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Round robin test for odour testing of migration waters

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ABSTRACT

For a round robin test for EN 1420-1 (Odour assessment for organic materials in contact with drinking water) with 14 contributing laboratories from 10 European countries segments of a plastic pipe were sent to the laboratories which performed a migration test and an odour analysis of the migration waters (water that had contact with the organic material) according to the procedure described in the standard from 1999. In addition reference substances (Methyl *tert*-butyl ether, 1-butanol and hexanal) were investigated for their suitability to qualify the panels and the individual panellists. Methyl *tert*-butyl ether (MtBE) and 1-butanol proved to be suitable for this purpose, whereas hexanal showed a wide distribution of the individual odour threshold concentrations. Both possible testing options (unforced and forced choice) were performed and gave similar results. However, with respect to the qualification of the panellists and the data analysis the unforced choice procedure showed advantages. As human olfactory perception is used for the analysis, the reproducibility and the comparability between laboratories is of particular concern. For the pipe material the TON results of the different laboratories were in a range of ±1.5 dilutions based on a dilution factor of 2. This might be improved by taking the individual sensitivities of the panellists into account more strongly. Appropriate measures for the improvement of the test method appear to be the use of the proposed reference substances for the training of the panellists as well as the auditing and the selection of the panellists. The results of this round robin test are used in the revision process of the standard.

1. Introduction

1.1. *Odour and flavour testing*

Odour and taste of drinking water are the consumers' first impressions of its quality and may cause rejection. Odours and flavours of drinking waters may originate from substances present in the water source. Additionally, materials in contact with drinking water may release substances into the drinking water which have a sensory impact (Rigal, 1992; Skjevrak et al., 2003; Tomboulian et al., 2004; Heim and Dietrich, 2007; Lund et al., 2011). The mass transfer of substances from organic materials into water is known as migration and the water that had contact with the material is specified as migration water. To ensure the aesthetic and hygienic quality of materials intended to come into contact with drinking water such products should be tested and certified or approved. For organic materials it is important that the organoleptic parameters are considered when products

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are tested. Although much progress has been achieved in identifying substances causing off-flavours of waters, an organoleptic analysis of the materials remains necessary because up to now odour and flavour perception by humans cannot be completely covered by instrumental analysis.

Odour and flavour tests of materials in contact with drinking water require a procedure both to prepare migration waters and to characterise their odour and flavour. The latter can follow a similar or the same procedure as is used for the determination of odour and flavour of real drinking waters. The European standard EN 1420-1 describes the preparation of migration waters and refers to EN 1622 for odour and flavour determination. The Australian and New Zealand standard AS/NZS 4020:2005 "Testing of products for use in contact with drinking water" includes the preparation of migration waters and a procedure to determine their taste, but does not require a test for odour. The American Standard NSF/ANSI 61 as the basis for the certification of products for the US market neither includes nor refers to an odour or flavour test.

For the determination of the odours or flavours of waters several testing procedures are in use (Bruvold and Pangborn, 1989). Besides the specific aroma, the intensity of the odour and flavour characterizes a sample. Intensity can be determined by diluting the samples down to a concentration at which panellists can no longer perceive the odour or flavour, giving a threshold odour number (TON) and a threshold flavour number (TFN). These numbers express the dilution ratio beyond which the diluted sample does not have any perceptible odour or flavour. Another approach is the flavour profile method (Krasner et al., 1985), which is a standardized method in the US (standard method 2170 in Standard Methods for the Examination of Water and Wastewater). This method uses a trained panel to determine the aroma and its intensity. The samples are not diluted and for the classification basic tastes standards at different intensities are used. The results are obtained after a discussion process by the entire panel. For testing foodstuff several descriptive sensory methods are used (Lawless and Heymann, 2010). A common method is the Quantitative Descriptive Analysis® (QDA) by Stone et al. (1974). According to this method the intensity of the different odours are quantified on a scale by each trained panellist individually. This allows a statistical evaluation of the results of the complete panel.

For the testing of materials in contact with drinking water EN 1420-1 describes the preparation of migration waters including the defined testing conditions. For the determination of odour and flavour intensity the standard refers to EN 1622 which describes the methods to determine the threshold odour number (TON) and the threshold flavour number (TFN) by a panel of persons. A shortage of the standard EN 1420-1 (1999) in combination with EN 1622 is the lack of requirements to ensure the reproducibility and the comparability between the results of different laboratories. The standard informs in its Annex E about a procedure to select new panellists for an existing panel. For this the results of the candidate are compared with the results of the accepted panellists. This allows ensuring the reproducibility for one laboratory. However, the standard does not include a procedure to ensure the comparability between different laboratories.

Additionally, EN 1622 (2006) gives different options for the determination of the threshold values (Rigal, 1995), i.e. the forced or the unforced choice test as well as a simplified method. According to the forced procedure the panellist has to select a sample even when he or she is unable to perceive a difference between a diluted sample and one or two blanks, whereas according to the unforced choice the panellist has only to choose one of the offered samples if he or she notes a difference. Another option given by the standard is the dilution ratio x in the range $1.3 < x < 3$ for the preparation of the sample dilutions. Therefore, the test method is not standardized in detail and it has never been investigated, whether these testing options have an influence on the test results.

Especially when products are tested for an approval or certification it is important that the results for odour and taste are reproducible and that laboratories performing these tests deliver comparable results. For this kind of test it is not necessary to represent the varieties of sensitivities in the population. However, by setting the acceptance criteria it has to be ensured that also sensitive consumers will not reject the drinking water due to odour when it came into contact with the accepted products. For the setting of the acceptance criteria it has also to be considered that the migration procedure described in EN 1420 will in most cases overestimate the migration of substances into the drinking water. EN 1420 does not include acceptance criteria, as for this a regulatory decision is required. As long as in Europe no harmonized acceptance procedure for materials or products in contact with drinking water exists, the acceptance criteria will have to be defined in each Member State individually.

1.2. *Qualification of panellists*

The determination of aromas and their intensities in waters strongly depends on the panel members (panellists). Individuals exhibits different sensitivities and also the sensitivity for one person is considerably variable (intra-individual variation) (Stevens et al., 1988). Others (Walker et al., 2003 or Linschoten et al., 2001) report a far less intra-individual variation for non-trained individuals. Based on our experiences with panels performing odour test of migration waters as a matter of routine we assume that training of panellists reduces the intra-individual variation.

The variation between persons (inter-individual variation) might be reduced by a qualification of the panellists. For this reason reference substances are used to train and qualify panellists (Ömür-Özbek and Dietrich, 2008). EN 13725 describes a procedure for the qualification and selection of panellists for the dynamic olfactometry of gaseous contaminations. For this a reference gas (1-butanol in nitrogen) is used (Capelli et al., 2010). A different approach for the qualification of panellists is the use of ranking procedures for the selection of panellists. Huber et al. (2002) used 5 different solutions of 1-butanol in water which the panellists have to rank according to their odour intensity. Experience with 1-butanol as reference substance is also available from its use to score the intensities of off-odours for the evaluation of package materials (Reinbach et al., 2011).

Another substance used as odour reference standard for sensory analysis of drinking water is hexanal (Ömür-Özbek