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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

Fifty-seventh session Geneva, 29 June-8 July 2020 Item 10 (c) of the provisional agenda Issues relating to the Globally Harmonized System of Classification and Labelling of Chemicals (GHS): testing of oxidizing substances Sub-Committee of Experts on the Globally Harmonized System of Classification and Labelling of Chemicals

Thirty-ninth session Geneva, 8-10 July 2020 Item 2 (a) of the provisional agenda Classification criteria and related hazard communication: work of the Sub-Committee of Experts on the Transport of Dangerous Goods (TDG) on matters of interest to the GHS Sub-Committee

# Tests for oxidizing liquids and oxidizing solids improvement regarding consideration for particle size, friable or coated materials

### Transmitted by the expert from France\*

# Introduction

1. During its ninth session the Committee approved the programme of work of its two sub-committees for the biennium 2019-2020 (see ST/SG/AC.10/46, para 14; ST/SG/AC.10/C.3/108, paragraph 139 and ST/SG/AC.10/C.4/72, annex II). This programme of work includes the tests for oxidizing liquids and oxidizing solids.

2. The programme of work focuses on improving the testing of materials of different particle sizes distribution and coated materials, as well as improvements to the testing methods for the Tests O.1, O.2 and O.3.

3. During the fifty-fifth session of the Sub-Committee of Experts on the transport of Dangerous Goods the expert from France proposed a schedule for the work foreseen (see ST/SG/AC.10/C.3/2019/20 - ST/SG/AC.10/C.4/2019/4).

4. The work progress was reported during the fifty-fifth session (see informal documents INF.44 (55<sup>th</sup> session) and INF.16 (37<sup>th</sup> session)) and the fifty-sixth session (see ST/SG/AC.10/C.3/2019/68 – ST/SG/AC.10/C.4/2019/11 and informal documents INF.39 (56<sup>th</sup> session) and INF.18 (38<sup>th</sup> session)). The present document reports the latest progress.



<sup>\* 2020 (</sup>A/74/6 (Sect.20) and Supplementary, Subprogramme 2)

#### **Results of the work programme**

5. A joint effort from several international laboratories (14 labs from 8 different countries) to improve the test methods for oxidizing liquids and oxidizing solids i.e. UN Tests O.1, O.2 and O.3 is on-going for several years. France wishes to report on behalf of this pool of laboratories to the sub-committees on the progress of work related to the tests for oxidizing liquids and oxidizing solids.

6. At the end of 2018, a Round Robin Test (RRT) was launched focusing on several aspects of the UN tests on oxidizing solids. It comprised three main sets of tests (see para. 7 and 8 for the set number 1 and para. 9-11 for the sets number 2 and 3).

7. The set number 1 determined the oxidizing potentials for the reference mixtures for both UN Tests O.1 and O.3 by: a) individual time take (stop watch) and b) burning rate (gravimetry) for each burning trial. The aims of this set were: 1) to confirm that the oxidizing potentials of the reference mixtures for both tests, O.1 and O.3, are comparable for classification means after phase-out of formerly widely used WHATMAN CF11 as combustibles and 2) to see if performing the UN Test O.3 with the reference solid oxidizer (calcium peroxide) obtained from other suppliers besides the initially identified provider yielded comparable results.

- 8. From the data collected during the RRT, the following observations were made:
  - For the determination of the oxidizing potential of a sample both UN Tests O.1 and O.3 performed similarly well when based on the burning time taken by the operator with a stop watch.
  - The discriminatory power between PG III, PG II and PG I assessed by the burning time is comparable for both reference oxidizers (potassium bromate and calcium peroxide).
  - The determination of the oxidizing potential based on the burning rate (calculated from the mass loss of cellulose) is satisfactory with both UN Tests O.1 and O.3.
  - The discriminatory power between Not Division 5.1, PG III, PG II and PG I assessed by the burning rate is acceptable in all cases. But for a conclusive discrimination between PG II and PG I with UN Test O.3, the use of burning time or expert judgment might be helpful in some cases.
  - Comparison of test results obtained in UN Test O.3 with two different sources of calcium peroxide showed some variations which could not be fully explained yet. There may be a need to refine the specifications of calcium peroxide as reference material.

9. The sets number 2 and 3 determined the oxidizing potentials of coated and uncoated test sample (sodium percarbonate) on one hand, and of granulated, but uncoated test sample (sodium nitrate) on the other hand. The purpose was to assess if the following note: "In the case of a substance coated to reduce or suppress its oxidizing properties with a significant content (> 10 % by mass) of particles less than 500  $\mu$ m, two sets of tests should be conducted: tests conducted with the substance as presented and tests conducted with particles less than 500  $\mu$ m that were obtained from sieving the substance as presented. The substance should not be ground before sieving or testing. The final classification should be based on the test results with the most stringent classification" would be valid and valuable as better defining the approach to testing coated substances.

- 10. From the data collected during the RRT the following observations were made:
  - (a) The oxidizing potential for the sample of coated sodium percarbonate as received was slightly lower than that of the sample of uncoated sodium percarbonate as received.
  - (b) The oxidizing potentials for the samples of coated or uncoated sodium percarbonate:

- (i) were lower for the sample tested as received compared to the sieved  $< 500 \mu m$  sample and the milled sample,
- (ii) the sieved samples exhibit a slightly higher oxidizing potential compared to the samples as received,
- (iii) the milled samples exhibit a significantly stronger oxidizing potential compared to the samples as received.
- (iv) The oxidizing potentials for the milled samples of coated or uncoated sodium percarbonate were comparable,
- (v) The oxidizing potential for the sample of sodium nitrate was lower for the sample tested as received compared to the sieved ( $< 500 \mu m$ ) sample, which was itself lower than for the milled sample,

11. From all the data and the observations made (see para. 10 above) it was concluded that when tested as received or after sieving ( $< 500\mu$ m) it is still possible to differentiate between a coated and an uncoated sample of a given substance. But when a coated and an uncoated sample are milled no differentiation between the two is possible. This observation indicates that the milling process significantly alters the oxidizing properties of the coated material. It is therefore important that coated oxidizing materials are not milled prior to testing to properly assess their oxidizing potential. This is taken as validation for the note proposed in paragraph 9.

12. In addition to the RRT activity, works continued to identify a proper way to define the friability aspect of a substance in reference to paragraphs 34.4.1.2.6 and 34.4.3.2.3 of the Manual of Tests and Criteria.

13. The goal is to find a simple criterium to define if a substance is friable or not. To this end a candidate test method has been identified from a review of ten existing test methods known to assess friability or attrition characteristics and used in different fields for solid granular materials.

14. The method selected is based on the MT 193 method "Attrition of Tablets" developed by the Collaborative International Pesticides Analytical Council (CIPAC). Its principle is to circulate/turn around the substance to be tested in a rotating dish with a build-in, bow-shaped baffle for a given time and rotation speed.

15. Preliminary tests have been carried out on substances in granulated form and in tablet form. For the moment the results obtained allow only to continue the investigation with the selected method. Further work is needed before the definition of any test protocol or test criteria at this stage.

### Proposal

16. Taking the outcome of the RRT into account and to better define the approach to properly assessing the oxidizing potential of coated materials, it is proposed to insert the following note at the end of paragraphs 34.4.1.2.6 and 34.4.3.2.3 of the Manual of Tests and Criteria:

"NOTE: In the case of a substance coated to reduce or suppress its oxidizing properties with a significant content (> 10 % by mass) of particles less than 500  $\mu$ m, two sets of tests should be conducted: tests conducted with the substance as presented and tests conducted with particles less than 500  $\mu$ m that were obtained from sieving the substance as presented. The substance should not be ground before sieving or testing. The final classification should be based on the test results with the most stringent classification."