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REMOTE HANDLING - BLENDING OF ENERGETIC MATERIALS

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ABSTRACT

A special handling-blending room fitted with manipulators was built to provide maximum safety for operators working with energetic materials. Background experimentation and data upon which the design was based, and the design of the room will be disucssed and illustrated.

INTRODUCTION

In 1974 to meet a need for a less sensitive, but still energetic, explosives initiator other than the commonly used primary explosives, such as lead azide, Monsanto Research Corporation started the development and production of a series of high energy pyrotechnics. It soon became apparent that these high energy pyrotechnics were not simple mixtures, easily handled, or subject to deflagration only. There was more to it. The safety literature and technical data on pyrotechnics did not deal fully with the materials under study. In-house work on small quantities, less than a gram, indicated that these metal/oxidizer blends were truly high energy mixtures.

Important production safety questions quickly surfaced. How large a batch could be blended safely? Was there a "critical mass" for detonation? How important was container shape and size? If these pyrotechnics detonated, what would be their TNT equivalency? On deflagration, what would be the size of the "fireball"?

Since Mound Facility does not have adequate space to have a range for testing up to 500-g charges, we contracted with the Engineering and Science Services Laboratory, NSTL Station, Mississippi (controlling office is U. S. Army ARRADCOM), to determine the TNT equivalency of $Ti/KClO_4$ mixtures (ref. 1), and to determine the adequacy

*Mound Facility is operated by Monsanto Research Corporation for the U. S. Department of Energy under Contract No. DE-AC04-76-DP00053.

of a special blending room we had planned, in which we hoped to blend up to 500-g quantities.

The objective of the test series was to determine the output energy of the titanium powder and potassium perchlorate mixture in a mechanical blender configuration representative of that used at Mound Facility. This was accomplished by measuring: (1) the free field air blast output equivalency as compared to an equal weight of TNT at the same scaled distances; (2) fireball diameter and duration; and (3) static pressure in a closed chamber.

EXPERIMENTAL METHODS

Materials

The composition tested consisted of one-third by weight of 2-micron particle size dry titanium powder and two-thirds by weight laboratory grade $KClO_4$. The number of tests and the quantities of ingredients for each test conducted are tabulated in Table 1. Five of the nine tests were conducted in a simulated blender configuration to determine free air equivalency, and the remaining four tests were conducted in closed chambers to measure static pressure.

TABLE 1

Pyrotechnic composition by ingredient weight for the various test objectives and configurations

Test Number	Ti Powder g(lb)	$KClO_4$ g(lb)	Total Weight g(lb)	Test objective
1	165 (0.364)	335 (0.739)	500 (1.102)	Explosive equivalency blending configuration
2	165 (0.364)	335 (0.739)	500 (1.102)	Explosive equivalency blending configuration
3	82.5 (0.182)	167.5 (0.369)	250 (0.551)	Explosive equivalency blending configuration
4	41.25 (0.091)	83.75 (0.185)	125 (0.276)	Explosive equivalency blending configuration
5	165 (0.364)	335 (0.739)	500 (1.102)	Explosive equivalency blending configuration
6	9.3 (0.021)	18.7 (0.041)	28 (0.062)	Static pressure 1.06 m^3 closed chamber
7	9.3 (0.021)	18.7 (0.041)	28 (0.062)	Static pressure 1.06 m^3 closed chamber
8	9.3 (0.021)	18.7 (0.041)	28 (0.062)	Static pressure 1.06 m^3 closed chamber
9	150 (0.331)	300 (0.661)	450 (0.992)	Static pressure 35 m^3 closed chamber

Test procedures for the blender test series

The test apparatus for the free field equivalency tests is shown in Fig. 1. This apparatus was designed to simulate the Mound Facility ball mill blender used for blending the two ingredients. Two light-weight metal containers were used: one was 2.5 liters (0.66 gal), and the other, 3.79 liters (1 gal). Relationship of test charge to pressure sensors is shown in Fig. 2.

The metal container was charged by weighing out the appropriate quantities of each ingredient and placing them in separate piles as shown in Fig. 1. With this procedure, the two ingredients were not initially in contact with one another. The lid was placed on the container and sealed with duct tape. An Atlas electric match head igniter was connected to the firing circuit via the slip ring assembly. The blender was then started remotely from the test control center and allowed to blend at 30 rpm for 30 min. Upon completion of the blending cycle the ball mill was stopped so that the match head igniter was positioned on the bottom of the container, and, consequently, submerged in the pyrotechnic material. The match head was then ignited.

Static pressure was measured in closed chambers of two different volumes. Fig. 3 shows the configuration of the smaller vessel, 1.07-m^3 (37.7-ft^3), used for static pressure measurement. This

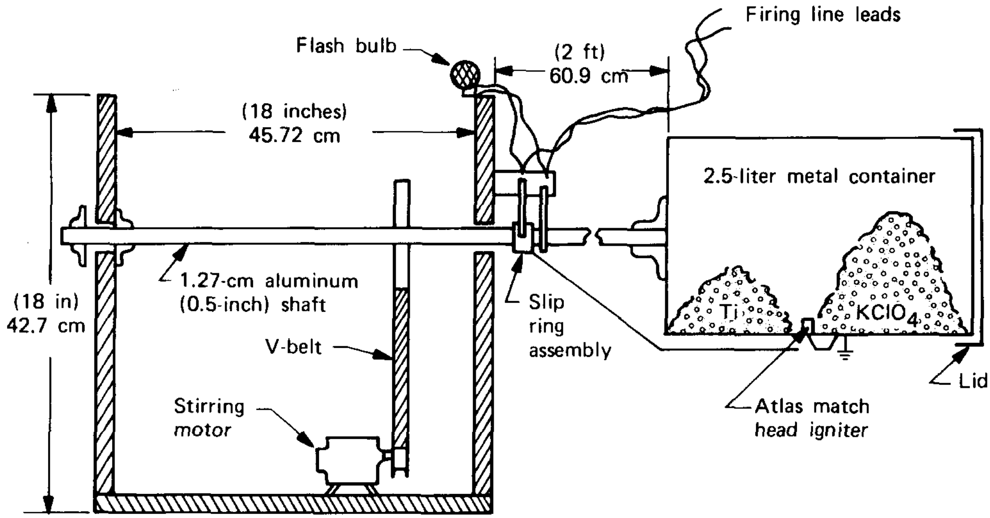

Fig. 1. Remote blender apparatus and firing circuit.

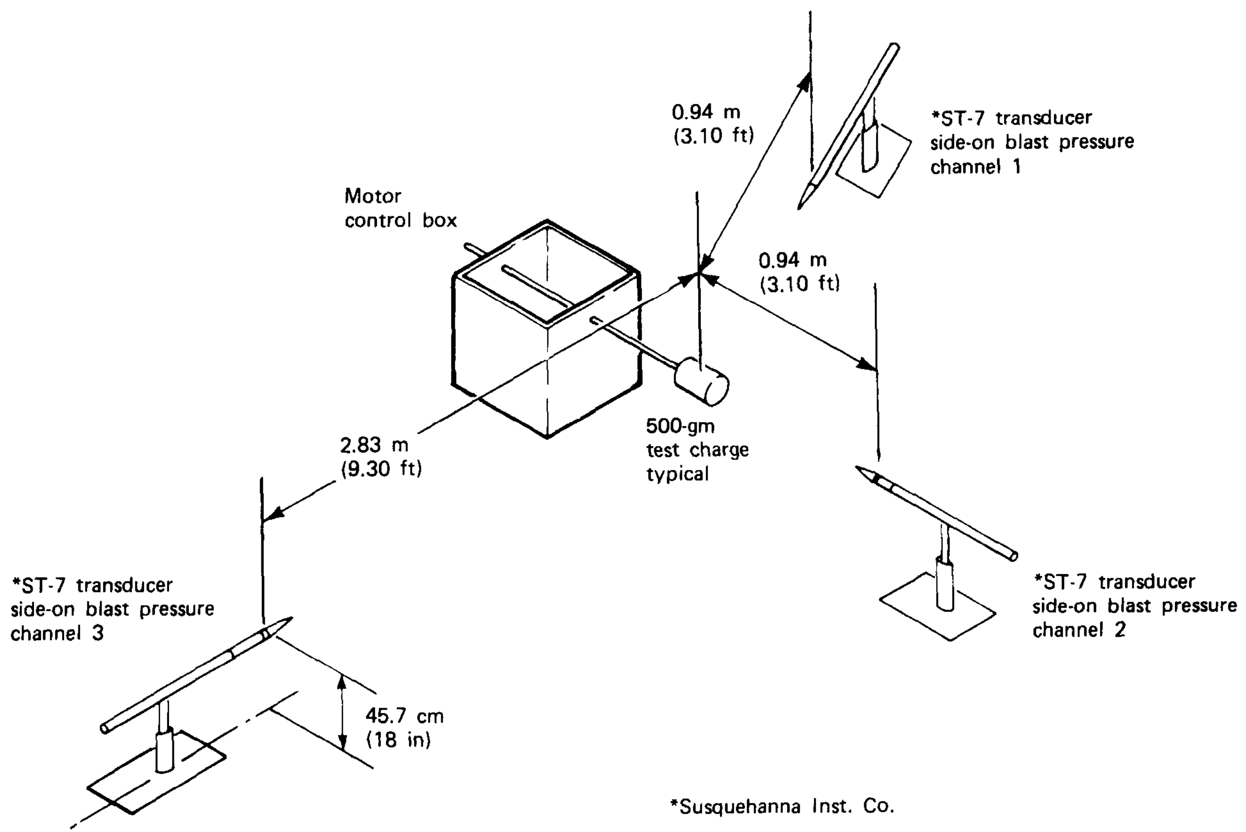

Fig. 2. Schematic of blast pressure gage deployment.

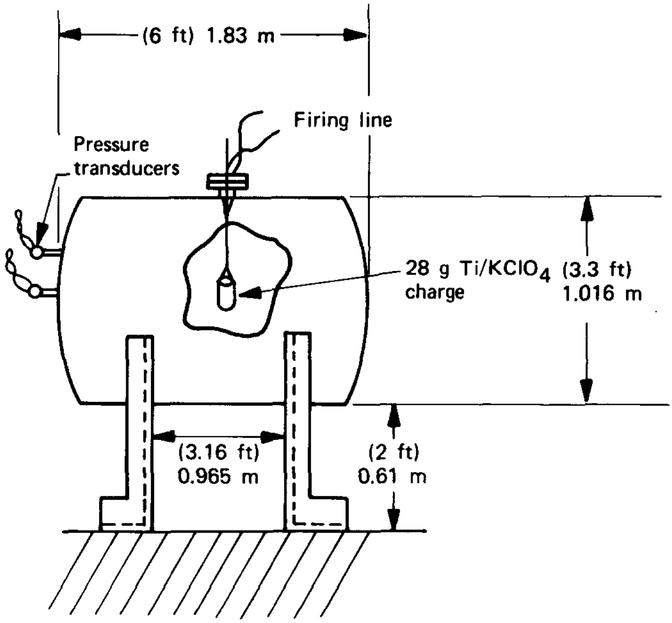

Fig. 3. Closed pressure vessel used for static pressure measurements Volume is 1.07 m^3 (37.7 ft^3).

chamber is rated at 517 kPa (75 psi). The charge weight, 28 g, was determined by scaling the volume of the proposed Mound Facility mixing cubicle, which has a calculated volume of 19.12 m^3 (675 ft^3), to that of the test chamber. The pyrotechnic composition was placed in a test tube and positioned in the approximate center of the vessel The composition was then ignited by an Atlas match head igniter. Two static pressure transducers and a bourdon-type pressure gage were installed in the chamber to measure static pressure.

Fig. 4 shows the larger test chamber used for static pressure measurement. A 450-g charge of the pyrotechnic composition was placed in a 10-cm (4-in.) diameter by 7.6 cm (3-in.) high Velostat plastic container. After being positioned inside the 35.4-m^3 (1250-ft^3) test chamber, the charge was ignited in the same manner as the charge in the small closed chamber. Six strain-gage-type pressure transducers were installed to measure the resultant static pressure.

RESULTS AND DISCUSSION

Data analysis

Peak blast pressures were recorded in digital form on the Biomation recorders. Average values of peak pressure were calculated for each

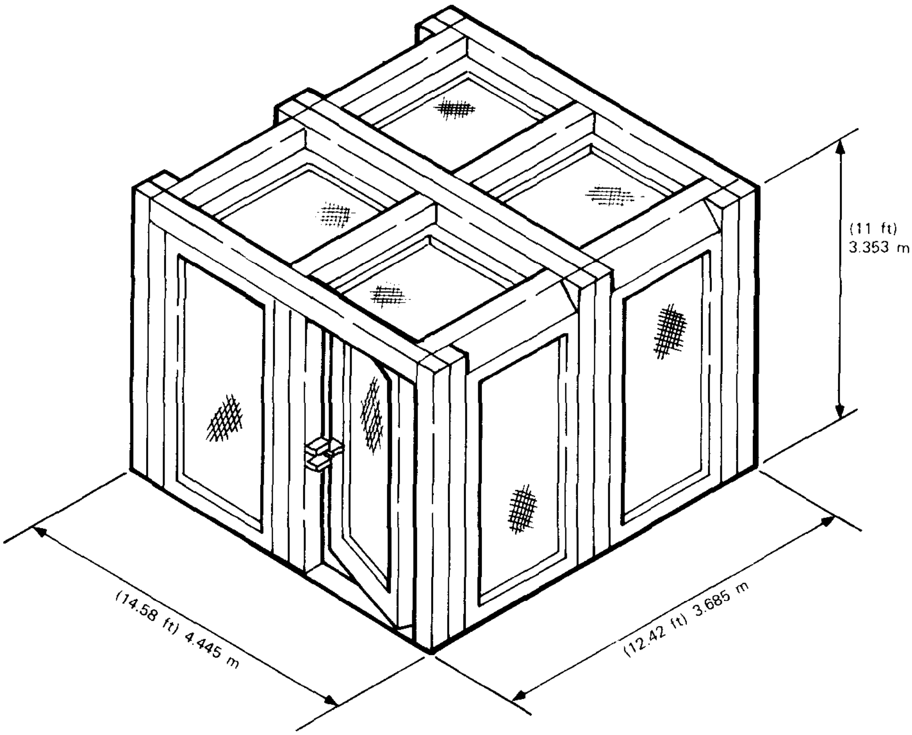

Fig. 4. Closed test chamber used for static pressure measurements. Volume is 34.5 m^3 (1250 ft^3).

weight and scaled distance. The average peak pressures were compared directly with a standard reference curve (ref. 2) to derive an explosive equivalency (Ep) as a percentage by weight based on equivalent side-on blast pressure at equal distance from the charge

$$Ep = 100\left[\frac{W_{exp}}{W_{Ti/KClO_4}}\right]_{\substack{\text{Constant}\\\text{Pressure \&}\\\text{Distance}}} = 100\left[\frac{Z^3_{Ti/KClO_4}}{Z^3_{exp}}\right]_{\substack{\text{Constant}\\\text{Pressure}}} \quad (1)$$

where W is the weight of the explosive, Z is the scaled distance, P is the peak blast pressure, and the subscripts refer to the explosive material. TNT equivalency is obtained by

$$E_{TNT} = Ep \times K \qquad (2)$$

where E_{TNT} is the TNT equivalent weight of the test explosive, Ep is the explosive equivalency derived in equation 1, and K is the constant between TNT and free air spherical pentolite used as the standard reference. In this case K is equal to 1.16 (ref 3).

The $Ti/KClO_4$ mixture was initiated by a single match head igniter which had little or no explosive output. Therefore, the total energy released in terms of peak blast overpressure was due to the pyrotechnic composition.

Peak static pressure calculations resulting from detonation of an explosive in a closed vessel have been developed by William S. Filler (ref. 4). In his investigation, good correlation of test results with calculated results was obtained using the equation:

$$P = \frac{H(\gamma - 1)}{V} \qquad (3)$$

where P is the pressure rise, H is the heat added to the gas, γ is the ratio of specific heats (C_P/C_V), and V is the volume of the container. For the pressures being considered and including conversion factors, this equation can be expressed as:

$$P = \frac{3844\ Wh}{V} \qquad (4)$$

where P is the pressure rise in pounds per square inch, W is the weight of the explosive in pounds, h is the heat of combustion in kcal/gram, and V is the volume of the container in cubic feet. TNT equivalency based upon static pressures resulting from a detonation in a closed vessel is a direct ratio of pressure observed to that calculated for the same weight of TNT in the same volume.

Test results

Average pressure and TNT equivalency results, with standard deviation, are summarized by test configurations and charge weight in Table 2. Fireball duration and diameter as measured from the high-speed motion pictures are summarized in Table 3. Static pressure values are summarized in Table 4.

TABLE 2

Summary of blast overpressure measurements of 1:2 $Ti/KClO_4$ thermally ignited in blender configuration

Charge Weight g(lb)	R m(ft)	Scaled Distance, Z $m/kg^{1/3}$ $(ft/lb^{1/3})$	Free Air Pentolite Pressure kPa(psi)	Peak Pressure kPa(psi)	TNT Equivalency %
500 (1.10)	0.91 (3.0)	1.15 (2.91)	781 (113.3)	466 ± 68 (67.2 ± 9.8)	53 ± 12
	2.74 (9.0)	3.45 (8.72)	63.6 (9.23)	55.8 ± 4.14 (8.1 ± 0.6)	75 ± 20
250 (0.55)	0.68 (2.23)	1.08 (2.72)	781 (113.3)	533.4 (80.28)	68
	2.03 (6.69)	3.24 (8.16)	63.6 (9.23)	49.22 (7.14)	71
125 (0.27)	0.54 1.77	1.08 (2.72)	781 (113.3)		No detonation
	1.62 (5.31)	3.24 (8.22)	63.6 (9.23)		

TABLE 3

Fireball duration and diameter

Charge Weight kg(lb)	Fireball diameter Predicted m(ft)	Maximum m(ft)	Fireball duration Predicted ms	Measured ms
0.500 (1.10)	3.09 (10.14)	3.07 (10.1)	240	260
0.250 (0.55)	2.48 (8.13)	3.2 (10.5)	190	200
0.125 (0.28)	1.98 (6.5)	4.57* (15)	154	625*

*Sample did not detonate.

TABLE 4

Summary of static pressure measurements in closed vessels

Sample Weight kg(lb)	Closed Vessel Volume $m^3(ft^3)$	Pressure Predicted* kPa(psi)	Measured kPa(psi)
0.450 (0.99)	35.4 (1250)	11.02 (1.60)	20.41 (2.96)
0.028 (0.06)	1.07 (37.7)	22.74 3.30	14.5 (2.1)

*Predicted pressure was based upon a heat of combustion of 1900 cal/g, derived from experiments performed by Mound.

The $Ti/KClO_4$ mixture exhibited characteristics of a detonation when thermally ignited in a light, metal container. For the 500-g (1.10 lb) charge, the TNT equivalent value was 53% at a scaled distance of 1.07 $m/kg^{1/3}$ (2.22 $ft/lb^{1/3}$) and approximately 75% equivalency at a scaled distance of 3.24 $m/kg^{1/3}$ (8.16 $ft/lb^{1/3}$). Equivalent values for the 250-g (0.55-lb) charge weight were 68% equivalency at the 1.07-$m/kg^{1/3}$. Because of the limited number of tests, it cannot be determined whether the apparent difference at the smaller distance is significant. The 125-g (0.28-lb) quantity failed to detonate. This was probably due in part to the volume of the container and the resultant depth of material.

Fireball characteristics were obtained from high-speed motion pictures taken during each test. The fireball diameter and duration were compared to predictions from equations given by High (ref. 5):

$$D = 3.86\ W^{0.320};\ T_O = 0.299\ W^{0.320} \quad (5)$$

where D is the fireball diameter in meters, W is the weight of the material in kilograms, and T_O is the duration in seconds.

The data from the 250-g (0.55 lb) and 500-g (1.10-lb) tests are in reasonably good agreement with the prediction. The 125-g (0.28-lb) test data are significantly different from predictions. The fireball was observed to expand from the open top of the container after the lid was ejected. This factor plus the longer burning time account for the larger diameter fireball for the smaller charge weight. The absence of blast overpressure, the larger fireball diameter, and the increased fireball duration all support the position that the 125-g (0.28-lb) charge resulted in a deflagration rather than a detonation.

Static pressure measurements

The average static pressure measurement for the 28-g (0.06-lb) sample in the 1.07-m^3 (37.7-ft^3) closed vessel was 14.5 kPa (2.1 psi). This represents a static pressure TNT equivalency of about 10%. The average pressure measurement for the 450-g charge in the 35.4-m^3 (1250-ft^3) closed vessel was 20.41 kPa (2.96 psi), which gives a TNT equivalency value of 27%. This variation is probably due in part to the fact that the smaller charge did not detonate.

CONCLUSIONS FROM TEST DATA

A $Ti/KClO_4$ mixture exhibited characteristics of a detonation when thermally ignited in 500-g (1.10-lb) and 250-g (0.55-lb) quantities in a simulated ball mill blender. The peak blast overpressure TNT equivalency varies from 53% at a scaled distance of 1.07 $m/kg^{1/3}$ (2.72 $ft/lb^{1/3}$) to 75% at a scaled distance of 3.24 $m/kg^{1/3}$ (8.16 $ft/lb^{1/3}$) when compared to TNT at the same charge weight and scaled distance. A 125-g sample of $Ti/KClO_4$ did not detonate when tested in the same blender configuration.

The static pressure measurements obtained were 14.5 kPa (2.1 psi) for 25 g of sample in a 1.07-m^3 (37.7-ft^3) closed vessel and 20.41 kPa (2.96 psi) for 450 g in a 35.4-m^3 (1250-ft^3) closed vessel, corresponding to a TNT equivalency of 10-27%.

The structural analysis of the proposed pyrotechnic blending facility was subcontracted to the Department of Ballistics and Explosive Sciences of the Southwest Research Institute of San Antonio, Texas (ref. 6). The proposed design is shown in Figure 5.

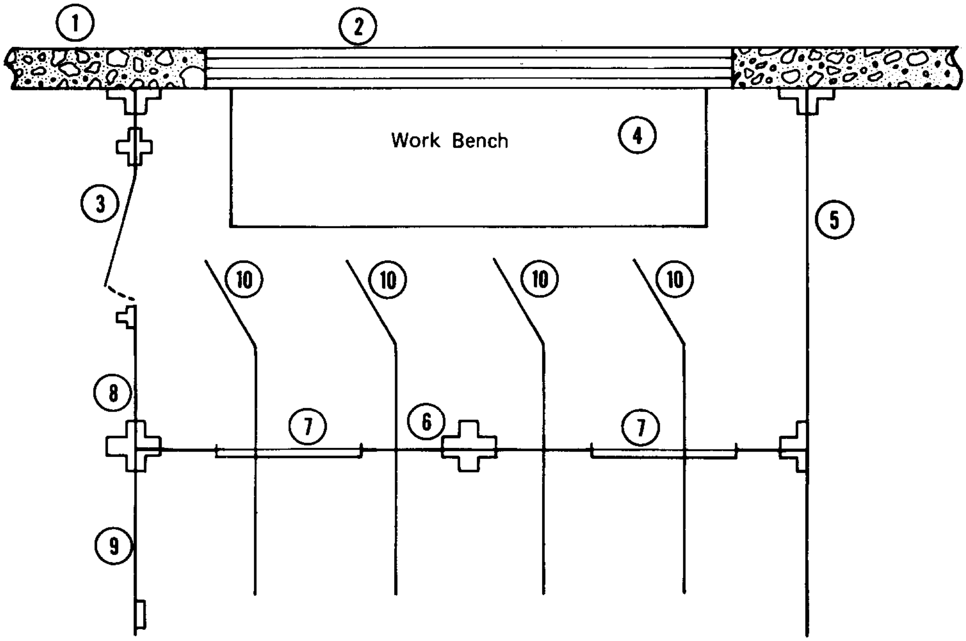

Fig. 5. Blending facility plan, showing masonry, exterior wall (1); blow-out panel (2); door (3); work bench (4); existing west wall (5); north wall (6); windows (7); east wall (8); east wall extension (9); and manipulators (10).

Southwest Research Institute based their calculations on a charge weight of 500 g of $Ti/KClO_4$. For blast wave loading effects, a TNT equivalency of 90% and a reflective factor of 1.5 for the charge being on the workbench were used.

$$\frac{500 \text{ g}}{453.6 \text{ g/lb}} (.90)(1.5) = 1.49 \text{ lb TNT} \qquad (5)$$

The value of 90% equivalency was based on the blast pressure measurements in the free field equivalency tests. We consider 90% quite conservative; 75% equivalency is nearer the experimental findings. For calculations of quasi-static pressure rise, an equivalency value of 30% was used, based on the data gained from the closed vessel test work using 450-g charges.

Using these values and the yield stress, elastic modulus, and weight density of the 0.5-in. thick A36 steel plates and angles, a structural analysis was made in detail. Recommended design modifications are listed below.

1. Increase the strength of the center beam column on the manipulator wall.
2. Add reinforcement at the 9-ft level.
3. Add a reinforcing ring around circular penetration for manipulators.
4. Add horizontal beams (3x3x¼ in. angles inside and out) above and below windows.
5. Replace laminated glass with high flex modulus polycarbonate.
6. Increase size of angles to 3x3x½ in.
7. Secure the steel walls to the floor using 6-in. long shields set in high-strength grout.
8. Secure the main column with 5/8-in. bolts; secure the other walls with 3/8-in. bolts on 15-in. centers..
9. Weld the full length and full fillet of all angles used for strengthening and/or connecting steel walls.

All these design modifications were incorporated along with a 0.5-in. thick steel ceiling. The latter not only helps to confine the pressure wave, and/or fireball, but also stiffens the side wall and protects the remainder of the building's ceilings from overpressure. The north wall (manipulator wall) was fitted with four Model G Master-Slave Manipulators made by Central Research Laboratories Inc. of Red Wing, Minnesota.

A fragment analysis was made on the assumption that a detonation of the pyrotechnic would occur in the hopper of the aluminum aliquot vessel. It was also assumed that the detonation would equal 1 lb of TNT, and that all available material would detonate, the worst case. The 0.5-in. thick steel walls will not stop a primary fragment, 1/8 x 1 x 1 in. aluminum, striking it at right angles.

The fragment danger can be eliminated if we avoid detonation. The work of ARRADCOM indicates that a 250-g mass of $Ti/KClO_4$ will detonate, but a 125-g mass will not, at least when in the physical configuration of the blender. (It should be noted that as little as 5 g of $Ti/KClO_4$ will detonate if confined in a minimum surface configuration, i.e., a sphere or a cube.) We have chosen at Mound, therefore, to limit the amount in the blender to about 125 g. The result is a blending facility which provides excellent protection to personnel from the fireball if ignition occurs, and good protection should a detonation occur.

These pyrotechnic mixtures are, of course, static sensitive. The blending room, therefore, is equipped with a conductive floor, and all benches, walls, and equipment are connected to the building static ground. The polycarbonate windows are covered on both sides with a transparent electro-conductive polyester film coated with gold. Light transmission is reduced to about 80% of clear glass. Conductivity across the surface is excellent.

The door is 26x66 in., stainless steel, and held closed by eight lugs rotated into position by a central lever. The door, tested at 10 psi by the manufacturer, is probably stronger than the walls. The door design is similar to that used by the Navy to close off bulkheads against fire and/or water.

Ventilation is provided by an armored, baffled intake at floor level and a filtered exhaust to the outside at ceiling level. The two-speed exhaust fan is switched into high speed via a sensitive heat, smoke, and flame detector located above the workbench.

SUMMARY

Data on detonation and deflagration characteristics of $Ti/KClO_4$, taken as representative of the metal/oxidizer pyrotechnics, were determined both in the open and in closed vessels. This information was used to design and build a steel powder blending room which will contain the detonation, and/or fireball, from the accidental ignition of up to 500 g of $Ti/KClO_4$ pyrotechnic. By use of manipulators in one wall, operators are able to blend, sieve and aliquot

the powders from outside the room. Operator safety has been greatly enhanced, and larger batches are permitted over the earlier hand operations.

ACKNOWLEDGEMENTS

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