

CORRIGENDA

Corrigendum to Commission Directive 87/94/EEC of 8 December 1986 on the approximation of the laws of the Member States relating to procedures for the control of characteristics of, limits for and resistance to detonation of straight ammonium nitrate fertilizers of high nitrogen content

(Official Journal of the European Communities No L 38 of 7 February 1987)

ANNEX II

METHOD 1

'2.2. Principle and definition

In an Erlenmeyer flask, heat the sample from ambient temperature to 50 °C and maintain at this temperature for a period of two hours (phase at 50 °C). Thereupon cool the sample until a temperature of 25 °C is achieved and maintain at that temperature for two hours (phase at 25 °C). The combination of the successive phases at 50 °C and 25 °C forms one thermal cycle. After being subjected to two thermal cycles, the test sample is held at a temperature of 20 ± 3 °C for the determination of the oil retention value.'

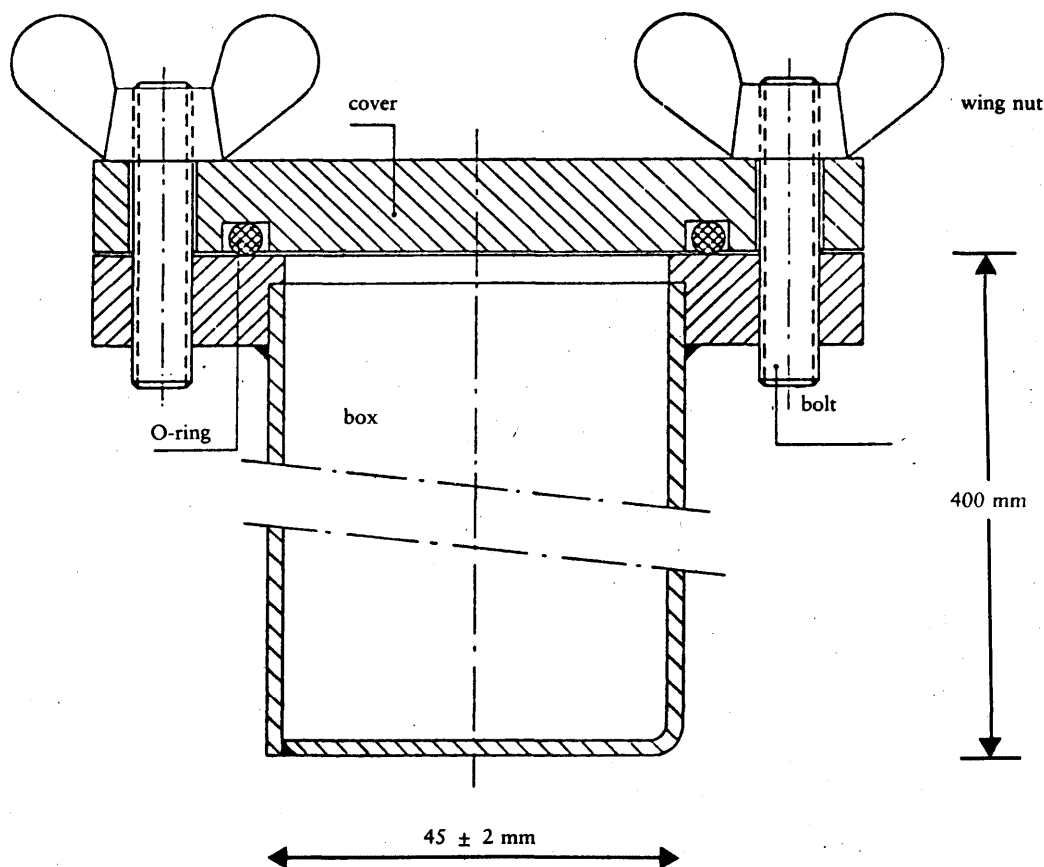
'3.2. Principle and definition

In a watertight box, heat the sample from ambient temperature to 50 °C and maintain at this temperature for a period of one hour (phase at 50 °C). Thereupon, cool the sample until a temperature of 25 °C is achieved and maintain at that temperature for one hour (phase at 25 °C). The combination of the successive phases at 50 and 25 °C forms one thermal cycle. After being subjected to the required number of thermal cycles, the test sample is held at a temperature of 20 ± 3 °C pending the execution of the detonability test.'

3.4. Procedure

'... One hour after the temperature at the centre has reached 25 °C heat the water to start the second cycle. In the case of two water baths, transfer the box to the other bath after each heating/cooling period.'

Figure 1



METHOD 3

- 3.1. Analytical-grade chromium (VI) trioxide CrO_3 .
- 3.2. Sulphuric acid, diluted to 60 % by volume — pour 360 ml of water into a one-litre beaker and carefully add 640 ml of sulphuric acid (density at 20 °C = 1,83 g/ml)
- 5.2. *Removal of carbonates*
'... At the end of this time there should be no more effervescence; if effervescence is seen, continue heating for 30 minutes. Allow solution to cool for at least 20 minutes with the nitrogen flowing through it.
...
Bubble a stream of nitrogen through for about 10 minutes. The solution must remain clear in the absorbers. If this does not happen, the carbonate removal process must be repeated.'
- 5.3. *Oxidation and absorption*
'... Place the crucible in the 600 ml beaker and add about 100 ml of boiled water (3.10). Introduce 50 ml of boiled water into each of the absorbers and pass nitrogen through the distributors for five minutes. ...'
- 5.4. *Measurement of the carbonates originating from organic material*
'... Add hydrochloric acid (3.5) drop by drop until the pink colour just disappears. Stir the solution well in the crucible to check that the pink colour does not reappear. ...'

METHOD 5

6. **Expression of results**
'Report the mean of the two values obtained for A on the one hand and for A + B on the other.'

METHOD 6

- 3.1. Acetone AR.'

METHOD 7

- 5.3.2. Preparation of the calibration solutions
By diluting the standard solution (3.6.1) with 0,5 M hydrochloric acid solution (3.3), prepare at least five standard solutions corresponding to the optimal measuring range of the spectrophotometer (0 to 5,0 $\mu\text{g/mlCu}$). Before making up to the mark, add to every solution ammonium nitrate (3.4) to give a final concentration of 100 mg per ml.'
- 5.4. *Measurement*
'Set up the spectrophotometer (4) at a wavelength of 324,8 nm. Use an oxidizing air-acetylene flame. Spray successively, in triplicate, the calibration solutions (5.3.2), the sample solution and the blank solution (5.3.1), washing the instrument through with distilled water between each spraying. ...'

ANNEX III

2. Principle

The test sample is confined in a steel tube and subjected to detonation shock from an explosive booster charge. Propagation of the detonation is determined from the degree of crushing of lead cylinders on which the tube rests horizontally during the test.'

- 3.6. Six lead cylinders
Diameter: 50 (\pm 1) mm
Height: 100 to 101 mm
Materials: soft lead, at least 99,5 % purity'

'3.10. Wooden disc

Diameter : 92 to 96 mm. diameter to be matched to the internal diameter of the plastic or cardboard cylinder (3.8)

Thickness : 20 mm'

'3.12. Dressmaking pins (maximum length 20 mm)'

4.1.1.2. '... Then insert a small dressmaking pin (3.12) transversally into the textile sleeve of each length of cord 5 to 6 mm from the end and apply adhesive around the outside of the lengths of cord in a band 2 cm wide adjacent to the pin. ...'

4.1.1.3. '... Adjust the height of the cylinder (64 to 67 mm) so that its top edge does not extend beyond the level of the wood. Finally, fix the cylinder to the wooden disc, for instance with staples or small nails, around its entire circumference.'

'4.1.2.2. Preparing the booster charge

Place the plastic explosive (3.1) into the cylinder (3.8) standing upright on a level surface, then press it down with a wooden die to give the explosive a cylindrical shape with a central recess. Insert the compressed pellet into this recess. Cover the cylindrically shaped explosive containing the compressed pellet with a wooden disc (3.10) having a central hole 7,0 to 7,3 mm in diameter for insertion of a detonator. Fix the wooden disc and the cylinder together with a cross of adhesive tape. Ensure that the hole drilled in the disc and the recess in the compressed pellet are coaxial by inserting the wooden rod (3.11).'

'4.3. *Filling and charging the steel tube*

(See figures 1 and 2)'

4.3.2. '...'

Repeat this charging method with another portion of the test sample. Finally, a further addition shall be made such that, after compaction by raising and dropping the tube 10 times and a total of 20 intermittent hammer blows, the charge fills the tube to a distance of 70 mm from its orifice.

...'

'4.4. *Positioning of the steel tube and lead cylinders (see figure 3)'*

4.4.2. '...'

Note : Make sure that the tube is in contact with all six lead cylinders ; a slight curvature of the tube surface can be compensated for by rotating the tube about its longitudinal axis ; if any of the lead cylinders is too tall, tap the cylinder in question carefully with a hammer until it is the required height.'

4.6. '...'

Record, for each of the marked lead cylinders, the degree of crushing expressed as a percentage of the original height of 100 mm. If the cylinders are crushed obliquely, record the highest and the lowest values and calculate the average.'

'4.7. A probe for continuous measurement of the detonation velocity can be used ; the probe should be inserted longitudinally to the axis of the tube or along its side wall.'

5. **Test report**

'...'

— the temperature of the tube and the sample shortly before firing,
— the packing density (kg/m³) of the sample in the steel tube,

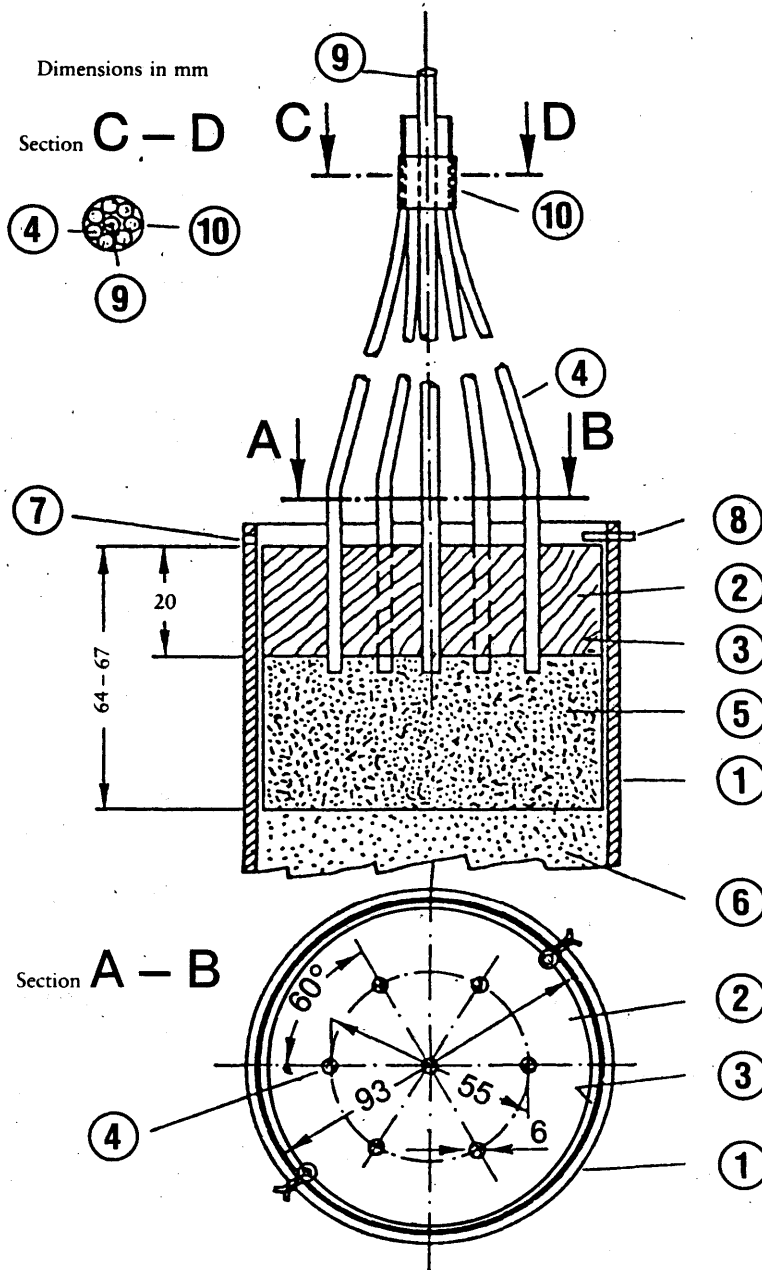
...'

'5.1. *Evaluation of test results*

If, in each firing, the crushing of at least one lead cylinder is less than 5 %, the test shall be considered conclusive and the sample in conformity with the requirements of Annex II to Directive 80/876/EEC.'

Figure 1

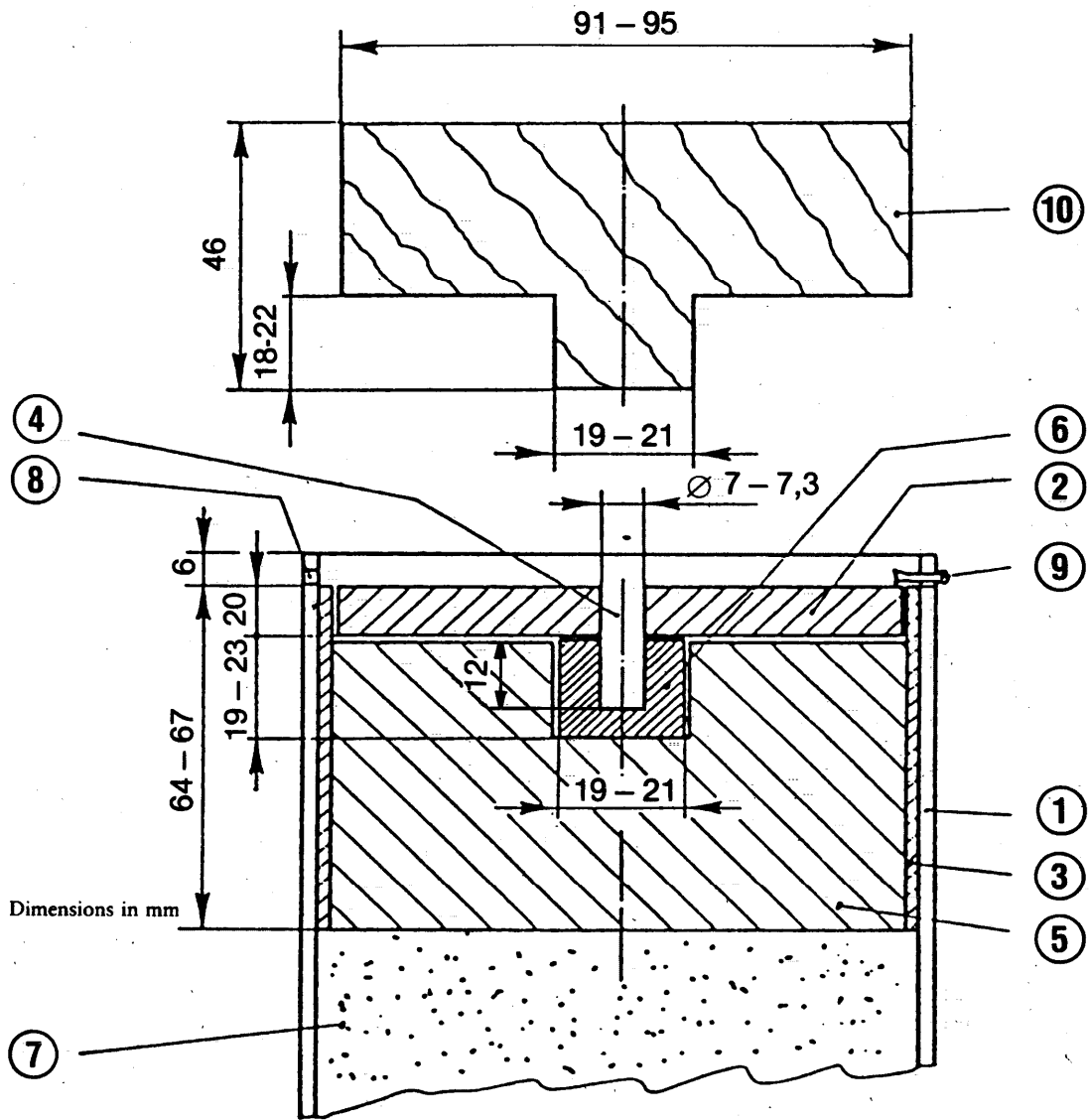
Booster charge with seven-point initiation



- | | |
|---------------------------------|---|
| ① Steel tube | ⑥ Test sample |
| ② Wooden disc with seven holes | ⑦ 4-mm-diameter hole drilled to receive split pin ⑧ |
| ③ Plastic or cardboard cylinder | ⑧ Split pin |
| ④ Detonating cords | ⑨ Wooden rod surrounded by ④ |
| ⑤ Plastic explosive | ⑩ Adhesive tape for securing ④ around ⑨ |

Figure 2

Booster charge with central initiation



Dimensions in mm

- ① Steel tube
- ② Wooden disc
- ③ Plastic or cardboard cylinder
- ④ Wooden rod
- ⑤ Plastic explosive
- ⑥ Compressed pellet
- ⑦ Test sample
- ⑧ 4-mm-diameter hole drilled to receive split pin ⑨
- ⑨ Split pin
- ⑩ Wooden die for ⑤