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PARTICLE SIZE EFFECT ON EXPLOSIVE BEHAVIOR OF AMMONIUM PERCHLORATE

Donna Price, et al.

U.S. Naval Ordnance Laboratory
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PARTICLE SIZE EFFECT ON EXPLOSIVE BEHAVIOR OF AMMONIUM PERCHLORATE

By

Donna Price, A. R. Clairmont, Jr., and I. Jaffe

ABSTRACT: Five different lots of ammonium perchlorate (AP) provided average particle sizes of 10 to 200 μ. They were used to show the particle size effect on the limit curves and the family of detonation velocity vs density curves. Decrease in particle size shifted the former to lower diameters and higher densities; the latter to higher detonation velocities and higher densities. Particle size did not affect ideal detonation velocities.

Use of the 10 μ and 25 μ AP data in the curved front theory gives a ratio of reaction times equal that of the average particle sizes. This result is consistent with heterogeneous reaction at the particle surfaces as is also the fact that shocked AP can undergo vigorous but fading reactions which can persist as far as nine diameters down the charge.

From earlier work, we had concluded that the extended gap test must be used for AP and that the gradient of the shock sensitivity curve (50% gap pressure vs % theoretical maximum density, P_g vs % TMD) is steeper than that for TNT. The present more thorough study has shown both of these conclusions to be wrong. Because granular AP can undergo a number of powerful explosive reactions which are not detonations, the regular gap test is actually preferable to the extended in testing it for shock sensitivity. At high porosities the gradient of the AP curve P_g vs % TMD is about the same as that for TNT, but the gradient of the AP curve increases rapidly at lower porosities because this material approaches a critical limit at the higher % TMD.

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This work was carried out under ORDTASK 033 102 F009 06 01 and is part of a continuing program on the systematic investigation of the explosive behavior of composite propellant models. The information of this report has been combined with that of the preceding report (NOLTR 67-71) into a paper which will be published in Combustion and Flame.

E. F. SCHREITER
Captain, USN
Commander

ALBERT LIGHTBODY
By direction

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INTRODUCTION

There is only a small amount of scattered information in the literature on the explosive behavior of composite oxidizer/fuel mixtures. Consequently a systematic study of composite propellant models has been initiated. The oxidizer chosen is ammonium perchlorate (AP) and the study has started with the explosive behavior of this component. It will continue with various AP/fuel mixtures and eventually include models containing common high explosives as components.

The explosive properties of particular interest are detonability, shock sensitivity and detonation behavior as functions of particle size, loading density, charge size, and chemical composition. The first report in this series[1] presented the detonability limits and detonation behavior for a finely ground AP. The object of this second report is to demonstrate the various effects of changing the particle size of the AP.

EXPERIMENTAL

Ammonium Perchlorate

All AP used was propellant grade and satisfied Navy Department specification OS 11354B except that the finest grinds (XP-17 and XP-11) contained 1% tricalcium phosphate. The manufacturer's analyses for other samples gave 99.3% AP and 0.15% tricalcium phosphate.

It has been the writers' experience that there is no satisfactory, rapid method of determining particle size, especially for

non-spherical particles. Exclusive reliance on one simple procedure such as the Micromerograph analysis can be very misleading. For example, one batch of AP was ordered with an average particle size of 45 μ. It arrived accompanied by a Micromerograph showing 44 μ at the mean (50% weight less than 44 μ diam.). However, on a Ro-Tap sieve analyzer 75% of this AP passed through the No. 325 screen (44 μ opening).

The differences we are looking for do not seem to justify the time and labor of a complete microscopic determination of particle size distribution on each batch. Consequently we have examined our samples in three ways: Micromerograph analyses, Ro-Tap sieve analyses, and by study of several photomicrographs (magnification of 60 -120 x) of each batch. Tables 1 and 2 contain the values for the first two sets of analyses, and Fig. 1 presents typical photomicrographs. Nominal mean sizes, which seem consistent with all the information, have been listed in Table 3. Typical Micromerographs are given in the Appendix.

Charge Preparation

The AP was dried (four or more hours at 50°C), handled, and made into cylindrical charges as in the previous work.[1] The charge preparation had to be modified according to the particle size, e.g., the lowest loading densities that could be obtained were 0.6, 0.9, and 1.3 g/cc, respectively, for the 10, 25, and 200 μ AP. Hence the charge density range that could be investigated narrowed as the AP became more coarse. The method of preparation for each charge is shown in the tables.

Measurements

The experimental set-up and instrumentation were the same as in the previous work.[1] As before, the details for the 70 mm smear

TABLE 1

Micromerograph Analyses*

AP	Injection pressure Psig	Sample Recovery, %	50 wt. % mean, μ	Source	Fig. No. of Record
XP-17	75	-	10.3	a	A1
	75	42	12.4	NOL	
	200	37	10	NOL	A2
XP-11	75	-	9.3	a	A3
N-125	200	60.4	25	NOL	
	200	53.2	31	NOL	
	200**	56.5	28	NOL	A4
	200	27	11.6	b	
N-126	200	30	26.5	NOL	A5
	75	16	26	NOL	
	-	-	44	c	

a. Thiokol Chemical Corp.
b. Naval Propellant Plant where this sample was ground.
c. American Potash & Chemical Corporation.

* Deagglomerator Slit Setting at 250μ.
** Sample dried in vacuo at 100°C. All others (at NOL) dried at 50°C in standard fashion.

TABLE 2

Ro-Tap Sieve Analyses

AP	Wt. % Retained on Sieve No: 100	140	200	230	270	325	Pan
XP-17	5.0[a]	2.7	1.1	0.6	0.9	0.4	86.5
	6.3[a]	2.6	1.4	0.9	1.0	1.7	84.4
XP-11	27.2[a]	3.8	1.7	1.1	0.9	1.1	60.8
	27.8[a]	3.9	1.6	1.0	0.8	1.2	60.9
N-125	0.4	3.3	7.0	6.8	10.0	10.3	61.1
	0.4	3.2	7.1	6.9	9.9	10.2	61.2
N-126	0	1.0	2.5	3.8	6.0	10.3	74.6
	0	1.1	2.4	3.0	4.1	10.1	74.8
N-119	74.6[b]	14.0	4.7	-	-	-	4.15 (50% wt. average of 200μ)

a. High percentage attributed to agglomeration, some portion of which might occur during the sieving operation (one hour in uncontrolled humidity). No clumps of this size (~150μ) were detected microscopically. Thiokol supplied these samples with the following sieve analysis:

	On screen No. 100	140	200	230	270
XP-17	0.5	1.0	1.6	0.5	0.3
XP-11	4.2	3.3	2.2	0.6	0.6

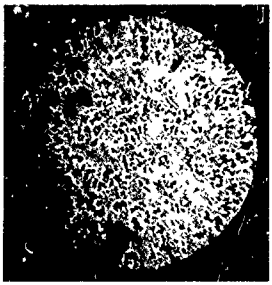
The separator speed was 3500 RPM and the equipment was probably the Alpine Air-Jet Sieve.[2]

b. Retained on sieves no. 40, 50, 70, and 100 were respectively 0, 3.4, 37.1, and 34.1%.

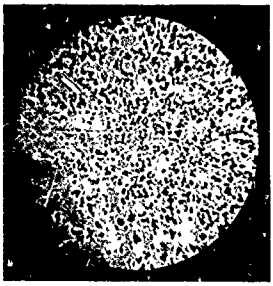
TABLE 3

Particle Size Assigned

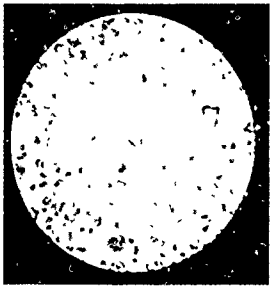
AP	Nominal mean particle diameter, μ	Comment
XP-17	10	Critical diameter at 1 g/cc close to those found by others on AP of 12-14μ (by Micromerograph).
XP-11	11	Assignment made to indicate higher percentage of coarser particles.
N-125	25	These samples appear much the same by all three methods.
N-126	25	
N-119	200	



XP-17



XP-11



N-125

N-126

N-119

100μ

FIG. 1 PHOTOMICROGRAPHS OF VARIOUS LOTS OF AP

camera records (film, flasher, length of charge observed, and writing speed) are given in the appendix (Table A-1). The records were reduced and interpreted by the methods developed and described earlier.[1]

RESULTS AND DISCUSSION

Detonation Behavior Pattern

Tables 4 and 5 contain the data obtained from charges prepared with a 25 μ AP (N-126) and a 200 μ AP (N-119); a summary of the failure or limit data appears at the end of Table 4. The detonability curve is shown in Figure 2, and the curves D vs ρ_o at various diameters are plotted in Fig. 3.

It was expected that increasing the particle size from 10 μ in the earlier work[1] to 25 μ would shift the detonability limit curve to higher critical diameters, and it did as Fig. 2 shows. The shift was such that detonation could be obtained only with charges of $d \geq 3.81$ cm. Consequently fewer D vs ρ_o curves could be obtained. Moreover, the range of experimental densities was reduced by the packing characteristics of the coarser AP; 0.9 g/cc was the lowest packing density that could be prepared. Consequently, the curves of Fig. 2 cover a lower range in ρ_o than that of the earlier work; the scatter of the data is also greater, probably because of greater charge heterogeneity.

As indicated in Table 5 and Fig. 2, the very coarse AP (200 μ) could not be detonated at its pour density of 1.29 g/cc in a 7.62 cm (3.0 in.) diameter charge. Nevertheless, a vigorous reaction, as indicated by the luminosity of the smear camera trace, was initiated and persisted over most of the length of the charge (to about 16 cm or 2.1 diam). Such fading but vigorous reactions are typical of AP (any particle size) and make exact determination of the limit

TABLE 4

Data from AP N-126 (25μ)

Shot No.	ρ_o[a] g/cc	D mm/μsec	Reaction Quenched at δ cm	diam	Shock Vel. mm/μsec	Comment
			Diameter = 2.54 cm; (ℓ/d) = 8.0			
185	0.91	F[b]	7.2	2.8	1.54	
186	1.00	F	6.8	2.7	1.53	
187	1.11	F	6.6	2.6	1.83	
			Diameter = 3.49 cm; (ℓ/d) = 5.8			
190	0.90	F	14.2	4.1		
196	0.97	F	16.7	4.8		
191	1.01	F	----	---	1.74	Poor record
195	1.01	F	19.5	5.6	1.63	Charge length of 30.5 cm; (ℓ/d) = 8.7
197	1.06	F	17.0	4.9		
198	1.06	F	19.7	5.6	1.98	
192	1.11	F	----	---	1.87	Poor record.
199	1.16	F	15.9	4.6	1.93	
200	1.16	F	16.9	4.9	1.92	
193	1.20	F	15.9	4.6	2.11	
			Diameter = 3.81 cm; (ℓ/d) = 5.3			
202	0.93	1.58	----	---		
203	1.02	1.75	----	---		
204	1.11	F	>20.3	>5.3	2.01	Very near critical value.
205	1.24**	F	14.9	3.8	2.06	

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TABLE 4 (Contd.)

Shot No.	ρ_o* g/cc	D mm/μsec	Reaction Quenched at δ cm	diam	Shock Vel. mm/μsec	Comment
			Diameter = 5.08 cm; (ℓ/d) = 4.0			
169	0.90	2.28				
194	0.95	2.36				
179	1.01	2.64				
178	1.02	2.60				
172	1.10	2.69				
181	1.20**	2.81				
180	1.20**	2.84				
183	1.28*	2.89				
182	1.29*	2.92				
171	1.33*	2.70				
201	1.35*	2.74				
189	1.36*	2.67				Charge 40.6 cm long; (ℓ/d) = 8
188	1.41*	F	18.5	3.6	2.73	
			Diameter = 7.62; (ℓ/d) = 2.7			
206	0.89	2.68				
207	1.10	3.19				
218	1.29*	3.58				
227	1.39*	3.67				
217	1.47*	3.45				
226	1.56*	F	>20.3	>2.7	3.10	Very near critical value.

TABLE 4 (contd.)

Summary of Detonability Data

Diam d(cm)	ρ_o +	ρ_o -	
2.54	None	0.9-1.1	
3.49	None	0.9-1.2	
3.81	1.02	1.11	Failure value close to critical.
5.08	1.36	1.41	
7.62	1.47	1.56	Failure value close to critical.

[a] Charges handpacked unless density value followed by * (isostatically pressed) or ** (hydraulically pressed).

[b] Failure.

TABLE 5

Data from AP N-119 (200 μ)

Shot	d cm	ρ_c	D	δ	Shock Velocity mm/μsec
239	7.62	1.29	F	15.8 cm 2.1 d	1.78

Observation length 6 in.,
writing speed 2 mm/μsec

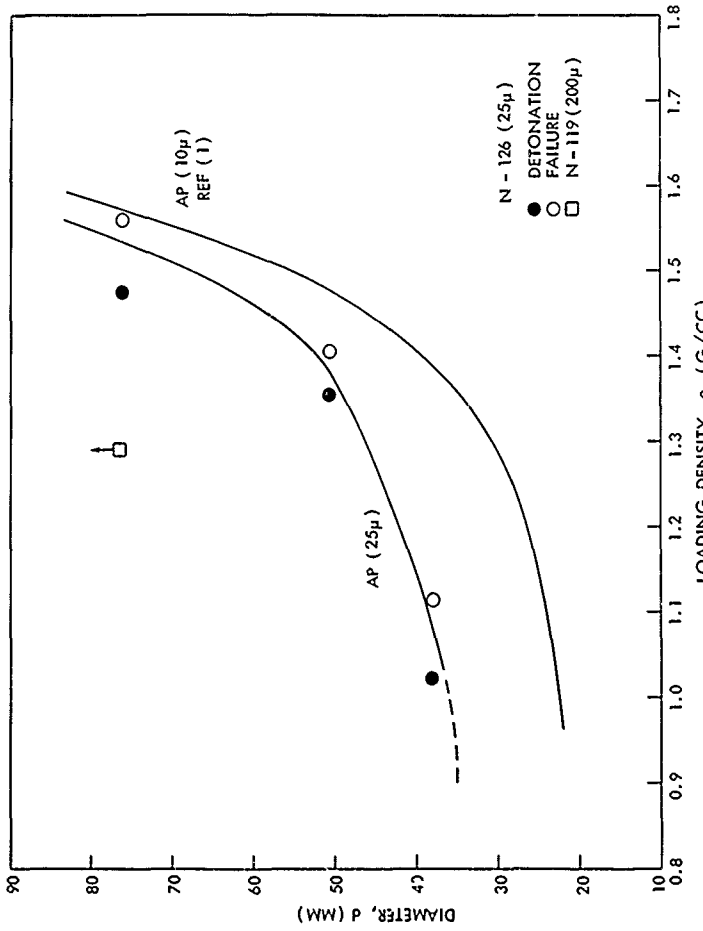

FIG. 2 EFFECT OF PARTICLE SIZE ON DETONABILITY LIMITS OF AP

curves very difficult. Table 4 shows a number of fading reactions persisting in the 25 μ AP to five diameters and more (Finer AP shows persistence to more than 9d; see Table 7). The fact that such reactions are of explosive proportions is indicated not only by luminosity, but also by conductance measurements[1], and by their ability to initiate high explosive witnesses. This will be described in a later section on shock sensitivity investigations.

The NOL firing regulations require that $d \leq 7.62$ cm; at the other end of the range, detonability requires that $d \geq 3.81$ cm. Consequently, as Fig. 3 shows, our data can be used only for a two-point extrapolation (D vs d^{-1}) to the infinite diameter detonation velocities D_i. These were obtained in the range 0.90 to 1.29 g/cc and are listed in Table 6; three values, those computed from measured velocities rather than interpolated values on the D vs ρ_o curves, are plotted in Fig. 3 together with the ideal curve derived for the 10 μ AP[1]. It is clear that well within the scatter of the present results and possible error (±0.1 mm/μsec) of the reference curve, the particle size of the AP has no effect on the ideal curve, i.e., the same D_i vs ρ_o relation will be obtained for AP of any particle size, provided measurements can be carried out on charges of sufficiently large diameter.

Comparison of Fig. 3 with the analogous curves (Fig. 9 of Ref. 1) readily shows that at a fixed diameter and loading density, the detonation velocity increases with decreasing particle size. To make a more quantitative comparison, the available 5.08 cm diam D vs ρ_o curves are compared in Fig. 4. (Data for the 11 μ AP are given in Table 7. This AP is slightly coarser than the 10 μ AP of Ref. 1; its limit curve lies between the two of Fig. 2 and is closer to the lower curve.) The plots of Fig. 4 show that although the two fine AP's are difficult to distinguish at low densities, they are readily differentiated at the higher densities by their limit behavior. They also demonstrate the existence of a critical

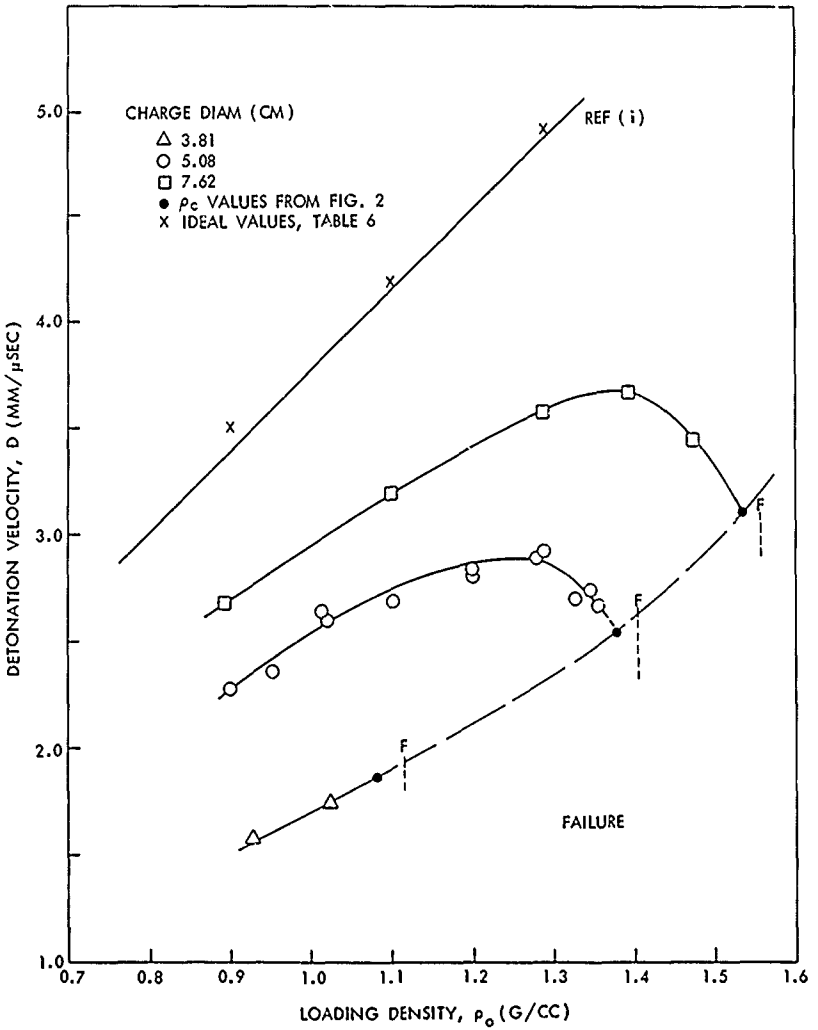

FIG. 3 DETONATION BEHAVIOR OF AP, N-126 (25μ)

TABLE 6

Infinite Diameter Values (D_1) Obtained by Two Point Extrapolation, AP (25μ)

Loading Density ρ_o (g/cc)	D_1* mm/μsec	$10^{-1}b$ $mm^2/\mu sec$	Nominal Reaction Zone length a (mm)
0.90	3.50	6.21	17.8
1.00	(3.76)	(6.21)	(16.5)
1.10	4.18	7.58	18.1
1.10	(4.08)	(6.82)	(16.7)
1.15	(4.25)	(7.27)	(17.1)
1.20	(4.47)	(8.18)	(18.1)
1.25	(4.71)	(9.25)	(19.6)
1.29	4.89	10.0	20.7

* $D=D_i - bd^{-1}$; $a=b/D_i$

Values in parentheses from smoothed curves of Fig. 3; others from data of Table 4

TABLE 7

Data from AP, XP-11, (11μ)

Shot No.	Loading Density[a] % g/cc	Detonation Velocity D mm/μsec	Reaction Quenched at δ cm	diam	Front Velocity at end of Record mm/μsec	Comment
			Diameter = 1.90 cm; (ℓ/d) = 10.7			
128	0.60	F[b]	7.6	4.7	--	
130	0.70	F	8.9	4.0	--	
135	0.70	F	8.9	4.0	--	Extra boosting used
129	0.30	F	7.6	4.7	--	
158	0.60	F	10.8	4.9	1.48	
156	0.70	F	16.7	7.5	1.39	
154	0.70	F	13.3	6.0	1.42	
155	0.80	F	13.3	6.0	1.54	
165	0.80	F	12.1	5.5	1.59	
166	0.90	F	19.0	8.6	1.75	
167	0.95	F	15.2	6.9	1.85	
168	1.00	F	15.8	7.1	1.87	
209	1.00	F	7.6	3.4	2.03	
210	1.00	F	9.3	4.2	1.91	
208	1.01	F	no trace	--	--	
161	1.01	F	>20.3	>9.2	2.06[c]	
212	1.24*	F	6.3	2.8	2.31	
			Diameter = 2.54 cm; (ℓ/d) = 8			
121	1.06	2.45	--	--		Resolution good.
122	1.10**	F	>20.3	>8	2.22	
123	1.10**	F	18.5	7.3	2.33	
125	1.15**	F	9.5	3.7	--	

TABLE 7 (Cont.)

Data from AP, XP-11, (11μ)

Shot No.	Loading Density[a] ρ_0 g/cc	Detonation Velocity D mm/μsec	Reaction Quenched at δ cm	diam	Front Velocity at end of Record mm/μsec	Comment
		Diameter = 5.08 cm; (ℓ/d) = 4.0				
233	0.68	2.23				
232	1.00	3.22				
231	1.15**	3.53				
224	1.32*	3.77				
230	1.36*	3.59				
229	1.37*	3.53				
223	1.39*	3.62				
228	1.45*	F	>20.3	>4	3.11	
221	1.49*	F	16.5	3.2	3.03	

Summary of Detonability Data on XP-11

Diam d(cm)	ρ_c +	ρ_c -	
1.90	None	0.6 to 0.8	
2.22	None	0.6 to 1.24	
2.54	1.06	1.10	
5.08	1.39	1.45	Failure value close to critical

[a] Charges handpacked unless density value followed by * (isostatically pressed) or ** (hydraulically pressed).

[b] Failure

[c] Trace appeared linear, but resolution poor since camera observed 17.8 cm length at only 1 mm/μsec writing speed. Coupled with failure of duplicate charge and of finer AP at these conditions, failure has been assumed here.

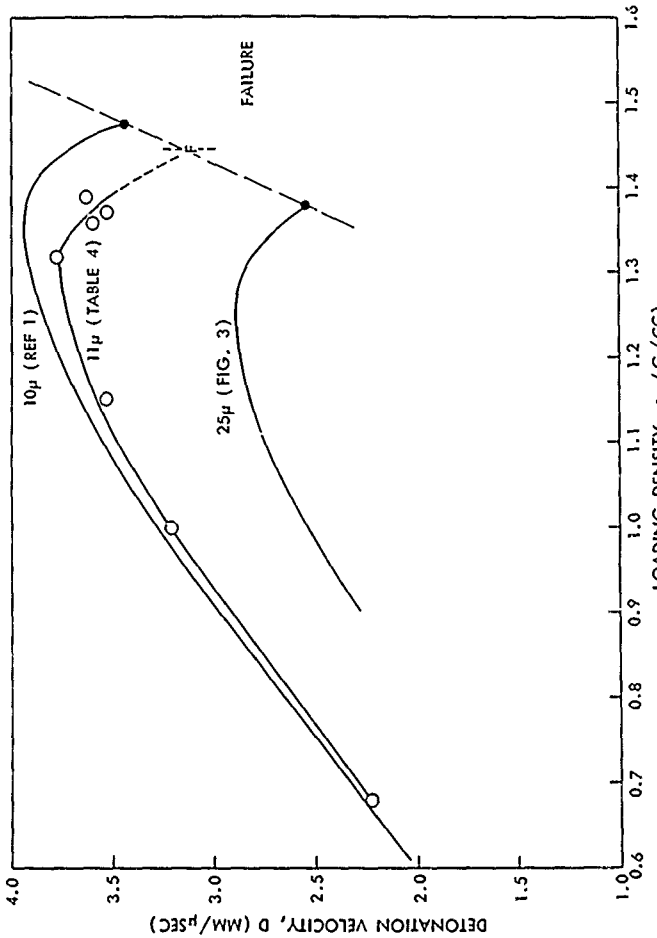

FIG. 4 EFFECT OF PARTICLE SIZE ON DETONATION VELOCITY OF AP IN 5.08 CM DIAMETER CHARGES

D vs critical ρ_o limit curve determined by variation in particle size at a fixed charge diameter. This is analogous to the limit curve D_c vs ρ_c of Fig. 3 determined by variation in charge diameter at fixed particle size and implies the existence of a limit curve, particle size vs ρ_o, analogous to the limit curve d vs ρ_o of Fig. 2.

In an earlier study[3] of the effect of particle size of AP on the detonation velocity at fixed diameter of a composite explosive, it was found that D appeared to vary linearly with the particle size, i.e.

$$D = D_1 - A\alpha \qquad (1)$$

where D_1 = detonation velocity at zero particle size
A = constant > 0
α = particle diameter.

It is interesting to treat some of the present data as if (a) the mean particle size assigned is an adequate characterization of the material and (b) the relationship of Eq (1) is applicable. This has been done by making a two point extrapolation of the velocities measured on the largest diameter charges (7.62 cm) of 10 μ AP[1] and of 25 μ AP (Fig. 3). The resultant zero particle size D_1's are listed in Table 8 and compared there with the corresponding D_i values. Over the ρ_o range of 1.0 to 1.2 g/cc, where the D vs ρ_o curves are practically linear for both AP's, such extrapolated values equal the corresponding ideal values. At $\rho_o > 1.2$, the particle size extrapolation gives $D_1 < D_i$. Such may indeed be the case, since the higher the density, the less ideal the behavior of AP. On the other hand, for reasons discussed in Ref. 1, the derived D_i values may be too high at the higher densities.

Reaction Zone Length, Reaction Time

As in the previous work[1], the Eyring curved front theory[4]

TABLE 8

Extrapolation to Zero Particle Size of 7.62 cm Diameter AP Charge Data

ρ_o g/cc	Detonation Velocity (mm/μsec) N-126 25μ	XP-17* 10μ	Extrap. 0μ	Ideal D_i*
1.0	2.92	3.43	3.77	3.77
1.1	3.19	3.78	4.17	4.15
1.2	3.40	4.00	4.40	4.52
1.3	3.60	4.27	4.72	4.90**

* Reference 1
** Extrapolated value

has been used to calculate nominal reaction zone lengths. According to this theory

$$D = D_i - aD_i d^{-1} \tag{2}$$

and the reaction zone length (a) is given by

$$a = b/D_i \tag{3}$$

where b is the slope of the D vs d^{-1} curve. Over the range of linear D vs d^{-1}, the reaction zone length is assumed independent of the diameter. Moreover, this zone length is related to the reaction time τ by

$$a = (D - \bar{u})\,\tau \tag{4}$$

where $\bar{u}$ is the average particle velocity between the leading shock (von Neumann pressure) and the C-J plane. Measurements in organic explosives give a variation in this region of $u_N = 1.25$ to $1.55\ u_j$* $\cong 1.4\ u_j$, where the subscripts j, N refer to the C-J and von Neumann planes, respectively. If we now approximate $\bar{u} \cong 1.2\ u_j$, Eq. (4) becomes

$$a \cong (D - 1.2\ u_j)\,\tau \tag{5}$$

In Eq. (5), D and u_j are functions of the diameter; so too must τ be if a is to be independent of d. Since pressure and temperature will vary with diameter, it is reasonable to expect that the reaction time will also vary. If we now apply Eq. (5) to the infinite diameter situation, it becomes

$$a \cong (D_i - 1.2\ u_{ji})\,\tau_i \tag{6}$$

*Ref. 5

and we can use the approximation from hydrodynamic theory that

$$u_{j1} = D_1/(k + 1)$$

Eq. (6) then becomes

$$a \cong \left(\frac{k - 0.2}{k + 1}\right) D_1 \tau_1 \qquad (7)$$

where a, k, D_1, τ_1 are, for a given material, functions of ρ_o only, and a typical variation of k over the range of 1.0 to 2.0 g/cc would be 2.5 to 3.3. If we change the material by changing its particle size, D_1 and k (at a given density) will be unchanged, but a and τ_1 would be expected to vary. Moreover, from Eq. (7)

$$\frac{a_1}{a_2} \cong \frac{\tau_{i1}}{\tau_{i2}} \qquad \rho_o \text{ fixed.} \qquad (8)$$

According to the grain burning theory, reaction occurs sequentially in molecular layers and moves from the outer surface of the particle toward the interior until the entire particle has reacted. The time for this process would be

$$\tau = \frac{R_g}{\lambda K} \text{ and hence } \tau_1 = \frac{R_g}{\lambda K_1} \qquad (9)$$

where R_g is the particle radius, λ the molecular diameter and K is the specific reaction rate for a single molecule. In particular K_1 is the rate at the C-J temperature for the infinite diameter conditions and is independent of particle size. Hence Eq. (8) becomes

$$\frac{a_1}{a_2} \cong \frac{\tau_{i1}}{\tau_{i2}} = \frac{R_{g1}}{R_{g2}}, \quad \rho_{o1} = \rho_{o2} \qquad (10)$$

and the ratio of the reaction zone lengths should equal the ratio of the reaction times (infinite diameter conditions) and the ratio of the particle sizes.

Values for the nominal reaction zone length, a, have been given as a function of ρ_c for the 10 μ AP in Ref. 1 and in Table 6 of this report for the 25 μ AP. They are plotted in Fig. 5 which shows a continuous, smooth variation* of a with ρ_o. The zone length, a, first decreases slightly with increasing ρ_o, and then increases. Unfortunately, the conventional trend, decreasing a with increasing ρ_o, occurs in the lower part of the density range where preparation of uniform and reproducible charges is most difficult. However, the appearance of such a trend for both APs strengthens the possibility that it is real.

In Fig. 5, as in other functional relations, the 25 μ AP produces much the same form of curve as does the 10 μ AP; it is merely compressed into the smaller density range possible for the larger sized material. If we now compare the ratio of the zone lengths, we find that over the ρ_o range of 0.90 to 1.20 g/cc $a(10\ \mu)/a(25\ \mu) = 0.40 \pm 10\%$. This compares very favorably with the particle size ratio (10/25) = 0.40 and is consistent with the effect of particle size on reaction time as deduced from the grain-burning theory. However, the consistent increase in the ratio with increasing ρ_o, particularly the increased gradient at ρ_o = 1.26 g/cc (see Table 9) indicates some fault in the computed values of a or the derived values of b and D_1 or both.

There is another way that the ratio of reaction zone lengths, and hence of ideal reaction times, can be considered. That is by use of the measured failure conditions, d_c and D_c. It is generally agreed that the critical or failure diameter is that diameter for

*One value derived from smoothed D vs ρ_o data has been indicated in Table 6; it has been used in place of that computed from the raw data to obtain the 25 μ curve of Fig. 5.

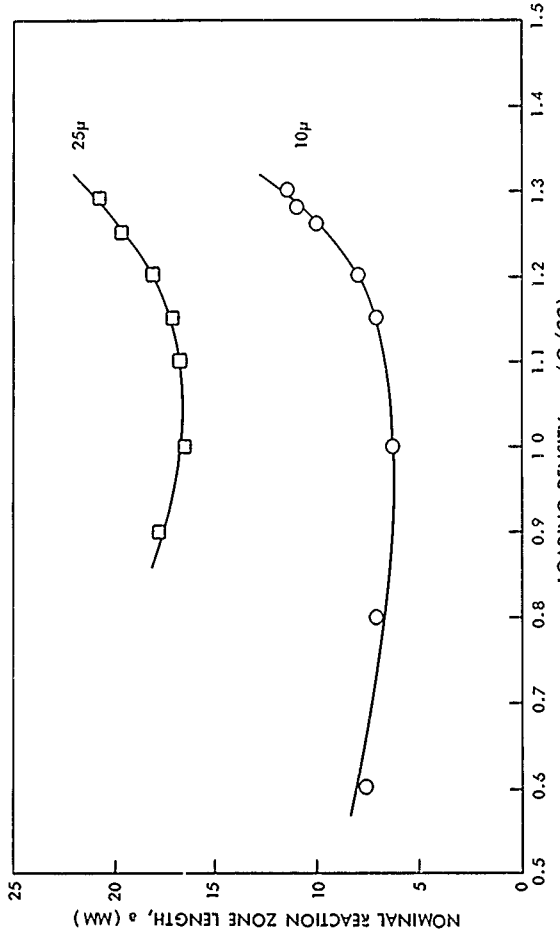

FIG. 5 NOMINAL REACTION ZONE LENGTHS FOR AP AS A FUNCTION OF DENSITY AND PARTICLE SIZE

TABLE 9

Ratio of Nominal Reaction Zone Lengths

Density ρ_o (g/cc)	Ratio* a_1/a_2
0.90	0.36
1.00	0.38
1.10	0.40
1.20	0.44
1.26	0.50

*a_1 nominal reaction zone of 10 μ AP
a_2 nominal reaction zone of 25 μ AP

which lateral rarefactions enter the reaction zone and quench enough of the reaction so that insufficient energy is released for self-propagation. Consequently d_c will be a function of the true reaction zone length, curvature of the detonation front, and sound velocity (velocity of the rarefaction wave behind the front).

Dremin[6] maintains that there is a linear relation between the reaction zone length and d_c. He has shown a straight line plot (4 points) between the experimental values for TNT. But, in general, such a simple relationship would not be expected; it certainly does not exist between the nominal reaction zone a and d_c, as Fig. 6 illustrates.

However, if despite the fact that Eyring's assumed independence of a and d fails near d_c, i.e., the D vs d^{-1} curves become non-linear as D approaches D_c, we assume that the equation

$$a = d\left(1 - \frac{D}{D_i}\right) \tag{11}$$

still holds, we can derive

$$a_c = d_c\left(1 - \frac{D_c}{D_i}\right) \tag{12}$$

In regions of slowly varying D_c/D_i (as ρ_o changes), a_c vs d_c will, of course, appear to vary linearly.

The Wood and Kirkwood[7] modification of the curved front theory gives

$$z = (S/3.5)\,(1 - D/D_i) \tag{13}$$

where z is reaction zone length and S is the radius of curvature of the reaction front. With the added assumption that at any failure limit,

FIG. 6 CRITICAL DIAMETER AS A FUNCTION OF NOMINAL REACTION ZONE LENGTH

$$S_c = s\, d_c$$

Eq. (13) becomes identical to Eq. (12) except for the constant (S/3.5)*. Both of these equations give

$$\frac{z_{c1}}{z_{c2}} = \frac{a_{c1}}{a_{c2}} = \frac{d_{c1}}{d_{c2}} \frac{D_{i2}}{D_{i1}} \frac{D_{i1} - D_{c1}}{D_{i2} - D_{c2}} = \tag{15}$$

$$f(D_{i1}, D_{i2}) \frac{d_{c1}}{d_{c2}}$$

Moreover, by definition

$$z_c = (D_c - \bar{u}_c)\, \tau_c \tag{16}$$

Hence Eq. (15) can be written

$$\frac{z_{c1}}{z_{c2}} = \frac{a_{c1}}{a_{c2}} = f \frac{d_{c1}}{d_{c2}} = \frac{\tau_{c1}}{\tau_{c2}}, \qquad D_{c1} = D_{c2} \tag{17}$$

Data chosen from the limit curves, D_c vs ρ_o, (e.g., dashed curve of Fig. 3) so that $D_{c1} = D_{c2}$ for the 10 μ and 25 μ AP, can be used in Eq. (17) to determine the ratio of the critical reaction times. These are reaction times at T_c in contrast to those at T_i considered earlier.

Table 10 contains data taken from the small common range of D_c on the two limit curves for 10 μ and 25 μ AP. The last column shows that the ratio for the reaction times at T_c is 0.37 ± 0.01, again a good check for the ratio of the average particle sizes.

The constant S is quite commonly taken equal to 0.5 at critical conditions.

TABLE 10

Ratio of Critical Diameters for Constant D_c

D_c mm/μsec	ρ_{o_1} (g/cc)	d_{c_1} (mm)	ρ_{o_2} (g/cc)	d_{c_2} (mm)	f	d_{c_1}/d_{c_2}	τ_{c_1}/τ_{c_2} *
2.0	0.86	(20.5)	1.15	40.2	0.71	0.51	0.36
2.2	0.96	(21.7)	1.24	43.5	0.74	0.50	0.37
2.4	1.05	23.0	(1.32)	47.2	0.76	0.49	0.37

Subscripts: 1. 10 μ AP
2. 25 μ AP

*From Eq (14)

The curved front theory, therefore, leads to relative reaction zone length values that give relative reaction times consistent with the grain burning theory and an effective particle size of the APs represented by the mean values used here. The distinction between a and z in the two versions of the theory has been shown because they differ in absolute value; at the critical conditions ($s \cong 0.5$), $a_c = 7\ z_c$.

Shock Sensitivity

All shock sensitivity measurements were carried out with the standard gap test procedures reported in Ref. 8. Among these is the use of a steel witness plate in the regular test and use of an explosive witness in the extended test. An explosive witness is used when the detonation reaction is insufficiently powerful to punch the steel plate. Cast Comp B is most frequently used as witness in the extended test but any explosive can be used. Table 11 shows the sensitivity values found for three typical explosive witnesses. They were measured by both the regular and extended test; in the latter the very sensitive explosive, cast pentolite, was used as the witness to emphasize any difference that might be found between endpoints with steel and explosive witnesses.

Within experimental error, the measured 50% pressure P_g is the same whether a steel plate or a very sensitive explosive is used as a witness. This is true despite the fact that detonation must be established nearer the shocked end of the acceptor cylinder to punch the steel plate than to initiate cast pentolite because a longer impulsive loading is required to punch the plate. However, although the measured P_g values are the same within experimental error for the two tests, the 50% gaps vary from 3 to 13 cards for the most and least sensitive material, respectively. This same trend and these small differences will be found in some of the later tables.

TABLE 11

Shock Sensitivity of Common H. E. Witnesses

Comparison of Regular and Extended Test Results

Cast Explosive	Density g/cc	Regular Test 50% Values		Extended Test* 50% Values	
		gap (No. cards)	P_g (kbar)	gap (No. cards)	P_g (kbar)
TNT	1.61	108	46	121	42
Comp B	1.70	207	16	212	16
Pentolite 50/50	1.67	284	9	287	9

*Explosive witness used was cast pentolite.

a. Explosive but Non-detonating Reactions. Although common organic explosives can be tested in either the extended or the regular gap test to obtain the same well defined result, this is not the case for granular AP. As the detonation and detonability patterns have already shown, AP undergoes a number of powerful explosive reactions which are not detonations but which persist as fading reactions for many diameters down the charge. Just as this situation mades selection of the failure limits difficult, it also complicates measuring the shock sensitivity. A powerful but fading reaction can damage the witness, but the measured gap will be irrelevant to a shock sensitivity to detonation. Consequently gap testing of AP must be accompanied by a number of supplementary tests.

As a result of a few tests made some years ago, we had concluded that the detonation reaction of granular AP is so weak that the extended rather than the regular gap test must be used for this material. The present work has shown that the earlier conclusion is wrong, and that exploding but non-detonating AP can frequently initiate an explosive witness. Consequently all AP charges are now first fired at zero gap in the regular test; if the plate is punched, the 50% point is determined by the regular procedure. Otherwise the extended test is used.

When it is suspected that damage to the witness is caused by a powerful but fading reaction, the situation can be checked by instrumentation of the test (probes) or by increasing the length of the acceptor charge so that the reaction fades before it reaches the witness. In this work, fading reactions were suspected from the known pattern of detonation behavior, and the first check used was doubling the acceptor length. If the longer acceptor still produced a positive result, probes were used to follow the reaction initiated at zero gap.

An example of gap test results obtained without detonation is seen in the coarse AP (200 μ). This material reacted violently but did not detonate at its pour density (where it would be most shock sensitive) in the 7.62 cm unconfined charge. It was therefore suspected that the confinement of the gap test (core diam 3.65 cm) was inadequate for detonation despite the fact that positive results were obtained in the extended gap test. This turned out to be the case for both coarse AP and a similarly coarse ammonium nitrate (AN) as the data of Table 12 show. It is interesting that the fading reaction in AP at 1.29 g/cc was still sufficiently strong, after a travel of 8.6 core diameters, to initiate the Comp B witness.

b. Shock Sensitivity as a Function of Porosity. AP lots N-125 and N-126 were ordered from two different sources; the average particle sizes requested were 10 μ and 45 μ respectively. While the gap tests of Table 13 were carried out, the additional investigation of particle size, described in the first section of this report, was also made. Unfortunately, the two lots were found to be practically indistinguishable and of an average particle size of 25 μ. In agreement with this result, their measured 50% gap pressures fall on the same P_g vs % TMD curve, as shown in Fig. 7. The only information about the effect of particle size on P_g is that given by a single test previously carried out on AP, XP-11, (11 μ), and that result is so close to the curve that the difference may not be significant. However, the difference is in the direction generally reported for granular explosives: the coarser material is more shock sensitive. The particle size effect will be further investigated when another sample of finely ground AP can be obtained.

The data of Table 13 indicate that the regular test value rather than the extended is preferable in gap testing AP. They also show, plotted in Fig. 7, a typical shock sensitivity curve for a material approaching its critical density. The lowest density for failure is shown as the No-Go line at 83.5% TMD. (The critical

TABLE 12

Extended Gap Test Results from Non-Detonating Acceptor

Material	ρ_o g/cc	%TMD	Extended Test* 50% point No. cards	Acceptor 30.5 cm long, zero gap ρ_o	%TMD	Effect on witness	
AP, N-119 (200μ)	1.60	81.8	17	1.57	80.5	No-go	AP recovered
	1.58	80.9	74	1.43	73.2	No-go	
	1.43	73.2	98	1.29	66.1	Go	Probes at 5.08 cm intervals registered successive velocities of 4.2, 2.5, 2.3, 2.0, and 1.9 mm/μsec.
	1.29	66.0	153				
AN, N-124 (200μ)	1.69	97.9	x	0.76	44.0	No-go	No unburned material left and Comp B witness tube showed pressure rupture.
	1.30	75.4	x				
	1.20	69.7	~1				
	1.13	65.5	~31				
	0.86	49.6	53				

* Witness of cast Comp B
x Negative result

TABLE 13

Shock Sensitivity Data for APs

AP	ρ_o g/cc	%TMD	50% Gap No. cards Regular	50% Gap No. cards Extended*	50% Pressure P_g (kbar)
XP-11 (11u)	1.25	64.1	150<N<176	178	23.5[d]
N-126 (25u)	1.65	84.4	x	33[a]	x
	1.58	81.1	x	98	50.0[e]
	1.52	77.7	131	—	38.0
	1.25	64.1	173	—	22.0
N-125 (25u)	1.90	97.3	—	x	x
	1.76	90.0	—	x	x
	1.66	85.5	x[b]	32[c]	x
	1.60	81.9	—	98	—
	1.58	81.1	93	—	52.0
	1.47	75.5	143	143	33.7
	1.25	64.1	178	—	22.0
	1.11	56.9	198	209	17.8

* Cast Comp B witness. x Failure or no-go.

a. No detonation. Double length acceptor of ρ_o = 1.63 g/cc (83.5% TMD) failed to initiate cast Comp B witness.
b. Original result: Go at zero cards, no-go at one card. Failed with double-length acceptor.
c. Failed with double length acceptor.
d. Extended test value corrected to regular test value with data of Table 11.
e. Pressure from extended test; all other values from regular test.

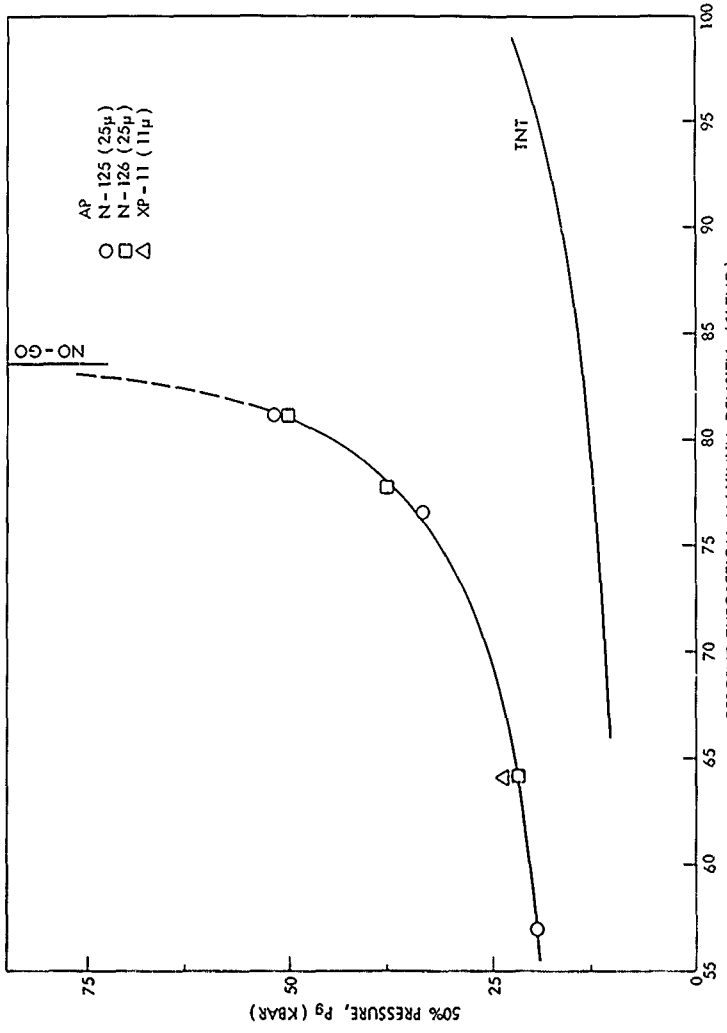

FIG. 7 SHOCK SENSITIVITY VS POROSITY FOR THREE LOTS OF AP

value lies somewhere between this and 81.9% TMD where detonation occurred.) As the % TMD approaches the critical value, the gradient of the P_g vs % TMD curve increases very rapidly. In earlier work, this steep portion of the AP curve was compared to the TNT curve typical of organic explosives, and it was remarked that the AP curve was characterized by a much higher gradient than that of the H.E. Fig. 7 contains the TNT curve for another such comparison. Here it is evident that at lower % TMD, say 70% or less, the gradient of the AP curve is almost identical to that of the TNT. It is only with the approach to the critical % TMD (not present in TNT) that the two differ appreciably.

SUMMARY AND CONCLUSIONS

1. Study of the lots of AP used in the present work showed that a reasonable assignment for the average particle size was 10, 11, 25 (two lots), and 200 μ.

2. Detonability curves (d vs ρ_o) and the pattern of D vs ρ_o curves showed that increasing the AP particle size from 10 to 25 μ:

a. Shifted the limit curve to higher d_c and lower ρ_c.

b. Lowered the D vs ρ_o curve for any given finite diameter.

c. Did not affect the infinite diameter values (D_1 vs ρ_o).

d. Demonstrated the existence of a limit curve, particle size vs ρ_o at fixed diameter.

3. The shifts described above are so large that work at NOL should be done on AP with an average particle size of 10 μ or less.

4. The curved front theory of diameter effect gave a ratio of reaction times at the ideal and critical conditions equal to the

ratio of the average particle sizes. This is consistent with the grain burning theory of reaction.

5. Linear extrapolation to zero particle size of the 7.62 cm detonation velocities gave the D_i values.

6. Insufficient data were obtained to show the particle size effect on shock sensitivity, but it appears to be either no change or a decreased sensitivity with decreased particle size.

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APPENDIX

SUPPLEMENTARY DATA

Table A1 contains experimental details of the conditions for each photographic record: flasher, film, charge length viewed and writing speed of the camera.

Figures A-1 through A-5 are typical of the Micromerographs which supplied the data of Table 1 in the text.

TABLE A1

EXPERIMENTAL CONDITIONS FOR THE SHOTS

Shot No.	Flasher a	Film b	Length Observed in.	Writing Speed W mm/μsec
185			7.5	1
186			7.5	2
187			7.5	2
190			7.5	2
196			7.5	1
191			7.5	1
195			7.5	1
197			2.5	2
198			2.5	2
192			7.5	1
199			2.5	2
200			2.5	2
193			7.5	1
202			2.5	2
203			2.5	2
204			2.5	2
205	M		2.5	2
169		P	7.5	1
194			7.5	1
179			2.5	3
178			2.5	3
158			7.5	1
156			7.5	1
164			7.5	1
155			7.5	1
165			7.5	1
172		P	7.5	1
181	N		2.5	3
180	N		2.5	3
183	N		2.5	3
182	N		2.5	3
171	M	P	7.5	1
201	M		7.5	1
189	N		7.5	1
188	N		7.5	1
206			2.5	2
207			2.5	2
218	M		2.5	2
227	M		2.5	2
217	M		7.5	1
226	M		6.0	2
239			6.0	2
128	M over A		2.5	3
130	M over A		7.5	1
135			7.5	1
129	M over A		7.5	1
223	M		2.5	3.0
228	M		2.5	3.0
221	M		6.0	2.0

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TABLE A1 (Contd.)

Shot No.	Flasher a	Film b	Length Observed in.	Writing Speed W mm/μsec	Shot No.	Flasher a	Film b	Length Observed in.	Writing Speed W mm/μsec
160			7.5	1					
167			7.5	1					
168			7.5	1					
209			7.5	1					
210			7.5	1					
208			2.5	2					
161			7.5	1					
212	S		7.5	2					
121			2.5	3					
122	N		2.5	2					
123	S		2.5	2					
125	M		7.5	1					
233			2.5	3					
232			2.5	3					
231	M		2.5	3					
224	M		2.5	3					
230	M		2.5	3					
229	M		2.5	3					

a. Cellulose acetate used unless other specified. Symbols are: A cellulose acetate; M Magic Tape; N None; S Scotch Tape.

b. T Tri-X film used unless P (Panatomic-X) specified.

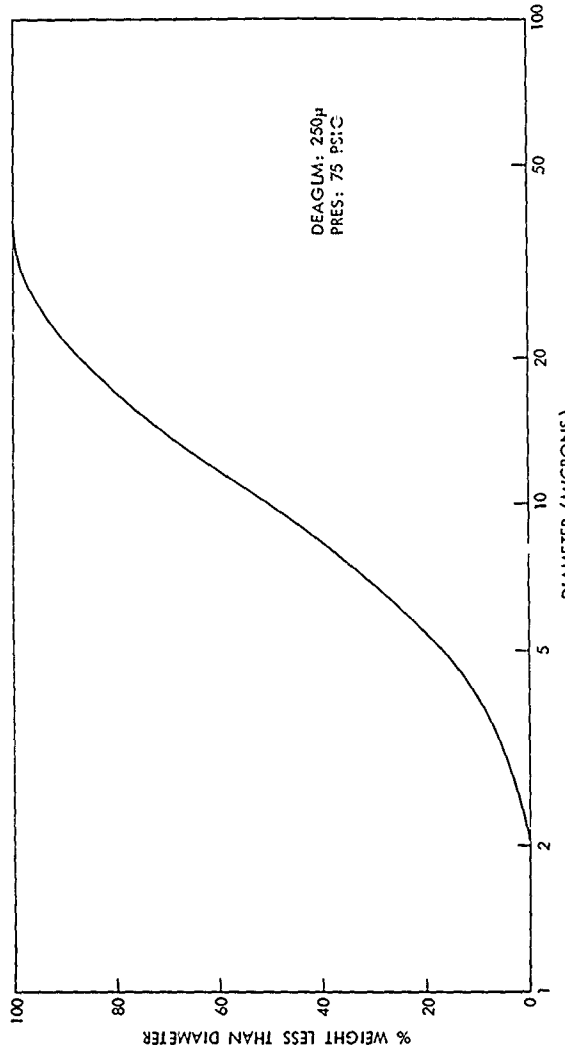

FIG. A-1 MICROMEROGRAPH OF AP (XP-17)

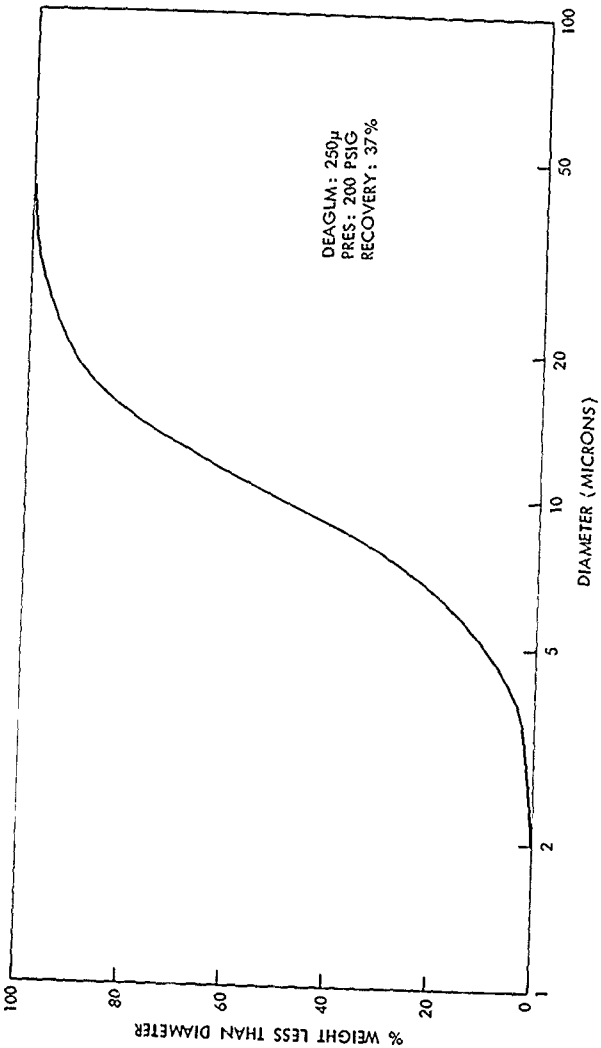

FIG. A-2 MICROMEROGRAPH OF AP (XP-17)

FIG. A-3 MICROMEROGRAPH OF AP (XP-11)

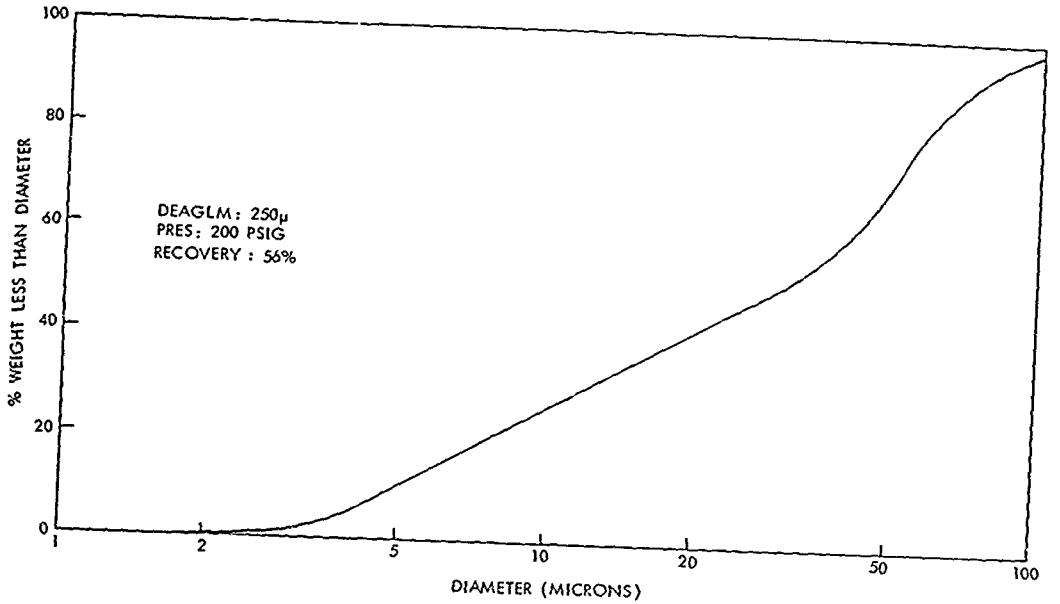

FIG. A-4 MICROMEROGRAPH OF AP (N-125)

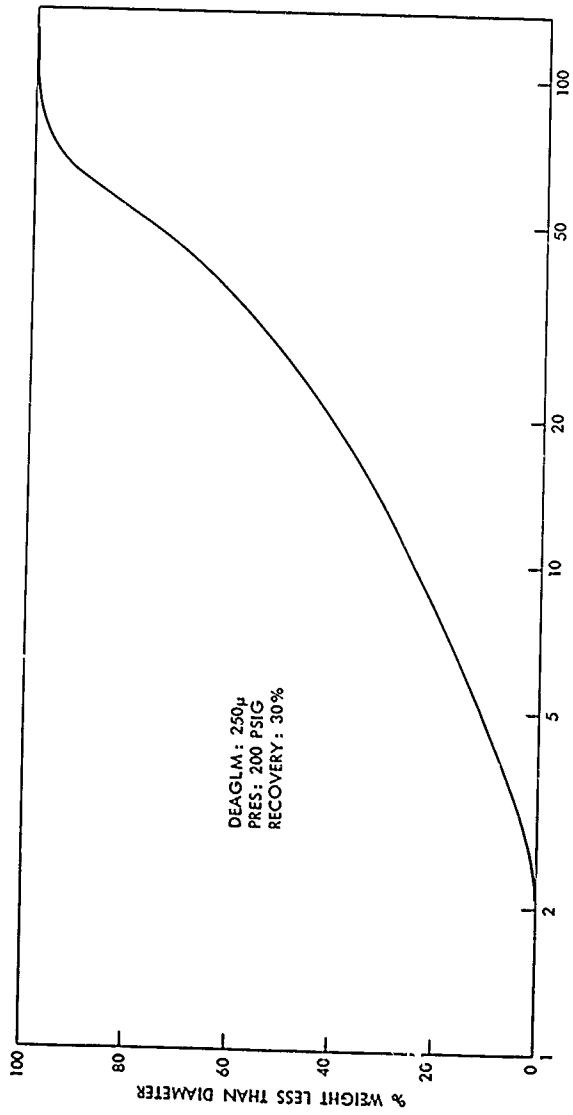

FIG. A-5 MICROMEROGRAPH OF AP (N-126)

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4 DESCRIPTIVE NOTES *(Type of report and inclusive dates)*

5 AUTHOR(S) *(Last name, first name, initial)*

Donna Price, A. R. Clairmont, Jr., and I. Jaffe

6 REPORT DATE	7a TOTAL NO OF PAGES	7b NO OF REFS
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13 ABSTRACT

The detonation velocity and critical density of ammonium perchlorate (AP) increased, whereas the critical diameter decreased, with decreasing particle size. Relative reaction times varied as the particle diameter. Ideal detonation velocities were unaffected.

The regular gap test is recommended for AP and the gradient of its P_g vs ρ_o curve at higher porosities is shown to be like that of the TNT curve.

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14 KEY WORDS	LINK A		LINK B		LINK C	
	ROLE	WT	ROLE	WT	ROLE	WT
Ammonium perchlorate Detonation Velocity Detonability Shock Sensitivity Critical diameter Critical density Effect of particle size						

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