

Designation: F 764 – 82 (Reapproved 1994)

Standard Practice for Compatibility of Materials with High-Energy Propellants (Impact Sensitivity Threshold Technique)¹

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1. Scope

1.1 This practice covers the threshold procedure for impact testing in high-energy propellants, and differs from Test Method D 2512 only in the additional safeguards and restraints required when handling these dangerous materials, both for safety and for prevention of interfering reactions.

2. Referenced Documents

2.1 ASTM Standards:

D 2512 Test Method for Compatibility of Materials with Liquid Oxygen (Impact Sensitivity Threshold and Pass-Fail Technique)²

3. Significance and Use

3.1 When this practice is employed to measure the threshold impact sensitivity of a material-propellant pair, a relative sensitivity assessment is obtained which permits the ranking of materials.

3.2 The parent test method, D2512, is widely used for acceptance-testing materials for use in liquid oxygen systems, and this practice is usable in a similar way for many propellant systems. Twenty separate samples of the material submerged in liquid propellant are subjected to 98J (72 ft-lbf) or as specified impact energy delivered through a 12.7-mm ($\frac{1}{2}$ -in.) diameter contact. More than one indication of sensitivity is cause for immediate rejection. A single explosion, flash, or other indication of sensitivity during the initial series of 20 tests requires that an additional 40 samples be tested without incident to ensure acceptability of the material for use with that propellant.

4. General

4.1 In conducting tests with toxic, corrosive, hypergolic, or other dangerously reactive rocket propellants, the following constraints, in addition to those for liquid oxygen impact testing, must be met:

4.1.1 Assure safety for personnel.

4.1.2 Assure that substances known to react with the test

propellant are excluded from the test system.

4.1.3 Assure that reactive propellant is not diluted with inert material.

4.1.4 Dispose of unreacted propellant and reaction products after test in a safe manner.

4.1.5 Minimize the quantity of propellant used.

4.1.6 Eliminate interaction of the propellant with functional parts of the test apparatus.

4.1.7 Assure proper post-test examination of specimens.

4.1.8 Miscellaneous considerations.

4.1.9 Reporting.

4.2 The procedures described in this practice deal with precautions necessary for handling the high-energy propellants safely and for preventing interfering reactions while conducting the impact tests. The basic Test Method D 2512, of the impact test is not altered. The emphasis is on test system design principles, not specifics, because each high-energy propellant has different properties that must be considered in the final design of the system, and new propellants with new properties are frequently introduced into the test laboratory. Many of the precautions and techniques described are appropriate in tests other than impact tests. Tables 1 and 2 list the physical properties of some fluids that are used or are under consideration as rocket propellants—oxidizers and fuels respectively.

5. Safety Precautions

5.1 The main requirement for personnel safety is prevention of any of the propellant, reaction intermediates, or reaction products from coming in contact with the personnel. Complete isolation of personnel from the test apparatus when the latter contains propellant will prevent such contact.

5.2 Complete isolation is assured by locating the test apparatus in an enclosure and behind a barricade. The operator is stationed in a control room on the other side of the barricade. Visual observation of tests shall be through armored glass viewing ports and a mirror system or periscope. Manipulation of test apparatus, specimens, cups, striker pins, etc., must be carried out using a remote manipulator. (Many types exist; they were developed for nuclear applications and may need relatively simple modifications to be satisfactory.) Valving for propellants, refrigerants, purge gasses, etc., must be operated remotely from the control room. Controls for plummet raising, lowering and dropping, and for lights and ventilation must be

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² *Annual Book of ASTM Standards*, Vol 15.03.

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TABLE 1 Physical Properties of Propellant Oxidizers

Oxidizer	Molecular Formula	Molecular Weight	Melting Point, ° R (K)	Boiling Point, ° R (K)	T_c ° R (K)	P_c , psia (MPa)	Liquid Range, ° R (K)
Chlorine pentafluoride	ClF ₅	130.45	306.3 (551.3)	469.1 (844.9)	749.1 (1348.9)	769 (5.3)	432.8 (779.0)
Chlorine trifluoride	ClF ₃	92.45	352.6 (634.9)	512.9 (923.2)	805 (1449.0)	838 (5.7)	452 (813.6)
Dioxygen difluoride	O ₂ F ₂	70.00	197 (354.6)	399.4 dec (718.9)	200 est (360)
70 % FLOX ^A	...	36.20	NA	244 est (439.2)	160 est (288)
		(average)					
Fluorine	F ₂	38.00	96.4 (173.5)	153.1 (275.6)	259.1 (466.4)	808.3 (5.6)	162.7 (292.9)
MON-10 ^B	...	85.8
		(average)					
Nitrogen tetroxide	N ₂ O ₄	92.02	447.9 (806.2)	529.8 (953.6)	777 (1398.6)	1470 (10.1)	329 (592.2)
Nitrogen trifluoride	NF ₃	71.01	116.7 (210.1)	260.8 (469.4)	421.3 (758.3)	657 (4.5)	304.6 (548.3)
Nitryl fluoride	NO ₂ F	65.01	192 (345.6)	361.3 (650.3)	629.1 (1132.4)	...	437.1 (786.8)
Oxygen	O ₂	32.00	97.8 (176.0)	162.3 (292.1)	278.6 (501.5)	736.9 (5.1)	180.8 (325.4)
Oxygen difluoride	OF ₂	54.00	89 (160.2)	231 (415.8)	388 (698.4)	719 (5.0)	299 (538.2)
Perchloryl fluoride	ClO ₃ F	102.45	228.9 (412.0)	407.5 (733.5)	663 (1193.4)	779 (5.4)	434 (781.2)
Tetrafluorohydrazine	N ₂ F ₄	104.02	197.7 (355.9)	358.7 (645.7)	559.2 (1006.6)	625 (4.3)	361.5 (650.7)

^A Seventy weight percent of fluorine in oxygen.

^B Ten weight percent of nitric oxide in nitrogen tetroxide.

TABLE 2 Physical Properties of Propellant Fuels

Fuel	Formula	Molecular Weight	Melting Point, ° R (K)	Boiling Point, ° R (K)	T_c ° R (K)	P_c , psia (MPa)	Liquid Range, ° R (K)
Aerozine 50	^A	45 (avg)	478–481 (860.4–865.8)	618 (1112.4)	1094 (1969.2)	1696 (11.7) (calc)	614 (1105.2)
Ammonia	NH ₃	17.03	351.7 (633.0)	431.6 (776.9)	730.0 (1314.0)	1638.6 (11.3)	378.3 (680.9)
Diborane	B ₂ H ₆	27.69	193.7 (348.7)	325.1 (585.2)	521.8 (939.2)	581 (4.0)	328 (590.4)
Ethane	C ₂ H ₆	30.07	161.8 (291.2)	331.8 (597.2)	549.7 (989.5)	709.8 (4.9)	387.9 (698.2)
Ethylene	C ₂ H ₄	28.05	187.2 (337.0)	305.0 (549.0)	509.5 (917.6)	742.2 (5.1)	322.3 (580.1)
Hydrazine	N ₂ H ₄	32.04	494.4 (889.9)	696.0 (1252.8)	1176 (2116.8)	2132 (14.7)	682 (1227.6)
Hydrogen (normal)	H ₂	2.016	25.1 (45.2)	36.7 (66.1)	59.7 (107.5)	190.8 (1.3)	34.6 (62.3)
Methane	CH ₄	16.04	163.2 (293.8)	201.0 (361.8)	343.2 (617.8)	673.1 (4.6)	180.0 (324.0)
Monomethylhydrazine	(CH ₃) ₂ NH ₂	46.08	397.2 (715.0)	652.2 (1174.0)	1054 (1897.2)	1195 (8.2)	647 (1164.6)
Monomethylhydrazine monohydrate	(CH ₃) ₂ NH ₂ ·H ₂ O	64.10
Pentaborane	B ₅ H ₉	63.17	407.6 (733.7)	596.8 (1074.2)
Propane	C ₃ H ₈	44.10	153.9 (277.0)	415.9 (748.6)	665.9 (1198.6)	617.4 (4.3)	512.1 (921.8)
Trimethylborane	B(CH ₃) ₃	55.92	201.0 (361.8)	455.7 (820.3)	690 (1242) (est)	...	488 (878.4) (est)
UDMH (unsymmetrical dimethylhydrazine)	(CH ₃) ₂ N ₂ H ₂	60.08	388.7 (699.7)	606 (1090.8)	942 (1695.6)	786 (5.4)	553 (995.4)

^A Fifty weight percent hydrazine in UDMH.

electrical switches operated from the control room. Inert gas pneumatic controls are acceptable substitutes; hydraulic controls should *not* be used.

5.3 All ports and leads through the walls and barricade must be carefully sealed and continuously purged with nitrogen to prevent gas diffusion in either direction. Air-line respirators must be available in the control room. Their use can be optional when testing with easily detected propellants such as chlorine trifluoride, but mandatory for very toxic and insidious propellants such as pentaborane and nitrogen tetroxide.

5.4 The control room must have a ventilating system separate from that of the test enclosure. The intake should be remote from the exhaust of the test enclosure. Atmospheric pressure in the control room should be slightly greater than that in the test enclosure so that there is resistance to gas flow into the control room even if the port purges fail. When detectors for the propellant being tested are available, a unit should be set up in the control room and connected to an audible alarm. If air leakage from the control room to the test enclosure can result in an explosion hazard in the atmosphere of the test enclosure because of the presence of fuel vapors, the control room must be purged free of oxygen by means of an inert gas, and the operator must wear an air line respirator. The usual

precautions of a “buddy” in the same room and the wearing of a rescue harness are mandatory.

6. Exclusion of Reactive Impurities

6.1 Many high-energy propellants will react readily with common materials, either spontaneously or under shock and impact initiation. These various reactions with impurities must be prevented as they would be false positives. Thus, liquid fluorine will react explosively under impact with frozen water, solid CO₂, and most common organic materials including polytetrafluoroethylene. Liquid pentaborane will inflame in dilute gaseous oxygen (air plus excess nitrogen) at impact, even when the oxygen content is too low (<4 %) to cause spontaneous inflammation. Some fluorocarbon and chlorofluorocarbon liquids will explode when torqued in contact with aluminum, such as the sample cups. Trace residues from chlorocarbon cleaning solvents are impact sensitive in N₂O₄ and they inflame spontaneously in some fluorinating agents (the behavior in NF₃ is not reported).

6.2 There are two stages to exclusion of reactive impurities. The first is the prevention of their introduction during preparation for tests. The apparatus, enclosure, and specimens must be cleaned in such a manner that all reactive materials are

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removed. Thus, for propellants that are sensitive to moisture, the whole test enclosure must be thoroughly dried. A warm-air gun is effective for small enclosures. Grease, oil, and dust particles must be removed, in accordance with Test Method D 2512, Section 8, from all portions of the apparatus that might allow them to drop or condense in the sample cup. Long-ray ultraviolet illumination may be used to check for cleanliness. Test specimens, sample cups, striker pins, and guide bars must be thoroughly cleaned and dried. The procedures commonly called “FLOX Cleaning” specified in Test Method D 2512, Section 8 are sufficient provided that the standards are truly met. Since most cleaning solvents are reactive with most high-energy propellants, absorbed traces of the solvents must be removed. All porous or permeable materials such as metal castings and sinterings, ceramics and cements, plastics and elastomers, must be baked dry in a vacuum after cleaning. Two hours at 105°C, 1 torr (133 Pa) is sufficient for the specimens used in impact tests.

6.3 Reactive species must be removed from the atmosphere of the test enclosure as well. The best arrangement is a continuous purge with filtered dry gaseous nitrogen. The purge should be turned on after completion of all operations that require the presence of the operator in the test enclosure. The enclosure is then sealed except for entrance and exhaust of the purge gas. Depending on the size of the enclosure and the initial and final maximum concentrations of the species to be removed, the duration of the purge period before testing can be estimated. However, an actual check of the concentration should be made before starting the tests. In cryogenic, water-sensitive tests, turning on the refrigerant supply will suffice; if water vapor is present, it will be shown by frosting on tubing or fogging in the atmosphere. A gas oxygen concentration analyzer³ is needed for determining the safe level for tests in which oxygen is detrimental, such as with boron hydrides.

6.4 Reactive impurities must be prevented from entering the test enclosure during the tests also. The enclosure must not be opened during a test series. Gases and vapors can be prevented from entering, or swept out if they penetrate, by continuing the gas nitrogen purge throughout the test period. The impurities that must be avoided are usually specific to the tests being conducted. The test designer must decide which substances must be barred, and what levels of other impurities can be tolerated. In tests with cryogenic fluorinating oxidizers, the moisture level must be held so low that frost formation does not occur on parts at -196°C. In tests with boron hydrides, the oxygen content should be below 0.25 % to prevent smoke or fume formation which can obscure tests. Possible interactions of impurities with the materials being tested must also be considered.

6.5 If reaction occurs in an impact test, partially reacted particles are splashed around the test area. These particles may react hypergolically with propellant splashed out of the sample cup in subsequent tests, causing false positives. The test area must be cleaned out after each positive reaction. A gaseous

nitrogen clean-off gun on a flex line can be controlled by the remote manipulator to blow particles out of the test area.

6.6 Commercial cryogenic refrigerants such as liquid nitrogen must conform to the requirements of this method with regard to concentrations of solid impurities. The concentration of solids is often increased where locally refilled portable containers are used to supply liquid nitrogen. Many of the particles consist of species that are reactive with propellants—ice, CO₂, frozen oils. To prevent contamination of the test, the liquid nitrogen must be filtered through a 40- μ m nominal cryogenic filter. If the purity of purge gases is not assured, they must be filtered through a 10- μ m filter.

7. Prevention of Dilution of Propellant

7.1 The use of purge gases and separate refrigerants (see Section 9) introduces the possibility that the propellant may be diluted by a foreign, nonreactive material, and so give a false negative test. Sources of such dilution are the atmosphere in the test enclosure and the refrigerant used in tests conducted at temperatures below ambient.

7.2 The only condition under which the atmosphere is likely to act as a diluent exists when the temperature of the test fluid is below the condensation temperature of the atmospheric gases. At liquid nitrogen temperature (-196°C), oxygen will condense (boiling point—182°C). However, if the test enclosure is swept free of oxygen with gas nitrogen, then only nitrogen is present. Condensation of the latter into a test fluid at -196°C is slow enough that it can be ignored, and if the test fluid is warmer than -196°C, no appreciable condensation will occur in the interval between filling the sample cup and dropping the plummet. If tests are conducted at temperatures below -196°C, neon or helium should be used as the inert gas purge.

7.3 Dilution by a liquid refrigerant is a more serious possibility, and the apparatus must be designed to prevent such an occurrence. Generally, high-energy propellants are not used as their own refrigerants in the fashion done with liquid oxygen. An inert refrigerant is used instead. It may be used to condense the propellant from the gaseous state, and it is generally used to chill parts of the test apparatus. Liquid nitrogen used as a refrigerant for fluorine and oxygen difluoride tests is an example. In such systems, the sample cup must be positioned so that the refrigerant cannot spill into the cup. This care must be taken in both prechilling the cups and maintaining them at a controlled temperature during the test itself. The sample cup must always be located so that its lip is at least 6.4 mm (1/4 in.) above the maximum spillover tubing for the refrigerant. Fig. 1 shows one design which accomplishes this separation.

8. Disposal and Decontamination

8.1 It is necessary to remove residues or propellant and gaseous reaction products after each test.

8.2 Gaseous propellant will be removed on the stream of the gas used to purge the test enclosure. If the toxicity or reactivity of this vaporized propellant is not too great, the vapors may be carried from the enclosure through a stack, so that they are well diluted before reaching ground level again. More dangerous vapors must be absorbed or rendered harmless in some fashion.

³ A dual-range Model 320 Portable Oxygen Detector, manufactured by Teledyne Analytic Instruments, San Gabriel, Calif., is satisfactory.