Committee of Experts on the Transport of Dangerous Goods
and on the Globally Harmonized System of Classification
and Labelling of Chemicals 26 November 2010

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

Thirty-eight session
Geneva, 29 November–7 December 2010
Item 11 of the provisional agenda

Issues related to the Globally Harmonized System of Classification and Labelling of Chemicals (GHS)

Twentieth session
Geneva, 7–9 December 2010
Item 2 (a) of the provisional agenda

Physical hazards

Test results on UN Test N.5

Transmitted by the expert from Germany

Introduction

1. In documents ST/SG/AC.10/C.3/2008/68 and ST/SG/AC.10/C.4/2008/19, the experts from France and Germany have proposed to work on UN test method N.5 of the Manual of Tests and Criteria and have given reasoning for the proposed action.

2. In document UN/SCETDG/35/INF.54 (submitted by France and Germany) the informal intersessional correspondence group on test method N.5 agreed to start an intense phase of collecting and exchanging data on UN test N.5.

3. In the UN test N.5 no special laboratory apparatus/measuring technique to determine the rate of evolution of flammable gas is required and no reference material is prescribed.

4. Therefore different techniques are used in the laboratories to determine the rate of evolution of flammable gas, e.g. gravimetry or volumetry.

5. As demonstrated in the past by a Round Robin test, the gas evolution rate measured with different systems may vary in a wide range.

6. The BAM Federal Institute for Material Research and Testing has tested two substances (magnesium and aluminium) according to the UN test N.5 and would like to present the test results as well as some results on the calibration of the used test apparatus.

Experimental set-up of the apparatus

7. At BAM a gravimetric set-up is used to determine the rate of evolution of flammable gas according to the UN Manual of Tests and Criteria, test N.5.

8. This test is performed three times at ambient temperature (20 °C) and atmospheric pressure. An additional “blank test” is used to identify and account for influencing atmospheric parameters like air pressure and temperature.
Calibration of the test apparatus

9. The reaction of 0.05 g magnesium with a solution of 10 % hydrochloric acid has been used for the calibration of the apparatus. The measured total volume of hydrogen gas was compared to the theoretical value. The error was in the range of 4 %.

Results of the N.5 test

10. The aim of the tests was to identify the smallest measurable volume of evolved gas with an acceptable error. Therefore two test series were performed. The first one with magnesium by varying the mass of magnesium from 0.02 g to 10 g. The second varying the mass of aluminium from 0.2 g to 10 g. Contrary to the calibration for these tests distilled water was used.

11. The graph shows the evolved gas volume after 2 hours in relation to the initial mass for aluminium and magnesium.

12. 1g magnesium leads to a gas production of 6 ml during the first 2 h with an error of about 17 %. The gas production of ≤ 0.5 g magnesium is constant, means independent on mass. But the error is almost 50 %. We conclude that 1 g magnesium which is corresponding to an absolute gas amount of 6 ml seems to be the borderline for an acceptable accuracy for this experimental set-up. The gas production rate doesn’t increase linearly with mass. One reason could be that magnesium obviously doesn’t react completely.

13. The results for aluminum differ. Regardless of the initial mass the measured gas volume was about 4 ml. Most probably the oxide layer of the aluminum inhibits the reaction of aluminum with water.
Further action

14. It is advised to proceed the work on the UN test N.5.

15. Other laboratories are asked to perform and communicate the calibration of their test apparatus, preferably also with the quantitative reaction of magnesium with hydrochloric acid.

16. Furthermore, these laboratories are asked to test the used set-up for the accurate determination of small gas volumes, e.g. performing test series where the initial mass of the substance is varied.

17. Additional information about the test set-up used at BAM can be requested from: Dr. Heike Michael-Schulz, email: heike.michael-schulz@bam.de or Dr. Marcus Malow, email: marcus.malow@bam.de.