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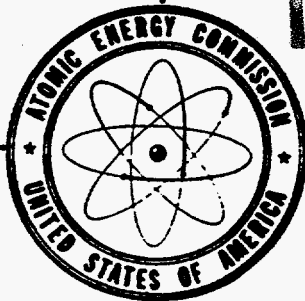
SANL 601-005 & 601-009

A. G. Osborn

April, May, June 1969

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## FORMULATION FACILITY

*A. G. Osborn*

April, May, June 1969  
SANL's 601-005 & 009

### ABSTRACT

Two 250-pound batches of LX-09 were made for BAACP for special studies.

Twenty Micronizer runs were made to ascertain the effects of various fluids and wetting agents upon the stability of high surface area PETN and to establish the limits within which repeatability can be expected. One sample, after being treated with the wetting agent dodecyl sodium sulfate, stabilized at 7400 cm<sup>2</sup>/gm after the 300-hour bake at 100°C.

A study was begun to determine the effects of deaeration upon the rheological and firing properties of Extex. Rheological data indicate that deaeration has little effect on extrudability. Firing data are currently being obtained.

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DISCUSSION

In conjunction with the Process Engineering Group, we have been making an attempt to determine the effect of deaeration upon Extex made at Burlington and at Pantex. It was hoped that differences in extrudability caused by deaeration would be picked up with the Rossi-Peakes or Burrell-Severs rheometers and that differences in firing would show up in a specially designed 8-inch diameter firing plate.

Deaeration was accomplished in the small rolling diaphragm slit plate deaerator designed at Pantex and formerly used by Production. Approximately 900 grams of HE can be deaerated each time by extruding it in thin sheets into an evacuated chamber.

Basically the deaerator consists of an 80 cubic inch chamber into which the HE is placed. The HE rests on a six-inch diameter plate containing several 0.020 inch wide slits. Beneath this plate is an 82 cubic inch vacuum chamber into which the HE is to be extruded. Vacuum is applied and when 200  $\mu$  is reached, hydraulic oil is pumped into the top rolling diaphragm piston arrangement which forces the Extex through the slits thus creating exposure of the increased surface of the thin sheets. Vacuum is continually applied for a period of 30 minutes to 1 hour until 150  $\mu$  is reached, indicating that virtually all of the air is removed. Then as the top diaphragm piston remains pressurized to hold the slit plate in place, the bottom diaphragm piston is raised to extrude the HE into three connecting 3 inch diameter x 1 inch thick plastic lined modules

spaced around the chamber. This then yields a convenient pre-packaged pattie of Extex ready for subsequent processing or testing. Approximately 4 pounds of the Pantex Extex, Lot No. 8142, and 2 pounds of the Burlington Extex, No. 8140, were deaerated.

To eliminate differences caused by mechanical extrusion, a sample of each lot, undeaerated, was placed in the deaerator into the lower chamber and simply extruded without deaeration into the modules for forming the patties. The deaerator was evacuated just to clear the bulk of air from the modules into which the Extex was to be loaded.

Rossi-Peakes viscometer<sup>1</sup> data are presented in Fig. 1 and the Burrell-Severs<sup>2</sup> extrusion rheometer data are given in Figs. 2 and 3. It may be seen there apparently are some differences between the deaerated and undeaerated Extex; however, the sensitivity of the tests is not adequate to establish any clear trends. The Burlington HE appears to be somewhat less viscous than the Pantex HE. For the Rossi-Peakes test, two samples of deaerated Pantex Extex and one deaerated BAACP sample were run. These were compared with the respective undeaerated HE. Each curve is an average of duplicate runs on each sample with many of the points coinciding.

<sup>1</sup>See LASL Specification 13Y-104-481

<sup>2</sup>The Burrell Severs rheometer consists of a reservoir 18 inches long, 1.5 inch in diameter with an opening at the bottom designated as a capillary orifice. Capillary length is 1.27 inch. The K<sub>2</sub> orifice with a 0.130-inch diameter capillary was used for this work. Approximately 50 to 75 gm of extrudable HE are placed over the orifice, then they are placed in the bottom of the reservoir with precautions being taken to prevent the HE from touching the side walls of the reservoir. The reservoir is then sealed and the selected nitrogen pressure applied. Data taken include weight, time required, and density of HE extruded. From this, viscosity is calculated.

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Fig. 1

Rossi-Peakes Extrudability Data

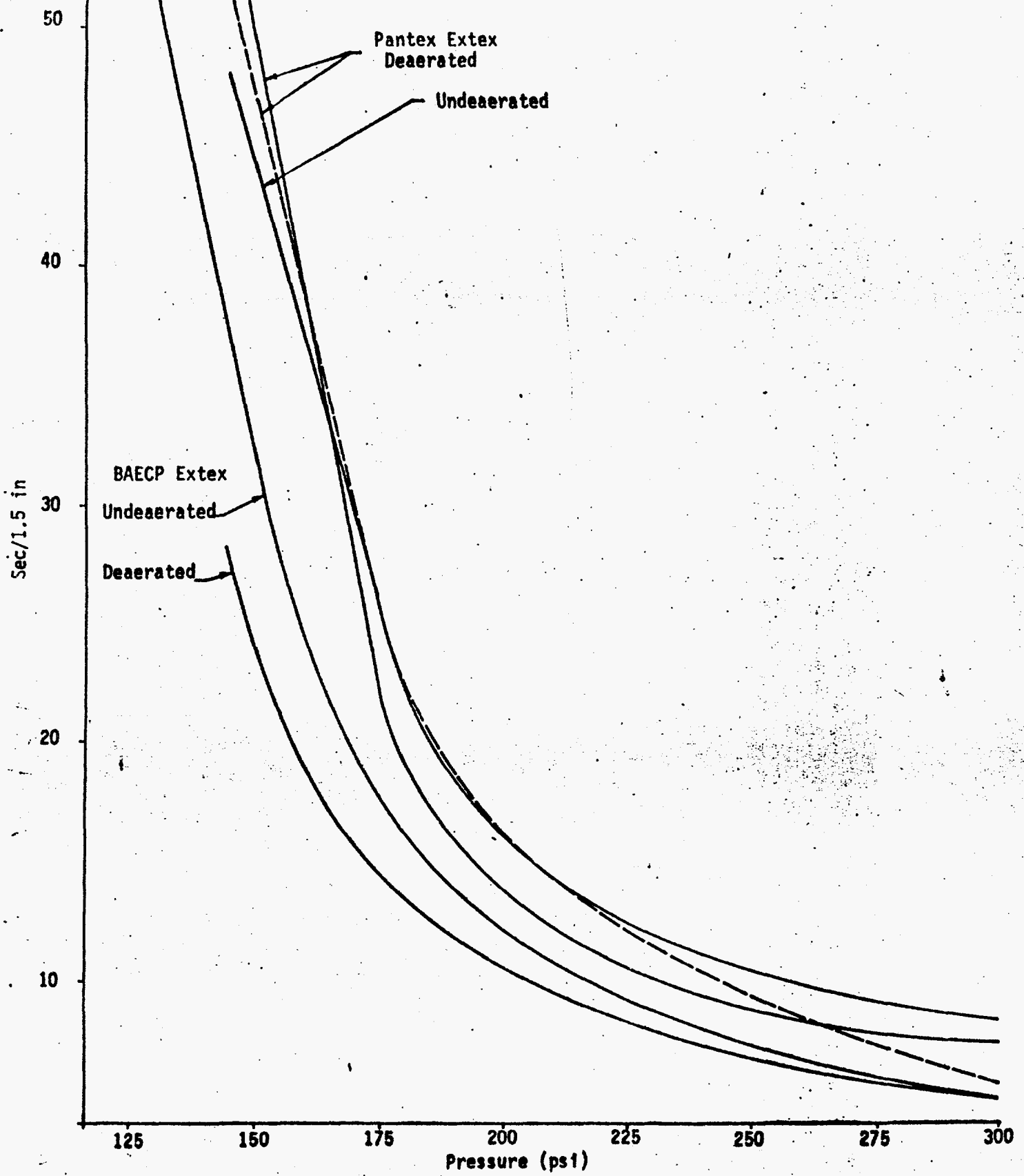


Fig. 7  
Burrell Severs Data

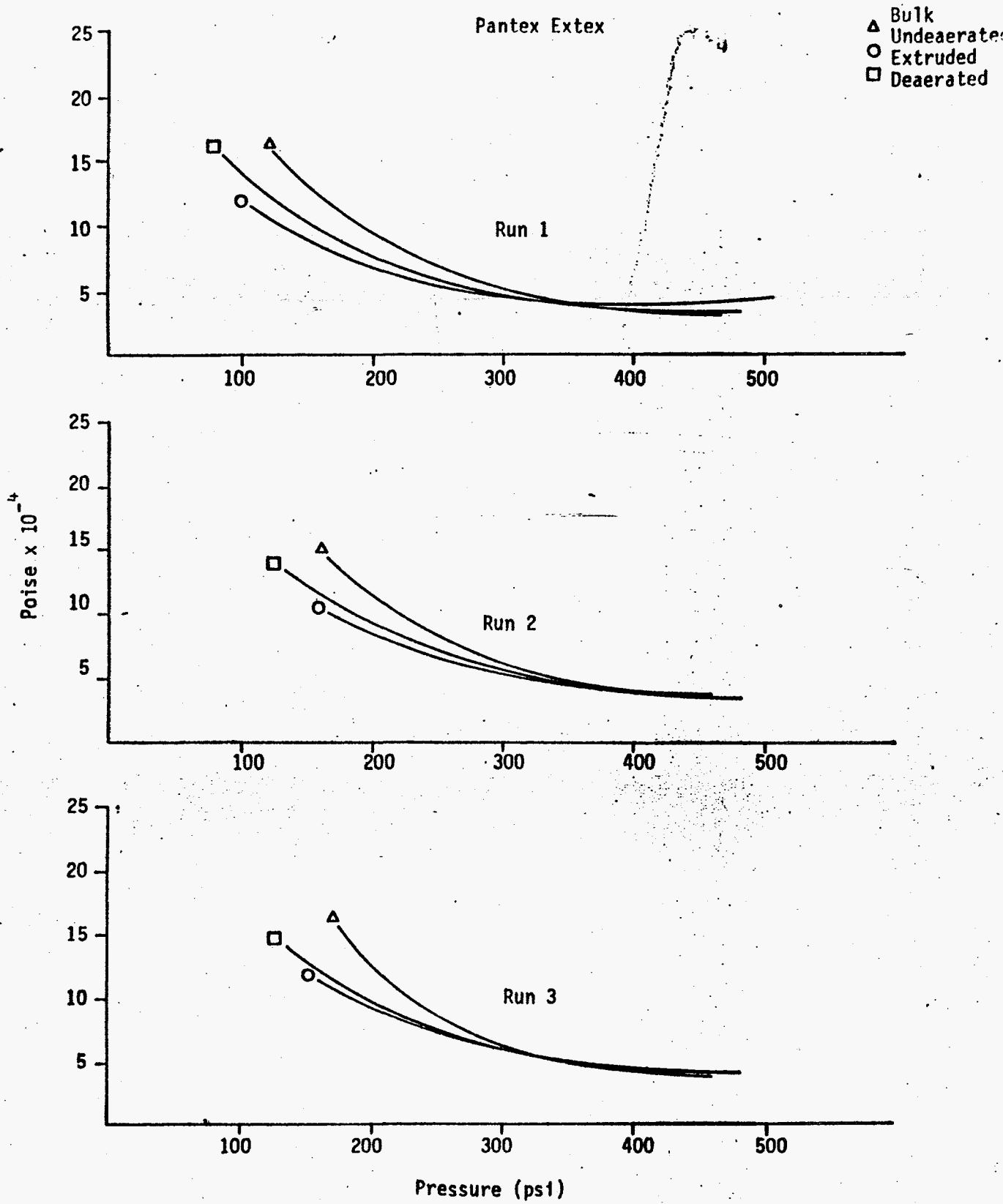
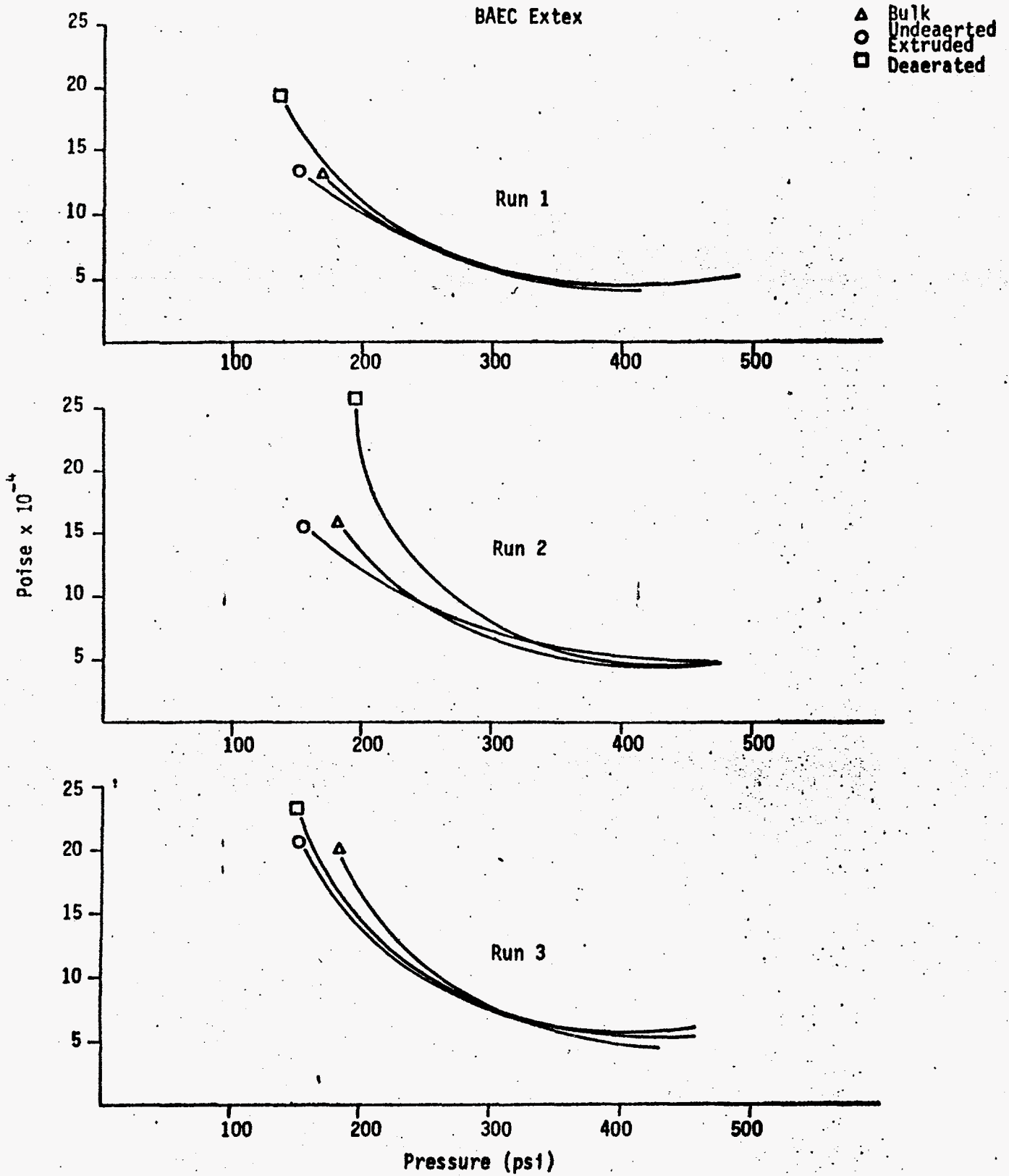


Fig. 3.  
Burrell Severs Data



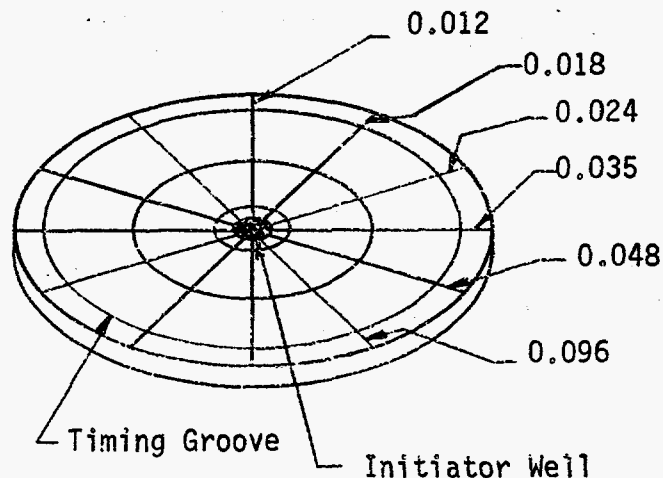


The Burrell-Severs extrusion rheometer data in Fig. 2 and 3 include three successive runs on each of the three samples from BAACP and Pantex. The samples consist of:

- (1) the bulk material after manufacture as it comes off of the roll mill.
- (2) undeaerated but extruded into patties in the deaerator.
- (3) deaerated and extruded into patties.

The Burrell Severs' curves seem to verify the Rossi-Peakes data showing little difference between any of the samples except that the Burlington material is slightly less viscous than the Pantex; however, there may be some difference at low pressures.

Firing is currently in progress. First preliminary results are rather inconclusive indicating that probably little difference will be seen in velocity at several diameters between the deaerated and undeaerated material. The firing plate is somewhat similar in design to the original LRL Sunburst test; but the various diameter grooves are of constant cross-section as they traverse across the plate. Groove sizes are 0.012, 0.018, 0.024, 0.035, 0.048, and 0.096 inch.



PETN

Experiments were begun to explore the capabilities of the Micronizer for reliably reproducing PETN of a given surface area. Also high surface area PETN was treated with two wetting agents, dodecyl sodium sulfate (DSS) and pentaerythritol tetraacetate (PTA), to ascertain their effect on stabilization of surface area,  $S_0^P$ , as judged by the  $S_0^D$  before and after exposure at elevated temperatures. A reasonably high stabilized surface area of 5950 cm<sup>2</sup>/gm was attained with PTA and an even higher surface area of 7400 cm<sup>2</sup>/gm was attained with DDS. The experiments and surface area data are summarized in Tables I and II with the results again presented in Figs. 4 and 5 for quick comparison. (Initial surface area is shown first followed by samples from ovens 1 and 2 respectively.) Some comparative photomicrographs are given in Figs. 6, 7, and 8.

From past experience the effect of any given oven cycle on the particular surface area obtained has been suspect. For this reason two series of experiments were conducted with samples being split and placed in two ovens. For the first series, the controls were set so the top of the thermostatic excursions for both ovens would be the same (220°F), but with the mean temperature being different—212.5°F for oven 1 and 216°F for oven 2. In the second series of experiments the size of the excursions was lowered somewhat, with the mean temperature of both ovens being maintained at 202°F.

Table I  
Series I - Micronizer Runs

Treatment	Batch No	Initial Surface Area	Stab Time (Hrs)	Final Surface Area (cm <sup>2</sup> /gm)	
				Oven 1	Oven 2
				(212.5 ± 7.5°F)	(216 ± 4°F)
5% PETN/Acetone into water-water washed	9126-A	16,150	310	3,700	3,750
	9139-A	17,200	328	4,250	4,500
5% PETN/Acetone into water-water and alcohol wash	9126-B	10,350	310	1,850	1,900
	9139-B	12,950	208	2,850	3,250
5% PETN/Acetone into 1/3 alcohol and 2/3 water - water wash	9126-C	14,850	267	3,750	4,500
	9139-C	18,500	208	4,600	4,700
5% PETN/Acetone into alcohol and 2/3 water-alcohol and water wash	9126-D	14,100	110	3,950	4,650
	9139-D	15,400	160	3,750	4,600
5% PETN/Acetone into 1/3 alcohol and 2/3 water-alcohol and water, and Freon wash	9126-E	9,400	110	3,250	4,250
	9139-E	14,600	136	4,000	4,700
5% PETN/acetone into 1/3 alcohol and 2/3 water-alcohol and water and ether wash	9126-F	10,350	199	3,650	3,200
	9139-F	10,950	184	3,750	4,900
5% PETN/acetone into water - alcohol and water and ether wash	9126-G	9,550	184	3,250	3,600
	9139-G	5,750	173	3,300	2,950

Table II  
Series 2 - Micronizer Runs

Treatment	Batch No.	Initial Surface Area	Stab. Time (Hrs)	Final Surface Area (cm <sup>2</sup> /gm)	
				Oven 1 (202 ± 10°F)	Oven 2 (201.5 ± 4.5°F)
5% PETN/Acetone into water w/DSS*-water washed	9156-A	19,250	304	1,250***	7,350
5% PETN/Acetone into water-water DSS wash	9156-B	20,900	304	2,000***	6,900
5% PETN/Acetone into water w/PTA*** - water wash	9156-C	19,700	304	2,800***	5,950
5% PETN/Acetone into water-water PTA wash	9156-D	16,550	304	3,200	4,250
5% PETN/Acetone into water-water wash	9157-E	21,050	304	4,200	4,350
2.5% PETN/Acetone into water-water wash	9158-F	22,750	309	4,000	4,000

\*DSS-Dodecyl Sodium Sulfate

\*\*PTA-Pentaerythritol Tetra-acetate

\*\*\*Excessive lumping noted in sample

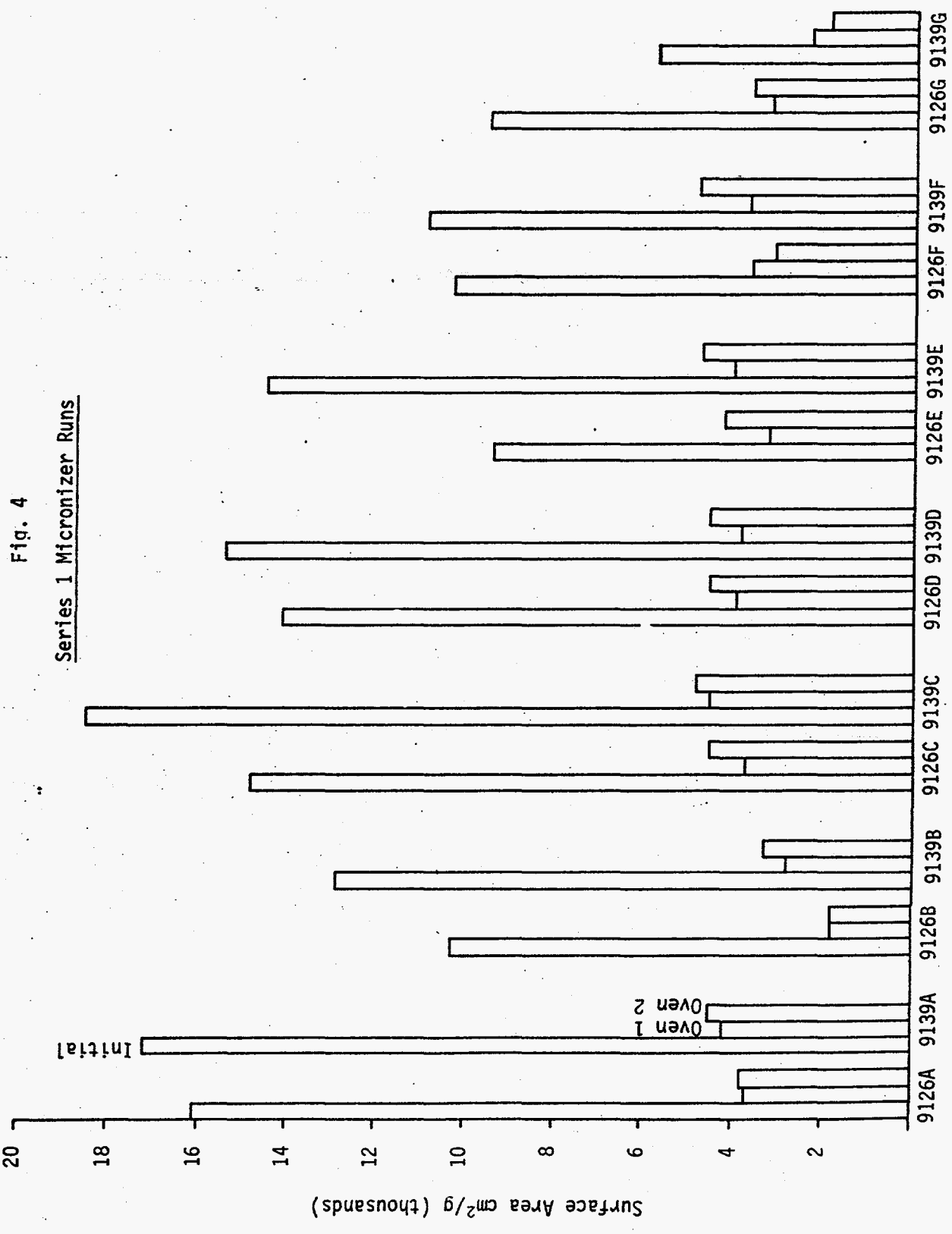


Fig. 5  
Series 2 Micronizer Runs

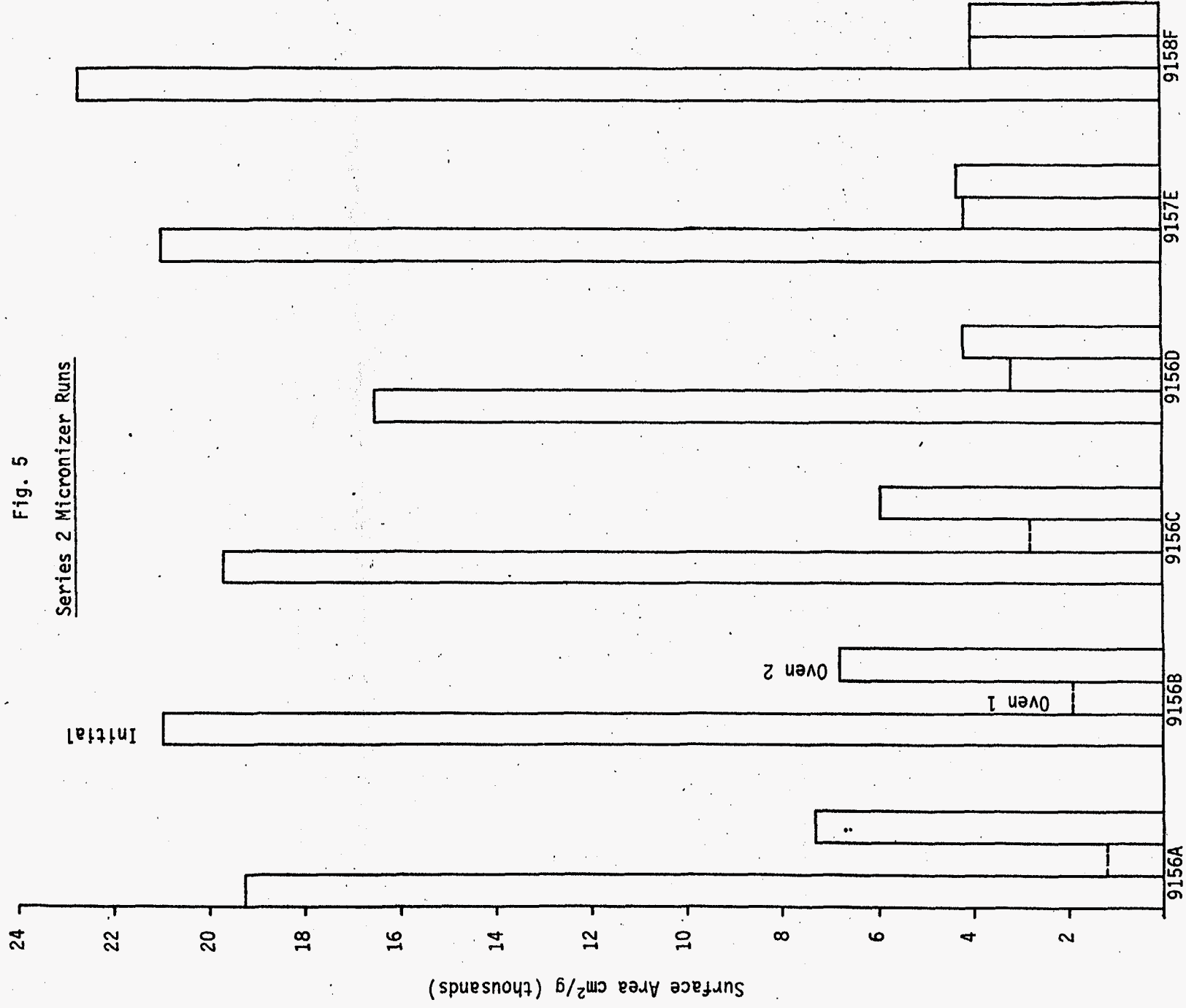
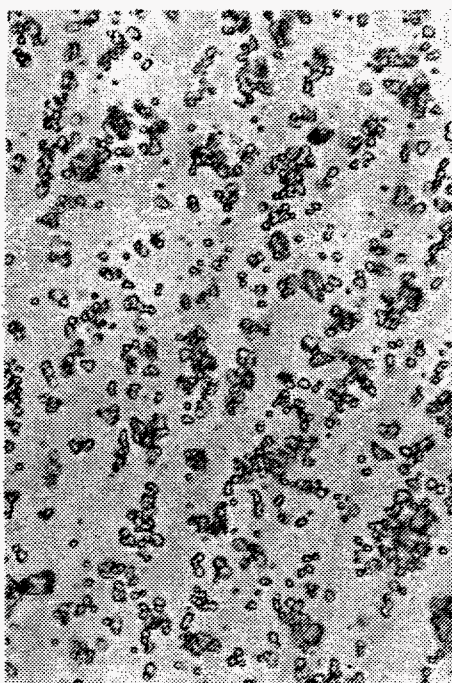




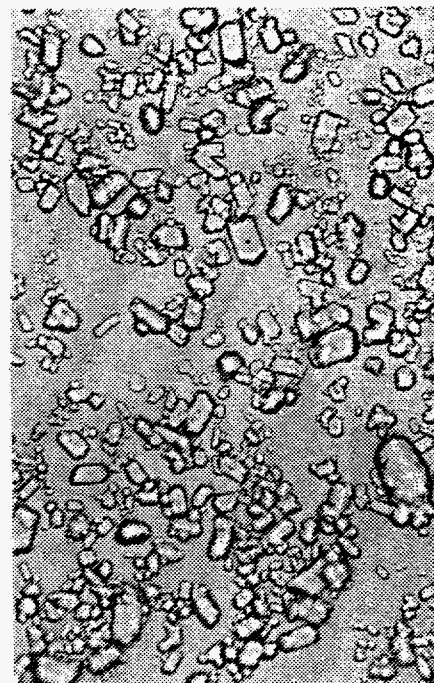
Fig. 6



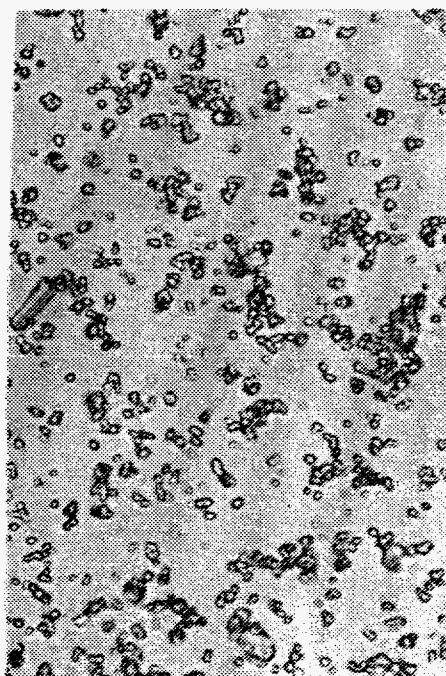
PETN 9126A 940X  
Vac Dried 16150 cm<sup>2</sup>/g



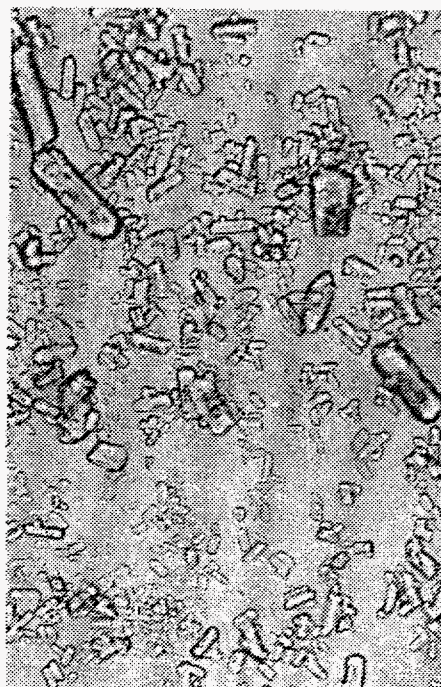
PETN 9126A<sub>1</sub> 320X  
310 Hrs 3700 cm<sup>2</sup>/g



PETN 9126A<sub>2</sub> 320X  
310 Hrs 3750 cm<sup>2</sup>/g



PETN 9139A 940X  
Vac Dried 17200 cm<sup>2</sup>/g



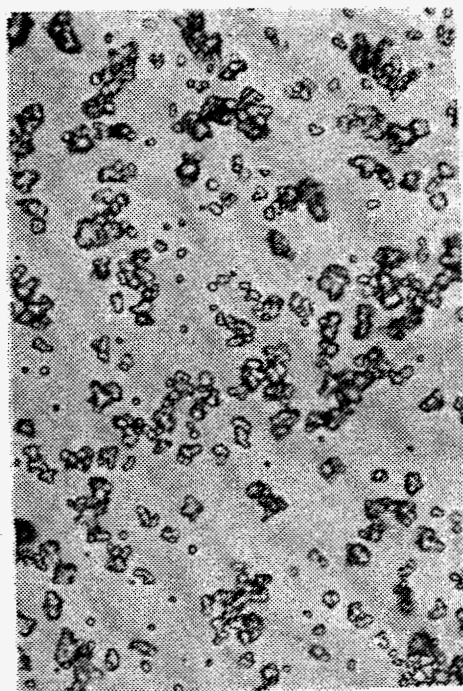
PETN 9139A<sub>1</sub> 320X  
328 Hrs 4250 cm<sup>2</sup>/g



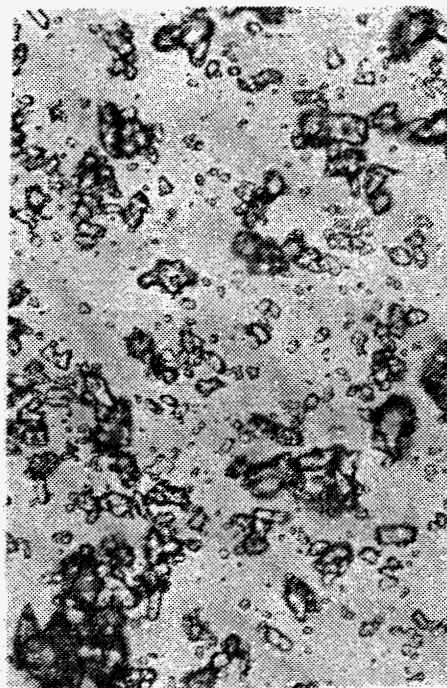
PETN 9193A<sub>2</sub> 320X  
328 Hrs 4500 cm<sup>2</sup>/g



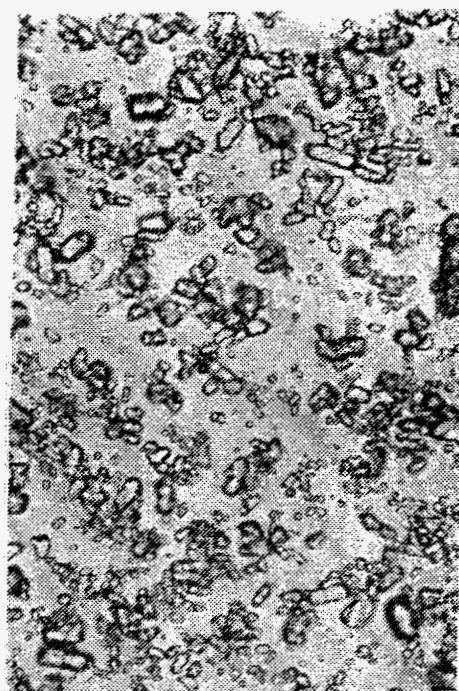
Fig. 8



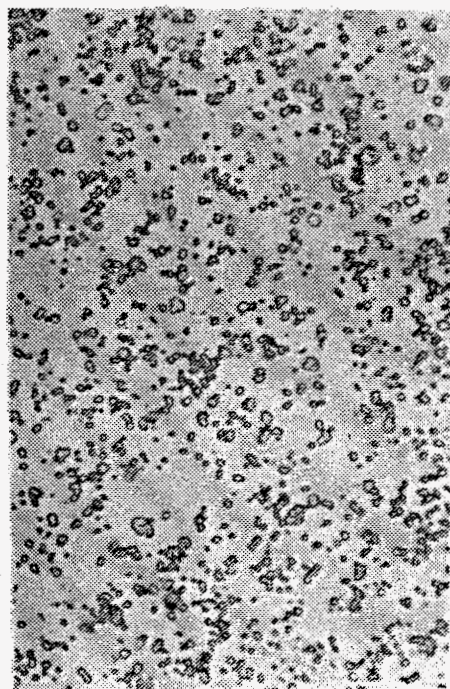
PETN 9156C 940X  
Vac Dried 19700 cm<sup>2</sup>/g



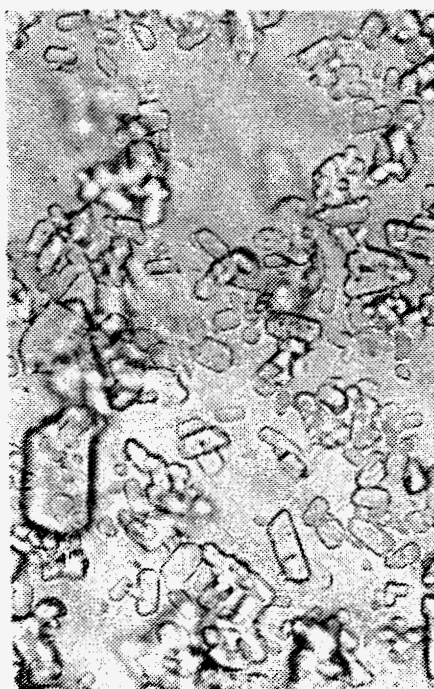
PETN 9156C<sub>1</sub> 585X  
304 Hrs 2800 cm<sup>2</sup>/g



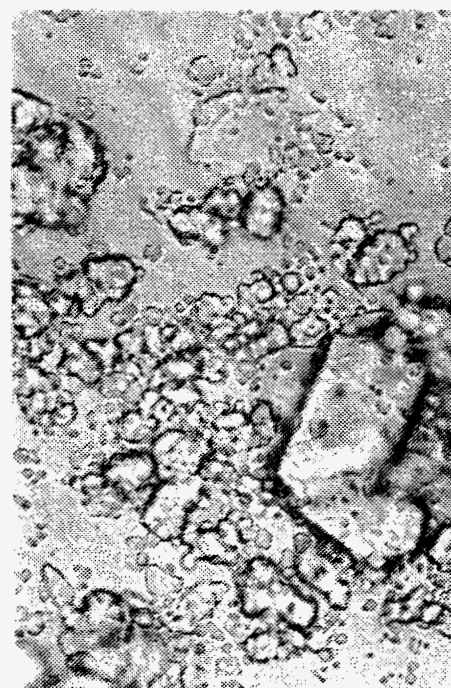
PETN 9156C<sub>2</sub> 585X  
304 Hrs 5950 cm<sup>2</sup>/g



PETN 9158F 940X  
Vac Dried 22750 cm<sup>2</sup>/g



PETN 9158F<sub>1</sub> 585X  
309 Hrs 4000 cm<sup>2</sup>/g



PETN 9158F<sub>2</sub> 585X  
309 Hrs 4000 cm<sup>2</sup>/g



Also in the first group of experiments an attempt was made to duplicate conditions by making repeat runs. As seen in Fig. 4, the stabilized surface areas were reasonably reproducible. Note, that with sample G as the only exception, the initial surface areas of the first group were always somewhat lower than the corresponding runs. Oven 2 with its smaller temperature cycle produced slightly higher surface areas than oven 1 though the mean temperature—and the maximum temperature—was higher.

As may be noted from Table I, all of the aging periods are not exactly matched because all samples were not continued for the full 300-hour cycle since the particle sizes continued to increase and the relative surface areas were dropping beyond any reasonable usefulness. Thus in the interest of time, the aging cycle was prematurely discontinued and the second series of experiments were begun. Again for this group as shown in Fig. 5 the narrower oven cycle—and lower maximum temperature—can be noted as producing the higher surface area. In Table II the different methods used for applying the wetting agents and the corresponding results are given. In Fig. 5 these results are graphically illustrated. The significance of the large difference in surface area between oven 1 and 2 for samples 9156A, B, C cannot be assessed because of the excessive caking of the material.

Of the two wetting agents investigated, pentaerythritol tetraacetate and dodecyl sodium sulfate, the DSS seems to be the most promising, yielding the higher surface area, 7400 cm<sup>2</sup>/gm.

LX-09

The two 250-pound batches of deviate composition LX-09 ordered by Burlington (for hydro testing experiments) were made in the 300-gallon kettle using a procedure discussed in detail in previous reports. The composition of the first batch was slightly low in FEFO and may require reworking. The composition of the second batch was very close to the desired composition. The composition of one lot was planned to be on the high side of the nominal tolerance range for LX-09 while the other lot was to be on the low side for studying the effect of variations in compositions. The following is a tabulation of the desired composition and the actual composition:

<u>LX-09-AA No. 9168</u>	<u>HMX</u>	<u>DNPA</u>	<u>FEFO</u>	<u>DNPA + FEFO</u>	<u>Ratio</u>
Desired	$92 \pm \begin{matrix} 0.0 \\ 0.5 \end{matrix}$	5.41	2.84	$8 \pm \begin{matrix} 0.5 \\ 0.0 \end{matrix}$	$1.90 \pm 0.35$
Actual	92.51	5.44	2.05	7.49	2.65
<u>LX-09-BB No. 9177</u>	<u>HMX</u>	<u>DNPA</u>	<u>FEFO</u>	<u>DNPA + FEFO</u>	<u>Ratio</u>
Desired	$94 \pm \begin{matrix} 0.5 \\ 0.0 \end{matrix}$	3.77	1.98	$6 \pm \begin{matrix} 0.0 \\ 0.6 \end{matrix}$	$1.9 \pm 0.35$
Actual	94.35	3.74	1.91	5.65	1.96

PBX Development (G. T. West)

The purpose of this project is to develop a PBX system, composed of HMX and a suitable binder, which will yield an energy output within 2% of that of pure crystal density HMX, as measured in the cylinder test at 6 mm expansion.

Upon studying this concept, it became evident that 1) formulations must contain fairly low concentrations, probably less than 3 weight percent, of energetic materials used as binders (e.g., PETN, nitroguanidine, ammonium perchlorate, etc.) and be pressed to 97.5% TMD or greater, or 2) formulations must contain very little, 1% or less, non-energetic binder and be pressed to 99% TMD or greater.

Efforts using the first alternative, namely using energetic materials as binders, proved fruitless, because formulation was difficult, and pressing usable billets to the required density was not achieved.

The latter alternative was explored by formulating a 99%/1% HMX/Vistanex L-100 PBX, which was to be pressed and repressed, using known techniques, either until 99% TMD was reached (at which point a cylinder test would be fired), or until the billet broke. It was believed that, if 99% TMD could not be reached with this PBX (which had the best pressing properties of most formulations tested), it would be unlikely that the purpose of the project could be attained. After eight different repressing attempts (all isostatic), varying such things as pressure, pre-heat conditions, dwell, cycling, relaxation time, etc., the billet had advanced from 97.8% TMD (originally attained by hydrostatic pressing) to 98.6% TMD, at which time the billet cracked extensively. Due to decrease in interest of the high energy explosive, no additional work of this type is planned.

FUTURE WORK; COMMENTS; CONCLUSIONS

The experiments with the different ovens and temperature cycles show that they affect the way the crystal grows and stabilizes. Until more is known about stabilization cycles, to duplicate any given PETN batch it is apparent that one oven should be used exclusively for PETN stabilization and the cycles conditioned and monitored. Numerous other experiments could be conducted related to the particular way that a particular oven stabilizes PETN; a few are being planned, if equipment with the necessary controls can be located to explore the effect of a few degrees variation versus maximum temperature. Most of the work being planned for PETN in the near future involves studying the flow and concentration parameters of the Micronizer and exploring the potential of a few wetting agents.

Appropriate traps have been ordered to convert our large vacuum chamber to a freeze drying chamber to try to obtain a non-caking powder.