



BSI Standards Publication

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

National foreword

This British Standard is the UK implementation of ISO 17091:2013.

The UK participation in its preparation was entrusted to Technical Committee EH/2/2, Work place atmospheres.

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

This publication does not purport to include all the necessary provisions of a contract. Users are responsible for its correct application.

© The British Standards Institution 2013. Published by BSI Standards Limited 2013

ISBN 978 0 580 77732 5

ICS 13.040.30

Compliance with a British Standard cannot confer immunity from legal obligations.

This British Standard was published under the authority of the Standards Policy and Strategy Committee on 30 September 2013.

Amendments issued since publication

Date	Text affected
------	---------------

**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





COPYRIGHT PROTECTED DOCUMENT

© ISO 2013

All rights reserved. Unless otherwise specified, no part of this publication may be reproduced or utilized otherwise in any form or by any means, electronic or mechanical, including photocopying, or posting on the internet or an intranet, without prior written permission. Permission can be requested from either ISO at the address below or ISO's member body in the country of the requester.

ISO copyright office
Case postale 56 • CH-1211 Geneva 20
Tel. + 41 22 749 01 11
Fax + 41 22 749 09 47
E-mail copyright@iso.org
Web www.iso.org

Published in Switzerland

Contents

Page

Foreword	v
Introduction	vi
1 Scope	1
2 Normative references	1
3 Terms and definitions	2
3.1 General definitions.....	2
3.2 Particle size fraction definitions.....	3
3.3 Sampling definitions.....	3
3.4 Analytical definitions.....	4
3.5 Statistical terms.....	6
4 Principle	7
5 Requirement	7
6 Reagents	8
7 Apparatus	9
7.1 Sampling equipment.....	9
7.2 Laboratory apparatus.....	10
8 Occupational exposure assessment	11
8.1 General.....	11
8.2 Personal sampling.....	11
8.3 Static sampling.....	12
8.4 Selection of measurement conditions and measurement pattern.....	12
9 Sampling	13
9.1 Preliminary considerations.....	13
9.2 Preparation for sampling.....	15
9.3 Sampling position.....	15
9.4 Collection of samples.....	16
9.5 Transportation.....	16
10 Analysis	16
10.1 Preparation of test and calibration solutions.....	17
10.2 Instrumental analysis.....	17
10.3 Estimation of detection and quantification limits.....	18
10.4 Quality control.....	19
10.5 Measurement uncertainty.....	20
11 Expression of results	21
12 Method performance	21
12.1 Sampling efficiency and sample storage.....	21
12.2 Quantification limit.....	21
12.3 Upper limits of the working range.....	21
12.4 Bias and precision.....	22
12.5 Uncertainty of sampling and analysis method.....	22
12.6 Interferences.....	22
13 Test report	22
13.1 Test record.....	22
13.2 Laboratory report.....	23
Annex A (informative) Temperature and pressure correction	24
Annex B (normative) Filter materials	25
Annex C (informative) Sampler wall deposits	26

Bibliography27

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).

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

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 m³ 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 M Ω ·m (usually expressed by manufacturers of water purification systems as 18 M Ω ·cm).

6.2 Sulfuric acid (H₂SO₄) 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 (H₂SO₄) 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, polyvinyl chloride (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 $^{\circ}\text{C}$ to 50 $^{\circ}\text{C}$, graduated in divisions of 1 $^{\circ}\text{C}$ 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).

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

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 $\pm 5\%$. 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 $\text{Ca}(\text{OH})_2$ 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 $\text{Ca}(\text{OH})_2$ 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 $\pm 5\%$, 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).

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

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 CO₂ 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](#));

- h) the time at the start and at the end of the sampling period, and the duration of the sampling period, in minutes;
- i) the mean flow rate during the sampling period, in litres per minute;
- j) the mean atmospheric temperature and pressure during the sampling period, if appropriate (see [9.1.3](#));
- k) the volume of air sampled, in litres, at ambient conditions;
- l) the name of the person who collected the sample;
- m) the time-weighted average mass concentration of LiOH, NaOH, KOH, and Ca(OH)₂ found in the air sample (in milligrams per cubic metre), at ambient temperature and pressure, or, if appropriate, adjusted to reference conditions;
- n) the analytical variables used to calculate the result, including the concentrations of Li, Na, K, and Ca in the sample and blank solutions, the volumes of the sample and blank solutions, and the dilution factor, if applicable;

NOTE If necessary data (e.g. the volume of air sampled) are not available to the laboratory for the above calculations to be carried out, the laboratory report may contain the analytical result in micrograms of LiOH, NaOH, KOH, and Ca(OH)₂ per filter sample.

- o) the type(s) of instrument(s) used for sample preparation and analysis, and unique identifier(s);
- p) the estimated instrumental detection limits, method detection limits, and quantification limits under the working analytical conditions; the measurement uncertainty determined in accordance with ISO/IEC Guide 98-3:2008[4]; and, if requested by the customer, quality control data;
- q) any operation not specified in this International Standard, or regarded as optional;
- r) the name of the analyst(s) [or other unique identifier(s)];
- s) the date of the analysis;
- t) any inadvertent deviations, unusual occurrences, or other notable observations.

13.2 Laboratory report

The laboratory report shall contain all information required by the end user, regulatory authorities, and accreditation organizations.

Annex A (informative)

Temperature and pressure correction

A.1 Temperature and pressure correction for the indicated volumetric flow rate

A.1.1 Bubble flow meters are preferred for measuring the volumetric flow rate because the readings they give are independent of temperature and pressure. For other flow meters, it might be necessary to apply a correction to the indicated volumetric flow rate if the temperature and pressure at the time of measurement is different to when the calibration of the flow meter was checked.

A.1.2 A typical example of the need for a temperature and pressure correction is when a constant pressure drop, variable area flow meter is used to measure the volumetric flow rate. In this instance, use Formula (A.1) to calculate a corrected air sample volume.

$$V_{\text{corr}} = q_v \times t \times \sqrt{\frac{p_1 \times T_2}{p_2 \times T_1}} \quad (\text{A.1})$$

where

V_{corr} is the corrected volume, in litres;

q_v is the mean flow rate, in litres per minute;

t is the sampling time, in minutes;

p_1 is the atmospheric pressure, in kilopascals, during calibration of the sampling pump flow meter;

p_2 is the mean atmospheric pressure, in kilopascals, during the sampling period;

T_1 is the temperature, in kelvin, during calibration of the sampling pump flow meter;

T_2 is the mean temperature, in kelvin, during the sampling period.

A theoretical calculation shows that a 5 % deviation in the air sample volume at the reference atmospheric pressure of 101,3 kPa occurs at 91,9 kPa and 112,2 kPa. Both these values are outside the normal weather conditions at sea level, but this pressure difference corresponds to an altitude change of about 800 m (–0,1 kPa approximately 8 m increased altitude) at normal atmospheric pressure at sea level. Similarly, a 5 % deviation in the air sample volume at the reference temperature of 293 K occurs at 264 K and 323 K.

A.1.3 Any other flow meter can also require a correction for variation in pressure and temperature. Follow the manufacturer's instructions for such corrections.

Annex B (normative)

Filter materials

B.1 Filters

The purpose of the filters is to collect particulate hydroxide.

LiOH, NaOH, KOH, and Ca(OH)₂ are powerful hydroxides that react with many filter materials. Therefore, correct selection of the filter used for collection of particulate hydroxides is of paramount importance. It has to be manufactured from a material that does not react with the hydroxides.

Quartz fibre filters are generally suitable for use as filters. However, the level and variability of blanks can vary significantly depending on the filter supplier and the individual filter batch (see [12.6](#)). It is therefore advisable to verify that each batch of filters is fit for use by determining the quantification limit following the procedure prescribed in [10.3.2](#).

NOTE Glass fibre filters that are by principle suitable for sampling hydroxides lead to very high blank values of Na and Ca.

Annex C (informative)

Sampler wall deposits

C.1 General

This annex provides information on methods for accounting for deposits of particulate matter on the interior walls of a transport cassette or sampler (see [10.1.2](#)).

C.2 Samplers

Samplers for aerosols typically consist of a filter supported in a holder. The entire device is considered to be an aerosol sampler. The sampling efficiency of an aerosol sampler is considered to be the air concentration calculated from the particulates collected by the sampler compared to the undisturbed concentration in air. All aerosol samplers exhibit a decrease in sampling efficiency with increasing particulate aerodynamic diameter. Size-selective samplers are designed for a specific sampling efficiency over a range of aerodynamic diameters, known as a sampling convention (see ISO 7708:1995), in which case the actual sampling efficiency of the sampler is considered with reference to the relevant sampling convention.

C.3 Collection efficiency

The collection efficiency of an aerosol sampler has four components: (i) aspiration efficiency, (ii) transfer efficiency within the sampler (either from sampler inlet to the collection substrate or, if an internal separator is present, both from the sampler inlet to the internal separator and from the internal separator to the collection substrate), (iii) penetration efficiency (through the internal separator, if present), and (iv) the capture efficiency of the collection substrate (e.g. filtration efficiency, when the collection substrate is a filter). For any given design of sampler, the various components are functions of particle aerodynamic size and air flow rate through the sampler. The aspiration efficiency also depends on wind speed and direction, while the sampler's angle to the vertical influences both aspiration and transport efficiency. Part of the sample will deposit on internal surfaces of the sampler as a result of losses during passage within the sampler. In addition, if the sampler is transported after sampling, particles deposited on the substrate can become dislodged and add to deposits already on the internal surfaces of the sampler (although this is likely of lesser importance, except when the collection substrate is overloaded with sample). If the design specification for the sampler is to include all aspirated particles, these losses need to be taken into account.

For some samplers, the sample deposited on the collection substrate is considered to be the entire sample, i.e. wall deposits are not considered to be part of the sample. For other samplers, it is recommended that the wall deposits are evaluated.

C.4 Procedures

There exist several procedures that could be used to account for wall deposits. One method is extraction or sample dissolution within the body of the sampler. Another procedure, often followed, is to rinse the internal deposit into the extraction or sample dissolution vessel containing the collection substrate. This can be quantitative if the deposit is very soluble or easily displaced, which is the case for LiOH, NaOH, KOH, and Ca(OH)₂.

Bibliography

- [1] ISO 3534-2:2006, *Statistics — Vocabulary and symbols — Part 2: Applied statistics*
- [2] ISO 3585, *Borosilicate glass 3.3 — Properties*
- [3] ISO 8756, *Air quality — Handling of temperature, pressure and humidity data*
- [4] ISO 9004-4:1993, *Quality management and quality system elements — Part 4: Guidelines for quality improvement*
- [5] ISO/IEC Guide 98-3:2008, *Uncertainty of measurement — Part 3: Guide to the expression of uncertainty in measurement (GUM:1995)*
- [6] ISO/IEC Guide 99:2007, *International vocabulary of metrology — Basic and general concepts and associated terms (VIM)*
- [7] ASTM E 882, *Standard guide for accountability and quality control in the chemical analysis laboratory*
- [8] ASTM E 1370, *Standard guide for air sampling strategies for worker and workplace protection*
- [9] ASTM D 4840, *Standard practice for chain of custody procedures*
- [10] EN 482, *Workplace atmospheres — General requirements for the performance of procedures for the measurement of chemical agents*
- [11] EN 689, *Workplace atmospheres — Guidance for the assessment of exposure to chemical agents for comparison with limit values and measurement strategy*
- [12] EN 1540:2011, *Workplace exposure — Terminology*
- [13] EN 13890, *Workplace exposure — Procedures for measuring metals and metalloids in airborne particles — Requirements and test methods*
- [14] EN 14902:2005, *Ambient air quality — Standard method for the measurement of Pb, Cd, As and Ni in the PM₁₀ fraction of suspended particulate matter*
- [15] EUROPEAN COMMISSION. Council Directive 98/24/EC of 7 April 1998 on the protection of the health and safety of workers from the risks related to chemical agents at work (fourteenth individual Directive within the meaning of Article 16(1) of Directive 89/391/EEC). Available (2011-11-06) at <http://eur-lex.europa.eu/LexUriServ/LexUriServ.do?uri=OJ:L:1998:131:0011:0023:EN:PDF>
- [16] American Conference of Government Industrial Hygienists (ACGIH), *Threshold Limit Values for Chemical Substances and Physical Agents; Biological Exposure Indices*, ACGIH, Cincinnati, OH, USA (updated annually) <http://www.acgih.org/>
- [17] EUROPEAN COMMISSION. Commission Directive 2006/15/EC of 7 February 2006 establishing a second list of indicative occupational exposure limit values in implementation of Council Directive 98/24/EC and amending Directives 91/322/EEC and 2000/39/EC. Available (2011-11-06) at <http://eur-lex.europa.eu/LexUriServ/LexUriServ.do?uri=OJ:L:2006:038:0036:0039:EN:PDF>
- [18] International Labour Organization (ILO). Programme on Safety and Health at Work and the Environment (SafeWork), Chemical Exposure Limits. Available (2011-11-06) at http://www.ilo.org/safework/areasofwork/lang--en/WCMS_118291/index.htm
- [19] INSTITUT FÜR ARBEITSSCHUTZ DER DEUTSCHEN GESETZLICHEN UNFALLVERSICHERUNG (IFA). Database : GESTIS — International limit values for chemical agents. Available (2011-11-06) at http://www.dguv.de/ifa/en/gestis/limit_values/index.jsp

- [20] BREUER D. Hydroxide (LiOH, NaOH, KOH, Ca(OH)₂), In: BGIA Working Folder, Sheet No. 7683. Erich-Schmidt Verlag GmbH & Co, Berlin, Germany, 2009. ISBN 3 503 020853. Available (2011-11-06) at <http://www.myesv.info/login.html>
- [21] Dionex, Dionex reference library, manuals, technical documents, applications, product literature, CD-ROM, Dionex corporation (updated regularly). Available (2011-11-06) at www.dionex.com
- [22] KENNEDY E.R., FISCHBACH T.J., SONG R., ELLER P.M., SHULMAN S. *Guidelines for air sampling and analytical method development and evaluation*. U.S. Department of Health and Human Sciences, Public Health Service, Centers for Disease Control and Prevention, National Institute for Occupational Safety and Health, Cincinnati, OH, 1995. DHHS (NIOSH) Publication pp, 95-117. Available (2011-11-06) at <http://www.cdc.gov/niosh/docs/95-117/>
- [23] CURRIEL A. Nomenclature in evaluation of analytical methods including detection and quantification capabilities (IUPAC Recommendations 1995). *Pure Appl. Chem.* 1995, **67** pp. 1699-1723
- [24] BAM. Database EPTIS (European Information System on Proficiency Testing Schemes). Federal Ministry of Economics and Technology (BMWi), Federal Institute for Materials Research and Testing (BAM), Berlin, Germany. Available (2011-11-06) at www.eptis.bam.de
- [25] ELLISON S.L.R., & BARWICK V.J. Estimating measurement uncertainty: Reconciliation using a cause and effect approach. *Accredit. Qual. Assur.* 1998, **3** pp. 101-513
- [26] ELLISON S.L.R., & BARWICK V.J. Using validation data for ISO measurement uncertainty investigation: Part 1. Principles of an approach using cause and effect analysis. *Analyst (Lond.)*. 1998, **123** pp. 1387-1392

British Standards Institution (BSI)

BSI is the national body responsible for preparing British Standards and other standards-related publications, information and services.

BSI is incorporated by Royal Charter. British Standards and other standardization products are published by BSI Standards Limited.

About us

We bring together business, industry, government, consumers, innovators and others to shape their combined experience and expertise into standards-based solutions.

The knowledge embodied in our standards has been carefully assembled in a dependable format and refined through our open consultation process. Organizations of all sizes and across all sectors choose standards to help them achieve their goals.

Information on standards

We can provide you with the knowledge that your organization needs to succeed. Find out more about British Standards by visiting our website at bsigroup.com/standards or contacting our Customer Services team or Knowledge Centre.

Buying standards

You can buy and download PDF versions of BSI publications, including British and adopted European and international standards, through our website at bsigroup.com/shop, where hard copies can also be purchased.

If you need international and foreign standards from other Standards Development Organizations, hard copies can be ordered from our Customer Services team.

Subscriptions

Our range of subscription services are designed to make using standards easier for you. For further information on our subscription products go to bsigroup.com/subscriptions.

With **British Standards Online (BSOL)** you'll have instant access to over 55,000 British and adopted European and international standards from your desktop. It's available 24/7 and is refreshed daily so you'll always be up to date.

You can keep in touch with standards developments and receive substantial discounts on the purchase price of standards, both in single copy and subscription format, by becoming a **BSI Subscribing Member**.

PLUS is an updating service exclusive to BSI Subscribing Members. You will automatically receive the latest hard copy of your standards when they're revised or replaced.

To find out more about becoming a BSI Subscribing Member and the benefits of membership, please visit bsigroup.com/shop.

With a **Multi-User Network Licence (MUNL)** you are able to host standards publications on your intranet. Licences can cover as few or as many users as you wish. With updates supplied as soon as they're available, you can be sure your documentation is current. For further information, email bsmusales@bsigroup.com.

BSI Group Headquarters

389 Chiswick High Road London W4 4AL UK

Revisions

Our British Standards and other publications are updated by amendment or revision.

We continually improve the quality of our products and services to benefit your business. If you find an inaccuracy or ambiguity within a British Standard or other BSI publication please inform the Knowledge Centre.

Copyright

All the data, software and documentation set out in all British Standards and other BSI publications are the property of and copyrighted by BSI, or some person or entity that owns copyright in the information used (such as the international standardization bodies) and has formally licensed such information to BSI for commercial publication and use. Except as permitted under the Copyright, Designs and Patents Act 1988 no extract may be reproduced, stored in a retrieval system or transmitted in any form or by any means – electronic, photocopying, recording or otherwise – without prior written permission from BSI. Details and advice can be obtained from the Copyright & Licensing Department.

Useful Contacts:

Customer Services

Tel: +44 845 086 9001

Email (orders): orders@bsigroup.com

Email (enquiries): cservices@bsigroup.com

Subscriptions

Tel: +44 845 086 9001

Email: subscriptions@bsigroup.com

Knowledge Centre

Tel: +44 20 8996 7004

Email: knowledgecentre@bsigroup.com

Copyright & Licensing

Tel: +44 20 8996 7070

Email: copyright@bsigroup.com



...making excellence a habit.™