

The background features a dark blue gradient with faint, light blue circular patterns and a scale. The scale is a large arc on the left side, with numerical markings from 140 to 260 in increments of 10. Several smaller circles and arcs are scattered across the background, some with arrows indicating direction.

PLENARIA UNICHIM 17 APRILE 2018

DISCUSSIONE DEI RISULTATI DELLE PROVE INTERLABORATORIO
E AGGIORNAMENTO SUI METODI DI PROVA

ASTM D1322 – 15 E1 PUNTO FUMO (MANUAL APPARATUS)

8. Sampling and Preparation of Samples

Allow all samples to come to ambient temperature ($20 \pm 5^\circ\text{C}$), without artificial heating.

9. Preparation of Apparatus

9.1 Place the lamp in a vertical position in a room where it can be completely protected from drafts.

9.1.1 If the room is not completely draft-free, place the lamp in a vertical position in a box constructed of heat-resistant material (not containing asbestos), open at the front. The top of the box shall be at least 150 mm above the top of the chimney and the inside of the box painted dull black.

Il campione viene portato a $20 \pm 5^\circ\text{C}$?

La lampada è posizionata in un ambiente privo di correnti d'aria?

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9.3 Extract all wicks, either new or from a previous determination, for at least 25 cycles in an extractor, using a mixture of equal volumes of toluene and anhydrous methanol. Allow the wicks to dry partially in a hood before placing in the oven, or use a forced-draft and explosion-proof oven for drying wicks, or both. Dry for 30 min at 100 to 110°C, and store in a desiccator until used.

9.3.1 Extracted wicks are commercially available and may be used, provided that they have been certified as being extracted by the procedure outlined in 9.3. Store purchased extracted wicks in a desiccator over desiccant until use. After use, extract these wicks as in 9.3 before using again.

**Eseguiamo la procedura qui
descritta?**

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10. Calibration of Apparatus

10.1 Confirm calibration of the apparatus in accordance with 10.1.3 or calibrate, if needed, in accordance with 10.1.1 prior to first use of the day. Recalibrate when there has been a change in the apparatus or operator, or when a change of more than 0.7 kPa occurs in the barometric pressure reading.

Facciamo la taratura dello strumento come previsto dal paragrafo 10?

Ogni volta che ci sono modifiche all'apparecchiatura?

Ogni volta che cambia l'operatore?

Ogni volta che c'è una variazione di più di 0,7 Kpa nella pressione barometrica?

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TABLE 1 Reference Fuel Blends

Standard Smoke Point at 101.3 kPa	Toluene	2,2,4-trimethylpentane
mm	% (V/V)	% (V/V)
14.7	40	60
20.2	25	75
22.7	20	80
25.8	15	85
30.2	10	90
35.4	5	95
42.8	0	100

10.1.1 Calibrate the apparatus by testing two of the reference fuel blends specified in 7.4, using the procedure specified in Section 11 and, if possible, bracketing the smoke point of the sample. If this is not possible, use the two test blends having their smoke points nearest to the smoke point of the sample.

10.1.2 Determine the correction factor, f , for the apparatus from the equation:

$$f = \frac{(A_s / A_d) + (B_s / B_d)}{2}$$

Quali miscele vengono utilizzate come riferimento?

Calcoliamo il fattore di correzione?

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11.5 Light the candle and adjust the wick so that the flame is approximately 10 mm high and allow the lamp to burn for 5 min. (See Fig. 6 and Fig. 7.) Raise the candle until a smoky tail appears, then lower the candle slowly through the following stages of flame appearance:

11.5.1 A long tip; smoke slightly visible; erratic and jumpy flame.

11.5.2 An elongated, pointed tip with the sides of the tip appearing concave upward as shown in Fig. 6 (Flame A).

11.5.3 The pointed tip just disappears, leaving a very slightly blunted flame as shown in Fig. 6 (Flame B). Jagged, erratic, luminous flames are sometimes observed near the true flame tip; these shall be disregarded.

11.5.4 A well rounded tip as shown in Fig. 6 (Flame C). Determine the height of Flame B to the nearest 0.5 mm. Record the height observed.

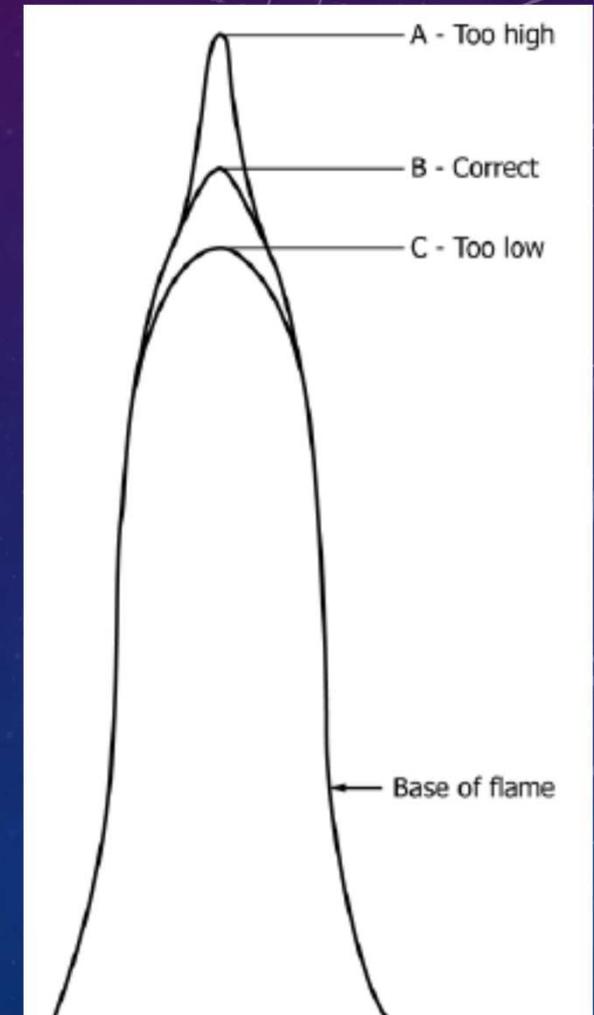


FIG. 6 Typical Flame Appearances

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11.5.5 Make three separate observations of the flame height at the smoke point by repeating the flame-appearance sequence specified in 11.5. If these values vary over a range greater than 1.0 mm, repeat the test with a fresh sample and another wick.

Aspettiamo che il campione bruci per 5 minuti?

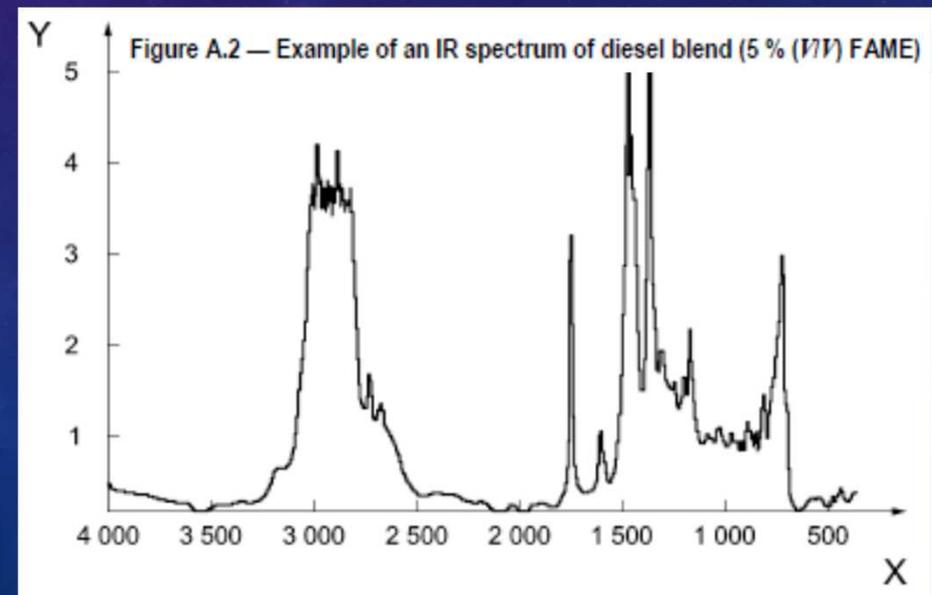
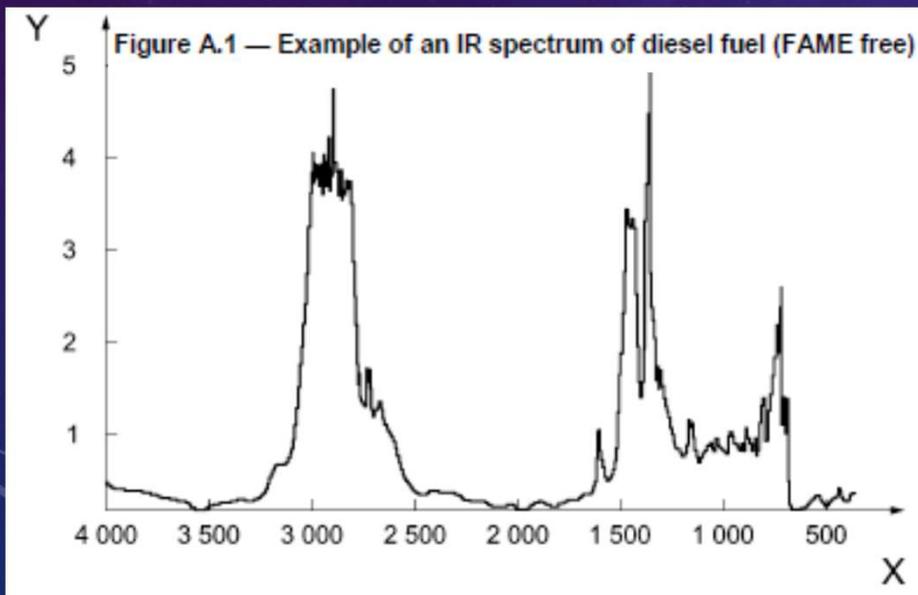
Adottiamo la procedura descritta dal metodo?

Facciamo 3 distinte letture applicando la sequenza descritta?

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4.2 FAME free middle distillate as solvent for dilution and as reference material for the measurement of the background spectrum. In particular, a middle distillate suitable for the type of sample (diesel fuel or domestic heating oil) shall be used for range A in order to avoid spectral decompensation as far as possible. In this context, the property “FAME free” means middle distillates without any absorption bands in the IR signal range typical for FAME.

Controlliamo che il FAME free middle distillate sia effettivamente FAME free?



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7.1 Selection and treatment of the cell

The path length of the cell (5.2) shall be selected so that adequate net signal intensities can be obtained (in at least two decimal places, see marked cells in Table A.1). Signal intensities shall still be within the linear detector range. Specific adjustments shall be followed (see recommendations given in Table A.1) depending on measurement range A, B or C:

- range A: path length as long as possible and measurement preferably without dilution;
- range B: shorter path length and dilution adapted to the anticipated FAME content;
- range C: shorter path length and a dilution higher than for range B, adapted to the anticipated FAME content.

Le celle che utilizziamo sono adeguate per i range di concentrazione attesi?

Quali rapporti di diluizione vengono utilizzati?

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The path length shall be known or determined exactly. If in use for a longer period of time it should be checked more frequently. A specific and individual calibration shall be made for every cell in use, retaining all other measurement parameters for every applied measurement range. The use of several or different cells during the same calibration is not permissible.

If cells sensitive to water are used the path length shall be checked more frequently. In case the path length has changed, path length determination and calibration shall be carried out anew.

Controlliamo inizialmente e periodicamente lo spessore della cella?

La calibrazione viene fatta specificatamente per ogni cella?

Usiamo celle sensibili all'acqua?

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7.2 Cleaning of the cells

After every measurement the cell shall be carefully cleaned with a solvent (4.3). This is particularly important after measuring samples with high FAME content and extremely important when the measurement of samples with low FAME contents is prepared. The cell may also be cleaned by rinsing repeatedly with FAME free middle distillate (4.2).

Puliamo attentamente le celle con solvente o FAME free distillate?

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7.3.1 Range A

For from approx. 0,05 % (V/V) to approx. 3 % (V/V) cells with a long path length (e.g. KBr) of approx. 0,5 mm, known to the nearest 0,01 mm) have proven to be well suited. Other materials and path lengths are also permissible, though on no account should the path length be chosen less than about 0,2 mm to enable a signal and signal-to-noise ratio as favourable as possible.

7.3.2 Range B

For FAME contents from approx. 3 % (V/V) to approx. 20 % (V/V) a path length of approx. 0,1 mm, known to the nearest 0,01 mm, is recommended in combination with a dilution rate of 1 : 5 (refer to data given in Table A.1). Other cells and path lengths are also possible; however, dilution should be arranged within a comparable dimension in order to avoid any major dilution errors.

Le celle che utilizziamo sono adeguate per i range di concentrazione attesi?

Quali rapporti di diluizione vengono utilizzati?

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7.4.2 A set of at least five calibration solutions (preferably more) with precisely known concentrations of FAME (4.1) in FAME free middle distillate (4.2) shall be prepared for the measurement range of interest by weighing FAME into appropriate graduated flasks and filling to the mark with FAME free middle distillate.

Quanti punti di calibrazione usiamo per la costruzione della retta?

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This method detects all FAME components by measurement of the C=O IR absorption at approximately 1745 cm⁻¹ and C8 to C22 molecules, as specified in EN 14214. The accuracy of the measured results depends on the best match of molecular weights (proportional to chain length) between the FAME material used for calibration and the FAME material in the analyzed sample. This means that presence of FAME with lower average chain length, determined with a calibration using FAME of longer average chain length, will result in considerable overestimation, and vice versa. For a correct result it is recommended to use FAMEs for calibration (4.1) that have a similar average molecular weights to the product being tested.

Quale FAME viene utilizzato per la costruzione della retta di taratura?

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Based on the FAME content (X) and the relevant normalised extinction coefficients E_{corr} (Y), for all calibration solutions the straight calibration line is calculated by linear regression using the model according to Formula (2):

$$Y(i) = a \times X(i) + b \quad (2)$$

With

$Y(i)$ is the determined corrected extinction E_{corr} for calibration sample (i);

$X(i)$ is the adjusted FAME content, in grams FAME per litre (g FAME/l), of calibration sample (i);

a, b are the regression coefficients (slope and intercept) obtained from the linear regression.

The regression coefficient b (“Y-axis intercept”) should ideally be zero in case of accurate work. However, it shall not be set to zero at random. Deviation from zero results from the regular statistical spread of the measuring points around the line of best fit and from the leverage of calibration samples with higher FAME contents. In case of high or striking values for the y-axis intercept the calibration should be rechecked carefully.

Other calibration models are not permitted.

Quanto è il coefficiente R2 della retta di taratura?

Il coefficiente b della retta risulta uguale a zero oppure no?

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7.6.1 General instructions

If multiple scans are possible, at least 16 scans (identical for all records, see also 7.4.1) shall be used. All further steps apply to both the calibration samples and for the samples to be tested. It is important that all other settings of the IR spectrometer are also retained.

7.6.2 Background and reference spectrum

At every calibration a background spectrum shall be taken and recorded as a reference spectrum for the compensation which is carried out with every measurement. For that the appropriate base fuel for the relevant type of sample (refer to 7.4.1 and 4.2) shall be used.

Quante scansioni IR del campione facciamo?

Eseguiamo il background spectrum ad ogni taratura?

Quale appropriate base fuel utilizziamo?

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7.6.4 Determination of the Extinction and the corrected Extinction

First of all a tangent is drawn between approximately $1\,670\text{ cm}^{-1}$ and approximately $1\,820\text{ cm}^{-1}$ as base line. Thereafter the extinction E_{meas} is determined as the vertical distance from the base line to the absorption band maximum at $1\,745 \pm 5\text{ cm}^{-1}$. For the assessment, the correct definition of the base line, a background correction as sound as possible and sufficient signal-to-noise ratio shall be given attention to. During calculation the used dilution rates shall be considered accordingly. The corrected extinction E_{corr} is the extinction measured in the IR spectrum (E_{meas}), recalculated (normalised or adjusted) for the undiluted sample.

E_{corr} is calculated according to:

$$E_{\text{corr}} = E_{\text{meas}} \cdot \left(\frac{V_{\text{VF}}}{V_{\text{SV}}} \right) \quad (3)$$

and applies only for the specific cell used.

V_{VF} is the volume of the volumetric flask in millilitres (ml) used for diluting the sample

V_{SV} is the sample volume in millilitres (ml) for dilution.

If an undiluted sample is tested the dilution factor is equal to one. In that case, for both volumes a value of 1,0 shall be used.

NOTE Unlike to the extinction E_{meas} which should be in the linear extinction range of the detector (see above remarks) the normalisation on undiluted samples according to Formula (3) may lead to significantly higher values for E_{corr} if higher FAME contents are tested.

Eseguiamo quanto indicato in paragrafo 7.6.4?

Il coefficiente E_{meas} viene determinato al massimo nell'intervallo $1740\text{-}1750\text{ cm}^{-1}$?

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Table A.1 gives additional information for the selection of path length and dilution factor. All values in Table A.1 should be regarded as approximate reference values. The cells with bold figures indicate the presumed possible main range for the test method. Pay attention to the linearity. The best results can be achieved by determining the extinction preferably in the medium extinction range. Therefore, based on preliminary tests the optimum combination of cell and dilution factor should be selected. These approximate values may well differ entirely due to varying cell materials.

**Utilizziamo la tabella A.1
(informative)?**