone		

6.2 Sample preparation

Take a representative sample from the leather or leather component. When possible sample according to ISO 2418.

The leather specimen shall be cut into pieces in accordance with ISO 4044. The dimensions of the pieces shall not be larger than 2 mm to 3 mm. Do not condition the sample.

6.3 Extraction procedure

Weigh accurately approximately 1 g of the leather pieces with the analytical balance (5.1) and place them into the sealable jar (5.2). Add 10 ml of acetone and extract the leather at (50 ± 2) °C in an ultrasonic bath for (1,0 ± 0,1) h.

After cooling to room temperature, filter the solution through a PTFE membrane filter (5.8) and transfer to a vial (5.9), and seal with a PTFE cap. This solution is now ready for instrumental analysis, which shall be performed within 24 h.

If there is to be any delay in analysing the prepared sample then it may be stored at (4 ± 2) °C for up to 24 h.

7 Chromatographic determination

The detection of the *N*-methyl-2-pyrrolidone may be performed using the GC-MS chromatographic technique given in Annex A.

If the amount of NMP in the sample is above the highest calibration point, a dilution step is necessary, which has to be considered in the calculation in Clause 8.

8 Quantification

The content of *N*-methyl-2-pyrrolidone is calculated as a mass fraction, *w*, in mg/kg, according to Formula (1):

$$w = \frac{A_{\text{NMP-S}} \cdot C_{\text{NMP-Std}} \cdot V}{A_{\text{NMP-Std}} \cdot m_{\text{S}}} \quad (1)$$

where

$A_{\text{NMP-S}}$ is the peak area of NMP in the sample;

$A_{\text{NMP-Std}}$ is the peak area of NMP in the calibration standard;

$C_{\text{NMP-Std}}$ is the concentration of NMP in the calibration standard, in µg/ml;

V is the final volume of sample, in ml, (V = 10 ml according to 6.3);

m_{S} is the mass of the sample as received, in g.

9 Performance of the method

The quantification limit shall be sufficient to ensure the analysis complies with any specification or legislative framework requirements, for example less than 1 000 mg/kg for *N*-methyl-2-pyrrolidone in the leather. The typical limit of quantification for this method is 5 mg/kg.

10 Test report

The test report shall include at least the following:

a) a reference to this International Standard, i.e. ISO 19070;

b) the date of the test;

c) all details necessary for complete identification of the sample tested;

d) the amount of *N*-methyl-2-pyrrolidone determined in the leather sample, in mg/kg;

e) the limit of detection for this method;

f) any deviation from the analytical procedure specified in this International Standard.

Annex A
(informative)

Chromatographic analysis

A.1 Preliminary remark

As the instrumental equipment (5.7) of the laboratories may vary, no generally applicable instructions can be provided for chromatographic analyses. The following procedures have been tested and used.

A.2 Gas chromatography with mass selective detector (GC-MS)

Column:	5 % phenyl – 95 % dimethylpolysiloxane, 30 m × 0,25 mm × 0,25 µm
Carrier gas:	helium; flow 1,2 ml/min
Injection system:	splitless
Injector temperature:	250 °C
Injection volume:	1 µl
Temperature programme:	60 °C at 4 min, 15 °C/min to 140 °C and 30 °C/min to 300 °C for 1 min
Transfer line temperature:	300 °C
Source Temperature:	230 °C
Quadrupole Temperature:	150 °C
MS-Detection (SIM/SCAN Mode):	SCAN: m/z 60–110; SIM: m/z 99

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