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THE EFFECT OF LOW TEMPERATURES
ON IMPACT SENSITIVITY OF TNT

by

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The Effect of Low Temperatures

On Impact Sensitivity of TNT

by

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I. INTRODUCTION

Since 2,4,6,-trinitrotoluene (TNT) is one of the most popular commercial and military explosives used today, it is also one of the most widely studied. Numerous experiments have been conducted to test the various characteristics of TNT (impact sensitivity, thermal sensitivity, crystalline structure, etc.). However, little is known of some of these characteristics due to the lack of TNT samples of proper size and crystalline configuration.

The purpose of this investigation was to observe the behavior of impact sensitivity of TNT in a temperature range of -110°C to room temperature (+22°C). A secondary objective was to investigate by differential thermal analysis any possible crystalline change which might affect the impact sensitivity at low temperature.

The investigation of impact sensitivity at low temperatures is important because the knowledge obtained may be used to increase reliability of explosive devices used in space. Also, the possible usage of a "cold soak" for increased safety in disarming bombs may be derived from this investigation. Impact sensitivity has been investigated down to a temperature of -40°C by Picatinny Arsenal. A further reduction in temperature was considered necessary to gain more information about the impact sensitivity behavior at low temperatures.

TNT is known to be a relatively insensitive substance to impact even though it is a powerful explosive when properly detonated. TNT can be safely handled in both the liquid and solid phases. It exists under normal conditions as a crystalline solid below 80°C. When TNT is used in military or commercial explosive devices, it is usually mixed with other high explosives and formed or cast from the liquid phase to a hard packed solid. This enhances the handling capability of such devices while the high destructive capability is still maintained.

TNT may exist at normal temperatures (80°C and below) in any one of several polymorphic states. The experiments of Burkhardt and Bryden [2] have detected both monoclinic and orthorhombic forms of TNT as well as many variants of each of these two crystalline forms. These forms are dependent on their formation from the melt. The orthorhombic form is obtained by quick-cooling the melt (quenching) while the monoclinic form is obtained by slow-cooling the melt and condensation of crystals on a surface (annealing) [10]. The monoclinic form may also be associated with the crystals formed by sublimation onto a probe at constant temperature. The TNT samples used in this investigation were assumed to be monoclinic since they were formed by sublimation. However, no X-ray nor crystallographic study was made to substantiate this assumption because of the time involved and the lack of the necessary equipment. Previous tests have shown that the orthorhombic forms are more sensitive to impact than the monoclinic forms [10].

The various crystalline forms of TNT are very temperature dependent. Crystal growth studies have been made on TNT in the temperature range from -15°C to +80°C [3,4,6,7]. Liquid TNT can be supercooled to as low as -15°C before crystallizing. At -70°C the orthorhombic form (and its variants) has been observed in TNT [2]. There have been no studies done on the possible crystalline changes of TNT as the temperature is increased from very cold or decreased from a warmer solid phase. An important phase of this study was to investigate any possible structure change of TNT with temperature by using the DTA-900 (Differential Thermal Analyzer). Any change would appear as an exotherm or endotherm on a recorder trace (thermogram). Since the orthorhombic form of TNT is considered more sensitive to impact than the monoclinic form, it is possible that any change at lower temperatures from monoclinic to orthorhombic might increase the sensitivity.

Impact tests were originally used as a means of relating one explosive to another. This measure of sensitivity to impact was important in classifying explosives with regard to their relative potential hazard in handling. The original methods of impact testing (with some modifications) are still used in modern laboratory studies.

The drop test, a method of dropping a weight of known mass onto a small quantity of explosive beneath a steel striker cylinder, is the simplest impact test yielding the most reproducible results. The test consists of a simple transfer of the potential energy of the drop weight

to the explosive sample. Sensitivity can be expressed as the height at which an explosion will occur 50% of the time. The relative sensitivity of different explosives can be compared to determine potential handling hazards.

Impact testing is still considered by many to be an art rather than an exact scientific test. Other testing methods such as the gap test [5] may yield more conclusive data, but the drop weight tester is simpler and more economical to operate. The data obtained from impact tests closely approximates that of other methods in regard to relative sensitivity.

The mechanism of impact initiation of detonation is complex and difficult to analyze because of the variables involved in the test. The process, however, obeys the laws of thermal theory. It is known that the process occurs within an interval of about 10^{-4} second and includes; (1) the initial delay before visible reaction, (2) the propagation of a slow flame at velocities of 10-50 meters/second and (3) the explosion with propagation velocities ranging from several meters/second to 2 kilometers/second [12]. The basic premise in developing initiation theory is that the explosive sample is raised to some temperature by any one of several methods proposed by Bowden and Yoffe [1]. Many investigators have found that the primary factor affecting impact initiation was the influence of temperature on the reaction rate arising from the heat balance equation [5].

Impact creates local "hot spots" in the explosive with temperatures high enough for decomposition to take place at such a rate that the reaction is considered adiabatic. The reaction accelerates until detonation occurs. The time delay from impact to explosion is so short that transient conditions are considered not to exist. Thus, the reaction is considered an adiabatic decomposition process. It seems logical to assume that if the ambient temperature of the explosive is lowered then more energy would be required to initiate explosion. Additional impact energy would be expended in raising the temperature to create "hot spots" and initiate decomposition.

II. METHODS OF INVESTIGATION

The investigation was divided into two distinct phases. Phase I involved the analysis of TNT samples at low temperatures with the DTA-900 (Differential Thermal Analyzer). Phase II consisted of impact tests conducted at 10°C intervals between -110°C and room temperature (+22°C). Both phases were conducted in the High Energy Laboratory at the Naval Postgraduate School. The TNT samples for this investigation were prepared from sublimed crystals produced in the High Energy Laboratory. The large crystals were pulverized and shaken through a 100 mesh Tyler screen in a Ro-Tap machine.

A. PHASE I

The Dupont DTA-900 is one of several laboratory instruments used to make thermal analyses of materials. The DTA-900 consists of a standard cell assembly (with its associated thermocouple arrangement) and a recorder unit which records differential thermal signals from the sample. Figure 1 shows the arrangement of the DTA. The cell assembly is located on the upper right of the apparatus (Fig. 1 - center). A Varian G-4000 X-Y Recorder with a thermocouple lead in the heating block was used as a cross-check for the DTA temperature recording (Fig. 1 - right). A standard laboratory stop timer (Fig. 1 - upper left) was used to cross-check the heating rate of the DTA.

FIGURE 1. Differential Thermal Analyzer - DTA 900

Figure 2 is a schematic of the analyzer. The heater is electrically controlled through the Temperature Program Controller. All temperatures were referenced to 0°C by a thermocouple leading from an ice bath to the inner portion of the cell assembly. When a change in thermal properties of the sample is sensed by the differential thermocouples, a signal is sent to the recorder via high gain amplifiers and is traced on a grid chart.

A small sample of TNT (about .05cc) was placed in the sample tube of the heating block. Glass beads (about .05cc) were placed in the reference tube of the heating block. Three chromel-alumel thermocouples were connected to their respective leads and inserted into the sample, reference and temperature control tubes (see Figure 2A). A fourth thermocouple was placed in a drilled hole in the heating block and connected to the Varian Recorder. The entire cell assembly was covered with a glass bell jar in order to evacuate the assembly and inject liquid nitrogen coolant. Before the coolant was admitted to the cell assembly, the block was heated to melt the TNT sample and to establish a sample temperature of approximately 90°C. The temperature control switch was turned to COOL and the liquid nitrogen (LN) was admitted into the cell. The COOL function of the DTA allows the heating block to cool at a desired rate with small additions of heat to the block. The cell assembly was allowed to cool to -100°C. This temperature was verified by the Varian Recorder.

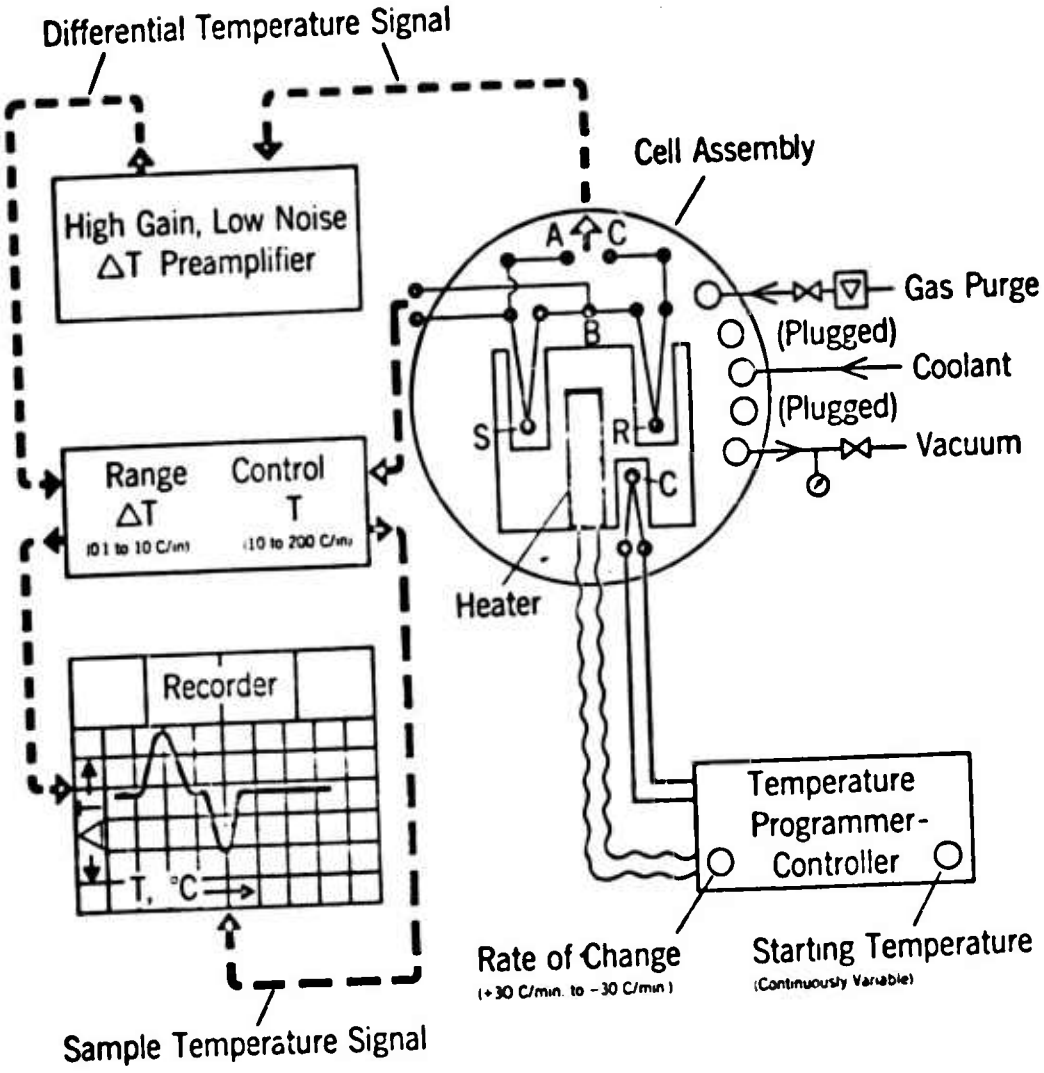

FIGURE 2. Analyzer Schematic [13]

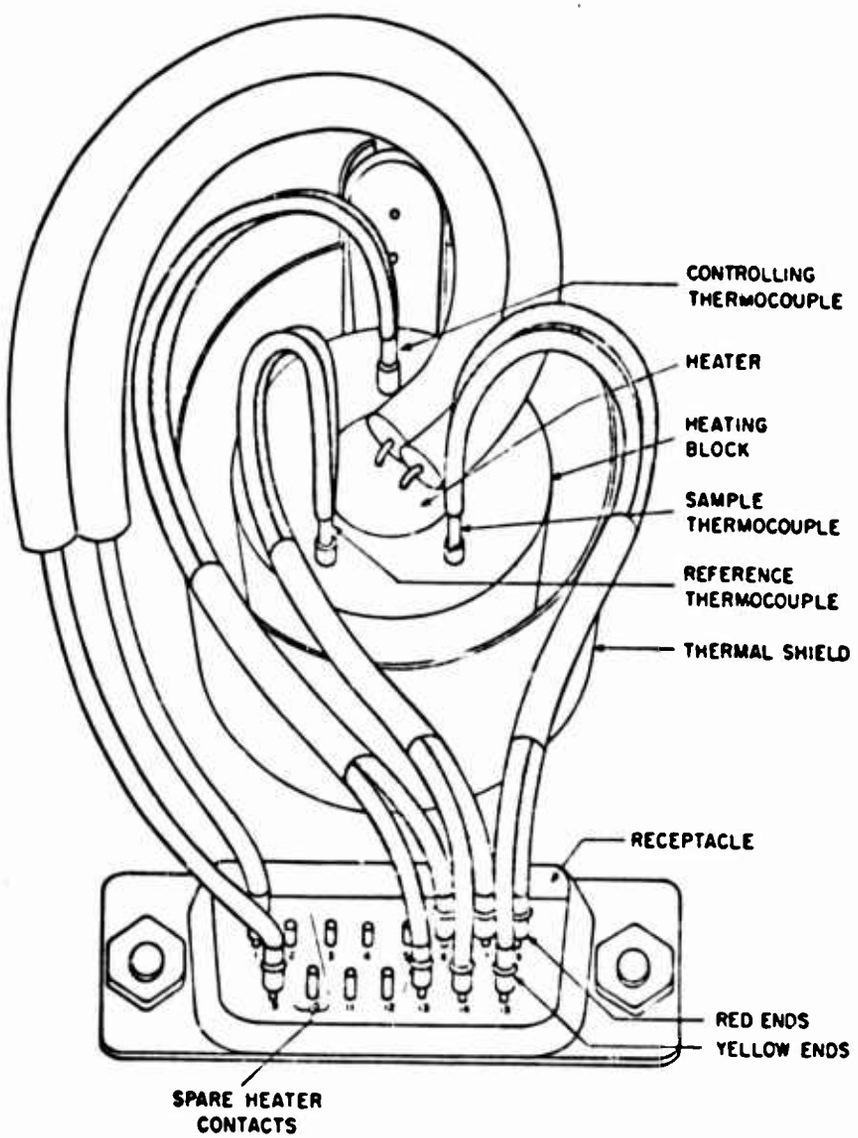

FIGURE 2A. Thermocouple - Heating Clock Arrangement [13]

A second run was conducted using a different procedure. The heating block and its associated thermocouples and heating element were placed in a Quick-Cool cell. This special cell allowed the sample and reference tubes to be quickly cooled without submerging the tubes and wetting the sample. The heating block was cooled to -147°C with LN admitted through the top of the Quick-Cool cell. When the temperature would register no lower, the LN was allowed to boil off and the temperature control was turned to HEAT. There was an initial surge in the recorder due to the fact that not all the LN had disappeared. The block and sample were heated until the sample decomposed (approximately 350°C). Only the temperature region from -147°C to +160°C was recorded since this included the temperature range of interest.

The two runs were conducted at heating/cooling rates of 10°C/min. and 20°C/min. respectively.

B. PHASE II

The impact tester used in this phase of the investigation was of original design. Although several impact testers were available through commercial sources, none satisfied the requirement for being easily cooled, easily operated and economically constructed. The impact tester and cooling system are shown in Figure 3.

The cooling system consisted of a cylinder of inert gas (argon), a 60 liter Dewar flask of liquid nitrogen, a Union Carbide Temperature Controller (TC-1), cooling coils around the impact tester chamber and a thermistor. The inert gas was used to pressurize the Dewar flask.

FIGURE 3. Impact Tester and Cooling System

The liquid nitrogen, pressurized from the top, was forced through a tube extending to the bottom of the Dewar. The inert gas was not used continuously since the boiling LN created its own pressure to force itself out. The LN entered the TC-1 temperature controller where it was expanded through a diaphram to become a cold gas. The cold nitrogen gas went through the coils of the impact tester and was released to the atmosphere. At the exit of the cooling coils was a thermistor which sensed temperature fluctuations and sent a signal to the temperature controller to control the speed of the diaphram and the nitrogen gas discharge. Coordination of the thermistor and the temperature controller maintained temperatures in the chamber at any desired setting to within 1°C. Chamber temperatures could be regulated from approximately liquid nitrogen temperatures to +10°C. Figure 4 shows approximate TC-1 dial settings for any desired temperature. The tubing connecting the apparatus was made of flexible gum rubber which became rigid when the system was operating. The tubing was insulated with plastic packaging material to reduce losses to the atmosphere.

The impact tester consisted of a cylindrical test chamber attached to a 1-inch thick steel base plate. The base plate was situated on 34-inch steel legs to give the operator comfortable access to the test chamber. The chamber supported three 200cm drop weight guide rails on its outer shell. The drop weight was a stainless steel tank ball bearing weighing 1.044kg. Figure 5 is a close-up view of the impact tester and related equipment. All components of the tester were constructed of high strength steel.

FIGURE 4. Approximate Temperature Controller Dial Settings for Chamber Temperatures

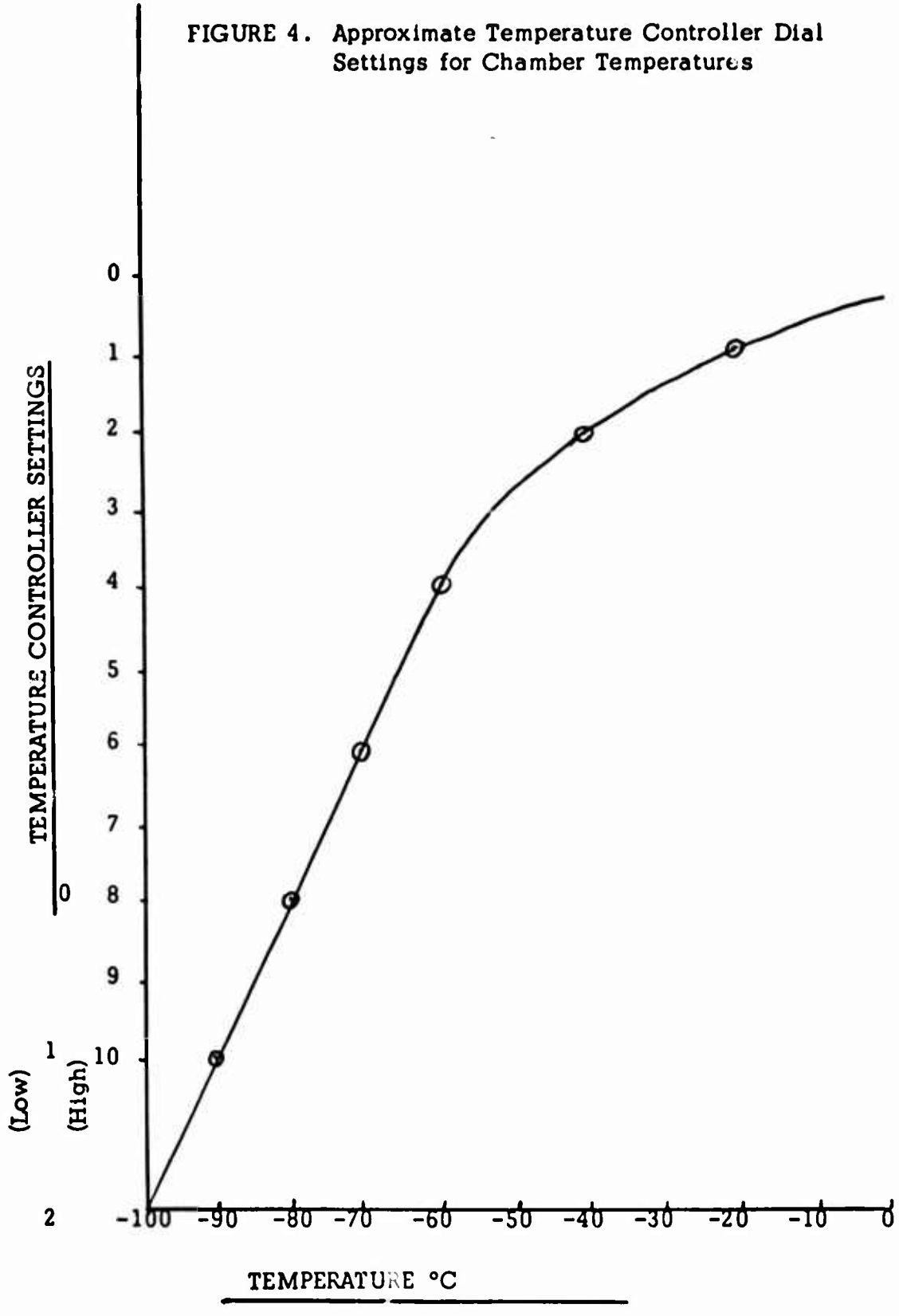

FIGURE 5. Impact Tester

The interior of the test chamber is shown in Figure 6. The test chamber consisted of a hollow cylinder (1-inch inside diameter) which housed the anvil, floating hammer, TNT sample, garnet paper and indicator paper. The chamber was surrounded by 1/4 inch copper cooling coils which cooled the entire chamber interior. Chamber temperatures were determined by a metal thermometer inserted through a rubber stopper in the top of the chamber. As the chamber was cooled, the temperature was monitored and adjusted by adjusting the "high" or "low" dial on the temperature controller. Once the desired temperature was attained and maintained for 30 minutes, the thermometer was removed and the impact test was commenced.

The hammer and anvil were machined from high strength tool steel. Their relative sizes and positions are shown in Figure 7 with the hammer on the left representing the top of the test chamber. The TNT sample was placed between the hammer and anvil on a small disc of garnet paper. The garnet paper was used to increase the relative sensitivity of the small sample of TNT (23.0 ± 2.0 mg.) in order to keep the drop heights within the limits of the apparatus. Each garnet paper disc was cut from the same sheet to reduce experimental error. The proper sample size and specifications of the garnet paper were determined in a previous experiment by Professor J. E. Sinclair [11]. The TNT sample was covered with a thin stainless steel foil disc to prevent marring of the hammer surface. A piece of plain white indicating paper surrounded the TNT sample. It was fitted loosely between the hammer and anvil to prevent

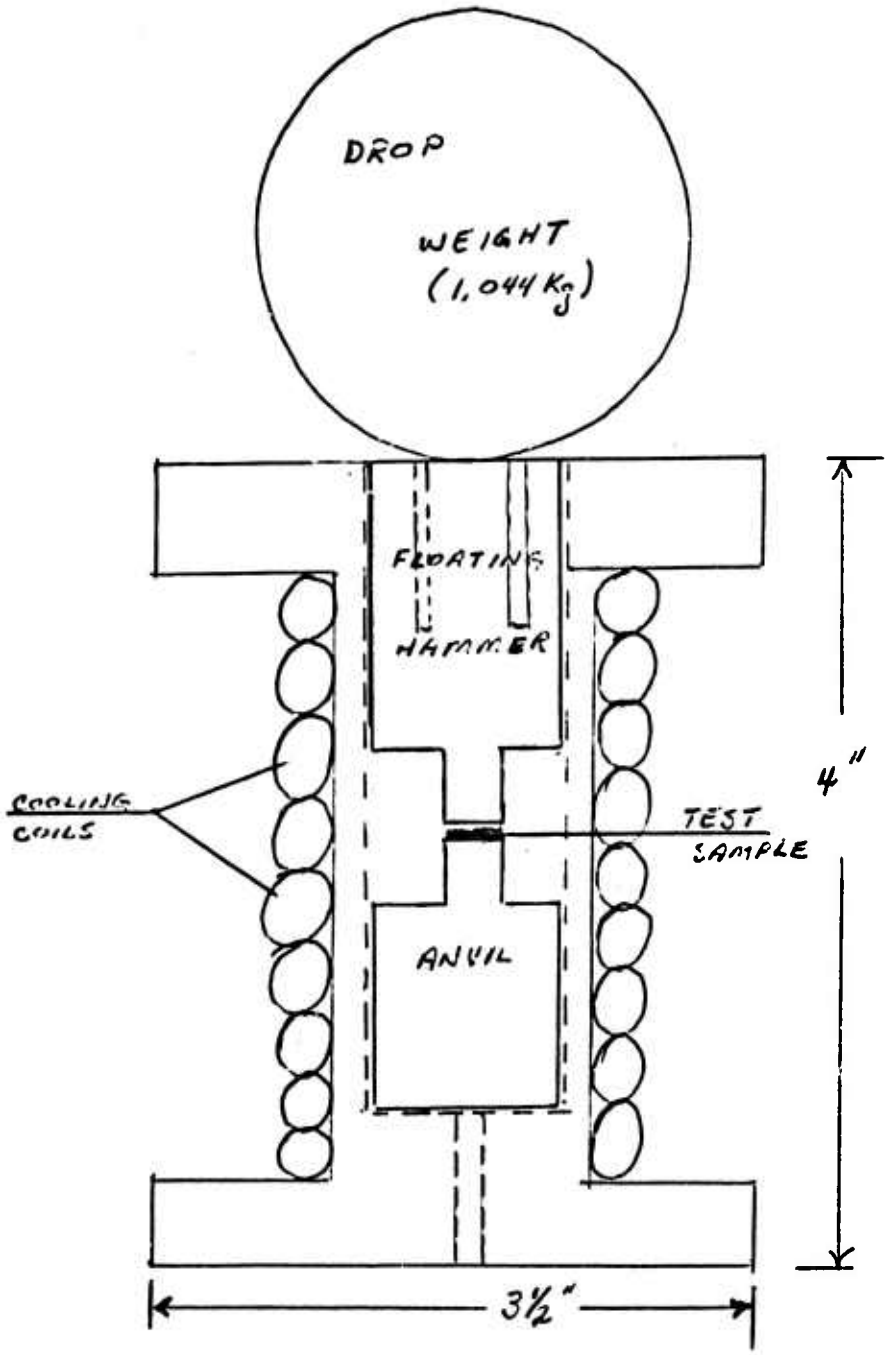

FIGURE 6. Schematic of Impact Tester Chamber

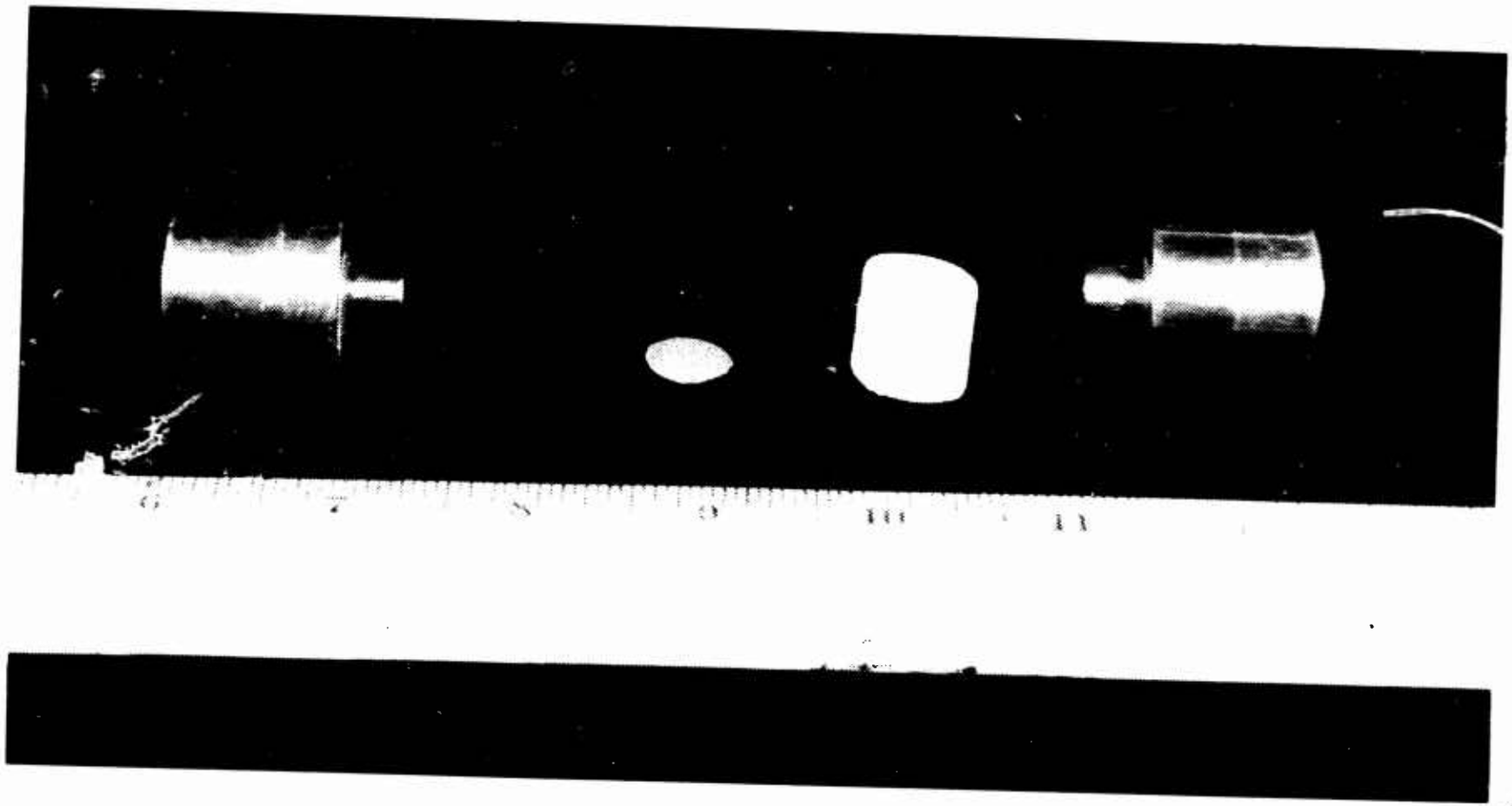

FIGURE 7. Internal Components of Test Chamber

interference with the impact. An explosion of the TNT sample produced a burn mark on the indicator paper and/or the garnet paper.

Since reproducibility of results is considered a major problem in impact testing [9, 10], a set of rules was established so that each of the runs would give meaningful statistical results. All runs were made under the same rules:

1). Each run at a specified temperature consisted of at least 20 recorded drops. Additional drops were made until the next change occurred - either no explosion (N) or explosion (E). (See Figure 10.)

2). Each run commenced with a series of ranging drops arbitrarily started at 180cm. If an explosion occurred, the height was reduced 20cm. If no explosion occurred, the drop height was increased 20cm. This pattern continued until the first change (either N or E). The next drop was made 5cm above the change point if N or 5 cm below if E. The recording of the drop data was started at this point.

3). If the occasion arose where it was too difficult to judge if an explosion had occurred (as indicated by any burn marks), the drop was considered no explosion (N) and the next drop height was increased 5cm.

4). The general procedure for drop height determination was that if N occurred, the next drop height was increased 5cm or that if E occurred, the next drop height would be reduced 5cm.

5). Determination of the 50% point (FPP), i.e., the height that yielded 50% explosions, was made after the run was completed. The explosion

percentages for the various drop heights were plotted on a probability graph. The point where the best straight line crossed 50% probability was the FPP for the run.

A concept employed by Sinclair was also used in this investigation [11]. If on any given drop an explosion occurred it was assumed that an explosion would have occurred at a greater height for that particular drop. This was denoted by e. The same was true if no explosion occurred in that any height below this was assumed to have no explosion occurring. This was denoted by n. (See Figure 10.)

A factor, called RFP, for defining reproducibility or reliability level was determined for each run. An empirical formula was developed by Sinclair [11] to represent RFP:

$$1 - \frac{n-2}{T-1} = RFP$$

where n = the number of 5cm increments of drop height used in the run between 0% E and 100% E, and T = the number of recorded drops for the run.

The actual procedures for conducting each run were practiced before any test runs were made. This was done in order to reduce the handling time of the explosive in the cold environment, to reduce the time that the inner parts of the test chamber were exposed to the atmosphere, and to better standardize procedures to give more reliable results. A typical run was conducted using the following procedures:

1). The liquid nitrogen Dewar was pressurized with inert gas. This required about 100 cubic inches of gas at 2 psig.

2). The TC-1 dial was set for the desired temperature. The thermistor was connected, and the system was cooled to the desired temperature. This took about 2 hours at the lower temperatures.

3). The chamber temperature was adjusted to within 1°C and was allowed to equilibrate for 30 minutes to correct for system fluctuations.

4). The hammer and anvil were placed in the test chamber to chill for 20 minutes.

5). The hammer and anvil were removed from the chamber, and the temperature was checked to insure correctness.

6). The anvil was prepared for testing. This particular step was done as quickly as possible but with reasonable accuracy. The procedure included placing the garnet paper on the head of the anvil, measuring the TNT sample with a specially constructed glass spoon, placing the sample in the center of the garnet paper, placing the steel foil disc over the sample and placing the white indicator paper around the sample.

7). The anvil was placed in the chamber. Laboratory tongs had to be used and handling the anvil was quite difficult.

8). The hammer was placed in the chamber so that it met the anvil head to head. Raising and lowering the hammer and anvil in the chamber was accomplished by means of a rod attachment rising through the base of the chamber and base plate.

9). Once the sample was in place in the chamber, a 2 - 5 minute waiting period began to allow the sample to cool to chamber temperature.

10). The drop was made from a predetermined height.

11). The hammer and anvil were removed to inspect for explosion. Results were recorded. The hammer and anvil were cleaned, and steps 6 - 11 were repeated for the next drop.

III. RESULTS AND DISCUSSION

A. PHASE I

The results of the DTA investigation are shown in Figures 8 and 9. Figure 8 is the thermogram obtained from the first DTA run. The cooling rate was 10°C/min. The large exotherm shown on the upper scale represents the heat released as the liquid TNT supercooled and turned to solid crystals. The lower scale is the temperature range of particular interest. There was no significant indication of any endothermic or exothermic reaction.

Figure 9 is the thermogram obtained from the second DTA run. The TNT was initially cooled to -147°C and then heated at 20°C/min. to decomposition. The upper scale shows a large sloping curve which was caused by irregular heating in the block from a voltage surge to the heater element. The temperature range of interest (the upper scale) again showed no significant exo- or endothermic reaction. However, McCrone [8], in his work on crystallization of TNT, has mentioned that a change may occur in the structure of TNT without showing any significant endothermic or exothermic reaction. A slower heating rate might have yielded some indication of change. However, it was not feasible to heat at a slower rate because of uncontrolled atmospheric heating nor was it feasible to cool at a slower rate because of the limited liquid nitrogen supply.

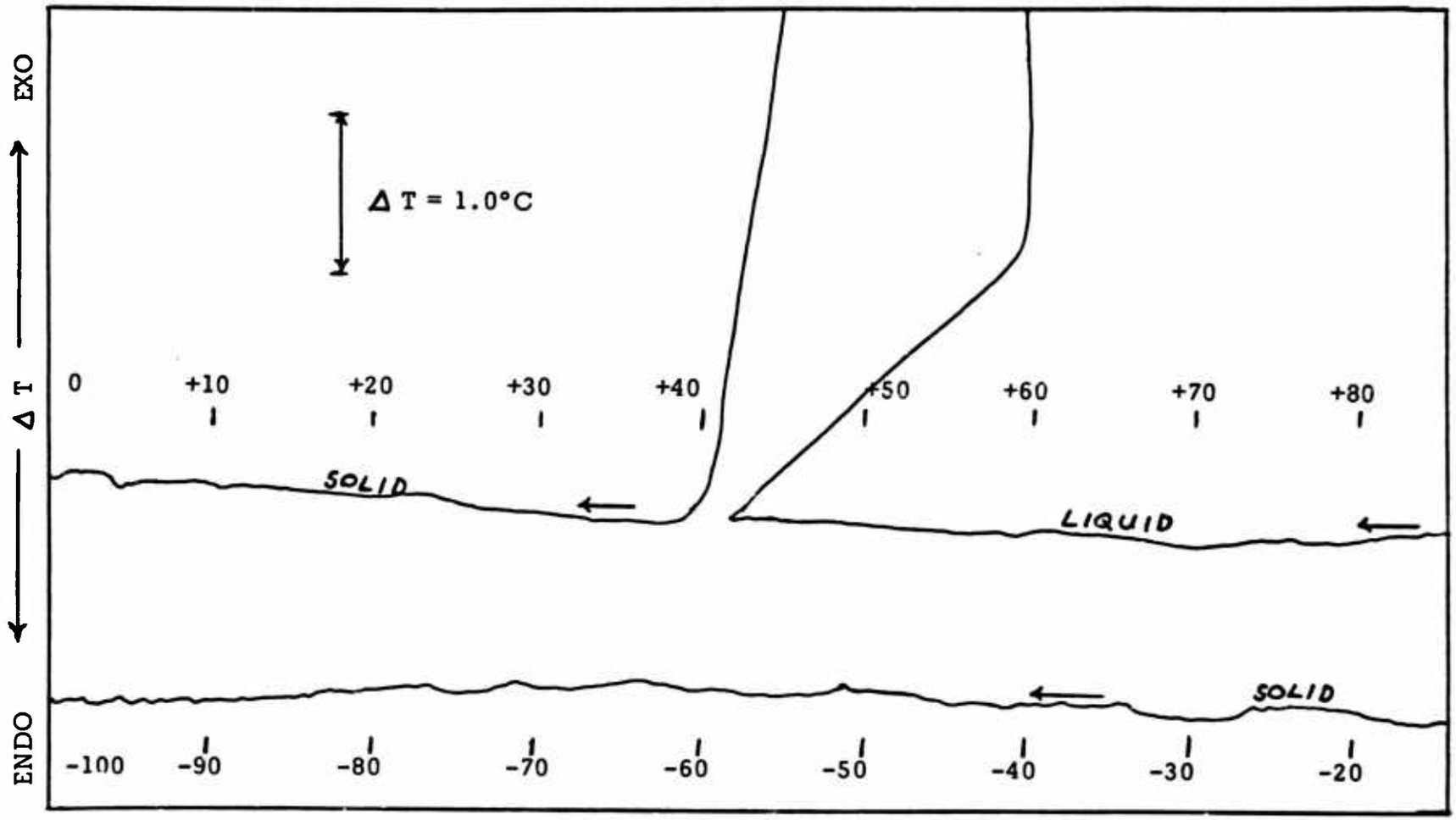

FIGURE 8. DTA Thermogram (#1)

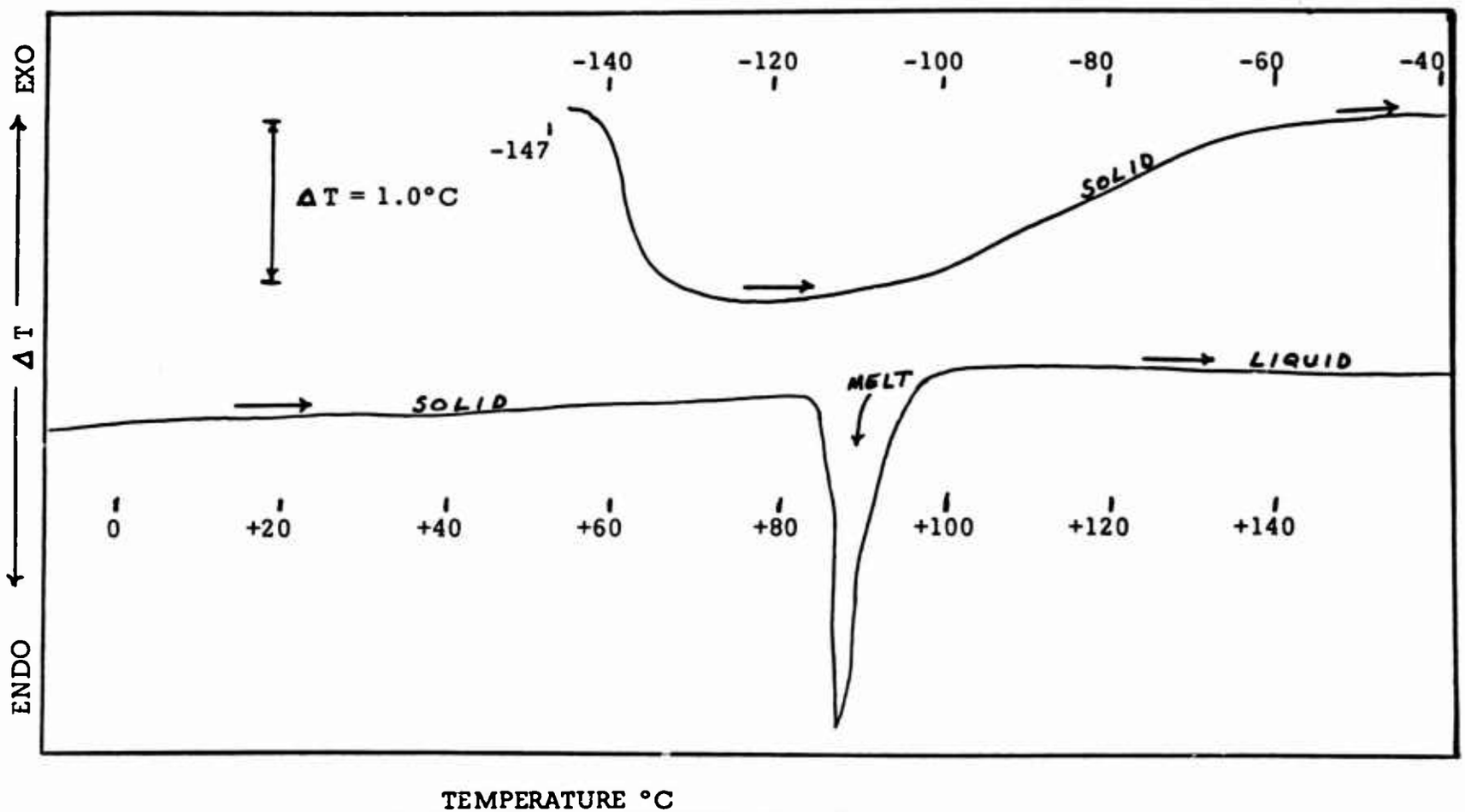

FIGURE 9. DTA Thermogram (#2)

B. PHASE II

Figure 10 is a sample data sheet used for all runs in the impact tests. This particular example is for data collected on Run 1 at -100°C. The upper case letters, N and E, represent the actual drop recordings. The lower case letters, n and e, represent the assumed no explosion - explosion points described previously. The probability curve at the bottom of Figure 10 is typical of that used to find the FPP for all runs. Experimental data are summarized in Table I.

The FPP or mean explosion point for each run was plotted against temperature (Figure 11). The FPP increases linearly as the temperature decreases which suggests that, as the explosive is cooled to a sufficient temperature (near absolute zero), it becomes very insensitive to impact.

The potential energy of the drop weight associated with each FPP (Table I) was also plotted against temperature. The potential energy, assumed to be totally transmitted to the TNT sample, was converted to units of kcal/mole for the 23.0mg sample of TNT. If the energy losses due to friction and to elastic collision between the ball and hammer are neglected, the potential energy of the drop weight may approximate the energy necessary for the explosive reaction to occur. However, the losses in impact testing are too great for this to be considered a valid argument. A portion of this potential energy must be used to raise the sample temperature to the point where "hot spots" occur and the explosion takes place. This additional energy required to raise the

FIGURE 10. Sample of Impact Data Sheet

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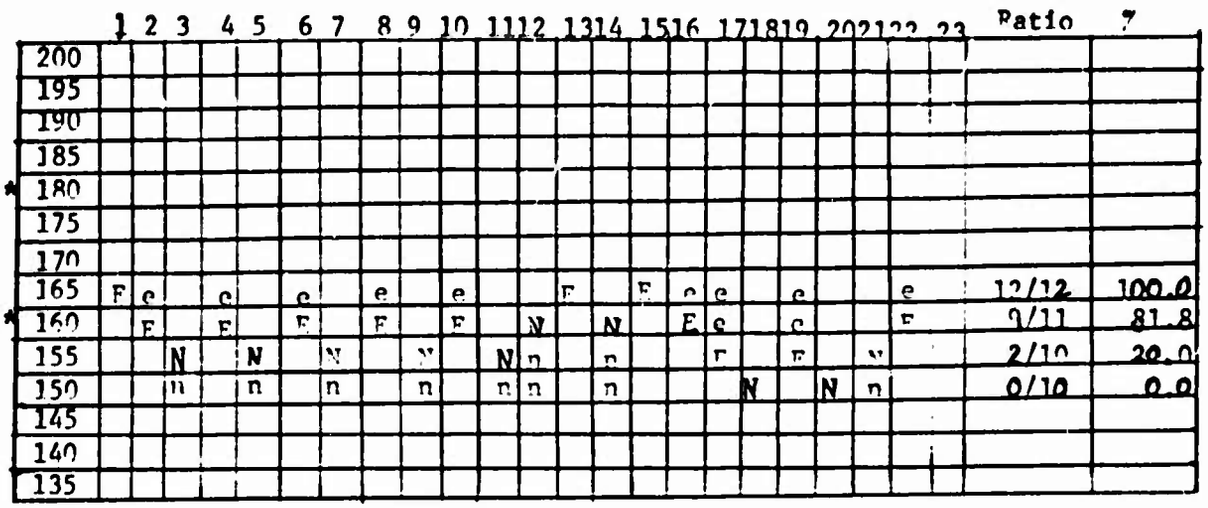
Impact Test Data Sheet



* Denotes ranging shot

n = 4, T = 22 RFP = $1-\left[\frac{n-2}{T-1}\right]$ = 1 - 2/21 = .9045

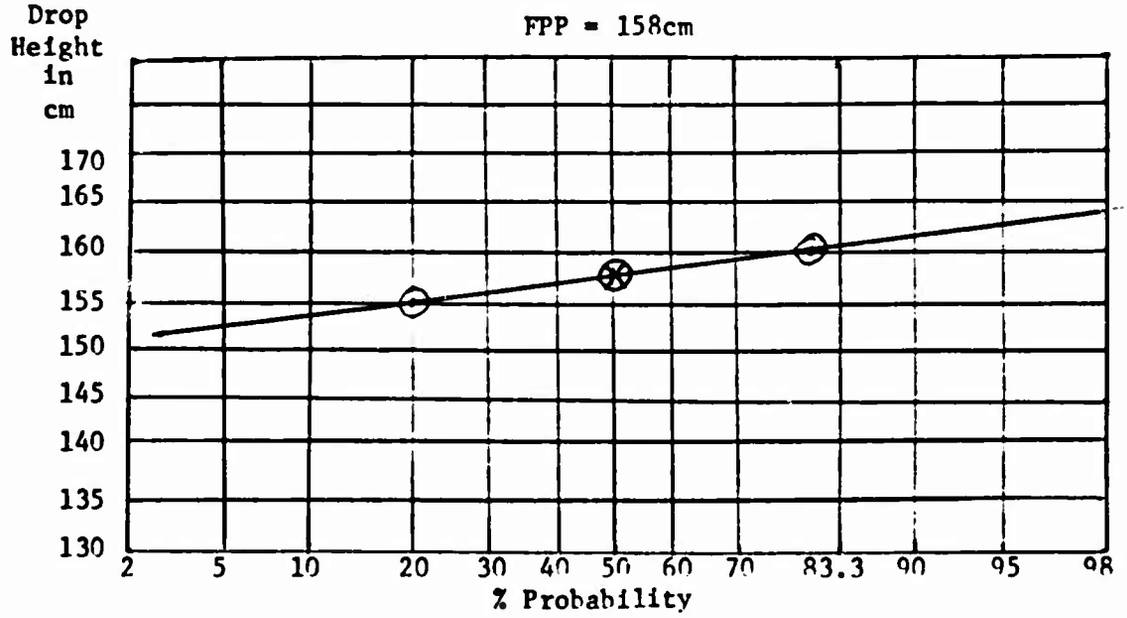


TABLE I

IMPACT SENSITIVITY DATA FOR PURE TNT

Run	Chamber Temperature	FPP (cm)	RFP (%)	Energy Input at FPP (kcal/mole)	Test Date
1	-100°C	158	90.5	38.1	11-16-71
2	- 90°C	149	80.5	36.0	11-17-71
3	- 80°C	147	85.0	35.4	11-19-71
4	- 70°C	148	81.0	35.7	11-23-71
5	- 60°C	143	85.7	34.5	12-2-71
6	- 50°C	133	85.0	32.2	12-7-71
7*	- 40°C	144	85.0	34.8	12-10-71
8	- 30°C	128	86.0	30.9	1-11-72
9	- 20°C	115	75.0	27.8	1-12-72
10	- 10°C	104	80.0	25.1	1-13-72
11	0°C	108	90.0	26.2	1-17-72
12	+ 22°C	110	86.0	26.4	1-19-72
13	-110°C	167	65.0	40.4	1-19-72
14*	- 40°C	93	70.0	22.5	1-25-72

Drop weight = 1.044kg

Steel disc thickness = .13mm

TNT sample weight = 23.0 $\pm$ 2.0mg

Sample density = greater than 1.0 g/cc

Hammer strike surface area = 1.33 cm^2

FIGURE 11. Fifty Percent Point Versus Chamber Temperature

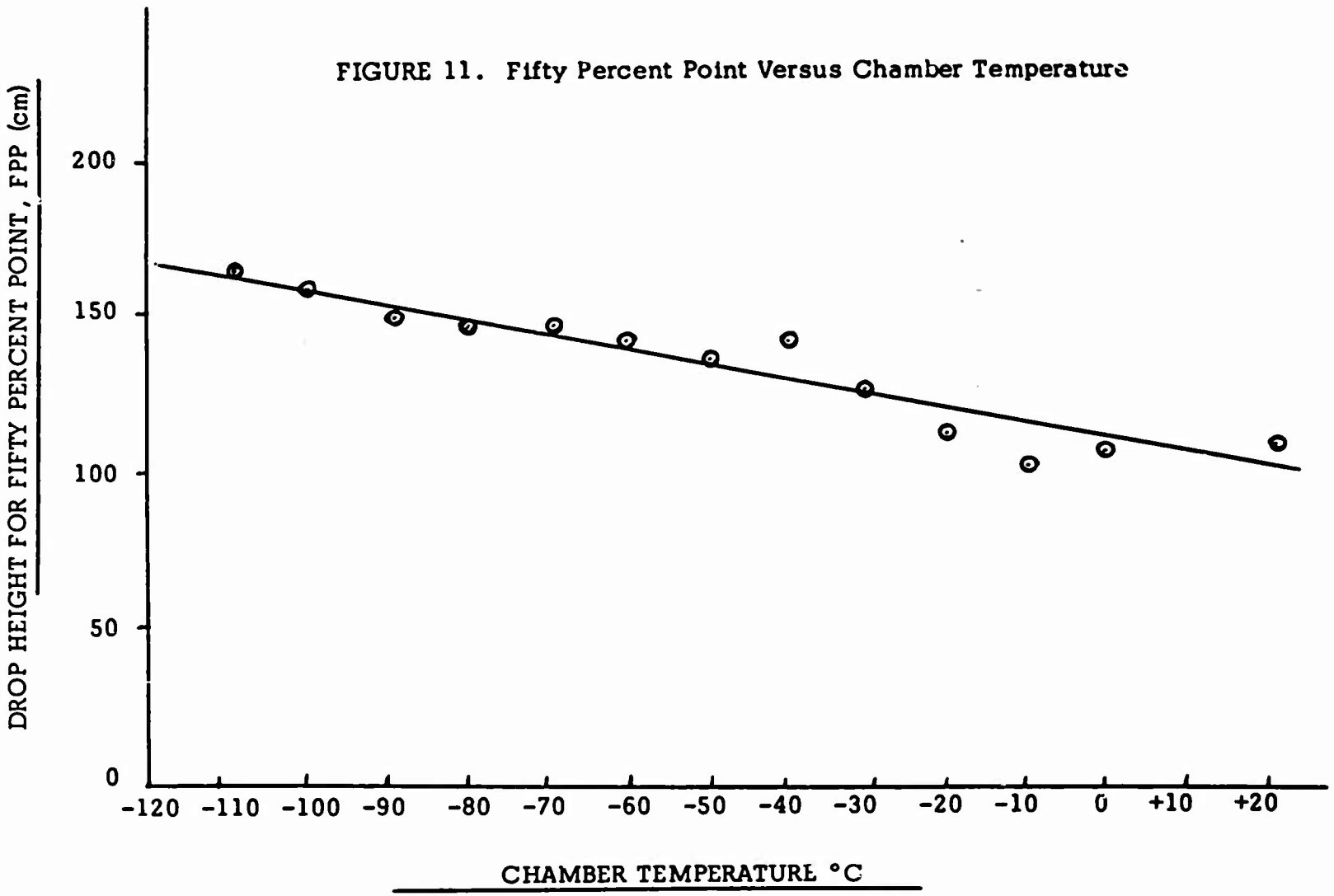
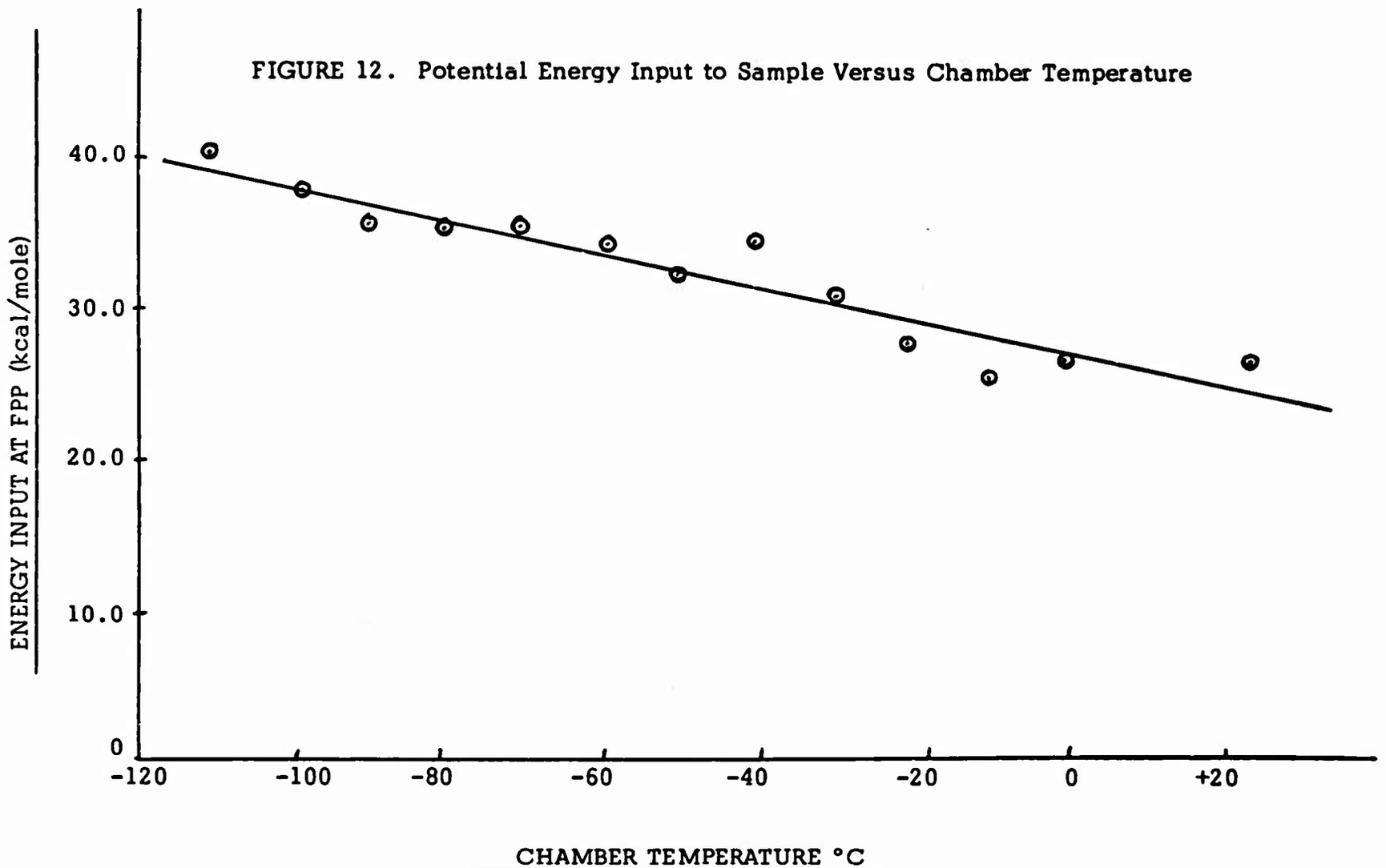


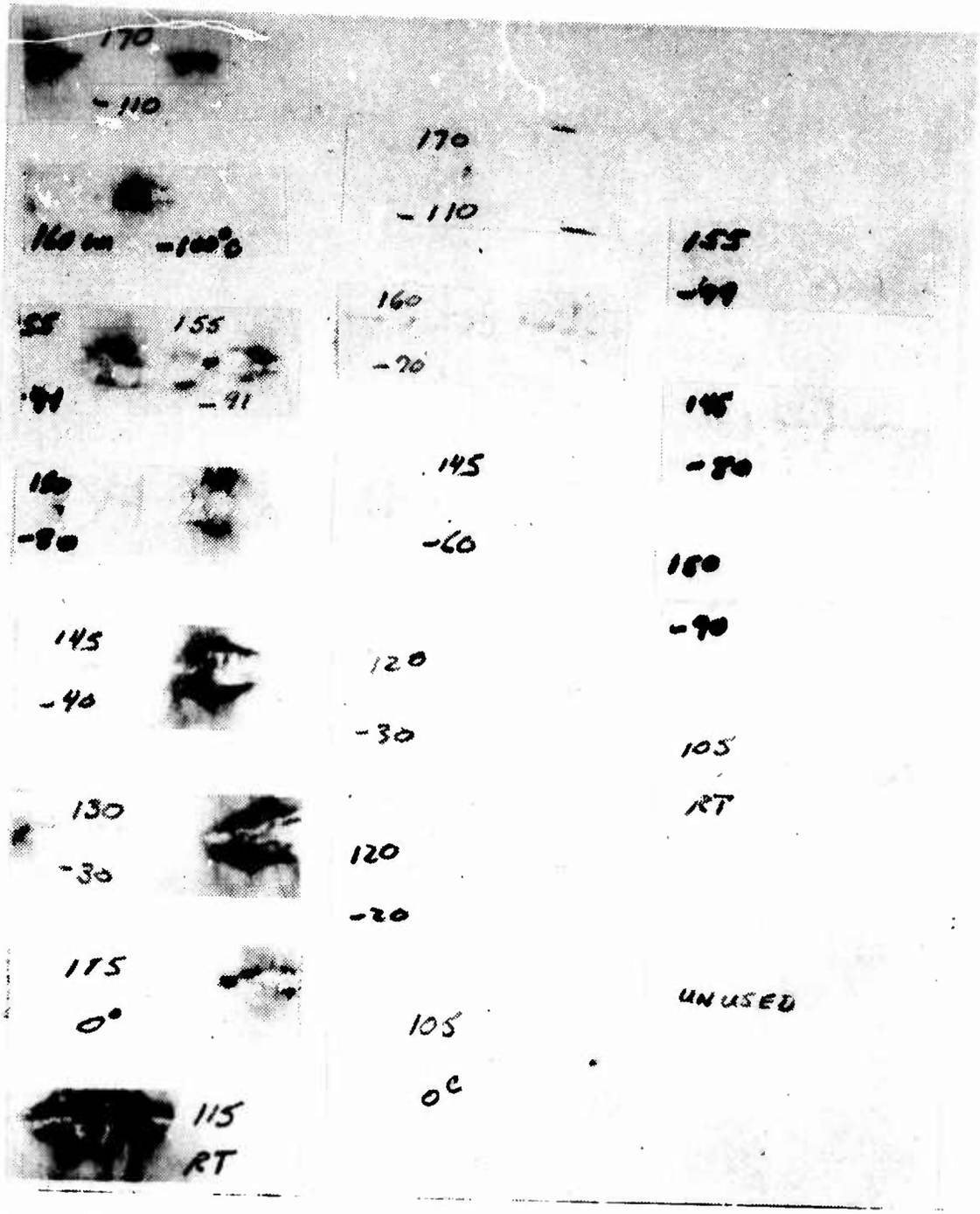
FIGURE 12. Potential Energy Input to Sample Versus Chamber Temperature



temperature to the explosion point may account for the linearity of the energy-temperature relation in Figure 12. The slope of Figure 12 is the heat capacity/unit mass of the sample or specific heat of the TNT sample. The value of the slope (.50 cal/gm -°C) was compared to tabulated values of specific heat for TNT. There was significant error between the tabulated and experimental values, but these errors may be attributed to the energy losses at impact and other system anomalies. From the work of Smith and Richardson on interpretation of sensitivity data [12], it appears that energy alone is not adequate for interpreting impact data. A rate term such as force/time or kinetic energy/time should be included in the analysis. However, the energy data obtained in this investigation fall within the range of activation energies tabulated from other experiments [5].

Sample density is an important consideration when interpreting impact data. The fine grain sample used in this investigation had a measured density of 0.6 g/cc. The sample was compressed by the hammer when placed in the chamber, but the density became only slightly greater than 1.0 g/cc.

Figure 13 shows samples of explosion indicator paper after drops were made. The left column shows definitely that an explosion occurred while the right column shows that no explosion occurred. The center column shows cases of only slight explosion and indicates the difficulty in judging between explosion and no explosion. There was, consequently, a certain amount of judgement error involved which may have influenced the determination of the FPP.



a. Positive b. Slight c. No Explosion

FIGURE 13. Samples of Explosion Indications

Figure 14 is a microphotograph of an exploded sample on garnet paper. The blackened spot in the center of the photo was the only indication of any reaction. This is again indicative of the difficulty in determining some results. Figure 15 is the same type of photo except in this case, there was definitely no explosion. Both of these photos are of samples after drops at room temperature (+22°C). Note that the sample has been compressed to a brittle, shiny substance.

Another observation which proved to be significant was the formation of ice in and around the test chamber. Ice must also have formed between the sample particles; however, this could not be seen. The ice created little interference with the impact since it was continually cleaned away. However, in the temperature range of -50°C to -30°C, there seemed to be a greater amount of fine ice crystals which made movement of the hammer and anvil in or out of the chamber quite difficult. The FPP at -40°C seemed quite out of line with the surrounding data points and was, therefore, rechecked (Run 14). Again, the FPP did not appear correct. The relative humidity in the laboratory had been held between 45 and 50 percent. However, the relative humidity outside the laboratory was significantly higher during Run 14 than during Run 7 or any other run. This perhaps accounts for the greater ice build up, and, hence the disparity in FPP.

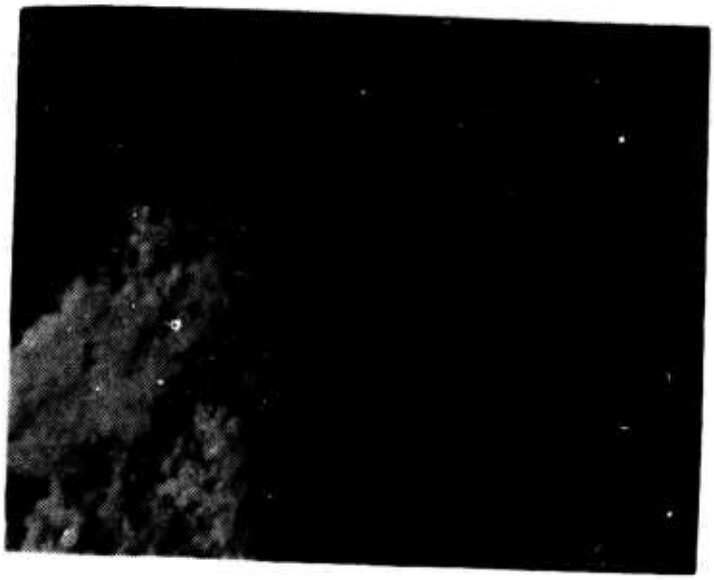


FIGURE 14.

Microphoto of Exploded TNT at Room Temperature

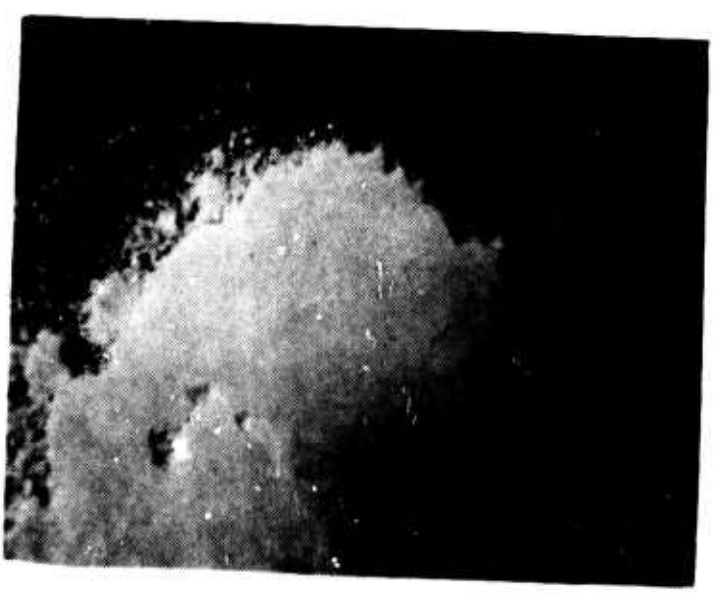


FIGURE 15.

Microphoto of Unexploded TNT at Room Temperature

IV. CONCLUSIONS

Experience has shown impact sensitivity of explosives to be a mysterious characteristic. Explosions have occurred when unfused bombs drop a few feet from a handling sling, yet a fused bomb may drop from high altitude only to land as a "dud." In general, though, data gathered statistically can give meaningful sensitivity characteristics.

The sensitivity of TNT is decreased by lowering the temperature. This may be represented as an increased FPP or input energy with decreasing temperatures. DTA results using moderate heating/cooling rates indicate that reduced temperatures do not affect crystalline structure of TNT. If the DTA results provide a correct indication of crystalline structure change, the results indicate that improved safety factors and handling conditions exist at lower temperatures.

It would be difficult to transpose these experimental results for TNT to another explosive substance. Other explosives, such as ammonium nitrate, show definite transformations in crystalline structure at lower temperatures and may exhibit quite different sensitivity at lower temperatures. However, the results could possibly be applied to a great many high explosives which contain large amounts of TNT.

It is recommended that further studies be conducted on impact sensitivity at lower temperatures than those included in this investigation. The results of decreases in temperature may show that the

sensitivity or impact energy required is not linearly related to temperature at all but follows a higher order relation. Consequently, the impact sensitivity near absolute zero may be reduced to zero. Work in this area is presently being undertaken by the research laboratories at the Picatinny Arsenal in New Jersey under the auspices of the Justice Department.

Further applications of these studies may be directed toward the explosive devices used in space. The low temperatures encountered in space may desensitize these devices enough to cause system failure or create a need for greater initiation energy.

The apparatus used in the impact test of this experiment were simply constructed with only minor regard for material, measurement of other data and effects of atmospheric conditions. Future experiments in this area should be conducted in a dry atmosphere (less than 10% relative humidity). This would reduce the formation of ice in the chamber and produce more realistic results since most explosive devices are hermetically sealed in a dry atmosphere. The apparatus should be constructed of high carbon steels to reduce deformations from impact. Other measurements might also be incorporated, such as a force gage to make force-time measurements. Incorporating other devices in or around the test chamber increases the difficulty in insulating the chamber for efficient cooling or in handling the sample.

Research in this field cannot, of course, be limited to TNT but should include all high explosives, boosters, primers and propellants. Improved knowledge of sensitivity improves safety and benefits both the users and manufacturers.

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