Determination of the concentration of dispersed particles in diesel fuel – Automatic Particle Counter (APC) Light Obscuration Method

0 Introduction

This test method includes three procedures (A,B and C) to determine the concentration of dispersed particles in diesel fuel. Procedure A tests the sample without a co-solvent, and procedures B and C use co-solvents. Due to the risk of particles settling, good sampling and laboratory technique is particularly important in the application of this test method. Results from each procedure can differ due to the interaction of the particles with the co-solvents when used. This test method only uses standard apparatus that is calibrated in accordance with ISO 11171.

1 Scope

This standard describes a method for determining the concentration of dispersed particles in diesel fuel, diesel fuel containing hydrogenated vegetable oil (HVO) and diesel fuel containing fatty acid methyl esters (FAME) and FAME (Procedure B only). Results are reported for particles ≥4 μm(c), ≥6 μm(c) and ≥14 μm(c) per/ml and also in terms of ISO 4406 codes. The method covers three procedures; A which tests the sample without treatment to dissolve soft particles; B and C which dissolve soft particles using co-solvent prior to measurement. The precision statement was derived using two manufacturers' apparatus (known as Type 1 and Type 2). Details are provided in Normative Annex A and B.

NOTE 1 When using a co-solvent the particle counts represent the amount of hard particles as defined by this method.

NOTE 2 For the purposes of this method, water droplets are considered as particles and agglomerated particles are detected as a single obscuration event. Although the projected area of a particle is measured, this is expressed as a diameter for the purposes of this method.

NOTE 3 During use of a co-solvent to dissolve soft particles and if hard particles are agglomerated with soft particles, an increase in particles and consequently an increase in particle count can also occur. Where such effects cancel out, or no soft particles are present, counts can remain unchanged.

WARNING – The use of this standard may involve hazardous materials, operations and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety, health and environmental practices and determine the applicability of regulatory limitations prior to use.

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

IP 475: Petroleum liquids – Manual sampling (EN ISO 3170)

ISO 4406: Hydraulic fluid power - Method for coding the level of contamination by solid particles.

ISO 11171 Hydraulic fluid power – Calibration of automatic particle counters for liquids.

3 Definitions

For the purposes of this standard, the following terms and definitions apply:

3.1
cumulative count

the total number of particles, counted per ml, in pre-determined particle size channels including at least ≥4 μm(c), ≥ 6 μm(c) and ≥14 μm(c).

3.2
particle size, μm(c)

the projected area equivalent diameter of particles passing through the detecting cell as defined in ISO 11171.

3.3
soft particles

particles typically comprising free water or other material which are dissolved by the specified co-solvents.

NOTE The action of dissolving the soft particles is to make them undetectable during the analysis by this method.

3.4
hard particles

particles that are not dissolved by specified co-solvents, that can be abrasive, typically comprising iron and/or silicon.

NOTE Further information on hard and soft particles can be found in technical report CEN TR/17548.

4 Principle

A test portion is prepared by tumbling to generate a homogeneous particle dispersion. If required, soft particles are dissolved through the addition of a co-solvent (see Procedure B or C), and in the case of Procedure B, warming the sample to aid dissolution. Without delay, the test portion is introduced into the APC for analysis. A purging and test sequence is commenced. In the test sequence 10 ml of the prepared test portion passes through the optical cell and is measured. During the measurement, light, from a laser, is passed through the test portion to an optical sensor. The optical sensor and associated electronics record the light intensity in the form of a voltage. As a particle obscures the light and casts a shadow on the optical sensor the reduction in light intensity causes a voltage drop which is proportional to the particles size. Software calculates the diameter of the particle and counts the number of particles in the size bands ≥4 μm(c), ≥ 6 μm(c) and ≥14 μm(c). Two further 10 ml aliquots of the test portion are measured and the average result is calculated.

5 Reagents and materials

5.1 Co-solvent 1, for use with procedure B containing specified amounts (see Normative Annex C) of solvents (5.1.1 and 5.1.2).

5.1.1 Propan-2-ol, technical grade or better with a the particle count less than 100 counts per ml for the ≥4 μm**(c)** measurement.

5.1.2 Toluene, technical grade or better with a the particle count less than 100 counts per ml for the ≥4 μm**(c)** measurement.

5.2 Co-solvent 2, a proprietary material[1, 2] for use with procedure C.

5.3 Membrane Filter, PTFE, 0.45 μm pore size.

5.4 Verification fluid[1]**,** containing Medium Test Dust (MTD).

5.5 Heptane, technical grade filtered. Prepare the heptane by filtering through a 0,45 μm filter (5.3) contained in a filter apparatus (6.4). Store in a container made of suitable material, previously rinsed three times with filtered heptane.

6 Apparatus

6.1 Automatic Particle Counter (APC)[1]

A self-contained light obscuration based particle counter for portable or laboratory use. The APC shall meet the requirements of and be calibrated in accordance with ISO 11171. Acceptable APC's are described in Annex A (Type 1) and Annex B (Type 2). Details of the manufacturers are available from the Energy Institute and such apparatus was used to derive the method precision statements - see Table 6.

Any new or proposed equipment, or changes to current equipment, shall be approved by the Energy Institute using the Code of Practice for communicating changes to Apparatus Specified in IP Test Methods.

6.2 Test portion container, cylindrical, made of glass, of approximately 250 ml volume and a cap with suitable internal seal.

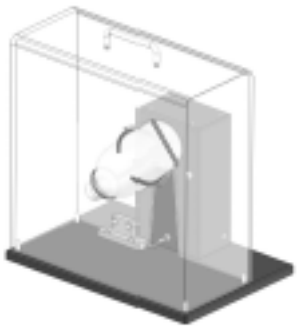
6.3 Waste container, for collecting the tested test portion.

6.4 Filter apparatus, containing a 0,45 μm pore size filter (5.3)

NOTE It is advisable to take appropriate precautions to avoid static charge generation when filtering flammable solvents. An example of suitably grounded filtration apparatus can be found in IP 440.

1 Details of suitable materials and apparatus are available from the Energy Institute, technical@energyinst.org.
2 Supplied in single test vials with particle count for the ≥4 μm(c) size band ≤100/ml for convenience of use.

6.5 Mechanical tumbler (optional), as shown, to tumble a 250 ml glass container at a speed of 1 revolution per second.



6.6 Hot water bath, capable of maintaining 40 °C ± 1 °C with a water depth of approximately 100 mm.

7 Apparatus verification and calibration

7.1 General

The operator shall check that the calibration of the APC is up to date.

7.2 Verification

Verify the correct operation of the APC at least every 6 months in accordance with the manufacturer's instructions using a verification fluid, or by participation in a proficiency testing scheme (PTS) meeting the requirements of ISO 17043 and reported in accordance with ISO 4259-3.

7.2.1 Verification fluid

Run a test using the verification fluid (5.4) in accordance with instructions given in clause 11 and the manufacturer's instructions for the verification fluid. The result obtained shall be within R/√2 plus the uncertainty, of the accepted reference value for the verification fluid, for the certified ≥4 μm(c) count, where R is the reproducibility of the test. If the result obtained does not meet this requirement, ensure the verification fluid preparation is in accordance with the manufacturers' instructions, check the verification fluid's date for validity, and run a further test using filtered heptane; then repeat the verification. If the result is not within the allowed tolerance, contact the apparatus manufacturer.

7.2.2 Proficiency testing scheme (PTS)

Run a test in accordance with instructions given in clause 11 and instructions provided by the PTS. The result obtained shall be within R/√2 plus the uncertainty, of the PTS consensus value for the ≥4 μm**(c)** count, where R is the reproducibility of the test. If the result obtained does not meet this requirement, ensure the test was conducted within the requirements of the PTS instructions, check the PTS sample date for validity, and contact the apparatus manufacturer.

7.3 Flow Rate

Verify the nominal flow rate in Table 1 in accordance with the manufacturer's instructions. If the flow rate is incorrect contact the manufacturer.

Table 1 - Flow Rate

Apparatus	Flow rate required (ml/min)
Type1 (Annex A)	30
Type 2 (Annex B)	25

7.4 Calibration

Confirm the calibration of the APC according to the manufacturer's instructions at least every 12 months or immediately after any maintenance to the measuring system.

8 Samples and sample handling

8.1 Unless otherwise specified take a sample of at least 400 ml, in accordance with IP 475 (EN ISO 3170) along with the additional requirements of 8.2 to 8.4.

8.2 Sample containers shall be capable of transporting the sample without contamination. Containers shall be of fully epoxy-lined metal, amber coloured glass or polytetrafluoroethylene (PTFE). The use of amber glass containers is preferred and can help avoid any potential problems with particles adhering to the insides of the containers due to static electricity or magnetism which could occur with some samples.

8.3 Prior to taking the sample, the sample container shall be flushed at least three times with the container 10 % to 20 % filled with the product to be sampled. For each flush the container shall be closed and shaken vigorously for a minimum of 5 seconds and the product drained. Fit the cap on the sample container immediately after filling.

8.4 Ensure that the sample container includes 10 % to 20 % ullage so that the particles are re-dispersed during tumbling.

NOTE The ILS research report titled Inter-Laboratory Study to Establish Precision Statements for IP PM FA/21: Determination of the concentration of dispered particles in diesel fuel - Automated Particle Counter (APC) Light Obscuration Method contains information regarding the subsampling of samples for dispatch to multiple laboratories. The data indicated that the procedure used was reliable for the comparison of bulk stock.

9 Apparatus preparation

9.1 Follow the manufacturer's instructions to prepare the APC.

9.2 Remove the in-line filter, if fitted.

9.3 If available select the correct mode of operation according to the manufacturer's instructions. This mode may be identified on the result print out.

9.4 If the instrument has been used to test samples where the ≥4 μm(c) particle count was more than 20 000/ml, run a sample of filtered heptane through the instrument and verify that it is clean (see Clause 9.7) before commencing testing.

9.5 Clean the input tube by washing the outside in filtered heptane.

9.6 At the start of any daily testing regime, clean the APC using filtered heptane in accordance with the manufacturer's instructions and Table 2.

Table 2 - Heptane Volume Required

Apparatus	Volume required (ml)
Annex A (Type 1)	120
Annex B (Type 2)	400

9.7 Confirm the condition of the measurement cell by testing a sample of filtered heptane, see Annex A and B for the applicable measurement cell cleanliness acceptance criteria.

9.8 When using Procedure B, before every test, clean the APC using filtered heptane in accordance with the manufacturer's instructions and Table 2. Ensure the cleanliness requirements in Annex A or B are achieved.

10 Test portion preparation

10.1 Procedure A

10.1.1 The test portion shall be prepared immediately prior to testing.

10.1.2 Prepare the test portion by tumbling the sample container and its contents end over end for 60 revolutions with a frequency of approximately 1 revolution per second either manually or using an automated tumbler.

10.1.3 Immediately pour 200 ml into a clean 250 ml glass test portion container of sufficient size which has been previously rinsed three times with the sample and fit a clean cap.

NOTE Ullage is important so that the particles are re-dispersed during tumbling.

10.2 Procedure B

10.2.1 The test portion shall be prepared immediately prior to testing.

10.2.2 Prepare the test portion by tumbling the sample container and its contents end over end for 60 revolutions with a frequency of approximately 1 revolution per second either manually or using an automated tumbler.

10.2.3 Using a clean measuring cylinder, which has been rinsed three times with the sample, measure 100 ml of sample into a clean 250 ml glass test portion container which has been previously rinsed three times with co-solvent 1 (5.1). Volumes shall be measured to an accuracy of ±2% of the required volume or better.

10.2.4 Using a clean measuring cylinder, which has been rinsed three times with the co-solvent 1 measure 100 ml of co-solvent 1 (5.1) and add it to the 250 ml glass test portion container and fit a clean cap. Volumes shall be measured to an accuracy of ±2% of the required volume or better.

10.2.5 Tumble the test portion container 5 times and place it in a hot water bath set at 40°C such that the water level is above the top level of the test portion. After 10 minutes tumble the test portion container a further 5 times and then leave in the bath for a total of 1 hour ±5 mins.

10.3 Procedure C

10.3.1 The test portion shall be prepared immediately prior to testing.

10.3.2 Prepare the test portion by tumbling the sample container and its contents end over end for 60 revolutions with a frequency of approximately 1 revolution per second either manually or using an automated tumbler.

10.3.3 Using a clean measuring cylinder, which has been rinsed three times with the sample, measure 200 ml of sample into a clean 250 ml glass test portion container which has been previously rinsed three times with sample. Volumes shall be measured to an accuracy of ±2% of the required volume or better.

10.3.4 Empty the contents of the 10 ml vial of co-solvent 2 into the 250 ml glass test portion container and fit a clean cap.

10.4 For test portion preparation weighing is an alternative to volumetric measurement for procedures A, B and C, by calculating the required mass of sample and/or co-solvent based upon the respective density and volume required.

11 Procedure

11.1 Prepare the APC according to Clause 9, with particular attention to cleaning the input tube by washing the outside in filtered heptane.

11.2 Prepare the test portion according to the required procedure in Clause 10.

11.3 Immediately, before commencing a test, tumble the test portion, prepared in accordance with the procedure given in Clause 10, end over end for 60 revolutions with a frequency of approximately 1 revolution per second either manually or using an automated tumbler. Since some settling of particulates can occur during the course of a test, always make a fresh test portion in accordance with Clause 10 for every test.

11.3.1 In order to prevent air entrainment, do not over vigorously mix the test portion.

11.4 Insert the cleaned input tube of the APC into the test portion container ensuring that it is between 10 mm and 20 mm from the bottom of the container. The input tube shall not touch the sides of the test portion container.

11.5 Start the automatic test sequence in accordance with the manufacturer's instructions.

11.6 The following steps occur automatically:

11.6.1 50 ml of the test portion passes through the apparatus to flush and clean.

11.6.2 10 ml of the test portion is measured in the optical measurement cell at ≥ 4 μm(c) , ≥ 6 μm(c) and ≥14 μm(c) size channels.

NOTE 1 Other size channels can be measured, however the precision has not been determined.

NOTE 2 Information on free water and particle counting is available in a document titled Dispersed Water and Particulates in Jet Fuel:Size Analysis under Operational Conditions and Application to Coalescer Disarming (refer to bibliography).

11.6.3 Repeat 11.6.2 two more times. Calculate the mean. If there is a difference of over 10% of the mean, or 200 particles per ml, whichever is the larger, between the three ≥ 4 μm(c) measurements then discard the result. A new test portion shall be prepared in accordance with Clause 10.

11.7 Calculations

11.7.1 For Procedure A the average cumulative count calculated in 11.6.3 does not require further calculation.

11.7.2 For Procedures B and C calculate the average cumulative number of particles per millilitre present in the original sample by multiplying the cumulative particle count obtained for each size channel by the multiplication factor specific for each procedure as shown in Table 3. These factors correct the result for the dilution caused by the co-solvent used. An example is given in Table 4 and 5.

Table 3 - Multiplication factors

Procedure	Multiplication Factor
Procedure B	2,00
Procedure C	1,05

11.7.3 Report the average cumulative counts per ml as the result of the test.

11.7.4 From the average cumulative counts per ml, calculate the ISO code for each size channel according to ISO 4406.

11.8 Clean the apparatus

11.8.1 If any of the measurements exceed 20,000 particles/ml at 4 μm(c), flush the apparatus with filtered heptane and confirm cleanliness by following Clauses 9.6 and 9.7 prior to testing the next sample. This helps to avoid cross contamination between samples.

11.8.2 At the end of daily testing flush the apparatus with filtered heptane by following Clause 9.6.

Table 4 - Example calculation for Procedure B

Cumulative particle count size channel	Counts obtained in Procedure B for sample prepared with co-solvent 1	Multiplication Factor	Average cumulative counts per ml (value to report)
≥4 μm(c)	3500,0	2,00	7000,0
≥6 μm(c)	250,0	2,00	500,0
≥14 μm(c)	10,0	2,00	20,0

Table 5 - Example calculation for Procedure C

Cumulative particle count size channel	Counts obtained in Procedure C for sample prepared with co-solvent 2	Multiplication Factor	Average cumulative counts per ml (value to report)
≥4 μm(c)	6667	1,05	7000,4
≥6 μm(c)	476,2	1,05	500,0
≥14 μm(c)	19,0	1,05	20,0

12 Expression of results

12.1 Record the average cumulative counts per ml for ≥4 μm(c), ≥ 6 μm(c) and ≥14 μm(c) to 1 decimal place, ensuring that the multiplication factor (Table 3) has been applied.

12.2 Record the ISO codes, according to ISO 4406, as integers.

13 Precision

13.1 General

An inter-laboratory study was carried out in 2021 on diesel, diesel containing hydrogenated vegetable oil (HVO) and diesel containing up to 20 % fatty acid methyl ester (FAME), with test results in the range 277.2 particles per ml ≥4μm(c) to 23453.6 particles per ml ≥4μm(c), using 10 laboratories/locations with Type 1 or Type 2 apparatus.

The precision evaluation study as detailed above did not conform to one of the requirements of ISO 4259-1, due to the degrees of freedom being less than 30 for reproducibility. As permitted in ISO 4259-1 the precision is accepted due to these exceptional circumstances: 1) industry requested a multi-site study; 2) this method is the current state of the art; 3) there is an urgent industry need.

A second inter-laboratory study for Procedure B was completed in 2024 on diesel, diesel containing hydrogenated vegetable oil (HVO) and blends of diesel containing up to 50% fatty acid methyl ester (FAME) and FAME (B100), using 9 laboratories/locations with Type 1 apparatus and 7 laboratories/locations with Type 2 apparatus.

The precision, as determined by statistical examination following the procedures in ISO 4259-1 of interlaboratory test results is given in Table 6.

13.2 Repeatability (r)

The difference between two independent results obtained in the normal and correct operation of the test method by the same operator in a given laboratory applying the same method, for test material considered to be the same, within a short interval of time, with the same apparatus under constant operating test conditions, that is expected to be exceeded with a probability of 5 % due to random variation, can be calculated using the functions in Table 6:

13.3 Reproducibility (R)

The difference between two independent results obtained by different operators in the normal and correct operation of the same method, for test material considered to be the same, in different laboratories using different apparatus under different operating test conditions, that is expected to be exceeded with a probability of 5 % due to random variation, can be calculated using the functions in Table 6:

13.4 Relative bias

The degree of agreement between Type 1 and Type 2 apparatus has not been determined.

NOTE 1 During the 2024 ILS regarding Procedure B there were not sufficient labs or samples to meet the requirements in ISO 4259-5, however following similar mathematical principles, using the results from the 8 ILS samples, there is no evidence of a statistically significant bias between the Type 1 and Type 2 instruments.

NOTE 2 For guidance, statisical analysis for procedure A determined that a value less than or equal to 7789 particles per ml ≥4 μm(c) provides a 95% probability that the sample has a true particle count of less than 10000. For more information refer to research report titled Inter-Laboratory Study to Establish Precision Statements for IP PM FA/21: Determination of the concentration of dispersed particles in diesel fuel - Automated Particle Counter (APC) Light Obscuration Method. Users may also refer to ISO 4259-1 Section 6:Assessment of quality conformance to specification.

NOTE 3 The relative bias between procedures has not been determined.

14 Test report

The test report shall contain at least the following information:

a) a reference to this standard;
b) the type and complete identification of the product tested;
c) the procedure used (A, B or C);
d) the result of the test from clause 12;
e) any deviation by agreement or otherwise from the procedure specified;
f) the date and time of the test.
g) the apparatus used (Type 1 or Type 2).

Table 6 - Precision

Particles size band μm(c)	Scope Limits	repeatability, particles per ml	DF	Reproducibility, particles per ml	DF
Procedure A (Type 1 and Type 2 apparatus)[1]					
≥4	458 to 23453	0,09246(x + 2000)	61	0,3382(x + 400)	22
≥6	111 to 11610	0,1425(x + 150)	54	0,6084x	17
≥14	13 to 2531	0,1598(x + 50)	46	0,5884x	23
Procedure B (Type 1 apparatus)[2]					
≥4	1983 to 25188	0,0534x + 757.2	70	0,2796x + 514.9	63
≥6	799 to 9350	0,2832(x + 300)	69	0,3850(x + 300)	102
≥14	127 to 363	0,4124(x + 30)	68	0,6816(x + 30)	37
Procedure B (Type 2 apparatus)[3]					
≥4	3825 to 27880	0,1633(x + 5000)	51	0,2808(x + 5000)	46
≥6	908 to 11125	0,1659(x + 1500)	52	0,3477(x + 1500)	59
≥14	91 to 468	0,4442(x + 16)	52	0,7037(x + 16)	33
Procedure C (Type 1 and Type 2 apparatus)[1]					
≥4	4837 to 21914	0,1681(x + 4000)	63	0,5233(x + 4000)	20
≥6	514 to 6110	0,1447(x + 600)	60	0,3699(x + 600)	51
≥14	40 to 826	0,2212(x + 20)	63	0,6305(x + 20)	44

Where x is the average cumulative counts per ml, and DF is the degrees of freedom.

1 From 2021 ILS.
2 From 2024 ILS. The ≥4 μm(c) precision functions were derived following ISO 5725.
3 From 2024 ILS.

Annex A
(normative)

Automatic particle counter assembly Type 1

A.1 Apparatus[1]

A.1.1 General description

Light obscuration based particle counter, fully integrated apparatus consisting of a microprocessor and display, optical measurement cell, a computer controlled double pump and an automatic changeover valve.

The APC shall meet the requirements of and be calibrated in accordance with ISO 11171.

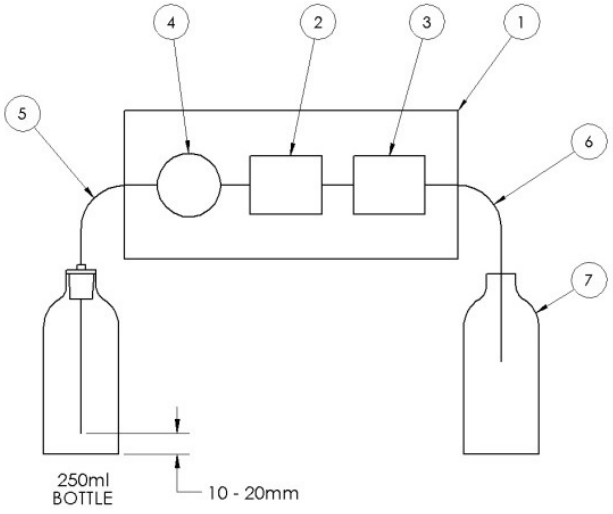
Certain attributes of the apparatus are particular to the apparatus contained in this Annex as in Table A.1. The specific apparatus configuration is shown in Figure A1.1.

Table A.1 Specific apparatus attributes

Attribute	Value
Flow rate	30 ml/min
Maximum particle concentration (per ml)	60 000
Sample volume used during test	80 ml
Number of measurements per test	3
Acceptance criteria for the test	Maximum difference allowed of less than 10% of the mean, or 200 particles per ml, whichever is the larger, between the three ≥ 4 μm(c) measurements
Heptane cleaning volume	120 ml
Measurement cell cleanliness acceptance criteria	≤50 counts in the ≥4μm channel count
Pump location	Downstream of optical measurement cell
Minimum number of size channels	8

Figure A.1 Apparatus

Item	Description
1	Light obscuration based particle counter
2	Automatic changeover valve
3	Double pump
4	Optical measurement cell
5	Sample delivery tube assembly
6	Outlet tube
7	Waste container



1 Details of suitable materials and apparatus are available from the Energy Institute, technical@energyinst.org.

A.1.1.1 Automatic changeover valve, to enable flushing and measurements to be carried out automatically under computer control, while maintaining single directional flow of the sample directly from the test portion container to the optical measurement cell.

A.1.1.2 Double pump, a pump driven by a constant speed motor under computer control to ensure a constant nominal flow rate of 30 ml/min through the optical measurement cell.

A.1.1.3 Optical measurement cell, comprising an optical sensor and a 5 mW laser diode light source, wavelength 670 nm ± 5 nm.

A.1.1.4 Sample delivery tube assembly, comprising:

a) flexible transparent tubing for connection to the input connector;

b) adjustable length metal tube adaptor to enable the use of different dimension test portion containers;

c) test portion container cap to avoid dust ingress.

A.1.1.5 Protective Filter (Optional), metal mesh filter of pore size 250 μm, used to protect the apparatus, following manufacturer's instructions.

A.1.1.6 Printer, to record full details of the measurements and average results.

NOTE Results are printed out automatically at the end of each test. Results and analyses of previous measurements can also be printed (see manufacturer's instructions).

Annex B
(normative)

Automatic particle counter assembly Type 2

B.1 Apparatus[1]

B.1.1 General description

Light obscuration based particle counter, fully integrated apparatus consisting of a microprocessor and display, optical measurement cell, a microprocessor controlled metering pump.

The APC shall meet the requirements of and be calibrated in accordance with ISO 11171.

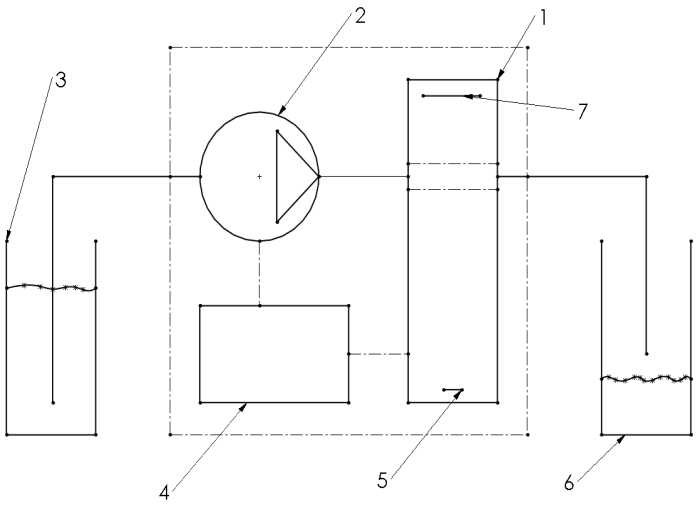
Certain attributes of the apparatus are particular to the apparatus contained in this Annex as in Table B.1. The specific apparatus configuration is shown in Figure B1.1.

Table B.1 Specific apparatus attributes

Attribute	Value
Flow rate	25 ml/min
Maximum particle concentration (per ml)	60 000
Sample volume used during test	80 ml
Number of measurements per test	3
Acceptance criteria for the test	Maximum difference allowed of less than 10% of the mean, or 200 particles per ml, whichever is the larger, between the three ≥ 4 μm(c) measurements
Heptane cleaning volume	400 ml
Measurement cell cleanliness acceptance criteria	≤100 counts in the ≥4μm channel count
Pump location	Upstream of optical measurement cell
Minimum number of size channels	8

Figure B.1 Apparatus

Item	Description
1	Sensor
2	Metering pump
3	Test portion container
4	Microcontroller system
5	Laser diode
6	Waste container
7	Optical sensor



1 Details of suitable materials and apparatus are available from the Energy Institute, technical@energyinst.org.

B.1.1.1 Metering pump, a pump driven by a constant speed stepper motor under microprocessor control to ensure a constant nominal flow rate of 25 ml/min through the optical measurement cell.

B.1.1.2 Optical measurement cell, comprising an optical sensor and a 5 mW laser diode light source, wavelength 670 nm ± 5 nm.

B.1.1.3 Sample delivery tube assembly, flexible transparent tubing for connection to the input and output connectors.

B.1.1.4 Printer, to record full details of the measurements and results.

NOTE Results are printed out automatically at the end of each test. Results and analyses of previous measurements can also be printed (see manufacturer's instructions).

Annex C
(normative)

Preparation of co-solvent 1

C.1 Preparation of co-solvent 1

C.1.1 Mix sufficient co-solvent 1 to run the required number of tests.

C.1.2 Prepare co-solvent 1 by mixing 75 volume % toluene and 25 volume % propan-2-ol .

Warning – Extremely flammable (Category II flammable liquid). Hazardous to health. Consult Safety Data Sheet for full information

C.1.3 Store co-solvent 1 in a clean container made of a suitable material, previously rinsed three times with co-solvent 1.

C.1.4 Measure the particle count of each batch of co-solvent 1 following Clause 11 using either apparatus detailed in Annex A or B and record the total counts per ml.

If the particle count is greater than 100 counts per ml for the ≥4 μm measurement then the batch of co-solvent 1 shall not be used. Co-solvent 1 shall be replaced or filtered through a membrane filter with a 0.45 μm pore size (5.3) contained in a filter apparatus (6.4). Repeat C.1.4 until the criteria are met.

NOTE It is advisable to take appropriate precautions to avoid static charge generation when filtering flammable solvents. An example of suitably grounded filtration apparatus can be found in IP 440.

C.1.5 Following completion of clause C.1.4 flush the apparatus with filtered heptane by carrying out a test following clause 9.6 to remove co-solvent 1 from the apparatus.

NOTE A possible reason for particle counts greater than 100 counts per ml is the cleanliness of the APC or that co-solvent 1 can contain entrained air bubbles.

Bibliography

[1] CEN TR/17548 Automotive fuels - Diesel fuel market issues – Abrasive particles investigation report

[2] ISO 17043 Conformity assessment — General requirements for proficiency testing

[3] ISO 4259-3 Petroleum and related products — Precision of measurement methods and results — Part 3: Monitoring and verification of published precision data in relation to methods of test

[4] Dispersed Water and Particulates in Jet Fuel: Size Analysis under Operational Conditions and Application to Coalescer Disarming. Industrial & Engineering Chemistry Research 2011, 50, 9, 5749–5765 Publication Date: March 29, 2011

[5] ISO 4259-1 Petroleum and related products - Precision of measurements and results - Part 1: Determination of precision data in relation to methods of test.

[6] Inter Laboratory Study to Establish Precision Statements for IP PM 630/22 Procedure B: Determination of the concentration of dispersed particles in diesel fuel –Automated Particle Counter (APC) Light Obscuration Method