INTERNATIONAL STANDARD

**ISO 8573-2**

Third edition
2018-02

# Compressed air — Contaminant measurement —

## Part 2: Oil aerosol content

*Air comprimé — Mesurage de contaminants —*

*Partie 2: Teneur en aérosols d'huile*



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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. www.iso.org/directives

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For an explanation on the voluntary nature of standard, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT), see the following URL: www.iso.org/iso/foreword.html

This document was prepared by Technical Committee ISO/TC 118, *Compressors and pneumatic tools, machines and equipment*, Subcommittee SC 4, *Air treatment technology*.

This third edition cancels and replaces the second edition (ISO 8573-2:2007), which has been technically revised.

A list of all the parts in the ISO 8573 series can be found on the ISO website.

# Introduction

This document requires the use of solvents to extract the oil captured on the sampling disc used in the sampling process. As a result of world-wide agreements such as the Montreal Protocol on the reduction of ozone depleting substances, a number of solvents used, for example 1,1,2 trichlorotrifluoroethane (TCTFE) have become subject to application restrictions. The revision of this document in 2007 did not identify a solvent but indicated the required characteristics.

This revision introduces the use of equipment that does not require the use of specific solvents and also an alternative solvent with reduced properties for the current method.

This revision will also include guidance to methods which provide an indication of oil aerosol content in compressed air.

# Compressed air — Contaminant measurement —

# Part 2: Oil aerosol content

## 1 Scope

This document specifies test methods for the sampling and quantitative analysis of liquid oil and oil aerosols that can typically be present in compressed air. Test methods for oil vapour are excluded from this document as they are covered by ISO 8573-5.

Two different methods are described, Method A and Method B. Method B is subdivided into two parts to clearly distinguish between procedures for obtaining the quantity of oil for analysis.

Method A describes an oil collection technique using inline coalescing filters whereas Method B utilizes sampling discs in a holder from which the collected oil is extracted with a solvent and analysed by infrared spectrometry or gas chromatography with flame ionization detection.

This document also includes descriptions of alternative oil aerosol detection by the use of indicator type devices, see Annex E.

## 2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 3857-4, *Compressors, pneumatic tools and machines — Vocabulary — Part 4: Air treatment*

ISO 8573-1, *Compressed air — Part 1: Contaminants and purity classes*

ISO 8573-5, *Compressed air — Part 5: Test methods for oil vapour and organic solvent content*

ISO 12500-1, *Filters for compressed air — Test methods — Part 1: Oil aerosols*

DIN 32645, *Chemical analysis — Decision limit, detection limit and determination limit under repeatability conditions - Terms, methods, evaluation*

## 3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 3857-4 and ISO 8573-1 apply.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at http://www.electropedia.org/

## 4 Units

General use of SI units as given throughout this document is recommended, see ISO 80000-1. However, in agreement with accepted practice in the pneumatic field, some non-preferred SI units, accepted by ISO, are also used.

1 bar = 100 000 Pa

NOTE bar (e) is used to indicate effective pressure above atmospheric.

1 l (litre) = 0,001 $m^3$

## 5 Reference conditions

Reference conditions for oil aerosol content volume statements are as follows:

— air temperature: 20 °C;

— absolute air pressure: 100 kPa [1 bar (a)];

— relative water vapour pressure: 0.

## 6 Guidance for selection of sampling method

The sampling methods can be used at any point in the compressed air system. The selection of Method A or B depends upon the actual level of oil contamination present in the compressed air system, as shown in Table 1. Where wall-flow is present, then Method A shall be used.

**Table 1 — Guidance for the selection of sampling method**

| Parameter | Method A<br>Full flow | Method B1<br>Full flow | Method B2<br>Partial flow |
|---|---|---|---|
| Min/max detection limit | >1 mg/$m^3$ | 0,001 mg/$m^3$ to 10 mg/$m^3$ | |
| Sampling time (typical) | 50 h to 200 h | 10 min to 10 h | |
| Filter construction | Coalescing line filter | Sampling disc | |

## 7 Method A — Description, measuring procedure and calculation of results

### 7.1 Description of sampling equipment and method

#### 7.1.1 General

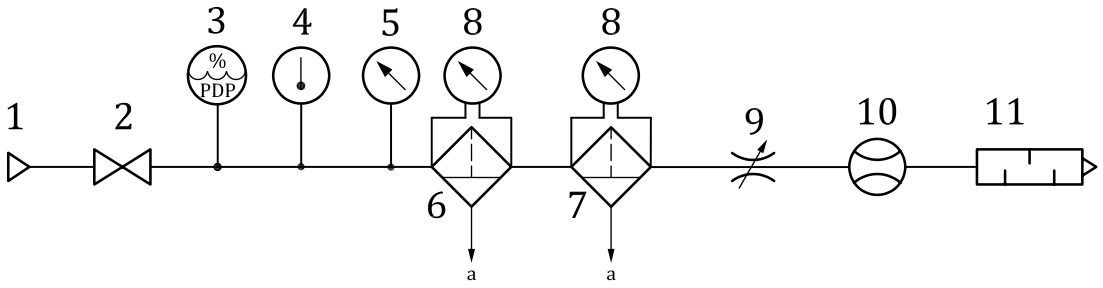
This sampling method is suitable for full flow only and samples all of the air flow that is passed through two high efficiency coalescing filters in series and measures oil in both aerosol and wall-flow forms.

This sampling method may be used at any point in a compressed air system where heavy contamination levels of oil are believed to exist.

#### 7.1.2 Sampling equipment

##### 7.1.2.1 General description

The typical arrangement of equipment used in Method A is shown in Figure 1. The sampling equipment should not influence the collection sample. An explanation of the equipment is included in the listing as follows.




**Key**

| | | | |
|---|---|---|---|
| 1 | compressed air sampling point | 8 | differential pressure sensing/measuring |
| 2 | full-flow ball valve | 9 | multi-turn flow control valve |
| 3 | pressure dewpoint sensing/measuring | 10 | flow sensing/measuring |
| 4 | temperature sensing/measuring | 11 | silencer |
| 5 | pressure sensing/measuring | a | To liquid collection. |
| 6 | sampling filter | | |
| 7 | back-up filter | | |

**Figure 1 — Typical arrangement for Method A**

a) Compressed air sampling point (see Figure 1, key item 1).

The compressed air sampling point is a test point at a nominated location in the compressed air system under investigation.

b) Full-flow ball valve (see Figure 1, key item 2).

This is an optional item for convenient connection to the compressed air sampling point and has the same bore as that of the pipe to which it is attached to prevent restrictions.

c) Pressure dewpoint sensing/measuring (see Figure 1, key item 3).

A pressure dewpoint sensing/measuring device is used to determine the moisture content of the compressed air being sampled.

d) Temperature sensing/measuring (see Figure 1, key item 4).

A temperature sensing/measuring device is used to indicate the compressed air sampling point temperature at the time of the test.

e) Pressure sensing/measuring (see Figure 1, key item 5).

A pressure-sensing/indicating device is used to confirm that the coalescing filters are operating within manufacturer's specifications.

f) Sampling filter (see Figure 1, key item 6).

The sampling filter is a high efficiency, coalescing filter capable of removing the oil whose concentration is being measured from the upstream concentration and of reducing the downstream concentration to 0,01 mg/m$^3$ or less as determined by ISO 12500-1.

The sampling filter shall be operated within the manufacturer's recommendations.

The measurements are only valid once this filter has reached steady state conditions (see Figure 2).

g) Back-up filter (see Figure 1, key item 7).

This filter is identical to the sampling filter and, in the event of malfunction of the sampling filter, collects any oil that passes through it.

h) Differential pressure gauge (see Figure 1, key item 8).

These gauges determine the pressure drop across the sample and back-up filters.

i) Flow control valve (see Figure 1, key item 9).

In order to adjust the flow accurately, a valve with fine adjustment is required.

j) Flow sensing/measuring (see Figure 1, key item 10).

A suitable flow meter with an accuracy of ±5 % of the actual value is used to determine the air sample volume, which shall be referred to reference conditions.

k) Silencer (see Figure 1, key item 11).

This is to limit the noise during the test and assist in meeting any local noise reduction requirements.

## 7.2 Sampling procedure

### 7.2.1 Start-up

The user shall ensure that the equipment selected for the measurement is safe for use at the operational pressure and temperature at which the liquids are collected and compatible with the collected liquids.

Open full-flow ball valve (see Figure 1, key item 2) fully to pressurize the sampling equipment. Adjust flow using flow control valve (see Figure 1, key item 9) to required flow conditions shown on the flow sensing/measuring device (Figure 1, key item 10).

### 7.2.2 Stabilizing sampling filter

The sampling filter element (see Figure 1, key item 6) operates in a saturated equilibrium condition and time shall be allowed for this condition to be reached. Equilibrium is considered to have been achieved when liquid oil is observed in the bottom of the filter housing in which the sampling filter is contained and the rate of change in pressure drop is less than 1 %/h of the measured pressure drop.

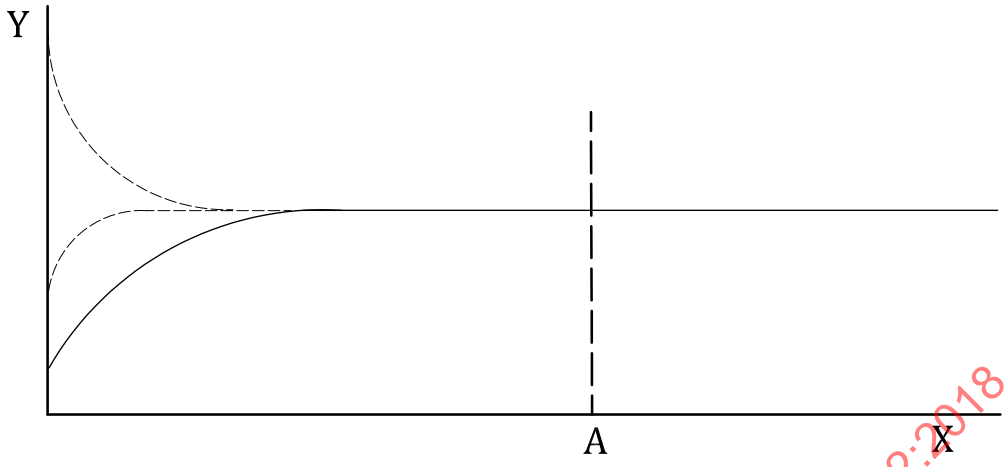
Starting from this point, the liquid collected from the drainage of the sampling and back-up filters (see Figure 1, items 6 and 7, respectively), is discharged to a collection device and the mass or volume is measured with a suitable measuring device.

Necessary precautions when discharging the liquid, include taking care in controlling the liquid flow and any subsequent rapid escape of compressed air that can cause the collected oil to foam. In addition, if air bubbles appear in the collected liquid, then allow time for settling before taking a reading of volume. The mass of the oil can be directly measured in milligrams by weighing.

Measurement shall be taken only when the differential pressure of the sampling filter reaches the stable part of the graph (from point A to point X, see Figure 2) and oil is visible in the filter bowl of the sampling filter (Figure 1, key item 6).






**Key**

| | |
|---|---|
| X | time |
| Y | pressure drop across sampling filter |
| A | position of pressure drop equilibrium (change in pressure drop is less than 1 %/h of the measured pressure drop) |
| ———— | characteristic curve for unused sampling filter |
| — — — — | characteristic curve for previously used sampling filters |

**Figure 2 — Typical characteristic curves for sampling filters**

A stable pressure drop is indicated by the differential pressure gauge (see Figure 1, key item 8). An unused sampling filter may take longer to reach a stable condition than a filter that has previously been used. The time required to reach a stable pressure drop depends on the oil/water loading.

### 7.2.3 Oil measurement

Drain the collected liquid for measurement from the sampling filter (see Figure 1, key item 6) and transfer to a suitable volumetric measuring cylinder. Measuring intervals depend upon the amount of liquid collected. Allow the collected oil to separate in order to avoid incorrect readings due to foaming, and take care during measurement to account for the meniscus. Record the volume of oil collected, *V*, in millilitres. Alternatively, the collected oil may be weighed and the mass, *m*, recorded in milligrams.

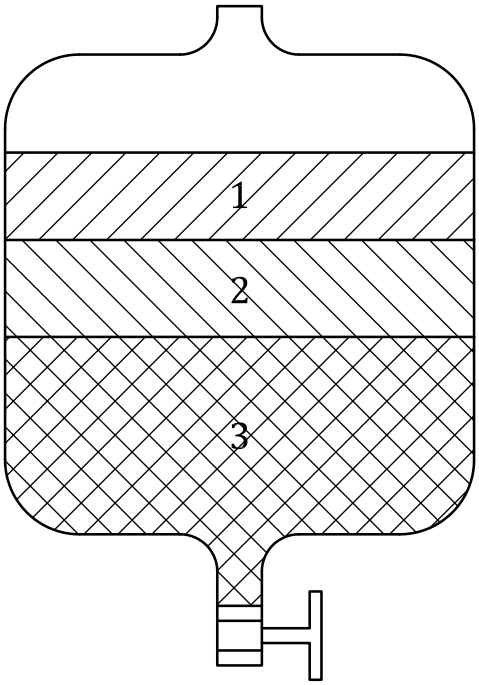
The first sampling filter (see Figure 1, key item 6) collects the oil to the required accuracy. The back-up filter (see Figure 1, key item 7) is used to ensure the first sampling filter has functioned correctly. Any sign of oil in the second filter may indicate that it is necessary to replace the first filter element.

### 7.2.4 Oil/water measurements

The liquid collected consists of water, oil/water emulsion and oil. Depending on the type of oil, separation of the oil/water emulsion can occur, allowing the water to be drained off and the oil to be measured; see Figures 3 and 4.

If a water/oil emulsion zone occurs, drain the oil-free water then add a measured quantity of solvent and stir to dissolve the oil; see Figure 4.

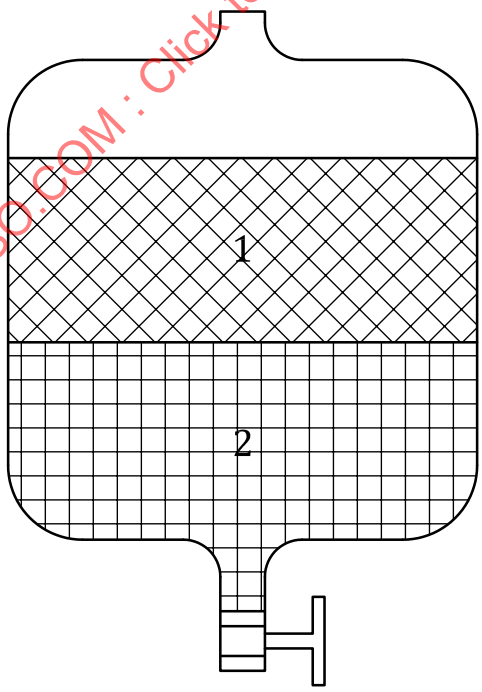
The collected oil and solvent may be weighed and the mass recorded in milligrams having subtracted the solvent mass.




**Key**

1 oil
2 oil/water emulsion
3 water

**Figure 3 — Oil/water separator**




**Key**

1 water
2 oil/solvent solution

**Figure 4 — Oil-solvent/water separator**

Drain the heavier oil/solvent solution and measure the actual quantity of oil collected by subtracting the measured quantity of solvent from the total. Record the volume of oil collected, *V*, in millilitres. Alternatively, the collected oil may be weighed and the mass, *m*, recorded in milligrams.

#### 7.2.5 Air flow-rate (discharge)

The air flow-rate measurement should have an accuracy of better than 5 % at the actual flow being measured.

#### 7.2.6 Temperature

The temperature is measured in degrees Celsius with an accuracy of better than 1 °C.

### 7.3 Calculation of test results

#### 7.3.1 General

The accuracy of the test is dependent on the volume of oil collected and increases with increasing volume of oil and collection time. It is necessary to ensure that results are stable, repeatable and presented in a form that shows that this has been achieved.

#### 7.3.2 Oil content

When the volume of the collected oil is measured, the oil content, *X*, in milligrams oil per cubic metre air, is calculated using Formula (1):

$$X = \frac{V \times \rho}{q \times H \times 3,6} \tag{1}$$

where

*V* is the volume of oil collected, expressed in millilitres;

$\rho$ is the specific density of the oil, expressed in kilograms per cubic metre;

*q* is the air flow-rate, expressed in litres per second at reference conditions; see Clause 5;

*H* is the duration of the test, expressed in hours.

When the mass of the collected oil is measured, the oil content, *X*, is calculated using Formula (2):

$$X = \frac{m}{q \times H \times 3,6} \tag{2}$$

where *m* is the mass of oil, expressed in milligrams.

## 8 Method B — Description, measuring procedure and calculation of results

### 8.1 General description of sampling equipment and method

Method B deals with the sampling and analysis of oil aerosols at constant flow rate. Within the constraints detailed above, this method permits the quantification of oil aerosols present in a compressed air system, provided wall-flow contamination is not present.

The method is subdivided into procedures B1 and B2. Method B2 uses the same sampling equipment employed in Method B1; with the addition of a sampling probe to allow partial-flow sampling under isokinetic conditions from the main pipe flow if the velocity constraints of the air flowing through the sampling disc of Method B1 are exceeded. Accuracy and limitations are as stated in Method B1.

The optimum duration for sample measurement may be determined after an initial test to determine the approximate oil concentration present. When carrying out full-flow sampling, it is possible to route the air back into the compressed air system, preventing loss of the product. Conversely, it is also possible to vent the flow to the atmosphere. Flow measurement is required to determine the volume of air sampled, whichever method is adopted. As the sampling apparatus is portable, different sample locations may be chosen provided the stated parameters are not exceeded and suitable connections for insertion of the sampling equipment into the circuit exists. Obvious precautions to prevent shock depressurization, which can damage the sampling discs, or ingress of atmospheric contamination are necessary.

The sampling and analysing equipment used as described give an accuracy of better than ±10 % over the range from 0,001 mg/m$^3$ to 10 mg/m$^3$ oil content with a minimum sampling time calculated to collect sufficient oil to meet the requirements of the oil mass-per-volume of solvent used when determining the response characteristics of the measuring equipment. The upper limit for the air velocity (at operating pressure) in front of the sampling disc is 1 m/s.

As this method concerns the measurement of relatively low concentrations of oil aerosol in air, particular attention shall be paid to the cleanliness of the sampling equipment and other precautions shall be taken, e.g. valve purging and stabilization to constant test conditions. Good analytical techniques help improve the confidence level of the measurements. At very low oil concentration the recommended sampling time should be increased.

### 8.1.1 Sampling disc

In order to obtain good measuring accuracy, a high-efficiency binder free microfibre sampling disc or similar shall be used. To achieve the accuracy specified for this method, three or more layers of sampling discs in series and in intimate contact shall be used and the individual sampling discs should meet the requirements given in Table 2.

**Table 2 — Typical high-efficiency microfibre glass sampling disc properties**

| Parameter | Specification |
|---|---|
| Particle removal efficiency (1 and 2) % | >99,995 |
| Surface mass, g/m$^2$ | 130 to 150 (for glass fibre)<br>80 to 90 (for quartz fibre) |

NOTE 1 The particle removal efficiency is typically measured according to EN 1822-3[3].

NOTE 2 An equivalent particle penetration rating is a 1 µm particle retention in liquid filtration. Suitable binder free glass fibre sampling discs typically have a thickness of 0,7 mm, a surface mass of 140 g/m$^2$ and air resistance of 95 mbar per disc, binder free quartz fibre sampling discs a thickness of 0,4 mm, a surface mass of 85 g/m$^2$ and air resistance of 50 mbar per disc.

### 8.1.2 Sampling disc support

In order to prevent the collection sampling disc from bursting, it shall be supported by a robust, inert material that is sufficiently strong to withstand the differential pressures of the discs in use during sampling. The pressure drop losses from the support should be minimized to allow the sampled compressed air flow to pass with a minimum of resistance; see Annex B.

### 8.1.3 Pipes and valves

It is important that the pipe inner diameter from the connection point in the compressed air system to the sampling disc holder be constant and crevice free to minimize system loss.

The valves (for example see Figure 5, key item 8) should be full-flow ball type and the hole in the ball should have approximately the same diameter as the bore of the pipe.

The bypass pipe (see Figure 5, key item 6) may consist of a flexible tube and although a full-flow ball valve (see Figure 5, key item 8) is indicated, this may be of any convenient type.

### 8.1.4 Sampling disc holder

The design of the sampling disc holder shall be such that the air flow is evenly distributed across the surface to prevent jetting which can cause an uneven oil loading or even damage to the sampling disc surface. One such design of sampling disc holder can be seen in Figure B.3.

In circumstances where only a portion of the sampling disc is to be analysed by solvent extraction, tests shall establish that the oil is distributed evenly throughout and the measured oil quantity corrected for the ratio of the analysed area to the total area used during sampling.

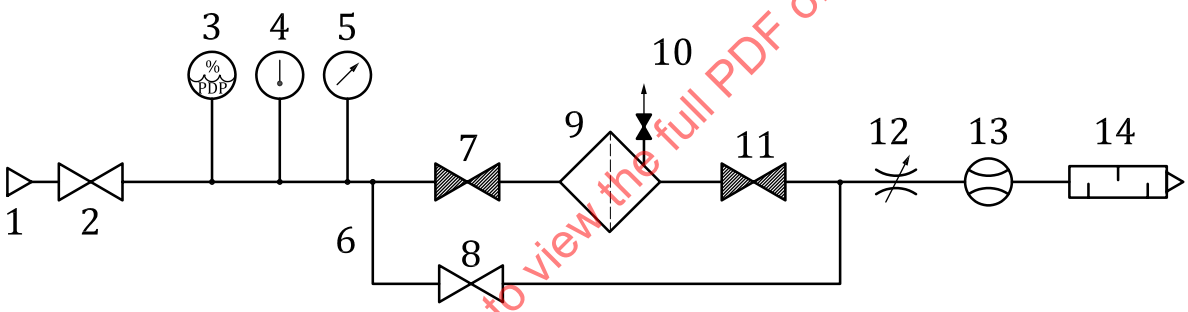
### 8.1.5 Construction materials

Aluminium and its alloys shall not be used for any component that can come into contact with the analysis solvent.

## 8.2 Sampling equipment arrangement

### 8.2.1 Sampling equipment Method B1 — Full flow sampling

A general arrangement of typical sampling equipment is shown in Figure 5.




**Key**

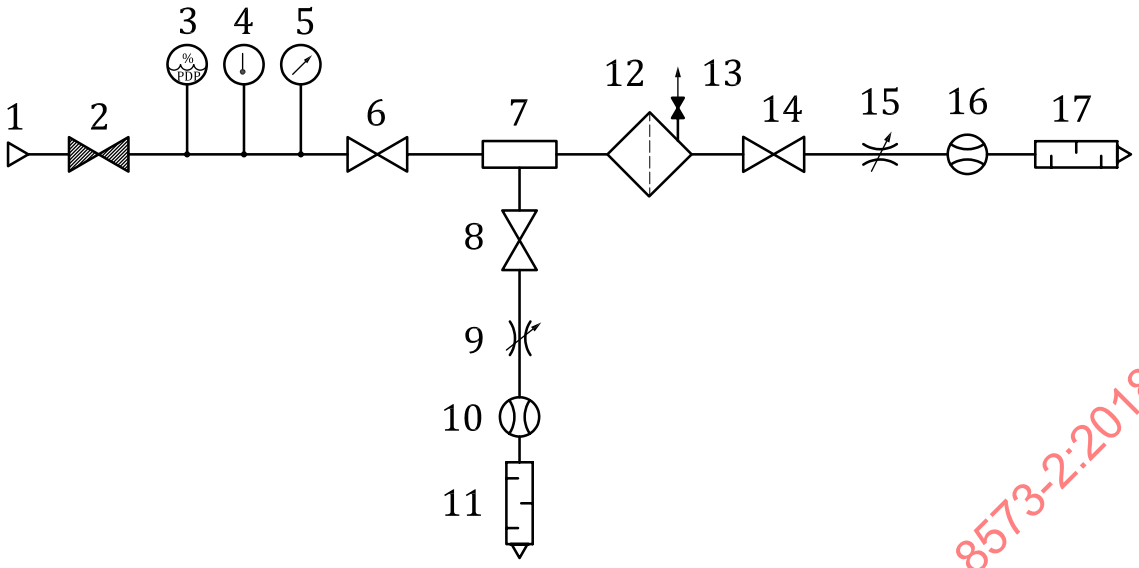
| | | | |
|---|---|---|---|
| 1 | compressed air sampling point | 8 | full-flow ball valve (open) |
| 2 | full-flow ball valve (open) | 9 | sampling disc holder |
| 3 | pressure dewpoint sensing/measuring | 10 | sampling disc holder depressurising valve |
| 4 | temperature sensing/measuring | 11 | full-flow ball valve (closed) |
| 5 | pressure sensing/measuring | 12 | multi-turn flow control valve |
| 6 | bypass pipe | 13 | flow sensing/measuring |
| 7 | full-flow ball valve (closed) | 14 | silencer |

**Figure 5 — Typical arrangement for Method B1**

In Method B1 all of the sampled air flow is diverted through the sampling equipment via suitable in-line valves, which have been previously checked to ensure they do not contribute to the level of oil contamination already present.

### 8.2.2 Sampling equipment Method B2 — Partial flow sampling

A general arrangement of typical sampling equipment is shown in Figure 6.




**Key**

| | | | |
|---|---|---|---|
| 1 | compressed air sampling point | 10 | flow sensing/measuring |
| 2 | full-flow ball valve (closed) | 11 | silencer |
| 3 | pressure dewpoint sensing/measuring | 12 | sampling disc holder |
| 4 | temperature sensing/measuring | 13 | sampling disc holder depressurising valve |
| 5 | pressure sensing/measuring | 14 | full-flow ball valve (open) |
| 6 | full-flow ball valve (open) | 15 | multi-turn flow control valve |
| 7 | isokinetic probe insertion point — see Figure 7 | 16 | flow sensing/measuring |
| 8 | full flow ball valve (open) | 17 | silencer |
| 9 | multi-turn flow control valve | | |

**Figure 6 — Typical arrangement for Method B2**

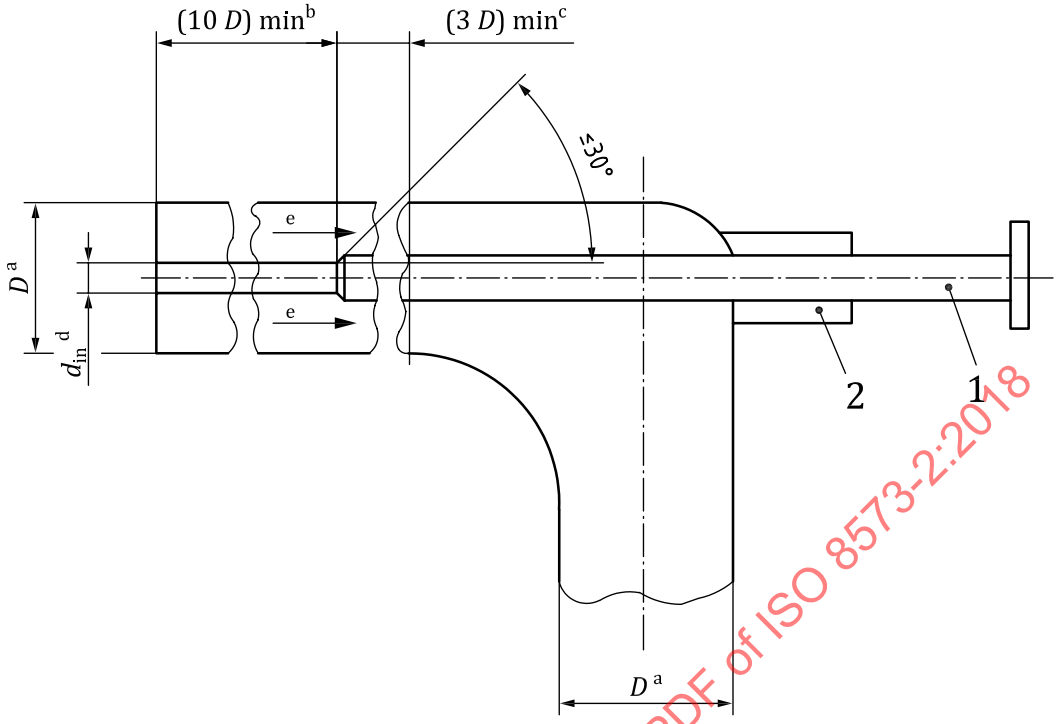
In Method B2 a proportion of the air flow is sampled using a sample probe operated at isokinetic conditions, and allows a sample of air to be taken from the compressed air supply under identical velocity conditions. The test apparatus can be attached to any section of the compressed air system using suitable connections and valves, which have been previously checked to ensure they do not contribute to the level of oil contamination already present. The probe may be inserted to an approximately central position across the main pipe diameter and it is recommended that a number of preliminary tests be made.

It is necessary to know both compressed air supply pipe flow and sample flows to define the sampling conditions.

The pressure seals used in the probe/holder connectors shall not release any hydrocarbon into solution when in contact with the analysing solvent. It is impractical to return the sample flow to the main pipe flow downstream from the sampling disc holder, and it is usual to vent this flow to atmosphere.

### 8.2.3 Equipment set-up for isokinetic sampling

The set-up for the isokinetic sampling probe at the insertion point of the compressed air system under investigation is shown in Figure 7.




**Key**

| | | | |
|---|---|---|---|
| 1 | isokinetic sampling probe in the main pipe | a | Main pipe inside diameter, $D$. |
| 2 | adjustable gland to allow adjustment of probe | b | Minimum straight length in front of probe, ≥10 × $D$. |
| | | c | Probe insertion point at minimum of ≥3 × $D$. |
| | | d | Internal probe diameter, $d_{in}$. |
| | | e | Direction of flow. |

**Figure 7 — Typical set-up of probe insertion for isokinetic sampling**

For sampling from high-flow systems, isokinetic sampling may be used when wall-flow is not present.

### 8.2.4 Compressed air flow-rates for isokinetic conditions

The air velocities within the compressed air pipeline and within the probe shall be identical throughout the sampling period. This is accomplished through adjustment of the flow controllers to provide appropriate readings on the flow meters.

Verification of identical pipe and probe velocities can be evaluated by the following procedures.

**If sampling disc size is known**

| | |
|---|---|
| Start | |
| ↓ | |
| Select a disc size | |
| ↓ | |
| Check that flow in the main pipe is turbulent | $q_m > D/20$ |
| ↓ | |
| Calculate the maximum probe diameter | $d_{in} \leq 0{,}028 \times D_m \times D \times \left[(p+1)/q_m\right]^{0,5}$ |
| Select probe diameter for possible use that is $\leq d_{in}$ | |
| ↓ | |
| Calculate sample flow using actual probe diameter selected | $q_p = q_m \times (d_{in}/D)^2$ |
| ↓ | |
| Check minimum sampling disc diameter is OK | $D_m \geq 35{,}7 \times \left[q_p/(p+1)\right]^{0,5}$ |
| ↓ | |
| Test | |

**If probe size is known**

| | |
|---|---|
| Start | |
| ↓ | |
| Select a probe size | |
| ↓ | |
| Check that flow in main pipe is turbulent | $q_m > D/20$ |
| ↓ | |
| Calculate sample flow rate for isokinetic conditions | $q_p = q_m \times (d_{in}/D)^2$ |
| ↓ | |
| Calculate minimum sampling disc diameter | $D_m \geq 35{,}7 \times \left[q_p/(p+1)\right]^{0,5}$ |
| ↓ | |
| Select a sampling disc holder | |
| ↓ | |
| Test | |

where

$q_m$ is the flow through the compressed air supply pipe, expressed in litres per second (at reference conditions);

$q_p$ is the flow through the isokinetic sample probe, expressed in litres per second (at reference conditions);

$d_{in}$ is the internal diameter of probe, expressed in millimetres;

$D$ is the internal diameter of pipe, expressed in millimetres;

$D_m$ is the sampling disc diameter, expressed in millimetres;

$p$ is the system pressure, expressed in bar(e).

NOTE The check for turbulent conditions is based upon a calculated Reynolds number of >4 000.

## 8.3 Equipment and sampling disc preparation

The oil aerosol sample collection method on to the sampling discs is the same for Methods B1 and B2. Where the concentration of oil aerosol is unknown, an initial test can be carried out to establish the approximate level.

### 8.3.1 Equipment preparation

The sampling equipment shall be free from oil and other contaminants before it is connected to the system. This is particularly important for the parts between the connection point and the sampling disc holder. Once the holder has been cleaned, handle it only using polyethylene gloves to protect from finger-borne grease contamination. Using tweezers, pre-load the holder (see B.3) with suitable sampling discs and insert a suitable seal on the inlet flow side of the outside diameter of the sampling discs to provide an integral seal.

NOTE Equipment cleaning is of particular importance when the equipment is used at different test locations or sites.

The chamber may then be suitably clamped externally to allow pressurization to system pressure. Mount the holder in the sampling pipe as shown schematically in Figures B.1 and B.2.

The assembly should not contribute any hydrocarbon background of its own; checks to establish this can be performed by analysis of the solvent used to cleanse the assembly prior to use.

### 8.3.2 Oil vapour

To prevent condensation of oil vapour, isothermal conditions should be maintained between the main stream and the sample stream for the period of the test.

### 8.3.3 Temperature

The temperature is measured in degrees Celsius with accuracy better than 1 °C.

### 8.3.4 Handling

Clean sampling discs shall be stored such that they are protected against dust and atmospheric contamination. The use of a pair of tweezers is recommended when the sampling discs are placed in and taken out of the sampling disc holder. After the measurement is completed and before analysis, the exposed sampling discs shall be stored and protected from extraneous contamination.

NOTE An amber glass bottle with stopper is most suitable, so that the sample is sealed from the air and light.

Do not store the sampling discs in containers made from organic or hydrocarbon materials. The sampling discs should be stored in a cool place below 6 °C as soon as practicable to prevent sample loss through evaporation.

#### 8.3.5 Sampling disc contamination check

In order to check that the sampling discs are clean, choose a new sampling disc at random, commonly known as a "blank sample". Analyse this disc to make sure that it does not contain oil. Additionally, the following applies:

a) laboratory blank — taken from the original packaging;

b) field blank — taken to the point of measurement, exposed to the measurement location.

Analyse the laboratory blank before beginning the sampling. Analyse the field blank on its return to the laboratory. For the field blank, where oil is found that will be reported.

### 8.4 Compressed air sampling procedure

#### 8.4.1 Typical sampling Method B1

##### 8.4.1.1 Start-up

The aerosol oil content is measured by means of collection sampling discs (see Figure 5 and 8.2.1), which are placed in the sampling disc holder. Before inserting the sampling discs, divert the air through the bypass pipe as follows.

a) Turn full-flow ball valve in the bypass pipe (see Figure 5, key item 8) to the open position.

b) Close valves (see Figure 5, key item 7 and key item 11). The air flow is now diverted through the bypass tube (see Figure 5, key item 6).

c) Evacuate the sampling disc holder by means of the valve (see Figure 5, key item 10) and the sampling disc holder can be removed.

d) Open the sampling disc holder (see Figure 5, key item 9) and place a supporting disc and three or more sampling discs in to the holder (the supporting disc shall be placed after the sampling discs (downstream); see also Figure B.2 for more details relating to the arrangement of the sampling discs in the holder).

e) Close the depressurising valve (see Figure 5, key item 10) and insert the sampling disc holder.

##### 8.4.1.2 Sampling

The aerosol measuring device is now ready and is used in the following way.

a) Open the sample holder inlet valve (see Figure 5, key item 7) carefully, so that the sampling disc holder is pressurized.

b) Turn valve (see Figure 5, key item 11) to the open position and carefully close valve (see Figure 5, key item 8), the flow control valve (see Figure 5, key item 12) having been pre-set to provide the required flow through the sampling disc as displayed on the flow sensing/measuring device (see Figure 5, key item 13). Ensure that the maximum flow rate through the sampling disc does not exceed a velocity of 1 m/s, see 8.1.

c) Measure the time required for collection, which should be at least 2 min, stop the test by opening valve (see Figure 5 key item 8) and closing valve (see Figure 5, key item 11).

d) Close the sample holder inlet valve (see Figure 5, key item 7) and evacuate the sampling disc holder using valve (see Figure 5, key item 10).



e) Either remove the sampling discs, or place the whole assembly, in a hydrocarbon-free container, seal and protect from contaminants.

For methods of analysis and calculation of the amount of oil present, see Clause 9.

### 8.4.2 Typical sampling Method B2

#### 8.4.2.1 Start-up

The aerosol oil content is measured by means of sampling discs (see Figure 6 and 8.2.2), which are placed in the sampling disc holder. Before inserting the sampling discs, release the pressure from the sampling disc holder as follows.

a) Turn full-flow ball valves (see Figure 6, key items 6, 8 and 14) to the closed position to isolate the apparatus from the compressed air supply.

b) Evacuate the sampling disc holder and supply lines by means of the valve (see Figure 6, key item 13) and remove the sampling disc holder (see Figure 6, key item 12).

c) Open the sampling disc holder (see Figure 6, key item 12) and place a supporting disc and three layers or more of sampling discs in to the holder (the support disc shall be placed after the sampling discs (downstream); see also Figure B.2).

d) Re-install the sampling disc holder on to the isokinetic probe insertion point (see Figure 6, key item 7).

e) Close the depressurising valve (see Figure 6, key item 13).

#### 8.4.2.2 Sampling

The aerosol sampling device is now ready and is used in the following way.

a) Open the compressed air supply valves carefully (see Figure 6, key item 2 and key item 6), so that the sampling disc holder is pressurized.

b) Turn valve (see Figure 6, key item 8) to the open position, the flow control valve (see Figure 6, key item 9) having been pre-set to provide the required supply flow as displayed on the flow sensing/measuring device (see Figure 6, key item 10).

c) Turn valve (see Figure 6, key item 14) to the open position, the flow control valve (see Figure 6, key item 15) having been pre-set to provide the required flow through the sampling disc holder as displayed on the flow sensing/measuring device (see Figure 6, key item 16). Ensure that the maximum flow rate through the sampling disc does not exceed a velocity of 1 m/s, see 8.1.

d) Measure the time required for collection, which should be at least 2 min, stop the test by closing valve (see Figure 6, key item 14).

e) Turn valve (see Figure 6, key item 8) to the closed position and then close the compressed air supply valve (see Figure 6, key item 6) and evacuate the sampling disc holder using valve (see Figure 6, key item 13).

f) Remove the sample disc holder and isokinetic sample probe from the isokinetic probe insertion point (see Figure 6, key item 7) and either remove the sampling discs, or place the whole assembly, in a sealed hydrocarbon-free container and protect from extraneous contaminants.

For methods of analysis and calculation of the amount of oil present, see Clause 9.

# 9 Analytical procedure for Methods B1 and B2

## 9.1 General

Examples of the analytical procedure to be followed for Method B1 and B2 are given in Annex C for the IR method and in Annex D for the GC FID method.

## 9.2 Apparatus

### 9.2.1 Usual laboratory glassware

Clean all glassware by the usual procedures for this type of analysis and check for cleanliness.

### 9.2.2 Infrared spectrometer (IR)

Double-beam or Fourier-transform infrared spectrometer covering a wave number range of at least 3 400 $cm^{-1}$ to 2 500 $cm^{-1}$ and with a transmittance reproducibility of better than 0,3 %.

### 9.2.3 Gas chromatograph and flame ionization detector (GC-FID)

A gas chromatograph equipped with a non-discriminating inlet system and flame ionization detector (GC-FID).

## 9.3 Analytical procedure linearity check

A straight-line least-squares regression analysis shall be performed to establish that the instrument response from either the IR or GC-FID analytical methods used was linear across the full instrument response range. The computed linearity, as indicated by the coefficient of determination, $R^2$, shall be no less than 0,995; otherwise, the analysis shall be repeated.

## 9.4 Analytical procedure — Oil recovery coefficient

To establish that the solvent selected has the correct level of solvency for the oil being investigated a sampling disc is dosed with a predetermined mass of oil which is subsequently extracted and the mass calculated by use of the response coefficient (see C.1.7.3 and D.1.7.2). The mass extracted is compared directly to the original dosed mass and the recovery coefficient calculated.

This method is performed 3 times at each of 10 %, 50 % and 90 % of the maximum allowable mass to be collected on the sampling discs.

The average oil recovery coefficient calculated from all nine dosed sampling discs shall not deviate by more than 1 ± 0,15. The recovery coefficient is equal to 1 if the extracted oil from the sampling discs is exactly equal to the amount of oil that it was dosed on the sampling discs.

To determine the dosed oil recovery coefficient, the procedure detailed in C.1.5 Spectroscopy or D.1.5 GC-FID can be used.

## 9.5 Limit of detection

The limit of detection shall be determined in accordance with DIN 32645.

## 9.6 Calculation of test results

## 9.7 General

The mass of oil, *m*, determined to have been extracted from the sampling disc holder, sampling discs and isokinetic sample probe (when used), obtained from the analytical procedures in Annex C or Annex D,



is then substituted in Formula (2) to determine the oil content of the compressed air sample under investigation.

## 10 Presentation of results

Minimum data to be recorded and their form of presentation are shown in Annex A, which includes the following:

— date of test;

— location;

— equipment;

— duration of sampling;

— oil type and reference;

— oil concentration;

— temperature;

— pressure;

— full flow;

— sample flow;

— analytical technique used (IR or GC-FID);

— instrument response data;

— coefficient of linearity;

— recovery coefficient;

— test method.

## 11 Uncertainty of the method

Using the test apparatus as described, the uncertainty of the method is better than ±10 % of the measured value.

# Annex A
(informative)

# Typical test report

When measured under the conditions mentioned below, the compressed air sample, analysed according to method (A, B1 or B2) has been found to contain $X$ mg/m$^3$ of oil in liquid or aerosol form.

Conditions:

The sample was taken from (e.g. receiver, main pipe, branch pipe, etc.).

At the sampling point, the following conditions prevailed:

| | |
|---|---|
| Test reference: | Date of test: |
| Location: | |
| Pressure: | kPa [bar (e)] |
| Temperature: | °C |
| Full flow: | l/s |

The compressed-air system had the following configuration (specify if appropriate):

| |
|---|
| Equipment: |
| Test method [ A: B 1: B 2] |
| Instrument response data: Analytical technique [IR – FT/IR – GC/FID] |
| Oil type and reference: |
| Duration of sampling (h): Sample flow: l/s |
| Coefficient of linearity $R^2$: Oil recovery coefficient $O_a$: |
| Oil concentration: $X$ mg/m$^3$ |

General

| | |
|---|---|
| Compressor 1 was running at | % load |
| Compressor 2 was running at | % load |
| Compressor 3 was running at | % load |
| Compressor n was running at | % load |

Additional Notes

| |
|---|
| |
| |
| |
| |

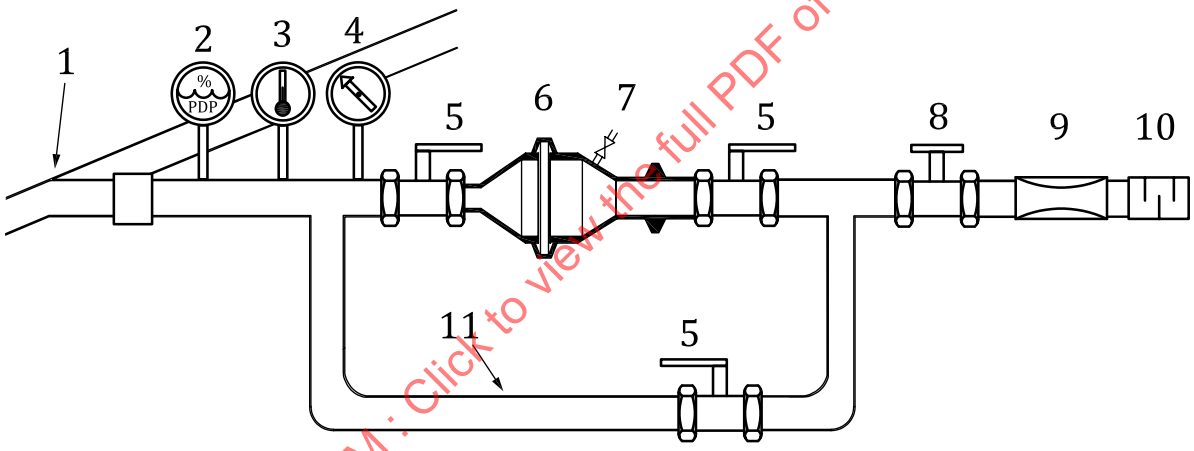
# Annex B
(informative)

# Typical equipment layout and dimensional details

## B.1 Method B1

The general arrangement of the test equipment at the point of measurement is shown in Figure B.1. The sampling disc holder shown is that for which a design is included in B.3. The type of holder may actually be any commercially available type which utilizes the same circular section as the sampling disc holder (key item 6).

The test apparatus, including sampling disc holder assembly, especially upstream of the sampling disc should be conditioned to be at the same temperature as the test air, otherwise condensation or evaporation can occur.




**Key**

1 compressed air sampling point
2 pressure dewpoint sensing/measuring
3 temperature sensing/measuring
4 pressure sensing/measuring
5 full-flow ball valve
6 sampling disc holder
7 sampling disc-holder depressurising valve
8 multi-turn flow-control valve
9 flow sensing/measuring
10 silencer
11 bypass pipe

**Figure B.1 — Typical test equipment for Method B1: Full flow sampling**

## B.2 Method B2

### B.2.1 Isokinetic sampling — General

The test apparatus including the isokinetic sample probe and sampling disc holder assemblies especially upstream of the sampling disc should be conditioned to be at the same temperature as the test air, otherwise condensation or evaporation can occur.

Isokinetic sampling devices should exhibit the following characteristics.

a) The probe should be a minimum distance of 10 pipe diameters from upstream bends or restrictions and three diameters from downstream bends or restrictions.

b) The size of the probe should not influence the air stream. The nozzles may vary in shape and construction.

c) Precautions are necessary to prevent surface condensation of oil vapours.

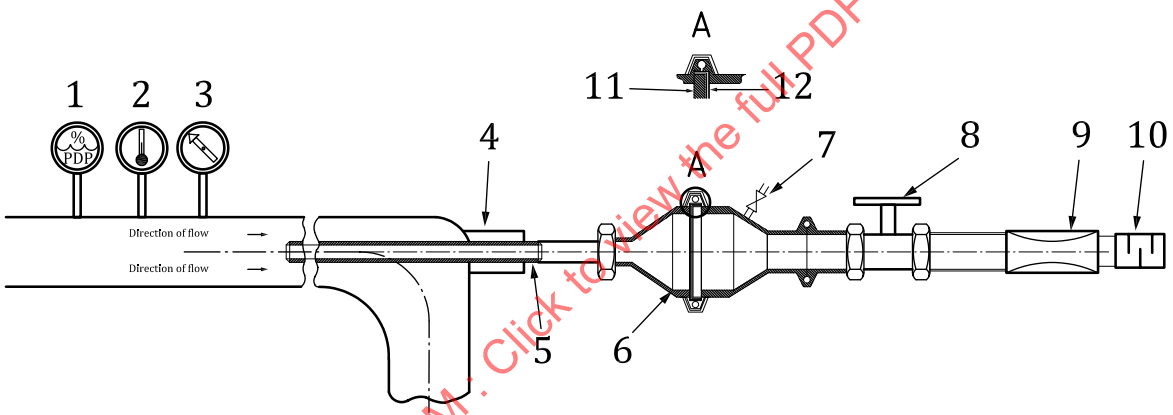
Turbulent flow conditions within the main air stream are required for sampling (Reynolds number greater than 4 000).

In normal industrial use, compressed air is in a state of turbulent flow, which occurs when the flow, $q$, expressed in litres per second at reference conditions, in the pipe meets the condition in Formula (B.1):

$$q_m > D/20 \quad \text{(B.1)}$$

where $D$ is the pipe bore, expressed in millimetres.

The test set-up for isokinetic sampling comprises the elements as shown in Figure B.2. The valve (key item 8) and flow meter (key item 9) allow adjustment and measurement of sample flow, respectively.



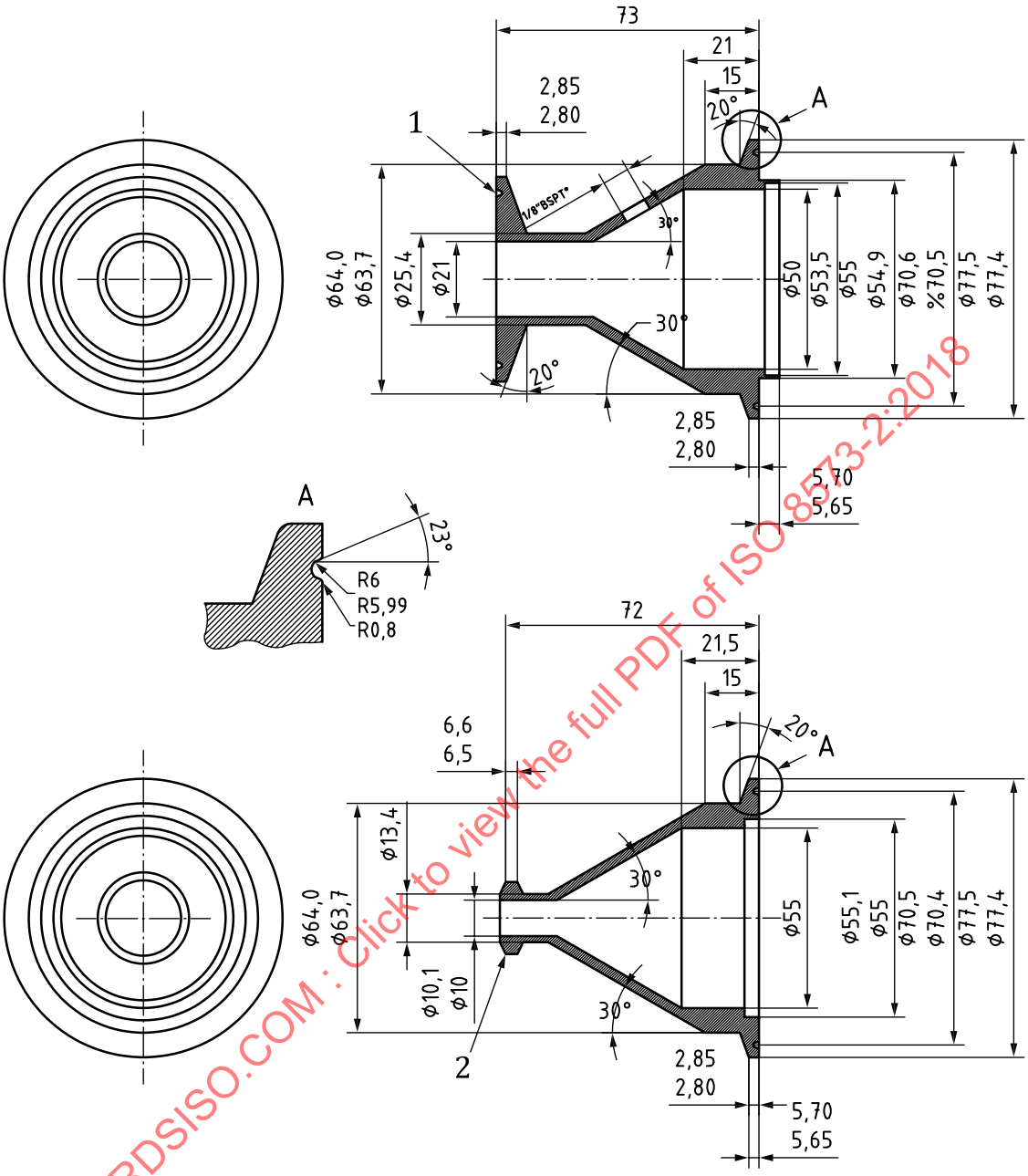

**Key**

| | | | |
|---|---|---|---|
| 1 | pressure dewpoint sensing/measuring | 7 | sampling disc-holder depressurising valve |
| 2 | temperature sensing/measuring | 8 | multi-turn flow-control valve |
| 3 | pressure sensing/measuring | 9 | flow sensing/measuring |
| 4 | nozzle with gland | 10 | silencer |
| 5 | isokinetic probe | 11 | three or more sampling disc layers |
| 6 | sampling disc holder | 12 | stainless steel support disc |

**Figure B.2 — Typical test equipment for Method B2, isokinetic sampling (shown for bend insertion of probe)**

## B.3 Design of sampling disc holder

Figure B.3 provides guidance on the dimensioning of the various elements used in the construction of the sampling disc holder. The design shown in Figure B.3 facilitates the use of 55 mm sampling discs.




**Key**

1 typical crevice-free joint

2 crevice-free joint to suit probe

**Figure B.3 — Typical sampling disc holder**

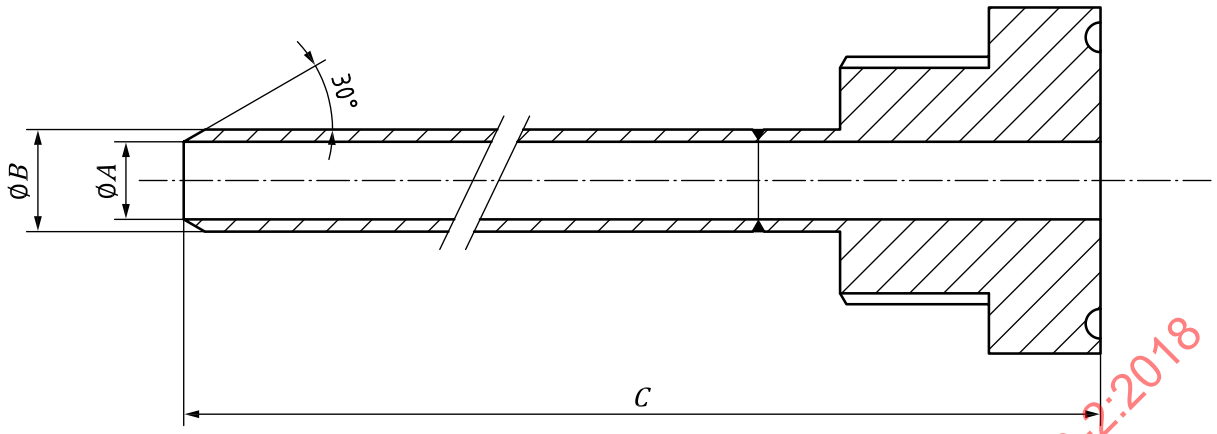


## B.4 Design of the isokinetic probe

The general construction of the probe is shown in Figure B.4 and is intended for use at reference conditions at 700 kPa (7 bar) to match the velocity of that in the main line for sampling when used with a suitable sampling disc holder designed for a 55 mm diameter standard disc.

The probe should be of circular cross-section, the open end having a thickness of less than 1,5 mm and the internal and external surfaces having an inclination not greater than 30° to the axis of the nozzle; see Figure B.4.

The angle at the nozzle minimizes the effect of impact onto the end of the probe.




| Typical probe sizes | A<br>mm | B<br>mm | C<br>mm |
|---|---|---|---|
| 1 | 7 | 9,6 | 200 |
| 2 | 10 | 12,6 | 200 |
| 3 | 17 | 19,6 | 400 |

**Figure B.4 — Typical isokinetic sampling probes**

## B.5 Probe installation

An isokinetic sampling probe should not create a problem, providing the general design in Figure B.4 is followed. A simple compression seal may be used, provided a fluorocarbon elastomer (or similar) seal is used to prevent contamination during the analysis procedure.

For the insertion of the isokinetic sampling tube, a gland with an integrated seal can be used. This seal shall be capable of maintaining the probe in the pipe up to the maximum working pressure. Ideally this gland should allow insertion of the probe to differing lengths if necessary.

# Annex C
(informative)

# Example IR analysis and calculation of test results for Method B

## C.1 Analytical procedure

### C.1.1 General

The following describes an example procedure based on IR spectroscopy that has been proven to be suitable for the determination of the mass of oil, *m*, expressed in milligrams (mg) collected by the sampling apparatus.

The response of the IR spectrometer is performed to determine the instrument response and linearity thus establishing the operational range over which the measurements are made. In addition, the degree of recovery of oil from the sampling discs using solvent is also determined. These parameters shall meet the minimum levels required by the document.

Once the response, linearity and the oil recovery coefficient have been established the amount of oil collected on the sampling apparatus can be analysed and the concentration of oil in compressed air calculated.

### C.1.2 Principle

Oil collected on the sampling disc, sample disc holder and isokinetic sample probe if present (Method B2) is extracted from the apparatus with a suitable optical grade solvent and the amount collected determined by infrared spectroscopy.

The concentration of oil in solution is proportional to the absorbance of infrared (IR) light at two or more wave numbers and is characteristic for each oil under investigation. These are normally in the range 2 960 $cm^{-1}$ and 2 860 $cm^{-1}$, but the exact peak positions and number are dependent on the solvent and oil type.

The absorbance obtained due to the presence of an unknown amount of oil in the solvent is converted to an oil mass concentration in solvent by use of the response coefficient.

Due to variation in instrument response the oil used to perform the response and level of recovery analysis shall be the same as that found in the compressed air and if necessary samples shall be obtained from the compressed air system for this purpose. The same absorbance peaks shall be employed in the analysis.

### C.1.3 Solvent

For the purpose of oil extraction from the sampling discs and holder, a suitable solvent, for example tetrachloroethylene, shall be chosen that has a good solvency for the oil in question, contains no C-H bonds in its chemical composition and has an optical transparency at the infrared wave numbers of interest. The reagents should be handled with care and in accordance with manufacturer or supplier instructions. The solvents chosen should be specifically prepared for use in infrared spectroscopy. An example of the typical purity of tetrachloroethylene is given in Table C.1 for guidance.

NOTE Tetrachloroethylene is also known under the systematic name tetrachloroethene, or perchloroethylene.

**Table C.1 — Example of typical purity of tetrachloroethylene**

| Designation | Specification |
|---|---|
| Chemical formula: | $C_2Cl_4$ |
| Ultrapure, Spectrophotometric Grade | >99 % purity |
| CAS number: | 127–18–4 |
| Molecular weight: | 165,83 g/mol |
| Residual water: | ≤0,05 % |
| Residue on evaporation: | <0,000 5 % |

## C.1.4 Apparatus

### C.1.4.1 Glassware

Glassware used for determination of volume shall be of Class A according to ISO 1042, borosilicate glass with glass stopper and supplied with a batch certificate. It shall be cleaned before use and checked to ensure that it does not contribute any hydrocarbons during the analysis process.

### C.1.4.2 Infrared Spectrometer

Double-beam or Fourier-transform infrared spectrometer covering a wave number range of at least 3 400 $cm^{-1}$ to 2 500 $cm^{-1}$ and with a transmittance reproducibility of better than 0,3 %.

### C.1.4.3 Infrared Spectrometer Cuvettes

Various designs of cuvettes are available for application in IR spectroscopy. Quartz glass, lidded cells have been found to be suitable and where more than one cell is required (i.e. twin beam spectrometers) in the analysis all cells should be matched pairs. A suitable cuvette path-length should be determined to match the response and linearity required by the method. Cuvette path-lengths between 1 cm and 4 cm have been found to prove suitable in the majority of cases.

## C.1.5 Instrument response, linearity and oil recovery coefficients

The instrument response, linearity and oil recovery coefficient shall be determined as follows;

### C.1.5.1 Preparation of the response graph

Using an analytical balance or other precision weighing instrument with a resolution of no less than 0,01 mg, weigh out 100 mg of the oil under investigation and dilute it to a volume of 100 ml with clean solvent to create a stock solution with an oil concentration of 1 mg/ml. If the exact amount of oil cannot be dispensed then the actual amount weighed out shall be recorded for use later in calculation of the sample concentrations.

From this initial stock solution, prepare six samples that span the expected range of oil concentrations. Samples should be prepared by measuring a prescribed volume of the initial stock solution and diluting it with clean solvent to a volume of 50 ml. Prescribed volumes of the initial stock solution and the resulting oil concentrations of the diluted samples are given in Table C.2.

**Table C.2 — Dilution of the stock solution**

| Volume of initial stock solution<br>ml | Sample oil concentration (after dilution of the stock solution with 50 ml of clean solvent)<br>$C_s$<br>µg/ml |
|---|---|
| 0,1 | 2 |
| 0,5 | 10 |
| 1,0 | 20 |
| 2,0 | 40 |
| 3,5 | 70 |
| 5,0 | 100 |

It should be noted that the resulting concentrations shown in Table C.2 assume that the mass of oil weighed out in preparation of the stock solution is exactly 100 mg. In practice, this is unlikely and resulting concentrations should be calculated based on the actual mass of the oil weighed out above.

For each sample, record an IR spectrum (see Figure C.1) and calculate the absorbance.

The IR absorbance, $A$, a dimensionless ratio, is calculated as given in Formula (C.1):

$$A = \lg\left(\frac{I_0^n}{I_1 \times I_n}\right) \tag{C.1}$$

where

$I_0$ baseline light intensity;

$I_1$ IR transmittance at one characteristic wavenumber;

$n$ number of peaks at which the absorbance has been obtained for a specific oil type.

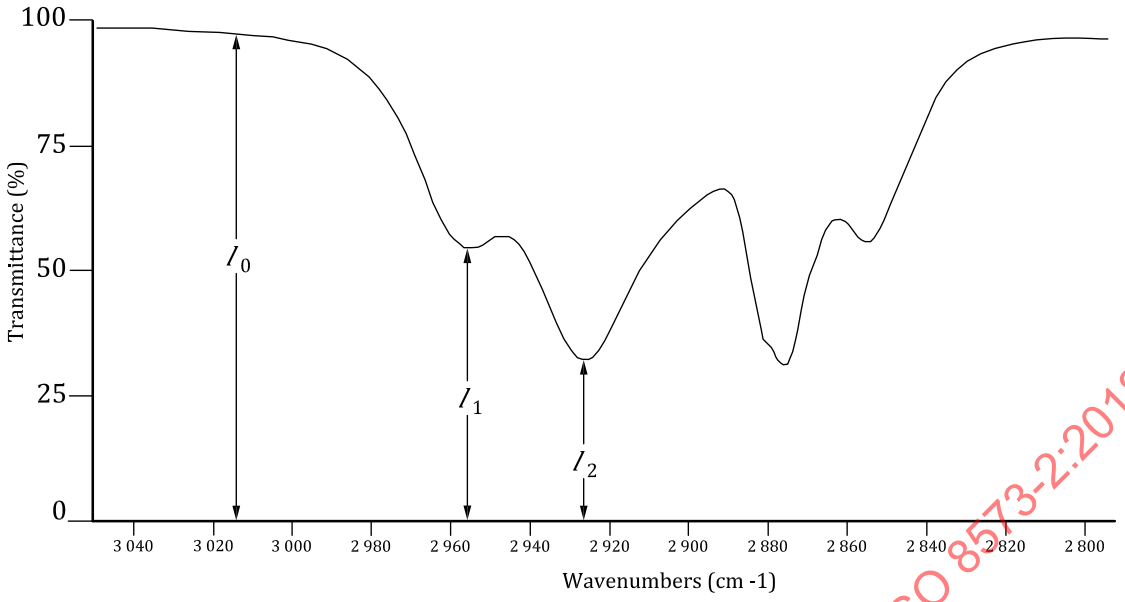



**Figure C.1 — Example transmittance spectra**

Example absorbance data are given in Table C.3.

**Table C.3 — Infrared absorbance example data**

| Volume of stock solution<br>ml | Oil concentration<br>$C_s$<br>µg/ml | Infrared absorbance<br>$A$ |
|---|---|---|
| 0,1 | 1,990 | 0,006 10 |
| 0,5 | 9,950 | 0,030 00 |
| 1,0 | 19,90 | 0,056 00 |
| 2,0 | 39,80 | 0,100 00 |
| 3,5 | 69,65 | 0,190 00 |
| 5,0 | 99,50 | 0,268 00 |

In the above data the original stock solution contained 99,5 mg of oil diluted in to 100 ml of solvent creating a stock solution concentration of oil in solvent of 995 µg/ml.

Using the results obtained, construct a response curve by plotting absorbance against oil concentration, as shown in Figure C.2.

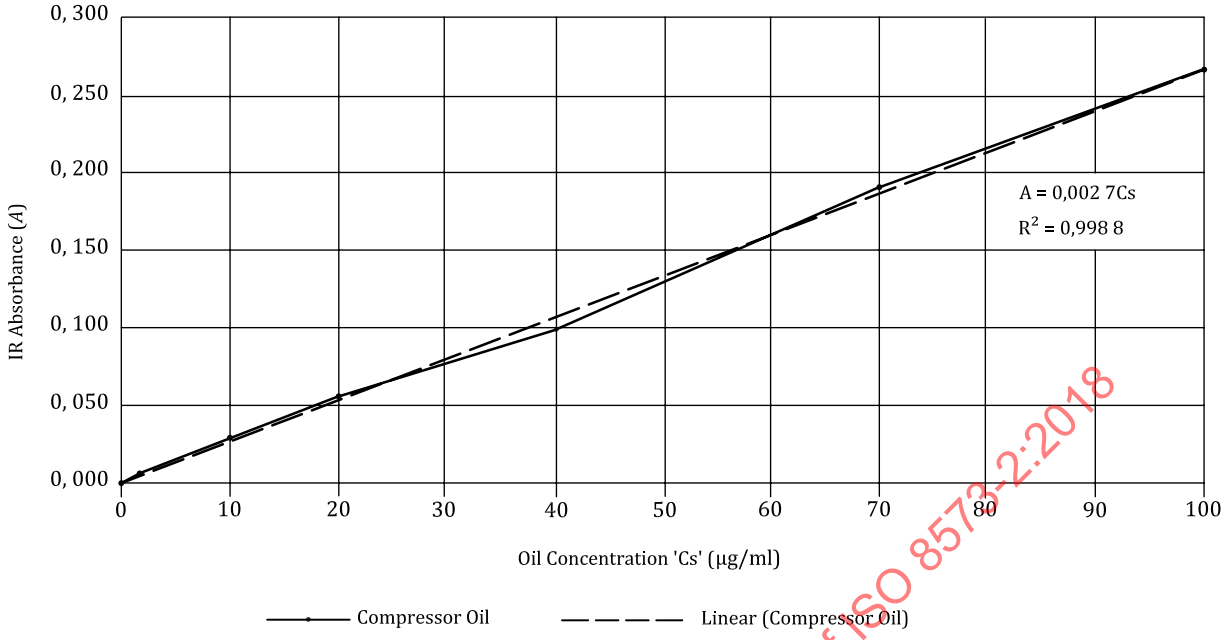



**Figure C.2 — IR Absorbance against variation in oil concentration in solvent**

### C.1.5.2 Instrument response coefficient and linearity

The instrument response and linearity for increasing oil concentration in solvent is determined by performing a least spares regression analysis for a straight line on the collected data that was used to construct the response curve shown in Figure C.2. Regression analysis determines the instrument response coefficient, $C_c$, and coefficient of determination, $R^2$, for which the intercept, $C$, is zero.

$$A = (C_c \times C_s) + C \tag{C.2}$$

where

$A$ absorbance (dimensionless);

$C_c$ represents the instrument response coefficient;

$C_s$ oil in solvent concentration, expressed in micrograms per millilitre;

$C$ intercept at the origin of the plot (normally 0).

In the example curve Figure C.2 the instrument response coefficient. $C_c$, is equal to 0,002 7 and the coefficient of determination, $R^2$, is equal to 0,998 8.

The instrument response curve varies according to the degree of oxidation the oil has undergone. Therefore, when constructing the instrument response curve, oil should be collected from the compressor supplying the compressed air. If it is not practical to collect oil from the compressor, the oil used in the determination of the response curve should be of the same manufacture as the oil in use in the compressor.

The instrument response curve is valid only for the specific oil analysed and the instrumentation and cuvettes used during the test. A separate response curve shall be constructed for each oil type and also for each batch of the same oil and shall not be more than 3 months old.

### C.1.6 Oil recovery coefficient

To establish that the solvent selected has the correct level of solvency for the oil being investigated a sampling disc is dosed with a predetermined mass of oil which is subsequently extracted and the mass calculated by use of the response coefficient, $C_c$, (see C.1.5). The mass extracted is compared directly to the original dosed mass and the recovery coefficient, $O$, calculated.

This method is performed at 10 %, 50 % and 90 % of the maximum allowable mass to be collected on the filter discs when in use and the average oil recovery calculated for the nine samples measured.

#### C.1.6.1 Preparation of dosed sampling discs

Using an analytical balance or other precision weighing instrument with a resolution of no less than 0,01 mg, weigh out 200 mg of the oil under investigation and dilute it to a volume of 20 ml with clean solvent to create a stock solution with an oil concentration of 10 mg/ml.

From this initial stock solution, dose three individual sampling discs with each of the following volumes (see Table C.4) of stock solution to create nine samples and allow the solvent to evaporate. The mass of dosed oil, $O_d$, remaining is then ready for extraction and the IR absorbance determined. Care should be exercised during dosing to prevent loss of the dosed volume of solvent and oil solution to secondary surfaces such as those supporting the sampling discs since this would result in an apparent under recovery of the oil.

**Table C.4 — Dilution of the stock solution**

| Volume of stock solution added to the sampling disc<br>ml | Mass of oil dosed on the sampling disc<br>$O_d$<br>mg |
|---|---|
| 0,03 | 0,3 |
| 0,15 | 1,5 |
| 0,27 | 2,7 |

It should be noted that the resulting concentrations shown in Table C.4 assume that the mass of oil weighed out in preparation of the stock solution is exactly 200 mg. In practice, this is unlikely and resulting concentrations should be calculated based on the actual mass of the oil weighed out when formulating the stock solution.

#### C.1.6.2 Mass of oil by extraction from dosed sampling discs

Each of the oil dosed sampling discs is then extracted with clean solvent and the IR Absorbance measured. The mass of oil extracted "$O_e$" from the sampling disc is then computed from Formula (C.3) as follows:

$$O_e = \frac{A}{C_c} \times V_s \qquad \text{(C.3)}$$

where

$O_e$ mass of oil extracted from the sampling disc;

$A$ absorbance of the solvent oil mixture by IR spectroscopy;

$C_c$ response coefficient for the specific oil under investigation;

$V_s$ volume of solvent (ml) used to extract the oil from the sampling disc.

All nine results are recorded and used to determine the average oil recovery coefficient.

### C.1.6.3 Calculation of oil recovery coefficient

The recovery coefficient, $O$, is calculated from Formula (C.4) for each of the nine sampling discs as follows:

$$O = \frac{O_e}{O_d} \tag{C.4}$$

where

$O$ oil recovery coefficient for a single sampling disc (dimensionless);

$O_e$ recovered mass from the sampling disc by extraction (µg);

$O_d$ dosed oil on the sampling disc before extraction (µg).

Example oil recovery coefficient data are given in Table C.5.

**Table C.5 — Example oil recovery data and calculated oil recovery coefficient**

| Sampling disc reference | Dosed oil $O_d$ mg | Recovered mass $O_e$ mg | Oil recovery coefficient $O$ |
|---|---|---|---|
| 1 | 0,298 5 | 0,334 9 | 1,122 |
| 2 | 0,298 5 | 0,329 8 | 1,105 |
| 3 | 0,298 5 | 0,336 6 | 1,283 |
| 4 | 1,492 5 | 1,508 | 1,010 |
| 5 | 1,492 5 | 1,516 | 1,016 |
| 6 | 1,492 5 | 1,498 | 1,004 |
| 7 | 2,686 5 | 2,692 | 1,002 |
| 8 | 2,686 5 | 2,687 | 1,000 |
| 9 | 2,686 5 | 2,664 | 0,992 |
| | | **Average oil recovery coefficient, $O_a$** | 1,042 |

NOTE In the above example data the original stock solution contained 199,0 mg of oil diluted in to 20 ml of solvent creating a stock solution of 9,950 mg/ml.

## C.1.7 Quality control

### C.1.7.1 General

The linearity of the method can be checked by the degree of fit to a straight line as indicated by the coefficient of determination, $R^2$. In addition, the oil recovery coefficient $O$, checks the effectiveness of the solvent to recover oil from a sampling disc and establishes that the solvent chosen has the correct level of solvency for the oil under investigation.

### C.1.7.2 Linearity

The linearity to a straight line is indicated by the coefficient of determination, $R^2$, this shall be no less than 0,995; otherwise, the analysis shall be repeated. In the example data (see Figure C.2) the value of the coefficient of determination, $R^2$ is 0,998 8 and is thus accepted.

NOTE The $R^2$ value represents the deviations of the data points from a straight line and indicates how well the data fits this line; when multiplied by 100, the $R^2$ value gives a percentage degree of fit.

### C.1.7.3 Sampling disc oil recovery coefficient

The average oil recovery coefficient, $O_a$, is calculated from Formula (C.5) below:

$$O_a = \frac{1}{n} \times \sum_{i=1}^{n} O \tag{C.5}$$

where

$O_a$ calculated average oil recovery coefficient;

$N$ number of tests performed (in this case, nine);

$O$ individual values of the oil recovery coefficient determined for each test performed.

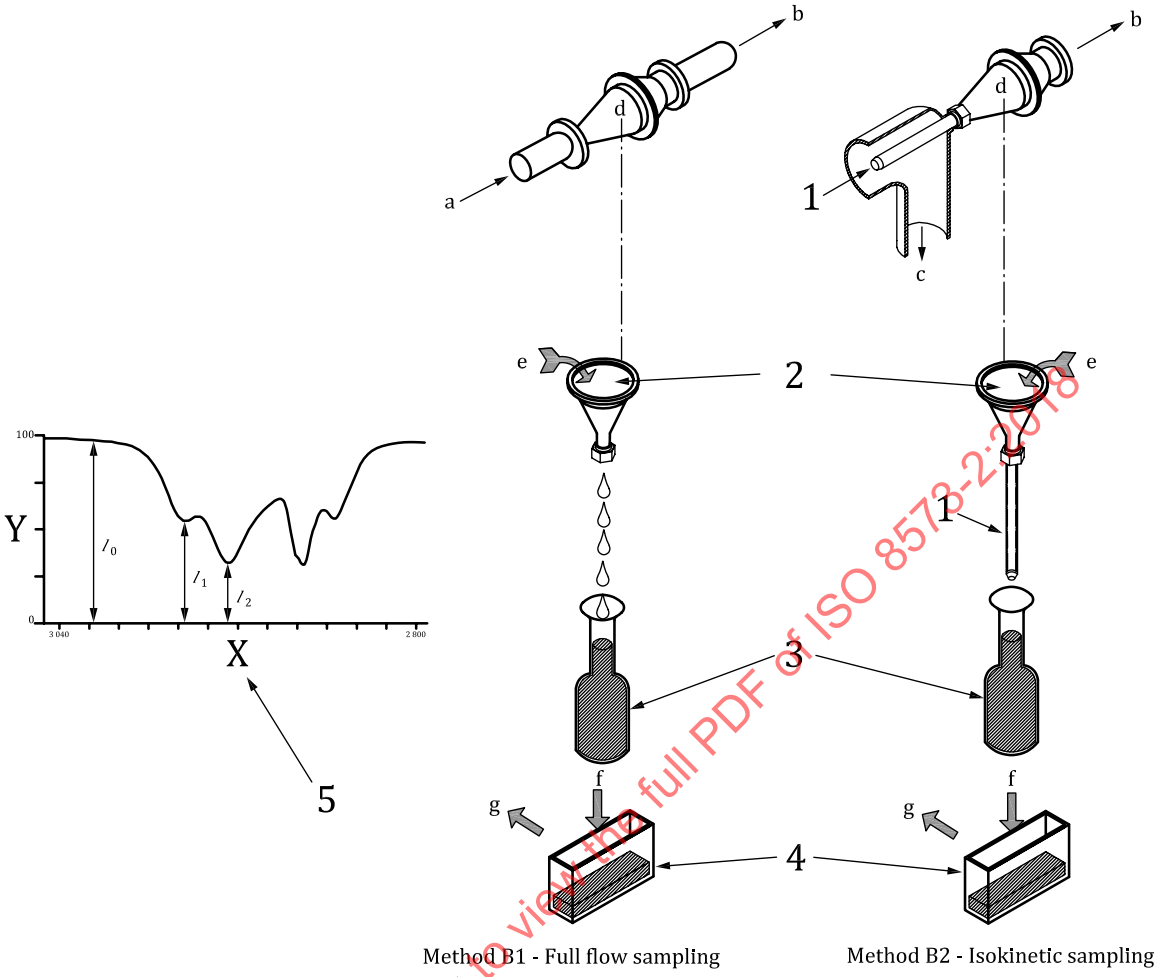
This method is performed 3 times at each of 10 %, 50 % and 90 % of the maximum allowable mass to be collected on the sampling discs.

The results from all nine dosed sampling discs shall not deviate by more than 1 ± 0,15. Wherein, if the extracted oil from the sampling discs were to exactly equal the amount of oil with which the discs were dosed, then the recovery coefficient would be equal to 1.

In the example data of Table C.5 the average oil recovery coefficient, $O_a$, is calculated as being 1,042 representing a 4,2 % over recovery from the dosed sampling discs, which remains within the allowed tolerance and is therefore accepted.

## C.1.8 Oil collection apparatus analysis procedure

Extract the oil from the collection apparatus, including sampling discs, sample disc chamber and isokinetic sample probe (if present), by adding a known amount of solvent to the surface of the sampling discs and allowing it to drain back through the upstream half of the sample disc holder and through the probe (if present). Record the spectrum from 3 400 $cm^{-1}$ to 2 500 $cm^{-1}$. The analysis procedure is shown schematically in Figure C.3. The component parts of the oil collection apparatus can be analysed separately but the individual amounts shall be summated to determine the total mass of oil collected in the apparatus. To ensure that all of the sample is extracted by the solvent, pass the solvent through the sampling apparatus a minimum of 3 times.




**Key**

| | | | |
|---|---|---|---|
| 1 | isokinetic probe | a | From control valve. |
| 2 | sampling discs | b | To control valve and flow meter. |
| 3 | oil plus solvent | c | To main flow meter. |
| 4 | infrared spectrometer cuvette | d | Remove sample disc holder, sampling disc and isokinetic probe if present. |
| 5 | typical infrared spectrum | e | Add solvent; see C.1.3 and C.1.8. |
| | x-axis wave number, expressed in reciprocal centimetre | f | Fill cuvette with sample. |
| | y-axis transmittance, expressed in percent | g | To infrared spectrometer. |
| | $I_0$, $I_1$, and $I_2$ infrared spectrum peak height | | |

**Figure C.3 — Schematic representation of the analysis of the oil collection apparatus**



## C.1.9 Calculation of oil mass in the oil collection apparatus

Once the IR transmittance spectrograph of the oil in solvent has been obtained the absorbance *"A"* of the spectrograph is calculated using Formula (C.1) for the same individual peaks used during the process detailed in C.1.5.1.

After determining the absorbance of the spectrograph the total oil mass, *m*, (mg) on the oil collection apparatus can be determined using Formula (C.6)

$$m = \frac{A}{1\,000 \times C_c} \times V_s \tag{C.6}$$

where

$m$ mass of oil extracted from the oil collection apparatus (mg);

$A$ absorbance of the solvent oil mixture by IR spectroscopy;

$C_c$ instrument response coefficient for the specific oil under investigation;

$V_s$ total volume of solvent (ml) used to extract the oil from the oil collection apparatus.

In instances where individual components of the oil collection apparatus have been extracted with solvent separately the mass of oil determined for each component shall be summated.

The oil aerosol concentration measured in the compressed air can then be calculated by substituting "*m*" in to Formula 2.

### C.1.10 Limit of detection

The limit of detection shall be determined in accordance with DIN 32645.

# Annex D
(informative)

# Example GC FID analysis and calculation of test results for Method B

## D.1 Analytical procedure

### D.1.1 General

The following text describes an example procedure based on gas chromatography fitted with flame ionization detection (GC-FID) that has been proven to be suitable for the determination of the mass of oil, *m*, expressed in milligrams collected by the sampling apparatus.

Calibration of the GC-FID is performed to determine the instrument response and linearity thus establishing the operational range over which the measurements are made. In addition, the degree of recovery of oil from the sampling disc using solvent is also determined. These parameters shall meet the minimum levels required by the document.

Once the response, linearity and the oil recovery coefficients have been established the amount of oil collected on the sampling apparatus can be analysed and the concentration of oil in compressed air calculated.

### D.1.2 Principle

The oil collected on the sampling apparatus is dissolved in a suitable solvent (preferred is *n*-hexane) and the amount determined by gas chromatography with flame ionization detector (GC-FID).

The concentration of oil in solution is proportional to the area under the spectrum between the limits of *n*-decane ($C_{10}$, boiling point = 175 °C) and *n*-tetracontane ($C_{40}$, boiling point 525 °C).

This area is compared with oil standard solutions (synthesized with reference oil) in the range of 0,002 mg/ml and 0,3 mg/ml and the amount of oil is calculated with regard to the regression graph. For higher concentrations the validity of the response curve shall be tested with a suitable standard. If the measured amount differs by more than 10 % of the expected amount, a new response curve with higher standard concentrations shall be performed.

### D.1.3 Solvent

For the purpose of oil extraction from the sampling discs and holder, a solvent shall be chosen that has a good solvency for the oil in question. The reagents should be handled with care and in accordance with manufacturer or supplier instructions. The solvents chosen should be specially prepared for use in gas chromatography (e.g. for residue analysis) and shall not interfere with the marker $C_{10}$.

The preferred solvent is *n*-hexane because of its available purity and good manageability. Other solvents, such as *n*-pentane or *n*-heptane are also suitable, however certain oil types may not be soluble in *n*-hexane, in which case it is suggested that acetone is used. An example of the typical purity of *n*-hexane is given in Table D.1 for guidance.

**Table D.1 — Example of typical purity of *n*-hexane**

| Designation | Specification |
|---|---|
| Molecular formula: | $CH_3(CH_2)4CH_3$ |
| Gas chromatography FID Grade | ≥98 % purity |
| CAS number: | 110–54–3 |
| Molecular weight: | 86,18 g/mol |
| Residual water: | ≤0,01 % |
| Residue on Evaporation: | ≤3,0 mg/l |

### D.1.4 Apparatus

#### D.1.4.1 Glassware

Glassware used for determination of volume shall be Class A to ISO 1042, borosilicate glass with a glass stopper and supplied with a batch certificate. It shall be cleaned before use and checked to ensure that it does not contribute any hydrocarbons during the analysis process.

#### D.1.4.2 GC-FID typical operating conditions

Analyser: Gas chromatography with flame ionization detector (GC-FID):
Trace GC Ultra

Carrier gas: Helium, const. flow 1,2 ml/min

Column: MEGA SE-52-HT
Film thickness $d_f$ = 0,25 µm;
Column length $l$ = 15 m;
Column internal diameter (mm) $d_i$ = 0,32 mm
Pre-column [IP deactivated guard column by (2 m × 0,53 mm)]

PTV-Injector: Splitless
80 °C isothermal for 0,05 min
10 °C/sec. to 320 °C
320°C isothermal for 10 min

Injection volume: 5 µl

Temperature program: 50 °C isothermal for 2,5 min
25 °C/min to 100 °C
50 °C/min to 330 °C
330 °C isothermal for 4,3 min

Detector temperature: 360 °C

#### D.1.4.3 Sampling disc preparation

The sampling discs shall be prepared before they can be used. The sampling discs are heated at 800 °C in an oven for approximatively 1 h to remove all hydrocarbons.

#### D.1.4.4 Blank sample preparation

A blank sample is prepared by extracting a clean 3 layered sampling disc and determining the area of the peaks in the chromatogram between $C_{10}$ and $C_{40}$.

### D.1.5 Instrument response, linearity and recovery coefficients

The GC-FID instrument response, linearity and oil recovery coefficients shall be determined as follows.

#### D.1.5.1 Preparation of the instrument response graph

Using an analytical balance or other precision weighing instrument with resolution of no less than 0,01 mg, weigh out approximately 0,5 g of the oil under investigation and dilute it to a volume of 25 ml with clean solvent to give a stock solution with an oil concentration of 20 mg/ml. It should be noted that the resulting concentrations shown in Table D.2 assume that the mass of oil weighed out in preparation of the stock solution is exactly 0,5 g. In practice, this is unlikely and resulting concentrations should be calculated based on the actual mass of the oil weighed out above. From this initial stock solution, prepare at least 10 samples that span the expected range of oil concentration. To prepare the samples, different volumes of the initial stock solution are diluted with clean solvent and internal marker (0,2 ml $C_{10}/C_{40}$) to 10 ml. This results in oil concentrations between 0,002 mg/ml and 0,3 mg/ml (see example Table D.2). The solvent that is intended to be used for extraction and analysis, also called a blank sample (i.e. only the solvent), is also analysed.

**Table D.2 — Preparing standard solutions in the range 0,002 and 0,311 mg/ml**

| Volume of stock solution<br>µl | Oil mass<br>µg | Added volume of $C_{10}/C_{40}$<br>µl | Added volume of hexane<br>ml | Resulting standard solution<br>mg/ml |
|---|---|---|---|---|
| 1 | 20 | 200 | 9,799 | 0,002 |
| 2 | 40 | 200 | 9,798 | 0,004 |
| 3 | 60 | 200 | 9,797 | 0,006 |
| 4 | 80 | 200 | 9,796 | 0,008 |
| 5 | 100 | 200 | 9,795 | 0,010 |
| 6 | 120 | 200 | 9,794 | 0,012 |
| 7 | 140 | 200 | 9,793 | 0,014 |
| 8 | 160 | 200 | 9,792 | 0,016 |
| 9 | 180 | 200 | 9,791 | 0,018 |
| 10 | 200 | 200 | 9,790 | 0,020 |
| 20 | 400 | 200 | 9,780 | 0,041 |
| 30 | 600 | 200 | 9,770 | 0,061 |
| 40 | 800 | 200 | 9,760 | 0,082 |
| 50 | 1 000 | 200 | 9,750 | 0,103 |
| 60 | 1 200 | 200 | 9,740 | 0,123 |
| 75 | 1 500 | 200 | 9,725 | 0,154 |
| 100 | 2 000 | 200 | 9,700 | 0,206 |
| 125 | 2 500 | 200 | 9,675 | 0,258 |
| 150 | 3 000 | 200 | 9,650 | 0,311 |

For each sample, record a GC-FID chromatograph and determine the total peak area by integration. An example chromatograph is shown in Figure D.1.