## 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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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 MTC

# **Proposed changes to the Koenen test specifications – Response to informal document INF.40**

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

# I. Background

1. This document provides responses to the comments provided in informal document INF.40 and proposed language to address concerns raised. The baseline text for this document is informal document INF.34.

- 2. There are three main points raised in informal document INF.40:
  - (a) Koenen Tube Wall Thickness Measurements

Notably that the measured wall thickness of tubes examined by Germany were lower compared to those stated in the report of the round robin test program.

(b) Purging air from the dynamic burst pressure test apparatus

Testing performed by Germany with air purged and with air remaining in the system indicate no marked difference in performance.

(c) Dynamic Burst Pressure Test rate of rise

Germany agreed with the need to mandate a minimum pressure rise rate in the burst pressure test procedure, but expressed concern that the proposed minimum 60 MPa/s rate as measured between 5-35 MPa appears low, especially compared to the rates they have observed in their burst pressure tests ranging between 110-120 MPa/s.

3. Recognizing variances in existing tube wall thicknesses it is proposed to maintain the existing (8<sup>th</sup> Revised Edition of the Manual of Tests and Criteria) wall thickness dimensions of  $0.5 \pm 0.05$  mm. It is proposed to make purging of air from the dynamic burst pressure test apparatus optional. It is additionally proposed to make amendments to the text referencing the dynamic burst pressure test rate of rise to be calculated from 5 to 25 MPa, and raising the minimum rise rate to a more conservative 100 MPa/s between these points.

4. Absent the adoption of these amendments, the Manual of Tests and Criteria will lack test configuration and procedural steps to accurately and repeatably perform the Koenen tube burst pressure testing. This results in most, if not all, tubes on the market today not being in compliance with the existing requirements. The proposals below address the concerns raised in informal document INF.40 and a decision can be taken to adopt the amendments at this time. For further work, it is recommended that the informal correspondence group continue to examine the points raised in informal document INF.40 and present additional proposals and data to the EWG to further refine the test method if deemed appropriate.

# II. Sustainable Development Goals (SDGs)

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

6. 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 target 16.6); and

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

### III. Proposals

7. Changes in this document from the consolidated text in informal document INF.34 are shown as: new text underlined and bold and deleted text is shown in double strikethrough. Amend the *Manual of Tests and Criteria* to read as follows:

(a) In 11.5.1.2.1:

11.5.1.2 Apparatus and materials

11.5.1.2.1 The apparatus consists of a non-reusable steel tube, with its re-usable closing device, installed in a heating and protective device. The tube is deep drawn from sheet steel conforming to specification DC04 (EN 10027-1), or equivalent A620 (AISI/SAE/ASTM), or equivalent SPCEN (JIS g 3141).<sup>1</sup> The dimensions are given in figure 11.5.1.1. The open end of the tube is flanged. The closing plate with an orifice, through which the gases from the decomposition of the test substance escape, is made from heat-resisting chrome steel and is available with the following diameter holes: 1.0 - 1.5 - 2.0 - 2.5 - 3.0 - 5.0 - 8.0 - 12.0 - 20.0 mm. The dimensions of the threaded collar and the nut (closing device) are given in figure 11.5.1.1.

For quality control of the steel tubes, 1 % of the tubes from each production lot shall be subjected to quality control and the following data shall be verified:

(a) The mass of the tubes shall be  $26.5 \pm 1.5 \text{ g} 27.5 \pm 3 \text{ g}$ , tubes to be used in one test sequence shall not differ in mass by more than 1 g;

(b) The length of the tubes shall be  $75 \pm 0.5$  mm;

(c) The wall thickness of the tubes measured 20 mm from the bottom of the tube shall be  $0.5 \pm 0.05$  mm  $\frac{0.65 \pm 0.1 \text{ mm}}{0.65 \pm 0.1 \text{ mm}}$ ; and

(d) The bursting pressure as determined by quasi-static dynamic load through an incompressible fluid a liquid shall be  $29 \pm 4$  MPa. (i.e., the pressure device should be able to measure a pressure rise from 5-35 MPa). The pressure rise rate should be not less than 100 MPa/s, e.g. calculated from the pressure rise from 5 to 25 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.

<u>Footnote <sup>1</sup></u> <u>Tubes manufactured from sheet steel not meeting these specifications may be</u> used provided conditions a-d are met and the tubes are qualified as having the required <u>limiting diameters listed in Section A.12.3 of Appendix A.12.</u>

### (b) In 12.5.1.2<del>.1</del>

### 12.5.1.2 *Apparatus and materials*

12.5.1.2.1 The apparatus consists of a non-reusable steel tube, with its re-usable closing device, installed in a heating and protective device. The tube is deep drawn from sheet steel conforming to specification DC04 (EN 10027-1), or equivalent A620 (AISI/SAE/ASTM), or equivalent SPCEN (JIS g 3141).<sup>1</sup> The dimensions are given in figure 12.5.1.1. The open end of the tube is flanged. The closing plate with an orifice, through which the gases from the decomposition of the test substance escape, is made from heat-resisting chrome steel and is available with the following diameter holes: 1.0 - 1.5 - 2.0 - 2.5 - 3.0 - 5.0 - 8.0 - 12.0 - 20.0 mm. The dimensions of the threaded collar and the nut (closing device) are given in figure 12.5.1.1.

For quality control of the steel tubes, 1 % of the tubes from each production lot shall be subjected to quality control and the following data shall be verified:

(a) The mass of the tubes shall be  $\frac{26.5 \pm 1.5 \text{ g}}{27.5 \pm 3 \text{ g}}$ , tubes to be used in one test sequence shall not differ in mass by more than 1 g;

(b) The length of the tubes shall be  $75 \pm 0.5$  mm;

(c) The wall thickness of the tubes measured 20 mm from the bottom of the tube shall be  $0.5 \pm 0.05$  mm  $\frac{0.65 \pm 0.1 \text{ mm}}{0.65 \pm 0.1 \text{ mm}}$ ; and

(d) The bursting pressure as determined by quasi-static dynamic load through an incompressible fluid a liquid shall be  $29 \pm 4$  MPa. (i.e., the pressure device should be able to measure a pressure rise from 5-35 MPa). The pressure rise rate should be not less than 100 MPa/s, e.g. calculated from the pressure rise from 5 to 25 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.

<u>Footnote 1</u> <u>Tubes manufactured from sheet steel not meeting these specifications may be</u> 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.

(c) In 18.6.1.2.1

18.6.1.2.1 Apparatus and materials

18.6.1.2.1 The apparatus consists of a non-reusable steel tube, with its re-usable closing device, installed in a heating and protective device. The tube is deep drawn from sheet steel conforming to specification DC04 (EN 10027-1), or equivalent A620 (AISI/SAE/ASTM), or equivalent SPCEN (JIS G 3141).<sup>1</sup> The dimensions are given in figure 18.6.1.1. The open end of the tube is flanged. The closing plate with an orifice, through which the gases from the decomposition of the test substance escape, is made from heat-resisting chrome steel and is available with numerous sized orifices. For this test the following diameter holes are used:

- 1.5 mm for the closing plate used in the heating calibration procedure; and
- 2.0 mm for the closing plate used in the test

The dimensions of the threaded collar and the nut (closing device) are given in figure 18.6.1.1.

For quality control of the steel tubes, 1 % of the tubes from each production lot shall be subjected to quality control and the following data shall be verified:

(a) The mass of the tubes shall be  $\frac{26.5 \pm 1.5 \text{ g}}{27.5 \pm 3 \text{ g}}$ , tubes to be used in one test sequence shall not differ in mass by more than 1 g;

(b) The length of the tubes shall be  $75 \pm 0.5$  mm;

(c) The wall thickness of the tubes measured 20 mm from the bottom of the tube shall be  $0.5 \pm 0.05$  mm  $\frac{0.65 \pm 0.1 \text{ mm}}{0.65 \pm 0.1 \text{ mm}}$ ; and

(d) The bursting pressure as determined by quasi-static <u>dynamic</u> load through an incompressible fluid a <u>liquid</u> shall be 29 ± 4 MPa. (i.e., the pressure device should be able to measure a pressure rise from 5-35 MPa). The pressure rise rate should be not less than 100 MPa/s, e.g. calculated from the pressure rise from 5 to 25 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.

<u>Footnote 1</u> <u>Tubes manufactured from sheet steel not meeting these specifications may be</u> 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.

(d) In 25.4.1.2.1:

25.4.1.2.1 Apparatus and materials

25.4.1.2.1 The apparatus consists of a non-reusable steel tube, with its re-usable closing device, installed in a heating and protective device. The tube is deep drawn from sheet steel conforming to specification DC04 (EN 10027-1), or equivalent A620 (AISI/SAE/ASTM), or equivalent SPCEN (JIS g 3141).<sup>1</sup> The dimensions are given in figure 25.4.1.1. The open end of the tube is flanged. The closing plate with an orifice, through which the gases from the decomposition of the test substance escape, is made from heat-resisting chrome steel. For classification the following diameter holes shall be used: 1.0 - 1.5 - 2.0 - 2.5 - 3.0 - 5.0 - 8.0 - 12.0 - 20.0 mm. In addition, other diameters can be used for hazard assessment. The dimensions of the threaded collar and the nut (closing device) are given in figure 25.4.1.1.

For quality control of the steel tubes, 1 % of the tubes from each production lot shall be subjected to quality control and the following data shall be verified:

(a) The mass of the tubes shall be  $26.5 \pm 1.5 \text{ g} 27.5 \pm 3 \text{ g}$ , tubes to be used in one test sequence shall not differ in mass by more than 1 g;

(b) The length of the tubes shall be  $75 \pm 0.5$  mm;

(c) The wall thickness of the tubes measured 20 mm from the bottom of the tube shall be  $0.5 \pm 0.05$  mm  $\frac{0.65 \pm 0.1 \text{ mm}}{0.65 \pm 0.1 \text{ mm}}$ ; and

(d) The bursting pressure as determined by quasi-static <u>dynamic</u> load through an incompressible fluid a <u>liquid</u> shall be 29 ± 4 MPa. (i.e., the pressure device should be able to measure a pressure rise from 5-35 MPa). The pressure rise rate should be not less than 100 MPa/s, e.g. calculated from the pressure rise from 5 to 25 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.

<u>Footnote  $^{1}$ </u> <u>Tubes manufactured from sheet steel not meeting these specifications may be</u> used provided conditions a-d are met and the tubes are qualified as having the required <u>limiting diameters listed in Section A.12.3 of Appendix A.12.</u>

(e) Incorporate the following tube tolerance amendments into Figures 11.5.1.1, 12.5.1.1, 18.6.1.1 and 25.4.1.1. The underlined text is explanatory. Maintain the "key" below the figures.



(f) Add a new appendix to the *Manual of Tests and Criteria* titled "Koenen Tube Qualification Test Procedures", containing both the Dynamic Burst Pressure Test Procedure and a Table of required Limiting Diameters for use of alternative tube alloys. This new appendix is contained in annex 1 to this document.

### 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

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  $29 \pm 4$  MPa.

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 <del>completely purge</del> Koenen tubes of all air and apply pressures of 35 MPa or greater using a noncorrosive liquid, and optionally capable of purging Koenen tube and apparatus of all air. 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*.

(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. As a best practice it is recommended at this step that the entire system is purged of air under vacuum, followed by closure of the vacuum valve and examination 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  $29 \pm 4$  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	$1 \text{ mm}^1$

*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).

<sup>&</sup>lt;sup>1</sup> For a type "A" effect.