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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

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Explosives and related matters: Review of tests in parts I, II and III of the Manual of Tests and Criteria **Sub-Committee of Experts on the Globally Harmonized System of Classification and Labelling of Chemicals**

Forty-fifth session

Geneva, 6-8 December 2023
Item 2 (a) of the provisional agenda
Work on the Globally Harmonized System
of Classification and Labelling of Chemicals:
Work of the Sub-Committee of Experts on the
Transport of Dangerous Goods on matters of interest to
the Sub-Committee of Experts on the Globally
Harmonized System of Classification and Labelling of
Chemicals

Comments related to the Koenen test and INF.16 (TDG, 63rd session) - INF.6 (GHS 45th session) from Germany

Transmitted by the Sporting Arms & Ammunition Manufacturers' Institute (SAAMI)

I. Background

- 1. The steel alloys originally specified for production of the steel tubes for the Koenen test used for the classification of explosives, self reactives and organic peroxides are no longer available on the market. The *Manual of Tests and Criteria* (MTC) specifications (i.e., dimensions, mass, etc.) and the associated tolerances were based on tubes made from the original alloys. The steel specification and tube design can significantly influence the outcome of classifications for explosives, self reactives and organic peroxides.
- 2. The United Kingdom (UK) and the United States of America (USA) brought concerns regarding the tube specification to the TDG Sub-Committee at the sixty session in 2022, and the Explosives Working Group was supportive of their initiative to review the critical parameters of the tubes. Subsequently, thirteen laboratories comprised of competent authorities and NGOs, including Germany, volunteered to perform Koenen comparative testing ("round-robin" testing) which would be used to refine the Koenen tube specifications including burst pressure and other properties. This round robin testing group under the leadership of the UK and USA is functioning in a manner consistent with an informal correspondence group (ICG). We value Germany as an important contributor to this initiative based on their expertise and experience.
- 3. The following INF papers document the collaborative efforts of this ICG:
 - (a) Informal document INF.15 (TDG, 60th session) from the UK and USA entitled "Parameters for specification of Koenen apparatus" provided the technical basis for the Koenen tube specifications discussion by the Explosives Working Group (EWG) during the 60th session of the TDG Sub-Committee.
 - (b) Informal document INF.44 (60th session) from the Chair of the Explosives Working Group (EWG) "Report of the Working Group on Explosives" agenda item 2(c) 7 documents the discussion and concluded that "The Explosives Working Group was supportive on this work proceeding with round robin tests in which several experts expressed interest in participating."

- (c) Informal document INF.36 (TDG, 62nd session) from the UK and USA entitled "Explosive Working Group Koenen tube round-robin initial test results" conveys the initial results of the round-robin testing being performed by thirteen international laboratories.
- 4. Participating laboratories in the Round-Robin ICG include:

ATF - USA	HSE - UK
BAM - Germany	NOURYON - Netherlands
BAYER - Germany	ORICA – USA/Australia
CERL - Canada	Sandia National Laboratory - USA
EMRTC - USA	Safety Management Services, Inc USA
INERIS - France	TNO - Netherlands
IPO - Poland	

- 5. The objective of this round-robin testing is "to evaluate the potential effect on the limiting orifice diameters for UN listed standard substances when using Koenen tubes with different hydrostatic burst pressures."
- 6. Based on availability for all participating laboratories, the UN standard substances selected by consensus for testing included two (2) explosive substances and one (1) peroxide.
- 7. Informal document INF.16 (TDG, 63rd session) INF.6 (45th session) transmitted by the expert from Germany specifically references INF.15 (TDG, 60th session) which served as a discussion starter for the greater work found in INF.36 (62nd session) entitled "Explosive Working Group Koenen tube round-robin initial test results." The German informal paper focuses on the testing specifications associated with determining the "bursting pressure as determined by quasi-static load through an incompressible fluid," referenced in the MTC paragraphs 11.5.1.2, 12.5.1.2, 18.6.1.2, and 25.4.1.2. This bursting pressure is commonly referred to as the "quasi-static bursting pressure."

II. Discussion

- 8. The UK and USA are currently leading an EWG initiative to evaluate the potential effect on the limiting orifice diameters for UN listed standard substances when using Koenen tubes with different hydrostatic burst pressures (quasi-static bursting pressures) based on round-robin testing by participating laboratories.
- 9. An integral part of this EWG initiative is to perform quasi-static bursting pressure testing of Koenen tubes produced by different manufacturers during different time periods, which requires a uniform test method to ensure consistency. Since the MTC does not specify a burst pressure method, the testing participants agreed to utilize Fauske and Associates, a third-party independent test laboratory with international recognition, to perform the quasi-static bursting pressure tests on the specified Koenen steel tubes.
- 10. Germany provided a copy of INF.4 (TDG, 27th session) entitled "Explosives, Self-Reactive Substances and Organic Peroxides" which resulted in the current wording found in paragraphs 11.5.1.2, 12.5.1.2, 18.6.1.2, and 25.4.1.2 of the MTC. However, the paper included "quasi-static" bursting pressure graphs that pressurized the Koenen tubes to 27-28 MPa within approximately one (1) second. By definition, "Quasi-static means that a load or deformation cycle is applied to a test specimen in a slow, controlled, and predetermined manner. The loading is applied so slowly that the structure deforms in a static manner." In other words, a quasi-static pressure rise should allow time for the tube to reach equilibrium (periodic static conditions) as the pressure is increased in reasonable increments.

This ensures that the inertial effects of the fluid are negligible, producing a static load on the tube¹. The method used by Germany appears to be inconsistent with this approach.

- 11. The German graph in INF.4 (TDG, 27th session) shows a constant pressure ramp rate until failure of the tube with no time allowed for the tube to reach any kind of equilibrium. This rapid and continuous pressure rise, and the associated pressurization momentum will result in higher (less conservative) Koenen tube bursting pressure measurements than tubes tested with a true quasi-static pressurization procedure.
- 12. For this reason, Germany was requested to provide a copy of their quasi-static bursting pressure test procedure and configuration for consideration in the development of the quasi-static bursting pressure testing procedure to be conducted as part of the round-robin testing. As recorded in the EWG report associated with the TDG 62nd session (INF.47, paragraph 7) "Germany stated that they are working to provide their test procedure for determining the quasi-static burst pressure to the round-robin lead." The requested procedure has not yet been provided.
- 13. The Koenen tube burst pressure test procedure included in INF.36 (TDG, 62nd session) on behalf of the round robin group more closely reflects quasi-static pressure increases by starting at 15 MPa and then increasing the pressure in "steps of approximately one-tenth or less of the maximum expected burst pressure (2 MPa). The step should be held for ten seconds before increasing pressure again." Since three (3) Koenen tubes from each of the various manufacturers and production years were collected for quasi-burst pressure testing, it was decided to test one (1) Koenen tube from each production year using this procedure. The group is waiting to test the remaining tubes with hopes that Germany can provide their procedure which was requested at the last session. This will facilitate the finalization of the quasi-static test procedure in a February 2024 intersessional ICG review, in preparation for agreement at the 64th TDG session.
- 14. The following points should be noted:
 - (a) Even with the amended specifications in the MTC intended to accommodate the new tubes, there are no Koenen tubes that have been produced since 2000 that meet all specifications outlined in the MTC (see INF.36 (TDG, 62nd session). For example, Koenen tubes produced by Reichel in Germany and OZM in the Czech Republic all have square tube bottoms versus the curvature specifications outlined in the MTC. This square transition of the tube bottom serves as a stress riser and can lead to premature failure of the tube compared to a tube having the specified tube curvature. All other things being equal, a square tube may therefore fail the test criteria and cause a substance to be classed as an explosive that might be excluded in a curved tube. Similar impacts could be seen for the classification severity of self reactives and organic peroxides. Such discrepancies are systematically being identified by the ICG to aid in the EWG's efforts to re-evaluate the Koenen tube specifications/tolerances and the associated testing methods to accommodate the changing availability of steel alloys.
 - (b) In informal document INF.16, the "Exploratory examination as regards the equivalency of the different bursting pressure test methods" does not fully reflect the rigour of the Fauske and Associates test method. The test method "simulated at BAM with the help of a manual hand pump in a way that the bursting pressure was achieved by means of manually effected strokes via several pressure stages" does not represent the test method used by Fauske and Associates since no manual "hand pump" was used. Hand pumps with manual stokes introduce several testing variables that will be adversely reflected in the test results.
 - (c) Apparatus is readily available to produce the pressure-time curves provided in INF.4 (TDG, 27th session) if needed.

¹ See *Textbook of Engineering Thermodynamics, 3rd edition* by R.K. Rajput, and ASME BPVC.VIII.1-2019.

15. The intent of the MTC is to provide sufficient specificity that any member country with a reasonable technical competency can utilize the test methods and criteria to reach proper classifications. It is reasonable to expect that countries will continue to want to perform their own tests such as the "quasi-static bursting pressure" test.

III. Conclusions

- 16. Although required by the MTC in paragraphs 11.5.1.2, 12.5.1.2, 18.6.1.2, and 25.4.1.2, the MTC gives no guidance or method for determining the Koenen tube "bursting pressure as determined by quasi-static load through an incompressible fluid." The ICG has created a procedure to address this that is under review.
- 17. The Koenen tube fabrication and quality assurance specifications in the MTC are provided to enable countries to produce their own Koenen tubes without dependence on the originator of the test method(s) and criteria.
- 18. We believe the current EWG Koenen Round-Robin testing initiative led by the UK and USA and supported by 13 participating organizations is the optimal way forward. This ongoing initiative includes:
 - (a) Addressing Koenen tube specifications considering the likelihood that available steel alloys will continue to change with time.
 - (b) Developing a standard Koenen tube quasi-static bursting pressure test procedure that can be referenced by the MTC.
 - (c) Coordinating with IGUS EPP and EOS participants and other stakeholders regarding potential changes to avoid unintended consequences.

IV. Proposal

19. We propose that the UK and USA continue to collaborate with any willing participants to address this important classification initiative.

4