DEVELOPMENT OF AN IMPROVED GASEOUS OXYGEN IMPACT TEST SYSTEM

W. R. Blackstone

FINAL REPORT

Prepared for

The Manned Spacecraft Center
National Aeronautics and Space Administration
Houston, Texas, 77058

July 1970

SOUTHWEST RESEARCH INSTITUTE
SAN ANTONIO HOUSTON

NASA CR 108529

SOUTHWEST RESEARCH INSTITUTE
Post Office Drawer 28510, 8500 Culebra Road
San Antonio, Texas 78228

DEVELOPMENT OF AN IMPROVED GASEOUS OXYGEN IMPACT TEST SYSTEM

W. R. Blackstone

FINAL REPORT

Prepared for

The Manned Spacecraft Center
National Aeronautics and Space Administration
Houston, Texas, 77058

July 1970

Approved:

Robert C. DeHart

Robert C. DeHart, Director
Department of Structural Research

FOREWORD

This report was prepared by W. R. Blackstone, Senior Research Engineer, at the Structural Research Laboratory of Southwest Research Institute in San Antonio, Texas. The work was conducted under NASA Contract No. NAS 9-9543 and SwRI Project No. 03-2631, entitled "Development of an Improved Gaseous Oxygen Impact Test System."

The work reported occurred June 1969 to May 1970 and was conducted under the guidance of L. U. Rastrelli, Assistant Director of the Department of Structural Research of Southwest Research Institute and under the general supervision of Dr. Robert C. DeHart, Director of that Department. The study was administered under the auspices of the Manned Spacecraft Center of the National Aeronautics and Space Administration by Mr. Martial Davoust, Contracting Officer. The Technical Monitor was Mr. Herschel M. Jamison.

ABSTRACT

This report describes consultative services provided by Southwest Research Institute to the Manned Spacecraft Center in a program to develop an improved gaseous oxygen impact test system. The services provided included a critical evaluation of background information derived from related tests already in used, general consultations with MSC staff in the areas of material compatibilities with liquid and gaseous oxygen, assistance to the staff of the White Sands Test Facility in the areas of equipment design and impact test procedures, and assistance to the MSC Apollo 13 Investigation Team.

TABLE OF CONTENTS

I. INTRODUCTION

A. Report Summary

This report summarizes the work performed at Southwest Research Institute in the period of June 10, 1969 through May 10, 1970, under Contract NAS 9-9543, entitled "Development of an Improved Gaseous Oxygen Impact Test System." Experimental efforts in this program were conducted at the Manned Spacecraft Center by NASA personnel; SwRI's role was to provide consultation services to expedite accomplishment of the program goals. The services included assistance in planning of the experimental program, analysis of experimental results, assistance in design and improvement of the experimental equipment, and general consultation in the area of GOX- and LOX-material compatibility.

B. Background and Scope of Efforts

It is common knowledge that many materials can burn or explode if subjected to external stimuli in the presence of oxygen. The space industry, with its widespread use of both liquid and gaseous oxygen, is particularly concerned with this problem, and much effort has been expended in attempting to define the degree of hazard associated with the various materials under various types of external stimuli. Impact is the mode of stimulus which has perhaps received the most attention, as it is known that impacts can and do occur in nearly all oxygen environments.

One of the first problem areas to be recognized was that of the severe mechanical and/or hydraulic impact conditions in liquid oxygen (LOX) systems. Consequently, much research has gone into the development of LOX impact test methods to aid in the selection of materials for these systems, and from this a more or less standardized LOX impact test technique has evolved. The body of data accumulated to date with this procedure is substantial.

On the other hand, relatively little attention has been paid to the problem of impact hazard in gaseous oxygen (GOX) systems, and no proven impact test methods exist in this area. As a result, the designers selecting GOX system materials have had to rely on the rather indirectly applicable results of LOX impact tests. Considering only the great physical differences between LOX and GOX reveals this to be an unsatisfactory situation, but, as will be seen later, there are also serious questions concerning the meaningfulness of LOX impact test data with respect to LOX systems - not to mention any indirect applications.

In an attempt to alleviate this, the Manned Spacecraft Center (MSC) is presently engaged in a program to develop a reliable GOX impact test for use in selecting materials for the many GOX systems employed in the space program. This work was initiated early in 1968 in the Thermochemical Test Branch (TTB) of the MSC with the fabrication of three replicates of the first generation high-pressure GOX impact test system. This system was designed to operate at pressures up to 10,000 psi and consisted essentially of a miniaturized ABMA-type* drop weight apparatus completely enclosed within a high-pressure chamber. The purpose of this first-generation system (known as the E&D tester) was to serve as a vehicle for parametric studies, and succeeding generations would of course be redesigned to account for vagaries pinpointed in the initial exploratory testing. It was recognized that the exploratory tests would be of questionable value with regard to definitive compatibility ratings of the various materials; such data were to come after the redesign phase.

It soon became apparent, however, that the need for reliable compatibility data was becoming critical. Moreover, the E&D tester has a maximum data output rate of only about 9 test drops per day, and this was much too slow to accommodate the growing demand. As a consequence of these developments, it was decided to increase the level of effort by going ahead with two or three alternate tester designs before completing the exploratory work with the E&D tester. This brought manpower availability into the picture, and eventually, in mid-1969, led to transfer of the entire experimental effort to the NASA White Sands Test Facility (WSTF). With the delays involved in the transfer and in the program redirection, it was not until early 1970 that any of the test systems actually became operational.

SwRI's proposal for this work was submitted in June 1968, and the scope of the proposed efforts was keyed to the program being pursued at MSC at that time. Contract negotiations, however, were not completed until June 1969, and at that time the NASA program redirection was being effected. As a result, some changes in emphasis had to be made with regard to SwRI's efforts in the program.

The major part of the proposed effort was to have been directed toward assistance in planning of the experimental program and in analysis of the experimental results with the goal of a carefully developed and reliable test procedure. With the combination of SwRI's background in these areas and MSC's experience with the equipment being developed, it was felt that this goal could be achieved within one to two years. However, the unanticipated equipment transfer resulted in a long delay in the experimental work together with the replacement of the MSC personnel with WSTF personnel who were relatively inexperienced in impact sensitivity testing. As a result, the SwRI efforts have consisted mostly of general consultation with the MSC in the area of GOX- and LOX-material compatibility, and assistance to WSTF

---

*The ABMA drop weight apparatus will be described in Section II.

in the basic techniques of sensitivity testing and in critical areas of equipment design.

Another aspect which influenced the course of the work was a decision early in 1970 to postpone the test system evaluation work in order to put the new equipment to immediate use in "qualifying" materials for the Apollo program. This decision is still in effect at this writing, and there appear to be no plans for any system evaluation work before September 1970, as the systems are fully committed to such work until that time. It is noted that this particular redirection was opposed by SwRI because of the detrimental effects a similar approach had in the early development of the LOX impact test. As will be brought out in detail in later sections, impact test results are highly system dependent. They usually exhibit a very poor reproducibility between test systems, even when the differences between the systems are seemingly minor. It thus stands to reason that the material response obtained in a test system could easily be different from that in the service system. Further, the material response is usually categorized as a "go/no-go" type of response, i.e., the material either does or does not ignite. Considering all these aspects together, it appears that, even with a carefully developed test system, there is always some risk that the material which did not ignite in the test system will do so in the service system. It is felt that these risks are compounded by "qualifying" materials in a test system which has not been evaluated as to whether the material responses it predicts are realistic with respect to the service system in which the material will be used.

In view of the fact that the materials in question are already in use and that we obviously need an evaluation as quickly as possible, the above point may seem a bit trifling at first. Indeed, it would seem that in this case a small amount of early information would be preferable to waiting out a possibly lengthy system evaluation. However, if that preliminary test rates a material as "safe" when in fact it is not, then the user who is not completely aware of the development status of the test may be mislead with expensive consequences. Moreover, as will be brought out in discussing the LOX test development, such an approach tends to result in the accumulation of preliminary data on a large number of materials before the test system evaluation is started. Then, if the test system evaluation is conducted at all, it will inevitably show that the necessarily arbitrary procedures and equipment designs for the preliminary data gathering phase are in need of change. These indicated changes, however, will probably not be made, because it will then be realized that this would result in the possible negation of the large amount of data already in hand. The usual result is that the original test system design is eventually standardized, and those vagaries that happen to be associated with the original system are perpetuated.

The next section discusses the three investigative areas in which SwRI provided consultative services during this program. Following these discussions are the conclusions drawn about program progress in these areas and recommendations concerning the direction of future work.

II. AREAS OF CONSULTATION

The consultative services provided by SwRI during the course of this program were:

(1) A critical evaluation of background information available to the GOX impact test method development for TTB and an assessment of the expected problem areas.

(2) Technical consultation with WSTF throughout the contract period. This took the forms of providing WSTF with a source of background experience in the problems peculiar to impact sensitivity research (WSTF personnel had no prior experience in this area), recommendations on equipment design concepts, recommendations of appropriate approaches to test system evaluation, and analysis of the small amount of data generated by the end of the contract period.

(3) Assistance to Panel 8, High Pressure Oxygen Systems Survey, MSC Apollo 13 Investigation Team.

Work accomplished in these areas is described in the following paragraphs.

A. Pre-Program Status of Oxygen Impact Test Development

As indicated previously, almost no work had been done in GOX impact testing prior to this program. Consequently, not only have LOX impact test results been the only information available to designers, but, also, they have formed the only background information for guidance in developing a GOX impact test. Since the LOX test was developed many years ago and has produced voluminous amounts of data, it would seem that there would be no trouble in developing a GOX test along similar lines. However, it was also mentioned previously that there are still serious questions concerning the meaning of the data from the test in its most widely used form. Considering this, it is appropriate to discuss the LOX test in more detail, followed by an assessment of what little work has been done recently in GOX.

1. The LOX Impact Test

In the late 1950's, the Army Ballistic Missile Agency (ABMA)* devised a drop-weight impact tester and a test method for measuring impact sensitivity (Ref. 1). Over the years, considerable effort has been expended

---

*Now Marshall Space Flight Center, National Aeronautics and Space Administration.

in refinement and standardization of this technique, and these efforts have resulted in the adoption of standard impact sensitivity test methods in the forms of USAF Specification Bulletin 527 (Ref. 2) and NASA Specification MSFC-SPEC-106B (Ref. 3). Recently, the similar characteristics of these two methods have been combined in the ASTM Test Method D-2512 (Ref. 4). All of these test methods employ the same basic drop-weight type tester with only minor variations in test procedures and in the details of handling the test materials. In all instances, the test material is placed in an aluminum specimen cup in the presence of liquid oxygen, and upon the test material is placed a stainless steel striker pin. A plummet is released from a known height, and observations are made as to whether a reaction has or has not taken place. After each test drop, the sample remains are discarded, and a new sample is positioned for the next drop. In all three methods, the basic concept used is that of attempting to measure the "threshold value" of a material by finding the highest drop height at which the material will withstand 20 consecutive test drops without reaction. The threshold value thus obtained is used as the relative sensitivity rating for the material. In the Air Force and NASA methods, a variation of the threshold value technique is widely used to qualify materials for service. In this variation, 20 or more drops are made from a fixed height of 42 in. (or 43.3 in.), and the materials are accepted or rejected on a "go/no-go" basis. Clearly, for a material that yields no reactions in this test, its threshold value is, by definition, either 42 in. (or 43.3 in.) or higher.

Unfortunately, these methods have been plagued from their inception with rather poor repeatability and reproducibility, as is brought out in the precision statement (Table 1) published in ASTM Method D-2512.

TABLE 1

PRECISION STATEMENT OF ASTM TEST METHOD D-2512

Threshold value drop height (in.)	Repeatability, one operator and apparatus (in.)	Reproducibility, diff. operator and apparatus (in.)
24	17.0	40.8
15	10.6	25.2
6	4.3	10.1

Such figures show that the current test methods do not yield results sufficiently precise for reliable compatibility evaluations. In spite of its long history of errant behavior, however, this test method remains today as the primary method by which materials are chosen for NASA LOX systems.

Insofar as is known, all the LOX impact test facilities in the country, save one, are set up in adherence to this procedure.

The one test facility set up along different lines is at Southwest Research Institute. This facility was originally installed under Air Force contract in the late 1950's, and for some years was operated in accordance with the Bulletin 527 procedure mentioned previously. Generally, the research program followed during this period was similar to those of other organizations in the field: a systematic investigation of the effects of various equipment adjustments in an attempt to determine the cause or causes of the poor precision of the "threshold value" method. In the mid-1960's, however, both the Institute and the Air Force came to the conclusion that the prime source of trouble lay not in the equipment but in the procedure itself. This conclusion came as a result of reviewing impact sensitivity research in the field of condensed phase explosives and liquid monopropellants. It was learned that these people had at first followed the "threshold value" approach, but had long since abandoned this as unworkable from the standpoint of the statistical theory involved. The much more efficient replacement method involved measurement of the 50-50 "go/no-go" point, with fundamental principles of probability mathematics being applied in determining the S-curve from which to establish this point. Apparently, LOX impact sensitivity testing had run afoul of the same problem and could possibly benefit from the same solution. The Institute then set out, with Air Force approval, to attempt to apply these techniques.

It was only after a considerable period of "trial-and-error" redesign of the striker pin and specimen cup that the new technique could be used. Once operational, however, the new technique, which is known as the "up-and-down," or "Bruceton" method, proved to be quite efficient and very repeatable. Figures 1 and 2 present some typical results for various liquids and greases, ranging from common hydrocarbons to fluorochemicals.

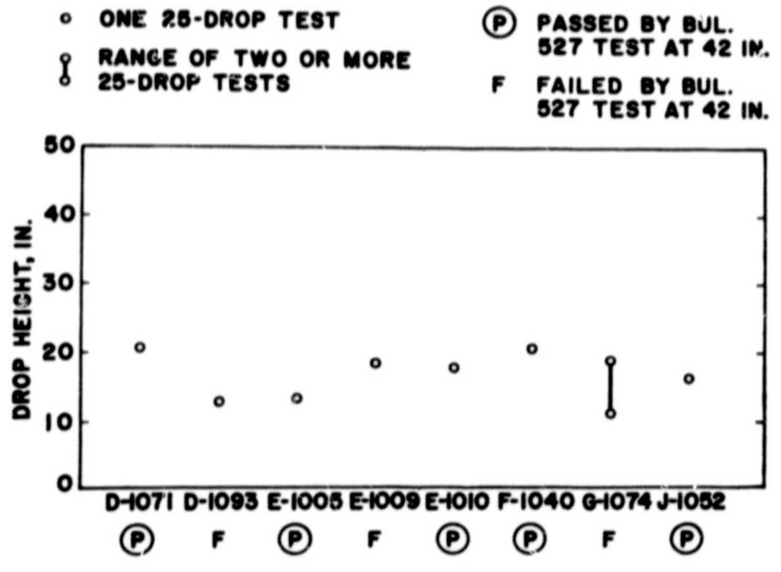

FIGURE 1. 50-PERCENT POINTS OF LIQUIDS

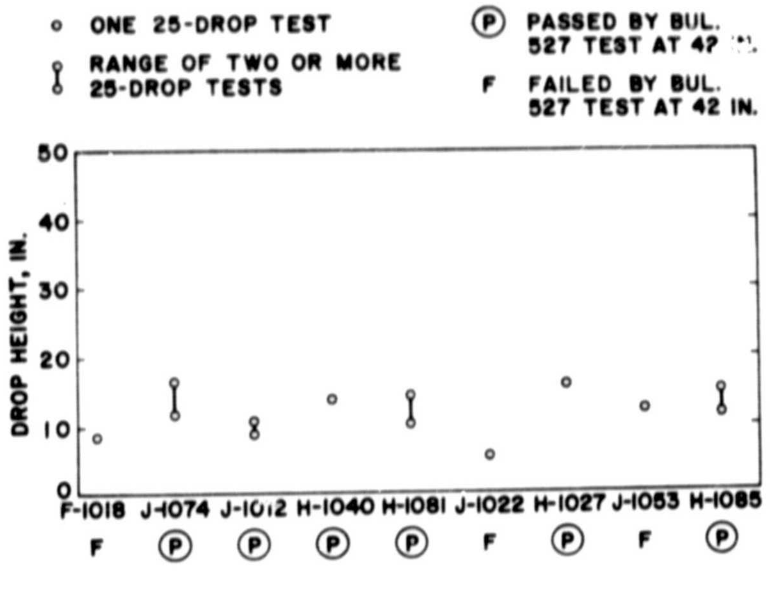

FIGURE 2. 50-PERCENT POINTS OF GREASES

In both figures, the highest and lowest ratings are shown for materials tested two or more times. The encircled letter "P" under the sample code indicates that the sample has passed the USAF Specification Bulletin 527 test at 42 in.; similarly, the letter "F" denotes that the sample has failed that test. Table 2 identifies the test material codes.

It was found that the precision with which sensitivity could be rated by the up-and-down test was much improved over that of the current test methods. However, with improved precision, it can also be seen that the sensitivities of most of these materials are so close together as to make significant distinction between them difficult. Moreover, just as one would suspect intuitively (from the types of materials tested), some of these were very violent reactors while others yielded at worst only small char marks--no flash, no sound. Clearly, the violent reactors would be more hazardous in a LOX system than would the mild reactors; yet, this intuitively obvious difference was not being defined quantitatively by sensitivity measurements alone. This, in turn, made it clear that the degree, or intensity, of reaction is not only a fundamental part of the full definition of hazard per se, but in some cases is the only property which can distinguish unambiguously between "good" and "bad" materials. Significantly, Brown, et al., independently and simultaneously arrived at the same conclusion with regard to the impact sensitivity testing of solid explosives (Ref. 5).

Considering the above, the next logical step seemed to be the development of some means for making quantitative measurements of reaction intensity during impact tests. The equipment subsequently developed for this purpose proved quite satisfactory, and the results of an extensive test program utilizing this apparatus were published in an Air Force Technical Report (Ref. 6) in December 1967, and in the ASLE Transactions in July 1968. The method also appears in the ASTM Standards, Part 18 (Second Edition), October 1969.

TABLE 2

TEST MATERIALS

Sample code	Sample type	Description
D-1071	Liquid	Heptacosafluorotributylamine
D-1093	Liquid	Silicone
E-1005	Liquid	Fluorosilicone
E-1009	Liquid	Chlorinated biphenyl
E-1010	Liquid	Fluorinated polymers of chlorotrifluoroethylene
F-1040	Liquid	A fluorochemical, composition unknown
G-1074	Liquid	Silicone
J-1050	Liquid	Mineral oil, MIL-L-6032, Grade 1100
J-1052	Liquid	A fluorochemical, composition unknown
J-1080	Liquid	Polyglycol
K-1081	Liquid	Different lot of J-1080
K-1082	Liquid	Different lot of E-1010
F-1018	Grease	MoS_2 and petroleum base oil
G-1051	Grease	Unknown
G-1052	Grease	Unknown
G-1071	Grease	Unknown
G-1077	Grease	Graphite in fluorocarbon oil
H-1012	Grease	MoS_2 in fluorocarbon oil
H-1013	Grease	Tetrafluoroethylene in fluorocarbon oil
H-1027	Grease	Unknown
H-1032	Grease	Chlorinated polymer of chlorotrifluoroethylene
H-1040	Grease	Graphite in chlorinated biphenyl
H-1081	Grease	Fluorosilicone
H-1085	Grease	Different lot of H-1032
J-1012	Grease	Graphite and chlorinated biphenyl
J-1022	Grease	Silicone
J-1053	Grease	Hydrocarbon
J-1074	Grease	Different lot of H-1032
J-1120	Grease	Unknown
K-1001	Grease	Fluorosilicone
K-1002	Grease	Different lot of H-1032
K-1015	Grease	Polymeric fluorinated oil with PTFE thickener
K-1022	Grease	Perfluorotrialkylamine with PTFE thickener
None	Solid	Acetate, clear
None	Solid	Phenolic, canvas base
None	Solid	Polyester
None	Solid	Polyimide
None	Solid	PTFE (polytetrafluoroethylene)
None	Solid	Vinyl

Briefly, the reaction intensity measuring system consists of a probe device which makes a relative measurement of the peak air shock pressure generated by a reaction. For such test drops, two such devices are located near the specimen cup at points 180° apart. The standard test procedure calls for 20 test drops from 43.3 in., and this yields a total of 40 peak pressure readings. The highest individual peak pressure reading of the 40 thus obtained is defined as the maximum peak pressure and is taken as the reaction intensity rating of the material under test.

Figure 3 presents the results of 69 different 20-drop reaction intensity tests on 23 liquids and greases. For each material, the two circles connected by a line show the range of maximum peak pressures (the maximum peak pressure is the highest individual peak pressure reading of the 40 such readings taken in a 20-drop test) obtained in all tests on that material. For example, one 20-drop test on H-1081 yielded a maximum peak pressure of 50 psi, another test gave 32 psi, while the remaining eight tests yielded maximum peak pressures somewhere between these two values.

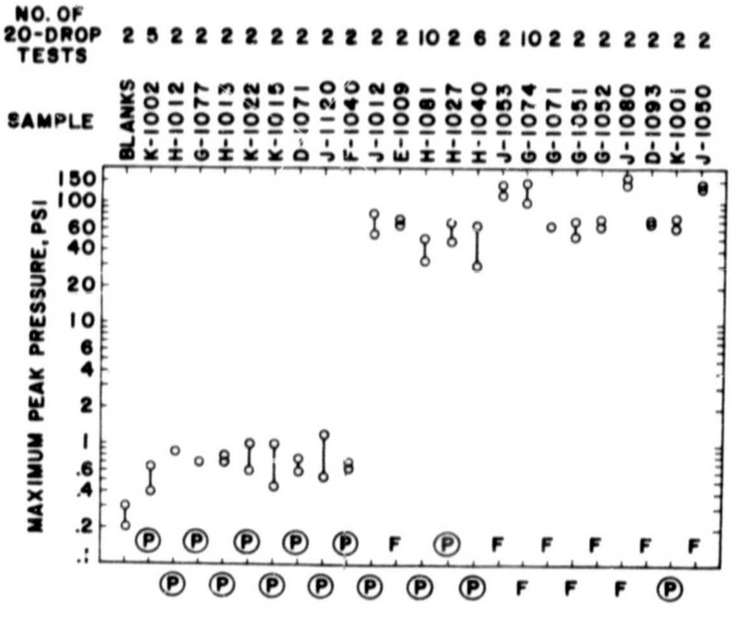

FIGURE 3. MAXIMUM PEAK PRESSURES OF VARIOUS LIQUIDS AND GREASES. LINE CONNECTING CIRCLES DENOTES THE RANGE OF MAXIMUM PEAK PRESSURES MEASURED IN THE APPLICABLE NUMBER OF TESTS

Before proceeding further with the discussion of these data, it is felt that some physical frame of reference for the various levels of peak pressure would be helpful. First, the maximum peak pressure readings obtained in two 20-drop tests on "blanks" (specimen cup with liquid oxygen but without any sample material) are included in Figure 3. These readings (0.2 to 0.3 psi) amount to background noise--the sound pressure generated by the steel-on-steel impact. Second, the readings from about 0.4 to about 2 psi are those of sounds only very slightly above the background noise. Toward the lower part of this range, these sounds are practically indistinguishable from the background noise. Toward the upper part, the sounds are distinct but still very low, and are usually not accompanied by any visible flash. Third, readings in the neighborhood of 5 to 9 psi are the same as those produced by "Chinese firecrackers" (approximately 3/16-in. diam. × 1.5-in. long) exploded individually in the specimen cup. Finally, readings of 50 to 70 psi are the same as those produced by "cherry bombs" (spherical firecrackers of about 1.0-in. diam.) exploded in the specimen cup.

With this background, the significance of the maximum peak pressure readings produced by the various materials can be evaluated. Most noteworthy is the separation of the 23 materials into two distinct groups: one with high maximum peak pressures and the other with low maximum peak pressures. Judging from the physical references discussed, it is not unreasonable to assume that the 30 to 160 psi reactions are dangerous explosions and that occurrence of such reactions in an oxidizer system would probably abort the mission of the system. Furthermore, those materials in Figure 3 that yielded such explosive reactions are obviously capable of doing the same thing in an oxidizer system if stimulated. Considering this together with the fact that the impact type of stimulus is very difficult to predict in a dynamic system, it appears that this group of materials should be classified as extremely hazardous and excluded from use in oxidizer systems. On the other hand, the reactions of the other group of materials in Figure 3 in no instance yielded peak pressures exceeding 1.2 psi; reactions of this order are so mild that their occurrence in a system would not appear likely to cause damage, or even be noticed. Therefore, this group could be classified as relatively inert.

It should be emphasized that the classifications of extremely hazardous or relatively inert developed above are based upon a premise radically different from that of the current test methods. The current test methods try to determine whether a material does or does not react with liquid oxygen under impact, with the object of accepting those which do not. This premise becomes untenable when liquids and greases of almost every conceivable structure show some type of evidence of reaction at almost the same stimulus levels. The object of the reaction intensity test, then, is to identify those materials which do not appear capable of reacting to an extent which would affect the system. This premise would also be difficult if one had to specify the exact level of intensity just sufficient to cause system damage. Fortunately, this problem is avoided in the case of liquids and greases, since the compatibility differences between the two groups are large and obvious.

Also notable is the fact that no less than five of the materials shown to be capable of powerful explosions in Figure 3 have previously passed the Bulletin 527 test at 42 inches. These are J-1012, H-1081, H-1027, H-1040, and K-1001. Considering that for the past several years, this test method and its very similar NASA counterpart, MSFC-SPEC-106B, have handled nearly all the acceptance testing in this area, it is a logical assumption that such hazardous materials may be in service at present.

Another very interesting aspect of material behavior uncovered by the reaction intensity test involves the material contamination problem. It has been generally accepted that minute quantities of a contaminant can render an ordinarily inert material impact sensitive. The data in Table 3, on the other hand, suggest a somewhat different interpretation.

TABLE 3

REACTION INTENSITIES OF MIXTURES OF TWO LIQUIDS

Sample	Avg. peak press. (psi)	Max. peak press. (psi)
K-1082	0.18	0.36
90% K-1082, 10% K-1081	0.20	0.54
	0.27	0.65
	0.21	0.40
	0.27	0.65
	0.24	0.40
80% K-1082, 20% K-1081	0.17	0.25
	0.25	0.35
	0.24	0.55
	0.22	0.75
	0.48	2.25
70% K-1082, 30% K-1081	0.49	6.8
	0.18	2.5
	0.23	1.5
	0.35	5.5
	1.33	21.0
50% K-1082, 50% K-1081	6.3	46.0
30% K-1082, 70% K-1081	38.0	100.0
K-1081	48.0	124.0

This table presents the results of reaction intensity tests on various mixtures of a violently explosive liquid K-1081 and a relatively inert liquid K-1082. In considering these results, it should be recalled that maximum peak pressures up to about 2.0 psi are exceedingly mild. With this in mind, it can be seen that a really significant reaction did not occur until the proportion of the explosive liquid was increased to 20 percent. Even then, only one such reaction, and it was of low order, occurred in five 20-drop tests. Further, it was necessary to increase the proportion of the explosive liquid to 50 percent before highly explosive reactions became evident.

It is apparent that this particular case presented a hazardous situation only when the amount of explosive material far exceeded what might be considered a trace contaminant. Now, there is no intended implication that reasonable efforts to avoid contamination are not needed; rather, it is emphasized that an explosive reaction obtained with a supposedly inert material in a test or service situation cannot necessarily be blamed on the presence of trace contaminants. Indeed, considering the previously discussed performance of the current test methods, the prime suspect in such a case should be the supposedly inert material.

Considerably more discussion could be devoted to the problem areas uncovered during more than ten years of impact testing in LOX. It is felt, however, that the foregoing provides an adequate picture of the "state-of-the-art" in this test and of the difficulties that are as yet unresolved. It is also worthy of mention at this time that many of these difficulties are conceptual in nature rather than mechanical, and that we can thus expect some familiar situations to develop in the GOX program.

2. The GOX Impact Test

In contrast to the situation of the LOX test, early experiments with GOX 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 GOX tests have to take into account two additional and significant environmental variables not faced in the LOX test: initial pressure and initial temperature.

As mentioned previously, very little work has been done in the area of impact testing of materials in GOX environments. In fact, outside of the small amount gathered to date in the subject program, there appears to be no data at all at pressures greater than 50 psi. Low-pressure data are also quite limited, with the only significant amount being that gathered in a recently completed project at Marshall Space Flight Center. The equipment used for this work was capable of pressures to 50 psi and consisted of the basic ABMA LOX impact tester along with a modified anvil region assembly. The test procedure used was essentially that of MSFC-SPEC-106B; namely, obtaining the reaction frequency in 20 drops of a 20-lb plummet from 43.3 inches. Considerable data were gathered in this manner, and it was stated that these data showed a good general correlation with LOX impact data. From this, it was concluded that the 106B procedure would continue as MSFC's method for choosing materials for both LOX and GOX service.

The MSC-WSTF program is of course concerned with systems operating at considerably higher pressures. Considering this together with the previously discussed problems with the LOX test, it was concluded that there was a definite need for high-pressure impact response data that would not be served by the 106B test.

One attractive feature of the MSFC equipment is the apparent capability for generating relatively large amounts of data in a short time. If the current program should indicate that impact test results at high pressures relate in a predictable manner to those at lower pressures, then this approach might prove useful for qualification and/or verification test procedures.

B. Assistance to the Experimental Program

As mentioned previously, a major reason for the equipment transfer was to seek a more rapid development program consummation by simultaneous development of several test system concepts. The E & D test system, of course, was already in an operational status but it was reasoned that the alternate concepts could be designed and built while the E & D system was being transferred. As of this writing, the E & D system transfer had been completed; however, no further testing had been accomplished, though one alternate system had been built and used in a limited amount of exploratory testing. Data from these tests were used for guidance in designing and building a second alternate system, which was put in operation in April 1970.

A detailed description of these systems would constitute a lengthy paper in itself. Therefore, we will atempt here to describe only the basic concepts being pursued. Essentially, the two alternate mechanical impact test systems under development differ from the E & D test system only in that the drop-weight apparatus is external to the high-pressure sample chamber. This of course introduces a pressure sealing problem at the striker pin together with the question of the additional impact resistance posed by the sample chamber pressure on the striker pin. Both the alternate systems attack these problems by utilizing a secondary pressure chamber around a collar on the upper portion of the striker pin. The secondary pressure chamber is pressurized with nitrogen, exerting a downward force on the striker pin that counterbalances the upward force from the oxygen pressure chamber. The second alternate system differs from the first primarily in that it is designed for quick disassembly, which in turn allows a much improved test output of 5 or more test drops per hr. A more subtle difference concerns the amount of impact energy used and the manner in which the sample material is confined; the significance of this will be discussed later.

At the outset, it was decided that SwRI's role in this work would be to serve as a technical consultant to offset the relative inexperience of the WSTF staff in the area of impact sensitivity testing. This was effected by three different trips to WSTF and numerous telephone contacts. The discussions involved mainly the statistical techniques to be used in developing the test procedure, and the equipment design features considered necessary in the light of the probable applicability of the "hot spot" theory of impact-induced ignition.

It might seem at first glance that the evaluation of the new test systems would be a relatively straightforward procedure, but, unfortunately, there are a number of well-hidden "pitfalls" here. As an illustration, consider the apparently simple matter of choosing the appropriate impact energy capabilities for the new systems. It would seem logical to start with some arbitrary figure such as, say, 70 ft-lb, and then determine whether this energy level was sufficient to react the various materials already well known to be reactive (for example, automotive engine oil). If not, then other energy levels would be tried. Once the appropriate energy level was found, then materials would be rated as "safe" or "unsafe" in accordance with their response at this level. Now, as stated previously, this appears to be a reasonable approach, but the "pitfall" lies in the assumption that a 70 ft-lb impact gives the same order of magnitude of ignition stimulus in the service system as in the test system. Just how much in error this assumption can be may be demonstrated in the LOX impact test with any of several materials. One material, for example, can be made to explode violently on every trial at 35 ft-lb, or to be inert in every trial at 70 ft-lb, with the type of response being dependent on the configuration of the striker pin and specimen cup. Further evidence of this is afforded by preliminary data from the two alternate GOX test systems. In tests at 10 ft-lb on the same materials, one system yields significantly greater sensitivity ratings.

The probable reasons for the above may be found by examination of the generally accepted theory of the impact ignition mechanism. This theory holds that impact produces ignition through concentration of energy into hot spots; not through general compression of the bulk sample material. Since much of the total impact energy delivered does, however, go into deformation of the sample, sample cup, etc., it follows that only a portion shows up at a hot spot site as ignition stimulus. From this, it can be seen that the amount of actual ignition stimulus delivered depends not only upon the gross impact stimulus applied, but also on how efficiently that stimulus is localized into hot spots. This, in turn, is a function of test method, specimen cup, striker pin, compression, state of the substance tested, and so on. Clearly, then, it can be very dangerous to conclude from an impact test that a certain material is safe at so many ft-lb of impact--in the service system, a much smaller energy input might cause ignition through better localization. In other words: impact sensitivity is a highly system-dependent phenomena.

There are of course other problems to be faced in the test system evaluation, but most are connected to some degree with the system dependence aspect. To combat this, considerable attention was paid to the formulation of an evaluation plan. A general description of this plan, along with the reasoning involved, is presented in the following paragraphs.

1. Selection of Reference Materials

In order to expedite the evaluation procedures, one should initially use a group of test materials which have already been reasonably well characterized from previous LOX and/or GOX. These materials should be readily available, and should include types expected to be highly reactive, marginal, and relatively inert from each of the categories of solids, liquids, and semi-solids (greases). A list of materials tentatively selected is presented in Table 4.

TABLE 4

TENTATIVE LIST OF TEST MATERIALS SYSTEM EVALUATION PROGRAM

Material Type	Expected Degree of Reactivity	Material
Solid	Reactive	Acetate, Nylon
	Marginal	Mylar
	Inert	Teflon, Kel F
Liquid	Reactive	Mineral Oil
	Marginal	Versilube, FS-1265, Aroclor 1254
	Inert	Krytox, Halocarbon 411V
Grease	Reactive	Hydrocarbon Grease
	Marginal	FS-1281
	Inert	Krytox

2. Verification of Test System Response Ranges

One should begin testing with each system using one of the materials expected to be highly reactive, along with the maximum design impact capability.

The reason for this approach is that, as stated previously, the "effective" input stimulus is known from past experience to be quite sensitive to the configuration of the striker pin and specimen cup holder. The magnitude of the sensitivity, however, is not well known; hence, we have not available "a priori" estimate of what this effect will be in the new systems. On the other hand, it is known that the difference between the design input and the "effective" input can be large, and it is quite possible that the initial system designs may be unable to produce reactions on even the most reactive materials. Obviously, such a system would be of limited value for measuring relative material reactivities; a redesign of the striker pin and specimen holder would be in order. Thus the system evaluations can be expected to be iterative procedures involving perhaps a series of test-redesign sequences.

3. Definition of Response Distributions

The most accurate method of defining the impact sensitivity of a material is to subject the material to a large number of impacts at each of several drop heights. The reaction frequencies obtained are then plotted as an estimate of the response distribution. Such a procedure is not practical, however, for day-to-day testing; it is too costly and too slow. To circumvent this, several "short-cut" procedures have been developed, of which the 50-percent point test is the most accurate with a limited number of test drops. Like any other "short-cut" procedure, however, this procedure cannot be used with confidence without at least a general idea of the response distribution for the material being tested. Therefore, the development of any impact test procedure must proceed at first with the more cumbersome reaction frequency plotting on materials thought to be representative of the "spectrum" of materials which will eventually be tested. The "short-cut" procedure is brought in when judgment and experience dictate that an adequate estimate of the "spectrum" behavior has been obtained.

4. Selection of Test Procedures

At this point, about all that can be said about the final test procedures and equipment is that both impact sensitivity and reaction intensity will have to be taken into account. Preliminary data taken with the E & D test system indicate that materials which normally explode in LOX tend to burn at a considerably slower rate in GOX; this may necessitate a different approach to intensity measurement than was used with LOX. Beyond this, any further speculation is considered to be decidedly premature.

As noted in Section I, all system evaluation work has been postponed in order to "qualify" some of the materials being used in the Apollo program. The test procedure chosen arbitrarily for this work involves a 7-1/2-lb plummet dropped from 16 in., using the second alternate test system mentioned previously. The test samples are 1/4 in. in diam. The pressure, rather than the drop height, is being varied, starting with four test drops at 2000 psi. If a reaction is observed, the pressure is lowered and four more test drops are made. A considerable amount of data has been generated in this work. However, with the statistical inadequacies of this approach, and without a system evaluation background, it is difficult to derive meaningful judgments of the materials from such data.

C. Assistance in Apollo 13 Investigation

In late April, SwRI was asked to provide assistance to Panel 8, High Pressure Oxygen Systems Survey, MSC Apollo 13 Investigation Team. The Panel met in Houston during the week of April 27, for the purpose of conducting a comprehensive survey of the state-of-the-art in aircraft and commercial oxygen system design. Particular emphasis was placed on techniques, standards, and criteria used to minimize fire and explosion hazards. The final report on the Panel findings was distributed in May.

In addition to participation in the Panel investigations, SwRI was asked to perform an assessment of 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. The document ensuing from this effort was distributed in June in the form of an addendum to the final report of the Panel.

III. CONCLUSIONS AND RECOMMENDATIONS

It can be seen from the foregoing discussions that the development of GOX impact test is still in a very early stage. Hence, it is premature to attempt to draw any conclusions at this time concerning the probability of success of the effort. On the other hand, the small amount of data obtained thus far is encouraging in that the more efficient test system being developed is producing results roughly comparable with those from the first-generation system (the E&D tester).

In all candor, however, it must be concluded that the decision to postpone a careful evaluation of the test system in favor of immediate use of the equipment for "qualification" tests was unwise. As shown herein, the data from such tests can at best be of only limited usefulness, and it is felt that the net result of this approach has been a lengthy delay in the achievement of a reliable GOX impact test. It is recommended that the evaluation phase be resumed as soon as possible.

LIST OF REFERENCES

1. Lucas, W. R., and Riehl, W. A., "An Instrument for the Determination of Impact Sensitivity of Materials in Contact with Liquid Oxygen," ABMA Rept. DSN-TN-2-58, October 15, 1958.

2. Liquid Oxygen Compatibility Impact Sensitivity Test Method, USAF Specification Bulletin 527, May 1, 1961.

3. Testing Compatibility of Materials for Liquid Oxygen Systems, NASA Specification MSFC-SPEC-106B, September 16, 1966.

4. Method of Test for Compatibility of Materials with Liquid Oxygen (Impact Sensitivity Threshold Technique), ASTM Designation D2512, 1966.

5. Brown, J. A., Garabrant, A. R., Coburn, J. F., et al., Esco Research and Engineering Co., "Sensitivity and Rheology Studies on Lead Azide-RDX-Silica Mixtures," Final Technical Report No. GR-1SRS-69 for U. S. Army Picatinny Arsenal, January 1969.

6. Blackstone, W. R., Baber, B. B., and Ku, P. M., "Development of New Test Techniques for Determining the Compatibility of Materials with Liquid Oxygen under Impact," AFAPL Tech. Rept. 67-41, December 1967.