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TECHNICAL REPORT ARLCD-TR-79045

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EXPLOSIBILITY OF EXPLOSIVE AND PROPELLANT DUSTS

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LARGE CALIBER
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DOVER, NEW JERSEY

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REPORT DOCUMENTATION PAGE		READ INSTRUCTIONS BEFORE COMPLETING FORM
1. REPORT NUMBER Technical Report ARLCD-TR-79045	2. GOVT ACCESSION NO.	3. RECIPIENT'S CATALOG NUMBER
4. TITLE *(and Subtitle)* EXPLOSIBILITY OF EXPLOSIVE AND PROPELLANT DUSTS		5. TYPE OF REPORT & PERIOD COVERED Final
		6. PERFORMING ORG. REPORT NUMBER
7. AUTHOR(s) W. T. Moore, P. Lu, S. Roorda B. Fishburn, and N. Slagg R. M. Rindner, Project Leader W. Seals, Project Engineer		8. CONTRACT OR GRANT NUMBER(s)
9. PERFORMING ORGANIZATION NAME AND ADDRESS ARRADCOM, LCWSL Energetic Materials Division (DRDAR-LCE) Dover, NJ 07801		10. PROGRAM ELEMENT, PROJECT, TASK AREA & WORK UNIT NUMBERS
11. CONTROLLING OFFICE NAME AND ADDRESS ARRADCOM, TSD STINFO (DRDAR-TSS) Dover, NJ 07801		12. REPORT DATE July 1980
		13. NUMBER OF PAGES 39
14. MONITORING AGENCY NAME & ADDRESS*(if different from Controlling Office)* ARRADCOM, LCWSL Energetic Systems Process Division (DRDAR-LCM-SP) Dover, NJ 07801		15. SECURITY CLASS. *(of this report)* Unclassified
		15a. DECLASSIFICATION/DOWNGRADING SCHEDULE

16. DISTRIBUTION STATEMENT *(of this Report)*

Approved for public release; distribution unlimited.

17. DISTRIBUTION STATEMENT *(of the abstract entered in Block 20, if different from Report)*

18. SUPPLEMENTARY NOTES

19. KEY WORDS *(Continue on reverse side if necessary and identify by block number)*

Manufacturing safety	Propellant dust
Ammunition plants	Dust explosibility
Hazards analysis	Munitions manufacturing technology
Propellants	

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

A specially designed flow system was constructed to obtain more detailed data on the effect of particle size and concentration on the spark ignition of propellant and explosive dusts. With this apparatus the concentration can be controlled over a 1.5 m (5 ft), and streak photography can be employed to determine the velocity of flames and/or explosion waves. Thus information on both ignition and propagation can be obtained. The ignition characteristics of M1 propellant, RDX, HMX, and ball powder were determined. The concentration

20. ABSTRACT (Continued)

was varied from 0.15 to 1.5 g/liter, and the particle size was varied from 75 to 225 micrometers. Ignition with subsequent flame propagation required a concentration of at least 0.15 to 0.3 g/liter for all dusts tested. Values from previous studies conducted in a Hartmann apparatus ranged from 0.1222 g/liter to 0.5 g/liter for some of the same materials with similar particle sizes. Thus the lower limits of ignition are in good agreement; however, due to the relatively small size of the Hartmann apparatus, direct comparisons of ignition at upper limits are difficult.

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SUMMARY

The minimum explosible concentrations of M1 propellant, RDX, HMX, and ball powder were determined with a specially constructed, steady flow, spark ignition apparatus. Results were in general agreement with data that have been obtained using Hartmann apparatus, which produces less homogeneous clouds.

Dust-concentration sampling was carried out at three Army ammunition plants to assess the explosion hazard present during some loading operations.

TABLE OF CONTENTS

INTRODUCTION

Background

Although dust explosions have been a recognized hazard for about 200 years, it has been in the last 25 years that scientific studies have been undertaken. These were chiefly motivated by the need to find the cause of coal mine explosions and the means to prevent them. Dusts can be classified into two types: (1) capable of ignition with a resultant flame propagating from the ignition, and (2) capable of ignition but without any flame propagation. It is only those of type 1 that constitute a hazard.

This study was undertaken to evaluate the possibility of fire and explosion in Army manufacturing and loading installations due to dust dispersions of energetic materials (explosives or propellants) in air. The study consisted of two efforts. One was an experimental program to measure the minimum dust concentrations of various materials required to present a type 1 hazard. The other was a sampling program in the manufacturing plants to determine the dust concentrations produced during production operations.

Spark Ignition

The most likely ignition source to be encountered in practice is a spark discharge. Since sparks are known to be effective ignition sources and can be well controlled and reproduced in the laboratory, spark ignition was chosen for this study. The goal was to measure a minimum concentration of dust at which ignition and flame propagation will occur for a particular energetic material. The manner in which ignition energy varies with dust concentration provides a basis for the minimum dust concentration concept. Figure 1 shows schematically a typical curve of ignition energy versus explosible dust concentration. There are two critical values associated with this curve. One is the minimum ignition energy (E_{min}) required to ignite the dust at its most favorable concentration, and the other is the minimum dust concentration (C_{min}) where the required ignition energy becomes extremely large.

Previous studies that attempted to measure the minimum ignition energy have resulted in ambiguous data. For example, studies of spark ignition of a gaseous methane-air mixture resulted in a minimum spark energy of 0.28×10^{-3} joule for an 8.5% methane-air mixture (refs 1 to 7). Then Priede demonstrated that if a series resistance of 5×10^4 ohms is introduced in the capacitive discharge circuit, the minimum energy can be reduced to about half of

1

that obtained when the resistance was removed (ref 8). He postulated that the resistance lengthened the spark time, that leads to a reduction in energy losses from heat conduction (ref 9).

A similar situation exists in determining the minimum energy for dusts. Line et al. employing a high-speed camera, showed that sparks can disturb the dust cloud adjacent to the spark (ref 10). His photographs demonstrate the existence of a small dust-free region adjacent to the spark. Eckhoff found a similar result (refs 11 and 12). The studies by Boyle and Llewellyn (ref 13), Priede (ref 8), and Moore et al. (ref 14) have shown that a series resistance in the spark discharge circuit can dramatically affect the minimum energy for ignition. Studies using a wide variety of spark circuits have confirmed that long duration sparks are more efficient in producing ignition than short-duration sparks (ref 15). Eckhoff postulates how the theoretical aspect of the spark disturbs the dust cloud and causes this effect (refs 9 and 12).

In contrast to this situation, the value of C_{min} is not apparatus dependent. Since the slope of the curve becomes very large, changes in the ignition energy do not produce minimum explosible concentrations that differ noticeably from C_{min}. Measurements of C_{min} can be made by choosing a sufficiently large ignition energy to insure operation on the nearly vertical section of the curve and by simply varying the dust concentration until no ignition is obtained. This approach was adopted in this study (ref 16).

A problem with closed bomb measurements of C_{min} is obtaining a uniform dust concentration throughout the test volume. Usually a weighed amount of dust is dispersed in the closed bomb by a blast of air. Inhomogeneity of the dust within the bomb is not characterized, although, as pointed out by Eckhoff (refs 11 and 12), concentration gradients are likely to occur. The spark ignition apparatus designed for this study attempted to eliminate this problem by providing a steady flow of dusty air past the electrodes. Dust concentrations were determined through measurements of dust collected in a given period of time.

Types of Explosions

A dust in which reaction propagates by the combustion of particles with the suspending gas is considered explosible. Because explosives and monopropellants contain their own oxidizers, they do not require external oxidizers to undergo a reaction. Therefore, dust from explosives and monopropellants may explode in inert atmospheres. However, in practice, airborne dusts use atmospheric oxygen during an explosion. All tests conducted in this study examine the explosible property of these airborne dusts.

Within the definition of the general term "explosion" there are two processes to be considered: deflagration and detonation. A deflagration is a flame propagation through the dust at velocities less than the local speed of sound. Pressure waves associated with this process are typically mild and overpressures developed in an unconfined environment are less than 101.3 kPa (1 atmosphere). A detonation is a reaction propagation through the dust at velocities much greater than the local speed of sound. Strong shock waves are associated with such an event. Some dusts will support both types of processes depending on the details of the ignition. Strauss has demonstrated the detonability of aluminum dust in oxygen, and Lu et at. have demonstrated detonability characteristics in aluminum dust-air mixtures (refs 17 and 18). Another study has shown that coal dust-air mixtures can be made detonable if they are sensitized by addition of methane (ref 19). A detonation of a dust is much more destructive and far reaching than a deflagration of the same dust.

Deflagrations in some materials can undergo transition to detonation. Generally, this transition requires some confinement or obstacle within the volume of explosive material. Deflagration of part of the explosive mixture sets up a flow of the unreacted mixture along the confinement or obstacle. Turbulence is generated in the flow ahead of the flame. When the flame interacts with this turbulence, a localized autoexplosion may occur which is the beginning of the detonation which consumes the remainder of the explosive mixture. What starts as a slow deflagration becomes a detonation, producing a strong blast wave. The small region where the autoexplosion occurs is called an "exothermic center." Currently, it is not possible to accurately prescribe the conditions leading to the formation of these centers, although some materials are much more likely to form them than others. A dust's potential to undergo transition should be evaluated along with its potential to become ignited and propagate a flame.

EXPERIMENTAL PROCEDURES

Dust Ignition Experiment Apparatus

An apparatus was constructed that allowed a spark discharge to be passed through a flowing dust-air mixture of uniform dust concentration (figs. 2 and 3). Dust concentration could be controlled. The procedure was to fix the spark energy at a sufficiently high value and to vary the dust concentration to determine

3

the concentration below which no ignition will occur. Minimum spark energies for HMX/TNT and RDX/TNT dusts have been measured by Willim and Nicholson and have values of 0.016 and 0.022 joules, respectively (ref 15). In addition, the study by Gehring and Friesenhahn (ref 20) on the explosibility of M1 propellant dust indicates that the steep region of the ignition energy versus explosible dust concentration curve begins at about 0.3 joules (ref 21). A spark energy ten times this value (3 joules) was chosen in this study to insure operation on the steep part of the curve.

The apparatus was set up in a test chamber with control panels (fig. 4), oscilloscope, and streak camera located in an adjoining room. Primary airflow through the dust fluidizer was registered on the control panels,as well as secondary airflow used to dilute the primary dust stream to desired concentrations. The resulting dust-air mixture entered the vertical tube test volume from the top. A Tektronics type 555 dual trace oscilloscope was used to record output from photodiodes that monitored flame propagation. A Beckman and Whitley model 370A streak camera also monitored flame motion. Valve openings and spark discharge were synchronized with a Jenor Model 2410 automatic rogrammer.

In the electrical circuit for the spark ignitor the voltage was variable from 1 to 10 kilovolts and the capacitance was variable from 1 to 5 μF in 1-μF increments.

Dust Ignition Experiment Operation

Samples were taken from production grade RDX, HMX, M1 propellant and ball powder. These materials were ball milled and particles larger than 250 μm were removed by dry sieving the M1 and ball powder and wet sieving the RDX and HMX with freon (RDX and HMX are not soluble in freon). Sieving was accomplished using either a sonic sifter (Fisher-Scientific) or large standard screens and brush. Residual moisture or solvent was removed from the RDX and HMX samples by placing them in a drying oven at 80°C for one hour. These materials are not affected by 80°C temperature. Table 1 gives the composition of M1 and ball powder used in the tests.

A dust sample was placed in the dust reservoir, and the programmer was energized to permit flow of air through the dust bed and into the 6 foot long, 2-inch-inner diameter plastic vertical tube (figs. 2 and 3). Another signal started the firing sequence, causing the programmer to automatically close control valves and within 50 msec, discharge the capacitors through electrodes mounted in the column. The amount of energy in the discharge was calculated in the usual way using the $E = \frac{1}{2} CV^2$ formula, although the

4

resulting value does not represent the true energy absorbed by the dust.

Dust concentration during a run was measured by collecting the dust exiting the vertical tube for a fixed time interval and relating this to the amount of air flow during this interval. Air flow was measured by rotameters in both the primary and secondary air lines. Dust concentration in this airflow was determined through measurements as staying within 10% of the average value throughout a test run. Variations in dust concentration between runs with the same initial conditions were within 20%.

Any propagating reaction in the dust column was detected both by the photodiodes and the streak camera. The time required for a flame to pass between the two photodiodes was recorded so the flame velocity could be calculated. A separate calculation of the flame velocity was made from the simultaneous streak photograph.

Manufacturing Site Sampling Apparatus

Air pumps were used to draw dusty air through millipore polyvinyl chloride (PVC) or cellulose ester filters that had been tared and placed in millipore aerosol analysis monitor holders. The flow rate through the filters was calibrated to a rotameter in the line to the pump. Calibration was accomplished by connecting a graduated 1000 mL tube to the intake of the filters. The pump was operated for a length of time sufficient to stabilize the flow rate. A dish containing soap solution was momentarily brought into contact with the bottom of the inverted intake, and the time for the bubble-air interface to sweep out a particular volume of the graduated tube was measured (fig. 5). This value was converted to liters per minute flow rate for that particular rotameter setting. In this way, a chart relating rotameter setting to flow rate was constructed. All pumps were individually standardized.

Manufacturing Site Sampling Operation

At the manufacturing site, likely locations for sampling were noted during a walk-through inspection. The schedules of the workers and the magnitude and frequency of the operation were noted. A schedule was then devised to allow the sampling pumps to be set up in such a way as to cause minimum interference with production and to allow a sampling time of 2 hours or more. Most of the filters used were the polyvinyl chloride type since they are not as sensitive to humidity variation as are cellulose ester filters. After the pumps were emplaced and attendant filters connected, the pumps

were started and adjusted to a flow rate of between 1.5 and 2.5 liters per minute. The sample volume was determined by multiplying the flow rate and the total pump operation time.

The used filters were preweighed and weighed again after dust samples were collected. The difference in weight represents the weight of the dust. To obtain the concentration the quantity of dust collected is divided by the sample volume.

RESULTS OF SPARK IGNITION STUDIES

Table 2 summarizes the results obtained here with HMX and shows that for the 3 joule discharge, the minimum explosible dust concentration is 0.3 g/liter for particles in the 125 to 177 μm range and between 0.15 to 0.3 g/liter for particles in the 88 to 125-μm range. No ignition was observed for concentrations below 0.15 g/liter or with larger particles. These results are in general agreement with the results obtained by Hazard Research Inc. with a Hartman apparatus (ref 21). Exact agreement is not expected since the Hartman apparatus produces a relatively inhomogeneous dust cloud. They found that for class 1 HMX particles of median diameter 100 μm, ranging from 65 to 320 μm, the minimum explosible concentration was 0.5 g/liter. Considering their less homogeneous dust concentration and their larger range of particle sizes, this result seems consistent with that obtained here.

Table 3 shows the results obtained with RDX. The minimum explosible dust concentration is 0.3 grams per liter for particle sizes in the range of 63 to 88 μm. For particle sizes in the range of 177 to 250 μm, ignition was not observed. The RDX powder had a tendency to form clumps, which caused the particle size to change during an experimental run; therefore, the particle size associated with these results may not be the same as the initial particle size. However, the results are consistent with the HMX results, where no agglomeration occurs.

Dusts that were roughly similar to those studied here were tested by Willim and Nicholson (ref 15). They tested 60/40 RDX/TNT and 75/25 HMX/TNT mixtures by sparking dust clouds formed after a blast of air was passed into a known amount of powder. No provision for observing flame propagation was made. For their RDX/TNT dust, with particle sizes ranging from 124 to 154 μm, they found a

6

minimum ignitable dust concentration of approximately 0.09 to 0.12 grams per liter. In contrast, our results for RDX dust, with particle sizes ranging from 125 to 177 μm, indicate a minimum explosible dust concentration of 0.75 to 1.5 grams per liter.

Since Willim and Nicholson did not determine whether a flame would propagate through their low concentration after its ignition, their values may not represent actual explosible concentrations. Normally, the concentration required for ignition alone is lower than the concentration required for ignition followed by flame propagation. This fact could account for most of the difference in values. The greater nonuniformity of dust concentration in their experiment also contributes to this difference.

If the data obtained here are plotted on a curve of concentration versus particle size (figs. 6 and 7), a region in which ignition can be expected becomes apparent. The average particle sizes for the four ranges presented in tables 2 and 3 were used in figures 6 and 7. (There is a general view that the ignitability becomes easier as the particle size decreases.)

Table 4 gives results obtained with M1 propellant. Minimum concentration for ignition is less than 0.06 grams per liter, but no subsequent flame propagation occurs through the low density dust. This value agrees with the value found by Hazards Research, Inc., of 0.05 to 0.06 grams per liter using their Hartmann apparatus (ref 21). A minimum dust concentration of 0.3 grams per liter was sufficient for ignition followed by flame propagation in the particle size range of 44 to 150 μm. The minimum value lies between 0.15 grams per liter and 0.3 grams per liter. This value compares well with the minimum explosible dust concentration of between 0.122 and 0.244 grams per liter determined for the partiles ranging in size from 75 to 105 μm found by Gehring and Friesenhahn of Southwest Research Institute (ref 20).

Severe agglomeration was encountered in testing ball powder. An ignition was observed for a concentration of 3 grams per liter, but no subsequent flame propagation occurred (table 5).

Although the minimum explosible dust concentrations are determined, factors that may occur in the manufacturing plants can significantly change the minimum values. The most likely factor is the presence of solvent vapor in the dust-air mixture. Solvent-air mixtures of 0.05 grams per liter of solvent are detonable, and the ignition energy for these gaseous mixtures is low. In addition, other modes of dust ignition are possible. An explosion from another source may generate a strong shock wave which propagates into the dust, causing ignition. This method of ignition may cause

7

ignition of concentrations or particle sizes that are not ignitable by a spark.

With regard to differentiating between deflagration and detonation during these tests, no indication of deflagration-to-detonation transition was observed, although all the ignitions were followed by flame propagation down the tube. The lucite vertical tube used as a test volume was not destroyed during any of the propagations, indicating only low pressures were developed.

Streak photographs provide the best evidence that no transition to detonation occurred. An example of the appearance of a streak photograph taken of deflagration-to-detonation transition is shown in figure 8. The mixture was stoichiometric propane-oxygen that was spark ignited. Transition to detonation is indicated where the sharp change of slope occurs. Once started, the detonation propagates at great speed both into the partially reacted mixture already traversed by the flame front (called retonation) and into the undisturbed mixture.

A representative streak photographs of flame propagation through three RDX powder dusts is shown in figure 9. All the streak records indicate flame speeds of approximately 300 m/sec, which is close to the velocity of sound in air. The flow field generated during an actual accident situation is more complicated than the flow induced in the vertical tube by the flame motion. Therefore, transition to detenonation in these dusts cannot be ruled out based on the results of this study alone.

RESULTS OF MANUFACTURING SITE SAMPLING STUDIES

Trips were made to Radford, Indiana, and Milan Army Ammunition Plants to obtain data on explosive dust concentrations which could be used, together with the laboratory ignition studies, to assess the hazards at these plants.

The operations investigated and the concentrations of dust observed at Radford Army Ammunition Plant are given in table 6. These concentrations are significantly lower than the concentrations of explosive and propellant dusts required for ignitione (about 0.15 to 0.3 grams per liter). Therefore, the values observed at Radford do not constitute an obvious hazard; however, some additional points follow. Since most operations are batch-wide and are remotely controlled, sampling must be controlled from a remote site, or the sampling must be accomplished over a time

8

period greater than an actual batch period of operation. The results obtained may not be indicative of the maximum dust concentration that occurs during a batch run.

Data obtained at Indiana Army Ammunition Plant from the sites indicated in figure 10 are presented in tables 7 and 8. The concentrations appear to be several orders of magnitude below that required for ignition; however, if the dust reservoir that accumulates near the bag-loading machinery was blown into the air, hazardous concentrations could be obtained. Based on conversations with maintenance personnel responsible for blowing down dust from weighing hoppers, large lot-to-lot variations in dust levels exist, with some lots appreciably more dusty than those sampled here. It might be inferred from the frequency of blowing down of dust at Indiana Army Ammunition Plant that dusts may reach four to five times the levels determined during our sampling visit.

The data obtained at Milan Army Ammunition Plant, presented in table 9, do not indicate a hazard. However, drilling and facing operations on M105 shells generate a large quantity of dust which is exhausted through a 50.8 mm (2 in.) diameter aluminum collector tube to a dry scrubber. Based on conversations with the operator, it is estimated that 34 kg of dust are obtained from three collectors per hour in the cyclone precipitators. Thus the collecting line and precipitators contain enough dust in layer form that, if dispersed by some event, a hazardous concentrations in air could result.

CONCLUSIONS

1. Studies of the ignition of energetic dust, using a flow system which permitted good control and characterization of dust concentration, gave results in essential agreement with previous studies using the Hartman apparatus.

2. The dust concentration required for ignition was a function of particle size.

3. Measurements made at Army ammunition plants of normal, air-suspended dust did not reveal concentrations sufficient to present an obvious explosive dust hazard.

4. Sufficient dust accumulates at certain sites in the plants that an external event such as a nearby explosion could stir up dust clouds with hazardous concentrations that can transition from a deflagration to detonation reaction.

9

RECOMMENDATIONS

1. A study should be conducted to determine the ignition parameters associated with dust layers that are fluidized by an external source.

2. A study should be conducted to determine the sympathetic detonation parameters associated with shockwaves impacting dust layers.

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Table 1. Composition of Ml propellant and ball powder (%)

Compound	Ml propellant	Ball powder
Nitrocellulose (13% N)	84.2	97.7
2-Nitrodiphenylamine	--	1.3
Dinitrotoluene	9.9	--
Dibutylphthalate	4.9	--
Diethylphthalate	--	1.0
Diphenylamine	1.0	--
Total	100.0	100.0
Moisture and volatiles*	1.25	1.0

*Not normally included in composition specification values, but considered an added ingredient.

13

Table 2. Ignition characteristics of HMX dusts[a]

Particle size (μm)	Dust concentration (g/liter)			
	1.5	0.75	0.3	0.15
177 – 250	no flame	no flame	no flame	no flame
125 – 177	flame propagated	flame propagated	flame propagated[b]	no flame
88 – 125	flame propagated	flame propagated	flame propagated	no flame

[a]Ignition energy = 3 joules.

[b]Inconsistent ignition.

14

Table 3. Ignition characteristics of RDX dusts[a]

Particle size (μm)	Dust concentration[b] (g/liter)			
	1.5	0.75	0.3	0.15
177 - 250	no flame	no flame	no flame	no flame
125 - 177	flame propagated	no flame	no flame	no flame
88 - 125	flame propagated	flame propagated	no flame	no flame
63 - 88	flame propagated	flame propagated	flame propagated	no flame

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[a]Ignition energy = 3 joules.

[b]Some coalescence of material.

Table 4. Ignition characteristics of M1 propellant

Particle size (μm)	Concentration (g/liter)	Ignition Characteristics[a]
75 – 150	0.1	No flame
44 – 150	0.6	No flame
44 – 75	0.3	Flame propagation

[a]Ignition energy = 3 joules.

Table 5. Ignition characteristics of ball powder
(dust concentration – 3.0 grams per liter)[a]

Particle size distribution		Ignition characteristic
Percentage	Size range (μm)	
0.11	20 – 30	Slow burning
0.45	30 – 50	Slow burning
0.28	50 – 70	Slow burning
0.04	70 – 100	Slow burning

[a]Ignition energy = 3 joules.

Table 6. Controlled sampling at Radford Army Ammunition Plant

Operation and location of sample	Sampling time (min)	Dust concentration (g/liter x 10^{-6})
Tray dumping of AHH* (at top of bin; bin vented on two of four sides)	2	< 18.3
Blending of ingredients for N5 roll powder (at mouth of blending barrel; one top and one bottom)	5	< 7.4
	5	< 12.4
Grinding of 2 DNPA and KNO_3 (behind each grinder)	5	< 6.7
	15	< 4.2
Block breaking of nitrocellulose (at output chute of block-breaker)	8	< 4.6
	8	< 7.8
Packing of can with M30 propellant (105 mm) (at bottom level where propellant dumps into bin)	17	64.0

*Refer to reference 22.

Table 7. Controlled sampling at Indiana Army Ammunition Plant

Location of sample*	Sampling time (hr)	Dust concentration (g/liter x 10^{-6})
A	2	58
B	2	4
C	2-1/4	1
D	1-1/4	10
E	1-1/4	19
F	2-1/4	85
G	3-1/4	294
H	1-1/6	242
I	2	20
J	1-1/6	165
K	1-1/6	78
L	2	6
M	1-1/6	199

*Refer to figure 11.

Table 8. Uncontrolled sampling[a] at Indiana Army Ammunition Plant

Location of sample[b]	Dust collected (g)
N	0.165
N	0.748
O	0.016
P	0.020

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[a]These samples cannot be related to a known flow rate of air, but instead relate to dust fallout over a 10.74 cm^2 area during a 90-minute period.

[b]Refer to figure 11.

Table 9. Controlled sampling at Milan Army Ammunition Plant

Operation and location of sample	Sampling time (hr)	Dust concentration (g/liter x 10^{-6})
Comp B flake dump:		
at mouth of hood no. 2	3-1/4	Negligible
at mouth of hood no. 3	3-1/4	Negligible
Conveyor:		
behind hood no. 2	4-5/12	1.28
behind hood no. 3	0.5	Negligible
behind hood no. 3	4-1/3	1.92
Riser scrap addition (above riser sump chute)	5-3/4	0.87
Riser knockout:		
at mouth of hood no. 1	4-1/4	Negligible
at mouth of hood no. 2	4-1/4	0.49
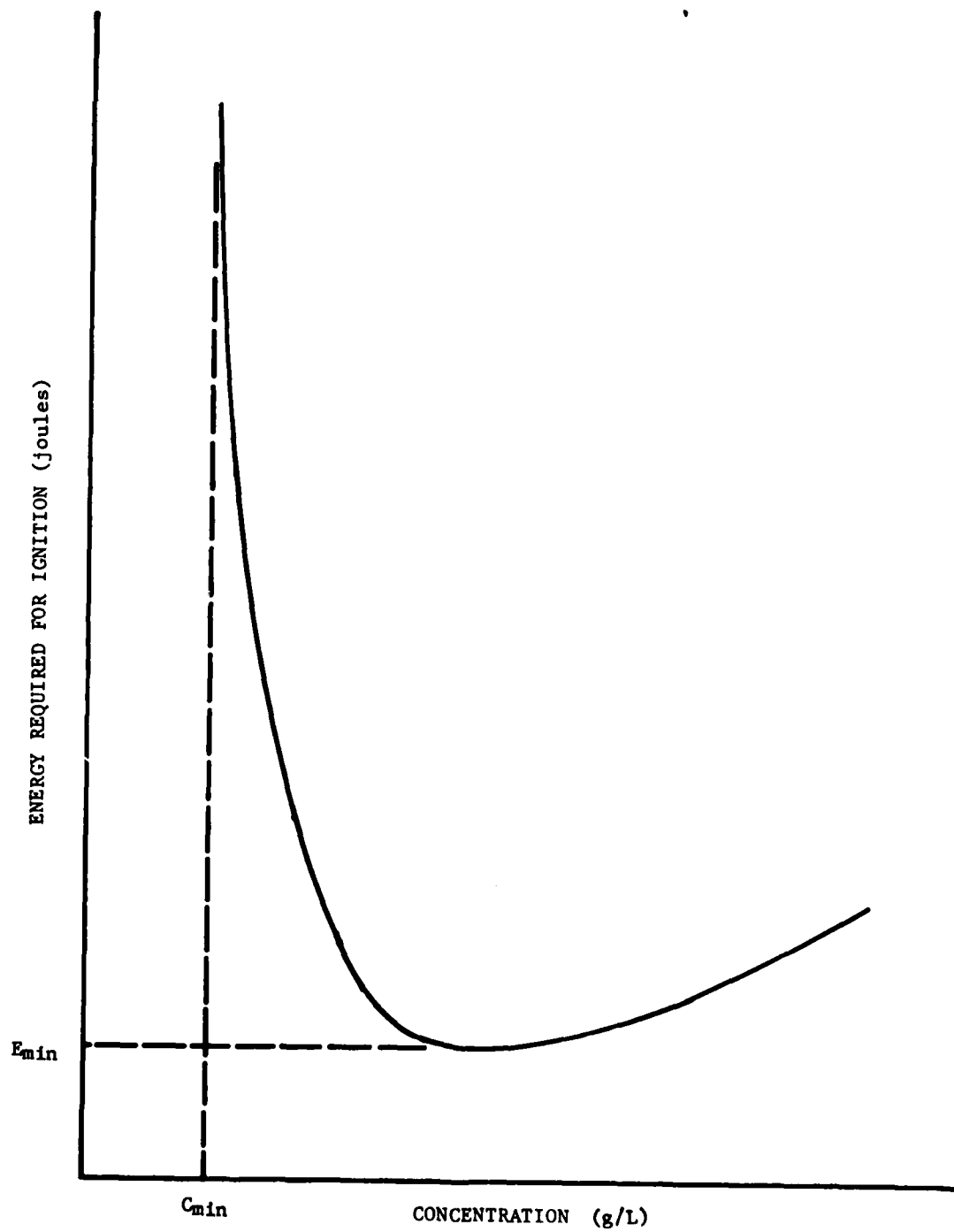
Figure 1. Typical ignition energy versus explosible
dust concentration curve.
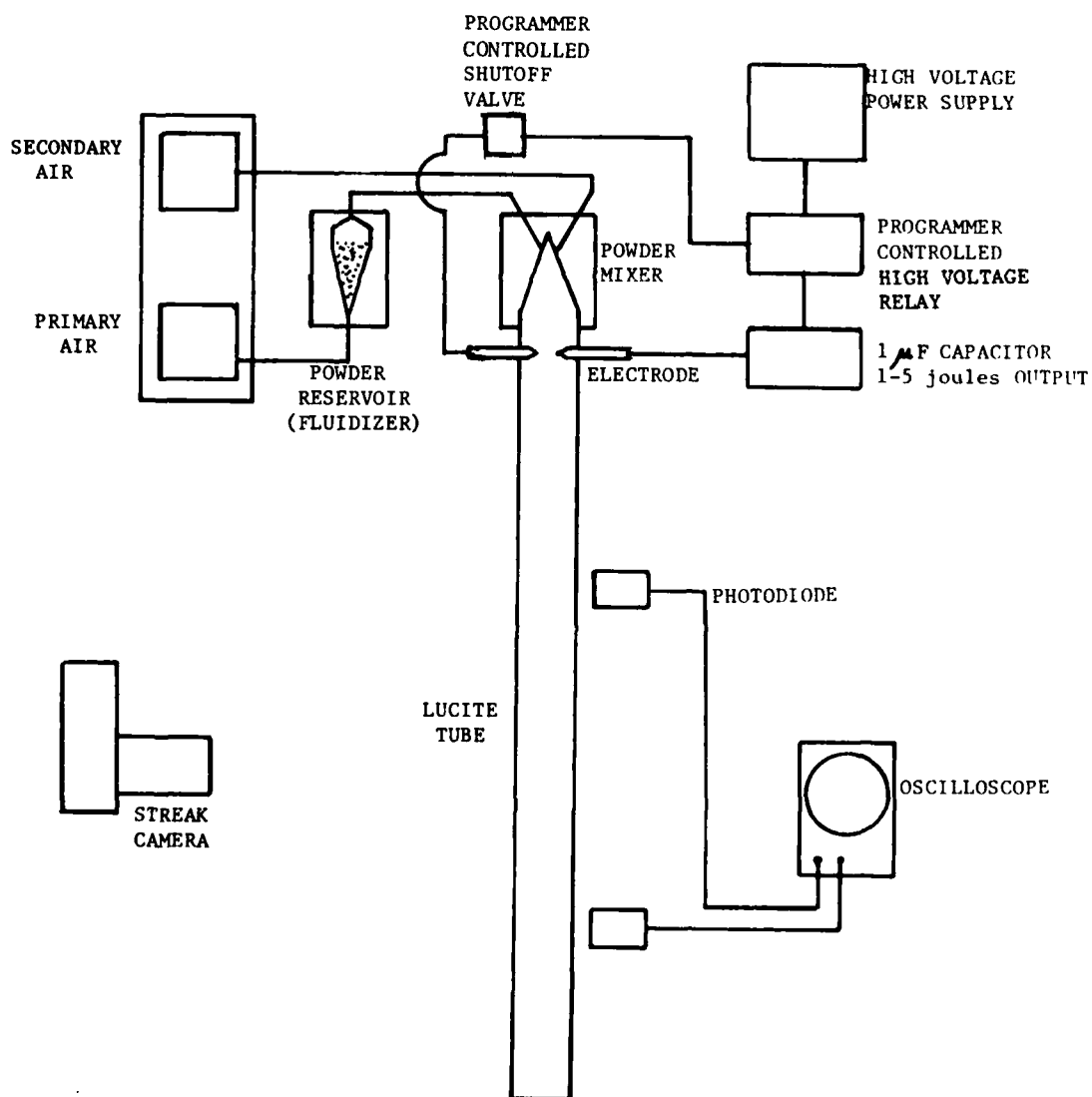
Figure 2. Schematic of spark ignition test apparatus.

22

CAPACITOR
BANK

DUST &
SECONDARY AIR
MIXING SECTION

DETONATION
TUBE

PHOTODIODES

STREAK PHOTOGRAPH REFERENCE MARKER

AIR HANDLING SYSTEM CONTROLS
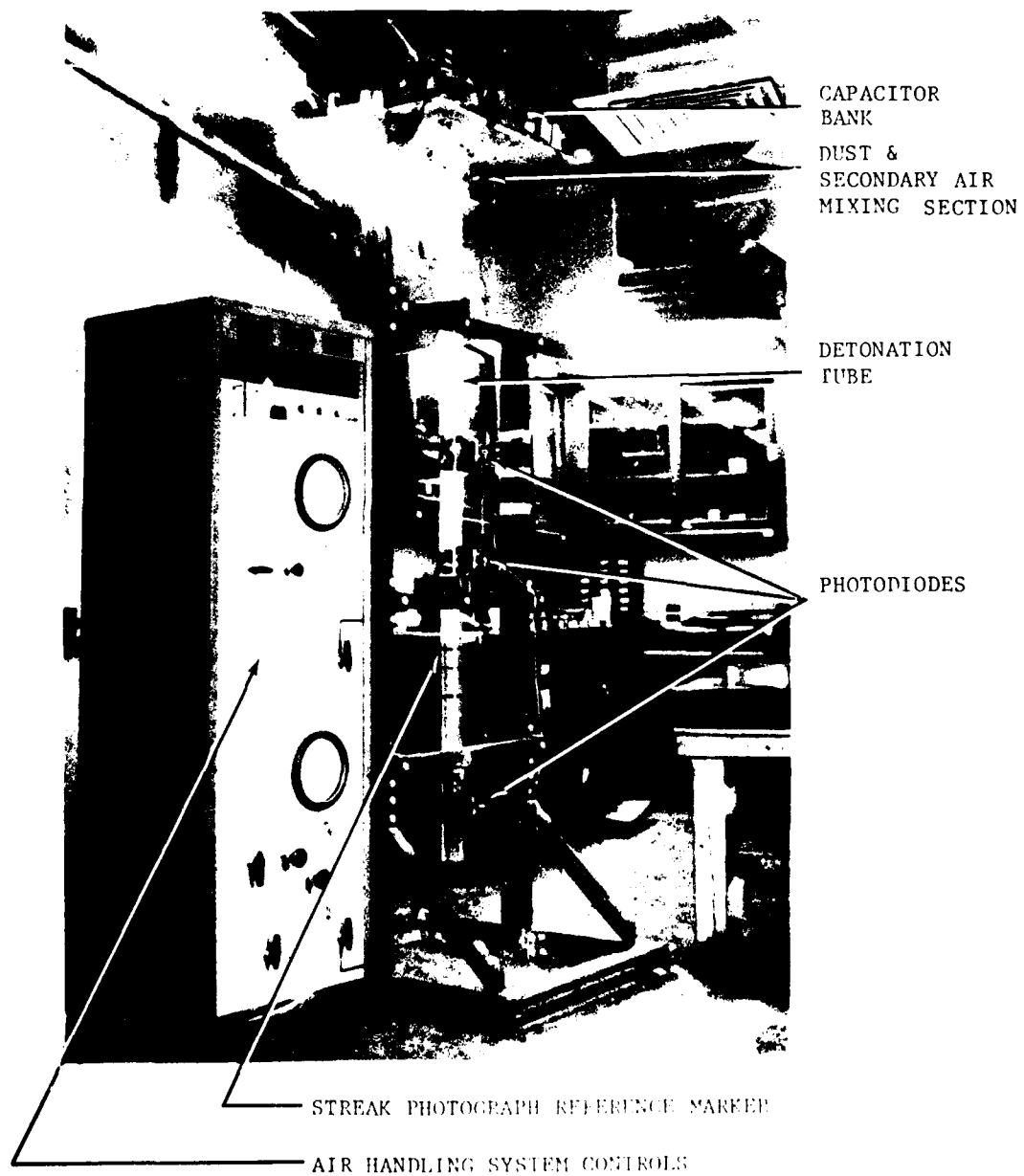
Figure 3.   Spark ignition test apparatus.
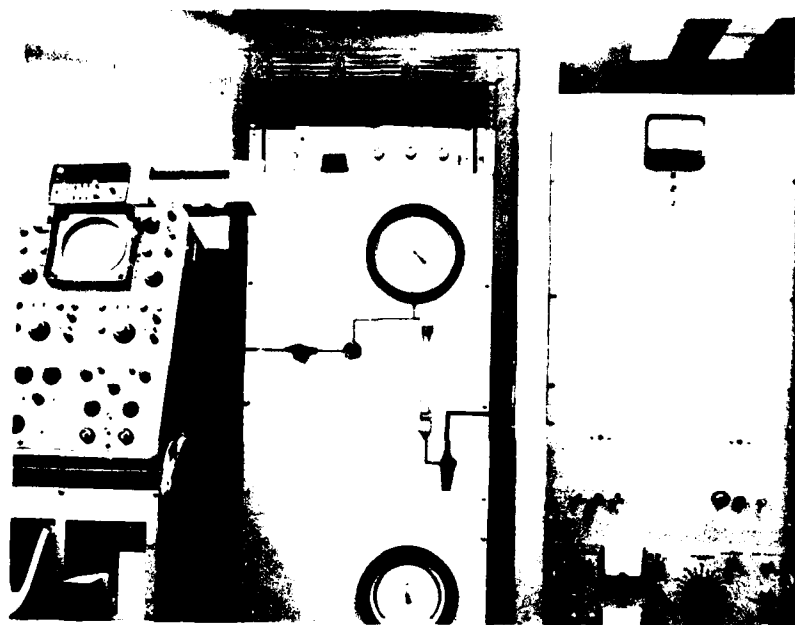
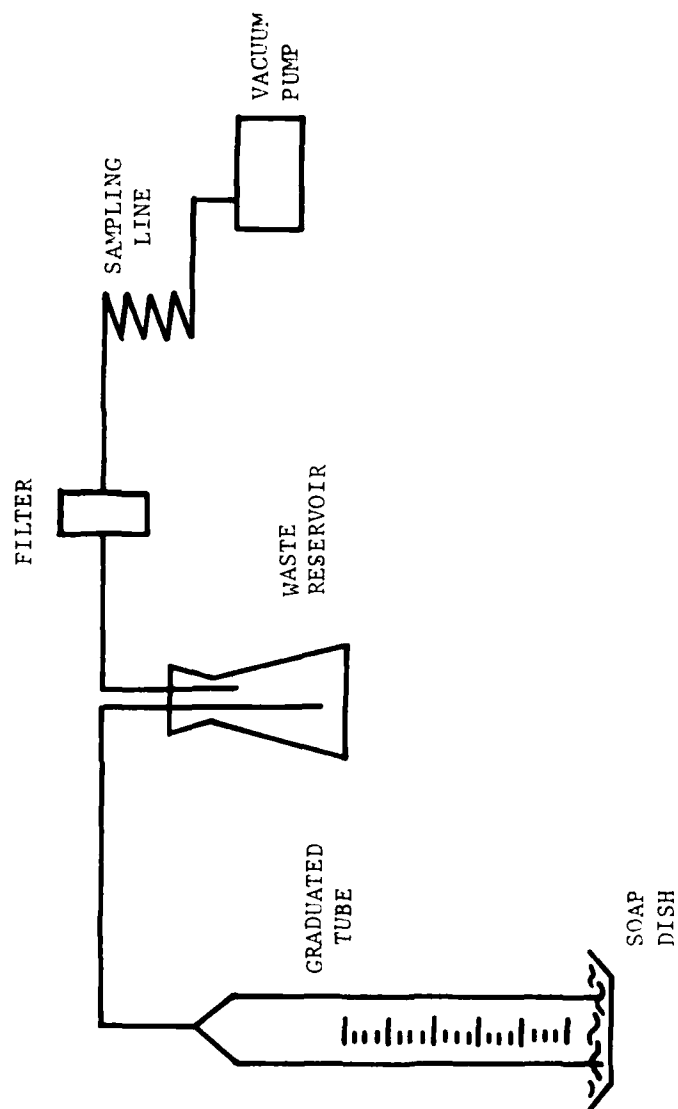| OSCILLOSCOPE | AIR HANDLING SYSTEM CONTROL | HIGH VOLTAGE POWER SUPPLY |

Figure 4. Spark ignition test apparatus control panels.

Figure 5. Apparatus for calibrating sampling pumps.
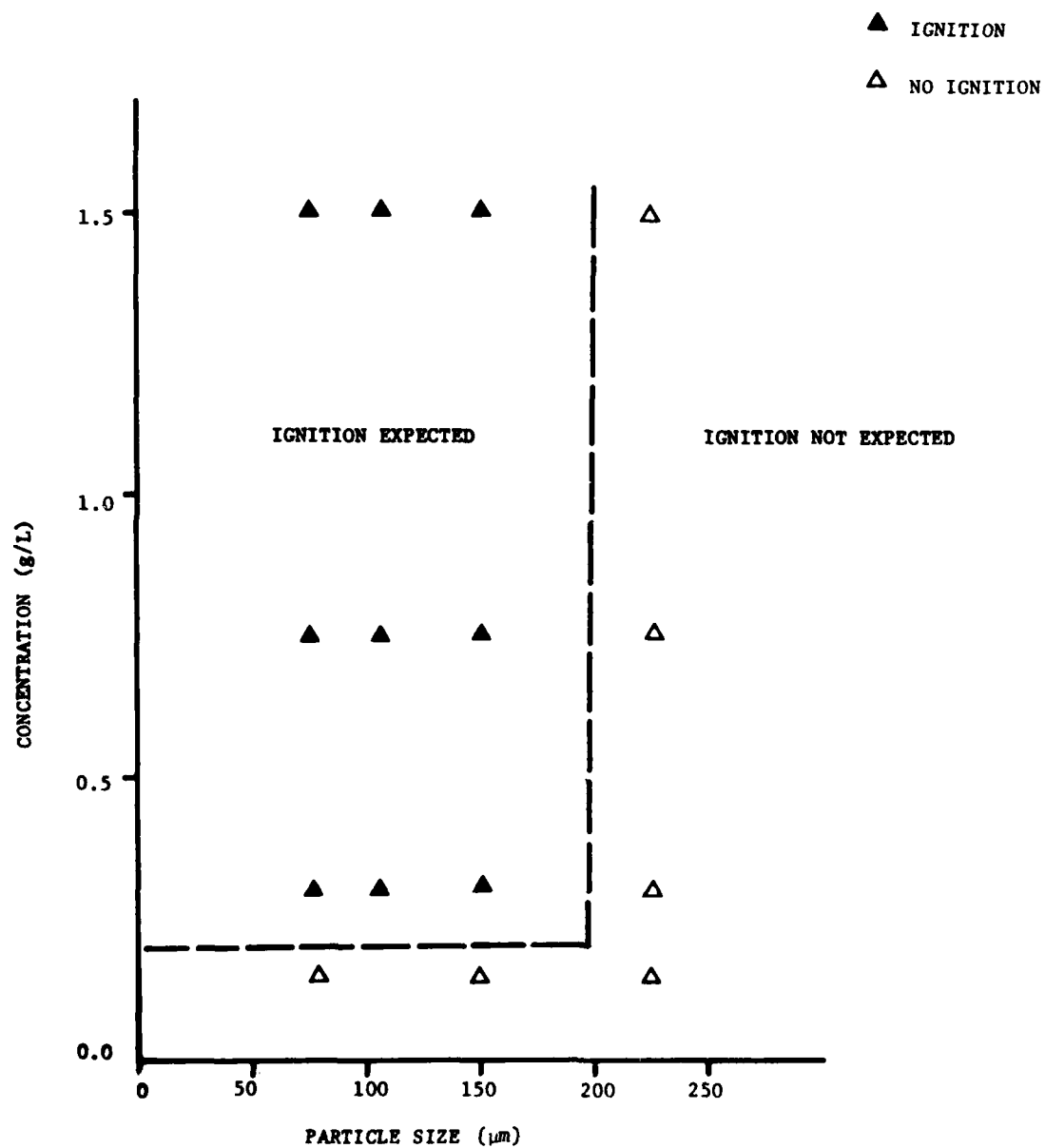
Figure 6.   Explosible dust concentration versus particle size for HMX.
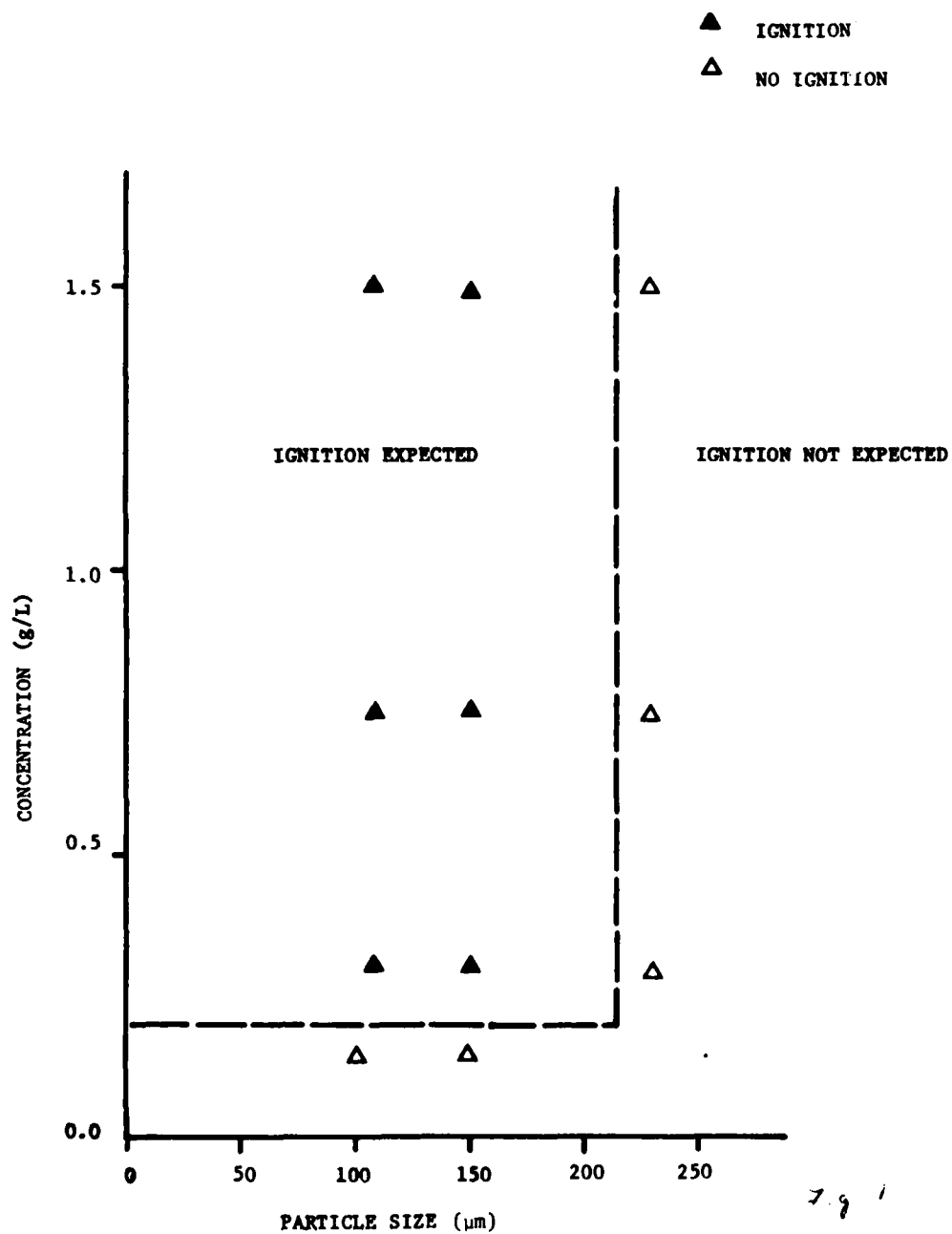
Figure 7. Explosible dust concentration versus particle size for RDX.
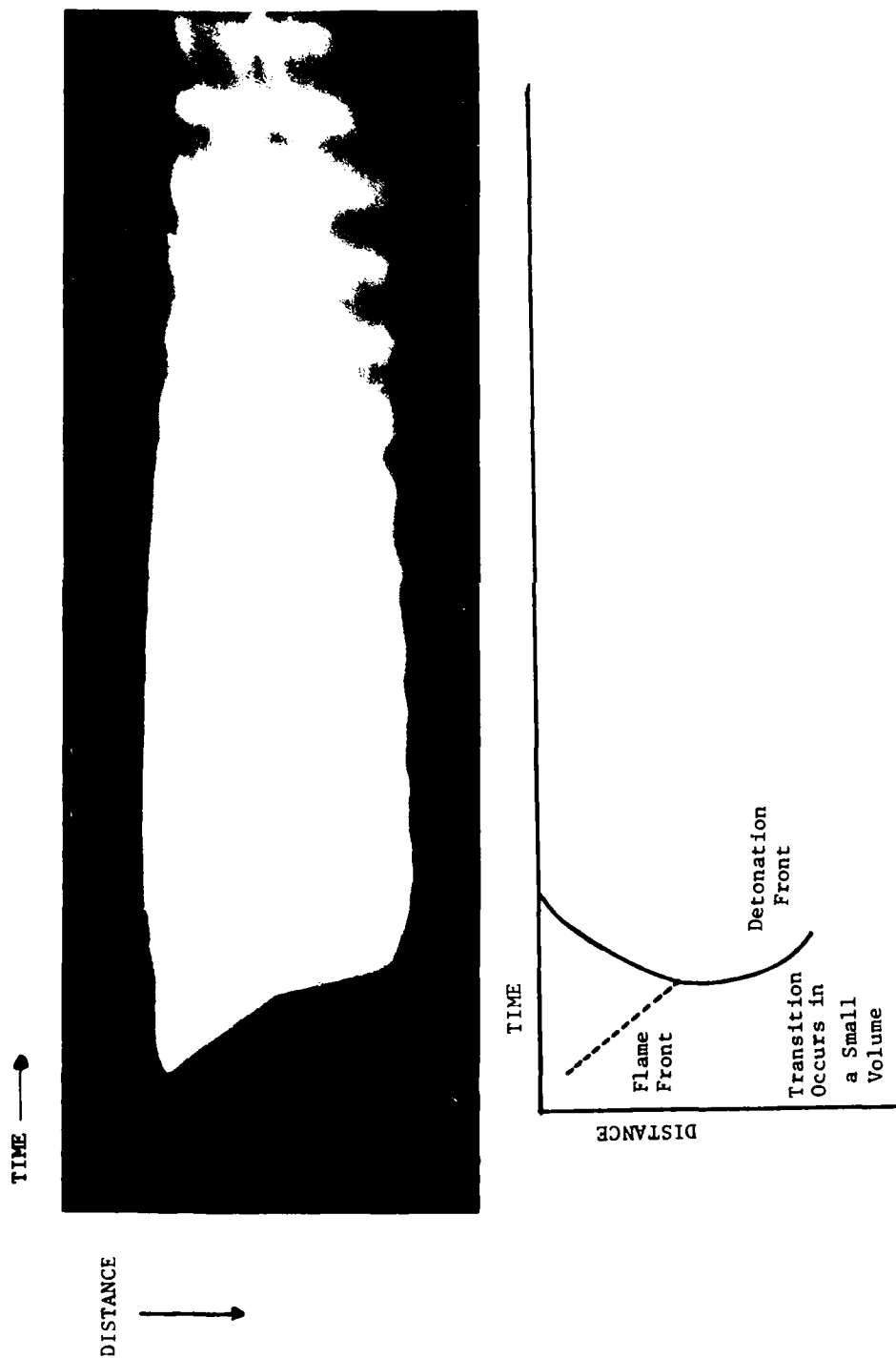
Figure 8. A deflagration-to-detonation transition for a spark initiated, stoichiometric propane-oxygen mixture.
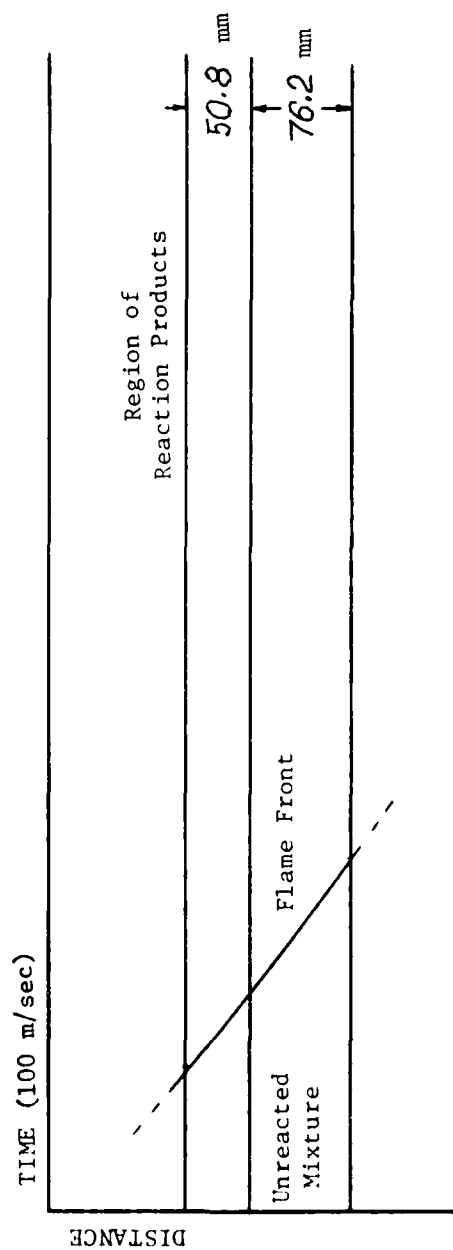
28

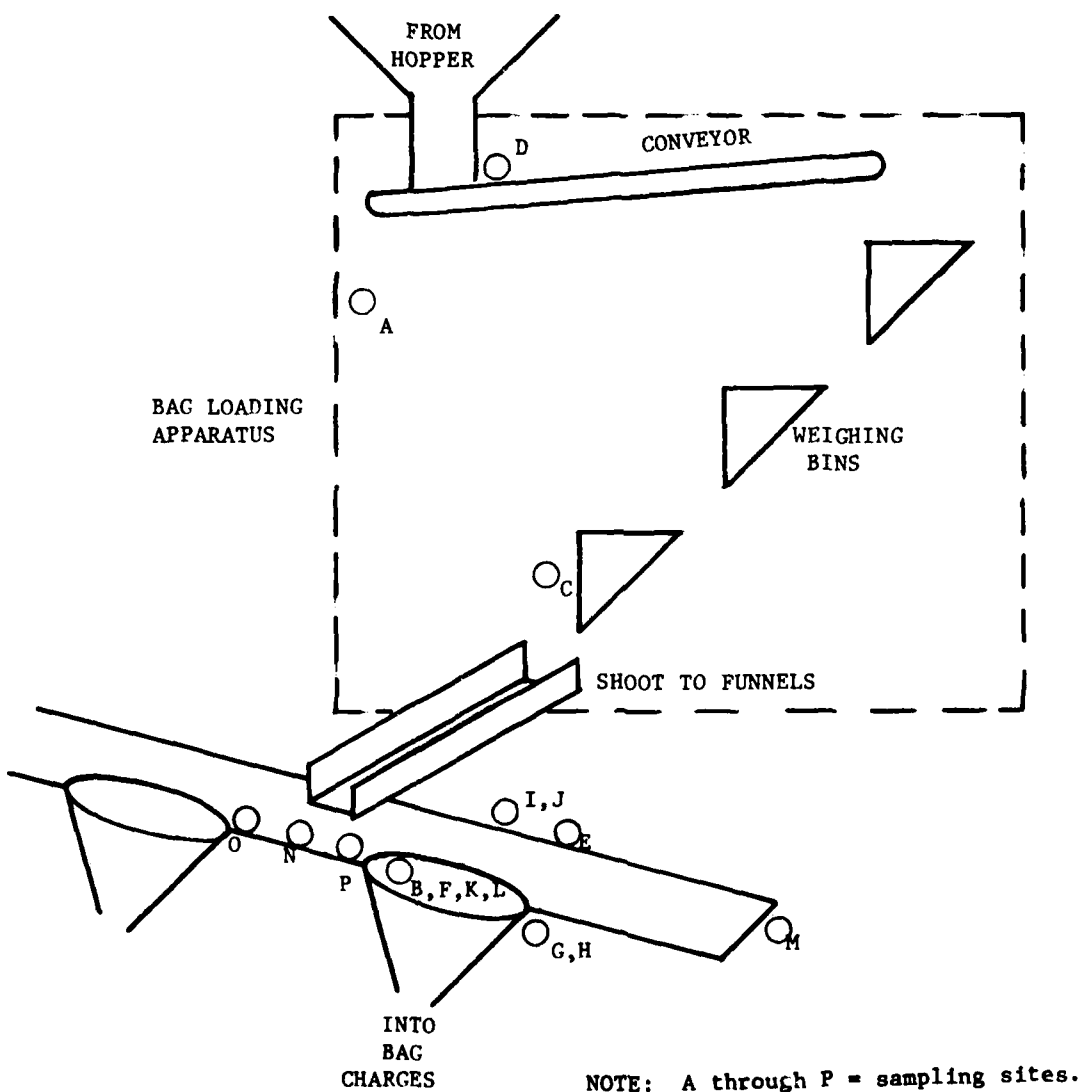Figure 9. Flame propagation through RDX powder dispersed in the air.

FROM
HOPPER

CONVEYOR

O D

O A

BAG LOADING
APPARATUS

WEIGHING
BINS

O C

SHOOT TO FUNNELS

O I,J

O E

O
O
O
N

O
B,F,K,L

P

O
G,H

O M

INTO
BAG
CHARGES

NOTE: A through P = sampling sites.

Figure 10.   Locations of sampling sites at Indiana Army
Ammunition Plant loading operation.

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