NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

MSC APOLLO 13 INVESTIGATION TEAM

FINAL REPORT

PANEL 8

HIGH PRESSURE OXYGEN SYSTEMS SURVEY

ADDENDUM 1

JUNE 1970

MANNED SPACECRAFT CENTER
HOUSTON, TEXAS

PANEL 8

HIGH PRESSURE OXYGEN SYSTEM PANEL

ADDENDUM TO FINAL REPORT

June 5, 1970

Richard S. Johnston, Chairman

FOREWARD

As a consequence of the Apollo 13 oxygen tank failure, this survey was undertaken to assess the current test techniques for determining the fire or explosion hazards of materials exposed to liquid or gaseous oxygen with the purpose in mind of developing more significant materials qualification tests. This survey was made by W. R. Blackstone of Southwest Research Institute.

AN ASSESSMENT OF CURRENT TEST TECHNIQUES FOR DETERMINING THE FIRE OR EXPLOSION HAZARDS OF MATERIALS EXPOSED TO LIQUID OR GASEOUS OXYGEN

INTRODUCTION

Historically, the qualification of materials for use in either LOX (liquid oxygen) or GOX (gaseous oxygen) has evolved from prior successful usage or by testing the material under the expected use conditions for the most probable mode of ignition, generally impact or friction generated ignition. As a consequence, test methods have concentrated on impact and friction as virtually the only causes of ignition in LOX or GOX systems. For systems containing electrical power, this is not a valid assumption. For a material to be hazardous, sometimes more than ignition is required in most applications, that is, the material must propagate the reaction to a degree where system damage or malfunction occurs.

The types of materials to be tested are those that might react to an external stimulus of some sort, and not those that tend to react with liquid or gaseous oxygen during static exposure, since these would not be considered for spacecraft use.

The types of ignition stimuli would include sparks, electrical shorts, friction, mechanical impact, hydrodynamic shock, pneumatic shock, bulk heating, etc. Note that all of these involve either direct heating of the material or conversion of some other form of energy into heat. Since the heating rates differ widely among these various stimuli, the thermal conductance of the material becomes important to ignition. Once ignition has occurred, interest should be focused on propagation rates to determine if the material will self-quench, burn slowly, burn rapidly, or explode.

This report summarizes the findings of a review of current test methods in both liquid and gaseous oxygen and provides an assessment of their competence in determining the hazards of candidate materials for spacecraft usage.

LIQUID OXYGEN TESTS

Tests which considered the ignition sources available to a candidate material such as electrical shorts, sparks, friction, etc. would be useful, but insofar as it was determined efforts in these areas have been limited to "one shot" experiments to resolve specific problems and did not generate data of wide applicability.

Apparently, the only widely used LOX ignition test methods that exist in specification form are those employing mechanical impact. These impact tests employ a method and procedure originally developed at the Army Ballistic Missile Agency (now NASA - Marshall Space Flight Center). This method now exists in three standardized forms: USAF Specification Bulletin 527, NASA Specification MSFC-SPEC-106, and ASTM Test Method D-2512. The NASA and Air Force tests are actually almost identical, and the ASTM method is no more than a marriage of the two. In all three, a weight is dropped onto a striker pin which rests on a sample immersed in LOX. The test data consist simply of observations as to whether ignition did or did not occur. A material is considered acceptable for LOX service if it produces no reactions in twenty test drops, using a new sample for each test drop.

A major difficulty with the LOX impact testing has been one of extremely poor precision in evaluating the results. For example, the repeatability and reproducibility of results vary so widely that it is virtually impossible to determine significant differences on impact sensitivity with any confidence.

Also worthy of mention is the fact that some materials are presently categorized in the current LOX impact tests as having"batch-to-batch" variation of sensitivity. One of these is a fluorosilicone grease, and NASA caompatibility ratings show that, on the average, about one-fourth of the jars of this material submitted for evaluation are accepted as "satisfactory" for LOX service. The remainder are rejected as "unacceptable." However, scrutiny of the test results on which these decisions were made reveals that the reaction frequencies obtained were about what one would expect to obtain in repeated tests on the same batch of material, and that there was no statistical justification for deciding that some of the batches were "acceptable" while others were not. Moreover, a jar from another batch of this material was submitted to repeated tests by the current standards (at another laboratory) and the same behavior was observed: both "passes" and "fails" were obtained.

These contradictory results are partly attributable to differences in test equipment at the two laboratories, but the basic problem is conceptual in nature rather than mechanical. The current standards are grossly inadequate from a statistical standpoint, and they do not take into consideration the reaction propagation which is necessary to the determination of the potential hazard of a material.

Until recently, the idea of measuring reaction propagation rates in LOX was largely ignored and materials were rated solely on the basis of the standard LOX impact test. In 1969, however, ASTM published for information a new method for rating materials on the basis of reaction intensity (Book of Standards, Part 18, Second Edition, October 1969). This method is to be balloted for tentative status in June 1970, and appears likely to be accepted. The test employs impact as the ignition stimulus, but does not attempt to measure minimum ignition levels. Rather, the material is impacted from a level known to be sufficient to produce ignition, and a measurement is made of the explosive shock pressure produced.

A considerable body of data have been produced on a wide range of materials (reference 1) and it has been determined that all liquids and greases tested fall conclusively into one of two distinct categories; either relatively inert or explosively reactive.

Much additional discussion could be devoted to the problems of LOX impact testing, but this would get quickly beyond the scope of this review. It should be noted, however, that with regard to testing for fire and explosion hazards in LOX, there are only two more or less standardized methods in existence and both are impact tests. The more extensively used of these methods measures susceptibility to ignition only and is known to be deficient in precision. The other appears to be very accurate, but measures reaction intensity only and thus does not provide the very necessary knowledge of susceptibility to ignition. From these facts, it is clear that the LOX test area is in need of considerably more attention.

GASEOUS OXYGEN TESTS

In contrast to the situation of the LOX test, early experimenters with gaseous oxygen had the background advantage of a considerable number of ignition and flammability test procedures already worked out for other similar environments such as air. A considerable disadvantage, however, was the fact that gaseous oxygen tests have to take into account two additional and significant environmental variables not faced in the LOX test; initial pressure and initial temperature. In addition, consideration must be given to a much broader range of variables with respect to the rate at which ignition stimuli are applied (bulk heating, for example, becomes a relevant source of ignition). Probably as a result of the added complexities, standardization of test methods for GOX does not appear to have progressed as far as it has in the LOX area.

Among the many ignition tests that have applied experimentally to materials in GOX are types such as spark, hot wire, flashbulb, open flame, friction, hotplate, compression, oxidation bomb, shock tube, explosive charge, and impact. Generally, each of these has been used only for a few specific investigations, and within these usages, there is considerable variation in initial pressures. In 1956, the oxygen industry adopted an acceptance policy (reference 2) for anti-friction compounds which closely followed the basic tenets in the present Military Specification MIL-T-5542D. The test conditions covered a range of pressures from 0 to 500 psi and temperatures up to 250° F. The specified tests included a bomb oxidation test, a calorimeter tests, a "surge pressure" test, and the familiar LOX impact test. According to Reynales (reference 3), these tests were felt to be adequate for relatively low pressure systems used in the oxygen industry, but quite inadequate for missile systems with the attendant higher magnitudes of pressure, temperature, etc.

In 1967, NASA-MSC issued a revised specification for material qualification tests entitled "Procedures and Requirements for the Evaluation of Spacecraft Nonmetallic Materials," MSC-A-D-66-3, Revision A. This was followed early in 1968 by a similar specification entitled "Apollo Spacecraft Nonmetallic Material Requirements." According to McCain (reference 4), these documents set forth the first comprehensive hazard test program concerning oxygen compatible materials for spacecraft environments. The tests for fire hazard in these specifications include upward and downward propagation rate tests, a combined thermogravimetric analysis and spark ignition test, an electrical wire insulation and accessory flammability test, an electrical potting and coatings flammability test, a flash point and fire point test, and guidelines for friction and impact tests. A further breakdown was made with regard to the service environment to be faced by the candidate material, and the specific tests to be imposed upon a material were chosen in accordance with this breakdown. A subcategory was identified to differentiate between material with major or minor exposure to the environment.

The MSC specification represents a major improvement in GOX testing logic, and considerable data have been obtained. Most of these data have been obtained for GOX pressures below 20 psia but some work is being done at higher pressures.

CONCLUSIONS AND RECOMMENDATIONS

Based on this review, it appears that, at present, there is no single test, or group of tests, for either LOX or GOX tests over a wide range of conditions. The LOX impact reaction intensity test appears promising, but this test has only recently been published and has not been used outside the laboratory where it was developed.

The NASA GOX testing methods represent a considerable improvement in the state-of-the-art, and should provide an accurate basis for GOX material compatibility screening tests and propagation hazards.

Attention should be given to the development of a means of combining the results of several tests which represent the overall ignition, explosion, and propagation hazard of the material. Numerical comparison, rather than "Go-No-Go" criteria, should be instigated wherever possible to provide a quantitative ranking of materials as to their flammability hazard.

Ignition methods used in the tests should reflect the ignition sources available to the candidate material in its intended usage.

While there is considerable flammability data available, and more accurate materials screening information can be generated through the improvement of test techniques and the combination of the results of several tests, experience at NASA indicates the fact that is difficult to relate the results of material screening test to the degree of hazard associated with the spacecraft system. The experimental work strongly indicates the need to conduct tests in an accurate simulation of the intended use environment.

Screening tests will provide a basis for the selection of materials in the design phase and are an excellent means of evaluating "batch-to-batch" sensitivity.

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PANEL 8, HIGH PRESSURE OXYGEN PANEL

MEMBER	MAIL ADDRESS
Dr. R. A. Mahugh	The Boeing Co. P. O. Box 3707 Org. 6-8503, Mail Stop 54-60 Seattle, Washington 98124
J. M. Lea	The Boeing Co. P. O. Box 3707 Org. 6-7021, Mail Stop 78-81 Seattle, Washington 98124 206-237-4183
John Kennedy	Dept. 93-14 Garrett-AiResearch 2525 W. 190th Torrance, California 90509 213-323-9500, ext. 2208
Dr. J. E. Johnson	Code 6180 Naval Research Lab Washington, D. C. 20390
C. M. Ramsey	McDonnell-Douglas St. Louis, Mo.
W. R. Blackstone	Southwest Research Institute 8500 Culebra Road San Antonio, Texas 78228
R. S. Johnston	NASA-MSC (PA) ext. 2593
W. H. Bush (3)	NASA-MSC (EC4) ext. 4986
H. H. Jamison	NASA-MSC (EP6) ext. 4991
D. F. Hughes	NASA-MSC (EC3) ext. 5536
J. H. Kimzey	NASA-MSC (ES8) ext. 5281
Dr. C. K. La Pinta	NASA-MSC (EC4) ext. 4291
W. T. Kitts	NASA-MSC (EL2) ext. 2815

MEMBER	MAIL ADDRESS
J. M. Conlon	NASA-MSC (SM) ext. 5231

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