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**Heavy-duty engines — Measurement of  
gaseous emissions from raw exhaust gas  
and of particulate emissions using partial  
flow dilution systems under transient test  
conditions**

*Moteurs de poids lourds — Détermination, sur cycle transitoire, des  
émissions de polluants gazeux par mesure des concentrations dans les  
gaz d'échappement bruts et des émissions de particules en utilisant un  
système de dilution partielle*



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## 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.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 3.

The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

Attention is drawn to the possibility that some of the elements of this International Standard may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 16183 was prepared by Technical Committee ISO/TC 22, *Road vehicles*, Subcommittee SC 5, *Engine tests*.

Annexes A, B and C form a normative part of this International Standard. Annex D is for information only.

## Introduction

Today's emission measurement systems depend on the type of test cycle — steady-state or transient — and the type of pollutant to be measured.

In a steady-state cycle, the mass of gaseous emissions is calculated from the concentration in the raw exhaust gas and the exhaust flow of the engine, which can easily be determined. For particulate matter (PM), partial-flow dilution systems, in which only a portion of the exhaust gas is diluted, are widely used.

In a transient cycle, real time exhaust flow determination is more difficult. Therefore, the constant volume sampling (CVS) principle has been used for many years because exhaust mass flow measurement is not required with this system. The total exhaust gas is diluted, the total flow as the sum of dilution air and exhaust gas volume is kept virtually constant, and the emissions (both gaseous and PM) are measured in the diluted exhaust gas. The space and cost requirements of such a system are considerably higher than for the partial-flow dilution systems used in steady-state cycles. Nevertheless, raw exhaust measurement and partial flow systems can only be applied to transients if sophisticated control systems and calculation algorithms are used.

The mass emission determination in a raw exhaust sample and the measurement of the exhaust gas mass flow rate is a state-of-the-art procedure for light duty vehicle development on chassis dynamometers. There it is called modal analysis. However, it is usually done in conjunction with the mass emission evaluation on a full-flow CVS with bag analysis, where quality of the modal results can easily be verified by comparison with the CVS bag results. For heavy-duty engines, the CVS system is a large and costly system.

The aim of this International Standard is to provide an optional, stand-alone measurement procedure. By the nature of the transient mass emission calculation, small changes could result in large deviations of the final results, for example, by a wrongly performed time alignment caused by a wrong response time determination or by a system fault resulting in a change of the response time behaviour of the system. Therefore, the quality assurance procedure of a carbon dioxide-based carbon balance check, in line with highly sophisticated verification procedures for the partial flow particulate measurement, have been established in this International Standard.

NOTE CVS systems are covered in detail in various exhaust emissions regulations for both light- and heavy-duty vehicles as well as by ISO 8178-1. They are therefore not included in this International Standard. Since they are considered to be the reference systems for exhaust emission measurement on transient cycles, extensive studies have been commissioned by ISO/TC 22/SC 5/WG 2 on the correlation between CVS systems and the systems covered by this International Standard, with the results having been taken into consideration in its development.



# Heavy-duty engines — Measurement of gaseous emissions from raw exhaust gas and of particulate emissions using partial flow dilution systems under transient test conditions

## 1 Scope

This International Standard specifies methods for the measurement and evaluation of gaseous and particulate exhaust emissions from heavy-duty engines under transient conditions on a test bed. The procedures it defines can be applied to any transient test cycle that does not require extreme system response times; it can therefore be used as an option to the regulated measurement equipment of certification test cycles — usually CVS-type systems — with the approval of the certification agency [among certification test cycles in place are the European transient cycle (ETC) and the US heavy-duty transient cycle (FTP)].

This International Standard is applicable to heavy-duty engines for commercial vehicles primarily designed for road use, but can also be applied to passenger car engines and to engines used for non-road applications. The test equipment specified in this International Standard can also be used in steady-state test cycles, however, if so, the calculation procedures will need to be replaced by those applicable to the particular test cycle.

## 2 Normative references

The following normative documents contain provisions which, through reference in this text, constitute provisions of this International Standard. For dated references, subsequent amendments to, or revisions of, any of these publications do not apply. However, parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the normative documents indicated below. For undated references, the latest edition of the normative document referred to applies. Members of ISO and IEC maintain registers of currently valid International Standards.

ISO 5167-1, *Measurement of fluid flow by means of pressure differential devices — Part 1: Orifice plates, nozzles and Venturi tubes inserted in circular cross-section conduits running full*

ISO 5725-2, *Accuracy (trueness and precision) of measurement methods and results — Part 2: Basic method for the determination of repeatability and reproducibility of a standard measurement method*

ISO 8178-5:1998, *Reciprocating internal combustion engines — Exhaust emission measurement — Part 5: Test fuels*

SAE paper 770141, *Optimization of Flame Ionization Detector for the Determination of Hydrocarbons in Diluted Automobile Exhaust*, Glenn D. Reschke

SAE J 1936:1989, *Chemical methods for the measurement of non-regulated diesel emissions*

SAE J 1937:1995, *Engine testing with low-temperature charge air-cooler systems in a dynamometer test cell*

## 3 Terms, definitions, symbols and abbreviations

For the purposes of this International Standard, the following terms and definitions, and symbols and abbreviations (see Table 1), apply.

**3.1**

**particulate matter**

**PM**

any material collected on a specified filter medium after diluting exhaust with clean filtered air to a temperature of  $\leq 325$  K (52 °C), as measured at a point immediately upstream of the filter; it is primarily carbon, condensed hydrocarbons, and sulfates with associated water

NOTE Regulatory agencies choosing to use ISO 16183 could adapt this definition to their particular needs. For example, US regulations after 2007 will define particulate matter at a temperature greater than 42 °C and less than 52 °C.

**3.2**

**gaseous pollutant**

gas considered to be polluting to the atmosphere: carbon monoxide, hydrocarbons or non-methane hydrocarbons, or both these, oxides of nitrogen [expressed in nitrogen dioxide (NO<sub>2</sub>) equivalent], formaldehyde and methanol

**3.3**

**partial-flow dilution method**

process of separating a part of the raw exhaust from the total exhaust flow, then mixing it with an appropriate amount of dilution air prior to the particulate sampling filter

**3.4**

**full-flow dilution method**

process of mixing dilution air with the total exhaust flow prior to separating a fraction of the diluted exhaust stream for analysis

NOTE It is common in many full flow dilution systems to dilute this fraction of pre-diluted exhaust a second time to obtain appropriate sample temperatures at the particulate filter.

**3.5**

**specific emission**

mass emission expressed in grams per kilowatt hour

**3.6**

**steady-state test cycle**

test cycle comprising a sequence of engine test modes in which the engine is given sufficient time to achieve defined speed, torque and stability criteria at each mode

**3.7**

**transient test cycle**

test cycle comprising a sequence of normalized speed and torque values that vary relatively quickly with time

**3.8**

**response time**

difference in time between a rapid change of the component to be measured at the reference point and the appropriate change in the response of the measuring system, whereby the change of the measured component is at least 60 % FS (full scale) and takes place within less than 0,1 s

See Figure 1.

NOTE 1 The system response time,  $t_{90}$ , consists of the delay time to the system and of the rise time of the system.

NOTE 2 The response time can vary, depending on where the reference point for the change of the component to be measured is defined: either at the sampling probe or directly at the port entrance of the analyser. For the purposes of this International Standard, the sampling probe is defined as the reference point.

**3.9**

**delay time**

time between the change of the component to be measured at the reference point and a system response of 10 % of the final reading,  $t_{10}$

See Figure 1.

NOTE 1 For the gaseous components, this is basically the transport time of the measured component from the sampling probe to the detector.

NOTE 2 The delay time can vary, depending on where the reference point for the change of the component to be measured is defined: either at the sampling probe or directly at the port entrance of the analyser. For the purposes of this International Standard, the sampling probe is defined as the reference point.

### 3.10

#### rise time

time between the 10 % and 90 % response of the final reading ( $t_{90} - t_{10}$ )

See Figure 1.

NOTE 1 This is the instrument response after the component to be measured has reached the instrument.

NOTE 2 The rise time can vary, depending on where the reference point for the change of the component to be measured is defined: either at the sampling probe or directly at the port entrance of the analyser. For the purposes of this International Standard, the sampling probe is defined as the reference point.

### 3.11

#### transformation time

time between the change of the component to be measured at the reference point and a system response of 50 % of the final reading,  $t_{50}$

See Figure 1.

NOTE 1 The transformation time is used for the signal alignment of different measurement instruments.

NOTE 2 The transformation time can vary, depending on where the reference point for the change of the component to be measured is defined: either at the sampling probe or directly at the port entrance of the analyser. For the purposes of this International Standard, the sampling probe is defined as the reference point.

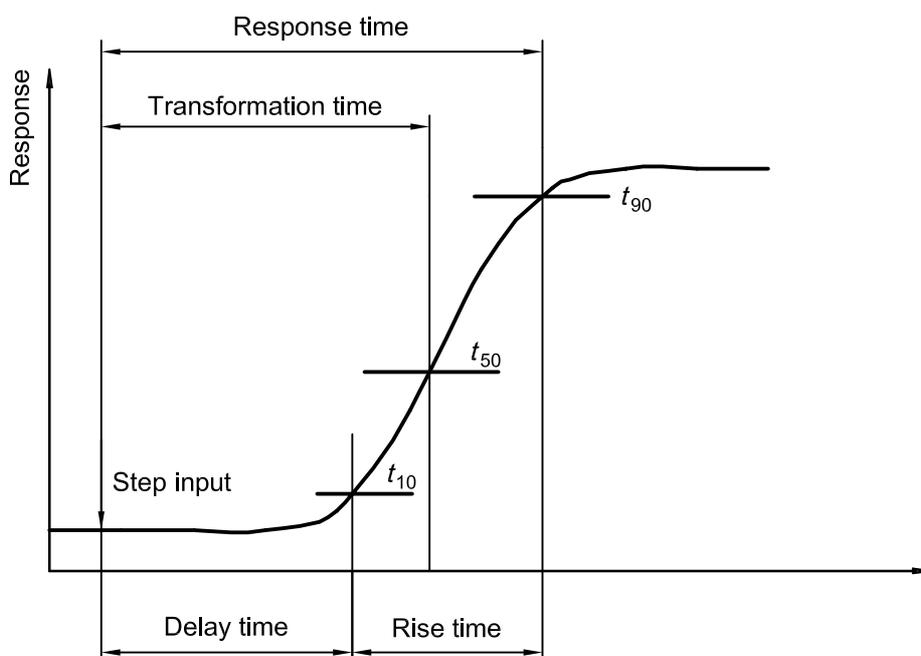


Figure 1 — Definitions of system response

Table 1 — General symbols and abbreviations used in this International Standard

| Symbol/Abbreviation | Unit                                   | Meaning  |
|---------------------|--|--|
| $A/F_{st}$          | —                                      | Stoichiometric air-to-fuel ratio                                       |
| $c$                 | ppm <sup>a</sup> (µl/l) or % by volume | Concentration  |
| $C_c$               | —                                      | Slip factor  |
| $d_e$               | m                                      | Exhaust pipe diameter  |
| $d_p$               | m                                      | Sampling probe diameter  |
| $d_{PM}$            | m                                      | Particle diameter  |
| $f$                 | Hz                                     | Data sampling rate   |
| $f_a$               | —                                      | Laboratory atmospheric factor  |
| $E_{CO_2}$          | %                                      | CO <sub>2</sub> quench of NO <sub>x</sub> analyser                     |
| $E_E$               | %                                      | Ethane efficiency  |
| $E_{H_2O}$          | %                                      | Water quench of NO <sub>x</sub> analyser                               |
| $E_M$               | %                                      | Methane efficiency   |
| $E_{NO_x}$          | %                                      | Efficiency of NO <sub>x</sub> converter                                |
| $\eta$              | Pa·s                                   | Dynamic viscosity of exhaust gas                                       |
| $H_a$               | g/kg                                   | Absolute humidity of the intake air                                    |
| $i$                 | —                                      | Subscript denoting an instantaneous measurement (e.g. 1 Hz)            |
| $k_f$               | —                                      | Fuel specific factor   |
| $k_{h,D}$           | —                                      | Humidity correction factor for NO <sub>x</sub> for CI engines          |
| $k_{h,G}$           | —                                      | Humidity correction factor for NO <sub>x</sub> for SI engines          |
| $k_w$               | —                                      | Dry to wet correction factor for the raw exhaust gas                   |
| $\lambda$           | —                                      | Excess air ratio   |
| $m_{edf}$           | kg                                     | Mass of equivalent diluted exhaust gas over the cycle                  |
| $m_f$               | mg                                     | Particulate sample mass collected                                      |
| $m_{gas}$           | g                                      | Mass of gaseous emissions (over the test cycle)                        |
| $m_{PM}$            | g                                      | Mass of particulate emissions (over the test cycle)                    |
| $m_{se}$            | kg                                     | Exhaust sample mass over the cycle                                     |
| $m_{sed}$           | kg                                     | Mass of diluted exhaust gas passing the dilution tunnel                |
| $m_{sep}$           | kg                                     | Mass of diluted exhaust gas passing the particulate collection filters |
| $M_{gas}$           | g/kWh                                  | Specific emission of gaseous emissions                                 |
| $M_{PM}$            | g/kWh                                  | Specific emission of particulate emissions                             |
| $M_{r,e}$           | —                                      | Molecular mass <sup>b</sup> of exhaust                                 |
| $M_{r,gas}$         | —                                      | Molecular mass of exhaust component                                    |

Table 1 (continued)

| Symbol/Abbreviation | Unit                 | Meaning  |
|---------------------|----------------------|--|
| $n$                 | —                    | Number of measurements                                       |
| $p_a$               | kPa                  | Saturation vapour pressure of the engine intake air          |
| $p_b$               | kPa                  | Total atmospheric pressure                                   |
| $p_r$               | kPa                  | Water vapour pressure after cooling bath                     |
| $p_s$               | kPa                  | Dry atmospheric pressure                                     |
| $P$                 | —                    | Particle penetration   |
| $q_{mad}$           | kg/s                 | Intake air mass flow rate on dry basis                       |
| $q_{maw}$           | kg/s                 | Intake air mass flow rate on wet basis                       |
| $q_{mCe}$           | kg/s                 | Carbon mass flow rate in the raw exhaust gas                 |
| $q_{mCf}$           | kg/s                 | Carbon mass flow rate into the engine                        |
| $q_{mCp}$           | kg/s                 | Carbon mass flow rate in the partial-flow dilution system    |
| $q_{mdew}$          | kg/s                 | Diluted exhaust gas mass flow rate on wet basis              |
| $q_{mdw}$           | kg/s                 | Dilution air mass flow rate on wet basis                     |
| $q_{medf}$          | kg/s                 | Equivalent diluted exhaust gas mass flow rate on wet basis   |
| $q_{mew}$           | kg/s                 | Exhaust gas mass flow rate on wet basis                      |
| $q_{mex}$           | kg/s                 | Sample mass flow rate extracted from dilution tunnel         |
| $q_{mf}$            | kg/s                 | Fuel mass flow rate  |
| $q_{mp}$            | kg/s                 | Sample flow of exhaust gas into partial-flow dilution system |
| $q_{vs}$            | l/min                | System flow rate of exhaust analyser system                  |
| $q_{vt}$            | cm <sup>3</sup> /min | Tracer gas flow rate   |
| $r_d$               | —                    | Dilution ratio   |
| $r_h$               | —                    | Hydrocarbon response factor of the FID                       |
| $r_m$               | —                    | Methanol response factor of the FID                          |
| $r_s$               | —                    | Average sample ratio   |
| $\rho_e$            | kg/m <sup>3</sup>    | Exhaust gas density (wet, at 273 K and 101,3 kPa)            |
| $\rho_{gas}$        | kg/m <sup>3</sup>    | Density of exhaust component (at 273 K and 101,3 kPa)        |
| $\rho_{PM}$         | kg/m <sup>3</sup>    | Particle density (at 273 K and 101 kPa)                      |
| $\sigma$            |                      | Standard deviation   |
| $T$                 | K                    | Absolute temperature   |
| $T_a$               | K                    | Absolute temperature of the intake air                       |
| $T_e$               | K                    | Exhaust gas temperature                                      |
| $t_{10}$            | s                    | Time between step input and 10 % of final reading            |
| $t_{50}$            | s                    | Time between step input and 50 % of final reading            |
| $t_{90}$            | s                    | Time between step input and 90 % of final reading            |
| $\tau$              | s                    | Particle relaxation time                                     |
| $u$                 | —                    | Ratio between densities of gas component and exhaust gas     |
| $V_s$               | l                    | Total volume of exhaust analyser system                      |

Table 1 (continued)

| Symbol/Abbreviation  | Unit | Meaning  |
|--|------|--|
| $W_{act}$  | kWh  | Actual cycle work of the respective test cycle |
| $v_e$  | m/s  | Gas velocity in the exhaust pipe               |
| $v_p$  | m/s  | Gas velocity in the sampling probe             |
| <b>Symbols specific to fuel composition</b>                            |      |  |
| $w_{ALF}$  | —    | Hydrogen content of fuel, % by mass            |
| $w_{BET}$  | —    | Carbon content of fuel, % by mass              |
| $w_{GAM}$  | —    | Sulfur content of fuel, % by mass              |
| $w_{DEL}$  | —    | Nitrogen content of fuel, % by mass            |
| $w_{EPS}$  | —    | Oxygen content of fuel, % by mass              |
| $\alpha$   | —    | Molar hydrogen ratio (H/C)                     |
| $\beta$  | —    | Molar carbon ratio (C/C)                       |
| $\gamma$   | —    | Molar sulfur ratio (S/C)                       |
| $\delta$   | —    | Molar nitrogen ratio (N/C)                     |
| $\varepsilon$  | —    | Molar oxygen ratio (O/C)                       |
| Referring to a fuel $C_\beta H_\alpha O_\varepsilon N_\delta S_\gamma$ |      |  |
| <b>Symbols and abbreviations for chemical components</b>               |      |  |
| ACN  | —    | Acetonitrile                                   |
| C1   | —    | Carbon 1 equivalent hydrocarbon                |
| CH <sub>4</sub>  | —    | Methane  |
| CH <sub>3</sub> OH   | —    | Methanol                                       |
| C <sub>2</sub> H <sub>6</sub>  | —    | Ethane   |
| C <sub>3</sub> H <sub>8</sub>  | —    | Propane  |
| CO   | —    | Carbon monoxide                                |
| CO <sub>2</sub>  | —    | Carbon dioxide                                 |
| DNPH   | —    | Dinitrophenyl hydrazine                        |
| DOP  | —    | Di-octylphtalate                               |
| HC   | —    | Hydrocarbons                                   |
| HCHO   | —    | Formaldehyde                                   |
| H <sub>2</sub> O   | —    | Water  |
| NMHC   | —    | Non-methane hydrocarbons                       |
| NO <sub>x</sub>  | —    | Oxides of nitrogen                             |
| NO   | —    | Nitric oxide                                   |
| NO <sub>2</sub>  | —    | Nitrogen dioxide                               |
| PM   | —    | Particulate matter                             |
| RME  | —    | Rapeseed oil methyl ester                      |

Table 1 (continued)

| Symbol/Abbreviation  | Unit | Meaning                               |
|--|------|---------------------------------------|
| <b>Other abbreviations</b>   |      |                                       |
| CLD  | —    | Chemiluminescent detector             |
| FID  | —    | Flame ionization detector             |
| FTIR   | —    | Fourier transform infrared (analyser) |
| GC   | —    | Gas chromatograph                     |
| HCLD   | —    | Heated chemiluminescent detector      |
| HFID   | —    | Heated flame ionization detector      |
| HPLC   | —    | High pressure liquid chromatograph    |
| NDIR   | —    | Non-dispersive infrared (analyser)    |
| NMC  | —    | Non-methane cutter                    |
| % FS   | —    | Percentage of full scale              |
| SIMS   | —    | Soft ionization mass spectrometer     |
| <i>St</i>  | —    | Stokes number                         |
| <p><sup>a</sup> "Parts per million (ppm)" is a deprecated unit, i.e. not accepted by the International System of Units, SI. It is used exceptionally in this International Standard, immediately followed by the SI unit of equivalent value in parentheses, in order to correspond to other, closely related and already published standards. The accepted SI form for the expression of a volume fraction is in units of microlitres per litre (µl/l), or, alternatively, as 10<sup>-6</sup> or as a percentage by volume (% by volume); for mass fractions it is expressed in micrograms per gram (µg/g). See ISO 31-0:1992, 2.3.3, and ISO 31-8-15:1992.</p> <p><sup>b</sup> Formerly called molecular weight.</p> |      |                                       |

## 4 Test conditions

### 4.1 Engine test conditions

#### 4.1.1 Test condition parameter

The absolute temperature ( $T_a$ ) of the engine air at the inlet to the engine, expressed in kelvin, and the dry atmospheric pressure ( $p_s$ ), expressed in kilopascals, shall be measured, and the parameter  $f_a$  determined in accordance with the following provisions. In multi-cylinder engines having distinct groups of intake manifolds, such as in a "vee" engine configuration, the average temperature of the distinct groups shall be taken.

a) For compression-ignition engines:

— Naturally aspirated and mechanically supercharged engines

$$f_a = \left( \frac{99}{p_s} \right) \times \left( \frac{T_a}{298} \right)^{0,7} \quad (1)$$

— Turbocharged engines, with or without cooling of the intake air

$$f_a = \left( \frac{99}{p_s} \right)^{0,7} \times \left( \frac{T_a}{298} \right)^{1,5} \quad (2)$$

b) For spark-ignition engines:

$$f_a = \left( \frac{99}{p_s} \right)^{1,2} \times \left( \frac{T_a}{298} \right)^{0,6} \quad (3)$$

#### 4.1.2 Test validity

For a test to be recognized as valid,  $f_a$  shall be such that  $0,96 \leq f_a \leq 1,06$ .

### 4.2 Engines with charge air cooling

The charge air temperature shall be recorded and be within  $\pm 5$  K of the maximum charge air temperature specified by the manufacturer at the speed of the declared maximum power and full load. The temperature of the cooling medium shall be at least 293 K (20 °C).

If a test shop system or external blower is used, the charge air temperature shall be set to within  $\pm 5$  K of the maximum charge air temperature specified by the manufacturer at the speed of the declared maximum power and full load. Coolant temperature and coolant flow rate of the charge air cooler at the above set point shall not be changed for the whole test cycle. The charge air cooler volume shall be based upon good engineering practice and typical vehicle/machinery applications.

Optionally, the setting of the charge air cooler may be done in accordance with SAE J 1937.

### 4.3 Power

The basis of specific emissions measurement is uncorrected net or gross power, depending on the regulation.

Certain auxiliaries necessary only for the operation of the vehicle that can be mounted on the engine should be removed for the test.

**EXAMPLE** Air compressor for brakes, power steering compressor, air conditioning compressor or pumps for hydraulic actuators.

Where such auxiliaries have not been removed, the power absorbed by them shall be determined in order to adjust the set values and calculate the work produced by the engine over the test cycle.

### 4.4 Engine air intake system

An engine air intake system shall be used presenting an air intake restriction within  $\pm 300$  Pa of the value specified by the engine manufacturer for a clean air cleaner and in accordance with the respective regulation.

### 4.5 Engine exhaust system

A vehicle exhaust system or a test shop system shall be used presenting an exhaust backpressure within  $\pm 650$  Pa of the value specified by the engine manufacturer and in accordance with the respective regulation. The exhaust system shall conform to the requirements for exhaust gas sampling in 5.6.2 and 7.2.3, EP.

If the engine is equipped with an exhaust after-treatment device, the exhaust pipe shall have the same diameter as found in-use for at least four pipe diameters upstream to the inlet of the beginning of the expansion section containing the after-treatment device. The distance from the exhaust manifold flange or turbocharger outlet to the exhaust after-treatment device shall be the same as in the vehicle configuration or within the distance specifications of the manufacturer. The exhaust backpressure or restriction shall follow these same criteria, and may be set with a valve. The after-treatment container may be removed during dummy tests and during engine mapping and replaced with an equivalent container having an inactive catalyst support.

### 4.6 Cooling system

An engine cooling system with sufficient capacity to maintain the engine at normal operating temperatures prescribed by the manufacturer shall be used.

## 4.7 Lubricating oil

The lubricating oil shall be as specified by the manufacturer; the specifications of the lubricating oil used for the test shall be recorded and presented with the results of the test.

## 4.8 Test fuel

Fuel characteristics influence the engine exhaust gas emission. Therefore, the characteristics of the fuel used for the test should be determined, recorded and declared with the results of the test. Where fuels designated as reference fuels are used, the reference code and the analysis of the fuel shall be provided. For all other fuels, the characteristics to be recorded shall be those listed in the appropriate universal data sheet of ISO 8178-5.

The fuel temperature shall be in accordance with the manufacturer's recommendations.

# 5 Determination of gaseous and particulate components

## 5.1 General

For the purpose of this International Standard, the gaseous components are measured in the raw exhaust gas on a real time basis, and the particulates are determined using a partial-flow dilution system.

The instantaneous concentration signals of the gaseous components are used for the calculation of the mass emissions by multiplication with the instantaneous exhaust mass flow rate. The exhaust mass flow rate may be measured directly or calculated in accordance with 5.4.4 (intake air and fuel flow measurement), 5.4.5 (tracer method) or 5.4.6 (intake air and air/fuel ratio measurement). Special attention shall be paid to the response times of the different instruments. These differences shall be accounted for by time aligning the signals in accordance with 5.5.3.

For particulates, the exhaust mass flow rate signals given in 5.4 are used for controlling the partial-flow dilution system in order that a sample proportional to the exhaust mass flow rate can be taken. The quality of proportionality is checked by applying a regression analysis between sample and exhaust flow in accordance with 5.6.2.

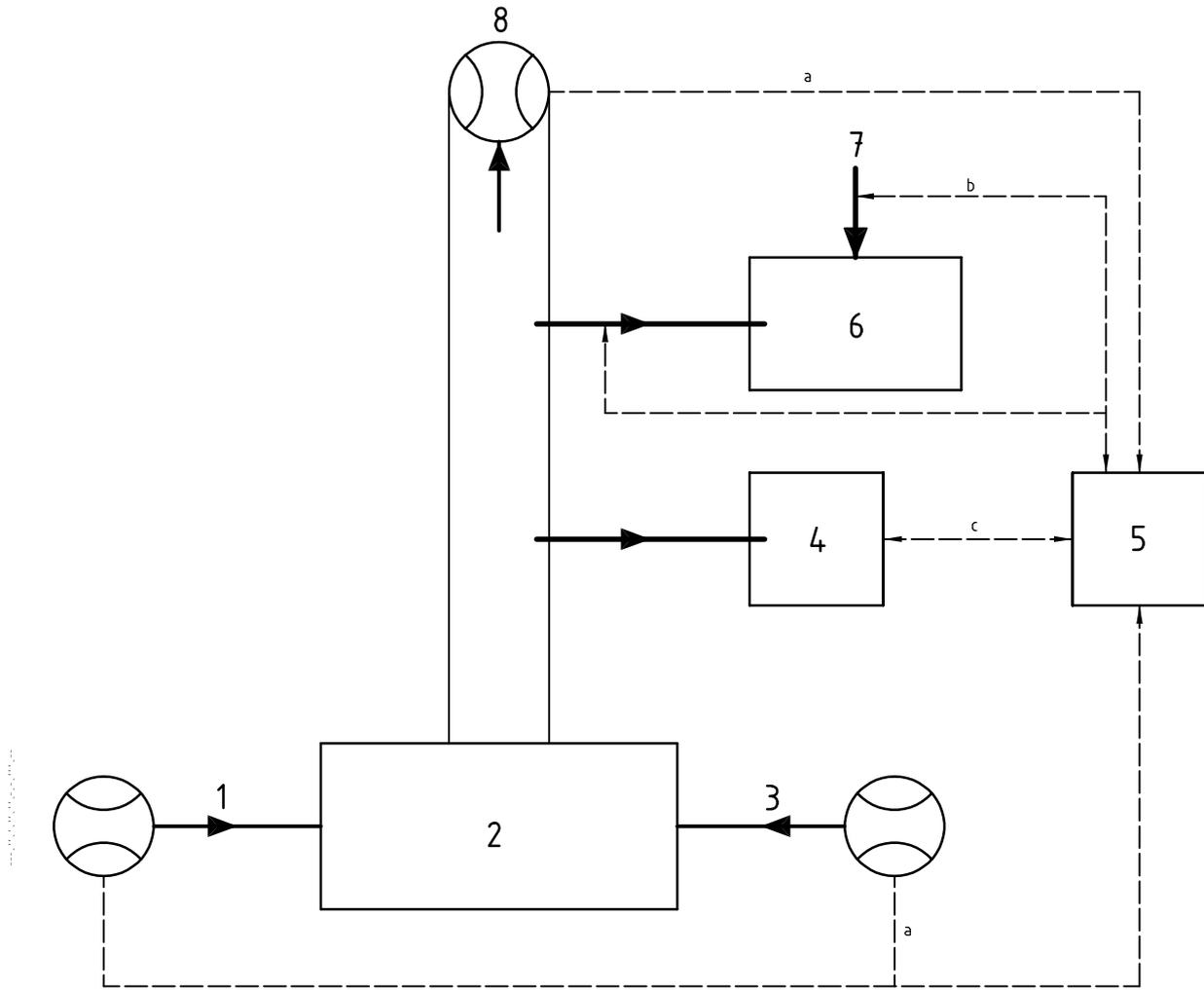
The complete test set up is shown schematically in Figure 2.

## 5.2 Equivalence

The emission of gaseous and particulate components by the engine submitted for testing shall be measured in accordance with clauses 6 and 7. These describe the recommended analytical systems for the gaseous emissions (see clause 6) and the recommended particulate dilution and sampling systems (see clause 7).

Other systems or analysers may be accepted if they yield equivalent results. The determination of system equivalency shall be based on a seven-sample pair (or larger) correlation study between the system under consideration and one of the systems accepted by this International Standard. *Results* refers to the specific cycle weighted emissions value. The correlation testing is to be performed at the same laboratory, on the same test cell, and the same engine, and is preferably to be run concurrently. The test cycle to be used shall be the appropriate cycle on which the engine will be run. The equivalency of the sample pair averages shall be determined by "t"-test statistics as given in Annex A, obtained under these laboratory cell and engine conditions. Outliers shall be determined in accordance with ISO 5725-2 and excluded from the database. The systems to be used for correlation testing shall be declared prior to the test and shall be agreed upon by the parties involved.

For the introduction of a new system into the standard, the determination of equivalency shall be based upon the calculation of repeatability and reproducibility in accordance with ISO 5725-2.



**Key**

- Exhaust sample
- Flow measurement
- Signals for system control and computation

- 1 Fuel flow
- 2 Engine
- 3 Intake air flow
- 4 Exhaust gas analyser
- 5 Control unit
- 6 Partial-flow dilution system
- 7 Dilution air
- 8 Exhaust flow
- a Flow values
- b Flow control
- c Computation

**Figure 2 — Schematic of measurement system**

### 5.3 Accuracy

The equipment described in this International Standard shall be used for emissions tests of engines. This International Standard does not specify particular flow, pressure or temperature measuring equipment. Instead, only the accuracy requirements of such equipment necessary for conducting an emissions test are given. The instruments shall be calibrated as required by internal audit procedures or by the instrument manufacturer.

The calibration of all measuring instruments shall be traceable to relevant national and international standards and shall be in accordance with Table 2.

**Table 2 — Permissible deviations of instruments**

| Number | Measurement instrument    | Permissible deviation  |
|--------|---------------------------|--|
| 1      | Engine speed              | $\pm 2\%$ of reading or $\pm 1\%$ of engine's maximum value, whichever is greater  |
| 2      | Torque                    | $\pm 2\%$ of reading or $\pm 1\%$ of engine's maximum value, whichever is greater  |
| 3      | Fuel consumption          | $\pm 2\%$ of engine's maximum value  |
| 4      | Air consumption           | $\pm 2\%$ of reading or $\pm 1\%$ of engine's max. value, whichever is greater     |
| 5      | Exhaust gas flow          | $\pm 2,5\%$ of reading or $\pm 1,5\%$ of engine's max. value, whichever is greater |
| 6      | Temperatures $\leq 600$ K | $\pm 2$ K absolute   |
| 7      | Temperatures $> 600$ K    | $\pm 1\%$ of reading   |
| 8      | Exhaust gas pressure      | $\pm 0,2$ kPa absolute   |
| 9      | Intake air depression     | $\pm 0,05$ kPa absolute  |
| 10     | Atmospheric pressure      | $\pm 0,1$ kPa absolute   |
| 11     | Other pressures           | $\pm 0,1$ kPa absolute   |
| 12     | Absolute humidity         | $\pm 5\%$ of reading   |
| 13     | Dilution air flow         | $\pm 2\%$ of reading   |
| 14     | Diluted exhaust gas flow  | $\pm 2\%$ of reading   |

### 5.4 Determination of exhaust gas mass flow

#### 5.4.1 Introduction

In order to calculate the emissions in the raw exhaust gas and control a partial-flow dilution system, it is necessary to know the exhaust gas mass flow rate. For the determination of the exhaust mass flow rate, either of the methods specified in 5.4.3 to 5.4.6 may be used.

#### 5.4.2 Response time

For the purpose of emissions calculation, the response time of either method shall be less than or equal to the requirement for the analyser response time, in accordance with 6.3.5.

For the purpose of controlling a partial-flow dilution system, a faster response is required. For partial-flow dilution systems with online control, a response time of  $\leq 0,3$  s is required. For partial-flow dilution systems with look ahead control based on a pre-recorded test run, a response time of the exhaust flow measurement system of  $\leq 5$  s with a rise time of  $\leq 1$  s is required. The system response time shall be as specified by the instrument manufacturer. The combined response time requirements for exhaust gas flow and partial-flow dilution systems are given in 5.6.3.

**5.4.3 Direct measurement method**

Direct measurement of the instantaneous exhaust flow may be done by systems including

- pressure differential devices, such as flow nozzle (see ISO 5167-1),
- ultrasonic flow meters, and
- vortex flow meters.

Precautions shall be taken to avoid measurement errors that could have an impact on emission value errors. Such precautions include the careful installation of the device in the engine exhaust system in accordance with the instrument manufacturers' recommendations and good engineering practice. In particular, engine performance and emissions shall not be affected by the installation of the device.

The flow meters shall meet the accuracy specifications of 5.3.

**5.4.4 Air and fuel measurement method**

This involves measurement of the air and fuel flows with suitable flow meters. The calculation of the instantaneous exhaust gas flow is as follows:

$$q_{mew,i} = q_{maw,i} + q_{mf,i} \tag{4}$$

(for wet exhaust mass)

The flow meters shall meet the accuracy specifications of 5.3, but shall be accurate enough to also meet the accuracy specifications for the exhaust gas flow.

**5.4.5 Tracer measurement method**

This involves measurement of the concentration of a tracer gas in the exhaust.

A known amount of an inert gas (e.g. pure helium) shall be injected into the exhaust gas flow as a tracer. The gas is mixed and diluted by the exhaust gas, but shall not react in the exhaust pipe. The concentration of the gas shall then be measured in the exhaust gas sample.

In order to ensure complete mixing of the tracer gas, the exhaust gas sampling probe shall be located at least 1 m or 30 times the diameter of the exhaust pipe, whichever is larger, downstream of the tracer gas injection point. The sampling probe may be located closer to the injection point if complete mixing is verified by comparing the tracer gas concentration with the reference concentration when the tracer gas is injected upstream of the engine.

The tracer gas flow rate shall be set so that the tracer gas concentration at engine idle speed after mixing becomes lower than the full scale of the trace gas analyser.

The calculation of the exhaust gas flow is as follows:

$$q_{mew,i} = \frac{q_{vt} \times \rho_e}{60 \times (c_{mix,i} - c_a)} \tag{5}$$

where

- $q_{mew,i}$  is the instantaneous exhaust mass flow, in kilograms per second;
- $q_{vt}$  is the tracer gas flow, in cubic centimetres per minute;
- $c_{mix,i}$  is the instantaneous concentration of the tracer gas after mixing, in parts per million (microlitres per litre);

$\rho_e$  is the density of the exhaust gas, in kilograms per cubic metre (cf. Table 3);

$c_a$  is the background concentration of the tracer gas in the intake air, in parts per million (microlitres per litre).

The background concentration of the tracer gas ( $c_a$ ) may be determined by averaging the background concentration measured immediately before and after the test run.

When the background concentration is less than 1 % of the concentration of the tracer gas after mixing ( $c_{mix,i}$ ) at maximum exhaust flow, the background concentration may be neglected.

The total system shall meet the accuracy specifications for the exhaust gas flow, and shall be calibrated in accordance with 6.3.8.

#### 5.4.6 Air flow and air-to-fuel ratio measurement method

This involves exhaust mass calculation from the air flow and the air-to-fuel ratio. The calculation of the instantaneous exhaust gas mass flow is as follows:

$$q_{mew,i} = q_{maw,i} \times \left( 1 + \frac{1}{A/F_{st} \times \lambda_i} \right) \quad (6)$$

with

$$A/F_{st} = \frac{138,0 \times \left( \beta + \frac{\alpha}{4} - \frac{\varepsilon}{2} + \gamma \right)}{12,011 \times \beta + 1,007\,94 \times \alpha + 15,999\,4 \times \varepsilon + 14,006\,7 \times \delta + 32,065 \times \gamma} \quad (7)$$

$$\lambda_i = \frac{\beta \times \left( 100 - \frac{c_{CO} \times 10^{-4}}{2} - c_{HC} \times 10^{-4} \right) + \left( \frac{\alpha}{4} \times \frac{1 - \frac{2 \times c_{CO} \times 10^{-4}}{3,5 \times c_{CO_2}}}{1 + \frac{c_{CO} \times 10^{-4}}{3,5 \times c_{CO_2}}} - \frac{\varepsilon}{2} - \frac{\delta}{2} \right) \times (c_{CO_2} + c_{CO} \times 10^{-4})}{4,764 \times \left( \beta + \frac{\alpha}{4} - \frac{\varepsilon}{2} + \gamma \right) \times (c_{CO_2} + c_{CO} \times 10^{-4} + c_{HC} \times 10^{-4})} \quad (8)$$

where

$A/F_{st}$  is the stoichiometric air-to-fuel ratio, in kilograms;

$\lambda$  is the excess air ratio;

$c_{CO_2}$  is the dry CO<sub>2</sub> concentration, in percent by volume;

$c_{CO}$  is the dry CO concentration, in parts per million (microlitres per litre);

$c_{HC}$  is the HC concentration, in parts per million (microlitres per litre).

NOTE  $\beta$  can be 1 for fuels containing carbon and 0 for hydrogen fuel.

The air flow meter shall meet the accuracy specifications of 5.3, the CO<sub>2</sub> analyser used shall meet those of 6.1, and the total system shall meet the accuracy specifications for the exhaust gas flow.

Optionally, air-to-fuel ratio measurement equipment such as a zirconia type sensor may be used for the measurement of the excess air ratio in accordance with 6.2.9.

## 5.5 Determination of gaseous components

### 5.5.1 General

The gaseous components emitted by the engine submitted for testing shall be measured in accordance with clause 6. They shall be determined in the raw exhaust gas. Optionally, they may be determined in the diluted exhaust gas of a *fractional sampling type* partial-flow dilution system used for particulate determination, in accordance with 7.2.2. Data evaluation and calculation procedures given in 5.5.3 and 5.5.4 refer to raw emissions measurement only. If, optionally, the diluted emissions measurement is used, the data evaluation and calculation procedures shall be agreed by the parties involved.

### 5.5.2 Sampling for gaseous emissions

The gaseous emissions sampling probes shall be fitted at least 0,5 m or three times the diameter of the exhaust pipe, whichever is the larger, upstream of the exit of the exhaust gas system, but sufficiently close to the engine to ensure an exhaust gas temperature of at least 343 K (70 °C) at the probe.

In the case of a multi-cylinder engine with a branched exhaust manifold, the inlet of the probe shall be located sufficiently far downstream to ensure that the sample is representative of the average exhaust emissions from all cylinders. In multi-cylinder engines having distinct groups of manifolds, such as in a "vee" engine configuration, it is recommended that the manifolds be combined upstream of the sampling probe. If this is not practical, it is permissible to acquire a sample from the group with the highest CO<sub>2</sub> emission. Other methods which have been shown to correlate with these procedures may be used. For exhaust emission calculation, the total exhaust mass flow shall be used.

If the engine is equipped with an exhaust after-treatment system, the exhaust sample shall be taken downstream of the exhaust after-treatment system.

### 5.5.3 Data evaluation

For the evaluation of the gaseous emissions, the emission concentrations (HC, CO and NO<sub>x</sub>) and the exhaust gas mass flow rate shall be recorded and stored with a sample rate of at least 2 Hz on a computer system. All other data may be recorded with a sample rate of at least 1 Hz. For analog analysers, the response will be recorded, and the calibration data may be applied online or offline during the data evaluation.

For calculation of the mass emission of the gaseous components, the traces of the recorded concentrations and the trace of the exhaust gas mass flow rate shall be time-aligned by the transformation time. Therefore, the response time of each gaseous emissions analyser and of the exhaust gas mass flow system shall be determined in accordance with 6.3.5 and 5.4.2, respectively, and recorded.

### 5.5.4 Calculation of mass emission

#### 5.5.4.1 General

The mass, expressed in grams per test, of the pollutants shall be determined by calculating the instantaneous mass emissions from the concentrations of the pollutants and the exhaust mass flow, aligned for the transformation time as determined in accordance with 5.5.3, and integrating the instantaneous values over the cycle in accordance with 5.5.4.2. Preferably, the concentrations should be measured on a wet basis. If measured on a dry basis, the dry/wet correction in accordance with 5.5.5 shall be applied to the instantaneous concentration values before any further calculation is done.

Optionally, the mass emissions may be calculated using the exact formulae of 5.5.4.3, with the prior agreement of the parties involved. In any case, the exact formulae shall be used if the fuel used for the test is not specified in Table 3, or under multi-fuel operation.

See annex D for an example of the calculation procedures.

**5.5.4.2 Calculation method based on tabulated values**

The following formula shall be used:

$$m_{\text{gas}} = \sum_{i=1}^{i=n} u_{\text{gas}} \times c_{\text{gas},i} \times q_{\text{mew},i} \times \frac{1}{f} \tag{9}$$

(expressed in grams per test)

where

- $u_{\text{gas}}$  is the ratio between the density of the exhaust component and the density of the exhaust gas;
- $c_{\text{gas},i}$  is the instantaneous concentration of the respective component in the raw exhaust gas, in parts per million (microlitres per litre);
- $q_{\text{mew},i}$  is the instantaneous exhaust mass flow, in kilograms per second;
- $f$  is the data sampling rate, in hertz;
- $n$  is the number of measurements.

For the calculation of  $\text{NO}_x$ , the humidity correction factor  $k_{h,D}$ , or  $k_{h,G}$ , as applicable, determined in accordance with 5.5.6, shall be used.

Values for  $u_{\text{gas}}$  are given in Table 3 for selected components and a range of fuels.

**Table 3 — Values of  $u_{\text{gas}}$  and density for various exhaust components**

| Gas  | $\text{NO}_x$ | CO  | HC        | $\text{CO}_2$          | $\text{O}_2$ | $\text{CH}_4$ | HCHO      | $\text{CH}_3\text{OH}$ |           |
|--|---------------|---|-----------|------------------------|--------------|---------------|-----------|------------------------|-----------|
| $\rho_{\text{gas}}$ (kg/m <sup>3</sup> )   | 2,053         | 1,250                                     | 2,053     | a                      | 1,9636       | 1,4277        | 0,716     | 1,340                  |           |
| Fuel   | $\rho_e$      | Coefficient $u_{\text{gas}}$ <sup>b</sup> |           |                        |              |               |           |                        |           |
| Diesel   | 1,293 9       | 0,001 587                                 | 0,000 966 | 0,000 479              | 0,001 518    | 0,001 103     | 0,000 553 | 0,001 035              | 0,001 105 |
| RME  | 1,295 0       | 0,001 585                                 | 0,000 965 | 0,000 536              | 0,001 516    | 0,001 102     | 0,000 553 | 0,001 035              | 0,001 104 |
| Methanol   | 1,260 7       | 0,001 628                                 | 0,000 991 | 0,001 133              | 0,001 558    | 0,001 132     | 0,000 568 | 0,001 063              | 0,001 134 |
| Ethanol  | 1,275 6       | 0,001 609                                 | 0,000 980 | 0,000 805              | 0,001 539    | 0,001 119     | 0,000 561 | 0,001 050              | 0,001 121 |
| Natural gas <sup>c</sup>   | 1,265 6       | 0,001 622                                 | 0,000 987 | 0,000 523 <sup>d</sup> | 0,001 552    | 0,001 128     | 0,000 565 | 0,001 059              | 0,001 130 |
| Propane  | 1,280 5       | 0,001 603                                 | 0,000 976 | 0,000 511              | 0,001 533    | 0,001 115     | 0,000 559 | 0,001 046              | 0,001 116 |
| Butane   | 1,283 1       | 0,001 600                                 | 0,000 974 | 0,000 505              | 0,001 530    | 0,001 113     | 0,000 558 | 0,001 044              | 0,001 114 |
| Gasoline   | 1,297 7       | 0,001 582                                 | 0,000 963 | 0,000 471              | 0,001 513    | 0,001 100     | 0,000 551 | 0,001 032              | 0,001 102 |
| <sup>a</sup> Depending on fuel.<br><sup>b</sup> At $\lambda = 2$ , dry air, 273 K, 101,3 kPa.<br><sup>c</sup> $u$ accurate within 0,2 % for mass composition of: C = 66 % to 76 %; H = 22 % to 25 %; N = 0 % to 12 %.<br><sup>d</sup> NMHC on the basis of $\text{CH}_{2,93}$ (for total HC the $u_{\text{gas}}$ coefficient of $\text{CH}_4$ shall be used. |               |   |           |                        |              |               |           |                        |           |

**5.5.4.3 Calculation method based on exact formulae**

The mass emission shall be calculated using Equation (9). Instead of using the tabulated values, the following formulae shall be applied for the calculation of  $u_{\text{gas}}$ . It is assumed in the following formulae that the concentration  $c_{\text{gas}}$  is measured in, or converted to, parts per million.

$$u_{\text{gas},i} = M_{\text{gas}} / (M_{r,e,i} \times 1\,000) \tag{10}$$

or

$$u_{\text{gas},i} = \rho_{\text{gas}} / (\rho_{e,i} \times 1\,000) \tag{11}$$

where

$$\rho_{\text{gas}} = M_{\text{gas}} / 22,41 \tag{12}$$

or, optionally, is taken from Table 3.

The densities,  $\rho_{\text{gas}}$ , are given for a number of exhaust gas components in Table 3. The molecular mass of the exhaust,  $M_{r,e}$  shall be derived for a general fuel composition  $C_{\beta}H_{\alpha}O_{\varepsilon}N_{\delta}S_{\gamma}$  under the assumption of complete combustion, as follows:

$$M_{r,e,i} = \frac{1 + \frac{q_{mf,i}}{q_{maw,i}}}{\frac{q_{mf,i}}{q_{maw,i}} \times \frac{\frac{\alpha}{4} + \frac{\varepsilon}{2} + \frac{\delta}{2}}{12,011 \times \beta + 1,007\,94 \times \alpha + 15,999\,4 \times \varepsilon + 14,006\,7 \times \delta + 32,065 \times \gamma} + \frac{\frac{H_a \times 10^{-3}}{2 \times 1,007\,94 + 15,999\,4} + \frac{1}{M_{r,\text{air}}}}{1 + H_a \times 10^{-3}}} \tag{13}$$

The exhaust density  $\rho_e$  shall be derived as follows:

$$\rho_{e,i} = \frac{1\,000 + H_a + 1\,000 \times (q_{mf,i} / q_{mad,i})}{773,4 + 1,243\,4 \times H_a + k_f \times 1\,000 \times (q_{mf,i} / q_{mad,i})} \tag{14}$$

where

$$k_f = 0,055\,584 \times w_{\text{ALF}} - 0,000\,108\,3 \times w_{\text{BET}} - 0,000\,156\,2 \times w_{\text{GAM}} + 0,007\,993\,6 \times w_{\text{DEL}} + 0,006\,997\,8 \times w_{\text{EPS}} \tag{15}$$

**5.5.4.4 Calculation of NMHC with non-methane cutter**

The concentration of NMHC shall be calculated as follows:

$$c_{\text{NMHC}} = \frac{c_{\text{HC(w/oCutter)}} \times (1 - E_M) - c_{\text{HC(wCutter)}}}{E_E - E_M} \tag{16}$$

where

$c_{\text{HC(wCutter)}}$  is the HC concentration with the sample gas flowing through the NMC;

$c_{\text{HC(w/oCutter)}}$  is the HC concentration with the sample gas bypassing the NMC;

$E_M$  is the methane efficiency as determined in accordance with 6.3.10.5.2;

$E_E$  is the ethane efficiency as determined in accordance with 6.3.10.5.3.

NOTE If a non-methane cutter is used, the system response time could exceed 10 s.

### 5.5.5 Dry/wet correction

The instantaneously measured concentration shall be converted to wet basis in accordance with the following formulae, if not already measured on a wet basis.

$$c_{\text{wet}} = k_W \times c_{\text{dry}} \quad (17)$$

$$k_W = \left( 1 - \frac{1,243\,4 \times H_a + 111,12 \times w_{\text{ALF}} \times \frac{q_{\text{mf},i}}{q_{\text{mad},i}}}{773,4 + 1,243\,4 \times H_a + \frac{q_{\text{mf},i}}{q_{\text{mad},i}} \times k_f \times 1\,000} \right) \times 1,008 \quad (18)$$

or

$$k_W = \left( 1 - \frac{1,243\,4 \times H_a + 111,12 \times w_{\text{ALF}} \times \frac{q_{\text{mf},i}}{q_{\text{mad},i}}}{773,4 + 1,243\,4 \times H_a + \frac{q_{\text{mf},i}}{q_{\text{mad},i}} \times k_f \times 1\,000} \right) / \left( 1 - \frac{p_r}{p_b} \right) \quad (19)$$

or

$$k_W = \frac{1}{1 + \alpha \times 0,005 \times (c_{\text{CO}_2} + c_{\text{CO}}) - 0,01 \times c_{\text{H}_2}} - k_{\text{W}2} \quad (20)$$

with

$$k_{\text{W}2} = \frac{1,608 \times H_a}{1\,000 + (1,608 \times H_a)} \quad (21)$$

$$c_{\text{H}_2} = \frac{0,5 \times \alpha \times c_{\text{CO}} \times (c_{\text{CO}} + c_{\text{CO}_2})}{c_{\text{CO}} + 3,5 \times c_{\text{CO}_2}} \quad (22)$$

where

$p_r$  is the water vapour pressure after cooling bath, in kilopascals;

$p_b$  is the total atmospheric pressure, in kilopascals;

$\alpha$  is the molar hydrogen ratio of the fuel;

$c_{\text{CO}_2}$  is the dry  $\text{CO}_2$  concentration, in percent by volume;

$c_{\text{CO}}$  is the dry CO concentration, in percent by volume;

$c_{\text{H}_2}$  is the dry  $\text{H}_2$  concentration, in percent by volume (four-stroke gasoline engines only);

$H_a$  is the intake air humidity, in grams of water per kilogram of dry air;

$$k_f = 0,055\,584 \times w_{\text{ALF}} - 0,000\,108\,3 \times w_{\text{BET}} - 0,000\,156\,2 \times w_{\text{GAM}} + 0,007\,993\,6 \times w_{\text{DEL}} + 0,006\,997\,8 \times w_{\text{EPS}} \quad (15)$$

### 5.5.6 NO<sub>x</sub> correction for temperature and humidity

As the NO<sub>x</sub> emission depends on ambient air conditions, the NO<sub>x</sub> concentration shall be corrected for ambient air temperature and humidity with the factors given in the following formulae.

a) For compression-ignition engines:

$$k_{h,D} = \frac{1}{1 - 0,018\ 2 \times (H_a - 10,71) + 0,004\ 5 \times (T_a - 298)} \quad (23)$$

with

$T_a$  the temperature of the intake air, in kelvin;

$H_a$  the humidity of the intake air, in grams of water per kilogram of dry air, where  $H_a$  may be derived from relative humidity measurement, dewpoint measurement, vapour pressure measurement or dry/wet bulb measurement using the generally accepted formulae.

b) For spark-ignition engines:

$$k_{h,G} = 0,627\ 2 + 44,030 \times 10^{-3} \times H_a - 0,862 \times 10^{-3} \times H_a^2 \quad (24)$$

where  $H_a$  may be derived from relative humidity measurement, dewpoint measurement, vapour pressure measurement or dry/wet bulb measurement using the generally accepted formulae.

### 5.5.7 Calculation of the specific emissions

The specific emissions (grams per kilowatt hour) shall be calculated for each individual component as follows:

$$M_{\text{gas}} = m_{\text{gas}} / W_{\text{act}} \quad (25)$$

where  $W_{\text{act}}$  is the actual cycle work as determined over the respective test cycle, in kilowatt hours, in accordance with the relevant international or national regulations or standards.

## 5.6 Particulate determination

### 5.6.1 General

The determination of the particulates calls for a dilution system. For the purposes of this International Standard, dilution shall be accomplished by a partial-flow dilution system. The flow capacity of the dilution system shall be large enough to completely eliminate water condensation in the dilution and sampling systems, and maintain the temperature of the diluted exhaust gas at or below 325 K (52 °C) immediately upstream of the filter holder. Dehumidification of the dilution air before it enters the dilution system is permitted, and is especially useful if dilution air humidity is high. The temperature of the dilution air shall be higher than 288 K (15 °C) in close proximity to the entrance into the dilution tunnel. The particulate level of the dilution air may be determined by passing the dilution air through the particulate filter.

The partial-flow dilution system has to be designed to extract a raw exhaust sample of constant mass ratio from the engine exhaust stream, while responding to excursions in the exhaust stream flow rate and introducing dilution air to this sample to achieve 325 K (52 °C) or below at the test filter. For this, it is essential that the dilution ratio or the sampling ratio,  $r_d$  or  $r_s$ , be determined such that the accuracy requirements of 7.1.4 are fulfilled. Different extraction methods can be applied, whereas the type of extraction used dictates to a significant degree the sampling hardware and procedures to be used (see clause 7).

To determine the mass of the particulates, a particulate sampling system, particulate sampling filters, a microgram balance, and a temperature and humidity-controlled weighing chamber, are required. The details of the systems are given in clause 7.

### 5.6.2 Particulate sampling

In general, the particulate sampling probe shall be installed in close proximity to the gaseous emissions sampling probe, but sufficiently distant so as to not cause interference. Therefore, the installation provisions of 5.5.2 also apply to particulate sampling. The sampling line shall be in accordance with clause 7.

In the case of a multi-cylinder engine with a branched exhaust manifold, the inlet of the probe shall be located sufficiently far downstream to ensure that the sample is representative of the average exhaust emissions from all cylinders. In multi-cylinder engines having distinct groups of manifolds, such as in a “vee” engine configuration, it is recommended that the manifolds be combined upstream of the sampling probe. If this is not practical, it is permissible to acquire a sample from the group with the highest particulate emission. Other methods shown to correlate with the above methods may be used. For exhaust emission calculation, the total exhaust mass flow shall be used.

### 5.6.3 System response time

For the control of a partial-flow dilution system, a fast system response is required. The transformation time for the system shall be determined in accordance with 7.3.3. If the combined transformation time of the exhaust flow measurement (see 5.4.2) and the partial flow system is less than 0,3 s, online control may be used. If the transformation time exceeds 0,3 s, look-ahead control based on a pre-recorded test run shall be used. In this case, the rise time shall be  $\leq 1$  s and the delay time of the combination  $\leq 10$  s.

The total system response shall be designed to ensure a representative sample of the particulates,  $q_{mp,i}$ , proportional to the exhaust mass flow. To determine the proportionality, a regression analysis of  $q_{mp,i}$  versus  $q_{mew,i}$  shall be conducted at a minimum 5 Hz data acquisition rate, and the following criteria shall be met:

- the correlation coefficient  $R^2$  of the linear regression between  $q_{mp,i}$  and  $q_{mew,i}$  shall be not less than 0,95;
- the standard error of estimate of  $q_{mp,i}$  on  $q_{mew,i}$  shall not exceed 5 % of  $q_{mp}$  max.;
- $q_{mp}$  intercept of the regression line shall not exceed  $\pm 2$  % of  $q_{mp}$  max.

Optionally, a pre-test may be run, and the exhaust mass flow signal of the pre-test used for controlling the sample flow into the particulate system (look-ahead control). Such a procedure is required if the transformation time of the particulate system,  $t_{50,P}$  or the transformation time of the exhaust mass flow signal,  $t_{50,F}$ , or both, are  $> 0,3$  s. A correct control of the partial dilution system is obtained if the time trace of  $q_{mew,pre}$  of the pre-test, which controls  $q_{mp}$ , is shifted by a look-ahead time of  $t_{50,P} + t_{50,F}$ .

For establishing the correlation between  $q_{mp,i}$  and  $q_{mew,i}$ , the data taken during the actual test shall be used, with  $q_{mew,i}$  time aligned by  $t_{50,F}$  relative to  $q_{mp,i}$  (no contribution from  $t_{50,P}$  to the time alignment). That is, the time shift between  $q_{mew}$  and  $q_{mp}$  is the difference in their transformation times, determined in accordance with 7.3.3.

### 5.6.4 Data evaluation

At least 1 h before the test, the filter shall be placed in a petri dish, protected against dust contamination and allowing air exchange, and then placed in a weighing chamber for stabilization. At the end of the stabilization period, the filter shall be weighed and the tare mass shall be recorded. The filter shall then be stored in a closed petri dish or sealed filter holder until needed for testing. The filter shall be used within 8 h of its removal from the weighing chamber.

The particulate filter shall be returned to the weighing chamber no later than 1 h after completion of the test. It shall be conditioned in a petri dish, protected against dust contamination and allowing air exchange, for at least 1 h, but for not more than 80 h, and then weighed. The gross mass of the filter shall be recorded and the tare mass subtracted, resulting in the particulate sample mass  $m_f$ . For the evaluation of the particulate concentration, the total sample mass ( $m_{sep}$ ) through the filter over the test cycle shall be recorded.

With the prior approval of the parties involved, the PM mass may be corrected for the PM level of the dilution air, as determined in 5.6.1, in line with good engineering practice and the specific design features of the particulate measurement system.

**5.6.5 Calculation of mass emission**

The mass, expressed in grams per test, of particulates shall be calculated by either of the following methods (for an example of the calculation procedures, see Annex D).

a) Method 1:

$$m_{PM} = \frac{m_f}{m_{sep}} \times \frac{m_{edf}}{1\,000} \tag{26}$$

where

- $m_f$  is the particulate mass sampled over the cycle, in milligrams;
- $m_{sep}$  is the mass of diluted exhaust gas passing the particulate collection filters, in kilograms;
- $m_{edf}$  is the mass of equivalent diluted exhaust gas over the cycle, in kilograms.

The total mass of equivalent diluted exhaust gas mass over the cycle shall be determined as follows:

$$m_{edf} = \sum_{i=1}^{i=n} q_{medf,i} \times \frac{1}{f} \tag{27}$$

$$q_{medf,i} = q_{mew,i} \times r_{d,i} \tag{28}$$

$$r_{d,i} = \frac{q_{mdew,i}}{(q_{mdew,i} - q_{mdw,i})} \tag{29}$$

where

- $q_{medf,i}$  is the instantaneous equivalent diluted exhaust mass flow rate, in kilograms per second;
- $q_{mew,i}$  is the instantaneous exhaust mass flow rate, in kilograms per second;
- $r_{d,i}$  is the instantaneous dilution ratio;
- $q_{mdew,i}$  is the instantaneous diluted exhaust mass flow rate through dilution tunnel, in kilograms per second;
- $q_{mdw,i}$  is the instantaneous dilution air mass flow rate, in kilograms per second;
- $f$  is the data sampling rate, in hertz;
- $n$  is the number of measurements.

b) Method 2:

$$m_{PM} = m_f / (r_s \times 1\,000) \tag{30}$$

where

- $m_f$  is the particulate mass sampled over the cycle, in milligrams;
- $r_s$  is the average sample ratio over the test cycle

with

$$r_s = \frac{m_{se}}{m_{ew}} \times \frac{m_{sep}}{m_{sed}} \quad (31)$$

where

- $m_{se}$  is the sample mass over the cycle, in kilograms;
- $m_{ew}$  is the total exhaust mass flow over the cycle, in kilograms;
- $m_{sep}$  is the mass of diluted exhaust gas passing the particulate collection filters, in kilograms;
- $m_{sed}$  is the mass of diluted exhaust gas passing the dilution tunnel, in kilograms.

NOTE In the case of the total sampling type system,  $m_{sep}$  and  $m_{sed}$  are identical.

### 5.6.6 Calculation of the specific emissions

The specific particulate emission (in grams per kilowatt hour) shall be calculated by

$$M_{PM} = m_{PM}/W_{act} \quad (32)$$

where

- $W_{act}$  is the actual cycle work as determined over the respective test cycle, in kilowatt hours, in accordance with the relevant international or national regulations or standards.

## 6 Measurement equipment for the gaseous components

### 6.1 Analyser specifications

#### 6.1.1 General

The analysers shall have a measuring range and response time appropriate to the accuracy required to measure the concentrations of the exhaust gas components under transient conditions. The analyser range shall be selected such that the average concentration measured over the test cycle falls between 15 % and 100 % FS, the accuracy of the analyser not exceeding  $\pm 2$  % of reading for the average concentration.

If read-out systems (computers, data loggers) can provide sufficient accuracy and resolution below 15 % FS, measurements below 15 % FS are also permitted. In this case, additional calibrations of at least four, non-zero, nominally equally spaced points shall be made to ensure the accuracy of the calibration curves in accordance with 6.3.6.3.

The electromagnetic compatibility (EMC) of the equipment shall be on a level to minimize additional errors.

#### 6.1.2 Accuracy

The analyser shall not deviate from the nominal calibration point by more than  $\pm 2$  % of the reading or  $\pm 0,3$  % FS, whichever is larger. The accuracy shall be determined in accordance with the calibration requirements given in 6.3.6.

NOTE For the purposes of this International Standard, accuracy is defined as the deviation of the analyser reading from the nominal calibration values using a calibration gas ( $\equiv$  true value).

### 6.1.3 Precision

The precision, defined as 2,5 times the standard deviation of 10 repetitive responses to a given calibration or span gas, shall be not greater than 1 % FS concentration for each range used above 155 ppm (= 155 µl/l) or 155 ppm C (= 155 µl/l C), or 2 % of each range used below 155 ppm (= 155 µl/l) or 155 ppm C (= 155 µl/l C).

### 6.1.4 Noise

The analyser peak-to-peak response to zero and calibration or span gases over any 10 s period shall not exceed 2 % FS on all ranges used.

### 6.1.5 Zero drift

Zero response is defined as the mean response, including noise, to a zero gas during a 30 s time interval. The drift of the zero response during a 1 h period shall be less than 2 % FS on the lowest range used.

### 6.1.6 Span drift

Span response is defined as the mean response, including noise, to a span gas during a 30 s interval. The drift of the span response during a 1 h period shall be less than 2 % FS on the lowest range used.

### 6.1.7 Rise time

The rise time of the analyser installed in the measurement system shall not exceed 2,5 s.

NOTE Simply evaluating the response time of the analyser alone will not clearly define the suitability of the total system for transient testing. Volumes and, especially, dead volumes throughout the system will not only affect the transportation time from the probe to the analyser, but also the rise time. In addition, transport times inside an analyser are considered as analyser response time — as for the converter or water traps inside a NO<sub>x</sub> analyser. See 6.3.5 for the determination of the total system response time.

### 6.1.8 Gas drying

Exhaust gases may be measured wet or dry. A gas-drying device, if used, shall have minimal effect on the composition of the measured gases. Chemical dryers shall not be used to remove water from the sample.

## 6.2 Analysers

### 6.2.1 General

The measurement principles to be used are given in 6.2.2 to 6.2.9. A detailed description of the measurement systems is given in 6.4. The gases to be measured shall be analysed using the following instruments. For non-linear analysers, the use of linearizing circuits is permitted.

### 6.2.2 Carbon monoxide (CO) analysis

The carbon monoxide analyser shall be of the non-dispersive infrared (NDIR) absorption type.

### 6.2.3 Carbon dioxide (CO<sub>2</sub>) analysis

The carbon dioxide analyser shall be of the non-dispersive infrared (NDIR) absorption type.

### 6.2.4 Hydrocarbon (HC) analysis

The hydrocarbon analyser shall be of the heated flame ionization detector (HFID) type with detector, valves, pipe work, etc. heated to maintain a gas temperature of 463 K ± 10 K (190 °C ± 10 °C). Optionally, for NG-fuelled and SI

engines, the hydrocarbon analyser may be of the non-heated flame ionization detector (FID) type, depending on the method used (see 6.4.3).

### 6.2.5 Non-methane hydrocarbon (NMHC) analysis

The determination of the non-methane hydrocarbon fraction shall be performed with a heated, non-methane cutter (NMC) operated in line with an FID in accordance with 6.4.4 by subtraction of the methane from the hydrocarbons.

### 6.2.6 Oxides of nitrogen (NO<sub>x</sub>) analysis

If measured on a dry basis, the oxides of nitrogen analyser shall be of the chemiluminescent detector (CLD) or heated chemiluminescent detector (HCLD) type, with a NO<sub>2</sub>/NO converter. If measured on a wet basis, an HCLD with converter maintained above 328 K (55 °C) shall be used, provided the water quench check (see 6.3.11.3.2) is satisfied. For both CLD and HCLD, the sampling path shall be maintained at a wall temperature of between 328 K and 473 K (55 °C to 200 °C) — up to the converter for dry measurement, and up to the analyser for wet measurement.

### 6.2.7 Formaldehyde (HCHO) analysis

For continuous measurement in the raw exhaust gas, an FTIR (Fourier transform infrared) or an SIMS (soft ionization mass spectrometric) analyser shall be used in accordance with the instrument supplier's instructions.

The FTIR analyser shall be equipped with an algorithm for generating interference-free concentration values from an infrared spectrum. It shall also be equipped with a dedicated spectral data base for each instrument, in order to avoid interference caused by differences in the spectra between the instruments.

The SIMS analyser shall be equipped with a control library for generating interference decoupled values of the formaldehyde concentration. The internal energy of the ionizing ion shall be above 11,6 eV (e.g. Xe<sup>+</sup> with an internal energy of 12,2 eV). If measured on mass 30, the interference from NO<sub>2</sub> shall be compensated with the help of the known ionization efficiency ratio of NO<sub>2</sub> for masses 46 and 30. The interference decoupling shall be performed with a maximum cycle time of 300 ms. Measurement of formaldehyde on mass 29 is acceptable if the additional signal due to higher aldehydes is either desired, accepted or compensated (measurement on mass 29 gives an upper limit for the formaldehyde concentration).

If measured in the dilute exhaust gas of a partial-flow dilution system, formaldehyde shall be determined by passing the diluted exhaust sample with a constant flow rate through an impinger containing an acetonitrile (ACN) solution of DNPH reagent or through a silica cartridge coated with 2,4-DNPH. The sample collected shall be analysed by a high-pressure liquid chromatograph (HPLC) using UV detection at 365 nm (see 6.4.5). If the dilute gas is sampled from a total sampling type partial-flow dilution system, the provisions for the accuracy of the flow measurement given in 7.1.4. shall be met using the procedure given in 7.1.4.2.

### 6.2.8 Methanol (CH<sub>3</sub>OH) analysis

For continuous measurement in the raw exhaust gas, an FTIR or SIMS analyser shall be used in accordance with the instrument supplier's instructions.

The FTIR analyser shall be equipped with an algorithm for generating interference-free concentration values from an infrared spectrum. It shall also be equipped with a dedicated spectral data base for each instrument, in order to avoid interference caused by differences in the spectra between the instruments.

The SIMS analyser shall be equipped with a control library for generating interference decoupled values of the methanol concentration. The internal energy of the ionizing ion shall be above 11,2 eV (e.g. Xe<sup>+</sup> with an internal energy of 12,2 eV). This enables measurement of methanol on mass 31. The only substances interfering with this mass could be fragments of ethanol and propanol, substances which usually do not occur in exhaust gases. For an accurate measurement of methanol, the interference decoupling shall nevertheless be performed with the help of the known ionization efficiency ratios of these substances on their unfragmented masses and mass 31.

Optionally, methanol may be determined with an HFID. In this case, the HFID calibrated on propane shall be operated at  $385\text{ K} \pm 10\text{ K}$  ( $112\text{ °C} \pm 10\text{ °C}$ ). The methanol response factor shall be determined at several concentrations in the range of concentrations in the sample, in accordance with 6.3.10.4.

If measured in the dilute exhaust gas of partial-flow dilution system, methanol shall be determined by passing the diluted exhaust sample with a constant flow rate through an impinger containing deionized water. The sample shall be analysed by a gas chromatograph (GC) with FID (see 6.4.6). If the dilute gas is sampled from a total sampling type partial-flow dilution system, the provisions for the accuracy of the flow measurement given in 7.1.4. shall be met using the procedure given in 7.1.4.2.

### 6.2.9 Air-to-fuel measurement

The air-to-fuel measurement equipment used to determine the exhaust gas flow in accordance with 5.4.6 shall be a wide-range air-to-fuel ratio sensor or lambda sensor of zirconia type.

The sensor shall be mounted directly on the exhaust pipe where the exhaust gas temperature is high enough to eliminate water condensation.

The accuracy of the sensor with incorporated electronics shall be within

- $\pm 3\%$  of reading       $\lambda < 2$
- $\pm 5\%$  of reading       $2 \leq \lambda < 5$
- $\pm 10\%$  of reading      $5 \leq \lambda$

To fulfil this accuracy, the sensor shall be calibrated as specified by the instrument manufacturer.

## 6.3 Calibration

### 6.3.1 General

Each analyser shall be calibrated as often as necessary to fulfil the accuracy requirements of this International Standard. The calibration method to be used is specified here for the analysers indicated in 6.2 and 6.4.

### 6.3.2 Calibration gases

#### 6.3.2.1 General requirements

The shelf life of all calibration gases shall be respected. The expiry date of the calibration gases stated by the manufacturer shall be recorded.

#### 6.3.2.2 Pure gases

The required purity of the gases is defined by the contamination limits given below. The following gases shall be available for operation.

a) Purified nitrogen:

- Contamination       $\leq 1\text{ ppm}$  (=  $1\text{ }\mu\text{l/l}$ ) C<sub>1</sub>,       $\leq 1\text{ ppm}$  (=  $1\text{ }\mu\text{l/l}$ ) CO,       $\leq 400\text{ ppm}$  (=  $400\text{ }\mu\text{l/l}$ ) CO<sub>2</sub>,  
     $\leq 0,1\text{ ppm}$  (=  $0,1\text{ }\mu\text{l/l}$ ) NO.

b) Purified oxygen:

- Purity > 99,5 % by volume O<sub>2</sub>

## c) Hydrogen-helium mixture:

- $(40 \pm 2)$  % hydrogen, balance helium
- Contamination  $\leq 1$  ppm (= 1  $\mu\text{l/l}$ ) C1,  $\leq 400$  ppm (= 400  $\mu\text{l/l}$ ) CO<sub>2</sub>,

## d) Purified synthetic air:

- Contamination  $\leq 1$  ppm (= 1  $\mu\text{l/l}$ ) C1,  $\leq 1$  ppm (= 1  $\mu\text{l/l}$ ) CO,  $\leq 400$  ppm (= 400  $\mu\text{l/l}$ ) CO<sub>2</sub>,  
 $\leq 0,1$  ppm (= 0,1  $\mu\text{l/l}$ ) NO.
- Oxygen content of between 18 % and 21 % by volume.

**6.3.2.3 Calibration and span gases**

Mixtures of gases having the following chemical compositions shall be available:

- C<sub>3</sub>H<sub>8</sub> and purified synthetic air (see 6.3.2.2);
- CO and purified nitrogen;
- NO<sub>x</sub> and purified nitrogen — the amount of NO<sub>2</sub> contained in this calibration gas shall not exceed 5 % of the NO content;
- CO<sub>2</sub> and purified nitrogen;
- CH<sub>4</sub> and purified synthetic air;
- C<sub>2</sub>H<sub>6</sub> and purified synthetic air.

Other gas combinations are allowed, provided the gases do not react with one another.

The true concentration of a calibration or span gas shall be within  $\pm 2$  % of the nominal value, and shall be traceable to national and international standards. All concentrations of calibration gas shall be given on a volume basis (in parts per million or percent by volume or as a volume fraction in microlitres per litre).

**6.3.2.4 Use of precision blending devices**

The gases used for calibration and span may also be obtained by means of precision blending devices (gas dividers), diluting with purified N<sub>2</sub> or with purified synthetic air. The accuracy of the mixing device shall be such that the concentration of the blended calibration gases is accurate to within  $\pm 2$  %. This accuracy implies that primary gases used for blending shall be known to an accuracy of at least  $\pm 1$  %, traceable to national or international gas standards. The verification shall be performed at between 15 % FS and 50 % FS for each calibration incorporating a blending device. An additional verification may be performed using another calibration gas, if the first verification fails.

Optionally, the blending device may be checked with an instrument by nature linear, e.g. using NO gas with a CLD. The span value of the instrument shall be adjusted with the span gas directly connected to the instrument. The blending device shall be checked at the used settings and the nominal value shall be compared to the measured concentration of the instrument. This difference shall in each point be within  $\pm 1$  % of the nominal value.

Other methods may be used based on good engineering practice and with the prior agreement of the parties involved.

The precision gas divider with an accuracy within  $\pm 1$  % is recommended, in order that an accurate analyser calibration curve can be established. The gas divider shall be calibrated by the instrument manufacturer.

**6.3.2.5 Oxygen interference check gases**

Oxygen interference check gases are a blend of propane, oxygen and nitrogen. They shall contain propane with 350 ppm (= 350 µl/l) C ± 75 ppm (= 75 µl/l) C hydrocarbon. The concentration value shall be determined to calibration gas tolerances by chromatographic analysis of total hydrocarbons plus impurities or by dynamic blending. The oxygen concentrations required for gasoline and diesel engine testing are given in Table 4, with the remainder being purified nitrogen.

**Table 4 — Oxygen interference check gases**

| Type of engine      | O <sub>2</sub> concentration (volume fraction)<br>% by volume |
|---------------------|---|
| Diesel              | 21 (20 to 22)   |
| Diesel and gasoline | 10 (9 to 11)  |
|                     | 5 (4 to 6)  |
| Gasoline            | 0 (0 to 1)  |

**6.3.3 Operating procedure**

The operating procedure for analysers shall follow the start-up and operating instructions of the instrument manufacturer, and the minimum requirements given in 6.3.4 to 6.3.9.

**6.3.4 Leakage test**

A system leakage test shall be performed. Disconnect the probe from the exhaust system and unplug the end. Switch on the analyser pump. After an initial stabilization period, all flow meters are to read zero. If not, the sampling lines shall be checked and the fault corrected.

The maximum allowable leakage rate on the vacuum side shall be 0,5 % of the in-use flow rate for the portion of the system being checked. The analyser flows and bypass flows may be used to estimate the in-use flow rates.

Alternatively, the system may be evacuated to a pressure of at least 20 kPa vacuum (80 kPa absolute). After an initial stabilization period, the pressure increase Δ*p* (in kilopascals per minute) in the system should not exceed

$$\Delta p = p / V_s \times 0,005 \times q_{vs} \tag{33}$$

where

*V<sub>s</sub>* is the system volume, in litres;

*q<sub>vs</sub>* is the system flow rate, in litres per minute.

Another method is the introduction of a concentration step change at the beginning of the sampling line by switching from zero to span gas. If, after an adequate period of time, the reading is about 1 % low compared to the introduced concentration, this points to calibration or leakage problems.

**6.3.5 Response time check of analytical system**

The system settings for the response time evaluation shall be exactly the same as during measurement of the test run (i.e. pressure, flow rates, filter settings on the analysers and all other response time influences). The response time determination shall be done with gas switching directly at the inlet of the sample probe. The gas switching shall be done in less than 0,1 s. The gases used for the test shall cause a concentration change of at least 60 % FS.

Record the concentration trace of each single gas component. The response time is defined as the difference in time between the gas switching and the appropriate change of the recorded concentration. The system response time ( $t_{90}$ ) consists of the delay time to the measuring detector and the rise time of the detector.

For time alignment of the analyser and exhaust flow signals, the transformation time is defined as the time from the change ( $t_0$ ) until the response is 50 % of the final reading ( $t_{50}$ ).

The system response time shall be  $\leq 10$  s with a rise time  $\leq 2,5$  s in accordance with 6.1.7 for all limited components (CO, NO<sub>x</sub>, HC or NMHC) and all ranges used.

### 6.3.6 Calibration procedure

#### 6.3.6.1 Instrument assembly

Calibrate the instrument assembly and check the calibration curves against standard gases. The same gas flow rates shall be used as when sampling exhaust.

#### 6.3.6.2 Warming-up time

The warming-up time should be in accordance with the recommendations of the manufacturer. If not specified, a minimum of 2 h is recommended for warming up the analysers.

#### 6.3.6.3 Establishment of calibration curve

This shall be done using the following procedure.

- a) Calibrate each normally used operating range.
- b) Using purified synthetic air (or nitrogen), set the CO, CO<sub>2</sub>, NO<sub>x</sub> and HC analysers to zero.
- c) Introduce the appropriate calibration gases into the analysers, record the values and establish the calibration curve.

The calibration curve shall be established by at least six calibration points (excluding zero) approximately equally spaced over the operating range. The highest nominal concentration shall be greater than or equal to 90 % FS.

The calibration curve shall be calculated by the method of least-squares. A best-fit linear or non-linear equation may be used.

The calibration points shall not differ from the least-squares best-fit line by more than  $\pm 2$  % of reading or  $\pm 0,3$  % FS, whichever is the larger.

- d) Recheck the zero setting and repeat the calibration procedure, if necessary.

#### 6.3.6.4 Alternative methods

If it can be shown that alternative methods — computer, electronically controlled range switch, etc. — offer an equivalent accuracy, they may be used.

### 6.3.7 Verification of calibration curve

Each normally used operating range shall be checked prior to each engine test in accordance with the following procedure.

Check the calibration using a zero and span gas whose nominal value is more than 80 % FS of the measuring range.

If, for the two points considered, the value found does not differ by more than  $\pm 4$  % FS from the declared reference value, the adjustment parameters may be modified. Should this not be the case, verify the span gas or establish a new calibration curve in accordance with 6.3.6.3.

### 6.3.8 Calibration of tracer gas analyser for exhaust flow measurement

Calibrate the analyser for measurement of the tracer gas concentration using the standard gas.

The calibration curve shall be established by at least 10 calibration points (excluding zero) spaced so that half the calibration points are placed between 4 % to 20 % of the analyser's full scale and the rest are between 20 % FS to 100 % FS. The calibration curve is calculated by the method of least squares.

The calibration curve shall not differ by more than  $\pm 1$  % FS from the nominal value of each calibration point, in the range from 20 % FS to 100 % FS. It also shall not differ by more than  $\pm 2$  % of reading from the nominal value in the range from 4 % FS to 20 % FS.

Set the analyser to zero and span it prior to the test run using a zero and span gas with a nominal value of  $> 80$  % of the analyser's full scale.

### 6.3.9 Efficiency test of NO<sub>x</sub> converter

#### 6.3.9.1 General

The efficiency of the converter used for the conversion of NO<sub>2</sub> into NO shall be tested in accordance with 6.3.9.2 to 6.3.9.9 (see Figure 3).

#### 6.3.9.2 Test set-up

Using the test set-up as schematically shown in Figure 3 and the following procedure, test the efficiency of converters by means of an ozonator.

#### 6.3.9.3 Calibration

Calibrate the CLD and the HCLD in the most common operating range following the manufacturer's specifications using a zero and span gas (the NO content of which shall amount to about 80 % of the operating range and the NO<sub>2</sub> concentration of the gas mixture to less than 5 % of the NO concentration). The NO<sub>x</sub> analyser shall be in the NO mode so that the span gas does not pass through the converter. Record the indicated concentration.

#### 6.3.9.4 Calculation

The % efficiency of the NO<sub>x</sub> converter is calculated by

$$E_{\text{NO}_x} = \left( 1 + \frac{a - b}{c - d} \right) \times 100 \quad (34)$$

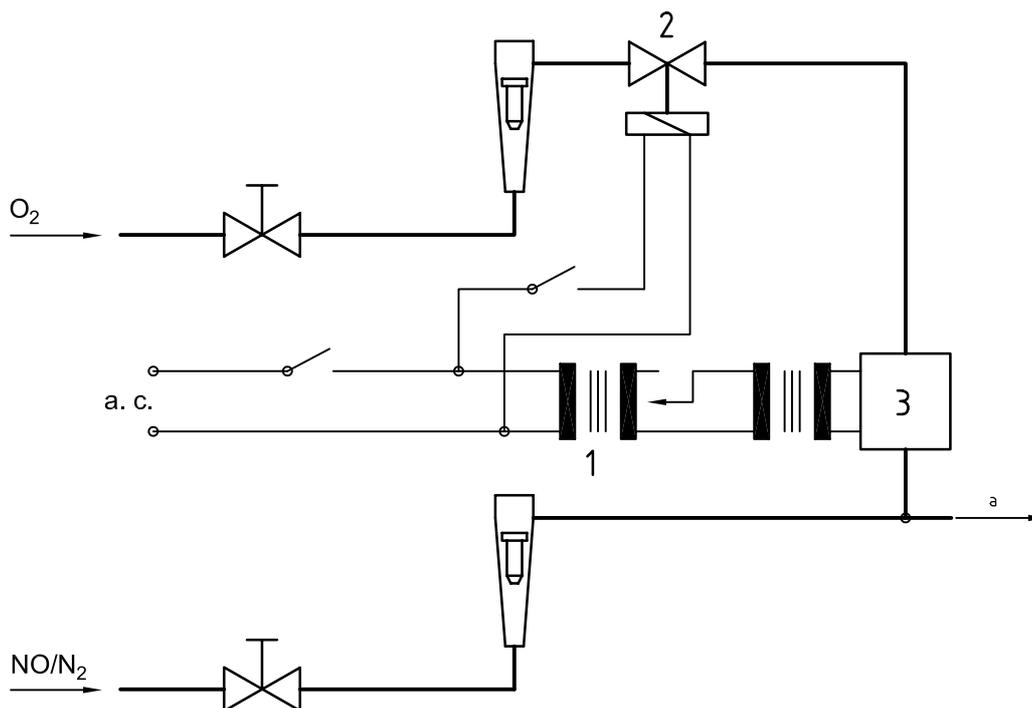
where

*a* is the NO<sub>x</sub> concentration in accordance with 6.3.9.7;

*b* is the NO<sub>x</sub> concentration in accordance with 6.3.9.8;

*c* is the NO concentration in accordance with 6.3.9.5;

*d* is the NO concentration in accordance with 6.3.9.6.



#### Key

- 1 Variac (variable transformer)
  - 2 Solenoid valve
  - 3 Ozonator
- a To analyser.

Figure 3 — Schematic of NO<sub>2</sub> converter efficiency device

#### 6.3.9.5 Adding oxygen

Via a T-fitting, add oxygen or zero air continuously to the gas flow until the indicated concentration is about 20 % less than the indicated calibration concentration from 6.3.9.3 (with the analyser in the NO mode).

Record the indicated concentration, *c*. The ozonator remains deactivated throughout the process.

#### 6.3.9.6 Activation of the ozonator

Activate the ozonator in order to generate enough ozone to bring the NO concentration down to about 20 % (minimum 10 %) of the calibration concentration given in 6.3.9.3. Record the indicated concentration, *d* (with the analyser in the NO mode).

#### 6.3.9.7 NO<sub>x</sub> mode

Switch the NO analyser to the NO<sub>x</sub> mode so that the gas mixture (consisting of NO, NO<sub>2</sub>, O<sub>2</sub> and N<sub>2</sub>) now passes through the converter. Record the indicated concentration, *a* (with the analyser in the NO<sub>x</sub> mode).

#### 6.3.9.8 Deactivation of ozonator

Deactivate the ozonator. The mixture of gases described in 6.3.9.7 will pass through the converter into the detector. Record the indicated concentration, *b* (with the analyser in the NO<sub>x</sub> mode).

#### 6.3.9.9 NO mode

Switching to NO mode with the ozonator deactivated also shuts off the flow of oxygen or synthetic air. The NO<sub>x</sub> reading of the analyser shall not deviate by more than  $\pm 5\%$  from the value measured in accordance with 6.3.9.3 (with the analyser in the NO mode).

#### 6.3.9.10 Test interval

Test the efficiency of the converter prior to each calibration of the NO<sub>x</sub> analyser.

#### 6.3.9.11 Efficiency requirement

The efficiency of the converter,  $E_{\text{NO}_x}$ , shall be not less than 90 %, but a higher efficiency of 95 % is strongly recommended.

If, with the analyser in the most common range, the ozonator cannot give a reduction from 80 % to 20 % in accordance with 6.3.9.6, then the highest range giving the reduction shall be used.

### 6.3.10 Adjustment of FID

#### 6.3.10.1 Optimization of detector response

The FID shall be adjusted as specified by the instrument manufacturer. A propane-in-air span gas should be used to optimize the response on the most common operating range.

- a) With the fuel and air flow rates set at the manufacturer's recommendations, introduce a 350 ppm (= 350  $\mu\text{l/l}$ ) C  $\pm$  75 ppm (= 75  $\mu\text{l/l}$ ) C span gas into the analyser. The response at a given fuel flow shall be determined from the difference between the span gas response and the zero gas response.
- b) Incrementally adjust the fuel flow above and below the manufacturer's specification. Record the span and zero response at these fuel flows. Plot the difference between the span and zero response and adjust the fuel flow to the rich side of the curve.

This is the initial flow rate setting, which may need further optimization depending on the results of the hydrocarbon response factors and the oxygen interference check specified in 6.3.10.2 and 6.3.10.3.

- c) If the oxygen interference or the hydrocarbon response factors do not meet the following specifications, incrementally adjust the air flow above and below the manufacturer's specifications, repeating 6.3.10.2 and 6.3.10.3 for each flow.

The optimization may optionally be conducted using the procedures outlined in SAE paper 770141.

#### 6.3.10.2 Hydrocarbon response factors

The analyser shall be calibrated using propane in air and purified synthetic air, in accordance with 6.3.6.

Response factors shall be determined when introducing an analyser into service and after major service intervals. The response factor,  $r_h$ , for a particular hydrocarbon species is the ratio of the FID C1 reading to the gas concentration in the cylinder expressed in parts per million (microlitres per litre) of C1.

The concentration of the test gas shall be at a level that gives a response of approximately 80 % FS. The concentration shall be known to an accuracy of  $\pm 2\%$  in reference to a gravimetric standard expressed in volume. In addition, the gas cylinder shall be preconditioned for 24 h at a temperature of 298 K  $\pm$  5 K (25 °C  $\pm$  5 °C).

The test gases to be used and the recommended relative response factor ranges are

- methane and purified synthetic air  $1,00 \leq r_h \leq 1,15$
- propylene and purified synthetic air  $0,90 \leq r_h \leq 1,1$
- toluene and purified synthetic air  $0,90 \leq r_h \leq 1,1$

These values are relative to an  $r_h$  of 1 for propane and purified synthetic air.

### 6.3.10.3 Oxygen interference check

The oxygen interference check shall be made when introducing an analyser into service and after major service intervals.

NOTE The oxygen interference check need only be conducted if the measurement is done in the raw exhaust gas.

A range shall be chosen where the oxygen interference check gases (see 6.3.2.5) will fall in the upper 50 %. The test shall be conducted with the oven temperature set as required. Oxygen interference check gas specifications shall be in accordance with 6.3.2.5.

- a) Set the analyser to zero.
- b) The analyser shall be spanned with the 0 % oxygen blend for gasoline-fuelled engines. Diesel engine instruments shall be spanned with the 21 % oxygen blend.
- c) Recheck the zero response. If it has changed by more than 0,5 % FS, repeat steps a) and b).
- d) Introduce the 5 % and 10 % oxygen interference check gases.
- e) Recheck the zero response. If it has changed by more than  $\pm 1$  % FS, repeat the test.
- f) Calculate the oxygen interference, %  $O_I$ , for each mixture given in step d), as follows:

$$\% O_I = (B - \text{analyser response}) \times 100 / B \quad (35)$$

$$\text{where analyser response is } (A / \% \text{ FS at } A) \times (\% \text{ FS at } B) \quad (36)$$

where

$A$  is the hydrocarbon concentration of the span gas used in step b), in parts per million (microlitres per litre) of C;

$B$  is the hydrocarbon concentration of the oxygen interference check gases used in step d), in parts per million (microlitres per litre) of C.

The oxygen interference shall be less than  $\pm 3,0$  % for all required oxygen interference check gases prior to testing.

- g) If the oxygen interference is greater than  $\pm 3,0$  %, incrementally adjust the air flow above and below the manufacturer's specifications, and repeat 6.3.10.1 for each flow.
- h) If the oxygen interference is greater than  $\pm 3,0$  % after adjusting the air flow, vary the fuel flow and, thereafter, the sample flow, and repeat 6.3.10.1 for each new setting.
- i) If the oxygen interference is still greater than  $\pm 3,0$  %, repair or replace the analyser, FID fuel or burner air prior to testing. Then repeat the procedure with the repaired or replaced equipment or gases.

**6.3.10.4 Methanol response factor,  $r_m$**

When the FID analyser is to be used for the analysis of hydrocarbons containing methanol, the methanol response factor,  $r_m$ , of the analyser shall be established.

Inject a known volume of methanol ( $a$ , in millilitres), using a microlitre syringe, into the heated mixing zone [395 K (122 °C)] of a septum injector, which is then vaporized and swept into a tedlar<sup>1)</sup> bag with a known volume of zero-grade air ( $b$ , in cubic metres). The air volume or volumes shall be such that the methanol concentration in the bag is representative of the range of concentrations found in the exhaust sample.

Analyse the bag sample using the FID, and calculate  $r_m$  as follows:

$$r_m = \frac{x_{FID}}{c_{SAM}} \tag{37}$$

where

$x_{FID}$  is the FID reading, in parts per million (microlitres per litre) of C;

$c_{SAM}$  is the methanol concentration in the sample bag, as calculated from  $a$  and  $b$ :  $c_{SAM} = 594 \times a/b$ , in parts per million (microlitres per litre) of C.

**6.3.10.5 Efficiency of non-methane cutter (NMC)**

**6.3.10.5.1 General**

The NMC is used for the removal of the non-methane hydrocarbons from the sample gas by oxidizing all hydrocarbons except methane. Ideally, the conversion for methane is 0 %, and for the other hydrocarbons represented by ethane, 100 %. For the accurate measurement of NMHC and CH<sub>4</sub>, the two efficiencies shall be determined and used for the calculation of the NMHC emission mass flow rate (see 5.5.4.4).

**6.3.10.5.2 Methane efficiency**

Methane calibration gas shall be flown through the FID with and without bypassing the NMC and the two concentrations recorded. The efficiency shall be determined as follows:

$$E_M = 1 - \frac{c_{HC(wCutter)}}{c_{HC(w/oCutter)}} \tag{38}$$

where

$c_{HC(wCutter)}$  is the HC concentration, with CH<sub>4</sub> flowing through the NMC, in parts per million (microlitres per litre) of C;

$c_{HC(w/oCutter)}$  is the HC concentration, with CH<sub>4</sub> bypassing the NMC, in parts per million (microlitres per litre) of C.

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1) Tedlar® is the trade name of a product supplied by DuPont. This information is given for the convenience of users of this International Standard and does not constitute an endorsement by ISO of the product named. Equivalent products may be used if they can be shown to lead to the same results.

### 6.3.10.5.3 Ethane efficiency

Ethane calibration gas shall be flown through the FID with and without bypassing the NMC and the two concentrations recorded. The efficiency shall be determined as follows:

$$E_E = 1 - \frac{c_{\text{HC(wCutter)}}}{c_{\text{HC(w/oCutter)}}} \quad (39)$$

where

$c_{\text{HC(wCutter)}}$  is the HC concentration with,  $\text{C}_2\text{H}_6$  flowing through the NMC, in parts per million (microlitres per litre) of C;

$c_{\text{HC(w/oCutter)}}$  is the HC concentration with,  $\text{C}_2\text{H}_6$  bypassing the NMC, in parts per million (microlitres per litre) of C.

### 6.3.11 Interference effects

#### 6.3.11.1 General

Gases other than the one being analysed can interfere with the reading in several ways. Positive interference occurs in NDIR instruments where the interfering gas gives the same effect as the gas being measured, but to a lesser degree. Negative interference occurs in NDIR instruments by the interfering gas broadening the absorption band of the measured gas, and in CLD instruments by the interfering gas quenching the reaction. The interference checks in 6.3.11.2 and 6.3.11.3 shall be performed prior to an analyser's initial use and after major service intervals.

#### 6.3.11.2 CO analyser interference check

Water and  $\text{CO}_2$  can interfere with the CO analyser performance. Therefore, a  $\text{CO}_2$  span gas having a concentration of 80 % FS to 100 % FS of the maximum operating range used during testing shall be bubbled through water at room temperature and the analyser response recorded. The analyser response shall not be more than 1 % FS for ranges greater than or equal to 300 ppm (= 300  $\mu\text{l/l}$ ) or more than 3 ppm (= 3  $\mu\text{l/l}$ ) for ranges below 300 ppm (= 300  $\mu\text{l/l}$ ).

#### 6.3.11.3 $\text{NO}_x$ analyser quench checks

The two gases of concern for CLD (and HCLD) analysers are  $\text{CO}_2$  and water vapour. Quench responses to these gases are proportional to their concentrations and therefore require test techniques in order to determine the quench at the highest expected concentrations experienced during testing.

##### 6.3.11.3.1 $\text{CO}_2$ quench check

Pass a  $\text{CO}_2$  span gas having a concentration of 80 % FS to 100 % FS of the maximum operating range through the NDIR analyser and record the  $\text{CO}_2$  value as  $A$ . Then dilute it approximately 50 % with a NO span gas and pass it through the NDIR and CLD, recording the  $\text{CO}_2$  and NO values as  $B$  and  $C$ , respectively. Then shut off the  $\text{CO}_2$  and pass only the NO span gas through the (H)CLD; record the NO value as  $D$ .

The percentage quench, which shall not be greater than 3 % FS, shall be calculated as follows:

$$E_{\text{CO}_2} = \left[ 1 - \left( \frac{C \times A}{D \times A - D \times B} \right) \right] \times 100 \quad (40)$$

where

- A* is the undiluted CO<sub>2</sub> concentration measured with NDIR, in percent by volume;
- B* is the diluted CO<sub>2</sub> concentration measured with NDIR, in percent by volume;
- C* is the diluted NO concentration measured with (H)CLD, in parts per million (microlitres per litre);
- D* is the undiluted NO concentration measured with (H)CLD, in parts per million (microlitres per litre).

Alternative methods of diluting and quantifying of CO<sub>2</sub> and NO span gas values, such as dynamic mixing/blending, may be used.

### 6.3.11.3.2 Water quench check

This check applies to wet gas concentration measurements only. Calculation of water quench shall consider dilution of the NO span gas with water vapour and scaling of water vapour concentration of the mixture to that expected during testing.

Pass a NO span gas having a concentration of 80 % FS to 100 % FS of the normal operating range through the (H)CLD and record the NO value as *D*. Then bubble the NO span gas through water at room temperature and pass it through the (H)CLD, recording the NO value as *C*. Determine the water temperature and record it as *F*. Determine the mixture's saturation vapour pressure corresponding to the bubbler water temperature (*F*) and record it as *G*. The water vapour concentration (in percent) of the mixture shall be calculated by

$$H = 100 \times (G / p_b) \quad (41)$$

and recorded as *H*. The expected diluted NO span gas (in water vapour) concentration shall be calculated by

$$D_e = D \times (1 - H / 100) \quad (42)$$

and recorded as *D<sub>e</sub>*. For diesel exhaust, the maximum exhaust water vapour concentration (in percent) expected during testing shall be estimated, under the assumption of a fuel H/C ratio of 1,8:1, from the maximum CO<sub>2</sub> concentration in the exhaust gas *A* as follows:

$$H_m = 0,9 \times A \quad (43)$$

This is then recorded as *H<sub>m</sub>*.

The percentage water quench, which shall not be greater than 3 %, shall be calculated as follows:

$$E_{H_2O} = 100 \times [(D_e - C) / D_e] \times (H_m / H) \quad (44)$$

where

- D<sub>e</sub>* is the expected diluted NO concentration, in parts per million (microlitres per litre);
- C* is the measured diluted NO concentration, in parts per million (microlitres per litre);
- H<sub>m</sub>* is the maximum water vapour concentration, in percent;
- H* is the actual water vapour concentration, in percent.

NOTE It is important that the NO span gas contain minimal NO<sub>2</sub> concentration for this check, since absorption of NO<sub>2</sub> in water has not been accounted for in the quench calculations.

### 6.3.12 Calibration intervals

The analysers shall be calibrated in accordance with 6.3.6 at least every three months, or whenever a system repair or change is made that could influence calibration.

## 6.4 Analytical system

### 6.4.1 General

The following provides an overall description of the recommended sampling and analysing systems. Since various configurations can produce equivalent results, exact conformance with the system shown in Figure 4 is not required. Components such as instruments, valves, solenoids, pumps, flow devices and switches may be used to provide additional information and coordinate the functions of the component systems. Other components not needed to maintain the accuracy on some systems may be excluded, provided their exclusion is based upon good engineering judgement.

NOTE Although fully adequate for measuring the gaseous components, full-flow CVS systems are not covered by this International Standard. Such systems are described in detail in the regulations listed in the Bibliography.

### 6.4.2 Description of the analytical system

An analytical system for the determination of the gaseous emissions in the raw exhaust gas (see Figure 4) is based on the use of

- an HFID or FID analyser for the measurement of hydrocarbons,
- NDIR analysers for the measurement of carbon monoxide and carbon dioxide, and
- an HCLD or CLD analyser for the measurement of the oxides of nitrogen.

The sample for all components should be taken with one sampling probe and internally split to the different analysers. Optionally, two sampling probes located in close proximity may be used. Care shall be taken that no unintended condensation of exhaust components (including water and sulfuric acid) occurs at any point of the analytical system.

### 6.4.3 Components (see Figure 4)

#### 6.4.3.1 Exhaust gas sampling probe (SP)

A stainless steel, straight, closed-end multi-hole probe is recommended. The inside diameter shall be not greater than the inside diameter of the sampling line. The wall thickness of the probe shall not be greater than 1 mm. There shall be a minimum of three holes in three different radial planes sized to sample approximately the same flow. The probe shall extend across at least 80 % of the diameter of the exhaust pipe. One or two sampling probes may be used.

#### 6.4.3.2 Heated pre-filter (HF1) — Optional

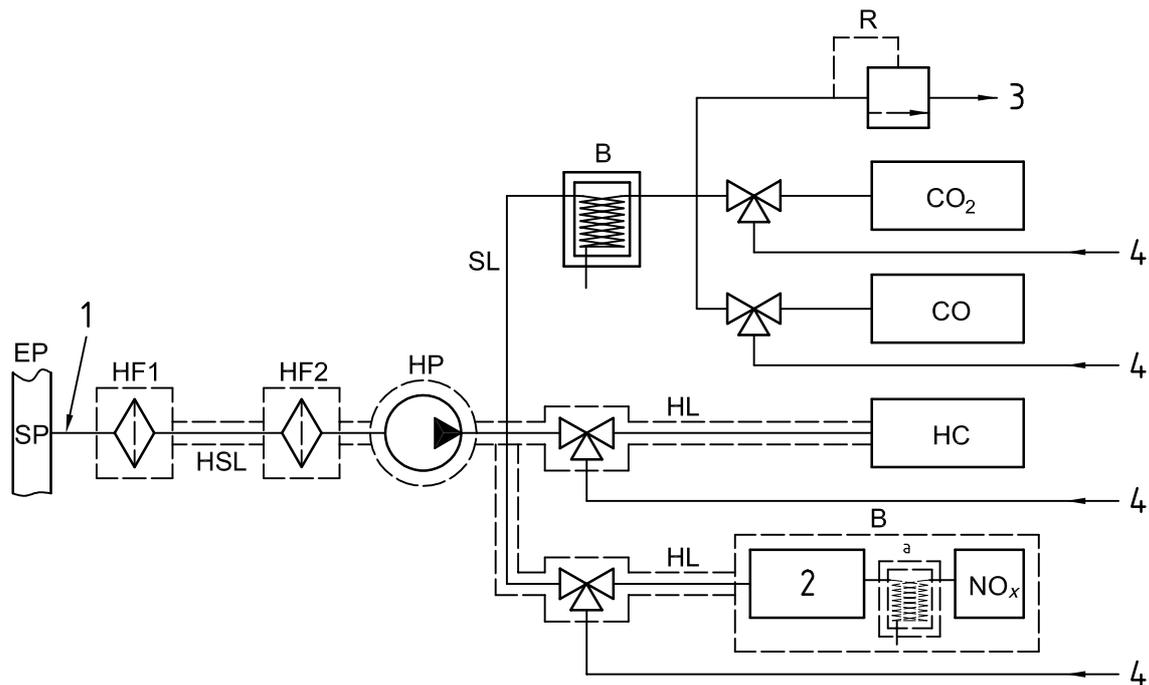
The temperature shall be the same as that for the heated sampling line (see 6.4.3.4).

#### 6.4.3.3 Heated filter (HF2)

This filter shall extract any solid particles from the gas sample prior to the analyser. The temperature shall be the same as the heated sampling line. The filter shall be changed as needed.

#### 6.4.3.4 Heated sampling line (HSL)

The heated sampling line provides a gas sample from a single probe to the split point or points and the HC analyser.



**Components**

- |                     |  |   |                           |
|---------------------|--|---|---------------------------|
| EP                  | Exhaust pipe                             | 1 | Test gas                  |
| SP                  | Exhaust gas sampling probe               | 2 | NO <sub>x</sub> converter |
| HF1                 | Heated pre-filter                        | 3 | Vent                      |
| HF2                 | Heated filter                            | 4 | Zero, span gas            |
| HSL                 | Heated sampling line                     |   |                           |
| HL                  | Heated NO <sub>x</sub> sampling line     |   |                           |
| HP                  | Heated sampling pump                     |   |                           |
| SL                  | Sampling line for CO and CO <sub>2</sub> |   |                           |
| HC                  | HFID analyser                            |   |                           |
| CO, CO <sub>2</sub> | NDIR analyser                            |   |                           |
| NO <sub>x</sub>     | CLD analyser                             |   |                           |
| B                   | Cooling bath                             |   |                           |
| R                   | Pressure regulator                       |   |                           |
| a                   | Optional                                 |   |                           |

**Figure 4 — Schematic flow diagram of exhaust gas analysis system for CO, CO<sub>2</sub>, NO<sub>x</sub>, HC**

The sampling line shall

- have a 4 mm minimum and a 13,5 mm maximum inside diameter,
- be made of stainless steel or PTFE,
- maintain a wall temperature of 463 K ± 10 K (190 °C ± 10 °C) as measured at every separately controlled heated section if the temperature of the exhaust gas at the sampling probe is ≤ 463 K (190 °C),
- maintain a wall temperature greater than 453 K (180 °C) if the temperature of the exhaust gas at the sampling probe is > 463 K (190 °C), and
- maintain a gas temperature of 463 K ± 10 K (190 °C ± 10 °C) immediately before the heated filter and the HFID.

**6.4.3.5 Heated NO<sub>x</sub> sampling line (HL)**

The sampling line shall

- maintain a wall temperature of between 328 K and 473 K (55 °C to 200 °C) up to the converter for dry measurement and up to the analyser for wet measurement, and
- be made of stainless steel or PTFE.

**6.4.3.6 Heated sampling pump (HP)**

This pump shall be heated to the temperature of HSL.

**6.4.3.7 Sampling line for CO and CO<sub>2</sub> (SL)**

The line shall be made of PTFE or stainless steel. It may be heated or unheated.

**6.4.3.8 HFID analyser (HC)**

The heated flame ionization detector (HFID) or flame ionization detector (FID) is used for determining the hydrocarbons. The temperature of the HFID shall be maintained at a temperature of between 453 K and 473 K (180 °C to 200 °C).

**6.4.3.9 NDIR analyser for CO and CO<sub>2</sub>**

NDIR analysers are for the determination of carbon monoxide and carbon dioxide, and are optional for determining the dilution ratio for PT measurement.

**6.4.3.10 CLD analyser (NO<sub>x</sub>)**

The CLD or HCLD analyser is for the determination of the oxides of nitrogen. If a HCLD is used, it shall be maintained at a temperature of between 328 K and 473 K (55 °C to 200 °C).

**6.4.3.11 Cooling bath (B) — Optional for NO measurement**

This is used to cool and condense water from the exhaust sample. It is optional if the analyser is free from water vapour interference in accordance with 6.3.11.3.2. If water is removed by condensation, the sample gas temperature or dew point shall be monitored either within the water trap or downstream. The sample gas temperature or dew point shall not exceed 280 K (7 °C). Chemical dryers shall not be used to remove water from the sample.

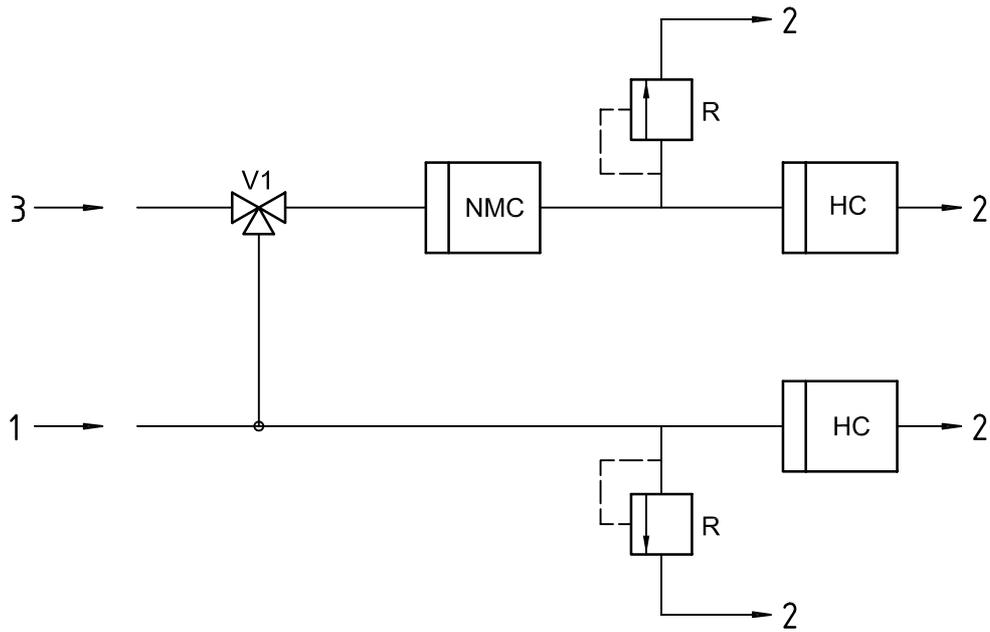
**6.4.3.12 Pressure regulator (R)**

This is used to control the pressure in the sampling lines and the flow to the analysers.

**6.4.4 NMC method****6.4.4.1 General**

The non-methane cutter oxidizes all hydrocarbons except CH<sub>4</sub> to CO<sub>2</sub> and H<sub>2</sub>O, so that by passing the sample through the NMC, only CH<sub>4</sub> is detected by the HFID. In addition to the usual HC sampling train (see 6.4.2 and Figure 4), a second HC sampling train shall be installed, equipped with a cutter as shown in Figure 5. This allows simultaneous measurement of total HC and NMHC.

The cutter shall be characterized at or above 600 K (327 °C) prior to test work with respect to its catalytic effect on CH<sub>4</sub> and C<sub>2</sub>H<sub>6</sub> at H<sub>2</sub>O values representative of exhaust stream conditions. The dewpoint and O<sub>2</sub> level of the sampled exhaust stream must be known. The relative response of the FID to CH<sub>4</sub> and C<sub>2</sub>H<sub>6</sub> shall be determined in accordance with 6.3.10.5.



**Components**

|     |                    |   |                       |
|-----|--------------------|---|-----------------------|
| NMC | Non-methane cutter | 1 | Sample                |
| HC  | HFID or FID        | 2 | Vent                  |
| V1  | Selector valve     | 3 | Zero gas and span gas |
| R   | Pressure regulator |   |                       |

**Figure 5 — Schematic flow diagram of methane analysis with the NMC**

**6.4.4.2 Components** (see Figure 5)

**6.4.4.2.1 Non-methane cutter (NMC)**

This is used to oxidize all hydrocarbons except methane.

**6.4.4.2.2 HFID or FID (HC)**

The heated flame ionization detector (HFID) or flame ionization detector (FID) is used to measure the HC and CH<sub>4</sub> concentrations. The temperature of the HFID shall be maintained at between 453 K and 473 K (180 °C to 200 °C).

**6.4.4.2.3 Selector valve (V1)**

This is used for selecting the zero gas and span gas.

**6.4.4.2.4 Pressure regulator (R)**

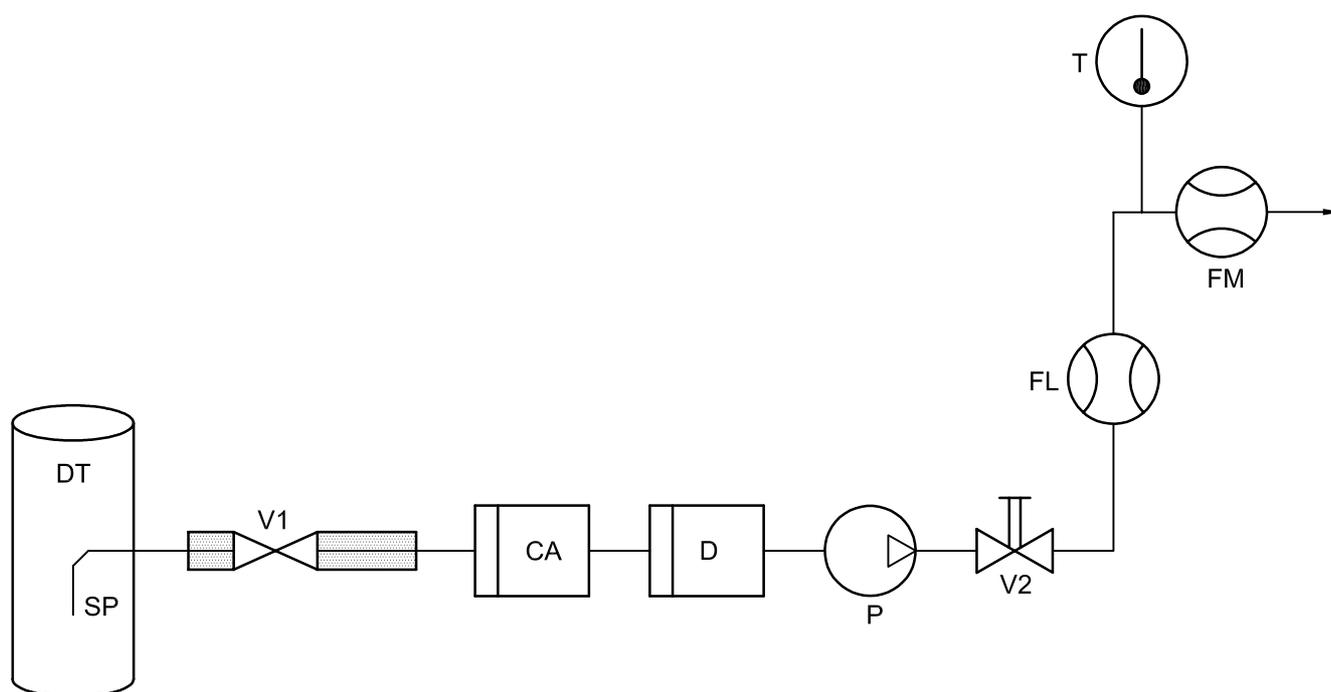
This is used to control the pressure in the sampling line and the flow to the HFID.

## 6.4.5 Formaldehyde analysis

### 6.4.5.1 General description

The exhaust sample is passed through two ice-cooled impingers placed in series containing an ACN solution of DNPH reagent or through a silica cartridge coated with 2,4-DNPH, as shown in Figure 6. An HCHO concentration in the collectors of at least 1 mg/l is recommended.

A small measured volume of the sample is injected onto an analytical column of the HPLC (high pressure liquid chromatograph) through which it is swept by an inert liquid under pressure. Separation, elution and detection of the components shall be in accordance with 6.4.5.3 and the instrument suppliers' instructions.



#### Components

|     |                         |
|-----|-------------------------|
| DT  | Dilution tunnel         |
| SP  | Sampling probe          |
| HSL | Heated sampling line    |
| CA  | Cartridge collector     |
| D   | Dryer (optional)        |
| P   | Sampling pump           |
| V1  | Solenoid valve          |
| V2  | Needle valve            |
| T   | Temperature sensor      |
| FL  | Flow meter (optional)   |
| FM  | Flow measurement device |

Figure 6 — Schematic flow diagram of formaldehyde sampling

**6.4.5.2 Components** (see Figure 6)

**6.4.5.2.1 Sampling probe (SP)**

The probe shall be in the same plane of the dilution tunnel as the sampling probes for the gaseous components (if applicable) and the particulates, but sufficiently distant from other probes and the tunnel to be free from the influence of any wakes or eddies.

**6.4.5.2.2 Heated sampling line (HSL)**

The temperature of the heated sampling line shall be between the maximum dewpoint of the mixture and 394 K (121 °C). Heating of the sampling line may be omitted, provided the sample collection system is close-coupled to the sampling pump, thereby preventing loss of sample due to cooling and resulting condensation in the line.

**6.4.5.2.3 Cartridge collector (CA)**

This collects the formaldehyde in the sample.

**6.4.5.2.4 Dryer (D) — Optional**

This removes water from the sample.

**6.4.5.2.5 Solenoid valve (V1)**

This directs the sample to the collection system.

**6.4.5.2.6 Needle valve (V2)**

This regulates the sample flow through the collection system.

**6.4.5.2.7 Temperature sensor (T)**

This monitors the temperature of the sample.

**6.4.5.2.8 Flow meter (FL) — Optional**

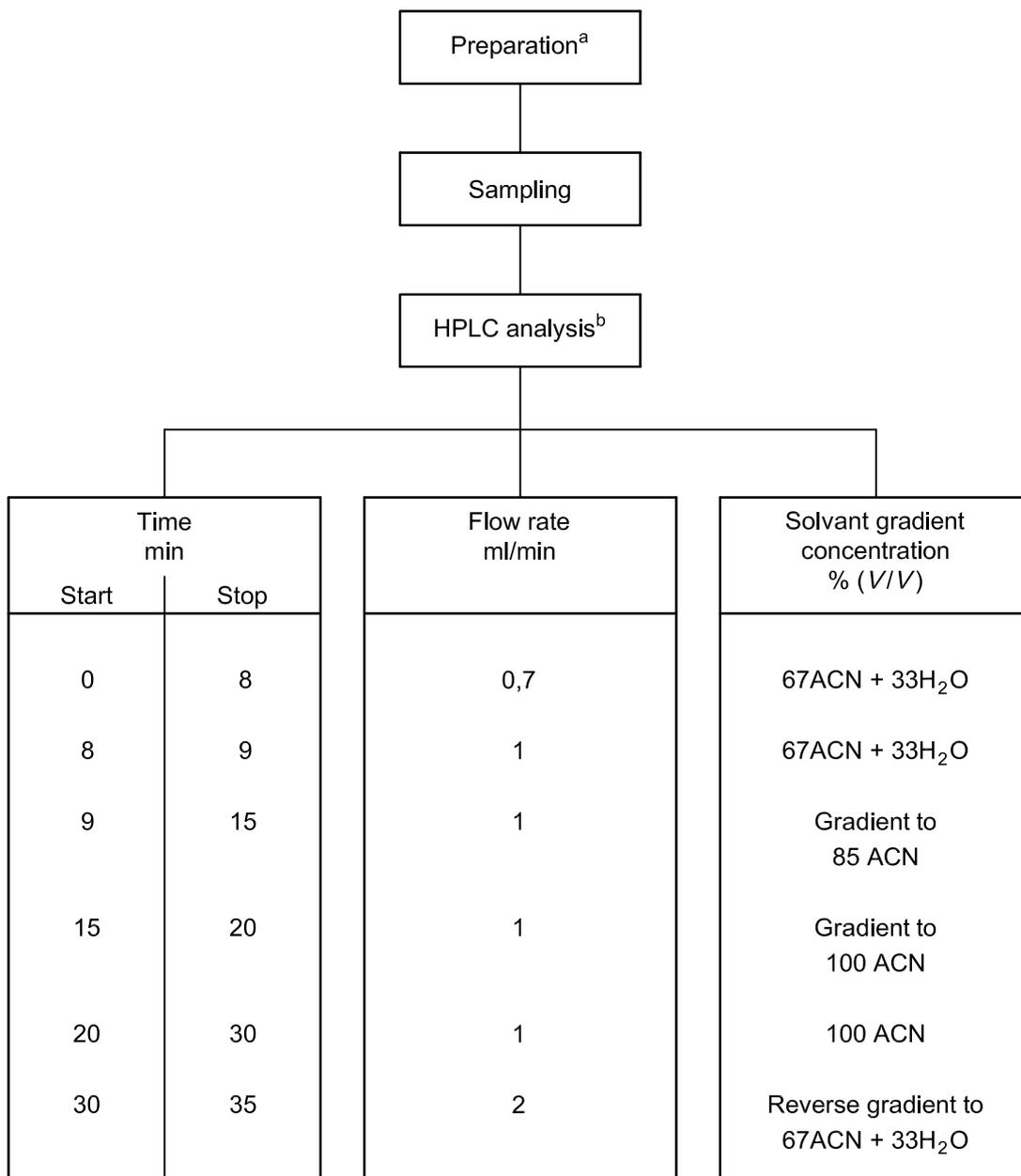
This measures the sample flow rate through the collection system.

**6.4.5.2.9 Flow measurement device (FM)**

This gas meter or other flow instrument measures the flow through the collection system during the sampling period.

**6.4.5.3 Gradient elution system**

A sample from the collector is injected into the HPLC, preferably not later than 24 h after the test. If it is not possible to perform the analysis within 24 h, the sample should be stored in a dark cold environment at 277 K to 283 K (4 °C to 10 °C) until analysis. HCHO is separated from the other carbonyl components by gradient elution (Figure 7) and detected with an UV detector at 365 nm. The HPLC is calibrated with standards of HCHO-DNPH derivatives. See SAE J 1936.



a DNP solutions standard

b HPLC 4,6 mm × 250 mm column 5 m Zorbax ODS<sup>2</sup>; 4,135 MPa (initial); UV 365 nm, sensitivity 0,2 AUFS.

**Figure 7 — Schematic representation of formaldehyde gradient elution**

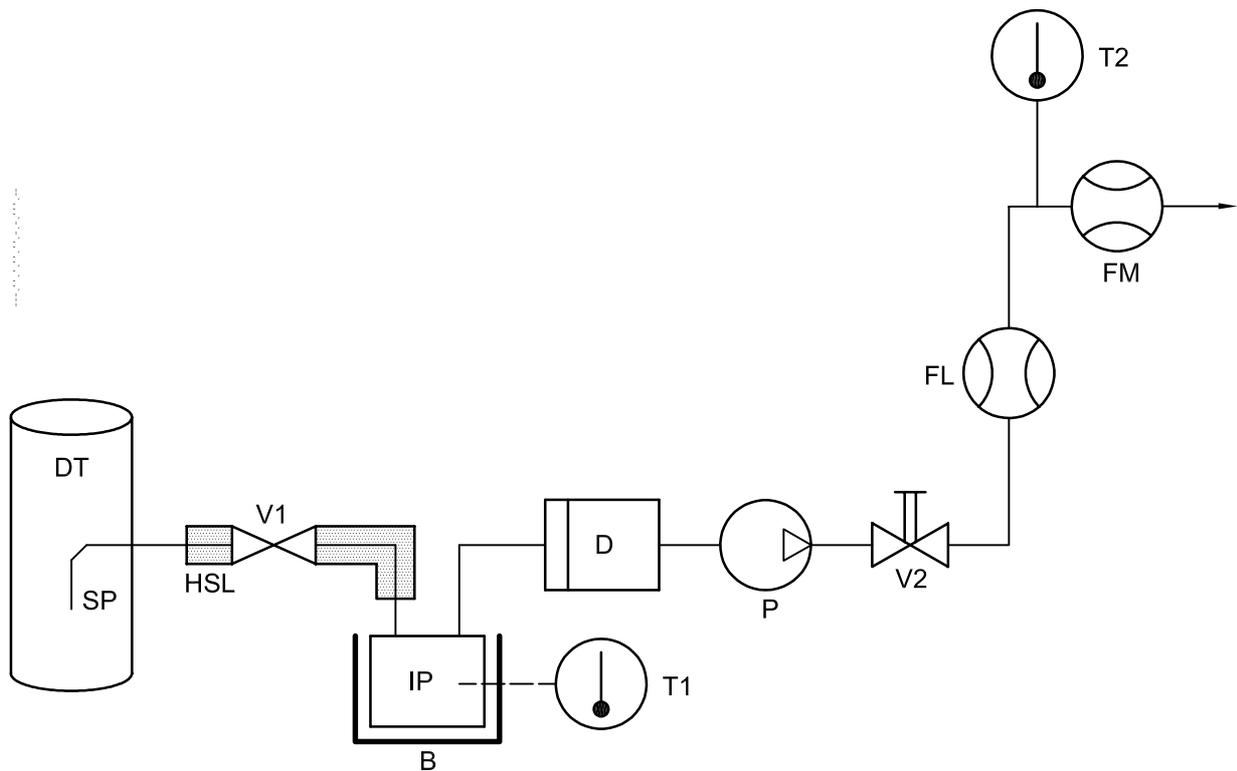
2) Zorbax ODS is an example of a suitable product available commercially.

6.4.6 Methanol analysis

6.4.6.1 General description

The exhaust sample is passed through two ice-cooled impingers placed in series containing deionized water, as shown in Figure 8. Sampling time and flow rate shall be such that a recommended CH<sub>3</sub>OH concentration of at least 1 mg/l can be reached in the primary impinger. The CH<sub>3</sub>OH concentration in the second impinger shall not be more than 10 % of the total amount collected. These requirements do not apply to background measurements.

A sample from the impingers is injected into the GC, preferably not later than 24 h after the test. If it is not possible to perform the analysis within 24 h, the sample should be stored in a dark, cold environment at 277 K to 283 K (4 °C to 10 °C) until analysis. CH<sub>3</sub>OH is separated from the other components and detected with an FID. The GC is calibrated with known amounts of CH<sub>3</sub>OH standards.



Components

- DT Dilution tunnel
- SP Sampling probe
- HSL Heated sampling line
- B Cooling bath
- IP Impinger
- D Dryer (optional)
- P Sampling pump
- V1 Solenoid valve
- V2 Needle valve
- T1, T2 Temperature sensors
- FL Flow meter (optional)
- FM Flow measurement device

Figure 8 — Schematic flow diagram of methanol sampling

#### **6.4.6.2 Components** (see Figure 8)

##### **6.4.6.2.1 Sampling probe (SP)**

The probe shall be in the same plane of the dilution tunnel (DT) as the sampling probes for the gaseous components, (if used), and the particulates, but sufficiently distant from other probes and the tunnel wall to be free from the influence of any wakes or eddies.

##### **6.4.6.2.2 Heated sampling line (HSL)**

The temperature of the heated sampling line shall be between the maximum dewpoint of the mixture and 394 K (121 °C). Heating of the sampling line may be omitted, provided the sample collection system is close-coupled to the sampling pump, thereby preventing loss of sample due to cooling and resulting condensation in the line.

##### **6.4.6.2.3 Impinger (IP)**

This collects methanol in the sample.

##### **6.4.6.2.4 Cooling bath (B)**

The impingers shall be cooled with ice or a refrigeration unit.

##### **6.4.6.2.5 Dryer (D) — Optional**

This removes water from the sample.

##### **6.4.6.2.6 Solenoid valve (V1)**

This directs the sample to the collection system.

##### **6.4.6.2.7 Needle valve (V2)**

This regulates the sample flow through the collection system.

##### **6.4.6.2.8 Temperature sensor (T1)**

This monitors the temperature of the cooling bath.

##### **6.4.6.2.9 Temperature sensor (T2)**

This monitors the temperature of the sample.

##### **6.4.6.2.10 Flow meter (FL) — Optional**

This measures the sample flow rate through the collection system.

##### **6.4.6.2.11 Flow measurement device (FM)**

This gas meter or other flow instrument measures the flow through the collection system during the sampling period.

## 7 Measurement equipment for particulates

### 7.1 Specifications

#### 7.1.1 General

To determine the mass of the particulates, a particulate sampling system, particulate sampling filters, a microgram balance, and a temperature and humidity controlled weighing chamber, are required. The particulate sampling system shall be designed to ensure a representative sample of the particulates proportional to the exhaust flow under transient engine operating conditions in accordance with 5.6.2.

#### 7.1.2 Particulate sampling filters

The diluted exhaust shall be sampled by a filter meeting the requirements of 7.1.2.1 and 7.1.2.2 during the test sequence.

##### 7.1.2.1 Filter specification

Fluorocarbon-coated glass-fibre filters or fluorocarbon membrane filters are required. All filter types shall have a 0,3 µm DOP (di-octylphthalate) collection efficiency of at least 99 % at a gas face velocity of between 35 cm/s and 100 cm/s.

##### 7.1.2.2 Filter size

Particulate filters with a diameter of 47 mm are recommended. Larger diameter filters are acceptable (7.1.2.4), but smaller diameter filters shall not be used.

##### 7.1.2.3 Filter face velocity

A gas face velocity through the filter of from 0,35 m/s to 1 m/s shall be achieved. The pressure drop increase between the beginning and the end of the test shall be no more than 25 kPa.

##### 7.1.2.4 Filter loading

The required minimum filter loadings for the most common filter sizes are given in Table 5. For larger filter sizes, the minimum filter loading shall be 0,065 mg/1 000 mm<sup>2</sup> filter area.

**Table 5 — Minimum filter loadings**

| Filter diameter<br>mm | Minimum loading<br>mg |
|-----------------------|-----------------------|
| 47                    | 0,11                  |
| 70                    | 0,25                  |
| 90                    | 0,41                  |
| 110                   | 0,62                  |

If, based on previous testing, the required minimum filter loading is unlikely to be reached on a test cycle after optimization of flow rates and dilution ratio, the test cycle may be run repeatedly on the same filter without removing it from the filter holder, provided there is agreement of the parties involved. Also, a lower filter loading may be acceptable, with the agreement of the parties involved, if it can be shown to meet the accuracy requirements of 7.1.3.2 (e.g. with a balance of 0,1 µg resolution).

### 7.1.3 Weighing chamber and analytical balance specifications

#### 7.1.3.1 Weighing chamber conditions

The temperature of the chamber (or room) in which the particulate filters are conditioned and weighed shall be maintained to within  $295 \text{ K} \pm 3 \text{ K}$  ( $22 \text{ }^\circ\text{C} \pm 3 \text{ }^\circ\text{C}$ ) during all filter conditioning and weighing. The humidity shall be maintained to a dewpoint of  $282,5 \text{ K} \pm 3 \text{ K}$  ( $9,5 \text{ }^\circ\text{C} \pm 3 \text{ }^\circ\text{C}$ ) and a relative humidity of  $45 \% \pm 8 \%$ .

#### 7.1.3.2 Reference filter weighing

The chamber (or room) environment shall be free of any ambient contaminants (such as dust) that might settle on the particulate filters during their stabilization. Disturbances to weighing room specifications as described in 7.1.3.1 are permitted if the duration of the disturbances does not exceed 30 min. The weighing room should meet the required specifications prior to personnel entering the weighing room. At least two unused reference filters shall be weighed within 4 h of, but preferably at the same time as, the sample filter weighing. They shall be the same size and material as the sample filters.

If the average mass of the reference filters changes between sample filter weighing by more than  $10 \mu\text{g}$ , then all sample filters shall be discarded and the emissions test repeated.

If the weighing room stability criteria outlined in clause 7.1.3.1 is not met, but the reference filter weighing meets the above criteria, the engine manufacturer has the option of accepting the sample filter masses, or voiding the tests, fixing the weighing room control system and rerunning the test.

#### 7.1.3.3 Analytical balance

The analytical balance used to determine the filter mass shall have a precision (standard deviation) of at least  $2 \mu\text{g}$  and a resolution of at least  $1 \mu\text{g}$  (1 digit =  $1 \mu\text{g}$ ), specified by the balance manufacturer.

#### 7.1.3.4 Elimination of static electricity effects

If unstable or irreproducible filter weighing is observed due to the effects of static electricity, the filters shall be neutralized prior to weighing, for example, by a Polonium neutralizer or similar device.

### 7.1.4 Specifications for flow measurement

#### 7.1.4.1 General

Absolute accuracies of flow meter or flow measurement instruments shall be in accordance with 5.3. For partial-flow dilution systems, the accuracy of the sample flow  $q_{mp}$  is of special concern if not measured directly but determined by differential flow measurement:

$$q_{mp} = q_{mdew} - q_{mdw} \quad (45)$$

In this case, an accuracy of  $\pm 2 \%$  for  $q_{mdew}$  and  $q_{mdw}$  is insufficient to guarantee acceptable accuracies of  $q_{mp}$ . If the gas flow is determined by differential flow measurement, the maximum error of the difference shall be such that the accuracy of  $q_{mp}$  is within  $\pm 5 \%$  when the dilution ratio is less than 15. It can be calculated by taking the root-mean-square of the errors of each instrument.

Acceptable accuracies of  $q_{mp}$  can be obtained by any one of the following methods:

- a) The absolute accuracies of  $q_{mdew}$  and  $q_{mdw}$  are  $\pm 0,2 \%$ , which guarantees an accuracy of  $q_{mp}$  of  $\leq 5 \%$  at a dilution ratio of 15. However, greater errors will occur at higher dilution ratios.
- b) Calibration of  $q_{mdw}$  relative to  $q_{mdew}$  is carried out such that the same accuracies for  $q_{mp}$  as in a) are obtained (see 7.3.2).

- c) The accuracy of  $q_{mp}$  is determined indirectly from the accuracy of the dilution ratio as determined by a tracer gas (e.g. CO<sub>2</sub>). Again, accuracies equivalent to method a) for  $q_{mp}$  are required.
- d) The absolute accuracy of  $q_{mdew}$  and  $q_{mdw}$  is within  $\pm 2\%$  of full scale, the maximum error of the difference between  $q_{mdew}$  and  $q_{mdw}$  is within 0,2 %, and the linearity error is within  $\pm 0,2\%$  of the highest  $q_{mdew}$  observed during the test.

**7.1.4.2 Correction for sample flow**

If measurement of methanol or formaldehyde is done with a total sampling type partial-flow dilution system, it is necessary to extract a sample flow  $q_{mex}$  from the tunnel, i.e. before the flow measuring device for  $q_{mdew}$ . Then,  $q_{mex}$  shall be determined with the flow measurement device (FM) shown in Figures 6 and 8, and will usually be much smaller than  $q_{mdew}$ , but not negligible ( $q_{mex} > 0,01 \times q_{mdew}$ ).

In order to guarantee an accuracy of  $q_{mp}$  in accordance with 7.1.4 and 7.3.2.1, any of the methods given here may be used, but with  $q_{mdew}$  replaced by  $q_{mdew} + q_{mex}$ .

For the calculation of the PM mass emission (5.6.5), the mass of  $q_{mex}$  extracted from the tunnel shall be taken into account by correcting the particulate mass  $m_f$ , as follows:

$$m_{f,corrected} = m_f \times \frac{q_{mdew}}{(q_{mdew} - q_{mex})} \tag{46}$$

**7.1.5 Additional specifications**

All parts of the dilution system and the sampling system — from the exhaust pipe to the filter holder — in contact with raw and diluted exhaust gas shall be designed to minimize deposition or alteration of the particulates. All parts shall be made of electrically conductive materials that do not react with exhaust gas components, and shall be electrically grounded to prevent electrostatic effects.

**7.2 Dilution and sampling system**

**7.2.1 General**

The following (see Figures 9 to 11), provides a general description of the recommended dilution and sampling systems. Since various configurations can produce equivalent results, exact conformance with the figures is not required. Additional components such as instruments, valves, solenoids, pumps and switches may be used to provide additional information and coordinate the functions of the component systems. Other components not needed to maintain the accuracy on some systems may be excluded if their exclusion is based upon good engineering judgment.

NOTE Although fully adequate for measuring particulates, full-flow CVS systems are not specified by this International Standard. Such systems are described in detail in the regulations listed in the bibliography.

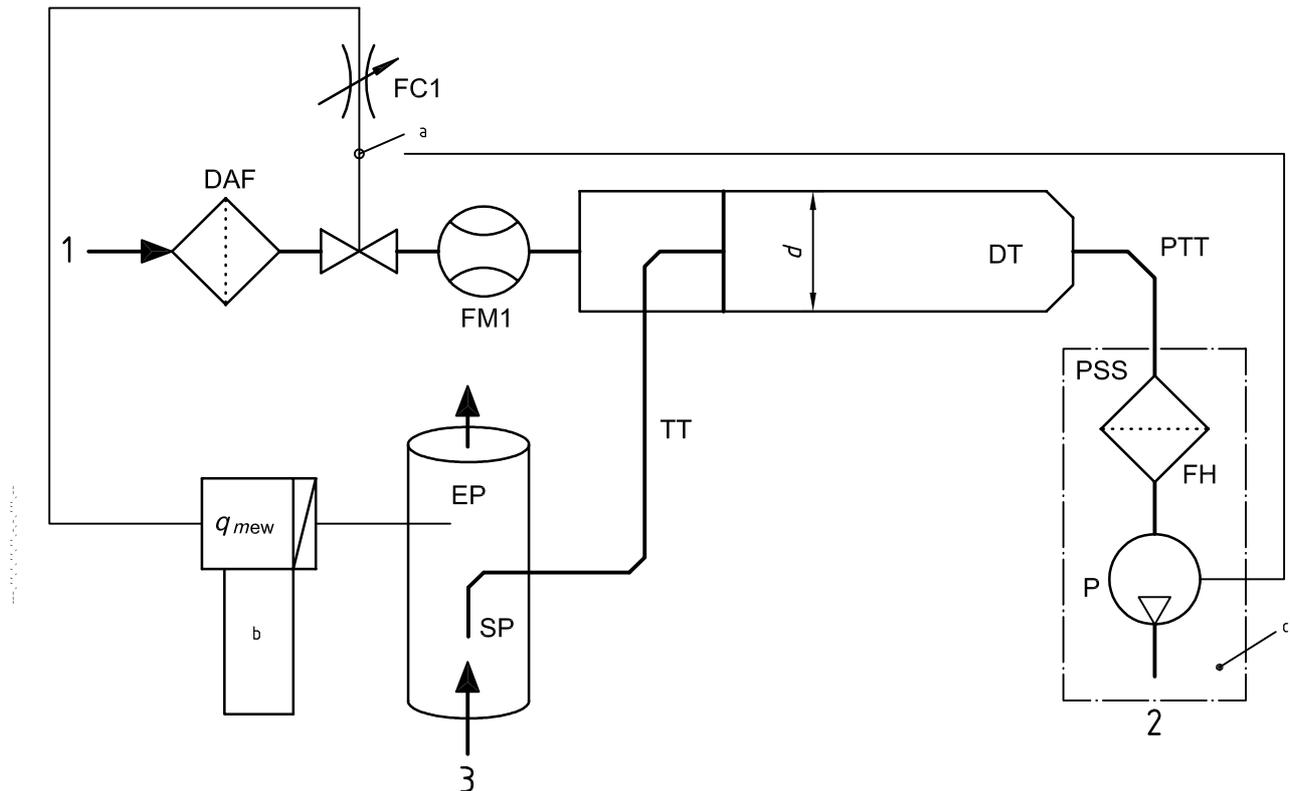
**7.2.2 Partial-flow system**

The dilution system described here is based upon the dilution of part of the exhaust stream. Splitting of the exhaust stream and the following dilution process may be done by different dilution system types. For subsequent collection of the particulates, the entire dilute exhaust gas or only a portion of the dilute exhaust gas is passed to the particulate sampling system. The first method is referred to as being a total sampling type, the second method as being a fractional sampling type. The calculation of the dilution ratio depends upon the type of system used.

With the total sampling system as shown in Figure 9, raw exhaust gas is transferred from the exhaust pipe (EP) to the dilution tunnel (DT) through the sampling probe (SP) and the transfer tube (TT). The total flow through the tunnel is adjusted with the flow controller (FC2) and the sampling pump (P) of the particulate sampling system (see Figure 11). The dilution air flow is controlled by the flow controller (FC1), which may use  $q_{mew}$  or  $q_{maw}$  and  $q_{mf}$  as command signals, for the desired exhaust split. The sample flow into DT is the difference of the total flow and the dilution air flow. The dilution air flow rate is measured with the flow measurement device (FM1), and the total flow

rate with the flow measurement device (FM3), of the particulate sampling system (see Figure 11). The dilution ratio is calculated from these two flow rates.

With the fractional sampling system as shown in Figure 10, raw exhaust gas is transferred from the exhaust pipe (EP) to the dilution tunnel (DT) through the sampling probe (SP) and the transfer tube (TT). The total flow through the tunnel is adjusted with the flow controller (FC1) connected either to the dilution air flow or to the suction blower for the total tunnel flow. The FC1 may use  $q_{mew}$  or  $q_{maw}$  and  $q_{mf}$  as command signals for the desired exhaust split. The sample flow into DT is the difference of the total flow and the dilution air flow. The dilution air flow rate is measured with the flow measurement device FM1, the total flow rate with the flow measurement device FM2. The dilution ratio is calculated from these two flow rates. From DT, a particulate sample is taken with the particulate sampling system (see Figure 11).



**Components**

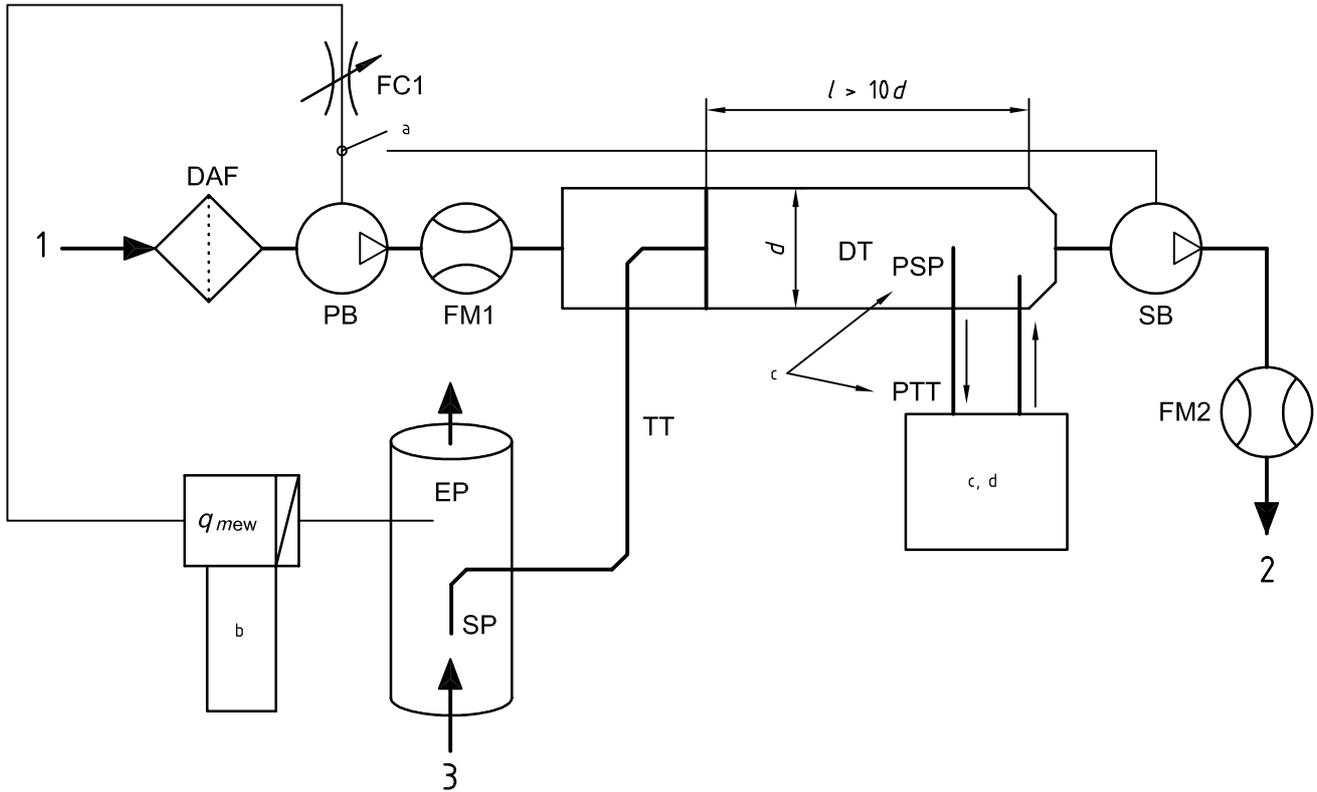
|     |                             |   |                                     |
|-----|-----------------------------|---|-------------------------------------|
| EP  | Exhaust pipe                | 1 | Air                                 |
| SP  | Sampling probe              | 2 | Vent                                |
| TT  | Exhaust transfer tube       | 3 | Exhaust                             |
| FC1 | Flow controller             | d | See 7.2.3.10 for required diameter. |
| FM1 | Flow measurement device     |   |                                     |
| DAF | Dilution air filter         |   |                                     |
| DT  | Dilution tunnel             |   |                                     |
| PTT | Particulate transfer tube   |   |                                     |
| FH  | Filter holder               |   |                                     |
| PSS | Particulate sampling system |   |                                     |
| P   | Sampling pump               |   |                                     |

a Optional to P, PSS.

b Or  $q_{maw}$  and  $q_{mf}$

c See Figure 11.

**Figure 9 — Schematic of partial-flow dilution system (total sampling type)**

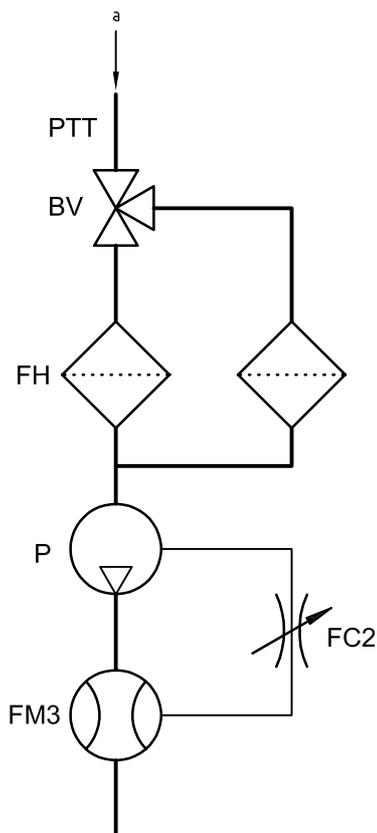


**Components**

- |     |                             |          |                                     |
|-----|-----------------------------|----------|-------------------------------------|
| EP  | Exhaust pipe                | 1        | Air                                 |
| SP  | Sampling probe              | 2        | Vent                                |
| TT  | Exhaust transfer tube       | 3        | Exhaust                             |
| FC1 | Flow controller             | <i>d</i> | See 7.2.3.10 for required diameter. |
| FM1 | Flow measurement device     |          |                                     |
| DAF | Dilution air filter         |          |                                     |
| DT  | Dilution tunnel             |          |                                     |
| PTT | Particulate transfer tube   |          |                                     |
| FH  | Filter holder               |          |                                     |
| PSS | Particulate sampling system |          |                                     |
| FM2 | Flow measurement device     |          |                                     |
| PB  | Pressure blower             |          |                                     |
| SB  | Suction blower              |          |                                     |

- a To PB or SB.
- b Or  $q_{maw}$  and  $q_{mf}$
- c See Figure 11.
- d To particulate sampling system.

**Figure 10 — Schematic of partial-flow dilution system (fractional sampling type)**



### Components

|     |                         |     |                           |
|-----|-------------------------|-----|---------------------------|
| FM3 | Flow measurement device | FH  | Filter holder             |
| P   | Sampling pump           | BV  | Ball valve                |
| FC2 | Flow controller         | PTT | Particulate transfer tube |

<sup>a</sup> From dilution tunnel. See Figure 9 or 10.

**Figure 11 — Schematic of particulate sampling system**

## 7.2.3 Components (see Figures 9 to 11)

### 7.2.3.1 Exhaust pipe (EP)

The exhaust pipe may be insulated. Reducing the thermal inertia of the exhaust pipe by a thickness to a diameter ratio of  $\leq 0,015$  is recommended. The use of flexible sections shall be limited to a length to diameter ratio of  $\leq 12$ . Bends shall be minimized to reduce inertial deposition. If the system includes a test bed silencer, the silencer may also be insulated. A straight pipe of six pipe diameters upstream and three pipe diameters downstream of the tip of the probe is recommended.

### 7.2.3.2 Sampling probe (SP)

The type of probe shall be either

- an open tube facing upstream on the exhaust pipe centreline, or
- an open tube facing downstream on the exhaust pipe centreline, or
- a multiple hole probe (SP in Figure 4), or
- a hatted probe facing upstream on the exhaust pipe centreline (see Figure 12).

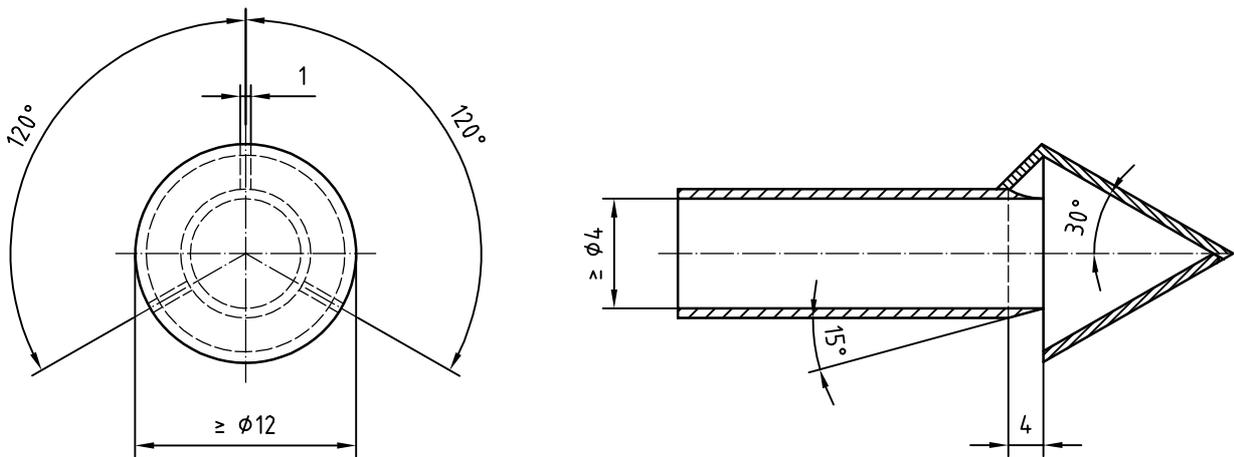


Figure 12 — Hatted probe

The minimum inside diameter of the probe tip shall be 4 mm. The minimum diameter ratio between exhaust pipe and probe shall be 4 for probes b) to d). For probe a), the exhaust pipe diameter, the probe diameter and the flow rates shall be set to allow a maximum sampling error of  $0,98 \leq P \leq 1,02$ , determined in accordance with Annex B.

When using an open tube facing upstream on the exhaust pipe centreline, special caution should be taken with regard to the impact of large particles unrelated to combustion products from the test cycle. Such particles may easily enter the probe, resulting in an erroneous PM measurement. A typical example of such particles unrelated to the actual test are particles from the muffler or particles re-entrained from the exhaust pipe into the exhaust flow. Therefore, an inertial preclassifier (cyclone or impactor) with a 50 % cut point of between 2,5 µm and 10 µm, installed immediately upstream of the filter holder, is recommended.

### 7.2.3.3 Exhaust transfer tube (TT)

The transfer tube shall be

- as short as possible, but not more than 1 m in length,
- greater than or equal to the probe diameter, but not more than 25 mm in diameter, and
- shall exit on the centreline of the dilution tunnel and be pointing downstream.

The tube shall be insulated with material having a maximum thermal conductivity of 0,05 W/(m · K) with a radial insulation thickness corresponding to the diameter of the probe, or shall be heated.

### 7.2.3.4 Flow controller (FC1)

A flow controller may be used to control the flow of the pressure blower or the suction blower or both. It may be connected to the exhaust flow sensor signals specified in 5.4. The flow controller may be installed either upstream or downstream of the respective blower. When using a pressurized air supply, the flow controller directly controls the air flow.

### 7.2.3.5 Flow measurement device (FM1)

This is a gas meter or other flow instrument used for measuring the dilution air flow. It is optional if the pressure blower is calibrated to measure the flow.

**7.2.3.6 Dilution air filter (DAF)**

It is recommended that the dilution air be filtered and charcoal scrubbed to eliminate background hydrocarbons. The dilution air shall have a temperature higher than 288 K (15 °C) and may be dehumidified. The dilution air may be sampled in accordance with good engineering practice to determine the background particulate levels, which can then be subtracted from the values measured in the diluted exhaust (see 5.6.4).

**7.2.3.7 Flow measurement device (FM2) — Fractional sampling type** (Figure 10 only)

A gas meter or other flow instrumentation for measuring the diluted exhaust gas flow, FM2 is optional if the suction blower is calibrated to measure the flow.

**7.2.3.8 Pressure blower (PB) — Fractional sampling type** (Figure 10 only)

This is used to control the dilution air flow rate. It may be connected to flow controllers FC1 or FC2, but is not required when using a butterfly valve. The pressure blower may be used to measure the dilution air flow, if calibrated.

**7.2.3.9 Suction blower (SB) — Fractional sampling type** (Figure 10 only)

For fractional sampling systems only, this may be used to measure the diluted exhaust gas flow, if calibrated.

**7.2.3.10 Dilution tunnel (DT)**

The dilution tunnel

- shall be of a sufficient length to completely mix the exhaust and dilution air under turbulent flow conditions for a fractional sampling system (complete mixing is not required for a total sampling system),
- shall be constructed of stainless steel,
- shall be at least 75 mm in diameter for the fractional sampling type,
- is recommended to be at least 25 mm in diameter for the total sampling type,
- may be heated to a maximum 325 K (52 °C) wall temperature, and
- may be insulated.

**7.2.3.11 Particulate sampling probe (PSP) — Fractional sampling type** (Figure 10 only)

The particulate sampling probe, which is the leading section of the particulate transfer tube (PTT),

- shall be installed facing upstream at a point where the dilution air and exhaust gas are well mixed, i.e. on the dilution tunnel (DT) centreline approximately 10 tunnel diameters downstream of the point where the exhaust enters the dilution tunnel,
- shall have a minimum inside diameter of 12 mm,
- may be heated to a maximum 325 K (52 °C) wall temperature, and
- may be insulated.

#### 7.2.3.12 Particulate transfer tube (PTT)

The particulate transfer tube shall not exceed 1 020 mm in length, and shall be minimized in length whenever possible.

The dimensions are valid for both

- the fractional sampling type from the probe tip to the filter holder, and
- the total sampling type from the end of the dilution tunnel to the filter holder.

The transfer tube

- may be heated to a maximum 325 K (52 °C) wall temperature, and
- may be insulated.

#### 7.2.3.13 Filter holder (FH)

The filter holder

- may be heated to a maximum 325 K (52 °C) wall temperature, and
- may be insulated.

An inertial preclassifier with a 50 % cut point between 2,5 µm and 10 µm may be installed immediately upstream of the filter holder. The use of the preclassifier is strongly recommended if an open tube sampling probe facing upstream is used.

#### 7.2.3.14 Sampling pump (P) — Particulate sampling system (Figure 11 only)

The sampling pump shall be used for drawing the particulate sample from the exhaust gas.

#### 7.2.3.15 Flow controller (FC2) — Particulate sampling system (Figure 11 only)

This flow controller shall be used for controlling the particulate sample flow rate.

#### 7.2.3.16 Flow measurement device (FM3) — Particulate sampling system (Figure 11 only)

A gas meter or flow instrument used for determining the particulate sample flow through the particulate filter, the flow measurement device FM3 may be installed upstream or downstream of the sampling pump.

#### 7.2.3.17 Ball valve (BV) — Optional — Particulate sampling system (Figure 11 only)

The ball valve shall have an inside diameter not less than the inside diameter of the particulate transfer tube, and a switching time of < 0,5 s.

If the ambient temperature in the vicinity of PSP, PTT, SDT and FH is < 293 K (20 °C), precautions should be taken to avoid particle losses on to the cool wall of these parts. Therefore, heating or insulating of these parts, or both, within the limits given in the respective descriptions, is recommended. It is also recommended that the filter face temperature during sampling be > 293 K (20 °C).

### 7.3 Calibration

#### 7.3.1 General

The calibration of the particulate measurement is limited to the flow meters used to determine sample flow and dilution ratio. Each flow meter shall be calibrated as often as is necessary to fulfil the accuracy requirements of this International Standard. The calibration method to be used shall be in accordance with 7.3.2.

## 7.3.2 Flow measurement

### 7.3.2.1 Periodical calibration

In order to fulfil the absolute accuracy of the flow measurements specified in 5.3, the flow meter or flow measurement instrument shall be calibrated with an accurate flow meter traceable to international or national standards or both.

If the sample gas flow is determined by differential flow measurement, the flow meter or flow measurement instrument shall be calibrated using one of the following procedures, such that the probe flow  $q_{mp}$  into the tunnel fulfils the accuracy requirements of 7.1.4.

- Connect the flow meter for  $q_{mdw}$  in series to the flow meter for  $q_{mdew}$ , and calibrate the difference between the two flow meters for at least five set points with flow values equally spaced between the lowest  $q_{mdw}$  value used during the test and the value of  $q_{mdew}$  used during the test. The dilution tunnel may be bypassed.
- Connect a calibrated mass flow device in series to the flow meter for  $q_{mdew}$  and check the accuracy for the value used for the test. Connect the calibrated mass flow device in series to the flow meter for  $q_{mdw}$ , and check the accuracy for at least five settings corresponding to a dilution ratio between 3 and 50, relative to  $q_{mdew}$  used during the test.
- Disconnect the transfer tube from the exhaust, and connect a calibrated flow measuring device with a suitable range to measure  $q_{mp}$  to the transfer tube. Then set  $q_{mdew}$  to the value used during the test, and set  $q_{mdw}$  sequentially to at least five values corresponding to dilution ratios  $q$  between 3 and 50. Alternatively, a special calibration flow path may be provided, in which the tunnel is bypassed but the total and dilution air flow passed through the corresponding meters as in the actual test.
- Feed a tracer gas into the exhaust transfer tube. This tracer gas may be a component of the exhaust gas, such as  $CO_2$  or  $NO_x$ . After dilution in the tunnel, measure the tracer gas component. This shall be carried out for five dilution ratios between 3 and 50. The accuracy of the sample flow shall be determined from the dilution ratio  $r_d$  as

$$q_{mp} = q_{mdew} / r_d \quad (47)$$

The accuracies of the gas analysers shall be taken into account in order to guarantee the accuracy of  $q_{mp}$ .

### 7.3.2.2 Carbon flow check

A carbon flow check using actual exhaust is strongly recommended for detecting measurement and control problems and verifying the proper operation of the partial flow system. The carbon flow check should be run at least each time a new engine is installed, or whenever there is a significant change in the test cell configuration.

The engine shall be operated at peak torque load and speed or any other steady-state mode that produces  $\geq 5\%$  of  $CO_2$ . The partial flow sampling system shall be operated with a dilution factor of about 15 to 1.

If a carbon flow check is conducted, the procedure given in Annex C shall be applied. The carbon flow rates  $q_{mCf}$ ,  $q_{mCe}$  and  $q_{mCp}$  shall be calculated in accordance with equations C.1, C.2 and C.3, respectively. All carbon flow rates should agree to within 6%.

### 7.3.2.3 Pre-test check

A pre-test check shall be performed within 2 h before the test run, as follows.

Check the accuracy of the flow meters by the same method used for calibration (see 7.3.2.1) for at least two points, including flow values of  $q_{mdw}$  corresponding to dilution ratios between five and 15 for the  $q_{mdew}$  value used during the test.

If it can be demonstrated by records of the calibration procedure in accordance with 7.3.2.1 that the flow meter calibration is stable over a longer period of time, the pre-test check may be omitted.

### 7.3.3 Determination of transformation time

The system settings for the transformation time evaluation shall be exactly the same as those during measurement of the test run. The transformation time shall be determined by the following procedure.

- a) Place an independent reference flow meter with a measurement range appropriate for the probe flow in series with, and closely coupled to, the probe. This flow meter shall have a transformation time of less than 100 ms for the flow step size used in the response time measurement, with flow restriction sufficiently low not to affect the dynamic performance of the partial-flow dilution system, and consistent with good engineering practice.
- b) Introduce a step change to the exhaust flow (or air flow if exhaust flow is calculated) input of the partial-flow dilution system, from a low flow to at least 90 % FS. The trigger for the step change should be the same as the one used to start the look-ahead control in actual testing. Record exhaust flow step stimulus and the flow meter response at a sample rate of at least 10 Hz.
- c) From this data, determine the transformation time for the partial-flow dilution system, which is the time from the initiation of the step stimulus to the 50 % point of the flow meter response. In similar manner, determine the transformation times of the  $q_{mp}$  signal of the partial-flow dilution system and of the  $q_{mew,i}$  signal of the exhaust flow meter. These signals are used in the regression checks performed after each test (see 5.6.3).
- d) Repeat the calculation for at least 5 rise-and-fall stimuli, and average the results. Subtract the internal transformation time (< 100 ms) of the reference flow meter from this value. This is the look-ahead value of the partial-flow dilution system, which shall be applied in accordance with 5.6.3.

### 7.3.4 Calibration intervals

The instruments shall be calibrated as required by internal audit procedures or the instrument manufacturer.

## Annex A (normative)

### Determination of system equivalence

In accordance with 5.2, other systems or analysers (candidates) may be accepted if they yield equivalent results. The determination of system equivalency shall be based on a seven-sample pair (or larger) correlation study between the candidate system and one of the accepted reference systems of this International Standard using the appropriate test cycle. The equivalency criterion to be applied shall be the two-sided Student  $t$  test.

This statistical method examines the hypothesis that the population mean value for an emission measured with the candidate system does not differ from the population mean value for that emission measured with the reference system. The hypothesis shall be tested on the basis of a 1 % significance level of the  $t$  value. The  $t$  values for seven to 10 sample pairs are given in Table A.1. If the  $\tau$  value calculated in accordance with Equation A.1 is greater than the tabulated  $t$  value, the candidate system is not equivalent.

The following procedure shall be followed. The subscripts R and C refer to the reference and candidate system, respectively.

- a) Conduct at least seven tests with the candidate and reference systems, preferably operated in parallel. The number of tests is referred to as  $n_R$  and  $n_C$ .
- b) Calculate the mean values  $x_R$  and  $x_C$  and the standard deviations  $s_R$  and  $s_C$ .
- c) Calculate the test value  $\tau$ , as follows:

$$\tau = \frac{x_C - x_R}{\sqrt{(n_C - 1) \times s_C^2 + (n_R - 1) \times s_R^2}} \times \sqrt{\frac{n_C \times n_R \times (n_C + n_R - 2)}{n_C + n_R}} \quad (\text{A.1})$$

- d) Compare  $\tau$  with the tabulated  $t$  value corresponding to the respective number of tests given in Table A.1. If larger sample sizes are selected, calculate the degrees of freedom as  $(n_C + n_R - 2)$  and the  $t$  value taken from statistical tables for 1 % significance (99 % confidence).

**Table A.1 —  $t$  value for selected sample sizes**

| Sample size | Degrees of freedom | $t$ value |
|-------------|--------------------|-----------|
| 7           | 12                 | 3,055     |
| 8           | 14                 | 2,977     |
| 9           | 16                 | 2,921     |
| 10          | 18                 | 2,878     |

- e) Determine the equivalency, as follows:
  - 1) if  $\tau < t$ , then the candidate system is equivalent to the reference system in accordance with this International Standard;
  - 2) if  $\tau \geq t$ , then the candidate system is different from the reference system in accordance with this International Standard.

## Annex B (normative)

### Determination of system sampling error

An empirical formula for estimating the particulate sampling error was developed by Belyaev and Levin<sup>[7]</sup> and published in [8], which gives the following formula for the penetration of a particle,  $P$ , when sampled from a larger flow stream by a coaxial sampling probe:

$$P = 1 + \left( \frac{v_e}{v_p} - 1 \right) \left[ 1 - \frac{1}{1 + \left( 2 + 0,62 \times \frac{v_p}{v_e} \right) St} \right] \quad (\text{B.1})$$

where

- $v_e$  is the gas velocity in the exhaust pipe, in metres per second;
- $v_p$  is the gas velocity in the sampling probe, in metres per second;
- $St$  is the Stokes number for target particles.

The parameters for equation B.1 shall be calculated as follows:

gas velocity in exhaust pipe —

$$v_e = \frac{q_{mew} \times 4}{\rho_e \times \pi \times d_e^2} \quad (\text{B.2})$$

gas velocity in sampling probe —

$$v_p = \frac{q_{mp} \times 4}{\rho_e \times \pi \times d_p^2} \quad (\text{B.3})$$

Stokes number —

$$St = \frac{\tau \times v_e}{d_p} \quad (\text{B.4})$$

particle relaxation time —

$$\tau = \frac{\rho_{PM} \times d_{PM}^2 \times C_c}{18 \times \eta} \quad (\text{B.5})$$

$$\eta = \frac{1,458 \times 10^{-6} \times T_e^{1,5}}{T_e + 110,4} \quad (\text{B.6})$$

where

- $q_{mew}$  is the exhaust gas mass flow rate, in kilograms per second;
- $\rho_e$  is the exhaust gas density, in kilograms per cubic metre;

- $d_e$  is the exhaust pipe diameter, in metres;
- $q_{mp}$  is the sample mass flow rate, in kilograms per second;
- $d_p$  is the sampling probe diameter, in metres;
- $\rho_{PM}$  is the particle density, in kilograms per cubic metre;
- $d_{PM}$  is the particle diameter, in metres;
- $C_c$  is the slip factor;
- $\eta$  is the dynamic viscosity of exhaust gas, in pascal seconds (Pa·s);
- $T_e$  is the exhaust gas temperature, in kelvins.

For the purposes of this International Standard, the following values are applicable:

$$\rho_{PM} = 1\,000 \text{ kg/m}^3$$

$$d_{PM} = 1,7 \times 10^{-7} \text{ m}$$

$$C_c = 4,35$$

For further information on these parameters, see [7] and [8] in the Bibliography.

## Annex C (normative)

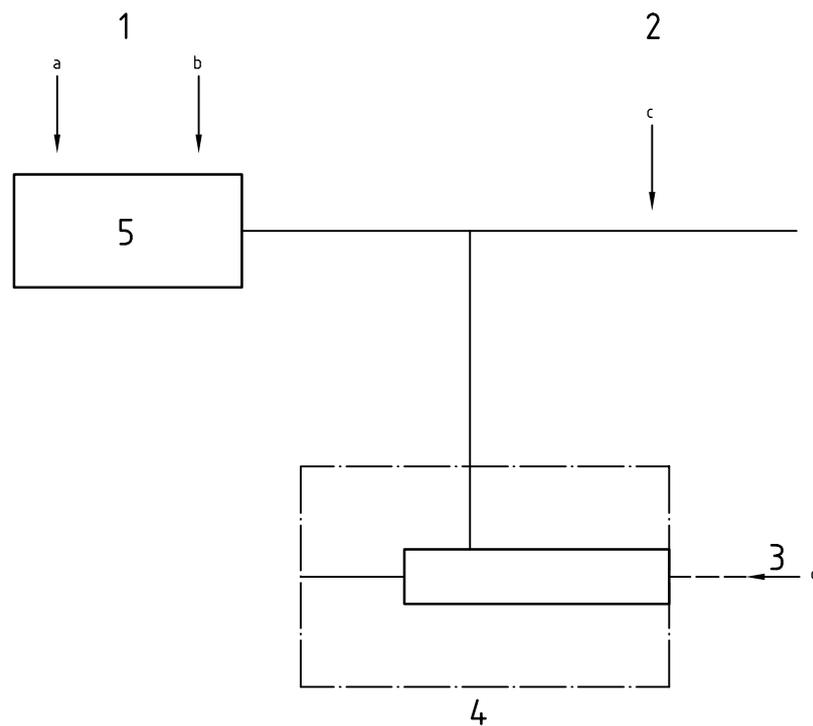
### Carbon flow check

#### C.1 General

All but a tiny part of the carbon in the exhaust comes from the fuel, and all but a minimal part of this is manifest in the exhaust gas as CO<sub>2</sub>. This is the basis for a system verification check based on CO<sub>2</sub> measurements.

The flow of carbon into the exhaust measurement systems is determined from the fuel flow rate. The flow of carbon at various sampling points in the emissions and particulate sampling systems is determined from the CO<sub>2</sub> concentrations and gas flow rates at those points.

In this sense, the engine provides a known source of carbon flow, and observing the same carbon flow in the exhaust pipe and at the outlet of the partial flow PM sampling system provides verification of leak integrity and flow measurement accuracy. This check has the advantage that the components are operating under actual engine test conditions of temperature and flow. The carbon flows shall be checked at the sampling points shown in Figure C.1. The specific equations for the carbon flows at each of the sample points follow.



**Key**

- |   |                     |   |                     |
|---|---------------------|---|---------------------|
| 1 | Location 1          | a | Air                 |
| 2 | Location 2          | b | Fuel                |
| 3 | Location 3          | c | CO <sub>2</sub> raw |
| 4 | Partial flow system | d | CO <sub>2</sub> PFS |
| 5 | Engine              |   |                     |

**Figure C.1 — Measuring points for carbon flow check**

## C.2 Carbon flow rate into engine (location 1)

The carbon mass flow rate into the engine for a fuel  $C_\beta H_\alpha O_\varepsilon$  is given by

$$q_{mCf} = \frac{12\beta}{12\beta + \alpha + 16\varepsilon} \times q_{mf} \quad (\text{C.1})$$

where  $q_{mf}$  is the fuel mass flow rate, in kilograms per second.

## C.3 Carbon flow rate in raw exhaust (location 2)

The carbon mass flow rate in the exhaust pipe of the engine shall be determined from the raw  $\text{CO}_2$  concentration and the exhaust gas mass flow rate as follows:

$$q_{mCe} = \left( \frac{c_{\text{CO}_2,r} - c_{\text{CO}_2,a}}{100} \right) q_{mew} \times \frac{12}{M_r} \quad (\text{C.2})$$

where

$c_{\text{CO}_2,r}$  is the wet  $\text{CO}_2$  concentration in the raw exhaust gas, in percent by volume;

$c_{\text{CO}_2,a}$  is the wet  $\text{CO}_2$  concentration in the ambient air, in percent by volume (around 0,04 %);

$q_{mew}$  is the exhaust gas mass flow rate on wet basis, in kilograms per second;

$M_r$  is the relative molecular mass<sup>3)</sup> of the exhaust gas.

If the  $\text{CO}_2$  is measured on a dry basis, it shall be converted to a wet basis in accordance with 5.5.5.

## C.4 Carbon flow rate in partial-flow dilution system (location 3)

For the partial-flow dilution system, the splitting ratio also needs to be taken into account. The carbon flow rate shall be determined from the dilute  $\text{CO}_2$  concentration, the exhaust gas mass flow rate and the sample flow rate:

$$q_{mCp} = \left( \frac{c_{\text{CO}_2,d} - c_{\text{CO}_2,a}}{100} \right) q_{mdew} \times \frac{12}{M_r} \times \frac{q_{mew}}{q_{mp}} \quad (\text{C.3})$$

where

$c_{\text{CO}_2,d}$  is the wet  $\text{CO}_2$  concentration in the dilute exhaust gas at the outlet of the dilution tunnel, in percent by volume;

$c_{\text{CO}_2,a}$  is the wet  $\text{CO}_2$  concentration in the ambient air, in percent by volume (around 0,04 %);

$q_{mdew}$  is the diluted exhaust gas mass flow rate on wet basis, in kilograms per second;

$q_{mew}$  is the exhaust gas mass flow rate on wet basis, in kilograms per second;

$q_{mp}$  is the sample flow of exhaust gas into partial-flow dilution system, in kilograms per second;

$M_r$  is the relative molecular mass<sup>3)</sup> of the exhaust gas.

If the  $\text{CO}_2$  is measured on a dry basis, it shall be converted to wet basis in accordance with 5.5.5.

3) Formerly called molecular weight.

## Annex D (informative)

### Calculation procedure — Example

#### D.1 Basic data for stoichiometric calculations

The basic data is given in Table D.1.

Table D.1

|   |           |       |
|---|-----------|-------|
| Atomic mass <sup>a</sup> of hydrogen        | 1,007 94  |       |
| Atomic mass of carbon                       | 12,011    |       |
| Atomic mass of sulfur                       | 32,065    |       |
| Atomic mass of nitrogen                     | 14,006 7  |       |
| Atomic mass of oxygen                       | 15,999 4  |       |
| Atomic mass of argon                        | 39,9      |       |
| Molecular mass of water                     | 18,015 34 | g/mol |
| Molecular mass of carbon dioxide            | 44,01     | g/mol |
| Molecular mass of carbon monoxide           | 28,011    | g/mol |
| Molecular mass of oxygen                    | 31,998 8  | g/mol |
| Molecular mass of nitrogen                  | 28,011    | g/mol |
| Molecular mass of nitric oxide              | 30,008    | g/mol |
| Molecular mass of nitrogen dioxide          | 46,01     | g/mol |
| Molecular mass of sulfur dioxide            | 64,066    | g/mol |
| Molar volume of water                       | 22,401    | l/mol |
| Molar volume of carbon dioxide              | 22,262    | l/mol |
| Molar volume of oxygen                      | 22,392    | l/mol |
| Molar volume of nitrogen                    | 22,390    | l/mol |
| Molar volume of nitric oxide                | 22,391    | l/mol |
| Molar volume of nitrogen dioxide            | 21,809    | l/mol |
| Molar volume of sulfur dioxide              | 21,891    | l/mol |
| Molar volume of argon                       | 22,4      | l/mol |
| <sup>a</sup> Formerly called atomic weight. |           |       |

NOTE If these basic component values are used in the emissions calculation in accordance with 5.5.4.3, the final results could be slightly different from the results calculated on the basis of the tabulated  $\mu$  values in accordance with 5.5.4.2.

## D.2 Gaseous emissions — Diesel fuel

The measurement data of an individual point of the test cycle (data sampling rate of 1 Hz) for the calculation of the instantaneous mass emission are given in Table D.2. In this example, CO and NO<sub>x</sub> are measured on a dry basis, HC on a wet basis. The HC concentration is given in propane equivalent (C3) and has to be multiplied by three to obtain the C1 equivalent. The calculation procedure is identical for the other points of the cycle.

Table D.2

| $T_{a,i}$ | $H_{a,i}$ | $W_{act}$ | $q_{mew,i}$ | $q_{maw,i}$ | $q_{mf,i}$ | $c_{HC,i}$ | $c_{CO,i}$ | $c_{NO_x,i}$ |
|-----------|-----------|-----------|-------------|-------------|------------|------------|------------|--------------|
| K         | g/kg      | kWh       | kg/s        | kg/s        | kg/s       | ppm (µl/l) | ppm (µl/l) | ppm (µl/l)   |
| 295       | 8,0       | 40        | 0,155       | 0,150       | 0,005      | 10         | 40         | 500          |

The fuel composition given in Table D.3 is considered.

Table D.3

| Component | Molar ratio              | % mass            |
|-----------|--------------------------|-------------------|
| H         | $\alpha = 1,852\ 9$      | $w_{ALF} = 13,45$ |
| C         | $\beta = 1,000\ 0$       | $w_{BET} = 86,50$ |
| S         | $\gamma = 0,000\ 2$      | $w_{GAM} = 0,050$ |
| N         | $\delta = 0,000\ 0$      | $w_{DEL} = 0,000$ |
| O         | $\varepsilon = 0,000\ 0$ | $w_{EPS} = 0,000$ |

### a) Step 1: Dry/wet correction (see 5.5.5)

Formula (15):

$$k_f = 0,055\ 584 \times 13,45 - 0,000\ 108\ 3 \times 86,5 - 0,000\ 156\ 2 \times 0,05 = 0,738\ 2$$

Formula (18):

$$k_W = \left( 1 - \frac{1,243\ 4 \times 8 + 111,12 \times 13,45 \times \frac{0,005}{0,148}}{773,4 + 1,243\ 4 \times 8 + \frac{0,005}{0,148} \times 0,738\ 2 \times 1000} \right) \times 1,008\ 5 = 0,933\ 1$$

Formula (17):

$$c_{CO,i}(\text{wet}) = 40 \times 0,9331 = 37,3$$

$$c_{NO_x,i}(\text{wet}) = 500 \times 0,9331 = 466,6$$

both results expressed in parts per million (microlitres per litre).

### b) Step 2: NO<sub>x</sub> correction for temperature and humidity (see 5.5.6)

Formula (23):

$$k_{h,D} = \frac{1}{1 - 0,018\ 2 \times (8,00 - 10,71) + 0,004\ 5 \times (295 - 298)} = 0,965\ 4$$

c) **Step 3: Calculation of the instantaneous mass emission using  $u$  values from Table 3** (see 5.5.4.2)

Formula (9):

$$m_{HC,i} = 0,000\ 478 \times 10 \times 3 \times 0,155 = 0,002\ 22$$

$$m_{CO,i} = 0,000\ 966 \times 37,3 \times 0,155 = 0,005\ 58$$

$$m_{NO_x,i} = 0,001\ 587 \times 466,6 \times 0,965\ 4 \times 0,155 = 0,110\ 8$$

all three results expressed in grams per second.

d) **Step 4: Integration of the instantaneous mass emission over the cycle** (see 5.5.4.2)

The following calculation is assumed for the ETC cycle (1 800 s) and the same emission in each point of the cycle.

Formula (9):

$$m_{HC} = \sum_{i=1}^{1800} 0,002\ 22 = 3,96$$

$$m_{CO} = \sum_{i=1}^{1800} 0,005\ 58 = 10,04$$

$$m_{NO_x} = \sum_{i=1}^{1800} 0,110\ 8 = 199,44$$

all three results expressed in grams per test.

e) **Step 5: Calculation of the specific emissions** (see 5.5.7)

Formula (25):

$$HC = 3,96/40 = 0,10$$

$$CO = 10,04/40 = 0,25$$

$$NO_x = 199,44/40 = 4,99$$

all three results expressed in grams per kilowatt hour.

### D.3 Particulate Emission — Diesel fuel

Particulate measurement is based on the principle of sampling the particulates over the complete cycle, while determining the sample and flow rates ( $M_{SAM}$  and  $G_{EDF}$ ) during the individual points of the cycle. The calculation of  $G_{EDFW}$  depends on the system used. In the following example, it is a system with flow measurement in accordance with 5.6.5, method a).

The measurement data in Table D.4 are assumed in this example.

**Table D.4**

| $W_{act}$<br>kWh | $q_{mew,i}$<br>kg/s | $q_{mf,i}$<br>kg/s | $q_{mdw,i}$<br>kg/s | $q_{mdew,i}$<br>kg/s | $m_f$<br>mg | $m_{se}$<br>kg |
|------------------|---------------------|--------------------|---------------------|----------------------|-------------|----------------|
| 40               | 0,155               | 0,005              | 0,001 5             | 0,002 0              | 1,700       | 1,515          |

a) **Step 1: Calculation of  $m_{\text{edf}}$**  (see 5.6.5)

Formula (29):

$$r_{\text{dil},i} = \frac{0,002}{(0,002 - 0,0015)} = 4$$

Formula (28):

$$q_{m\text{edf},i} = 0,155 \times 4 = 0,62$$

in kilograms per second.

Formula (27):

$$m_{\text{edf}} = \sum_{i=1}^{1800} 0,62 = 1116$$

in kilograms per test.

b) **Step 2: Calculation of particulate mass emission** (see 5.6.5)

Formula (26):

$$m_{\text{PM}} = \frac{1,7}{1,515} \times \frac{1116}{1000} = 1,252$$

in grams per test.

c) **Step 3: Calculation of specific emissions** (see 5.6.6)

Formula (32):

$$\text{PM} = 1,252/40 = 0,031$$

in grams per kilowatt hour.

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