



**Committee of Experts on the Transport of Dangerous Goods
and on the Globally Harmonized System of Classification
and Labelling of Chemicals****Sub-Committee of Experts on the Transport of Dangerous Goods****Forty-eight session**

Geneva, 30 November – 9 December 2015

Item 2 (c) of the provisional agenda

Explosives and related matters: review of tests in parts I and II of the Manual of Tests and Criteria**On the use of the minimum burning pressure test as a
replacement for some of the Series 8 Tests****Transmitted by the expert from Canada¹****Introduction**

1. There is considerable dissatisfaction with the current Series 8 tests for ANEs. The Koenen test (8(c)) attempts to assess fire hazards but it does not work well for ANEs, in part because the orifice often becomes blocked with sample during testing. From the recent report of the Working Group on Explosives of the forty-seventh session of the Sub-Committee (informal document INF.53 (47th session)) there was a general consensus that the Koenen test is not suitable for evaluating ANEs and that research into what test might be a suitable replacement should be considered. Thus, a repeatable, small-scale test that could assess the hazards posed by exposure of ANEs to fire is highly desirable. The Vented Pipe Test (8(d)), which also attempts to assess fire hazards, requires large test facilities due to the possibility of explosion of the large mass of sample tested (60 kg) and its repeatability is poor.

2. At the thirty-seventh session of the Sub-Committee, the expert from Canada provided a description of the Canadian Explosives Research Laboratory (CanmetCERL) Minimum Burning Pressure (MBP) test. As part of this description provided in informal document INF.41 (37th session), several test results were presented for a wide variety of common emulsion formulations, while demonstrating the repeatability of test results. It was demonstrated that the results show a clear potential for the CanmetCERL MBP test to

¹ In accordance with the programme of work of the Sub-Committee for 2015–2016 approved by the Committee at its seventh session (see ST/SG/AC.10/C.3/92, paragraph 95 and ST/SG/AC.10/42, para. 15).

differentiate among ANEs that would be typical precursors for “bulk” emulsion explosives or to “packaged” emulsion explosives. Insensitive ANEs that are typically transported in tanks have MBPs in the range 6 - 12 MPa (855 to 1725 psig), while ANEs that are normally used in packaged products have MBPs in the range 0.45 – 3 MPa (50 – 420 psig).

3. The attractiveness of the MBP test within Series 8 is based on the fact that, ANEs having a high MBP are harder to ignite, support combustion less readily and show a lower propensity for deflagration-to-detonation behaviour than ANEs having a low MBP. On a more pragmatic basis, the test differentiates between those materials that have historically been considered safe to transport in bulk and those that have not. As demonstrated by the results in Section 9 and the analyses previously provided in informal document INF.41 (37th session), the water content is the major factor controlling the MBP value of ANEs. Other ingredients such as sodium nitrate or urea can have a significant effect on the MBP value.

Proposal

4. The expert from Canada proposes to amend Test Series 8 in order to replace the current Tests 8 (c) (Section 18.6.1 *Koenen Test*) and 8 (d) (Section 18.7.1 *Vented Pipe Test* and Section 18.7.2 *Modified Vented Pipe Test*) with the CERL MBP Test described below in the annex. It is further proposed that inclusion in the UN 3375 and Division 5.1 be restricted to those products having a MBP above 5.6 MPa (800 psig).

Annex

The CanmetCERL MBP Test

1. *Introduction*

This test is used to determine the sensitiveness of a candidate ammonium nitrate emulsion or suspension or gel, intermediate for blasting explosive, to the effect of intense localised thermal ignition under high confinement.

This test also determines the suitability of the candidates for transport in tanks.

2. *Apparatus and materials*

The following items are needed:

(a) The samples should be loaded in small cylindrical steel pipes (so-called test cells) having a length of 7.6 cm and an internal diameter of at least 1.6 cm. Each test cell should have a 3-mm wide slit machined along the axis to allow combustion gases to escape during the tests (Figure 1). The interior of each test cell should be painted with high-temperature non-conductive paint. Introduction of the sample into the cell should be done with caution to avoid causing crystallization of the sample and introducing air voids in the sample. Once the ignition wire has been introduced in the sample (see (b) below), the ends of the cell are closed off with No. 0 neoprene stoppers which must be reamed at their inside face to accommodate the splice connectors of the ignition wire assembly.

(b) Ignition is provided by a 60/16 Ni/Cr wire having a diameter of 0.51 mm (nominal resistance of $5.5 \Omega \text{ m}^{-1}$ at 20°C) and a length of 7 cm. Both ends of the ignition wire should be spliced onto 50 cm lengths of 14 AWG solid core bare copper wire using appropriate butt-end splice connectors. The ignition wire should be introduced in the sample, along the axis of the test cell. The neoprene stoppers are then inserted in place and the bare copper wires are pulled apart and bent at a 90° angle in order to ensure the ignition wire is held straight onto the axis.

(c) The above test cell should be introduced in a pressure vessel so that the axis of the cell is held horizontal with the slit on top (Figure 2). A minimum volume of 4-L and an operating pressure resistance of 20.8 MPa (or 3000 psig) are recommended for this pressure vessel. The vessel must be equipped with two insulated rigid feedthrough electrodes capable of carrying an electric current up to 20 A and sealed so as to have a pressure rating equivalent to that of the vessel itself. For safety reasons, it is recommended that the vessel be installed in a protected test room and should be equipped with a rupture disc assembly designed to vent the vessel at a pressure slightly lower than its maximum operating pressure. The vessel should also be equipped with an inlet and an outlet. In order to vent the vessel after a test, the outlet should be equipped with a high-pressure valve that can be operated remotely. The inlet should be used to pressurize the vessel to a predetermined initial pressure before the test. For convenience, it is recommended that the vessel also be equipped with a 0-25 MPa pressure transducer.

(d) A gas manifold system operated from a nearby protected room (the instrument room) capable of pressurizing the pressure vessel to a chosen initial pressure using pressurized cylinders of argon. For convenience, this manifold should be equipped with a needle valve that can be used as a bleed valve to adjust the initial pressure in the vessel.

- (e) A constant current power supply capable of delivering a constant current up to 20 A. The current can be monitored by measuring the voltage across a high precision shunt resistor (few m Ω) connected in series with the ignition wire.
- (f) An oscilloscope or PC-based data acquisition system capable of acquiring the pressure transducer signal and the ignition wire current. Minimum acquisition rate should be 100 Hz for time periods up to 5 minutes.
- (g) A multi-meter capable of measuring electrical resistance in the range 0.1 Ω to 10 M Ω .

3. *Testing Procedure*

3.1. A test cell prepared as in 2 (a) and (b) above is introduced in the pressure vessel with its axis being horizontal. The bare copper wires from the cell are connected to the vessel's electrodes inside the vessel and the vessel is closed.

3.2 Using the multi-meter (see 2 (g) above) the operator should check that there is no electrical contact between each electrode and the body of the pressure vessel. Once this has been established, the leads from the power supply (see 2 (e) above) are connected to the electrodes. If any contact is detected between the electrodes and the body of the vessel, the reason(s) for it must be found and the contact eliminated before testing can proceed.

3.3 The operator exits the test room and enters the instrument room. The vessel outlet is closed while the vessel inlet is opened. The vessel is then remotely pressurized, from the instrument room, approximately to the required initial pressure for the test. If this is the first test with a given substance, this pressure should be an educated guess as the expected MBP, based on the formulation of the sample. The inlet is then closed and the vessel is left pressurized for several minutes before ignition in order to check that the system has no significant leaks. Once this is established, the pressure is finely adjusted to the required initial value and the vessel inlet is closed.

3.4 The data acquisition (or oscilloscope) is then started manually and a 10.5 A current is allowed to flow through the ignition wire. The current should remain on for a few seconds until the sample ignites and melts the ignition wire. When this happens, the power supply should be shut off.

3.5 If the sample burns completely (combustion front reaching wall of the test cell; small amount of sample can be left on the neoprene stoppers), the result is deemed to be a 'go'. The pressure should be decreased for the next test. Otherwise the result is deemed to be a 'no-go' and the pressure should be increased for the next test (Figure 3). The pressure record from the transducer can also be used as evidence of sustained combustion or not (Figure 4).

3.6 Once the test is completed, the outlet valve is opened and all combustion gases should be vented to an exhaust system. A slow purge with argon for a few minutes is also recommended to remove all toxic gas species before opening the vessel.

3.7 The leads from the power supply are disconnected from the vessel's electrodes and the vessel is opened. The test cell is recuperated and all visual observations are noted. These evidences can also be further documented by taking photographs. The vessel is then cleaned thoroughly.

3.8 Steps 3.1 to 3.7 are repeated while gradually decreasing the pressure increments (or decrements) until the MBP has been determined to the desired degree of precision (see typical examples below). A minimum of 12 tests using this 'up-

and-down' methodology should be performed. The MBP should be quoted as the mean between the initial pressure of the highest 'no-go' event and that of the lowest 'go' event (Figure 5).

4. *Test criteria and method of assessing results*

The result of the test is considered positive (“+”) if the measured MBP of the candidate ammonium nitrate emulsion or suspension or gel, intermediate for blasting explosive is greater or equal to 5.6 MPa (or 800 psig). In this case the candidate intermediate can be included in UN 3375 and Division 5.1, and is considered to be suitable for transport in tanks.

If the measured MBP is lower than 5.6 MPa (or 800 psig), the result is considered negative (“-”).

5. *Examples of results*

Substances	MBP/MPa(psi)	Result
69.75 ammonium nitrate / 24.8 water / 5.45 oil+wax	14.24 (2051)	+
77.82 ammonium nitrate / 16.6 water / 5.58 oil+wax	8.69 (1246)	+
66.36 ammonium nitrate / 10.36 sodium nitrate / 17.9 water / 5.38 oil+wax	4.68 (649)	-
69.32 ammonium nitrate / 10.45 sodium nitrate / 14.70 water / 5.53 oil+wax	4.16 (589)	-
51.79 ammonium nitrate / 19.25 sodium nitrate / 20.6 water / 8.36 oil+wax	7.97 (1141)	+
57.66 ammonium nitrate / 18.42 calcium nitrate / 17.22 water / 6.70 oil+wax	7.79 (1115)	+
69.26 ammonium nitrate / 5.71 sodium nitrate / 6.42 sodium perchlorate / 7.80 water / 5.71 oil+wax / 5.00 Aluminum / 0.2 plastic μ S*	1.59 (216)	-
72.54 ammonium nitrate / 6.15 sodium perchlorate / 8.10 water / 5.26 oil+wax / 4.97 aluminum / 2.98 glass μ S*	0.94 (122)	-
76.92 ammonium nitrate / 7.46 urea / 10.20 water / 5.42 oil+wax	6.05 (863)	+

* μ S refers to micro-spheres

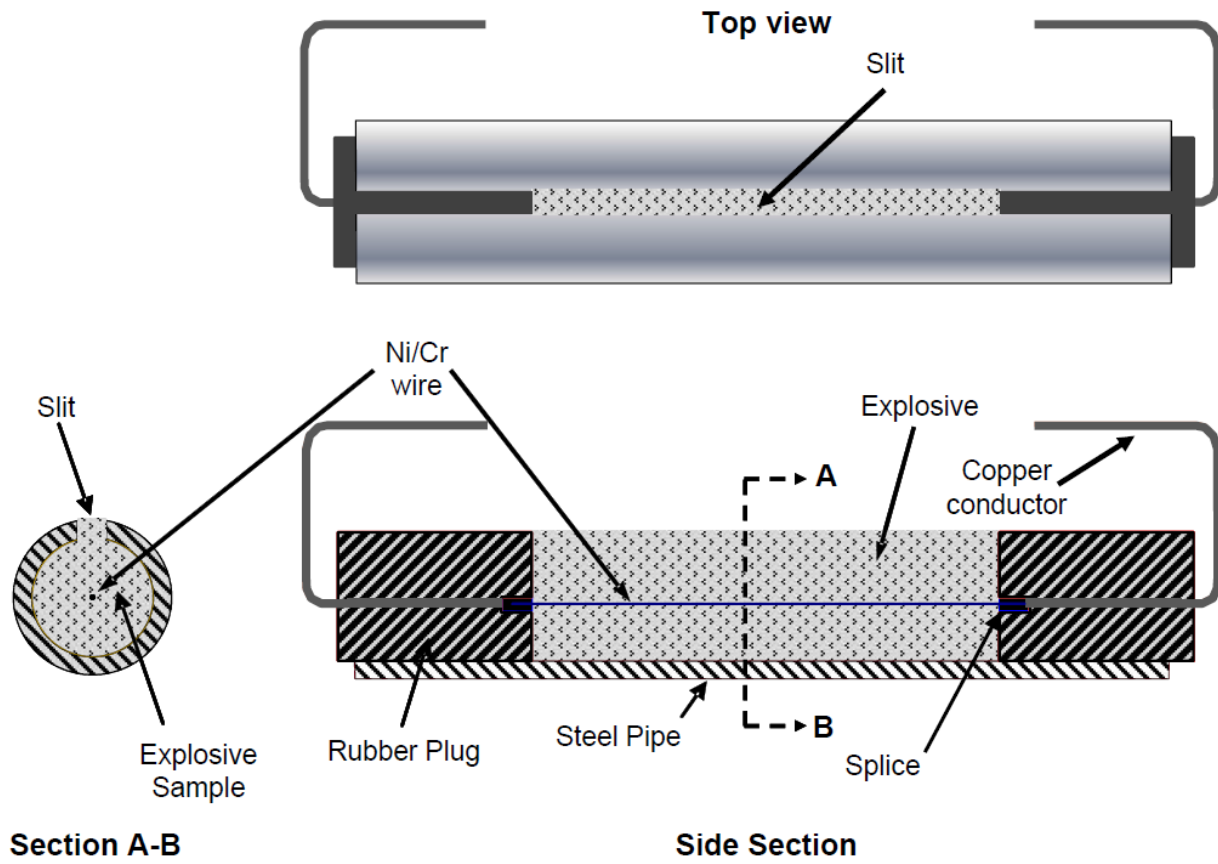


Figure 1
Test Cell for CanmetCERL MBP Test



Figure 2

Test cell mounted horizontally under the cover of the pressure vessel (copper conductors connected to vessel's fixed electrodes)

Go



No-Go

Figure 3

Typical aspect of the test cell after a 'go' (left) and 'no-go' (right) event

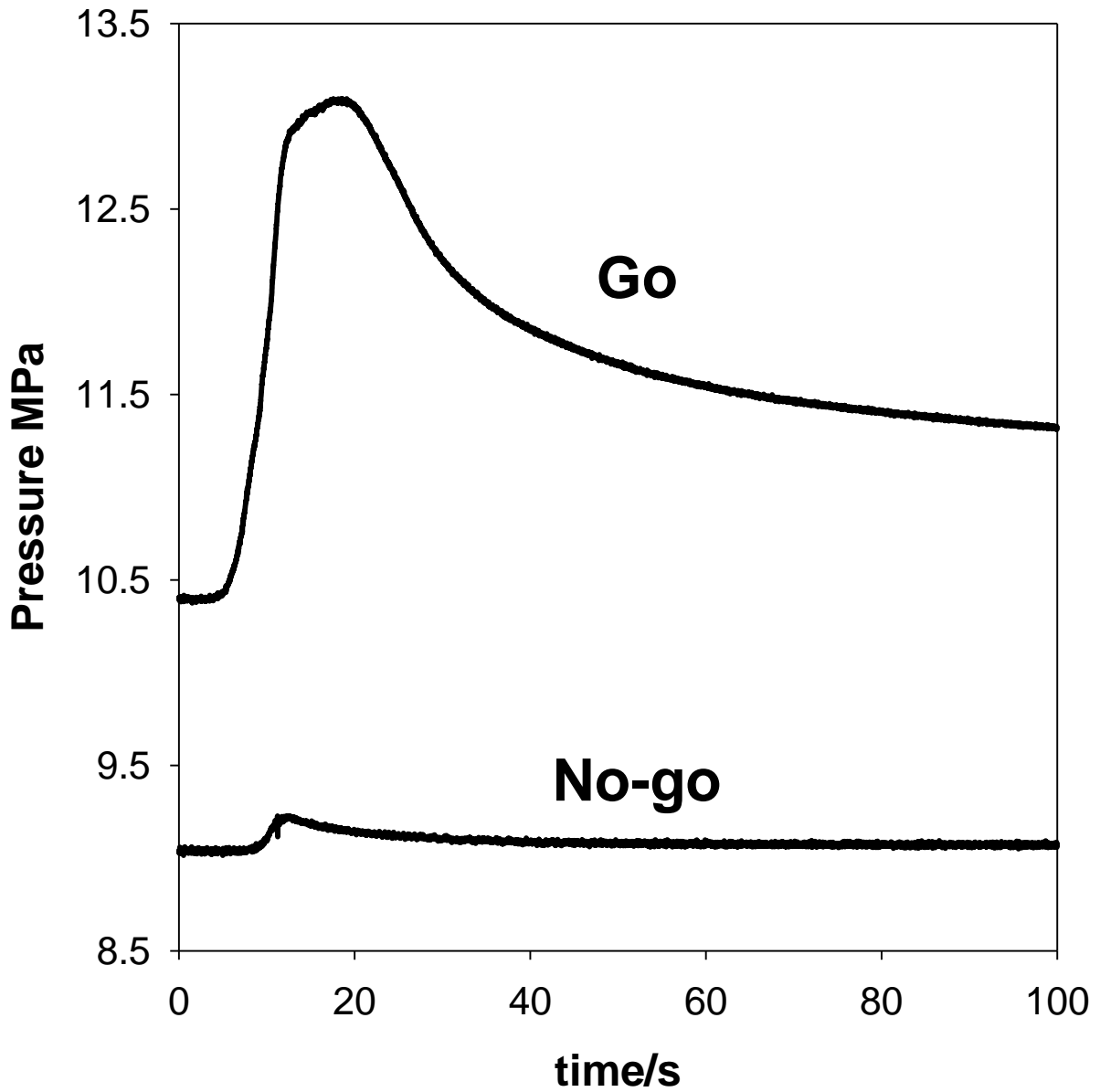


Figure 4
Typical pressure records for 'Go' and 'No Go' events

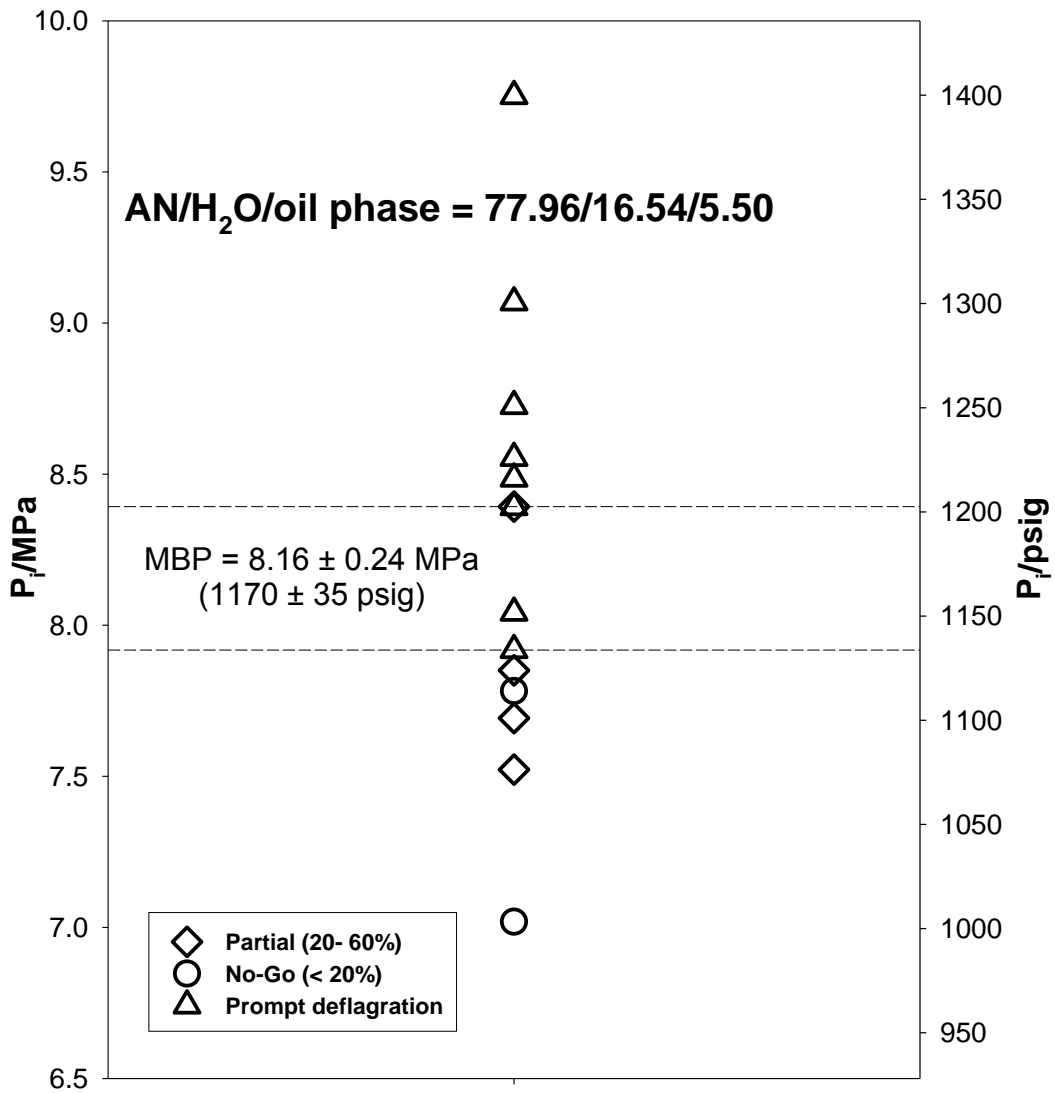


Figure 5
Typical MBP result for an ANE