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NAVAL ORDNANCE LABORATORY MEMORANDUM 10289

21 December 1949

J. M. Rosen  
Explosives Division Files

A New Apparatus for the Determination of the Ignition  
Temperature of Explosives. (NOL-37-Re2c-19-1 (a))

A new type of apparatus for use in determining the "ignition temperature" of explosives has been devised. It consists of a tapered aluminum bar, heated at one end, and drilled with a uniformly spaced series of sample holes along length of the bar. In this way a number of different and relatively constant temperature levels is obtained. The hot bar is enclosed in a Transite box, and is operated usually at a temperature of about 460°C at the hot end. Iron-constantan thermocouples spaced along the bar are used to make the temperature measurements. Several trials are made on each explosive using the up and down method and a 50% ignition temperature is reported. For each trial about 10-30 mg. of explosive are used. The 50% ignition temperature seems to be fairly reproducible for a number of explosives. This method gives a larger difference between the values of the ignition temperature for difference explosives than other methods commonly used.

Fwd: The apparatus and technique described for determining ignition temperatures have proved useful in studying the thermal properties of explosives. It is believed that all statements made and data given are correct. However, The Naval Ordnance Laboratory is not committed to endorse either.

- Ref:
- (a) OSRD 830
  - (b) Picatinny Arsenal Technical Report 1372
  - (c) OSRD 787
  - (d) Naval Powder Factory Methods Book
  - (e) OSRD 1986
  - (f) Dennis and Shelton, J. Am. Chem. Soc., 52, 3128 (1930)
  - (g) A.M.P. Report No. 101.1 R

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Encl: (1) Plates 1 and 2  
 (2) Figures 1 and 2  
 (3) Tables I through IV

## INTRODUCTION

1. The "ignition temperature" has been used as one of the criteria of the stability of an explosive, references (a) and (b). It is the lowest temperature at which ignition will take place under a particular set of conditions and usually is determined by one of the following methods. A small sample of explosive is sprinkled on the surface of a heated Wood's metal bath, reference (c), and the lowest temperature at which ignition takes place is noted. In another procedure, a sample is placed in a glass test tube immersed in a Wood's metal bath which is heated at a specified rate, reference (d). The lowest temperature at which the sample ignites is reported as the ignition temperature. In a third method, reference (e); the ignition temperature is reported as well as the time lag before ignition takes place. This is accomplished by lowering a cap or container which holds the sample into a heated Wood's metal bath. The copper container is covered with a metal cap to which has been soldered a fine wire. As soon as the container touches the heated bath an electrical circuit is completed which starts a timer. When the sample ignites, the cap is blown off which breaks the circuit and stops the timer. The average time for ignition of the explosive at a series of temperatures is measured.

2. The three procedures described are rather slow and also involve the use of a liquid metal bath which is sometimes hazardous and inconvenient.

3. It was our aim to find a suitable method for determining the ignition temperature not involving the use of a liquid metal bath. A knowledge of the ignition point would be used to judge whether or not it was safe to subject explosive samples to heat for such purposes as drying operations or gas evolution studies. Another objective was to gain more information about the ignition temperature in order to correlate this with other properties.

4. The "hotbar" is commonly used by organic chemists to get a rapid approximation of the melting point, reference (1). It consists of a metal bar usually about 36" long and 1" square, which is heated at one end. There is a temperature gradient along the bar, so that a large number of temperature levels are available at any one time. The ignition point apparatus described below is a modification of the "hotbar" idea.

## APPARATUS

1. The ignition point apparatus consists of a tapered aluminum bar 1 1/2" long. It tapers from 2" diameter near one end to 3/4" diameter at the other end. A top view of the bar is shown

in Figure 1. A 2" diameter cylindrical section about 7" long at the large end is wound with 24 turns of #22 Nichrome wire. This Nichrome spiral is insulated with about 1" of asbestos and connected to the 115 volt power supply through a Variac. Sample holes are  $7/32$ " diameter and  $1/4$ " deep with a flat bottom. Adjacent holes are  $7/16$ " apart on center. A bar with the above dimensions will have sixty holes along the long axis, providing sixty different temperature levels. In order to reduce the heat loss from the surface, the bar is covered with  $1/32$ " asbestos paper. Iron-constantan thermocouples made from #30 wire, are located at every fifth temperature level and these are connected to a selector switch. The thermocouples are inserted in a small hole reaching  $1/4$ " below the surface. The temperature at any one of the several locations may be determined by means of a potentiometer which is connected to the selector switch. A Leeds and Northrup potentiometer No. 8862 has been found satisfactory. The bar is mounted in a box about 7" wide, 7" deep, and 35" long, made of  $1/2$ " Transite. Plate 1 is a closeup view of the bar. The top is covered with a number of 6" x 9" sections of Transite, each of which has a window opening covered with mica. Plate 2 shows the complete ignition temperature apparatus ready for operation.

#### BAR TEMPERATURE

6. A temperature curve can be constructed as shown by Figure 2. A fairly smooth curve is obtained by plotting the temperature against temperature level. In reality a whole series of curves can be made for the bar, depending upon the power input. Figure 2 shows curves for 80 and 92 volts. The most useful temperature range has been obtained by operating at 92 volts. This yields a high temperature of  $460^{\circ}\text{C}$ , which is high enough to ignite T.T., and a low of  $150^{\circ}\text{C}$ , which is below the ignition temperature of almost all of the materials studied. It requires about 2-3 hours to heat the bar, after which time it remains surprisingly constant. Adjacent temperature levels at the high range differ by  $9^{\circ}\text{C}$  and by about  $4^{\circ}\text{C}$  at the low range.

#### PROCEDURE

7. The sample, measured by means of a small scoop, is placed in one of the sample holes of the heated bar. A top cover is lifted high enough to allow entry of the scoop. After discharging the sample, the scoop is rapidly withdrawn and the cover is lowered in place. Ignitions can be determined readily by viewing the bar through the mica window. The A.M.P. "up and down" method, reference (g), is used. When 50 trials are made on a sample the relatively few trials (10-20) are made the 50% ignition point is estimated by plotting per cent probability against temperature and noting the temperature where the 50% level is crossed.

Ignition generally occurs within a few seconds after introduction of the explosives sample or not at all. Delays beyond perhaps 10 seconds are unusual. As the heated sample has generally partially or wholly decomposed if ignition does not occur, a fresh sample must be used for each trial.

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8. The sample holes are cleaned by means of a flat end high-speed twist drill. Carbon or other residue can be removed by turning the drill in the sample hole.

#### SAMPLE SIZE

9. Samples in the range of 10-30 mg. are used generally. For most of the explosives studied the 50% ignition point is not dependent on sample size when working in this range as shown by examples in Table I. For primary explosives smaller samples of 3-5 mg. are used.

#### REPRODUCIBILITY

10. The 50% ignition point seems to be reproducible for a number of the explosives using the apparatus and procedure described. Table I gives an indication of the degree of reproducibility which can be achieved.

11. The 50% ignition point of aluminized Composition A was determined eight times, each determination being made on a different day. The data are given in Table II. There is a difference of 12° between the high and low value. Although the ignition point of aluminized Composition A is fairly reproducible, the data are not homogeneous at the 95% level when treated statistically by the method of reference (g).

#### IGNITION TEMPERATURE OF EXPLOSIVES

12. Using the procedure described, the ignition points of a number of explosives were determined. Average values obtained are shown in Table III. An interesting feature of the procedure is the wide spread between the ignition temperatures of the explosives. Up to the present time no attempt has been made to correlate ignition temperature with other properties. In Table IV is shown ignition temperatures as determined by three different methods. The HLL values are not too different from the H0L values. These two are determined in a rather similar way in principle - the rapid application of high temperature. On the other hand, the MFP ignition temperatures are obtained by heating a sample at a specified rate (5-10° per minute) until the explosive ignites. The starting temperature is 100-125°C. It should be pointed out that the ignition temperatures as determined by means of the aluminum hot bar are 50% points - representing a temperature at which there is a 50% probability of ignition.

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.218 DIA. SAMPLE HOLES .25 DEEP  
60 HOLES SPACED .437 APART

26.50

7.0

2.0

INSULATION

NICHROME COIL

NO. 60 (40) DRILL THERMOCOUPLE HOLES  
.25 DEEP SPACED BESIDE EVERY 5TH SAMPLE  
HOLE.

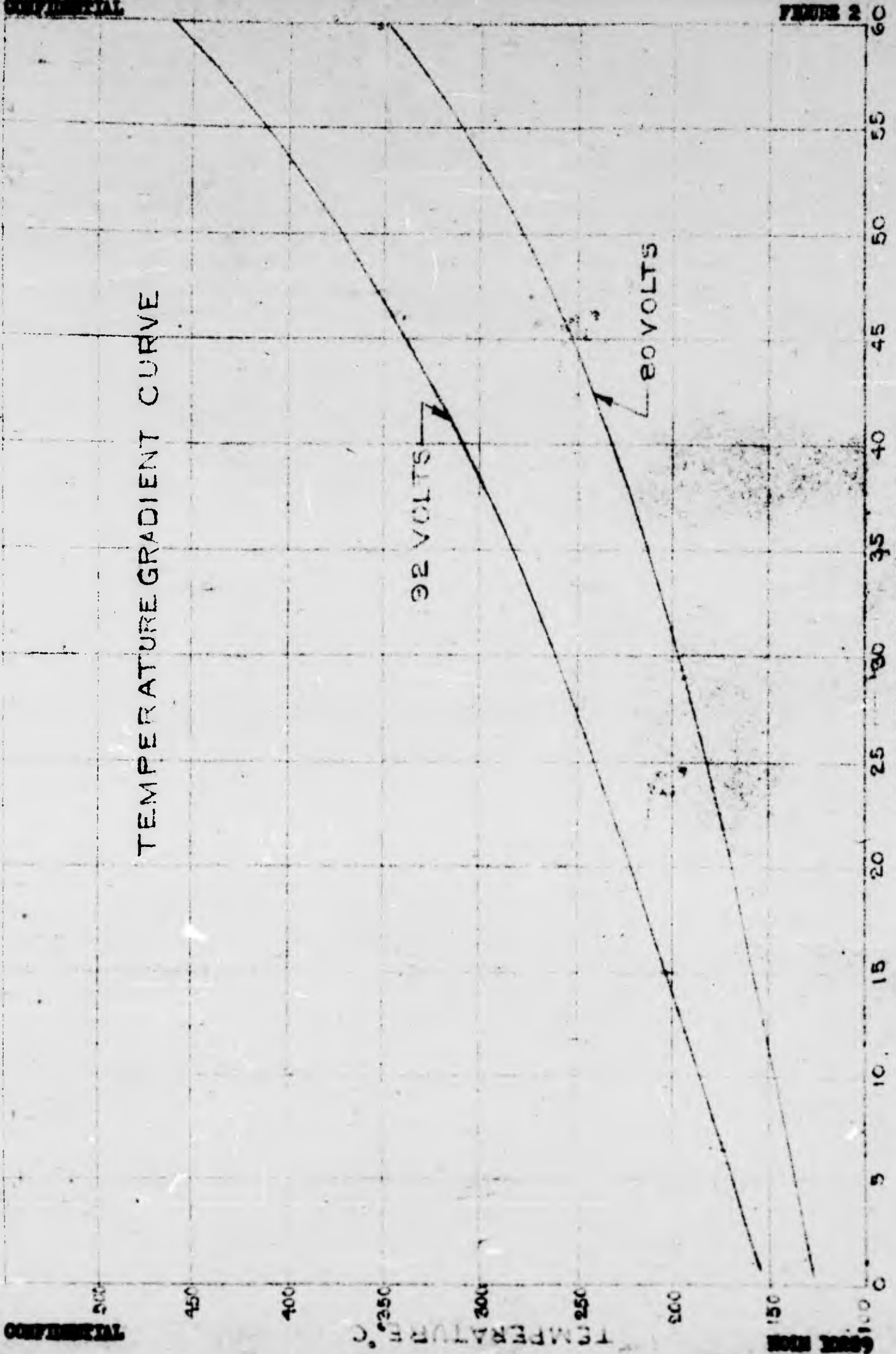
# ALUMINUM IGNITION TEMPERATURE BAR

ALL DIMENSIONS ARE IN INCHES

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# TEMPERATURE GRADIENT CURVE

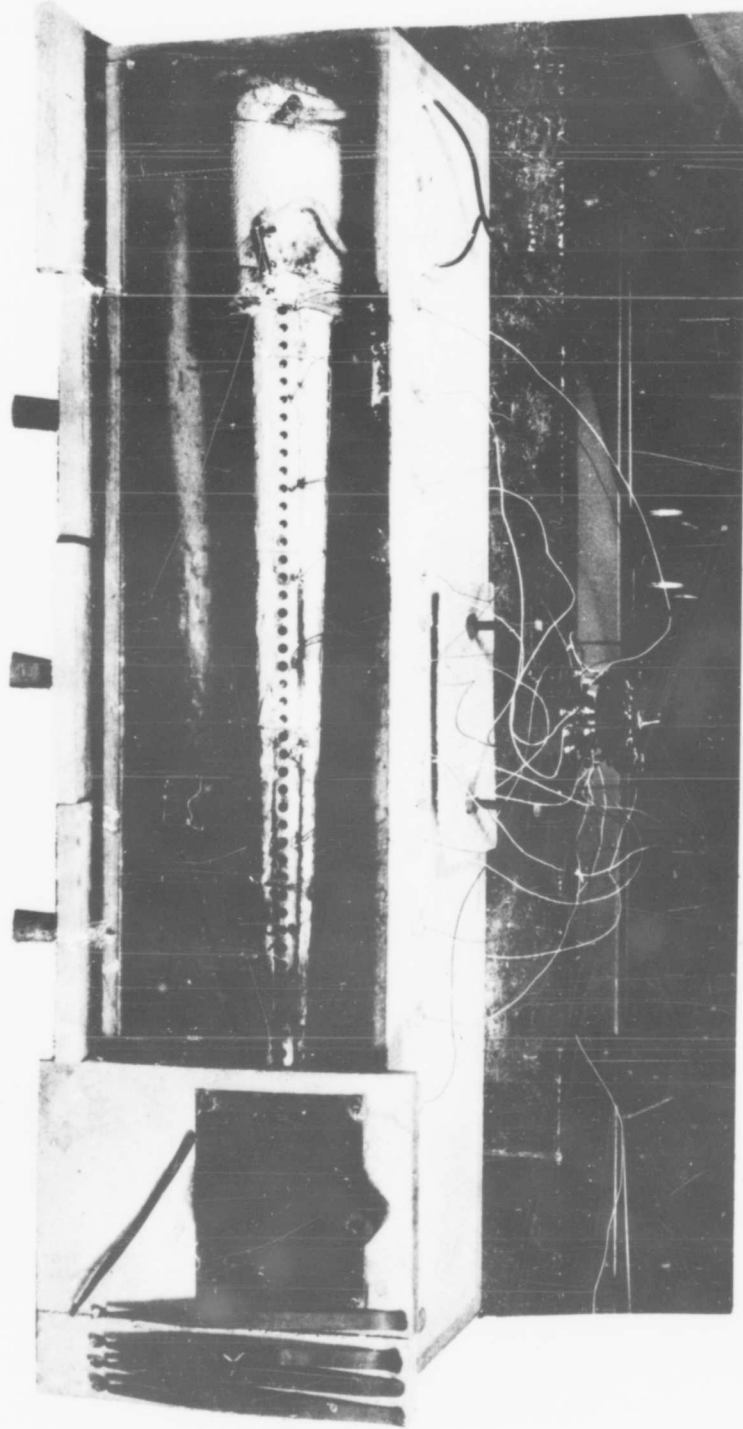


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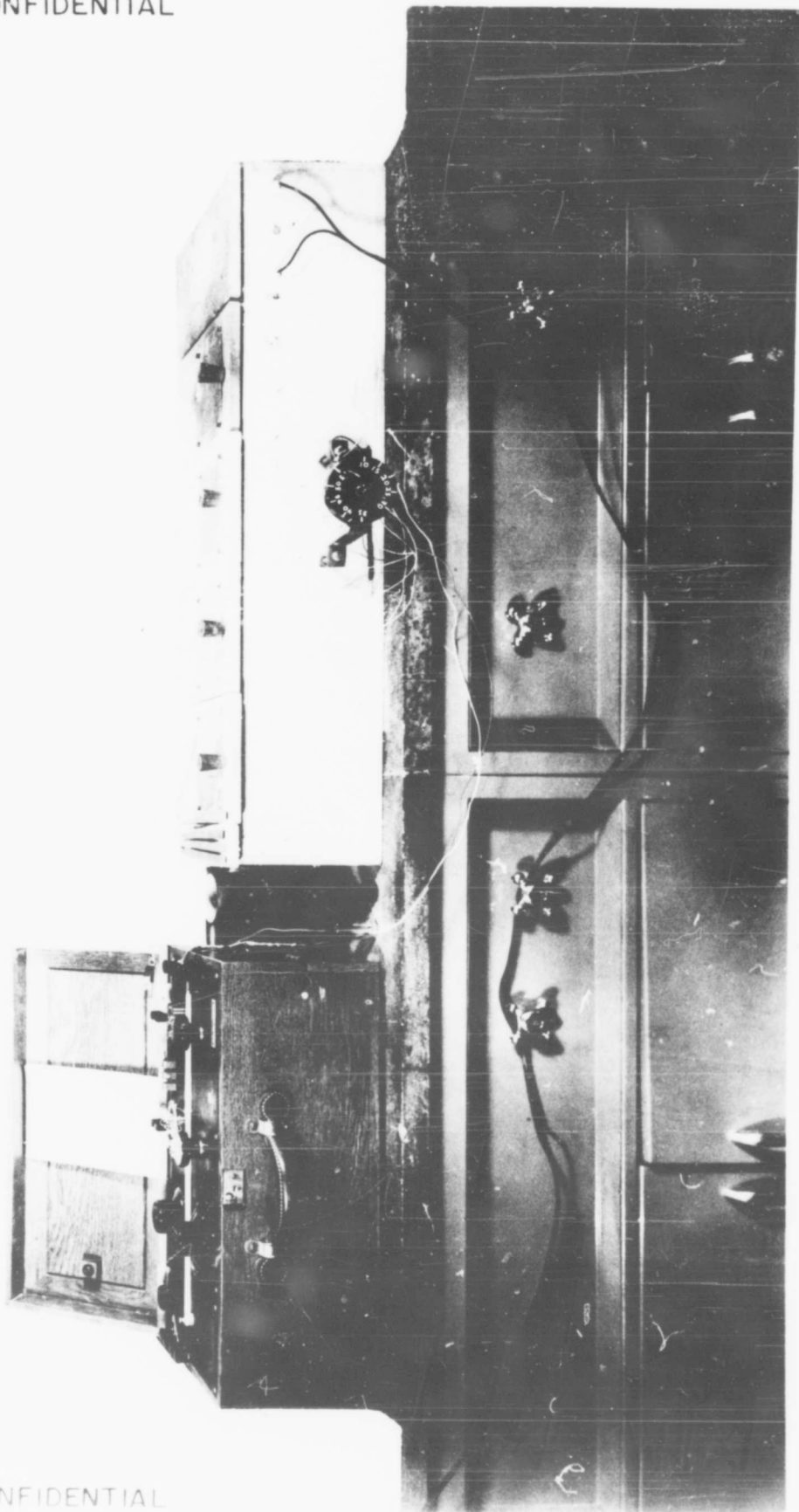
FIGURE 2

TEMPERATURE IN DEGREES C

TEMPERATURE LEVEL



ALUMINUM IGNITION TEMPERATURE BAR



IGNITION TEMPERATURE APPARATUS

TABLE I

Dependence of Ignition Temperature on Sample Weight

Sample Weight, Pentolite	<u>50% point, °C</u>	
	<u>12 mg</u>	<u>25 mg</u>
	238	238
	238	238
		238
Sample Weight, TNT	<u>18 mg</u>	<u>28 mg</u>
	454	454
	454	454
		462
		463
		473

TABLE II

Ignition Temperature of Aluminized Composition A,  
X41 Sample Size - 14 mg.

	<u>50% point, °C</u>
1.	364.3
2.	376.8
3.	383.7
4.	382.0
5.	372.3
6.	383.2
7.	384.3
8.	<u>374.9</u>
Av.	380.3

TABLE III

Ignition Temperature of Explosives

<u>Sample</u>	<u>50% point, °C</u>
Baritol	457
TNT	456
HBX	440
Comp B	436
Ammonium Picrate	418
Picric Acid	418
Aluminized Comp a	380
Comp A-3	373
Tetryl	326
RDX	316
Hydrazine Nitrate	307
Lead Azide	292
Lead Styphnate	260
Pentolite	238
PEIH	232
Haleite	222
DINA	219
Mercury Fulminate	195
Potassium 1,1-dinitroethane	179

TABLE IV

Comparison of Ignition Temperatures

	<u>ERE</u> <sup>1</sup>	<u>NPF</u> <sup>2</sup>	<u>NOLM</u> <sup>3</sup>
Mercury Fulminate	175 °C	177-180 °C	195 °C
Halate	150	--	222
PICN	215	170-175	232
Lead Styphnate	280	--	260
Lead azide	315	Indef.	292
RDX	above 360	203-208	316
Tetryl	265	160-164	326
Picric acid	265	300-304	418
ammonium Picrate	above 360	288-291	418
Comp A	"	197-204	400
Comp B	"	188-194	436
HMX	"	200-202	440
TNT	"	238-290	456
Baritol	"	276-283	457

1. Explosives Research Laboratory, reference (e)
2. Naval Powder Factory, reference (d)
3. This Memorandum

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