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Workplace air — Determination of lithium hydroxide, sodium hydroxide, potassium hydroxide and calcium dihydroxide — Method by measurement of corresponding cations by suppressed ion chromatography

Air des lieux de travail — Détermination de la teneur en hydroxyde de lithium, hydroxyde de sodium, hydroxyde de potassium et dihydroxyde de calcium — Méthode par mesurage des cations correspondants par chromatographie ionique

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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

The committee responsible for this document is ISO/TC 146, *Air quality,* Subcommittee SC 2, *Workplace atmospheres*.

Introduction

The health of workers in many industries is at risk through exposure by inhalation of lithium hydroxide, sodium hydroxide, potassium hydroxide, and calcium dihydroxide. Industrial hygienists and other public health professionals need to determine the effectiveness of measures taken to control workers' exposure, and this is generally achieved by making workplace air measurements. This International Standard has been published in order to make available a method for making valid exposure measurements for lithium hydroxide, sodium hydroxide, potassium hydroxide, and calcium dihydroxide in use in industry. It will be of benefit to: agencies concerned with health and safety at work; industrial hygienists and other public health professionals; analytical laboratories; industrial users of lithium hydroxide, sodium hydroxide, potassium hydroxide, and calcium dihydroxide and their workers; etc.

It has been assumed in the drafting of this International Standard that the execution of its provisions and the interpretation of the results obtained are entrusted to appropriately qualified and experienced people.

Workplace air — Determination of lithium hydroxide, sodium hydroxide, potassium hydroxide and calcium dihydroxide — Method by measurement of corresponding cations by suppressed ion chromatography

1 Scope

This International Standard specifies a method for the determination of the time-weighted average mass concentration of lithium hydroxide (LiOH), sodium hydroxide (NaOH), potassium hydroxide (KOH), and calcium dihydroxide [$Ca(OH)_2$] in workplace air by collection of the particulate hydroxides on a filter and analysis of the corresponding cations using ion chromatography.

For aerosol sampling, the method is applicable to the personal sampling of the inhalable fraction of airborne particles, as defined in ISO 7708, and to static (area) sampling.

The method is applicable to the determination of masses of 0,005 mg to at least 2,5 mg of lithium per sample and 0,01 mg to at least 5 mg of sodium, potassium, and calcium per sample.

The concentration range of particulate LiOH, NaOH, KOH, and $Ca(OH)_2$ in air for which the measuring procedure is applicable is determined by the sampling method selected by the user. For a 1 m^3 air sample, the working range is approximately 0,002 mg m^{-3} to at least 20 mg m^{-3} for all four hydroxides. For a 30 l air sample, the lower limit of the working range is approximately 0,1 mg m^{-3} for all four hydroxides.

The procedure does not allow differentiation between the hydroxides and their corresponding salts if both are present in the air. If the cations are present alone in the form of hydroxides, the method is specific for these basic compounds. In other circumstances, the results obtained represent the highest concentration of the hydroxides that could be present in the sampled air. (See 12.6.)

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 1042, *Laboratory glassware — One-mark volumetric flasks*

ISO 7708:1995, *Air quality — Particle size fraction definitions for health-related sampling*

ISO 8655-1, *Piston-operated volumetric apparatus — Part 1: Terminology, general requirements and user recommendations*

ISO 8655-2, *Piston-operated volumetric apparatus — Part 2: Piston pipettes*

ISO 8655-6, *Piston-operated volumetric apparatus — Part 6: Gravimetric methods for the determination of measurement error*

ISO 13137:—[1)], *Workplace atmospheres — Pumps for personal sampling of chemical and biological agents — Requirements and test methods*

EN 13205-1, *Workplace atmospheres — Assessment of performance of instruments for measurement of airborne particle concentrations — Part 1: General requirements*

1) To be published.

3 Terms and definitions

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

3.1 General definitions

3.1.1
breathing zone
<general definition> space around the worker's face from which breath is taken

[SOURCE: EN 1540:2011, 2.4.5, modified]

3.1.2
breathing zone
<technical definition> hemisphere (generally accepted to be 0,3 m in radius) extending in front of the human face, centred on the midpoint of a line joining the ears; the base of the hemisphere is a plane through this line, the top of the head, and the larynx

Note 1 to entry: The definition is not applicable when respiratory protective equipment is used.

[SOURCE: EN 1540:2011, 2.4.5, modified]

3.1.3
chemical agent
any chemical element or compound, on its own or admixed, as it occurs in the natural state or as produced, used, or released, including release as waste, by any work activity, whether or not produced intentionally and whether or not placed on the market

[SOURCE: Council Directive 98/24/EC, Art. 2(a)]

3.1.4
exposure (by inhalation)
situation in which a chemical agent is present in the air that is inhaled by a person

[SOURCE: EN 1540:2011, 2.4.1, modified]

3.1.5
occupational exposure limit value
limit value
limit of the time-weighted average of the concentration of a chemical agent in the air within the breathing zone of a worker in relation to a specified reference period

[SOURCE: Council Directive 98/24/EC, Art. 2(d)]

EXAMPLE Threshold limit values® (TLVs) established by the ACGIH,[15] indicative occupational exposure limit values (IOELVs) promulgated by the European Commission,[16] and national limit values. Information on national limit values is available from the International Labour Organization (ILO)[17] and on the GESTIS database.[18]

3.1.6
measuring procedure
measurement procedure
set of operations, described specifically, used for the sampling and analysis of chemical agents in air

Note 1 to entry: A measuring procedure for the sampling and analysis of chemical agents in air usually includes the following steps: preparation for sampling, sampling, transportation and storage, preparation of samples for analysis, and analysis.

[SOURCE: ISO/IEC Guide 99:2007, modified]

3.1.7
operating time
period during which the pump can be operated at a specified flow rate and pressure drop without recharging or replacing the battery

[SOURCE: ISO 13137:—, 3.12]

3.1.8
reference period
specified period of time for which the occupational exposure limit value of a chemical agent applies

Note 1 to entry: The reference period is usually 8 h for long-term measurements and 15 min for short-term measurements.

Note 2 to entry: Examples for different reference periods are short-term and long-term limit values, such as those established by the ACGIH.[15]

[SOURCE: EN 1540:2011, 2.4.7, modified]

3.1.9
workplace
designated area or areas in which the work activities are carried out

[SOURCE: EN 1540:2011, 2.5.2]

3.2 Particle size fraction definitions

3.2.1
inhalable convention
target specification for sampling instruments when the inhalable fraction is of interest

[SOURCE: ISO 7708:1995]

3.2.2
inhalable fraction
mass fraction of total airborne particles which is inhaled through the nose and mouth

Note 1 to entry: The inhalable fraction depends on the speed and direction of air movement, on breathing rate, and other factors.

[SOURCE: ISO 7708:1995]

3.2.3
total airborne particles
all particles surrounded by air in a given volume of air

Note 1 to entry: Because all measuring instruments are size-selective to some extent, it is often impossible to measure the total airborne particles concentration.

[SOURCE: ISO 7708:1995]

3.3 Sampling definitions

3.3.1
air sampler
device for separating chemical agents from the surrounding air

Note 1 to entry: Air samplers are generally designed for a particular purpose, e.g. for sampling gases and vapours or for sampling airborne particles.

[SOURCE: EN 1540:2011, 3.2.1, modified]

3.3.2
personal sampler
sampler, attached to a person, that collects gases, vapours, or airborne particles in the breathing zone to determine exposure to chemical agents

[SOURCE: EN 1540:2011, 3.2.2]

3.3.3
personal sampling
process of sampling carried out using a personal sampler

[SOURCE: EN 1540:2011, 3.3.3]

3.3.4
static sampler
area sampler
sampler, not attached to a person, that collects gases, vapours, or airborne particles at a particular location

[SOURCE: EN 1540:2011, 3.2.3]

3.3.5
static sampling
area sampling
process of air sampling carried out in a particular location

[SOURCE: EN 1540:2011, 3.3.4]

3.4 Analytical definitions

3.4.1
analysis
all operations carried out after sample preparation to determine the amount or concentration of the analyte(s) of interest present in the sample

[SOURCE: EN 14902:2005, 3.1.1, modified]

3.4.2
blank solution
solution prepared by taking a reagent blank, laboratory blank, or field blank through the same procedure used for sample dissolution

3.4.3
calibration blank solution
calibration solution prepared without the addition of any working standard solution

Note 1 to entry: The concentrations of Li, Na, K and Ca in the calibration blank solution are taken to be zero.

[SOURCE: EN 14902:2005, 3.1.3, modified]

3.4.4
calibration solution
solution prepared by dilution of the working standard solution, containing Li, Na, K, and Ca at concentrations that are suitable for use in calibration of the analytical instrument

[SOURCE: EN 14902:2005, 3.1.3, modified]

3.4.5
field blank
filter that is taken through the same handling procedure as a sample, except that it is not used for sampling, i.e. it is loaded into a sampler, transported to the sampling site, and then returned to the laboratory for analysis

3.4.6
laboratory blank
unused filter, taken from the same batch used for sampling, that does not leave the laboratory

3.4.7
linear dynamic range
range of concentrations over which the calibration curve for Li, Na, K, or Ca is linear

Note 1 to entry: The linear dynamic range extends from the detection limit to the onset of calibration curvature.

3.4.8
reagent blank
all reagents used in sample dissolution, in the same quantities used for preparation of laboratory blank, field blank, and sample solutions

3.4.9
sample dissolution
process of obtaining a solution containing Li, Na, K, and Ca from a sample, which might or might not involve complete dissolution of the sample

[SOURCE: EN 14902:2005, 3.1.25, modified]

3.4.10
sample preparation
all operations carried out on a sample, after transportation and storage, to prepare it for analysis, including transformation of the sample into a measurable state, where necessary

[SOURCE: EN 14902:2005, 3.1.24, modified]

3.4.11
sample solution
solution prepared from a sample by the process of sample dissolution

Note 1 to entry: A sample solution might need to be subjected to further operations, e.g. dilution, in order to produce a test solution that is ready for analysis.

[SOURCE: EN 14902:2005, 3.1.22, modified]

3.4.12
stock standard solution
solution, used for preparation of the calibration solutions, containing Li, Na, K, or Ca at a certified concentration that is traceable to national standards

[SOURCE: EN 14902:2005, 3.1.26, modified]

3.4.13
test solution
blank solution or sample solution that has been subjected to all operations required to bring it into a state in which it is ready for analysis

Note 1 to entry: "Ready for analysis" includes any required dilution. If a blank solution or sample solution is not subject to any further operations before analysis, it is a test solution.

[SOURCE: EN 14902:2005, 3.1.30, modified]

3.4.14
working standard solution
solution, prepared by dilution of the stock standard solution, that contains Li, Na, K, and Ca at concentrations that are better suited to preparation of calibration solutions than the concentrations of Li, Na, K, or Ca in the stock standard solutions

[SOURCE: EN 14902:2005, 3.1.32, modified]

3.5 Statistical terms

3.5.1
analytical recovery
ratio of the mass of analyte measured in a sample to the known mass of analyte in that sample

Note 1 to entry: The analytical recovery is usually given as a percentage.

[SOURCE: EN 1540:2011, 5.1.1]

3.5.2
bias
difference between the expectation of a test result or measurement result and a true value

Note 1 to entry: Bias is the total systematic error as contrasted to random error. There may be one or more systematic error components contributing to the bias. A larger systematic difference from the true value is reflected by a larger bias value.

Note 2 to entry: The bias of a measuring instrument is normally estimated by averaging the error of indication over an appropriate number of repeated measurements. The error of indication is the "indication of a measuring instrument minus a true value of the corresponding input quantity".

Note 3 to entry: In practice, the accepted reference value is substituted for the true value.

Note 4 to entry: In the case of measurement procedures for the sampling and analysis of chemical agents in air, the accepted reference value can be, for example, the certified value of a reference material, the concentration of a standard test atmosphere, or the target value of an interlaboratory comparison.

[SOURCE: ISO 3534-2:2006, 3.3.2]

3.5.3
coverage factor
k
numerical factor used as a multiplier of the combined standard uncertainty in order to obtain an expanded uncertainty

Note 1 to entry: A coverage factor, k, is typically in the range from 2 to 3.

[SOURCE: ISO/IEC Guide 98-3:2008]

3.5.4
combined standard uncertainty
u_c
standard uncertainty of the result of measurement when that result is obtained from the values of a number of other quantities, equal to the positive square root of a sum of terms, the terms being the variances or covariances of these other quantities weighted according to how the measurement result varies with changes in these quantities

[SOURCE: ISO/IEC Guide 98-3:2008]

3.5.5
expanded uncertainty
quantity defining an interval about a result of a measurement, expected to encompass a large fraction of the distribution of values that could reasonably be attributed to the measurand

[SOURCE: ISO/IEC Guide 98-3:2008]

3.5.6
precision
closeness of agreement of independent test/measurement results obtained under stipulated conditions

Note 1 to entry: Precision depends only on the distribution of random errors and does not relate to the true value or the specified value.

Note 2 to entry: The measure of precision is usually expressed in terms of imprecision and computed as a standard deviation of the test results or measurement results. Less precision is reflected by a larger standard deviation.

Note 3 to entry: Quantitative measures of precision depend critically on the stipulated conditions. Repeatability conditions and reproducibility conditions are particular.

[SOURCE: ISO 3534-2:2006, 3.3.4]

3.5.7
true value
value which characterizes a quantity or quantitative characteristic perfectly defined in the conditions which exist when that quantity or quantitative characteristic is considered

Note 1 to entry: The true value of a quantity or quantitative characteristic is a theoretical concept and, in general, cannot be known exactly.

[SOURCE: ISO 3534-2:2006, 3.2.5]

3.5.8
uncertainty (of measurement)
parameter associated with the result of a measurement that characterizes the dispersion of the values that could reasonably be attributed to the measurand

Note 1 to entry: The parameter may be, for example, a standard deviation (or a given multiple of it), or the width of a confidence interval.

Note 2 to entry: Uncertainty of measurement comprises, in general, many components. Some of these components may be evaluated from the statistical distribution of the results of a series of measurements, and can be characterized by standard deviations. The other components, which also can be characterized by standard deviations, are evaluated from assumed probability distributions based on experience or other information. ISO/IEC Guide 98-3:2008[4] refers to these different cases as type A and type B evaluations of uncertainty, respectively.

[SOURCE: ISO/IEC Guide 99:2007, modified]

4 Principle

4.1 A known volume of air is drawn through a filter mounted in an inhalable sampler (7.1.1) to collect particulate LiOH, NaOH, KOH, and $Ca(OH)_2$.

4.2 The filter is extracted with water or eluent solution (see 6.3 and 10.1.1), without heating, to solubilize the particulate hydroxides.

4.3 Aliquots of the sample solution are subjected to ion chromatography in order to separate the extracted Li, Na, K, and Ca from other cations. Following this separation, Li, Na, K, and Ca are measured using a conductivity detector.

4.4 Analytical results are obtained by plotting the measured conductivity as a function of concentration. They can be used for the assessment of occupational exposure to LiOH, NaOH, KOH, and $Ca(OH)_2$ in air.

5 Requirement

The measuring procedure shall comply with any relevant International, European, or National Standard that specifies performance requirements for procedures for measuring chemical agents in workplace air (e.g. EN 482[9]).

6 Reagents

During the analysis, use only reagents of recognized analytical grade and only water as specified in 6.1.

NOTE Na, K, and Ca are found ubiquitously in the environment, and their presence in reagents will lead to high blank values. It is therefore advisable to check the blank values of all chemicals before use.

6.1 Water, from a purification system that delivers ultrapure water having a resistivity greater than 0,18 MΩ·m (usually expressed by manufacturers of water purification systems as 18 MΩ·cm).

6.2 Sulfuric acid (H_2SO_4) solution, 2,5 mol l^{-1}.

NOTE A commercial solution may be used or 2,5 mol l^{-1} sulfuric acid may be prepared from concentrated sulfuric acid.

6.3 Sulfuric acid (H_2SO_4) solution, 0,004 5 mol l^{-1}, for use as eluent and for extraction of the sampling filters (see 10.1.3).

Dilute 1,8 ml of 2,5 mol l^{-1} sulfuric acid solution (6.2) into a 1 000 ml one-mark volumetric flask (7.2.2), dilute to the mark with water, stopper, and mix thoroughly.

6.4 Cartridge, for generation of eluent for chemically suppressed ion chromatography, suitable for use with the eluent generation system (7.2.6.2).

6.5 Standard solutions

6.5.1 Lithium stock standard solution

Use a commercial standard solution with a certified lithium concentration, e.g. 1 000 mg l^{-1} of Li, traceable to national standards. Observe the manufacturer's expiration date or recommended shelf life.

6.5.2 Sodium stock standard solution

Use a commercial standard solution with a certified sodium concentration, e.g. 1 000 mg l^{-1} of Na, traceable to national standards. Observe the manufacturer's expiration date or recommended shelf life.

6.5.3 Potassium stock standard solution

Use a commercial standard solution with a certified potassium concentration, e.g. 1 000 mg l^{-1} of K, traceable to national standards. Observe the manufacturer's expiration date or recommended shelf life.

6.5.4 Calcium stock standard solution

Use a commercial standard solution with a certified calcium concentration, e.g. 1 000 mg l^{-1} of Ca, traceable to national standards. Observe the manufacturer's expiration date or recommended shelf life.

6.5.5 Cation working standard solution, 25 mg l^{-1} of Li, 50 mg l^{-1} of Na, K, and Ca.

Accurately pipette appropriate volumes, e.g. 0,5 ml, of the Li stock standard solution (6.5.1) and 1,0 ml of the of Na, K, and Ca stock standard solutions (6.5.2 to 6.5.4) into a 20 ml plastic one-mark volumetric flask (7.2.3.1), dilute to the mark with water (6.1), stopper, and mix thoroughly. Prepare this solution fresh monthly.

7 Apparatus

7.1 Sampling equipment

7.1.1 Samplers, designed to collect the inhalable fraction of airborne particles, complying with EN 13205-1, manufactured from a material that does not react with alkaline hydroxides.

NOTE 1 If samplers have an internal filter cassette, this too has to be manufactured from a material that does not react with the hydroxides.

NOTE 2 Materials which do not react with hydroxides from which samplers and internal filter cassettes can be manufactured include polytetrafluoroethylene (PTFE) and other fluorinated polymers, polyvinylchloride (PVC), polyethylene, polypropylene, and polycarbonate.

7.1.2 Filters, of a diameter suitable for use with the samplers (7.1.1) with a collection efficiency of not less than 99,5 % for particles with a 0,3 µm diffusion diameter (see 2.2 of ISO 7708:1995), manufactured from a material that does not react with hydroxides, e.g. quartz fibre.

NOTE Na, K, and Ca are found ubiquitously in the environment and their presence in filter materials can lead to high blank values. It is therefore essential to check the blank values of each batch of filters used.

7.1.3 Sampling pumps, complying with the requirements of ISO 13137 and capable of maintaining the selected flow rate (see 9.1.1.2) to within ±5 % of the nominal value throughout the sampling period (see 9.1.2).

7.1.4 Flow meter, portable, with an accuracy that is sufficient to enable the volumetric flow rate (see 9.1.1.2) to be measured to within ±5 %.

The calibration of the flow meter shall be checked against a primary standard, i.e. a flow meter whose accuracy is traceable to national standards. If appropriate (see 9.1.3), record the atmospheric temperature and pressure at which the calibration of the flow meter was checked.

It is advisable that the flow meter used is capable of measuring the volumetric flow rate to within ±2 % or better.

7.1.5 Ancillary equipment

7.1.5.1 Flexible tubing, of a diameter suitable for making a leak-proof connection from the samplers (7.1.1) to the sampling pumps (7.1.3).

7.1.5.2 Belts or harnesses, to which the sampling pump can conveniently be fixed for personal sampling (except where the sampling pumps are small enough to fit in workers' pockets).

7.1.5.3 Tweezers, manufactured from or tipped with PTFE, for loading and unloading filters into samplers (9.2.2 and 10.1.3.1).

7.1.5.4 Thermometer, 0 °C to 50 °C, graduated in divisions of 1 °C or better, for measurement of atmospheric temperature, if required (see 9.1.3).

For applications at temperatures below freezing, the range of the thermometer shall extend to the appropriate desired range.

7.1.5.5 Barometer, suitable for measurement of atmospheric pressure, if required (see 9.1.3).

7.2 Laboratory apparatus

Use ordinary laboratory apparatus, and the following.

NOTE 1 It is preferable to use plastic labware rather than glassware.

NOTE 2 Na, K, and Ca are found ubiquitously in the environment. This can lead to elevated blanks so it is especially important to take great care that all disposable plastic labware is checked for Na, K, and Ca contamination and that all reusable laboratory apparatus is thoroughly clean before use.

7.2.1 Disposable gloves, impermeable, to protect the hands from contact with toxic and corrosive substances.

PVC gloves are suitable.

7.2.2 Glassware, beakers and one-mark volumetric flasks, of suitable capacities, between 100 ml and 1 000 ml capacity, complying with the requirements of ISO 1042, made of borosilicate glass (3.3) complying with the requirements of ISO 3585, cleaned before used with water (6.1).

Alternatively, the flasks may be cleaned using a laboratory washing machine and afterwards rinsed thoroughly with water.

7.2.3 Plastic labware

7.2.3.1 One-mark volumetric flasks, of suitable capacities, between 10 ml and 1 l.

7.2.3.2 Screw-cap polyethylene vessels, disposable, of a suitable capacity, e.g. 10 ml.

7.2.3.3 Beakers, of a suitable capacity, e.g. 50 ml.

7.2.3.4 Graduated centrifuge tubes, with caps, of a suitable capacity, e.g. 10 ml.

7.2.3.5 Filter funnels, of a size suitable for use in transferring washings from the internal surfaces of the sampler (7.1.1) into a tube.

7.2.3.6 Disposable filters, PTFE, pore size 0,45 µm, for use in ion chromatography.

7.2.3.7 Disposable syringes, of a suitable capacity, e.g. 2 ml or 5 ml, with luer lock connector, for use with disposable filters (7.2.3.6).

7.2.3.8 Autosampler vials, of a suitable capacity, e.g. 1,5 ml to 2 ml.

7.2.4 Piston-operated volumetric instruments, complying with the requirements of ISO 8655-1 and tested in accordance with ISO 8655-6, including pipettors, with capacities of 10 µl to 5 ml, complying with the requirements of ISO 8655-2, for the preparation of standard solutions, calibration solutions, and dilution of samples.

7.2.5 Ultrasonic bath, preferably with a timer, suitable for use in the extraction of hydroxides.

7.2.6 Ion chromatograph, having the components listed in 7.2.6.1 to 7.2.6.9. Components and tubing that come into contact with the sample solution or eluent shall, as far as possible, be comprised of inert materials, e.g. poly ether ether ketone (PEEK).

7.2.6.1 Pump, capable of delivering a constant flow within the range 0,1 ml min^{-1} to 5 ml min^{-1} at a pressure of 15 MPa to 150 MPa.

7.2.6.2 Eluent generation system, for producing an eluent suitable for use with the selected separator column (7.2.6.5 and e.g. Reference [20]).

7.2.6.3 Sample injection system, comprising of a low dead-volume, electronically controlled metallic valve fitted with a sample loop having a volume of up to 500 µl, for injecting the sample solution into the eluent stream.

7.2.6.4 Guard column, placed before the separator column (7.2.6.5) to protect it from fouling by particles or strongly adsorbed organic constituents of the sample solution.

7.2.6.5 Separator column

7.2.6.5.1 Separator column for chemically suppressed ion chromatography, packed with high-capacity pellicular cation exchange resin, suitable for resolving Li, Na, K, and Ca from other cations.

7.2.6.5.2 Separator column for non-suppressed ion chromatography, packed with high-capacity pellicular cation exchange resin, suitable for resolving Li, Na, K, and Ca from other cations.

7.2.6.6 Suppressor module for chemically suppressed ion chromatography, suitable for use with the separator column (7.2.6.5.1).

7.2.6.7 Conductivity detector, flow through, low volume, with a non-metallic flow path.

NOTE A conductivity detector can be used with both chemically suppressed and non-suppressed ion chromatography.

7.2.6.8 Recorder, integrator, or computer, compatible with detector output, capable of recording detector response as a function of time, for the purpose of measuring peak height or area.

The use of an automated system is recommended.

7.2.6.9 Container, suitable for use as a reservoir for storing eluent or water used for eluent generation (7.2.6.2).

7.2.7 pH meter

8 Occupational exposure assessment

8.1 General

This International Standard pertains to the taking of personal and static samples. Refer to relevant International, European, or National Standards (e.g. EN 482,[9] EN 689,[10] ASTM E 1370,[7] etc.) for guidance on how to develop an appropriate assessment strategy and for general guidance on measurement strategy.

8.2 Personal sampling

Exposure of workers to LiOH, NaOH, KOH, and $Ca(OH)_2$ shall normally be determined by personal sampling, since the concentration of LiOH, NaOH, KOH, and $Ca(OH)_2$ in the breathing zone can be different from the background level in the workplace.

8.3 Static sampling

Static sampling may be carried out, if appropriate, to assess the exposure of workers in a situation where personal sampling is not possible (see NOTE in 9.1.2.1 for an example of such a situation), to characterize the background level of LiOH, NaOH, KOH, and $Ca(OH)_2$ in the workplace in order to give an indication of the efficiency of ventilation, or to provide information on the location and intensity of an emission source.

8.4 Selection of measurement conditions and measurement pattern

8.4.1 General

8.4.1.1 Sampling shall be carried out in such a way as to cause the least possible interference with the worker and the normal performance of the job, and to provide samples that are representative of normal working conditions and that are compatible with the analytical method.

8.4.1.2 The pattern of sampling shall take into consideration practical issues, such as the nature of the measurement task and the frequency and duration of particular work activities.

8.4.2 Screening measurements of variation of concentration in time and/or space

Screening measurements of variation of concentration in time and/or space are used to

— provide information on the likely pattern of concentration of chemical agents,

— identify locations and periods of elevated exposure,

— provide information on the location and intensity of emission sources, and

— estimate the effectiveness of ventilation or other technical measures.

8.4.3 Screening measurements of time-weighted average concentration and worst case measurements

8.4.3.1 Screening measurements of time-weighted average concentration are performed to obtain relatively crude quantitative information on the exposure level in order to decide whether an exposure problem exists at all and if so to appraise its possible seriousness. These measurements can also be used to determine if the exposure is well below or well above the limit value.

8.4.3.2 Screening measurements of time-weighted average concentration are typically carried out in the initial stages of a survey to assess the effectiveness of control measures. Sampling may be carried out during representative work episodes to obtain clear information about the level and pattern of exposure, or worst case measurements may be made.

NOTE Screening measurements of time-weighted average concentration made to identify clearly work episodes during which highest exposure occurs are typically referred to as 'worst case measurements'.

8.4.4 Measurements near an emission source

Measurements may be performed near an emission source to provide information on the location and intensity of the source. In association with other information, they can allow the elimination of a suspected source as a significant contributor to exposure.

8.4.5 Measurements for comparison with limit values and periodic measurements

8.4.5.1 Measurements for comparison with limit values

8.4.5.1.1 Measurements for comparison with limit values are performed to provide accurate and reliable information on, or allow the prediction of, the time-weighted average concentration of a specific chemical agent in the air that could be inhaled (see EN 482[9]).

8.4.5.1.2 For making measurements for comparison with a short-term exposure limit, the sampling time shall be as close as possible to the reference period, which is typically 15 min.

8.4.5.1.3 For making measurements for comparison with a long-term exposure limit, samples shall be collected for the entire working period, if possible, or during a number of representative work episodes (see 9.1.2.1 for the minimum sampling time).

NOTE The best estimate of long-term exposure is obtained by taking samples for the entire working period, but this is often not practicable (e.g. because of the possibility of overloading the filter).

8.4.5.2 Periodic measurements

Periodic measurements are used to determine whether exposure conditions have changed since the measurements for comparison with limit values were performed, or whether control measures remain effective.

9 Sampling

9.1 Preliminary considerations

9.1.1 Selection and use of samplers

9.1.1.1 Select samplers (7.1.1) designed to collect the inhalable fraction of airborne particles, as defined in ISO 7708:1995, manufactured from a material that does not react with LiOH, NaOH, KOH, and $Ca(OH)_2$.

If possible, the samplers selected should be manufactured from conducting material, since samplers manufactured from non-conducting material have electrostatic properties that can influence representative sampling.

9.1.1.2 Use the samplers at their design flow rate and in accordance with the instructions provided by the manufacturer.

9.1.2 Sampling period

9.1.2.1 Select a sampling period that is appropriate for the measurement task (see 8.4), but ensure that it is long enough to enable LiOH, NaOH, KOH, and $Ca(OH)_2$ to be determined with acceptable uncertainty (see 3.5.8) at levels of industrial hygiene significance. For example, estimate the minimum sampling time required to ensure that the amount collected is above the lower limit of the working range of the analytical method when LiOH, NaOH, KOH, and/or $Ca(OH)_2$ are present in the test atmosphere at an appropriate multiple of their limit values, using Formula (1).

$$t_{\min} = \frac{m_{\text{lower}}}{q_v \times F \times \rho_{LV}} \quad (1)$$

where

$t_{\min}$ is the minimum sampling time, in minutes;

m_{lower} is the lower limit of the analytical range, in micrograms;

q_v is the design flow rate of the sampler, in litres per minute;

F is an appropriate multiple of the limit value (e.g. 0,1 times for an 8 h time-weighted average limit value or 0,5 times for a short-term limit value);

ρ_{LV} is the limit value, in milligrams per cubic metre.

NOTE If the minimum sampling time is not short enough for the method to be useful for the intended measurement task, consider the possibility of using a sampler designed to be used at a higher flow rate.

9.1.2.2 When high concentrations of airborne particles are anticipated, select a sampling period that is not so long as to risk overloading the filter with particulate matter.

9.1.3 Temperature and pressure effects

9.1.3.1 Effect of temperature and pressure on flow rate measurements

Refer to the manufacturer's instructions to determine if the indicated volumetric flow rate of the flow meter (7.1.4) is dependent upon temperature and pressure. Consider whether the difference between the atmospheric temperature and pressure at the time of calibration of the flow meter and during sampling is likely to be great enough to justify making a correction to take this into account, e.g. if the error could be greater than ±5 %. If a correction is necessary, measure and record the atmospheric temperature and pressure at which the calibration of the flow meter was checked (7.1.4) and measure and record the atmospheric temperature and pressure at the start and at the end of the sampling period (see 9.4.1 and 9.4.2).

NOTE An example of temperature and pressure correction for the indicated volumetric flow rate is given in A.1 for a constant pressure drop, variable area flow meter.

9.1.3.2 Expression of results

Consider whether it is necessary to recalculate the concentration of LiOH, NaOH, KOH, and $Ca(OH)_2$ in air to reference conditions (see ISO 8756[2]). If so, measure and record the atmospheric temperature and pressure at the start and at the end of the sampling period (see 9.4.1 and 9.4.2) and use the formula given in A.1.2 to apply the necessary correction.

NOTE The concentration of LiOH, NaOH, KOH, and $Ca(OH)_2$ in air is generally stated for actual environmental conditions (temperature and pressure) at the workplace.

9.1.4 Sample handling

To minimize the risk of damage or contamination, only handle filters (7.1.2) in a clean area where the concentration of Li, Na, K, and Ca compounds in air is as low as possible and only handle filters using tweezers (7.1.5.3).

9.2 Preparation for sampling

9.2.1 Cleaning of samplers

Clean the samplers (7.1.1) before use, unless disposable sampling cassettes are used. Disassemble the samplers, rinse thoroughly with water, wipe with absorbent tissue, and allow to dry before reassembly. Alternatively, use a laboratory washing machine.

9.2.2 Loading the samplers with filters

Load each clean sampler (see 9.2.1) with suitable filters (7.1.2), label each sampler so that it can be uniquely identified, and seal with its protective cover or plug to prevent contamination.

9.2.3 Setting the volumetric flow rate

Perform the following in a clean area, where the concentration of Li, Na, K, and Ca compounds is minimal.

Connect each loaded sampler (see 9.2.2) to a sampling pump (7.1.3) using flexible tubing (7.1.5.1), ensuring that no leaks can occur. Remove the protective cover or plug from each sampler, switch on the sampling pump, attach the flow meter (7.1.4) to the sampler so that it measures the flow through the sampler inlet orifice(s), and set the required volumetric flow rate (see 9.1.1.2). Switch off the sampling pump and seal the sampler with its protective cover or plug to prevent contamination during transport to the sampling position.

If necessary, allow the sampling pump operating conditions to stabilize before setting the volumetric flow rate.

9.2.4 Field blanks

Retain as blanks one unused loaded sampler from each batch of 10 prepared, subject to a minimum of three. Treat these in the same manner as those used for sampling in respect of storage and transport to and from the sampling position, but draw no air through the filters.

9.3 Sampling position

9.3.1 Personal sampling

9.3.1.1 Position the sampler in the worker's breathing zone, as close to the mouth and nose as is reasonably practicable, e.g. fastened to the worker's lapel. Attach the sampling pump to the worker in a manner that causes minimum inconvenience, e.g. to a belt (7.1.5.2) around the waist, or place it in a convenient pocket.

9.3.1.2 Give consideration to whether the nature of the process is likely to result in a significant difference between the actual exposure of the worker and the concentration of LiOH, NaOH, KOH, and $Ca(OH)_2$ measured by a sampler mounted on the lapel. If this is the case, make special arrangements to mount the sampler as close as possible to the worker's nose and mouth.

9.3.2 Static sampling

9.3.2.1 If static sampling is carried out to assess the exposure of a worker in a situation where personal sampling is not possible, position the sampler in the immediate vicinity of the worker and at breathing height. If in doubt, take the sampling position to be the point where the risk of exposure is considered to be greatest.

9.3.2.2 If static sampling is carried out to characterize the background level of Li, Na, K, and Ca in the workplace, select a sampling position that is sufficiently remote from the work processes, such that results will not be directly affected by Li, Na, K, and Ca from emission sources.

9.4 Collection of samples

9.4.1 When ready to begin the sampling, remove the protective cover or plug from the sampler and switch on the sampling pump. Record the time and volumetric flow rate at the start of the sampling period. If the sampling pump is fitted with an integral timer, check that this is reset to zero. If appropriate (see 9.1.3), measure the atmospheric temperature and pressure at the start of the sampling period using the thermometer (7.1.5.4) and barometer (7.1.5.5), and record the measured values.

NOTE If the temperature or pressure at the sampling position is different from that where the volumetric flow rate was set (see 9.2.3), the volumetric flow rate could change and it might need to be re-adjusted before the sampling.

9.4.2 At the end of the sampling period (see 9.1.2), record the time and calculate the duration of the sampling period. Check the malfunction indicator and/or the reading on the integral timer, if fitted, and consider the sample to be invalid if there is evidence that the sampling pump was not operating properly throughout the sampling period. Measure the volumetric flow rate at the end of the sampling period using the flow meter (7.1.4), and record the measured value. If appropriate (see 9.1.3), measure the atmospheric temperature and pressure at the end of the sampling period using the thermometer (7.1.5.4) and barometer (7.1.5.5), and record the measured values.

9.4.3 Carefully record the sample identity and all relevant sampling data (see Reference [13]). Calculate the mean volumetric flow rate by averaging the volumetric flow rates at the start and at the end of the sampling period and, if appropriate (see 9.1.3), calculate the mean atmospheric temperature and pressure. Calculate the volume of air sampled, in litres, at atmospheric temperature and pressure, by multiplying the mean flow rate in litres per minute by the duration of the sampling period in minutes.

9.5 Transportation

9.5.1 Samplers with an internal filter cassette

For samplers with an internal filter cassette, remove the filter cassette from each sampler and fasten with its lid or transport clip.

9.5.2 Samplers of the disposable cassette type

For samplers of the disposable cassette type, transport the samples to the laboratory in the samplers in which they were collected.

9.5.3 Transport of samples to the laboratory

9.5.3.1 Transport the samples (9.5.1 and 9.5.2) to the laboratory in a container which has been designed to prevent damage to the samples in transit and which has been labelled to ensure proper handling.

9.5.3.2 Ensure that the documentation which accompanies the samples is suitable for a 'chain of custody' to be established (see, for example, ASTM D 4840[8]).

10 Analysis

CAUTION Use suitable personal protective equipment (including suitable gloves, face shield or safety glasses, etc.) while carrying out the analysis.

10.1 Preparation of test and calibration solutions

10.1.1 Selection of sample preparation method

Decide whether to use water (6.1) or eluent (6.3) to prepare test solutions for determination of LiOH, NaOH, KOH, and $Ca(OH)_2$, depending on the analytical technique and separator column used. See e.g. Reference [20] for further guidance.

10.1.2 Action to be taken regarding sampler wall deposits

Prior to opening filter transport cassettes or samplers, consider the possibility that particles may have deposited on the interior walls of the cassette or sampler during the sampling event, and actions that may be required to include such particles in the sample. Additional information is provided in Annex C.

10.1.3 Preparation of test solutions

10.1.3.1 Open each filter cassette or sampler (see 9.5) and transfer the filter into individual, labelled screw cap vessels (7.2.3.2) or beakers (7.2.3.3 or 7.2.2) using clean tweezers (7.1.5.3), ensuring that the side of the filter on which the sample was collected is facing upwards. Follow the same procedure for the blank filters (see 9.2.4).

NOTE It is possible to carry out the extraction in samplers of the disposable cassette type if they are of sufficient capacity and are watertight when the sample outlet orifice is sealed with its protective plug. In this case, the extraction solution (see 10.1.3.2) should be added to the sampler via the air inlet orifice and the samplers should be maintained in an upright position while in the ultrasonic bath (see 10.1.3.3) to avoid spillage and contamination of the sample solutions.

10.1.3.2 Accurately pipette 10,0 ml of water (6.1) or eluent (6.3) into each screw cap vessel or beaker. If the sampler used was of a type in which airborne particles deposited on the internal surfaces of the sampler form part of the sample (see C.2), use the water or eluent to carefully wash any particulate material adhering to the internal surfaces of the sampler into the beaker. In the case of PTFE filters, add 0,1 ml of ethanol because of the hydrophobic nature of these filters.

10.1.3.3 Swirl gently to mix the contents, ensuring that the filter remains completely immersed. Sonicate for 15 min in an ultrasonic bath (7.2.5) and then allow the immersed filters to sit for 1 h at room temperature, swirling or agitating occasionally.

10.1.3.4 Filter each sample solution through a PTFE filter (7.2.3.6), e.g. by using a disposable syringe (7.2.3.7), dispensing each filtrate into an individual, labelled, autosampler vial (7.2.3.8).

10.1.4 Preparation of calibration solutions

Prepare a minimum of five calibration solutions to cover the required concentration range, e.g. from 0,3 mg l^{-1} to 3 mg l^{-1} for Li and 0,6 mg l^{-1} to 6 mg l^{-1} for Na, K, and Ca. Accurately pipette appropriate volumes of cation working standard solution (6.5.5) into individual, labelled one-mark volumetric flasks (7.2.3.1 or 7.2.2) or graduated centrifuge tubes (7.2.3.4), dilute to the mark with water (6.1), close, and mix thoroughly. Prepare these calibration solutions fresh daily.

10.2 Instrumental analysis

10.2.1 Setting up the instrument

10.2.1.1 Set up the ion chromatograph in accordance with manufacturer's instructions.

10.2.1.2 Install a sample loop that gives a suitable injection volume.

10.2.1.3 Adjust the detector to measure to a suitable measuring range.

10.2.1.4 Adjust the flow rate of the eluent (6.4) to a value that is compatible with the columns used.

10.2.1.5 Adjust the flow rate of the regeneration solution to a suitable value.

10.2.2 Analysis

10.2.2.1 Inject the calibration solutions (see 10.1.4) into the ion chromatography system in the order of increasing concentration and measure hydroxide peaks for each calibration solution in peak area mode.

10.2.2.2 Use the instrument's computer to generate a calibration function using a linear regression. Repeat the calibration if the coefficient of determination, R^2, is not > 0,999.

NOTE 1 If $R^2 < 0{,}999$, it might be possible to remove an erroneous calibration point and reprocess the data to obtain an acceptable calibration.

10.2.2.3 Inject the blank and sample test solutions (see 10.1.3) into the ion chromatography system and make measurements for each solution. Use the stored calibration function (see 10.2.2.2) to determine the hydroxide concentrations in mg l^{-1}.

10.2.2.4 Analyse the calibration blank and a mid-range calibration solution after the initial calibration and then after every 10 test solutions. If the measured concentration of Li, Na, K, and Ca in the continuing calibration blank is above the method detection limit, as determined in 10.3.2, or if the measured concentration of Li, Na, K, and Ca in the continuing calibration verification has changed by more than ±5 %, take one of the following corrective measures. Either use the instrument software to correct for the sensitivity change (reslope facility), or suspend analysis and recalibrate the instrument. In either case, reanalyse the test solutions that were analysed during the period in which the sensitivity change occurred, or if this is not possible, reprocess the data to take account of the sensitivity change.

10.2.2.5 Analyse reagent blank solutions and laboratory blank solutions, as specified in 10.4.1, and quality control solutions, as prescribed in 10.4.2.1, and use the results to monitor the performance of the method, as prescribed in 10.4.2.2.

10.2.2.6 If concentrations of Li, Na, K, and Ca are found to be above the upper limit of linear calibration range, dilute the test solutions in order to bring them within the linear range and repeat the analysis. Add an appropriate volume of extraction solution when making dilutions, so that the diluted test solutions and the calibration solutions are matrix-matched, and record the dilution factor (DF).

NOTE For samples expected to have very high concentrations of LiOH, NaOH, KOH, and $Ca(OH)_2$, it might be necessary to dilute the test solutions before they are first analysed.

10.3 Estimation of detection and quantification limits

10.3.1 Estimation of the instrumental detection limit

10.3.1.1 Estimate the instrumental detection limit under the working analytical conditions following the procedure described in 10.3.1.2 and 10.3.1.3, and repeat this exercise whenever the experimental conditions are changed significantly.

NOTE The instrumental detection limit is of use in identifying changes in instrument performance, but it is not a method detection limit (see Reference [21]). The instrumental detection limit is likely to be lower than the method detection limit because it only takes into account the variability between individual instrumental readings; determinations made on one solution do not take into consideration contributions to variability from the matrix or sample.

10.3.1.2 Prepare a test solution with hydroxide concentrations near the anticipated instrumental detection limits by diluting the cation working standard solution (6.5.5) by an appropriate factor.

10.3.1.3 Make at least 10 ion chromatographic measurements on the test solution and calculate the instrumental detection limit as three times the sample standard deviation of the mean concentration value.

10.3.2 Estimation of the method detection limits and quantification limits

10.3.2.1 Estimate the method detection limits and quantification limits under the working analytical conditions following the procedure described in 10.3.2.2 and 10.3.2.3 (which is based upon the approach described in Reference [22]), and repeat this exercise whenever the experimental conditions are changed significantly.

10.3.2.2 Fortify at least 10 filters (7.1.2) with Li, Na, K, and Ca near the anticipated detection limits, e.g. 1 µg of each cation, by spiking each filter with 0,01 ml of a solution prepared by diluting the cation working standard solution (6.5.5) by an appropriate factor. Prepare test solutions following the sample dissolution procedure used to prepare the sample test solutions (see 10.1.3).

10.3.2.3 Make ion chromatographic measurements on the test solutions derived from each spiked filter (see 10.3.2.2) and calculate the method detection limit and the quantification limit as 3 times and 10 times the sample standard deviation of the mean concentration value, respectively.

NOTE An alternative procedure for estimating the method detection limit involves the analysis of filter samples fortified with the analyte of interest at values spanning the predicted detection limit (see Reference [21]).

10.4 Quality control

10.4.1 Laboratory blanks

Carry reagent blanks (water and reagents) and media blanks (unspiked filters) throughout the entire sample preparation and analytical process to determine whether the samples are being contaminated from laboratory activities. Process reagent blanks according to a frequency of at least 1 per 20 samples or a minimum of one per batch.

10.4.2 Spiked samples and spiked duplicate samples

10.4.2.1 Carry spiked samples and spiked duplicate samples throughout the entire sample preparation and analytical process to estimate the method accuracy on the sample batch, expressed as a percent recovery relative to the true spiked value. Spiked samples and spiked duplicate samples consist of filters to which known amounts of Li, Na, K, and Ca have been added. (This can be accomplished by spiking with known volumes of working cation standard solution at amounts within the linear dynamic range of the instrument. The working cation standard solution used shall be prepared from stock standard solutions from a different source than that used for preparing the calibration solutions.) Process these quality control samples according to a frequency of at least 1 per 20 samples or a minimum of one per batch.

10.4.2.2 Monitor the performance of the method by plotting control charts of the relative percent recoveries and of the relative percent differences between the spiked samples and the spiked duplicate samples. If quality control results indicate that the method is out of control, investigate the reasons for this, take corrective action, and reanalyse the samples if necessary. See ASTM E 882[6] for general guidance on the use of quality control charts.

10.4.3 Certified reference materials (CRMs)

If available, suitable certified reference materials (CRMs) for Li, Na, K, and Ca shall be analysed prior to routine use of the method to establish that the percent recovery relative to the certified value is

satisfactory. CRMs are available from European Reference Materials (ERM), the Institute for Reference Materials and Measurements (IRMM), and the National Institute for Standards and Technology (NIST).

10.4.4 External quality assessment

If the laboratory carries out the analysis of hydroxides in workplace air samples on a regular basis, it is recommended that the laboratory participates in a relevant external quality assessment scheme or proficiency testing scheme, if such a scheme exists and if the laboratory has access to it.

NOTE Information about existing proficiency testing schemes, refer, for example, to the database EPTIS[23]or to a national accreditation organization.

10.5 Measurement uncertainty

It is strongly recommended that the laboratory estimates and reports the uncertainty of its measurements in accordance with ISO/IEC Guide 98-3:2008.[4] This entails first constructing a cause and effect diagram[3] to identify the individual sources of random and systematic error in the overall sampling and analytical method. The standard uncertainties associated with these errors are then estimated, determined experimentally, or both, and combined in what is referred to as an uncertainty budget. The combined standard uncertainty is ultimately multiplied by an appropriate coverage factor to produce an expanded uncertainty. A coverage factor of 2 is ordinarily recommended, giving a level of confidence of approximately 95 % in the calculated value.

NOTE 1 The application of cause and effect analysis to analytical methods is described in ISO/IEC Guide 98-3:2008[4] and in References [24] and [25].

NOTE 2 EN 13890[12] provides guidance on including the uncertainty associated with sampling in the uncertainty budget.

NOTE 3 Terms that contribute to the random variability of an analytical method are generally accounted for in the measurement precision, which can be estimated from quality control data. Error associated with instrumental drift can be estimated, assuming a rectangular probability distribution, by dividing the allowable drift before recalibration by $\sqrt{3}$ (see 10.2.2.4). Systematic errors of an analytical method include, for example, those associated with analytical recovery, preparation of working standard solutions, dilution of test solutions, etc.

11 Expression of results

Calculate the mass concentration of LiOH, NaOH, KOH, and $Ca(OH)_2$ in the air samples at ambient conditions using Formula (2).

$$\rho_{\text{hydroxide}} = \frac{\left(\rho_{\text{cation},1} \times V_1 \times f_{\text{dilution}}\right) - \left(\rho_{\text{cation},0} \times V_0\right)}{V} \times f_{\text{conversion}} \quad (2)$$

where

$\rho_{\text{hydroxide}}$ is the mass concentration of LiOH, NaOH, KOH, and $Ca(OH)_2$ in the air sample, in milligrams per cubic metre;

$\rho_{\text{cation},0}$ is the mean concentration of Li, Na, K, and Ca in the blank test solutions, in milligrams per litre;

$\rho_{\text{cation},1}$ is the concentration of Li, Na, K, and Ca in the sample test solution, in milligrams per litre;

V is the volume, in litres, of the air sample;

V_0 is the volume, in millilitres, of the blank test solution;

V_1 is the volume, in millilitres, of the sample test solution;

f_{dilution} is the dilution factor ($f_{\text{dilution}} = 1$ in the absence of dilution);

$f_{\text{conversion}}$ is the factor to convert from cation to hydroxide concentration [3,45 for LiOH, 1,73 for NaOH, 1,43 for KOH, and 1,85 for $Ca(OH)_2$].

12 Method performance

12.1 Sampling efficiency and sample storage

Laboratory testing with filters spiked with LiOH, NaOH, KOH, and $Ca(OH)_2$ yielded a recovery of >95 % after four weeks sample storage. See Reference [19] for further information.

12.2 Quantification limit

The quantification limit of the method has been determined[19] to be 0,5 mg l^{-1} for Li and 1 mg l^{-1} for Na, K, and Ca. For a sample solution volume of 10 ml and an air sample volume of 420 l, this is equivalent to 0,041 mg m^{-3} for LiOH and NaOH, 0,034 mg m^{-3} for KOH, and 0,044 mg m^{-3} for $Ca(OH)_2$.

12.3 Upper limits of the working range

The upper limit of the analytical range is governed by the maximum permissible loading of the sample filter. It has been demonstrated[19] that no breakthrough occurs for quartz fibre filters at sample loadings of up to 1 mg.

12.4 Bias and precision

12.4.1 Analytical bias

Laboratory experiments have shown that the analytical method does not exhibit significant bias. The mean analytical recovery, determined from the analysis of spiked filters, has been found[19] to be in the range 100 % ± 2 % for LiOH, NaOH, KOH, and $Ca(OH)_2$.

12.4.2 Analytical precision

The component of the coefficient of variation of the method that arises from analytical variability, CV (analysis), determined from spiked samples, has been found[19] to be in the range 0,7 % to 1,3 % for LiOH, 0,9 % to 3,0 % for NaOH, 0,8 % to 1,9 % for KOH, and 1,4 % to 3,4 % for $Ca(OH)_2$.

12.5 Uncertainty of sampling and analysis method

The expanded uncertainty of the method, using a coverage factor of 2, has been estimated[19] to be <22 % for particulate hydroxides.

12.6 Interferences

Na, K, and Ca are ubiquitous in the environment and, if present in the sampled air in high amounts, other soluble Na, K, and Ca salts can significantly affect measurements of NaOH, KOH, and $Ca(OH)_2$ made using the methodology described in this International Standard. However, there are also selectivity and sensitivity issues when measuring hydroxides by titrimetry. For example, mixed exposure to more than one hydroxide can occur and the presence of carbonates and reaction of sampled hydroxides with CO_2 can be confounding factors. The methodology described in this International Standard has been selected because it gives worst case results, i.e. false-positive results are possible but false-negative results are not.

13 Test report

13.1 Test record

A comprehensive record of the test performed shall be maintained, including the following information:

a) a statement to indicate the confidentiality of the information supplied, if appropriate;

b) a complete identification of the air sample, including the date of sampling, the place of sampling, the type of sample (personal or static), either the identity of the individual whose breathing zone was sampled (or other personal identifier) or the location at which the general occupational environment was sampled (for a static sample), a very brief description of the work activities that were carried out during the sampling period, and a unique sample identification code;

c) a reference to this International Standard;

d) the make, type, and diameter of filters used;

e) the make and type of sampler used;

f) the make and type of sampling pump used, and its identification;

g) the make and type of flow meter used, the primary standard against which the calibration of the flow meter was checked, the range of flow rates over which the calibration of the flow meter was checked, and the atmospheric temperature and pressure at which the calibration of the flow meter was checked, if appropriate (see 9.1.3);