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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

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Item 2 (b) of the provisional agenda
Explosives and related matters:
Improvement of test series 8

Amendments to UN 8(e) (Minimum Burning Pressure) Test

Transmitted by the expert from Japan*

I. Introduction

1. The test series 8 of the *Manual of Tests and Criteria*, in particular type 8 (e) test (hereafter referred to as "UN 8(e) test") is utilized to determine the sensitivity of substances, including ammonium nitrate emulsions (ANEs). The test determines a minimum burning pressure (MBP) of a substance to evaluate the sensitivity to thermal ignition under hermetically sealing. This test method was developed by Canada. This test has been conducted in Canada, Germany, France, South Africa, Japan, and other countries.

2. In the UN 8(e) test, a sample is loaded in a cylindrical steel pipe with a slit (a test cell) while avoiding crystallization of the sample and air voids in the sample. An ignition wire is introduced in the sample, and then both ends of the pipe are closed by stoppers.

3. The test cell is held in a pressure vessel. Argon is introduced into the pressure vessel such that the vessel is pressurized to the initial pressure of the test. A current of 10.5 A or higher is flown through the ignition wire.

4. If the sample burns completely, the result is a "go", otherwise a "no-go". The MBP is calculated as the average between the highest initial pressure of "no-go" and the lowest initial pressure of "go". If the MBP is less than 5.6 MPa, the result of the UN 8(e) test is positive ("+"), and the substance should not be classified in Division 5.1.

5. The National Institute of Advanced Industrial Science and Technology (AIST) of Japan performed the UN 8(e) test to introduce the test of the Japanese Industrial Standards (JIS). Its test results can be found in the annex.

6. Based on the test results by Japan, this document proposes amend the UN 8(e) test terms of both apparatus and the procedure of the test.

* A/78/6 (Sect. 20), table 20.5.

II. Discussion

7. The UN 8(e) test is determined to utilize a small cylindrical steel pipe, called a test cell, having a 3-mm wide slit along the axis. The test cell has a nominal length of 7.6 cm and an internal diameter of at least 1.6 cm. The test requires introducing the sample into the test cell, avoiding sample crystallization and air voids.

8. The test does not indicate the method to install the sample into the test cell. As some samples with low moisture are sticky, it is difficult to fill the sample into the test cell through the slit, avoiding air voids.

9. Japan examined how to introduce the sample into the test cell and adopted syringes as an auxiliary tool. In the UN 8(e) test demonstrated by Japan, samples were filled into the test cells with syringes, and the test was successfully performed.

10. The utilization of syringes as an auxiliary tool is proposed.

11. In the UN 8(e) test, the sample in the test cell is ignited by an ignition wire. The standard of the ignition wire is American Wire Gauge (AWG), i.e., a nominal diameter of 0.51 mm (nominal resistance of 5.5 Ω m^{-1} at 20 °C).

12. Countries that use the metric system face difficulties in supplying AWG products. Japan performed the UN 8(e) test utilizing an ignition wire in the metric system, which had a nominal diameter of 0.50 mm and a nominal resistance of 5.68 Ω m^{-1}.

13. Joule heat generated in the unit length of the metric wire increases by 4 per cent from that of AWG. The UN 8(e) test utilizing the metric wire is a slightly rigorous evaluation because the possibility of ignition of ANEs increases with the amount of heat. Also, the UN 8(e) test allows the flow of a current higher than 10.5 A such that a larger Joule heat is applied to the sample. However, because the variation in combustion conditions exceeds 4 per cent, the impact of a 4 per cent increase in energy input on the results is limited.

14. For metric wires, Japan proposes accepting ranges in diameter and resistance of an ignition wire.

15. The test cell is set in a pressure vessel, and the pressure vessel is pressurized with argon to an initial pressure. The pressure vessel is left for several minutes after closing the gas inlet for leak check.

16. Temperature increment due to adiabatic compression should be considered in the gas introduction. The gas temperature in the pressure vessel theoretically increases to 487 K from 293 K when the vessel is adiabatically pressurized by 14.24 MPa of argon, which is MBP of substance 9 in section 18.8.1.5 of the *Manual of Tests and Criteria*. It has been reported that higher sample temperatures may result in lower MBP. Differences in sample temperature due to differences in pressure could lead to inconsistent experimental results.

17. To avoid the effect of high ambient temperature on MBP, Japan proposes leaving the pressure vessel for an appropriate duration until the gas temperature drops to room temperature. A thermocouple should be equipped in the pressure vessel to monitor the gas temperature.

III. Proposal

18. It is proposed to amend the first paragraph of section 18.8.1.2.1 of the *Manual of Tests and Criteria* as presented below (new text is indicated as underlined text):

"The samples should be loaded in small cylindrical steel pipes (so-called test cells) having a nominal 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 18.8.1). The interior of each test cell should be painted with high-temperature non-conductive paint. It is recommended that the sample is introduced into the test cell through the slit with a syringe, if the sample has not so high viscosity. 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 18.8.1.2.2), the ends of the cell are closed off with No. 0 neoprene, or similar, stoppers which must be reamed at their inside face to accommodate the splice connectors of the ignition wire assembly."

19. It is proposed to amend the first paragraph of section 18.8.1.2.2 of the *Manual of Tests and Criteria* as presented below (new text is indicated as underlined text):

"Ignition is provided by a Ni/Cr wire having a nominal diameter of 0.50 – 0.51 mm (nominal resistance of 5.50 – 5.75 $\Omega\ m^{-1}$ 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 (American Wire Gage) (1.628 mm) or larger 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 stoppers are then inserted in place."

20. It is proposed to amend the first paragraph of section 18.8.1.2.3 of the *Manual of Tests and Criteria* as presented below (new text is indicated as underlined text):

"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 18.8.2). A minimum volume of 4 litres 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. The vessel should also be equipped with an inlet and an outlet. 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 and a Type-K thermocouple to measure the gas temperature."

21. It is proposed to amend the first paragraph of section 18.8.1.3.3 of the *Manual of Tests and Criteria* as presented below (new text is indicated as underlined text):

"The vessel outlet is closed while the vessel inlet is opened. The vessel is then pressurized 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 in order to check that the system has no leak. Once this is established, the pressure is adjusted to the required initial value and the vessel inlet is closed. As an adiabatic gas compression raises its temperature, the test should be started after the gas temperature drops to room temperature. The value of the pressure transducer is then recorded as the initial pressure."

Annex

UN 8(e) Test**

1. AIST conducted the UN 8(e) test for two types of ANEs, low- and high-water contents. Table 1 presents the compositions of ANEs. The sample corresponded to the substances listed in the *Manual of Tests and Criteria* (MTC) as examples.

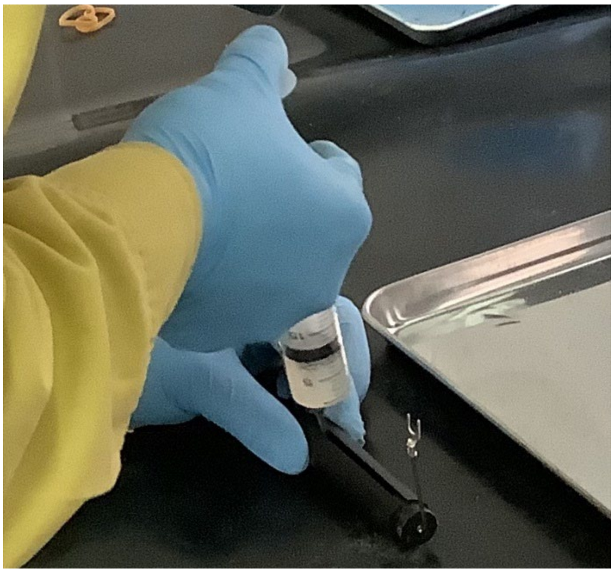
Table 1
Composition of ANEs

Composition [wt.%]	Low-water ANE (MTC #3)	High-water ANE (MTC #6)
ammonium nitrate AN	72.1	66.9
sodium nitrate SN	11.2	10.4
H_2O	11.2	17.2
Oil/Emulsifier	5.5	5.5

2. The samples were filled into the test cells with syringes (see Figure I). Ignition wires were installed at the center axis of the test cells (see Figure II). Metrenic Ni-Cr wires were utilized as the ignition wires instead of AWG wires. The nominal diameter and resistance of the wires were 0.50 mm and 5.68 Ω m^{-1}, respectively.

Figure I

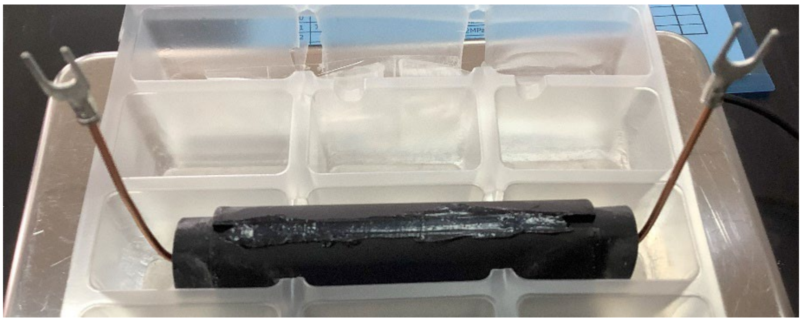
Installation of sample into test cell with syringe



** The author of the test results gave the authorization to use the materials contained in the annex for the purpose of the discussion at the sixty-fourth session of the Sub-Committee of Experts on the Transport of Dangerous Goods. For reproduction permission and all other issues, please contact ken.okada@aist.go.jp.

Figure II

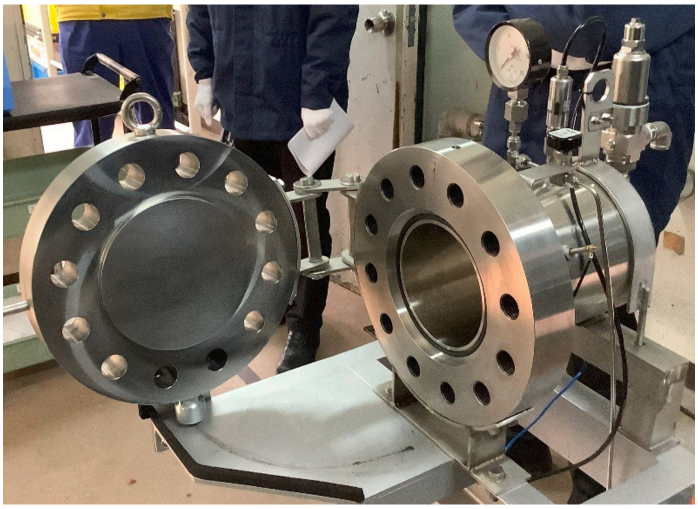
Test cell



3. The pressure vessel has an internal diameter of 160 mm, a depth of 220 mm, and a volume of 4.42 litres (see Figure III). The vessel was equipped with a pressure transducer and thermocouple to monitor the state of the gas. The test cell was horizontally held in the vessel by hanging the ignition wire from the terminal inside the vessel (see Figure IV). The vessel was pressurized by argon and left for several minutes until the gas temperature dropped to room temperature. Then, a current of 10.5–12.0 A was flown through the ignition wire.

Figure III

Pressure vessel



4. Japan performed 12 and 19 tests for the low- and high-water content ANEs (see Figures V and VI). In the initial stage of the tests, the initial pressure was set with reference to the MBP described in the MTC. The initial pressure of the vessel was increased or decreased to properly evaluate MBP according to GO/NOGO results.

Figure IV

Installation of test cell in pressure vessel

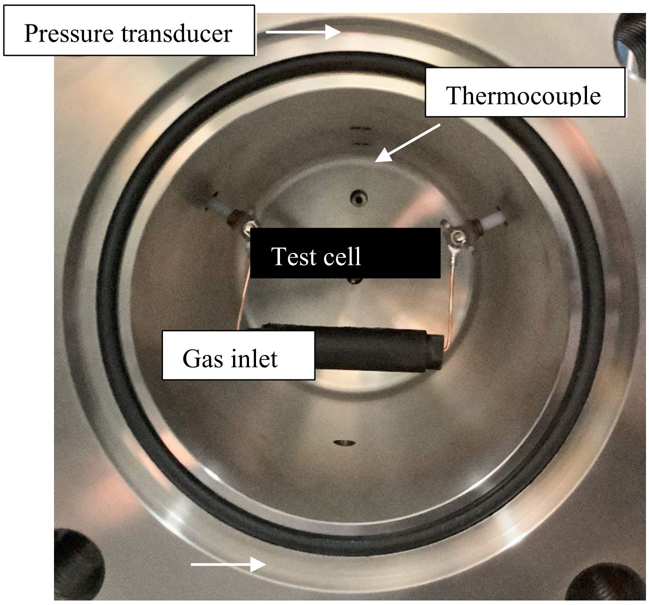


Figure V

Test history of low-water content ANE

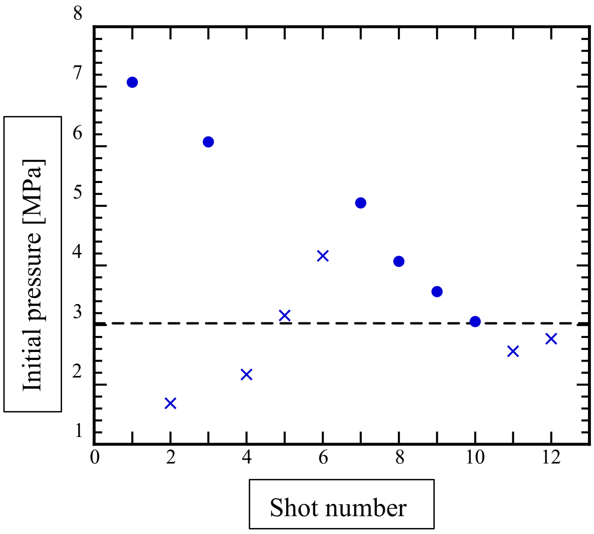
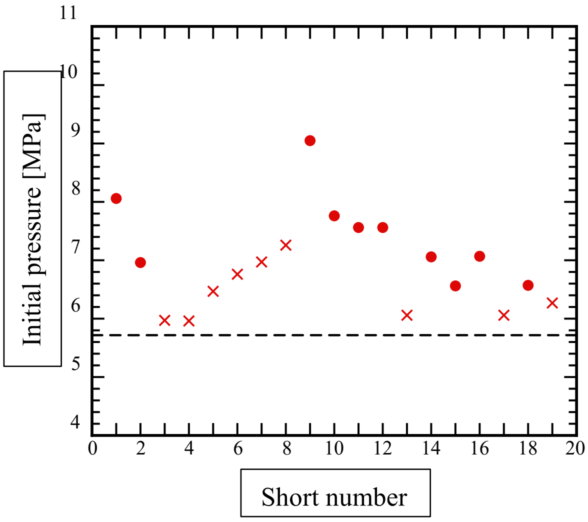


Figure VI

Test history of high-water content ANE



5. Figures VII and VIII present the photos and typical waveforms of GO result (low-water ANE, the initial pressure was 5.05 MPa). After the test, the ignition wire fused. No ANE remained in the test cell such that the weight of ANE in the test cell significantly decreased. The pressure vessel had water droplets at the top and water puddles due to chemical reactions on the bottom. The pressure and temperature in the pressure vessel increased rapidly when the current was applied.

Figure VII

Photos of GO trial

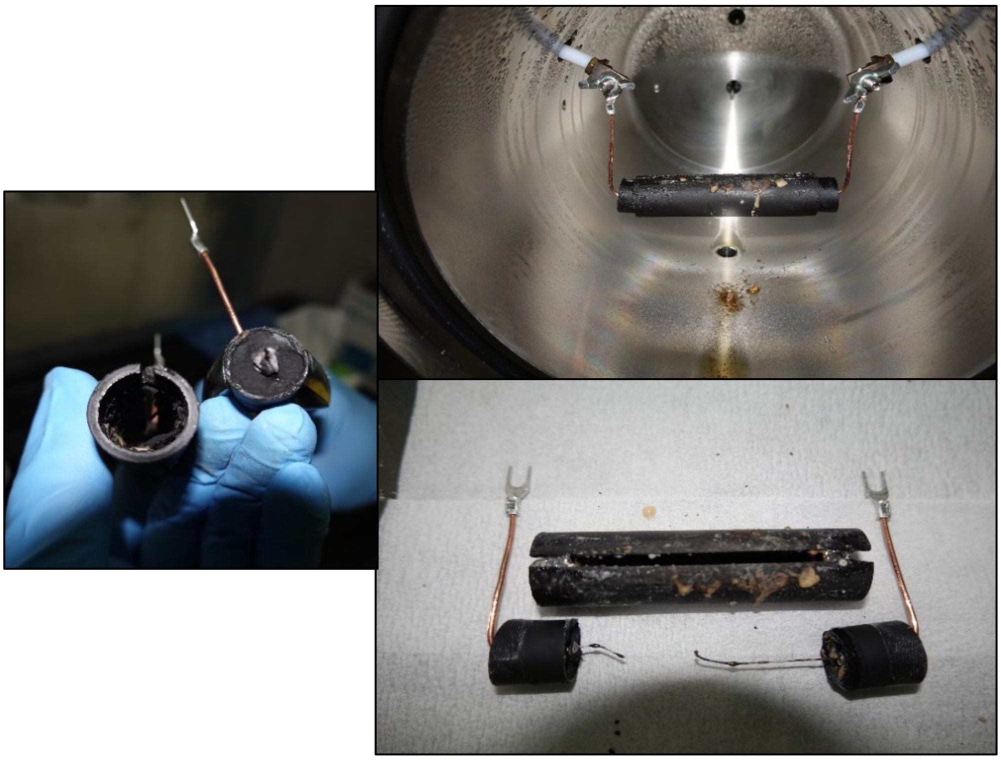
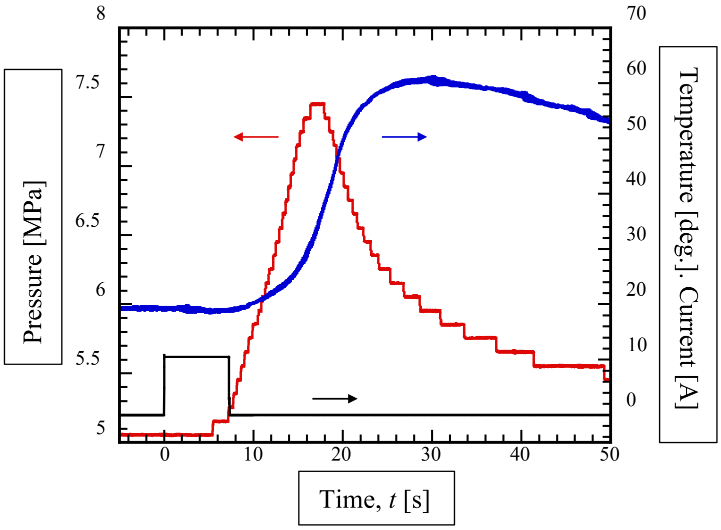


Figure VIII

Waveforms of GO trial



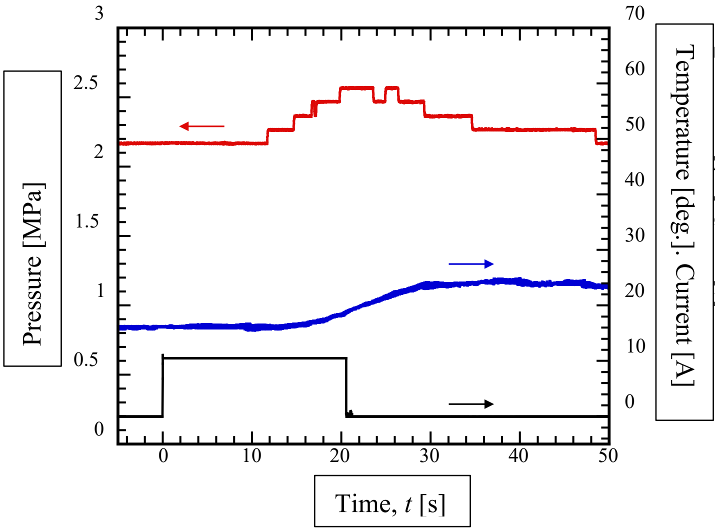
6. Figures IX and X present the photos and typical waveforms of NOGO result (low-water ANE, the initial pressure was 2.17 MPa). After the test, the ignition wire fused. Unburned ANE remained in the test cell, and the weight of ANE in the test cell did not decrease much. No water droplets appeared at the top in the pressure vessel, and a part of ANE spouted out on the bottom. The pressure and temperature in the pressure vessel hardly increased.

Figure IX

Photos of NOGO trial

Figure X

Waveforms of NOGO trial

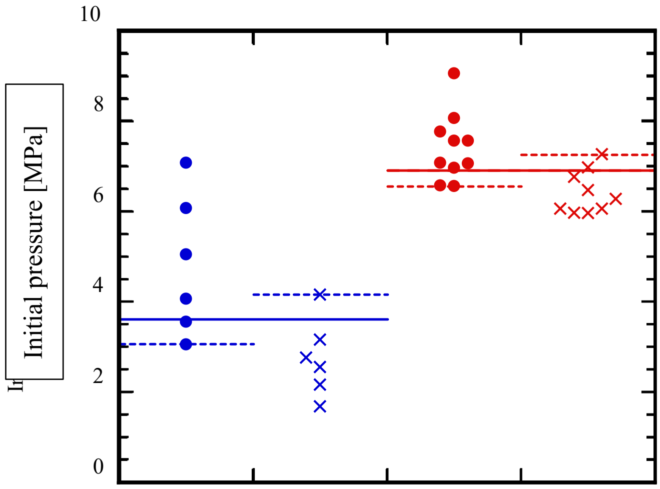


7. Figure XI illustrates GO/NOGO distributions and MBPs of the ANEs. The high initial pressure tended to be GO, and that of low initial pressure did NOGO. The range of the initial pressures overlapped for GO and NOGO. MBPs of the low- and high-water content ANEs were obtained as 3.61 MPa and 6.91 MPa, respectively.

8. To evaluate the scattering of the test, the standard deviation of the initial pressures of GO, which was less than MBP, and those of NOGO, which was higher than MBP, was calculated. The standard deviations were 0.45 MPa (12% of MBP) and 0.29 MPa (4% of MBP) for low- and high-water content ANEs, respectively.

Figure XI

GO/NOGO distributions



9. Table 2 compares MBPs and results obtained here with those listed in MTC. Figure XII compares MBPs obtained here with those of the previous studies. It also presents the MBP distribution of ammonium nitrate – sodium nitrate (AN-SN) emulsion, which has not been performed here. Both MBPs of AN and AN-SN emulsions linearly increased with the water content. The MBP of low-water content ANE almost coincided with the approximate line, whereas that of high-water content ANE was slightly higher than that of the line. Possible causes of the gap are the difference in oil, emulsifier, and viscosity of the samples. Also, the result of GO and NOGO are stochastic events. However, as indicated in Table 2, the results of the previous study were consistent with that of MTC. Therefore, Japan concluded that the UN 8(e) test here was performed correctly.

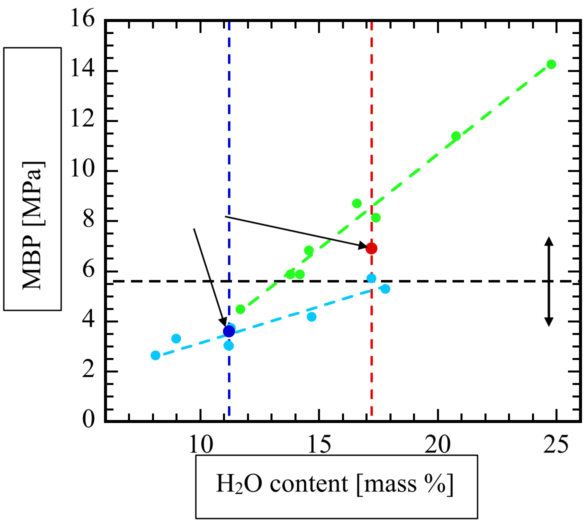
Table 2

Comparison of MBPs with previous works

MBP [MPa] (+/-)	Low-water content ANE	High-water content ANE
Present results	3.61 (+)	6.91 (-)
Ref 1 (MTC)	3.03 (+)	5.72 (-)

Figure XII

Comparison of MBPs with previous works



References:

1. “On the Use of the Minimum Burning Pressure Test as an Alternative Series 8 Test”, informal document INF.41 of the thirty-seventh session of the TDG Sub-Committee.

2. United Nations, *Manual of Tests and Criteria*, Rev.7 (2019).