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November 1970

FINAL REPORT ON THE DEVELOPMENT OF PELLETIZED HEAT SOURCES FOR THERMAL BATTERIES

UNIDYNAMICS PHOENIX, INC.

Phoenix, Arizona

FOR

SANDIA LABORATORIES, ALBUQUERQUE

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DEVELOPMENT OF PELLETIZED
HEAT SOURCES FOR
THERMAL BATTERIES

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I. INTRODUCTION

Under the terms of Sandia Purchase Order No. 25-8652, Unidynamics/Phoenix conducted a one-year study leading to the development of pelletized heat sources for thermal batteries. The purpose of this study was to further characterize the existing NX-1000 iron/potassium perchlorate heat powder and to improve the test equipment and techniques. The overall goal of the program was to collect the data necessary to revise and upgrade the present specification, "Preparation, Conductive Heat Powder, Thermal Battery", Sandia Drawing No. SS265866, Issue F, plus ACO's 312158-66P4 and 312158-68P4.

The program consisted of eight specific areas of investigation. These were:

- Calorimetry
- Ignition Sensitivity Testing
- Potassium Perchlorate Investigations
- Heat Powder Blending
- Pellet Density Versus Ignition Sensitivity
- Pellet Density Versus Pellet Breaking Strength
- Pellet Strength
- NX-1000 Iron Powder Evaluation

This report contains a summary of the major findings in each of these areas and presents Unidynamics' conclusions and recommendations.

II. TEST METHODS AND EQUIPMENT

A. General

The following heat powder parameters were measured routinely during the program.

- 1) calorific output
- 2) gas evolution
- 3) pellet breaking strength
- 4) ignition sensitivity
- 5) linear burn rate

During the course of the program, the methods and/or equipment used in three of these tests were modified. In each instance, the modification was made in the interest of increased accuracy and precision in data collection. The following paragraphs provide brief summary descriptions of the methods and equipment used in the five tests. More detailed descriptions of procedures may be found in Appendix D of this report.

B. Calorific Output

At the beginning of the program, calorific output determinations were performed using a Parr Model 1221 oxygen bomb calorimeter equipped with two matched mercury thermometers. However, measurement precision was not good, primarily due to the mass of the bomb. For this reason, one of the first program objectives was the evaluation of the smaller Model 1411 calorimeter, which in terms of precision, was found to be superior to the 1221 calorimeter for our use.

Later in the program, a further increase in both precision and accuracy was obtained with the substitution of a Hewlett-Packard Model 2801A quartz thermometer for the mercury thermometer. Subsequently, the testing of a five-gram sample of material in a Model 1411 calorimeter equipped with a quartz thermometer was selected as the standard method for determin-

ing calorific output.

C. Gas Evolution

During all previous heat powder programs at Unidynamics/Phoenix, gas evolution was determined by burning a weighed sample of material in a bomb and measuring the pressure developed after the temperature of the apparatus had been stabilized at 100°C. The specific gas evolution of the sample at STP conditions was then calculated. Several disadvantages to this method had become apparent even before the current program; and consequently an improved test, one which would be faster, easier to perform, and yield better precision was developed.

The improved test consisted of burning a weighed sample of heat powder in a bomb and measuring the volume of mercury which the gases displaced from a manometer tube at a known pressure. Both the bomb and the manometer were stabilized at ambient temperature (approximately 23°C). The gas evolution of the sample at STP conditions was then calculated. The manometric gas evolution procedure, which proved faster and more precise than the previously used pressure-dependent method, was selected as the method of choice for the current program.

D. Pellet Breaking Strength

The pellet breaking strength test was not altered during the current program. It consisted of measuring the force, in pounds, required to break a 1.25-inch-diameter heat pellet secured in a specially designed test fixture which supported approximately one-half of the pellet's surface. The test consisted principally of applying an increasing force to the pellet at a point not supported by the fixture. The force was continually monitored by a Chatillon dial push-pull gauge, and the gauge reading at the moment of pellet breakage was recorded.

E. Ignition Sensitivity

The capacitor discharge ignition sensitivity tester was modified in two significant respects during the current program. These included: 1) the addition of two new capacitance levels (0.35 microfarads and 0.65 microfarads), and 2) the incorporation of a heliarc-type gas shroud around the electrode to permit the use of an argon flow over the electrode and pellet during testing. The useful energy levels available after the addition of the new capacitors were 0.125, 0.175, 0.250, 0.325, 0.500 and 1.000 joules.

The standard test involved determining the ignition sensitivity of 0.50-inch-diameter heat pellets consolidated to a density of $3.50 \pm 0.03 \text{ gm/cm}^3$. The energy for 50% probability of ignition was determined by subjecting groups of 20 pellets each to the stimulus of selected energy levels and performing a probit analysis of the response obtained at the various levels.

The addition of the new capacitance levels significantly increased the statistical validity of the data. This was clearly shown by the higher correlation coefficients which resulted when the data from the modified tester was probited. The use of a 10 SCFH (standard cubic feet per hour) argon flow also appeared to enhance the reproducibility of the ignition sensitivity values obtained, possibly due to the exclusion of oxygen and atmospheric moisture from the area over and around the test pellets. Both modifications in the capacitor discharge tester were considered highly beneficial and were incorporated into the final design of the apparatus.

F. Linear Burn Rate

The linear burn rate test remained unchanged from that used previously. It consisted of measuring the time required for the burning front on a 1.25-inch-diameter heat pellet to cover the distance between two General Electric L9U

light-activated silicon rectifiers located 0.625 inches apart. The pellet was ignited electrically, and a Hewlett-Packard 523C electronic counter measured the time between triggering of the two light-activated rectifiers. The linear burn rate was calculated in inches/second by dividing 0.625 by the burn time in seconds.

III. AREAS OF INVESTIGATION

A. Calorimetry

1. Calibration of the Parr 1411 Calorimeter

One of the objectives of the program was to calibrate the Parr 1411 calorimeter and evaluate it for use with heat powder. During the course of this evaluation, three series of calibration tests were performed.

The initial calibration was performed using nominal 350 cal/gm Zr/BaCO₄ reference heat source material (SRM 1651) supplied by the National Bureau of Standards. This material was dried for two hours at 71°C under vacuum prior to use. Five calibration tests were conducted over a period of five days, with the following results.

<u>Sample Number</u>	<u>Water Equivalent (cal/°C)</u>
1	522.9
2	522.8
3	522.3
4	521.9
5	522.7

Average - 522.5 cal/°C

Standard deviation - 0.4 cal/°C

Numerous problems were experienced during the first series of calibration tests because of temperature variations in the area in which the calorimeter was located. For this reason, the instrument was relocated to an area with better temperature control. This necessitated recalibration.

The second calibration was performed using nominal 390 cal/gm Zr/BaCrO₄ reference heat source material (SRM 1652) also supplied by the National Bureau of Standards. This material was dried for 18 hours at 71°C under vacuum prior to use. A series of ten tests was conducted over a period of nine days,

with the following results.

<u>Sample Number</u>	<u>Water Equivalent (cal/°C)</u>
1	520.2
2	518.7
3	518.0
4	521.7
5	521.2
6	518.5
7	520.4
8	521.2
9	520.8
10	521.4
Average - 520.2 cal/°C	
Standard Deviation - 0.4 cal/°C	

Later in the program, it became necessary to calibrate the 1411 calorimeter a third time due to the installation of a Hewlett-Packard quartz thermometer probe in place of the mercury thermometer used previously. This calibration was performed using SRM 1652. Ten tests were conducted over a period of two days, with the following results.

<u>Sample Number</u>	<u>Water Equivalent (cal/°C)</u>
1	520.97
2	519.81
3	520.16
4	520.20
5	519.76
6	521.34
7	519.11
8	520.83
9	521.50
10	520.14
Average - 520.38 cal/°C	
Standard Deviation - 0.76 cal/°C	

2. Comparison of the Mercury and Quartz Thermometers

A series of calorific output determinations was conducted to compare the accuracy and precision of the mercury

thermometer with that of the quartz thermometer. These tests were performed using an 88/12 NX-1000 iron/potassium perchlorate composition.

Initially, five calorific output determinations were conducted using the quartz thermometer. The results were as follows:

<u>Sample Number</u>	<u>Calorific Output (cal/gm)</u>
1	220.36
2	220.55
3	221.03
4	220.19
5	220.62
Average	- 220.55 cal/gm
Standard Deviation	- 0.32 cal/gm

The water equivalent value used in these tests was 520.38 cal/°C.

The mercury thermometer was then reinstalled in the calorimeter, and five calorific output determinations were conducted on the same composition. The results were as follows:

<u>Sample Number</u>	<u>Calorific Output (cal/gm)</u>
1	219.9
2	220.3
3	219.8
4	220.9
5	221.4
Average	- 220.5 cal/gm
Standard Deviation	- 0.7 cal/gm

The water equivalent value used in these tests was 520.2 cal/°C.

These tests indicated that there was no significant difference in the accuracy of the quartz and mercury thermometers. However, based on a comparison of the standard deviation values, it was apparent that the quartz thermometer offered greater precision than the mercury thermometer.

3. Comparison of the Parr 1221 and 1411 Calorimeters

Early in the program, the accuracy and precision of the Parr 1221 and 1411 calorimeters were compared. Samples of 90/10, 88/12 and 86/14 NX-1000 iron/potassium perchlorate heat powders were used in this study. These three compositions were first tested in a 1221 calorimeter which had been calibrated with NBS benzoic acid. Then the same materials were tested in the 1411 calorimeter after its calibration with SRM 1651. The data obtained in these tests is summarized below.

<u>Composition</u>	<u>Calorific Output (cal/gm)</u>	
	<u>1221 Calorimeter</u>	<u>1411 Calorimeter</u>
90/10	183.1 $\pm$ 1.1	183.4 $\pm$ 0.6
88/12	218.3 $\pm$ 1.2	220.1 $\pm$ 0.6
86/14	257.9 $\pm$ 1.1	259.4 $\pm$ 1.0

NOTE: All tolerance values given in this table are equal to one standard deviation.

This data indicated that the 1411 calorimeter yielded better precision than the 1221 calorimeter. In addition, the values obtained using the 1411 calorimeter generally appeared to be slightly higher than those obtained with the 1221 calorimeter. The results of the comparison clearly confirmed that the 1411 calorimeter was the better instrument to use for determining the calorific output of heat powder.

4. Effects of Storage and Drying Conditions on the Calorific Output of Heat Powder

During the manufacture of thermal batteries, there are several operations during which the components of the battery are subjected to heat and/or vacuum for the purpose of removing or excluding moisture from the system. Since this results in the heat pellets being exposed to drying conditions, it was necessary to determine the effects of such conditions on the calorific output of iron/potassium perchlorate compositions. If any effect was observed, this would strongly indicate that a standard drying treatment should be used routinely on

heat powder prior to testing or use.

Three series of tests were conducted for the purpose of evaluating the effects of storage and/or drying conditions on the calorific output of 88/12 NX-1000 iron/potassium perchlorate heat powder.

In the first group of tests, samples of heat powder were exposed to various drying conditions for different periods of time, and the calorific output of each sample was then determined. The results of this study are summarized in Table 1.

The results of these initial tests indicated that none of the drying conditions evaluated had any pronounced effect on the calorific output of iron/potassium perchlorate heat powder with the possible exception of the six-week storage periods over the three desiccants.

In order to clarify the effects of long-term desiccation, two additional heat powder blends were prepared for evaluation. Both materials consisted of 88/12 NX-1000 iron/potassium perchlorate; however, one composition was prepared using potassium perchlorate which had been oven-dried at 120°C for 18 hours, while the other contained potassium perchlorate which had been taken directly from storage. Samples of each mixture were stored over the three desiccants for periods of twelve weeks, then tested to determine calorific output. Midway through the tests, at six weeks, samples of material were withdrawn from storage over anhydrous CaSO_4 and tested to determine calorific output. The purpose of these intermediate tests was to compare the results with those obtained previously under the same conditions.

A summary of the data obtained in the long-term desiccation studies is given in Table 2.

TABLE 1

Effects of Drying Conditions on the Calorific Output
of Heat Powder

Sample No.	Conditioning	Calorific Output (cal/gm)	Change in Calorific Output (cal/gm)
1-A	None	221.9 ±0.3	----
2-A	Desiccation over P ₂ O ₅ - three weeks	221.6 ±0.5	-0.3
2-B	Desiccation over P ₂ O ₅ - six weeks	220.5 ±0.6	-1.4
3-A	Desiccation over silica gel - three weeks	221.2 ±0.8	-0.7
3-B	Desiccation over silica gel - six weeks	219.8 ±0.8	-2.1
4-A	Desiccation over anhydrous CaSO ₄ - three weeks	221.8 ±0.7	-0.1
4-B	Desiccation over anhydrous CaSO ₄ - six weeks	220.1 ±0.8	-1.8
5-A	70°C - four days	221.0 ±0.6	-0.9
6-A	23°C and 10 mm of Hg - four days	221.1 ±0.8	-0.8
7-A	70°C and 10 mm of Hg - twenty-four hours	220.9 ±0.9	-1.0

NOTE: All tolerance values given in this table are equal to one standard deviation.

TABLE 2

Effects of Long-Term Desiccation on the Calorific
Output of Heat Powder

Sample No.	Conditioning	Calorific Output (cal/gm)	Change in Calorific Output (cal/gm)
1-A ¹⁾	None	222.1 ±0.8	----
1-B	Anhydrous CaSO ₄ - six weeks	220.3 ±0.6	-1.8
1-C	Anhydrous CaSO ₄ - twelve weeks	219.9 ±0.4	-2.2
1-D	P ₂ O ₅ - twelve weeks	220.3 ±0.4	-1.8
1-E	Silica gel - twelve weeks	219.8 ±0.4	-2.3
2-A ²⁾	None	221.9 ±0.7	----
2-B	Anhydrous CaSO ₄ - six weeks	220.2 ±0.1	-1.7
2-C	Anhydrous CaSO ₄ - 12 weeks	219.9 ±0.4	-2.0
2-D	P ₂ O ₅ - twelve weeks	219.8 ±0.4	-2.1
2-E	Silica gel - twelve weeks	219.2 ±0.2	-2.7

NOTE: All tolerance values given in this table are equal to one standard deviation.

1) KClO₄ taken directly from storage.

2) KClO₄ dried at 120°C for 18 hours prior to use.

Analysis of the data from the long-term desiccation studies, as well as that obtained during the first series of tests, disclosed that a statistically significant loss in calorific output occurred in 88/12 NX-1000 iron/potassium perchlorate compositions stored over anhydrous CaSO_4 , P_2O_5 or silica gel for periods of six weeks or longer. Furthermore, in both series of tests, silica gel always caused a greater drop in calorific output than either of the other two desiccants evaluated. The loss in calorific output which occurred after only three weeks of desiccation was shown not to be statistically significant. In addition, the use of oven-dried potassium perchlorate did not appreciably affect the loss in calorific output occurring during long-term desiccation.

Later in the program, a third series of tests was performed to evaluate the effects of selected drying cycles or storage conditions on the calorific output of heat powder. This new test series was considered necessary for two reasons: 1) in all previous tests, a mercury thermometer had been installed in the calorimeter, rather than the more precise quartz thermometer and 2) in the earlier tests, particularly those involving long-term desiccation, the procedures evaluated had not been practical for routine laboratory use. In the third series of tests, the specific objective was to evaluate several practical drying cycles, one of which was to be selected for routine use on heat powders prior to testing.

The composition used in this third test series was an 88/12 NX-1000 iron/potassium perchlorate mixture prepared using oven-dried potassium perchlorate. The data obtained for this composition before and after exposure to several drying cycles and storage conditions is shown in the table on the following page.

Sample No.	Conditioning	Calorific Output (cal/gm)	Change in Calorific Output (cal/gm)
1	None	221.96 $\pm$ 0.67	---
2	Ambient - 20 hours	221.17 $\pm$ 0.59	-0.79
3	70°C and 10 mm of Hg - two hours	221.52 $\pm$ 0.45	-0.44
4	70°C and 10 mm of Hg - 16 hours	220.59 $\pm$ 0.63	-1.37
5	Desiccation over anhydrous CaSO ₄ - 48 hours	220.73 $\pm$ 0.42	-1.23

NOTE: All tolerance values given in this table are equal to one standard deviation.

Based on this series of tests, it was concluded that all heat powder samples should be dried for 16 hours at 70°C and 10 mm of Hg prior to conducting calorific output determinations.

B. Ignition Sensitivity Testing

1. Modification of the Capacitor Discharge Ignition Sensitivity Tester

Two modifications were made in the capacitor discharge ignition sensitivity tester. These consisted of the addition of two new capacitance levels (0.35 microfarads and 0.65 microfarads) and the incorporation of a heliarc gas shroud to permit a flow of argon around the electrode during testing. A third modification involved replacing the tungsten carbide probe with a pure tungsten electrode from a heliarc welder. However, the pure tungsten electrode proved to be too brittle, so the tungsten carbide probe was reinstalled.

2. Evaluation of the Modified Capacitor Discharge Ignition Sensitivity Tester

In an effort to determine the effect of the argon flow, the arc was photographed before and after modification

of the tester. In each series of tests, the arcs from ten consecutive discharges were photographed. In the first series, no gas flow was used. In the second series, an argon flow of 10 SCFH (standard cubic feet per hour) was employed.

A constant one-microfarad capacitance level was used in all tests with pellets of NX-1000 iron powder substituted for heat pellets. The photographs were taken at a magnification of approximately 22X. The ten arc photographs taken before and after the modification of the tester are shown in Figures 1 and 2, respectively.

In both series of tests, the outlines of the arc were rather poorly defined, indicating that it was not localized, but covered a broad area below the tip. Some variations in the magnitude of the arc from test to test were observed, particularly in the first series of tests, which was conducted with no argon flow. In this series, a noticeably larger, brighter arc, accompanied by a distinctly audible "crack", was observed in tests 7 and 9. In these two tests, the arc was bright enough to cause a reflection from the Plexiglas side of the firing box. These reflections are clearly visible in Figure 1, photographs 7 and 9.

In general, it was observed that the arc discharged while the argon flow was in operation was noticeably smaller and dimmer than that produced without the argon flow. Since the capacitance and voltage settings remained unchanged throughout both series of tests, the argon flow apparently had an affect on the luminosity of the arc.

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FIGURE 1

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FIGURE 2

Color photographs were taken of the arc both with and without the argon flow in operation. They disclosed that the flash surrounding the arc discharged in argon was blue, with the arc itself a brilliant bluish-white. There was no indication of any red, orange or yellow color in or around the arc and no evidence of any burning iron particles. By comparison, the color photographs taken of the arc without the argon flow showed an appreciable fraction of yellow and orange around the probe tip and numerous glowing particles of iron which had been ejected from the pellet by the impact of the arc. The absence of these colors from the arc discharged in argon was considered responsible for its relative dimness.

From the photographs, it was not possible to detect any definite advantage to be gained from the use of an inert gas flow around the electrode. However, subsequent investigations disclosed that the tungsten carbide tips on the electrodes did not erode as rapidly when the argon flow was employed, presumably because the inert gas prevented or retarded their oxidation. For this reason, the use of a 10 SCFH argon flow was made standard practice in all ignition sensitivity testing.

In addition to comparing photographs of the arc produced by the capacitor discharge ignition sensitivity tester before and after modification, the modified apparatus was also evaluated by conducting ignition sensitivity tests. For these tests, an 88/12 iron/potassium perchlorate composition designated ELB 19753 was prepared using the same lot of NX-1000 iron powder that had been employed in a previous formulation (N 57644) which had been used to calibrate the tester prior to modification. By comparing the results obtained for the new lot of heat powder with those which had been obtained previously for N 57644, it was felt that a comparison of the ignition sensitivity

tester before and after modification, as well as a calibration of the apparatus in its modified form, could be obtained. A second objective of these tests was to determine the effect of a 10 SCFH argon flow on the ignition sensitivity of heat pellets.

Two series of tests were conducted, and the results are summarized in Table 3. Also included in this table are comparison values for N 57644. In both series of tests, the pellets used consisted of 0.50-inch-diameter pellets of ELB 19753 consolidated to a density of $3.58 \pm 0.03 \text{ gm/cm}^3$, the same density to which the N 57644 pellets tested previously had been consolidated.

In the first group of tests, a pure tungsten electrode was used on the capacitor discharge apparatus. This led to several very serious problems, particularly in those tests in which no argon flow was used. The most persistent of these problems were caused by the fact that the tungsten in the electrode oxidized very rapidly in the absence of an inert atmosphere. This resulted in extensive damage to the tips of the electrodes and was suspected of contributing to variations in the arc. After two or three firings, the tip would be burned away so badly that its surface would be rough and irregular and covered with a heavy coating of a yellow oxide of tungsten (probably WO_3). In some instances, particles of this material actually fell onto the surface of a pellet. Regardless of whether the argon flow was used or not, after several firings, the tip would become so brittle that it could not be sharpened without breaking. On several occasions, electrodes actually shattered during a test. Another problem related to the oxidation and resulting irregularity of the tip was multiple arcing. Examination of the pellets which failed to ignite revealed that

TABLE 3
Comparison of Ignition Sensitivity Characteristics
of Heat Pellets Tested Under Different Conditions

	N 57644	ELB 19753 Tested with a Tungsten Electrode		ELB 19753 Tested with a Tungsten Carbide Probe	
		<u>No Argon Flow</u>	<u>10 SCFH Argon Flow</u>	<u>No Argon Flow</u>	<u>10 SCFH Argon Flow</u>
<u>Probability of Ignition (%)</u>	<u>Energy Level (joules)</u>	<u>Energy Level (joules)</u>	<u>Energy Level (joules)</u>	<u>Energy Level (joules)</u>	<u>Energy Level (joules)</u>
1	0.353	-0.125	0.046	-0.015	0.133
5	0.386	0.139	0.275	0.122	0.224
10	0.404	0.280	0.397	0.195	0.273
25	0.434	0.516	0.601	0.317	0.354
50	0.467	0.778	0.828	0.453	0.444
75	0.500	1.104	1.105	0.588	0.533
90	0.530	1.127	1.126	0.710	0.614
95	0.548	1.141	1.138	0.783	0.663
99	0.581	1.168	1.161	0.920	0.754
Correlation Coefficient	0.891	0.957	0.988	0.987	0.964

some of them contained as many as three small burned spots where arcs had struck. With these factors all considered, the results of the first series of tests were not considered indicative of the true ignition sensitivity of the pellets being evaluated.

In view of the problems encountered with the pure tungsten electrode, a second series of ignition sensitivity tests was conducted with the tungsten carbide probe reinstalled. This change eliminated the problems of oxidation and breakage of the electrode.

Analysis of the results of the two series of tests disclosed that the addition of the two new capacitance levels (0.35 microfarads and 0.65 microfarads) had increased the statistical validity of the data, as evidenced by the higher correlation coefficients of the ELB 19753 probits compared to that of the recomputed N 57644 probit. The two 50% ignition energy values obtained for ELB 19753 tested with a tungsten carbide probe were not only very similar to each other, but were also very similar to the 50% ignition energy value previously determined for N 57644. This clearly indicated that the values obtained using the modified tester were comparable to those obtained with the apparatus prior to its modification and, furthermore, that the use of the argon flow did not significantly affect the results. However, it was apparent that the type of electrode used was very critical and, as stated previously, the tungsten carbide probe was selected on the basis of its durability. Following the second series of tests, the capacitor discharge ignition sensitivity tester was considered calibrated.

C. Potassium Perchlorate Investigations

1. Effects of Storage Conditions on the Particle Size of Potassium Perchlorate

To aid in establishing acceptable storage conditions for ground potassium perchlorate, a study was

conducted to determine the effects of a variety of environmental conditions on the particle size of this material. Immediately prior to the test, the potassium perchlorate was ground to an initial mean particle diameter of 5.8μ , as determined by the Eagle-Picher turbimeter.

The material was divided into 30 samples which were conditioned as follows:

Set I-A KClO_4 oven-dried at 120°C for 64 hours, then exposed to normal room conditions (23°C and 20-50% R.H.) in:

- a) an open container
- b) a sealed container (cover taped on to form an air-tight seal)
- c) a capped container (cover threaded on and hand tightened using normal pressure)

Set I-B KClO_4 not dried, then exposed to normal room conditions (23°C and 20-50% R.H.) in:

- a) an open container
- b) a sealed container (cover taped on to form an air-tight seal)
- c) a capped container (cover threaded on and hand tightened using normal pressure)

Four additional double sets of KClO_4 samples (one set oven-dried, the other set not dried) were placed in identical types of containers as those used for Sets I-A and I-B, then subjected to the following storage conditions.

Set II-A and II-B — Stored in an oven maintained at a constant temperature of 120°C .

Set III-A and III-B — Stored in a chamber with 66% R.H.

Set IV-A and IV-B — Stored in a chamber with 98% R.H.

Set V-A and V-B — Stored in a desiccator over phosphorus pentoxide (P_2O_5).

The average particle size of each of the 30 samples was determined after 35 days, 75 days and 115 days. The data obtained is shown graphically in Figures 3, 4, 5, 6 and 7.

Analysis of the data from the potassium perchlorate particle size investigations led to several conclusions.

- 1) Ground $KClO_4$ can be stored under normal room conditions (approximately $23^{\circ}C$ with 20-50% relative humidity) in either an open or a closed container for at least 115 days (approximately four months) with no significant change in its average particle size.

- 2) Ground $KClO_4$ can also be stored in an oven at $120^{\circ}C$ or in a very low humidity environment, as in a desiccator over P_2O_5 , for at least 115 days with no significant change in its average particle size. However, there is no apparent advantage to be gained from using these stringent storage conditions. The small particle size of the material is protected just as well by storing it in a closed container under normal room conditions.

- 3) When ground $KClO_4$ is stored in an environment containing either 66% or 98% relative humidity, its average particle size increases significantly. Furthermore, the rate of increase is much higher at the higher humidity level.

**KClO₄ OVEN DRIED AT 120°C FOR 64 HOURS,
THEN EXPOSED TO NORMAL ROOM CONDITIONS**

**KClO₄ NOT DRIED,
THEN EXPOSED TO NORMAL ROOM CONDITIONS**

SET I

LEGEND:
 AN OPEN CONTAINER
 — A SEALED CONTAINER
 (cover taped on to form an air tight seal)
 - - - A CAPPED CONTAINER
 (cover threaded on and hand tightened
 using normal pressure)

Figure 3

**KClO₄ OVEN DRIED AT 120°C FOR 64 HOURS,
THEN STORED IN AN OVEN MAINTAINED AT
A CONSTANT TEMPERATURE OF 120°C**

**KClO₄ NOT DRIED, THEN STORED
IN AN OVEN MAINTAINED AT
A CONSTANT TEMPERATURE OF 120°C**

SET II

LEGEND:

- AN OPEN CONTAINER
- A SEALED CONTAINER
(cover taped on to form an air tight seal)
- · - · - A CAPPED CONTAINER
(cover threaded on and hand tightened using normal pressure)

Figure 4

**KClO₄ OVEN DRIED AT 120°C FOR 64 HOURS,
THEN STORED IN A CHAMBER
WITH 66% RELATIVE HUMIDITY**

**KClO₄ NOT DRIED, THEN STORED IN A CHAMBER
WITH 66% RELATIVE HUMIDITY**

SET III

LEGEND:
 AN OPEN CONTAINER
 — A SEALED CONTAINER
 (cover taped on to form an air tight seal)
 - · - A CAPPED CONTAINER
 (cover threaded on and hand tightened
 using normal pressure)

Figure 5

**KClO₄ OVEN DRIED AT 120°C FOR 64 HOURS,
THEN STORED IN A CHAMBER
WITH 98% RELATIVE HUMIDITY**

**KClO₄ NOT DRIED, THEN STORED
IN A CHAMBER WITH 98% RELATIVE HUMIDITY**

SET IV

Figure 6

**KClO₄ OVEN DRIED AT 120°C FOR 64 HOURS,
THEN STORED IN A DESICCATOR
OVER PHOSPHORUS PENTOXIDE**

**KClO₄ NOT DRIED, THEN STORED
IN A DESICCATOR
OVER PHOSPHORUS PENTOXIDE**

SET V:

LEGEND:

- AN OPEN CONTAINER
- A SEALED CONTAINER
(cover taped on to form an air tight seal)
- · - · - A CAPPED CONTAINER
(cover threaded on and hand tightened using normal pressure)

Figure 7

4) In general, oven-drying ground KClO_4 at 120°C for 64 hours prior to storage has little or no effect on subsequent particle size changes which occur in the material during storage.

5) The "sealed" containers used in the tests were not hermetically sealed, as evidenced by the fact that samples of KClO_4 stored in these containers under high humidity conditions underwent increases in average particle size nearly as great as those of samples stored in open containers under the same humidity conditions. However, the method used to seal these containers (several wraps of friction tape) was identical to that normally employed in production operations, so they were representative of "sealed" potassium perchlorate storage containers.

2. Effects of Potassium Perchlorate Particle Size on the Properties of Heat Powder

To aid in selecting an allowable particle size range for potassium perchlorate for use in production heat powder, a study was conducted to determine the effects of variations in the particle size of this material on the calorific output, gas evolution, pellet breaking strength, ignition sensitivity and linear burn rate of 88/12 NX-1000 iron/potassium perchlorate compositions. Potassium perchlorate of the following nominal particle sizes was evaluated: 2.5μ , 5μ , 7.5μ , 10μ , 15μ and 20μ . The same lots of NX-1000 iron powder and potassium perchlorate were used throughout the study. The potassium perchlorate was dried for a minimum of four hours at 120°C prior to use, and all mixing operations were performed under controlled conditions of temperature and relative humidity. After blending, each composition was stored over anhydrous CaSO_4 for 64 hours prior to conducting calorific output determinations.

TABLE 4
Effects of Potassium Perchlorate Particle Size on the
Properties of Heat Powder

Composition Number	Calorific Output (cal/gm)	Gas Evolution (ml/gm)	Consolidation Pressure Required to Obtain a Nominal Density of 3.45 gm/cm ³ (psi)	Pellet ¹⁾ Breaking Strength at a Nominal Density of 3.45 gm/cm ³ (pounds)	Energy Level Required for 50% Probability of Ignition (joules)	Linear Burn Rate (in/sec.)
ELB 22117 2.5μ KC10 ₄	219.54 ± 0.55	0.72 ± 0.10	14,000	1.02	0.280	2.532 ± 0.049
ELB 22126 5μ KC10	221.38 ± 0.24	0.68 ± 0.14	13,000	1.01	0.274	3.065 ± 0.049
ELB 22193 7.5μ KC10 ₄	221.49 ± 0.48	0.60 ± 0.02	14,300	1.07	0.351	3.347 ± 0.130
ELB 22141 10μ KC10 ₄	221.20 ± 0.55	0.73 ± 0.06	13,800	0.98	0.415	3.381 ± 0.207
ELB 22156 15μ KC10 ₄	221.65 ± 0.82	0.65 ± 0.14	13,800	1.06	0.810	4.133 ± 0.064
ELB 22174- 15G 15μ KC10 ₄ (grown)	221.35 ± 1.00	0.57 ± 0.04	14,300	1.10	0.945	3.235 ± 0.401
ELB 22163 20μ KC10 ₄	219.70 ± 0.92	0.54 ± 0.19	13,800	1.24	2.028	4.388 ± 0.732

¹⁾ 1.25-inch-diameter pellet.

NOTE: All tolerance values given in this table are equal to one standard deviation.

All of the potassium perchlorate samples used in the study were obtained by grinding the raw material in a micropulverizer with the exception of a sample of 15-micron potassium perchlorate produced by "growing" the particles to the desired size in a controlled humidity environment.

The data obtained in the study relating potassium perchlorate particle size to the properties of heat powder is summarized in Table 4.

Evaluation of the data indicated that the particle size of the potassium perchlorate used in a heat powder blend had no significant effect on the calorific output or gas evolution of the material and little or no effect on its pellet breaking strength. However, it appeared to affect linear burn rate and very clearly influenced ignition sensitivity. The most pronounced effect was the sharp reduction in ignition sensitivity which occurred with increasing potassium perchlorate particle size, especially in the range of 15-20 μ .

The relationships of potassium perchlorate particle size to linear burn rate and ignition sensitivity are shown graphically in Figure 8.

D. Heat Powder Blending

A comparative evaluation of several types of blending equipment was conducted to determine the relative efficiency of each for the production of heat powder. The blenders evaluated were all modifications of either the V-blender or the twin-cone blender.

KClO_4 PARTICLE SIZE VERSUS
 PELLET IGNITION ENERGY (■)
 AND PELLET LINEAR BURN RATE (●●)

Figure 3

The blenders were evaluated by comparing the quality of the mixtures which they yielded at the end of a one-hour period of operation. The test consisted of mixing equal parts by weight of potassium perchlorate and red iron oxide in the blender for one hour, then removing a sample of the mixture for visual inspection and a streak test.

The two basic types of blending equipment evaluated in the tests were as follows:

- V-Blender - Nominal capacity - 300 grams (constant for all tests)
Rotation - 9.75 r.p.m. (constant for all tests)
- Twin-Cone
Blender - Nominal capacity - 150 grams (constant for all tests)
Rotation - 9.75 r.p.m. (constant for all tests)

The streak test was conducted by spreading a small portion of the test mixture with a spatula. This caused any small $KClO_4$ particles which were not readily apparent to leave a white or light-colored "streak".

In addition to the unmodified V-blender and the unmodified twin-cone blender, the following mixing equipment was evaluated.

- 1) V-Blender with a 1/4-inch-mesh stainless steel screen placed in each side arm at the midpoint, perpendicular to the side walls.
- 2) V-Blender with two one-inch-wide vanes inserted across the midpoint of each side arm of the blender (see sketch). The vanes were both

oriented 20 degrees from the longitudinal axis of the side arm and were mounted perpendicular to each other.

- 3) Twin-Cone Blender with a single one-inch-wide vane oriented 20 degrees from the vertical axis of the blender. This vane was mounted across the center of the blender.
- 4) Twin-Cone Blender with a grid of cross wires forming a 3/4-inch mesh mounted across the center of the blender.

Of the six types of mixing equipment evaluated, only the twin-cone blender with the 3/4-inch mesh grid across the center yielded a satisfactory blend after one hour. Since this blender appeared promising, it was evaluated to determine its efficiency in mixing 88/12 NX-1000 iron/potassium perchlorate. 132 grams of NX-1000 iron powder and 18 grams of potassium perchlorate were loaded into the blender, and it was operated for periods of 2, 4, 6 and 8 hours. Samples of the mixture were taken at the end of each of these time periods and checked for uniformity.

At the end of two hours, a streak test revealed some KClO_4 . Passing this mixture through a 60 mesh screen revealed that approximately two grams of the potassium perchlorate was still unmixed and in the form of very small agglomerates. At the end of four hours, no evidence of agglomerates was seen when the blend was passed through a 60 mesh screen. However, a streak test revealed that some of the potassium perchlorate was still unmixed. At the end of six hours, the blend appeared to be approaching a homogeneous state, since no agglomerates were visible, and a streak test revealed only very slight streaking. At the end of eight hours, a completely homogeneous mixture was obtained with no KClO_4 particles or agglomerates present.

It was concluded that the blending efficiency of the modified twin-cone blender was essentially equal to that of the rotary tumbler presently used for heat powder production, since both types of equipment require approximately eight hours to produce a satisfactory blend. Even with modifications, the V-blender did not produce a blend that was as homogeneous as that of the modified twin-cone blender.

With all factors considered, there appeared to be no reason to recommend changing from a rotary tumbler to a modified twin-cone blender for the production of large quantities of heat powder.

For laboratory preparations of heat powder, the present method of blending the ingredients by combining them and passing them six times through a 100-mesh sieve has proved very efficient, and no effort was expended in developing an alternate method.

E. Pellet Density Versus Ignition Sensitivity

A study was conducted to determine the effect of pellet density on the ignition sensitivity of 88/12 NX-1000 iron/potassium perchlorate heat powder. The pellet densities evaluated were 3.30, 3.40, 3.50, 3.60 and 3.70 gm/cm³. The nominal weight of each test pellet was 400 milligrams. The results of this study are summarized in Table 5 and shown graphically in Figure 9.

Table 5
Effect of Pellet Density on Ignition Sensitivity

<u>Nominal Pellet Density</u> <u>(gm/cm³)</u>	<u>Energy Required for 50%</u> <u>Probability of Ignition</u> <u>(joules)</u>
3.30	0.182
3.40	0.164
3.50	0.161
3.60	0.248
3.70	0.253

From the data obtained in this study, it appeared that two general levels of ignition sensitivity had been encountered in the test pellets, with a pronounced decrease in sensitivity occurring between pellets consolidated to a density of 3.50 gm/cm³ and those consolidated to a density of 3.60 gm/cm³. However, this phenomenon has not been observed previously in iron/potassium perchlorate heat pellets, and the indication of its occurrence in this series of tests may have been misleading.

The average thickness of the 400-milligram pellets used in these tests was only 0.035 inches, as compared to 0.055 inches for the 615-milligram pellets used in all previous tests. The effects of a reduction in pellet mass and/or thickness on the ignition sensitivity of iron/potassium perchlorate

PELLET DENSITY
VERSUS
IGNITION ENERGY

Figure 9

compositions has not been investigated. However, such effects may exist and could explain the apparent bilevel sensitivity characteristics of the 400-milligram pellets.

F. Pellet Density Versus Pellet Breaking Strength

A study was performed to determine the effect of density on the breaking strength of 88/12 NX-1000 iron/potassium perchlorate heat pellets. The pellet densities evaluated were 3.30, 3.40, 3.50, 3.60 and 3.70 gm/cm³. The nominal weight of each test pellet was 2.50 grams. The results of this study are summarized in Table 6 and shown graphically in Figure 10.

Table 6
Effect of Pellet Density on Breaking Strength

<u>Nominal Pellet Density</u> <u>(gm/cm³)</u>	<u>Required Consolidation</u> <u>Pressure (psi)</u>	<u>Average</u> <u>Breaking Strength</u> <u>(pounds)</u>
3.30	10,600	0.26
3.40	12,700	0.31
3.50	14,000	0.39
3.60	16,300	0.43
3.70	19,600	0.47

The data obtained in this study indicated that there was essentially a direct relationship between the density and breaking strength of 88/12 NX-1000 iron/potassium perchlorate heat pellets. This relationship has also been observed in previous studies.

G. Pellet Strength

1. Effects of Metal Grids on the Properties of
Heat Pellets

An investigation was conducted to determine the effects of metal grids on the density, breaking strength and

**PELLET DENSITY
VERSUS
PELLET STRENGTH**

Figure 10

ignition sensitivity of heat pellets. A sample of 7-2/0 Armco expanded metal (iron) manufactured by Ex Met Corporation of Bridgeport, Connecticut, was used as the grid material in this study. This material was cut into 1.25-inch-diameter and 0.50-inch inch-diameter discs for incorporation into the pellets.

It was determined by experimentation that the 1.25-inch-diameter pellets incorporating a metal grid should have a nominal weight of 2.63 grams to contain 2.50 grams of heat powder, while the 0.50-inch-diameter pellets incorporating a metal grid should have a nominal weight of 420 milligrams to contain 400 milligrams of heat powder.

Several 1.25-inch-diameter pellets were prepared with metal grids incorporated to determine the minimum density required for the pellets to withstand normal handling. This density was found to be 3.50 gm/cm^3 . Pellets consolidated to a lesser density developed areas of crumbling between the grid mesh, while pellets pressed to a greater density developed fractures along the face of the grid. This latter phenomenon was attributed to compression or buckling of the grid on consolidation, followed by a return to its original shape upon release of the forming pressure, causing the pellet surface to fracture.

It was determined that 1.25-inch-diameter pellets incorporating metal grids could be consolidated to a nominal density of 3.50 gm/cm^3 at a pressure of 15,800 psi. This compared to 14,000 psi for similar pellets without grids.

The average breaking strength of the pellets incorporating grids was 1.28 pounds, compared to 0.39 pounds for similar pellets without grids. In these tests, the breaking point was

taken as the first point at which any sign of breakage occurred. The pellets were actually still held together by the metal grids following the tests, but the consolidated layers of heat powder were fractured. The fractures that developed were of no set pattern or type and ranged from a clean linear break to a random crumbling of the pellet.

Ignition sensitivity tests were performed on 0.50-inch-diameter pellets consolidated to a density of 3.50 gm/cm^3 which incorporated metal grids. The tests were conducted with the modified capacitor discharge ignition sensitivity apparatus using a 10 SCFH argon flow. The energy required for 50% probability of ignition was found to be 0.337 joules, compared to 0.161 joules for similar pellets without grids.

From these tests, it was concluded that the incorporation of metal grids into 88/12 NX-1000 iron/potassium perchlorate heat pellets consolidated to a nominal density of 3.50 gm/cm^3 increased the breaking strength of the pellets approximately three times while reducing the ignition sensitivity to approximately one-half that of similar pellets not containing grids. From an overall standpoint, the use of metal grids did not appear to be advantageous.

2. Comparison of the Breaking Strengths of 3.80-gram and 2.50-gram Heat Pellets

Throughout most of the program, the 1.25-inch-diameter pellets which had been prepared had weighed 3.80 ± 0.05 grams. However, in the study relating pellet density to pellet breaking strength and the investigation of the effects of metal grids on the properties of heat pellets, 2.50-gram pellets had been used. For this reason, a test

was conducted to compare the average breaking strengths of 3.80-gram pellets and 2.50-gram pellets consolidated to equivalent densities. The density selected for comparison was 3.50 gm/cm^3 . The average breaking strengths of ten pellets each weighing 3.80 ± 0.05 grams and 2.50 ± 0.05 grams were 1.04 pounds and 0.39 pounds, respectively. Thus, the 3.80-gram pellets were approximately 2-1/2 times as strong as the 2.50-gram pellets.

H. NX-1000 Iron Powder Evaluation

1. Qualification Testing of NX-1000 Iron Powder, Lots 6 through 12

88/12 iron/potassium perchlorate blends were prepared using NX-1000 iron powder from Lots 6 through 12, and each composition was evaluated by a series of qualification tests similar to those anticipated for use in the revised heat powder specification. The following general procedures were employed.

a) Potassium Perchlorate - The same lot of KClO_4 was used in all compositions. The KClO_4 was ground to a mean particle diameter of 5.1μ , as determined using the Eagle-Picher turbimeter. After grinding, the KClO_4 was stored over a desiccant in a closed container and was oven-dried for a minimum of four hours at 120°C prior to use.

b) NX-1000 Iron Powder - All samples of iron powder used were passed through a 100-mesh sieve prior to blending with the KClO_4 to remove any foreign particulate matter.

c) Heat Powder Blending - The ingredients were weighed on a triple-beam balance accurate to 0.01 gram.

Blending was performed by passing the ingredients through a 100-mesh sieve together six times. Prior to blending, the relative humidity in the mixing area was determined. No mixing operations were performed unless the relative humidity was below the 50% limit established for production heat powder.

d) Determination of Calorific Output - Five calorific output tests were performed on each lot of heat powder. The nominal sample weight used was five grams. Each sample was dried at a temperature of $70 \pm 5^{\circ}\text{C}$ and a pressure of 10 mm of Hg for a minimum of 16 hours prior to testing. A Parr 1411 calorimeter with a quartz thermometer installed was used in all calorific output tests.

e) Determination of Gas Evolution - Five gas evolution determinations were performed on each lot of powder, using the manometric gas evolution apparatus. The sample weight used was five grams. All gas evolution values were reported in milliliters per gram corrected to standard temperature and pressure.

f) Consolidation of Pellets - All large pellets required for pellet breaking strength and linear burn rate tests weighed 3.80 ± 0.05 grams and were consolidated to a density of $3.50 \pm 0.03 \text{ gm/cm}^3$. All small pellets required for the ignition sensitivity tests weighed 615 ± 10 milligrams and were also consolidated to a density of $3.50 \pm 0.03 \text{ gm/cm}^3$. The nominal diameter of the large pellets was 1.25 inches, and the nominal diameter of the small pellets was 0.50 inches.

g) Determination of Pellet Breaking Strength - The standard pellet breaking strength test procedure was followed.

h) Determination of Linear Burn Rate - The standard linear burn rate test procedure was followed.

i) Determination of Ignition Sensitivity - The number of pellets required for conducting the ignition sensitivity test was determined by the response of the pellets when each group of 20 was subjected to one of the following energy levels: 0.125, 0.175, 0.250, 0.325, 0.500 and 1.000 joule. A sufficient number of pellet groups of each composition was tested to establish an all-fire level and a no-fire level for the material. The data was treated statistically to obtain a probit analysis. A 10 SCFH argon flow was used in all tests.

A summary of the data obtained in the qualification of the seven lots of NX-1000 iron powder is given in Table 7.

During qualification of the NX-1000 iron powder, significant discrepancies were encountered in the calorific output values obtained for several of the compositions. The cause of these discrepancies was not determined. However, it appeared to be related in some way to the 70°C vacuum drying treatment to which each lot of heat powder was exposed prior to determining its calorific output. Possibly, two or more factors were involved in causing these discrepancies, since the changes which occurred were not consistent in one direction. After exposure to 70°C and a pressure of 10 mm of Hg for 16 hours, the calorific outputs of some compositions had decreased, while those of others had increased. When certain of these discrepant values began to fall outside the tentative limits of the revised calorific output specification (221 ± 2 cal/gm), it became apparent that a problem might arise in the future in the qualification of production heat powder.

TABLE 7
Qualification Data for NX-1000 Iron Powder, Lots 6 Through 12

Composition Number	Calorific Output (cal/gm)	Gas Evolution (ml/gm)	Consolidation Pressure Required To Obtain a Nominal Density of 3.50 gm/cm ³ (psi)	Pellet ¹⁾ Breaking Strength at a Nominal Density of 3.50 gm/cm ³ (pounds)	Energy Required for 50% Probability of Ignition (joules)	Linear Burn Rate (in/sec)
ELB 23453 (Lot 6)	221.57 ± 0.55	0.63 ± 0.10	15,700	1.26	0.322	3.167 ± 0.055
ELB 23454 (Lot 7)	221.48 ± 0.57	0.60 ± 0.06	15,700	1.45	0.608	3.116 ± 0.048
ELB 23471 (Lot 8)	219.29 ± 0.71	0.23 ± 0.01	15,700	1.78	0.467	3.009 ± 0.050
ELB 23469 (Lot 9)	221.22 ± 0.84	0.17 ± <0.01	15,700	1.48	0.327	2.784 ± 0.090
ELB 23485 (Lot 10)	222.39 ± 0.20	0.32 ± 0.07	15,700	2.16	0.284	3.587 ± 0.258
ELB 23495 (Lot 11)	219.01 ± 0.23	0.22 ± 0.02	15,700	2.06	0.308	2.833 ± 0.063
ELB 23496 (Lot 12)	223.82 ± 0.70	0.12 ± 0.04	15,700	2.22	0.408	2.785 ± 0.062

¹⁾ 1.25-inch-diameter pellet.

NOTE: All tolerance values given in this table are equal to one standard deviation.

No discrepant calorific output values were encountered in the heat powder blends containing iron from Lots 6 and 7 (ELB 23453 and ELB 23454, respectively). However, the first composition prepared using Lot 8 iron, designated ELB 23468, yielded an average calorific output of only 217.03 cal/gm. This value did not agree with that established for an 88/12 NX-1000 iron/potassium perchlorate mixture, so the material was temporarily set aside. Although there was no reason to suspect that the constituents of ELB 23468 had been improperly weighed or blended, another 88/12 NX-1000/KClO₄ blend was prepared using Lot 8 iron powder. This composition was designated ELB 23471. While a 25-gram sample of ELB 23471 was being subjected to the vacuum drying procedure, two calorific output determinations were performed on a sample of ELB 23468 which had not been vacuum dried. The results of these tests were as follows:

<u>Sample Number</u>	<u>Calorific Output (cal/gm)</u>
1	222.57
2	221.82
Average - 222.20 cal/gm	

These results indicated that ELB 23468 was a true 88/12 iron/potassium perchlorate blend, so another 25-gram sample of this material was subjected to vacuum drying at 70°C and a pressure of 10 mm Hg for 16 hours. Five calorific output determinations were then performed on this sample. The results were as follows:

<u>Sample Number</u>	<u>Calorific Output (cal/gm)</u>
1	222.44
2	222.64
3	222.28
4	223.02
5	222.59
Average	- 222.59 cal/gm
Standard Deviation	- 0.28 cal/gm

These results indicated that the first 25-gram sample of ELB 23468 had become contaminated in some way which affected its calorific output. It was theorized that such contamination could have occurred during transit of the sample through a high humidity area prior to being subjected to the vacuum drying process. This could have resulted in the powder absorbing sufficient atmospheric moisture to cause an oxidation-reduction reaction between a portion of the iron powder and the potassium perchlorate when the sample was heated. Since this reaction would have consumed potassium perchlorate, it would have resulted in a reduction in the calorific output of the affected sample. When the calorific output of the master batch of ELB 23468 was found to be within the specification limits, this indicated that the material was acceptable for evaluation. However, in the interim, ELB 23471 had already been partially evaluated, so the evaluation of this composition was continued.

Compositions ELB 23469 and ELB 23485 (prepared using iron powder from Lots 9 and 10, respectively) both gave acceptable calorific output values, and these mixtures were subsequently evaluated. However, problems again arose when ELB 23486 was tested. This material, containing Lot 11 iron powder, yielded an average calorific output of 226.45 cal/gm. Since this was well above the upper limit established for heat powder, the testing of ELB 23486 was suspended. In order to verify that the blend was an actual 88/12 iron/potassium perchlorate mixture, two calorific output determinations were conducted on a portion of the master batch of ELB 23486 which had not been subjected to the vacuum drying procedure. The results of these tests were as follows:

<u>Sample Number</u>	<u>Calorific Output (cal/gm)</u>
1	218.75
2	220.76
Average - 219.76 cal/gm	

In view of the disparate results obtained for "dried" and "undried" ELB 23486, it was decided to suspend all further testing of this and all other materials until Sandia Corporation could be contacted concerning the advisability of discontinuing the vacuum drying procedure. However, it was disclosed that production heat pellets are always exposed to vacuum drying conditions during processing, so this same treatment of the calorific output sample was considered advisable.

It was decided not to expend any further effort on ELB 23486. Instead, this material was scrapped and another heat powder blend was prepared using Lot 11 iron. This composition was designated ELB 23495. Five calorific output determinations conducted on this material yielded an average of 219.01 cal/gm. Although this was on the low side of the allowable calorific output range, it did fall within the tentative limits, so ELB 23495 was accepted for evaluation.

An 88/12 mixture of Lot 12 iron powder and ground potassium perchlorate was prepared and designated ELB 23496. Five calorific output determinations were performed on this material. The results of these tests were as follows:

<u>Sample Number</u>	<u>Calorific Output (cal/gm)</u>
1	224.22
2	222.64
3	224.34
4	224.17
5	223.73
Average	- 223.82 cal/gm
Standard Deviation	- 0.70 cal/gm

Since the above average was out of specification on the high side, a sample of the master batch of ELB 23496 which had not been subjected to the vacuum drying procedure was tested to determine its calorific output. Three tests were conducted, with the following results.

<u>Sample Number</u>	<u>Calorific Output (cal/gm)</u>
1	221.34
2	218.96
3	219.60
Average - 219.97 cal/gm	

These results indicated that ELB 23496 was a true 88/12 blend, at least before the vacuum drying process, so the composition was accepted and its evaluation was resumed.

Analysis of the qualification data for the seven lots of NX-1000 iron powder resulted in two major conclusions. These were: 1) certain unexplained variations occurred in the calorific output values of some (but not all) NX-1000 iron/potassium perchlorate compositions after these materials had been exposed to heat and vacuum, and 2) definite lot-to-lot variations existed in the NX-1000 iron powder samples evaluated. Presumably the 70°C vacuum drying cycle to which the compositions were all exposed initiated a reaction or change of some type in certain lots of heat powder while having little or no effect on other lots. The reason for this was not determined. The basis for concluding that lot-to-lot variations existed in the NX-1000 iron powder was the data in Table 7 showing a general tendency toward increasing pellet breaking strength with higher lot numbers of iron and, in addition, the statistically significant lot-to-lot variations encountered in both the gas evolution and linear burn rate values. Also, the calorific output variations

which occurred in certain lots of heat powder after vacuum drying at elevated temperature could have been related in some way to the characteristics of the specific lots of iron powder used in their preparation.

IV. CONCLUSIONS

A number of conclusions relating to specific areas of investigation have been presented throughout this report. The following statements represent only the major conclusions of the entire program.

A. The test methods and equipment developed during this program for determining calorific output, gas evolution and ignition sensitivity are superior to those used previously.

B. The particle size of the potassium perchlorate used in 88/12 NX-1000 iron/potassium perchlorate heat powder affects the properties of the composition significantly. From an overall standpoint, a potassium perchlorate particle size of approximately five microns appears best for the Sandia application.

C. Either a rotary tumbler or a twin-cone blender with a break-up screen is satisfactory for blending NX-1000 iron/potassium perchlorate heat powders.

D. An increase in the density of 88/12 NX-1000 iron/potassium perchlorate heat pellets causes a corresponding increase in pellet breaking strength and a decrease in ignition sensitivity.

E. The incorporation of metal grids into iron/potassium perchlorate heat pellets increases pellet breaking strength but decreases ignition sensitivity. The use of metal grids is not recommended.

F. The vacuum drying of an NX-1000 iron/potassium perchlorate composition at elevated temperature may cause a significant change in its calorific output.

G. Lot-to-lot differences exist in NX-1000 iron powder. However, all lots of NX-1000 evaluated are acceptable for use in production heat powder.

V. RECOMMENDATIONS

A. Modify the pellet breaking strength test equipment to include a motorized device for the application of force.

B. Determine the effects of vacuum drying and other environmental conditioning on the properties of heat powder, with particular emphasis on calorific output.

C. Investigate the use of additives for the purpose of improving heat powder flow characteristics.

D. Investigate the use of additives for the purpose of increasing pellet breaking strength.

E. Investigate the feasibility of calibrating the Parr 1411 calorimeter using NX-1000 iron/potassium perchlorate heat powder as a standard.

F. Elucidate the nature of the combustion reaction occurring in 88/12 NX-1000 iron/potassium perchlorate heat powder.

G. Determine the nature and extent of any decomposition reactions occurring in 88/12 NX-1000 iron/potassium perchlorate heat powder or pellets on storage.

APPENDIX A
CONFERENCE REPORTS

CONFERENCE REPORT OF 8 MAY 1969

The first quarterly conference was held at Sandia Corporation on 8 May 1969. Messrs. Don Bush and Joe McDowell attended from Sandia; Mr. Charles Rittenhouse attended from Unidynamics.

The new pelletized heat source program (Sandia P.O. No. 25-8652; Unidynamics Contract C09-006) was discussed, and the work for the first quarter was planned. The tentative work plan was presented, and it was approved with only minor changes. A copy of the amended plan is attached to this report.

It was agreed that the initial work should be in the areas of calorimetry, ignition sensitivity testing and potassium perchlorate investigation.

Calorimetry

This work will follow the attached plan with no anticipated exceptions. There will be a delay in calibrating the Parr 1411 calorimeter, however, due to the necessity of ordering 350 cal/gm Zr/BaCrO_4 NBS standard heat powder for calibration. This material is preferred over the 390 cal/gm powder already on hand because its heating value has a smaller standard deviation and it is closer to that of 88/12 iron/potassium perchlorate.

It was agreed that a secondary standard 88/12 NX-1000 iron/potassium perchlorate heat powder should not be prepared at this time. However, such a material may be prepared later in the program.

It was disclosed that Sandia may purchase a quartz thermometer later this year. If so, it will be sent to Unidynamics for use on the 1411 calorimeter.

Ignition Sensitivity Testing

Basically, this work will follow the attached plan; however, a number of details were discussed which had not been included in the work plan.

One of the most critical tasks will be to eliminate as many operator-dependent variables as possible from the capacitor discharge tester. Three specific areas to check are the manner in which the probe tip is applied to the pellet, the uniformity of the underside pellet contact and the uniformity of the pellet surfaces. It was suggested that a metal shim of known thickness might be used during application of the probe to prevent the tip from actually touching the pellet. The thickness of the shim could then be compensated for in setting the gap. It was also suggested that the underside pellet contact be sanded and polished to improve its uniformity and, hopefully, cause the underside arc to occur at the same point in each test. It was recommended that consideration be given to the idea of identifying the pellet top and bottom surfaces and using the same surface each time in the ignition sensitivity tests. In this way, the effects of pellet surface imperfections and density gradients should cancel out.

Another modification of the capacitor discharge tester which should be considered is the use of a heliarc tip in place of the existing probe. It was suggested that we might

photograph the arc generated by the existing unit on a number of discharges to determine whether it wanders from test to test. If it does, then serious consideration should be given to installing a heliarc tip, possibly with dual gas flow. The inert gas shroud around the arc should serve both to exclude oxygen from the pellet surface and, hopefully, steer or guide the arc in the same direction on each discharge.

One final point discussed concerning the capacitor discharge tester was the addition of new capacitance levels. Tentatively, two new levels will be added, probably 0.35 and 0.65 microfarads. When added to the capacitors already available, this will give the apparatus the capability of producing 0.125, 0.175, 0.250, 0.325, 0.500 and 1.000 joules within the range of interest for heat powder testing.

It was stated that the overall objective of the ignition sensitivity study is first to optimize the capacitor discharge tester and then to be able to duplicate the apparatus. For this reason, a complete set of engineering drawings, blueprints, circuit diagrams and schematics, calibration procedures and operating procedures will be required.

Potassium Perchlorate Investigation

The work in this area will be conducted as outlined in the attached work plan with one exception. In addition to the five particle sizes listed, the smallest particle size of potassium perchlorate attainable (hopefully less than 5 microns) should also be evaluated. The various particle sizes of potassium perchlorate should be investigated beginning with the smallest sizes and working up to the coarser materials.

Heat Powder Blending

This work will follow the attached plan; however, nothing will be done in this area during the first quarter.

Ignition Sensitivity Versus Pellet Density and Pellet Strength Versus Pellet Density

This work will follow the attached plan; however, nothing will be done in this area during the first quarter.

Pellet Strength

This work will generally follow the attached plan; however, much more definition of objectives will be needed before reasonable strength-enhancing additives can be selected. In any event, no work will be done in this area during the first quarter.

NX-1000 Iron Powder Evaluation

This work will follow the attached plan; however, nothing will be done in this area during the first quarter.

SANDIA P.O. NO. 25-8652
PELLETIZED HEAT SOURCE STUDY
WORK PLAN

I. OBJECTIVE

The program will consist of a study to develop pelletized heat sources for thermal batteries. The primary purpose of this effort will be to characterize more completely the existing NX-1000 iron/potassium perchlorate heat powder and to improve the test equipment and techniques. The information obtained will be used to upgrade the present specification, "Preparation and Testing, Heat Powder, Conductive", Drawing 9926052.

II. STANDARD COMPOSITION

The standard composition to be used as a control will consist of 88% NX-1000 iron powder and 12% potassium perchlorate. Nominal values for heat pellets prepared from this composition will be as follows:

Diameter - 3.175 cm (1.250 inch)
Weight - 2.500 gm
Density - 3.50 gm/cm³

III. AREAS OF INVESTIGATION

There will be eight specific areas of investigation in the program. The sequence in which these areas are investigated will be decided by mutual agreement between representatives of Sandia Corporation and Unidynamics. The eight areas and the specific tasks to be performed under each are as follows:

A. Calorimetry

1. Calibrate the Parr 1411 calorimeter with 350 cal/gm zirconium/barium chromate reference heat source material from the National Bureau of Standards.

2. Prepare an 88/12 NX-1000 iron/potassium perchlorate composition and determine its calorific output as prepared, using the calibrated 1411 calorimeter. Expose samples of this composition to the action of selected desiccants, vacuum, elevated temperature and combinations of vacuum and elevated temperature. Determine the calorific output of the composition after each of these treatments and decide whether any of these treatments should be used routinely on iron/potassium perchlorate heat powders prior to testing or use.

3. Prepare a series of iron/potassium perchlorate compositions and perform calorific output tests in both the Parr 1221 and Parr 1411 calorimeters. Compare the values obtained in the two calorimeters.

B. Ignition Sensitivity Testing

1. Modify the capacitor discharge ignition sensitivity tester to include 0.35 and 0.65 microfarad capacitors to provide additional energy levels for use in the probit analyses.

2. Perform any equipment modifications which will minimize operator variables and investigate the use of a heliarc tip for the probe.

3. Prepare a detailed operating procedure for conducting the ignition sensitivity test. This procedure will contain updated drawings reflecting all equipment modifications.

C. Potassium Perchlorate Investigation

1. Prepare NX-1000 iron powder/potassium perchlorate compositions using potassium perchlorate of the following nominal particle sizes: 5 μ , 10 μ , 15 μ , 20 μ , and 25 μ . Determine the calorific output, gas evolution, ignition sensitivity, burning rate and pellet strength of each composition to evaluate the effect of the various particle sizes of potassium perchlorate. Establish the potassium perchlorate particle size range acceptable for heat powder preparation.

2. Prepare a specification describing the processing of potassium perchlorate for use in heat powder. This specification will include methods for grinding and conducting particle size determinations and recommended storage conditions for the ground material.

D. Heat Powder Blending

1. Evaluate several types of mixing equipment, such as V-blenders, twin-cone blenders, mullers, and rotary tumblers, to determine the applicability of each to blending iron/potassium perchlorate heat powder on either a laboratory or production scale.

2. Develop a rapid and reliable method for determining when a satisfactory mixture has been obtained.

E. Ignition Sensitivity Versus Pellet Density

1. Prepare and qualify 88/12 and 86/14 NX-1000 iron powder/potassium perchlorate compositions. Consolidate 0.5-inch-diameter pellets of each composition to the following densities: 3.00, 3.25, 3.40, 3.50, 3.60, 3.75 and 4.00 gm/cm³.

Determine the ignition sensitivity of each group of pellets and plot sensitivity versus pellet density for both compositions.

F. Pellet Strength Versus Pellet Density

1. Using the two compositions described in Paragraph E, consolidate 1.25-inch-diameter pellets of each composition to the following densities: 3.00, 3.25, 3.40, 3.50, 3.60, 3.75 and 4.00 gm/cm³. Determine the pellet strength of each group of pellets and plot pellet strength versus pellet density for both compositions.

G. Pellet Strength

1. Using the standard 88/12 NX-1000 iron/potassium perchlorate composition, consolidate 1.25-inch-diameter pellets of the material with grids to the following densities: 3.00, 3.25, 3.40, 3.50, 3.60, 3.75 and 4.00 gm/cm³. Determine the ignition sensitivity and pellet strength of each group of pellets and plot ignition sensitivity versus pellet density and pellet strength versus pellet density.

2. Prepare and evaluate NX-1000 iron powder/potassium perchlorate compositions containing additives such as metallic whiskers, colloidal silica, etc. Determine the effect of these additives on pellet strength versus pellet density and pellet density versus consolidation force.

H. NX-1000 Iron Powder Evaluation

1. Evaluate Pfizer NX-1000 iron powder lots 6 through 15 by blending each in an 88/12 ratio with potassium perchlorate and determining the calorific output, gas evolution, ignition sensitivity, burning rate and pellet strength of each mixture.

CONFERENCE REPORT OF 25 SEPTEMBER 1969

The second quarterly conference was held at Unidynamics/Phoenix on 25 September, 1969. Messrs. Joe McDowell, Don Bush and Bob Clark attended from Sandia. Mr. Charles Rittenhouse attended from Unidynamics.

The discussions centered on the work performed in the areas of calorimetry, ignition sensitivity testing and potassium perchlorate investigation. No major problems have been encountered in any of these areas. However, there were some questions raised concerning the conditions under which certain of the calorific output tests were conducted, and the consensus was that one group of tests should be repeated.

Calorimetry

Most of the work in this area has been completed. In general, the study was relatively straightforward and free of problems. However, there was some concern expressed over the apparent loss in calorific output which occurred in an 88/12 iron/potassium perchlorate composition after storage for several weeks over various drying agents. Certain calorific output results were considered questionable because the 1411 calorimeter had been relocated during one series of tests, necessitating recalibration of the instrument. It was agreed to repeat those tests where the results were subject to question.

It was learned that Sandia had ordered a quartz thermometer and that some of the parts had already been shipped to Unidynamics. A subsequent check disclosed that these parts had arrived, and they have now been accumulated in the Research Department.

Ignition Sensitivity Testing

Practically no testing has been completed in this area, so there was only a limited basis for discussion. We were conducting a series of tests on the day of the visit, so the Sandia representatives were able to observe the steps involved in each test. They saw some of the problems that were being encountered, the most persistent of which was repeated breaking of the tungsten electrodes.

It was requested that we make a series of photographs of the modified capacitor discharge apparatus fitted with a tungsten electrode. This should include a view of the tip before, during and after firing, preferably with the argon flow on during firing.

Potassium Perchlorate Investigation

The results of the tests to determine the effects of various storage conditions on the particle size of ground potassium perchlorate were discussed. It was agreed that none of the data indicated anything really new or unexpected. However, the results obtained in this study did confirm certain relationships which had intuitively been felt in the past but had never actually been proved. These include the following:

- 1) the particle size of ground potassium perchlorate increases in a high humidity environment, 2) the higher the humidity, the greater the rate of increase in particle size, and
- 3) the more the material is protected from the high humidity conditions (as in a well sealed container, for example), the less it is affected by the high humidity environment.

In addition, the tests indicated that ground potassium perchlorate can be stored under normal room conditions for at least two months with no appreciable increase in its average particle

size. These findings indicate that Unidynamics' normal handling procedures for ground potassium perchlorate are adequate to prevent appreciable particle size growth.

CONFERENCE REPORT OF 5 FEBRUARY 1970

The third quarterly conference was held at Unidynamics/Phoenix on 5 February 1970. Messrs. Bruce Van Domelin and Don Bush attended from Sandia. Mr. Charles Rittenhouse attended from Unidynamics.

The work accomplished since the last meeting was reviewed, and the tasks still remaining to be performed were discussed. An order of magnitude estimate of the total cost of completing all of the tasks in the original work plan was presented.

It was stated that Sandia could put approximately \$6,000 more into the study and that practically all of the money on the program, including the funds still remaining in the budget plus the additional \$6,000, would have to be spent and billed before 1 July 1970. Based on this requirement, it was agreed that we should stop all experimental work on the program by the end of May so the billing could be submitted to Sandia during June.

Subsequently it was agreed that there was not sufficient funding to accomplish all of the tasks originally planned. Therefore, it will be necessary to eliminate or limit the scope of certain of the investigations.

CONFERENCE REPORT OF 10 APRIL 1970

The fourth quarterly conference was held at Sandia Corporation on 10 April 1970. Messrs. Bruce Van Domelin and Don Bush attended from Sandia. Mr. Charles Rittenhouse attended from Unidynamics.

The discussions centered on the progress made since the last conference, especially the work conducted during the past month relating pellet density to breaking strength and ignition sensitivity. In general, the program is going well from the technical standpoint, and there do not appear to be any major problems. The principal points of the discussion pertained to the final report. They were as follows:

In discussing large and small heat pellets, the diameter of each type of pellet should be stated along with the weight.

The pellet forming pressure should be given in psi rather than merely giving the force in tons as was done in the monthly reports.

The test schedule for the calorific output study should not be included in the final report. Instead, the conditions (time, temperature, humidity, etc.) to which each sample was exposed should be reported along with the calorific output of the sample following exposure to these conditions.

In the appendix to the final report, the procedures should be given for determining calorific output, gas evolution, pellet breaking strength, linear burn rate and ignition sensitivity. The calorific output procedure will be basically the same as that in Parr Manual 128. The gas evolution procedure should be that for the manometric test. In addition to the test procedures, the appendix should also contain a specification for potassium perchlorate for use in heat powder.

APPENDIX B
SPECIFICATION FOR POTASSIUM PERCHLORATE

SPECIFICATION FOR POTASSIUM PERCHLORATE

Scope. This specification defines the procedures for the procurement, processing and storage of potassium perchlorate for use in iron/potassium perchlorate heat powder.

Procurement. For use in iron/potassium perchlorate heat powder, the potassium perchlorate must be certified to MIL-P-217A, Grade A, Class 3. An approved source for this material is:

American Potash and Chemical Corporation
3000 West Sixth Street
Los Angeles, California 90054

Processing. The processing of potassium perchlorate consists of three operations: 1) grinding, 2) drying and 3) particle size analysis.

Grinding. For use in iron/potassium perchlorate heat powder, the potassium perchlorate must be ground to a mean particle diameter of 5.0 ± 1.0 microns. This is best accomplished using a motor-driven pulverizer of the "hammermill" type. There are several manufacturers of this type of equipment. An approved machine is the Model 1SH Mikro-Pulverizer, which can be obtained from:

Pulverizing Machinery
Division of Slick Industrial Company
Summit, New Jersey

This machine has a nominal grinding speed of 9700 rpm and an approximate capacity of 50 pounds per hour.

NOTE

A grinding speed of approximately 10,000 rpm is required to reduce potassium perchlorate to a particle size of 5.0 ± 1.0 microns. Any model of pulverizer which is selected for this application should be capable of grinding speeds in this general range.

If a Model LSH Mikro-Pulverizer is used to grind the potassium perchlorate, the material should be passed through the machine two times at a moderate feed rate. A 0.010-inch herringbone screen should be used on the first pass, and 0.035-inch herringbone screen should be used on the second pass. The screens should be in good condition, since badly worn screens will result in inefficient grinding.

Drying. Following the second grinding of the potassium perchlorate, a sample of the ground material is dried at 120°C for a minimum of four hours, then tested to determine its mean particle size. If the particle size is within the range of 5.0 ± 1.0 microns, the entire lot of ground material is dried. Large quantities of ground potassium perchlorate are dried at 120°C for a minimum of 16 hours.

Particle Size Analysis. Particle size analysis of the ground potassium perchlorate is performed using an Eagle-Picher turbimeter. An accurately weighed sample containing 500 ± 100 milligrams is dispersed in 100 milliliters of odorless kerosene containing a small percentage of Twitchell Base 400-H (five milliliters of Twitchell Base 400-H per liter of kerosene). Dispersion of the sample is best accomplished using an ultrasonic vibrator or, if this is not available, an air-driven vibrator. When the potassium perchlorate has been dispersed completely in the kerosene, the suspension is poured immediately into the sample chamber of the turbimeter, and the initial absorption is determined. This absorption reading

should be between 60% and 80%. If the initial absorption is above 80%, prepare another sample containing less potassium perchlorate; if the initial absorption is below 60%, prepare another sample containing more potassium perchlorate. If the initial absorption is within the correct range, proceed with the test, following the directions given in the turbidimeter instruction manual. Determine the mean particle diameter of the potassium perchlorate. Then repeat the test two more times, and calculate the average particle size of the material.

If the average particle size of the potassium perchlorate is above 6.0 microns, grind the material a third time. If it is below 4.0 microns, set the material aside, and reject it for use in iron/potassium perchlorate heat powder. If the average particle size is within the required range of 5.0 ± 1.0 microns, place the material in storage unless it is to be used immediately.

Storage. The ground potassium perchlorate is stored in double-walled polyethylene bags sealed with several wraps of friction tape. Each double bag is placed into a metal container of appropriate size, and the container lid is also sealed with friction tape.

When protected in this manner, ground potassium perchlorate can be stored for as long as 30 days after grinding before being used in heat powder. If the material is stored longer than 30 days, its mean particle size must be redetermined to assure that it is still within the specified limits of 4.0-6.0 microns before it can be used. If the mean particle size of the material exceeds the upper limit of 6.0 microns after storage, it can be reclaimed by grinding once (twice, if necessary) through the Mikro-Pulverizer using a 0.035-inch herringbone screen.

APPENDIX C
STANDARD MIXING PROCEDURE

STANDARD MIXING PROCEDURE

Scope. This procedure defines the method for blending laboratory quantities (1000 grams or less) of NX-1000 iron/potassium perchlorate heat powder.

Ingredient Preparation.

- a. Remove a sufficient quantity of NX-1000 iron powder from stock to prepare the required quantity of heat powder. Then pass this material through a 100-mesh sieve to remove any foreign particulate matter.
- b. Using a triple-beam balance accurate to 0.01 gram, weigh the exact quantity of iron required into an aluminum can.
- c. Remove a sufficient quantity of ground potassium perchlorate of the correct particle size from stock to prepare the required quantity of heat powder.
- d. Using a triple-beam balance accurate to 0.01 gram, weigh the exact quantity of potassium perchlorate required into the aluminum can containing the iron powder.

NOTE

The potassium perchlorate must be dried for a minimum of four hours at 120°C within 48 hours prior to blending, then stored in a sealed container until ready for use. A particle size determination must be performed on the potassium perchlorate at the time of grinding and again prior to use if such use occurs more than 30 days after the date of grinding.

Blending.

- a. Blend the mixture by passing the ingredients through a 100-mesh sieve together six times.

packaging.

- a. Immediately after blending, place the mixture in a double-walled polyethylene bag, and seal the bag securely.
- b. Place the sealed bag into a metal container of appropriate size, and seal the container securely with friction tape.

NOTE

Mixtures which are to be used within one week of blending may be stored in a sealed metal container without the polyethylene bag.

APPENDIX D
TEST PROCEDURES

DETERMINATION OF CALORIFIC OUTPUT

Scope. This procedure defines the method for determining the calorific output of heat powder using a Model 1411 Parr Oxygen Bomb Calorimeter. Conventional calibration procedures for this calorimeter are included in Parr Instrument Company Manual No. 128, Supplement No. 1. However, the calorimeter may also be calibrated using one of the NBS Standard Reference Materials selected from the group consisting of SRM No. 1651, 1652 and 1653.

Procedure.

- a. Using an analytical balance, weigh a sample of the heat powder to be evaluated into a tared Parr fuel capsule. This sample should be sufficient to produce a temperature rise of approximately 2°C in the calorimeter.

NOTE

When testing 88/12 iron/potassium perchlorate, the sample weight should be approximately 5 grams.

- b. Cut a 6.96 cm length of No. 45C10 Parr fuse wire, and lace the wire onto the electrodes on the head of the bomb.
- c. Place the head of the bomb on the fuel capsule containing the sample, and ascertain that the fuse wire is in contact with the sample.
- d. Assemble the bomb.
- e. Purge the bomb ten times with 25-30 psig of argon, releasing the pressure after each purge. After the final purge, seal the bomb.

- f. Place the bomb in the calorimeter bucket, and attach the ignition terminal to the bomb.
- g. Measure 450.00 ± 0.05 grams of distilled water which is at a temperature of $18-20^{\circ}\text{C}$ into a volumetric flask, and pour the water into the calorimeter bucket.
- h. Close the calorimeter, and start the stirring mechanism.
- i. After three minutes, observe and record the exact temperature of the water in the calorimeter bucket. This temperature is T_3 .
- j. Let the temperature of the apparatus stabilize for nine minutes. Then observe and record the temperature again. This temperature is T_9 .
- k. Immediately after recording T_9 , ignite the sample in the bomb.
- l. Watch the thermometer closely after ignition. A rapid temperature rise confirms successful ignition of the sample. If no appreciable temperature rise occurs, remove the bomb from the calorimeter, and repeat steps a through k. If a rapid temperature rise occurs, proceed to step m.
- m. Six minutes after ignition of the sample, observe and record the temperature of the water in the calorimeter bucket. This temperature is T_{15} . Denote this as the final temperature.
- n. Continue the test until twelve minutes after ignition of the sample. Then observe and record the water temperature again. This temperature is T_{21} .

Calculations.

NOTE

If a mercury thermometer is used, the initial and final temperatures must be corrected using the correction chart supplied with the thermometer.

- a. Subtract the initial temperature (T_9) from the final temperature (T_{15}). This is the net temperature rise (NTR).
- b. Calculate the radiation correction (R.C.) using the formula:

$$R.C. = \frac{5 \times (T_{21} - T_{15}) + (T_9 - T_3)}{6}$$

- c. Subtract the radiation correction (R.C.) from the net temperature rise (NTR). This is the true net temperature rise (TNTR).
- d. Multiply the water equivalent value of the calorimeter (W) by the true net temperature rise (TNTR), and subtract from this the calorific yield of the fuse wire (1.13 calories). Then divide this value by the sample weight in grams (mass). The result obtained is the calorific output of the sample in calories per gram.

$$\text{Calorific Output (cal/gm)} = \frac{(W) \times (TNTR) - 1.13}{(\text{mass})}$$

LEGEND:

- A - Water-jacketed mercury manometer
- B - Combustion bomb
- C - Recirculating pump
- D - Adjustable clamp for leveling mercury columns

MANOMETRIC GAS EVOLUTION APPARATUS

DETERMINATION OF GAS EVOLUTION

Scope. This procedure defines the method for determining the gas evolution of heat powder using a manometric apparatus (see opposite page). The combustion bomb used in this apparatus is the bomb from the Model 1411 Parr Oxygen Bomb Calorimeter.

Procedure.

- a. Using an analytical balance, weigh a sample of the heat powder to be evaluated into a tared Parr fuel capsule. This sample should be sufficient to produce approximately five milliliters of gas.

NOTE

When testing 88/12 iron/potassium perchlorate, the sample weight should be approximately 5 grams.

- b. Cut a 6.96 cm length of No. 45C10 Parr fuse wire, and lace the wire onto the electrodes on the head of the bomb.
- c. Place the head of the bomb on the fuel capsule containing the sample, and ascertain that the fuse wire is in contact with the sample.
- d. Assemble the bomb.
- e. Purge the bomb ten times with 25-30 psig of argon, releasing the pressure after each purge. After the final purge, seal the bomb, and attach a short length of flexible plastic tubing to the purge valve of the bomb. This is the gas delivery tube.

- f. Place the bomb in the water bath, and attach the ignition terminal to the bomb.
- g. Start the recirculating water pump, and allow it to operate for five minutes. Then adjust the water bath temperature to equal the room temperature by the addition of hot or cold water.
- h. Observe and record the barometric pressure, applying a correction factor if necessary.
- i. After making certain that the water bath and room temperature are still equal, ignite the sample in the bomb.
- j. After five minutes, again adjust the water bath temperature to equal the room temperature. Record this temperature.
- k. Open the air inlet valve on the manometer, and equalize the levels of the mercury columns in the manometer and reservoir tubes to obtain a reading of zero on the manometer scale. Then close the air inlet valve, and attach the gas delivery tube from the combustion bomb to the gas inlet valve on the manometer.
- l. Slowly open the purge valve on the bomb to allow the evolved gases to enter the manometer. This will cause the level of the mercury column in the manometer tube to drop.
- m. Using a straight edge with an affixed level indicator, immediately equalize the levels of the mercury columns in the manometer and reservoir tubes. This will always require lowering the level of the column in the reservoir tube.

- n. With the levels of the mercury columns equalized, observe and record the volume of mercury displaced from the manometer tube by the evolved gases.

Calculations.

- a. Using the following formula, calculate the gas evolution of the sample.

$$V = \frac{273}{273 + T} \times \frac{P}{760} \times \frac{G}{W}$$

where V = Gas evolution in ml/gm at standard temperature and pressure.

T = Temperature of room and water bath in °C.

P = Corrected barometric pressure in mm of Hg.

G = Total gas evolved in ml.

W = Sample weight in gm.

DETERMINATION OF PELLET BREAKING STRENGTH

Scope. This procedure defines the method for determining the strength of 1.25-inch-diameter heat pellets consolidated to a density of $3.50 \pm 0.03 \text{ gm/cm}^3$.

Procedure.

- a. Procure the following equipment:
 - (1) Physical Strength Test Fixture
 - (2) Chatillon Model DPP-5 Dial Push-Pull Gauge
 - (3) Carver Laboratory Press
 - (4) 1.25-inch-diameter Pellet Die
 - (5) Loomis Press (25 ton capacity)
 - (6) Analytical Balance
 - (7) One-inch Micrometer
- b. Weigh at least ten heat powder samples (more if needed) containing 3.85 ± 0.02 grams each.
- c. Consolidate the first sample at a pressure of 15,900 psi on the Loomis press. On a 1.25-inch-diameter pellet, this pressure is equal to 19,500 pounds force or 9.75 tons force.

NOTE

If there is reason to believe that the required density can be achieved using a different pressure, use this pressure.

PELLET BREAKING STRENGTH TEST SETUP

- d. Record the weight, height and density of the pellet.
The weight should be 3.80 ± 0.05 grams, and the density should be 3.50 ± 0.03 gm/cm³.

$$\text{Density (gm/cm}^3\text{)} = \text{Weight (gm)}/\text{Height (in)} \times 20.18 \text{ (a constant)}$$

NOTE

If a pellet density of 3.50 ± 0.03 gm/cm³ is not obtained, additional testing must be performed until the proper consolidation pressure is determined.

- e. Record the consolidation pressure necessary to obtain pellets of the required density.
- f. Place the first pellet of the proper density in the recessed support of the test fixture (see opposite page) and clamp the pellet retaining block in position.
- g. Carefully insert the pellet ram through the guide until it just contacts the upper surface of the pellet.
- h. Place the loaded fixture on the table of the Carver press. A spacer block may be used if needed.
- i. Verify that the dial on the gauge reads 0; then push the LOCK button down.
- j. Slowly raise the table of the Carver press until the gauge ram touches the pellet ram. The gauge should indicate no greater than 0.15 pound.
- k. Slowly raise the table of the Carver press using one sweep of the handle until the pellet breaks. Record the gauge indication as the breaking strength of the pellet.

1. Test at least nine more pellets by the same procedure, and compute the average breaking strength for the ten pellets. Record this value as the average pellet breaking strength.

DETERMINATION OF IGNITION SENSITIVITY

Scope. This procedure defines the method for determining the capacitor discharge ignition sensitivity of 0.50-inch-diameter heat pellets consolidated to a density of $3.50 \pm 0.03 \text{ gm/cm}^3$. The energy required for 50% probability of ignition is determined by subjecting groups of 20 pellets each to the stimulus of selected energy levels and performing a probit analysis of the response obtained at the various levels.

Procedure.

a. Procure the following equipment:

- (1) Capacitor Discharge Ignition Sensitivity Tester with Safety Firing Box and Heliarc Gas Shroud
- (2) Argon Cylinder with Appropriate Gauges and Flowmeter
- (3) Wash Bottle containing a Mixture of 50/50 Distilled Water/Isopropyl Alcohol
- (4) Stiff Brush
- (5) Emery Cloth
- (6) Cup or Dish for Burned Pellets
- (7) Carver Laboratory Press
- (8) 0.50-inch-diameter Pellet Die
- (9) Analytical Balance
- (10) One-inch Micrometer

b. Weigh at least 60 heat powder samples (more if needed) containing 625 ± 10 milligrams each.

- c. Consolidate these samples at the pressure required to obtain a pellet density of $3.50 \pm 0.03 \text{ gm/cm}^3$.
- d. Record the weight, height and density of each pellet. The weight should be 615 ± 10 milligrams, and the density should be $3.50 \pm 0.03 \text{ gm/cm}^3$.
- e. Insert a 3/16-inch-diameter steel electrode with a freshly sharpened tungsten carbide tip into the electrode holder.

NOTE

Always use an electrode with a freshly sharpened tip, since a dull tip may alter the results of the test.

- f. Press the MAIN POWER button on the capacitor discharge ignition sensitivity apparatus, and allow the unit to warm up until the READY light comes on.
- g. Close the line valve between the argon cylinder and the flowmeter, and open the main valve on the cylinder. Observe the gas pressure in the cylinder. If the pressure is 1,000 psi or less, replace the cylinder.
- h. Adjust the flow pressure of the argon to 25-30 psig.

NOTE

No not exceed a flow pressure of 30 psig or the flowmeter will be damaged.

- i. Open the flow pressure valve to the flowmeter, and adjust the SCFH (standard cubic feet per hour) control valve on the flowmeter to 10 SCFH.

- j. Place a cup or dish in the firing box to catch the burned pellets, and position a pellet on the support directly under the electrode. Using the micrometer mechanism, adjust the distance between the tip of the tungsten carbide electrode and the upper surface of the pellet to 0.002 ± 0.0002 inch.
- k. Close both doors on the safety firing box.
- l. Press on the high voltage ON button, and disable the alarm. Using the 0-1500 volt scale on the dual-range voltmeter, adjust the capacitor charge level to 1000 volts.
- m. Set the selector switch on the capacitor bank at one of the following six capacitance levels:

Level 1 - $0.25 \mu\text{f}$ (0.125 joule)

Level 2 - $0.35 \mu\text{f}$ (0.175 joule)

Level 3 - $0.50 \mu\text{f}$ (0.250 joule)

Level 4 - $0.65 \mu\text{f}$ (0.325 joule)

Level 5 - $1.00 \mu\text{f}$ (0.500 joule)

Level 6 - $2.00 \mu\text{f}$ (1.000 joule)

NOTE

In general, $0.65 \mu\text{f}$ (0.325 joule at 1000 volts) is the best capacitance level to use in the initial test, since a group of 20 pellets tested at this level normally yields some ignitions as well as some no-fires.

- n. Allow the capacitor bank to charge to 1000 volts.
- o. Press the FIRE button.

- p. Regardless of whether the pellet ignites or not, remove it from the support with the pusher bar on the left side of the safety firing box immediately. In the case of those pellets which do ignite, this will prevent the base electrode and asbestos heat shield from overheating and influencing the ignition sensitivity of the next pellet tested.
- q. Record the response of the pellet to the electrostatic discharge, i.e., Ignited or No-Fire.
- r. Open the door on the safety firing box, and press the FIRE button to bleed off any residual charge stored in the capacitor bank.

NOTE

Opening the door breaks the circuit between the high voltage power supply and the capacitor bank, thus accomplishing the same thing as pressing the high voltage OFF button.

- s. Clean the pellet support with a stiff brush. Cool the support with the distilled water/isopropanol mixture, and polish the base electrode with a piece of emery cloth if necessary.
- t. Repeat steps j through s with each of the other 19 pellets in the group.
- u. Depending on the response of the pellets in the first group tested, continue testing groups of 20 pellets each at the other capacitance levels available (above and below 0.65 μ f) until both on all-fire and a no-fire level have been established.

- v. Submit the data obtained to a statistical probit analysis to determine the energy required for 50% probability of ignition.

DETERMINATION OF LINEAR BURN RATE

Scope. This procedure defines the method for determining the linear burn rate of 1.25-inch-diameter heat pellets.

Procedure.

a. Procure the following equipment:

- (1) Hewlett-Packard Model 523C Electronic Counter
- (2) Burn Rate Indicator with Accessories
- (3) Photocells, GE Type L9U LASCAR's or Equivalent
- (4) Cotton Swabs
- (5) Distilled Water
- (6) Spatula
- (7) Stiff Brush
- (8) Loomis Press (25 ton capacity)
- (9) 1.25-inch-diameter Pellet Die
- (10) Analytical Balance
- (11) One-inch Micrometer
- (12) Two-inch Micrometer

b. Weigh at least five heat powder samples (more if needed) containing 3.85 ± 0.02 grams each.

c. Consolidate these samples at the pressure required to obtain a pellet density of 3.50 ± 0.03 gm/cm³.

d. Record the weight, height and density of each pellet. The weight should be 3.80 ± 0.05 grams, and the density should be 3.50 ± 0.03 gm/cm³.

e. To set up the burn rate indicator:

- (1) Connect the power cord to a 110 volt, 60 cycle, AC source.
- (2) Connect the detector head to the proper panel input.
- (3) Set the power switch at ON.

f. To set up the HP 523C counter:

- (1) Connect the power cord to a 110 volt, 60 cycle, AC source.
- (2) Set the controls as follows:

Function Selector: Time Interval

Time Unit: Milliseconds

Display Time: Inf.

Start Trigger Level: -3

Multiplier: AC x 1

Stop Trigger Level: -3

Multiplier: AC x 1

Trigger Slope: - start - stop

Com-Sep: Sep.

- (3) Set the power switch at ON.

g. Connect one coaxial cable from the start output on the burn rate indicator panel to the start input on the counter.

h. Connect the other coaxial cable from the stop output on the burn rate indicator panel to the stop input on the counter.

- i. Attach the leads from the 5 volt, 20 ampere transformer to each end of the loop of wire on the base plate fixture.
- j. Place the heat powder pellet into the depression of the base plate fixture. Verify that the pellet is touching the loop of wire.
- k. Place the detector head onto the base plate fixture, matching the peg on the detector head with the notch in the base plate fixture.
- l. Slowly and firmly press the reset button on the counter. Hold for two or three seconds.
- m. Connect the power cord of the transformer to a 110 volt, 60 cycle, AC outlet, and allow time for the heat powder pellet to ignite. Disconnect the power cord immediately after ignition of the pellet.
- n. Remove the detector head from the base plate fixture immediately after complete combustion of the pellet. This will avoid overheating the detector head.
- o. Record the burn time displayed on the counter, and remove the burned pellet from the base plate fixture.
- p. Calculate the linear burn rate of the pellet in inches per second by dividing the distance between the two photocells (0.625 inch) by the burn time in seconds.
- q. Clean the base plate fixture with a stiff brush. Cool in distilled water, and scrape the asbestos bed and wire loop with a spatula if necessary.
- r. Clean the photocell portholes on the detector head if necessary.

CAUTION

Use moist cotton swabs. Do not spray water onto the photocells. Avoid using objects which would scratch the photocells.

- s. Test at least four more pellets by the same procedure, and compute the average linear burn rate for the five pellets. Record this value as the average pellet linear burn rate.

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