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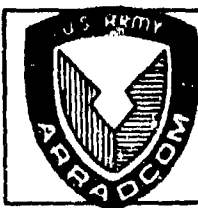
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FRICION SENSITIVITY OF PRIMARY EXPLOSIVES

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SEPTEMBER 1982



US ARMY ARMAMENT RESEARCH AND DEVELOPMENT COMMAND
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20. ABSTRACT (Continue on reverse side if necessary and identify by block number) A Bundesanstalt für Materialprüfung (BAM) small friction tester, manufactured in West Germany, has been used to rank primary explosives in their order of friction sensitivity. Primary explosives RD 1333 lead azide, dextrinated lead azide, polyvinyl-alcohol (PVA)-lead azide, colloidal lead azide, normal lead styphnate, basic lead styphnate, potassium dinitrobenzofuroxan, and tetrazene were tested to determine the 10% and 50% probability of friction initiation. The 50% probability of initiation level was used to rank primary (cont)		

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ABSTRACT (cont)

explosives for initiation of combustion by friction. The rankings for initiation by friction were compared with data for ranking primary explosives by impact sensitivity, autoignition temperature, and crystal density. Friction sensitivity values appeared to decrease with increase in the crystal density of primary explosives. Impact sensitivity and autoignition temperature values increased as the crystal density of primary explosives increased.

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INTRODUCTION

Friction is the resistance to relative motion between two bodies in contact. It is a force which opposes the motion of one body moving over the other or the force needed to overcome adhesion and the interlocking of the asperities of two bodies. Frictional force is proportional to the normal force pressing two surfaces together, independent of the areas in contact, and within limits independent of the velocity. Thus

$$f = \mu N$$

f = frictional force

μ = constant of proportionality known as the coefficient of friction

N = the normal force (mass $\times$ gravity)

In order to overcome the resistance to motion (static friction), energy must be imparted to the system to overcome the forces opposing compression. The products of this expenditure of energy are movement of the bodies and heat. The greater the normal force, the more heat will be released as a by-product of the energy expended overcoming friction. Assuming that explosive reactions are initiated by "hot spots," the greater the load (N), the more likely that initiation will take place (other forces remaining constant). Thus hot spot temperatures may be dependent on the ability of a material to resist motion due to a combination of rough surface, hardness, and load.

The BAM friction tester was developed in the German Federal Republic in the 1950's as a response to dissatisfaction with the existing mortar and pestle technique. This method was satisfactory for certifying explosives packed according to regulations, but it proved unsuitable for loose materials. Loose materials certified for transportation by the mortar and pestle technique were the cause of frequent accidents when exposed to friction (ref 1). These new regulation and test methods were required for the assessment of the friction hazards associated with the preparation, handling, packing, transportation, and use of explosive materials.

In order to develop a more qualitative test than the mortar and pestle method, the porcelain mortar was replaced by a small flat porcelain plate. The surfaces of this plate were roughened and the pestle was replaced by a small cylindrical porcelain peg. The porcelain plate was attached to a platform which moved with an oscillatory motion powered by an electric motor. The variables of motion, surface, and volume of powder were then eliminated. This technique left the weight of the normal force pressing the surfaces together as the only variable. Thus a qualitative test for friction sensitivity was created to initiate the numerical value of force. This test is used in many NATO countries, and its users have requested that it be adopted as a NATO standard.

The apparent advantages of the BAM friction tester are:

1. Variables, other than normal force, have been essentially eliminated. Test conditions have been standardized by the use of fresh surfaces of uniformly-manufactured porcelain plates and pegs.

2. The numerical results are measurements of rubbing alone rather than a composite of friction and impact values that are characteristic of the results of the friction pendulum device.

3. Relative numerical results are produced by the BAM, which enables the sensitivities of energetic materials to be compared.

The characteristics of the BAM friction test which may be considered to be disadvantageous are:

1. The expendable porcelain parts are expensive.
2. Small test sample size may not be considered to be characteristic of an entire batch. This can be overcome by testing many small samples.
3. A heavy duty friction tester is needed if materials other than initiating explosives are to be tested.

The objectives of this project were:

1. To evaluate the BAM tester as an instrument method to obtain friction sensitivity.
2. To develop a safe operational procedure for testing initiating explosives (see app).
3. To rank the friction sensitivity of initiating explosives.
4. To attempt to correlate the friction sensitivity with other physical properties of the explosives.

EXPERIMENTAL PROCEDURE

The BAM friction sensitivity tester is manufactured by the Julius Peters Company of West Berlin, Federal Republic of Germany. The explosive material is placed on a roughened porcelain plate and a porcelain peg is placed on top of the explosive. Calibrated forces are applied to the peg by placing weights in slotted positions on an arm of the instrument. An electric motor moves the porcelain plate with the explosive under the fixed peg. The apparatus thus measures the friction sensitivity of the material in terms of the force in grams applied to the test material that lies between the porcelain peg and plate.

The normal forces on the small instrument are 10 g to 1000 g. By use of 2 or 3 calibrated weights in three slotted positions, the force loads were extended to 2075 g (the manufacturer claims calibration to only 1000 g). Some of the softer primary explosives and some mixes where the primary explosives were diluted with other materials tested very well in the 1000 g to 2000 g range. There did not appear to be any observable inaccuracies or problems involved in the use of multiple weights.

The 50% probability level of initiation was determined by an "up and down" Bruceton test (ref 2). Twenty-five to forty friction tests were conducted on energetic materials placed between unused surfaces of porcelain pegs and plates. The pegs are 10 mm in diameter, 10 mm in length, and rounded on top and bottom. The plates measure 25 mm x 25 mm x 5 mm. The plate is clamped to a sled which moves at a constant velocity of 4.7 cm to 5 cm per second over a 10 mm path (ref 3).

A 10% probability of initiation is defined as the force increment that is one level above the level at which no ignitions occurred in ten trials. It is, in all practicality, the level at which one ignition takes place in 10 attempts. The data reported at this level was later compared with the reported 1 in 6 initiations in reference 4. This method is not as precise as the Bruceton test method, which determines the 50% level (table 2).

The primary explosives which were tested are:

<u>Material</u>	<u>Lot numbers</u>
lead azide, RD 1333	OMC 2-2
lead azide, dextrinated	DU 52-127
lead styphnate, basic	4679
lead styphnate, normal	OMC 68-14
lead azide, colloidal	none
lead azide, PVA	none
potassium dinitrobenzofuroxan	none
tetrazene	407913
tetrazene	7902454

The mixes which were tested are: NOL 130 (basic lead styphnate, barium nitrate, lead azide, tetrazene, and antimony sulfide); PA 100 (normal lead styphnate, barium nitrate, tetrazene, lead dioxide, calcium silicide, and antimony sulfide); 60/40 lead styphnate, normal/potassium perchlorate. These materials were stored in a desiccator over indicating drierite and removed before each test.

Ball Drop Impact

The ball drop impact apparatus is based on a design used by duPont de Nemours Company in the United States and the Nobel Explosives Company in Scotland (ref 5). The instrument consists of a free falling steel ball, a movable platform on a vertical stand, and a hardened steel anvil block to hold the explosive powder.

The steel ball, a 1.27-cm diameter weighing 8.35 grams is dropped from heights varying by 2.54-cm increments onto the explosive powder spread in a 0.083-cm layer on a hardened and polished steel block. After each explosion, the residue deposited on the block is cleaned off and the ball is replaced. The ball is dropped by allowing it to roll off an inclined track, and is, therefore, rotating when it hits the sample. In the case of no-fire, the ball bounces away from the block, and it only impacts the sample once. This overcomes one of the inherent problems of multiple impacts produced by drop

hammers. The ball is made from chrome alloy steel, Grade 25 Gold Seal (by Atlas Ball Co., Philadelphia, PA); and it has a Rockwell C hardness between 64 and 66.

Activation Energy and Autoignition Temperature

The activation energies and autoignition temperatures of primary explosives (table 1) were obtained by techniques that are described in references 6 and 7. The techniques involve the use of peak temperatures of exotherms produced by heating the primary explosives in a differential thermal analyzer at variable rates of 1.3K to 20K/minute. A semi-log graph of heating rate versus the reciprocal of the absolute peak temperature produces a straight line from which the activation energy and intercept are obtained. Setting the left side of the Kissinger equation (ref 6),

$$\frac{E\phi}{RT_m^2} = A e^{-\frac{\Delta E}{RT_m}}$$

equal to a constant value of 0.05, T_m is calculated. The value, 0.05, was obtained by substituting known values for autoignition temperatures into the Kissinger equation.

- A - intercept or frequency factor (sec^{-1})
- ϕ - heating rate (K/min)
- ΔE - activation energy (calories)
- R - gas constant
- T_m - exothermic peak at autoignition temperature (K)

The autoignition temperature is located at the base of a straight line drawn tangent to the exotherm from the peak to the base line (the extrapolated onset). The autoignition temperature is obtained by subtracting the difference between the extrapolated onset and the peak at the lowest available heating rate (1.3 K/min); then subtracting this value from the peak value T_m .

Experimental procedure for obtaining T_m is to place explosive samples of less than the critical weight in the center of thermocups. Thermocups are steel cups to which thermocouples are welded. The critical weight is the weight at which explosives will begin to burn rapidly; the explosive decomposes below the critical weight. A two-pen recorder is used to record the data; one pen records the temperature of the reference material in an adjacent thermocup, while the other pen records the ΔT (difference in temperature K) of the two cups.

RESULTS

Table 1 is a description of different types of lead azide (ref 8). Table 2 includes BAM friction sensitivity, differential thermal analysis (DTA), and an 8.35-g steel ball drop impact test. The crystal density of each tested material is listed as a substitute for the more desirable hardness characteristic of each material. Hardness data are mostly unavailable. The explosives

are listed in order of increasing force load on the BAM small friction sensitivity to obtain the 50% probability of initiation. The 10% probability of initiation is listed in the third column of this table.

The DTA results in table 2 are the activation energies that are determined from the slow decomposition exotherms of each material, using the variable rate technique (ref 6) and the autoignition temperature (ref 7). Except where it is noted, this data was obtained at heating rates of 1.3 K, 2.6 K, 5.2 K, 10 K, and 20 K per minute.

The 8.35-g steel ball drop impact results are in table 2 (ref 9). The results were obtained from a 50% probability of initiation Bruceton type test. The densities of the primary explosives were obtained from references 4, 10, and 11.

The data in table 3 are from six individual friction tests on the same lot of dextrinated lead azide which was tested over a 6 month period. The lead azide was taken from the same container desiccated over the same desiccant for 6 months. The data listed are the 50% level of probability of initiation determined from tests on the BAM friction apparatus by means of a Bruceton type "up and down" technique. Table 4 is a comparison of the BAM 10% probability of friction initiation (ref 4).

Figure 1 shows the working surfaces of the BAM friction tester. Figures 2 and 3 are semi-log graphs of the 50% probability of friction initiation and ball drop impact initiation versus the crystal density of the primary explosives.

DISCUSSION OF RESULTS

Relationship of Physical Properties to Friction Sensitivity

Primary explosives readily initiate when subjected to sufficient impact and friction. A unique characteristic of primary explosives as a group is that initiation of reaction occurs before melting. Materials which melt before initiation are found to be difficult to initiate by friction due to the large heat sink represented by the heat of fusion.

The initiation of primary explosives by friction is reported to be dependent on such factors as hardness, thermal conductivity, shape and size of rubbing surfaces (ref 12, p 58). Hardness, as a first approximation, would appear to be the most important of these factors. Lacking data on the hardness for all of these materials, the crystal densities of primary explosives were graphed against results obtained from friction sensitivity testing. A possible exponential relationship was found between the friction load force which initiates reaction of primaries and crystal density (fig. 2).

The values for the BAM friction sensitivity test for primary explosives show an increased friction initiation sensitivity with increasing crystal density values (table 2 and fig. 2). The values for the ball drop impact

height to initiate reaction show a descending sensitivity with increase in crystal density (table 2 and fig. 3). The autoignition temperature values with the single exception of the value for basic lead styphnate also show a descending sensitivity with increase in crystal density (table 2).

The inverse relationship, which exists for the data from the friction and ball drop impact sensitivity tests, may indicate that different mechanisms for initiation are operating. One aspect to be considered is that impact sensitivity may depend on hot spots caused by the adiabatic compression of minute gas bubbles (ref 13). For only materials which decompose below their melting point, the ones which have the lowest autoignition temperature will also be the most impact sensitive. The autoignition temperatures in table 2 show that the softer, least dense materials do have the lowest initiation temperatures and are the most sensitive to impact.

Initiation of primary explosives by friction seems to involve hardness and density. The autoignition temperatures and activation energies of primary explosives in table 2 indicate that friction sensitivity does not relate to either of these latter characteristics. Factors, such as lattice structure, deformability, and plastic flow, may also be of importance to friction sensitivity; however, hardness seems to be the primary determinant of friction sensitivity of initiating explosives. Soft particles can be plastically deformed in a way that local concentrations of energy are not possible. Therefore, hard particles are more friction sensitive, provided the melting point of these particles is above the critical value.

Activation Energy

The values of 36-45 kcal/mole activation energy found for the various lead azide samples compare well with the value of 37-38 kcal/mole (ref 12) and Garner and Gomm's value of 42 kcal/mole. Lead azide activation energies seem to be more variable by lot than by type of lead azide. Such variability would indicate that impurities in the lead azide have an important affect on the activation energy of the lead azide.

The value of 40 kcal/mole for normal lead styphnate is the same value reported in the literature (ref 12). Activation energy for basic lead styphnate could not be found in the literature. The identical activation energy 40 kcal/mole found for basic lead styphnate indicates that a similar rate controlling step occurs to initiate decomposition as in normal lead styphnate.

There is a large variation in reported activation energies for tetrazene reported in the literature (ref 10). The variations can be due to differences in the complicated structure of tetrazene resulting from different manufacturing techniques, but it may also be caused by secondary reactions. Secondary reactions occurring in the gaseous state between decomposition products effect the process by which the kinetics of reaction are measured. The deviation of the E/R slope from a straight line for the

$$\frac{h}{T^2} \text{ versus } \frac{1}{T} \text{ (T = absolute temperature K)}$$

curve for tetrazene at heating rates of 10 K/minute and 20 K/minute are probably caused by the effect secondary reactions have on the decomposition peak temperatures; this nullifies the possibility for measurement of a constant activation energy by the variable rate technique. Forty-eight kcal/mole isothermally and 46 kcal/mole activation energies reported (ref 14) are not far from the 50 kcal/mole found for tetrazene lot 7902454, but 55 kcal/mole for tetrazene lot 407913 can be a variation. The activation energies reported here for tetrazene are only applicable between heating rates of 1 K to 5 K/minute.

A semi-log graph of friction sensitivity versus crystal density of primary explosives does not produce a straight line (fig. 1). It is anticipated that a graph of hardness versus friction sensitivity would approximate a better straight line when that data becomes available.

A graph of crystal density of the primary explosives versus the impact height from the ball drop impact test shows almost a straight line of the log of impact height versus crystal density of primary explosives (fig. 2). Apparently, there is a relationship between crystal density and impact height to this experimenter, however, the limited amount of data does not justify further speculation.

CONCLUSIONS

1. The small BAM friction sensitivity tester is suitable for the determination of a statistically significant friction sensitivity value for primary explosives.
2. By means of the small BAM friction sensitivity tester, new lots of primary explosives may be screened for acceptance.
3. A linear relationship exists between the results of the 50% probability of initiation height from ball drop impact data and the crystal density of the primary explosive.

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Table 1. Description of lead azide types

<u>Test</u>	<u>Dextrinated</u>	<u>RD 1333</u>	<u>PVA</u>	<u>Service</u>	<u>British Colloidal</u>
Lead azide, %	92.8	98.7	96.0	98.1	99.9
Total Lead, %	69.3	71.06	71.6	71.5	71.67
Particle size average, microns	24.5	34.5	19.0	55.0	3.4

Table 2. Sensitivity test results for primary explosives

Explosive	BAM friction sensitivity		DTA		Ball drop impact sensi- tivity values at 50% height (cm)	Crystal density (g/c)
	50% (g)	10% (g)	Activation energy (Kcal/mole)	Autoignition temperature (K)		
Colloidal lead azide	16 ± 7	-	40	540		4.8
PVA lead azide	50 ± 22	<10				<4.8
Special purpose lead azide			36	553	56.9 ± 5.3	4.8
RD 1333 51-49 lead azide			42	565		
RD 1333 51-13 lead azide			37	557		
RD 1333 OMC2-2 lead azide	81 ± 29	20	41	560	51.1 ± 8.9	
Dextrinated lead azide 51-127	157 ± 50	100	44	557	54.6 ± 8.4	4.4
Dextrinated lead azide 51-126	167 ± 90	100	44	561		4.4
Basic lead styphnate 4679	250 ± 100	40	40	508	37.3 ± 11.2	3.9
Normal lead styph- nate	285 ± 200	100	40	522	22.1 ± 4.1	3.1
KDRNF	400 ± 100	50	38	453		
Tetrazene 407913	850 ± 350	300	55	395		1.7
Tetrazene 7902454	900 ± 180	700	50	393		1.7
60/40 normal lead styphnate potassium perchlorate	1400 ± 600	600				
NOL 130	1350 ± 100	800	45	509	18.3	
PA 100	325 ± 100	200				

Table 3. Friction sensitivity results for dextrinated lead azide duPont 52-127

<u>Date</u>	<u>50% Initiation level (g)</u>
15 October 80	200.0 $\pm$ 022.6
27 October 80	167.0 $\pm$ 088.0
16 January 81	159.0 $\pm$ 045.0
09 March 81	201.0 $\pm$ 147.0
16 March 81	164.3 $\pm$ 048.0
24 April 81	191.5 $\pm$ 052.7

Table 4. A comparison of friction data at 10% probability of initiation

	10% Probability of initiation	
	Previous Work in 6 trials ^a (g)	Present work in 10 trials (g)
Lead azide	10	20 ^b
Lead styphnate	150	100
Tetrazene	800	700 ^c

^aFriction data from reference 4.

^bRD 1333 OMC 2-2

^cTetrazene, lot 7902454

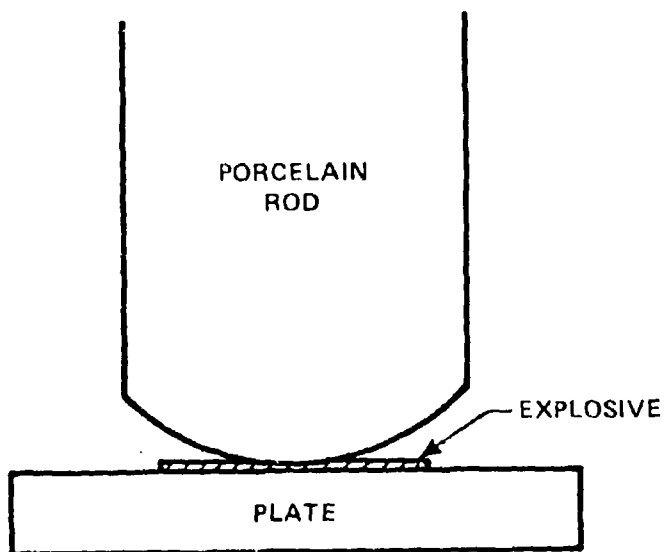


Figure 1. Working surfaces of BAM machine

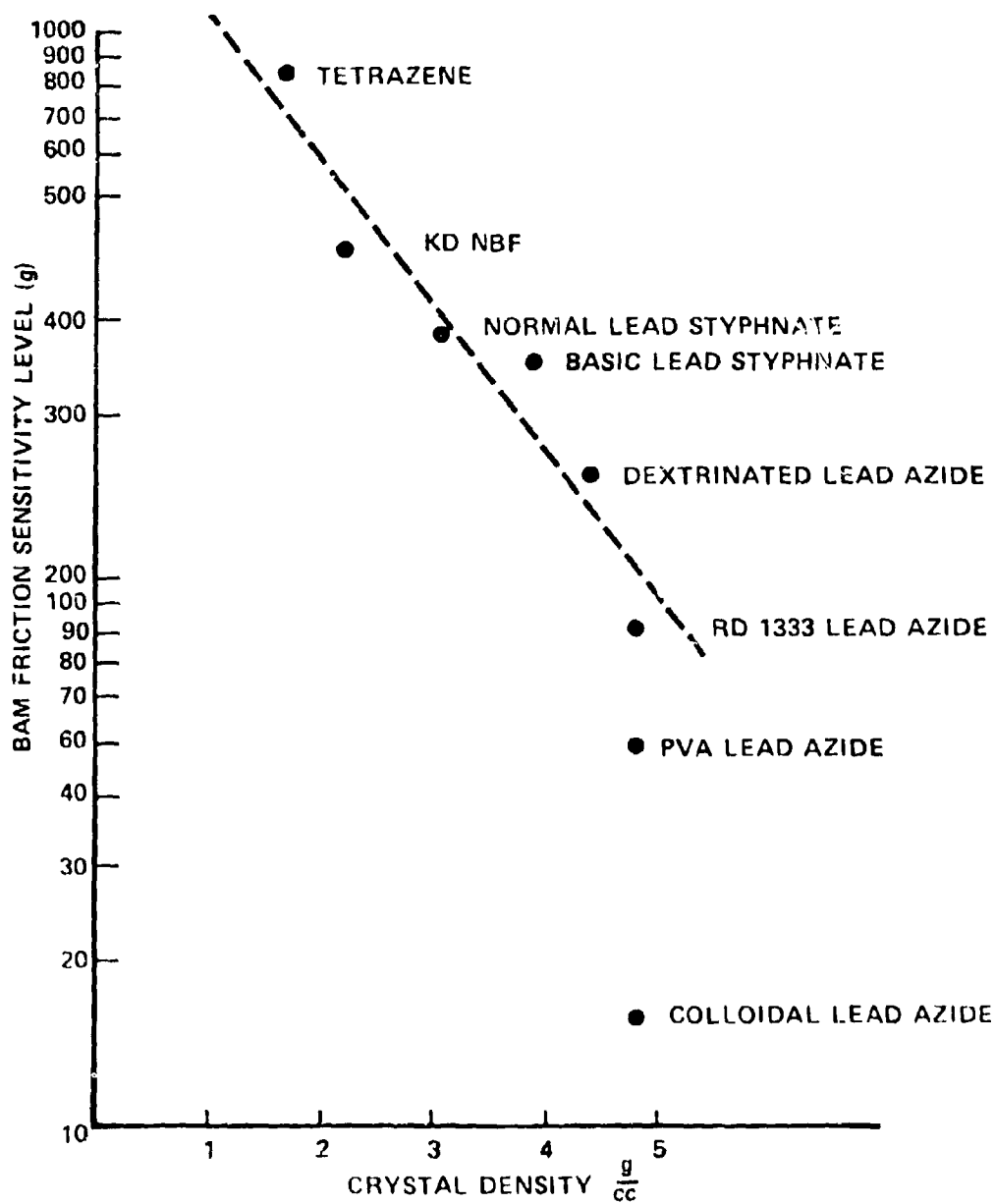


Figure 2. Friction sensitivity versus crystal density

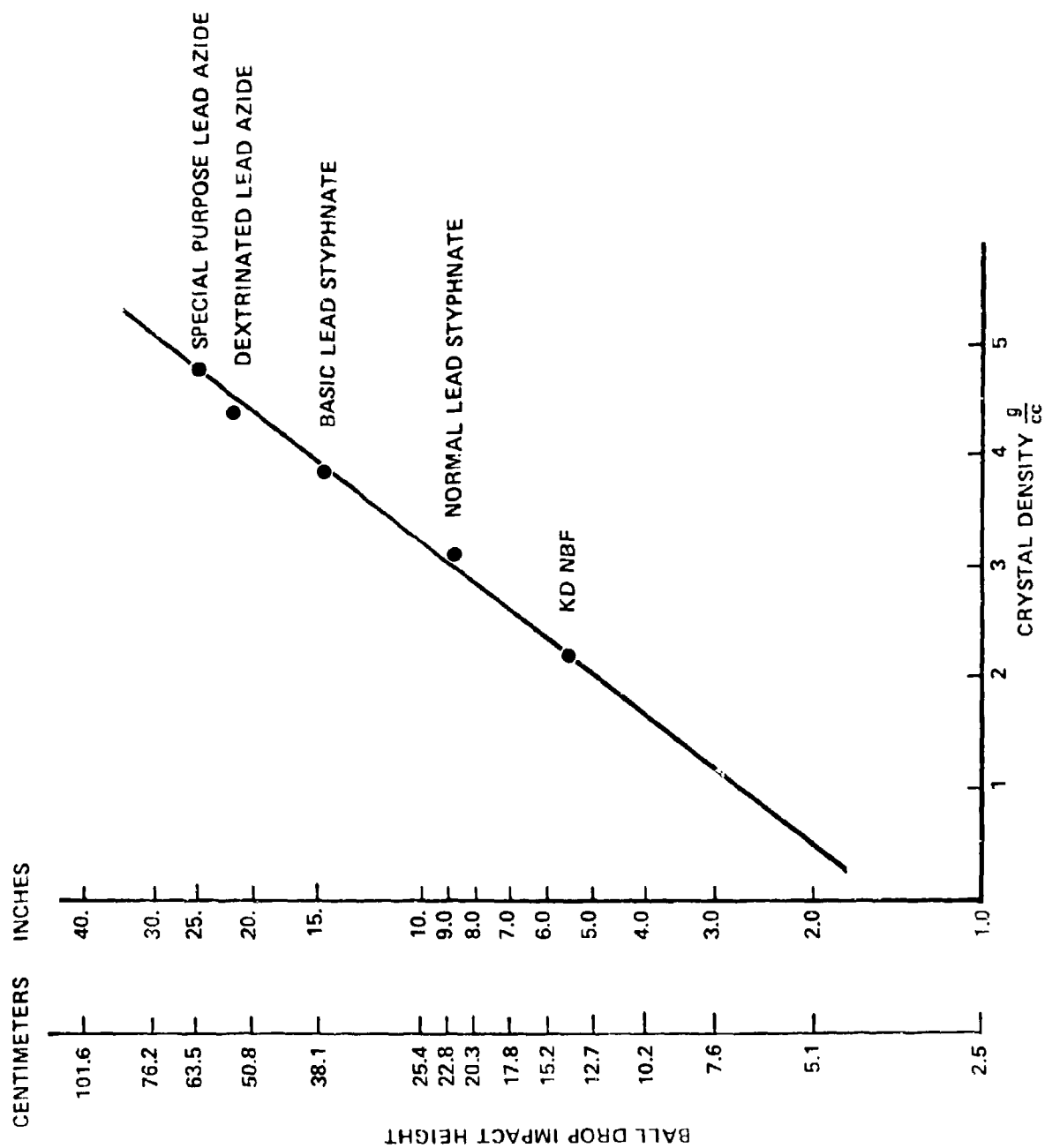


Figure 3. Ball drop impact height versus crystal density

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