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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-fifth session
Geneva, 25 November-3 December 2024
Item 2 (b) (iii) of the provisional agenda
Recommendations made by the Sub-Committee at its sixty-second, sixty-third and sixty-fourth sessions and pending issues: Explosives and related matters - Review of tests in parts I, II and III of the Manual of Tests and Criteria

Proposed changes to the Koenen test specifications

Transmitted by the experts from the United Kingdom of Great Britain and Northern Ireland, and the United States of America*

I. Background

1. The Koenen test is used in the classification of explosives, self-reactives, and organic peroxides. The *Manual of Tests and Criteria* (MTC) specifications (i.e., dimensions, mass, etc.) and the associated tolerances for the steel tubes used in the Koenen test were prescribed to achieve reproducible results on tubes made from specific steel alloys. While these steel specifications and tube design parameters can significantly influence the outcome of classifications, the original steel alloys are no longer available on the market.

2. The United Kingdom (UK) and the United States of America (USA) brought concerns regarding the tube specification to the Sub-Committee at its sixtieth 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 non-governmental organizations (NGOs) volunteered to perform Koenen comparative testing ("round-robin" testing) which would be used to refine the Koenen tube specifications including burst pressure and other dimensions. 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).

3. The following informal documents and report from the Sub-Committee document the collaborative efforts of this ICG:

(a) Informal document UN/SCETDG/60/INF.15 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 sixtieth session of the TDG Sub-Committee;

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

(b) Informal document UN/SCETDG/60/INF.44 “Report of the Working Group on Explosives” agenda item 2 (c), item 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 UN/SCETDG/62/INF.36 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;

(d) Informal document UN/SCETDG/63/INF.16-UN/SCEGHS/45/INF.6 from Germany proposed to have the Germany Federal Institute for Materials Research and Testing (BAM) lead the development of new equipment for performing the quasi-static bursting pressure test since their current test equipment is no longer available on the market;

(e) Informal document UN/SCETDG/63/INF.32-UN/SCEGHS/45/INF.14 from the Sporting Arms and Ammunition Manufacturers’ Institute (SAAMI) addressed the comments from Germany and that the current EWG Koenen Round-Robin testing, led by the UK and USA, includes standardization of the bursting pressure test;

(f) Paragraph 13 of the report of the sixty-third session (document ST/SG/AC.10/C.3/126): “The Sub-Committee welcomed the information on the bursting pressure test method for Koenen steel tubes presented in informal documents INF.16 and INF.32 and agreed on the need to review the provisions in the *Manual of Tests and Criteria*. The Sub-Committee acknowledged the complexity of this subject. All interested stakeholders were invited to contact the representative of SAAMI who will coordinate an inter-sessional discussion on the outcome of the round-Robin tests and come up with a proposal to be submitted to the Explosives Working Group (EWG) for a more detailed consideration at its next meeting. It was noted that the informal documents were also listed on the agenda of the GHS Sub-Committee. This decision should be brought to the attention of the GHS Sub-Committee;”

(g) Informal document UN/SCETDG/64/INF.20 from the UK and USA entitled “UN Explosive Working Group (EWG) Koenen Tube Round-Robin Updated Test Results and ICG Discussion Points” provided additional test results and proposals for modifying the Koenen test specifications based on ICG consensus; and

(h) Informal document UN/SCETDG/64/INF.67 “Report of the Working Group on Explosives” agenda item 2(c), item 7 documents the discussion on informal document INF.20 and concluded “There was unanimous support for the proposals.”

II. Discussion

4. The above referenced documents detail the substantial work of the EWG Koenen Test ICG including, testing objectives and protocols, test procedures, test results, ICG and EWG discussion points, and the basis for the proposals in this document.

5. Since the Koenen test is used in the classification of explosives, self reactives, and organic peroxides, the Koenen Round-Robin ICG is comprised of experts with experience in all of these areas. The test results and the proposals below were presented and discussed at the Joint IGUS EOS[1] and EPP/CIE[2] meetings held in Manchester England in April 2024. Consensus on these proposals was unanimous.

6. As stated above, the EWG is unanimous in their support of the proposals listed below.

7. In preparation for the authors providing a working paper for this session, the EWG asked that three items be considered. To supplement this working paper it is envisioned that an informal paper providing a complete underline/strikethrough version of the impacted

[1] Energetic and Oxidising Substances (EOS) working group of the International Group of Experts on the Explosion Risks of Unstable Substances (IGUS).

[2] IGUS’ Explosives, Propellants and Pyrotechnics (EPP) working group, International Conference of Chief Inspectors of Explosives (CIE).

MTC sections will be submitted prior to the meeting. The following lists the items and the associated resolution:

(a) Item 1: Determine which two of the three tube dimensions (inner diameter, outer diameter, and wall thickness) are relevant?

Resolution: The Koenen Round-Robin ICG reached consensus to specify the "inner diameter" (since it relates to tube volume) and "wall thickness" since they both are considered key parameters for the tubes.

(b) Item 2: Consider changing the term "incompressible fluid" to "liquid.";

Resolution: The Koenen Round-Robin ICG agreed to this change; and

(c) Item 3: Work with the secretariat on the best method for referencing the "Koenen Tube Dynamic Burst Pressure Test Procedure" and the "EWG Koenen Round-Robin Testing Procedure" in the MTC. The two options are to (1) include these procedures in a new appendix in the MTC or (2) modify Appendix 4 to include references to Germany, UK, and USA as national contacts for these procedures.

Resolution: The secretariat recommended including these procedures in a new appendix in the MTC. The secretariat also specified that the electronic files associated with these procedures must be submitted in an editable format.

III. Sustainable Development Goals (SDGs)

8. The work of the ICG is focused on contributing to sustainable delivery Goal 16, *Promote peaceful and inclusive societies for sustainable development, provide access to justice for all and build effective, accountable and inclusive institutions at all levels.*

9. In particular by providing greater clarity on the consumables and procedures necessary to effectively and reliably undertake Koenen tests it will:

(a) Develop effective, accountable and transparent institutions at all levels (SDG 16.6); and

(b) Broaden and strengthen the participation of developing countries in the institutions of global governance (SDG 16.8).

IV. Proposed amendments to the Manual of Tests and Criteria

10. In paragraphs 11.5.1.2.1, 12.5.1.2.1, 18.6.1.2.1 and 25.4.1.2.1:

- Insert a footnote reference at the end of the following sentence as shown below:

  "The tube is deep drawn from steel sheet conforming to specification DC04 (EN 10027-1), or equivalent A620 (AISI/SAE/ASTM), or equivalent SPCEN (JIS g 3141)[1]."

- Insert the following related footnote text and renumber subsequent footnotes accordingly:

  " [1] *Tubes manufactured from sheet steel not meeting these specifications may be used provided conditions a-d are met and the tubes are qualified as having the required limiting diameters listed in section A.12.3 of appendix A.12.".*

11. Amend sub-paragraphs 11.5.1.2.1 (d), 12.5.1.2.1 (d), 18.6.1.2.1 (d), and 25.4.1.2.1 (d) as follows (new text is underlined, deleted text is ~~crossed out~~):

"The bursting pressure as determined by ~~quasi-static~~ dynamic load through a~~n incompressible fluid~~ a liquid shall be 29 ± 4 MPa. The dynamic bursting pressurization rate is defined as a continuous and rapid pressure rate (i.e., 5-35 MPa in less than 0.5 seconds). The "Dynamic Burst Pressure Test Procedure" is located in Section A.12.2 of Appendix A.12.

12. Figures 11.5.1.1, 12.5.1.1, 18.6.1.1 and 25.4.1.1:

- Incorporate the following table of tube tolerances into figures 11.5.1.1, 12.5.1.1, 18.6.1.1 and 25.4.1.1 and modify the tube drawing in these figures to reflect the tolerances in this table.

Specification	Dimension ± tolerance
Outer Length	75 ± 0.5 mm
Inner Diameter	24 ± 0.3 mm
Wall Thickness	0.65 ± 0.1 mm
Bottom Thickness	0.6 ± 0.05 mm
Flange Diameter	32 ± 0.35 mm
Bottom Radius Height	4 ± 2 mm
Mass	27.5 ± 3 g

13. Amend sub-paragraphs 11.5.1.2.1 (a) and (c), 12.5.1.2.1 (a) and (c), 18.6.1.2.1 (a) and (c) and 25.4.1.2.1 (a) and (c) as follows (new text is shown is underlined, deleted text is ~~crossed-out~~):

11.5.1.2.1 (a) The mass of the tubes shall be ~~26.5 ± 1.5 g~~ 27.5 + 3 g, tubes to be used in one test sequence shall not differ in mass by more than 1 g.

11.5.1.2.1 (c) The wall thickness of the tubes measured 20 mm from the bottom of the tube shall be ~~0.5 ± 0.05 mm~~ 0.65 + 0.1 mm; and

12.5.1.2.1 (a) The mass of the tubes shall be ~~26.5 ± 1.5 g~~ 27.5 + 3 g, tubes to be used in one test sequence shall not differ in mass by more than 1 g.

12.5.1.2.1 (c) The wall thickness of the tubes measured 20 mm from the bottom of the tube shall be ~~0.5 ± 0.05 mm~~ 0.65 + 0.1 mm; and

18.6.1.2.1 (a) The mass of the tubes shall be ~~26.5 ± 1.5 g~~ 27.5 + 3 g, tubes to be used in one test sequence shall not differ in mass by more than 1 g.

18.6.1.2.1 (c) The wall thickness of the tubes measured 20 mm from the bottom of the tube shall be ~~0.5 ± 0.05 mm~~ 0.65 + 0.1 mm; and

25.4.1.2.1 (a) The mass of the tubes shall be ~~26.5 ± 1.5 g~~ 27.5 + 3 g, tubes to be used in one test sequence shall not differ in mass by more than 1 g.

25.4.1.2.1 (c) The wall thickness of the tubes measured 20 mm from the bottom of the tube shall be ~~0.5 ± 0.05 mm~~ 0.65 + 0.1 mm; and

14. Add the following new Appendix 12 (“Koenen tube qualification test procedures”) to the *Manual of Tests and Criteria*, containing both the dynamic burst pressure test procedure and a table of required limiting diameters for use of alternative tube alloys.

Annex

“APPENDIX 12

KOENEN TUBE QUALIFICATION TEST PROCEDURES

A12.1 Introduction

The purpose of this appendix is to provide sufficient procedural details to (1) obtain accurate dynamic burst pressure test results for quality control of the steel tubes specified by the Koenen test procedures (section A12.2), and (2) qualify new tubes produced from alloys other than those specified in Koenen procedures by confirming they will give identical limiting diameter results for specified substances (section A.12.3).

A12.2 Dynamic burst pressure test procedure

A12.2.1 *Introduction*

A12.2.1.1 This procedure details the test method to determine whether Koenen Tubes manufactured from a variety of manufacturers and manufacturing lots meet the dynamic burst pressure specifications in sections 11.5.1.2.1(d), 12.5.1.2.1(d), 18.6.1.2.1(d) and 25.4.1.2.1(d) of this *Manual.* The bursting pressure as determined by dynamic load through a liquid shall be 29 ± 4 MPa.

A12.2.1.2 Specific details of the Koenen apparatus are detailed in Tests 1(b), 2(b), 8(c), and Test Method E.1 of this *Manual.*

A12.2.2 *Apparatus and materials*

The following items are required:

(a) Burst pressure apparatus (figure A12.2.1) designed to completely purge Koenen tubes of all air and apply pressures of 35 MPa or greater using a non-corrosive liquid. The apparatus is equipped to accept a pressure transducer;

(b) A static pressure transducer. Any pressure-measuring device may be used provided it is calibrated with a measuring range above the allowable Koenen tube burst pressures detailed in 11.5.1.2.1 (d), 12.5.1.2.1 (d), 18.6.1.2.1 (d), or 25.4.1.2.1 (d) of this Manual and has a response time capable of detecting pressure changes at which they will occur during testing;

(c) A data acquisition (DAQ) system. Used to collect static pressure data with acquisition rate of suitable speed and resolution to accurately assess tube burst pressure. A minimum sampling rate of 10 kHz should be used to ensure peak pressure is captured with accuracy;

(d) A Koenen tube collar meeting the specifications detailed in Figures 11.5.1.1, 12.5.1.1, 18.6.1.1 of this Manual; and

(e) A modified orifice for hydraulic testing. The modified orifice allows connection of the Koenen tube to the burst pressure apparatus.

A12.2.3 *Procedure*

A12.2.3.1 The burst pressure testing apparatus is set up to purge air from the tube and introduce the test liquid. The pressure transducer and data acquisition system are attached (see figure A12.1) and tested to verify proper functioning and capability to accurately measure and record pressures above the maximum burst pressures specified in 11.5.1.2.1 (d), 12.5.1.2.1 (d), 18.6.1.2.1 (d) or 25.4.1.2.1 (d) of this *Manual*.

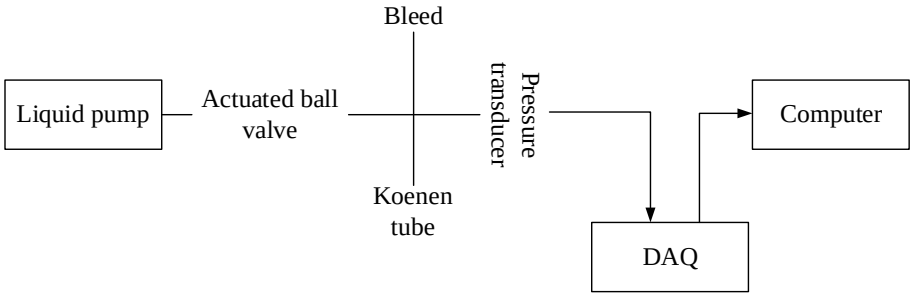
A12.2.3.2 The Koenen tube is labelled and inserted into the threaded collar with the modified orifice plate on top of the tube, then the collar nut is tightened to provide an effective seal.

A12.2.3.3 The modified orifice plate is connected to the burst pressure apparatus and placed in a protective area, then connections verified against leaks. The entire system is purged of air, then the vacuum valve is closed and examined for constant pressure. The actuated ball-valve is closed to prevent premature pressurization of the Koenen tube, after which the supply line to the closed valve is charged with the pump to approximately 35 MPa. The ball-valve is opened remotely and gauge pressure at which tube bursts is recorded.

A12.2.4 ***Test criteria and method of assessing results***

A12.2.4.1 The test results are interpreted in terms of whether the peak pressure recorded by the transducer before rupture of the Koenen tube falls within the required pressure range. The result is considered negative ("-") if the peak pressure is 29 ± 4 MPa and the lot from which the tested tubes were selected is considered to be qualified as meeting the Koenen Test burst pressure specifications.

Figure A12.2.1: Burst pressure apparatus setup



A12.3 Required limiting diameters for use of alternative tube alloys

A12.3.1 ***Introduction***

Tubes manufactured from sheet steel alloys other than those listed in 11.5.1.2.1, 12.5.1.2.1, 18.6.1.2.1 and 25.4.1.21 may be used provided that the each of the limiting diameters listed in A12.3.2 are met.

A12.3.2 Table of required limiting diameters

Substances	**Limiting diameter**
Guanidine Nitrate	1.5 mm
Ammonium Nitrate Powder	1 mm
Tert-Butyl peroxybenzoate	3.5 mm
Diluted tert-Butyl peroxybenzoate with 50 wt% Isododecane	1 mm[3]

Note: *The detailed protocol used to determine the limiting diameters given in the table above is available from the national contacts for test details in Germany, United Kingdom or United States of America (see appendix 4).*

[3] For a type "A" effect.