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| **Committee of Experts on the Transport of Dangerous Goods and on the Globally Harmonized System of Classificationand Labelling of Chemicals 20 November 2020** |
| **Sub-Committee of Experts on the Transport of Dangerous Goods**  |  |
| **Fifty-seventh session** |  |
| Geneva, 30 November - 8 December 2020Item 2(b) of the provisional agenda**Explosives and related matters** |  |

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| **UN/SCETDG/57/INF.38** |

 Report of the Working Group on Explosives

 Transmitted by the Acting Chairman of the Working Group

 Introduction

1. Due to travel restrictions related to the COVID-19 pandemic, the 57th Session of the Sub-Committee was postponed until 30 November – 8 December 2020; therefore, the working group’s regular meeting was also postponed. For that time period, those travel restrictions remain in place making it difficult to meet in person; however, issues were identified that needed the attention of the working group this biennium in support of the 57th Session of the Sub-Committee as well as, to some extent, the 39th Session of the GHS Sub-Committee. Therefore, the working group met via web conference on 17 and 18 November 2020 to conclude its business prior to and in support of the Sub-Committees. Participation in this meeting of the working group included 67 experts from Belgium, Canada, China, Finland, France, Germany, Japan, Korea (Republic of), Netherlands, Poland, Romania, Russia, Spain, Sweden, United Kingdom, United States of America, UN/ECE, Australian Explosives Industry and Safety Group (AEISG), Council on Safe Transportation of Hazardous Articles (COSTHA), European Association of Automotive Suppliers (CLEPA), European Chemical Industry Council (CEFIC), Hazmat Safety Consulting, Institute of Makers of Explosives (IME), Shanghai Institute of Chemical Industry, Responsible Packaging Management Association of Southern Africa (RPMASA), and Sporting Arms and Ammunition Manufacturers' Institute (SAAMI). Annex 1 of this report provides a list of participants. The group discussed technical matters related to official papers and informal papers as time allowed. Due to the unavailability of Mr. Ed de Jong (Netherlands), who normally serves as chair of the working group, Mr. Duane Pfund (USA) served as acting chair and Mr. David Boston (IME) as secretariat.
2. Throughout this report, the following abbreviations may be used:
* EWG – Working Group on Explosives
* GHS – Globally Harmonized System
* ICG – Informal Correspondence Group
* IGUS/EOS – International Group of Experts on the Unstable and Energetic Substances/ Energetic and Oxidizing Substances
* MR – Model Regulations
* MTC – Manual of Tests and Criteria
* TDG – Transportation of Dangerous Goods
* WR – Workspace Reviews of 15 May – 12 June 2020 and 20 October – 16 November 2020
1. As described in paragraph 4 (below), the following documents identified in agenda items 2(b), 6(a), and 11(a) of the revised provisional agenda for the 57th session[[1]](#footnote-2) were considered for discussion.

| **Document** | **Title** | **Paragraph** |
| --- | --- | --- |
| Agenda Item 2(b)(i) | Review of Test Series 6 |  |
| *ST/SG/AC.10/C.3/2020/4 (SAAMI)* | *Report of the informal correspondence group on the review of Test 6 (d) (Unconfined package test)* | [*5*](#para5) |
| Agenda Item 2(b)(ii) | Improvement of Test Series 8 |  |
| *UN/SCETDG/57/INF.13 (IME)* | *Recommendations on Test Series 8: Applicability of Test Series 8 (d)* | 6 |
| Agenda Item 2(b)(iii) | Review of tests in parts I, II and III of the Manual of Tests and Criteria |  |
| [*ST/SG/AC.10/C.3/2020/3 -ST/SG/AC.10/C.4/2020/4*](file:///H%3A%5CUN%5Cundocs%5CAC10%5CC3%5C2020%5C57th%20Session%5CST-SG-AC10-C3-2020-03e-ST-SG-AC10-C4-2020-04e.docx) *and* [*Add.1*](file:///H%3A%5CUN%5Cundocs%5CAC10%5CC3%5C2020%5C57th%20Session%5CST-SG-AC10-C3-2020-3a1e-ST-SG-AC10-C4-2020-4a1e.docx) *(Chair of the Working Group on Explosives)* | *Manual of Tests and Criteria, review of Test Series H: determination of self-accelerating decomposition temperature* | *7* |
| [*UN/SCETDG/57/INF.4-UN/SCEGHS/39/INF.3*](file:///H%3A%5CUN%5Cundocs%5CAC10%5CC3%5C2020%5C57th%20Session%5CINF%5CUN-SCETDG-57-INF04e-UN-SCEGHS-39-INF03e.docx) *(Chairman of the Working Group on Explosives)* | *Manual of Tests and Criteria, review of Test Series H: determination of self-accelerating decomposition temperature* |  |
| Agenda Item 2(b)(iv) | “UN” standard detonators |  |
| *No document* |  |  |
| Agenda Item 2(b)(v) | Review of packing instructions for explosives |  |
| *ST/SG/AC.10/C.3/2020/51 (United Kingdom)* | *Correction or amendment to packing instruction P137, special packing provision PP70* | *8* |
| *ST/SG/AC.10/C.3/2020/53 (United Kingdom)* | *Clarification on the applicability of PP70 in packing instruction P137* |  |
| Agenda Item 2(b)(iv) | Application of security provisions to explosives N.O.S. |  |
| *No document* |  |  |
| Agenda Item 2(b)(vii) | Test N.1 for readily combustible solids |  |
| *ST/SG/AC.10/C.3/2020/34 (China)* | *Proposals to amend Figure 33.2.4.1 (A) cross-section of the 250 mm long mould in the Manual of Tests and Criteria* | *9* |
| Agenda Item 2(b)(viii) | Review of Chapter 2.1 of the GHS |  |
| *See agenda item 11(a)* |  |  |
| Agenda Item 2(b)(ix) | Energetic samples |  |
| *No document* |  |  |
| Agenda Item 2(b)(x) | Issues related to the definition of explosives |  |
| *No document* |  |  |
| Agenda Item 2(b)(xi) | Review of packaging and transport requirements for ANEs |  |
| *No document* |  |  |
| Agenda Item 2(b)(xii) | Miscellaneous |  |
| *ST/SG/AC.10/C.3/2020/17 (Secretariat)* | *Inconsistency in the French description of the UN gap test in the Manual of Tests and Criteria* | *10* |
| *ST/SG/AC.10/C.3/2020/25 (France, COSTHA)* | *Terms of Reference for the work on “Exclusion from Class 1” within the Explosives Working Group* | *11* |
| *UN/SCETDG/57/INF.6 (COSTHA)* | *New entry for aerosol generating, fire suppression devices* |  |
| Agenda Item 6(a) | Other miscellaneous proposals |  |
| *ST/SG/AC.10/C.3/2020/58 (Romania)* | *Proposal of amendments concerning the use of the terms “risk” and “hazard/danger” in the Recommendations and Model Regulations* | *12* |
| Agenda Item 11(a) | Review of GHS Chapter 2.1 |  |
| *ST/SG/AC.10/C.3/2020/20-ST/SG/AC.10/C.4/2020/5 and Add.1 (ICG via Sweden)* | *A new Chapter 2.1 for the GHS* | *13* |
| *UN/SCETDG/57/INF.17-UN/SCEGHS/39/INF.14 (Sweden)* | *Review of GHS Chapter 2.1* |  |
| *UN/SCETDG/57/INF.23 (Sweden)* | *Decision logics and Precautionary statement matrices for the new GHS Chapter 2.1* |  |
| *ST/SG/AC.10/C.4/2020/18 (Sweden)* | *Decision logics for the new GHS Chapter 2.1* |  |
| *UN/SCEGHS/39/INF.22 (Germany)* | *Amendments to the decision logics for explosives in Chapter 2.1 as proposed in ST/SG/AC.10/C.4/2020/18* |  |
| *UN/SCEGHS/39/INF.25 (Sweden)* | *Comment to UN/SCEGHS/39/INF.22 – A (simple) fall back option for decision logic 2.1 (b) of new GHS Chapter 2.1* |  |
| *UN/SCETDG/57/INF.16-UN/SCEGHS/39/INF.13 (Sweden)* | *Status report on the review of GHS Chapter 2.1 – update on the open issues* |  |
| *ST/SG/AC.10/C.3/2020/22-ST/SG/AC.10/C.4/2020/7 (Sweden)* | *Amendments to the Manual of Tests and Criteria to accommodate the new GHS Chapter 2.1 – in principle* | *14* |
| *ST/SG/AC.10/C.3/2020/60-ST/SG/AC.10/C.4/2020/16 (Sweden and EWG Chair)* | *Amendments to the Manual of Tests and Criteria to accommodate the new GHS Chapter 2.1* |  |
| *UN/SCETDG/57/INF.15-UN/SCEGHS/39/INF.10 (USA, IME, SAAMI)* | *GHS Chapter 2.1: Revised exclusion and exemption criteria for explosive - containing articles not assigned to Class 1* | *15* |
| *UN/SCETDG/57/INF.33-UN/SCEGHS/39/INF.19 (ICG via Sweden)* | *Possible amendment to proposed new GHS Chapter 2.1 for products consisting of or containing explosives, which are not classified as Class 1 for transport* |  |

1. Led by Dr. Lorens van Dam (Sweden, ICG leader), the first day of the meeting (17 November) focused on finalizing the EWG input on proposals to the GHS Chapter 2.1 review. Led by Mr. Pfund, on the second day of the meeting (18 November), the EWG discussed papers submitted to the TDG, prioritizing those proposals that appeared closest to completion during this session based on the WR comments previously received. As time permitted, the EWG also discussed other explosives related working papers submitted to the 57th session.

There are three Annexes to this report:

* Annex 1 – List of Participants
* Annex 2 – Changes for the Model Regulations (21st Revised Edition)
* Annex 3 – Changes for the Test Manual (7th Revised Edition)

Agenda Item 2(b)(i) – Review of Test Series 6

1. **Subject.** Test Series 6(d)

*Documents: ST/SG/AC.10/C.3/2020/4 (SAAMI)*

*Informal documents: None submitted*

**Discussion:** SAAMI confirmed the comments received during the WR had been reviewed and that it will work with the respondents to address their comments. USA noted that the review should first consider the definition of 1.4S and to whom the protection of that classification is directed. USA believes this understanding is crucial to an effective review of the 6(d) acceptance criteria. There was support and no opposition expressed by the EWG to the continuation of this review.

**Conclusion:** The EWG recommends continuation of this review during the 2021/2022 biennium and that SAAMI continue to lead the review.

Agenda Item 2(b)(ii) – Improvement of Test Series 8

1. **Subject.** Test Series 8(d)

*Documents: None submitted*

*Informal documents: UN/SCETDG/57/INF.13 (IME)*

**Discussion:** IME confirmed that comments received during the WR had been reviewed and that it will work with the respondents to address those comments. Further:

* Spain opposed the proposal on the basis that the 8(d) test is necessary to qualify ANEs for containment in tanks.
* USA could not currently support the proposal, noting issues including that the minimum burning pressure (MBP) of emulsions is heavily dependent upon the test temperature, yet the test is carried out at ambient temperature. USA anticipates further technical conversation, including involvement of some of the technical experts from USA explosive test labs.
* AEISG supported the proposal in principle. It noted that the 8(e) MBP test examines the intrinsic properties of ANEs and, as such, is an acceptable alternative to, not a replacement for, the 8(d) test.
* Sweden questioned whether the 8(e) MBP test is applicable to the ANE in a portable tank citing the following reasons:
	+ the scale is very much different between Test 8(e) and portable tanks. Sweden believes that for non-ideal explosives such as ANEs, the performance characteristics of the explosive are largely dependent on the scale, which may imply the MBP of ANE in a tank is significantly lower than the MBP determined by test 8(e).
	+ the testing conditions in Test 8(e) are quite different from ANEs in a portable tank and especially under intensive fire.

**Conclusion:** IME will consider the comments provided above and plans to work intersessionially to develop a formal proposal for consideration in the 2021/2022 biennium.

Agenda Item 2(b)(iii) – Review of tests in parts I, II and III of the Manual of Tests and Criteria

1. **Subject.** Test Series H

*Documents: ST/SG/AC.10/C.3/2020/3-ST/SG/AC.10/C.4/2020/4 and Add.1 (Chair of the Working Group on Explosives)*

*Informal documents: UN/SCETDG/57/INF.4-UN/SCEGHS/39/INF.3 (Chairman of the Working Group on Explosives)*

**Discussion:** As the proposals to update Test Series H were the work and consensus of the IGUS/EOS working group, the EWG agreed to accept the proposed amendments as described in the above-referenced documents.

**Conclusion:** Revise section 28 of the MTC as shown in Annex 3, Amendment 1.

Agenda Item 2(b)(v) – Review of packing instructions for explosives

1. **Subject.** Orientation marks on packages of shaped charges (UN 0059, 0439, 0440, and 0441)

*Documents: ST/SG/AC.10/C.3/2020/51 (United Kingdom)
ST/SG/AC.10/C.3/2020/53 (United Kingdom)*

*Informal documents: None submitted*

**Discussion:** Having considered the WR replies to the questions in 2020/53 and that the majority of WR comments supported proposal 1 of 2020/51, the EWG supported, without opposition, adoption of that proposal. The EWG did not support proposal 4 in 2020/51. Additionally:

* IME and USA noted that 1.4S classifications for shaped charges (UN 0441) are rare since the implementation of the 6(d) test. They further noted that, most often, shaped charges are packaged in pairs and in such case the orientation marking requirement of PP70 would not apply. There are some cases of shaped charge packaging where they are not arranged in pairs and, in that case, the orientation markings are informative and desirable.
* While supporting proposal 1, Belgium suggested the possibility of adding references to shaped charges to section 5.2.1.7.1 of the MR to provide additional clarity.
* The explosives expert from the UK confirmed, based on his experience, the accuracy of the comments from the IME and USA regarding the rarity of 1.4S shaped charges and that of “singly packaged” shaped charges.
* Noting the two defined packaging options (in pairs or singly) described in P137 for shaped charges, Germany questioned if there might be other ways (a third option) of packaging shaped charges that should be considered. USA responded that, on very rare occasions, it had seen such third options, that were handled by issuance of a prescriptive packaging note on the approval.
* The packaging expert from the UK advised that proposal 1 was acceptable and that it was withdrawing proposals 2 – 4.

2020/53 contains a series of questions from the UK with the goal of better understanding shaped charge packaging. Noting that:

* IME advised that it had provided, in the WR replies, answers to all of the questions as well as a series of examples of shaped charge packaging. The UK had not seen those replies and IME advised that it would send them directly to the UK.
* Germany noted that the packaging in pairs is intended to cancel the jetting effect of the shaped charge in the event of an accidental initiation in the package. They further cautioned that this canceling effect could be lost if the shaped charges shift in the packaging such that they are no longer in true pairs. IME advised, and USA confirmed, that in most cases, the inner packagings for shaped charges are designed to maintain that orientation throughout the time period in which the shaped charges are in their packaging.
* The packaging expert from the UK noted that some confusion could arise for the difference in meaning and the use of the terms “in pairs” and “paired” and suggested some review should be completed to minimize that potential confusion.

**Conclusion:** Amend the first sentence of PP70 with proposal 1 of 2020/51 as shown in Annex 2, Amendment 1. The UK will review the comments received for both papers as it continues its review of packagings for shaped charges.

Agenda Item 2(b)(vii) – Test N.1 for readily combustible solids

1. **Subject.** Test N.1

*Documents: ST/SG/AC.10/C.3/2020/34 (China)*

*Informal documents: None submitted*

**Discussion:** After discussion of 2020/34, the EWG supported, with no opposition, adoption of proposal 2. Further, CEFIC and the Netherlands suggested to add the word “inner” before the words “width 20 mm” in section 33.2.4.2 of the MTC. This suggestion was accepted by the EWG with no opposition.

**Conclusion:** Amend Figure 33.2.4.1 (A) according to proposal 2 of 2020/34 as shown in [Annex 3, Amendment 2](#Annex3Amd2) and amend the first sentence of Section 33.2.4.2 of the MTC by adding the word “inner” before the words “width 20 mm” as shown in Annex 3, Amendment 3.

Agenda Item 2(b)(xii) – Miscellaneous

1. **Subject.** UN Gap Test (French description)

*Documents: ST/SG/AC.10/C.3/2020/17 (Secretariat)*

*Informal documents: None submitted*

**Discussion:** France confirmed the accuracy of the correction proposed in 2020/17. AEISG inquired as to whether other versions of the Gap Test in the MTC had been checked and the Secretariat advised that they had. The only occurrence of the error is in TS 1(a). France further confirmed that the correction reflects actual practice when performing the test. The EWG agreed, without objection, with the proposed correction in the above-referenced document.

**Conclusion:** Correct para. 11.4.1.2.1 of the MTC (French version) as shown in Annex 3, Amendment 4.

1. **Subject.** Exclusion from Class 1

*Documents: ST/SG/AC.10/C.3/2020/25 (France, COSTHA)*

*Informal documents: UN/SCETDG/57/INF.6 (COSTHA)*

**Discussion:** Document 2020/25 was discussed by the EWG to provide some guidance to France and COSTHA as they further pursue efforts to obtain some pathway out of Class 1 and into Class 9 for fire suppression dispersion devices that use technology similar to automotive safety devices for non-pyrotechnic effect. It is COSTHA’s opinion that the devices do not perform with an explosive effect and, in the contrary, function to provide a fire suppressing effect. They noted that INF.6 was produced to show what a new Dangerous Goods List (DGL) entry might comprise.

* Sweden observed that some formal guidance is needed in the MR to describe what type of explosives were acceptable for movement from Class 1 to Class 9 or as non-hazardous. It was also pointed out that classification in Class 9 can have direct consequences to future GHS classification, see paragraph 15 of this report, and that such guidance would be helpful also from that point-of-view.
* USA commented that auto safety devices, to which the devices described in 2020/25 are compared, are installed in transport vehicles and typically employ small amounts of explosives, whereas the devices described in 2020/25 generate large amounts of smoke and have no apparent size limit. They noted that articles may be fully excluded from Class 1 if they meet the exclusion criteria identified in the MR, whereas there is currently no technical criteria that differentiate Class 9 safety devices from the hazard of 1.4S articles. They agreed that the philosophical aspect raised by Sweden should be addressed before development of criteria or creation of new entries for the DGL.
* The UK pointed out that the new entry UN3548 ARTICLES CONTAINING MISCELLANEOUS DANGEROUS GOODS. N.O.S. may be an acceptable entry as opposed to creating new entries for articles in question. If the requirements of Section 2.1.3.6.4 of the MR are met, it may be possible to use the entry as the item would have been excluded from Class 1.
* In view of increasing requests for Class 9 classification of explosives other than safety devices, SAAMI stated support of new technical criteria for evaluating explosives for Class 9, and that this should be detached from the current rules for safety devices.
* CLEPA stated that they were hesitant to use the automotive safety device entry (UN 3268) to describe other devices such as those described in 2020/25.

Having considered the WR comments to these documents, and those received during the discussion on Day 2 of the EWG meeting, COSTHA advised they may return next biennium with a new proposal.

**Conclusion:** See Discussion.

Agenda Item 6(e) – Other miscellaneous proposals

1. **Subject.** Use of the terms “risk” and “hazard/danger”

*Documents: ST/SG/AC.10/C.3/2020/58 (Romania)*

*Informal documents: None submitted*

**Discussion:** Document TDG 2020/58 is not specifically addressed to the EWG; however, there are some instances where the subject of the paper would directly impact Class 1. Examples cited include PP52 and PP76 and other instances have also been identified. However, there was insufficient time to discuss the review of the use of the terms “risk” and “hazard/danger” as related to Class 1. The expert from Romania will forward his specific questions to the EWG for consideration intersessionally and for discussion during the next biennium.

**Conclusion:** Defer discussion until the 2021/2022 biennium.

Agenda Item 11(a) – Review of GHS Chapter 2.1

1. **Subject.** Final EWG recommendation on the new GHS chapter for explosives to the Sub-Committees

*Documents: ST/SG/AC.10/C.3/2020/20-ST/SG/AC.10/C.4/2020/5 and Add.1 (ICG via Sweden)
ST /SG/AC.10/C.4/2020/18 (Sweden)[[2]](#footnote-3)*

*Informal documents: UN/SCETDG/57/INF.16-UN/SCEGHS/39/INF.13 (Sweden)[[3]](#footnote-4)
UN/SCETDG/57/INF.17-UN/SCEGHS/39/INF.14 (Sweden)
UN/SCETDG/57/INF.23 (Sweden)[[4]](#footnote-5)
UN/SCEGHS/39/INF.22 (Germany)
UN/SCEGHS/39/INF.25 (Sweden)*

**Discussion:**

1. ***Chapter 2.1 as such.*** The EWG considered and endorsed the amendments to GHS Chapter 2.1 proposed by the GHS 2.1 ICG in TDG 2020/20 and Add.1 (GHS 2020/5 and Add.1), as consolidated in TDG INF.17 (GHS INF.14) with the following recommendations:
* That the first sentence of 2.1.1.2.2 should be amended as shown below, as no articles are listed in points (a) – (c) that follow:

*The following substances~~,~~ and mixtures ~~and articles~~ are excluded from the class of explosives:*

* That the text proposed for section 2.1.1.3.1 should be amended as shown (blue underscored text) below:

*The GHS classification of substances, mixtures and articles as explosives builds largely on the classification used for transport according to the UN Model Regulations. Information on their transport division and, when available, some of the underlying test results according to Part I of the Manual of Tests and Criteria, is therefore relevant for the GHS classification. Test data is not required when classification using expert judgement is possible based on available information from previous testing and characterization. Where appropriate, analogy to tested explosives may be used, taking into consideration whether changes to the configuration may affect the hazard posed compared to the tested configuration.**While the transport divisions are designed for the purpose of safe transportation of explosives, the GHS classification draws from this classification to ensure appropriate hazard communication in other sectors, in particular supply and use. In doing this, any mitigating effects of the transport configuration on the explosive behaviour, such as a particular packaging, are evaluated as they may not be present in sectors outside of transport.*

* That the text of second sentence of 2.1.1.3.4 should be amended as shown below:

*An explosive that was assigned to a division in a certain configuration, and hence classified in a sub-category within Category 2, may no longer retain~~s~~ that division when out of that configuration.*

* The last sentence of 2.1.1.3.5 appears to some to imply a requirement for a safety data sheet for articles, and further deliberations are needed.
* That, referring to the merged criteria table in 2.1.2, for Category 1, point (a)(ii) the word “effects” should be changed to “results”. It was confirmed the merged table as thus amended is correct in relation to previously agreed criteria.
* That proposed changes to 2.1.3, 2.1.4.2 and 2.1.4.3 were acceptable without comment.
1. ***Decision logics.*** The EWG considered and endorsed the amendments to GHS Chapter 2.1 proposed in GHS 2020/18, GHS INF.22, and GHS INF.25 as follows:
* The decision logic 2.1(a) in GHS 2020/18 is correct in relation to the criteria.
* The order of boxes in decision logic 2.1(a) should be changed as proposed in para. 5 of GHS INF.22 and the word “effects” in box 5A should be changed to “results”.
* The decision logic 2.1(b) in GHS 2020/18 is correct in relation to the criteria and was accepted. The alternative decision logic 2.1(b) in para. 6 of GHS INF.22 was not supported by the EWG.
* The footnote proposed in GHS INF.25 was amended and accepted as shown below:

*a In the absence of results from test 6 (a) or 6 (b), results from test 6 (d) may be used to assess whether there was a high hazard event, see 2.1.2.1. If the configuration includes attenuating features that are likely to mitigate a high hazard event, such as spacing or a specific orientation of explosive articles, Sub-category 2A may be assigned without the need to assess test data.*

**Conclusion:** Refer EWG recommendations in a) and b) above to the ICG for the review of GHS Chapter 2.1 and the Sub-Committees.

1. **Subject.** Amendments to the MTC

*Documents: ST/SG/AC.10/C.3/2020/22-ST/SG/AC.10/C.4/2020/7 (Sweden)
ST/SG/AC.10/C.3/2020/60-ST/SG/AC.10/C.4/2020/16 (Sweden and EWG Chair)*

*Informal documents: None submitted*

**Discussion** Noting that TDG 2020/22 (GHS 2020/7) contained no proposals, the EWG considered and endorsed the amendments to the MTC proposed in TDG 2020/60 (GHS 2020/16) with the amendments indicated below:

* Box 19 of Figure 10.2 is revised to read:

*Provisionally classify as an explosive. (Go to Figure 10.3)*

* Section 10.4.1.1. is corrected to read:

*:“… 2.1.1.2.1 (b) of the GHS”*

**Conclusion:** Refer EWG endorsement and amendments described above to the ICG for the review of GHS Chapter 2.1 ICG and the Sub-Committees.

1. **Subject. Additional provisions for Non-Class 1 products**

*Documents: None submitted*

*Informal documents: UN/SCETDG/57/INF.15-UN/SCEGHS/39/INF.10 (USA, IME, SAAMI)
UN/SCETDG/57/INF.33-UN/SCEGHS/39/INF.19 (ICG via Sweden)*

**Discussion:** The EWG considered and endorsed in principle the amendments to GHS Chapter 2.1 proposed by the GHS 2.1 ICG in TDG INF.33 (GHS INF.19). However, the Secretariat noted that the use of the term “products” in this context may be inappropriate. It was concluded that is not a technical issue to be resolved by the EWG, but that it is one that should be considered and resolved by the GHS Sub-Committee. Some believed that the scope for these provisions should cover only articles, and not be extended to cover substances or mixtures.

It was also pointed out that the provision for Class 6 needs to be restricted to Division 6.1, in order not to comprise infectious substances, and that the Note should be amended accordingly.

As they were generally incorporated into the proposals in TDG INF.33 (GHS INF.19), TDG INF.15 (GHS INF.10) was not discussed.

**Conclusion:** Refer EWG endorsement to the ICG on the review of GHS 2.1 ICG and the Sub-Committees. Refer the EWG comments about the term “products” discussed above to the GHS Sub-Committee.

Annex 1
Working Group on Explosives (17 – 18 November 2020)
List of Participants

| Name | Representing | Email address |
| --- | --- | --- |
| Arnaud Vandenbroucke | Belgium | arnaud.vandenbroucke@economie.fgov.be |
| Richard Bowes | Canada | richard.bowes@canada.ca |
| Michael Lafleur | Canada | michael.lafleur3@canada.ca |
| Yanchun- China | China |   |
| Chen Shuxin | China |  |
| Jian Zhou | China | zhoujian@ghs.cn  |
| Miina Grönlund | Finland | miina.gronlund@traficom.fi  |
| Mikko Ojala | Finland | mikko.ojala@tukes.fi  |
| Lionel Aufauvre | France | lionel.aufauvre@ineris.fr |
| Heike Michael-Schulz | Germany | heike.michael-schulz@bam.de |
| Alexander von Oertzen | Germany | alexander.von\_oertzen@bam.de |
| Cordula Wilrich | Germany | cordula.wilrich@bam.de  |
| Shu Usuba | Japan | shuusuba@live.jp |
| Jun-Hwa Ban | Korea, Republic of | warm2240@gmail.com  |
| Soedesh Mahesh | Netherlands | soedesh.mahesh@rivm.nl |
| Patrycja Ruskowska | Poland | Patrycja.Ruskowska@mr.gov.pl  |
| Mihai Cuciureanu | Romania | adr@arr.ro  |
| Olga Geraskina | Russia | o.geraskina@ciscenter.org |
| Agustin Chousa | Spain | achousa@miteco.es  |
| Ramon Gonzalez | Spain | reguren@maxamcorp.com |
| Shulin Nie | Sweden | shulin.nie@msb.se |
| Lorens van Dam | Sweden | lorens.van.dam@msb.se |
| Nathan Flood | UK | nathan.flood@hse.gov.uk  |
| Phil Smith | UK | philip.smith@hse.gov.uk |
| Deb Traynor | UK | deborah.traynor@hse.gov.uk  |
| Keith White | UK | keith.white@vca.gov.uk  |
| Britain Bruner | USA | britain.bruner@dot.gov |
| Andrea Dunham | USA | andrea.dunham@dot.gov |
| Michael Givens | USA | michael.givens@faa.gov  |
| Michael Klem | USA | michael.klem@dot.gov |
| Brent Knoblett | USA | brent.e.knoblett.civ@mail.mil  |
| Jennifer Lawless | USA | lawless.jennifer@dol.gov  |
| Kevin Leary | USA | kevin.leary@dot.gov |
| Joseph Nicklous | USA | joseph.nicklous@dot.gov |
| Duane Pfund | USA | duane.pfund@dot.gov  |
| Keith Ranck | USA | keith.ranck@faa.gov  |
| Maureen Ruskin | USA | ruskin.Maureen@dol.gov |
| Ryan Vierling | USA | ryan.vierling@dot.gov |
| Brian Vos | USA | brian.vos@dot.gov |
| Steven Webb | USA | steven.webb@dot.gov |
| Aaron Weiner | USA | Aaron.Wiener@dot.gov |
| Rosa Garcia Couto | UN/ECE | rosa.garciacouto@un.org |
| Romain Hubert | UN/ECE | romain.hubert@un.org  |
| Armando Serrano Lombillo | UN/ECE | serranolombillo@un.org |
| Ken Price | AEISG | ken@riskom.com.au |
| Bob Sheridan | AEISG | bob.sheridan@aeisg.org.au |
| Johann Zank | AEISG | johann.zank@orica.com |
| Dieter Heitkamp | CEFIC | dieter.heitkamp@bayer.com |
| Werner Lange | CEFIC | dr.werner.lange@icloud.com |
| Peter Schuurman | CEFIC | peter.schuurman@nouryon.com |
| Klaus Pilatus | CLEPA | klaus.pilatus@autoliv.com |
| Tom Ferguson | COSTHA | tom@costha.com |
| Lance Harry | COSTHA | lanceh@statx.com |
| Dave Madsen | COSTHA | dave.madsen@autoliv.com |
| Jason Newell | COSTHA | Jason.Newell@joysonsafety.com |
| Bob Richard | Hazmat Safety Consulting | brichard@hazmatsafety.com |
| David Boston | IME | dboston@ime.org |
| Noel Hsu | IME | noel.hsu@orica.com |
| Jackson Shaver | IME | jackson.shaver@dssa.daicel.com  |
| Kaylee Baker | RPMASA | kaylee.baker@aeciworld.com |
| Cheryl Kelly | RPMASA | cheryl.kelly@aeciworld.com  |
| Andre Pienaar | RPMASA | andre.pienaar@aeciworld.com  |
| Ben Barrett | SAAMI | ben.barrett@dgadvisor.com |
| Robert Ford | SAAMI | rford@smsenergetics.com |
| Matthew Spencer | SAAMI | mspencer@hornady.com |
| Yushuang Chen | Shanghai Institute of Chemical Industry Co., Ltd. | cys@ghs.cn  |
| Chaozheng Huang | Shanghai Institute of Chemical Industry Co., Ltd. | hcz@ghs.cn  |

Annex 2
Working Group on Explosives (17 – 18 November 2020)
Changes for the Model Regulations (21st Revised Edition)

Notes: Source of proposed change is indicated by *italicized text (Source: XXX)*

 ~~Red~~ indicates deleted text

 Blue indicates inserted text

**Section 4.1.4.1** – Amend the first sentence of PP70 of packing instruction P137 as shown below.

For UN Nos. 0059, 0439, 0440 and 0441, when the shaped charges are packed singly, the conical cavity shall face downwards and the package shall be marked as illustrated in figures 5.2.3 or 5.2.4 ~~in accordance with 5.2.1.7.1~~ on two opposite sides.

*Source: ST/SG/AC.10/C.3/2020/51, Para. 8 (Proposal 1) and Para. 8 of this report.*

Annex 3
Working Group on Explosives (17 – 18 November 2020)
Changes for the Test Manual (7th Revised Edition)

Notes: Source of proposed change is indicated by *italicized text (Source: XXX)*

 ~~Red~~ indicates deleted text

 Red indicates inserted text

**Section 28** – Amend section 28 as shown below. In Amendment 1 only, red text indicates inserted text and ~~red text~~ indicates deleted text.

*Source: ST/SG/AC.10/C.3/2020/3-ST/SG/AC.10/C.4/2020/4 and UN/SCETDG/57/INF.4-UN/SCEGHS/39/INF.3 (as amended by ST/SG/AC.10/C.3/2020/3/Add.1-ST/SG/AC.10/C.4/2020/4/Add.1) and Para. 7 of this report.*

SECTION 28

TEST SERIES H

28.1 Introduction

28.1.1 This test series contains test methods for the determination of the self-accelerating decomposition temperature (SADT) and the self-accelerating polymerization temperature (SAPT). The SADT is defined as the lowest temperature at which self-accelerating decomposition may occur with a substance in the packaging. The SAPT is defined as the lowest temperature at which self-accelerating polymerization may occur with a substance in the packaging. The SADT and SAPT are measures of the combined effect of the ambient temperature, reaction kinetics, package size and the heat transfer properties of the substance and its packaging. To assist in interpreting the results, the following models can be used[[5]](#footnote-6) ~~in which the main resistance to heat flow is~~:

 ~~(a) At the boundary i.e. packaging (Semenov model);~~

 ~~(b) Within the substance (Frank-Kamenetskii model); or~~

 ~~(c) A combination of these (Thomas model).~~

(a)    Semenov model, in which the main resistance to heat flow is at the boundary (i.e. packaging). This model is generally applicable to homogeneous liquids but can also be applied to solids in packagings (excluding IBC~~’~~s);

(b)   Frank-Kamenetskii model, in which the main resistance to heat flow is within the substance.  This model is generally applicable to solids in larger packagings, IBCs or tanks;

(c)     Thomas model, in which the resistance to heat flow is from both the boundary and the substance;

(d)    Non-stationary models, e.g. finite element methods (FEM), or computational fluid dynamics (CFD) all combined with thermal kinetic methods.

28.1.2 The text should be used in conjunction with the temperature control requirements given in sub-section 2.5.3.4 of the Model Regulations.

28.1.3  *Both the SADT and the SAPT may be influenced by factors such as aging of the sample, presence of stabilizers or impurities in the sample (including packaging materials in contact with the substance). These possible influencing factors should be considered when evaluating the results of an SADT or SAPT determination.*

28.2 Test methods

28.2.1 Test series H comprises tests and criteria concerning the thermal stability of substances or concerning the determination of whether a substance meets the definition of a self-reactive substance or a polymerizing substance.

28.2.2 Each test involves either storage at a fixed external temperature and observation of any reaction initiated or storage under near adiabatic conditions and measurement of the rate of heat generation versus temperature. The test methods included in test series H are given in Table 28.1. Each of the methods listed is applicable to solids, liquids, pastes and dispersions.

**Table 28.1: Test methods for test series H**

|  |  |  |
| --- | --- | --- |
| **Test code** | **Name of test** | **Section** |
| H.1 | United States SADT/SAPT test**a** | 28.4.1 |
| H.2 | Adiabatic storage test (AST)**b** | 28.4.2 |
| H.3 | Isothermal storage test (IST)**b** | 28.4.3 |
| H.4 | Heat accumulation storage test**c** | 28.4.4 |

 **a***Recommended test for substances contained in packagings.*

 **b***Recommended test for substances contained in packagings, IBCs or tanks.*

 **c** *Recommended test for substances contained in packagings, IBCs or small tanks.*

 The list of tests is not exhaustive; other tests may be used provided that they give the correct SADT or SAPT of the substance as packaged.

28.2.3 When temperature control is necessary (see Table 28.2) the control and emergency temperatures should be derived from the SADT or SAPT using Table 28.3.

Table 28.2: Criteria for temperature control

|  |  |
| --- | --- |
| **Type of substance** | **Criterion for temperature control** |
| Self-reactive substance | SADT ≤ 55 °C |
| Organic peroxide Type B and C | SADT ≤ 50 °C |
| Organic peroxide Type D showing medium effect when heated under confinementa | SADT ≤ 50 °C |
| Organic peroxides Type D showing low or no effect when heated under confinementa | SADT ≤ 45 °C |
| Organic peroxides Type E and F | SADT ≤ 45 °C |
| Polymerizing substance in packaging or IBC | SAPT ≤ 50 °C |
| Polymerizing substance in portable tank | SAPT ≤ 45 °C |
| a *As determined by test series E as prescribed in this Manual of Tests and Criteria, Part II.* |

Table 28.3: Derivation of control and emergency temperatures

|  |  |  |  |
| --- | --- | --- | --- |
| **Type of receptacle** | **SADT/SAPT a** | **Control temperature** | **Emergency temperature** |
| Single packagings and IBCs | ≤ 20 °C  | 20 °C below SADT/SAPT | 10 °C below SADT/SAPT |
| over 20 °C to 35 °C | 15 °C below SADT/SAPT | 10 °C below SADT/SAPT |
| over 35 °C | 10 °C below SADT/SAPT | 5 °C below SADT/SAPT |
| Portable tanks | ≤ 45 °C | 10 °C below SADT/SAPT | 5 °C below SADT/SAPT |
| a *i.e. the SADT/SAPT of the substance as packaged.* |

28.2.4 If a substance is being tested to determine whether it is a self-reactive substance, a test of series H, or a suitable alternative test, should be performed to determine if its SADT would be less than or equal to 75 °C when contained in a 50 kg package.

28.2.5 If a substance is being tested to determine whether it is a polymerizing substance, a test of series H, or a suitable alternative method, should be performed to determine if its SAPT would be less than or equal to 75 °C in its packaging, IBC or portable tank.

28.2.6 The results obtained for ~~the~~ larger~~st commercial~~ packages are applicable to smaller packages of similar construction and material provided that the heat transfer per unit mass is no smaller than from the larger package.

28.3 Test conditions

28.3.1 For organic peroxides and self-reactive substances the preliminary procedure (see section 20.3) should be performed and the effect of heating under confinement (test series E) determined before performing SADT tests. ***Safety precautions should be taken which allow for the possibility of catastrophic failure of the test vessel and for the hazards arising from the ignition of secondary fuel-air mixtures and the evolution of toxic decomposition products. Substances liable to detonate should only be tested with special precautions.***

28.3.2 The test selected should be conducted in a manner which is representative, both in size and material, of the package. For metal packagings, IBCs or tanks, it may be necessary to include a representative amount of the metal in the sample tested i.e. representative of both the metal(s) and the area of contact.

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

28.3.4 Samples which have been tested at a particular temperature and are apparently unreacted may be used again, for screening purposes only, provided extra care is taken. Fresh samples should be used for the final ~~actual~~ determination of the SADT or SAPT.

28.3.5 If the complete package is not tested, the heat loss data used for the determination of the SADT or SAPT should be representative of the package, IBC or tank. For all type of packagings up to 50 kg for solids or 200 kg/225 litres for liquids and for IBCs up to 1250 litres for liquids, a standard heat loss per unit of mass is given in Table 28.4. For other packagings, IBC’s or tanks or when there is a need for a heat loss value that deviates from the one as given in Table 28.4, the actual heat loss value per unit of mass has to be determined. In this case, ~~T~~the heat loss per unit of mass of the package, IBC or tank may be determined by calculation (taking account of the quantity of substance, dimensions of the package, heat transfer in the substance, ~~and~~ t~~he~~ heat transfer through the packaging and heat transfer from the outer wall of the packaging to the environment (see note)) or by measuring the half-time of cooling of the package filled with the substance or another substance having similar physical properties. The heat loss per unit mass, L (W/kg.K), can be calculated from the half-time of cooling, t1/2 (s), and the specific heat, Cp (J/kg.K), of the substance using the formula:



***NOTE:*** *For calculations an external heat transfer coefficient (i.e. heat transfer from the outer wall of the packaging to the environment) of 5 W/m2.K can be used.*

28.3.6 The half-time of cooling can be determined by measuring the period of time in which the temperature difference between the sample and its surroundings is decreased by a factor of 2. For example, for liquids, the packaging may be filled with silicone oil, apparent density 0.96 ± 0.02 at 20 °C and heat capacity 1.46 ± 0.02 J/g at 25 °C or dimethyl phthalate and this heated to about 80 °C. Water should not be used as erratic results may be obtained through evaporation/condensation. For solids for example, the packaging may be filled with dense soda ash (apparent density greater than 1 g/cm3) and this is heated to about 80 °C. The temperature drop is measured at the centre of the package over the temperature range which includes the expected SADT or SAPT. For scaling, it may be necessary continuously to monitor the temperature of the substance and surroundings and then use linear regression to obtain the coefficients of the equation:

ln {T - Ta} = co + ct

where: T = substance temperature (°C);

 Ta = ambient temperature (°C);

 co = ln{Initial substance temperature - initial ambient temperature}; and

 c = L/Cp;

 t = time (s).

28.3.7 Standard ~~Examples of the~~ heat loss characteristics of ~~some typical~~ packages, IBCs and tanks are given in Table 28.~~4~~. The actual value obtained will depend on the shape, wall thickness, surface coating etc. of the packaging.

**~~Table 28.3: HEAT LOSS PER UNIT MASS FROM PACKAGES, IBCs AND TANKS~~**

|  |  |  |  |
| --- | --- | --- | --- |
| **~~Type of receptacle~~** | **~~Normal capacity (litres)~~** | **~~Filling~~** | **~~Heat loss per unit mass (L) (mW/K.kg)~~** |
| *~~For liquids:~~* |
| ~~1A1~~ | ~~50~~ | ~~47.5 kg DMP~~**~~a~~** | ~~63~~ |
| ~~1H1~~ | ~~50~~ | ~~47.5 kg DMP~~**~~a~~** | ~~94~~ |
| ~~1H1~~ | ~~200~~ | ~~200 kg water~~ | ~~56~~ |
| ~~3H1 (black)~~ | ~~60~~ | ~~47.5 kg DMP~~**~~a~~** | ~~105~~ |
| ~~6HG2~~ | ~~30~~ | ~~35.0 kg DMP~~**~~a~~** | ~~69~~ |
| ~~IBC 31 HA1~~ | ~~500~~ | ~~500 kg water~~ | ~~51~~ |
| ~~Tank~~ | ~~3 400~~ | ~~3 400 kg water~~ | ~~18~~**~~b~~** |
| ~~Tank container (insulated)~~ | ~~20 000~~ | ~~14 150 kg isododecane~~ | ~~1.7~~ |
| *~~For solids:~~* |
| ~~1G~~ | ~~38~~ | ~~28.0 kg DCHP~~**~~c~~** | ~~35~~ |
| ~~1G~~ | ~~50~~ | ~~37.0 kg DCHP~~**~~c~~** | ~~29~~ |
| ~~1G~~ | ~~110~~ | ~~85.0 kg DCHP~~**~~c~~** | ~~22~~ |
| ~~4G~~ | ~~50~~ | ~~32.0 kg DCHP~~**~~c~~** | ~~27~~ |

**~~a~~** *~~DMP = dimethyl phthalate.~~*

**~~b~~** *~~Calculated using a heat transfer coefficient of 5 W/m~~~~2~~~~.K.~~*

**~~c~~** *~~Dicyclohexyl phthalate (solid).~~*

**Table 28.4: HEAT LOSS PER UNIT MASS FROM PACKAGES, IBCs AND TANKS**

|  |  |  |  |
| --- | --- | --- | --- |
| **Type of receptacle** | **Normal capacity (litres)** |  | **Heat loss per unit mass (L) (mW/K.kg)a** |
| *For liquids:* |
| Packagings up to 200 kg/225 l 40bPackagings larger than 200 kg/225 l no standard valuecIBCs up to 1250 l 30IBCs larger than 1250 l no standard valuecTanks no standard valuec*For solids:* |
| Packagings up to 50kg 30bPackagings larger than 50 kg no standard valuecIBCs - no standard valuecTanks - no standard valuec |

a *For test series H.4 the heat loss of the Dewar vessel to be used should be as close as possible to the value listed in the table.*

b*When determining the SADT for exemption or classification purposes, where the definition of the SADT is connected to a 50 kg package, a value of 60 mW/K.kg for liquids and 30 mW/K.kg for solids should be used.
Such is the case when the SADT is used either to exclude a new substance from Division 4.1 as a self-reactive substance (see 20.2.1 (e)), or to classify it under Type G as a self-reactive substance (see 20.4.2 (g)), or organic peroxide (see 20.4.3 (g)).*

c *Value should be determined for the actual configuration.*

28.4 Series H test prescriptions

28.4.1 *Test H.1: United States SADT/SAPT test*

28.4.1.1 *Introduction*

 This method determines the minimum constant temperature air environment at which self-accelerating decomposition or polymerization occurs for a substance in a specific package. Packages of up to 225 litres may be tested by this method. An indication of the explosion hazard from the decomposition ~~or polymerization reaction~~ can also be obtained.

28.4.1.2 *Apparatus and materials*

28.4.1.2.1 The test substance and packaging should be representative of that intended for commercial use. The packaging forms an essential part of the test.

28.4.1.2.2 The apparatus consists of a test chamber in which the air surrounding the package under test can be maintained at a constant temperature for a period of at least ten days.

28.4.1.2.3 The test chamber should be constructed such that:

 (a) It is well insulated;

 (b) Thermostatically controlled air circulation is provided so as to maintain a uniform air temperature within ± 2 °C of the desired temperature; and

 (c) The minimum separation distance for all sides from the package to the wall is 100 mm.

 Any type of oven may be used provided that it is capable of meeting the temperature control requirements and will not ignite any decomposition products. Examples of ~~a~~ suitable small-package and large-package ovens are described below. ~~given in 28.4.1.2.4 and 28.4.1.2.5 respectively.~~

28.4.1.2.3.1 Example 1

~~28.4.1.2.4~~ A small-package oven may be constructed from an open head 220 litre steel drum. This will readily accommodate packages of up to 25 litres capacity. Construction details are shown in Figure 28.4.1.1. Larger packages may be tested as long as a 100 mm clearance between the package and the oven wall is maintained.

28.4.1.2.3.2 Example 2

~~28.4.1.2.5~~ An expendable large-package oven may be made from 50 mm × 100 mm lumber formed into a cubic frame 1.2 m on each side, which is lined inside and out with 6 mm thick waterproof plywood and insulated all around with 100 mm fibreglass insulation. Construction details are shown in Figure 28.4.1.2. The frame should be hinged on one side to permit loading and unloading of test drums. The floor should be provided with 50 mm × 100 mm lumber on edge, spaced 200 mm on centres to keep the test container off the floor and allow free air circulation around the package. Cleats should run normal to the door to permit a fork-lift to move the drums. A circulating fan should be located on the side opposite the door. The air flow should be from the upper corner of oven to the fan discharge at the diagonally opposite lower corner. An electric heater of 2.5 kW is suitable for heating the air. Thermocouples should be placed in the air intake and outflow ducts as well as the top, middle and bottom of the oven. For substances with an SADT or SAPT below ambient temperature, the test should be performed in a cooling chamber or solid carbon dioxide should be used for cooling the oven.

28.4.1.2.3.3 Example 3

 For tests at temperatures up to 75 °C, a double walled metal chamber (minimum separation distance from the package to the wall is 100 mm) may be used with fluid from a temperature-controlled circulating bath passed between the walls at the desired temperature. The test chamber is loosely closed by an insulated lid (e.g. made from 10 mm thick polyvinyl chloride). The temperature control should allow the desired temperature for a liquid inert sample to be maintained with a deviation of not more than ± 2 K for up to 10 days.

28.4.1.2.~~6~~4 The package should be equipped with a thermowell which positions the thermocouple or Resistance Temperature Detector (RTD) at the package mid-point. The thermowell may be constructed of glass, stainless steel or other suitable material but should be introduced in a manner which does not reduce package strength or venting capability.

28.4.1.2.~~7~~5 Continuous temperature-measuring and recording equipment, which is protected from fire and explosion hazards, is required.

28.4.1.2.~~8~~6 ***The tests should be performed in an area which provides adequate protection from fire and explosion hazards, and from toxic fumes. A safety distance, e.g. 90*** ***m, from public roads and occupied buildings is recommended. If there may be toxic fumes, longer safety distances may be required.***

28.4.1.3 *Procedure*

28.4.1.3.1 The package is weighed. A thermocouple or RTD is inserted in the package to be tested such that the temperature in the centre of the sample can be monitored. If the required oven temperature is below ambient, the oven is switched on and the inside of the oven cooled to the desired temperature before placing the package in the oven. If the required oven temperature is at or above ambient temperature, the package is inserted in the oven at ambient temperature and the oven then switched on. There should be a minimum clearance of 100 mm between the package and the sides of the oven.

28.4.1.3.2 The sample is heated and the temperature of the sample and test chamber continuously monitored. The time is noted at which the sample temperature reaches a temperature 2 °C below the test chamber temperature. The test is then continued for a further seven days or until the sample temperature rises to 6 °C or more above the test chamber temperature if this occurs sooner. ~~Note the time taken for the sample to rise from the 2 °C below the test chamber temperature to its maximum temperature.~~

28.4.1.3.3 When the test is completed, cool the sample and remove it from the test chamber. Note the variation of temperature with time. If the package remains intact, note the percentage mass loss and determine any changes in composition. Dispose of the sample as soon as possible.

28.4.1.3.4 If the temperature of the sample does not exceed the oven temperature by 6 °C or more then repeat the test with a new sample in an oven at a 5 °C higher temperature. The SADT or SAPT is defined as the lowest oven temperature at which the sample temperature exceeds the oven temperature by 6 °C or more. If the substance is being tested to determine if temperature control is necessary, perform sufficient tests to determine the SADT or SAPT to the nearest 5 °C or to determine if the SADT or SAPT is greater than the applicable temperature specified in Table 28.2.~~or equal to 60 °C~~. If the substance is being tested to determine if it meets the SADT criterion for a self-reactive substance, perform sufficient tests to determine if the SADT for a 50 kg package is 75 °C or less. If the substance is being tested to determine if it meets the SAPT criterion for a polymerizing substance, perform sufficient tests to determine if the SAPT in the packaging as used is 75 °C or less.

28.4.1.4 *Test criteria and method of assessing results*

28.4.1.4.1 The SADT or SAPT is reported as the lowest temperature at which the sample exceeds the oven temperature by 6 °C or more. If the sample temperature does not exceed the oven temperature by 6 °C or more in any test, the SADT or SAPT is recorded as being greater than the highest oven temperature used.

28.4.1.5 *Examples of results*

|  |  |  |  |
| --- | --- | --- | --- |
| **Substance** | **Sample mass (kg)** | **Packaging** | **SADT/SAPT (°C)** |
| tert-Amyl peroxybenzoate | 18.2 | 6HG2, 22.8 litres | 65 |
| tert-Butyl peroxyacetate (60%) | 7.2 | 6HG2, 22.8 litres | 75 |
| Dibenzoyl peroxide  | 0.45 | 1G | 70 |
| Di-(4-tert-butylcyclohexyl) peroxydicarbonate | 43 | 1G | 40 |
| 2,5-Diethoxy-4-morpholinobenzene- diazonium zinc chloride (66%) | 30 | 1G, 50 litres | 50 |
| 2-(N-ethoxycarbonyl-N-phenylamino)- 3-methoxy-4-(N-methyl-N-cyclohexyl- amino)benzenediazonium zinc chloride (62 %)Didecanoylperoxide, technically pure2,2´-Azodi-(isobutyronitrile) | 102050 | 6HG1, 25 litres1G1G | 504050 |
|  |  |  |  |

**Figure 28.4.1.1: Small package oven (Example 1)**

(A) Insulation 25 mm thick (B) 220 litres open top drum

(C) 19 mm pipe (D) 9.6 mm eye bolt in steel cover

(E) Insulation on steel cover (F) 3 mm control cable

(G) Fan (H) Thermocouples and controls

(J) Drain (K) 25 mm angle stand

(L) 2 kW drum heater

**Figure 28.4.1.2: Large package oven (top view and side view) (Example 2)**

(A) Fan (B) Hinges (2)

(C) Insulation (D) Heater

(E) Drum (F) Latch

(G) Drum (e.g. 0.58 m x 0.89 m)

28.4.2 *Test H.2: Adiabatic storage test*

28.4.2.1 *Introduction*

28.4.2.1.1 This test method determines the rate of heat generation produced by a reacting substance as a function of temperature. The heat generation parameters obtained are used with the heat loss data ~~relating~~ of ~~to~~ the package to determine the SADT or SAPT of a substance in its packaging. The method is appropriate for every type of packaging, including IBCs and tanks. There are two versions of the adiabatic storage test:

(a) Open version: A Dewar vessel within an oven is used ~~for this version~~. This set-up uses a capillary to prevent pressure build-up and a cooling system to limit the temperature increase due to a thermal runaway reaction;

(b) Closed version ~~system~~: ~~In this version~~ a test vessel (e.g. Dewar or thin-walled vessel) is placed within an autoclave in an oven. Here the autoclave prevents the release of pressure to the surroundings during the test.

28.4.2.1.2 ~~Measurements can be performed in the temperature range from -20 °C to 220 °C.~~ The smallest temperature rise that can be detected with this method depends on the properties of the sample, but generally corresponds to a ~~rate of~~ heat generation rate of 15 mW/kg. The upper limit of the open version is determined by the capacity of the cooling system to safely cool the substance (up to 500 W/kg if water is used as ~~the~~ coolant). The closed version can disregard this limit if performed in a high-pressure autoclave. ~~Although the test is not perfectly adiabatic, the heat losses are less than 10 mW.~~ The maximum allowable error in heat generation is 30% at 15 mW/kg and 10% from 100 mW/kg to 10 W/kg. The detection limit of the adiabatic test should be suitable for assessing the heat loss from the package under consideration (e.g. 100 to 500 mW/kg for L=60 mW/K.kg). If significant extrapolation of heat production rates derived from adiabatic test data is required, a validation with additional isothermal tests is recommended.

28.4.2.1.3 An explosion may occur in the open version of the test i~~I~~f the cooling system is activated at a stage where the rate of heat generation exceeds the cooling capacity of the apparatus ~~an explosion may occur~~. For the closed version an explosion could lead to a rupture of the autoclave or its fittings. ***The test site should therefore be carefully selected to reduce to a minimum the possible hazards from an explosion and of a possible subsequent gas explosion of the decomposition products (secondary explosion).***

28.4.2.2 *Apparatus and materials*

28.4.2.2.1 Open version

 The apparatus consists of a glass Dewar vessel (max. 3~~1.0or 1.5~~ litre) to contain the sample, an insulated oven with a differential control system to keep the temperature in the oven to within 0.1 °C of the sample temperature, and an inert lid for the Dewar vessel. In special cases, sample holders of other construction materials may have to be used. An inert heating coil and cooling tube pass through the lid into the sample. Pressure build-up in the Dewar vessel is prevented by a~~2 m long PTFE~~ sufficiently long capillary tube made of an inert substance (e.g. 2 m long PTFE tube) ~~which passes~~ through the insulated lid. A constant power heating unit is used for internal heating of the substance to a ~~pre-set~~ temperature or for calibration. Internal heating and cooling may be stopped or started automatically at ~~pre-set~~ temperatures. In addition to the cooling system, a secondary safety device is used to~~fitted which~~ disconnect~~s~~ the power supply to the oven at a ~~pre-set~~ temperature. A schematic drawing of ~~the~~an open apparatus for the adiabatic storage test ~~AST~~ is given in Figure 28.4.2.1.

28.4.2.2.2 Closed version

28.4.2.2.2.1 The apparatus consists of a suitable inert vessel (e.g. Dewar or thin-walled test cell) to contain the sample, a high-pressure autoclave and an insulated oven with a differential temperature control system. Thin-walled test cells require the use of a pressure control system to balance the internal and external cell pressure.

28.4.2.2.2.2 The phi-factor (heat capacity of the set-up and the sample divided by heat capacity of the sample) of the system should be known and be considered in evaluating the tests results. Therefore, a suitable combination of phi-factor, insulation, and amount of substance should be chosen. The heat losses from the apparatus and detection limit of the system also have to be taken into consideration. An inert heating coil may be inserted into the sample. In addition to the high-pressure autoclave, a secondary safety device is used to disconnect the power supply to the oven at a pre-set temperature.

28.4.2.2.2.3 The closed version of the test is preferred for substances with a high vapour pressure at the test temperature to prevent mass loss due to evaporation or for substances that decompose with severe pressure rises (which in case of an open version of the test would throw off the insulated lid or eject the sample from the test cell). The weight of the sample should be determined after the measurement to detect mass loss during the test. Leakage from the system and the resulting evaporation cooling can result in a significant loss of sensitivity in the test and a large margin of error in the results. The suitability of a test run in the open version can be evaluated by determining the mass loss of the sample after the test.

28.4.2.2.3 The temperature of the substance is measured at its centre by means of thermocouples or platinum resistance sensors (RTD) placed in a steel or glass tube. The temperature of the surroundings ~~air~~ is measured at the same height as the sample temperature, also with thermocouples or platinum resistance sensors. Continuous temperature measuring and recording equipment is required to monitor the temperature of the substance as well as~~nd~~ (the air) in the oven. This equipment should be protected from fire and explosion. For substances with an SADT or SAPT below ambient temperature, the test should be performed with sufficient cooling~~in a cooling chamber or solid carbon dioxide should be used for cooling the oven~~.

28.4.2.3 *Procedure*

28.4.2.3.1 ~~Calibration~~Validation procedures

 (a) The ~~calibration~~validation procedure A is as follows:

(i) Fill the Dewar vessel with a suitable inorganic salt, preferably with similar physical properties to the test substance (e.g. sodium chloride or dense soda ash). Alternatively, an oil of known specific heat capacity at the temperature of interest (e.g. silicone oil, apparent density 0.96 ± 0.02 at 20 °C and heat capacity 1.46 ± 0.02 J/g at 25 °C) may be used ~~or another suitable oil and place it in the vessel holder of the AST oven~~;

(ii) Place the Dewar vessel in the vessel holder of the oven and h~~H~~eat the ~~sample calibration~~ validation substance in 20 °C steps using the internal heating system at a known power rating~~,~~ (e.g. 0.333 W or 1.000 W)~~,~~ and determine the heat losses at 40 °C, 60 °C, 80 °C and 100 °C;

(iii) Use the data to determine the heat capacity of the Dewar vessel and the test set-up using the method given in 28.4.2.4.

 (b) The validation procedure B is as follows:

(i) In order to perform validation procedure B the test set-up should be well characterized (e.g. by performing validation procedure A first);

(ii) The test set-up must be validated using the method described in 28.4.2.4 with at least two standard substances or mixtures. Suitable choices for these standards are dicumylperoxide in ethylbenzene[[6]](#footnote-7) (40:60% w/w, SADT for a heat loss of 60 mW/K.kg should be 90 °C), or any of the substances from the example of results tables in chapter 28.

 28.4.2.3.2 Test procedure

 The test procedure is as follows:

 (a) Fill the Dewar vessel/test cell with the weighed sample, including a representative amount of packaging material (if ~~metal~~necessary), and place it in the vessel holder of the ~~AST~~oven;

 (b) Start the temperature monitoring and then increase the sample temperature ~~using the internal heater~~ to a pre-set temperature at which detectable self-heating may occur. The specific heat of the substance can either be calculated from the temperature rise, heating time and heating power, or be determined by any suitable calorimetric test method beforehand;

 (c) ~~Stop the internal heating~~Heat the sample to the set temperature, maintain the oven temperature and monitor the sample temperature. If no temperature rise due to self-heating is observed ~~within 24 hours~~after temperature equilibration of the system (e.g. 24 h for the open system), increase the oven temperature by 5 °C. Repeat this procedure until self-heating is detected;

 For the closed version: The apparatus can be heated with < 0.5 W/kg until self-heating is detected. The heating power per unit mass should remain below the sensitivity for self-heating detection of the test equipment or autoclave;

1. When self-heating is detected, the sample is allowed to heat up under adiabatic conditions to a pre-settemperature, ~~where the rate of heat generation is less than the cooling capacity, upon~~ at which point the cooling system is activated or the oven temperature has reached its limit;

For the open version this temperature should be set so that the rate of heat generation does not exceed the cooling capacity of the system;

For the closed version this temperature is usually a pre-set maximum oven temperature. The sample may exceed this temperature under non-adiabatic conditions.

 ~~(e) On cooling, determine the loss in mass, if any, and determine the change in composition (if desired).~~

28.4.2.4 *Test criteria and method of assessing results*

28.4.2.4.1 Calculate the rate of temperature drop, A (~~°C~~K/h), of the Dewar vessel at the different temperatures used in the calibration procedure. Plot a graph through these values in order to allow determination of the rate of temperature drop at any temperature.

28.4.2.4.2 Calculate the heat capacity, H (J/~~°C~~K), of the Dewar vessel using the formula:

 

 where: E1 = power applied to the internal heater (W)

 A = rate of temperature drop at the temperature of calculation (~~°C~~K/h)

 B = slope of the curve on internal heating (~~calibration~~ validation substance) at the temperature of calculation (~~°C~~K/h)

 M1 = mass of ~~calibration~~ validation substance (kg)

 Cp1 = specific heat of ~~calibration~~ validation substance (J/kg.~~°C~~K)

28.4.2.4.3 Determine the heat loss, K (W), using:

 

at each desired temperature and draw a graph through these values.

28.4.2.4.4 Calculate the specific heat, Cp2 (J/K.kg~~°C~~), of the substance using:

 

 where: E2 = power applied to the internal heater (W)

 C = slope of the curve on internal heating (sample) at the temperature of calculation (~~°C~~K/h)

 M2 = mass of sample (kg)

28.4.2.4.5 Calculate the heat generation, QT (W/kg), of the substance at intervals of 5 °C using the following formula for each temperature:

 

 where: D = slope of the curve during the self-heating at the temperature of calculation (~~°C~~K/h)

28.4.2.4.6 Plot the calculated rates of heat generation per unit of mass (QT) as a function of the temperature in a graph with linear scales and draw the best-fit curve through the plotted points. Determine the heat losses per unit of mass, L (W/K.kg~~°C)~~ of the specific package, IBC or tank (see 28.3.5). Draw a straight line of gradient L tangential to the heat generation curve. The intersection of the straight line and the abscissa is the critical ambient temperature, i.e. the highest temperature at which the substance as packaged does not show self-accelerating decomposition. The SADT is the critical ambient temperature (°C) rounded to the next higher multiple of 5 °C. An example is given in Figure 28.4.2.2.

28.4.2.5 *Examples of results*

|  |  |  |  |  |
| --- | --- | --- | --- | --- |
| **Substance** | **Mass****(kg)** | **Packaging** | **Heat loss per unit mass (mW/K.kg~~.K~~)** | **SADT/SAPT****(°C)** |
| Azodicarbonamidea | 30 | 1G | 100 | > 75 |
| tert-Butyl peroxybenzoatea | 25 | 6HG2 | 70 | 55 |
| tert-Butyl peroxy-2-ethylhexanoatea | 25 | 6HG2 | 70 | 40 |
| tert-Butyl peroxypivalatea | 25 | 6HG2 | 70 | 25 |
| N-Vinylformamide | 1000 | 31H1 | 33 | 55 |

a *These historical examples were determined using heat losses that are higher than those currently recommended*

 *for classification purposes (see table 28.4).*

**Figure 28.4.2.1: Adiabatic storage test**



(A) Multi-point recorder and temperature controller (10 mV)

(B) External zero set

(C) Recorder set for greatest accuracy

(D) Control

(E) Relay

(F) Internal pre-heater

**~~Figure 28.4.2.2: Example of determination of SADT or SAPT~~**

~~~~

~~(A) Heat generation curve~~

~~(B) Line with gradient equal to the rate of heat loss and tangential to the heat generation curve~~

~~(C) Critical ambient temperature (intercept of heat loss line with the abscissa)~~

~~(D) Self-accelerating decomposition temperature (SADT) - critical ambient temperature rounded up to next higher multiple of 5 °C~~

~~(X) Temperature~~

~~(Y) Heat flow (generation or loss) per unit mass~~

**Figure 28.4.2.2: Example of determination of SADT or SAPT**



|  |  |  |  |
| --- | --- | --- | --- |
|  |  |  |  |
|  | Heat generation curve |  | Heat loss curve L = 0.06 W/K.kg ~~(Kg K)~~ |
| (A) | Critical ambient temperature (intercept of heat loss with the abscissa) | (B) | SADT or SAPT (Critical ambient temperature rounded up to next higher multiple of 5°C) |

28.4.3 *Test H.3: Isothermal storage test (IST)*

28.4.3.1 *Introduction*

28.4.3.1.1 This method determines the rate of heat generation produced by reacting or decomposing substances as a function of time at constant temperature. The heat generation parameters obtained are used with the heat loss data relating to the package to determine the SADT or SAPT of a substance in its packaging. The method is appropriate for every type of packaging including IBCs and tanks. Some substances may show an increase in the rate of heat generation with increasing decomposition (e.g. due to autocatalysis or induced decomposition). This property is also taken into account by this test method.

~~28.4.3.1.2 Measurements can be performed in the temperature range of -20 °C to 200 °C. Heat generation values can be measured from 5 mW/kg to 5 W/kg. The thermal resistance between the sample holder and aluminium block via the heat flow meters is about 0.1 W/°C. The apparatus is able to measure rates of heat generation from 15 mW/kg to 1 500 mW/kg with a maximum error of 30% at 15 mW/kg and 5% from 100 to 1 500 mW/kg.~~

28.4.3.1.~~3~~.2 The usually robust construction of ~~the~~ readily available apparatus, the relatively small sample size and the well-defined operating conditions enable the test to be performed in an ordinary laboratory. The effects of a thermal explosion, e.g. fragmentation of the sample holder and generation of pressure, should be contained within the apparatus.

28.4.3.2 *Apparatus and materials*

28.4.3.2.1 Isothermal Calorimetry (IC)

Appropriate isothermal calorimeters can be used. The equipment should be able to measure heat generation values of 1 mW/kg to 1500 mW/kg in a temperature range of -20 °C to 200 °C. The maximum error in the heat generation should be less than 5 %. The equipment should be capable of maintaining the temperature to within 0.2 °C of the set temperature. Sample mass of test material should be at least 200 mg. Closed pressure resistant sample holders should be used and the material of the sample holder should not have catalytic effect on the decomposition behaviour of the test substance. This can be achieved by selecting the appropriate materials for the sample holders or by an appropriate passivation method of the sample holders.

~~28.4.3.2.1 The apparatus consists of an air-insulated heat sink (an aluminium block) which is kept at a constant temperature by means of controlled heating. A cryostat is used to maintain temperatures below 40 °C. The heat controller is capable of maintaining the temperature to within 0.2 °C of the set temperature. The temperature of the heat sink is measured with a platinum-resistance sensor. The two holes drilled in the block contain heat flow meters (e.g. Peltier elements). A schematic drawing of the IST is given in Figure 28.4.3.1. For substances with an SADT or SAPT below ambient temperature, the test should be performed in a cooling chamber or solid carbon dioxide should be used for cooling the oven.~~

28.4.3.2.2 The sample ~~H~~holder~~s are~~ is placed on or around ~~both~~ heat flow meter~~s~~. ~~: one with the sample and one with an inert substance. Both holders are identical and have a volume of 70 cm~~~~3~~~~.~~ The amount of substance in ~~each~~ the sample holder is at least 200 mg ~~about 20 g~~. The material of the holder~~s~~ should be compatible with the sample. ~~are made of glass or stainless steel. The steel must be compatible with the test substance.~~ If an external reference is used it should be handled identical to the sample. ~~When using a glass holder, it is provided with a long capillary tube which prevents pressure build-up in the holder and evaporation of the sample.~~

28.4.3.2.3 The ~~voltage difference resulting from the difference in~~ heat flow from the sample ~~holder to the heat sink and inert substance holder to the heat sink~~ is continuously recorded as a function of time (differential measurement) by a recorder or computer.

28.4.3.3 *Procedure*

28.4.3.3.1 Calibration procedure

 Before a measurement can be performed, the blank signal and the sensitivity of the heat flow meter need to be determined by the ~~following~~ applicable calibration procedure for the equipment used covering the temperature range of the measurement.

 ~~(a) Set the IST at the selected test temperature;~~

 ~~(b) Insert a heating coil in the sample holder. Fill the sample and reference holders with inert material (e.g. sodium chloride or milled glass beads) ensuring that the heating coil is completely covered with material. Place both holders in the IST;~~

 ~~(c) Determine the blank signal (the output of the recorder when no electric power is applied to the heating coil);~~

 ~~(d) Determine the sensitivity of the heat flow meter using two or three different electrical heating powers which lie within the expected range of heat generation of the sample to be tested.~~

28.4.3.3.2 Test procedure

 The test procedure is as follows:

 (a) Set the ~~IST~~ apparatus for the desired test temperature.~~;~~ The temperature selected should be sufficient to give a rate of heat generation between 5 mW to 1000 mW per kg of substance or for tanks a maximum heat generation rate between 1 and 100 mW/kg;

 (b) Fill the sample holder with the weighed sample and with a representative quantity of packaging material (if metal) and insert the holder into the apparatus~~.~~; ~~The amount of sample should be sufficient to give a rate of heat generation between 5 mW to 1 500 mW per kg of substance;~~

 (c) Start monitoring the rate of heat production. ~~The results from the first 12 hours of the test should not be used because this period is required for temperature equilibration.~~ The duration of each test depends on the test temperature and on the rate of heat production. ~~The test should last for at least 24 hours after the 12 hour equilibration period but can then be stopped if the rate of heat generation is falling from the maximum or if the rate of heat generation is greater than 1.5 W/kg.~~ The measuring time as given in Figure 28.4.3.1 can be used as guidance unless it leads to unrealistic measuring times (e.g. greater than 1000 hours). These measuring times are given to achieve a certain degree of conversion of the substance in order to take auto-catalytic effects into account;[[7]](#footnote-8)

 (d) At the end of the test the change in sample mass should be determined;

 (e) The test is repeated with new samples at temperature intervals of 5 °C so that there are at least five ~~seven~~ results with a maximum heat generation rate between ~~1~~5 and 1~~5~~000 mW/kg or for tanks a maximum heat generation rate between 1 – 100 mW/kg.

Figure 28.4.3.1: Measuring period as a function of maximum heat generation measured

28.4.3.4 *Test criteria and method of assessing results*

~~28.4.3.4.1 Calculate the sensitivity, S (mW/mV), of the instrument at the various electrical powers used in the calibration procedure using the following formula:~~

~~~~

 ~~where: P = electric power (mW)~~

 ~~U~~~~d~~ ~~= dummy signal (mV)~~

 ~~U~~~~b~~ ~~= blank signal (mV)~~

~~28.4.3.4.2 Use these values and the test data to calculate the maximum rate of heat generation, Q (mW/kg), at the different test temperatures using the formula:~~

~~~~

 ~~where: U~~~~s~~ ~~= sample signal (mV)~~

 ~~M = mass (kg)~~

28.4.3.4.1~~3~~ Plot the calculated maximum rate of heat generation per unit of mass as a function of the test temperature on a graph with linear scales and draw the best-fit curve through the plotted points. Determine the heat losses per unit of mass, L (W/K.kg~~°C~~) of the specific package, IBC or tank (see 28.3.5). Draw a straight line of gradient L tangential to the heat generation curve. The intersection of the straight line and the abscissa is the critical ambient temperature i.e. the highest temperature at which the substance as packaged does not show self-accelerating decomposition. The SADT or SAPT is the critical ambient temperature (°C) rounded to the next higher multiple of 5 °C. An example is given in Figure 28.4.3.2.

28.4.3.5 *Examples of results*

|  |  |  |  |  |
| --- | --- | --- | --- | --- |
| **Substance** | **Mass****(kg)** | **Packaging** | **Heat loss per unit mass (mW/K.kg~~.K~~)** |  **SADT/ SAPT****(°C)** |
| Azodicarbonamidea | 30 | 1G | 100 | > 75 |
| tert-Butyl peroxybenzoatea | 25 | 6HG2 | 70 | 55 |
| tert-Butyl peroxy-2-ethylhexanoatea | 25 | 6HG2 | 70 | 40 |
| tert-Butyl peroxypivalatea | 25 | 6HG2 | 70 | 25 |
| 2,5-Diethoxy-4-morpholinobenzenediazonium zinc chloride (90%)a | 25 | 1G | 150 | 45 |
| 2,5-Diethoxy-4-morpholinobenzenediazonium tetrafluoroborate (97%)a | 25 | 1G | 15 | 55 |
| 2,5-Diethoxy-4-(phenylsulphonyl)- benzenediazonium zinc chloride (67%)a | 25 | 1G | 15 | 50 |
| 2-(N-ethoxycarbonyl-N-phenylamino)-3- methoxy-4-(N-methyl-N-cyclohexylamino)-  benzenediazonium zinc chloride (62%)a | 25 | 1G | 15 | 45 |
| 3-Methyl-4-(pyrrolidin-1-yl) benzenediazoniumtetrafluoroborate (95%)a | 25 | 1G | 15 | 55 |
| Cumyl peroxyneodecanoate (75%) | 25 | 3H1 | 40 | 10 |
| tert-Butyl peroxyneodecanoate | 25 | 3H1 | 40 | 15 |
| N-Vinylformamide | 1000 | 31H1 | 33 | 55 |

**a** *These historical examples were determined using heat losses that are higher than those currently recommended*

 *for classification purposes (see table 28.4).*

**~~Figure 28.4.3.1: Isothermal storage test~~**

~~~~

~~(A) Platinum resistance thermometer (B) Sample vessel~~

~~(C) Cylindrical holder (D) Air spaces~~

~~(E) Sample (F) Inert material~~

~~(G) Platinum resistance sensor (H) Platinum resistance sensor~~

 ~~for temperature control for safety control~~

~~(J) Peltier elements (K) Aluminium block~~

~~(L) Electric circuit (M) Air space~~

~~(N) Heating wires (O) Amplifier~~

~~(P) Recorder (Q) Temperature controller~~

~~(R) Glass wool~~

**~~Figure 28.4.3.2: Example of determination of SADT or SAPT~~**

~~~~

~~(A) Heat generation curve~~

~~(B) Line with gradient equal to the rate of heat loss and tangential to the heat generation curve~~

~~(C) Critical ambient temperature (intercept of heat loss line with the abscissa)~~

~~(D) SADT or SAPT - critical ambient temperature rounded up to next higher multiple of 5 °C~~

~~(X) Temperature~~

~~(Y) Heat flow (generation or loss) per unit mass~~

**Figure 28.4.3.2: Example of determination of SADT or SAPT**



|  |  |  |  |
| --- | --- | --- | --- |
|  |  |  |  |
|  | Heat generation curve |  | Heat loss curve L = 0.06 (W/K.kg) |
| (A) | Critical ambient temperature (intercept of heat loss with the abscissa) | (B) | SADT or SAPT (Critical ambient temperature rounded up to next higher multiple of 5°C) |

28.4.4 *Test H.4: Heat accumulation storage test*

28.4.4.1 *Introduction*

28.4.4.1.1 This method determines the minimum constant air environment temperature at which thermally unstable substances undergo exothermic decomposition or polymerization at conditions representative of the substance when packaged. The method is based on the Semenov theory of thermal explosion i.e. the main resistance to heat flow is considered to be at the vessel walls. The method can be used for the determination of the SADT or SAPT of a liquid substance in its packaging, including IBCs and small tanks (up to 2 m3) as well as for a solid substance in its packaging up to 50 kg.

28.4.4.1.2 The effectiveness of the method depends on selecting a Dewar vessel with heat loss per unit mass characteristics similar to the package.

28.4.4.2 *Apparatus and materials*

28.4.4.2.1 The experimental equipment consists of a suitable test chamber, appropriate Dewar vessels with closures, temperature probes and measuring equipment.

28.4.4.2.2 ***The test should be performed in a test chamber ~~cell~~ capable of withstanding fire and overpressure and, preferably, should be fitted with a pressure relief system e.g. a blowout panel.*** The recording system should be housed in a separate observation area.

28.4.4.2.3 For tests at temperatures up to 75 °C, a double walled metal chamber (ca. 250 mm inner diameter, 320 mm outer diameter and 480 mm high made from 1.5 mm to 2.0 mm thick stainless-steel sheet) ~~is~~ can be used with fluid from a temperature-controlled circulating bath passed between the walls at the desired temperature. The test chamber is loosely closed by an insulated lid (e.g. made from 10 mm thick polyvinyl chloride). The air temperature in the double walled metal chamber should be controlled ~~should allow~~ so that the desired temperature for a liquid inert sample in the Dewar vessel to be maintained with a deviation of not more than ± 1 °C for up to 10 days. The air temperature in the double walled metal chamber and the sample temperature in the Dewar should be measured and recorded.

28.4.4.2.4 Alternatively, and particularly for tests at temperatures above 75 °C, a thermostatically controlled drying oven (which may be fan-assisted) large enough to allow air circulation on all sides of the Dewar vessel may be used. The air temperature in the oven should be controlled so that the desired temperature for a liquid inert sample in the Dewar vessel can be maintained with a deviation of not more than ± 1 °C for up to 10 days. The air temperature in the oven and the sample temperature in the Dewar should be measured and recorded. It is recommended that the door of the oven be fitted with a magnetic catch or replaced by a loosely fitting insulated cover. The oven may be protected by an appropriate steel liner and the Dewar vessel housed in a wire mesh cage.

28.4.4.2.5 For tests at sub-ambient temperatures, a double walled chamber (e.g. a freezer) of suitable size provided with a loose door or lid (e.g. with a magnetic closure) may be used. The temperature of the air in the chamber should be controlled to ± 1 °C of the set temperature. The air temperature in the chamber and the sample temperature in the Dewar should be measured and recorded.

28.4.4.2.6 Dewar vessels, with their closure system, are used with heat loss characteristics which are representative of the maximum ~~size of~~ package size under investigation (see also table 28.4). The closure of the Dewar vessel should be made of inert material. Particularly for solids, cork or rubber bungs may be used. An example of a ~~A~~ closure system for use with liquids with low or medium volatility and wetted solids is illustrated in Figure 28.4.4.1. Samples which are highly volatile at the test temperature should be tested in a pressure-tight ~~metal~~ vessel that is made of a material compatible with the sample and fitted with a pressure relief valve. The pressure vessel is placed in the Dewar vessel and the effect of the heat capacity of the metal vessel taken into account by calculation.

28.4.4.2.7 The heat loss characteristics of the system used, i.e. Dewar vessel and closure, should be established (see 28.3.6) prior to performance of the test. Small adjustments to the heat loss characteristics of the Dewar can be achieved by varying the closure system. ~~Since the closure system has a significant effect on the heat loss characteristics, these can be adjusted to some extent by varying the closure system.~~ In order to achieve the required level of sensitivity, Dewar vessels with sample capacities below 0.~~5~~3 litres should not be used.

28.4.4.2.8 Dewar vessels with a volume of 300 – 500 ml, filled at 80% ~~with 400 ml of~~ with a liquid substance with a heat loss as given in Table 28.4 should be used. For larger packagings, IBCs or small tanks larger Dewar vessels with lower heat loss per unit of mass should be used (see Table 28.4). ~~of 80 to 100 less than or equal to 40 mW/K.kgK are normally suitable for representing a 225 l 50 kg package with a liquid substance.~~ For larger packages, IBCs or small tanks larger Dewar vessels with lower heat losses per unit mass should be used. ~~For example, spherical 1 litre Dewar vessels having heat loss characteristics in the range 16 to 34 mW/kg.K may be suitable for IBCs and small tanks.~~

28.4.4.3 *Procedure*

28.4.4.3.1 Set the test chamber at the selected storage temperature. Fill the Dewar vessel, to 80% of its capacity, with the substance to be tested ~~under test~~ and note the mass of the sample. Solids should be moderately compressed. In case of cylindrical Dewar vessel ~~Insert~~ the temperature probe is inserted centrally 1/3 of the inner height of the Dewar from the bottom of the Dewar vessel.  ~~into the centre of the sample.~~ Seal the lid of the Dewar in place and insert the Dewar vessel in the test chamber, connect the temperature recording system and close the test chamber.

28.4.4.3.2 The sample is heated and the temperature of the sample and test chamber continuously monitored. The time is noted at which the sample temperature reaches a temperature 2 °C below the test chamber temperature. The test is then continued for a further seven days or until the sample temperature rises to 6 °C or more above the test chamber temperature whichever  ~~if this~~ occurs sooner. ~~Note the time taken for the sample to rise from 2 °C below the test chamber temperature to its maximum temperature.~~

28.4.4.3.3 If the sample survives, cool and remove it from the test chamber and carefully dispose of it as soon as possible. The percentage mass loss and change in composition may be determined.

28.4.4.3.4 ~~Repeat the test with fresh samples, varying the storage temperature in 5 °C steps~~. If the substance is being tested to determine if temperature control is necessary, perform sufficient tests, in steps of 5 °C using fresh samples, to determine the SADT or SAPT to the nearest 5 °C or to determine if the SADT or SAPT is ~~greater than or~~ equal to or less than the applicable temperature specified in Table 28.2 ~~60 °C~~.

If the substance is being tested to determine if it meets the SADT criterion for a self-reactive substance, perform sufficient tests to determine if the SADT for a 50 kg package is 75 °C or less. If the substance is being tested to determine if it meets the SAPT criterion for a polymerizing substance, perform sufficient tests to determine if the SAPT in the packaging as used is 75 °C or less.

28.4.4.4 *Test criteria and method of assessing results*

28.4.4.4.1 The SADT or SAPT is reported as the lowest chamber temperature at which the sample temperature ~~undergoes~~ exceeds the ~~test~~ chamber temperature by 6 °C or more within the seven days testing time frame (see 28.4.4.3.2). If the sample temperature does not exceed the ~~test~~ chamber temperature by 6 °C or more in any test, the SADT or SAPT is recorded as being greater than the highest storage temperature used.

28.4.4.5 *Examples of results*

|  |  |  |  |
| --- | --- | --- | --- |
| **Substance** | **Sample mass (kg)** | **Dewar heat loss (mW/K.kg~~.K~~)** | **SADT/SAPT****(°C)** |
| Azodicarbonamidec | 0.28 | 74 | > 75 |
| Azodicarbonamide, 90% with 10% activatorc | 0.21 | 70 | 55 |
| 2,2'-Azodi(isobutyronitrile) | ~~0.18~~ 0.28 | ~~62~~ 27 | 50 |
| Benzene-1,3-disulphohydrazide, 50%c | 0.52 | 81 | 70 |
| tert-Butyl hydroperoxide, 80% with 12% di-tert-butyl peroxidec | 0.30 | 72 | 100**a** |
| tert-Butyl peroxyneodecanoate, 40%c | 0.42 | 65 | 25 |
| tert-Butyl peroxy-3,5,5-trimethylhexanoatec | 0.38 | 79 | 60 |
| Dibenzoyl peroxide, 50%c | 0.25 | 91 | 60 |
| Di-(4-tert-butylcyclohexyl) peroxydicarbonatec | 0.19 | 79 | 45 |
| 2,2-Di-(tert-butylperoxy)butane, 50%c | 0.31 | 88 | 80 |
| Di-(2-ethylhexyl) peroxydicarbonatec | 0.39 | 64 | 0 |
| 2,5-Diethoxy-4-morpholinobenzenediazonium  zinc chloride (66%)c | 0.25 | 58 | 45 |
| Diisotridecyl peroxydicarbonatec | 0.38 | 80 | 10 |
| Peroxyacetic acid, 15%, with 14% hydrogen peroxide  (type F)c | 1.00 | 33 | > 50**b** |
| Dilauroyl peroxide, technically pure | 0.16 | 26 | 50 |
| Didecanoyl peroxide technically pure | 0.20 | 28 | 40 |
| N-Vinylformamide | 0.40 | 33 | 55 |  |

**a** *In a pressure vessel contained with a 2 litres Dewar vessel.*

**b** *In a spherical, 1 litre Dewar vessel.*

**c** *These historical examples were determined using heat losses that are higher than those currently recommended for classification purposes (see table 28.4).*

**Figure 28.4.4.1: Dewar vessel with closure for testing liquids and wetted solids**



(A) PTFE capillary tube (B) Special screw fittings (PTFE or Al) with O-ring seal

(C) Metal strip (D) Glass lid

(E) Glass beaker base (F) Spring

(G) Glass protective tube (H) Dewar vessel

(J) Steel retaining device

**Section 33.2.4 (Test N.1)** – Replace inset A of Figure 33.2.4.1 with the figure shown below:



*Source: ST/SG/AC.10/C.3/2020/34, Para. 6 (Option 2) and Para. 9 of this report.*

1.

**Section 33.2.4.2** – In the first sentence, add the word “inner” immediately before “width 20 mm) as shown below:

A mould 250 mm long with a triangular cross-section of inner height 10 mm and inner width 20 mm is used to form the train for the burning rate test.

*Source: Para. 9 of this report.*

**Section 11.4.1.2.1 (French version)** – In the French version of the MTC, correct the reference to “40 ± 2 mm” to read “48 ± 2 mm”

*Source:* [*ST/SG/AC.10/C.3/2020/17*](file:///H%3A%5CUN%5Cundocs%5CAC10%5CC3%5C2020%5C57th%20Session%5CST-SG-AC10-C3-2020-17e.docx)*, Para. 5 and Para. 10 of this report.*

1. ST/SG/AC.10/C.3/113/Add.1/Rev.1 [↑](#footnote-ref-2)
2. GHS document referred to the EWG by TDG INF.23 [↑](#footnote-ref-3)
3. Status report of the review. Not discussed by the EWG. [↑](#footnote-ref-4)
4. Refers GHS 2020/18 to EWB for review [↑](#footnote-ref-5)
5. *References: N.N. Semenov, Z. Physik, 48, 1928, 571; D.A. Frank-Kamenetskii, Zhur. Fiz. Khim., 13, 1939, 738.; P.H. Thomas, Trans. Faraday Soc., 54, 1958, 60.* [↑](#footnote-ref-6)
6. *Reference: Dürrstein S., Kappler C., Neuhaus I., Malow M., Michael-Schulz H., Gödde M., 2016, Modell-based prediction of the adiabatic induction period and SADT of dicumyl peroxide solution and comparison large-scale experiments performed using 216.5-liter barrels in the H.1 test, Chemical Engineering Transactions, 48, 475-480.* [↑](#footnote-ref-7)
7. *References :*

1 *J. L. C. van Geel, Investigations into Self-Ignition Hazard of Nitrate Ester Propellants, Thesis, Technical University of Delft, The Netherlands, 1969.*

2 *Barendregt, R.B., Thermal Investigation of Unstable Substances, Including a Comparison of Different Thermal Analytical Techniques, Thesis, Technical University of Delft, The Netherlands, 1981.* [↑](#footnote-ref-8)