



The determination of chromium (VI): Standardisation status

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The co-existence of two different procedures for determining hexavalent chromium in leather across Europe has placed this hazardous substance once more in the midst of controversy.

Broadly, the German analytical procedure according to DIN 53314^(Legend 1) has been used. The European technical specification CEN/TS 14495 is based upon the German standard, but seeks to eliminate critical points of that procedure. So, for example, the possibility to obtain false positive results in the case of strongly coloured extracts was reduced by means of a discolouring step. The European test procedure was also adopted by EN 420 'Protective gloves—General requirements and test procedures' and by EN 20344 'Protective shoes—General requirements and test procedures'.

Inter-laboratory trials intended to validate the European test procedure have shown that due to the additional work step (discolouring of the extract) and different dilution steps worse characteristics of the analytical procedure in comparison to DIN 53314 were obtained. This applies especially to the detection and quantification limit: while DIN reaches a detection limit of 3 mg/kg, for the CEN procedure 10 mg/kg are achieved.

This 'increase' in the detection limit has met with criticism, for instance, from the side of consumer protection. Objections were even expressed from the protection of labour area: the test procedure does not comply with the protection level stated by the European PPE guideline. Accordingly, the decision was taken by the European Commission not to list the standard EN 420:2003 amongst harmonised standards of the PPE guideline.

Simultaneously, CEN was authorised to develop a test method with improved performance characteristics. This should be suitable to clearly determine chromium (VI) contents below the value considered as harmless by medical research. Of increasing importance for this purpose is a procedure using Ion Chromatography. This approach is also in the course of validation and, should the situation arise, will be able to realise a detection limit of explicitly below 3 mg/kg.

The introduction of CEN/TS 14495 in the lab routine showed several inconsistencies in this context, which produced a negative effect on the performance, precision and sensitivity of the photometric test procedure. The modifications proposed by

Lederinstitut Gerberschule, Reutlingen,^(Legend 2) were included in the standard. These were then validated by means of an inter-laboratory trial within the German standardisation board for chemical test methods (NMP 552) for leather. The evaluation of the ring trial mirrors a considerable improvement of the performance characteristics regarding repeatability and reproducibility.

Afterwards, the quality assurance procedure of the photometric test method was extended to the European area. Ten different laboratories located in France, Germany, Italy, Spain and the United Kingdom took part. Contrary to the ring trial of NMP 552, in this extended investigation some of the laboratories did not have any experience of the test method at that time (February 2006). Accordingly, this fact had an impact on the precision data of the procedure. While the precision in the lab remained consistently good, the reproducibility was somewhat impaired in comparison to the NMP 552 ring trial.

But both inter-laboratory trials showed that the procedure was improved. Subsequently, the proposed modifications to the test procedure, as well as the results of the inter-laboratory trial, were incorporated in the draft standard EN ISO 17075. After the voting procedure, the draft will be published as a full standard in the last quarter of 2007. Consequently, DIN 53314 will be withdrawn and CEN/TS 14495 replaced. In this way only one analytical procedure for the determination of hexavalent chromium able to consistently detect 3 mg/kg chromium (VI) in leather will be available.

The relevant standards of the occupational health and safety area (EN 420 and EN 20344) can therefore be revised, and EN 420 could be included in the list of harmonised standards of the PPE guideline. 

Legend

- 1) The European technical specification CEN/TS 14495 is identical to specification IUC 18.
- 2) Comments on the determination of Cr(VI) by test method IUC18/CEN/TS14495, R. Meyndt, C. Hauber, H-P Germann, (Lederinstitut Gerberschule Reutlingen): *World Leather*, December 2004/January 2005, p31-33.