



DEPARTMENT OF TRANSPORTATION
HAZARDOUS MATERIALS REGULATIONS BOARD
WASHINGTON, D.C. 20590

18534

[49 CFR Part 173]

[Docket No. HM-67; Notice 70-23]

TRANSPORTATION OF HAZARDOUS
MATERIALS

Flash Points of Flammable Liquids

The Hazardous Materials Regulations Board is considering amending §§ 173.115 and 173.119 of the Department's Hazardous Materials Regulations to specify use of the Tagliabue (Tag) closed-cup tester (ASTM D 56-70) to determine flash points of flammable liquids, instead of the Tagliabue (Tag) open-cup tester (ASTM D 1310-67), presently specified.

The flash point is generally accepted as a useful means to determine the flammability of flammable liquids, and therefore their potential fire hazard during transportation. The Tagliabue open-cup testing method, which has been in use with only minor modification for many years, lacks the precision, reliability, and reproducibility necessary to properly estimate the flammability hazard that may be encountered during transportation.

This notice is not intended to change the present established classification ranges or packaging of flammable liquids. Its purpose is to propose adoption of a more accurate method for determining flash points than the Tag open cup presently affords.

As part of the Department's overall review of the Hazardous Materials Regulations, the Board and the staff of the Office of Hazardous Materials (OHM) have been evaluating methods used for classification of materials according to the hazard presented during transportation. OHM contracted with the Safety Research Center, U.S. Bureau of Mines, to examine the limitations of the available flash point testers and to recommend the best method for adoption by DOT.

In reaching their conclusions, the Bureau of Mines measured the present state of the art against the following criteria:

1. Repeatability (data obtained by the same analyst in several determinations, using the same equipment and the same sample).
2. Reproducibility (data obtained by several analysts, each using a different piece of equipment of the same type, and using the same sample).
3. Reliability in assessing the fire or explosion hazard.

In addition, the Bureau of Mines considered and evaluated all comments received in response to that part of a prior notice of proposed rule making (NPRM)¹ dealing with definitions of flammable liquid, flashpoint, open-cup tester, and closed-cup tester. The results of 1 recommendations of the Bureau's study have been reported.²

The Bureau's report recommends that the Tag closed-cup method be used to determine flashpoints of flammable liquids for purposes of the DOT Hazardous Materials Regulations. The conclusions, proposing adoption of the closed-cup method, may be summarized as follows:

1. The closed-cup method is more precise and reliable than the open-cup method, gives more reproducible data, and provides a more conservative estimate of the hazard presented by the formation of flammable vapor-air mixtures under either confined or unconfined conditions.

2. "It is often proposed that an open-cup more nearly approximates the geometry of a spill situation than does a closed-cup. In our judgment, this is a trivial consideration in choosing among the variations of existing apparatus. The actual likelihood of ignition of a spill depends heavily upon factors which are beyond the scale of laboratory apparatus, such as the cooling of the liquid surface by evaporation or the gustiness of the atmosphere."

"The greatest explosion hazard results from leakage or spillage into surroundings that provide some confinement, such as a railroad box car, a van-type truck, or the hold of a ship. In this situation, convection currents aid the formation of homogeneous vapor-air mixtures and the magnitude of overpressures in confined combustion is usually greatest with homogeneous mixtures. Here again, the closed-cup gives the best definition of hazard." "Experience shows that spills and leaks in confinement are common accident situations and must be considered in the development of safety criteria."

3. Due to its greater reliability, the closed-cup method has been accepted by the National Fire Protection Association, the National Academy of Sciences, the United Nations Intergovernmental Maritime Consultative Organization (IMCO), and many western European industrial countries, including Great Britain, France, West Germany, Sweden, and the Netherlands.

Additional reasons supporting the closed-cup method may be found in a review of various technical publications and comments received on a prior notice of rule making.³ The following is quoted from the International Chamber of Shipping's statement which was attached to the IMCO October 15, 1969, communication to the sixth session of the Committee of Experts on the Transport of Dangerous Goods:

The closed-cup method of testing should be used rather than the open-cup method in view of the former's much better precision.⁴

Proponents of the open-cup method point out that improvement in technique in recent years has resulted in increased

precision and reproducibility of data. It is agreed that refinement of test methods has brought some improvement. However, in spite of this improvement, the Board believes that the open cup is still not equal to the closed-cup method for overall transportation safety purposes. For example, the report of Technical Subcommittee No. II of the Chicago Society for Paint Technology⁵ summarizes the testing done during 1968 with six different types of flashpoint testers and 27 solvents having flash points ranging from 20° F. to 190° F. The report concluded that, "All closed-cups were considerably more reliable and easier to work with than the other cups * * *."

Some comments received on Docket HM-3; Notice No. 68-2 stated that a closed-cup is not responsive to mixtures that contain low-volatility nonflammable components; it is, on the other hand, far too stringent for mixtures containing very small (less than 0.2 percent) amounts of highly volatile flammable compounds. During the test of a mixture, the closed-cup can concentrate nonflammable vapors as readily as flammable vapors. These nonflammable vapors can have a suppressant effect upon the flammability of the sample, thereby raising the flash point beyond the limit prescribed in the regulations for flammable liquids. In an open-cup, part or all of the vapors can escape, thus reducing this suppressant effect. On the other hand, comments noted that a non-flammable anti-knock compound containing less than 0.2 percent of dissolved hydrocarbon, because of trapping of the hydrocarbon traces in the vapor space of the apparatus, had a closed-cup flash point of 58°-73° F., compared to an open-cup flash point of 180°-245° F.

The board realizes that none of the presently available test methods accurately applies to all mixtures. To cover the unusual behavior of certain mixtures, the Board can issue the necessary rulings. For example, the Board could classify such mixtures according to the flash point of their major component. There may be alternative means to cover certain mixtures which do not lend themselves to the proposed testing procedure, and the Board welcomes any suggestions in this regard. The decision as to proper classification of exceptions could be based upon other data or experience showing that the liquid is more or less hazardous than the flash point data indicate. The exceptions should not govern the general rule, however, and the Board is concerned with covering the great majority of substances by a single test method.

In defining flammable liquid,⁶ the United Nations Organization recognizes both the open- and closed-cup methods. It is the Board's understanding that the U.N. included the open-cup method principally to accommodate the United States' regulations.

The United Nations Committee of Experts on the Transport of Dangerous Goods, in arriving at the value of 73° F. for the closed-cup as being equivalent to 89° F. in the open-cup test, considered all available information on the subject. The Paint Technology Report shows an average difference of 7° F. between the Tag open- and closed-cup methods.¹ A review of the pertinent literature confirms this relationship. Therefore, the Board intends to substitute 73° F. for 80° F. in the Hazardous Materials Regulations as the upper limit for flash points of regulated flammable liquids in implementing the change from the open-cup to the closed-cup method. The Board does realize that for a few materials the difference between methods may be much more. It is important to emphasize that this change is in no way an attempt to change the classification of the existing flammable liquids. It is recognized, however, that there may be some isolated cases where the classification would change based upon closed-cup test results. The Board would appreciate receiving advice on how to deal with such situations so as to minimize the hardship on industry.

Upon adoption of this proposal, all references in the Department's Hazardous Materials Regulations will be changed from open-cup to closed-cup.

The Board intends to retain the lower flash point limit of 20° F., as prescribed in § 173.119, open-cup for the closed-cup method. The corresponding flash point difference between the open- and closed-cup methods at this temperature range generally would be very slight, and therefore a change in the lower limit is considered unnecessary.

In the event that the new classification, "Combustible liquids," is established pursuant to proposed rule making,¹⁰ the test method proposed herein, conducted at an appropriately reduced heating rate, would be prescribed in place of the open-cup test. The equivalent closed-cup temperatures would be substituted for the adopted "Combustible liquid" open-cup temperatures.

Interested persons are invited to give their views on the amendment proposed herein. Communications should identify the docket number and be submitted in duplicate to the Secretary, Hazardous Materials Regulations Board, Department of Transportation, 400 Sixth Street SW., Washington, DC 20590. Communications received on or before March 2, 1971, will be considered before final action is taken on the proposal. All comments received will be available for examination by interested persons at the Office of the Secretary, Hazardous Materials Regulations Board, both before and after the closing date for comments.

In consideration of the foregoing, 49 CFR Part 173 would be amended as follows:

(A) In § 173.115 paragraph (a) would be amended; paragraphs (d) and (e) would be added to read as follows:

§ 173.115 Flammable liquids; definitions.

(a) For the purpose of Parts 170-189 of this chapter, "Flammable liquid" means any liquid having a closed-cup flash point at or below 73° F.

(d) "Flash point" of a liquid means the minimum temperature of the liquid at which it gives off vapor sufficient to form an ignitable mixture with the air near the surface of the liquid or within the container used.

(e) "Closed-cup" means the method of determining flash point as specified in the Standard Method of Test for Flash Point by the Tagliabue (Tag) Closed Tester (ASTM D 56 70) (American Society for Testing and Materials, 1916 Race Street, Philadelphia, PA). In determining the flash points of liquids having a viscosity of 4 centipoise or higher (at 100° F.) the prescribed 2° F./minute rate of the Tag test must be reduced to 0.5° F./minute, or the temperature differential between the sample and the bath must be maintained at 5° F. or less.

(B) In § 173.119 the introductory texts of paragraphs (b) and (l) would be amended to read as follows:

§ 173.119 Flammable liquids not specifically provided for.

(b) *Flammable liquids with flash point above 20° F.* Flammable liquids with flash point above 20° F. and having vapor pressure (Reid¹ test) not over 16 pounds per square inch, absolute, at 100° F., other than those for which special requirements are prescribed in this part, must be packaged in packagings of a design and constructed of materials that will not react dangerously with or be decomposed by the chemical packed therein, as follows (see paragraphs (c) through (l) of this section for high-pressure liquids and paragraph (m) of this section for flammable liquids which are also oxidizing materials or corrosive liquids):

(l) *Viscous flammable liquids with flash point above 20° F. and having a vapor pressure which does not exceed 18 pounds per square inch, absolute, at 100° F.* Viscous flammable liquids with flash point above 20° F. and having a vapor pressure which does not exceed 18 pounds per square inch, absolute, at 100° F. must be packaged as follows:

This proposal is made under the authority of sections 831-835 of title 18, United States Code, section 9 of the Department of Transportation Act (49 U.S.C. 1657), and title VI and section 902(h) of the Federal Aviation Act of 1958 (49 U.S.C. 1421-1430 and 1472(h)). U.S.C. 1421-1430 and 1472(h)).

¹ ASTM Test D323.

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By direction of Commandant,
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¹ Docket No. HM-3; Notice No. 68-2 (33 F.R. 3382, Feb. 27, 1968).

² Kuchta, Joseph M. and Burgess, David, Report No. S 4131, Apr. 29, 1970, Safety Research Center, U.S. Bureau of Mines. This document is available from the Clearing House for Federal Scientific and Technical Information, National Bureau of Standards, U.S. Department of Commerce, Springfield, Va. 22151 at a cost of \$3 per copy, or Microfiche copy at 65 cents.

³ Kuchta, Joseph M. and Burgess, David, Report No. S 4131, Apr. 29, 1970, Safety Research Center, U.S. Bureau of Mines, p. 5.

⁴ Ibid., p. 6.

⁵ Docket No. HM-3; Notice No. 68-2 (33 F.R. 3382, Feb. 27, 1968).

⁶ United Nations Economic and Social Council, E/CN.2/CONF.5/R.198.

⁷ Probst, K. G., Correlation of Apparatus for Measuring Flash Point of Solvents, J. of Paint Technology, Vol. 40, No. 527, pp. 576-81 (December 1968).

⁸ United Nations, Transport of Dangerous Goods (1966), Vol. I, p. 5, ST/ECA/81/Rev. 1, E/CN.2/Conf. 5/10/Rev. 1.

⁹ Probst, K. G., Correlation of Apparatus for Measuring Flash Point of Solvents, J. of Paint Technology, Vol. 40, No. 527, pp. 576-81 (December 1968).

¹⁰ Docket No. HM-42; Notice No. 70-3 (35 F.R. 3298, Feb. 21, 1970).