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Working Party on the Transport of Dangerous Goods

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Item 2 of the provisional agenda

REPORTS OF INFORMAL WORKING GROUPS

Informal Working Group "EN 15507"

Transmitted by the European Committee for Standardisation (CEN) on behalf
of the Working Group^{1,2}

Background

1. Reference is made to the following paragraphs of the reports of the last two sessions of the Joint Meeting and to the related information papers:

- (a) Paragraph 14 of ECE/TRANS/WP.15/AC.1/112 - Report of the session held in Geneva from 15 to 18 September 2008 together with informal document INF.11

¹ In accordance with the programme of work of the Inland Transport Committee for 2006-2010 (ECE/TRANS/166/Add.1, programme activity 02.7 (c)).

² Circulated by the Intergovernmental Organisation for International Carriage by Rail (OTIF) under the symbol OTIF/RID/RC/2009/45.

from the European Plastics Converters (EuPC), informal document INF.12 from CEN and informal document INF.19 from Belgium.

- (b) Paragraphs 7 and 8 of ECE/TRANS/WP.15/AC.1/114 - Report of the session held in Geneva from 15 to 18 March 2009 together with informal documents INF.31 from Belgium and INF.32 from EuPC.

2. These paragraphs and documents reflect a possible reference in ADR/RID to the European standard EN 15507:2008 Packaging — Transport packaging for dangerous goods — Comparative material testing of polyethylene grades, which had been at the formal vote stage in September 2008 and been published in March 2009.

3. A reference to EN 15507:2008 in subsections 6.1.5.2.5 and 6.5.6.3.4 as suggested by EuPC could not be agreed upon following the concerns expressed by several delegates. It was decided that an intersessional informal working group should address these concerns, hosted by EuPC and based upon detailed descriptions of the concerns in writing.

Information

4. EN 15507:2008 is aimed at providing requirements and test procedures for comparative testing of grades of high and medium molecular weight high density polyethylene (HDPE), used for the manufacture of plastics packagings and IBCs for the transport of dangerous goods.

The use of these test methods facilitate usage in selective testing procedures, eliminating or reducing the requirement to carry out the full design type test procedures each time a new grade of high density polyethylene is used for the manufacture of the same design type.

The need for the application of such a method increased during the past years as the manufacturers of high-molecular HDPE, due to reorganizations, mergers, takeovers, took several HDPE-types from the market so that alternatives have to be found at short notice. The application of the test method as described in EN 15507:2008 helped medium-sized manufacturers of plastics packaging for dangerous goods to run tests for the equivalence of HDPE-types at reasonable expenditure.

5. The tests described in EN 15507:2008 are essentially the same as the “laboratory methods” described in the “Requirements for the testing of plastic receptacles” published as an informal annex to RID.

However, whereas the RID laboratory methods compare the chemical impact of different substances (standard liquids or filling substances) on one plastics resin, EN 15507:2008 compares the chemical effect of a single substance on different resins (original design type resin and an alternative resin).

6. Compatibility testing is the most time consuming a costly element of the design type test regime for plastics packagings and IBC’s. Options to reduce these efforts are the driving force behind the establishment of EN 15507.

7. With respect to the compatibility testing of plastics packagings and IBC's, ADR/RID include, in addition the pre-storage of samples over 6 month and the accelerated pre-storage at elevated temperature in combination with the laboratory tests equivalent procedures recognized by the competent authority (6.1.5.2.5, last para. and 6.5.6.3.4).

Such procedures have been approved in some European countries and have triggered the evolution of EN 15507. However, European market needs require a multinational mutual acceptance of such procedures, which would be achieved by a reference of EN 15507 in ADR/RID.

8. Prior to the official start of the standardization work, EuPC had taken the initiative, together with APME (the former Association of Plastics Materials Manufacturers in Europe) in 1997 to promote the idea of a procedure to facilitate a change of plastics resins within the cover of existing type approvals. A series of eleven meetings of experts from plastics manufacturers and converters were organized over three years, some of them also involving representatives from competent authorities and test houses from Austria, Belgium, France, Germany, Netherlands and Norway.

9. Work on EN 15507 started in June 2001 on a first draft entitled:

'Packaging – Transport Packaging for dangerous goods – high density polyethylene materials for the manufacture of packagings and IBCs – material parameters and test procedures for comparative testing, for use in selective testing for packagings'.

It led to seven further drafts discussed during approximately 4 years. A Work Item No. was assigned in November 2002.

The target group (TG) "Plastics packaging" of CEN/TC 261/SC 5/WG 16 "Packaging of dangerous goods" which had established the two compatibility test standards EN 16101:2004 Packaging - Transport packaging for dangerous goods - Plastics compatibility testing (ISO 16101:2004) and EN 23667:2007 Packaging - Transport packaging for dangerous goods - Rigid plastics and plastics composite IBCs - Compatibility testing (ISO 23667:2007) was charged to develop the standard. This group was constituted of experts from Austria, Belgium, France, Germany, Netherlands and United Kingdom, representing test houses, resin manufacturers, packagings manufacturers and competent authorities.

10. The discussion on the method for assessing material degradation after immersion in nitric acid, in order to develop a satisfactory procedure with adequate reproducibility was hindered by changes in personnel at test laboratories and within the polyethylene raw materials industry due to mergers leading to reorganisation and was reason for protracted discussion. As a result the TG was unable to produce a Committee Draft for submission to the CEN Working Group (WG16) in the required time frame under new rules that had in the meantime been introduced by CEN.

11. However, progress had been made with the standard during this period including the a new title:

‘Packaging – Transport Packaging for dangerous goods – Comparative material testing of polyethylene grades’.

Following further development of the Molecular Degradation method with particular input from OTIF, Austria and BAM, Germany, it was felt that an improved procedure had been developed. Results to this method were discussed at TG meetings in March 2004 and April 2005 including an extensive report from BAM on experience with the German National Comparative testing procedure GGR003 which showed that the procedure was largely working successfully but there were still issues with the molecular degradation test. In order to prove the inter laboratory reproducibility, a Round Robin test was proposed between laboratories that had experience with this procedure. The issue of reproducibility of the Molecular Degradation method, which is still a discussion point, was provisionally resolved by specifying a larger number of test samples in this test procedure.

12. During this stage a new work Item No was applied for with CEN and granted in January 2006. It was then possible to submit a Committee draft to the WG16 and the standard was then progressed through the normal stages of Enquiry ending in August 2006. Following this the results of the Enquiry were returned to the TG, and new drafts were produced and discussed at further TG meetings incorporating the agreed changes from the Enquiry and further editorial changes from the CEN Secretariat, finally progressing to the WG16 in February 2008 as provisional standard prEN15507, which was sent out by CEN for Formal Vote from August-October 2008, resulting in acceptance and publication as a CEN standard in December 2008. A majority of 19 members against 3 negative votes and 4 XX abstaining votes was in favour of the standard.

Results of the meeting

13. On invitation by EuPC the working group met in Berlin on 10 June 2009 with participation from Austria, Belgium, France, Germany, Netherlands, Switzerland and United Kingdom. As required by the Joint Meeting, the meeting was dedicated to the concerns submitted in writing before the meeting. Full coverage on invitation, agenda, attendance, submitted documents and presentations and detailed discussion results will be compiled in an INF. document which will be provided before the September session of the Joint Meeting.

The meeting was chaired by the secretary of the CEN Target Group, Geoff Law, United Kingdom.

14. Documents with detailed description and reasoning of concerns about validity and reproducibility had been submitted by

- Belgium.
- Netherlands

and distributed to the participants.

15. To allow for a fruitful discussion at the workshop, BAM distributed questions in writing to the participants on 4 June, which had arisen during the study of the Belgium paper.

16. At the meeting contributions and presentations were given on:
- (a) Reasons and aims of the workshop by the CEN Consultant Transport of Dangerous Goods,
 - (b) A review of the standardization work on EN 15507 by the secretary of the Target Group of CEN/TC261/SC5/WG6 in charge of this work item; and
 - (c) Explanations on the conditions for the application, background principles and test procedures by the representative of BAM (Federal Institute for Materials Research and Testing).

17. The expert from Belgium highlighted his concerns based on the submitted document (“Belgian comments on EN 15507 based on tests effectuated by the Belgian Packaging Institute in the period 2007 – 2008”, dated 20-5-2009). This report summarizes the results of tests with jerricans of 140 different design types and producers. Most of them had been approved before based on resins which are supposed to be no longer available in the future and which had to be replaced by similar ones.

All samples manufactured with a new resin, comparable to the original ones (in terms of melt flow rate and density within the limits set by EN 15507) were initially retested positively with water and for most of them, as only a part of the material properties required by EN 15507 (NIS, FNCT and Ox) were available, with standard liquids for the additional and relevant tests as described in the EN15507 (taking in mind the worst case scenario that these material property values would be less favourable as for the old grades) with the standard liquid nitric acid. These test results would have led in relation with EN15507 to an extension of the design type approvals.

Following the EN 15507 tests additional full size tests or additional remaining tests were then performed after pre- storage with the standard liquids wetting agent, mixture of hydrocarbons and nitric acid (55%), as relevant due to the original type approvals based on the former resin. This led to a number of negative results.

The report comes to the following final conclusion:

*Based on the EN 15507 (selective testing), the selected 19 items (Annex 1) would have passed successfully.
Additional testing on the packaging’s in accordance with the usual type approval tests, not required by EN 15507, resulted in a lot of failures, leading to the refusal of certification or reduction in authorisation for all 19 items.*

18. The expert from the Netherlands explained his paper “Position paper of the Netherlands” which emphasizes that the test procedures should include a prototype testing with stress cracking agent. Furthermore he would welcome measures capable of ensuring a constant material quality in terms of properties relevant to the performance of plastics packagings.

The expert from the Netherlands informed that EN 15507 has not been applied in the Netherlands, so far. There has not been a need to apply the standard in The Netherlands as,

comparable to other European countries, a selective testing procedure has been applied when a manufacturer wants to change the material for a number of design types. This selective testing procedure restricts the testing with standard liquids considerably compared to what would be required by testing all design types involved and concerns critical design types in relation to specific standard liquids. This procedure is deemed to be more informative and not more costly than testing all design types involved with the standard liquid water, as required by the standard EN 15507.

As the paper did not document any failures in test procedures according to EN 15507 it was agreed to concentrate on the paper from Belgium.

19. A series of presentations followed deemed to give an overview on the latest state of knowledge on the subject. Some participants pointed out that an advance information about these presentation would have enhanced its understanding:

- (a) The representative of BAM summarized the results of completed investigations in the framework of her PhD- performed in BAM on the relationship of material parameters of polyethylene grades as used in EN 15507 and results of design type tests after pre- storage with standard liquids. Based on test samples (jerricans) of a single design type, manufactured by a single manufacturer on the same machine and the same batch of plastics material on one hand and the preparation of material samples of the same batch in a single laboratory on the other hand, the relationship of parameters and test results was shown. The main conclusions of this work were summarized as follows:

“The following material parameters of different polyethylene grades are in a systematic relationship to each other:

- N.I.S. at -30°C - marginal drop heights of design types in drop tests at – 18 °C,
- FNCT - times to failure of design types in stacking tests with wetting solution,
- Ox - marginal drop heights of design types in drop tests at -18 °C after conditioning with 55 % nitric acid and
- Ox - times to failure of design types in stacking tests with 55 % nitric acid.”

In the limited time available she concentrated on the presentation of the relationship between the FNCT-values with the times to failure of stacking tests with wetting agent (5%) saturated with n-Butylacetate as well as with nitric acid (55%);

- (b) The representative of the plastics materials manufacturer SABIC gave a report on the positive experience of his company with the application of EN 15507 and the German national rule GGR 003 describing the same procedures. It plays an important role in the development and marketing of new PE grades.

Based on initial test results according to EN 15507 the experiences with design type tests were very positive.

The unequal attitude of competent authorities with regard to the application of EN 15507 is seen as a considerable obstacle for his company with European and global marketing perspectives. The EN 15507 norm reduces complexity, costs in the value chain, valuable time and creates flexibility in the market.

He further expresses his expectations that the procedures in EN 15507 are further developed to possibly remove the requirements for an execution of the comparative tests in a single test house and the re-measurement of already available representative data measured according EN 15507;

- (c) The representative of the plastics packaging manufacturer Mauser demonstrated the condensed experience and collated results of comparative testing of his company about the relationship of material data and the results of the performance tests, both, for drum- and IBC grades of polyethylene. Since years, the procedures of EN 15507 (and of the German national rule GGR 003) have proven to be a reliable tool in packaging development and marketing.

Discussion

20. Resuming the presentations given so far, a discussion was started to find explanations for the diverging findings and test results. The following questions were identified to arrive at reasoned arguments for these differences. Basically, they are essentially the same as dispatched before the meeting (see para.15 above):

- (a) EN 15507 requires that the selection of alternative resins shall be based on a comparison of measured MFR-values “in the middle of the specified range in the supply specification for the grade” (Clause 5.1, 2nd para.). What MFR-values were used for the comparison?

This question has relevance with respect to the general suitability of an alternative resin. A margin of $\pm 30\%$ is set in clause 4.2.

- (b) The design type tests are documented for the new grades only. Have the tests with samples made from the original resin been tested by the same test house and same conditions (water temperature of hydraulic test, same wetting agent data)?

For valid invalidation of the standards procedures would need to include concurrent design type tests with samples from the old resin.

- (c) Have the test samples from the old and new resin been manufactured at the same site and with the same detailed specification (diameter and type of closure, tare mass etc)?

It is general experience that even slight variations in tare mass or modifications of the closure geometry may have great impact on test results.

21. Questions (a) and (b) initiated a discussion on the stability and limits of plastics materials specifications over time and the difficulty to clearly relate results of prototype performance tests to either the chemical impact of the pre- storage with standard liquids or to variations in material properties in terms of melt flow rate and density.

The expert from Belgium confirmed that the comparison of old and new grades is based of tests with prototypes manufactured in periods over 1-2 years, with the consequence that the material batches were of the specifications (within the limits) as valid at the time of manufacture. This was seen as a non-compliance with the rules of EN 15507 mentioned under a) above.

There was a general agreement that the performance of plastics packagings under chemical impact is not only determined by the properties of the plastics materials but also by the processing parameters (blow- moulding, i.e.), details in design (wall thickness and - distribution, neck- area), preparation of the test samples (closure torque, target hardness, temperature level, pressure increase rate) and that it is therefore difficult to relate negative test results to either of these parameters.

22. With respect to question (c) it was confirmed that design and manufacturing process of packagings made from old and new resin had been the same. The importance and relevance of this response was seen in context with the tolerances for production controls, for which the Belgian authority has set a limit of $\pm 4\%$ in tare mass of the plastics packagings, as an example and the level achieved in minimizing the material needed for production.

23. The discussion was additionally concerned with options to improve the standard aimed at further minimizing testing efforts, as indicated in the Dutch paper. In particular, it was hoped that the strong limitations in the standard which require a proof per design type and per manufacturer could be removed to arrive finally at ranking lists generally applicable for plastics materials used for dangerous goods packagings. There is thus a need to improve the application of the standard further, also in relation to the variations in resin properties.

Conclusion

24. With the agreement of all authors it was confirmed that all relevant documents and presentations distributed and given during the meeting would be compiled and made available to the participants of the meeting. After consultations with BAM, these data will be available on the following internet page:

http://www.tes.bam.de/gremien/gefahrguttransport/documents_WG_EN_15507.htm

It was agreed that the results of the meeting would be prepared and distributed to the participants by the CEN Consultant on 11 June with a deadline for comments till Monday, 15 June, 13.00. The agreed report would then be sent to the secretariat of the Joint Meeting in Geneva the same day in due time.

25. As a result of the meeting there were still some reservations (B, CH, NI) with respect to the application for a reference to EN 15507 in ADR/RID as applied for by EuPC.

26. It was pointed out that the reference text to EN 15507 as proposed by EuPC to be added to ADR/RID 6.1.5.2.5/ 6.5.6.3.4 (“EN 15507:2008 ...is considered to be such an approved equivalent procedure.”) would not be exclusive, but would allow competent authorities to keep the normal procedure or to approve further procedures. However, the reference text was not discussed. The Swiss representative would also accept wordings like “...may be such an equivalent procedure” or “...may be accepted as such an equivalent procedure.”

27. Under consideration of the information given during the meeting, the explanations found for the diverging test results, the outcome of latest investigations on the relationship between material properties determined by the laboratory tests and performance test results as described in EN 15507, in particular, which will be examined in detail till the Joint Meeting in September, it seemed possible that a consensus could be reached.

28. It was felt that the way how the standard shall be applied could be better described in more detail either in the standard itself or in a Note in ADR/RID. The results of the workshop indicate also room for additions and improvements of EN 15507 for which CEN should be encouraged to start with.
