CONF-850742--1

LA-UR--85-1023

DE85 009620

TITLE: THE EFFECT OF PRECIPITATION ON THE SPECIFIC SURFACE AND EXPLOSIVE CHARACTERISTICS OF PETN

AUTHOR(S): Robert H. Dinegar

MASTER

SUBMITTED TO: The International Pyrotechnics Society Symposium
Karlsruhe, Germany
July 3-5, 1985

Los Alamos

Los Alamos National Laboratory
Los Alamos, New Mexico 87545

THE EFFECT OF PRECIPITATION VARIABLES ON THE SPECIFIC SURFACE AND EXPLOSION CHARACTERISTICS OF PENTAERYTHRITOL TETRANITRATE

by

Robert H. Dinegar
Los Alamos National Laboratory
Los Alamos, New Mexico

I. INTRODUCTION

The particle size, shape, and texture of the crystals of pentaerythritol tetranitrate (PETN) precipitated from acetone solutions by the addition of water depend upon many crystallization factors. Among the most important of these are 1) the concentration of PETN in the acetone, 2) both the volume of water and the rate at which the nonsolvent is added, and 3) the amount of agitation of the solution during the precipitation process. Fortunately, all of these conditions can be controlled easily because the explosion characteristics of PETN are strong functions of the crystal description.

This paper quantitatively describes the effect of precipitation conditions on the type of PETN crystals obtained and the variation of both explosive sensitivity and explosion time with one important quantitative measure of crystal description: the area per unit mass or specific surface.

II. EXPERIMENTAL PROCEDURE

PETN was precipitated from acetone by the addition of water, with stirring, at ambient temperature (~20°C). The range of PETN concentration was from 12.5 to 100 g/l and one liter of solution was used in each experiment. The volume of the water that was added varied between 0.5 and 2.0 liters. The rate at which this precipitant was added varied from 8.2 to 100 cm^3/s.

Solutions were agitated with an aluminum stirrer, segment-shaped, of 3.0-in. radius and 2.5-$in.^2$ area. Precipitates were filtered by suction through medium-porosity (10-15 μm nominal maximum pore size) fritted glass filter, washed with distilled water, and air-dried for about 30 hours.

The specific surface (S_o) of each sample was measured by permeametric means. The density of the sample at which the surface was determined and that used in the pressings fired to measure the explosive sensitivity and explosion time were approximately the same, viz. 0.95 g/cm^3.

The explosive sensitivity was measured in the following fashion. Small amounts (approximately 0.3 g) of the explosives were compacted into brass cylinders, 0.75 mm in diameter and 6.2 mm in length, at a porosity of about 0.5. Each compact was in intimate contact with a gold bridgewire, 0.033 mm diameter x 1.0 mm long, soldered to brass lead-in wires. The assembly was connected by a firing cable of about 1-μH inductance to a 1-μF capacitor-discharge unit whose charging voltage could be varied. Upon the closing of a mechanical switch, the stored energy was delivered to the system. The bridgewire is heated extremely rapidly and bursts in less than 1 μs. The explosive next to the bridgewire is initiated to detonation by the deposition of this heat and shock energy. The voltage on the capacitor, which governs the amount of energy delivered to the wire, is used as a measure of the explosive sample's sensitivity - lower voltage at initiation signifies a more sensitive explosive sample. This voltage is called the initiation voltage and is designated $E_{0.5}$. The subscript means that the voltage value is that required for initiation of aliquots of the sample 50% of the time.

The explosion time of the initiated sample is designated as t_E. It is the time from the instant of firing-switch closure until the detonation wave appears at the end of the cylindrical pressing. This time includes 1) the bridgewire bursting time, 2) the explosive burning time as the reaction builds up to a self-sustaining detonation, and 3) the detonation time. The bridgewire bursting time is constant as each sample of explosive is initiated under the same electrical conditions. The burning or build-up time depends strongly on the specific surface. Previously reported experiments failed to detect any variation in explosive detonation velocity with specific surface over the range of particle size of our samples.[1] This report uses the variation in the experimentally determined explosion time as the measure of the effect of sample specific surface. The standard deviation associated with t_E is designated σ_E.

Twenty aliquots of each PETN sample were fired to determine an $E_{0.5}$ value. Ten PETN pressings were initiated for each t_E determination. Standard deviation values were about ±50 V in $E_{0.5}$ and 0.02 μs in t_E.

III. EXPERIMENTAL RESULTS

The specific surface of the sample increased when the water addition rate was increased. The sensitivity of the sample generally appeared to decrease as the specific surface became larger. The explosion time of the sample, however, decreased as the specific surface increased.

The magnitude of the effect of varying the total amount of water used in the precipitation process depended upon the rate of water addition. Qualitatively, all the changes were in the same direction but appeared to be greater at faster water-addition rates. A four-fold decrease in the water volume was accompanied by 1) a small decrease in specific surface, 2) an essentially unchanged bursting bridgewire initiation sensitivity, and 3) a longer explosion time.

The effect of decreasing the concentration of PETN in the acetone solution eight-fold was quite noticeable. The specific surface of the sample rose by over a factor of 2, the sensitivity dropped by 50%, and the explosion times decreased markedly.

Changing the stirrer speed by a factor of 5 produced a small change in specific surface, no change outside the precision of the measurement in sensitivity, and only a small percentage change in explosion time.

Because of the relatively large number of variables, the data are shown in both tabular form (Tables I through VIII) and graphically in Figs. 1 and 2.

The directions of the changes are quite understandable. In the precipitation process, a change in those variables that leads to large PETN nucleation rates and smaller coagulation rates produces more finely divided samples. These changes in variables are faster water addition and less concentrated solutions. The decrease in exploding bridgewire sensitivity with increase in specific surface is similar to the effect previously observed in the shock initiation of explosives in small-scale gap-test experimentation.[1,2] These results indicate that some mechanisms other than the purely surface burning reaction known to be present in ignition of propellants must be operative in the ignition of secondary explosives. The explosion-time variation with specific surface - on the other hand - indicates clearly that once the PETN is ignited a surface burning reaction governs the buildup to detonation.

Table I

SPECIFIC SURFACE (S_o), BRIDGEWIRE SENSITIVITY ($E_{0.5}$)
AND EXPLOSION TIME (t_E) AS A FUNCTION OF
THE RATE OF H_2O ADDITION IN THE PRECIPITATION PROCESS

Precipitation Conditions

PETN concentration: 75 g/l acetone
Volume H_2O added: 1.9 liters
Segment-type stirrer speed: 500 rpm

Rate of H_2O addition (cm^3/s)	t_E (μs)	σ_E (μs)	$E_{0.5}$ (V)	S_o (cm^2/g)
50.0	2.51	0.01	700	5500
16.7	2.81	0.01	600	3250
11.1	2.93	0.01	600	2900
8.2	3.25	0.03	550	2000

Table II

SPECIFIC SURFACE (S_o), BRIDGEWIRE SENSITIVITY ($E_{0.5}$)
AND EXPLOSION TIME (t_E) AS A FUNCTION OF
THE VOLUME OF H_2O ADDED IN THE PRECIPITATION PROCESS

Precipitation Conditions

PETN concentration: 75 g/l acetone
Rate of H_2O addition: 8.2 cm^3/s
Segment-type stirrer speed: 500 rpm

Volume H_2O added (cm^3)	t_E (μs)	σ_E (μs)	$E_{0.5}$ (V)	S_o (cm^2/g)
2000	2.84	0.01	650	3100
1500	3.09	0.02	650	2450
1000	3.07	0.01	650	2500
750	3.15	0.02	650	2400
500	3.02	0.02	650	2700

Table III

SPECIFIC SURFACE (S_o), BRIDGEWIRE SENSITIVITY ($E_{0.5}$) AND EXPLOSION TIME (t_E) AS A FUNCTION OF THE VOLUME OF H_2O ADDED IN THE PRECIPITATION PROCESS

Precipitation Conditions

PETN concentration: 75 g/l acetone
Rate of H_2O addition: 28.3 $cm^3/2$
Segment-type stirrer speed: 500 rpm

Volume H_2O added (cm^3)	t_E (μs)	σ_E (μs)	$E_{0.5}$ (V)	S_o (cm^2/g)
2000	2.49	0.01	750	5500
1500	2.49	0.01	700	5550
1000	2.48	0.01	700	5600
750	2.52	0.01	700	5300
500	2.58	0.01	700	4900

Table IV

SPECIFIC SURFACE (S_o), BRIDGEWIRE SENSITIVITY ($E_{0.5}$) AND EXPLOSION TIME (t_E) AS A FUNCTION OF THE VOLUME OF H_2O ADDED IN THE PRECIPITATION PROCESS

Precipitation Conditions

PETN concentration: 75 g/l acetone
Rate of H_2O addition: 50.0 cm^3/s
Segment-type stirrer speed: 500 rpm

Volume H_2O added (cm^3)	t_E (μs)	σ_E (μs)	$E_{0.5}$ (V)	S_o (cm^2/g)
2000	2.49	0.02	750	6000
1500	2.53	0.02	700	5900
750	2.52	0.02	700	5400
500	2.64	0.02	650	4150

Table V

SPECIFIC SURFACE (S_o), BRIDGEWIRE SENSITIVITY ($E_{0.5}$)
AND EXPLOSION TIME (t_E) AS A FUNCTION OF
THE CONCENTRATION OF PETN IN THE PRECIPITATION PROCESS

Precipitation Conditions

Volume of H_2O added: 1.9 liter
Rate of H_2O addition: 28.3 cm^3/s
Segment-type stirrer speed: 500 rpm

PETN concentration (g/l acetone)	t_E (μs)	σ_E (μs)	$E_{0.5}$ (V)	S_o (cm^2/g)
100.0	2.80	0.02	650	3550
75.0	2.60	0.01	750	4750
50.0	2.46	0.01	800	6500
25.0	2.43	0.01	800	6950
12.5	2.35	0.02	950	9200

Table VI

SPECIFIC SURFACE (S_o), BRIDGEWIRE SENSITIVITY ($E_{0.5}$)
AND EXPLOSION TIME (t_E) AS A FUNCTION OF
THE CONCENTRATION OF PETN IN THE PRECIPITATION PROCESS

Precipitation Conditions

Volume of H_2O added: 2.0 liters
Rate of H_2O addition: 50.0 cm^3/s
Segment-type stirrer speed: 500 rpm

PETN concentration (g/l acetone)	t_E (μs)	σ_E (μs)	$E_{0.5}$ (V)	S_o (cm^2/g)
100	2.56	0.01	700	5150
75	2.48	0.01	750	6050
50	2.45	0.01	800	6900
25	2.36	0.01	850	9200

Table VII

SPECIFIC SURFACE (S_o), BRIDGEWIRE SENSITIVITY ($E_{0.5}$)
AND EXPLOSION TIME (t_E) AS A FUNCTION OF
THE CONCENTRATION OF PETN IN THE PRECIPITATION PROCESS

Precipitation Conditions

Volume of H_2O added: 1.9 liters
Rate of H_2O addition: 100 cm^3/s
Segment-type stirrer speed: 500 rpm

PETN concentration (g/l acetone)	t_E (μs)	σ_E (μs)	$E_{0.5}$ (V)	S_o (cm^2/g)
100.0	2.59	0.02	700	5650
75.0	2.57	0.01	700	6150
50.0	2.58	0.01	700	6350
25.0	2.44	0.01	800	8700
12.5	2.33	0.02	1000	11900

Table VIII

THE SPECIFIC SURFACE (S_o), BRIDGEWIRE SENSITIVITY ($E_{0.5}$)
AND EXPLOSION TIME (t_E) AS A FUNCTION OF
THE STIRRING SPEED IN THE PRECIPITATION PROCESS

Precipitation Conditions

PETN concentration: 75 g/l acetone
Volume of water addition: 2.0 liters
Rate of water addition: 35 cm^3/s
Segment-type stirrer

Stirrer Speed (rpm)	t_E (μs)	σ_E (μs)	$E_{0.5}$ (V)	S_o (cm^2/g)
1000	2.63	0.02	650	4400
750	2.64	0.02	700	4600
500	2.64	0.02	700	4800
300	2.60	0.01	700	5400
200	2.58	0.03	700	5150

REFERENCES

1. R. H. Dinegar, R. H. Rochester, and M. S. Milligan, "The Effect of Specific Surface on the Explosion Times of Shock-Initiated PETN," Explosivstoffe Nr. 3/1967, pp. 51-54.

2. R. H. Dinegar, R. H. Rochester, and M. S. Milligan, "The Effect of Specific Surface on the Shock Sensitivity of Pressed Granular PETN," Explosivstoffe Nr. 9/1963, pp. 1-2.

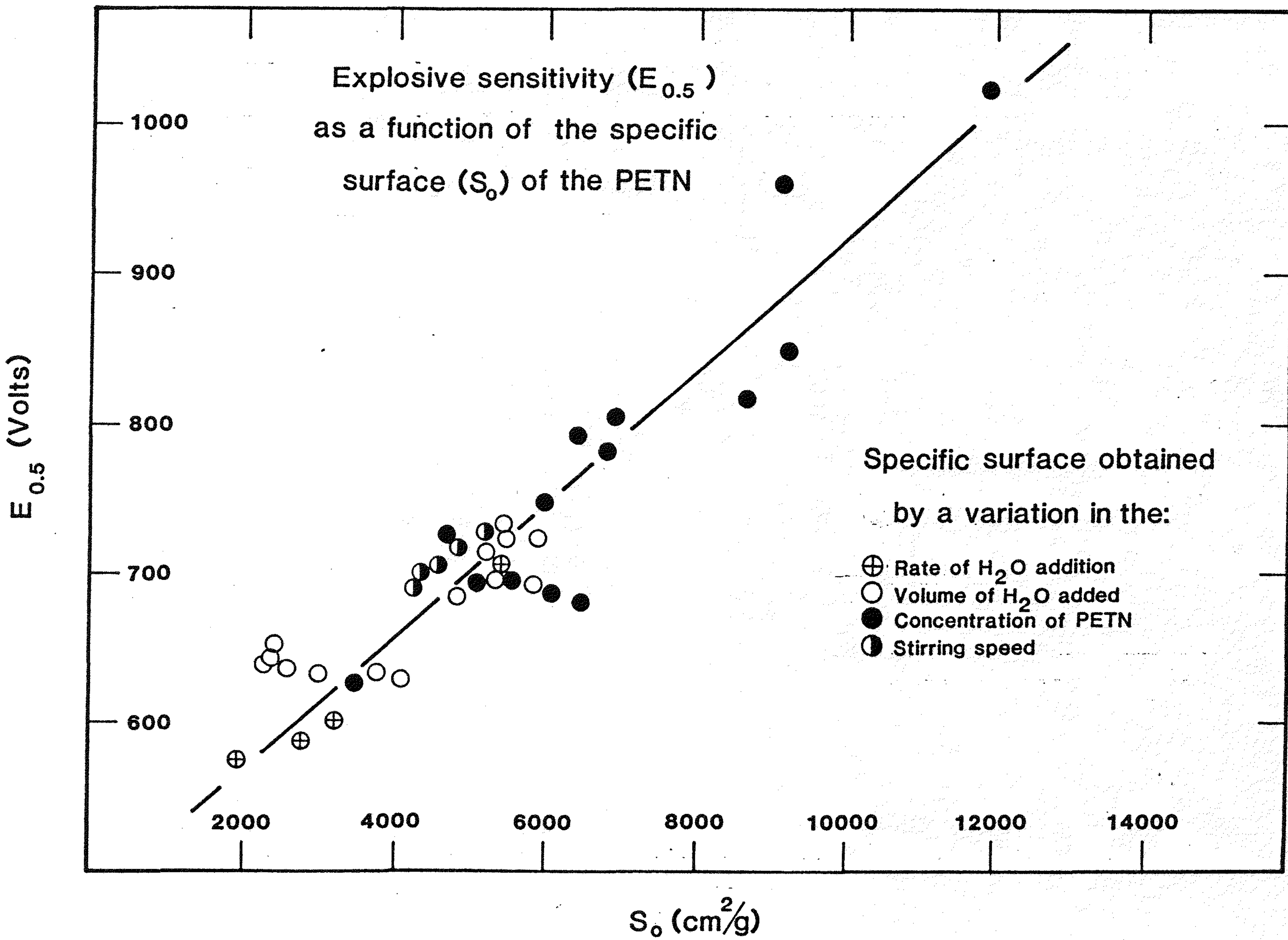
Explosive sensitivity ($E_{0.5}$)
as a function of the specific
surface (S_o) of the PETN
$E_{0.5}$ (Volts)
1000
900
800
700
600
2000
4000
6000
8000
10000
12000
14000
S_o (cm^2/g)
Specific surface obtained
by a variation in the:
Rate of H_2O addition
Volume of H_2O added
Concentration of PETN
Stirring speed

Fig. 1.

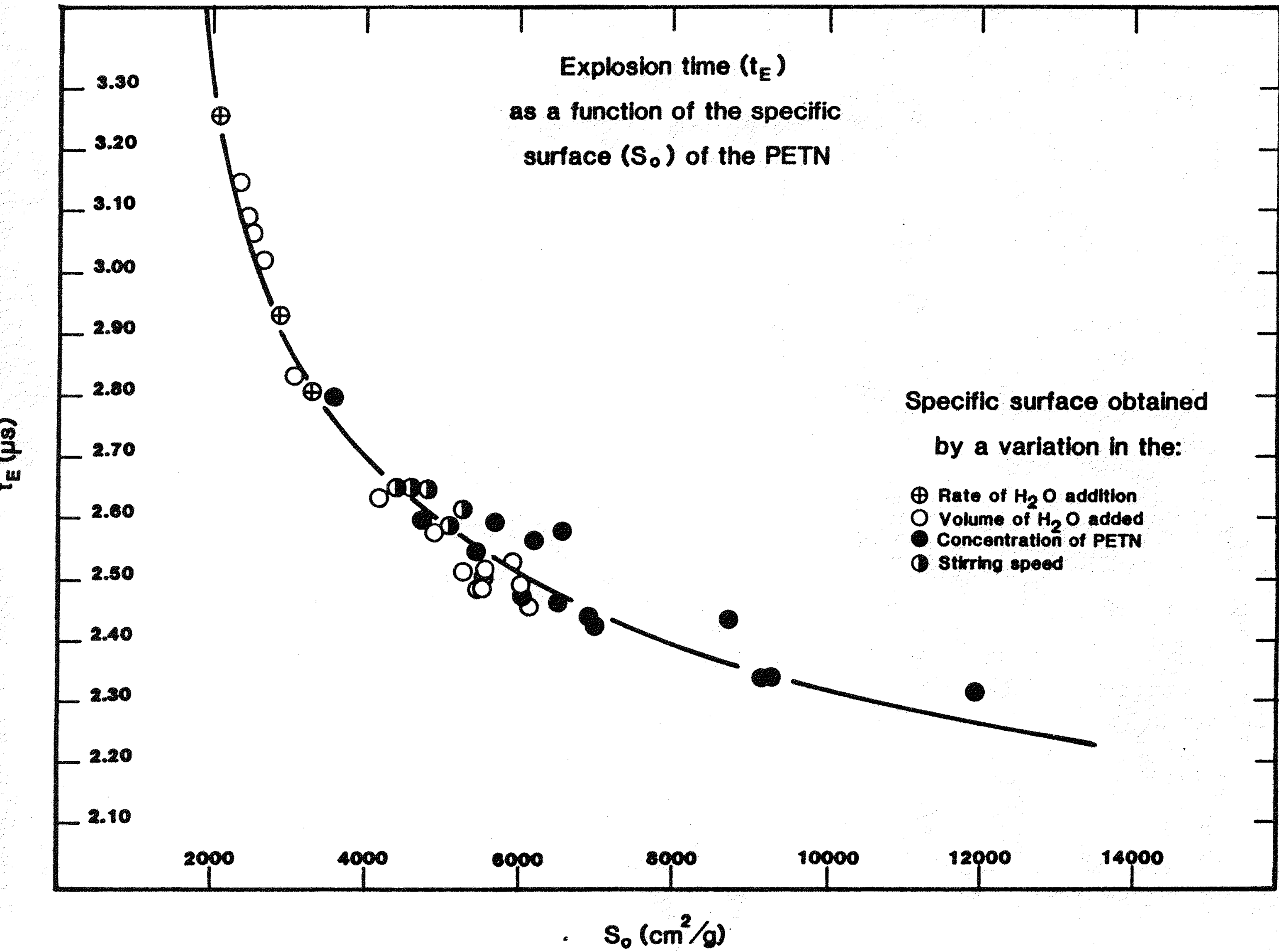
Explosion time (t_E)
as a function of the specific
surface (S_o) of the PETN
Specific surface obtained
by a variation in the:
⊕ Rate of H_2O addition
○ Volume of H_2O added
● Concentration of PETN
◑ Stirring speed
t_E (µs)
3.30
3.20
3.10
3.00
2.90
2.80
2.70
2.60
2.50
2.40
2.30
2.20
2.10
2000
4000
6000
8000
10000
12000
14000
S_o (cm^2/g)

Fig. 2.