

# **Recommendations on the TRANSPORT OF DANGEROUS GOODS**

## **Manual of Tests and Criteria**

*Fourth revised edition*



**UNITED NATIONS**  
**New York and Geneva, 2003**

**NOTE**

The designations employed and the presentation of the material in this publication do not imply the expression of any opinion whatsoever on the part of the Secretariat of the United Nations concerning the legal status of any country, territory, city or area, or of its authorities, or concerning the delimitation of its frontiers or boundaries.

ST/SG/AC.10/11/Rev.4
----------------------

Copyright © United Nations, 2003

*All rights reserved.*

*No part of this publication may, for sales purposes, be reproduced, stored in a retrieval system or transmitted in any form or by any means, electronic, electrostatic, magnetic tape, mechanical, photocopying or otherwise, without prior permission in writing from the United Nations.*

UNITED NATIONS PUBLICATION
<i>Sales No. E.03.VIII.2</i>
ISBN 92-1-139087-7

## FOREWORD

The recommendations in this Manual of Tests and Criteria supplement the "Recommendations on the Transport of Dangerous Goods" and the Model Regulations annexed thereto. They may also be used in relation to the "Globally Harmonized System of Classification and Labelling of Chemicals". They result from the decisions of the United Nations Committee of Experts on the Transport of Dangerous Goods at its thirteenth (1984), fourteenth (1986), fifteenth (1988), sixteenth (1990), seventeenth (1992), eighteenth (1994), nineteenth (1996), twentieth (1998) and twenty-first (2000) sessions, and those of the Committee of Experts on the Transport of Dangerous Goods and on the Globally Harmonized System of Classification and Labelling of Chemicals at its first (2002) session.

This fourth revised edition includes revised provisions for the classification of lithium batteries (sub-section 38.3) adopted by the Committee in 2000 (ST/SG/AC.10/27/Add.2) (already published, together with corrections to the third revised edition as ST/SG/AC.10/11/Rev.3/Amend.1); and new provisions for the classification of ammonium nitrate emulsions (section 18), flammable aerosols (section 31) and substances corrosive to metals (section 37) adopted by the Committee in 2002 (ST/SG/AC.10/29/Add.2).



## GENERAL TABLE OF CONTENTS

<u>Section</u>	<b>Page</b>
1. GENERAL INTRODUCTION (Introduction, Layout, Precedence of Hazard Characteristics, Safety, General Conditions for Testing, Recommended Tests and Reporting).....	1
<b>PART I: CLASSIFICATION PROCEDURES, TEST METHODS AND CRITERIA RELATING TO EXPLOSIVES OF CLASS 1</b>	
10. INTRODUCTION TO PART I (Purpose, Scope, Acceptance procedure, Assignment Procedure, Examples of Test Report).....	13
11. TEST SERIES 1 (To determine if a substance has explosive properties).....	31
12. TEST SERIES 2 (To determine if a substance is too insensitive for inclusion in Class 1) .....	51
13. TEST SERIES 3 (To determine if a substance is thermally stable and not too dangerous to transport in the form in which it was tested).....	69
14. TEST SERIES 4 (To determine if an article, packaged article or packaged substance is too dangerous for transport) .....	123
15. TEST SERIES 5 (To determine if a substance may be assigned to Division 1.5).....	129
16. TEST SERIES 6 (To assign a substance or article to Division 1.1, 1.2 1.3 or 1.4 or exclude it from Class 1) .....	143
17. TEST SERIES 7 (To determine if an article may be assigned to Division 1.6).....	155
18. TEST SERIES 8 (To determine if an ammonium nitrate emulsion, suspension or gel, intermediate for blasting explosives (ANE), is insensitive enough for inclusion in Division 5.1, and to evaluate the suitability for transport in tanks).....	177
<b>PART II: CLASSIFICATION PROCEDURES, TEST METHODS AND CRITERIA RELATING TO SELF-REACTIVE SUBSTANCES OF DIVISION 4.1 AND ORGANIC PEROXIDES OF DIVISION 5.2</b>	
20. INTRODUCTION TO PART II (Purpose, Scope, Preliminary Procedure, Classification Procedure, Example of Test Report).....	201
21. TEST SERIES A (To determine if there is propagation of detonation) .....	213
22. TEST SERIES B (To determine if there is detonation in the package).....	229
23. TEST SERIES C (To determine if there is propagation of deflagration).....	233
24. TEST SERIES D (To determine if there is rapid deflagration in the package) .....	245
25. TEST SERIES E (To determine the effect of heating under confinement).....	249
26. TEST SERIES F (To determine the explosive power).....	265
27. TEST SERIES G (To determine if there is thermal explosion in the package).....	289
28. TEST SERIES H (To determine the Self-accelerating Decomposition Temperature) .....	295

**GENERAL TABLE OF CONTENTS (continued)**

<b><u>Section</u></b>	<b>Page</b>
<b>PART III: CLASSIFICATION PROCEDURES, TEST METHODS AND CRITERIA RELATING TO CLASS 3, CLASS 4, DIVISION 5.1 AND CLASS 9</b>	
30. INTRODUCTION TO PART III (Purpose, Scope).....	323
31. CLASSIFICATION PROCEDURES, TEST METHODS AND CRITERIA RELATING TO FLAMMABLE AEROSOLS OF CLASS 2 .....	325
32. CLASSIFICATION PROCEDURES, TEST METHODS AND CRITERIA RELATING TO LIQUID DESENSITIZED EXPLOSIVES AND FLAMMABLE LIQUIDS OF CLASS 3 .....	341
33. CLASSIFICATION PROCEDURES, TEST METHODS AND CRITERIA RELATING TO CLASS 4 .....	351
34. CLASSIFICATION PROCEDURES, TEST METHODS AND CRITERIA RELATING TO OXIDIZING SUBSTANCES OF DIVISION 5.1 .....	369
35. <i>Reserved for classification procedures, test methods and criteria relating to Class 6</i> .....	381
36. <i>Reserved for classification procedures, test methods and criteria relating to Class 7</i> .....	383
37. CLASSIFICATION PROCEDURES, TEST METHODS AND CRITERIA RELATING TO SUBSTANCES OF CLASS 8 .....	385
38. CLASSIFICATION PROCEDURES, TEST METHODS AND CRITERIA RELATING TO CLASS 9 .....	389
<b>APPENDICES</b> .....	403
Appendix 1 SPECIFICATIONS OF STANDARD DETONATORS.....	407
Appendix 2 BRUCETON AND SAMPLE COMPARISON METHODS.....	411
Appendix 3 CAVITATION OF SAMPLES .....	415
Appendix 4 NATIONAL CONTACTS FOR TEST DETAILS .....	419
Appendix 5 EXAMPLE OF A TEST METHOD FOR VENT SIZING .....	421
Appendix 6 SCREENING PROCEDURES.....	427

## SECTION 1

### GENERAL INTRODUCTION

#### 1.1 Introduction

1.1.1 The purpose of this text is to present the United Nations schemes for the classification of certain types of dangerous goods and to give descriptions of the test methods and procedures considered to be the most useful for providing competent authorities with the necessary information to arrive at a proper classification of substances and articles for transport. The Manual of Tests and Criteria should be used in conjunction with the latest version of the Recommendations on the Transport of Dangerous Goods (hereafter referred to as the Recommendations) and of the Model Regulations on the Transport of Dangerous Goods annexed to these Recommendations (hereafter referred to as the Model Regulations).

1.1.2 It should be noted that the Manual of Tests and Criteria is not a concise formulation of testing procedures that will unerringly lead to a proper classification of products. It therefore assumes competence on the part of the testing authority and leaves responsibility for classification with them. The competent authority has discretion to dispense with certain tests, to vary the details of tests, and to require additional tests when this is justified to obtain a reliable and realistic assessment of the hazard of a product. In some cases, a small scale screening procedure may be used to decide whether or not it is necessary to perform larger scale classification tests. Suitable examples of procedures are given in the introductions to some test series and in Appendix 6.

#### 1.2 Layout

1.2.1 The classification procedures, test methods and criteria are divided into three parts:

Part I: those relating to assignment of explosives to Class 1;

Part II: those relating to assignment of self-reactive substances to Division 4.1 and of organic peroxides to Division 5.2;

Part III: those relating to assignment of substances or articles to Class 3, Class 4, Division 5.1 or Class 9.

Part III contains some classification procedures, test methods and criteria which are also given in the Model Regulations. There are also a number of appendices which give information common to a number of different types of tests, on the National Contacts for Test Details, on an example method for emergency relief vent sizing of portable tanks for the transport of organic peroxides and self-reactive substances and on screening procedures.

1.2.2 The methods of test identification are given in Table 1.1.

**Table 1.1: TEST IDENTIFICATION CODES**

Part of manual	Test series	Test type	Test number	Example of test identification code
I	1 - 8	(a), (b), etc.	(i), (ii), etc. <sup>a</sup>	2 (a) (i)
II	A - H	-	1, 2, etc.	A.1
III	L - T	-	1, 2, etc.	L.1

<sup>a</sup> *If only one test is given for a test type, the Roman numerals are not used.*

1.2.3 Each test is given a unique identification code and is edited as follows:

- x.1 *Introduction*
- x.2 *Apparatus and materials*
- x.3 *Procedure* (including observations to be made and data to be collected)
- x.4 *Test criteria and method of assessing results*
- x.5 *Examples of results*

**NOTE:** *Examples of results are not normally given for tests on articles as these are too specific to the article tested and do not allow validation of the test procedure. Results on substances may vary from those given in the "Examples of results" if the physical form, composition, purity etc. of the substance is different. The results given should not be regarded as standard values.*

**Figures** x.1, x.2, x.3 etc. (i.e. diagrams of apparatus etc.)

**NOTE:** *Unless otherwise indicated, the dimensions given on the diagrams are in millimetres.*

### 1.3 Precedence of hazard characteristics

1.3.1 The table in 2.0.3.3 of Chapter 2.0 of the Model Regulations may be used as a guide in determining the class of a substance, mixture or solution having more than one risk, when it is not named in the Dangerous Goods List in Chapter 3.2 of the Model Regulations. For goods having multiple risks, which are not specifically listed by name in Chapter 3.2 of the Model Regulations, the most stringent packing group denoted to the respective hazard of the goods takes precedence over other packing groups, irrespective of the precedence of hazard table in 2.0.3.3 of Chapter 2.0 of the Model Regulations.

1.3.2 The precedence of hazard characteristics of the following are not dealt with in the Precedence of Hazard Table in Chapter 2.0 of the Model Regulations, since these primary characteristics always take precedence:

- Substances and articles of Class 1;
- Gases of Class 2;
- Liquid desensitized explosives of Class 3;
- Self-reactive substances and solid desensitized explosives of Division 4.1;
- Pyrophoric substances of Division 4.2;
- Substances of Division 5.2;
- Substances of Division 6.1 with a Packing Group I inhalation toxicity;
- Substances of Division 6.2; and
- Material of Class 7.

1.3.3 Self-reactive substances, except for type G, giving a positive result in the self-heating test for Division 4.2, should not be classified in Division 4.2 but in Division 4.1 (see paragraph 2.4.2.3.1.1 of the Model Regulations). Organic peroxides of type G having properties of another class or division (e.g. UN 3149) should be classified according to the requirements of that class or division.

### 1.4 Safety

1.4.1 For the safety of laboratory personnel, the producer or other applicant for classification of a new product should provide all available safety data on the product e.g. the toxicity data.

1.4.2 Particularly when explosive properties are suspected, it is essential for the safety of workers that small scale preliminary tests are carried out before attempting to handle larger quantities. This involves tests for determining the sensitiveness of the substance to mechanical stimuli (impact and friction), and to heat and flame.

1.4.3 In tests involving initiation of potentially explosive substances or articles, a safe waiting period, prescribed by the test agency, should be observed after initiation.

1.4.4 Extra care should be taken when handling samples which have been tested since changes may have occurred rendering the substance more sensitive or unstable. Tested samples should be destroyed as soon as possible after the test.

## **1.5 General conditions for testing**

1.5.1 The conditions given in the test prescriptions should be followed as closely as possible. If a parameter is not specified in the test prescription then the conditions given here should be applied. Where tolerances are not specified in the test prescription, it is implied that the accuracy is according to the number of decimal places given in any dimension e.g. 1.1 implies 1.05 to 1.15. In cases where conditions during a test deviate from those prescribed, the reason for the deviation should be stated in the report.

1.5.2 The composition of the test sample should be as close as possible to the concentration of the substance intended for transport. The contents of active substance(s) and diluent(s) should be specified in the test report with at least an accuracy of  $\pm 2\%$  by mass. Components which can have a major effect on a test result, such as moisture, should be specified as accurately as possible in the test report.

1.5.3 All test materials in contact with the test substance should be such that, as far as possible, they do not affect the test results e.g. catalyse decomposition. In cases where such an effect cannot be excluded, special precautions should be taken to prevent the result being affected, e.g. passivation. The precautions taken should be specified in the test report.

1.5.4 The tests should be performed under the conditions (temperature, density etc.) which are representative of the expected circumstances of transport. If the transport conditions are not covered by the test conditions specified, supplementary tests may need to be performed which are specifically designed for the anticipated transport conditions e.g. elevated temperature. Where appropriate, e.g. when the result is particle size dependent, the physical conditions should be specified in the test report.

## **1.6 Recommended tests**

1.6.1 The manual gives descriptions of tests and criteria used to provide the necessary information to arrive at a proper classification. In some cases, there is more than one test for a particular property. As a result of comparative work with some of these tests, it has been possible to identify one test as the recommended test in a set of equivalent tests. The recommended tests for classifying explosive substances and articles (Part I of the manual) are listed in Table 1.2 and for classifying self-reactive substances and organic peroxides (Part II of the manual) in Table 1.3. All test methods given in Part III of the manual are recommended tests as only one test is given for each property. The other tests in a set are considered to be alternative tests and may continue to be used for classification purposes.

1.6.2 As a result of comparative work, some tests have been deleted. However, as some countries maintain databases referenced by the test number, the tests currently given in the test manual have not been renumbered unless existing tests have been assigned to different test types.

1.6.3 The aim is to have only one United Nations test, or combination of tests, for each property. However, until the recommended tests have been used more widely, it is not possible to do this in all cases at present.

1.6.4 If new tests are proposed for inclusion in the manual, the proposer should be able to provide justification that the new test is a significant improvement on the existing recommended test. In such cases, the new test may be included as an alternative test until it has been tried by laboratories of other countries.

**Table 1.2: RECOMMENDED TESTS FOR EXPLOSIVES AND EXPLOSIVE ARTICLES**

<b>Test series</b>	<b>Test type</b>	<b>Test code</b>	<b>Test name</b>
1	(a)	1 (a)	UN gap test
1	(b)	1 (b)	Koenen test
1	(c)	1 (c)(i)	Time / pressure test
2	(a)	2 (a)	UN gap test
2	(b)	2 (b)	Koenen test
2	(c)	2 (c)(i)	Time / pressure test
3	(a)	3 (a)(ii)	BAM Fallhammer
3	(b)	3 (b)(i)	BAM Friction apparatus
3	(c)	3 (c)	Thermal stability test at 75 °C
3	(d)	3 (d)	Small-scale burning test
4	(a)	4 (a)	Thermal stability test for unpackaged articles and packaged articles
4	(b)	4 (b) (i)	Steel tube drop test for liquids
4	(c)	4 (b)(ii)	Twelve metre drop test for unpackaged articles, packaged articles and packaged substances
5	(a)	5 (a)	Cap sensitivity test
5	(b)	5 (b)(ii)	USA DDT test
5	(c)	5 (c)	External fire test for Division 1.5
6	(a)	6 (a)	Single package test
6	(b)	6 (b)	Stack test
6	(c)	6 (c)	External fire (bonfire) test
7	(a)	7 (a)	EIDS cap test
7	(b)	7 (b)	EIDS gap test
7	(c)	7 (c)(ii)	Friability test
7	(d)	7 (d) (i)	EIDS bullet impact test
7	(e)	7 (e)	EIDS external fire test
7	(f)	7 (f)	EIDS slow cook-off test
7	(g)	7 (g)	1.6 article external fire test
7	(h)	7 (h)	1.6 article slow cook-off test
7	(j)	7 (j)	1.6 article bullet impact test
7	(k)	7 (k)	1.6 article stack test
8	(a)	8(a)	Thermal stability test for ANE
8	(b)	8(b)	ANE gap test
8	(c)	8(c)	Koenen test
8	(d)	8(d)	Vented pipe test <sup>a</sup>

<sup>a</sup> *This test is intended for evaluating the suitability for transport in tanks.*

**Table 1.3: RECOMMENDED TESTS FOR SELF-REACTIVE SUBSTANCES AND ORGANIC PEROXIDES**

Test series	Test code	Test name
A	A.6	UN detonation test
B	B.1	Detonation test in package
C	C.1	Time/pressure test
C	C.2	Deflagration test
D	D.1	Deflagration test in the package
E	E.1	Koenen test
E	E.2	Dutch pressure vessel test
F	F.4	Modified Trauzl test
G	G.1	Thermal explosion test in package
H	H.1	United States SADT test (for packages)
H	H.2	Adiabatic storage test (for packages, IBCs and tanks)
H	H.4	Heat accumulation storage test (for packages, IBCs and small tanks)

## 1.7 Reporting

1.7.1 Classifications for Chapter 3.2 of the Model Regulations are made on the basis of consideration of data submitted to the Committee by governments, intergovernmental organisations and other international organisations in the form recommended in Figure 1 of the Recommendations. Supplementary data is required for the classification of:

Substances and articles of Class 1 (see 10.5);  
Self-reactive substances of Division 4.1 (see 20.5); and  
Organic peroxides of Division 5.2 (see 20.5).

1.7.2 Where tests are performed on packaged substances or articles, the test report should contain the quantity of substance or number of articles per package and the type and construction of the packaging.

