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EVALUATION OF TEST METHODS FOR PYROTECHNIC HAZARD CLASSIFICATION

by

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March 1975

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Storage	Initiation sensitivity	Instrumentation

20. ABSTRACT (Continue on reverse side if necessary and identify by block number)

The hazard classification procedures of TB 700-2 are improperly applied to pyrotechnics. Forty-six test methods were proposed as replacements for the currently applied methods. The proposed test methods were subjected to engineering review and analysis including the testing of two materials by six methods. Fifteen test methods were recommended for inclusion in a pyrotechnics supplement to TB 700-2.

PREFACE

The work described in this report was authorized under US Army MIPR B4030 and TWR EA-4D01. It was performed at the NASA National Space Technology Laboratories (NSTL) for the Edgewood Arsenal Resident Laboratory (EARL) and NASA-NSTL by the General Electric Company under Contract No. NAS8-27750. This work was initiated on 24 September 1973 and completed in August 1974.

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Summary

This study covers the engineering reviews and analyses of 46 testing methods proposed for determining the hazard classification of pyrotechnic bulk materials and munition end items during transportation and storage.

Six test methods were applied to Green Smoke IV and Violet Smoke IV to demonstrate the validity of the tests.

The 15 most definitive bulk and end item test procedures are recommended for inclusion in a supplement to TB 700-2 for pyrotechnics. The recommended test procedures are intended to replace the explosives related tests that are now being improperly applied to pyrotechnics in TB 700-2.

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EVALUATION OF TEST METHODS FOR PYROTECHNIC HAZARD CLASSIFICATION

1.0 INTRODUCTION

1.1 Objective. The objective of this study was to provide engineering evaluations and analyses of test methods to be utilized for the classification of pyrotechnic bulk materials and munition end items. This study is intended to contribute toward the preparation of pyrotechnics hazard classification procedures that will be integrated into documentation for use by the Department of Defense, the Department of Transportation, and the North Atlantic Treaty Organization.

1.2 Authority. The work described in this report was authorized by National Space Technology Laboratories Technical Work Request (TWR) EA-4D01, dated 24 September 1973.

1.3 Background. The Explosives Hazard Classification Procedures, US Army Technical Bulletin 700-2, Change 1, 1968, sets forth testing procedures for determining the reactions of explosives, solid propellants, pyrotechnics, and end items to initiating influences such as heat, mechanical impact, hydrodynamic shock, and open flame.

The Bulletin provides for hazard classification of bulk materials on the strength of the above testing as shown in figure 1. The classification thus obtained applies only to transportation and storage and does not apply to the various stages of manufacturing and assembly.

The classification procedures that now appear in TB 700-2 consist of test methods that produce only "go" or "no go" results. The tests and their interpretation were devised specifically for mass detonating materials. They do not adequately provide for the true hazard classification of pyrotechnics (see paragraph 3.3). TB 700-2 is nevertheless applied to the classification of pyrotechnics.

This program was conducted to assemble, evaluate, and recommended hazard classification test procedures intended specifically for pyrotechnics to be included in a revised edition of TB 700-2.

2.0 TECHNICAL APPROACH

2.1 Test Review and Evaluation. Each candidate test method was:

- Reviewed and evaluated to determine its applicability to pyrotechnic hazards classification.
- Classified according to its nature and the physical parameters involved.
- Ranked on the bases of relatability, quantification, scalability, and cost.

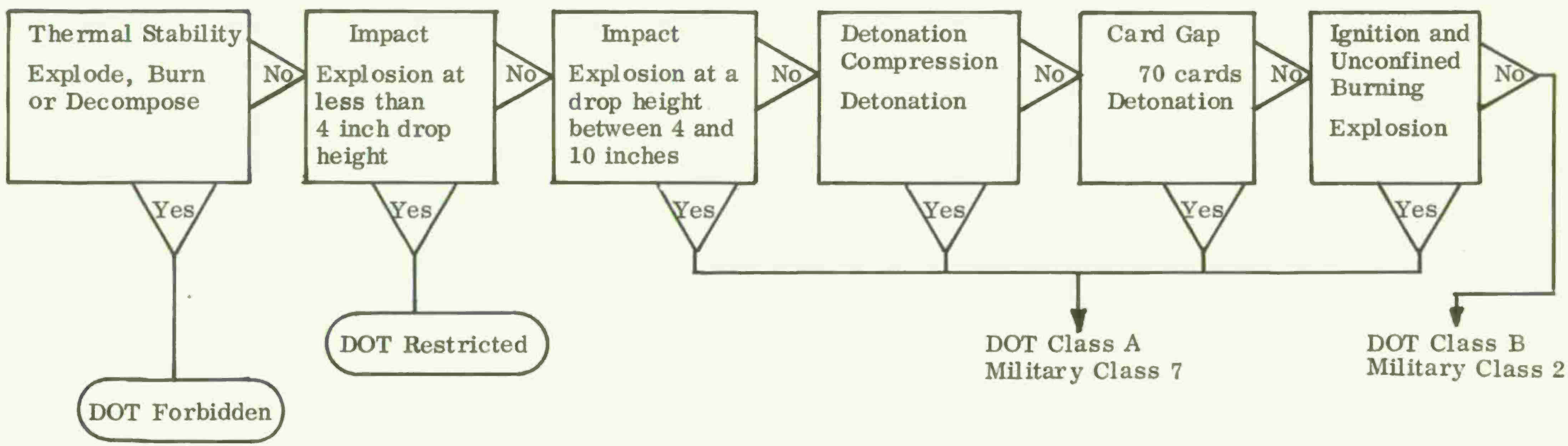

Figure 1. Classification of Bulk Pyrotechnics in Accordance with Paragraph 3-13 of TB700-2

Two materials, Green Smoke Mix IV and Violet Smoke Mix IV were selected as reference materials with which to demonstrate the suitability of the candidate test methods. They were selected because of the large amount of available test data.

2.1.1 Candidate Test Method Sources. The candidate test methods required to be reviewed and evaluated were obtained from the following:

- Test Methods for Pyrotechnic Materials Hazards Evaluation, A. Levine and D. Kone (appendix B)

- TB 700-2, Explosive Hazard Classification Procedures (including Change, 1, 1968)

- PEMA 4932, Project 5744099, Exhibit P-16, Paragraph 1a.

Additional candidate test methods were selected from the hazards evaluation experience at the National Space Technology Laboratories.

The candidate test methods are listed in table 1 on pages 11 and 12.

2.1.2 Ranking Criteria and Methods. For expediency, the relevant factors and characteristics were extracted from each test method for independent evaluation.

Each parameter was analyzed to determine the extent to which the resulting data could contribute to the proper hazard classification of a pyrotechnic bulk material or munition end item. To that end, evaluations were made on the basis of:

- Potential contribution of the parameter to hazardous situations.

- The ability of the test to evaluate the parameter.

- Whether an alternative test method is available.

- Cost.

Consequently, numerical values were assigned to the ranking criteria to facilitate the comparison and recommendation of specific test methods.

2.1.2.1 Relatability. Relatability refers herein to the extent to which the test method simulates a relevant parameter or initiation mechanism found in the transportation or storage environment. The numerical values assigned to relatability were:

4 Found and expected in one or both of the transportation or storage environment.

3 Possibly found in one or both environments; the probability of occurrence is not known.

2 Possible but less probable in either environment.

1 Occurrence improbable in either environment.

0 Not considered to be relevant to hazard evaluation.

2.1.2.2 Quantitative. Quantitative is the term used to reflect the ability of the test method to quantitatively measure the particular parameter. The numerical values assigned were:

2 Precise, quantitative and objective measurement of the parameter.

2 Only available test method for the parameter.

1 Subjective quantitative measurement of the parameter.

1 Qualitative but objective measurement.

0 Measurement that is both qualitative and subjective.

2.1.2.3 Scalability. Scalability describes the confidence with which results from the test can be extrapolated and applied to full-scale situations. The numerical values assigned were:

3 Full-scale test.

3 Amply demonstrated scalability.

2 Scalability not thoroughly demonstrated but believed to be valid.

2 Only test method available.

1 Scalability considered to be poor.

0 Scalability demonstrated to be poor.

2.1.2.4 Cost. Cost becomes a relatively minor item in the context of hazard classification. The cost of classification testing can be regarded as part of a materials development cost, and as such, it makes an insignificant contribution to the unit cost for production quantities. Furthermore, the cost of hazard classification testing is small compared to the potential consequences of a single incident where proper classification could have resulted in reduced casualty losses. The numerical values assigned to cost were:

1 Relative cost less than $400 per bulk material or end item.

0 Relative cost greater than $401 per bulk material or end item.

2.1.2.5 Application of Ranking Values. The ranking values, except cost, determined as described above are applied additively for each test method. Cost was considered only when other factors were equal. A "perfect" test method would have a ranking value of 10.

2.2 Tests Performed. Tests were conducted utilizing those candidate methods for which sufficient references were not available.

2.2.1 Differential Thermal Analysis. Differential thermal analysis testing was conducted in accordance with the procedure outlined in appendix B, method 112.

2.2.2 Parr Bomb Calorimeter. Parr Bomb calorimeter testing was conducted in accordance with the procedure outlined in appendix B, method 117.

2.2.3 Hygroscopicity. The procedure outlined in appendix B, method 303, was used to conduct hygroscopicity testing.

2.2.4 Moisture (Desiccation Method). Moisture testing by the desiccation method was conducted in accordance with the procedure outlined in appendix B, method 304.

2.2.5 Moisture and Volatiles (Vacuum Oven Method). Moisture and volatiles testing by the vacuum oven method was conducted in accordance with the procedure outlined in appendix B, method 305.

2.2.6 Isothermal Analysis (Multipount DTA). This is a variation of standard differential thermal analysis, the difference being in the controlled rate of heat applied externally. Isothermal analysis is supplemental to the standard DTA and can result in a more definitive evaluation of the potential thermal hazards of materials exhibiting rate-controlled reactions due to prolonged exposures at near-ignition temperatures.

A standard DTA is first performed. If no exotherm is observed below 500°C, the material is considered thermally safe. From the standard DTA results, a temperature is selected for the isothermal analysis. A 25 milligram sample is weighed into the sample tank, a thermocouple is placed into the material and set aside until the temperature block has stabilized. The sample tank is then introduced into the block, and the time is noted on the recorder. Within four to five minutes, the temperature of the sample will stabilize. The sample is observed for 20 to 30 minutes for endotherms and exotherms. A typical diagram of an isothermic analysis system is shown in figure 2.

3.0 RESULTS AND DISCUSSION

3.1 Tests Performed. The pyrotechnic bulk materials tested were:

- Green Smoke Mix IV, Drawing Number B 143-2-1.
- Violet Smoke Mix IV, Drawing Number B 143-5-1.

3.1.1 Differential Thermal Analysis. The average of 10 test runs on each sample produced the following results:

- Green IV
    - Exhibited exotherms at 166.49°C and 221.68°C, the decomposition temperature.

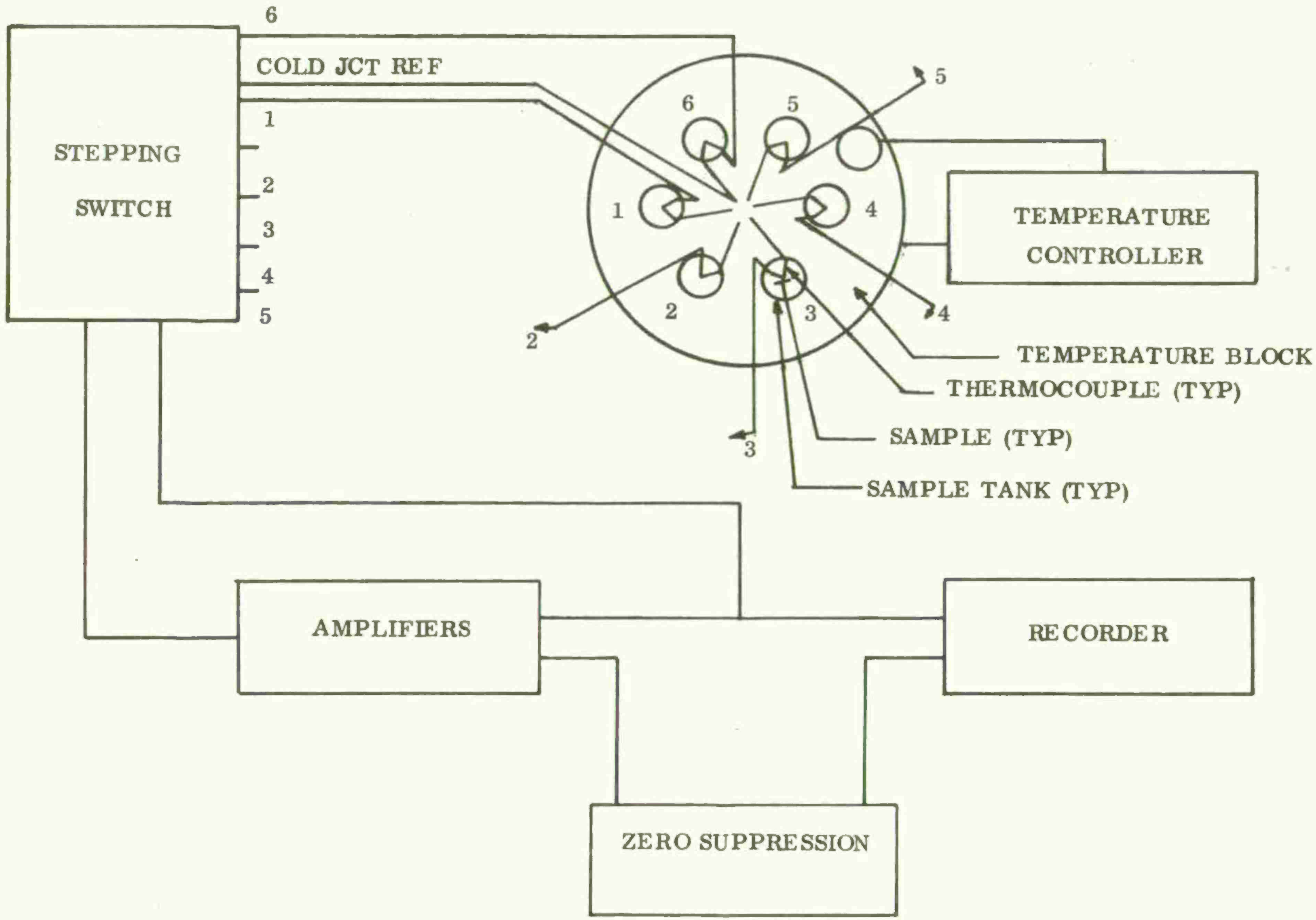

Figure 2. Block Diagram of the Isothermal Analysis Apparatus

- Exhibited endotherms at 68.78°C (transition of crystalline state), 117.75°C (melting of sulfur), and 178.64°C (sulfur - potassium chlorate reaction).

- Violet IV

  - Exhibited exotherms at 117.59°C (small peak), 175.45°C, and 239.88°C, the decomposition temperature.

  - Exhibited endotherms at 70.22°C (transition of crystalline state), 119.34°C (melting of sulfur), and 200.80°C (sulfur - potassium chlorate reaction).

The differential thermal analysis is a valid test that detects the chemical and physical changes occurring within the specimen as a function of temperature. However, the results may not be scalable because of the small sample size and variation in consolidation density. The test relates to initiation sensitivity and stability and may be more meaningful than either thermal stability or ignition and unconfined burning tests.

3.1.2 Parr Bomb Calorimeter. The average of 11 test runs on each sample produced the following results:

- Green IV - Gross heat of combustion = 3.432 Kcal/gm = 6177 BTU/lb.

- Violet IV - Gross heat of combustion = 2.816 Kcal/gm = 5069 BTU/lb.

While the Parr Bomb calorimeter does not provide results directly applicable to hazards classification, it does provide specific output energy available in the material. The specific output energy thus obtained can be applied to assessment of the consequence of functioning full-scale quantities, and it can influence quantity-distance and protective measures requirements. The scalability of Parr Bomb calorimeter testing is questionable because the sample is small and very likely not representative of the consolidation density of a full-scale mixture.

3.1.3 Hygroscopicity. Three samples of Green Smoke IV absorbed an average of 3.45 percent by weight of moisture under prolonged exposure to 30 $\pm$ 2°C and 90 percent relative humidity. Three samples of Violet Smoke IV absorbed an average of 26.1 percent of moisture under the same conditions. Hygroscopicity testing does not correlate with sensitivity or output and is not relevant to hazards classification except in cases where moisture content is known to significantly affect the reaction of a material.

3.1.4 Moisture (Desiccation Method). Three samples of Green Smoke IV were found to contain an average of 0.53 percent by weight of moisture; Violet Smoke IV, 0.76 percent. Moisture content of a material is not relevant to hazards classification.

3.1.5 Moisture and Volatiles (Vacuum Oven Method). Sixteen samples of Green Smoke IV were tested and found to contain 0.621 percent by weight of moisture and volatiles. Similarly, 17 samples of Violet Smoke IV were found to contain 0.524 percent moisture and volatiles. Moisture and volatile content of a material is not relevant to hazards classification.

3.1.6 Isothermal Analysis. Three samples each of Green Smoke IV and Violet Smoke IV were tested by isothermal analysis techniques. Neither material could be tested at a temperature greater than 169.25°C without decomposition. Both materials were tested at 169.25°C for 2 hours and decomposed without producing measurable exotherms or endotherms.

Results of testing with the two sulfur-based smokes are inconclusive, but it is believed that isothermal analysis, in conjunction with differential thermal analysis can be a valuable tool for assessing the reactivity of a pyrotechnic bulk material.

3.2 Tests Evaluated. Table 1 is a listing of the candidate test methods and references. The classification test method summary for each test presents the findings of engineering reviews and analyses. The application, parametric and ranking value results for all tests are summarized in table 2. A complete set of classification method summary sheets appear in appendix A.

3.3 Discussion. TB 700-2 does not adequately provide for the hazard classification of pyrotechnic bulk materials and munition end items. The TB 700-2 classification procedures are based on the presumption of an explosive material having a critical diameter of less than 1-1/2 or 2 inches. Classifications determined in accordance with TB 700-2 are based solely upon initiation sensitivity with no regard being given to output consequences of a reaction. It is implied that output damage potential is related to initiation sensitivity. That implication is not supported by actual experience. Recent experience with the testing of 70 pyrotechnic materials (as reported in GE-MTSD-R-059, et al.) has shown that a Class 7 explosion hazard results have been obtained only from the impact sensitivity test (106). The other tests invariably produced results corresponding to Class 2, fire hazard. The risk attendant to recognition that a pyrotechnic material, Class 2, might be transported or stored in a configuration greater than its particular critical diameter demands concern.

3.3.1 Initiation Sensitivity. Initiation sensitivity of a pyrotechnic material is of importance in determining hazards classification. The stimuli of interest are open flame, indirect thermal, mechanical impact, hydrodynamic shock, and electrostatic discharge. Sensitivity to friction stimuli and dust explosibility are for the most part irrelevant to the transportation or storage environments.

3.3.1.1 Open Flame. It is assumed that a pyrotechnic material is sensitive to initiation by open flame since that is an inherent characteristic. The essential question is whether the material once initiated will undergo transition to detonation. The thermal ignition test (417), ranking value 8, provides the required data on a full-scale basis. The ignition and unconfined burning test (103), ranking value 4, serves only to demonstrate that a pyrotechnic will burn in a fire.

3.3.1.2 Indirect Thermal. Indirect thermal initiation, sensitivity, and thermal stability are of paramount concern in determing the hazard classification of a pyrotechnic material. Those parameters are the following tests:

Table 1. Candidate Test Methods

Number	Test	Reference
101	Thermal Stability (75 $^{\circ}$C Oven Method)	Appendix B
102	Thermal Stability (Tube Method)	
103	Ignition and Unconfined Burning	
104	Burning Propagation Rate (Screen)	
105	Burning Propagation Rate (Tube)	
106	Impact Sensitivity (Bureau of Explosives Apparatus)	
107	Bullet Impact Friction	
108	Electrical Spark Sensitivity	
112	Differential Thermal Analysis	
113	Detonation - Compression	
114	Card Gap	
115	High Explosive Equivalency	
116	Closed Bomb	
117	Parr Bomb Calorimeter	
201	Propagation/Transition Test A	
202	Propagation/Transition Test B	
203	External Heat Test C	
204	Transporation Rough Handling	
205	Crash Safety (40 Foot Drop)	
301	Bulk Density	
302	Compatibility (Reactivity with Surroundings)	
303	Hygroscopicity	
304	Moisture (Desiccation Method)	
305	Moisture and Volatiles (Vacuum Oven Method)	
306	Moisture and Total Volatiles (Gas Chromatographic Method)	

Table 1. Candidate Test Methods (Cont'd)

Number	Test	Reference
401	75°C International Heat Test	AMCP 385-177
402	100°C Heat Test	AMCP 385-177
403	Explosion Temperature Test	AMCP 385-177
404	Hot Bar Test	AMCP 385-177
405	Impact Sensitivity Test (Bureau of Mines Apparatus)	106 (Different Apparatus)
406	Impact Sensitivity Test (Picatinny Arsenal Apparatus)	106 (Different Apparatus)
407	Friction Pendulum Test	AMCP 385-177
408	Friction Sensitivity Test	EA-FR-4D11
409	Impingement Reaction Test	EA-FR-4D11
410	Abel Heat Test	*
411	Isothermal Analysis	TES-20-73-2
412'	Hartmann Dust Sensitivity	EA-FR-1D0X
413	Large Scale Parr Bomb	EA-FR-4D11
414	Carrier Medium Test	EA-FR-4D21
415	Charging and Blending Sequence Test	EA-FR-4D21
416	Mass-Effects Test	EA-FR-4D21
417	Thermal Ignition Test	EA-FR-4D21
418	Full-Scale Blending Test	EA-FR-4D21
419	End Item Electrostatic Sensitivity	**
420	Transporation Simulation Test	EA-FR-4D71 and GE-MTSD-R-058
421	Modified Detonation Test B	Method 421 Summary Sheet

*S. Fordham, "High Explosives and Propellants", Pergamon Press, 1966.

**C. Pique, "M139 Bomblet Electrostatic Testing", (Unpublished), Edgewood Arsenal Resident Laboratory Project 4G07.

Table 2. Classification Test Accumulation and Ranking Summary

Test No.	Category			Applica-bility		Type				Parameter															Ranking Value		
	Bulk	End Item	Misc.	Transpor-tation	Storage	Sensitivity	Stability	Output	Property	Tempera-ture	Heat	Friction	Impact	Hydrodynamic Shock	Electric Spark	Reaction Rate	Energy	Air Blast	Propagation	Transition	Density	Reactivity	Moisture Content	Volatile Content	Triboelectrifi-cation	Parametric	Cost	Overall
101	X			X	X	X				X																6	1	7
102	X			X	X	X				X																6	1	7
103	X			X	X			X												X						4	1	5
104	X			X	X			X								X										6	1	7
105	X			X	X			X								X										6	1	7
106	X			X	X	X							X													6	1	7
107	X			X	X	X						X	X													3	1	4
108	X			X		X									X											7	1	8
112	X				X	X	X			X	X															8	1	9
113	X			X	X	X								X												4	1	5
114	X			X	X	X								X												5	1	6
115	X			X	X			X										X								7	0	7
116	X			X	X			X								X	X									6	1	7
117			X	X	X			X									X									8	1	9
201		X		X	X			X											X							8	1	9
202		X		X	X			X											X							8	1	9
203		X		X	X			X											X							8	0	8
204		X		X		X							X													5	0	5
205		X		X		X							X													5	0	5

Table 2. Classification Test Accumulation and Ranking Summary (Cont'd)

Test No.	Category			Applicability		Type				Parameter																Ranking Value		
	Bulk	End Item	Misc.	Transportation	Storage	Sensitivity	Stability	Output	Property	Temperature	Heat	Friction	Impact	Hydrodynamic Shock	Electric Spark	Reaction Rate	Energy	Air Blast	Propagation	Transition	Density	Reactivity	Moisture Content	Volatile Content	Triboelectrification	Parametric	Cost	Overall
301			X	X	X				X												X					5	1	6
302			X		X		X															X				7	1	8
303			X		X				X														X			4	1	5
304			X		X				X														X			4	1	5
305			X		X				X														X	X		4	1	5
306			X		X				X														X	X		4	1	5
401	X				X		X			X																6	1	7
402	X				X		X			X																4	1	5
403	X			X	X	X					X															4	1	5
404	X			X	X	X					X															4	1	5
405	X			X	X	X							X													6	1	7
406	X			X	X	X							X													6	1	7
407	X			X		X						X														3	1	4
408	X			X		X						X														5	1	6
409	X			X		X						X	X													4	1	5
410	X				X		X				X															7	1	8
411	X				X	X				X																8	1	9
412	X				X	X					X						X									6	1	7
413	X			X	X			X		X							X									6	1	7
414			X						X																X	7	1	8

Table 2. Classification Test Accumulation and Ranking Summary (Cont'd)

Test No.	Category			Application		Type				Parameter																Ranking Value		
	Bulk	End Item	Misc.	Transportation	Storage	Sensitivity	Stability	Output	Property	Temperature	Heat	Friction	Impact	Hydrodynamic Shock	Electric Spark	Reaction Rate	Energy	Air Blast	Propagation	Transition	Density	Reactivity	Moisture Content	Volatile Content	Triboelectrification	Parametric	Cost	Overall
415			X						X																X	8	1	9
416	X			X	X	X		X						X				X		X						7	0	7
417	X			X	X	X		X									X			X						8	0	8
418	X								X																X	6	0	6
419		X		X		X									X											6	1	7
420		X		X				X											X							7	1	8
421		X		X	X			X											X							8	1	9

	Ranking Value
• Thermal Stability (75°C oven method) (101)	6
• Thermal Stability (tube method) (102)	6
• Differential Thermal Analysis (112)	8
• 75°C International Heat Test (401)	6
• 100°C Heat Test (402)	4
• Explosion Temperature Test (403)	4
• Hot Bar Test (404)	4
• Isothermal Analysis (411)	8

The two thermal stability tests (101 and 102) are functionally similar in providing basic thermal stability data under reasonable maximum transportation and storage environmental conditions.

Differential thermal analysis (112) and isothermal analysis (411) provide meaningful data basic to an understanding of the chemical reactivity and physical changes of the pyrotechnic material.

3.3.1.3 Mechanical Impact. Mechanical impact initiation sensitivity is an important parameter to be considered in determining hazard classification because potential initiation sources are constantly present in the transportation and storage environment. Mechanical impact sensitivity is the subject of the following test methods:

	Ranking Value
• Impact Sensitivity (Bureau of Explosives Apparatus) (106)	6
• Bullet Impact Friction (107)	3
• Impact Sensitivity (Bureau of Mines Apparatus) (405)	6
• Impact Sensitivity (Picatinny Arsenal Apparatus) (406)	6
• Impingement Reaction Test (409)	4

The impact sensitivity test (106) using the Bureau of Explosives apparatus provides meaningful data relevant to initiation sensitivity of a pyrotechnic material. The same test using different apparatus (405 or 406) has not yet been shown to correlate with the Bureau of Explosives apparatus.

3.3.1.4 Hydrodynamic Shock. Hydrodynamic shock sensitivity is less relatable to pyrotechnic materials as it is to explosives. Hydrodynamic shock is the intended initiation stimulus for most explosives, whereas pyrotechnics are usually designed to be flame initiated. Pyrotechnics have been shown (GE-MTSD-R-059, et al.) to be shock insensitive, but the possibility should be considered.

The tests for hydrodynamic shock sensitivity are:

	Ranking Value
• Detonation-Compression (113)	4
• Card Gap (114)	5
• Mass-Effects Test (416)	7

The mass-effects test (416) is superior to the other two methods for evaluation of a pyrotechnic material, principally because quantities of material more representative of pyrotechnic handling are used. The test combines hydrodynamic shock sensitivity determination with a limit level test of critical diameter and provides blast output measurements if the material does explode or detonate. There has been little experience to date with the mass-effects test (416), but the results are promising (EA-FR-4D21).

3.3.1.5 Electrostatic Discharge. Electrostatic discharge as an initiation stimulus is more relevant to manufacturing hazards than to transportation and storage. It must be recognized, however, that thermoplastic materials are increasingly replacing metals for munition end item cases and for bulk and end-item packaging. Pyrotechnics are no longer necessarily afforded the electrostatic protection of a conductive enclosure.

Electric spark sensitivity testing (108), ranking value 7, can provide data useful in assessing the extent to which a material might be vulnerable to electrostatic initiation.

3.3.2 Output Energy Release. The output energy release characteristics of a pyrotechnic are as important as sensitivity in determining hazard classification. Output data will contribute to the establishment of quantity-distance separation criteria. The following tests provide output data:

	Ranking Value
• Ignition and Unconfined Burning (103)	4
• Burning Propagation Rate (Screen) (104)	6
• Burning Propagation Rate (Tube) (105)	6
• High Explosive Equivalency (115)	7
• Closed Bomb (116)	6
• Parr Bomb Calorimeter (117)	8
• Hartmann Dust Sensitivity (412)	6
• Large Scale Parr Bomb (413)	6
• Mass-Effects Test (416)	7
• Thermal Ignition Test (417)	8

Either of the burning propagation rate tests (104 or 105) provides useful data relative to pyrotechnic performance, and is superior to ignition and unconfined burning (103). Since more representative quantities of material are used, mass-effects test (416) provides more meaningful data than does the high explosive equivalency test (115).

The closed bomb (116) and the parr bomb calorimeter (117) tests provide basic output energy data for a pyrotechnic, but data therefrom are not always scalable to large quantities. Eventual hazard classification should be based upon larger samples, approaching full-scale, such as the mass-effects test (416) and the thermal ignition test (417). The Hartmann dust sensitivity test (412) (as noted in paragraph 3.3.1) is not relevant.

3.3.3 End-Item Testing. Propagation/transition tests A and B (201 and 202), ranking value 8 for both, provide meaningful data for hazards evaluation. These tests answer two basic questions:

- If an end item functions within its shipping container, will the reaction propagate to other similar items in the container?

- If there is propagation within a container, will the reaction propagate to other similar items in an adjacent container under free air conditions?

The answers to these questions guide the classifying authority in the establishment of quantity-distance requirements for the item under test. The modified detonation test B (421), ranking value 8, refines the test by modifying the procedure for arranging the containers in a "B" test in those cases where the standard "A" or "B" test resulted in container rupture. As before, TB 700-2 is found to be presuming an explosive in which case proximity rather than configuration is paramount. However, pyrotechnic end items frequently exhibit a directional output, especially if the end item is propulsive. In such cases, the greatest propagation hazard is in the direction of the donor output, and this modified procedure places the acceptor in the most vulnerable position.

Another area of concern is whether the transportation carrier contributes confinement that would produce a more severe output from a "B" test. The transportation simulation test (420), ranking value 7, is intended to subject the end items to partial confinement, such as within a carrier, in a reduced scale propagation test. Results to date (GE-MTSD-R-058 and EA-FR-4D71) are inconclusive but this approach is worth of further study.

The external heat test C (203), ranking value 8, is intended to provide the classifying agency with data on the performance of a quantity of packaged end items enveloped in a fire. The results of the "C" test contribute significantly to the establishment of quantity-distance requirements.

In its present form, the end-item electrostatic sensitivity test (419), ranking value 6, is used to assure that an end item is insensitive by several orders of magnitude to electrostatic initiation. As pointed out in paragraph 3.3.1.5, use of containers other than metal increases vulnerability of an end item to electrostatic stimulation.

3.3.4 Thermal Output. Since pyrotechnics burn rather than explode, the greatest energy output hazard is thermal flux rather than blast phenomena. Knowledge of the thermal output characteristics of pyrotechnic bulk material in large quantities is essential to assessment of hazard potentials and for determination of proper classification. Heat flux data is occasionally gathered in the course of other testing, but no procedures are available for specifically evaluating this parameter, nor have performance standards and limits been established.

4.0 RECOMMENDATIONS

4.1 Specific Test Methods. The results gathered from the test data suggest that a supplement to Technical Bulletin 700-2 be prepared and issued to include hazard classification testing procedures for pyrotechnic bulk materials and munition end items. The DOD component responsible for an item is at liberty to require additional testing in accordance with paragraph 1.3 of TB 700-2. It is believed that the supplemental tests will eventually gain acceptance throughout the pyrotechnic community.

4.1.1 Recommended Tests for Inclusion. The following hazard classification tests for pyrotechnics are recommended for inclusion into a supplement to TB 700-2 and for eventual inclusion into a revised TB 700-2:

Number	Bulk Test Material	Remarks
101*	Thermal Stability (75°C Oven Method)	102 Optional
102	Thermal Stability (Tube Method)	101 Optional
104	Burning Propagation Rate (Screen)	105 Optional
105	Burning Propagation Rate (Tube)	104 Optional
106*	Impact Sensitivity (Bureau of Explosives Apparatus)	
108	Electrical Spark Sensitivity	
112	Differential Thermal Analysis	
116	Closed Bomb	
117	Parr Bomb Calorimeter	
301	Bulk Density	
411	Isothermal Analysis	With 112
416	Mass Effects Test	
417	Thermal Ignition Test	

* Performance now required for TB 700-2 compliance.

Number	End-Item Material	Remarks
201*	Propagation/Transition Test A	
202*	Propagation/Transition Test B	
203	External Heat Test C	
421	Modified Detonation Test B	

4.1.2 Exclusions. The following hazard classification tests for pyrotechnics are recommended for exclusion from a supplement to TB 700-2 and eventual exclusion from a revised TB 700-2.

Number	Test
103*	Ignition and Uncontinued Burning
107	Bullet Impact - Friction
113*	Detonation - Compression
114*	Card Gap
115	High Explosive Equivalency
204	Transportation Rough Handling
205	Crash Safety (40 foot Drop)
302	Compatibility (Reactivity with Surroundings)
303	Hygroscopicity
304	Moisture (Desiccation Method)
305	Moisture and Volatiles (Vacuum Oven Method)
306	Moisture and Total Volatiles (Gas Chromatograph Method)
401	75°C International Heat Test
402	100° C Heat Test
403	Explosion Temperature Test
404	Hot Bar Test
405	Impact Sensitivity Test (Bureau of Mines Apparatus)
406	Impact Sensitivity Test (Picatinny Arsenal Apparatus)
407	Friction Pendulum Test
408	Friction Sensitivity Test
409	Impingement Reaction Test

* Performance now required for TB 700-2 compliance.

Number	Test
410	Abel Heat Test
412	Hartmann Dust Sensitivity
413	Large Scale Parr Bomb
414	Carrier Medium Tests
415	Changing and Blending Sequence Test
418	Full-Scale Blending Test

4.1.3 Test Recommended for Further Development. It is recommended that hazard classification tests of the following types be made the subject of additional projects to develop criteria, apparatus and procedures and to demonstrate their validity:

Number	Test
419	End Item Electrostatic Sensitivity
420	Transportation Simulation Test
None	Thermal Output, paragraph 3.3.4

4.2 Other Recommendations. The following statements comprise other recommendations resulting from the research conducted.

- Additional projects should be initiated to supplement this study by validating the classification tests with pyrotechnic materials other than smoke mixes. The broadened scope of test validation would enhance the credibility of findings and recommendations.

- Some nonflammable wicking material such as asbestos or sand should be substituted for the sawdust in the ignition and unconfined burning test (103). Sawdust is a variable material, and some other material would be more reproducible.

- The blast transducers should be rearranged in the high explosive equivalency test (115). The spiral array of transducers now used can result in inconclusive data because a pyrotechnic sample frequently results in pneumatic rupture of the test vessel and the resulting airblast overpressures are not cylindrically symmetric. It would be more desirable to arrange the transducers in four quadrants in each of two concentric circles. Data analysis would then reveal airblast asymmetry and allow corrections to be applied.

- A program should be conducted to document hazard classification procedures for pyrotechnic manufacture and assembly operations. TB 700-2 procedures now being applied to pyrotechnic manufacture do not properly assess the hazards involved. No account is taken of either the properties of pyrotechnics or the nature of the manufacturing environment.

SELECTED REFERENCES

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P. V. King, Sr. and D. M. Koger, GE-MTSO-R-035, "Pyrotechnic Hazards Classification and Evaluation Program, Phase I, May 1970, National Space Technology Laboratories, Bay St. Louis, MS 39520, General Electric Co., Contract NAS8-23524.

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P. V. King, Sr. and D. M. Koger, GE-MTSD-R-059, "Pyrotechnic Hazards Classification and Evaluation Program, Phase III, Segments 1-4, Investigation of Sensitivity Test Methods and Procedures for Pyrotechnic Hazards Evaluation and Classification", April 1971, National Space Technology Laboratories, Bay St. Louis, MS 39520, General Electric Co., Contract NAS8-23524.

CLASSIFICATION TEST METHOD SUMMARY

NUMBER	TEST
101	Thermal Stability (75^oC Oven method)

CATEGORY	APPLICABILITY	TYPE	PARAMETER(S)
Bulk	Trans. & Storage	Sensitivity	Temperature

DESCRIPTION

The sample is placed in an explosion-proof oven at a temperature of 75^oC and maintained at this temperature for a 48-hour period to determine whether it is physically and chemically stable.

RATIONALE

The sample is subjected to elevated temperatures to permit the observation of characteristic tendencies of the sample material to detonate, ignite, decompose or change in configuration under adverse storage conditions.

APPARATUS

Explosion proof oven regulated from 50^oC - 200^oC

INSTRUMENTATION

Balance $\pm$ 0.2 milligram accuracy
Thermocouple and temperature recorder

SAMPLE SIZE	NUMBER OF TESTS FOR VALIDITY
75 - 300 grams	1 test for a 48 hour duration.

SPECIAL REQUIREMENTS

Sample is pre-weighed and identified as to: Sample designation, lot number, manufacturer, date manufactured, lot size, and date sampled.

TESTING EXPERIENCE	RESULTS	REPORT
GREEN IV	No reaction	GE-MTSD-R035 & R059
VIOLET IV	No reaction	GE-MTSD-R035 & R059

PARAMETER	RELATABILITY +	QUANTATIVE +	SCALABILITY =	TOTAL
Temperature	4	0	2	6

APPROXIMATE COST PER MATERIAL OR END ITEM TESTED

$125 per material — RANK 1

REMARKS

This test represents the universally accepted high temperature environment at 75^oC. The addition of a thermocouple with the sample provides an indication of reactivity. The test subjects the sample material to one storage parameter; it cannot stand alone if results indicate instability.

CLASSIFICATION TEST METHOD SUMMARY

NUMBER	TEST
102	Thermal Stability (Tube Method)

CATEGORY	APPLICABILITY	TYPE	PARAMETER(S)
Bulk	Trans. & Storage	Sensitivity	Temperature

DESCRIPTION This test is conducted to determine if a sample material is stable at a temperature of 75°C and evaluate potential hazards due to an explosion, ignition or a marked change in configuration indicated by a change in color or an excessive weight loss (>10%) that may occur at the 75°C temperature.

RATIONALE
If the sample material explodes, ignites, or shows marked change in configuration due to a change in color or gross loss in weight (> 10%) the material is incompatible for shipping or storage by standard transportation and storage modes.

APPARATUS
Stainless steel tube 3/8" OD by 8" length with a .035" wall thickness. Nichrome ribbon heater is wrapped on the outside of the tube. The tube is covered by 1-inch thickness of asbestos insulation.

INSTRUMENTATION
Balance ± 0.2 milligram accuracy
Temperature regulator for controlling the heating tape
Copper constantan thermocouple - 2 each

SAMPLE SIZE	NUMBER OF TESTS FOR VALIDITY
5 grams	1 test 48 hours duration

SPECIAL REQUIREMENTS The sample is pre-weighed and identified as to: Sample designation, lot number, lot size, manufacturer's name and plant designation, date sampled, date loaded. Consolidation to end item configuration may be simulated.

TESTING EXPERIENCE	RESULTS	REPORT
GREEN IV	None	
VIOLET IV	None	

PARAMETER	RELATABILITY +	QUANTATIVE +	SCALABILITY =	TOTAL
Temperature	4	0	2	6

APPROXIMATE COST PER MATERIAL OR END ITEM TESTED

$145 per material — RANK 1

REMARKS

Test evaluation is essentially qualitative. Test method cannot stand alone as a method of classification. This test represents the universally accepted high temperature environment at 75°C. This test is a suitable alternative to the oven method and it is not limited to solids. No testing by this method was conducted at NSTL because it is functionally no different from the 75°C oven method, No. 101.

CLASSIFICATION TEST METHOD SUMMARY

NUMBER	TEST
103	Ignition and Unconfined Burning

CATEGORY	APPLICABILITY	TYPE	PARAMETER(S)
Bulk	TRANS. & STORAGE	Output	Transition

DESCRIPTION

A 2" cube is placed on a kerosene soaked sawdust bed, and the sawdust is ignited. The sample specimen is observed for signs of detonation or deflagration. The time of the reaction is measured. This test is run in two configurations: single cube and multiple cubes (4).

RATIONALE

This test determines whether a pyrotechnic, propellent or explosive material will undergo transition from deflagration to detonation when exposed to an open flame.

APPARATUS

Steel Pan	Kerosene
Sawdust	Match-head igniter

INSTRUMENTATION

Stop watch

SAMPLE SIZE	NUMBER OF TESTS FOR VALIDITY
50-120 gm	3 tests (2 each single cube and one each multiple cube test)

SPECIAL REQUIREMENTS

Sample screened through a No. 50 sieve and the temperature of the specimen stabilized to ambient prior to test.

TESTING EXPERIENCE	RESULTS	REPORT
GREEN IV	No Detonation	GE-MTSD-R035, R059
VIOLET IV	No Detonation	GE-MTSD-R035, R059

PARAMETER	RELATABILITY	QUANTITATIVE	SCALABILITY	TOTAL
Transition	2	1	1	4

APPROXIMATE COST PER MATERIAL OR END ITEM TESTED

$30 per material

RANK 1

REMARKS This test was initially used for high explosive and I-C-T will result if the critical diameter of the sample specimen is less than 2". For pyrotechnics and propellants this test only demonstrates that the propellants and pyrotechnics will burn. No evidence is available indicating that a pyrotechnic material ever did more than burn during this test. Test results can vary due to wicking of the kerosene into the sample specimen.

CLASSIFICATION TEST METHOD SUMMARY

NUMBER	TEST
104	Burning Propagation Rate (Screen)

CATEGORY	APPLICABILITY	TYPE	PARAMETER(S)
Bulk	TRANS. & STORAGE	Output	Rate of Reaction

DESCRIPTION

A bed of material is ignited at one end, and the transit time of the reaction front is measured to determine a burning propagation rate under uncontrolled conditions.

RATIONALE

This test determines an open-air burning rate and, hence, the rate of energy release can be deduced.

APPARATUS

100-mesh stainless steel screen to support bed of specimen materials propane torch for ignition.

INSTRUMENTATION

Fuse wire and electric V timer

SAMPLE SIZE	NUMBER OF TESTS FOR VALIDITY
11 cubic inches	5

SPECIAL REQUIREMENTS

Apparatus must be screened from wind. Determine bulk density. Identify sample.

TESTING EXPERIENCE	RESULTS	REPORT
GREEN IV	None	--
VIOLET IV	None	--

PARAMETER	RELATABILITY	QUANTITATIVE	SCALABILITY	TOTAL
Rate of reaction	2	2	2	6

APPROXIMATE COST PER MATERIAL OR END ITEM TESTED

$240 per material RANK 1

REMARKS

More meaningful data are obtained than from Test No. 3, ignition and uncontinued burning. Applicable only to solid or granulated materials. This test can be utilized as an alternative to the tube method, No. 105.

CLASSIFICATION TEST METHOD SUMMARY

NUMBER	TEST
105	Burning Propagation Rate (Tube)

CATEGORY	APPLICABILITY	TYPE	PARAMETER(S)
Bulk	TRANS. & STORAGE	Output	Rate of Reaction

DESCRIPTION

A cylinder of material partially confined in a steel tube is ignited at one end, and the transit time of the reaction front is measured to determine a burning propagation rate under partially confined conditions.

RATIONALE

The test determines burning rate; it is possible to obtain a rate of energy release under such conditions at confinement.

APPARATUS

Prepared steel tube to hold sample material
Ventilated hood
Propane torch for ignition

INSTRUMENTATION

Fuse wire and electric V timer

SAMPLE SIZE	NUMBER OF TESTS FOR VALIDITY
2.5 cubic inches	5

SPECIAL REQUIREMENTS

Determine bulk density. Identify sample material.

TESTING EXPERIENCE	RESULTS	REPORT
GREEN IV	None	---
VIOLET IV	None	---

PARAMETER	RELATABILITY	QUANTITATIVE	SCALABILITY	TOTAL
Rate of Reaction	2	2	2	6

APPROXIMATE COST PER MATERIAL OR END ITEM TESTED

$230 per material — RANK 1

REMARKS

More meaningful data are obtained than from Test No. 3, ignition and unconfined burning. Could be applied to liquid as well as solid or granulated materials. This test can be utilized as an alternative to the screen method, No. 104.

CLASSIFICATION TEST METHOD SUMMARY

NUMBER	TEST
106	Impace Sensitivity Test (Bureau of Explosives Apparatus)

CATEGORY	APPLICABILITY	TYPE	PARAMETER(S)
Bulk	TRANS. & STORAGE	SENSITIVITY	Impace

DESCRIPTION

A 10 mg. sample is subjected to impact by a weight falling from a predetermined height.

RATIONALE

This test determines the sensitivity of a pyrotechnic, propellant or explosive mixture to decomposition or detonation as a result of mechanical shock caused by impact.

APPARATUS

Bureau of Explosives Test Apparatus

INSTRUMENTATION

None required. Strain gage, load cell, or Piezolectic crystal to measure force of impact and rebound of the falling weight is optional.

SAMPLE SIZE	NUMBER OF TESTS FOR VALIDITY
10 milligram	10 tests at each predetermined drop height.

SPECIAL REQUIREMENTS Sieving sample through 50 mesh screen. Temperature of the sample stabilized to 25 $\pm$ 50^{o}C prior to test. Weigh material to 10 mg. identify sample material as to type, manufacture date, lot number, date tested, etc. NOTE: Test must be performed under controlled humidity conditions.

TESTING EXPERIENCE	RESULTS	REPORT
GREEN IV	No Reaction @ 3-3/4 or 10"	GE-MTSD-R035-R059
VIOLET IV	No Reaction @ 3-3/4 Reaction @ 10"	GE-MTSD-R035-R059

PARAMETER	RELATABILITY	QUANTITATIVE	SCALABILITY	TOTAL
Impact	3	2	1	6

APPROXIMATE COST PER MATERIAL OR END ITEM TESTED

$170 per material — RANK 1

REMARKS The test as defined is a "go-no go" test. A reaction at either drop height constitutes a "no go" and the material is classified as DOT restricted or Class A. Sample size is too small for good statistical data. Data obtained with this apparatus do not correlate with those obtained on other test apparati; such as the Bureau of Mines or PA apparatus.

CLASSIFICATION TEST METHOD SUMMARY

NUMBER	TEST		
107	Bullet Impact (Friction)		
CATEGORY	**APPLICABILITY**	**TYPE**	**PARAMETER(S)**
Bulk	TRANS. & STORAGE	SENSITIVITY	Impact/Friction

DESCRIPTION A bulk material specimen is placed on a target table and a 0.30 caliber bullet is fired so that the bullet strikes perpendicular to the longitudinal axis of the specimen. Data is recorded to denote detonation, deflagration or no reaction.

RATIONALE This test determines the sensitivity or specifically the critical diameter of a bulk or end item pyrotechnic propellant, explosive mixtures to the combination impact and friction.

APPARATUS
30 caliber weapon, bench mounted.
Balance accurate to one gram
(cast iron pipe with threaded caps for bulk testing)

INSTRUMENTATION
Blast gages to measure peak overpressure.
Timing circuit to measure impact velocity.

SAMPLE SIZE	NUMBER OF TESTS FOR VALIDITY
50-200 gms. bulk. Unlimited mass end item.	5 trials using a single bullet against a single target. 5 trials using multiple bullets (5) against a single target.

SPECIAL REQUIREMENTS
Sample identification. Bulk samples must be screened through a number 50 sieve.

TESTING EXPERIENCE	RESULTS	REPORT
GREEN IV	None	---
VIOLET IV	None	---

PARAMETER	RELATABILITY	QUANTITATIVE	SCALABILITY	TOTAL
Impact/Friction	1	1	1	3

APPROXIMATE COST PER MATERIAL OR END ITEM TESTED

$310 per material — RANK 1

REMARKS Varied reactions can result depending upon where the bullet strikes the target. This is particularly noticeable in end item testing. This test is capable of imparting approximately 14 joules of energy to the sample but because of deflection some lesser value is obtained. Additionally, it is not known how the input energy is divided between impact and friction.

CLASSIFICATION TEST METHOD SUMMARY

NUMBER	TEST
108	Electrical Spark Sensitivity

CATEGORY	APPLICABILITY	TYPE	PARAMETER(S)
Bulk	TRANS.	SENSITIVITY	Electrical Spark

DESCRIPTION A small sample of material is placed on an anode, and an electrical spark is discharged through the material. The energy level at which the material initiates is determined.

RATIONALE This test determines the sensitivity of a pyrotechnic mixture to ignition by an electrical spark. The sensitivity is expressed in terms of minimum energy (Joules) required for initiation.

APPARATUS
Huhes Model 410 or equivalent H V Power supply 10,000 vdc. capacitors; 0.002, 0.01, 0.02, 0.05, 0.1 and 1, microfarad limiting resistors and switching device charging and discharging capacitors.

INSTRUMENTATION

10,000 vdc voltmeter

SAMPLE SIZE	NUMBER OF TESTS FOR VALIDITY
10-50 mg.	Staircase - approximately 30.

SPECIAL REQUIREMENTS

Sample material screened through a No. 50 sieve. Sample material stabilized.

TESTING EXPERIENCE	RESULTS	REPORT
GREEN IV	0.131 joule	GE-HERE-R059
VIOLET IV	0.161 joule	GE-HERE-R059

PARAMETER	RELATABILITY	QUANTITATIVE	SCALABILITY	TOTAL
Electrical Spark	3	2	2	7

APPROXIMATE COST PER MATERIAL OR END ITEM TESTED

$100 per material — RANK 1

REMARKS

This is the only test currently available to evaluate electrical spark sensitivity of bulk materials. The limitations of the test as it stands must be recognized:

.The apparatus is not standardized; results will vary among testers.

.Discharging the spark into a pile of material frequently results in the material being scattered.

.To date, only spark energy has been considered; voltage might also be a significant factor.

CLASSIFICATION TEST METHOD SUMMARY

NUMBER	TEST
112	Differential Thermal Analysis (DTA)

CATEGORY	APPLICABILITY	TYPE	PARAMETER(S)
Bulk	STORAGE	SENSITIVITY	Temperature and Heat

DESCRIPTION

Determine ignition temperature and reaction prior to ignition.

RATIONALE

Temperature of reacting composition can determine its hazard potential either in its decomposition or any physical change.

APPARATUS

Fisher Model 200 Differential Thermal Analyzer (DTA).

INSTRUMENTATION

DTA with platinel thermocouples and dual tract strip chart recorder.

SAMPLE SIZE	NUMBER OF TESTS FOR VALIDITY
50-100 mg.	3

SPECIAL REQUIREMENTS

Particle size: Sample 100-200 mesh. Reference (Alumina) 100-200 mesh.

TESTING EXPERIENCE	RESULTS	REPORT
GREEN IV	221.68°C	9 V. par. 3.1.1
VIOLET IV	239.88°C	9 V. par. 3.1.1

PARAMETER	RELATABILITY	QUANTITATIVE	SCALABILITY	TOTAL
Ignition Temperature	3	2	3	8

APPROXIMATE COST PER MATERIAL OR END ITEM TESTED

$190 per material RANK 1

REMARKS

Valid test, extremely useful in determining reaction characteristics of pyrotechnic materials, however, the controlled rate of heating being extremely applied can give suspect exotherms.

CLASSIFICATION TEST METHOD SUMMARY

NUMBER	TEST
113	Detonation - Compression

CATEGORY	APPLICABILITY	TYPE	PARAMETER(S)
Bulk	TRANS. & STORAGE	SENSITIVITY	Hydrodyromic Shock

DESCRIPTION

A two inch cube of a pyrotechnic mixture is placed atop a lead cylinder and a number 8 blasting cap in contact with the specimen is initiated.

RATIONALE

This test determines the sensitivity of a pyrotechnic material to the exposure of moderate shock and heat of a number 8 blasting cap.

APPARATUS

Open Field Test
Solid lead cylinder - 1-1/2" diameter by 4" long
Mild steel plate, SAE 1010-1030 1/2" thick by 12" square
Number 8 blasting cap.

INSTRUMENTATION

None Required

Go-No Go Gage

SAMPLE SIZE	NUMBER OF TESTS FOR VALIDITY
50-300 grams	5

SPECIAL REQUIREMENTS

Sample screened through a No. 50 sieve prior to test and temperature of the test specimen stabilized to ambient temperature.

TESTING EXPERIENCE	RESULTS	REPORT
GREEN IV	No Reaction	GE-MTSD-R035, R059
VIOLET IV	No Reaction	GE-MTSD-R035, R059

PARAMETER	RELATABILITY	QUANTITATIVE	SCALABILITY	TOTAL
Hydrodyromic Shock	2	1	1	4

APPROXIMATE COST PER MATERIAL OR END ITEM TESTED

$150 per material — RANK 1

REMARKS

This test is a qualitative test where the resultant reaction is the deformation of the lead cylinder. This is measured by a go/no-go gage with a known tolerance. This test is only valid for a material with a critical diameter equal to or less than 2 inches, generally excluding pyrotechnic mixtures.

CLASSIFICATION TEST METHOD SUMMARY

NUMBER	TEST
114	Card Gap Test

CATEGORY	APPLICABILITY	TYPE	PARAMETER(S)
Bulk	TRANS. & STORAGE	SENSITIVITY	Hydrodynamic Shock

DESCRIPTION A 1-1/2" x 5" schedule 80 pipe is filled with a pyrotechnic explosive mixture, two 1" x 2" diameter pentolite pellets are placed atop the tube. The tube is set in a 6" square x 3/8" thick witness plate. A J-2 engineers special blasting cap is initiated and the results are determined by observing a clean hole the diameter of the pipe in the witness plate.

RATIONALE

This test determines the reaction of a pyrotechnic material under the influence of external shock and heat of an explosion.

APPARATUS Cold drawn seamless tube 1-1/2" diameter, 5-1/2" length, .200±.20 wall thickness. Steel witness plate SAE 1010 steel with Rockwell hardness B 6" x 6" X 3/8". Two penolite pellets 1" x 2" diameter. Cellulose acetate cords 0.01" thick, 1/16" plastic spacers.

INSTRUMENTATION

Non Required.

SAMPLE SIZE	NUMBER OF TESTS FOR VALIDITY
50-300 grams	Trial and observation to obtain 50% value. Minimum 3 if no detonation.

SPECIAL REQUIREMENTS
Sample screened through a No. 50 sieve and stabilized to ambient temperature prior to test.

TESTING EXPERIENCE	RESULTS	REPORT
GREEN IV	Burned	GE-MTSD-R035, R059
VIOLET IV	Burned	GE-MTSD-R035, R059

PARAMETER	RELATABILITY	QUANTITATIVE	SCALABILITY	TOTAL
Hydrodynamic Shock	3	1	1	5

APPROXIMATE COST PER MATERIAL OR END ITEM TESTED

$240 per material RANK 1

REMARKS

Test presupposes that a detonation will occur. The value obtained is expressed in number of cards which equates to a form of classification. Test has value for determining explosives properties but not necessarily a good classification test. Detonation will not result with materials having critical diameters greater than 1.44", thus excluding pyrotechnic mixtures in general.

CLASSIFICATION TEST METHOD SUMMARY

NUMBER	TEST
115	High Explosive Equivalency

CATEGORY	APPLICABILITY	TYPE	PARAMETER(S)
Bulk	TRANS. & STORAGE	Output	High Explosive Equivalency

DESCRIPTION

A confined sample is initiated by a J-2 blasting cap.

RATIONALE

Determines ratio of amount of energy released in an explosive reaction of the sample to the energy released by a high explosive under the same conditions.

APPARATUS

Capped steel tube specimen holder and overhead support.

INSTRUMENTATION

Blast overpressure instrumentation system with data processing capability.

SAMPLE SIZE	NUMBER OF TESTS FOR VALIDITY
50-300 grams	7 (5 sample, 2 C-4 for reference)

SPECIAL REQUIREMENTS

Sieve sample through 50-mesh screen. Identify sample material.

TESTING EXPERIENCE	RESULTS	REPORT
GREEN IV	4.30% TNT	GE-MTSD-R035, R059
VIOLET IV	6.53% TNT	GE-MTSD-R035, R059

PARAMETER	RELATABILITY	QUANTITATIVE	SCALABILITY	TOTAL
Blast overpressure and impulse expressed as % of TNT	3	2	2	7

APPROXIMATE COST PER MATERIAL OR END ITEM TESTED

$720 per sample — RANK 0

REMARKS This test permits evaluation of a material's damage potential by obtaining an equivalent mass at high explosive, however, the materials airblast parameters could be applied directly to engineering problems. This test is meaningful only if the material reacts explosively. The equivalency data can be used to establish quantity-distance requirements for the material. The spiral array of pressure transducers described for Method 115 in Appendix B is subject to spurious results because the airblast may not be concentric. Frequently pyrotechnic mixtures result in pneumatic rupture of the vessel which produces a directional airblast. An array of eight transducers in four quandrants at two radii will result in more meaningful data.

CLASSIFICATION TEST METHOD SUMMARY

NUMBER	TEST
116	Closed Bomb (Instrumented Parr Bomb)

CATEGORY	APPLICABILITY	TYPE	PARAMETER(S)
Bulk	TRANS. & STORAGE	Output	Rate of Reaction and Energy Release

DESCRIPTION

The sample is ignited in a closed bomb. Pressure versus time data are recorded to obtain a rate of pressure rise value that is proportional to rate of energy released.

RATIONALE

The rates of reaction and energy release for the sample are compared to the same rates for a high explosive in an attempt to replace the samples output to a high explosives.

APPARATUS

200 cc closed bomb
Ignition system

INSTRUMENTATION

Dynamic pressure recording system
Analytical balance

SAMPLE SIZE	NUMBER OF TESTS FOR VALIDITY
10-40 grams	6

SPECIAL REQUIREMENTS

Identify sample material

TESTING EXPERIENCE	RESULTS	REPORT
GREEN IV	220 psig .8 sec/gm	GE-MTSD-059
VIOLET IV	200 psig .8 sec/gm	GE-MTSD-059

PARAMETER	RELATABILITY	QUANTITATIVE	SCALABILITY	TOTAL
Rate of reaction and energy release. "Relative Quickness" and "Relative Force"	3	2	1	6

APPROXIMATE COST PER MATERIAL OR END ITEM TESTED

$200 per sample — RANK 1

REMARKS

Does not correlate well with larger scale equivalency testing. Not representative of actual conditions because of small quantity tested.

CLASSIFICATION TEST METHOD SUMMARY

NUMBER	TEST		
117	Parr Bomb Calorimeter		
CATEGORY	**APPLICABILITY**	**TYPE**	**PARAMETER(S)**
Misc.	TRANS. & STORAGE	Output	Energy Output

DESCRIPTION

Determine the energy output per unit mass of reacting composition as heat of combustion and heat of explosion

RATIONALE

Energy output per unit mass can be used to determine hazard potential

APPARATUS

Parr Bomb Calorimeter Series 1300 and associated equipment.

INSTRUMENTATION

Thermocouple or thermometer recording of temperature versus time

SAMPLE SIZE	NUMBER OF TESTS FOR VALIDITY
1.0 gram	3

SPECIAL REQUIREMENTS

Sample is analyzed in the bulk granular state.

TESTING EXPERIENCE	RESULTS	REPORT
GREEN IV	6,177.84 Btu/lb.	9.V. par. 3.1.2
VIOLET IV	5,069.24 Btu/lb.	9.V. par. 3.1.2

PARAMETER	RELATABILITY	QUANTITATIVE	SCALABILITY	TOTAL
Energy Output	3	2	3	8

APPROXIMATE COST PER MATERIAL OR END ITEM TESTED

$320 per materials

RANK 1

REMARKS

Excellent test for evaluation of fuels and some pyrotechnics. Granular samples are mandatory. Spurious results can be obtained because of the high partial pressure of oxygen in the bomb. For instance, dyestuff might burn under 5 atmospheres of oxygen but not in air.

CLASSIFICATION TEST METHOD SUMMARY

NUMBER	TEST
201	Propagation/Transition Test A

CATEGORY	APPLICABILITY	TYPE	PARAMETER(S)
END ITEM	TRANS. & STORAGE	Output	Propagation

DESCRIPTION

This test is conducted on pyrotechnic end items which are packaged in an experimental or standard storage and shipping container. The test is performed on a single loaded end item container.

RATIONALE

This test determines whether the functioning of one round will propagate to surrounding end items within the same container.

APPARATUS

Open field testing employing end item packing container. J-2 engineers special blasting cap or, preferrably, device for initiating normal function of the end item.

INSTRUMENTATION

Heat Flux
Blast Measurement
Documentary Motion Picture
Before and After Still Photograph

SAMPLE SIZE	NUMBER OF TESTS FOR VALIDITY
Approx. 1.5 cu. ft. depending on pkg. size	Five or until propagation occurs or the outside container is ruptured.

SPECIAL REQUIREMENTS

Sample identification.

TESTING EXPERIENCE	RESULTS	REPORT
GREEN IV (Grenade)	No Propagation	GE-MTSD-R035, R059
VIOLET IV (Grenade)	No Propagation	GE-MTSD-R035, R059

PARAMETER	RELATABILITY	QUANTITATIVE	SCALABILITY	TOTAL
Propagation within container	3	2	3	8

APPROXIMATE COST PER MATERIAL OR END ITEM TESTED

$280 per item — RANK 1

REMARKS

Test must be performed on each end item and in each shipping container configuration. Results from this test do not lead directly to classification but serve to establish compatibility during storage and transportation.

CLASSIFICATION TEST METHOD SUMMARY

NUMBER	TEST
202	Propagation/Transition Test B

CATEGORY	APPLICABILITY	TYPE	PARAMETER(S)
END ITEM	TRANS. & STORAGE	Output	Propagation

DESCRIPTION

This test is conducted on pyrotechnic end item which are packaged in experimental or standard storage and shipping containers. This test is performed on two end item containers placed adjacent to one another.

RATIONALE

This test determines whether functioning of a donor round in a donor container propagates to an acceptor container placed adjacent to the donor container.

APPARATUS Open field testing employing two (2) end item packaging containers, J-2 engineers special blasting cap or, preferrably, device for initiating normal function at the end item.

INSTRUMENTATION

Heat Flux	Documentary Motion Picture
Blast Measurement	Before and After Still Photographs

SAMPLE SIZE	NUMBER OF TESTS FOR VALIDITY
Approx. 3 cu. ft. depending on pkg. size	Five or until propagation occurs.

SPECIAL REQUIREMENTS

Sample identification.

TESTING EXPERIENCE	RESULTS	REPORT
GREEN IV (Grenade)	Not required	GE-MTSD-R035, R059
VIOLET IV (Grenade)	Not required	GE-MTSD-R035, R059

PARAMETER	RELATABILITY	QUANTITATIVE	SCALABILITY	TOTAL
Propagation to adjacent container	3	2	3	8

APPROXIMATE COST PER MATERIAL OR END ITEM TESTED

$280 per item — RANK 1

REMARKS

This test is performed in the event the standard end item test, detonation test "A" results in propagation or the shipping container ruptures. This test is omitted if the results of the propagation/transition test A are negative. Due to the ambiguity of the placement of the donor & acceptor containers, test results can be varied. Results from this test do not lead directly to classification but serve to establish compatibility during storage and transportation.

CLASSIFICATION TEST METHOD SUMMARY

NUMBER	TEST
203	External Heat Test (C Test)

CATEGORY	APPLICABILITY	TYPE	PARAMETER(S)
END ITEM	TRANS. & STORAGE	Output	Propagation

DESCRIPTION

This test is conducted on pyrotechnic end items which are packaged in experimental or standard storage and shipping containers. One to six containers are used in this test.

RATIONALE

This test determines the potential hazards of transition from deflagration to detonation of packaged end items when they are enveloped in a hot open fire.

APPARATUS

Open field testing
One to six boxes placed atop a wooden pile 3' x 3' soaked with 50 gallons of diesel fuel

INSTRUMENTATION

Heat Flux
Optical Pyrometer or
(Optical Multichannel Analyzer)
Documentary Motion Picture
Before and After Still Photography

SAMPLE SIZE	NUMBER OF TESTS FOR VALIDITY
Approx. 10 cu. ft. depending on pkg. size	1

SPECIAL REQUIREMENTS

Sample identification

TESTING EXPERIENCE

	RESULTS	REPORT
GREEN IV (Grenade)	No explosion	GE-NTSD-R035, R059
VIOLET IV (Grenade)	No explosion	GE-NTSD-R035, R059

PARAMETER	RELATABILITY	QUANTITATIVE	SCALABILITY	TOTAL
Propagation	3	2	3	8

APPROXIMATE COST PER MATERIAL OR END ITEM TESTED

$740 per item — RANK 0

REMARKS

This test is comparable to "worst case" conditions in a transportation accident. Results of this test results in establishing compatibility during storage and transportation; they do not lead directly to classification.

CLASSIFICATION TEST METHOD SUMMARY

NUMBER	TEST
204	Transportation Rough Handling

CATEGORY	APPLICABILITY	TYPE	PARAMETER(S)
END ITEM	TRANS.	SENSITIVITY	Mechanical Shock and Vibration

DESCRIPTION

Various transportation shock and vibration stimuli are imposed on packaged end items.

RATIONALE

These tests are designed to simulate severeinduced shock and vibration environments to demonstrate container performance.

APPARATUS

Extensive shaker facility with high and low temperature capabilities.
Repetitive shock tester
Drop hook and drop pad

INSTRUMENTATION

None except that incident to apparatus operation.

SAMPLE SIZE	NUMBER OF TESTS FOR VALIDITY
2 end items or bulk material pkgs.	Numerous

SPECIAL REQUIREMENTS

Itentify sample items

TESTING EXPERIENCE	RESULTS	REPORT
GREEN IV	None	---
VIOLET IV	None	---

PARAMETER	RELATABILITY	QUANTITATIVE	SCALABILITY	TOTAL
Container mechanical performance		2	3	5

APPROXIMATE COST PER MATERIAL OR END ITEM TESTED

$20,000 per item RANK 0

REMARKS

These are container tests rather than classification tests. Four or five foot drop tests of an unpackaged end item in various attitudes would serve to demonstrate its invulnerability.

CLASSIFICATION TEST METHOD SUMMARY

NUMBER	TEST
205	Crash Safety (40 foot drop)

CATEGORY	APPLICABILITY	TYPE	PARAMETER(S)
END ITEM	TRANS.	SENSITIVITY	Mechanical Shock

DESCRIPTION

Forth foot drop test of packaged end items.

RATIONALE

Demonstrates containers ability to maintain integrity and contain product through severe mechanical shock.

APPARATUS

Structure to suspend packaged sample and drop it from a height of 40 feet.

INSTRUMENTATION

None except still documentary camera.

SAMPLE SIZE	NUMBER OF TESTS FOR VALIDITY
4 end items or bulk material packages	4

SPECIAL REQUIREMENTS

Identify samples

TESTING EXPERIENCE	RESULTS	REPORT
GREEN IV	None	---
VIOLET IV	None	---

PARAMETER	RELATABILITY	QUANTITATIVE	SCALABILITY	TOTAL
Container mechanical performance	0	2	3	5

APPROXIMATE COST PER MATERIAL OR END ITEM TESTED

$900 per item

RANK 0

REMARKS

This is a container test; it is not revelant to classification. This test is normally limited to containers for radioactive materials where loss or dispersal of contents is considered catastrophic.

CLASSIFICATION TEST METHOD SUMMARY

NUMBER	TEST
301	Bulk Density

CATEGORY	APPLICABILITY	TYPE	PARAMETER(S)
Misc.	TRANS. & STORAGE	Property	Density

DESCRIPTION

A measured volume of sample material is weighed to obtain the materials bulk density.

RATIONALE

Data can be used in calculating subsequent density related factors such as "critical mass"

APPARATUS

Graduated cylinder

INSTRUMENTATION

Analytical balance $\pm$ 10 mg accuracy.

SAMPLE SIZE	NUMBER OF TESTS FOR VALIDITY
100 ml	5

SPECIAL REQUIREMENTS

Identify sample

TESTING EXPERIENCE	RESULTS	REPORT
GREEN IV	.89 g/cc	EA-FR-1DOX
VIOLET IV	.76 g/cc	EA-FR-1DOX

PARAMETER	RELATABILITY	QUANTITATIVE	SCALABILITY	TOTAL
Density	0	2	3	5

APPROXIMATE COST PER MATERIAL OR END ITEM TESTED

$35 per material — RANK 1

REMARKS

This is a laboratory test that measures a material property; it does not relate to hazards or classification.

CLASSIFICATION TEST METHOD SUMMARY

NUMBER	TEST
302	Compatibility (Reactivity with Surroundings)

CATEGORY	APPLICABILITY	TYPE	PARAMETER(S)
Misc	STORAGE	Stability	Chemical Reactivity

DESCRIPTION

A 1-5 gram sample is dried and then placed in a glass heating tube. The tube is then placed in a constant temperature bath 90°C and evacuated to a pressure of 5mm mercury. The test is then continued for a minimum of 48 hours.

RATIONALE

This test determines the compatibility of a pyrotechnic mixture with other materials in which it comes in contact throughout its life cycle.

APPARATUS

Constant temperature bath
Specially constructed sample tube (compatibility apparatus)
Vacuum pump

INSTRUMENTATION

Balance accurate to 0.2 milligrams
Temperature recorder
Vacuum gage

SAMPLE SIZE	NUMBER OF TESTS FOR VALIDITY
1 - 5 grams	1 test at each desired temperature

SPECIAL REQUIREMENTS Pre-weigh sample material. Identify sample material, prepare constant temperature bath, perform determination of the sample material, contact material and the combination of sample material and contact material in the capillary.

TESTING EXPERIENCE	RESULTS	REPORT
GREEN IV	None	---
VIOLET IV	None	---

PARAMETER	RELATABILITY	QUANTITATIVE	SCALABILITY	TOTAL
Chemical Reactivity	3	2	2	7

APPROXIMATE COST PER MATERIAL OR END ITEM TESTED

$170 per material — RANK 1

REMARKS

Direct comparison of test values between different materials is not always possible. Test method does include more than one stability parameter. Similar test methods include: thermal stability oven and tube methods, 75 and 100°C heat tests and the Abel heat test. This test is probably not desirable where traditional materials are involved. It will be of interest to the designer or developer who introduces a novel material.

CLASSIFICATION TEST METHOD SUMMARY

NUMBER	TEST		
303	Hygroscopicity		
CATEGORY	**APPLICABILITY**	**TYPE**	**PARAMETER(S)**
Misc	Mfg. Storage	Property	Moisture Absorption

DESCRIPTION

Determine moisture absorptivity characteristics of pyrotechnics.

RATIONALE

Moisture has an important role in the formation of pyrotechnic mixes. Its role in sensitizing compositions must be known.

APPARATUS

Controlled environment. Desiccator and Sulfuric acid concentration variations.

INSTRUMENTATION

Analytic balance

SAMPLE SIZE	NUMBER OF TESTS FOR VALIDITY
10.0 gram	3

SPECIAL REQUIREMENTS

Relative humidity of 90%
1 density sample

TESTING EXPERIENCE	RESULTS	REPORT
GREEN IV	3.46% moisture absorbed	q.v. par. 3.1.3
VIOLET IV	26.1% moisture absorbed	q.v. par. 3.1.3

PARAMETER	RELATABILITY	QUANTITATIVE	SCALABILITY	TOTAL
Moisture absorption	0	2	2	4

APPROXIMATE COST PER MATERIAL OR END ITEM TESTED

$80 per material

RANK 1

REMARKS

Complex test having multiple interferrences including: Temperature, humidity, time. Actual use in determining hazard potential is questionable without assessing effect of moisture on the reaction.

CLASSIFICATION TEST METHOD SUMMARY

NUMBER	TEST
304	Moisture (Desiccation Method)

CATEGORY	APPLICABILITY	TYPE	PARAMETER(S)
Misc.	Storage	Property	Moisture

DESCRIPTION

Determine moisture content of pyrotechnic materials.

RATIONALE

While moisture has an important role in the functioning end items, its effect during manufacture could have adverse effects leading to potential hazards.

APPARATUS

Desiccator
Oven

INSTRUMENTATION

None

SAMPLE SIZE	NUMBER OF TESTS FOR VALIDITY
10.0 gram	3 tests routinely performed.

SPECIAL REQUIREMENTS

Identify sample.

TESTING EXPERIENCE	RESULTS	REPORT
GREEN IV	.53% by weight	q.v. par. 3.1.4
VIOLET IV	.76% by weight	q.v. par. 3.1.4

PARAMETER	RELATABILITY	QUANTITATIVE	SCALABILITY	TOTAL
Moisture	0	2	2	4

APPROXIMATE COST PER MATERIAL OR END ITEM TESTED

$80 per material RANK 1

REMARKS

Questionable benefit in assessing pyrotechnics hazards unless combined with some other test to indicate results from loss or change in moisture content. Results are suspect with highly hygroscopic material and invalid if volatiles are present.

CLASSIFICATION TEST METHOD SUMMARY

NUMBER	TEST
305	Moisture & Volatiles (Vacuum Oven Method)

CATEGORY	APPLICABILITY	TYPE	PARAMETER(S)
Misc	Storage	Property	% Moisture & Volatiles

DESCRIPTION

Determine moisture and volatiles by vacuum oven technique.

RATIONALE

Volatile substances which may be present in pyrotechnics will volatize by this technique.

APPARATUS

Vacuum oven
Desiccator

INSTRUMENTATION

None.

SAMPLE SIZE	NUMBER OF TESTS FOR VALIDITY
10 gram	3

SPECIAL REQUIREMENTS

Identify sample.

TESTING EXPERIENCE	RESULTS	REPORT
GREEN IV	.621% by weight	q.v. par. 3.1.5
VIOLET IV	.524% by weight	q.v. par. 3.1.5

PARAMETER	RELATABILITY	QUANTITATIVE	SCALABILITY	TOTAL
Moisture and Volatiles	0	2	2	4

APPROXIMATE COST PER MATERIAL OR END ITEM TESTED

$85 per material — RANK 1

REMARKS

Results obtained are unreliable when the sample is highly hygroscopic. This test by itself does not indicate hazards potential. It must be integrated with some other data to indicate the consequences of moisture and volatile content.

CLASSIFICATION TEST METHOD SUMMARY

NUMBER	TEST
306	Moisture and Total Volatiles (Gas Chromatographic Method)

CATEGORY	APPLICABILITY	TYPE	PARAMETER(S)
Misc	Storage	Property	Moisture and Volatile Content

DESCRIPTION

Moisture and volatiles (ethyl alcohol and diethyl ether) are extracted from the sample and analyzed by a gas chromatograph.

RATIONALE

Moisture and volatile matter content determinations are used to evaluate potential hazards on the basis of the amounts of moisture and volatiles in the material.

APPARATUS

Laboratory apparatus and reagents.

INSTRUMENTATION

Gas chromatograph equipped with thermal conductivity detector and recorder and integrator.

SAMPLE SIZE	NUMBER OF TESTS FOR VALIDITY
10 grams	1

SPECIAL REQUIREMENTS

Identify sample

TESTING EXPERIENCE	RESULTS	REPORT
GREEN IV	None	---
VIOLET IV	None	---

PARAMETER	RELATABILITY	QUANTITATIVE	SCALABILITY	TOTAL
Moisture and total volatiles	0	2	2	4

APPROXIMATE COST PER MATERIAL OR END ITEM TESTED

$180 per material

RANK 1

REMARKS

Only used when volatiles are suspected of constituting a hazard.

CLASSIFICATION TEST METHOD SUMMARY

NUMBER	TEST
401	75^{o}C International Heat Test

CATEGORY	APPLICABILITY	TYPE	PARAMETER(S)
Bulk	STORAGE	Stability	Temperature

DESCRIPTION

A 10-gram sample is subjected to an elevated temperature for 48 hours. The sample after this exposure is observed for signs of decomposition or volatility.

RATIONALE

This test determines the thermal stability of a given material.

APPARATUS

Oven, regulated from 50^{o}C - 400^{o}C.

INSTRUMENTATION

Balance accurate to 0.2 milligrams.

SAMPLE SIZE	NUMBER OF TESTS FOR VALIDITY
10 grams	1 test 48 hours

SPECIAL REQUIREMENTS

Pre-weigh sample, identify as to: Sample designation, lot number, lot size, manufacturer's name and plant designation, date sampled and date loaded.

TESTING EXPERIENCE	RESULTS	REPORT
GREEN IV	None	---
VIOLET IV	None	---

PARAMETER	RELATABILITY	QUANTITATIVE	SCALABILITY	TOTAL
Heat	4	0	2	6

APPROXIMATE COST PER MATERIAL OR END ITEM TESTED

$125 per material — RANK 1

REMARKS

This is a "Go/No-Go" test, and the test results cannot stand alone. If signs of volatility or decomposition are noted additional tests should be performed. This test method only attempts to validate one parameter found in prolonged storage and sample size. The 75^{o}C high temperature environment is universally accepted.

CLASSIFICATION TEST METHOD SUMMARY

NUMBER	TEST
402	100°C Heat Test

CATEGORY	APPLICABILITY	TYPE	PARAMETER(S)
Bulk	Storage	Stability	Temperature

DESCRIPTION

A sample is heated for two 48-hour periods at 100°C. It is also exposed for 100 hours at 100°C.

RATIONALE

This test determines if the sample material retains its properties during some specified period of time.

APPARATUS

Oven, regulated from 50°C - 200°C.

INSTRUMENTATION

Balance accurate to 0.2 milligrams
Method for determining proper temperature.

SAMPLE SIZE	NUMBER OF TESTS FOR VALIDITY
0.6 grams	3 tests (2 at 48 hours) (1 at 100 hours)

SPECIAL REQUIREMENTS

Pre-weigh sample, identify as to: Sample designation, lot number, manufacturer date, end date sampled.

TESTING EXPERIENCE	RESULTS	REPORT
GREEN IV	None	---
VIOLET IV	None	---

PARAMETER	RELATABILITY +	QUANTATIVE +	SCALABILITY =	TOTAL
Heat	2	0	2	4

APPROXIMATE COST PER MATERIAL OR END ITEM TESTED

$155 per material — RANK 1

REMARKS

This test is a "Go/No Go" test, and the test results cannot stand alone. Sample size is too small to put much weight in results. Additional tests are required if a "no Go" is the result. This test method only attempts to validate one parameter found in prolonged storage. The 100oC temperature is not representative of the actual environment; at best, it is an attempt at artificial aging.

CLASSIFICATION TEST METHOD SUMMARY

NUMBER	TEST
403	Explosion Temperature Test

CATEGORY	APPLICABILITY	TYPE	PARAMETER(S)
Bulk	Trans. & Storage	Sensitivity	Heat

DESCRIPTION

A sample specimen is placed in a gilding metal shell and is compacted by tamping. This loaded shell is then immersed to a fixed depth in a molten Wood's metal bath. The time required for detonation is noted and plotted on a time temperature-curve and the time-temperature required to cause flashing or explosion in 5 seconds is extrapolated.

RATIONALE

This test determines the temperature at which the specimen will flash or detonate when held at that temperature for a specified length of time.

APPARATUS

Electric Furnace
Molten Wood's Metal

INSTRUMENTATION

Pyrometer
Thermocouple
Timer

SAMPLE SIZE	NUMBER OF TESTS FOR VALIDITY
20 mg.	Sufficient number times to validate temperature-time curve.

SPECIAL REQUIREMENTS

Sample must be screened through a No. 50 sieve. Weighing of sample material. Temperature of the specimen sample must be stabilized at 25 $\pm$ 5°C prior to test. Sample specimen is then tamped in the metal shells.

TESTING EXPERIENCE	RESULTS	REPORT
GREEN IV	None	---
VIOLET IV	None	---

PARAMETER	RELATABILITY +	QUANTATIVE +	SCALABILITY =	TOTAL
Heat	2	1	1	4

APPROXIMATE COST PER MATERIAL OR END ITEM TESTED

$175 per material — RANK 1

REMARKS

Data obtained by indirect method after plotting temperature versus time. Data dependent upon explosion or deflagration and maximum temperature is the melting point of Wood's metal. Similar tests include Hot Bar Test. This test is inadequate when compared to DTA or ITA particularly in view of the small sample size.

CLASSIFICATION TEST METHOD SUMMARY

NUMBER	TEST
404	Test Hot Bar Test

CATEGORY	APPLICABILITY	TYPE	PARAMETER(S)
Bulk	Trans. & Storage	Sensitivity	Heat

DESCRIPTION

A sample specimen is dropped on to a hot bar or hot plate of known temperature and then required for detonating, deflagration or marked decomposition is plotted on a time-temperature curve.

RATIONALE

This test determines the temperature at which the specimen will flash or detonate when held at that temperature for a specified length of time.

APPARATUS

Hot bar or hot plate
Balance accurate to .01 grams.

INSTRUMENTATION

Pyrometer
Thermocouple
Timer

SAMPLE SIZE	NUMBER OF TESTS FOR VALIDITY
20 mg.	Sufficient number of tests to validate time-temperature curve.

SPECIAL REQUIREMENTS

Sample must be screened through a No. 50 sieve, weighing of sample material. Temperature of the specimen sample must be stabilized at 25 $\pm$ 5 ^{o}C prior to test.

TESTING EXPERIENCE	RESULTS	REPORT
GREEN IV	None	---
VIOLET IV	None	---

PARAMETER	RELATABILITY +	QUANTATIVE +	SCALABILITY =	TOTAL
Heat	2	1	1	4

APPROXIMATE COST PER MATERIAL OR END ITEM TESTED

$185 per material — RANK 1

REMARKS

Test is a variation of the Wood's metal explosion temperature test. Data are obtained by a direct measurement of time and temperature. This test is a quick method for determining the magnitude of a reaction for an unknown material. This test is inadequate when compared to DTA or ITA.

CLASSIFICATION TEST METHOD SUMMARY

NUMBER	TEST
405	Impact Sensitivity Test (Bureau of Mines Apparatus)

CATEGORY	APPLICABILITY	TYPE	PARAMETER(S)
Bulk	Trans. & Storage	Sensitivity	Impact

DESCRIPTION

A 20 mg sample is subjected to impact from a falling weight of a predetermined height.

RATIONALE

This test determines the sensitivity of a pyrotechnic mixture by obtaining the minimum drop height at which one reaction occurs out of 10 trials from a known drop height as a result of mechanical shock caused by impact.

APPARATUS

Bureau of Mines Impact Apparatus

INSTRUMENTATION

None required. Strain gage, load cell, or Piezoelectric crystal to measure force of the impact and rebound of the falling weight is optional.

SAMPLE SIZE	NUMBER OF TESTS FOR VALIDITY
20 mg.	10 tests at each drop height.

SPECIAL REQUIREMENTS

Sieving of the sample through 50 mesh screen. Temperature of the sample stabilized to $25^{0} \pm 5$ ^{0}C prior to test. Weighing of sample to 20 mg sizes, identify sample material. Test must be performed under controlled temperature and humidity conditions.

TESTING EXPERIENCE	RESULTS	REPORT
GREEN IV	None	---
VIOLET IV	None	---

PARAMETER	RELATABILITY +	QUANTATIVE +	SCALABILITY =	TOTAL
Impact	3	2	1	6

APPROXIMATE COST PER MATERIAL OR END ITEM TESTED

$170 per material

RANK 1

REMARKS

This test established the minimum energy required to initiate a given material. Sample size too small for good statistical data. Data obtained with this apparatus do not correlate with those obtained on other test apparatus such as PA apparatus or Bureau of Explosives apparatus. Sample holder in the apparatus consist of a deformable cup and may be directly used without modification for liquid. Also greater drop heights are obtainable as compared to the B of E device; device requires a substantial concrete rest.

CLASSIFICATION TEST METHOD SUMMARY

NUMBER	TEST
406	Impact Sensitivity (Picatinny Arsenal Apparatus)

CATEGORY	APPLICABILITY	TYPE	PARAMETER(S)
Bulk	Trans. & Storage	Sensitivity	Impact

DESCRIPTION

A 10 - 30 mg sample is subjected to impact from a falling weight of a predetermined height.

RATIONALE

This test determines the sensitivity of a pyrotechnic mixture by obtaining the minimum drop height at which five of ten reactions occur in ten trials from a known drop height as the result of mechanical shock caused by impact.

APPARATUS

Picatinny Arsenal Impact Apparatus.

INSTRUMENTATION

None required. Strain gage, load cell, or Piezoelectric crystal to measure the force of the impact and rebound of the falling weight is optional.

SAMPLE SIZE	NUMBER OF TESTS FOR VALIDITY
Varies - the cup must be full & level	10 tests at each drop height.

SPECIAL REQUIREMENTS

Sieving of sample material through 50 mesh screen. Temperature of the sample material stabilized at $25^{o} \pm 5^{o}C$ prior to test. Weigh of sample material.

TESTING EXPERIENCE	RESULTS	REPORT
GREEN IV	None	---
VIOLET IV	None	---

PARAMETER	RELATABILITY +	QUANTATIVE +	SCALABILITY =	TOTAL
Impact	3	2	1	6

APPROXIMATE COST PER MATERIAL OR END ITEM TESTED

$170 per material

RANK 1

REMARKS

This test method differs from the BM and BE apparatus; friction is introduced due to the tapered sample cup. This test establishes the energy required for initiation at the 50% value. Because of the variable sample size test results do not correlate with those obtained on any other test apparatus.

CLASSIFICATION TEST METHOD SUMMARY

NUMBER	TEST		
407	Friction Pendulum Test		

CATEGORY	APPLICABILITY	TYPE	PARAMETER(S)
Bulk	Transportation	Sensitivity	Friction

DESCRIPTION

A sample of pyrotechnic mixture is exposed to the action of steel or fiber shoe swinging as a pendulum at the end of a long steel rod.

RATIONALE This test determines the sensitivity of a pyrotechnic mixture as a result of frictional forces upon the sample material. The behavior of the material is described quantitatively to indicate its reaction to this experience, i.e., the most energetic reaction is an explosion and in decreasing order of severity or reaction, snaps, cracks and unaffected. Simulate operation where personnel are walking over dust covered floor.

Apparatus

Friction pendulum test apparatus.

INSTRUMENTATION

None required.

SAMPLE SIZE	NUMBER OF TESTS FOR VALIDITY
7 gram	1 - 10 trials

SPECIAL REQUIREMENTS

Laboratory test conditions with controlled temperature and humidity. Sample preparation includes stabilizing sample temperature to 25 $\pm$ 5^{0}C sieving through 50 - 100 mesh sieve.

TESTING EXPERIENCE	RESULTS	REPORT
GREEN IV	None	---
VIOLET IV	None	---

PARAMETER	RELATABILITY +	QUANTATIVE +	SCALABILITY =	TOTAL
Friction	2	0	1	3

APPROXIMATE COST PER MATERIAL OR END ITEM TESTED

$320 per material — RANK 1

REMARKS

Data of tests performed on this apparatus are plentiful but do not correlate other type friction tests. Variances in data are obtainable because of the fineness of the sample material and wear of either the steel or fiber shoe. Friction stimuli do not represent a significant initiation hazard during transportation and storage.

CLASSIFICATION TEST METHOD SUMMARY

NUMBER
408

TEST
Friction Sensitivity Test

CATEGORY	APPLICABILITY	TYPE	PARAMETER(S)
Bulk	Transportation	Sensitivity	Friction

DESCRIPTION

A 100 mg sample is subject to friction between a sliding bar and a fixed preload. Impact energy supplied by a pendulum weight.

RATIONALE

This test determines the frictional energy required to cause decomposition or detonation of a material.

APPARATUS

Apparatus described in EA-FR-4D11

INSTRUMENTATION

Strain gage for measuring preload on sample and a linear velocity transducer to measure the velocity of the plate.

SAMPLE SIZE	NUMBER OF TESTS FOR VALIDITY
100 mg.	10 tests at each energy level.

SPECIAL REQUIREMENTS

Sample screened to microns. Sample dryed 24 hours at 75°C. Test performed under controlled temperature and humidity conditions.

TESTING EXPERIENCE	RESULTS	REPORT
GREEN IV	No Reaction	EA-FR-4D11
VIOLET IV	No Reaction	EA-FR-4D11

PARAMETER	RELATABILITY +	QUANTATIVE +	SCALABILITY =	TOTAL
Friction	2	2	1	5

APPROXIMATE COST PER MATERIAL OR END ITEM TESTED

$380 per test

RANK 1

REMARKS

Tests are designed to measure the frictional energy required to cause a decompomposition or explosive reaction. This apparatus is significantly different from other friction devices because of the preloading of static pressure. Friction testing must be regarded as being in a developmental state; this apparatus is unproven and of questionable validity at this time. Friction stimuli during transportation or storage does not represent a significant initiation hazard.

CLASSIFICATION TEST METHOD SUMMARY

NUMBER	TEST
409	Inpingement Reaction Test

CATEGORY	APPLICABILITY	TYPE	PARAMETER(S)
Bulk	Transportation	Sensitivity	Impact/Friction

DESCRIPTION

A 100 mg sample is subjected to impact and friction by pneumatic acceleration of the sample against a variable angle and variable material target.

RATIONALE

This test will determine the safe velocity range for pneumatic transport of pyrotechnic materials and the velocity range above which decomposition or detonation may occur from induce mechanical friction or impact.

APPARATUS

Apparatus described in EA-FR-4D11

INSTRUMENTATION

Optical sensors tied to counter for measuring velocity.

SAMPLE SIZE	NUMBER OF TESTS FOR VALIDITY
100-300 mg.	10 tests at each predetermined velocity. $N_1 \ldots Nn$

SPECIAL REQUIREMENTS

Sieving samples to 297 microns. Samples dryed 22 hours at 75°C. Test area environment maintained a 50-60% RH and temperature 70-75°F. Visual observation of impact in darkened room.

TESTING EXPERIENCE	RESULTS	REPORT
GREEN IV	No reaction at 300 ft/sec.	EA-FR-4D11
VIOLET IV	No reaction at 300 ft/sec.	EA-FR-4D11

PARAMETER	RELATABILITY +	QUANTATIVE +	SCALABILITY =	TOTAL
Impact/Friction	1	2	1	4

APPROXIMATE COST PER MATERIAL OR END ITEM TESTED

$380 per material RANK 1

REMARKS

To transport granular materials a certain critical velocity must be maintained. The velocity is based on the material density and other physical factors. This test will provide a measurement of the velocity below which no reaction occurs on impact and above which decomposition or detonation may result when the material impacts the receiving vessel or a change in direction of the delivery pipe. The validity of scaling pneumatic transfer processes is questionable to the point of being invalid. This test must be regarded as a qualitative indicator at best. This test is not relevant to transportation or storage.

CLASSIFICATION TEST METHOD SUMMARY

NUMBER	TEST
410	Abel Heat Test (KI Test)

CATEGORY	APPLICABILITY	TYPE	PARAMETER(S)
Bulk	Storage	Stability	Heat

DESCRIPTION

A small amount of pyrotechnic mixture is heated to $160^{o}F$ for 10 minutes. A determination is made by which the gases liberated will produce a standard coloration on a starch-iodide paper.

RATIONALE

A quick test method for demonstrating the absence from the pyrotechnic, propellant or explosive material of impurities causing low thermal stability.

APPARATUS

Test tubes
Oil bath
Starch-iodide paper strips

INSTRUMENTATION

Temperature measurement of oil bath.

SAMPLE SIZE	NUMBER OF TESTS FOR VALIDITY
1-10 grams	1 - 5

SPECIAL REQUIREMENTS

Pre-weigh sample, sample identification

TESTING EXPERIENCE	RESULTS	REPORT
GREEN IV	None	--
VIOLET IV	None	--

PARAMETER	RELATABILITY +	QUANTATIVE +	SCALABILITY =	TOTAL
Heat	4	1	2	7

APPROXIMATE COST PER MATERIAL OR END ITEM TESTED

$80 per material — RANK 1

REMARKS

This test is limited to those materials that are nitrated organic compounds and assures the completeness of the purification during manufacturing and the freedom from contamination after manufacturing which might cause low thermal stability. It is a good test to perform on a material which has failed to pass a thermal stability test. Nitrated organic compounds are infrequently found in pyrotechnic materials.

CLASSIFICATION TEST METHOD SUMMARY

NUMBER	TEST
411	Isothermal Analysis

CATEGORY	APPLICABILITY	TYPE	PARAMETER(S)
Bulk	Storage	Sensitivity	Temperature

DESCRIPTION

Determine effect on pyrotechnic materials at prolonged exposure to temperature conditions of near ignition temperatures.

RATIONALE

Thermal stability of sample at elevated temperatures approaching ignition temperature may be different than at normal elevated storage temperatures.

APPARATUS

Fischer Model 200 Differential Thermal Analyzer (DTA)
Isothermal temperature regulator for furnace

INSTRUMENTATION

Dual trace strip chart recorder.

SAMPLE SIZE	NUMBER OF TESTS FOR VALIDITY
50-100 mg	3

SPECIAL REQUIREMENTS

Operate DTA in non-programmed mode.

TESTING EXPERIENCE	RESULTS	REPORT
GREEN IV	Decomposed at 169.25°C	q.v. par. 3.1.6
VIOLET IV	Decomposed at 169.25°C	q.v. par. 3.1.6

PARAMETER	RELATABILITY +	QUANTATIVE +	SCALABILITY =	TOTAL
Temperature	3	2	3	8

APPROXIMATE COST PER MATERIAL OR END ITEM TESTED

$190 per material — RANK 1

REMARKS

This test offers questionable benefits in assessing pyrotechnic reactivity but is an improvement over standard thermal stability test in TB700-2. The rate of heating in this ITA test is not a factor in decerning a materials exothernal behavior as is in the DTA test, Number 112. The isothermal analysis technique cannot produce meaningful data when applied to a material that decomposes at a temperature below its ignition temperature.

CLASSIFICATION TEST METHOD SUMMARY

NUMBER

412

TEST

Hartmann Dust Sensitivity

CATEGORY

Bulk

APPLICABILITY

Transportation

TYPE

Sensitivity

PARAMETER(S)

Heat and Output Energy

DESCRIPTION

An airborne suspension of sample material is ignited in a Hartmann apparatus. Results are related to a standard material.

RATIONALE

Airborne suspensions may be present in a manufacturing environment. Their initiation sensitivities and output may constitute hazards.

APPARATUS

Bureau of Mines Hartmann apparatus and associated equipment.
Ignition sources

INSTRUMENTATION

Dynamic pressure recording

SAMPLE SIZE

1-2 grams

NUMBER OF TESTS FOR VALIDITY

Approximately 80

SPECIAL REQUIREMENTS

Identify sample

TESTING EXPERIENCE

TESTING EXPERIENCE	RESULTS		REPORT
GREEN IV	Explosibility Index	.1 (Weak)	EA-FR-1DOX
VIOLET IV	Explosibility Index	.1 (Weak)	EA-FR-1DOx

PARAMETER	RELATABILITY +	QUANTATIVE +	SCALABILITY =	TOTAL
Heat	2	2	2	6

APPROXIMATE COST PER MATERIAL OR END ITEM TESTED

$390 per material

RANK 1

REMARKS

Good test for fuel components, but pyrotechnic mix particles tend to be too dense to make and maintain uniform suspensions. Conventional Hartmann apparatus cannot achieve concentrations and conditions typical of pneumatic transfer process. This test is not representative of transportation or storage environmental conditions.

CLASSIFICATION TEST METHOD SUMMARY

NUMBER	TEST
413	Large Scale Parr Bomb--38 cu.ft. Detonation Chamber

CATEGORY	APPLICABILITY	TYPE	PARAMETER(S)
Bulk	Trans. & Storage	Output	Pressure Temperature

DESCRIPTION

A sample of material is initiated in a vessel of large volume (38 cu. ft.). Pressure and temperature are recorded to reflect energy output.

RATIONALE

Rapid pressure and temperature rises can be measured before significant heat can be rejected by the vessel. Large samples are more

APPARATUS

Apparatus described in EA-FR-4D11

INSTRUMENTATION

Temperature and pressure recorders

SAMPLE SIZE	NUMBER OF TESTS FOR VALIDITY
20-50 grams	3

SPECIAL REQUIREMENTS

Identify sample
Sample must react rapidly

TESTING EXPERIENCE	RESULTS	REPORT
GREEN IV	1.5 psig	EA-FR-4D11
VIOLET IV	2.2 psig	EA-FR-4D11

PARAMETER	RELATABILITY +	QUANTATIVE +	SCALABILITY =	TOTAL
Output temperature and pressure	2	2	2	6

APPROXIMATE COST PER MATERIAL OR END ITEM TESTED

$380 per material RANK 1

REMARKS

Pyrotechnics, in general, and smoke mixes, in particular, react rather too slowly to obtain good results. Vessel affords a good closed chamber for general testing such as functioning and end item and sampling products for analysis.

CLASSIFICATION TEST METHOD SUMMARY

NUMBER	TEST
414	Carrier Medium Test

CATEGORY	APPLICABILITY	TYPE	PARAMETER(S)
Misc	Manufacturing	Property	Triboelectrification

DESCRIPTION

Test conducted in blender to determine the electrostatic potentials that result from triboelectrification between pyrotechnic components and the atmosphere within the blender.

RATIONALE

It is possible that some carrier media may be more or less susceptible than to electrostatic charge generation. Hazards can be reduced by the selection of less active media.

APPARATUS

Modified 100 cubic inch capacity Jet Airmix blender and associated pneumatic supplies and controls.

INSTRUMENTATION

Electrometer

SAMPLE SIZE	NUMBER OF TESTS FOR VALIDITY
400 gm	3

SPECIAL REQUIREMENTS

Temperature and humidity control at laboratory environment
Identify sample

TESTING EXPERIENCE	RESULTS	REPORT
GREEN IV	None	---
VIOLET IV	None	---

PARAMETER	RELATABILITY +	QUANTATIVE +	SCALABILITY =	TOTAL
Triboelectrification	3	2	2	7

APPROXIMATE COST PER MATERIAL OR END ITEM TESTED

$240 per material — RANK 1

REMARKS

This is a very useful method for obtaining basic triboelectrification data. It should be mandatory whenever a carrier medium other than air is contemplated for pneumatic processing.

CLASSIFICATION TEST METHOD SUMMARY

NUMBER	TEST
415	Charging and Blending Sequence Test For Full Scale Processing

CATEGORY	APPLICABILITY	TYPE	PARAMETER(S)
Misc	Manufacturing	Property	Triboelectrification

DESCRIPTION

Components of a pyrotechnic mixture are cycled in a blender individually and in combination. Electrostatic charge generation is measured to determine relative triboelectrification effects as a function of blending sequence.

RATIONALE

Components should be mixed in sequences that result in the minimum generation of electrostatic potentials to reduce attendant hazards of discharge.

APPARATUS

Modified 100 cubic inch capacity Jet Airmix blender and associated pneumatic supply and controls.

INSTRUMENTATION

Electrometer
Thermocouple

SAMPLE SIZE	NUMBER OF TESTS FOR VALIDITY
100-500 grams	3

SPECIAL REQUIREMENTS

Temperature and humidity control at laboratory environment
Identify sample

TESTING EXPERIENCE	RESULTS	REPORT
GREEN IV	None	---
VIOLET IV	None	---

PARAMETER	RELATABILITY +	QUANTATIVE +	SCALABILITY =	TOTAL
Triboelectrification	3	2	3	8

APPROXIMATE COST PER MATERIAL OR END ITEM TESTED

$240 per material

RANK 1

REMARKS

This is a most useful test method for obtaining meaningful triboelectrification data. This test method is virtually mandatory prior to actual or test full scale blending to prevent electrostatic discharge related accidents. A project is currently underway to perform this test on Green Smoke Mix IV and Violet Smoke Mix IV; results will be published in EA-FR-4D91.

CLASSIFICATION TEST METHOD SUMMARY

NUMBER	TEST
416	Mass Effects Test

CATEGORY	APPLICABILITY	TYPE	PARAMETER(S)
Bulk	Trans. & Storage	Output Sensitivity	Hydrodynamic Shock and Transition

DESCRIPTION

A 36 inch diameter sample is subjected to a plane shock wave to determine whether the material is sensitive to shock initiation and transition to detonation in large masses and diameters.

RATIONALE

Pyrotechnics are generally insensitive at small diameters; it is possible that large masses and diameters are sensitive. This test demonstrates the hazard (or lack thereof) explosive characteristics at full scale.

APPARATUS

Shock place generator
Sample holder and support

INSTRUMENTATION

Blast instrumentation
Velocity probes

SAMPLE SIZE	NUMBER OF TESTS FOR VALIDITY
500 pounds	3

SPECIAL REQUIREMENTS

Consolidation of sample to simulate a full vessel.
Identify sample.

TESTING EXPERIENCE	RESULTS	REPORT
GREEN IV	None	See Remarks
VIOLET IV	None	See Remarks

PARAMETER	RELATABILITY +	QUANTATIVE +	SCALABILITY =	TOTAL
Hydrodynamic shock sensitivity and transition	2	2	3	7

APPROXIMATE COST PER MATERIAL OR END ITEM TESTED

$720 per sample

RANK 0

REMARKS

This test, since it is performed at full scale, inspires the highest possible confidence. This test at once provides determinations of whether the material is sensitive to hydrodynamic shock, whether the material has a critical diameter at 36 inches or less and equivalency based on a larg mass. A project is currently underway to perform this test on Green Smoke Mix IV and Violet Smoke Mix IV; results will be published in EA-FR-4D91.

CLASSIFICATION TEST METHOD SUMMARY

NUMBER	TEST		
417	Thermal Ignition Test		
CATEGORY	**APPLICABILITY**	**TYPE**	**PARAMETER(S)**
Bulk	Trans. & Storage	Output Sensitivity	Energy (Airblast) Transition

DESCRIPTION

Full scale quantity of material is stimulated by an ignitor to determine whether the reaction will communicate and transition to detonation and whether the energy output is sufficient to rupture the containment vessel.

RATIONALE

This test is intended to demonstrate the hazards attendent to initiation of full scale quantities regardless of initiation source.

APPARATUS

Full scale containment vessel.

INSTRUMENTATION

Blast measurement system
Heat Flux

SAMPLE SIZE	NUMBER OF TESTS FOR VALIDITY
100-2000 pounds	1

SPECIAL REQUIREMENTS

Identify sample material

TESTING EXPERIENCE	RESULTS	REPORT
GREEN IV	None	See Remarks
VIOLET IV	None	See Remarks

PARAMETER	RELATABILITY	+ QUANTATIVE	+ SCALABILITY	= TOTAL
Transition and energy output	3	2	3	8

APPROXIMATE COST PER MATERIAL OR END ITEM TESTED

$920 per sample

RANK 0

REMARKS

This test is performed on full-scale thus inspiring the highest possible confidence in its results. The utility of the test would be greatly enhanced by the addition of thermal output measurements. A project is currently underway to perform this test on Green Smoke Mix IV and Violet Smoke Mix IV; results will be published in EA-FR-4D91.

CLASSIFICATION TEST METHOD SUMMARY

NUMBER	TEST
418	Full-Scale Blending Test

CATEGORY	APPLICABILITY	TYPE	PARAMETER(S)
Bulk	Manufacturing	Property	Triboelectrification

DESCRIPTION

The test consists of full-scale operation of blending equipment.

RATIONALE

It is desired to determine the hazards associated with full scale blending of a material with respect to the generation of electrostatic charges.

APPARATUS

Full scale blending equipment or simulator.

INSTRUMENTATION

Electrometer

SAMPLE SIZE	NUMBER OF TESTS FOR VALIDITY
100-2000 pounds	2

SPECIAL REQUIREMENTS

Identify sample material

TESTING EXPERIENCE	RESULTS	REPORT
GREEN IV	None	---
VIOLET IV	None	---

PARAMETER	RELATABILITY +	QUANTATIVE +	SCALABILITY =	TOTAL
Triboelectrification	1	2	3	6

APPROXIMATE COST PER MATERIAL OR END ITEM TESTED

$920 per sample RANK 0

REMARKS

That this test is a full-scale test makes it unquestionably representative of actual conditions. The primary drawback with this test is the uncertainty of the electrostatic measurements obtained. The development of adequate electrostatic measuring devices and techniques is eagerly awaited. Meanwhile, this approach is the best available. A project is currently under way to perform this test on Green Smoke Mix IV and Violet Smoke Mix IV; results will be published in EA-FR-4D91.

CLASSIFICATION TEST METHOD SUMMARY

NUMBER	TEST
419	End Item Electrostatic Sensitivity

CATEGORY	APPLICABILITY	TYPE	PARAMETER(S)
End Item	Transportation	Sensitivity	Electrical Spark

DESCRIPTION

The end item is subjected to an electrical spark discharge of very high energy (50 joules) to determine whether that stimulus will result in functioning of the munition.

RATIONALE

The energy level used for this test is several orders of magnitude higher than expected in practice (except for a lightning strike). No function of the round in test implies no function in practice.

APPARATUS

High voltage power supply, capacitor bank and control circuitry.

INSTRUMENTATION

10,000 vdc voltmeter

SAMPLE SIZE	NUMBER OF TESTS FOR VALIDITY
9 end item rounds	9 (3 tests at each of three locations of the item)

SPECIAL REQUIREMENTS

Identify sample rounds

TESTING EXPERIENCE	RESULTS	REPORT
GREEN IV	None	---
VIOLET IV	None	---

PARAMETER	RELATABILITY +	QUANTATIVE +	SCALABILITY =	TOTAL
Electrical spark sensitivity	1	2	3	6

APPROXIMATE COST PER MATERIAL OR END ITEM TESTED

$100 per material — RANK 1

REMARKS

This is an indirect test, but is nevertheless a good test. This test is valid as long as the end item does not function. The magnitude of electrostatic sensitivity at an end item is of no concern if it is greater than 50 joules. An end item with an electrostatic sensitivity below 50 joules would require an altered procedure and more critical evaluation.

CLASSIFICATION TEST METHOD SUMMARY

NUMBER	TEST
420	Transportation Simulation Test

CATEGORY	APPLICABILITY	TYPE	PARAMETER(S)
End Item	Transportation	Output	Propalation

DESCRIPTION

A number of end items containers are placed in a scaled-down chamber simulating the carrier with respect to burst pressure and loading density. A single item is functioned normally or by a J-2 blasting cap.

RATIONALE

This test demonstrates whether the functioning of a donor round will propagate or transition to detonation under conditions of confinement found in a typical carrier.

APPARATUS

Simulated carrier

INSTRUMENTATION

Internal pressure and temperature airblast instrumentation
Motion picture

SAMPLE SIZE	NUMBER OF TESTS FOR VALIDITY
Approx. 8 cu. ft.	2

SPECIAL REQUIREMENTS

Identify sample rounds

TESTING EXPERIENCE	RESULTS	REPORT
GREEN IV	None	---
VIOLET IV	None	---

PARAMETER	RELATABILITY +	QUANTATIVE +	SCALABILITY =	TOTAL
Propagation	3	2	2	7

APPROXIMATE COST PER MATERIAL OR END ITEM TESTED

$420 per item RANK 1

REMARKS

This test serves to evaluate the effects of confinement by a carrier. The test is in a developmental stage, and has not adequately been evaluated and validated. Results, to date are inconclusive. Standardized procedures and carrier performance parameters should be developed to provide development of this test method.

CLASSIFICATION TEST METHOD SUMMARY

NUMBER	TEST
421	Modified Detonation Test B

CATEGORY	APPLICABILITY	TYPE	PARAMETER(S)
End Item	Trans. & Storage	Output	Propagation

DESCRIPTION

This test is conducted on pyrotechnic end items which are packaged in experimental or standard storage and shipping containers. The test is performed on three or more end item containers placed adjacent to each other.

RATIONALE

This test determines whether functioning of a donor round in a donor container propagates to an acceptor container. The modification consists of arranging the acceptor containers so that they are exposed to the maximum output of the donor.

APPARATUS

Open field test employing three or more loaded containers. Device for initiating normal function of donor end item or J-2 blasting cap.

INSTRUMENTATION

Blast measurement
Heat Flux
Documentary still and motion picture

SAMPLE SIZE	NUMBER OF TESTS FOR VALIDITY
Approx. 6 cu. ft. depending on pkg. size	Five or until propagation occurs.

SPECIAL REQUIREMENTS

Sample identificatio
Container rupture in previous A or B test.

TESTING EXPERIENCE	RESULTS	REPORT
GREEN IV	None	---
VIOLET IV	None	---

PARAMETER	RELATABILITY +	QUANTATIVE +	SCALABILITY =	TOTAL
Propagation	3	2	3	8

APPROXIMATE COST PER MATERIAL OR END ITEM TESTED

$300 per item

RANK 1

REMARKS

This test is conducted in conjunction with a standard detonation "B" Test since it represents a more realistic approximation of the conditions of shipping and storage. This test is conducted to determine if the functioning of the acceptor item in one container would cause functioning of items in one or more adjacent containers due to additional confinement and varied configurations. This test is performed if the standard detonation Test "A" or Test "B" resulted in no communication within the container, and the

(Over)

REMARKS (Cont'd)

outside container was ruptured. The item in the donor container is primed and initiated by its own fuse or by an Engineer's Special J-2 blasting cap. The item primed in the donor container was the closest item to the explosive item in the acceptor container. This assured subjecting the acceptor explosives to maximum blast effects from donor material.

The test results of testing the M83A8 60mm Illuminating flare are shown below.

Test Item	Propagation	Blast Overpressure (x psig)	Fragmentation Mean No.	Fragmentation Max Distance in feet
Modified Detonation Test "B"	Yes	None	47	190
Propagation/Transition Test "B"	No	None	4	96

At the present time there are no criteria or accepted documentation that provide for the modified detonation test. Although similar tests have been performed and reported to the cognizant classifying agencies, it has not become a standard test. The test results clearly show that the reaction can be much more violent and total propagation can occur. The test configuration is more representative of those conditions found in shipping and storage. Repeated results would alter Q/D criteria, and the compatibility of the down grading from one classification to another would be offset. Additional testing with various end items and shipping containers should be conducted to validate the test and to guide the preparation of a standardized procedure.

APPENDIX B

TEST METHODS

FOR

PYROTECHNIC MATERIALS HAZARDS EVALUATION

by

A. Levine
D. Kone

CONTENTS

ALPHABETICAL INDEX OF TEST METHODS

NUMERICAL INDEX OF TEST METHODS

SECTION 1

INTRODUCTION

1. SCOPE

1.1 This document describes the general methods of sampling and testing pyrotechnic materials and end items.

2. REFERENCED DOCUMENTS

2.1 References are listed at the end of each individual test method.

SECTION 2

SAMPLING

1. SCOPE

1.1 This section specifies the procedures for sampling pyrotechnic materials and end items.

2. SAMPLING

2.1 Select the required test samples that are representative of a batch of pyrotechnic material or a lot of pyrotechnic end items.

2.2 Transfer the pyrotechnic materials to approved airtight containers and seal the containers immediately. Keep all containers and end items in a safe location at room temperature until ready for testing.

2.3 Label each pyrotechnic container with the following information:

a. Pyrotechnic designation

b. Lot number

c. Lot size

d. Manufacturer's name and plant designation

e. Date sampled

f. f. Date loaded

2.4 Before testing the pyrotechnic, inspect the sample container to see that it is not broken, unstoppered, or otherwise damaged. Also verify that it has been labeled correctly. Discard the contents of damaged or improperly labeled correctly.

2.5 Select samples as specified in the applicable test method.

SECTION 3

TEST METHODS

1. SCOPE

1.1 This section contains the examinations, tests and methods of analysis for pyrotechnic materials and end items as required for hazards evaluation.

1.2 Each test is considered as a separate method and is assigned an individual method number.

2. NUMBERING SYSTEM

2.1 Methods are arranged in three groups according to category of test. These groups are identified numerically by hundreds. Tests for pyrotechnic mixtures are in the 100 group, pyrotechnic end item tests are in the 200 group, and miscellaneous tests are in the 300 group.

2.2 Revision numbers are differentiated by the addition of tenths decimals to the test numbers. Revision numbers are assigned to basic numbers when changes are made in the method for clarification or to give additional details that will increase the reproducibility of the test results.

3. METHODS UNDER INVESTIGATION

3.1 The following methods are under investigation and are not included in the current edition of this document:

Dust Ignition Sensitivity (Hartman Apparatus)
Explosion Temperature
Ignition Temperature
Triboelectric Sensitivity
Small Arms Vulnerability
Photomicrographs

METHOD 101

THERMAL STABILITY (75° C OVEN METHOD)

1. SCOPE

1.1 This test is conducted to determine if a pyrotechnic mixture is stable at an elevated temperature and to evaluate potential hazards due to any explosion, ignition, or marked decomposition that may occur at this temperature.

2. SPECIMEN

2.1 This specimen shall consist of a 2 $\pm$ 1/4 inch cube formed from the pyrotechnic mixture to be tested.

3. MATERIALS

3.1 Materials required for this test are as follows:

(a) A ventilated, explosion proof oven with dual heat controls capable of maintaining a temperature of 75° $\pm$ 1° C for a period of 48 hours and equipped to continuously record the temperature.

(b) Three copper-constantan thermocouples connected to a temperature recorder.

(c) A balance accurate to 1 milligram (mg).

4. PROCEDURE

4.1 Weigh the 2-inch specimen cube to the nearest milligram and place it in the oven. Insert one thermocouple into the specimen and two thermocouples into the oven. Raise the oven temperature to 75° $\pm$ 1° C. Maintain the temperature for 48 hours. Cool the specimen to room temperature and weigh to the nearest milligram. Record any temperature changes in the specimen. Observe any evidence of explosion, ignition, or decomposition. Record any change in the weight of the specimen.

5. EVALUATION

5.1 This test was designed to determine the thermal stability of a pyrotechnic mixture or explosive. It is similar to the thermal stability test in TB 700-2 except for the addition of the thermocouples to record temperature changes in the specimen. The temperature changes indicate exothermix or endothermic reactions.

5.2 To date, the testing of pyrotechnic mixtures by this method has not produced any meaningful results. It is recommended that this test be replaced by Method 102, Thermal Stability (Tube Method).

6. REFERENCES

(a) TB 700-2

(b) MIL-STD-650

(c) MIL-STD-1234

(d) GE-MTSD-R-035

(e) GE-MTSD-R-059

(f) Henkin, OSRD Report No. 3401, 22 March 1944

(g) Rinkenbach and Clear, PATR 1401

(h) Taylor and Rinkenbach, Journal of Franklin Inst, 204, Sep 1927, 369.

(i) Tomlinson and Sheffeld, PATR 1740, Rev. 1.

(j) TM 9-1910

METHOD 102

THERMAL STABILITY (TUBE METHOD)

1. SCOPE

1.1 This test is conducted to determine if a pyrotechnic mixture is stable at an elevated temperature and to evaluate potential hazards due to any explosion, ignition, or marked decomposition that may occur at the elevated temperature.

2. SPECIMEN

2.1 The specimen shall consist of 5 grams of the pyrotechnic mixture to be tested. (If desired, the pyrotechnic mixture may be compressed to the same density as in the end item for testing under simulated end item conditions.)

3. MATERIALS

3.1 Materials required for this test are as follows:

(a) A stainless steel tube having an outside diameter of 3/8 inch, a wall thickness of 0.35 inch, and a length of 8 inches.

(b) Cotton.

(c) A heating tape of nichrome ribbon.

(d) Two chromel-alumel thermocouples connected to a temperature recorder.

(e) An asbestos insulating jacket.

(f) A temperature regulator for controlling the heating tape temperature.

(g) A balance accurate to 0.2 milligram (mg).

4. PROCEDURE

4.1 Weigh the specimen to the nearest 0.2 mg and place it in the stainless steel tube. Centrally locate one chromel-alumel thermocouple in the stainless steel tube. Plug both ends of the tube with loosely packed cotton. Locate the second thermocouple on the outside of the tube. Wrap the heating tape around the tube in a spiral, connect the temperature regulator to the heating tape, and cover the heating tape with the asbestos insulating jacket. (See figure 1.) Set the temperature regulator so that a temperature of $75° \pm 1°$ C is maintained within the tube. Maintain the temperture for 48 hours. Cool the specimen to room temperature and weigh to the nearest 0.2 mg. Record any temperature changes in the specimen. Observe any evidence of explosion, ignition, or decomposition. Record any change in the weight of the specimen.

5. EVALUATION

5.1 This test is recommended as the replacement for Method 101, Thermal Stability (75°C Oven Method) because it better approximates the storage conditions of pyrotechnic mixtures in munitions.

6. REFERENCES

(a) King and Lasseigne, Final Report TSA-20-72-5

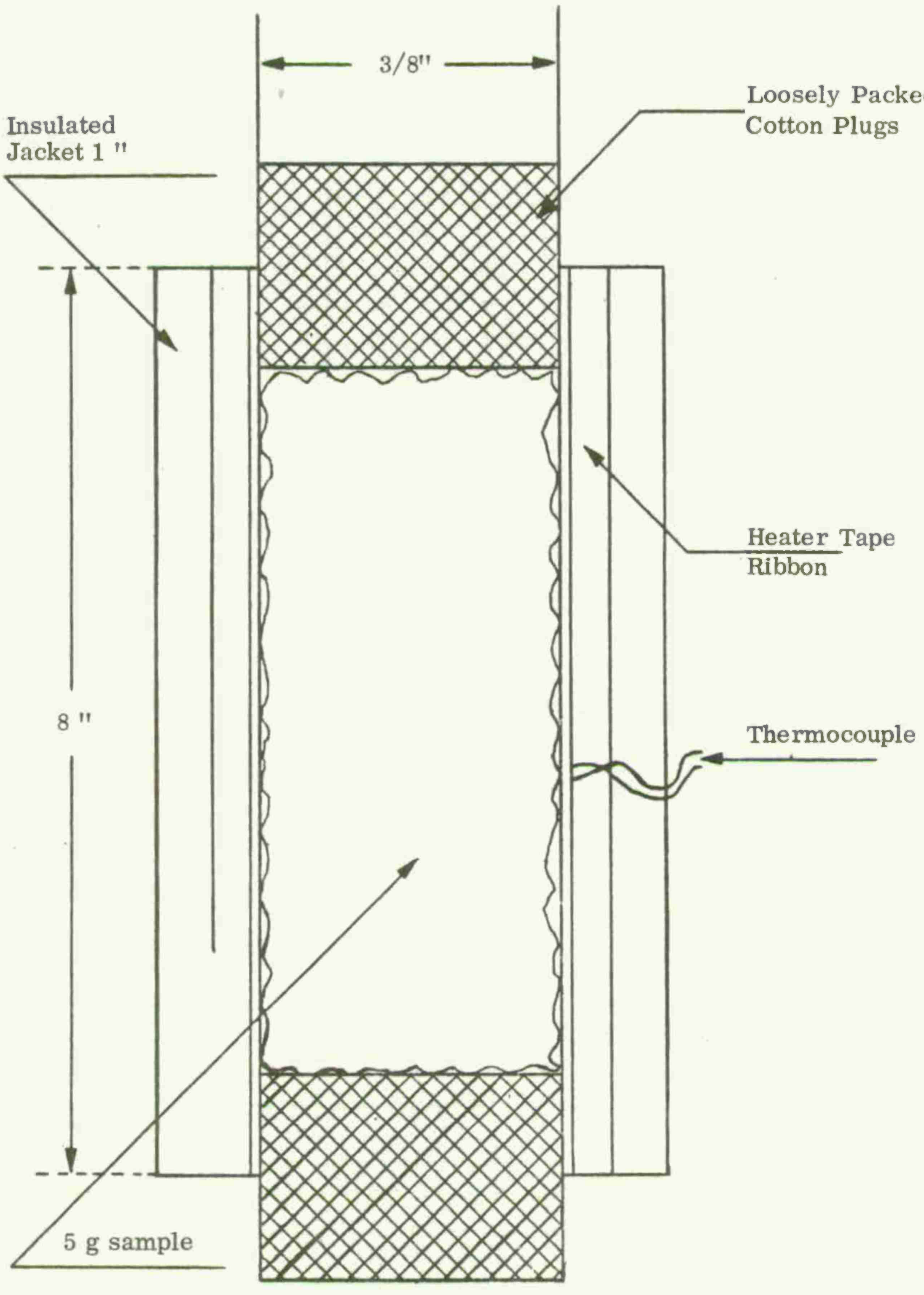


Figure 1. Bulk Thermal Stability Apparatus

METHOD 103

IGNITION AND UNCONFINED BURNING

1. SCOPE

1.1 This test was designed primarily to determine the probable gross fire hazards and transition to deflagration/detonation hazards of pyrotechnic mixtures.

2. SPECIMEN

2.1 The specimen shall consist of six 2 $\pm$ 1/4 inch cubes of the pyrotechnic mixture to be tested.

3. MATERIALS

3.1 Materials required for this test are as follows:

(a) A sheet metal tray 12 inches square by 1/4 inch deep.

(b) A quantity of kerosene-soaked sawdust.

(c) An electric match head igniter.

4. PROCEDURE

4.1 Place the sheet metal tray on a suitable fireproof surface and fill it with kerosene-soaked sawdust. Place a 2-inch specimen cube in the center of the tray. Place an electric match head igniter in the sawdust bed (see figure 1). Using approved safety operating procedures, ignite the sawdust with the electric match head igniter.

4.2 Repeat the procedure in 4.1 with another 2-inch specimen cube and then repeat it again with four 2-inch specimen cubes placed end-to-end in a single row in contact with each other (see figure 2). Observe any evidence of transition from burning to deflagration/detonation. Record the burning time.

5. EVALUATION

5.1 Explicit specifications should be written for the kerosene and sawdust materials. The variability in types of sawdust and kerosene available is too great and may cause the properties of the fire to which the specimen is subject to vary.

5.2 This method assumes that the material to be tested is a solid material that can be cut or machined into a 2-inch cube. Pyrotechnic mixtures are usually granular. A provision for granular materials should be made.

5.3 This test does not provide a definitive enough basis for determining burning rate. Moreover, the chance of detonation of the pyrotechnic mixture is extremely remote as tests have shown that these materials are not susceptible to a detonation reaction.

5.4 Based on evidence to date, it is concluded that this test does not adequately evaluate any potential hazards of pyrotechnic mixtures and therefore should not be used to test pyrotechnic mixtures.

6. REFERENCES

(a) TB 700-2

(b) GE-MTSD-R-035

(c) GE-MTSD-R-037

(d) Clear, PATR-FRL-TR 25

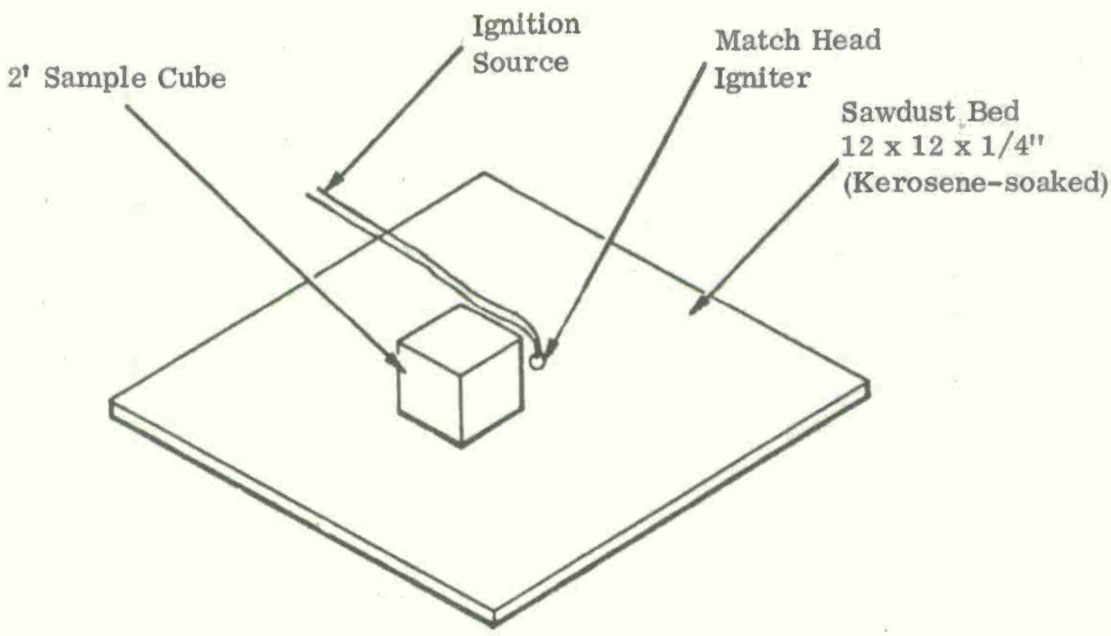


Figure 1. Test Configuration (One Specimen Cube)

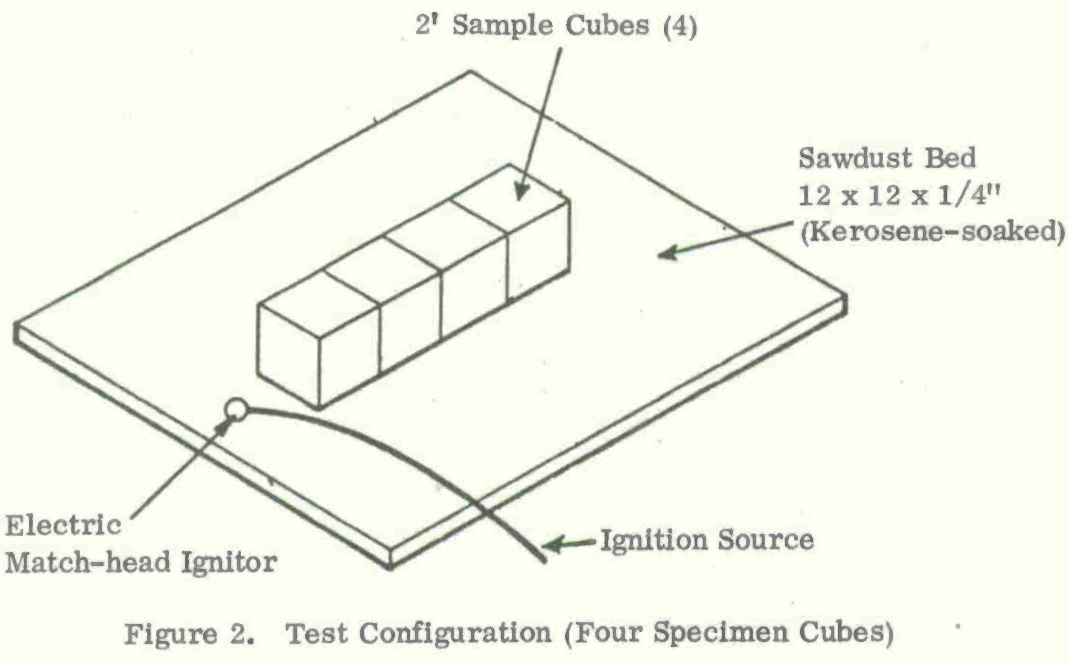


Figure 2. Test Configuration (Four Specimen Cubes)

METHOD 104

BURNING PROPAGATION RATE (SCREEN)

1. SCOPE

1.1 This test determines the burning propagation rate of pyrotechnic mixtures under unconfined conditions.

2. SPECIMEN

2.1 The specimen shall consist of the pyrotechnic mixture to be tested which has been prepared by sieving it through a 50-mesh screen prior to testing.

3. MATERIALS

3.1 Materials required for this test are as follows:

(a) A Hewlett Packard Model HP52336 electronic counter or equivalent

(b) Stainless steel screen, 100-mesh

(c) Ring stand support

(d) Wood and hardware for constructing frame for screen and propane burner support

(e) Ceramic stand-offs

(f) Propane torch

(g) Protective shield

(h) Lead fuse wire, 0.5 amp

(i) Electrical relay

4. PROCEDURE

4.1 Conduct the test in a well-ventilated hood. Set up a burning propagation rate apparatus as shown on figures 1 through 4 using the equipment described in 3.1. On the stainless steel screen, arrange a bed of the pyrotechnic mixture which is 11 inches long and which has a cross sectional area perpendicular to the length that is roughly triangular with a base width of 2 inches and a height of 1 inch. Place the lead fuse wires so that there is a distance of 5 $\pm$ 1/16 inch between them and so that there is a 3-inch length of the specimen bed from each fuse wire to its closest end of the bed. Catch any specimen that falls through the screen while the specimen bed is being prepared and return it to the top of the bed. Activate the electrical equipment and adjust the controls in accordance with the manufacturer's operating instructions. Turn on the exhaust hood. With the protective shield between the propane burner and the burning propagation rate apparatus, light the burner with a striker. Adjust the burner flame to a 2-inch pencil tip. Lower the hood door and apply the burner flame to the end of the specimen bed by means of the propane burner support taking care not to disturb the fuse wires. Record the time of burning between the fuse wires. Conduct five trials.

5. EVALUATION

5.1 This test provides a means of determining the burning propagation rate of a pyrotechnic mixture under unconfined conditions in inches per second. The results obtained from this test are more significant than the results obtained from the Ignition and Unconfined Burning test.

6. REFERENCES

(a) Clear, PATR-FRL-TR25

(b) King and Lasseigne, Final Report TSA 20-72-5

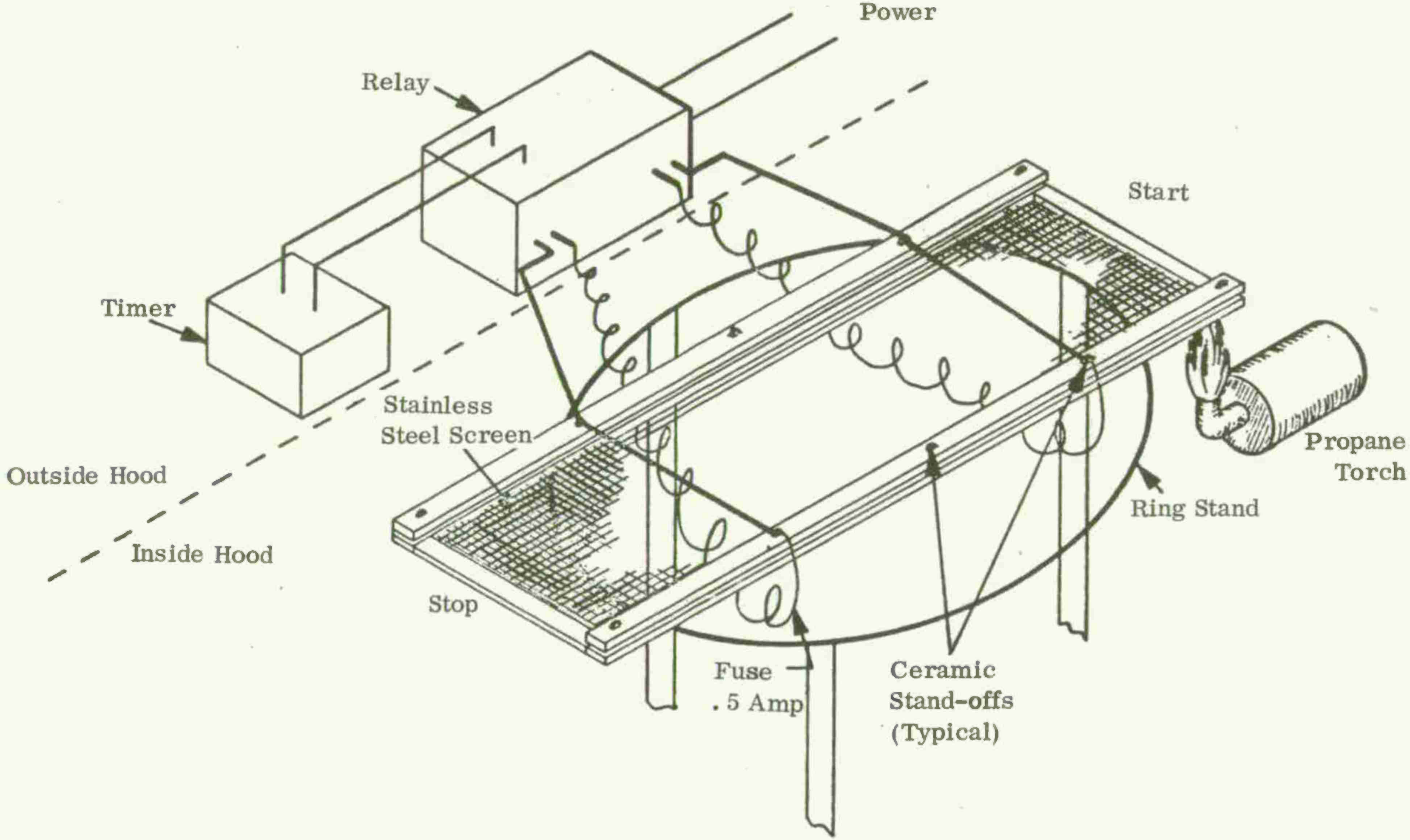


Figure 1. Burn Rate Apparatus

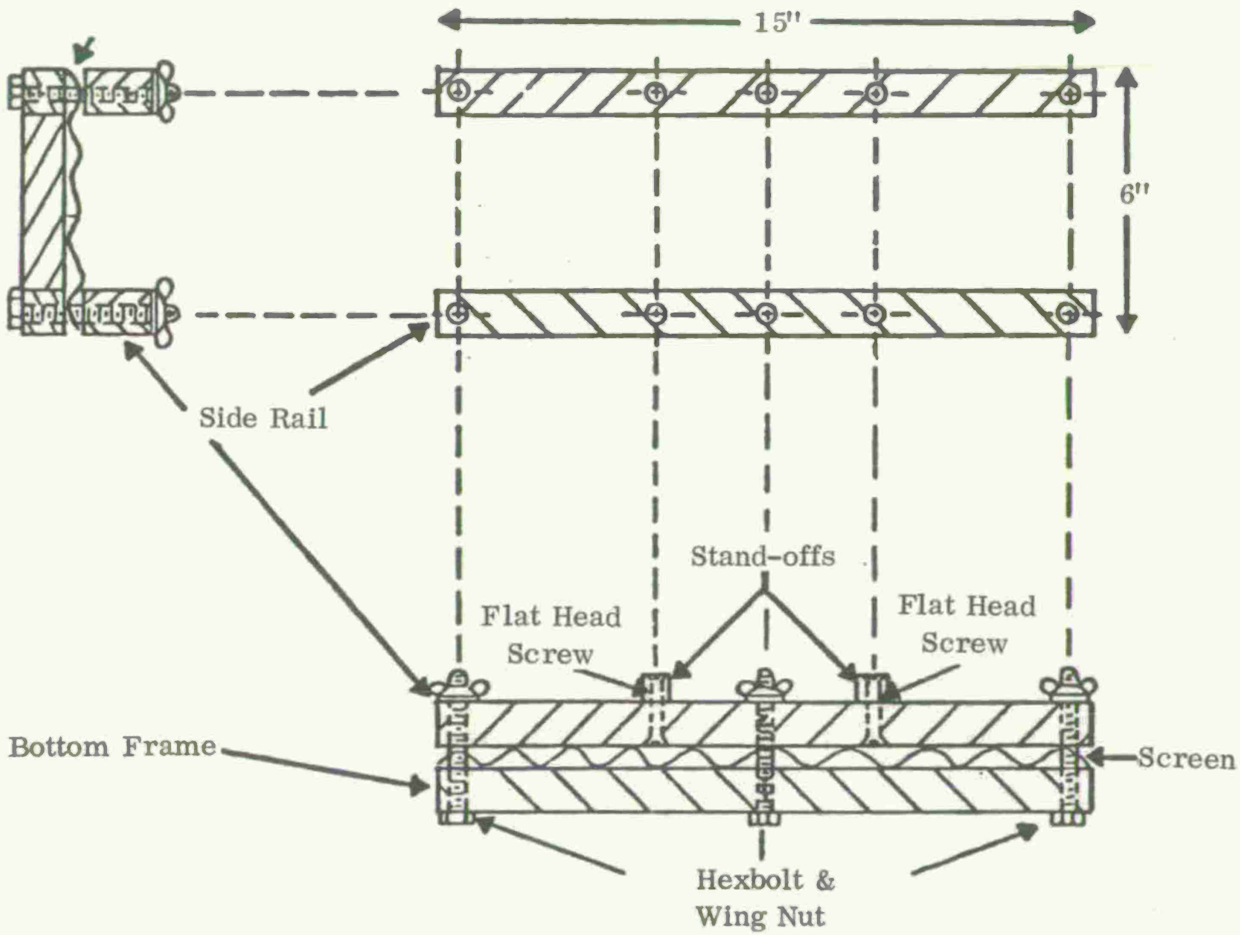


Figure 2. Burn Rate Apparatus - Bed

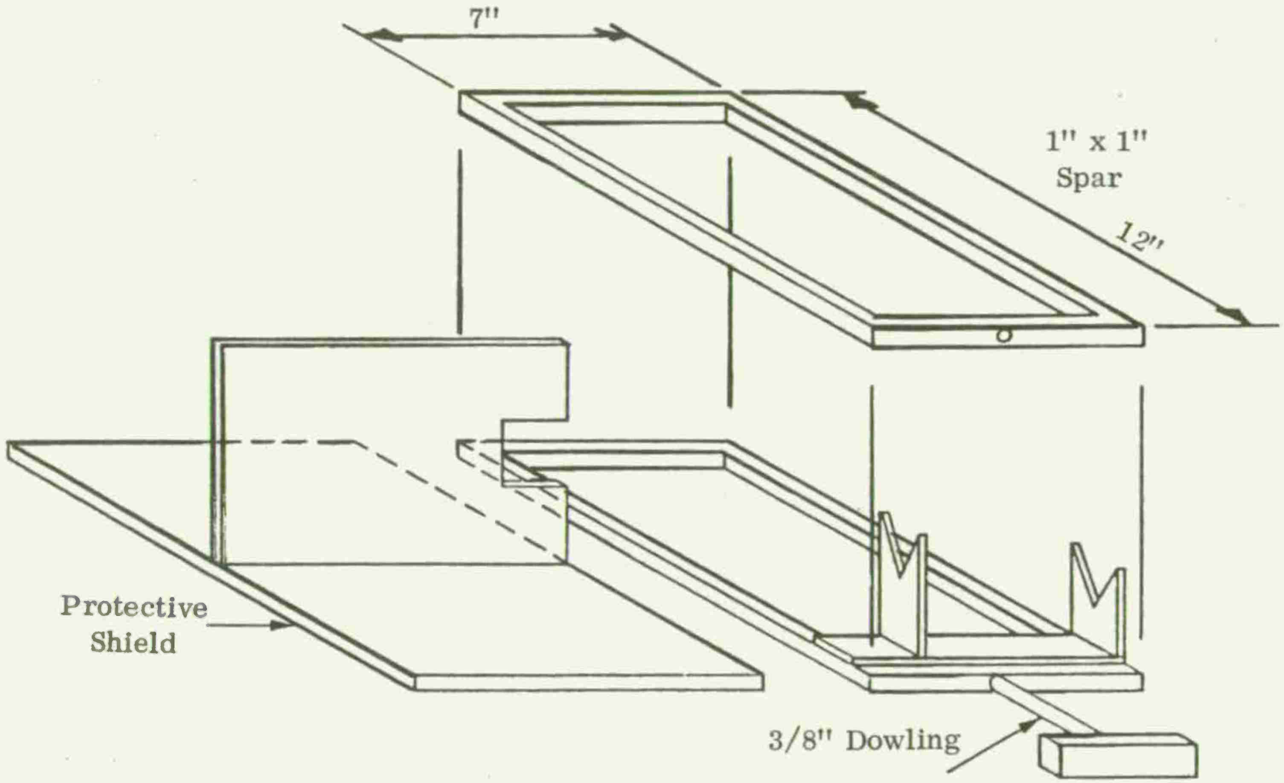


Figure 3. Propane Burner Support

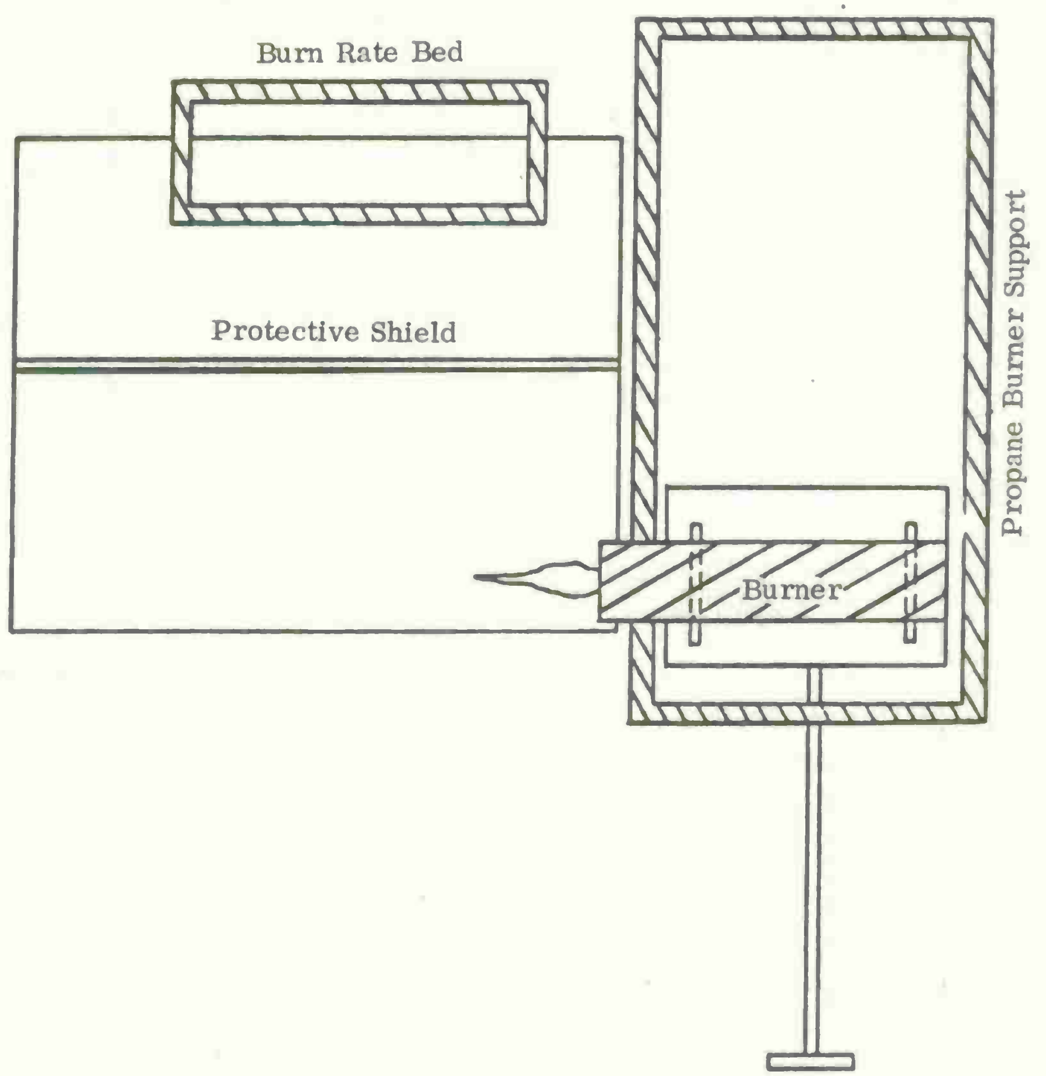


Figure 4. Burn Rate Test Set-up

METHOD 105

BURNING PROPAGATION RATE (TUBE)

1. SCOPE

1.1 This test determines the burning propagation rate of pyrotechnic mixtures under confined conditions.

2. SPECIMEN

2.1 The specimen for each trial shall consist of sufficient pyrotechnic mixture to fill a tube 12 inches long and approximately 1-2/3 inches in diameter. If desired the pyrotechnic mixture may be consolidated to the same density as in the end item in which it is used.

3. MATERIALS

3.1 Materials required for this test are as follows:

(a) A cold drawn seamless, 1015 composition, steel tube having a 1.875 inch outside diameter, a 0.219 $\pm$ 0.002 inch wall thickness, and a 12 inch length. The tube shall have two sawcuts perpendicular to the long axis as shown in figure 1.

(b) A Hewlett Packard Model HP52336 electronic counter and an electrical relay or equivalent equipment.

(c) Propane torch

(d) Protective shield

(e) Lead fuse wire, 0.5 amp

(f) Wood and hardware for constructing a propane burner support.

4. PROCEDURE

4.1 Conduct the test in a well-ventilated hood. Set up burning propagation rate apparatus by covering the saw cuts in the steel tube with tape and filling the tube with the pyrotechnic mixture specimen. Place the tube in a horizontal position with the saw cuts at the top, remove the tape and run separate lead fuse wires through each saw cut. Attach the electrical counting equipment as shown in figure 2, activate it, and adjust the controls in accordance with the manufacturer's operating instructions. Turn on the exhaust hood. With the protective shield between the propane burner and the tube, light the burner with a striker and adjust the burner flame to a 2-inch pencil tip. Lower the hood door and apply the burner flame to the specimen at the end of the tube which is 4 inches from a fuse wire by means of a propane burner support. Record the time of burning between the fuse wires. Conduct five trials.

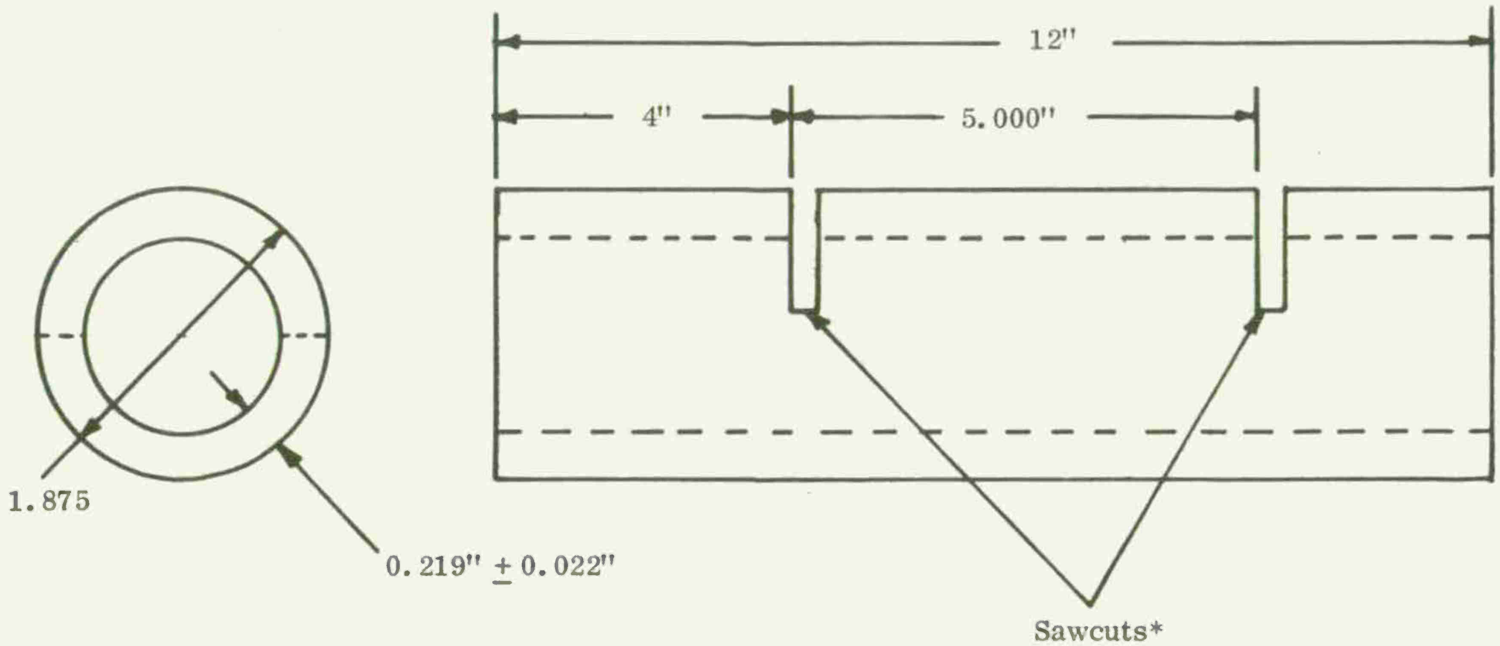
5. EVALUATION

5.1 This test is similar to the Burning Propagation Rate (Screen) test except that the pyrotechnic mixture is tested in a confined state. This simulates conditions that occur in pyrotechnic end items.

6. REFERENCES

(a) Clear, PATR-FRL-TR25

(b) King and Lasseigne, Final Report TSA 20-72-5



*Sawcuts will be covered with Texcel Tape prior to loading.

Material: Seamless steel tubing, 1015 cold drawn.

Tolerances

0.000 ± 0.010
0. ± 0.0625

Figure 1. Tube-Burning Rate Test

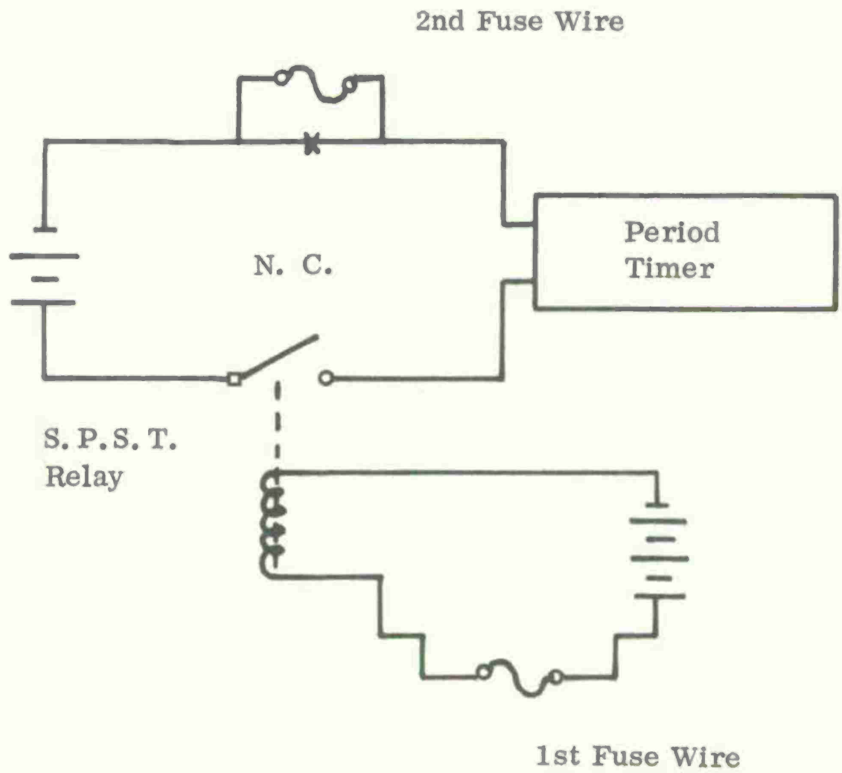


Figure 2. Wiring Diagram for Measuring Burn Time

METHOD 106

IMPACT SENSITIVITY

1. SCOPE

1.1 This test determines the probable sensitivity of a pyrotechnic mixture to decomposition or detonation as a result of mechanical shock caused by impact.

2. SPECIMEN

2.1 The specimen shall consist of 10 milligrams (mg) of the pyrotechnic mixture to be tested. The specimen shall be prepared by sieving it through a 50-mesh screen prior to testing. The temperature of the specimen at the time of test shall be 25° ± 5°C.

3. MATERIALS

3.1 Materials required for this test are as follows:

(a) A Bureau of Explosives impact apparatus. Drawings for the construction of the apparatus are available from the Bureau of Explosives, Association of American Railroads, 2 Penn Plaza, New York, NY 10001.

(b) Suitable cleaning materials and equipment for removing the decomposed residue from the cup and anvil of the impact apparatus.

4. PROCEDURE

4.1 Level the apparatus. Make sure that the cup and anvil are thoroughly clean and dry. Use a new cup if the old cup cannot be thoroughly cleaned. The apparatus should be at a temperature of 25° ± 5° C at the time of test. Place the 10-mg specimen in the test cup and place the impact apparatus. Using approved safety operating procedures, prepare the apparatus for testing and then initiate the test by releasing the weight. A typical test configuration is shown on figure 1.

4.2 Using the procedure in 4.1, conduct 10 trials with a drop height of 3-3/4 inches and 10 trials with a drop height of 10 inches. Use a new 10-mg specimen in a clean up for each trial. Observe any noise, smoke, flame, decomposed specimen in the cup, or lack of reaction.

5. EVALUATION

5.1 Instrumentation of the impact apparatus to determine dwell time, terminal velocity at impact, velocity of the falling weight, and force of impact should be considered. The use of an ionization probe to sense reactions should also be investigated.

5.2 The weight of specimen used should be optimized. The weight must be large enough to assure that a representative mixture is being tested. However, because sensitivity decreases with increased weight due to cushioning and heat sink effects, it must not be too large. The best weight probably will be in the 10 to 50 mg range.

5.3 The strike weight should also be considered. As sensitivity decreases, greater strike weights may be needed. The use of additional drop heights of 7 and 15 inches should be investigated.

5.4 Relative humidity conditions should be specified for the test.

6. REFERENCES

(a) TB 700-2

(b) MIL-STD-650, method 505.1

(c) MIL-STD-1234, method 505.1

(d) GE-MTSD-R-035

(e) GE-MTSD-R-056

(f) GE-MTSD-R-059

(g) AMCP 706-180

(h) Bureau of Mines Bulletin 346, p. 72

(i) Bureau of Mines Technical Paper 234

(j) Clear, PATR-FRL-TR-25 (1961)

(k) Downard, Fox, and Lawrence, ORSD 6627

(1) Eyster and Davis ORSD 5744

(m) Fox, ORSD 3185

(n) Fox, ORSD 3991

(o) Fox, ORSD 4962

(p) Rinkenbach and Clear, PATR 1401

(q) Taylor and Rinkenbach, Journal of the Franklin Institute <u>204</u> (1927), 369

(r) Tomlinson and Sheffeld, PATR 1740

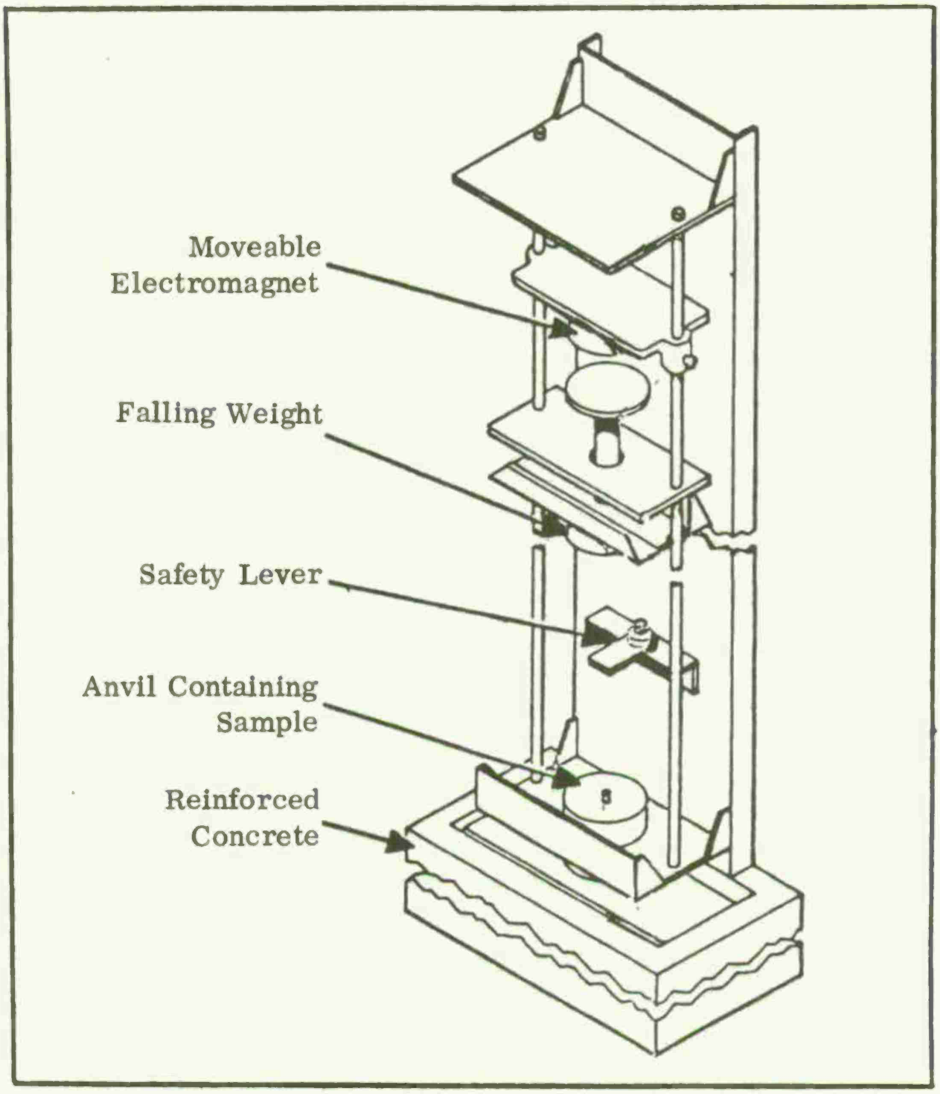


Figure 1. A Typical Test Configuration

METHOD 107

BULLET IMPACT - FRICTION

1. SCOPE

1.1 This test determines the sensitivity of bulk pyrotechnic mixtures to the combination of impact and friction produced by a small arms bullet in flight.

2. SPECIMEN

2.1 The specimen for each trial shall consist of sufficient pyrotechnic mixture to fill a pipe 3 inches long and 2 inches in diameter. If desired the pyrotechnic mixture may be consolidated to the same density as in the end item.

3. MATERIALS

3.1 Materials required for each trial are as follows:

(a) A piece of cast iron pipe 3 inches long having a 2-inch inside diameter and a 1/16-inch wall thickness and which is threaded at both ends.

(b) Two cast iron threaded caps for the ends of the pipe.

(c) A 0.30 caliber rifle and standard 0.30 caliber bullet.

(d) A balance accurate to 1 gram.

4. PROCEDURE

4.1 Screw one cap on the end of the pipe and weigh to the nearest gram. Fill the pipe with the specimen and reweigh. Screw on the second cap. Place the loaded pipe in a vertical position. Using approved safety operating procedures, fire the 0.30 caliber bullet through the pipe from a distance of 90 feet so that the bullet strikes between the two caps and perpendicular to the long axis of the pipe.

4.2 Conduct five trials. Record the weight of specimen used in each trial. Record the number of high order detonations, low order detonations and partial detonations (where the specimen burns), and the number of times there were no detonations.

5. EVALUATION

5.1 This test indicates potential hazards which may result from the sensitivity of a pyrotechnic mixture to a combination of friction and impact.

6. REFERENCES

(a) TM 9-1910

(b) Tomlinson and Sheffeld, PATR 1740.

(c) Eyster, E. H., Kistiakowsky, G. B., et al., "Sensitivity of Explosives to Projectile Impact," OSRD 3156, 1 January 1944.

(d) Eyster, E. H. and Rogers, W. H., "Physical Testing of Explosives, Part I, The Sensitivity of Explosives to Bullet Impact," OSRD 5745, 20 November 1945.

METHOD 108

ELECTRICAL SPARK SENSITIVITY

1. SCOPE

1.1 This test determines the sensitivity of pyrotechnic mixtures to ignition by electrostatic charge. The sensitivity is expressed in terms of the minimum energy in an electrical spark discharge which will ignite the pyrotechnic mixture.

2. SPECIMEN

2.1 The specimen shall consist of 10 to 15 milligrams of the pyrotechnic mixture to be tested. The specimen shall be prepared by sieving it through a 50-mesh screen prior to testing.

3. MATERIALS

3.1 Materials required for this test are as follows:

- (a) A Fluke Model 410B high voltage power supply or equivalent
- (b) Capacitors: 0.002, 0.01, 0.02, 0.05, 0.1 and 1 microfarad
- (c) Needle point voltage probe
- (d) Aluminum plate
- (e) A spark gap test fixture
- (f) A limiting resistor
- (g) Suitable switches for charging and discharging capacitor

4. PROCEDURE

4.1 Assemble the material described in 3.1 into the test configuration shown on figure 1. Initially, use the 1 microfarad capacitor. Connect the positive terminal of the condenser to the needle point voltage probe and the negative terminal to the aluminum plate. After verifying that the high voltage power supply is off, place the specimen in an even layer on the aluminum plate. Ground the specimen. Using approved safety operating procedures, turn on the high voltage power supply. Caution: Because of the high voltages present in the following procedure, use extreme caution to prevent accidental contact with points of high voltage. With all output voltage switches at zero, turn the high voltage power switch on.

With approximately five seconds between steps, advance the output voltage swithces to the test voltage. Record the final voltage on a data sheet similar to the on shown on figure 2.

Using the control knob, lower the spark gap probe toward the specimen until a spark occurs. Return the probe to this original position. Return the high voltage power supply output switches to zero. Observe the specimen for smoke, flame, or other evidence of ignition or for a lack of reaction and record the observation on the data sheet. Calculate the joules of energy to which the specimen was subjected as described on the data sheet.

4.2 In the first series of trials, use the 1 microfrared capacitor and the test voltages shown on figure 3. It is suggested that a test voltage of 1 kilovolt be used for the first trial. Conduct additional trials using the test voltages shown on figure 3 until at the lower of two consecutive test voltages no ignition is observed and at the higher test voltage evidence of ignition is observed. Then, using smaller capacitors and higher test voltages, determine as accurately as possible the amount of energy below which no ignition is observed and above which ignition is observed. Upon completion of all trials, turn the high voltage power supply off.

5. EVALUATION

5.1 Electrostatic charges may present potential hazards during the manufacturing, storage, and transportation of pyrotechnic mixtures because the energy involved may be great enough to cause ignition. This test provides information on the sensitivity of pyrotechnic mixture to the energy released by an electrostatic discharge.

6. REFERENCES

(a) GE-MTSD-R-057

(b) GE-MTSD-R-059

(c) TM9-1910

(d) Bureau of Mines Bulletin No. 346

(e) Tomlinson and Sheffeld, PATR 1740

(f) AMCP 706-177

(g) AMCP 706-186

(h) Bureau of Mines Report of Investigations 3852, (Brown, F. W. et al)

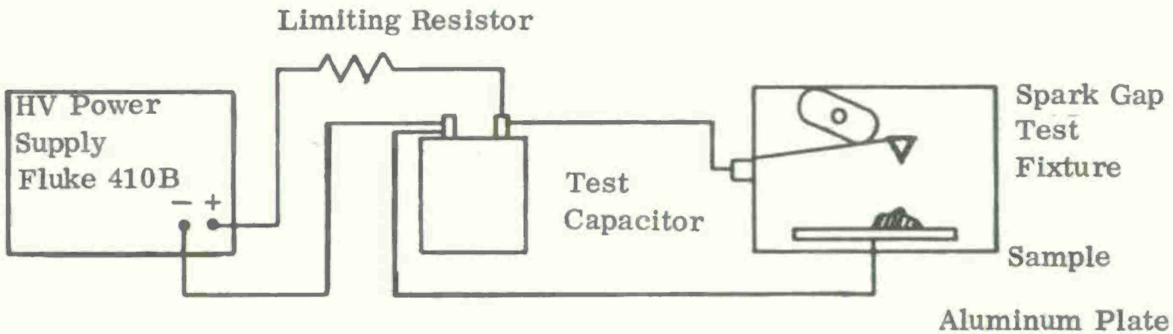


Figure 1. Electrostatic Ignition Susceptibility Test Setup

DATA SHEET

<table>
<tr><th rowspan="3">TEST NO.</th><th rowspan="3">TEST CONFIGURATION</th><th rowspan="3">VALUE OF CAPACITANCE (μF)</th><th rowspan="3">VOLTAGE LEVEL (VOLTS)</th><th rowspan="3">ENERGY $1/2\ CV^2$ (JOULES)</th><th>TEMPERATURE</th><th colspan="2">HUMIDITY</th><th>DATE</th></tr>
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<tr><th colspan="2">OBSERVATIONS</th><th colspan="2">COMMENTS</th></tr>
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</table>

Figure 2. Electrostatic Ignition Susceptibility Test Data Sheet

TRIAL #	VOLTAGE	ENERGY (JOULE)
1	10	.00005
2	100	.005
3	200	.02
4	300	.032
5	400	.08
6	500	.12
7	600	.18
8	700	.30
9	800	.32
10	900	.405
11	1KV	.5
12	2K	2.0
13	3KV	4.5
14	4K	8.0
15	5K	12.5
16	6K	18.0
17	7K	24.5
18	8K	32.0
19	9K	40.5
20	10K	50.0

Figure 3. Energy Discharge Values at One Microfarad Capacitance

METHOD 112

DIFFERENTIAL THERMAL ANALYSIS

1. SCOPE

1.1 This test determines ignition temperature and other physical and chemical reactions which may occur in a pyrotechnic mixture when the mixture is heated. The test measures the temperature difference between the pyrotechnic mixture and a thermally inert reference material as both are heated at a constant rate of increase in temperature.

2. SPECIMEN

2.1 The specimen shall consist of approximately 25 milligrams (mg) of the pyrotechnic mixture to be tested. The specimen shall be prepared by passing it through a 50-mesh screen prior to testing.

3. MATERIALS

3.1 Materials required for this test are as follows:

(a) A Fischer Series 200 differential thermal analyzer or similar equipment.

(b) A Varian Aerograph Model 20, dual-channel, strip-chart potentiometric recorder having a 1-millivolt full scale sensitivity on each channel or similar equipment.

(c) A thermally inert reference material such as quartz crystals, approximately 25 mg.

4. PROCEDURE

4.1 Using approved safety operating procedures and the manufacturer's operating instructions for the differential thermal analyzer and the recorder, obtain the thermogram of the specimen compared to the thermally inert reference material. The heating rate should be set at approximately 5°C per minute and the chart speed at approximately 10 inches per hour. Continue heating until the specimen is decomposed. A schematic diagram of the differential thermal analysis equipment is shown on figure 1.

5. EVALUATION

5.1 This test detects exothermic or endothermic changes that occur in the specimen while it is being heated. These changes may be related to dehydration, decomposition, crystalline transition, melting, boiling, vaporization, polymerization, oxidation, or reduction. The test is rapid and reasonably accurate. The results obtained provide significant information on the thermal stability and ignition sensitivity of the specimen.

5.2 The interpretation of the thermogram for determining where true ignition occurs needs further investigation. The first temperature differential associated with ignition usually represents an "on-set to ignition" characteristic which is not the true ignition temperature.

6. REFERENCES

(a) GE-MTSD-R-059

(b) EATR 4580

(c) Vold, Marjorie J., Analytic Chemistry, 21, 683 (1949).

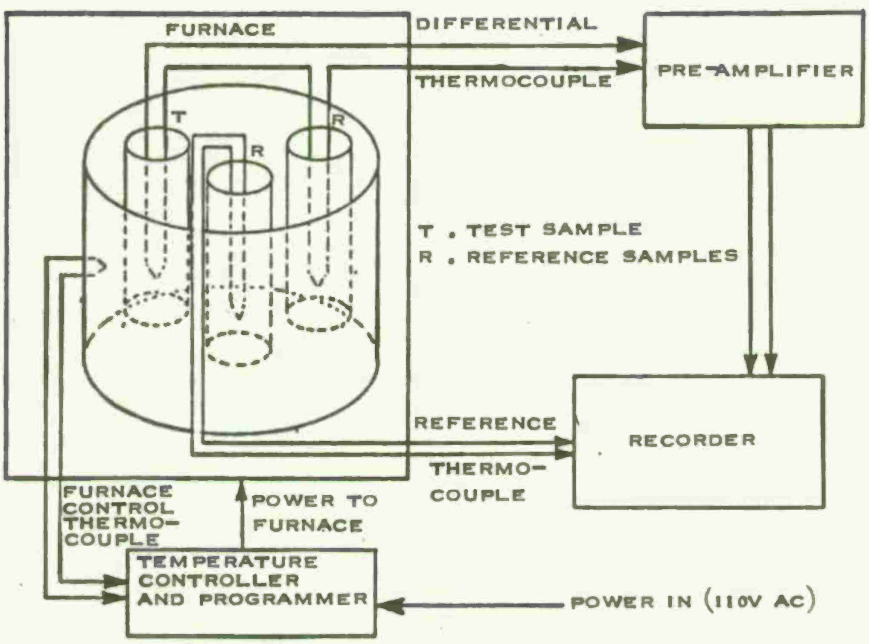


Figure 1. Differential Thermal Analyzer Diagram

METHOD 113

DETONATION - COMPRESSION

1. SCOPE

1.1 This test determines the probable sensitivity of pyrotechnic mixtures to detonation in free air.

2. SPECIMEN

2.1 The specimen shall consist of the pyrotechnic mixture to be tested in the form of a 2 $\pm$ 1/4 inch cube.

3. MATERIALS

3.1 Materials required for this test are as follows:

(a) A solid lead cylinder 1-1/2 inches in diameter by 4 inches long of known purity and hardness.

(b) A mild steel plate, SAE 1010 to 1030, 1/2 inch thick by 12 inches square.

(c) A number 8 blasting cap, a blasting machine, and connecting wire (blasting line).

(d) A block of wood 2 inches in diameter and as long as the blasting cap, drilled to receive the blasting cap through its vertical axis.

(e) A go-no-go gage for the 1-1/2 inch diameter lead block with a 1/16 inch tolerance.

4. PROCEDURE

4.1 Place the steel plate horizontally in a suitable facility. Place the lead cylinder on the center of the steel plate in a vertical standing position. Place the 2-inch specimen cube on top of the lead cylinder. Place the wood block containing the blasting cap on top of the specimen so that the cap is perpendicular to and in contact with the top surface of the specimen (see figure 1). Connect the blasting machine to the blasting cap and fire the blasting cap from a safe location using approved safety operating procedures.

4.2 Repeat the test on additional specimens until the detonation of a specimen occurs. Detonation is considered to have taken place if the lead cylinder is deformed (by mushrooming) so that the upper diameter has increased by at least 1/16 inch as indicated by the go-no-go gage. If no detonation of a specimen has occured after five trials, terminate the test.

5. EVALUATION

5.1 The test does not make provisions for granular specimens, nor for standard compression, tamping, or confinement of the material.

5.2 This test should not be used in the evaluation of potential hazards of pyrotechnic material since present evidence is that pyrotechnic mixtures are not susceptible to detonation in the unconfined state.

6. REFERENCES

(a) TB 700-2

(b) GE-MTSD-R-035

(c) GE-MTSD-R-059

(d) Monroe and Tiffany, B of M Bulletin 346.

(e) OSRD Report #1364

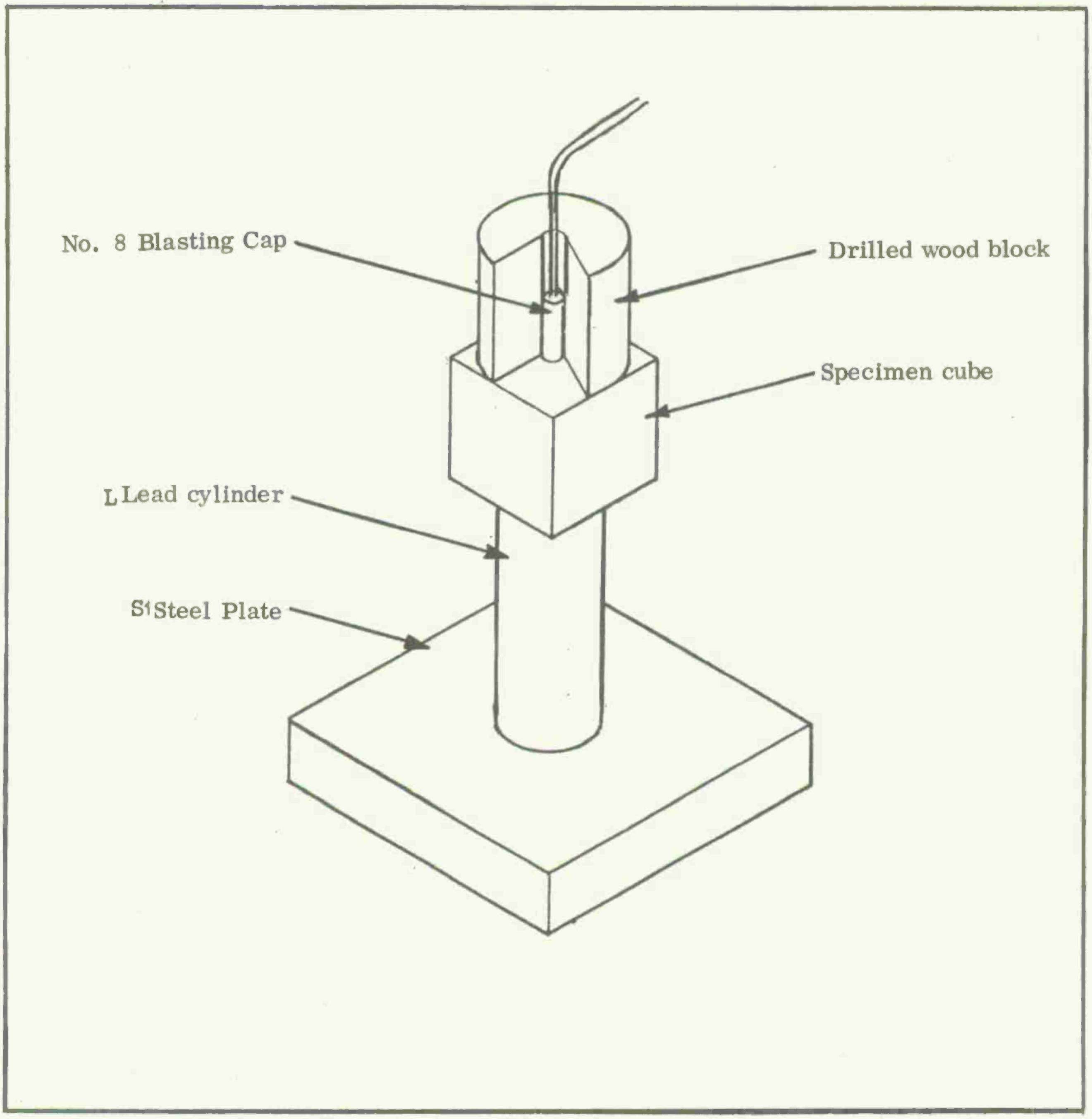


Figure 1. Detonation - Compression Test Configuration

METHOD 114

CARD GAP

1. SCOPE

1.1 This test determines the detonation sensitivity of pyrotechnic mixtures under the influence of an explosive shock wave.

2. SPECIMEN

2.1 The specimen for each trial shall consist of sufficient pyrotechnic mixture to fill a 1-7/8 inch diameter tube having a 5-1/2 inch length. The specimen shall be prepared by sieving it through a 50-mesh screen prior to testing.

3. MATERIALS

3.1 Materials required for each trial are as follows:

(a) A cold drawn seamless, 1015 composition, steel tube having a 1-7/8 inch outside diameter, a 0.219 $\pm$ 0.022 inch wall thickness, and a 5-1/2 inch length.

(b) A steel plate (witness plate), 6 inches square by 3/8 inch thick, made of SAE 1010 steel and having a Rockwell "B" hardness of 50 to 60 and a tensile strength of 60,000 to 65,000 pounds per square inch.

(c) An engineers special blasting cap, J-2.

(d) A blasting machine and wire.

(e) A block of wood 2 inches in diameter and as long as the blasting cap, drilled to receive the blasting cap through its major axis.

(f) Two pentolite pellets 2 inches in diameter by 1 inch long.

(g) A quantity of cellulose acetate cards 2 inches in diameter by 0.01 inch thick.

(h) Four pieces of plastic 1/16 inch thick by 1/2 inch square.

(i) Cardboard tube to hold materials in test configuration.

4. PROCEDURE

4.1 The temperatures of the specimen and the pentolite booster at the time of testing should be 25° $\pm$ 5°C. Place the witness plate in a horizontal position, supported along its edges, approximately 6 inches above the ground surface (see figure 1). Place the four pieces of plastic on the plate to support the tube containing the specimen in the center of the plate. Fill the steel tube with the specimen and place it on the plastic pieces so that there is a 1/16-inch space between the bottom of tube and the plate. The plastic pieces should not extend under the specimen. Place both pentolite pellets on top of the tube so that the bottom pellet it in contact with the specimen. Insert the blasting cap into the wooden block and place the block on top of the pentolite pellets so that the end of the blasting cap is in contact with the top pellet. Attach the blasting cap to the blasting machine and fire the blasting cap from a safe location using approved safety procedures. Detonation of the specimen is indicated by a clean hole cut through the witness plate. If detonation of the specimen does not occur, repeat the test procedures for two additional trials. If no detonation of the specimen occurs in the three trials, discontinue the test.

4.2 If detonation occurs in any of the first three trials, repeat the procedure in 4.1 adding cellulose acetate cards between the steel tube and the lower pentolite pellet as shown on figure 1. If detonation occurs, repeat the test using twice as many cards (16, 32, 64, 128, . . .) until detonation fails to occur. When detonation fails to occur, remove one-quarter of the number of cards for the next test. If detonation occurs or fails to occur, add or remove, respectively, one-eighth of the number of cards at which the first lack of detonation occured. Repeat this addition or removal of cards procedure by the factor of 1/16, 1/32, 1/64, 1/128, . . . until the number of cards is obtained at which the probability of detonation is 50 percent.

5. EVALUATION

5.1 The "go-no-go" characteristics of card gap warrants further examination with respect to its use as a means of determining degree of sensitivity. In this test, pyrotechnic mixtures do not detonate and cut a hole through the witness plate. As a result, the procedure using cellulose acetate cards is not employed. The pyrotechnic specimens will fall out of the steel tube unless they are compacted or unless the test is conducted in a 180° vertical orientation. It is recommended that the addition of instrumentation to measure overpressure and impulse be investigated in an effort to obtain more meaningful test results.

6. REFERENCES

(a) TB 700-2

(b) TM 9-1910

(c) GE-MTSD-R-035

(d) GE-MTSD-R-059

(e) Munroe and Tiffany, Bureau of Mines Bulletin No. 346

(f) AMCP 706-180

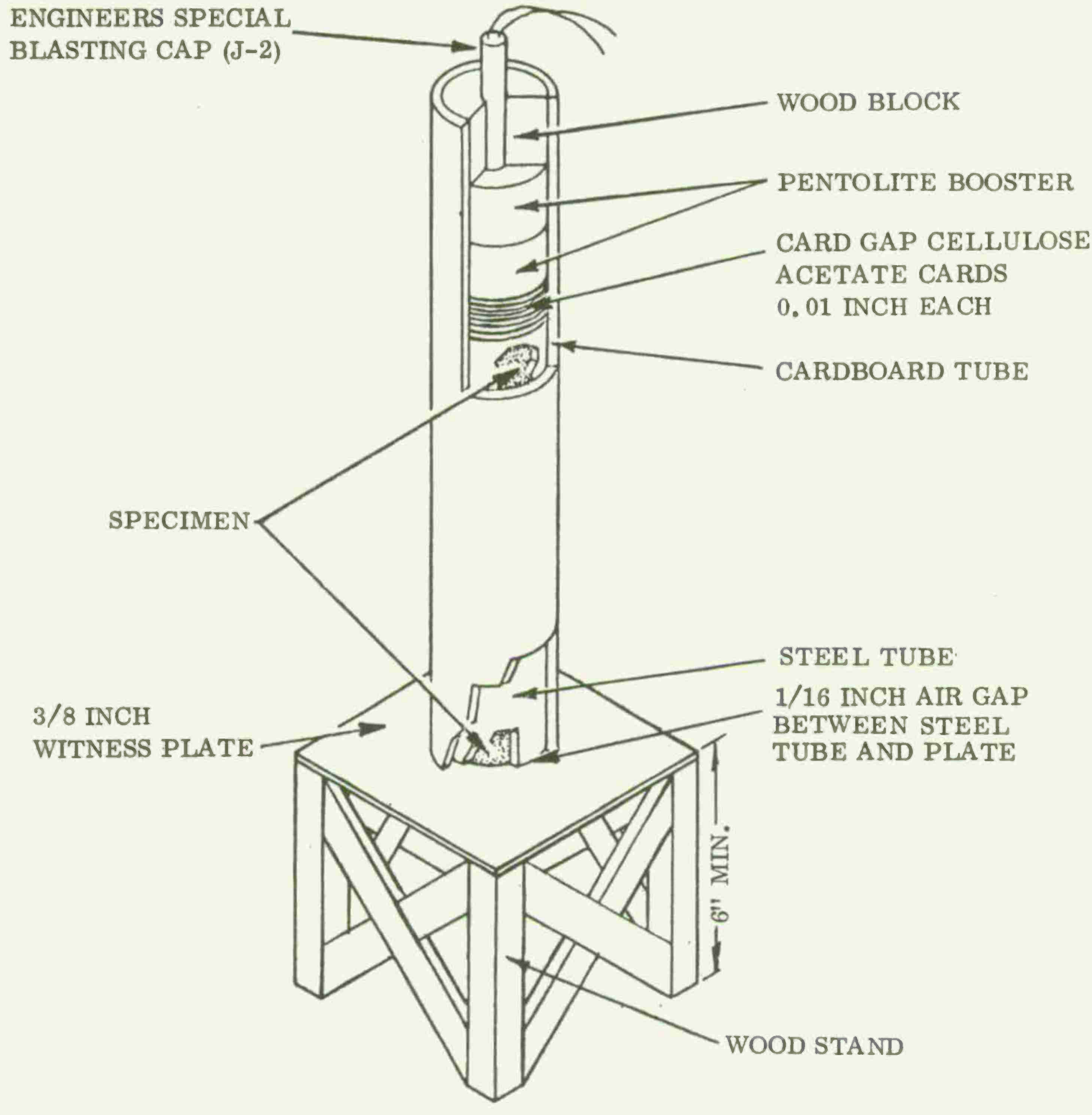


Figure 1. Card Gap Test Configuration

METHOD 115

HIGH EXPLOSIVE EQUIVALENCY

1. SCOPE

1.1 This test determines the ratio of the amount of energy released in a detonation reaction of a pyrotechnic mixture to the amount of energy released by a high explosive under the same conditions.

2. SPECIMEN

2.1 The specimen for each of five trials shall consist of 100 grams (g) of the pyrotechnic mixture to be tested. The specimen shall be prepared by sieving it through a 50-mesh screen prior to testing.

3. MATERIALS

3.1 Materials required for this test are as follows:

3.1.1 Explosive composition C-4, 100 g.

3.1.2 Seven high explosive equivalency test vessels. Each test vessel consists of the following materials:

(a) A cold drawn seamless, 1015 composition, steel tube having a 1-7/8 inch outside diameter, a 0.219 ± 0.022 inch wall thickness, and a 5-1/2 inch length. The tube is threaded at both ends to receive pipe caps.

(b) Two 3,000 psi forged steel pipe caps for the ends of the steel tube. One cap contains a hole to accept a blasting cap.

(c) A J-2 engineers special blasting cap and ignition wires.

(d) Epoxy resin for sealing the blasting cap in the pipe cap.

(e) Aluminum tape.

3.1.3 A test fixture for suspending the test vessel at a height of 9 feet above ground level.

3.1.4 A blast overpressure instrumentation system consisting of the following components:

(a) Eight blast overpressure transducers, Susquehanna Instruments, Model ST-7 or equivalent, mounted in aerodynamic probes.

(b) Eight in-line amplifiers, PCB Piezotronics, Inc. Model 402 or equivalent.

(c) Eight transient recorders, Biomation Model 610B or equivalent.

(d) Nine electronic counters, Hewlett-Packard Model 5233L or equivalent.

(e) One digital voltmeter, Hewlett-Packard Model 2501C or equivalent.

(f) One 12-channel "Dijitscan," available from Pievan Data Systems or equivalent recorder/memory interface.

(g) One X-Y plotter, Stromberg-Carlson 4020 or equivalent.

(h) One oscilloscope with camera pack, Tektronics Model 503 or equivalent.

4. PROCEDURE

4.1 Assemble the test vessel as shown on figure 1. Place the material to be tested in the vessel. Place the pipe cap (with a J-2 engineers special blasting cap preinstalled) on the threaded tube and tighten securely. Suspend the loaded vessel in the center of the instrumented test area at a height of 9 feet above ground level. Set up a blast overpressure instrumentation system as shown on figures 2 and 3 using the material described in 3.1.4. Aim the transducers at the test vessel and activate the instrumentation system. Initiate the test using approved safety operating procedures.

4.2 Conduct seven trials using the procedure described in 4.1. In the first and last trials use 100 g of explosive composition C-4 as the test material. In the second through the sixth trial use 100 to 200 g of the pyrotechnic mixture specimen as the test material.

5. CALCULATIONS

5.1 Calculate the high explosive equivalency of the pyrotechnic mixture in accordance with Appendix B of GE-MTSD-R-035. A computer program for processing the data is presented in GE-MTSD-R-070.

6. EVALUATION

6.1 This test determines maximum overpressure, function time, and positive impulse at eight scaled distances. These characteristics are used to provide a basis for comparing the "damage potential" of pyrotechnic mixtures under confined conditions to that of a standard high explosive (C-4). This approach is based on the assumption that the blast energy output of the pyrotechnic mixture results from detonation and therefore is comparable to a standard high explosive.

6.2 The test instrumentation used is a sophisticated version of the equipment required for blast pressure determination in chapter 5 of TB 700-2. It is based on the exploratory work of GE-MTSD at the Mississippi Test Facility of the National Aeronautics and Space

Administration. The high explosive equivalency test data which resulted from this work is not conclusive because of the following considerations:

(a) The weight of specimen varied from 50 to 125 g.

(b) The allowable void (in confinement) varied from 25 to 40 percent.

(c) The high explosive equivalencies based on maximum overpressure appeared to provide some useful information. However, they were not constant over the eight distances used.

(d) The high explosive equivalencies based on positive impulse were unsatisfactory.

6.3 It is therefore recommended that this test method be thoroughly investigated in order to define all test parameters. Modification of this test method will be necessary before it can be considered for inclusion in the revised version of TB 700-2.

7. REFERENCES

(a) GE-MTSD-R-035

(b) GE-MTSD-R-050

(c) GE-MTSD-R-059

(d) GE-MTSD-R-070

(e) GE-MTSD-FR-030

(f) TB 700-2

(g) TM 9-1910

(h) AMCP 706-180

(i) PATR 1740

(j) OSRD 1707

(k) BLR Report 1092

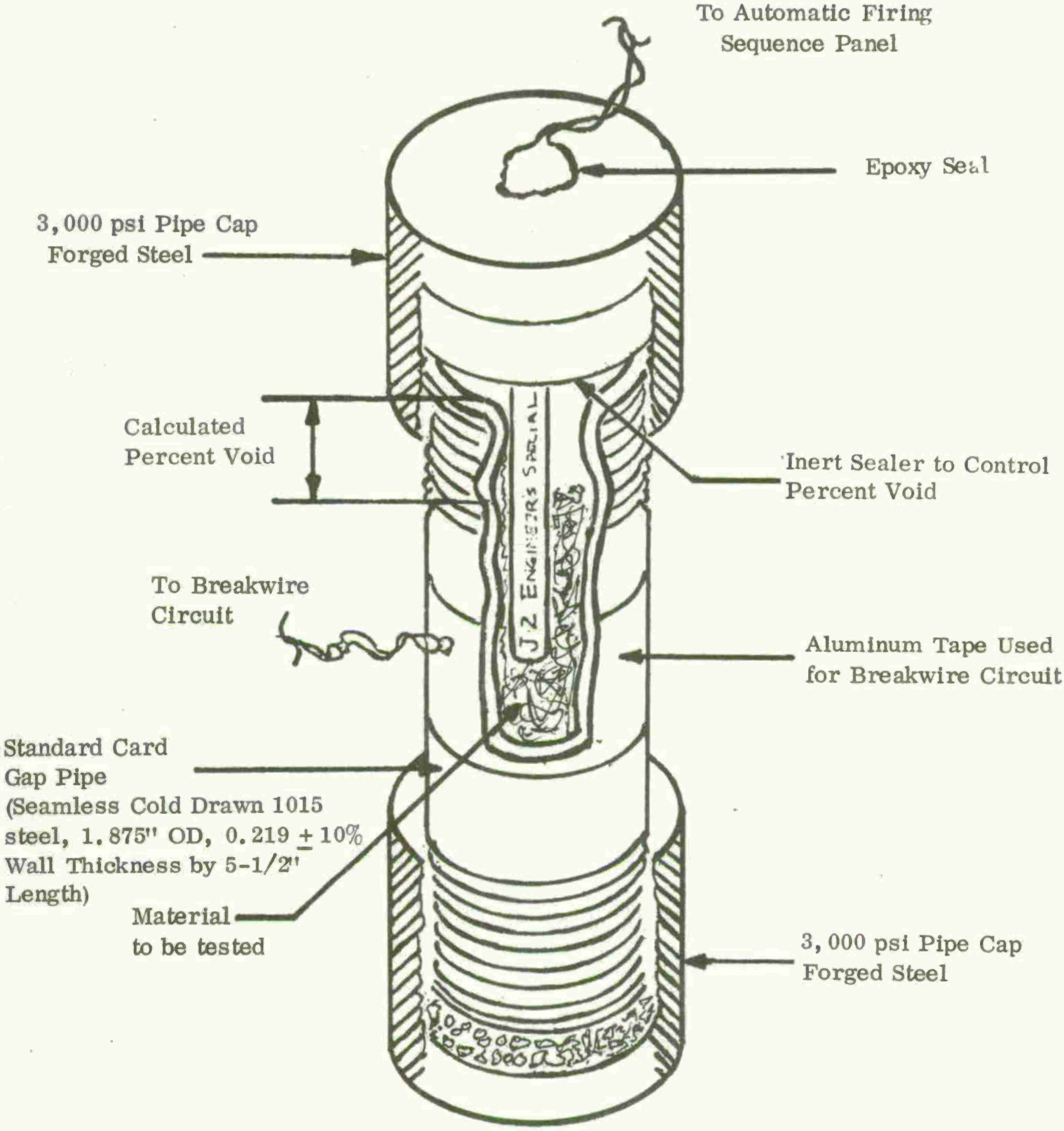
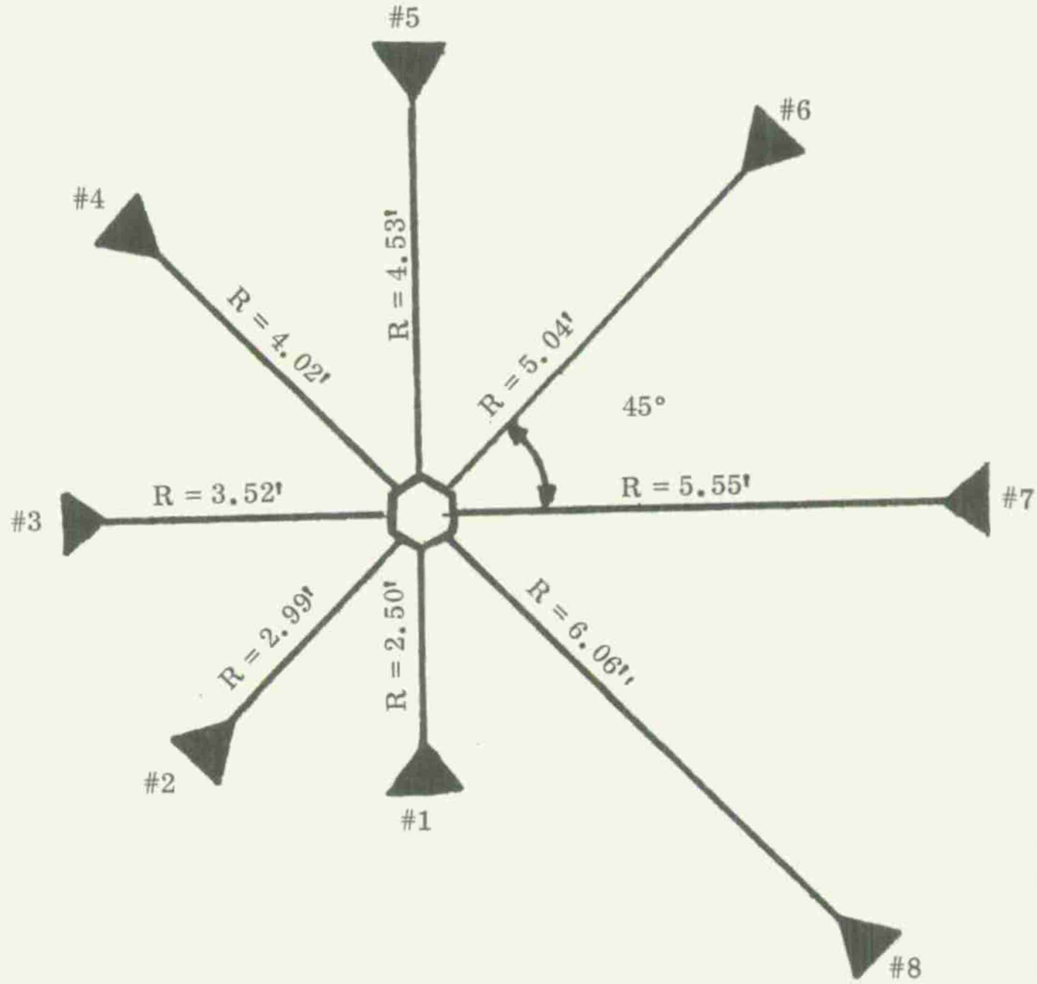


Figure 1. Test vessel configuration



Explosive charge suspended nine feet above ground surface so that only incident pressure is measured.

Pressure transducer and amplifier routed to transient recorder (digital readout).

R Radius from charge (feet).

Figure 2. Spiral Transducer Array

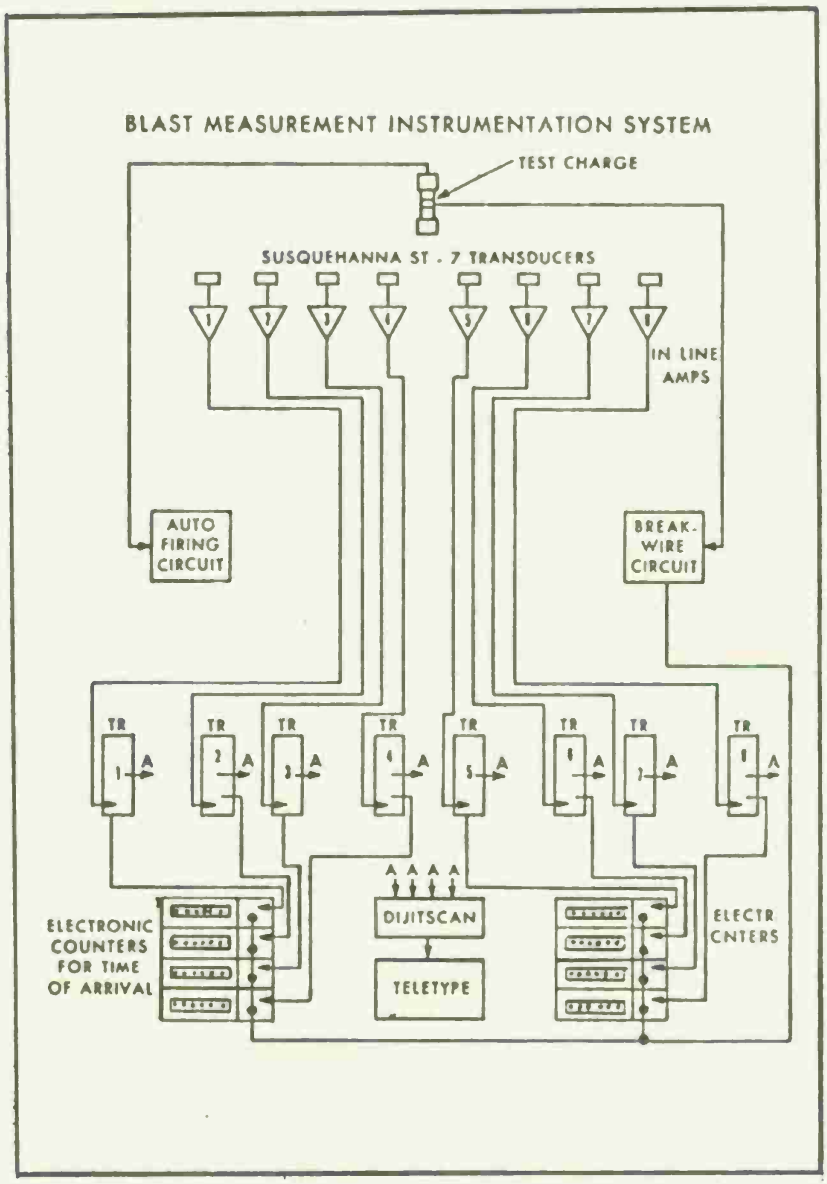


Figure 3. Blast Measurement Instrumentation System

METHOD 116

CLOSED BOMB

1. SCOPE

1.1 This method determines the relative quickness and relative force of a pyrotechnic mixture. The relative quickness value is defined as the average rate of change of pressure with respect to time (dp/dt) of the pyrotechnic mixture divided by that of a standard high explosive. The relative force value is the ratio of the maximum pressures developed by the pyrotechnic mixture and the standard high explosive.

2. SPECIMEN

2.1 The specimen for each trial shall consist of sufficient pyrotechnic mixture to produce a loading density of 0.2 gram (g) per cubic centimeter (cc) of bomb volume.

3. APPARATUS

3.1 The following apparatus is required:

(a) A 200-cc closed bomb capable of withstanding the pressures generated in the test. The inside diameter of the bomb should be about 2 inches. Equip the bomb with firing electrodes to effect ignition, a gas release valve, and a pressure transducer. Surround the bomb with a temperature controlled water jacket to maintain the required test temperature.

(b) A data acquisition system consisting of a pressure transducer, charge amplifier, D.C. amplifiers, differentiating amplifiers, and a cathode ray oscilloscope and equipment capable of photographing a trace; or an equivalent system capable of producing the required data.

(c) A piezo-electric type pressure transducer with a frequency response of at least 25 Kilohertz and a response to applied pressure which is linear over a range of 0 to 40,000 pounds per square inch (psi). Calibrate the transducer at least once per month with a dead weight tester or similar device which has an accuracy of $\pm$ 0.1 percent. If significant changes in guage sensitivity are noted, discontinue its use.

(d) An ignition system consisting of an electric squib and 0.5 to 1.0 g of black powder or an equivalent system.

(e) A balance accurate to 0.05 g.

4.0 PROCEDURE

4.1 Determine the volume of the closed bomb by filling it with water and measuring the volume of the water. Condition the material to be tested to 90° ± 2° F. Use a loading density of 0.2 g per cc for the specimen. The loading density of the standard high explosive will depend on the material used. Weigh the material put into the bomb to the nearest 0.05 g.

4.2 Make a warm-up shot prior to the start of the firing series. Alternate firing of the standard and the specimen and obtain records of the resulting dp/dt vs pressure traces. Conduct at least three firings of both the standard and the specimen. Clean the closed bomb thoroughly after each firing.

5. CALCULATIONS

5.1 The relative quickness values are taken at pressure points between approximately 25 percent and 75 percent of maximum pressure. Make Vy measurements at Vx values of 0.50, 0.75, 1.00, and 1.25 volts. Calculate the relative quickness at each Vx value as follows:

$$\text{Relative quickness} = \frac{100\ (\text{Average Vy value of specimen})}{(\text{Average Vy value of standard})}$$

Average the four results to obtain the relative quickness value of the pyrotechnic mixture.

5.2 Make the maximum pressure (Vmax) measurement at the point where a line tangent to the furtherest portion (from the Y axis) of the firing trace intersects the X axis. Calculate the relative force value as follows:

$$\text{Relative force} = \frac{100\ (\text{Average Vmax of specimen})}{(\text{Average Vmax of standard})}$$

The sensitivity settings of the recording instruments should be chosen so that the maximum pressure value falls between 1.75 and 2.00 volts.

6. EVALUATION

6.1 This method provides information on the rate of reaction and the force generated by a pyrotechnic mixture compared to a standard high explosive when both materials are separately ignited under confined conditions. The selection of a standard high explosive will be made after testing begins.

7. REFERENCES

(a) MIL-STD-286B, Method 801.1.1

(b) OSRD 1707

(c) Munroe and Tiffany, Bureau of Mines Bulletin 346

Closed Bomb

Rate of pressure rise (dp/dt) vs. Pressure (P)

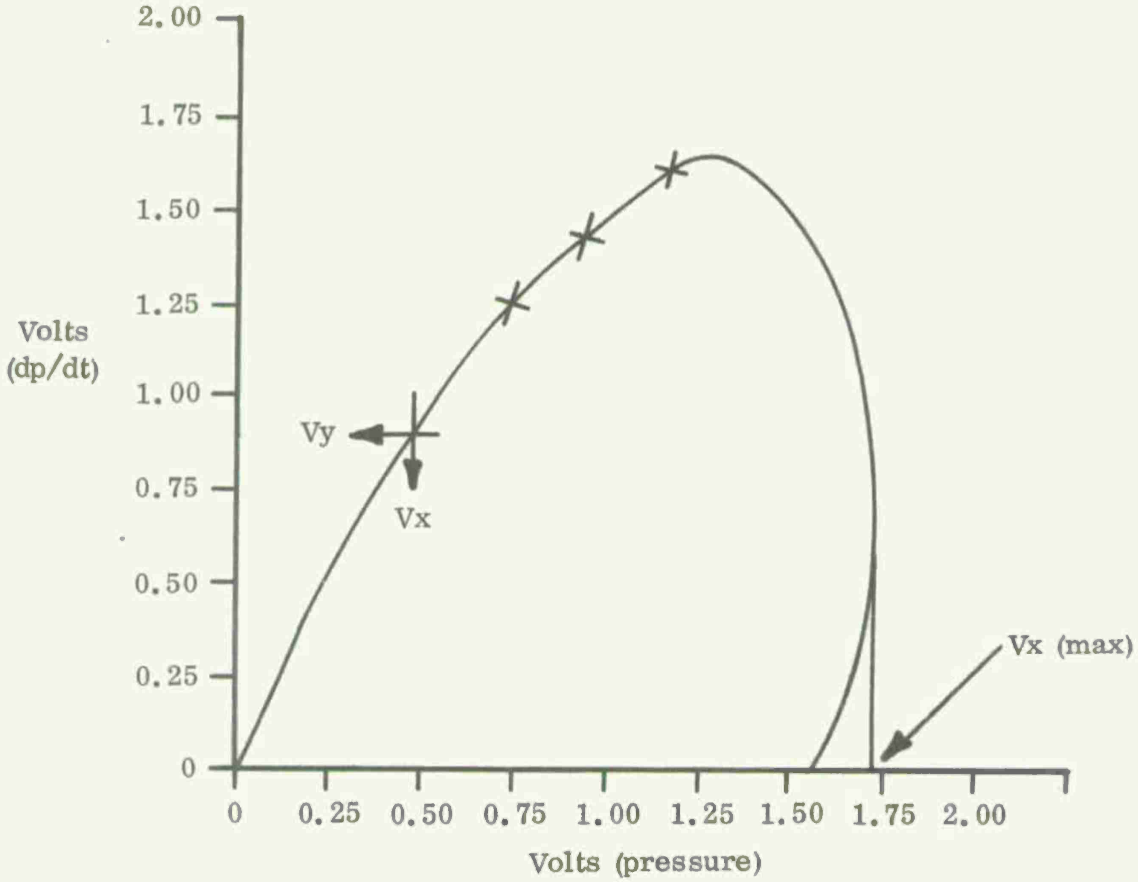


Figure 1. A Typical Trace Recording

METHOD 117

PARR BOMB CALORIMETER

1. SCOPE

1.1 This test determines the gross heat of combustion and gross heat of explosion of a pyrotechnic mixture. The gross heat of combustion is measured by burning the pyrotechnic mixture in an oxygen filled bomb submerged in water in an adiabatic chamber and measuring the rise in water temperature. The gross heat of explosion is measured by using nitrogen in the bomb in place of the oxygen.

2. SPECIMEN

2.1 The specimen shall consist of the quantity of pyrotechnic mixture necessary to cause a 2° to 3° C rise in water temperature. (If the approximate amount of specimen needed is unknown, use 1 to 2 grams (g) of specimen in the first trial.) The specimen shall be prepared by sieving it through a 50-mesh screen prior to testing.

3. MATERIALS

3.1 Materials required for this test are as follows:

(a) A Parr Instrument Co. Series 1200 adiabatic oxygen bomb calorimeter and related equipment or a similar adiabatic oxygen bomb calorimeter.

(b) A Parr Instrument Co. Series 2900 calorimeter temperature controller or similar equipment.

(c) A differential thermometer with a 5° C range and 0.01°C gradations.

(d) A balance with 0.2 g sensitivity and 5 kilogram capacity.

(e) A balance with 0.1 milligram (mg) sensitivity.

(f) Prepurified nitrogen having an oxygen content of no more than 0.01 percent.

(g) Pure oxygen.

(h) Calorimetric standard banzoic acid pellets.

(i) Distilled water.

4. PROCEDURE

4.1 Standard benzoic acid. Weigh to the nearest 0.1 mg approximately 1 g of the standard benzoic acid into a tared calorimeter combustion cup. Attach a 10-centimeter length of fuse wire to the electrodes in accordance with the manufacturer's instructions.

Place the combustion cup in its holder beneath the bomb head and bring the fuse wire in contact with the benzoic acid using the procedure described in the manufacturer's instruction manual. Place the assembled bomb head, along with 1 milliliter of distilled water, in the bomb cylinder and screw on the retaining cap. Tare the calorimeter bucket and fill with 2000.0 $\pm$ 0.2 g of distilled water. Pressurize the bomb with 25 to 30 atmospheres of pure oxygen and submerge it in the calorimeter bucket. Observe the submerged bomb for gas leakage as evidenced by bubbles. Do not fire the bomb if there is evidence of leakage. Place the calorimeter bucket inside the calorimeter jacket and attach the ignition wire to the firing terminal of the bomb. Close the cover and lower the thermometers into position. Turn on the automatic temperature controller and allow 10 minutes to establish temperature equilibrium between the calorimeter bucket and the calorimeter jacket. (Adiabatic conditions may be established and maintained by manual manipulation of the hot and cold water supplies.) Record the initial temperature. Using approved safety operating procedures, fire the charge. Allow the automatic temperature controller to equilibrate the calorimeter bucket and the calorimeter jacket temperatures after firing. Record the final maximum temperature when three identical readings have been made at 1-minute intervals. Remove the bomb from the calorimeter, release the pressure in a hood, disassemble, and clean. Measure the unburned fuse wire so that the measured heat of combustion may be corrected for the heat contributed by the fuse wire. Calibrate each calorimeter system monthly. Calculate the water equivalent of the calorimeter as follows:

$$\text{Water equivalent, calories per °C} = \frac{(6{,}318W + C)}{T}$$

where: C = Correction for combustion of fuse wire in calories,
T = Temperature rise, °C, corrected for thermometer error and emergent stem, and
W = Weight of standard benzoic acid in grams.

4.2 Heat of combustion. Weigh the specimen to the nearest 0.1 mg into a tared combustion cup. Repeat the procedure used for the standard benzoic acid in 4.1 except use a bomb pressure of 5 atmospheres of oxygen. Calculate the heat of combustion as follows:

$$\text{Heat of combustion, calories per gram} = \frac{ET}{W}$$

where: E = Water equivalent in calories per °C calculated in 4.1,
T = Temperature rise, °C, corrected for thermometer error and
W = Weight of specimen in grams.

4.3 Heat of explosion. Weigh the specimen to the nearest 0.1 mg into a tared combustion cup. Repeat the procedure used for the standard benzoic acid in 4.1 except omit the addition of a 1 ml of distilled water to the bomb and pressurize the bomb with 25 atmospheres of prepurified nitrogen after purging the bomb twice with the nitrogen. Calculate the heat of explosion as follows:

$$\text{Heat of explosion, calories per gram} = \frac{ET}{W}$$

where: E = Water equivalent in calories per °C calculated in 4.1,
T = Temperature rise, °C, corrected for thermometer error and emergent stem, and
W = Weight of specimen in grams.

5. EVALUATION

5.1 The heat of combustion and the heat of explosion of pyrotechnic mixtures give an indication of heat liberation potential and explosive power potential. These potentials are directly related to a pyrotechnic mixture's hazard potential. Another important factor which should be taken into consideration is the rate of pressure rise within the bomb as a function of time. The feasibility of instrumenting a Parr bomb to record such pressure rises should be examined. The rate of pressure rise gives an indication of the rate of reaction of the pyrotechnic mixture and can also be used to calculate the volume of gas liberated during the reaction. Consideration should also be given to determining the heat of combustion and heat of explosion in a bomb whose internal volume more nearly approaches the volume of the specimen. Any free volume in the bomb tends to influence the composition of the end products and thus affects the total heat content. This approach is reflected in the current Closed Bomb method.

6. REFERENCES

(a) MIL-STD-286B, method 802.1

(b) ASTM D240-64

(c) ORSD 293

(d) ORSD 702

(e) ORSD 1707

(f) PATR 1740

(g) GE-MTSD-R-059

(h) TM 9-1910

(i) Burlot and Thomas, Memoirs Poudres <u>29</u>, 1939, 262.

(j) Munroe and Tiffany, Bureau of Mines Bulletin 346.

METHOD 201

PROPAGATION/TRANSITION TEST A

1. SCOPE

1.1 This test is conducted on pyrotechnic end items which are packaged in experimental or standard storage and shipping containers. The test determines the potential hazards associated with the propagation of functioning from one end item (donar) in the container to surrounding end items (receptors) in the container.

2. TEST ITEMS

2.1 The test items required for each trial shall consist of the pyrotechnic end items packaged in an experimental or standard storage and shipping container.

3. MATERIAL

3.1 Materials required for each trial are as follows:

- (a) One electrical initiator: M2 squib or Engineers special electric blasting cap, J-2, as required.
- (b) One blasting machine or equivalent for use with the electrical initiator.
- (c) Wire (blasting line) for connecting blasting machine to electrical initiator.
- (d) One still camera.
- (e) One motion picture camera, 24 frames per second.
- (f) One motion picture camera, 1,000 frames per second.
- (g) Instrumentation to record blast overpressure, impulse, and thermal flux.
- (h) Meteorological equipment.
- (i) Sampling equipment for effluent gases.

4. PROCEDURE

4.1 Open the shipping container. Prime the most centrally positioned pyrotechnic end item in the container with the electrical initiator and connect the blasting line to the initiator. Reclose the container and place it on a suitable pad for remote controlled ignition. Attach the blasting line to the blasting machine and fire the primed pyrotechnic end item (donor) from a safe location using approved safety procedures.

4.2 This test should be conducted until propagation to receptor pyrotechnic end items occurs. If no propagation has occurred after five trials, terminate the test. If propagation to receptor end items does occur, discontinue this test, record the results, and conduct Propagation/Transition Test B.

4.3 Observe the number of end items within the container that have functioned and the condition of the container. During the test, sample any effluent gases for subsequent chemical analysis.

5. DOCUMENTATION

5.1 The documentation of this test shall include the following information:

(a) Item designation (and Federal Stock No. if available).

(b) Item lot number.

(c) Item sublot number.

(d) Item serial number.

(e) Detailed quality control report on location and size of defects if any, in test item.

(f) Date of manufacture of item.

(g) Date of test.

(h) Meteorological data: ambient air temperature, barometric pressure, wind velocity and direction, and relative humidity.

(i) Schematic drawing of test setup prior to test showing location, type, and distance of instrumentation, cameras, and test item.

(j) Photographs of the actual test items in the test configuration and photographs of the test position after firing.

(k) Motion pictures of actual test at 24 and 1,000 frames per second.

(l) Overpressure in psi and impulse vs distance curve (including calibration test).

(m) A map locating the radial and angular positions of unexploded items and missiles of any form, including metal fragments and propellant fragments (burned and unburned) with respect to the test position. Missile type and estimated weight will be recorded on the map.

(n) Crater dimensions.

(o) Thermal flux measurements.

(p) Qualitative analysis of effluent gases produced during the test.

(q) A chronology of events such as that shown on figure 1.

(r) A report of test.

6. EVALUATION

6.1 This test is satisfactory for determining the potential hazards associated with the propagation of functioning from a donor pyrotechnic end item to receptor pyrotechnic end items in the same container. The potential hazards include fire, missiles, blast (over-pressure) and effluent gases.

7. REFERENCES

(a) TB 700-2

(b) GE-MTSD-R-035

(c) GE-MTSD-R-037

TIME IN SECONDS	EVENT
T+ 0	Ignition
T+ 2	CS Burning, Donor Canister
T+ 7	Box on Fire
T+ 24	Entire Box Reacting Violently - Propagating to Other Containers
T+ 49	High Flame Engulfing Box
T+ 55	Temperature Reading
T+ 64	Grass on Fire
T+ 66	Three Canisters Blown Out of Box
T+ 76	Additional Eruption
T+ 134	Case of Canisters on Ground Stopped Burning
T+ 179	END OF TEST

Figure 1. A Typical Chronology of Events

METHOD 202

PROPAGATION/TRANSITION TEST B

1. SCOPE

1.1 This test is conducted on pyrotechnic end items which are packaged in two experimental or standard storage and shipping containers. The test determines the potential hazards associated with the propagation of functioning from a pyrotechnic end item (donor) in one container to pyrotechnic items (receptors) in an adjacent container.

2. TEST ITEMS

2.1 The test items required for each trial shall consist of pyrotechnic end items packaged in two experimental or standard storage and shipping containers.

3. MATERIAL

3.1 Materials required for each trial are as follows:

(a) One electrical initiator: M2 squib or Engineers special electric blasting cap, J-2, as required.

(b) One blasting machine or equivalent for use with the electrical initiator.

(c) Wire (blasting line) for connecting blasting machine to electrical initiator.

(d) One still camera.

(e) One motion picture camera, 24 frames per second.

(f) One motion picture camera, 1,000 frames per second.

(g) Instrumentation to record blast overpressure, impulse and thermal flux.

(h) Meteorological equipment.

(i) Sampling equipment for effluent gases.

(j) A quantity of steel banding and banding equipment.

4. PROCEDURE

4.1 Open one of the shipping containers. Use the electrical initiator to prime the pyrotechnic end item which will be closest to the receptor pyrotechnic end items in the second container. If several items will be equally close to the receptor end items in the second container, prime the most centrally positioned one. Connect the blasting line to the electrical initiator and reclose the container. Band the two containers together with steel straps in a manner which provides the minimum separation between the primed donor end item and the receptor end items. Place the two containers on a suitable pad for remote controlled ignition. Attach the blasting line to the blasting machine and fire the primed donor pyrotechnic end item from a safe location using approved safety procedures.

4.2 This test should be conducted until propagation to receptor pyrotechnic end items occurs. If no propagation has occurred after five trials, terminate the test.

4.3 Observe the number of receptor pyrotechnic end items in the second container that have functioned as a result of functioning the donor pyrotechnic end item in the first container. During the test, sample any effluent gases for subsequent chemical analysis.

5. DOCUMENTATION

5.1 The documentation of this test shall include the following information:

(a) Item designation (and Federal Stock No. if available).

(b) Item lot number.

(c) Item sublot number.

(d) Item serial number.

(e) Detailed quality control report on location and size of defects, if any, in test item.

(f) Date of manufacture of item.

(g) Date of test.

(h) Meteorological data: ambient air temperature, barometric pressure, wind velocity and direction, and relative humidity.

(i) Schematic drawing of test setup prior to test showing location, type, and distance of instrumentation, cameras, and test item.

(j) Photographs of the actual test items in the test configuration and photographs of the test position after firing.

(k) Motion pictures of actual test at 24 and 1,000 frames per second.

(l) Overpressure in psi and impulse vs distance curve (including calibration test).

(m) A map locating the radial and angular positions of unexploded items and missiles of any form, including metal fragments and propellant fragments (burned and unburned) with respect to the test position. Missile type and estimate weight will be recorded on the map.

(n) Crater dimensions.

(o) Thermal flux measurements.

(p) Qualitative analysis of effluent gases produced during the test.

(q) A chronology of events such as that shown on figure 1.

(r) A report of test.

6. EVALUATION

6.1 This test is satisfactory for determining the potential hazards associated with the propagation of functioning from a donor pyrotechnic end item in one container to receptor pyrotechnic end items in an adjacent container. The potential hazards include fire, missiles, blast (overpressure), and effluent gases (chemical).

7. REFERENCES

(a) TB 700-2

(b) GE-MTSD-R-035

(c) GE-MTSD-R-037

TIME IN SECONDS	EVENT
T+ 0	Ignition
T+ 2	CS Burning, Donor Canister
T+ 7	Box on Fire
T+ 24	Entire Box Reacting Violently - Propagating to Other Containers
T+ 49	High Flame Engulfing Box
T+ 55	Temperature Reading
T+ 64	Grass on Fire
T+ 66	Three Canisters Blown Out of Box
T+ 76	Additional Eruption
T+ 134	Case of Canisters on Ground Stopped Burning
T+ 179	END OF TEST

Figure 1. A Typical Chronology of Events

METHOD 203

EXTERNAL HEAT TEST

1. SCOPE

1.1 This test is conducted on pyrotechnic end items which are packaged in experimental or standard storage and shipping containers. The test determines the potential hazards involved when the containers are enveloped by a hot open fire.

2. TEST ITEMS

2.1 The items to be tested shall consist of pyrotechnic end items packaged in four experimental or standard storage and shipping containers.

3. MATERIALS

3.1 Materials required for the test are as follows:

(a) A quantity of steel banding and banding equipment.

(b) A quantity of scrap lumber.

(c) Approximately 50 gallons of kerosene or diesel fuel.

(d) Two electric squibs.

(e) One blasting machine.

(f) Wire (blasting line) for connecting blasting machine to squibs.

(g) Four ounces of smokeless powder.

(h) One still camera.

(i) One motion picture camera, 24 frames per second.

(j) One motion picture camera, 1,000 frames per second.

(k) Instrumentation to record blast overpressure, impulse, and thermal flux.

(l) Meteorological equipment.

(m) Sampling equipment for effluent gases.

4. PROCEDURE

4.1 Using the scrap lumber, build a crib approximately 30 inches high and of sufficient dimensions to hold the stack of shipping containers. Band the four shipping containers together with steel straps in a configuration that best approximates a cube. Place the banded containers on the crib and stack additional scrap lumber around and over the containers to ensure a sustained hot fire. A typical test configuration is shown on figure 1. Pour approximately 50 gallons of kerosene or diesel fuel over the entire crib. At each of two locations, on opposite sides of the crib, place an electric squib in 2 ounches of smokeless powder. Use the blasting line to connect the squibs to a blasting machine. Start the test by firing the squibs from a safe location using approved safety procedures. During the test, sample any effluent gases for subsequent chemical analysis.

5. DOCUMENTATION

5.1 The documentation of this test shall include the following information:

(a) Item designation (and Federal Stock No. if available).

(b) Item lot number.

(c) Item sublot number.

(d) Item serial number.

(e) Detailed quality control report on location and size of defects if any, in test item.

(f) Date of manufacture of item.

(g) Date of test.

(h) Meteorological data: ambient air temperature, barometric pressure, wind velocity and direction, and relative humidity.

(i) Schematic drawing of test setup prior to test showing location, type, and distance of instrumentation, cameras, and test item.

(j) Photographs of the actual test items in the test configuration and photographs of the test position after firing.

(k) Motion pictures of actual test at 24 and 1,000 frames per second.

(l) Overpressure in psi and impulse vs distance curve (including calibration test).

(m) A map locating the radial and angular positions of unexploded items and missles of any form, including metal fragments and propellant fragments (burned and unburned) with respect to the test position. Missle type and estimated weight will be recorded on the map.

(n) Crater dimensions.

(o) Thermal flux measurements.

(p) Qualitative analysis of effluent gases produced during the test.

(q) A chronology of events such as that shown on figure 2.

(r) A report of test.

6. EVALUATION

6.1 This test is satisfactory for determining the potential hazards involved when containers of pyrotechnic end items are enveloped by a hot open fire. The potential hazards include missiles, blast (overpressure), and effluent gases.

7. REFERENCES

(a) TB 700-2

(b) GE-MTSD-R-035

(c) GE-MTSD-R-037

External Heat Test

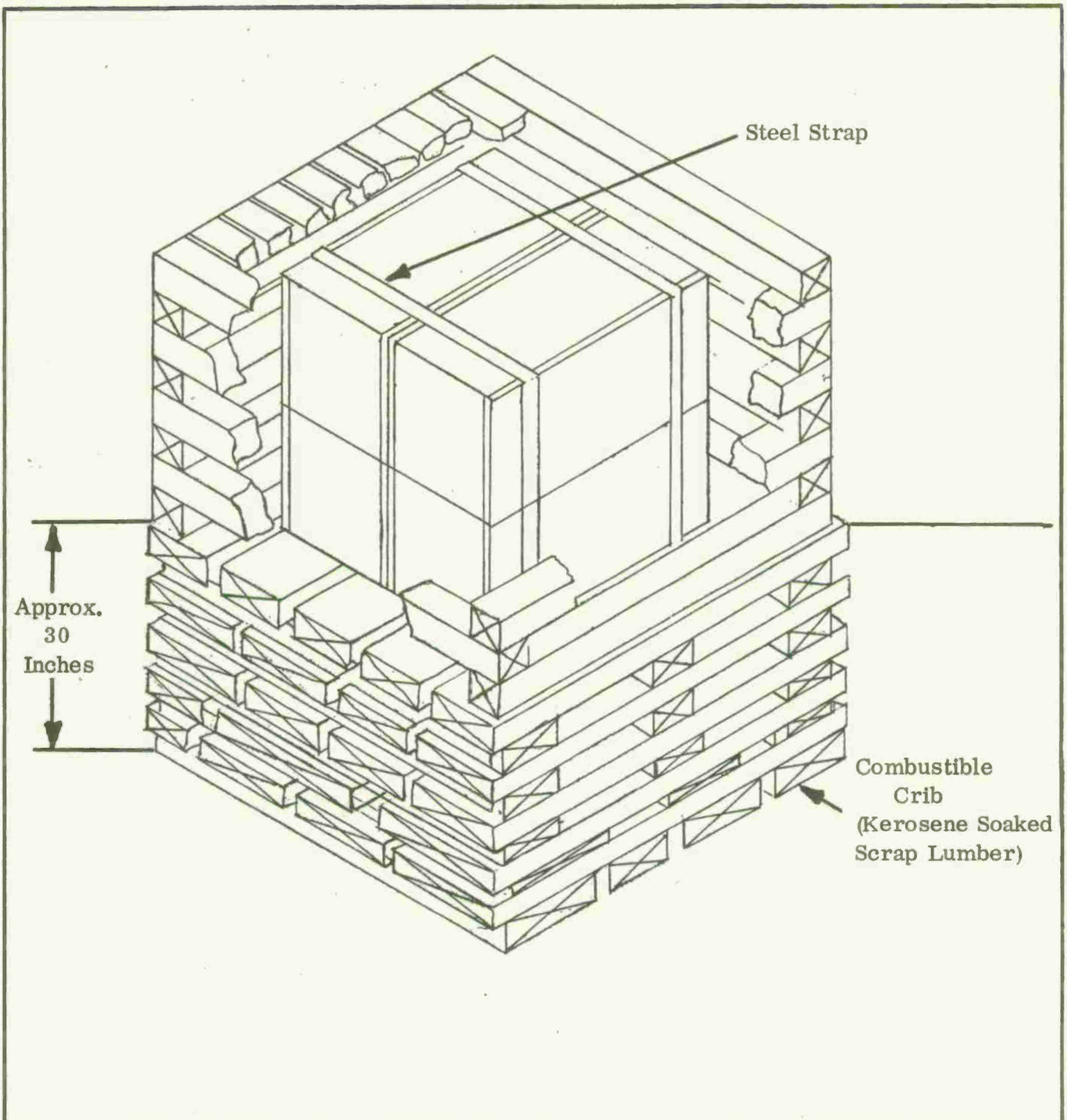


Figure 1. A Typical Test Configuration

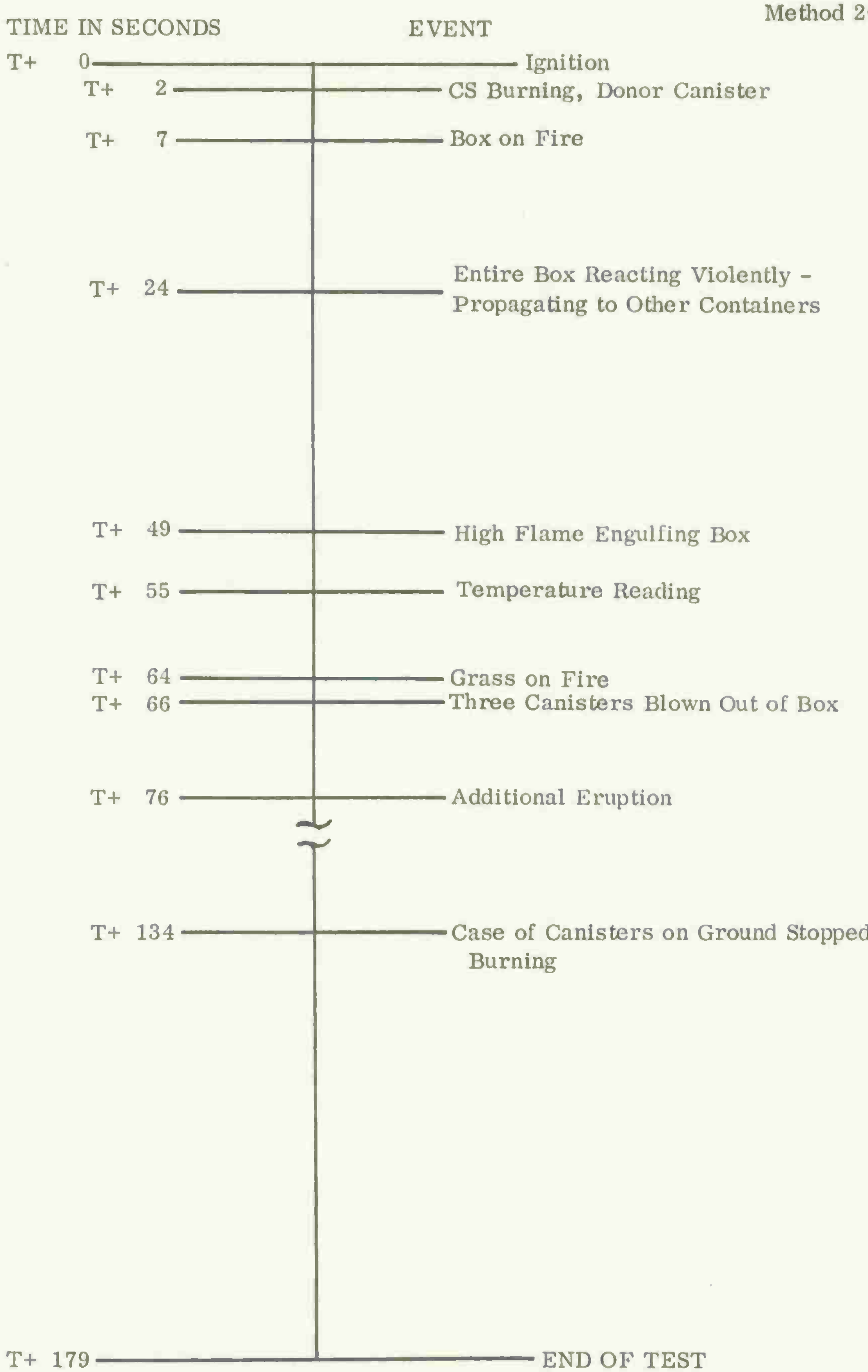


Figure 2. A Typical Chronology of Events

METHOD 204

TRANSPORTATION ROUGH HANDLING

1. SCOPE

1.1 This test determines the potential hazards of pyrotechnic end items packaged in shipping containers when the containers experience rough handling associated with transporttion. The rough handling conditions used in the test are vibration, shock and 5-foot drop.

2. SPECIMEN

2.1 The specimen shall consist of two shipping containers filled with the pyrotechnic end items to be tested.

3. EQUIPMENT

3.1 The following equipment is required:

(a) Vibration equipment as specified in MIL-STD-810B.

(b) L.A.B. package testing machine.

(c) Equipment for dropping containers from a height of 5 feet onto an armor plate over concrete.

(d) Equipment for conditioning the containers at -65°F and at 155°F.

(e) A still camera.

4. PROCEDURE

4.1 Vibration. Condition one specimen container at -65° F for 48 hours and condition the other specimen container at 155° F for 48 hours. Maintain the conditioning temperature during the vibration test. Subject each container to high frequency vibration in accordance with method 514, procedure X of MIL-STD-810B. Use vibration test curve AB of figure 514-6 of MIL-STD-810B and time schedule IV of table 514-II of MIL-STD-810B.

4.2 Shock. After completion of the procedure in 4.1, recondition the specimen containers for 24 hours at the same conditioning temperatures used in 4.1 on the table of a L.A.B. package testing machine. Operate the table at a speed that will impact with an acceleration of $\pm$ 1 g. Bounch the containers for a total of 2 hours 40 minutes with the longitudinal axes of the containers in the horizontal plane and parallel to the throw of the machine, 40 minutes with the longitudinal axes in the horizontal plane and perpendicular to the throw, and 40 minutes with the longitudinal axes in the vertical plane.

METHOD 205

CRASH - SAFETY (40 FOOT DROP)

1. SCOPE

1.1 This test determines the potential hazards of pyrotechnic end items packaged in shipping containers in the event of an accidental drop or crash.

2. SPECIMEN

2.1 The specimen shall consist of four shipping containers filled with the pyrotechnic end items to be tested.

3. EQUIPMENT

3.1 The following equipment is required:

(a) Equipment for dropping containers from a height of 40 feet onto a steel plate over concrete.

(b) A still camera.

4. PROCEDURE

4.1 Drop each specimen container separately from a height of 40 $\pm$ 1/2 feet onto a steel plate over concrete. Drop each of the four containers one time so as to impact in the following respective altitudes:

(a) Corner

(b) Bottom, edge

(c) Top, flat

(d) End, flat

4.2 Record and photograph any resulting damage or deformation. Report in detail any evidence of flaming, explosion, fragmentation, or functioning giving duration, range, and other pertinent information.

5. EVALUATION

5.1 This test provides a reliable means of evaluating the potential hazards of shipping containers filled with pyrotechnic end items which are subjected to a violent accidental crash or drop.

6. REFERENCES

(a) Fed. Test Method Std. No. 101a.

METHOD 301

BULK DENSITY

1. SCOPE

1.1 This test determines the bulk or apparent density of pyrotechnic mixtures. Bulk density is the weight per unit of outside volume, which may include voids.

2. SPECIMEN

2.1 The specimen shall consist of sufficient pyrotechnic mixture to fill a 100-milliliter (ml) graduated cylinder.

3. MATERIALS

3.1 The materials required for this test are as follows:

(a) A 100-ml graduated cylinder.

(b) A balance accurate to 0.01 gram.

4. PROCEDURE

4.1 Make all weighings to the nearest 0.01 gram. Weigh the empty graduated cylinder. Fill the graduated cylinder with the specimen by gravity feed. Allow the cylinder to stand undisturbed for 10 minutes. Read the fill volume to the nearest milliliter graduation. Weigh the cylinder and specimen.

5. CALCULATIONS

5.1 Calculate the (apparent) bulk density in grams per cubic centimeter as follows:

$$\text{Bulk density} = \frac{(A - B)}{C}$$

where: A = Weight of cylinder and specimen in grams,
B = Weight of empty cylinder in grams, and
C = Volume of specimen in cylinder in milliliters.

6. EVALUATION

6.1 In order to assess potential hazards of pyrotechnic mixtures, it is necessary to know certain physical characteristics of the mixture. Bulk density is one of the important physical characteristics in determining the "critical mass" of a pyrotechnic mixture.

7. REFERENCES

(a) MIL-STD-286B, method 507.1

(b) MIL-STD-650, method 201.1

METHOD 302

COMPATIBILITY (REACTIVITY WITH SURROUNDINGS)

1. SCOPE

1.1 This test determines the compatibility of a pyrotechnic mixture with other materials in which it comes in contact throughout its life cycle.

2. SPECIMEN

2.1 The specimen shall consist of 5 grams (g) of the pyrotechnic mixture to be tested and 5 g of the contact material to be tested. The pyrotechnic mixture specimen shall be prepared by sieving it through a 50-mesh screen prior to testing. If the contact material is metal, it shall be tested as fine milled chips or filings. If the contact material is film, cloth, or paper, it shall be cut into 1/8-inch squares. Other solid contact materials shall be milled to a fineness of approximately 12 mesh.

3. APPARATUS

3.1 The test apparatus shall consist of the following:

(a) Constant temperature bath capable of maintaining a temperature of $100° \pm 1°$ C.

(b) Compatibility apparatus as shown on figure 1.

(c) Vacuum pump.

4. PROCEDURE

4.1 Standardize the compatiblity apparatus as follows: Determine the volume of the heating tube by filling it with mercury from a buret until the mercury reaches the level at which it will contact the ground glass joint of the capillary tube. Determine the unit capacity of the capillary by placing exactly 10 g of mercury in its cup, and manipulating the tube so that all the mercury passes into the long (85-centimeter) section of the capillary. Be sure that the mercury remains as a continuous column. Measure the length of the mercury column at three positions in the long section of the capillary, and average the three measurements. Calculate the unit capacity of the capillary as follows:

$$\text{Unit capacity of capillary, ml per mm} = \frac{W}{13.59L}$$

where: W = Weight of mercury in the column in grams and
L = Average length of mercury column in millimeters.

4.2 Perform three determinations. In the first determination, place 2.5 g of the pyrotechnic mixture specimen in the heating tube. In the second determination, place 2.5 g of the contact material in the heating tube. In the third determination, place 2.5 g of the pyrotechnic mixture specimen and 2.5 g of the contact material in the heating tube.

4.3 Perform each determination as follows: Coat the ground glass joint of the capillary tube with a light film of petroleum jelly, and make an airtight connection between the heating tube and the capillary by pressing the tube up against the capillary with a twisting motion. Mount the apparatus on a rack so that the long section of the capillary is nearly vertical, and the cup at the bottom rests on a solid support. Fill the cup with 7.0 millimeters (ml) of mercury and connect a vacuum line to the mouth of the cup. Evacuate the capillary to an absolute pressure of approximately 5 millimeters (mm) of mercury. (Evacuation will be facilitated by tilting the apparatus until the capillary opening in the bottom of the cup is free of mercury.) When the pressure has been reduced to 5 mm of mercury, remove the vacuum line and allow the mercury to enter the capillary. Record the following data:

(a) Length of capillary from heating tube joint to surface of mercury pool in cup (C_1).

(b) Height of mercury column above the surface of the mercury pool (H_1).

(c) Barometric pressure in millimeters of mercury (P_1).

(d) Temperature of room in degrees Centigrade (t_1).

4.4 Immerse the heating tube in the constant temperature bath, being careful not to loosen the connection between the heating tube and the capillary. Heat the tube at a temperature of 100° ± 1° C for 40 hours. Remove the tube from the constant temperature bath and allow it to cool to room temperature. Record the following data:

(a) Length of capillary from heating tube joint to the surface of the mercury pool in the cup (c).

(b) Height of mercury column above the surface of the mercury pool (H).

(c) Barometric pressure in millimeters of mercury (P).

(d) Temperature of the room in degrees Centigrade (t).

5. CALCULATIONS

5.1 Calculate the volume of gas (at standard temperature and pressure) liberated during the test as follows:

$$\text{Volume of gas, ml} = (A + B)(C - H)\,\frac{273\,(P - H)}{760(273 + t)} - (A+B)(C_1 - H_1)\,\frac{273\,(P_1 - H_1)}{760\,(273 + t_1)}$$

where: A = Volume of heating tube (less 5 ml allowance for specimen) in milliliters,

B = Unit capacity of capillary, milliliters per millimeter calculated in 4.1,

C = Length of capillary from heating tube joint to top of mercury column at end of test in millimeters,

C_1 = Length of capillary from heating tube joint to top of mercury column at beginning of test in millimeters,

H = Height of mercury column above the surface of the mercury pool at end of test in millimeters,

H_1 = Height of mercury column above the surface of the mercury pool at beginning of test in millimeters,

P = Atmospheric pressure at end of test in millimeters of mercury,

P_1 = Atmospheric pressure at beginning of test in millimeters of mercury,

t = Room temperature, °C, at end of test, and

t_1 = Room temperature, °C, at beginning of test.

5.2 Calculate the amount of gas produced by the mixture of contact material and pyrotechnic specimen in excess of the amount of gas evolved by the materials separately as follows:

$$\text{Gas due to reactivity, milliliters} = A - (B + C)$$

where: A = Milliliters of gas evolved by mixture of contact material and pyrotechnic specimen in the third determination,

B = Milliliters of gas evolved by the pyrotechnic specimen in the first determination, and

C = Milliliters of gas evolved by the contact material in the second determination.

6. EVALUATION

6.1 Compatibility, which is the ability of a pyrotechnic mixture to remain unaffected when in contact with other material, is useful in evaluating the potential hazards of a pyrotechnic mixture due to its reaction upon contact with other materials.

7. REFERENCES

(a) MIL-STD-650, method 504.1

(b) MIL-STD-1234, method 504.1

(c) AMCP 760-177 - Engineering Design Handbook, Properties of Explosives of Military Interest, Part I, January, 1971.

(d) AMCP 760-186 - Engineering Design Handbook, Military Pyrotechnic Series, Part Two - Safety, Procedures, and Glossary, October, 1963.

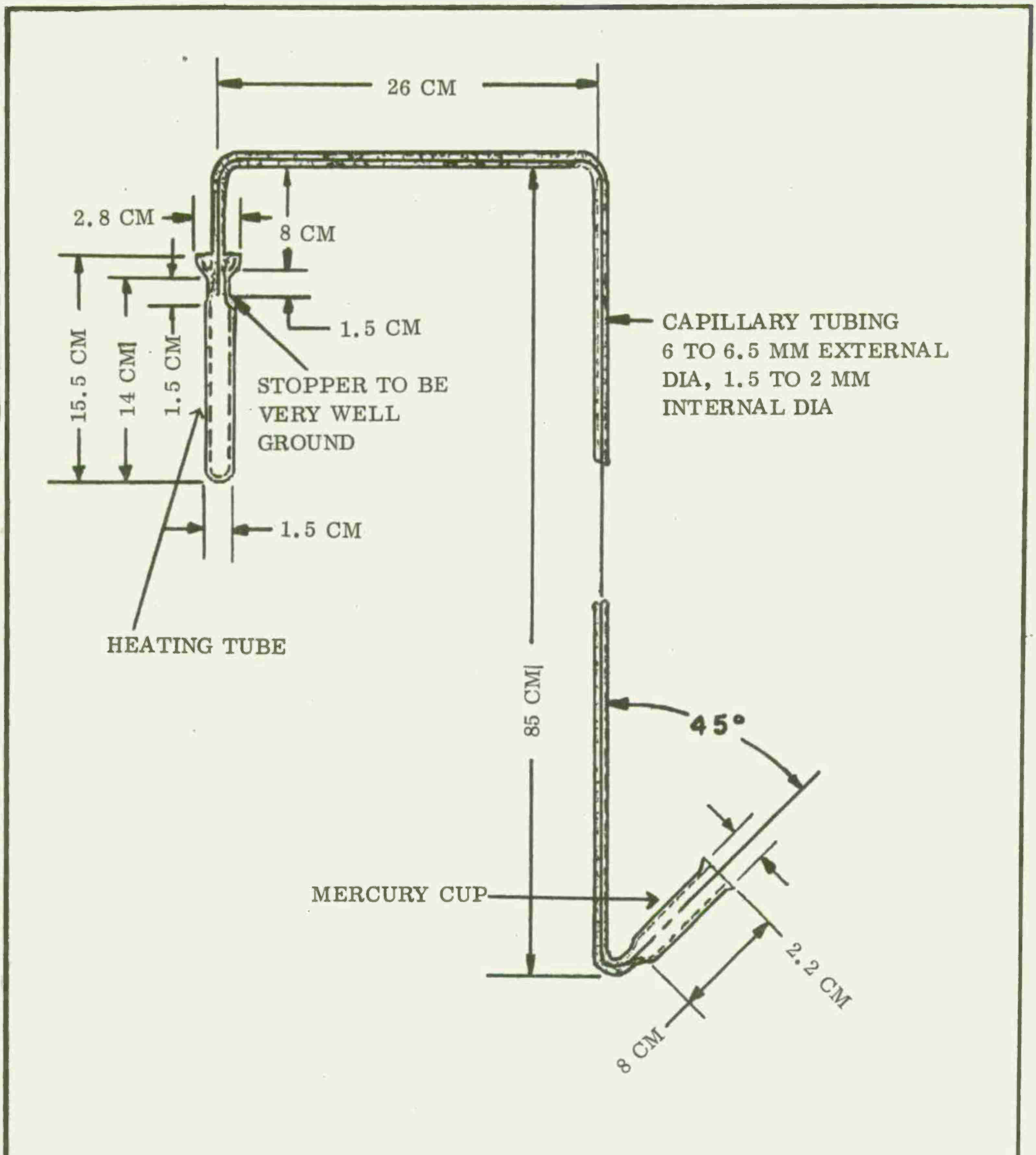


Figure 1. Compatibility Apparatus

METHOD 303

HYGROSCOPICITY

1. SCOPE

1.1 This test determines the amount of moisture absorbed by pyrotechnic mixtures when they are subjected to a relative humidity of 90 percent.

2. SPECIMEN

2.1 The specimen shall consist of 14 to 15 grams of the pyrotechnic mixture to be tested. The specimen shall be prepared by sieving it through a 50-mesh screen prior to testing.

3. MATERIALS

3.1 Materials required for this test are as follows:

(a) A glass weighing bottle approximately 70 millimeters (mm) in diameter and 33 mm high with ground glass cover.

(b) A desiccator containing a solution of 18.6 ± 0.5 percent by weight sulfuric acid in water for producing a 90-percent relative humidity at 30° C.

(c) A desiccator containing an indicating desiccant.

(d) An oven capable of maintaining a constant temperature of 30° ± 2° C.

(e) A balance accurate to 0.2 milligram (mg).

4. PROCEDURE

4.1 Make all weighings to the nearest 0.2 mg. Weigh the weighing bottle and cover. Place the specimen in the weighing bottle, cover, and reweigh. Place the weighing bottle and contents in the desiccator containing the sulfuric acid solution and remove the bottle cover. Cover the desiccator and place it in an oven which is maintained at a constant temperature of 30° ± 2°C. Remove the weighing bottle from the oven after 2 days, cover, cool to room temperature in a desiccator containing an indicating desiccant, and weigh. Place the weighing bottle back in the desiccator containing the sulfuric acid solution in the oven. Reweigh the bottle and contents at 2-day intervals until the change in weight between successive weighings is no more than 0.2 mg.

5. CALCULATIONS

5.1 Calculate the percent by weight of hygroscopic moisture absorbed by the specimen as follows:

$$\text{Percent hygroscopic moisture absorbed} = \frac{100\ (A - B)}{(B - C)}$$

where: A = Final weight of covered weighing bottle and contents,
B = Initial weight of covered weighing bottle and contents, and
C = Weight of empty covered weighing bottle.

6. EVALUATION

6.1 This test gives some indication of a pyrotechnic mixture's tendency to absorb atmospheric moisture. This information can be used as a guide in the processing, storing, and handling of the pyrotechnic mixture up to the time of testing for potential hazards. Excessive moisture absorption by a pyrotechnic mixture prior to testing for potential hazards may produce misleading evaluations.

7. REFERENCES

(a) MIL-STD-286B, method 503.1.3

(b) MIL-STD-650, method 208.1

(c) Henkin, H., "Methods of Stability Testing", ORSD 3401, 22 March 1944.

(d) Tomlinson, W. R. and Sheffeld, O. E., "Properties of Explosives of Military Interest," Picatinny Arsenal Technical Report 1740 (Revision 1), April, 1958.

(e) AMCP 706-177

(f) AMCP 706-186

METHOD 304

MOISTURE (DESICCATION METHOD)

1. SCOPE

1.1 This test determines the moisture content of pyrotechnic mixtures which do not contain volatile ingredients. The moisture content determined is based on the loss of weight of the pyrotechnic mixture in a desiccated atmosphere.

2. SPECIMEN

2.1 The specimen shall consist of approximately 10 grams of the pyrotechnic mixture to be tested. The specimen shall be prepared by sieving it through a 50-mesh screen prior to testing.

3. MATERIALS

3.1 Materials required for this test are as follows:

(a) A glass weighing bottle approximately 70 millimeters (mm) in diameter and 33 mm high with ground glass cover.

(b) A desiccator approximately 250 mm in diameter, or one of equivalent volume, which has been filled nearly to the plate with a suitable desiccant such as calcium chloride or anhydrous calcium sulfate.

(c) A balance accurate to 0.2 milligram (mg).

4. PROCEDURE

4.1 Make all weighings to the nearest 0.2 mg. Weigh the weighing bottle and cover. Place the specimen in the weighing bottle, cover, and weigh. Place the weighing bottle and contents in the desiccator and remove the bottle cover. Cover the desiccator and maintain at a temperature of 25° ± 5° C. Weigh the covered bottle and contents at 24-hour intervals until the loss in weight between successive weighings is no more than 1 mg.

5. CALCULATIONS

5.1 Calculate the percent by weight moisture in the specimen as follows:

$$\text{Percent moisture} = \frac{100\,(A - B)}{A - C}$$

where: A = Weight of covered bottle and specimen before desiccation,
B = Weight of covered bottle and specimen after desiccation, and
C = Weight of empty covered weighing bottle.

6. EVALUATION

6.1 Moisture content determination of pyrotechnic mixtures is required so that subsequent evaluations of potential hazards of the pyrotechnic mixtures can be correlated to the amount of moisture in the specimen.

7. REFERENCES

(a) MIL-STD-286B, method 101.3.2

(b) MIL-STD-650, method 101.2

(c) MIL-STD-1234, method 101.1.1

METHOD 305

MOISTURE AND VOLATILES (VACUUM OVEN METHOD)

1. SCOPE

1.1 This test determines the moisture and volatile matter content of pyrotechnic mixtures. The determination is based on the loss of weight of the pyrotechnic mixture in an oven under vacuum.

2. SPECIMEN

2.1 The specimen shall consist of approximately 10 grams of the pyrotechnic mixture to be tested. The specimen shall be prepared by sieving it through a 50-mesh screen prior to testing.

3. MATERIALS

3.1 Materials required for this test are as follows:

(a) A glass weighing bottle approximately 70 millimeters (mm) in diameter and 33 mm high with ground glass cover.

(b) A desiccator containing a suitable desiccant such as calcium chloride or anhydrous calcium sulfate.

(c) A vacuum oven.

(d) A balance accurate to 0.2 milligram (mg).

4. PROCEDURE

4.1 Make all weighings to the nearest 0.2 mg. Weigh the weighing bottle and cover. Place the specimen in the weighing bottle, cover, and weigh. Place the weighing bottle and contents in the vacuum oven, remove the bottle cover, and heat the specimen for 6 hours at $55° \pm 2°$ C under an absolute pressure of 80 ± 10 mm of mercury. Cool the bottle and specimen to room temperature in the desiccator. Cover and reweigh.

5. CALCULATIONS

5.1 Calculate the percent by weight moisture and volatiles in the specimen as follows:

$$\text{Percent moisture and volatiles} = \frac{100\,(A - B)}{(A - C)}$$

where:
- A = Weight of covered bottle and specimen before heating,
- B = Weight of covered bottle and specimen after heating, and
- C = Weight of empty covered weighing bottle.

6. EVALUATION

6.1 A moisture and volatiles content determination of pyrotechnic mixtures is required so that subsequent evaluations of potential hazards of the pyrotechnic mixtures can be correlated to the amount of moisture and volatiles in the specimen.

7. REFERENCES

(a) MIL-STD-286B, Method 101.1.2

(b) MIL-STD-1234, Method 102.2.1

METHOD 306

MOISTURE AND TOTAL VOLATILES (GAS CHROMATOGRAPHIC METHOD)

1. SCOPE

1.1 This test determines water, ethyl alcohol, and diethyl ether content of pyrotechnic mixtures. It is based on the extraction of the solvents from the pyrotechnic mixture with a solution of predried methyl ethyl ketone and secondary butyl alcohol.

2. SPECIMEN

2.1 The specimen shall consist of approximately 10 grams (g) of the pyrotechnic mixture to be tested. The specimen shall be prepared by sieving it through a 50-mesh screen while keeping exposure to the atmosphere to a minimum in order to reduce loss of volatiles or absorption of water.

3. APPARATUS

3.1 The test apparatus shall consist of a gas chromatograph equipped with a thermal conductivity detector and a 1-millivolt recorder and integrator. The chromatographic column shall be made of 1/4-inch outside diameter stainless steel tubing and shall have a length of 8 feet. The column shall be packed with 80 to 100 mesh "Porapak Q."

4. MATERIAL

4.1 Materials required for this test are as follows:

(a) Erlenmeyer flasks, 125-milliliter (ml), with rubber stoppers.

(b) Syringe, 50-microliter.

(c) Flow meter, 10-cubic centimeter (cc).

(d) Serum bottles, 30-ml capacity, with rubber stoppers.

(e) Volumetric pipets, 25-ml and 50-ml.

(f) Shaker, horizontal (for flasks).

(g) Acetone, reagent grade.

(h) Methyl ethyl ketone, certified reagent grade (Fisher Scientific Co. No. M-209 or equal), 1 gallon.

(i) Sec-butyl alcohol, reagent grade (Eastman Organic Chemicals No. 943 or equal), 1 gallon.

(j) Molecular sieves, type 4A, 1/16-inch pellets.

(k) Ethyl alcohol, absolute, dried.

(l) Water, distilled.

(m) Diethyl ether, reagent grade, dried.

(n) Helium, commercial grade.

(o) Balance accurate to 0.2 milligram (mg).

5. PROCEDURE

5.1 Preparation of extraction solution. Dry the extraction solvents by adding 1-inch layers of molecular sieves directly to the gallon containers of methyl ethyl ketone and sec-butyl alcohol 2 days prior to mixing. Prepare a solution of 1 part by volume dry methyl ethyl ketone to 3 parts by volume dry sec-butyl alcohol. Add a 1-inch layer of molecular sieves to the container of the mixed solvents.

5.2 Preparation of standard. Pipet 30 ml of the dry, mixed solvents into a 30-ml serum bottle, stopper, and weigh to the nearest 0.2 mg. Using a suitable syringe, inject through the rubber stopper approximately 0.10 ml each of distilled water, dry ethyl alcohol, and diethyl ether. Reweigh the bottle to the nearest 0.2 mg after each injection to determine the weight of each component added. Be careful not to get any of the solvents in the stopper during injection. Record the weight of each component.

5.3 Extraction. Add approximately 10 g of specimen, weighed to the nearest 0.2 mg, to a 125-ml Erlenmeyrer flask and stopper immediately. Pipet 50 ml of the extracting solvent solution prepared in 5.1 into the flask and immediately stopper the flask. Place the flask on the horizontal shaker at low speed and ambient conditions to extract any solvents from the specimen. Extract for at least 2 hours. Remove the flask from the shaker and allow 15 minutes for most of the solids to settle.

5.4 Chromatography. Check the instrument settings and make any necessary adjustments to obtain the following conditions:

(a) Injection port temperature of 160° C.

(b) Oven temperature of 150°C.

(c) Detector temperature of 180° C.

(d) Bridge current of 200 milliamperes.

(e) Helium flow rate of 60 cc per minute.

(f) Helium inlet pressure of 50 psig.

Without disturbing the settled solids, sample 20 microliters (free of bubbles) of the liquid portion of the sample with a 50-microliter syringe that has been cleaned with acetone and dried with forced air. Wipe the tip of the syringe with a tissue and then draw up 1.0 microliter of air. Immediately inject the sample into the gas chromatograph and allow the component peaks to evolve at their respective attenuations. See figure 1. The sequence of separation of the components will be air, water, ethyl alcohol, impurity from methyl ethyl ketone (only seen when chromatograph is set at high sensitivity), diethyl ether, and extraction solvent mixture. Depending upon specimen concentration, it may be necessary to change the attenuation in order to keep the component peaks on the chart. It is not necessary to keep the methyl ethyl ketone - sec-butyl alcohol peak on the chart. The attenuation should never be changed during the evolving of a peak; and if possible, it is desirable to preattenuate so that a peak between 30 to 95 percent of the chart can be obtained. At times it may be necessary to rezero the recorder and integrater after switching attenuator settings or after evolution of a component. True zero is when the integrator runs in a straight line. Record on the chart the specimen identification, the attenuation, and the integrator reading of each component peak.

5.5 Standard and water correction tests. Repeat the procedure in 5.4 using the standard prepared in 5.2. Repeat the procedure in 5.4 using the methyl ethyl ketone - sec-butyl alcohol solution at an attenuation of 1X to obtain the water correction (if necessary).

6. CALCULATIONS

6.1 Calculate the percent by weight alcohol, ether, water, and total volatiles as follows:

$$\text{Percent alcohol} = \frac{(A)(B)(W_1)(100)(E)}{(C)(D)(W_2)}$$

$$\text{Percent ether} = \frac{(A)(B)(W_1)(100)(E)}{(C)(D)(W_2)}$$

$$\text{Percent ether} = \frac{(AB - FG)\ (W_1)(100)(E)}{(CD - FG)\ (W_2)}$$

Percent total volatiles = Percent alcohol + Percent ether + Percent water

Where:
- A = Attenuator setting for specimen extract
- B = Area of peak for specimen extract
- C = Attenuator setting for standard
- D = Area of peak for standard
- W_1 = Grams of component per 25 ml of standard
- W_2 = Specimen weight

E = Ratio of solvent between specimen and standard (equals 2 when 50 ml is used for specimen and 25 ml is used for standard)
F = Attenuator setting for water in the extraction solvent
G = Area of peak for water in the extraction solvent.

7. EVALUATION

7.1 Moisture and volatile matter content determination of pyrotechnic mixtures is required so that subsequent evaluations of potential hazards of the pyrotechnic mixtures can be correlated to the amount of moisture and volatiles in the specimen.

8. REFERENCES

(a) MIL-STD-286B, method T103.5

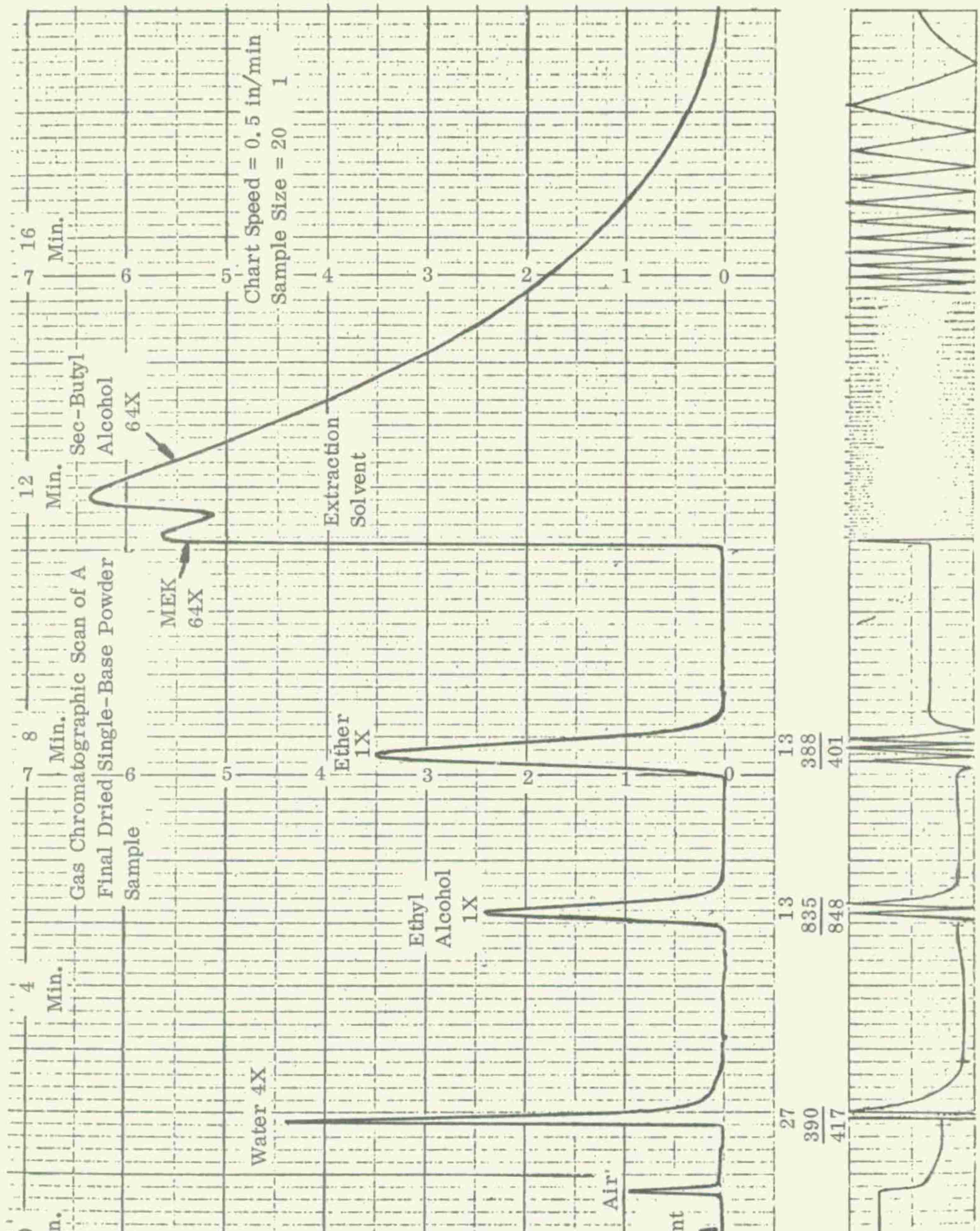


Figure 1. A Typical Gas Chromatographic Scan

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