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This report covers work conducted from November 1959 to February 1961.

This report has been preceded by a series of four Quarterly Technical Summary Reports which described the objectives and approaches to the experimental program and presented the interim results.

The principal scientists working on the project at Pennsalt were Mr. Henry C. Miller, Group Leader, and Mr. John C. Grigger, Senior Research Chemist. They were assisted by Mr. Stanley Yoslov, Technician. Acknowledgment is made of the analytical services provided by the Analytical Department under the direction of Dr. Paul A. Munter. This report was prepared by Messrs. Grigger and Miller and approved by Dr. G. Barth-Wehrenalp, Director of Inorganic Research.

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ABSTRACT


Compatibility and corrosion rates of alloys of aluminum, copper, magnesium, nickel, titanium, steel and stainless steel, and columbium, molybdenum, carbon, graphite and fluorocarbon plastics in chlorine trifluoride, perchloryl fluoride and mixtures of these at 30°C. were investigated. Titanium, columbium, molybdenum, carbon and graphite were rapidly attacked in ClF_3 . Corrosion rates of others were extremely low in all liquids. In the vapors, instances of higher corrosion rates were noted. Teflon and Kel-F adsorbed moderate amounts of ClF_3 and ClO_3F . Passivation by ClF_3 was unnecessary for reducing corrosion of properly cleaned metals. Corrosion in wet ClO_3F was characterized by localized attack, but some stainless steels were resistant.

Titanium exhibited increasing impact ignition in liquid ClO_3F beginning at 19 ft.-lbs., but even at 140 ft.-lbs. burning was not sustained. No other metals showed impact ignition in ClF_3 or ClO_3F . In explosive shock tests, ClO_3F gave a stronger interaction with the metals tested than did ClF_3 , and aluminum showed a greater interaction with the fluorine chemicals than low carbon or stainless steel. Greatest enhancement of explosive shock occurred with titanium and ClO_3F . In explosive denting and perforation of steel and aluminum cylinders containing ClF_3 , ClO_3F and their mixtures, no enhancement occurred. A high order explosive interaction occurred between ClO_3F and titanium cylinders perforated by a shaped explosive charge.

PUBLICATION REVIEW

This report has been reviewed and is approved.

FOR THE COMMANDER:


I. PERLMUTTER
Chief, Physical Metallurgy Branch
Metals and Ceramics Laboratory
Materials Central

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I. INTRODUCTION

The importance of storable liquid oxidizers for missile use is well recognized, and the compatibility and handling characteristics of the low-cost, medium-performance, liquid oxidizers like nitric acid, dinitrogen tetroxide, and hydrogen peroxide have been thoroughly studied, but the compatibility of the high-performance, storable, liquid oxidizers - chlorine trifluoride, perchloryl fluoride and mixtures of these- had not been studied in detail prior to this contract.

Chlorine trifluoride is a very reactive substance. It is incendiary to most materials, including surface-contaminated metals, nearly all organic materials, and even silicate compositions like asbestos. The handling procedures used for this very reactive oxidizer until now have been developed by exploiting the chemical similarity of chlorine trifluoride to liquid fluorine. This has resulted in the design of apparatus with a high safety factor and in no way gives adequate information for the development of procedures and materials for handling chlorine trifluoride to the limit needed in modern large-scale rocketry.

Perchloryl fluoride is a much less reactive material than chlorine trifluoride. Chemically it behaves much like liquid oxygen, and it can be handled as a liquid at atmospheric temperatures, where it exhibits a vapor pressure of about 150 lbs. psig. Practical handling procedures for perchloryl fluoride have been developed from laboratory and semi-works experience with this material, and from its similarity to liquid oxygen. Before operational use of perchloryl fluoride in rocketry could be undertaken, however, compatibility data of this oxidizer under extreme static and dynamic conditions were desirable.

Recently, considerable interest has been shown in mixtures of chlorine trifluoride and perchloryl fluoride as storable liquid oxidizers. Experience with these mixtures was limited to laboratory demonstration of chemical compatibility of the two compounds, and to measurements of some of the physical and chemical properties of the mixed liquids. The compatibility of these mixtures with materials of construction used in rocket design had not been studied.

The object of this project was to develop information on the compatibility, including quantitative corrosion rates, of metals, carbon, graphite and plastic materials with chlorine trifluoride, perchloryl fluoride, and mixtures of these to the limit needed in modern large-scale rocketry.

In order to prepare and safely use liquid chlorine trifluoride-perchloryl fluoride mixtures, vapor pressure, density and liquid-vapor equilibrium measurements were made on the system. Preliminary compatibility tests under maximum

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safe conditions were carried out in liquid ClF_3 , ClO_3F and the 25% ClO_3F -75% ClF_3 mixture on several metals, carbon, graphite and plastics with which there was no experience or which were suspected as being reactive.

A large part of the experimental effort was given to 21-day static immersion tests at 30°C . in the anhydrous liquids - ClF_3 , ClO_3F and 25% ClO_3F -75% ClF_3 . Yearly corrosion rates were calculated for these unstressed test materials which included alloys of aluminum, copper, magnesium, nickel, titanium (ClO_3F alone), steel and stainless steel as well as Teflon and Kel-F plastics. Stressed specimens of eight selected alloys exposed under the same conditions were observed for possible stress corrosion. Corrosion rates resulting from exposure at 30°C . to the vapors of ClF_3 and ClO_3F of ten selected alloys in unstressed condition were also determined. An evaluation of passivation and preservation by chlorine trifluoride was made by a cyclic exposure of twelve selected alloys to chlorine trifluoride and laboratory atmosphere.

Previous experience indicated that moisture at 100 ppm. or higher can result in severe and localized attack in perchloryl fluoride. Therefore, 21-day immersion tests at 30°C . in both the liquid and vapor of wet perchloryl fluoride were completed for 30 alloys. The average corrosion rates which were calculated were hedged by the pitting attack observed in many instances.

The above static tests were complemented by a series of dynamic tests which included impact tests, cylinder denting and rupturing tests, and high explosive shock tests. These dynamic, drastic exposure tests were made on several metals of greatest practical interest including low carbon steel, stainless steel, aluminum and titanium (ClO_3F alone).

An evaluation of corrosion measurement by the electrical resistance method revealed that probe units have not yet been developed which can safely be used in contact with chlorine trifluoride.

A discussion of corrosion mechanism in chlorine trifluoride is presented based on experience and literature information. A critical evaluation is made of the unclassified literature survey on compatibility of materials with chlorine trifluoride and perchloryl fluoride, which was prepared for this project.

II. EXPERIMENTAL WORK AND RESULTS

1. Test Chemicals

All chlorine trifluoride used in this work was first qualitatively checked to have its vapor pressure correspond closely to that (15.3 psig at 30°C.) reported in the literature.⁽¹⁾ Cylinders with higher pressure, indicating a possible chlorine or chlorine monofluoride contamination, were vented to the normal pressure. The purity of the ClF₃ was then checked by infrared analysis using metal cells with barium fluoride or silver sulfide coated silver chloride windows for gas sampling. Normally only trace amounts of ClO₂, ClO₂F, CO₂ and CF₄ were found by infrared, and only occasionally were trace amounts of HF noted. To ensure freedom from HF, all ClF₃ was scrubbed free of HF before use by being vaporized through a tower of sodium fluoride pellets.

Pennsalt perchloryl fluoride (PF*) was used in all compatibility tests both in mixtures with ClF₃ and alone. Previous experience indicated that moisture content is an important factor in corrosion by ClO₃F. Therefore, the moisture content was precisely determined by the P₂O₅ method.** Quantitative analysis of the ClO₃F was performed by gas chromatographic and infrared techniques. In addition, tests were made for chlorine and easily reducible chlorine compounds by bubbling the ClO₃F through a water solution of silver nitrate containing hydrogen peroxide at 150 ml./min. for five minutes, and no AgCl precipitate in this time was taken as indicating no such compounds. Analyses on the two 100-lb. cylinders of ClO₃F used were (all % as vol. %):
(a) 14.2 ppm H₂O, 0% CO₂, 0.17% Air, 99.83% ClO₃F, no other reactive Cl;
(b) 9.9 ppm H₂O, 0% CO₂, 0.25% Air, 99.75% ClO₃F, no other reactive Cl.

2. Physical Measurements in ClO₃F-ClF₃ System

Vapor Pressure Measurements: In order to prepare and safely use liquid ClO₃F-ClF₃ mixtures, it was necessary to determine their vapor pressure and density properties to a practical degree of precision.

Solutions of varying concentrations were prepared by mixing precisely weighed amounts of ClF₃ and ClO₃F in steel pressure cylinders. In these measurements the vapor-to-liquid ratios were kept small, so that the amount of material in the gas phase would represent a small part of the total charge. This eliminated the necessity for making a correction in the concentration of the liquid because of material loss to the vapor phase.

*Trade mark - Pennsalt Chemicals Corporation,

**As described in Pennsalt procedure for moisture in Isotron specification tests (Pennsalt Standard TI-1) with equipment modification to eliminate possible reaction with ClO₃F.

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All weights were known to better than 0.5 per cent, the temperatures were known to $\pm 0.2^{\circ}\text{C}$., while pressures were measured to ± 3 psi. It is believed that the vapor pressures are good to ± 4 psi. More reliance could be placed on these data if more compositions were measured and if duplicate measurements were run. However, these data were more than adequate for our needs.

The results of the measurements are shown in Table 1. The vapor pressures at even temperature intervals and at even compositions as determined from a plot of the above data are given in Table 2.

These measurements confirmed the previously reported observation that $\text{ClO}_3\text{F}-\text{ClF}_3$ mixtures form a single phase system with no compound formation, and that the vapor pressure of the mixture has a large positive deviation from Raoult's Law.

Density Measurements: The density of mixtures of $\text{ClO}_3\text{F}-\text{ClF}_3$ was determined by filling an evacuated calibrated volume by direct liquid transfer, and then transferring this mixture into a small weighing tube by a vacuum distillation technique. Densities measured at one temperature of the two mixtures 25% and 75% ClO_3F , checked closely those calculated from the densities of the pure materials using the method of additive volumes. Calculated densities from $20-30^{\circ}\text{C}$. for the 25% and 75% ClO_3F mixtures together with the measured values are listed in Table 3.

Liquid-Vapor Diagram for $\text{ClO}_3\text{F}-\text{ClF}_3$ System: In the preparation of liquid $\text{ClO}_3\text{F}-\text{ClF}_3$ mixtures for corrosion and other tests, it was necessary to have some knowledge of liquid-vapor equilibrium compositions. To provide this information a series of five determinations was made as listed in Table 4, from which a liquid-vapor diagram (Figure 1) of sufficient accuracy for this research could be drawn. The diagram shows a large positive deviation from an ideal solution with respect to ClO_3F .

The five liquid mixtures were prepared in steel pressure cylinders by weight with care taken to leave only a relatively small vapor volume. In three cases, analyses of the liquid and vapor were made. Since the liquid analyses checked the make-up composition, only vapor analyses were made on the remaining mixtures.

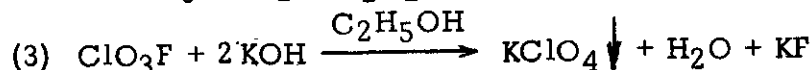
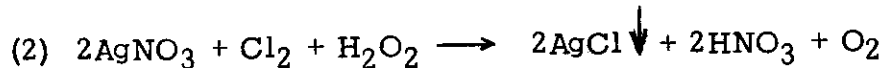
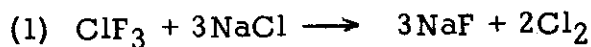
Each $\text{ClO}_3\text{F}-\text{ClF}_3$ mixture was passed as a vapor through an absorption train. This consisted, in order, of:

- (1) 1/4 -inch S.S. tube packed with 14-20 mesh sodium chloride crystals, which was heated and purged with nitrogen prior to use.

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- (2) two bubblers, one open end and one fritted, charged with acidic silver nitrate solution containing hydrogen peroxide.
- (3) two open end bubblers charged with 5% alcoholic KOH.

The analytical procedure is based on the following over-all reactions in the absorption train:



The chlorine absorbed in the silver nitrate bubblers is indirectly determined by the Volhard method of titration for silver remaining in solution. Absorbed perchloryl fluoride is determined by an acid titration of the KOH bubbler solutions.

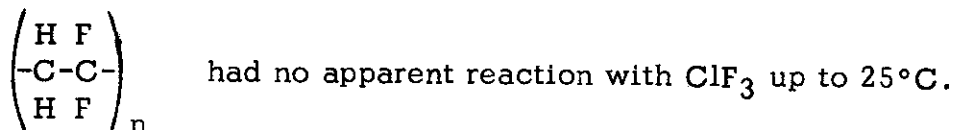
3. Preliminary Compatibility Tests

Test materials with which there was no experience or which were suspected as being reactive were first individually contacted with ClF_3 and ClO_3F liquid under controlled temperature and pressure behind a safety barricade. Fractional gram test pieces were used. For ClF_3 contact the test piece was held in a 3/4" by 6" Kel-F trap which was connected to a metal vacuum manifold system. The Kel-F traps were unaffected in these test exposures. For contact with 25% ClO_3F -75% ClF_3 mixture or pure ClO_3F a copper trap was used. Sufficient ClF_3 , ClO_3F or mixture was charged as a gas to the evacuated trap cooled by liquid nitrogen or dry ice-trichloroethylene mixture to completely immerse the test piece in condensed liquid. After charging, the cold bath was removed. Observations were made in the Kel-F trap on the reaction and change occurring during the period of warming to room temperature, but in the copper trap only after holding about one hour at 25°C. and venting. All test materials were recovered for observation and reweighing if still in compact form.

As noted in Section A of Table 5, all titanium alloys as well as molybdenum are rapidly dissolved in ClF_3 below 25°C. Columbium metal reacts with incendiary violence on contacting ClF_3 , even at dry ice temperature. Of all carbons and graphites exposed, only Graphitar 39 exhibited reasonable resistance to disintegration and powdering up to 25°C. Even here there was apparently some adsorption of ClF_3 . These results provide striking examples where liquid ClF_3 is more active than either liquid or gaseous fluorine. The rapid reduction of massive samples of both graphite and carbon to a finely

divided powder by liquid ClF_3 is comparable to the known action of fluorine in forming interstitial compounds with graphite and under some conditions with carbon at temperatures below the ignition point. However, the rate of attack by fluorine is not rapid under these conditions and many forms of carbon show no action at all.

Vinylidene fluoride resin, Kynar, * with the structure



Adsorption of ClF_3 is suspected, since the exposed sample fused during oven drying considerably below its crystal melting point. Use of Kynar for exposure to chlorine trifluoride is not recommended without further study because of the favorable free energy for the reaction with carbon and hydrogen in the plastic molecule to form hydrogen fluoride and carbon tetrafluoride.

Data in Section B of Table 5 show that the 25% ClO_3F -75% ClF_3 liquid mixture is about as effective as pure ClF_3 in completely dissolving titanium, columbium, and molybdenum. The attack on carbon and graphite was slower and possibly less drastic in its powdering action; however, complete failure by fracture into smaller pieces still occurred within two to three hours.

A 2 mil thick Teflon tape was used in place of white lead dope as thread lubricant in metal connections on test equipment and cylinder valves. The likelihood of firing of this thin gauge Teflon if accidentally exposed within a pipe or vessel holding ClF_3 or ClO_3F was checked. It was found that exposure to either liquid at 25°C . had no effect on the Teflon tape (Sections B and C, Table 5).

A few selected materials including Teflon, CaF_2 -filled Teflon, chromium, aluminum, magnesium and lead were additionally exposed to liquid ClF_3 in a stainless steel cylinder at up to 80°C . During the warming period from the dry ice charging temperature, the cylinder was checked for any abnormalities in pressure or local autonomous heating as an indication of a possible rapid sample failure. As shown in Table 6 only silver and lead underwent a moderate surface reaction.

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4. Immersion Tests of Unstressed and Stressed Specimens in Anhydrous Liquids

Test Pieces: The procedure used in preparation of test specimens for immersion testing is outlined in Exhibit 1, and a description of the standard unstressed test piece is given in Figure 2.

A complete test materials listing which includes identification, temper or grade, composition, density and supplier is given in Table 7.

Information on the size and the shaping of the standard stressed test specimen together with a listing of the eight alloys selected for stress corrosion testing is included in Exhibit 2.

Test Facilities and Procedures: Unstressed test specimens were held in a rack assembly by being gripped against their ends in C-clamp type holders, with each specimen insulated from its steel holder by two CaF_2 -filled Teflon inserts in the holder jaws. Stressed specimens were supported in the holders by bolting the end of the stressing bolt on each specimen to the holder. Each assembly holding 12 unstressed or 8 stressed test pieces was placed in a flanged, steel test tank (6-3/4" O.D. x 7" H.) fitted with a bolted lid and sealed with a Flexitallic gasket formed of 304 stainless steel and Teflon. Each tank lid was suitably valved to a metal manifold and also fitted with a frangible disc safety head set at 125 or 250 pounds bursting pressure. All safety heads were piped to an outdoor forced-air vent. Four test tanks were housed in each of three isolation cabinets to provide a test capacity of 144 specimens. The cabinets (52" L. x 16" W. x 22" H.), formed of aluminum sheet with acrylic plastic front windows, were each furnished with a 500-Watt strip heater and an air-circulating blower, and were thermostatted to $30 \pm 2^\circ\text{C}$. All vacuum pumps used on lines and equipment handling ClF_3 or ClO_3F were doubly protected by a liquid nitrogen chilled glass trap and a metal trap packed with a 1:1 mixture of 4-8 mesh soda lime and rock salt.

A test liquid charge was made by vaporizing the liquid (ClF_3 , ClO_3F or mixture) from a cylinder held on a scale and connected through a flexible joint to an evacuated test tank chilled in a dry ice-trichloroethylene bath. The test tank was previously loaded with the test pieces and mounted in the isolation cabinet. When the charge, sufficient to completely immerse all test pieces in liquid at 30°C . was completed, the tank was pumped on briefly to remove possible non-condensibles. Then the tank was closed off, cold bath removed and the cabinet brought up to the 30°C . test temperature for the test period, usually 21 days. The pressure in each test tank was checked at frequent intervals during the test period.

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Special techniques used in the preparation and charging of the 25% ClO_3F -75% ClF_3 liquid mixture are outlined in Exhibit 3. The step by step procedure used at the end of each exposure period for discharging the test tanks and handling the test pieces to obtain suitable corrosion data are given in Exhibit 4. This handling procedure was designed to avoid adventitious corrosion after the end of the formal test period.

Data Processing: Corrosion rates expressed as mils./yr. were calculated by the formula set up in Exhibit 5. The successive steps followed in converting the raw corrosion data into figures usable for corrosion rate calculation are outlined in Exhibit 6. Observed weight gains were assumed to be fluoride in the case of metals exposed to ClF_3 and ClO_3F - ClF_3 mixtures, and oxide for metals exposed to pure ClO_3F . Factors for converting these weight gains to equivalent weights of reacted metal were calculated for each of the alloys tested and are listed in Table 8. The procedure used in calculating these weight gain factors is outlined as a footnote to Table 8.

Two corrosion rate figures are presented as noted in the outline of Exhibit 6 and in the tabulation of corrosion rates for the 21-day immersion tests in Table 9. The first corrosion rate, C.R.', is based on the initial weight change observed immediately after exposure and drying of the test specimen. This would be the normally reported corrosion rate for the case where the reaction products are completely dissolved in the liquid corrosion medium. Because of the apparently adherent corrosion products or films resulting from exposure to ClF_3 and ClO_3F , a second or cumulative corrosion rate, C.R'', was calculated to include a correction for this adherent corrosion product as described above.

The precision of weighing and measurement permits reporting corrosion rates to 0.01 mils/yr. The results in Table 9 are reported to 0.01 mils/yr. to bring out a comparative difference among the various alloys and the several corrosion media, especially since the total corrosion rates obtained are all below 0.5 mils/yr. Nevertheless, it is recognized that in the extrapolation of 21-day data to yearly rates, reliable absolute corrosion rates should not be expected to a precision greater than about 0.1-0.2 mils/yr.

Results:

Unstressed metals: All unstressed metals exhibited very low corrosion rates at 30°C. in the two pure liquids, ClF_3 and ClO_3F , and in the liquid mixture 25% ClO_3F -75% ClF_3 . These corrosion rates (Table 9) are generally less than 0.2 mils/yr., with a maximum of 0.4 mils/yr. No evidence of pitting or other localized corrosion which could result in failure at low apparent corrosion rates was found under these conditions. These results confirm previous qualitative experience and show

that more drastic exposure conditions are needed to bring out differences in corrosion resistance.

Plastics: All fluorinated plastics showed weight gains on exposure to each of the three test liquids. The observed slow release of this adsorbed ClF_3 and ClO_3F could present a corrosion problem for equipment with Teflon or Kel-F packings, gaskets, etc., during standby or atmospheric exposure periods.

After removing the unstressed Teflon, calcium fluoride-filled Teflon, and Kel-F from the 21-day ClF_3 immersion tests and heating them at 135°C . in a nitrogen atmosphere for 45 minutes, they still carried a strong ClF_3 odor and etched the glass desiccator in which they were being stored. These samples (#1 and 2 of classes 5-1 to 5-3) were then held in the hot vapor over boiling trichloroethylene (87°C .) for 10 minutes and oven dried at 120°C . for 30 minutes in a further effort to release the ClF_3 . However, some odor still remained. On weighing these plastic pieces, weight gains of 1.02% for the Kel-F, 0.70% for the Teflon and 0.16% for the CaF_2 -filled Teflon were observed due to the ClF_3 exposure even after this vigorous treatment. There was no change in appearance of these plastics except for a partial bleaching of the tan color of the CaF_2 -filled Teflon.

By continued heating at 120°C ., an attempt was made to bring these plastics to constant weight. For the Teflon and CaF_2 -filled Teflon, constant weight was reached in 118.5 hours, and the final weights were essentially the original weights before ClF_3 exposure. In the same heating period, the Kel-F lost only 70% of its weight gain, and even after 888 hours at 120°C ., the Kel-F pieces retained 12% of their weight gain.

These plastics showed somewhat smaller weight gains on 21-day exposure to pure ClO_3F . After drying at 135°C . for 45 minutes, the weight gains were 0.81% for Kel-F, 0.32% for Teflon and 0.03% for CaF_2 -filled Teflon. However, drastic physical changes occurred during this heating. Large, permanent blisters were raised on both faces of the Teflon pieces. On the faces of the Kel-F, a clear, water-white glaze about 10 mils thick was formed. This glaze was smooth and hard at room temperature. The interior of the Kel-F turned a dense, opaque, milk-white color. There were also a few small blisters on the surface. The CaF_2 -filled Teflon was unchanged. No attempt was made to heat the PF-exposed plastics to constant weight.

The fluorinated plastics Kel-F and Teflon showed a moderate weight gain on exposure to the 25% ClO_3F -75% ClF_3 liquid mixture just as they did in the individual liquids. On drying at 135°C . for 45 minutes after removal from the test liquid and prior to weighing, the Kel-F was blistered and "crazed", with a water-white glaze of varying depth formed on the faces. The CaF_2 -filled Teflon showed only a small weight gain on exposure to the 25% ClO_3F -75% ClF_3 liquid. Polyvinylidene fluoride plastic, Kynar, on exposure to liquid perchloryl fluoride exhibited a weight gain about the same as Teflon, and less than Kel-F.

Several instances of complete failure or firing of CaF_2 -filled Teflon buttons used as insulators for metal corrosion specimens occurred during exposure to ClF_3 and ClF_3 - ClO_3F mixtures. It was surmised that the reaction was initiated by an impurity on the surface.

Stressed metals: The problem of stress corrosion cracking of metals in contact with ClF_3 , ClO_3F and mixtures of these does not appear to have been previously studied. The present, limited tests were designed to detect obvious susceptibility to stress corrosion of eight typical alloys chosen from the complete list of Table 7. The test metals are listed in Exhibit 2, together with information on sample size and shape, and methods used for detecting stress cracking after exposure.

Duplicate U-bend specimens of each of the eight alloys, after initial forming, were further deformed by a bolt across the ends to ensure stressing in the plastic range beyond the proportional limit and approaching the yield strength. These were exposed to liquid ClF_3 , ClO_3F and a 25% ClO_3F -75% ClF_3 mixture at 30°C . for 21 days in the same test tanks used for the unstressed specimens. No evidence of stress corrosion was found on any of the exposed metals either by visual examination or by the dye penetrant inspection procedure.

5. Immersion Tests of Unstressed Metals in Anhydrous Vapors

A 21-day exposure of ten selected alloys to ClF_3 and ClO_3F vapors at 30°C . was carried out using the same procedures and test equipment as for the liquid immersion tests previously reported.

For vapor exposure, each test tank containing mounted test pieces was evacuated and then charged to 5 psig with ClF_3 gas or to 100 psig with ClO_3F gas. Because of the small amount of tank charge (17 g. at 5 psig) in the case of ClF_3 gas, the charge was pumped out every five days and fresh ClF_3 gas immediately recharged to 5 psig. This procedure provided a correction for any

depletion in the ClF_3 concentration by reaction with exposed metal. As usual, all ClF_3 was freed of residual HF by vaporizing through a sodium fluoride tower prior to use.

Yearly corrosion rates were calculated based on the 21-day exposure data of the ten metals in ClF_3 and ClO_3F vapor, and are listed in Table 10. In ClF_3 vapor, corrosion rates of aluminum 1100, copper ETP, Ampco 8, magnesium AZ31B, "A" nickel, Monel, 403 S.S., 316 S.S. and low carbon steel 1010 were all very slight (<0.2 mils/yr.) and essentially the same as in ClF_3 liquid. Only yellow brass suffered a noticeably greater corrosion in ClF_3 vapor (0.6 mils/yr.) than in the liquid (0.03 mils/yr.). In ClO_3F vapor, corrosion rates of aluminum 1100, copper ETP, Ampco 8, 403 S.S., 316 S.S. and low carbon steel 1010 were very slight (<0.2 mils/yr.) and essentially the same as in ClO_3F liquid. Moderately higher corrosion rates in ClO_3F vapor as compared to those in the liquid were imposed on yellow brass (0.78 vs. 0.18 mils/yr.) and "A" nickel (0.53 vs. 0.01 mils/yr.). A striking increase in corrosion in ClO_3F vapor as compared to that in the liquid occurred with magnesium AZ31B (4.5 vs. 0.15 mils/yr.) and Monel (3.8 vs. 0.02 mils/yr.). Just as in the liquids, there was no pitting or other localized corrosion due to ClF_3 or ClO_3F vapor exposure.

In general, metal corrosion rates in ClF_3 and ClO_3F vapor are of the same order as those in the respective liquids. However, the possibility of sharply greater corrosion of a certain few metals in the vapors, particularly ClO_3F , exists. Therefore vapor contact of metals not reported here should be checked by preliminary exposure tests.

6. Passivation-Preservation Tests

The literature on ClF_3 generally assumes that a separate, conditioning exposure of metals to ClF_3 acts to passivate the metal surfaces and thus to reduce the general corrosion rate on exposures in ClF_3 service, as compared to ClF_3 service of untreated metals. There is a common impression that this initial exposure or passivation also acts to preserve the metal during stand-by or storage periods, possibly even with atmospheric exposure, between ClF_3 service periods. These pretreatment practices have apparently developed through analogous comparison with qualitative experience in elemental fluorine service.

To provide some quantitative information on this problem, a check series of passivation-preservation tests was completed in which 12 selected alloys (Table 11) were exposed in the following sequence: (1) liquid ClF_3 for two days, (2) laboratory atmosphere for 50 days, and (3) liquid ClF_3 for seven days. A duplicate control set of these alloys was also carried through steps

(2) and (3). All exposures were made in a 30°C. constant temperature cabinet vented to the laboratory atmosphere. Data on these tests are listed in Table 11.

At the end of the exposure series, very little difference either in appearance or weight change was found between the controls and the test set which had a prior passivation in liquid ClF_3 . It was concluded that ClF_3 passivation of the common construction metals is not necessary as a means of reducing the general corrosion rate in ClF_3 or its mixtures with ClO_3F . The advantage of prior ClF_3 contact under controlled, moderating conditions is to slowly and safely burn out any traces of foreign matter which may have escaped the cleaning operation. A rigorous precleaning and drying is the prime essential for metals to be exposed to ClF_3 or ClF_3 - ClO_3F mixtures. Contamination between exposures should be avoided as a matter of course by isolation of the system, and for sensitive installations by flushing and filling to a moderate pressure with dry nitrogen gas. Where contamination or heavy corrosion has occurred or is suspected either in passivated or virgin metal systems, a complete chemical cleaning is the only safe procedure before ClF_3 or ClF_3 - ClO_3F exposure. Such a cleaning cycle might include: (1) solvent or alkaline degreasing, (2) acid pickle, with mechanical abrasion where necessary to remove heavy, tenacious scales or deposits, (3) water rinse, (4) mild alkali (Na_2CO_3) neutralization, (5) water rinse, (6) thorough drying, preferably above 100°C. and with dry nitrogen flushing of vessels or other enclosed spaces.

7. Corrosion in Wet Perchloryl Fluoride

Previous experience at Pennsalt has shown that while most metals are highly resistant to corrosive attack in pure, dry ClO_3F , the presence of moisture in the range of 100 ppm. and higher can result in severe and often localized attack.

Corrosion rates of 30 alloys were observed at 30°C. in the liquid and vapor phases of perchloryl fluoride to which had been added 0.2 to 1% water, and are listed in Table 12. Metal specimens were precleaned by the standard procedure of Exhibit 1, and were suspended in the test tank on glass or plastic racks through drilled holes, with CaF_2 -filled Teflon spacers acting as additional electrical insulators. The charged tank was held in an air cabinet maintained at $30 \pm 2^\circ\text{C}$. for the 21-day exposure periods.

In the Test Series #1, the test tank was low carbon steel, protected internally with a plastic coating. At the end of the test period, the tank coating was found to have broken loose from the steel wall and to have several cracks, with severe and deep rusting of the tank at these points. There was no attack on the plastic itself. Loose, crumbly and voluminous corrosion de-

posits were formed on those test metals such as low carbon steel which exhibited high corrosion rates. Since a moisture analysis of the liquid ClO_3F from the test tank showed only 20 ppm. H_2O remaining, the extremely low corrosion rates observed for the stainless steels and titanium alloys could not be considered as a true measure of their corrosion resistance to wet ClO_3F .

The Test Series #2 represented a repeat of the corrosion testing of stainless steels and titanium in wet ClO_3F , but with 1% added H_2O and use of an all-stainless (304) tank system. Prior to use, the tank was pickled with a 1/1 (V/V) nitric acid solution at 60-65 °C. for 15 minutes, rinsed, washed with a 5% sodium carbonate solution, then rinsed and dried at 120°C. At the end of Test Series #2 no corrosion of the tank interior was found, although the 304 S.S. inside rim of the Flexitallic gasket was corroded at several points in what appeared to be a localized, crevice-type corrosion. Moisture analysis of the recovered ClO_3F gave 543 ppm. H_2O in the vapor phase. Judging from previous control analyses, it is likely that the liquid ClO_3F was still saturated with H_2O and water remained in excess as a separate phase on the surface of the ClO_3F liquid. Corrosion rates listed in the second part of Table 12 prove that in the absence of more easily corroded metals and at high moisture content, the stainless steels and titanium are attacked by wet ClO_3F .

At the end of Test Series #3, in which the procedures of Test Series #2 were repeated with additional types of stainless steels, there occurred during discharge of the ClO_3F a perforation of the tank wall (0.094" thick) at four points of pitting attack in the general area of the liquid level during test. A heavier walled 304 S.S. tank was fabricated by welding a 6-inch welding cap, as bottom, to a 6-inch welding neck flange and closing with a 6-inch bolting lid, using standard 150 lb. parts. A more effective passivation than nitric acid alone was obtained by pickling the tank interior in an HF- HNO_3 solution:

305 ml. 70% HNO_3
120 ml. 50% HF
diluted to 3000 ml.
temp. - 70-80°C.
time - 15 min.

The pickling was followed by rinsing, washing with a 5% sodium carbonate solution, rinsing and drying at 120°C. This passivation treatment was given before each wet PF exposure in Test Series 4 to 6. No pitting or other localized attack on this passivated stainless steel was observed after 67 days total exposure at 30°C. to PF containing 1% H_2O . Another 304 S.S. cylinder

which had not been passivated and which had held wet ClO_3F mixtures for several weeks for control moisture determinations was found to contain a large amount of small, irregular nodules of a dark brown pasty solid together with a brown wet slime. This corrosion product was washed and dried at 120°C . to give a brown-black powder composed of fine, shiny flakes, and identified as Fe_3O_4 by X-ray diffraction analysis.

The predominant and serious form of attack in metal exposure to wet ClO_3F was pitting at local concentration cells. Such points of attack were common at crevices formed about the support hole of the test piece, but sometimes occurred on fully exposed faces. Carpenter #20-Cb appeared to be the most resistant of the wrought stainless steels, and its cast modification, Durimet 20 was also highly resistant. Other high nickel alloys exhibited low corrosion rates as shown in Table 12. Gold and platinum were completely resistant to attack, while silver underwent a mild uniform etch. Because of the susceptibility to unpredictable pitting attack of many metals in wet PF, any metal contact, excepting gold and platinum, with PF suspected of having a moisture contact above 100 ppm. should first be checked by adequate in-service testing.

8. Shock Tests

The static immersion tests reported above did not, except in wet perchloryl fluoride and in the complete failure of several special materials in screening type exposures, bring out any dramatic differences in the corrosion resistance of the materials listed in Table 7. The quantitative data from these immersion tests, on which a large part of the effort of this project was expended, are of course of considerable value in confirming the compatibility of these structural materials with chlorine trifluoride, perchloryl fluoride and mixtures of these under normal ambient conditions. In addition, however, dynamic tests which will clearly delineate the range of and the differences in compatibility by the imposition of drastic exposure conditions are necessary for a complete stability and safety evaluation of materials of interest. The application of chlorine trifluoride and perchloryl fluoride as missile oxidizers in particular requires a background of dynamic materials compatibility in anticipation of unusual and trying service conditions.

The dynamic tests reported here including detonation shock tests, cylinder denting and perforation tests, and plate impact tests provide an introduction to dynamic compatibility for several metals of greatest practical interest. These metals include low carbon steel, stainless steel, aluminum and titanium (ClO_3F alone).

Grenade Tests: When a liquid oxidizer in a combustible container, like steel or aluminum, is subjected to detonation shock from an internally placed priming explosive, the force of the primer is enhanced, presumably by interaction of the oxidizer with the fragmenting container. If the liquid oxidizer is replaced by an explosive compound or by a thermodynamically unstable compound such as hydrogen peroxide, a high order explosion takes place which is many orders of magnitude more powerful than the primer charge. This type of test can be used to give a rough measure of the stability of a compound as well as a measure of its oxidizing power. Past tests of this type have been carried out cooperatively by Pennsalt and McCullough Tool Company of Houston, Texas.

Perchloryl fluoride has a small positive free energy of formation, so that gradual decomposition into the component elements is possible. However, no such decomposition has been observed, except at elevated temperatures. The heat of formation of perchloryl fluoride is negative, so that in decomposition into its elements heat would have to be absorbed from the surroundings. Therefore, a fast explosive decomposition cannot be expected, and this may explain why the past shock tests noted above failed to initiate any signs of explosive behavior other than the oxidizer-container interactions which was shown by all the liquid oxidizers tested, including liquid chlorine, which, being an element, cannot undergo decomposition.

It was considered desirable to make a comparative determination of how grenades made from low-carbon steel, stainless steel and aluminum filled with liquid chlorine trifluoride, perchloryl fluoride and mixtures of these, and titanium grenades filled only with liquid perchloryl fluoride, titanium being entirely incompatible with chlorine trifluoride, would behave under fragmenting shock. These tests as well as the cylinder perforations and dentings were done at the West Hanover, Mass. testing range of the American Potash and Chemical Corporation's National Northern Division and at the Red Lion, N.J. testing range of The Drum Co. of Bristol, Pa.. Still and motion pictures were taken of the tests.

Metal grenades of the type shown in Figure 3 were made from steel, stainless steel, aluminum and titanium. They were filled with approximately equal amounts of the chemicals under test and sealed. The center well was filled with 9 RDX 97-3 pellets, 0.475" D. x 0.500" H. and 2.7 g. each, with a number six electric blasting cap for initiation taped against the last pellet. The loaded grenade was placed in the center of a sand-filled 39-gallon caustic drum fitted with a tightly

clamped cover. These steps are pictured in Figure 4.

The effects of the grenade shots on the drums are described in Table 13 and are illustrated in Figure 5. The increased energy which was developed in these tests over that from the primer alone against a water or air charge is considered to come from the reaction between the chemical and the hot fragments of the center tube of the grenade. An additional energy arises from the chemical reacting with the vapor-exposed upper side wall of the grenade and with the explosion products of the primer.

Over-all, perchloryl fluoride was judged to have a higher level of effectiveness in enhancing the primer explosion. In the chlorine trifluoride tests there were more rapid upward propulsions of drum cover and sand, but less bulging of the drum and distortion of the cover than in the pure PF or PF-ClF₃ mixtures tests.

A noticeably greater enhancement of the primer charge was noted in the tests using grenades of aluminum as compared to low carbon steel and stainless steel, suggesting a somewhat greater interaction of the test chemicals with aluminum than with steel. This metal interaction is also shown to a small degree by the primer, but it is most pronounced with perchloryl fluoride and its mixtures. The grenade shots show that PF has a somewhat greater interaction with aluminum than does chlorine trifluoride.

By far the greatest enhancement of primer explosive was obtained in the shots of titanium grenades containing liquid perchloryl fluoride. The considerably higher order of explosion caused a more drastic rending of the sand drums, more complete scattering of the contained sand, and even jagged perforations of the drum sheet by small grenade fragments. In all other shots, the grenade fragments were relatively large and sufficiently cushioned by the sand to prevent cutting the drum. Also, after the Ti-ClO₃F shots only, small brush fires were started in a circular area about 50 feet in diameter apparently by small, hot grenade fragments.

Examination of the grenade fragments showed that in all cases there was a deep grooving and burning of vapor-exposed upper side wall. Small fragments of the center tube were also found embedded in this area. This phenomenon was noted in previous Pennsalt and McCullough tests as well as by National Northern Division in some of their other tests.

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Cylinder Denting: In the first denting shots, the effect of severe mechanical shock on mixtures of perchloryl fluoride and chlorine trifluoride contained in steel shipping cylinders was observed. The cylinders, which were ICC 3A480, 5-pound capacity chlorine trifluoride type, 4-1/4" O.D. x 13-1/2" shoulder height, were each liquid half-filled with a test mixture. At the National Northern test site, the cylinder, with shipping cap removed, was securely clamped to a vertical steel ground post and a 1/2-inch RDX 97-3 pellet with a number six electric blasting cap was taped near the top of the cylinder for the gas phase tests and near the bottom for the liquid phase tests.

Mixtures of 25% and 75% perchloryl fluoride in chlorine trifluoride were shock tested both in the liquid and gas phases. In every case the cylinder wall was severely dented by the explosive charge but the cylinder wall was never ruptured. In no case was there any sign that the chemical was sensitive to this kind of shock, nor was there any sign of chemical attack on the steel at the point of shock, as determined by sectioning and internal examination of the cylinders. The same results were obtained by Pennsalt in previous independent shock tests with chlorine trifluoride and perchloryl fluoride individually.

The shocking and denting of titanium A-55 vessels containing liquid ClO_3F by #6 or #E-91 blasting caps, or by a combination of an RDX pellet (2.7 g.) and a #6 blasting cap taped to the side walls was not sufficient to initiate a sustained or explosive interaction of the titanium and ClO_3F . A double wrap of RDX high velocity primacord around a titanium vessel charged with liquid ClO_3F caused a shallow dent circle and sufficient internal interaction to blow off the welded lid. These titanium vessels were specially fabricated using the grenade design of Figure 3, but eliminating the center well and increasing the body length from 5-1/2 to 12-1/2 inches.

Cylinder Perforation: The first cylinder perforation shots were made to observe the effect of firing an explosive charge into the liquid and gaseous phases of chlorine trifluoride, perchloryl fluoride, and mixtures of these contained in steel shipping cylinders. Cylinders used were ICC 3A480, 5-pound ClF_3 type. These were liquid half-filled to a level about 6 inches from the bottom. Two cylinders were charged with each of the liquids, pure ClF_3 , 25% ClO_3F -75% ClF_3 , 75% ClO_3F -25% ClF_3 and pure ClO_3F , for a total of 8 cylinders. At the test site, a cylinder, with shipping cap in place, was set upright on a steel block and fastened to a vertical steel ground post. A McCullough specialty three-inch jet perforation charge was taped near the top of the cylinder for vapor phase shots and near the bottom for liquid phase shots. An E-91 blasting cap and RDX primacord was used as detonator.

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In the case of shots into the liquid, the cylinders were propelled upward 15 to 20 feet; with the shots into the vapor space, the cylinders were simply knocked over on their sides 1 to 3 feet from the post. The chemical contents were more quickly vented and dispersed after shots into the liquid than after shots into the vapor. Pure ClF_3 took the longest time for venting and dispersal of contents. A 5/8-inch hole was cut completely through each cylinder in both liquid and vapor shots. There was no explosive enhancement by the cylinder contents in any of the shots. Cylinder damage was confined to the hole cutting and a minor burning in the hole area by the incendiary ClF_3 and its mixtures with ClO_3F . With pure ClO_3F there was no burning.

The second set of perforation shots was made into the liquid phase of chlorine trifluoride, perchloryl fluoride and water held in aluminum 1100-F pressure containers which were liquid half-filled. These containers (2-13/16" I.D. x 5-7/32" H. x 0.041" wall) had been impact formed in one piece by Alcoa. The closure was modified by welding a 1-inch section of 1-1/4-inch diameter aluminum 2024 rod to seal the opening, and a valve attached to this cap. For these light duty aluminum cylinders, the perforation charge taped to the cylinder wall consisted of one #E-91 blasting cap connected through a length of RDX high velocity primacord. In all three shots (ClF_3 , ClO_3F , H_2O) the result was simply the cutting of a 1/2-inch diameter hole in the front cylinder wall, with no explosive enhancement by the liquid contents nor any initiation of reaction with the aluminum.

In the third group, three-inch McCullough jet perforation shots were made into the liquid and gaseous phases of perchlory fluoride held in titanium A-55 vessels, which are described above under Cylinder Denting. These two titanium cylinders with the attached jet charges are pictured in Figure 6. Each of the two shots resulted in a tremendous explosive interaction of the titanium and the ClO_3F which, in turn, caused a complete shattering of the vessel into fine shrapnel. The power of this explosion was evidenced by the sharp bending of two steel angle ground posts, 3/8" x 2" x 2" x 4' L., against which the vessel was braced, and the uprooting and propulsion of these posts through the air about 75 feet from the shot point. In a control jet perforation shot through a titanium A-55 vessel containing air, the result was only an irregular 1/2-inch diameter hole cut completely through the cylinder, with no metal-explosive interaction.

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Impacts: Impact tests of a selected group of ten metals* in liquid ClF_3 and liquid ClO_3F at 30°C . were planned. The vapor pressures at 30°C . of ClF_3 (15 psig) and ClO_3F (185 psig) precluded the use of an open cup tester. Therefore a steel pressure vessel impact tester utilizing a compressed spring as energy source was designed and built for this use.

Figure 7 illustrates the features of this tester which after being fitted with metal striker and plate shown in Figure 8 and a spring of Table 14 was cocked, loaded and sprung following the procedure outlined in Exhibit 7. Striker and plate thickness were normally $1/8$ inch, but varied slightly according to plate thickness available. The tank height was designed to each spring length used such that with the impact tester assembled with a compressed spring, the usual vertical distance from striker tip to plate was equal to one-half the compression of the spring.

Two sizes of impact testers of Figure 7 were built to permit covering the impact energy range of 5 to 65 ft.-lbs.:

Impact Tester #1--Designed to hold die springs one inch in diameter by 4 inches long, for impact energies of 5 to 20 ft.-lbs. Vessel construction was based on use of a 3-inch standard 150 lbs. weld neck flange with welded extension of 3-inch pipe and a welded bottom plate. The lid assembly with spring housing, trigger rod and striker mechanism was fabricated on a 3-inch standard 150 lb. blind flange.

For flange seal, a Flexitallic gasket CG-1J with 304 S.S. and Teflon filler was used. The top gland for movement of the one-half inch tool steel trigger rod was packed with Garlock Teflon Chevron packing, 1" O.D. x $1/2$ " I.D. x $1-3/16$ " H. Each liquid charge was either 300 g. ClF_3 or 232 g. ClO_3F . This was sufficient liquid, 167 cc. at 30°C ., to completely immerse the striker at impact with the bottom horizontal plate.

Impact Tester #2--Embodied same type of construction as #1 Tester, with the following changes:

- a. Designed for die springs 2" D. x 5" L. (for impact energies of 20 to 65 ft.-lbs.)
- b. Based on 4-inch standard 150 lb. flange.
- c. Liquid charge was either 510 g. ClF_3 or 395 g. ClO_3F (284 cc. at 30°C .).

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- | | | |
|-------------------|--------------------|-------------------------------------------------------------|
| *1. Aluminum 1100 | 4. Magnesium AZ31B | 7. 316 S.S. |
| 2. Copper, ETP | 5. Nickel | 8. 403 S.S. |
| 3. Yellow Brass | 6. Monel | 9. Low Carbon Steel 1010 |
| | | 10. Titanium C-120AV-Ti
(in ClO_3F only) |

The calculation of energy release by the compressed spring on impact of striker and plate is covered in detail in Exhibit 8.

The effects obtained in 50 impacts made in ClF_3 , ClO_3F and air are described in Table 15. No signs of appreciable oxidation or burning were found with any of the metals in ClF_3 or air at impact levels up to about 65 ft.-lbs. Copper, magnesium and low carbon steel showed a slight staining or film formation as evidence of possible incipient oxidation in ClF_3 . A 403 S.S. striker was used with aluminum and magnesium plates at the highest impacts to avoid the cushioning obtained with the soft metal strikers. In ClO_3F impacts, all metals except titanium were essentially unaffected. Copper, magnesium and 403 S.S. underwent a slight surface staining.

Titanium in the form of the two alloys C-120AV-Ti and A-110AT-Ti exhibited an increasing reactivity in liquid ClO_3F proportional to the impact level, beginning with a metal fusion and oxidation at 19 ft.-lbs. At 64 ft.-lbs. there was a definite burning of the striker tip to form a black, sintered residue. However, the reaction was not sustained and was confined to a small impact area, except for some fused metal spattering. Several titanium impacts were made above the 65 ft.-lb. design limit of the tester, and up to 140 ft.-lbs. This was done by compressing the die springs beyond their normal, elastic limit. At the 140 ft.-lb. level, some initiation of burning of the plate as well as the striker resulted, but the reaction was not sustained.

As an extrapolation of the results obtained here, it is possible that at some reasonably higher impact level, perhaps 250-350 ft.-lbs., the burning reaction of titanium in liquid perchloryl fluoride might be sustained. Attention is directed to similar impact tests of titanium in liquid oxygen and nitrogen tetroxide as discussed in the Literature Survey of Section IV. In summary, the complete shock tests on titanium reported here cover the spectrum of possible exposure. The impact tests cover the lowest to the lower middle range; the cylinder perforation and grenade shots probe the high shock level at which the titanium- ClO_3F reaction is strongly sustained, and there is some overlapping with the cylinder denting shots.

9. Corrosion Measurement by the Electrical Resistance Method

Considerable interest has recently developed⁽²⁻⁶⁾ in the electrical resistance method of measuring corrosion rates. This method permits corrosion rates to be observed continuously and could be a powerful tool in determining the characteristics of the so-called protective films reported to account for the low corrosion rates of most metals in chlorine trifluoride. In this technique

the corroding specimen is a tube, strip or wire of the test metal, and is fabricated in the form of a probe unit with suitable external electrical connections. As the exposed specimen corrodes it becomes thinner and its electrical resistance increases. By using a balancing circuit and a sensitive galvanometer very small changes in resistance can be measured and, consequently, small amounts of corrosion can be detected.

An evaluation was made of the Model 2032 probe units of the Crest Instrument Company, Santa Fe Springs, California. These probes have a Teflon plug seal between the exposed metal element and the inner probe parts encased in epoxy resin. Unfortunately, the Teflon plug did not provide a perfect hermetic seal, and there was sufficient diffusion of ClF_3 past the Teflon plug to result in a violent incendiary reaction with the epoxy filler after short exposure periods. Therefore, it was not possible to make any meaningful corrosion measurements.

10. Conclusions

Typical alloys of aluminum, copper, magnesium, nickel, low carbon steel and stainless steel were found to be highly resistant to chlorine trifluoride, perchloryl fluoride and their mixtures at 30°C . and under severe shock. Therefore, the common construction metals are considered compatible in this service. The metals must be clean and dry, but no other special passivation is required.

Titanium, columbium, molybdenum, carbon and graphite are rapidly attacked by chlorine trifluoride. Titanium and perchloryl fluoride, although compatible under ambient conditions, interact under shock conditions, so titanium use is not recommended.

For wet perchloryl fluoride the following metals and alloys were found most resistant and are recommended: platinum, gold, various stainless steels, various high nickel alloys. No special treatment is required for the platinum or gold, but an acid pickling* is recommended for the stainless steels and high nickel alloys.

Teflon and Kel-F can be used with chlorine trifluoride and perchloryl fluoride, but only with extreme care and then only under mild conditions of heat and shock. Both plastics adsorb moderate amounts of oxidizers and may undergo structural changes. Occasionally they do fail, but the failure is not likely to spread to adjoining metal. These plastics are best used in vapor and when provided with good heat release. They should never be used in service with these oxidizers when subjected to heat, shock or flow conditions.

*Preferably HNO_3 -HF mixture.

III. CORROSION MECHANISM IN CHLORINE TRIFLUORIDE

The mechanism whereby metals resist the attack of elemental fluorine or of chlorine trifluoride is not completely understood. All metals are thermodynamically free to react with these corrodants at low temperatures, and a commonly held hypothesis is that a clean metal surface is at once coated with a layer of metallic fluorides which protects the metal from further attack.

Workers at the Oak Ridge Gaseous Diffusion Plant (U.S. Atomic Energy Commission)⁽⁷⁾ have measured the rate of film formation for nickel, and have shown the protective nature of these films at high temperatures. Although much less work has been done on the corrosion of metals in chlorine trifluoride, this same mechanism of a protective coat may apply and account for the low corrosion of most metals in chlorine trifluoride.

At low temperatures, where the films would be very thin, it is difficult to have direct proof that they still protect the metal surface and are responsible for the low corrosion rate observed. Knowledge of the corrosion mechanism would be helpful in selecting untested alloys to be used with chlorine trifluoride.

Although the direct proof of the mechanism for the corrosion of metals in chlorine trifluoride at atmospheric temperatures is beyond the scope of this project, the observation that only those metals that form volatile fluorides or fluorides soluble in chlorine trifluoride have high corrosion rates at low temperatures does give indirect evidence of a protective film of metal fluoride or mixed metal chloride and fluoride. Thus, in the present work it has been shown that iron, copper, nickel, aluminum, and magnesium all have very low corrosion rates. The fluorides of these metals have low vapor pressures up to several hundred degrees. Titanium, molybdenum, uranium, and columbium, on the other hand, all form fluorides that have high vapor pressures at atmospheric temperatures or that form complex ions that are soluble in liquid chlorine trifluoride. All of these metals show very high attack in liquid chlorine trifluoride at low temperatures.

If this mechanism is correct, then caution must be followed in using stainless steels that contain columbium and molybdenum for service with chlorine trifluoride. If these metals are present in very small amounts and as a solid solution, the alloy may be satisfactory. However, those alloys in which they are present in large amounts or in which they occur as granular or interstitial precipitates cannot be recommended for this service. This would be especially true for such structural elements as diaphragms, bellows, and fine wires, where a small surface corrosion could result in a serious change in the mechanical properties of the object.

IV. LITERATURE SURVEY

Compatibility of Materials with Chlorine Trifluoride and Perchloryl Fluoride

(All reference numbers in this section apply to Exhibit 9)

An extensive literature survey on chlorine trifluoride and perchloryl fluoride was condensed to the pertinent bibliography of 34 references attached to this report as Exhibit 9. This bibliography is generally limited to materials compatibility with ClF_3 and ClO_3F . For the analogous problems in the handling of elemental fluorine, reference (1) is made to concurrent WADD contract studies by Air Products, Inc., the technical report including a literature survey on materials compatibility.

The original classical report on chlorine trifluoride by Ruff (31) includes the characteristics of the reaction of liquid ClF_3 with a large number of substances, including metals. Details of conditions are not given and apparently the metals are finely divided. Other review articles and bibliographies (10, 11, 27) contain similar listings of the reactions of ClF_3 . A number of sources (2, 6, 13, 19, 21, 22, 25, 26) report the suitability of the common metals such as nickel, Monel, copper, mild steel, brass, aluminum, magnesium and stainless steel as materials of construction for handling ClF_3 , with nickel and Monel being preferred for service at elevated temperatures and in the presence of moisture. It is generally assumed that an initial, superficial reaction of ClF_3 to form a protective coating of the metal fluoride that prevents further reaction is the basic reason for the excellent compatibility of most compact metals with ClF_3 . The prime necessity for extreme cleanliness of all surfaces to be contacted with ClF_3 in order to prevent initiation of reaction by impurities and foreign materials is also strongly stressed.

Fused silica and Pyrex glass are reported (19) as not being attacked by ClF_3 up to 100°C . if HF is completely absent. For normal, non-metallic contact, only the fluoroplastics such as Teflon and Kel-F, either unfilled or filled with an inert material such as calcium fluoride, are recommended (2, 13, 14, 19, 23, 24) as resistant to ClF_3 at ambient temperature. Use of fluoroplastics in flow conditions is not recommended.

The bulk fluorination of metals by ClF_3 to form the corresponding fluorides has also been studied. Hüchel (20) describes conditions for the formation of HgF_2 , AgF_2 , CuF_2 , TiF_3 , CoF_3 , PtF_4 and PbF_4 as well as SeF_4 . Application of ClF_3 dissolution of uranium in atomic energy processes is described by several workers (4, 9, 18, 32). Uranium metal dissolved in liquid ClF_3 at $25-70^\circ\text{C}$. in a smooth reaction, and ignited in ClF_3 vapor at 205°C . Bernhardt(9) and Gustison (18) found that ClF_3 containing HF is more corrosive than ClF_3 alone in reaction with uranium. Stein and Vogel (4, 32) report that thorium is attacked very little by ClF_3 up to 350°C . while zirconium is unstable and ignites in ClF_3 vapor at 340°C .

Contrails

The literature contains no information on the mechanism of fluorination of metals by ClF_3 . Farrar and Smith (16, 17), however, have studied the fluorination of nickel oxide by chlorine trifluoride. They found the reaction taking place in two steps. First, there is a rapid uptake of gas to form a thin film of fluoride sufficient to prevent an uncontrolled reaction taking place, the initial fluoride film increasing in thickness by a diffusion process. Then, at a critical thickness of fluoride film a recrystallization process is reported taking place with a change from a more or less continuous film to a mosaic network of crystallites with open grain boundary paths. The sorption mechanism then is said to involve migration of the ClF_3 down micro-channels between crystallites of the porous NiF_2 particles, followed by diffusion through the transition zone. The rate controlling step then is diffusion through the transition zone of relatively constant thickness. The completely converted NiF_2 contained ClF_3 that could be removed by evacuation. It was concluded that the excess ClF_3 was sorbed in the inter-crystallite boundaries.

Worthington (34) in attempting the stabilization of copper and nickel apparatus against corrosion by anhydrous HF found that prefluorination with HF had no protective effect. Prefluorination with ClF_3 prevented subsequent attack when it was performed at a temperature of 40 to 50°C. higher than the reaction temperature subsequently used with the HF.

Experience associated with application of ClF_3 as a rocket oxidizer is being reported (6, 12, 27, 30, 33) with increasing frequency. All confirm qualitatively the compatibility of materials with ClF_3 previously noted. However, Rocketdyne's (27) literature survey in Table 2 erroneously includes carbon as one of the materials compatible with liquid ClF_3 . We have found that almost all grades of compact carbon and graphite are rapidly disintegrated to powders or flakes on contact with liquid ClF_3 at or slightly below ambient temperature. Rocketdyne's literature survey includes a private communication on impact stability which states that attempts to decompose ClF_3 by mechanical shock induced by a high brisance explosion caused no apparent change. Cylinders of ClF_3 were pierced by high-velocity, steel-jacketed rifle bullets with no indication of adverse reaction from the shock. Instances of ignition of the ruptured metal about the bullet hole were, however, reported.

Because of special interest in titanium compatibility, several background references (3, 7, 8) are included. Ericson (8) found that titanium exhibited promising corrosion resistance to liquid and gaseous fluorine between -320°F. and +220°F. This was opposite to our experience with ClF_3 in which titanium rapidly dissolved with vigorous reaction below ambient temperature. A similar experience on titanium exposure to ClF_3 was noted by Rocketdyne (28). This is striking proof that analogous comparisons of compatibility in ClF_3 and fluorine should not be made in the absence of experience. In impact tests (3, 7) of titanium in liquid oxygen and nitrogen tetroxide it appeared that although ignition might occur at high impact values, a combination of several factors is necessary to propagate the reaction.

Contrails

Published compatibility information on perchloryl fluoride is very limited (15,29) and qualitative in nature.

References on the physical and other chemical properties were not specifically added to the bibliography of Exhibit 9. However, summaries of the properties of ClF_3 are included in references (2,10,11,19,31) and of ClO_3F in reference (29).

V. REFERENCES

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FIGURE 1

LIQUID-VAPOR DIAGRAM FOR ClO_3F - ClF_3 SYSTEM
(Per cent by Weight at $25^\circ \pm 1^\circ\text{C}.$)

(See Table 4 for equilibrium data)

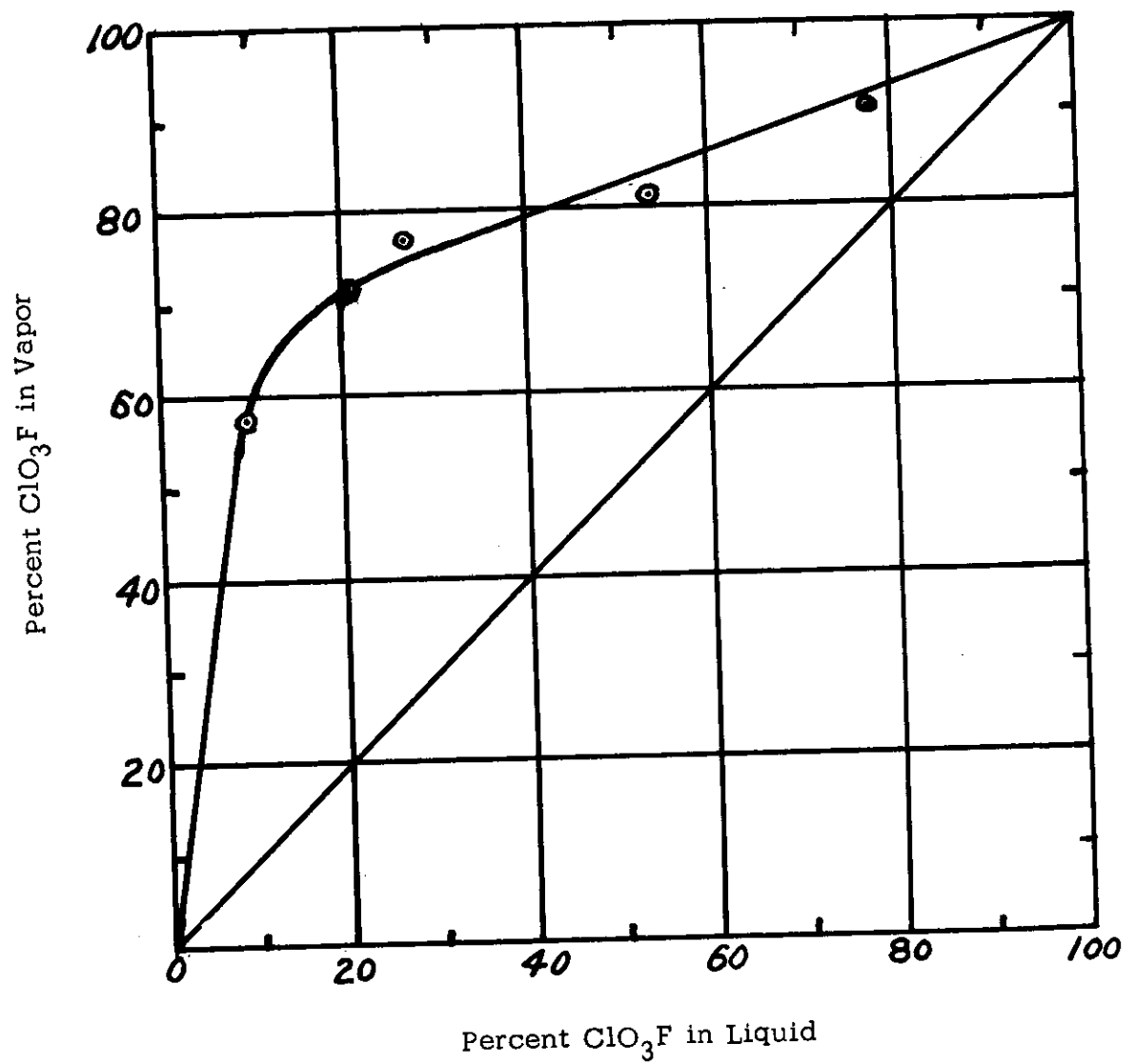


FIGURE 2

SURFACE AREA OF UNSTRESSED TEST PIECE
IN IMMERSION TESTING

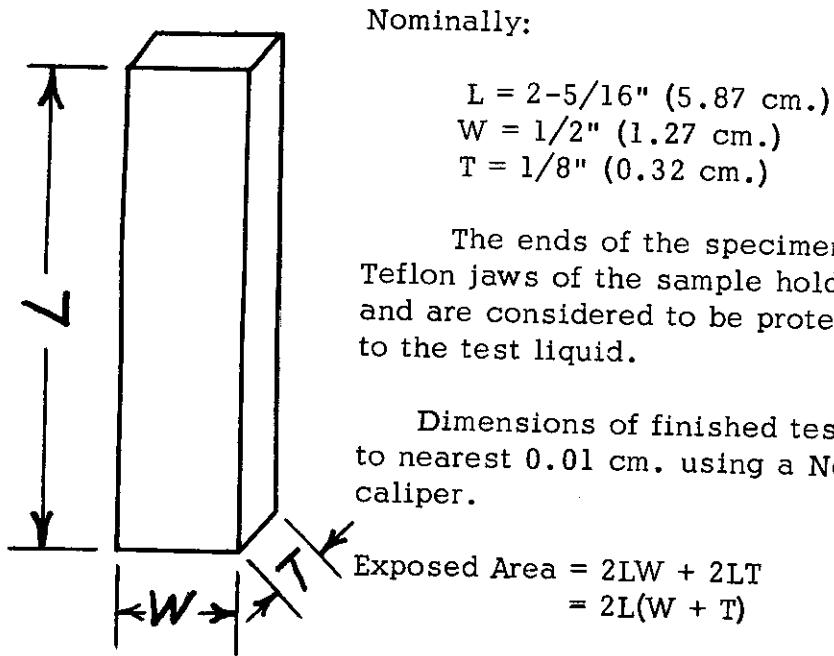


FIGURE 3

CHEMICAL GRENADE

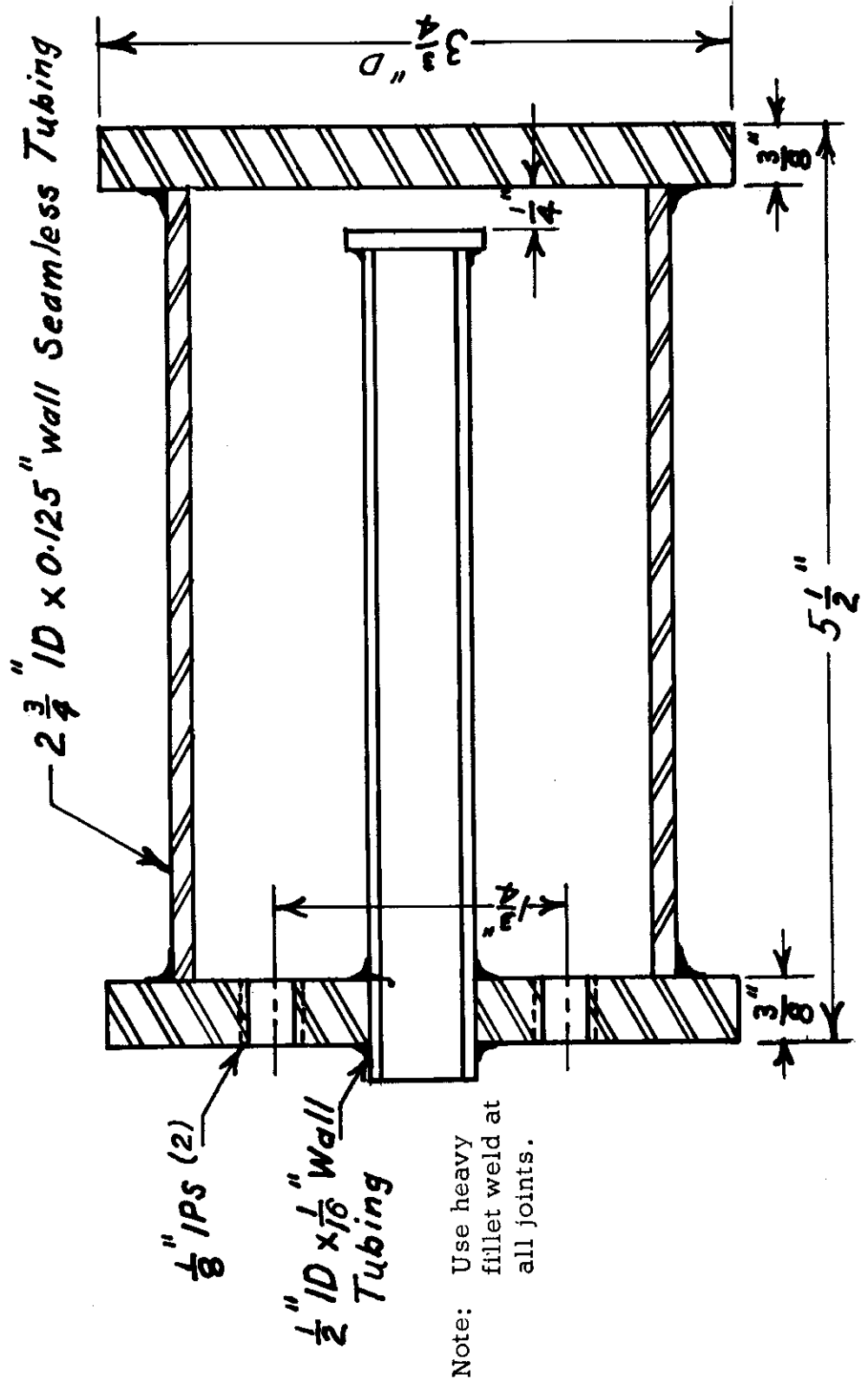
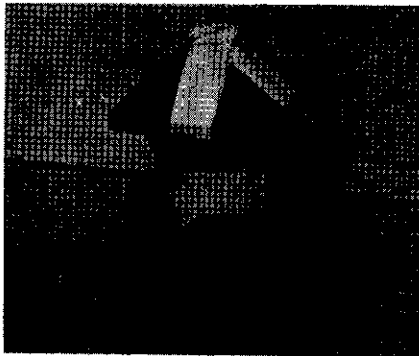


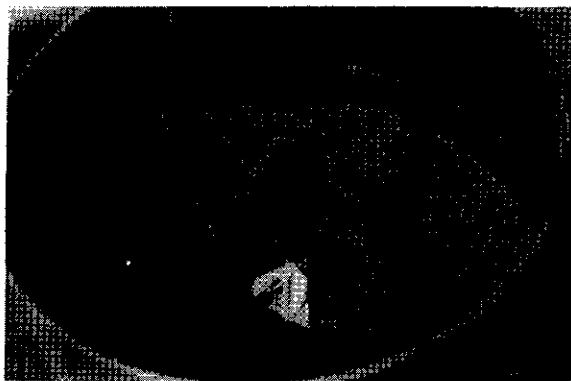
FIGURE 4

GRENADE TEST ARRANGEMENT



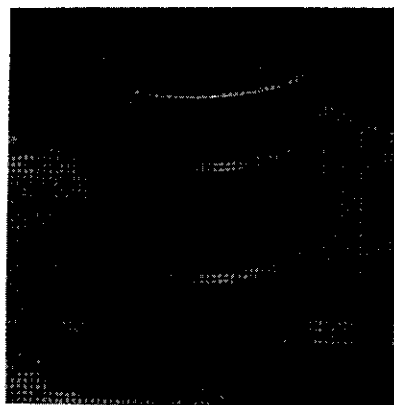
4a

Detonation Charge Fastened to Grenade



4b

Grenade Placed in Sand Drum

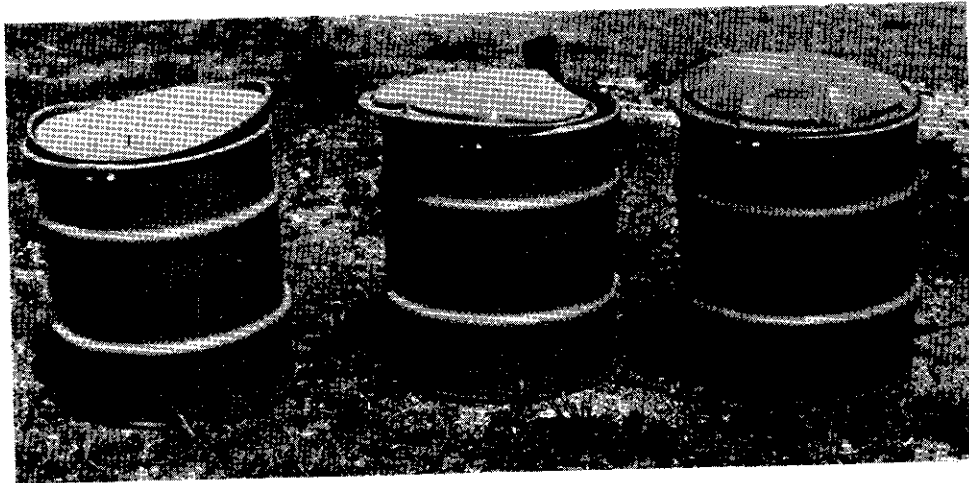


4c

Sand-Filled Covered Drum Ready for Shot

FIGURE 5

EFFECT OF GRENADE SHOTS ON DRUMS
(See Table 13 for Details)



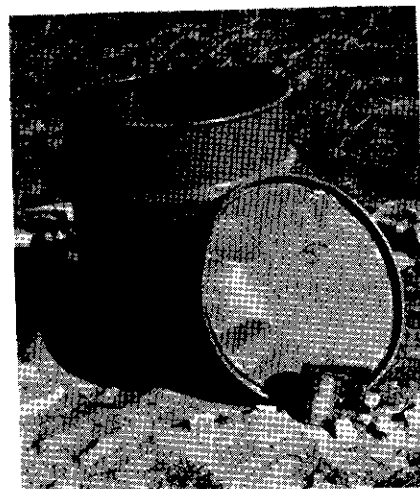
5a

Shot 1-NN. H₂O in Low Carbon Steel Grenade
Shot 2-NN. H₂O in Aluminum 2024 Grenade
Shot 12-NN. Air in Aluminum 2024 Grenade



5b

Shot 1-DC. Air in
Aluminum 6061
Grenade



5c

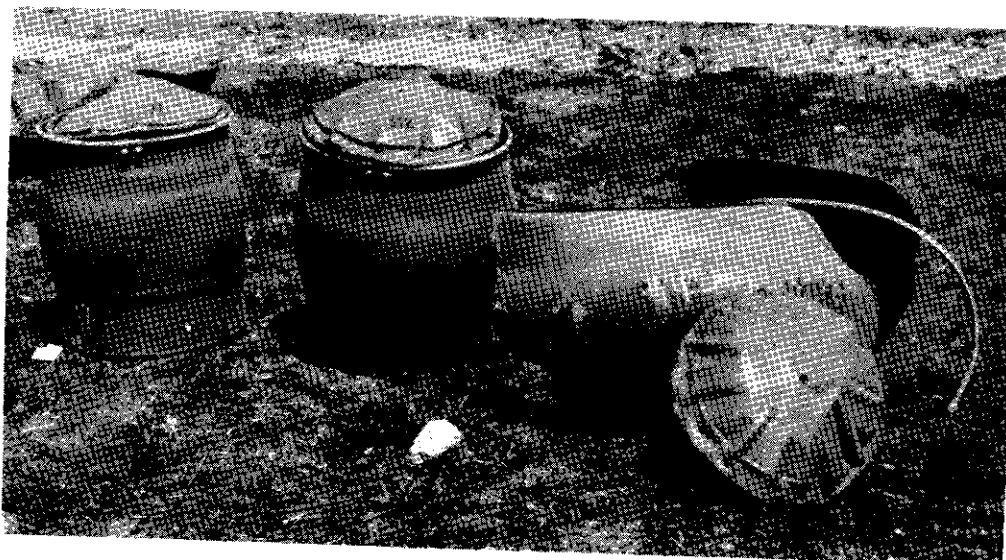
Shot 6-DC. Air in
Titanium A-55
Grenade

FIGURE 5 (Cont.)



5d

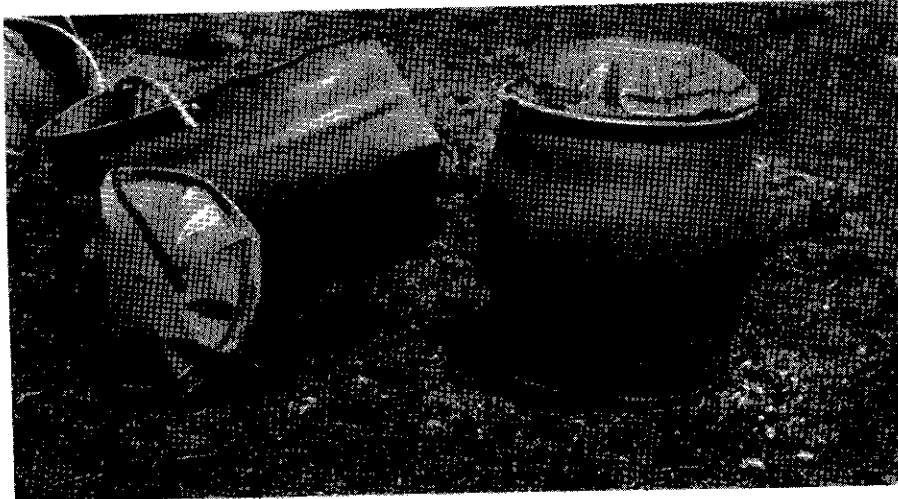
- Shot 3-NN. ClF_3 in Low Carbon Steel Grenade
- Shot 4-NN. ClF_3 in 316 Stainless Steel Grenade
- Shot 5-NN. ClF_3 in Aluminum 2024 Grenade



5e

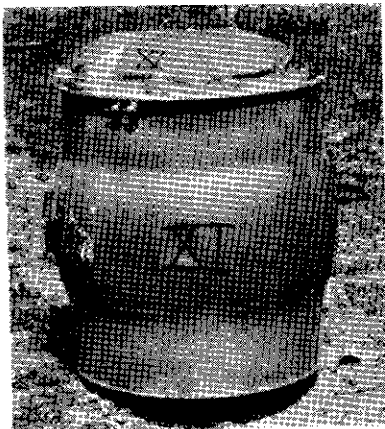
- Shot 6-NN. 25% ClO_3F -75% ClF_3 in Low Carbon Steel Grenade
- Shot 7-NN. 25% ClO_3F -75% ClF_3 in 316 Stainless Steel Grenade
- Shot 8-NN. 25% ClO_3F -75% ClF_3 in Aluminum 2024 Grenade

FIGURE 5(Cont.)



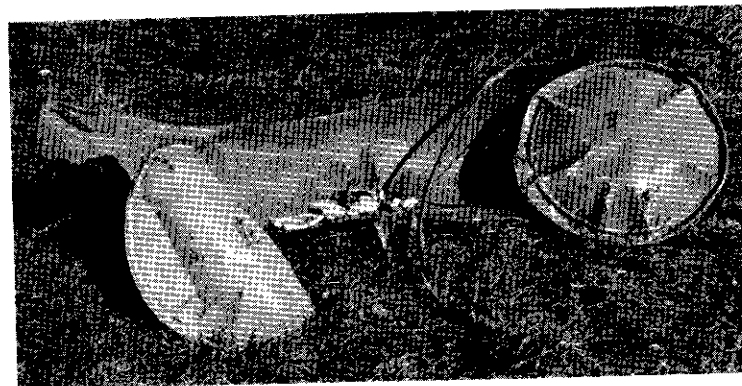
5f

Shot 9-NN. 75% ClO_3F -25% ClF_3 in Low Carbon Steel Grenade
Shot 10-NN. 75% ClO_3F -25% ClF_3 in 316 Stainless Steel Grenade



5g

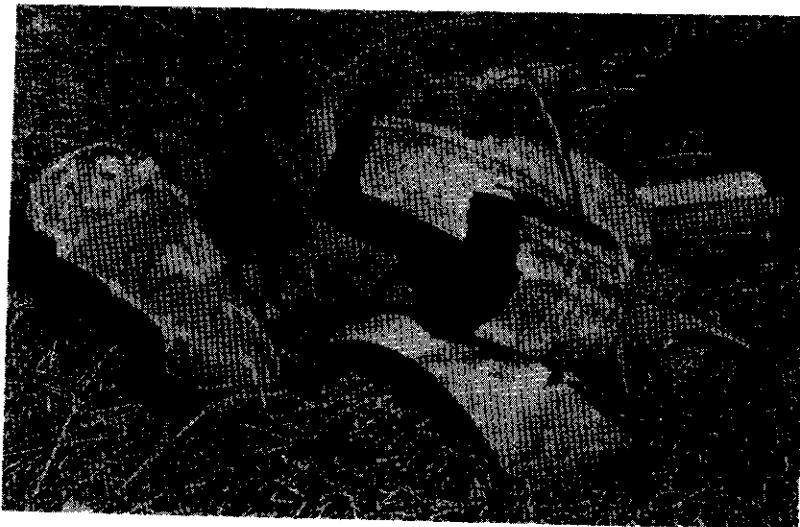
Shot 11-NN. ClO_3F in
Low Carbon Steel
Grenade



5h

Shot 3-DC. ClO_3F in Aluminum 6061 Grenade

FIGURE 5 (Cont.)



5i

Shot 2-DC. ClO_3F in Titanium A-55 Grenade

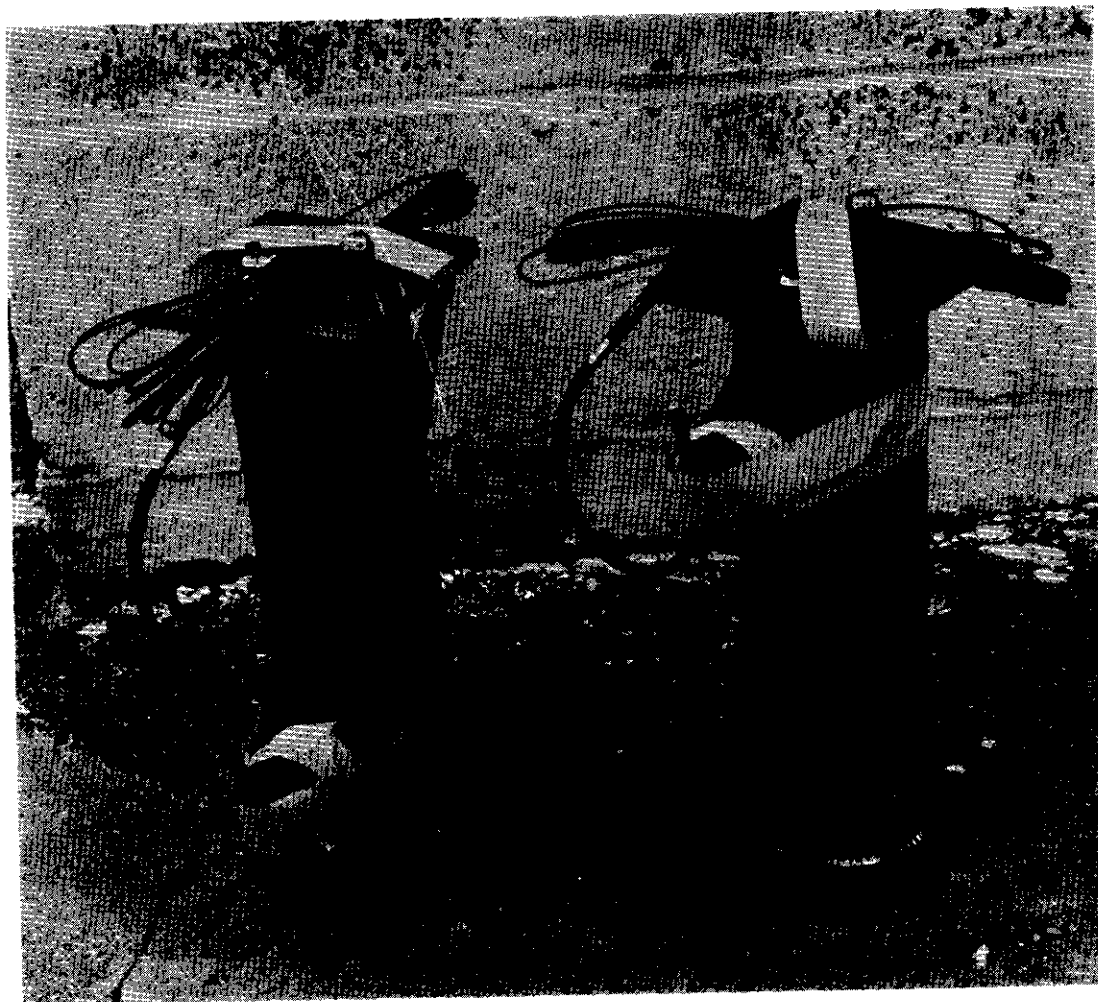


5j

Shot 5-DC. ClO_3F in Titanium A-55 Grenade

FIGURE 6

ClO_3F LOADED TITANIUM A-55 CYLINDERS WITH ATTACHED
3-INCH JET PERFORATION CHARGES



Left - Jet Charge Directed Against Liquid in Lower Half of Cylinder
Right - Jet Charge Directed Against Vapor in Upper Half of Cylinder

FIGURE 7

IMPACT TESTER FOR SUPERATMOSPHERIC PRESSURES

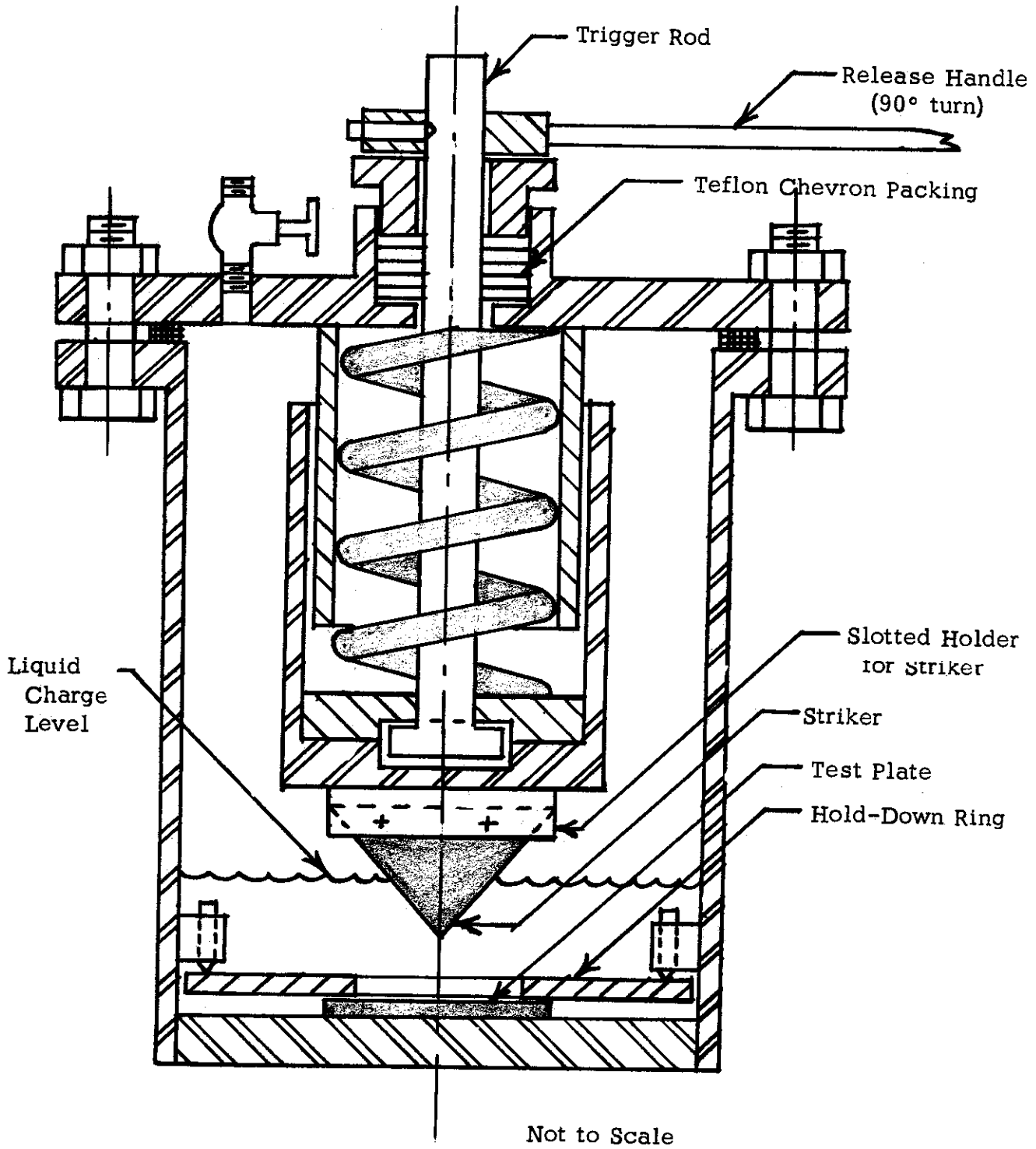


FIGURE 8

METAL STRIKER AND PLATE FOR IMPACT TESTS

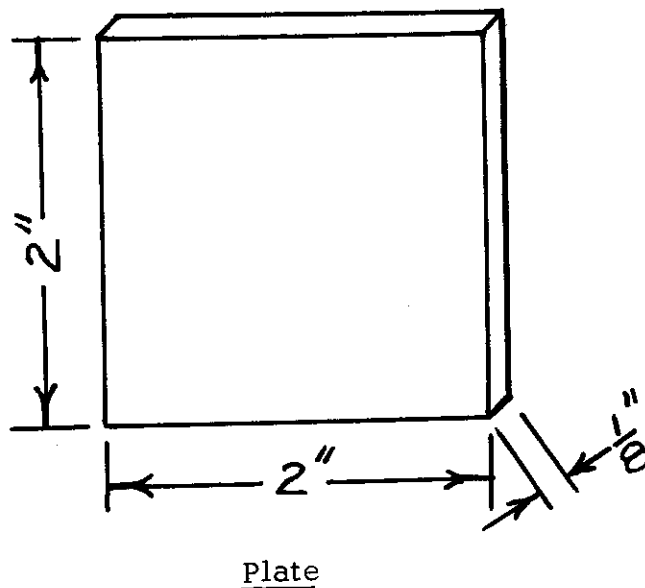
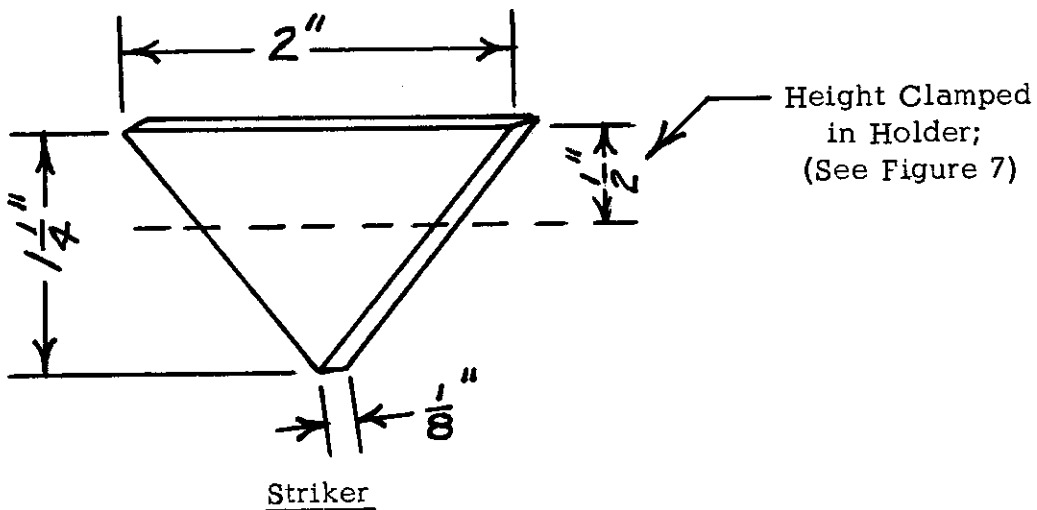


TABLE 1
 VAPOR PRESSURE MEASUREMENTS OF ClO₃F-ClF₃
 MIXTURES

Composition	Vapor Pressure (psia)		
	0°C.	25°C.	54.5°C.
9.7% ClO ₃ F 90.3% ClF ₃	34	65	130
19.8% ClO ₃ F 81.2% ClF ₃	48	88	168
24.9% ClO ₃ F 75.1% ClF ₃	57	103	190
73.5% ClO ₃ F 26.5% ClF ₃	81	155	307

TABLE 2
VAPOR PRESSURES OF $\text{ClO}_3\text{F}-\text{ClF}_3$ MIXTURES
AT 10°C . INTERVALS

Weight % of ClO_3F	Vapor Pressure (psia)						
	0°C .	10°C .	20°C .	30°C .	40°C .	50°C .	60°C .
10.0%	34	44	56	74	94	118	142
25.0%	57	75	93	119	147	180	218
73.5%	81	107	139	177	225	280	350

TABLE 3

LIQUID DENSITIES OF ClO₃F-ClF₃ MIXTURES

Density for 25% ClO ₃ F-75% ClF ₃ Mixture		
Temp. -°C.	Density g/cc	
	Calculated	Measured
20	1.708	--
25	1.690	--
27	1.682	1.68
30	1.672	--

Density for 75% ClO ₃ F-25% ClF ₃ Mixture		
Temp. -°C.	Density - g/cc	
	Calculated	Measured
20	1.514	--
25	1.493	--
29.5	1.473	1.48
30	1.471	--

TABLE 4
LIQUID-VAPOR EQUILIBRIUM COMPOSITIONS
FOR $\text{ClO}_3\text{F}-\text{ClF}_3$ MIXTURES AT 25°C .

Liquid Composition wt. per cent ClO_3F	Vapor Composition wt. per cent ClO_3F
9.4	57.4
20.7	71.9
26.8	77.0
53.8	81.2
77.2	90.5

(See Figure 1 for liquid-vapor diagram.)

TABLE 5

PRELIMINARY COMPATIBILITY TESTS
AT 30°C. MAXIMUM TEMPERATURE

A. Exposure to ClF₃ Liquid in Kel-F Tube

Test Material	Form	Initial Weight g.	Weight Change g.	Observations
Aluminum, AA 1100	.05" sheet	0.1848	-0.0001	No change.
Aluminum, AA 1100	foil	0.0317	--	Vigorous reaction initiated by heat lamp used in warming the ClF ₃ during venting; powder residue left in Kel-F tube.
Aluminum, AA 1100	foil	0.0277	0.0000	No change; no external heat source used in venting the ClF ₃ .
Carbon, Acheson GA grade, National Carbon Co.	block	0.1878	--	Expanded and crumbled to a powder as ClF ₃ temp. rose from 0°C. to 25°C.
Carbon, spectroscopic grade	rod	0.1441	--	Same as for GA carbon
Carbon, Karbate #15, epoxy filler	block	0.2462	--	Crumbled to a granular powder on warming of ClF ₃ to 25°C.
Carbon, high density, Code 82, National Carbon Co.	block	0.2226	--	Crumbling to a fine powder begins well below 25°C. to give a black suspension of carbon powder, in liquid ClF ₃ .
Chromium, 99.3% purity	lump	0.5704	-0.0002	No change.

(Continued)

TABLE 5 (Cont.)

Test Material	Form	Initial Weight g.	Weight Change g.	Observations
Columbium	sheet	0.9034	--	Violent incendiary reaction with bright, bluish-white flame on contact with ClF ₃ at dry ice temp., burning out Kel-F tube.
Columbium	sheet	0.2861	--	Repeat test using liquid N ₂ bath for condensing ClF ₃ ; same incendiary reaction when bath removed and ClF ₃ -Cb warms slightly.
Graphite, spectroscopic grade	rod	0.1224	--	Expanded and crumbled to a powder as ClF ₃ temp. rose from 0° to 25°C.
Graphite, AUT-72 (100% lampblack base, phenolic filler), National Carbon Co.	block	0.2046	--	Disintegrated into flakes on warming of ClF ₃ to 25°C.
Graphite, Karbate #25, epoxy filler	block	0.2516	--	Crumbled to a fine powder on warming of ClF ₃ to 25°C.
Graphite, high density, Code 82, National Carbon Co.	block	0.2827	--	Same as for Code 82 carbon above.
Graphite, Graphitar 39, The U.S. Graphite Co.	block	0.3909	+0.0001	Holds original shape and size, and no disintegration in ClF ₃ at 25°C. Some powdering from surface, therefore apparently some adsorption of ClF ₃ .

(Continued)

TABLE 5 (Cont.)

Test Material	Form	Initial Weight g.	Weight Change g.	Observations
Graphite, Graphitar 39 (Cont.)			-0.0014	Weight loss on heating of ClF ₃ exposed G#39 at 120°C. for 65 hours.
Graphite, Graphitar 67, The U.S. Graphite Co.	block	0.2875	--	Expansion and breakup into smaller, soft lumps and a fine powder on warming ClF ₃ to 25°C.
Kynar* (polyvinylidene fluoride resin)	sheet	0.2757	--	No apparent reaction with ClF ₃ up to 25°C. Sample lost by fusion on drying at 135°C., below the normal fusion point of this plastic (cryst. m.p. 171°C.)
Lead (QQ-L-201a)	sheet	1.8391	-0.0023	Loose, white coating removed by water wash and hand wipe.
Magnesium-AZ31B	sheet	0.0702	-0.0001	No change.
Molybdenum	sheet	0.7393	--	As for Ti reaction, but somewhat longer time necessary for complete solution of the Mo.
Silver	sheet	0.3875	-0.0036	Brown to black film formed on silver; this powdery film washed off by water leaving a dull white surface.
Teflon tape (Permacel)	0.002" thick	0.0611	+0.0001	Unchanged after 1-1/2 hours at 25°C.

*Trade mark - Pennsalt Chemicals Corp.

(Continued)

TABLE 5 (Cont.)

Test Material	Form	Initial Weight g.	Weight Change g.	Observations
Titanium, Ti-100A	sheet	0.0760	--	Vigorous reaction with ClF ₃ below 25°C. until Ti dissolved; normal v.p. for ClF ₃ .
Titanium, comm. pure (Rem-Cru)	sheet	0.2574	--	Same as for Ti-100A alloy.
Titanium, C-120AV-Ti (6Al-4V)	sheet	0.6724	--	Vigorous reaction with ClF ₃ below 25°C. until all Ti dissolved in two hours. Normal ClF ₃ v.p. at end of reaction.
Titanium, A-110AT-Ti	sheet	0.9985	--	As for C-120AV-Ti, except 60 psig pressure in system at end of reaction. IR of gas shows bonds in region where metal hexafluorides show absorption.

B. Exposure to 25% ClO₃F-75% ClF₃ Liquid Mixture in Copper Tube

Test Material	Form	Initial Weight g.	Weight Change g.	Observations
Carbon - spectroscopic grade	rod	0.1962	--	Piece broken into smaller chunks with some powdering and flaking on warming liquid to 25°C.
Columbium	sheet	0.2847	--	After one hr. reaction at 25°C., metal completely reacted leaving a white residue on evaporation of ClF ₃ .

TABLE 5 (Cont.)

Test Material	Form	Initial Weight g.	Weight Change g.	Observations
Graphite - spectroscopic grade	rod	0.2053	--	Graphite expanded and broken into smaller chunks with some powdering on warming liquid to 25°C.
Molybdenum	sheet	0.7775	--	Metal dissolved completely in one hour at 25°C.
Titanium, .C-120AV-Ti	sheet	0.3106	--	Metal dissolved completely in 1-1/2 hours at 25°C.

C. Exposure to ClO₃F Liquid

Test Material	Form	Initial Weight g.	Weight Change g.	Observations
Teflon tape (Permacel), in Copper tube	0.002" thick	0.0528	0.0000	Unchanged after 1 hour at 25°C.
Steel wool (Grade No. 2, Fed. Spec. FF-W-556)	Liquid ClO ₃ F at dry ice temp. poured over wad of steel wool at 25°C. in Monel beaker.			No reaction, even when steel wool wadded into pool of liquid ClO ₃ F at bottom of Monel beaker.

TABLE 6

PRELIMINARY ClF_3 COMPATIBILITY TESTS IN
STAINLESS STEEL CYLINDER UP TO 80°C.

Test Material	Exposure Time at 25°C. hours	Exposure Time at 70-80°C. hours	Initial Weight g.	Weight Change g.	Observations
Teflon (unfilled)	333	--	3.0527	-0.0030	Unchanged
Teflon (unfilled)	71	31.5	2.6662	-0.0007	Unchanged
CaF ₂ filled Teflon (AEC)	333	--	4.7078	-0.0022	Lighter in color
CaF ₂ filled Teflon (AEC)	71	31.5	4.7842	-0.0003	Lighter in color
CaF ₂ filled Teflon (Garlock)	333	--	2.5325	-0.0015	Unchanged (Lot #1)
CaF ₂ filled Teflon (Garlock)	71	31.5	2.5445	+0.0004	Unchanged (Lot #1)
CaF ₂ filled Teflon (Garlock)	17	3	2.9685	+0.0044	Lighter in color (Lot #2)
"			3.3794	+0.0026	Lighter in color (Lot #2)
Chromium, 99.3% purity	40	25.5	2.2743	-0.0002	No change
Aluminum sheet, #1100	36	29.5	1.4460	0.0000	No change
Aluminum foil	36	29.5	0.3363	-0.0003	No change
Silver	23	26	0.0861	-0.0001	No change
"			1.0386	-0.0068	Loose black coating removed by water washing and hand wipe.
"			0.9425	-0.0069	"
Magnesium, AZ31B	22	25	0.2087	-0.0004	Brown tinted film with scattered brown-black powdery residue.
"			0.1795	-0.0004	"

(Continued)

TABLE 6 (Cont.)

Test Material	Exposure Time at 25°C. hours	Exposure Time at 70-80°C. hours	Initial Weight g.	Weight Change g.	Observations
Lead, QQ-L-201a	22.5	25	4.4749	-0.0018	Etched and discolored to shades of yellow, gray and black with small amount of powdery residue.
			4.8043	-0.0043	"

TABLE 7

MATERIALS FOR ClF₃-ClO₃F TESTING

A. Temper and Supplier

Material	Temper	Supplier
Aluminum Alloys		
AA 1060	-0, annealed	Aluminum Co. of America
AA 1100	-0, annealed	Whitehead Metal Products Co., Inc.
AA 2024	-0, annealed	Whitehead Metal Products Co., Inc.
AA 3003	-0, annealed	Whitehead Metal Products Co., Inc.
AA 5052	-0, annealed	Whitehead Metal Products Co., Inc.
AA 7079	-0, annealed	Aluminum Co. of America
Copper Alloys		
ETP Copper	hot rolled, soft	Whitehead Metal Products Co., Inc.
Phosphorized Cop- per (DHP)	hard	The American Brass Company
Beryllium Copper, 2%(Berylco 25)	annealed	The Beryllium Corporation
Phosphor Bronze, 5% Grade A	spring	Whitehead Metal Products Co., Inc.
Aluminum Bronze, 8% (Ampco 8)	annealed	Ampco Metal, Inc.
Yellow Brass	half-hard	The American Brass Company
Rule Brass	half-hard	Whitehead Metal Products Co., Inc.
Nickel Silver, 18% Alloy A	annealed	Whitehead Metal Products Co., Inc.
Cupro-Nickel, 30%	annealed	The American Brass Company
Magnesium Alloys		
AZ31B	-0, annealed	A.R. Purdy Co., Inc.
HK31A	-0, annealed	The Dow Metal Products Co.
HM21A	-T8, solution heat treated, cold worked, artificially aged	The Dow Metal Products Co.
Nickel Alloys		
"A" Nickel	annealed	The International Nickel Co., Inc.
Monel	annealed	The International Nickel Co., Inc.
Inconel	annealed	The International Nickel Co., Inc.
Incoloy	annealed	The International Nickel Co., Inc.

TABLE 7 (Cont.)

Material	Temper	Supplier
Non-Metallics		
*Carbon	various grades	National Carbon Company
*Graphite	various grades	National Carbon Company and The U.S. Graphite Company
Polytetrafluoro- ethylene(Teflon)		The Garlock Packing Co.
25-35% CaF ₂ -filled Teflon		The Garlock Packing Co.
Polychlorotrifluoro- ethylene (Kel-F)	300 grade	Walter B. Gallagher Co., Conshohocken, Pa.
Refractory Metals		
*Molybdenum		Fansteel Metallurgical Corp.
*Columbium		Fansteel Metallurgical Corp.
Stainless Steels		
AISI 304	annealed	The Carpenter Steel Company
AISI 316	annealed	The Carpenter Steel Company
AISI 347	annealed	The Carpenter Steel Company
AISI 403	annealed	The Carpenter Steel Company
Carpenter #20-Cb	annealed	The Carpenter Steel Company
PH 15-7 Mo	RH 950	Armco Steel Corporation
PH 15-7 Mo	TH 1050	Armco Steel Corporation
Low Carbon Steel		
AISI 1010	cold rolled, annealed	Armco Steel Corporation
Titanium Alloys		
*Ti-100A	annealed	Titanium Metals Corp. of America
*C-120AV-Ti (6Al-4V)	annealed	Crucible Steel Co. of America
*A-110AT-Ti	annealed	Crucible Steel Co. of America

*Failed in preliminary exposure to ClF₃; not included in 21-day immersion tests.

(Continued)

TABLE 7 (Cont.)

Additional Metals Tested Only in Wet ClO₃F:

The metals listed below were received and used in various coupon sizes other than the standard form of Figure 2.

Material	Temper	Supplier
Chlorimet 3 (cast)	water quenched	The Duriron Co., Inc.
Durimet 20 (cast)	water quenched	The Duriron Co., Inc.
Gold	annealed	J. Bishop & Co. Platinum Works
Hastelloy C (cast)	solution heat-treated	Haynes Stellite Division, Union Carbide Corp.
Hastelloy C (wrought)	solution heat-treated	Haynes Stellite Division, Union Carbide Corp.
Illium G (cast)	solution heat-treated	Stainless Foundry & Engineering, Inc.
Illium 98 (cast)	solution heat-treated	Stainless Foundry & Engineering, Inc.
Illium R	hot rolled	Stainless Foundry & Engineering, Inc.
Ni-o-nel	annealed	The International Nickel Co., Inc.
Platinum	annealed	J. Bishop & Co. Platinum Works
Silver	annealed	J. Bishop & Co. Platinum Works

(Continued)

TABLE 7 (Cont.)

B. Composition and Density of Alloys

Material	Density g./cc.	Analysis	Composition - Wt. %
Aluminum Alloys AA 1060	2.71	Typical	Al 99.60 min., Cu 0.05, Fe 0.35, Si 0.25, Mn 0.03, Mg.0.03, Zn 0.05, Ti 0.03, Other 0.03 max. each.
AA 1100	2.71	Typical	Al 99.0 min., Cu 0.20, Fe + Si 1.0 max., Mn 0.05, Zn 0.10, Other 0.15 max. total.
AA 2024	2.77	Typical	Al-Bal., Cu 3.8-4.9, Fe 0.50, Si 0.50, Mn 0.3-0.9, Mg.1.2-1.8, Zn 0.25, Cr 0.10, Other 0.15 max. total.
AA 3003	2.73	Typical	Al-Bal., Cu 0.20, Fe 0.7, Si 0.6, Mn 1.0-1.5, Zn 0.10, Other 0.15 max. total.
AA 5052	2.68	Typical	Al-Bal., Cu 0.10, Fe + Si 0.45 max., Mn 0.10, Mg.2.2-2.8, Zn 0.20, Cr 0.15-0.35, Other 0.15 max. total.
AA 6061 (Used only for fabrication of grenades for shock tests)	2.70	Typical	Al-Bal., Cu 0.15-0.40, Fe 0.7, Si 0.4-0.8, Mn 0.15, Mg.0.8-1.2, Zn 0.25, Cr 0.15-0.35, Ti 0.15, Other 0.15 max. total.
AA 7079	2.73	Typical	Al-Bal., Cu 0.4-0.8, Fe 0.40, Si 0.30, Mn 0.10-0.30, Mg.2.9-3.7, Zn 3.8-4.8, Cr 0.10-0.25, Ti 0.10, Other 0.15 max. total.

(Continued)

TABLE 7 (Cont.)

B. Composition and Density of Alloys - (Cont.)

Material	Density g./cc.	Analysis	Composition - Wt. %
Copper Alloys			
Copper, ETP	8.92	Typical	Cu + Ag 99.90 min., O ₂ 0.04
Copper, DHP	8.94	No.74729	Cu + Ag 99.954, P 0.024
Beryllium Copper, 2%(Berylco 25)	8.26	Typical	Be 1.80-2.05, Ni or Co or both 0.20 min., Ni + Co + Fe 0.60 max., Cu + Be + additive elements 99.50 min.
Phosphor Bronze, 5% Grade A	8.86	Typical	Cu 94.75, Sn 5.00, P 0.25
Aluminum Bronze, 8%(Ampco 8)	7.78	Typical	Cu-Bal., Al 6.00-8.00, Fe 1.50- 3.00, Others 0.50 max.
Yellow Brass	8.47	Typical	Cu 64.0-68.5, Pb 0.15 max., Fe 0.05 max., Zn-Bal.
Rule Brass	8.50	Typical	Cu 62.50, Zn 35.00, Pb 2.50
Nickel Silver, 18% Alloy A	8.73	Typical	Cu 65.00, Zn 17.00, Ni 18.00
Cupro-Nickel, 30%	8.94	No.74345	Cu 69.01, Zn <0.10, Pb 0.005, Fe 0.50, Mn 0.32, Ni 30.16 (by difference)
Gold	19.3	Typical	Pure, 24 carat sheet
Magnesium Alloys AZ31B	1.77	Typical	Mg.-Bal., Al 2.5-3.5, Mn 0.20 min., Zn 0.6-1.4, Si 0.10 max., Cu 0.05 max., Ni 0.005 max., Fe 0.005 max., Cu 0.04 max., Others 0.30 max. total.
HK 31A	1.79	Typical	Mg.-Bal., Th 2.5-4.0, Zr 0.45- 1.0, Mn 0.15 max., Others 0.10 max. each, 0.30 max. total.
HM 21A	1.77	Typical	Mg.-Bal., Th 0.45-1.1, Others 0.30 max. total.
Nickel Alloys "A" Nickel	8.89	No.N5849A	Ni 99.52, C 0.02, Mn 0.190, Fe 0.15, S 0.005, Cu 0.050, Si 0.04 Ni 65.57, C 0.06, Mn 0.90, Fe 1.52, S 0.005, Cu 31.77, Si 0.15

TABLE 7 (Cont.)

B. Composition and Density of Alloys - (Cont.)

Material	Density g./cc.	Analysis	Composition - Wt. %
Inconel	8.51	No. NX9933	Ni 75.06, C 0.04, Mn 0.30, Fe 8.11, S 0.007, Cu 0.27, Si 0.31, Cr 15.88
Incoloy	8.02	No. HH6333A	Ni 33.55, C 0.06, Mn 0.76, Fe 44.86, S 0.007, Cu 0.34, Si 0.36, Cr 20.04
Ni-o-nel	8.00	Typical	Ni 38.0-46.0, Cr 19.5-23.5, Mo 2.5-3.5, Cu 1.5-3.0, Mn 1.0 max., Si 0.5 max., C 0.05 max., Ti 0.6-1.2, Fe-Bal.
Chlorimet 3	9.00	No. 01197	Ni 58.59, Cr 18.30, Mo 19.00, Si 1.07, C 0.034, Cu 0.13, Mn 0.75, Fe 2.12, S 0.014
Illium G	8.31	Typical	Ni 56, Cr 22.5, Mo 6.4, Fe 6.5, Cu 6.5, Mn 1.25, Si 0.65, C 0.20
Illium 98	8.3	Typical	Ni 55, Cr 28, Mo 8.5, Cu 5.5, Mn 1.25, Si 0.70, Fe 1.00, C 0.05
Illium R	8.31	Typical	Ni 68, Cr 21, Mo 5.0, Cu 3.0, Fe 1.0, Mn 1.25, Si 0.70, C 0.05
Hastelloy C(cast)	8.94	No. 1484	Ni-Bal., Cr 16.01, W 4.34, Fe 5.78, C 0.07, Si 0.55, Co 0.91, Mn 0.83, V 0.30, Mo 17.00, P 0.007, S 0.014
Hastelloy C (wrought)	8.94	No. 3256	Ni-Bal., Cr 15.82, W 3.34, Fe 5.22, C 0.08, Si 0.52, Co 1.96, Mn 0.71, V 0.26, Mo 16.34, P 0.008, S 0.008
Platinum	21.45	Typical	Pure, electrode grade
Refractory Metals Molybdenum	10.2	Typical	Mo-Bal., C 0.005, O 0.003, N 0.001, Fe 0.003, Ni 0.003, Ca 0.001, Si <0.01, W <0.01

(Continued)

TABLE 7 (Cont.)

B. Composition and Density of Alloys - (Cont.)

Material	Density g./cc.	Analysis	Composition - Wt. %
Columbium (Niobium)	8.55	Typical	Nb-Bal., O 0.03, N 0.03, C 0.015, Ta 0.05, Fe 0.015, Ti 0.01, Zr <0.02, W <0.01, Ni <0.007
Silver	10.5	Typical	Pure, 999 + fine
Stainless Steels			
AISI 304	7.9	No.801744	Fe-Bal., Ni 9.45, Cr 18.65, C 0.06, Mn 0.86, Si 0.65, Mo 0.18
AISI 316	7.98	No.801764	Fe-Bal., Ni 12.48, Cr 17.39, C 0.06, Mn 1.80, Si 0.58, Mo 2.21
AISI 347	8.0	No.47357	Fe-Bal., Ni 11.03, Cr 18.27, C 0.05, Mn 1.58, Si 0.64, Mo 0.40, Cb + 0.86, Ta 0.07
AISI 403	7.75	No.801667	Fe-Bal., Cr 12.15, C 0.13, Mn 0.55, Si 0.40, P 0.012, S 0.011
Carpenter #20-Cb	8.0	No. 18021	Fe-Bal., Ni 28.41, Cr 19.83, C 0.08, Mn 0.73, Si 0.72, Mo 2.39, Cu 3.30, Cb + 0.72
Durimet 20	7.95	No.93005	Fe-Bal., Ni 28.02, Cr 20.35, C 0.04, Si 1.08, Mo 2.38, Cu 3.35
PH 15-7 Mo	7.68	Typical	Fe-Bal., Ni 6.50-7.75, Cr 14.00- 16.00, C 0.09 max., Mn 1.00 max., Si 1.00 max., P 0.04 max., S 0.03 max., Mo 2.00-3.00, Al 0.75-1.50
Low Carbon Steels			
AISI 1010	7.87	Typical	Fe-Bal., C 0.08-0.13, Mn 0.30- 0.60, P 0.040 max., S 0.050 max.
AISI 1018	7.87	Typical	Fe-Bal., C 0.15-0.20, Mn 0.60- 0.90, P 0.040 max., S 0.050 max.

(Continued)

TABLE 7 (Cont.)

B. Composition and Density of Alloys - (Cont.)

Material	Density g./cc.	Analysis	Composition - Wt. %
Titanium Alloys Ti-100A	4.54	No.60-212-417-1	Ti 99.5, Fe 0.2, N 0.10, C 0.06, W 0.01
Ti-55A (Used only for fabrication of pressure vessels for shock tests)	4.5	Typical	Ti 99.4; commercially pure titanium
C-120AV-Ti	4.43	No.G5794	Ti-Bal., C 0.04, H 0.0095, N 0.02, V 4.0, Al 5.6, Fe 0.16, O 0.20
A-110 AT-Ti	4.46	No.D7727	Ti-Bal., C 0.03, H 0.0117, N 0.01, Al 5.4, Sn 2.7

TABLE 8

FACTORS FOR CONVERTING WEIGHT GAIN TO
EQUIVALENT WEIGHT OF REACTED METAL

Material	Alloy Code No.	Weight Gain Factor as Ratio:	
		Alloy/F	Alloy/O
Aluminum 1060	1-1	0.473	1.12
" 1100	1-2	0.473	1.12
" 2024	1-3	0.492	1.17
" 3003	1-4	0.478	1.13
" 5052	1-5	0.477	1.17
" 7079	1-6	0.493	1.17
Copper ETP	2-1	1.68	3.98
" DHP	2-2	1.68	3.98
Beryllium Copper, 2% (Berylco 25)	2-3	1.50	3.55
Phosphor Bronze, 5% Grade A	2-4	1.67	3.96
Aluminum Bronze, 8% (Ampco 8)	2-5	1.40	3.32
Yellow Brass	2-6	1.69	4.00
Rule Brass	2-7	1.72	4.07
Nickel Silver, 18% Alloy A	2-8	1.66	3.93
Cupro-Nickel, 30%	2-9	1.63	3.86
Magnesium AZ31B	3-1	0.638	1.51
" HM21A	3-2	0.651	1.54
" HK31A	3-3	0.658	1.56
"A" Nickel	4-1	1.55	3.67
Monel	4-2	1.57	3.72
Inconel	4-3	1.33	3.15
Incoloy	4-4	1.10	2.61
Stainless Steel 403	7-1	0.972	2.30
" 316	7-2	1.10*	2.41
" 347	7-3	1.01	2.39
" 304	7-4	1.00	2.37
" Carp. #20 Cb	7-5	1.19*	2.58
" PH 15-7 Mo	8-1 } 8-2 }	1.13*	2.29

*Includes correction for loss of volatile fluorides of Mo and Si.

TABLE 8 (Cont.)

Material	Alloy Code No.	Weight Gain Factor as Ratio:	
		Alloy/F	Alloy/O
Low Carbon Steel 1010	8-3	0.980	2.32
	8-4		
	8-5		
Titanium Ti-100A	9-1	--	1.50
" C-120AV-Ti	9-2	--	1.48
" A-110AT-Ti	9-3	--	1.50

(Continued)

TABLE 8 (Cont.)

Procedure for weight gain factor calculation:

In the case of metals exposed to ClF_3 or mixtures of ClF_3 and ClO_3F , and observed weight gain is assumed to be fluoride, the weight ratio Alloy/Fluorine was calculated for each of the alloys used by the following procedure:

- a. The nominal alloy composition in weight % was set down. All minor constituents below 1% were included in the main element weight %.
- b. The weight ratio of Fluorine/Metal was determined for each of the pure metal constituents, assuming metal reaction to the highest oxidation state stable fluoride.
- c. These weight ratios F/M were multiplied by each of the constituent nominal weight % figures to give the weight F/100 g. Alloy contributed by each alloy constituent. The sum of these figures gave the total weight F/100 g. Alloy.

d. Then the weight ratio: Alloy/Fluorine =
$$\frac{1}{100 \left(\frac{\text{WtF}}{100 \text{ g. Alloy}} \right)}$$

This assumes that the fluorine of ClF_3 will react with all of the alloy constituents in the same proportion as their weight percentages in the alloy.

Where the formation of volatile fluorides such as MoF_6 and SiF_4 is indicated, the entire weight of such metal fluoride is subtracted from the total F/100 g. Alloy for the other constituents to give a net effective F/100 g. Alloy weight gain.

For metals exposed to pure ClO_3F , and the observed weight gain is assumed to be oxide, the weight ratio Alloy/Oxygen was calculated by:

- a. In the weight ratio Alloy/F, the proportion of F is taken as 1.00, and $1.00 \times 16/38 = 0.422$ equivalent oxygen
- b. Alloy/O = Alloy/F (1/0.422)

TABLE 9

CORROSION RATES IN 21-DAY LIQUID
IMMERSION TESTS AT 30°C.

Material	Sample Code No.	Liquid Comp. ClO ₃ F-ClF ₃ wt. %	Hours of Exposure	Av. Corrosion Rate mils/yr.	
				C.R. '	C.R. "
Aluminum Alloys					
AA 1061	1-1-1,2	0-100	524	0.01	0.01
"	1-1-5,6	25-75	522	0.00	0.00
"	1-1-11,12	100-0	519	0.01	0.01
AA 1100	1-2-1,2	0-100	524	0.01	0.01
"	1-2-5,6	25-75	522	0.01	0.02
"	1-2-11,12	100-0	519	0.01	0.01
AA 2024	1-3-1,2	0-100	524	0.01	0.01
"	1-3-5,6	25-75	522	0.01	0.01
"	1-3-11,12	100-0	519	0.00	0.00
AA 3003	1-4-1,2	0-100	524	0.01	0.01
"	1-4-5,6	25-75	522	0.01	0.05
"	1-4-11,12	100-0	519	0.01	0.01
AA 5052	1-5-1,2	0-100	524	0.01	0.01
"	1-5-5,6	25-75	522	0.01	0.01
"	1-5-11,12	100-0	519	0.01	0.10
AA 7079	1-6-1,2	0-100	524	0.01	0.01
"	1-6-5,6	25-75	522	0.00	0.00
"	1-6-11,12	100-0	519	0.01	0.16
Copper Alloys					
ETP Copper	2-1-1,2	0-100	520	0.09	0.11
"	2-1-6	25-75	598	0.06 ^a	0.10 ^a
"	2-1-3,4	100-0	520	0.13	0.13
DHP Copper	2-2-1,2	0-100	520	0.12	0.12
"	2-2-5,6	25-75	598	0.13	0.18
"	2-2-3,4	100-0	520	0.17	0.17
Beryllium Copper, 2% (Berylco 25)	2-3-1,2	0-100	520	0.03	0.11
"	2-3-5,6	25-75	598	0.08	0.15
"	2-3-3,4	100-0	520	0.19	0.19

(Continued)

TABLE 9 (Cont.)

Material	Sample Code No.	Liquid Comp. ClO ₃ F-ClF ₃ wt. %	Hours of Exposure	Av. Corrosion Rate mils/yr.	
				C.R. '	C.R. "
Copper Alloys(Cont.)					
Phosphor Bronze, 5% Grade A	2-4-1, 2	0-100	520	0.13	0.15
"	2-4-5, 6	25-75	598	0.10	0.10
"	2-4-3, 4	100-0	520	0.12	0.12
Aluminum Bronze, 8% Ampco 8)	2-5-1, 2	0-100	520	0.03	0.03
"	2-5-5, 6	25-75	598	0.02	0.05
"	2-5-3, 4	100-0	520	0.10	0.10
Yellow Brass	2-6-1, 2	0-100	524	0.03	0.03
"	2-6-5, 6	25-75	596	0.04	0.15
"	2-6-3, 4	100-0	415	0.33 ^b	0.33 ^b
"	2-6-9, 10	100-0	567	0.11 ^c	0.18 ^c
Rule Brass	2-7-1, 2	0-100	524	0.04	0.07
"	2-7-5, 6	25-75	596	0.04	0.24
"	2-7-3, 4	100-0	415	0.30 ^b	0.30 ^b
"	2-7-9, 10	100-0	567	0.10 ^c	0.12 ^c
Nickel Silver, 18% Alloy A	2-8-1, 2	0-100	524	0.02	0.03
"	2-8-5, 6	25-75	596	0.01	0.06
"	2-8-3, 4	100-0	415	0.21 ^b	0.21 ^b
"	2-8-9, 10	100-0	567	0.11 ^c	0.11 ^c
Cupro-Nickel, 30%	2-9-1, 2	0-100	524	0.01	0.03
"	2-9-5, 6	25-75	596	0.00	0.04
"	2-9-3, 4	100-0	415	0.18 ^b	0.18 ^b
"	2-9-9, 10	100-0	567	0.05 ^c	0.05 ^c
Magnesium Alloys					
AZ31B	3-1-1, 2	0-100	520	0.03	0.03
"	3-1-5, 6	25-75	520	0.03	0.09
"	3-1-3, 4	100-0	524	0.09	0.15
HK31A	3-3-1, 2	0-100	520	0.01	0.13
"	3-3-5, 6	25-75	520	0.03	0.03
"	3-3-3, 4	100-0	524	0.08	0.22
HM21A	3-2-1, 2	0-100	520	0.03	0.05
"	3-2-5, 6	25-75	520	0.04	0.12
"	3-2-3, 4	100-0	524	0.13	0.40

(Continued)

TABLE 9 (Cont.)

Material	Sample Code No.	Liquid Comp. ClO ₃ F-ClF ₃ wt. %	Hours of Exposure	Av. Corrosion Rate mils/yr.	
				C.R. ¹	C.R. ²
Nickel Alloys					
"A" Nickel	4-1-1,2	0-100	522	0.00	0.00.
"	4-1-5,6	25-75	526	0.01	0.01
"	4-1-3,4	100-0	521	0.01	0.01
Monel	4-2-1,2	0-100	522	0.01	0.01
"	4-2-5,6	25-75	526	0.01	0.01
"	4-2-3,4	100-0	521	0.02	0.02
Inconel	4-3-1,2	0-100	522	0.00	0.00
"	4-3-5,6	25-75	526	0.00	0.00
"	4-3-3,4	100-0	521	0.00	0.00
Incoloy	4-4-1,2	0-100	522	0.01	0.01
"	4-4-5,6	25-75	526	0.01	0.01
"	4-4-3,4	100-0	521	0.00	0.00
Non-Metallics					
Teflon	5-1-1,2	0-100	520	0.70%	Wt. Gain
"	5-1-5,6	25-75	520	2.92%	" "
"	5-1-3,4	100-0	524	0.32%	" "
Teflon, CaF ₂ -filled	5-2-1,2	0-100	520	0.16%	" "
"	5-2-5,6	25-75	520	0.03%	" "
"	5-2-3,4	100-0	524	0.03%	" "
Kel-F	5-3-1,2	0-100	520	1.02%	" "
"	5-3-5,6	25-75	520	4.44%	" "
"	5-3-3,4	100-0	524	0.81%	" "
Kynar, polyvinyl- idene fluoride resin	5-4-3,4	100-0	520	0.37%	" "
Stainless Steels					
AISI 304	7-4-1,2	0-100	383	0.00 ^b	0.02 ^b
"	7-4-11,12	0-100	568	0.00 ^c	0.03 ^c
"	7-4-5,6	25-75	574	0.01	0.02
AISI 316	7-2-1,2	0-100	383	0.02 ^b	0.03 ^b
"	7-2-11,12	0-100	568	0.01 ^c	0.06 ^c
"	7-2-5,6	25-75	574	0.01	0.07
"	7-2-3,4	100-0	521	0.01	0.05
AISI 347	7-3-1,2	0-100	383	0.05 ^b	0.05 ^b
"	7-3-11,12	0-100	568	0.03 ^c	0.09 ^c
"	7-3-5,6	25-75	574	0.02	0.06
"	7-3-3,4	100-0	521	0.00	0.05

(Continued)

TABLE 9 (Cont.)

Material	Sample Code No.	Liquid Comp. ClO ₂ F-ClF ₃ wt. %	Hours of Exposure	Av. Corrosion Rate mils/yr.	
				C.R. '	C.R. "
Stainless Steels(Cont.)					
AISI 403	7-1-1,2	0-100	383	0.02 ^b	0.04 ^b
"	7-1-11,12	0-100	568	0.01 ^c	0.07 ^c
"	7-1-5,6	25-75	574	0.02	0.07
"	7-1-3,4	100-0	521	0.01	0.03
Carpenter #20-Cb	7-5-1,2	0-100	383	0.01 ^b	0.02 ^b
"	7-5-11,12	0-100	568	0.01 ^c	0.05 ^c
"	7-5-5,6	25-75	574	0.00	0.04
"	7-5-3,4	100-0	521	0.01	0.03
PH 15-7 Mo (Cond. RH 950)	8-1-1,2	0-100	522	0.01	0.01
"	8-1-5,6	25-75	571	0.01	0.01
"	8-1-3,4	100-0	521	0.01	0.01
PH 15-7 Mo (Cond. TH 1050)	8-2-1,2	0-100	522	0.01	0.01
"	8-2-5,6	25-75	571	0.01	0.01
"	8-2-3,4	100-0	521	0.01	0.01
Low Carbon Steel					
AISI 1010	8-3-1,2	0-100	523	0.00	0.01
"	8-3-5,6	25-75	571	0.01	0.02
"	8-3-3,4	100-0	521	0.01	0.01
AISI 1010 (Coated w. Fosbond 40*)	8-4-1,2	0-100	523	0.00	0.04
"	8-4-5,6	25-75	571	0.00	0.06
"	8-4-3,4	100-0	521	0.00	0.00
AISI 1010 (Coated w. Fosbond 27*)	8-5-1,2	0-100	523	0.01	0.01
"	8-5-5,6	25-75	571	0.02	0.02
"	8-5-3,4	100-0	521	0.02	0.02
Titanium Alloys					
Ti-100A	9-1-1,2	100-0	524	0.04	0.04
C-120AV-Ti	9-2-1,2	100-0	524	0.04	0.04
A-110AT-Ti	9-3-1,2	100-0	524	0.07	0.07

(Continued)

TABLE 9 (Cont.)

Notes:

^aSingle sample; duplicate damaged in handling.

^bRupture disc on test tank failed before scheduled end of test; entire test repeated.

^cRepeat test.

*Trade mark - Pennsalt Chemicals Corporation

Fosbond 40 - a zinc phosphate type coating

Fosbond 27 - an alkali phosphate type coating

C.R.' - Based on ΔW_a (Weight change first observed after exposure and drying of test piece.)

C.R." - Based on ΔW_e (Cumulative weight change, including correction for fluoride or oxide film removed by standard erasure procedure.)

See Exhibit 6 for outline of corrosion rate calculations.

TABLE 10

CORROSION RATES IN 21-DAY VAPOR IMMERSION TESTS AT 30°C.

Material	Sample Code No.	Vapor Exposure	Hours of Exposure	Av. Corrosion Rate mils/yr.	
				C.R. '	C.R. "
Aluminum 1100	1-2-7, 8	ClF ₃	501	0.02	0.02
"	1-2-9, 10	ClO ₃ F	497	0.02	0.02
Copper, ETP	2-1-7, 8	ClF ₃	501	0.08	0.11
"	2-1-9, 10	ClO ₃ F	497	0.11	0.13
Aluminum Bronze, 8% (Ampco 8)	2-5-7, 8	ClF ₃	501	0.10	0.15
"	2-5-9, 10	ClO ₃ F	497	0.12	0.12
Yellow Brass	2-6-7, 8	ClF ₃	501	0.22	0.60
"	2-6-15, 16	ClO ₃ F	497	0.78	0.78
Magnesium AZ31B	3-1-7, 8	ClF ₃	501	0.04	0.20
"	3-1-9, 10	ClO ₃ F	497	4.5	4.5
"A" Nickel	4-1-7, 8	ClF ₃	501	0.01	0.06
"	4-1-9, 10	ClO ₃ F	497	0.53	0.53
Monel	4-2-7, 8	ClF ₃	501	0.02	0.02
"	4-2-9, 10	ClO ₃ F	497	3.8	3.8
Stainless Steel 403	7-1-15, 16	ClF ₃	501	0.11	0.13
"	7-1-17, 18	ClO ₃ F	497	0.03	0.08
Stainless Steel 316	7-2-15, 16	ClF ₃	501	0.02	0.09
"	7-2-17, 18	ClO ₃ F	497	0.03	0.11
Low Carbon Steel 1010	8-3-7, 8	ClF ₃	501	0.04	0.07
"	8-3-9, 10	ClO ₃ F	497	0.03	0.05

Notes: C.R. ' based on W_a (Weight change first observed after exposure and drying.)
 C.R. " based on W_e (Cumulative weight change, including correction for fluoride or oxide film removed by standard erasure procedure.)

TABLE 11

WEIGHT CHANGES IN PASSIVATION - PRESERVATION TESTS

Material	Sample Code No.	Successive Exposures	Exposure Time hours or days	Weight Change per Exposure mg.
Aluminum 1100 " " " Copper, ETP " " Aluminum Bronze, 8% (Ampco 8) "	1-2-15	ClF ₃ Liq.	65 h	0.0
		Air	51 d	+0.1
		ClF ₃ Liq.	164 h	+0.1
	1-2-16	ClF ₃ Liq.	64 h	0.0
		Air	51 d	+0.1
		ClF ₃ Liq.	164 h	+0.1
	1-2-17	(Control)		
		Air	51 d	+0.2
		ClF ₃ Liq.	164 h	+0.1
	1-2-18	(Control)		
		Air	51 d	+0.1
		ClF ₃ Liq.	164 h	+0.1
2-1-11	2-1-11	ClF ₃ Liq.	65 h	+0.5
		Air	51 d	+0.1
		ClF ₃ Liq.	161 h	+0.9
	2-1-12	ClF ₃ Liq.	65 h	+0.6
		Air	51 d	+0.1
		ClF ₃ F Liq.	161 h	+1.1
	2-1-15	(Control)		
		Air	51 d	+0.1
		ClF ₃ Liq.	161 h	+1.0
	2-1-16	(Control)		
		Air	51 d	0.0
		ClF ₃ Liq.	161 h	+1.4
2-5-11	2-5-11	ClF ₃ Liq.	65 h	+0.2
		Air	51 d	+0.2
		ClF ₃ Liq.	161 h	+0.5
	2-5-12	ClF ₃ Liq.	65 h	+0.2
		Air	51 d	+0.2
		ClF ₃ Liq.	161 h	+0.4

(Continued)

TABLE 11 (Cont.)

Material	Sample Code No.	Successive Exposures	Exposure Time hours or days	Weight Change per Exposure mg.
Aluminum Bronze, 8% (Ampco 8) - Cont.	2-5-15	(Control)		
		Air	51 d	+0.1
		ClF ₃ Liq.	161 h	+0.5
"	2-5-16	(Control)		
		Air	51 d	+0.1
		ClF ₃ Liq.	161 h	+0.4
Yellow Brass	2-6-17	ClF ₃ Liq.	65 h	+0.5
		Air	51 d	+0.2
		ClF ₃ Liq.	161 h	+0.5
"	2-6-18	ClF ₃ Liq.	65 h	+0.5
		Air	51 d	+0.2
		ClF ₃ Liq.	161 h	+0.6
"	2-6-19	(Control)		
		Air	51 d	+0.2
		ClF ₃ Liq.	161 h	+0.7
"	2-6-20	(Control)		
		Air	51 d	+0.2
		ClF ₃ Liq.	161 h	+0.7
Magnesium AZ31B	3-1-11	ClF ₃ Liq.	65 h	+0.2
		Air	51 d	+2.0
		ClF ₃ Liq.	164 h	0.0
"	3-1-12	ClF ₃ Liq.	65 h	+0.1
		Air	51 d	+2.5
		ClF ₃ Liq.	164 h	0.0
"	3-1-15	(Control)		
		Air	51 d	+1.5
		ClF ₃ Liq.	164 h	+0.2
"	3-1-16	(Control)		
		Air	51 d	+1.5
		ClF ₃ Liq.	164 h	+0.2

(Continued)

TABLE 11 (Cont.)

Material	Sample Code No.	Successive Exposures	Exposure Time hours or days	Weight Change per Exposure mg.	
"A" Nickel	4-1-15	ClF ₃ Liq.	65 h	+0.1	
		Air	51 d	+0.2	
		ClF ₃ Liq.	164 h	0.0	
	"	4-1-16	ClF ₃ Liq.	65 h	+0.1
			Air	51 d	+0.2
			ClF ₃ Liq.	164 h	+0.1
	"	4-1-17	(Control)		
			Air	51 d	+0.2
			ClF ₃ Liq.	164 h	+0.1
	"	4-1-18	(Control)		
			Air	51 d	+0.3
			ClF ₃ Liq.	164	0.0
Monel	4-2-15	ClF ₃ Liq.	65 h	+0.1	
		Air	51 d	+0.3	
		ClF ₃ Liq.	163 h	+0.3	
	"	4-2-16	ClF ₃ Liq.	65 h	+0.1
			Air	51 d	+0.2
			ClF ₃ Liq.	163 h	+0.3
	"	4-2-17	(Control)		
			Air	51 d	+0.3
			ClF ₃ Liq.	163 h	+0.4
	"	4-2-18	(Control)		
			Air	51 d	+0.3
			ClF ₃ Liq.	163 h	+0.4
Stainless Steel 403	7-1-19	ClF ₃ Liq.	65 h	-0.1	
		Air	51 d	+0.3	
		ClF ₃ Liq.	163 h	+0.2	
	"	7-1-20	ClF ₃ Liq.	65 h	-0.1
			Air	51 d	+0.2
			ClF ₃ Liq.	163 h	+0.2

(Continued)

TABLE 11 (Cont.)

Material	Sample Code No.	Successive Exposure	Exposure Time hours or days	Weight Change per Exposure mg.
Stainless Steel 403 (Cont.)	7-1-21	(Control)		
		Air	51 d	0.0
		ClF ₃ Liq.	163 h	-0.1
"	7-1-22	(Control)		
		Air	51 d	0.0
		ClF ₃ Liq.	163 h	0.0
Stainless Steel 316	7-2-19	ClF ₃ Liq.	65 h	-0.1
		Air	51 d	+0.2
		ClF ₃ Liq.	163 h	+0.2
"	7-2-20	ClF ₃ Liq.	65 h	-0.1
		Air	51 d	+0.1
		ClF ₃ Liq.	163 h	+0.1
"	7-2-21	(Control)		
		Air	51 d	0.0
		ClF ₃ Liq.	163 h	0.0
"	7-2-22	(Control)		
		Air	51 d	0.0
		ClF ₃ Liq.	163 h	-0.2
Low Carbon Steel 1010	8-3-13	ClF ₃ Liq.	65 h	+0.2
		Air	51 d	+2.3
		ClF ₃ Liq.	163 h	+1.1
	"	8-3-14	ClF ₃ Liq.	65 h
Air			51 d	+3.1
ClF ₃ Liq.			163 h	+1.1
"	8-3-15	(Control)		
		Air	51 d	+2.2
		ClF ₃ Liq.	163 h	+1.1
"	8-3-16	(Control)		
		Air	51 d	+2.5
		ClF ₃ Liq.	163 h	+1.3

(Continued)

TABLE 11 (Cont.)

Material	Sample Code No.	Successive Exposure	Exposure Time hours or days	Weight Change per Exposure mg.	
Low Carbon Steel 1010 with Fosbond 40* coating (zinc phosphate type)	8-4-7	ClF ₃ Liq.	20 h	-0.1	
		Air	49 d	-0.5	
		ClF ₃ Liq.	163 h	+1.0	
	8-4-8	ClF ₃ Liq.	20 h	0.0	
		Air	49 d	-1.2	
		ClF ₃ Liq.	163 h	+0.6	
	"	8-4-9	(Control)		
			Air	49 d	+0.4
	"	8-4-10	ClF ₃ Liq.	163 h	+0.3
			(Control)		
	Low Carbon Steel 1010 with Fosbond 27* coating (alkali metal phosphate type)	8-5-7	ClF ₃ Liq.	20 h	-0.2
			Air	49 d	+1.0
ClF ₃ Liq.			163 h	+0.4	
8-5-8		ClF ₃ Liq.	20 h	-0.4	
		Air	49 h	+1.8	
		ClF ₃ Liq.	163 h	0.0	
8-5-9		(Control)			
		Air	49 d	+0.5	
8-5-10		ClF ₃ Liq.	163 h	+0.5	
		(Control)			
8-5-10		Air	49 d	+0.5	
		ClF ₃ Liq.	163 h	+0.3	

*Trade mark--Pennsalt Chemicals Corporation

(Continued)

Metal Test Pieces:

These were of the standard form shown in Figure 2.

Exposed area was in the range of 18.4-20.1 cm.².

Weights ranged from 4.1 g. for magnesium to 23.8 g. for copper.

Temperature: 30°C. for all exposures.

TABLE 12

CORROSION RATES IN WET PERCHLORYL FLUORIDE AT 30°C.

Test Series #1: Charge--ClO₃F Containing 0.2 Wt. % H₂O; Hours of Exposure - 519

Material	Sample Code No.	Sample Exposure	Av. Corrosion Rate - C.R." mils/yr.	Exposed Surface After Cleaning
Aluminum 1100	1-2-15, 16	Vapor	3.6	Fine pitting etch of entire surface.
"	1-2-17, 18	Liquid	4.2	"
Copper, ETP	2-1-15, 16	Vapor	1.1	Ripple etch and a few shallow pits.
"	2-1-17, 18	Liquid	1.3	More uniform etch and less pitting than 15 & 16.
Aluminum Bronze, 8% (Ampco 8)	2-5-15, 16	Vapor	1.3	Mild, uniform etch
"	2-5-17, 18	Liquid	3.7	"
Yellow Brass	2-6-15, 16	Vapor	8.4	Scattered pitting type etch.
"	2-6-17, 18	Liquid	4.6	As for 15 & 16, but less severe.
Magnesium AZ31B	3-1-15, 16	Vapor	3.8	Heavy, pitting etch
"	3-1-17, 18	Liquid	2.2	Light, scattered etch
"A" Nickel	4-1-15, 16	Vapor	1.1	Several pits and etch spots.
"	4-1-17, 18	Liquid	2.4	Larger area of etch and pitting is finer than 15 & 16.
Monel	4-2-15, 16	Vapor	6.2	Severe pitting and etching.
"	4-2-17, 18	Liquid	3.8	Etching of surface and a few shallow pits.
Stainless Steel 403	7-1-15, 16	Vapor	0.02 ^a	Unchanged.
"	7-1-17, 18	Liquid	0.03 ^a	Unchanged.
Stainless Steel 316	7-2-15, 16	Vapor	0.03 ^a	Unchanged.
"	7-2-17, 18	Liquid	0.04 ^a	Unchanged.
Low Carbon Steel 1010	8-3-15, 16	Vapor	13.3	Severely etched and deeply pitted over entire surface.

(Continued)

TABLE 12 (Cont.)

Test Series #1 (Continued)

Material	Sample Code No.	Sample Exposure	Av. Corrosion Rate - C.R." mils/yr.	Exposed Surface After Cleaning
Low Carbon Steel 1010 (Cont.)	8-3-17, 18	Liquid	8.5	Etched, with a fine, shallow pitting over entire surface.
Titanium Ti-100A	9-1-15, 16	Vapor	0.01 ^a	Unchanged.
"	9-1-17, 18	Liquid	0.02 ^a	"
Titanium C-120AV-Ti	9-2-15, 16	Vapor	0.02 ^a	"
"	9-2-17, 18	Liquid	0.02 ^a	"

^aSee Section II-7 for a discussion of these apparently low corrosion rates.

Test Series #2: Charge--ClO₃F Containing 1.0 Wt. % H₂O; Hours of Exposure - 521

Stainless Steel 403	7-1-7, 8	Vapor	51.6	Severe and deep pitting over entire surface.
"	7-1-9, 10	Liquid	38.9	Somewhat less severe pitting than 7 & 8.
Stainless Steel 316	7-2-7, 8	Vapor	2.1	Several deep pits in area of support hole.
"	7-2-9, 10	Liquid	0.4	Pitting etch in area of support hole.
Titanium C-120AV-Ti	9-2-11, 12	Vapor	0.1	One sample showed etch in area of support hole.
"	9-2-13, 14	Liquid	11.4	Scattered etch.

Test Series #3: Charge--ClO₃F Containing 1.0 Wt. % H₂O; Hours of Exposure - 525

Stainless Steel 347	7-3-7, 8	Vapor	7.7	Crater-like pits around support hole.
"	7-3-9, 10	Liquid	2.7	As for 7 & 8, but less severe.
Stainless Steel 304	7-4-7, 8	Vapor	4.0	Crater-like pits around support hole.
"	7-4-9, 10	Liquid	0.6	As for 7 & 8, but less severe.

TABLE 12 (Cont.)

Test Series #3 (Continued)

Material	Sample Code No.	Sample Exposure	Av. Corrosion Rate - C.R." mils/yr.	Exposed Surface After Cleaning
S.S., Carp. #20-Cb	7-5-7-8	Vapor	0.5 ^a	One deep pit on #8 only at support hole.
"	7-5-9,10	Liquid	0.1	Two small pits on #9 only at support hole.

^aIndividual C.R."s: 7-5-7, 0.1 mils/yr.; 7-5-8, 0.9 mils/yr.

Test Series #4: Charge--ClO₃F Containing 1.0 Wt. % H₂O; Hours of Exposure - 518

Illium G	4-6-3,4	Vapor	0.2	Unchanged.
"	4-6-1,2	Liquid	0.4	"
Illium 98	4-7-3,4	Vapor	0.3	"
"	4-7-1,2	Liquid	1.7	"
Illium R	4-8-3,4	Vapor	0.2	Localized ripple etch at one edge.
"	4-8-1,2	Liquid	0.2	As for 3 & 4.
Hastelloy C (cast)	4-9-3,4	Vapor	0.2	Fine, shallow pitting at several points on faces.
"	4-9-1,2	Liquid	0.1	Shallow pitting on face of #1 only.
Hastelloy C (wrought)	4-10-3,4	Vapor	1.6	Satin smooth
"	4-10-1,2	Liquid	1.9	Satin Smooth

Test Series #5: Charge--ClO₃F Containing 1 Wt. % H₂O; Hours of Exposure - 569

Inconel	4-3-7,8	Vapor	0.4	Scattered, deep pin-point pits, more numerous on cut edges; no pitting at support hole.
"	4-3-9,10	Liquid	0.9	As for 7 & 8, but pits more numerous and larger.

(Continued)

TABLE 12 (Cont.)

Test Series #5: (Continued)

Material	Sample Code No.	Sample Exposure	Av. Corrosion Rate - C.R." mils/yr.	Exposed Surface After Cleaning
Incoloy	4-4-7, 8	Vapor	0.01	Unchanged.
"	4-4-9, 10	Liquid	0.4 ^a	A few pits on face of #10 only.
Chlorimet 3	4-5-1, 2	Vapor	6.2	Unchanged.
"	4-5-3, 4	Liquid	7.6	"
Durimet 20	7-6-1, 2	Vapor	0.04	"
"	7-6-3, 4	Liquid	0.04	"
Ni-o-nel	7-7-1, 2	Vapor	0.04	"
"	7-7-3, 4	Liquid	0.05	"

^aIndividual C.R."s: 4-4-9, 0.01 mils/yr.; 4-4-10, 0.9 mils/yr.

Test Series #6: Charge--ClO₃F Containing 1 Wt. % H₂O; Hours of Exposure - 524

Nickel Silver, 18% Alloy A	2-8-7, 8	Vapor	5.5	Localized etch and shallow pitting around support hole.
"	2-8-15, 16	Liquid	11.4 ^a	15 similar to 7 & 8; 16 had heavy but uniform etch.
Cupro-Nickel, 30%	2-9-7, 8	Vapor	2.2	Rough etch around support hole.
"	2-9-15, 16	Liquid	2.2	As for 7 & 8 plus scattered etch spots.
Gold	10-1-1, 2	Vapor	0.00	Unchanged.
"	10-1-3, 4	Liquid	0.02	"
Platinum	10-2-1, 2	Vapor	0.01	"
"	10-2-3, 4	Liquid	0.00	"
Silver ^b	10-3-1, 2	Vapor	0.4	Smooth, matte finish
"	10-3-3, 4	Liquid	0.4	"

^aIndividual C.R."s: 2-8-15, 6.6 mils/yr.; 2-8-16, 16.2 mils/yr.

^bSpecial cleaning of tarnished silver before test exposure:

- Pickle 10 secs. at 30°C. in 1/1 (V/V) HNO₃
- Rinse, dry
- Erase with rubber eraser to remove milky film
- Rinse, dry
- Vapor degrease in boiling trichloroethylene

(Continued)

TABLE 12 (Cont.)

Note: C.R." corrosion rate based on observed weight loss after removal of corrosion products by wet scrubbing with stiff nylon brush using a household type powder cleanser, followed by drying and the standard erasure cleaning described in Exhibit 4.

TABLE 13

GRENADE TESTS

NN - Shots at National Northern test range
DC - Shots at Drum Company test range

Shot No.	Grenade	Chemical Charge			Results
		Material	Wt. -g.	Calc. % Fill at 30°C.	
1-NN	Low Carbon Steel 1018	H ₂ O	340	81	Drum cover lifted only a few feet; no distortion of drum; slight bulging of cover.
2-NN	Aluminum 2024	H ₂ O	340	78	
12-NN	Aluminum 2024	Air, atm. p.	--	--	Drum cover lifted a little higher than in #1-NN with pronounced outward bulge; no distortion of drum.
1-DC	Aluminum 6061	Air, atm. p.	--	--	Drum cover lifted 10-15 feet with larger outward bulge than #2-NN; slight distortion of drum. Grenade fragments show grooving and burning over entire length of side wall.
6-DC	Titanium A-55	Air, atm. p.	--	--	About same as shot 12-NN.
3-NN	Low Carbon Steel 1018	ClF ₃	440	58	Effect on drum same as in #1-NN. Top and bottom of grenade blown off at weld, but body remained in one piece.

(Continued)

TABLE 13 (Cont.)

Shot No.	Grenade	Chemical Charge			Results
		Material	Wt. -g.	Calc. % Fill at 30°C.	
4-NN	Stainless Steel 316	ClF ₃	440	60	As for #3-NN, with greater bulging of drum so lower ring also is partially erased. Drum split lengthwise into 2 pieces; both rings largely erased; cover sharply bulged.
5-NN	Aluminum 2024	ClF ₃	427	55	
6-NN	Low Carbon Steel 1018	25% ClO ₃ F 75% ClF ₃	442	63	Drum cover bulge about equal to that of #4-NN. Drum bulge slightly greater than #6-NN so both rings are largely erased. As for #5-NN with a noticeably greater center bulging and drawing of the drum wall so that paint coating is broken at many points
7-NN	Stainless Steel 316	25% ClO ₃ F 75% ClF ₃	438	64	
8-NN	Aluminum 2024	25% ClO ₃ F 75% ClF ₃	433	59	
9-NN	Low Carbon Steel 1018	75% ClO ₃ F 25% ClF ₃	428	69	Drum split open along and near, but not at seam; both rings partially erased as in #6-NN.
10-NN	Stainless Steel 316	75% ClO ₃ F 25% ClF ₃	445	75	
4-DC	Aluminum 6061 (No photograph in Figure 5)	75% ClO ₃ F 25% ClF ₃	602	74	About same as #7-NN, with possibly greater erasure of the two rings. Explosive charge failed to detonate properly; imperfect shot.
11-NN	Low Carbon	ClO ₃ F	456	78	Possibly greater drum bulging than in #10; paint coating broken at several points; drum shows incipient splitting

(Continued)

TABLE 13 (Cont.)

Shot No.	Grenade	Chemical Charge			Results
		Material	Wt. -g.	Calc. % Fill at 30°C.	
13-NN	Stainless Steel 316	ClO ₃ F (Test not completed, and no photograph in Figure 5)			
3-DC	Aluminum 6061	ClO ₃ F	574	75	Similar to #9-NN, but with complete separation of drum bottom, and greater opening up of drum into a sheet.
2-DC	Titanium A-55	ClO ₃ F	525	69	
5-DC	Titanium A-55	ClO ₃ F	567	74	Very high order explosion; top and bottom of drum blown off and body split into 3 pieces. Only top and bottom and a small fragment of side wall of grenade recovered. About same as #2-DC

Notes:

*The sheet steel drums had reinforcing ring corrugations at the one-third and two-third levels. The degree to which these corrugations were erased in the explosive bulging of the drums was used as one measure of the explosive enhancement.

Grenades used in National Northern shots were 2.75" I.D.; those used in Drum Company shots were 3.068" I.D.

See Figure 5 for photographs showing effect of grenade shots on drums.

TABLE 14
DIE SPRINGS USED AS ENERGY SOURCE FOR IMPACT TESTS

Spring No.	Code No.	O.D. Inches	Length Inches	Force Constant of Spring*		Recommended Maximum Compression (Inches)
				Lbs./0.1" Deflection	k=lbs./ft.	
1	10-YM-40	1	4	18.5	2,220	1.48
2	10-YH-40	1	4	40	4,800	1.20
3	20-Y-50	2	5	42	5,040	2.50
4	20-YM-50	2	5	56	6,720	1.85
5	20-YH-50	2	5	104	12,480	1.50

Die springs made from chrome vanadium steel

*Taken from Catalog 600, Section C of Standard Die Set Co., Providence, R.I.

APPENDIX II

TABLE 15
IMPACT TESTS

Run No.	Test Metals		Liquid Charge	Energy Release by Spring ft. -lbs.	Crush of Striker Tip inches	Effect of Impact on: (naked eye and 100x)	
	Striker	Plate				Striker	Plate
3	Aluminum 1100	Aluminum 1100	None-(in air)	20	1/4	Tip compressed to rectangle.	Vee shaped dent 5/16" x 3/16"
32	403 S.S.	"	None-(in air)	66	1/16	Tip compressed slightly.	Pierced (1/8") by striker in vee shaped cut.
2	Aluminum 1100	"	ClF ₃	20	7/32	Tip compressed to rectangle.	Vee shaped dent 5/16 x 3/16"; fine, incipient pitting etch in ring 1/4" wide around dent.
22	403 S.S.	"	ClF ₃	62	1/16	Tip compressed to oval 1/16"x7/32".	Pierced (1/8") by striker in vee shaped cut; oval compressed area 1/4" x 11/32" on under side surrounding cut.
12	Aluminum 1100	"	ClO ₃ F	15	5/32	Tip compressed to rectangle; free end of striker bent to one side.	Deep, vee shaped dent
30	403 S.S.	"	ClO ₃ F	64	1/32	Tip compressed slightly.	Pierced (1/8") by striker in vee shaped cut.
8	Copper, ETP	Copper, ETP	None-(in air)	22	1/16	Tip compressed and rounded.	Vee shaped dent.

(Continued)

APPENDIX II

TABLE 15 (Cont.)

Run No.	Test Metals		Liquid Charge	Energy Release by Spring ft.-lbs.	Crush of Striker Tip inches	Effect of Impact on: (naked eye and 100x)	
	Striker	Plate				Striker	Plate
33	Copper, ETP	Copper, ETP	None (in air)	55	9/32	Tip compressed and bent to side	Vee shaped dent, 1/16" deep.
14	"	"	ClF ₃	19	1/16	Tip compressed to rectangle.	Deep, sharp-edged, vee shaped dent.
20	"	"	ClF ₃	62	1/4	Tip compressed to irregular rectangle; free end bent sharply to one side; light yellow stain on sides of striker.	Deep, vee shaped dent; light stain over undented area.
34	"	"	ClO ₃ F	66	9/32	Tip compressed and bent to side	Vee shaped dent, 1/16" deep; slight staining.
7	Yellow Brass	Yellow Brass	None (in air)	18	3/32	Tip compressed to irregular rectangle.	Slight, vee shaped dent.
5	"	"	ClF ₃	19	3/32	Tip compressed to irregular rectangle.	Shallow, vee shaped dent.
19	"	"	ClF ₃	64	5/32	Tip compressed to irregular rectangle.	Shallow, vee shaped dent.
35	"	"	ClO ₃ F	66	3/16	Tip compressed to irregular rectangle.	Shallow, vee shaped dent.
9	Magnesium AZ31B	Magnesium AZ31B	None (in air)	17	3/32	Part of crushed tip broken off; free end bent slightly to one side.	Oval, vee shaped dent.
29	403 S.S.	"	None	62	1/32	Tip compressed slightly.	Pierced (1/8") by striker in vee shaped cut.

(Continued)

APPENDIX II

TABLE 15 (Cont.)

Run No.	Test Metals		Liquid Charge	Energy Release by Spring ft. -lbs.	Crush of Striker Tip inches	Effect of Impact on: (naked eye and 100x)	
	Striker	Plate				Striker	Plate
10	Magnesium AZ31B	Magnesium AZ31B	ClF ₃	15	1/8	As for Run #9 except at 100x a purple coloration seen at tip as sign of incipient oxidation or burning.	Dented at 100x in dent area, a purple coloration seen.
23	403 S.S.	"	ClF ₃	64	<1/16	Tip compressed slightly	Pierced (1/8") by striker in vee shaped cut; oval compressed area on under side near cut had a brownish-gray scale. XRD of this scale showed MgF ₂ and MgCl ₂ .
11	Magnesium AZ31B	"	ClO ₃ F	15	1/8	As for Run #10	As for Run #10
31	403 S.S.	"	ClO ₃ F	64	1/32	Tip compressed slightly	Similar to that of Run #23.
16	"A" Nickel	"A" Nickel	ClF ₃	62	5/32	Tip compressed to irregular rectangle.	Vee shaped dent.
37	"	"	ClO ₃ F	66	5/32	Tip compressed to irregular rectangle.	Vee shaped dent.
15	Monel	Monel	ClF ₃	62	3/16	Tip compressed to irregular rectangle; free end bent slightly.	Deep, vee shaped dent.
36	"	"	ClO ₃ F	66	3/16	Tip compressed to irregular rectangle.	Vee shaped dent.

(Continued)

APPENDIX II

TABLE 15 (Cont.)

Run No.	Test Metals		Liquid Charge	Energy Release by Spring ft. lbs.	Crush of Striker Tip inches	Effect of Impact on:	
	Striker	Plate				Striker	Plate
17	316 S.S.	316 S.S.	ClF ₃	62	5/32	As for #15	Vee shaped dent.
38	"	"	ClO ₃ F	63	5/32	Tip compressed to irregular rectangle.	Shallow vee shaped dent.
28	403 S.S.	403 S.S.	None- (in air)	59	1/8	Tip compressed to oval.	Shallow, oval dent.
18	"	"	ClF ₃	60	3/32	Tip compressed to oval.	Shallow dent.
24	"	"	ClO ₃ F	62	3/32	Tip compressed to oval; rust coloration in this area which appears as a stain at 100x	Slight dent, with staining in dent area as on striker.
51	"	"	ClO ₃ F	131	5/32	Tip compressed to oval; slight staining.	Shallow dent.
13	Low Carbon Steel 1010	Low Carbon Steel 1010	ClF ₃	21	3/32	Tip compressed to irregular rectangle; faint, bluish purple coloration in compressed area seen at 100x.	Shallow dent; faint bluish purple coloration in dented area seen at 100x.
21	"	"	ClF ₃	64	3/16	As for #13; and free end bent slightly to one side.	As for #13.
39	"	"	ClO ₃ F	66	3/16	Tip compressed to irregular rectangle.	Shallow dent.

(Continued)

APPENDIX II

TABLE 15 (Cont.)

Run No.	Test Metals		Liquid Charge	Energy Release by Spring ft. -lbs.	Crush of Striker Tip inches	Effect of Impact on:	
	Striker	Plate				Striker	Plate
6	Titanium C-120AV - T1	Titanium C-120AV - T1	None (in air)	20	1/16	Tip compressed to thin oval; faint bluish purple stain at one end of compressed area seen at 100x; no apparent fusion of metal.	Slight dent; no evidence of reaction or fusion.
27	"	"	None (in air)	64	3/16	Tip compressed and bent to one side; light rust colored stain in compressed area.	Two shallow dents, showing evident bounce of striker; metal (60mg.) from striker tip mashed on and bonded tightly to plate; a few light rust colored stains in dent; no evidence of burning.
4	"	"	ClO ₃ F	19	3/32	At 100x on striker tip: dark purple crevices and craters; spots of bright, fused metal as spheres; bluish purple stain over most of contact area as evidence of oxidation.	At 100x; spherical drops of bright fused metal in dent area; bluish purple stain over most of contact area as evidence of oxidation.

(Continued)

APPENDIX II

TABLE 15 (Cont.)

Run No.	Test Metals		Liquid Charge	Energy Release by Spring ft. -lbs.	Crush of Striker Tip inches	Effect of Impact on: (naked eye and 100x)	
	Striker	Plate				Striker	Plate
25	Titanium C-120AV-Ti	Titanium C-120AV-Ti	ClO ₃ F	34	3/32	Compressed area has bluish purple staining in a wide groove seen with blue stain around groove; evidence of spattering and fusion of metal more severe than in Run #4.	Slight dent with light brown stains in contact area; at 100x saw metal drops fused to surface; evidence of burning and fusion more severe than in Run #4.
26	"	"	ClO ₃ F	64	5/32	Tip burned off (100 mg. loss), leaving rectangular end; free end also bent, tip surface had bluish purple stains with burning along edges; at 100x saw burning as in #25; two pieces of black, porous sintered residue fallen from tip and fused to plate.	Wide shallow dent; dark purple and black stains with definite signs of burning and fusion; at 100x, spattering of metal in impact area as well as staining already noted; definitely more reaction than in Run #25.

(Continued)

APPENDIX II

TABLE 15 (Cont.)

Run No.	Test Metals		Liquid Charge	Energy Release by Spring ft.-lbs.	Crush of Striker Tip inches	Effect of Impact on:	
	Striker	Plate				Striker	Plate
40	Titanium C-120AV-Ti	Titanium C-120AV-Ti	ClO ₃ F	68	3/16	Tip burned off (155 mg. loss); similar to #26, but more severe burning.	Spattering of fused metal beyond impact area; porous black ash and ball of metal from striker tip fused to plate in impact area.
41	"	"	ClO ₃ F	71	3/16	Tip burned off (143 mg. loss), and striker bent to side; similar to #26.	Similar to #40, but less spattering of fused metal.
42	"	"	ClO ₃ F	102	11/32	Tip burned (109 mg. loss) and striker bent sharply to side; lesser reaction due to energy loss in mechanical bending.	Part of striker tip fused to surface; much spattering of fused metal.
47	"	"	ClO ₃ F	102	7/32	Tip burned (59 mg. loss) and striker bent sharply to side; see #42.	Similar to #42.
46	403 S.S.	"	ClO ₃ F	99	5/32	Tip compressed to oval; slight stain, but no burning.	Very shallow dent; slight stain; faint trace of burning only at 100x.

(Continued)

APPENDIX II

TABLE 15 (Cont.)

Run No.	Test Metals		Liquid Charge	Energy Release by Spring ft. -lbs.	Crush of Striker Tip inches	Effect of Impact on:	
	Striker	Plate				Striker	Plate
44	Titanium A-110AT-Ti	Titanium C-120AV-Ti	ClO ₃ F	99	3/16	Tip burned (324 mg. loss).	Part of striker tip fused to surface; much spattering of fused metal; some black ash and stains at many points.
48	"	"	ClO ₃ F	140	7/32	Tip burned (227 mg. loss) and striker bent to side.	A 7/16" circle of black, porous ash formed in dome-like shape at impact point; plate burned in this circle to 1/32" depth; no unburned striker tip metal remained on plate. Highest degree of sustained burning re-action noted to this run.
49	"	"	ClO ₃ F	136	3/16	Tip burned (212 mg. loss)	Part of striker tip fused to surface; less burning of plate than in Run #48; much spattering of fused metal.

(Continued)

APPENDIX II

TABLE 15(Cont.)

Run No.	Test Metals		Liquid Charge	Energy Release by Spring ft.-lbs.	Crush of Striker Tip inches	Effect of Impact on:	
	Striker	Plate				Striker	Plate
43	Titanium A-110AT-Ti	Titanium A-110AT-Ti	ClO ₃ F	92	7/16	Tip burned(232 mg. loss)	Part of striker tip fused to surface; much spattering of fused metal; similar to Run #44.
45	"	"	ClO ₃ F	134	7/32	Tip burned(340 mg. loss)	Similar to Runs #43 and #44, but more evidence of burning of fused metal from striker.
50	"	"	ClO ₃ F	134	3/16	Tip burned(291 mg. loss)	Similar to Run #45.

Note: Thickness of titanium C-120AV-Ti striker, 3/32 inch.
 Thickness of titanium A-110AT-Ti striker, 1/8 inch.
 This accounts for greater resistance to bending by the A-110AT-Ti strikers.

EXHIBIT 1

PREPARATION OF SPECIMENS FOR IMMERSION TESTING

Standard Cycle:

1. Stamp code identification numbers at one end.
2. Remove ink print marks and gross dirt by wiping with muslin cloth wet with acetone.
3. Complete solvent degreasing by dipping into boiling trichloroethylene liquid.
4. a. Wipe with lintless cotton cloth to remove remaining fingerprint or other marks, or dry dirt particles.
or b. Acid etch or emery abrasion where necessary to remove mill or other heavy scale. Follow by water rinse and drying. See Special Treatment paragraph below.
5. Vapor degreasing and drying over boiling trichloroethylene.
6. Store cleaned specimens in individual polyethylene envelopes in desiccator.
7. Weigh test pieces to 0.1 mg. immediately before immersion testing.

Special Treatments:

Alloy 2-1 (ETP Copper)

Etch 5-30 seconds in 1/1 (V/V) nitric acid at 25-30°C. to remove dark mill scale.

Alloy 2-5 (Aluminum Bronze, Ampco 8)

Heavy black mill scale abraded with Behr-Manning #952 fine emery cloth. Some scale remains in fine pit marks in surface caused by rolling operation. Etch in 30% HNO₃-20% H₂SO₄ solution at 70°C. for 10-60 seconds to remove remainder of embedded scale.

EXHIBIT 2

STRESSED TEST PIECE INFORMATION

Alloys Included in Stress Corrosion Tests:

1. Aluminum 1100
2. Copper, ETP
3. Yellow Brass
4. Magnesium, AZ31B
5. "A" Nickel
6. Monel
7. Stainless Steel 316
8. Stainless Steel 403

Sample Size:

1/2" x 4" x 1/8"

Sample Shape:

Sample strip stressed into U-bend using a bending jig of design supplied by Carpenter Steel Company. Stress maintained in plastic range during test liquid exposure by a Teflon insulated steel bolt through drilled holes in U-bend ends.

Inspection Methods after Test Liquid Exposure:

1. Visual
2. Dye penetrant inspection procedure using Dy-Check kit (Turco Products, Inc.).

EXHIBIT 3

PREPARATION AND CHARGING PROCEDURE TO
TEST TANKS FOR 25% ClO_3F -75% ClF_3 LIQUID MIXTURE

1. Scrub residual HF from ClF_3 by vaporizing through a tower of sodium fluoride pellets. Condense purified ClF_3 into a 10-pound hold cylinder (H.C.) chilled in a dry ice-trichloroethylene bath.
2. Weigh out 2432 g. ClF_3 from H.C. into a tared evacuated 10-pound charge cylinder (C.C.) chilled as in 1. Close C.C.
3. Keep C.C. chilled; disconnect ClF_3 H.C.; connect ClO_3F H.C. (100 pounds). Charge 927 g. ClO_3F into C.C. Close C.C.
4. Warm C.C. to room temperature; check weight and mix contents well by rolling and inverting.
5. Clamp C.C. in inverted position in charge rack with cylinder outlet elevated above top of test tank.

Note: It was hoped to make a gravity liquid transfer from the C.C. into the evacuated test tank with the entire system at about 30°C . However, the liquid transfer was slow because of vapor lock in the connecting line. Therefore, the test tank was chilled with a dry ice-trichloroethylene bath to enable the transfer to be made in 5 to 10 minutes.

6. Allow system (C.C., line and test tank) to reach temperature and pressure equilibrium at 25 - 30°C .
7. Close valves on test tank and C.C. Disconnect C.C. and check weight for expected amount of ClO_3F - ClF_3 vapor mixture of composition given in Figure 1.

EXHIBIT 4

PROCEDURE FOR HANDLING IMMERSION TEST PIECES
AFTER
EXPOSURE TO ClF_3 , ClO_3F AND MIXTURES OF THESE

1. Discharge test liquid into evacuated cylinder chilled in a dry ice-trichloroethylene bath. Heat test tank with no warmer than 30°C . water bath to correct evaporative chilling, and complete test liquid discharge.
2. Flush test tank with H.P. dry nitrogen, maintaining 30°C . water bath.
3. Evacuate test tank.
4. Repeat steps 2 and 3.
5. Fill tank to 5 psig with H.P. dry nitrogen.
6. Open tank. Remove lid with suspended test pieces in holders and place quickly into 135°C . oven flushed with 3-5 l./min. nitrogen. Hold in oven for 45 minutes.
7. Place hot dried test piece assembly in nitrogen flushed box to cool.
8. Remove cool test pieces from holders and place in individual polyethylene envelopes. Store in desiccator.
9. Weigh test pieces to 0.1 mg.
10. Observe surface appearance before and after light wiping with a lintless cotton cloth. Make note of any material removal to cloth as a measure of ease of removal of corrosion film.
11. Carry out standard erasure cleaning of test pieces with the soft end of a #210 Union Eberhard-Faber white rubber eraser to remove corrosion film more completely. Observe effect of erasure.
12. Reweigh eraser cleaned test pieces and then replace in polyethylene envelopes. Store for future study or use.

EXHIBIT 5

FORMULA FOR CALCULATION OF CORROSION RATES

Corrosion Rate:

$$\text{mils/yr.} = \frac{3.449 \times 10^6 W}{DAT}$$

where:

W = weight loss in grams

D = specific gravity of test specimen

A = area of test specimen in sq. cm.

T = duration of test in hours

1 mil = 0.001 inch

Formula modified by change in units of measurement from that given by Fontana.*

*Fontana, Mars G., Corrosion, Hollenback Press, Columbus, Ohio (1957).

EXHIBIT 6

OUTLINE OF CORROSION RATE CALCULATIONS

Notation Definitions:

W_{initial}	- Weight of cleaned test piece before exposure to test medium.
W_a	- Weight of test piece after exposure to test medium and drying.
W_c	- Weight of test piece after exposure, drying, light cloth wiping and rubber erasure of surface.
W_u	- Weight of duplicate, unexposed, cleaned test piece after rubber erasure of surface.
ΔW_d	- Net weight loss by rubber erasure due to corrosion.
ΔW_m	- Weight of metal equivalent to the observed increase in weight, ΔW_a , due to exposure and assumed to be fluoride or oxide.
ΔW_e	- Total corrosion weight loss including the correction for adherent fluoride or oxide film removed by standard erasure procedure.
ΔW_{mm}	- Total weight loss by standard erasure procedure, expressed as metal loss.

Procedure:

1. Determine ΔW_a as weight gain (+), or weight loss (-).

$$\Delta W_a = W_a - W_{\text{initial}}$$

2. Determine ΔW_c as a weight loss (-) of exposed test piece.

$$\Delta W_c = W_c - W_a$$

3. Determine ΔW_u as average weight loss (-) of two duplicate, unexposed test pieces.

$$\Delta W_u = W_u - W_{\text{initial}}$$

4. Determine ΔW_d as net weight loss (-) of exposed test piece; if ΔW_d is a weight gain (+), ignore.

$$\Delta W_d = \Delta W_c - \Delta W_u$$

5. Calculate an initial corrosion rate, C.R.', based on ΔW_a

A. If ΔW_a is (-), weight loss is assumed to be metal.

- (1) Determine C.R.' in mils/yr. by standard formula (Exhibit 5), based on ΔW_a .

EXHIBIT 6 (Cont.)

Procedure: (Cont.)

B. If ΔW_a is (+), weight gain is assumed to be fluoride after exposure to ClF_3 or mixtures of ClF_3 and ClO_3F , and oxide after exposure to pure ClO_3F

(1) Determine ΔW_m as weight of metal equivalent to ΔW_a fluoride or oxide.

$$\Delta W_m = \Delta W_a \times \text{wt. ratio Alloy/F or,}$$

$$\Delta W_m = \Delta W_a \times \text{wt. ratio Alloy/O}$$

(weight gain factor or ratios Alloy/F and Alloy/O are given in Table 8)

(2) Determine C.R.' in mils/yr. by standard formula (Exhibit 5), based on ΔW_m .

6. Calculate a second or cumulative corrosion rate, C.R.", which includes a correction for fluoride and oxide film removed by standard erasure procedure.

Case I, when ΔW_a is (-)
then: $\Delta W_e = \Delta W_d + \Delta W_a$

(1) Determine C.R." in mils/yr. by standard formula (Exhibit 5), based on ΔW_e .

Case II, when ΔW_a is (+) and $\Delta W_d > \Delta W_a$ numerically
then: $\Delta W_e = \Delta W_d$
(That is, the total corrosion weight loss is equal to the net weight loss by erasure.)
also: ΔW_e includes ΔW_a

(1) Divide ΔW_e (or W_d) into two parts:
1st: ΔW_a , which is converted into an equivalent ΔW_m metal loss as in 5B above.
2nd: the difference
 $\Delta W_{mm} = \Delta W_e + \Delta W_a$ is taken as the algebraic sum.
If $\Delta W_{mm} > \Delta W_m$, then ΔW_{mm} is taken as the total corrosion weight loss, as metal.
If $\Delta W_{mm} < \Delta W_m$, then ΔW_m is taken as the total corrosion weight loss, as metal.

EXHIBIT 6 (Cont.)

Procedure (Cont.)

- (2) Determine C.R." in mils/yr. by standard formula (Exhibit 5), based on ΔW_{mm} or ΔW_m as explained in (1) above. Note that when $\Delta W_{mm} < \Delta W_m$, then C.R.' = C.R."

Case III, when ΔW_a is (+)
and $\Delta W_d < \Delta W_a$, numerically
then; $\Delta W_e = \Delta W_a$
and C.R.' = C.R."

EXHIBIT 7

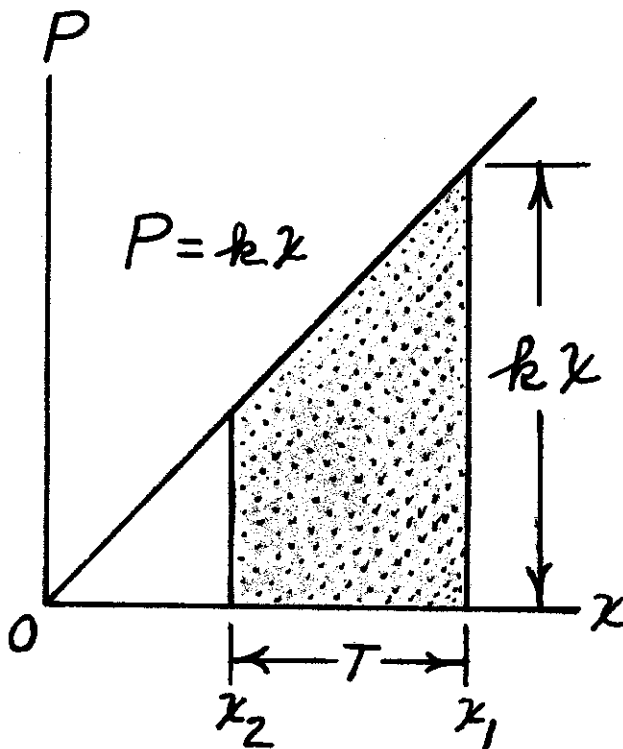
PROCEDURE FOR IMPACT TESTS

1. Weigh cleaned (Exhibit 1) striker and plate to 0.1 mg.
2. Place sample plate at bottom of tester. Place retaining ring on top of sample and lock in place with set screws.
3. Measure with steel rule to 1/32 inch the following:
 - a. Distance from top of Flexitallic gasket lying on flange to top of sample plate placed on bottom of tester.
 - b. Distance from under side of lid flange to end of striker sleeve, with spring expanded.
 - c. Same as (b), with spring compressed and locked.
 - d. Altitude of striker from base to tip.
 - e. Distance from tip of striker to lower end of striker sleeve, with striker mounted in sleeve.
4. Compress spring (see 3c) and lock striker sleeve in place.
5. Mount striker sample (see 3d) on striker sleeve.
6. Assemble lid and tank of tester; tighten bolts (5/8 inch chrome molybdenum alloy steel ASTM A193-B7) to 85-90 ft.-lbs.
7. Evacuate tank and charge with test liquid (either ClF₃ or ClO₃F).
8. Warm tank from dry ice charging temperature to 30°C. using electric heating mantle and heat lamp. Thermocouple, insulated from mantle, strapped to outside wall of tank one inch from bottom.
9. Release striker by remote control from outside of protective barricade.
10. Note pressure and temperature changes on impact.
11. Discharge liquid chemical from tank; flush with nitrogen and remove test pieces.
12. Inspect and weigh striker and plate, noting any changes.

EXHIBIT 8

ENERGY CALCULATIONS IN IMPACT TESTS

The calculation of energy release by the compressed spring on impact of striker against plate may be understood by referring to the following diagram and equation.



Ref.: Sears, Francis W. and Zemansky, Mark W., University Physics, 2nd Ed., Addison-Wesley Pub. Co., Inc., Reading, Mass. (1955)

P = force
 k = force constant of spring
 W = energy, ft.-lbs.

$$W = \int_{x_1}^{x_2} P dx = \int_{x_1}^{x_2} kx dx$$

$$W = 1/2 k(x_2^2 - x_1^2)$$

The spring is initially compressed the distance x_1 and then released the distance T with the striker tip hitting the plate (see Figure 7) when the spring has opened to the point x_2 . The energy release is given by the shaded area in the diagram, calculated as shown. The force constant, k , of the spring was given by the spring manufacturer as pounds required for 1/10 inch deflection. As shown in Table 14 this was converted to lbs./ft. to give W as ft.-lbs.

Energy release due to the gravity fall of the striker holder, striker and part of the spring was calculated to be about 0.06 ft.-lbs. for the small (#1) impact tester and about 0.15 ft.-lbs. for the large (#2) impact tester. The precision of measurement of spring compression and thus of impact energies was estimated to be about 5%, or 1/2 to 3 ft.-lbs. for impact levels of 10 to 65 ft.-lbs. Therefore, the energy contribution due to gravity fall was ignored.

EXHIBIT 9

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TRIFLUORIDE AND PERCHLORYL FLUORIDE

(Unclassified)

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