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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

#### Sixty-fourth session

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Item 2 (h) of the provisional agenda

**Explosives and related matters:**

**Miscellaneous**

### **Termination time and method of assessing results for the methyl violet paper test in appendix 10 of the Manual of Tests and Criteria**

Submitted by the expert from China\*

#### **I. Introduction**

1. During the conversion of appendix 10 of the *Manual of Tests and Criteria* into a national standard on the stability of nitrocellulose (NC) mixtures, the expert from China found two problems. It was also found that, based on the current text, the assessment of the results when the recorded test time is exactly 30 minutes is controversial.
2. First, the termination time for the test is not fully specified. In accordance with the current text, the test can only be discontinued “when the salmon pink end point is attained in any of the papers” (see paragraph A10.3.4.3 in appendix 10 of the *Manual of Tests and Criteria*). However, in some cases, the mixtures might be very stable, so that none of papers changes the colour completely over a long period of time. The judgement can certainly be made that the mixture is stable, but the test should be continued, which is a waste of time. The expert from China suggests setting another termination time and allowing that the test can also be discontinued if none of the papers changed colour completely in 40 minutes. Two examinations after the threshold of 30 minutes, at 35 and 40 minutes, should be sufficient to determine the stability of the tested nitrocellulose mixture.
3. As China believes most testers will choose, in real practice, to terminate the test at somewhere after 35 minutes, when no complete colour change is observed. Thus, the proposed amendments in proposal 1 below will not compromise the effectiveness of the test, but will only make the test procedure more complete.
4. The second problem is that, based on the current text, assessing how the result should be interpreted when the recorded test time is exactly 30 minutes is controversial. The current provision addressing interpretation of the test result reads:

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



“A10.3.4.4 The test result is considered "+" and the substance is classified as unstable if the test paper completely changes colour in less than 30 min. If the colour change exceeds 30 min the result is "-" and the substance is classified as stable.”

5. It is not clear whether the 30 minutes here means the recorded test time or natural time. If the 30 minutes in A10.3.4.4 means the recorded test time, the test result should obviously be considered "-" and the substance classified as stable when the recorded test time is exactly 30 minutes. In the case this interpretation is correct, China proposes to amend paragraph A10.3.4.4 and the table in A10.3.5 as shown in option 1 of proposal 2, to improve the clarity of text.

6. On the contrary, if the 30 minutes in A10.3.4.4 means natural time, the test result should be considered "+" and the substance classified as unstable when the recorded test time is exactly 30 minutes. Because the examinations of test papers are at 5 minutes intervals, instead of continuous, we cannot know the exact time for a complete colour change. We can only know the test phenomena at each examination. The complete colour change must be achieved in more than 25 minutes but less than 30 minutes, if the end point has been reached at 30 minutes, i.e. the recorded test time is 30 minutes. In this latter case, China proposes to amend A10.3.4.4 and the table in A10.3.5 as shown in option 2 of proposal 2.

7. To understand the original intention of the Sub-Committee, the expert from China traced back relevant documents from the past. On the basis of this research, we tend to believe that the 30 minutes in A10.3.4.4 refer to the recorded test time.

8. Document ST/SG/AC.10/C.3/2017/35, containing a proposal to introduce the methyl violet paper test for the first time, submitted by Germany at the fifty-second session of the Sub-Committee, stated the following in paragraph 7:

*“The chemical and thermal stability of NC and its mixtures are to be tested by means of the following tests:*

*(a) Bergmann Junk test: measurement of the amount of nitrogen oxides released over a period of two hours at 132 °C, where the amount of nitrogen oxides released shall not exceed 2.5 ml of NO/ g NC; or*

*(b) Methyl Violet Paper test: measurement of the amount of nitrogen oxides released at 134.5 °C over a period of at least 30 minutes, where the methyl violet paper shall not have completely changed its colour.”*

The methyl violet paper has completely changed its colour over the period of 30 minutes, if the recorded test time is 30 minutes. The mixture should be regarded as unstable with a “+” result.

9. However, in informal document INF.6 submitted by the European Chemical Industry Association (Cefic) at that same session, the criteria was paraphrased as follows:

*“It is proposed to require a tested thermal stability for nitrocellulose mixtures of class 1 (UN 0340, UN 0341, UN 0342 und UN 0343) and class 4.1 (UN 2555, UN 2556, UN 2557 und UN 3380). A NC-mixture is classified as stable for transport*

*- if the quantity of NO gas formed in the Bergmann Junk test within 2 hours at 132 °C is not higher than 2.5 ml NO gas per g of NC, or*

*- if a test time of min. 30 minutes is achieved with the Methyl Violet Paper Test, before the test paper has changed its colour completely.”*

The 30 minutes was clarified as a test time. The mixture is regarded as stable when the recorded test time is 30 minutes, which is different from that in the German document.

10. It seems that the Sub-Committee was more in favour of the understanding of Cefic, since it adopted the proposal in document ST/SG/AC.10/C.3/2018/19 (as amended by informal document INF.67) submitted by Cefic at the fifty-third session.

11. China would also like to refer to the military specification (“mil spec”) for nitrocellulose (MIL-DTL-244C) from the United States of America, which was cited as the origin of the methyl violet paper test in informal document INF.24 from the Sporting Arms

and Ammunition Manufacturers' Institute (SAAMI) submitted at the fifty-first session. Section 3.4.3 of the specification reads:

*"3.4.3 Stability.*

*For all classes of nitrocellulose, the nitrocellulose shall meet one of the following:*

*a) maximum of 2.5 mL NO/g NC for the 132 °C Bergman-Junk test*

*b) minimum of 30 minutes on the 134.5 °C methyl violet paper test".*

From the context, we tend to understand 30 minutes here as the test time.

12. Nevertheless, China considers option 2 as being more conducive to ensuring safety, because longer time before complete colour changes means better stability.

13. Although the recorded test time of exactly 30 minutes is not always achieved in real practice, China believes that it provides the most important judgement threshold for the classifier, and that this needs to be clarified. Both options 1 and 2 below are acceptable for the expert from China. The Sub-Committee is invited to take a decision.

## II. Proposal 1

14. Amend paragraph A10.3.4.3 in appendix 10 to the Manual of Tests and Criteria as follows (new text appears in **bold underlined**):

"A10.3.4.3 The test time is then recorded (for example, if the violet paper is not completely changed in 25 min, but is completely changed in 30 min, the time of the test is recorded as 30 min). The test is discontinued when the salmon pink end point is attained in any of the papers, **or at 40 min if none of the papers changes colour completely.**"

## III. Proposal 2

15. Amend paragraph A10.3.4.4 and the table in A10.3.5 in appendix 10 of the Manual of Tests and Criteria as proposed in either options below (deleted text appears in ~~strikethrough~~ and new text in **bold underlined**):

### Option 1

16. If the test time of 30 minutes is to be interpreted as "-".

Amend A10.3.4.4 and the table under A10.3.5 as follows:

"A10.3.4.4 The test result is considered "+" and the substance is classified as unstable if the **recorded time for test the test paper to completely changes colour is** ~~is~~ less than 30 min. If the **recorded test time for the test paper to completely change** colour ~~change exceeds~~ **is not less than** 30 min the result is "-" and the substance is classified as stable."

"A10.3.5 Examples of results

<b><u>Test time</u></b>	<b><u>Result</u></b>
25 min	+
<b><u>30 min</u></b>	<b><u>-</u></b>
35 min	-

".

### Option 2

17. If the test time of 30 minutes is to be interpreted as "+".

Amend A10.3.4.4 and the table under A10.3.5 as follows:

“A10.3.4.4 The test result is considered "+" and the substance is classified as unstable if the **recorded test time for the** test paper **to** completely changes colour **is not more than** ~~in less than~~ 30 min. If the **recorded test time for the test paper to completely change** colour ~~change exceeds~~ **is more than** 30 min the result is "-" and the substance is classified as stable.”

“A10.3.5 **Examples of results**

<b>Test time</b>	<b>Result</b>
25 min	+
<b>30 min</b>	<b>±</b>
35 min	-

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#### IV. Sustainable development goals

18. As explained above, the aim of this proposal is to make the current provisions more complete and explicit, which can help achieving target 16.6 of sustainable development goal 16, to “develop effective, accountable and transparent institutions at all levels”.

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