

HE FORMULATION

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DEVELOPMENT DIVISION

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SANL 900-003, -008

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ABSTRACT

A freeze drying process has been developed which alleviates the caking of high surface area HE powders upon drying and yields a light fluffy textured powder. Three batches of high surface area PETN treated with a surface active agent and two batches of high surface area (Micronizer) RDX have been freeze dried. The RDX powder had a low bulk density of about 0.25 g/cc. Batch sizes ranging from 2 to 10 pounds or more can be easily processed.

Samples of the RDX, along with Micronized PETN (surface agent treated and heat stabilized, and untreated), Extex PETN made by a semi-continuous process, and appropriate control samples were each made into 80/20 Sylgard compositions and hand-loaded into "Sunburst" assemblies. Some of these assemblies were subjected to 100 C for 30 hours and 300 hours. All of the samples loaded with Micronized PETN and Extex PETN fired satisfactorily, burning to 6-mil thickness of the tapered rays. The RDX fired well also, burning to ~ 20-mil thickness of the rays.

All the high surface area materials were, of course, very difficult to process at the 20% loading of Sylgard.

Several of these samples were also subjected to a detonation velocity test in varying diameter channels. The Micronized PETN behavior was similar to Extex-type PETN.

DISCUSSION

FREEZE DRIED PETN

Techniques now exist for producing not only thermally stable high surface area PETN powders, but also producing them in a fluffy non-caking form. As has been reported previously, Micronized PETN powders in the range of 20,000 cm²/g, after being treated with surface active agent, have been found to be essentially unaffected by heating at 116 C for 8 hours, which is our normal heat stabilization cycle. The surface area of samples did not drop appreciably. These powders, though, after being air dried were found to form hard, lumpy cakes. It has been difficult to break up these cakes to the point where they could be easily handled and loaded into parts. This caking problem is alleviated when the water is removed by freeze drying. Freeze drying yields a light fluffy material.

The first part of Table I includes data obtained from three batches of Micronized high surface area PETN which were freeze dried. They were treated with MRL 22A. As can be seen by comparing the surface areas before and after heating, thermal stability was not completely achieved for the first two batches, Nos. 1363 and 2006. The coating technique or method of applying the MRL 22A to the surface

of the crystals was different from that previously found to be successful; the modification hopefully would make the batch more readily adaptable to the freeze drying process. For example, the first batch, No. 1363, after being Micronized and collected in the centrifuge, (approx. 3 pounds of cake) was dispersed in 8 gallons of water containing 116 ppm of MRL 22A and then this water slurry without any further processing was placed directly into the freeze dryer. Had this worked, it would have greatly simplified the coating process. Other modifications were made for the next batch, No. 2006. The PETN was placed into 8 gallons of water containing 200 ppm 22A, filtered, washed, dispersed again in 8 gallons of water (no 22A) and placed into the freeze dryer. The appearance of the crystals may be noted in the photomicrographs in Fig. 1. Unfortunately, as shown from surface areas after heating, the coating of MRL 22A was not as complete or effective as it was hoped that it would be. Therefore, for the third batch, No. 2013 the technique used previously to effectively apply the MRL 22A was used. This involved dispersing the PETN in water containing 200 ppm of MRL 22A at a ratio of 9 gallons of water solution for each pound of HE. The cake is then again collected in the centrifuge, washed and then dispersed in 8 gallons of water and placed in the freeze drier. (The last dispersion in water is not a part of the coating process, but is incorporated as part of the freeze drying process.) Needless to say this technique involving larger quantities of water creates more filtration and handling. As noted in Table I and Fig. 2 the treatment of the MRL 22A was effective as the crystals were thermally stable. Additional samples were taken on this batch after various steps as reported in the results.

FREEZE DRIED RDX

RDX has been Micronized, using essentially the same procedure as used for PETN, in order to produce a high surface area powder with a low bulk density which has application to special equation of state studies*. Last quarter year, attempts to produce high surface area Micronized RDX yielded material with a bulk density of 0.45 g/cc which was somewhat higher than the desired 0.3 g/cc. This batch, though, was air dried in the conventional manner. The batches made this period, however, were freeze dried, and, as shown in Table I, had bulk densities of about 0.25 g/cc. Photomicrographs are in the upper part of Fig. 3. As a matter of interest, samples of this RDX were incorporated into Sylgard and loaded into "Sunbursts" and test fired. As discussed later these turned out rather well.

EXTEx OR LX-13 PETN

Also shown in Table I and Fig. 3 is an Extex or LX-13 type PETN made by a "semi-continuous" process or a "continuous batch" process similar to that used with the Micronizer. It differs from conventional Extex PETN reprecipitation. Instead of rapidly dumping acetone/PETN solution into a large volume of agitated water, the PETN solution and water are metered into a small turbulent chamber at a constant rate. The discharge is also held constant. In such a system none of

*By M. Finger, LLL.

the parameters vary as the recrystallization progresses. For this experiment the 30-liter reactor was used as the recrystallization chamber and the Lapp metering pumps were used to feed the fluids into the chamber. Limited firing data reported in Tables II and III (Sunburst and detonation velocity) indicates that this material is probably not different from conventionally made Extex. The firing tests and the other materials tested are discussed in more detail later.

This process has certain potential advantages for production. It should yield a consistent product, perhaps even to the point that between-batch differences would disappear. Other advantages would be the low cost of the equipment, and the cleanliness of the process. It can be kept in a closed circuit and all the solution feed lines can have in-line filters.

The evaluation of this type of approach actually began last quarter when a program was initiated to evaluate Micronized PETN for potential application to Extex. Desirable characteristics of the Micronization of PETN are the repeatability and cleanliness of the process. Also being included in the study was an evaluation of MRL 22A surface active agent.

The effort was initiated last quarter year by producing Micronized powders with surface areas in the range of $20,000 \text{ cm}^2/\text{g}$. They included untreated, treated, and heat stabilized powders. These were incorporated into the Sylgard binder (80% PETN loading) and tested this period. Processing was by methods similar to those used for making Extex; i.e., mixing followed by several passes on the 3-roll mill. Samples were small and the small laboratory 3-roll mill was used. These, along with other materials, were fired in the Sunburst, Table II (materials 2 through 4). Some limited detonation velocity data were also obtained, Table III.

The specific purposes of the experiments were primarily two fold: (1) to determine whether a starting material with a surface area in the range of $20,000 \text{ cm}^2/\text{g}$ (FSSS) will produce the same product as starting with a $5,000$ to $6,000 \text{ cm}^2/\text{g}$ powder and breaking the powder down to the range of $20,000 \text{ cm}^2/\text{g}$, as is now done during roll milling in the manufacture of Extex; and (2) to determine if the different crystal habit of the Micronized PETN would fire the same as Extex in the same geometric configurations. As mentioned, they were all tested in the LLL Sunburst configuration with the heat stabilized, treated sample, No. 2, also being tested for detonation velocity, Table III.

Several processing problems were encountered in incorporating 20% Sylgard into the higher surface area material. That was also true with RDX. Because of the high surface area, the proper wetting of the crystals was not properly achieved and the material was very dry and flaky. It was very difficult to hand-butter into the fixtures.

To explain the firing data more in detail, the Sunburst results are reported in Table II by giving the groove depth in thousandths of an inch at the point at which burning ceased. The Sunburst consists of 36 tapered grooves about 2.4 inches long which vary from 0.060 inch in depth to zero at the end of the ray. Width is held constant at 0.060 inch. Each Sunburst plate was divided in six

sections of six rays for each of the materials numbered in Table II. Also, as shown, there were 8 plates, four of which were heated. The factor limiting temperature was the Lexan from which the plates were made. Higher temperatures caused excessive distortion. Selected photographs of the loaded sunbursts are given in Figs. 4 through 7. These are typical in appearance of the other tests not shown. Also radiographs were made of the parts at the various stages, before and after heating, etc. Figs. 8 through 15 are photographs of the aluminum witness plates. Then, in Figs. 16 through 21 comparative photomicrographs were made of the HE powders loaded in the Sunburst, as the crystals appeared before they were incorporated into Sylgard, and then after they were incorporated into Sylgard.

Material No. 1 was of Development Extex (batch process) used as a control, 2 through 4 were PETN from the Micronizer, discussed earlier, No. 5 was from the semi-continuous batch of PETN, No. 6 was from a production batch of Extex, used as a second control, No. 7 was Micronized PETN, made much earlier and having one of the lowest surface areas ever to come out of the Micronizer, No. 8 was Du Pont Mil Spec PETN (which did not burn), No. 9 and 9A were the Micronized RDX samples, which burned two-third of the length of the rays, No. 10 is a batch of Class E RDX, which soon extinguished, and the last batch, No. 11, was made using the same high surface area PETN as in No. 2. It was made by a slightly different technique to see if wetting or the texture of the 80/20 Sylgard material could be improved by processing changes. Instead of adding the Sylgard to the powder and then adding the fluidizing agent (MF or TF Freon), the Sylgard was diluted with freon and the powder was added and stirred in slowly to see if crystal wetting could be improved. It was not. Thus, a technique has yet to be developed to successfully incorporate binder on PETN which has initially a high surface area.

Photographs of the Sunburst witness plates are essentially self explanatory, (Figs. 8 through 15). PETN loaded material is considered acceptable if it burns out to the scribed ring. As can be noted, in almost all cases the Extex and Micronized PETN burned passed the scribed mark. In Fig. 8, the failure of one of the rays in sunburst No. I was caused by a void in the ray, clearly visible in the X-ray (not shown). In Fig. 9, (Sunburst No. II) two sections of rays were not filled with any material. Fig. 12 shows the burnout of the Du Pont PETN and the Class E RDX. Note the length of the burn on the rays filled with Micronized RDX in this figure and also in Figs. 13 and 15. One of the individual RDX rays in Fig. 12 failed because of voids in the ray which are evident in the radiographs (not shown).

FUTURE WORK; COMMENTS; CONCLUSIONS

Basically the sunburst data were taken to show that the Micronizer PETN might fire as well as the Extex type PETN with the novel crystal habit, and that the processing or recrystallization of the Extex-type PETN by a semi-continuous process would not adversely affect firing. These experiments and data are intended only to establish general trends and point out areas warranting future work.

The purpose for obtaining the detonation velocity, reported in Table III, was to get a general indication of the burning rate of the Micronized PETN and the PETN made by the semi-continuous process. The detonation velocity blocks used were hand-loaded from non-deaerated material. Radiographs show many inclusions which would account for the fact that all of the 0.015-inch tracks were lost as well as some of the other tracks. Attempts were also made to test two detonation velocity blocks loaded with the same type of RDX/Sylgard used in the Sunbursts. These, though, did not burn because the 0.035 inch initiator track going from the detonator to the timing tracks was either too small in cross section, or too full of voids to ignite. The loading of RDX into the Sylgard will have to be reduced significantly or some technique found to wet the RDX w/Sylgard, in order to have a material with a texture and consistency which can be extruded.

Table I

Micronized PETN

Batch No.	Conc. in Acetone	Acetone Flow Rate (cc/min)	Water Flow Rate (cc/min)	Air Dried	FSSS Surface Areas cm ² /g		
					Treated with MRL 22A		
					Air Dried	Freeze Dried	Freeze Dried Heated & 116 C for 8 Hours
1362	2.5%	406	953	-	-	20,300	10,600
2006	2.5%	406	953	-	-	16,700	9,150
2013	2.5%	406	953	19,150	22,450	21,700	20,900

Micronized RDX

Batch No.	Conc. in Acetone	Acetone Flow Rate (cc/min)	Water Flow Rate (cc/min)	Surface Area	Bulk Density
1323	5%	406	953	14,300	0.25
1348	2%	406	953	12,000	0.23

Semi-Continuous Extex PETN

Batch No.	Conc. in Acetone	Acetone Flow Rate (cc/min)	Water Flow Rate (cc/min)	Dried @ 66 C for 30 Hrs. Surface Area
1328	15%	488	3288	4150

Table II. LLL Sunburst Test*

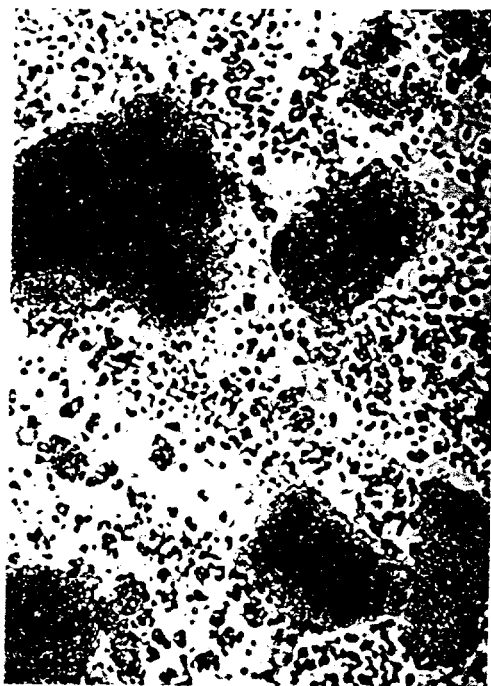
Material No.	Batch No.	Material Description	Surface Area	LLL Sunburst No.							
				Ambient Temp.				Elevated Temp.			
								30 hrs 300 hrs			
				@				@			
				100 C				100 C			
				I	II	III	V	IV	VI	VII	VIII
1	1288	PETN Dev Extex Type	5250	6	6	6	-	6	-	6	-
2	1286-2	PETN Mic 22A 8 hrs/116C	18600	6	-	6	-	6	-	6	-
3	1286-1	PETN Mic 22A	20700	6	-	6	-	6	-	6	-
4	1281	PETN Mic	19400	6	-	6	-	6	-	6	-
5	1328	PETN Semi-continuous Extex Type	4150	6	6	6	-	6	-	6	-
6	393	PETN Extex - Production Type	4850	6	6	6	6	6	6	6	6
7	9255	PETN Mic	9200	-	6	-	6	-	6	-	6
8	1340-301	PETN - Du Pont Mil Spec.	600	-	-	-	60	-	60	-	60
9	1340-322	RDX-Mic	13900	-	-	-	18	-	16	-	-
9A	1356	RDX-Mic	12000	-	-	-	-	-	-	-	21
10	1341-322	RDX-Class E	900	-	-	-	42	-	41	-	41
11	1341-301	PETN Mic 22A-8 hrs/116C	18600	-	-	-	6	-	6	-	6

*Results are expressed by giving groove depth in thousands of an inch at the point along the tapered groove where burning ceased.

Table III. Detonation Velocity

No.	Batch No.	Material Description	Surface Area	Detonation Velocity m/sec			
				Track Size			
				0.020	0.035*	0.080	0.015
2	1286-2	PETN Mic 22A -8 hrs/116 C	18,600	7,106	7,187	7,214	Lost
5	1328	PETN Extex Type Semi-cont.	4,150	7,142	7,227	7,253	Lost
6	393	PETN Extex Type Production	4,850	7,155	7,221	7,248	Lost
7	9255	PETN Micronizer	9,200	Lost	Lost	7,266	Lost

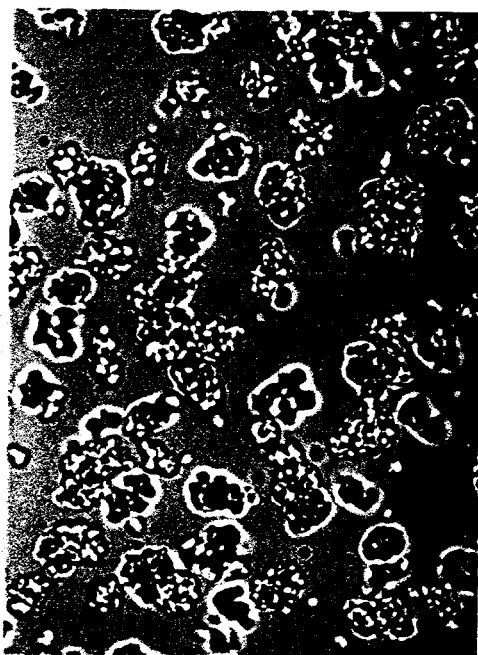
*Nominal 7270 ± 25



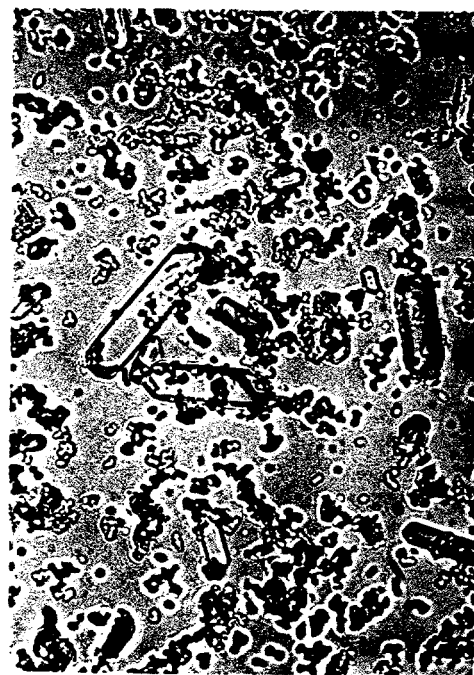
PETN No. 1363 400X, Treated
w/MRL 22A, Freeze Dried, So(P)
20,300 cm²/g



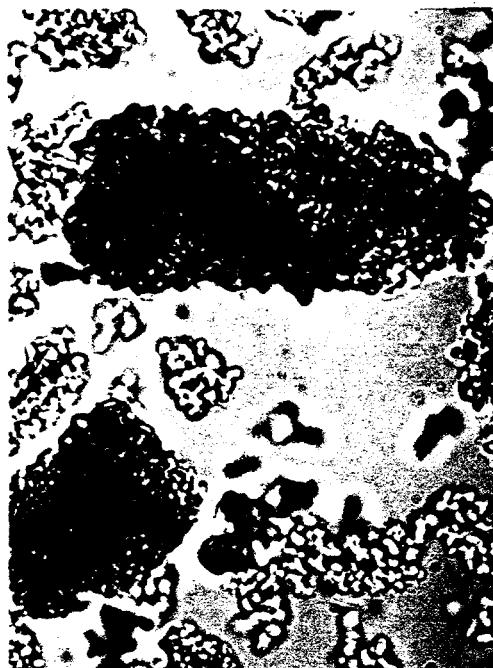
PETN No. 1363 800X, Treated
w/MRL 22A, Freeze Dried, Heat
Treated, 8 hrs @ 116 C, So(P)
10,600 cm²/g



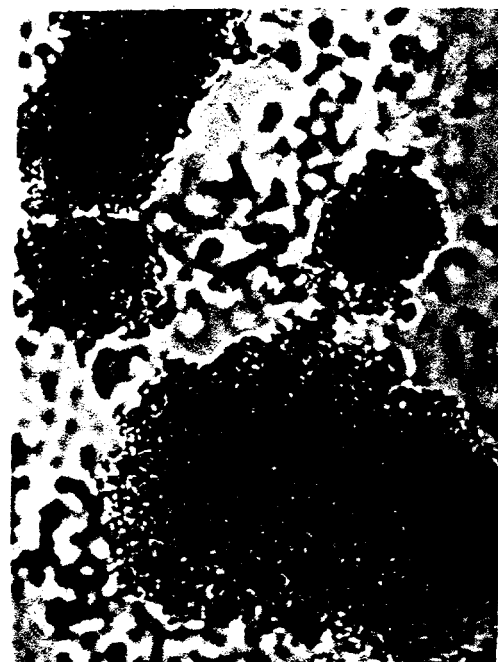
PETN No. 2006 800X, Treated
w/MRL 22A, Freeze Dried, So(P)
16,700 cm²/g



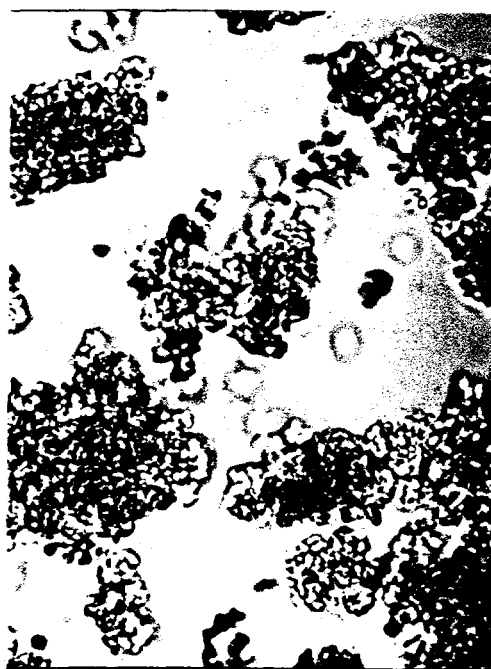
PETN No. 2006 400X, Treated
w/MRL 22A, Freeze Dried, Heat
Treated, 8 hrs @ 116 C, So(P)
9,159 cm²/g



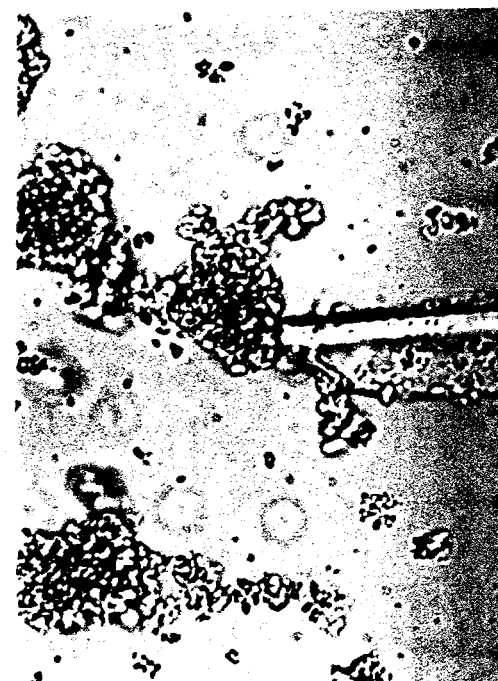
PETN No. 2013 800X, Air Dried
So(P) - 19,950 cm²/g



PETN No. 2013 800X, Treated
w/MRL 22A, Air Dried, So(P)
22,450 cm²/g

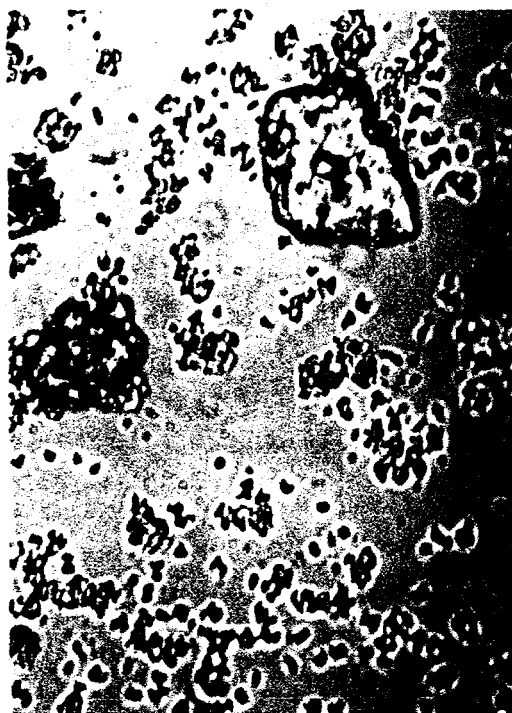


PETN No. 2013 800X, Treated
w/MRL 22A, Freeze Dried So(P)
21,700 cm²/g



PETN No. 2013 800X, Treated
w/MRL 22A, Freeze Dried, Heat
Treated , 8 hrs @ 116 C, So(P)
20,900 cm²/g

Fig. 2



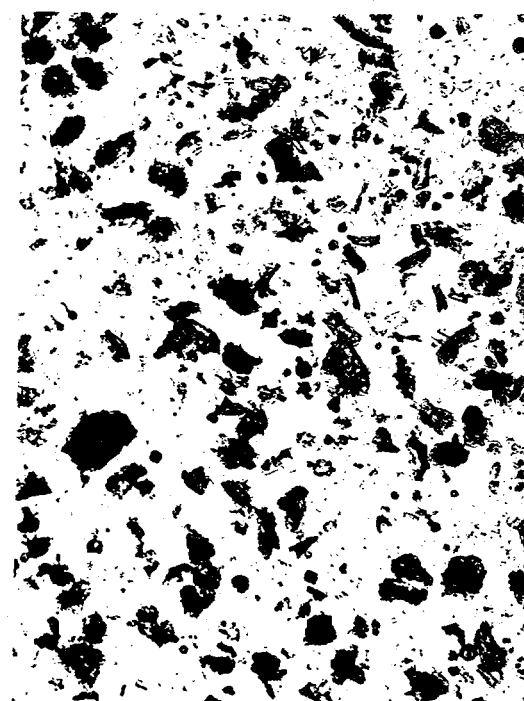
Micronized RDX No. 1323
800X, Freezed Dried, So(P)
14,300 cm²/g, 5% in Ace-
tone Bulk ρ 0.25



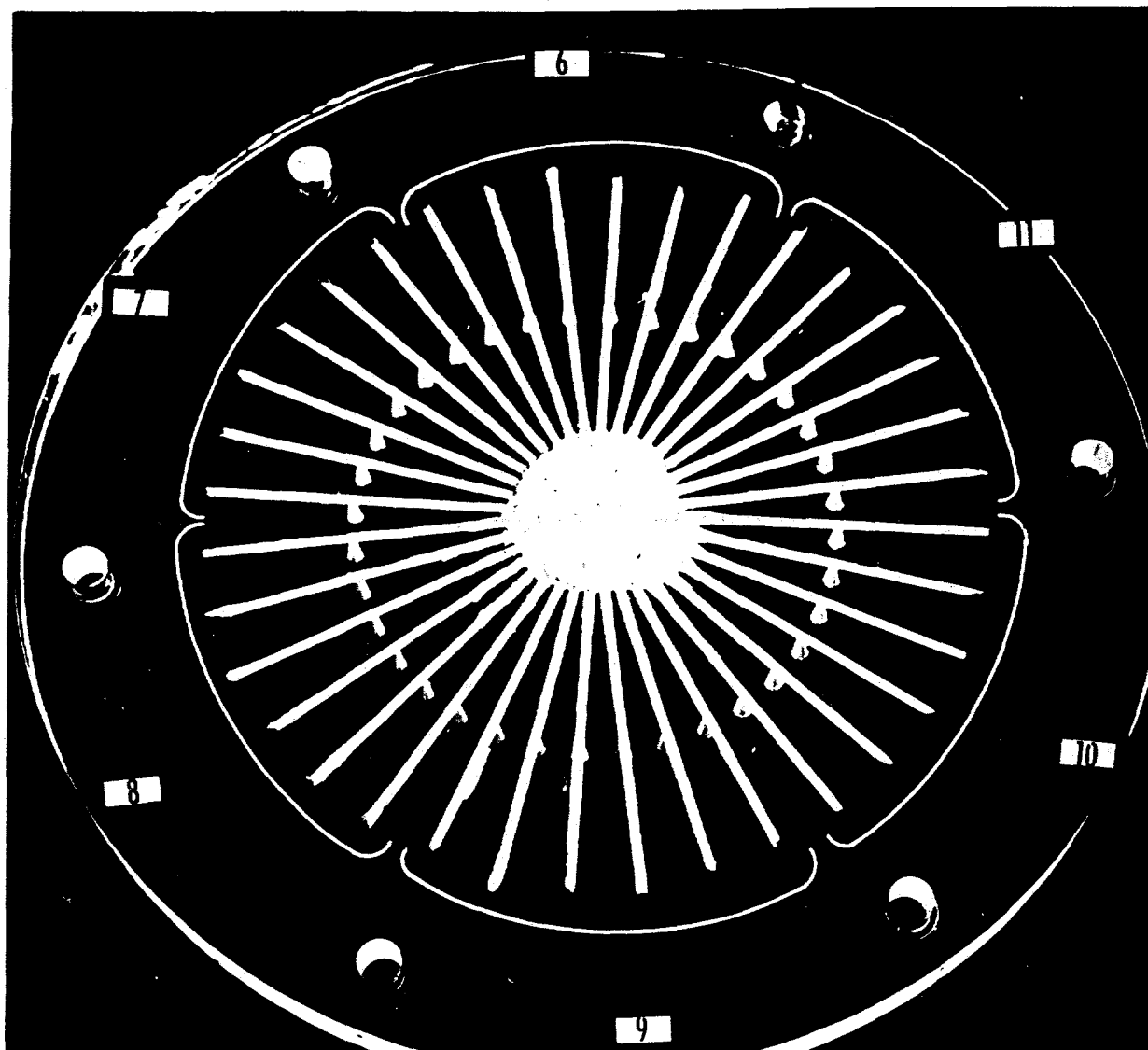
Micronized RDX No. 1348
800X, Freezed Dried,
So(P) - 12,000 cm²/g
2% in Acetone Bulk ρ 0.23



Extex PETN No. 1328 400X,
Semi-continuous Process
So(P) - 4,150 cm²/g

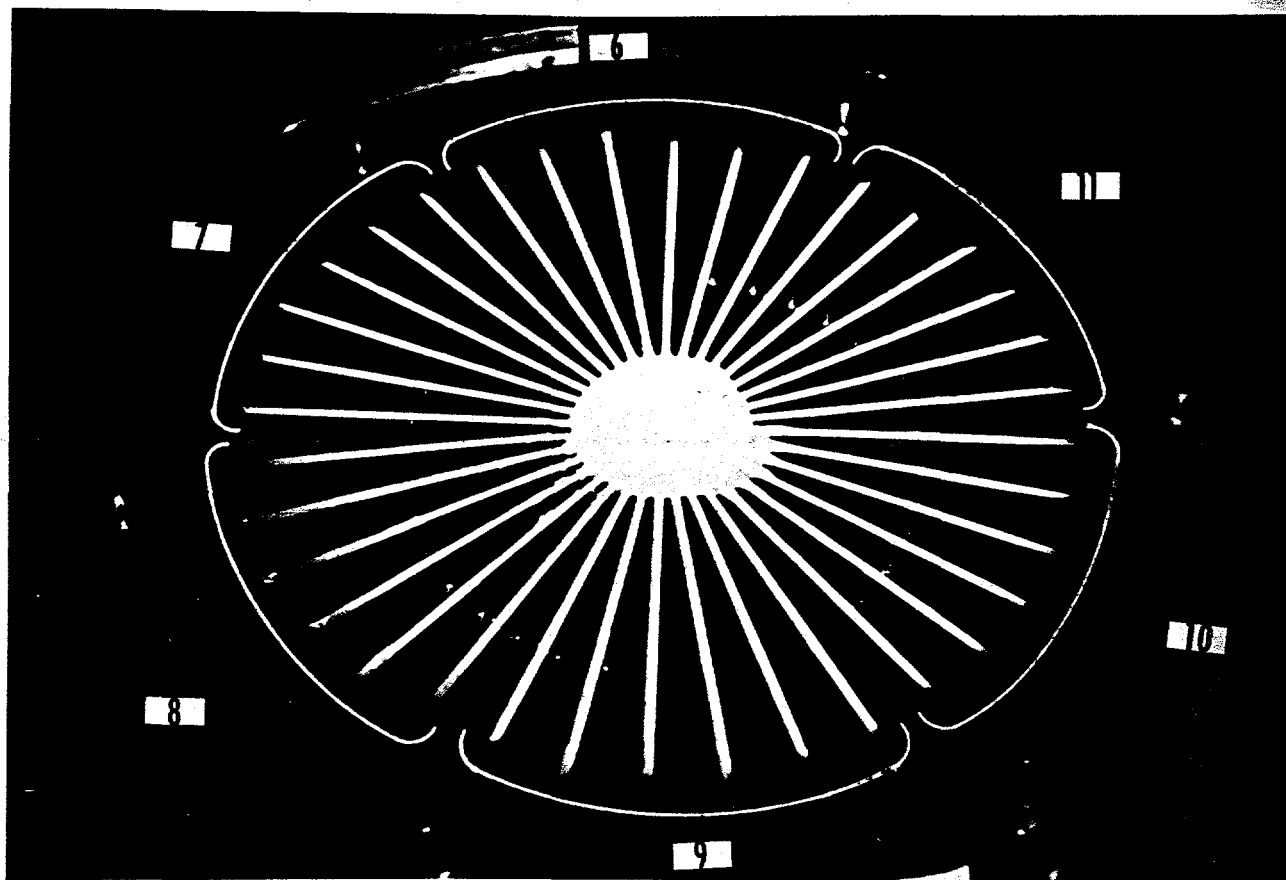


Extex PETN No. 1288 63X,
Batch Process-control So(P)
5,250 cm²/g



- No. 6, 393 PETN - Production Extex
- No. 7, 9255 PETN - Micronized (Low Surface Area)
- No. 8, 1340-301 PETN - Du Pont Mil Spec
- No. 9, 1340-322 RDX - Micronized
- No. 10, 1341-322 RDX - Class E
- No. 11, PETN - Micronized MRL 22A, 8 hrs/116 C

Fig. 4. LLL Sunburst No. VI Before Heating



No. 6, 393 PETN - Production Extex

No. 7, 9255 PETN - Micronized (Low Surface Area)

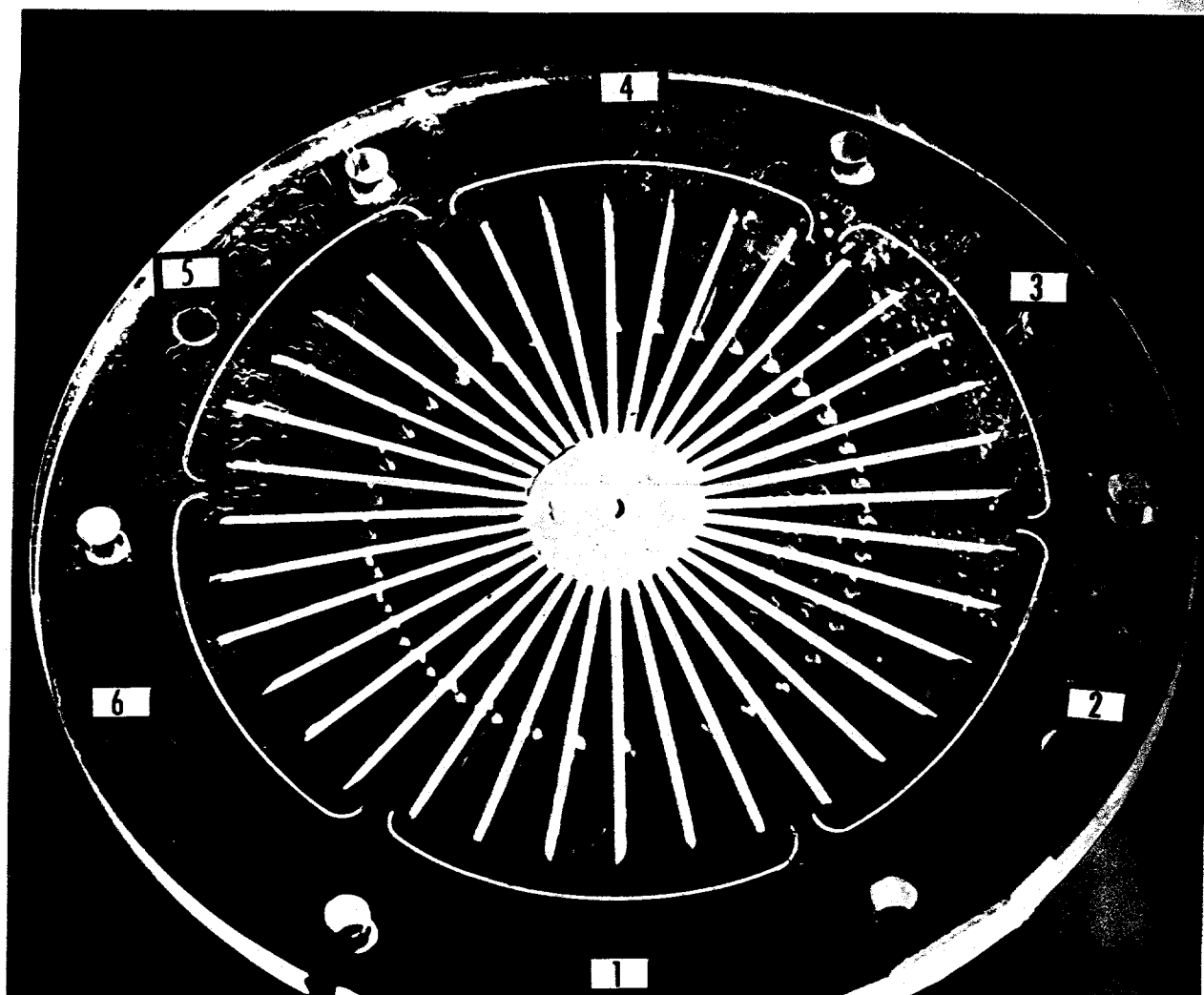
No. 8, 1340-301 PETN - Du Pont Mil Spec

No. 9, 1340-322 RDX - Micronized

No. 10, 1341-322 RDX - Class E

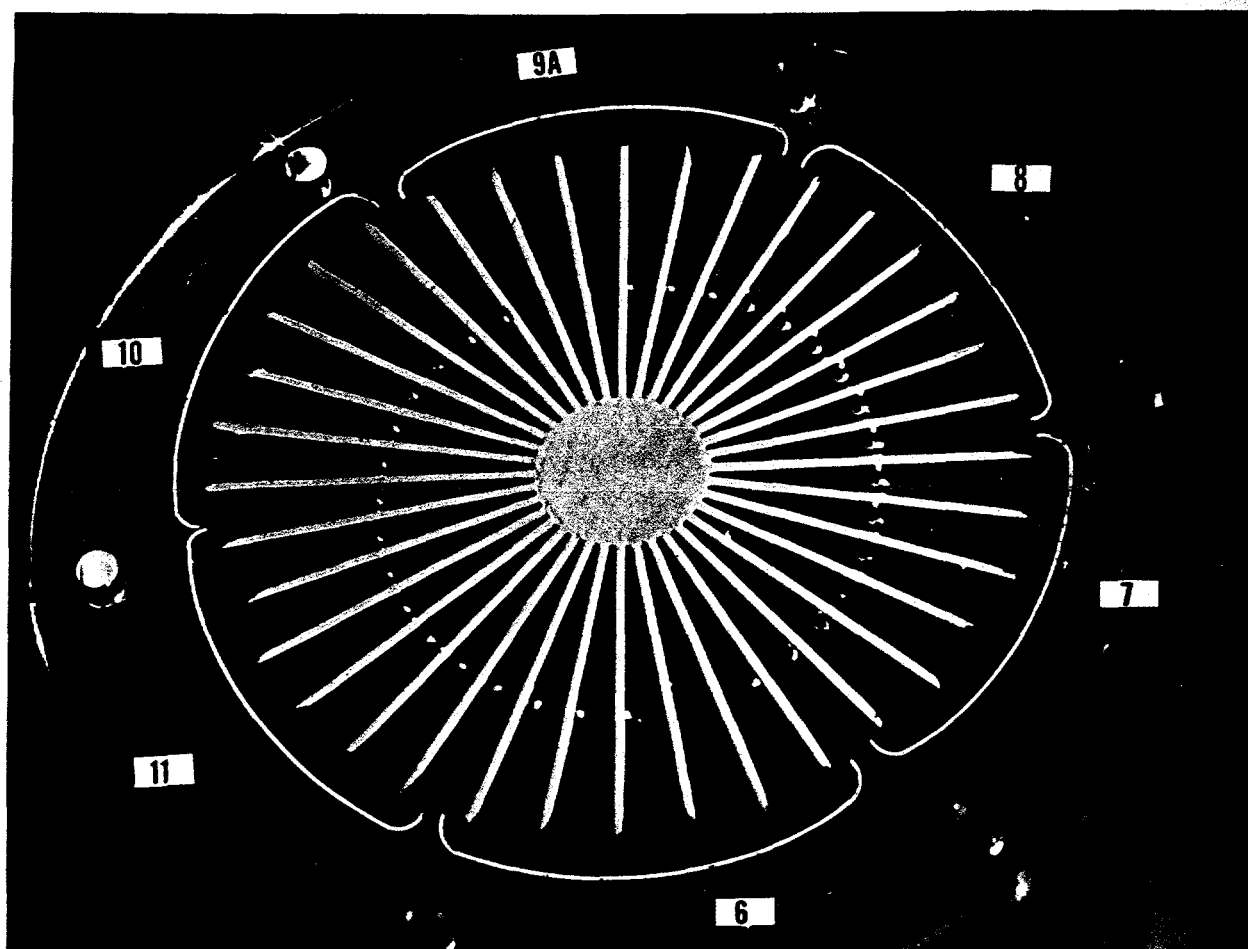
No. 11, PETN - Micronized MRL 22A, 8 hrs/100 C

Fig. 5. LLL Sunburst No. VI After 30 hrs/100 C



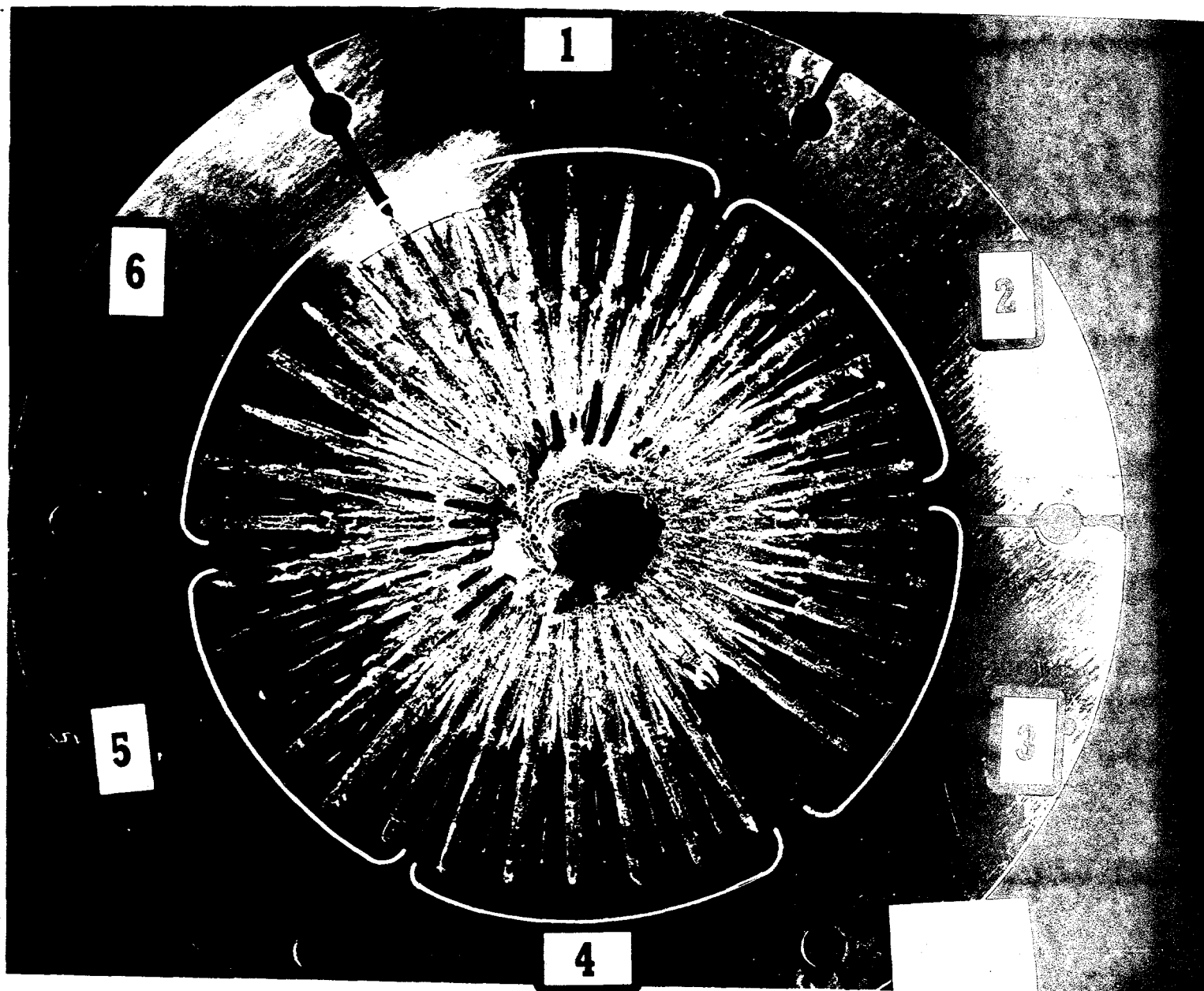
- No. 1, 1288 PETN - Development Extex
- No. 2, 1286-2 PETN - Micronized MRL 22A, 8 hrs/116 C
- No. 3, 1286-1 PETN - Micronized MRL 22A
- No. 4, 1281 PETN - Micronized
- No. 5, 1328 PETN - Semi-continuous, Batch Extex
- No. 6, 393 PETN - Production Extex

Fig. 6. LLL Sunburst No. VII After 300 hrs/100 C



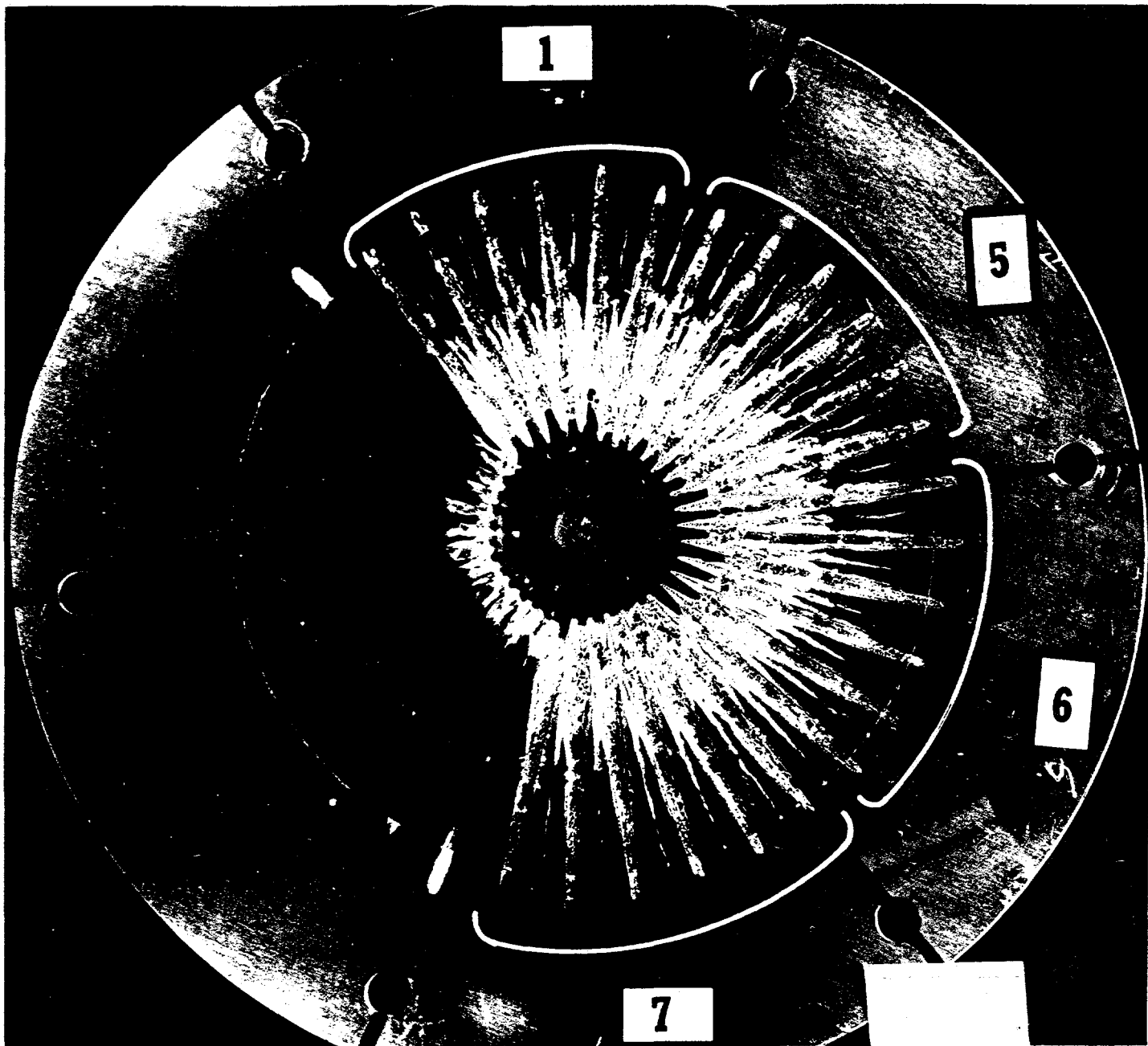
- No. 6, 393 PETN - Production Extex
- No. 7, 9255 PETN - Micronized (Low Surface Area)
- No. 8, 1340-301 PETN - Du Pont Mil Spec
- No. 9A, 1359 RDX - Micronized
- No. 10, 1341-322 RDX - Class E
- No. 11, 1341-301 PETN - Micronized MRL 22A, 8 hrs/116 C

Fig. 7. LLL Sunburst No. VIII After 300 hrs/100 C



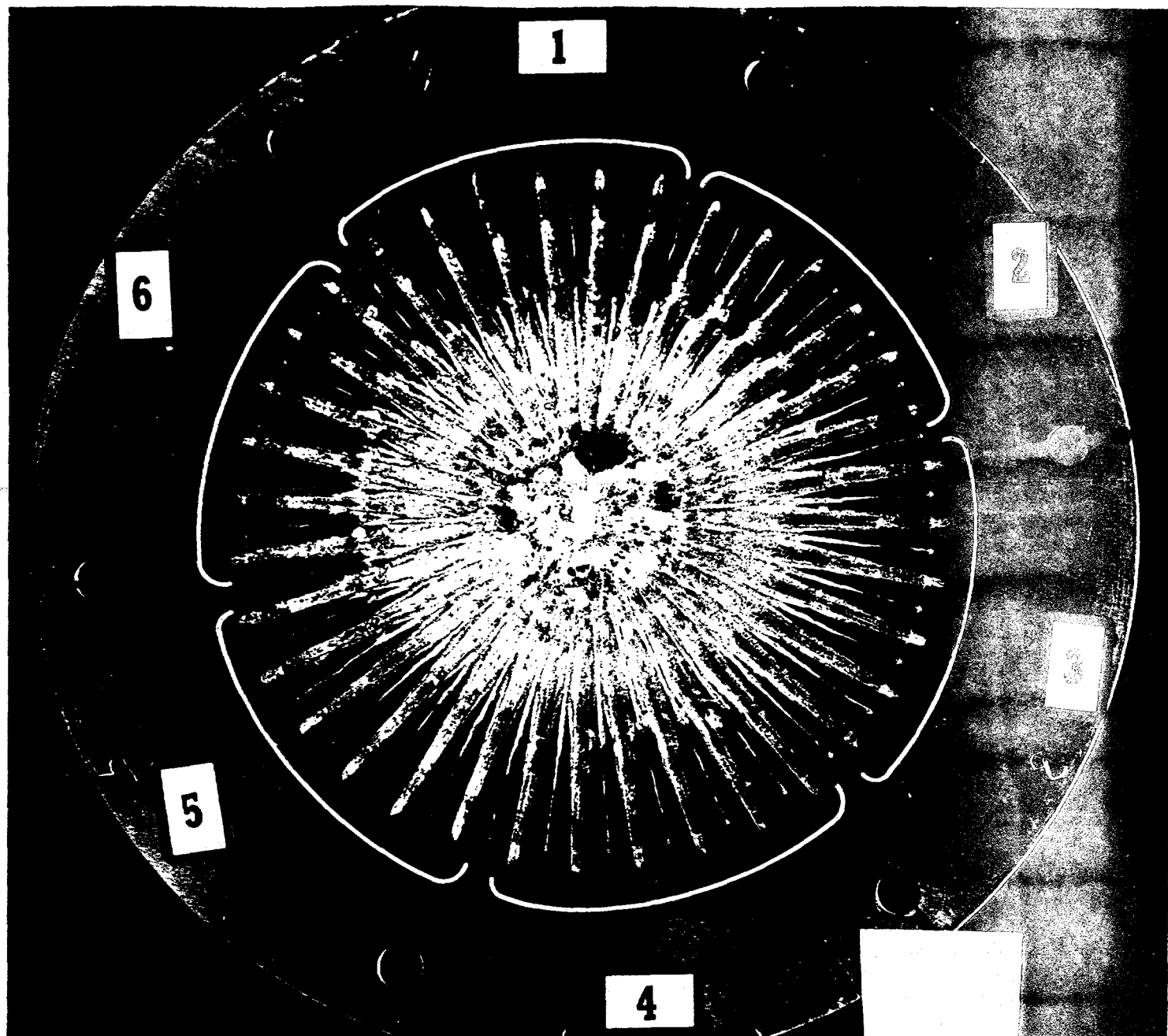
- No. 1, 1288 PETN - Development Extex
No. 2, 1286-2 PETN - Micronized MRL 22A, 8 hrs/116 C
No. 3, 1286-1 PETN - Micronized MRL 22A
No. 4, 1281 PETN - Micronized
No. 5, 1328 PETN - Semi-continuous, Batch Extex
No. 6, 393 PETN - Production Extex

Fig. 8. LLL Sunburst No. 1 Witness Plate



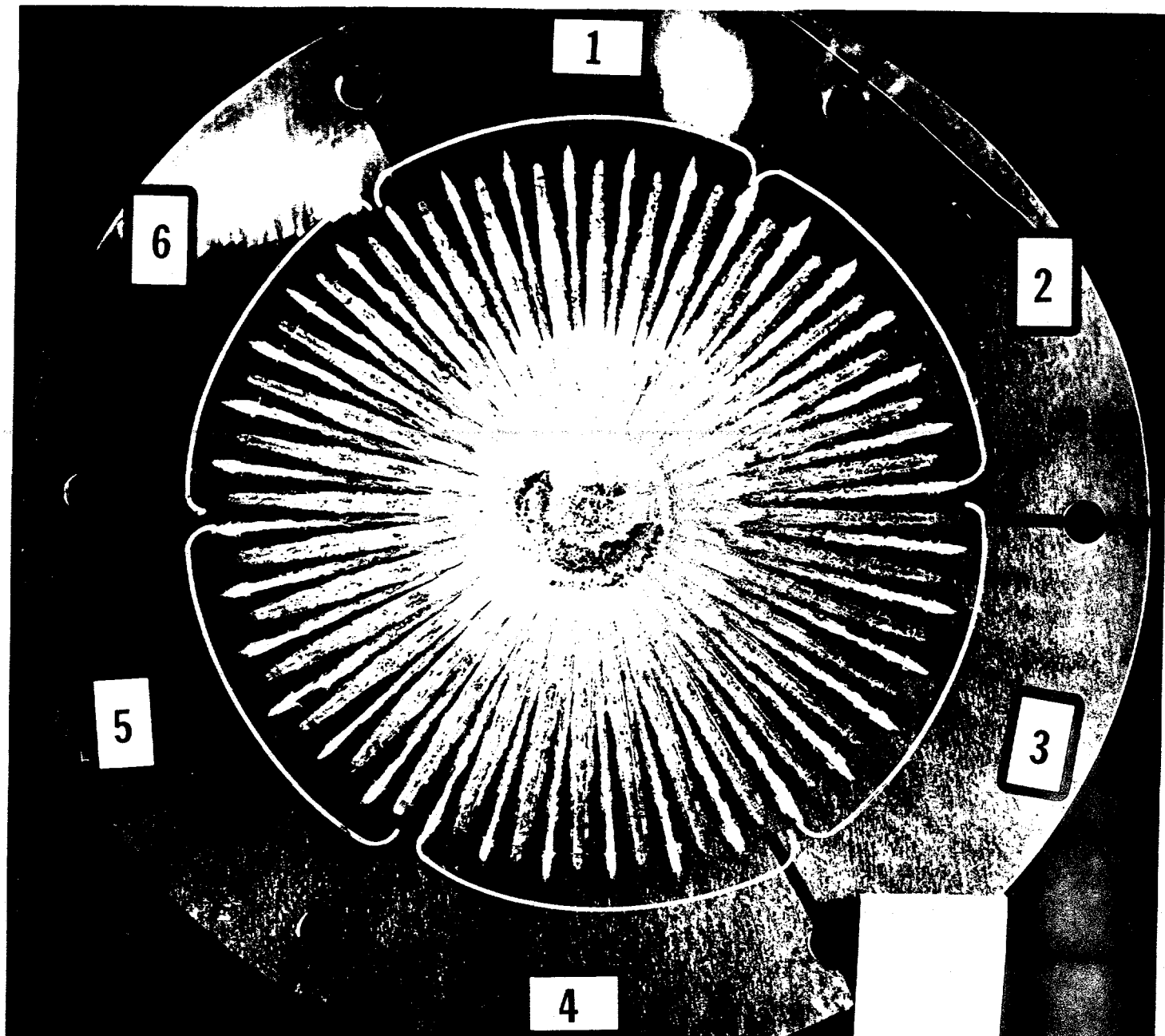
No. 1, 1288 PETN - Development Extex
No. 5, 1328 PETN - Semi-continuous, Batch Extex
No. 6, 393 PETN - Production Extex
No. 7, 9255 PETN - Micronized

Fig. 9. LLL Sunburst No. II Witness Plate



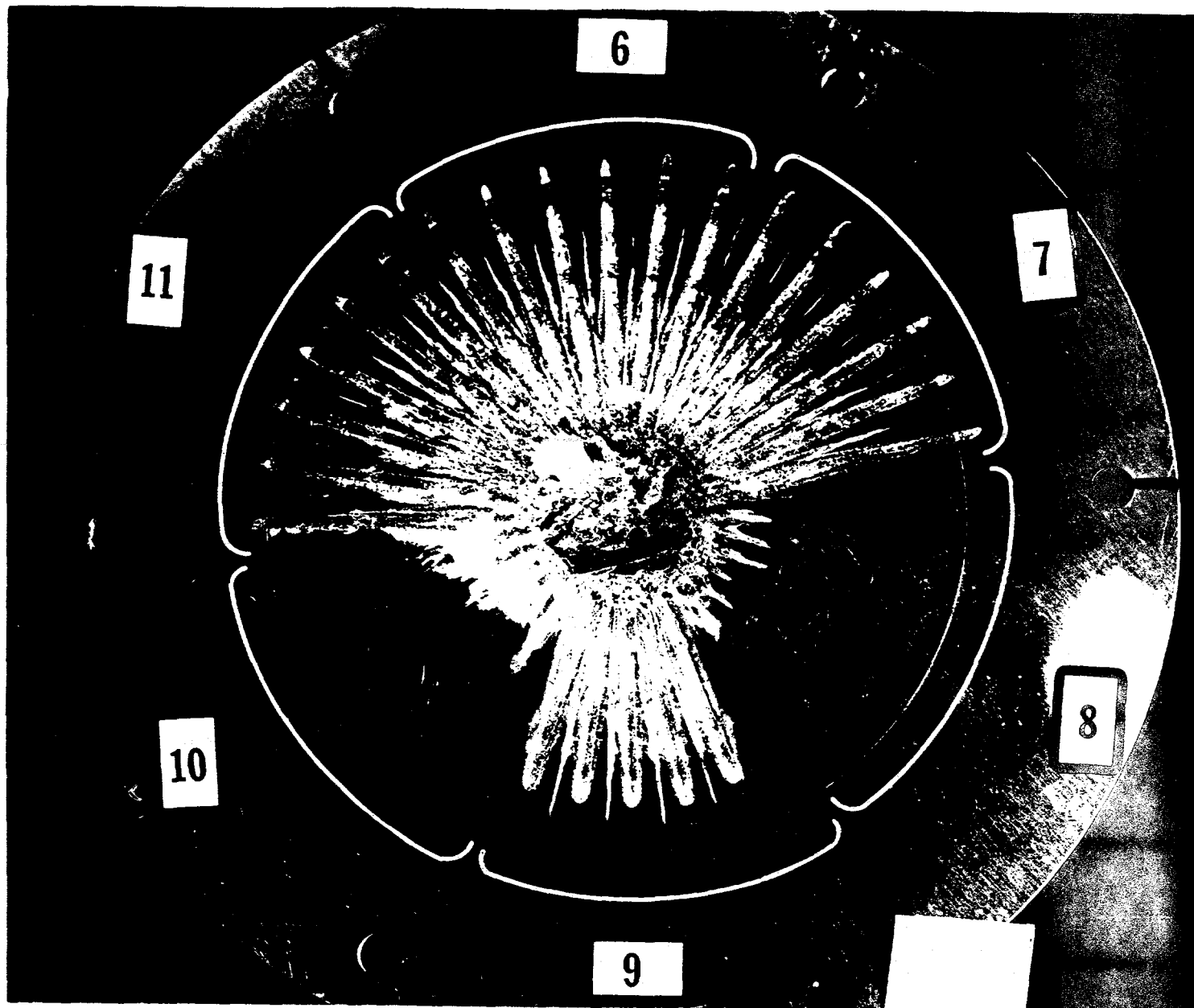
- No. 1, 1288 PETN - Development Extex
- No. 2, 1286-2 PETN - Micronized MRL 22A, 8 hrs/116 C
- No. 3, 1286-1 PETN - Micronized MRL 22A
- No. 4, 1281 PETN - Micronized
- No. 5, 1328 PETN - Semi-continuous, Batch Extex
- No. 6, 393

Fig. 10. LLL Sunburst No. 111



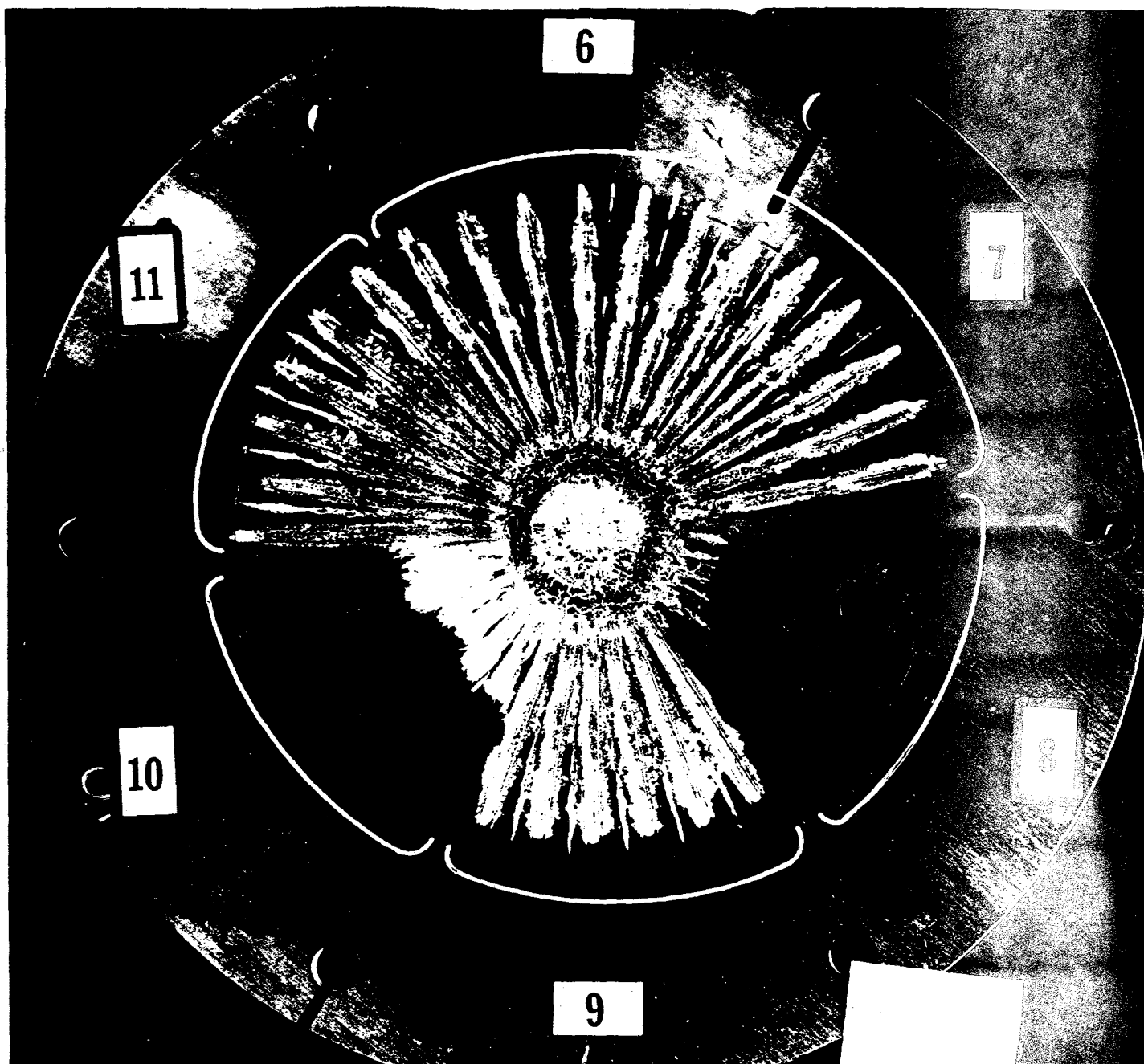
- No. 1, 1288 PETN - Development Extex
No. 2, 1286-2 PETN - Micronized MRL 22A, 8 hrs/116 C
No. 3, 1286-1 PETN - Micronized MRL 22A
No. 4, 1281 PETN - Micronized
No. 5, 1328 PETN - Semi-continuous, Batch Extex
No. 6, 393 PETN - Production Extex

Fig. 11. LLL Sunburst No. IV, 30 hrs/100 C
Witness Plate



- No. 6, 393 PETN - Production Extex
No. 7, 9255 PETN - Micronized
No. 8, 1340-301 PETN - Du Pont Mil Spec
No. 9, 1340-322 RDX - Micronized
No. 10, 1341-322 RDX - Class E
No. 11, 1341-301 PETN - Micronized MRL 22A, 8 hrs/116 C

Fig. 12. LLL Sunburst No. 11 Witness Plate



No. 6, PETN - Production Extex

No. 7, PETN - Micronized

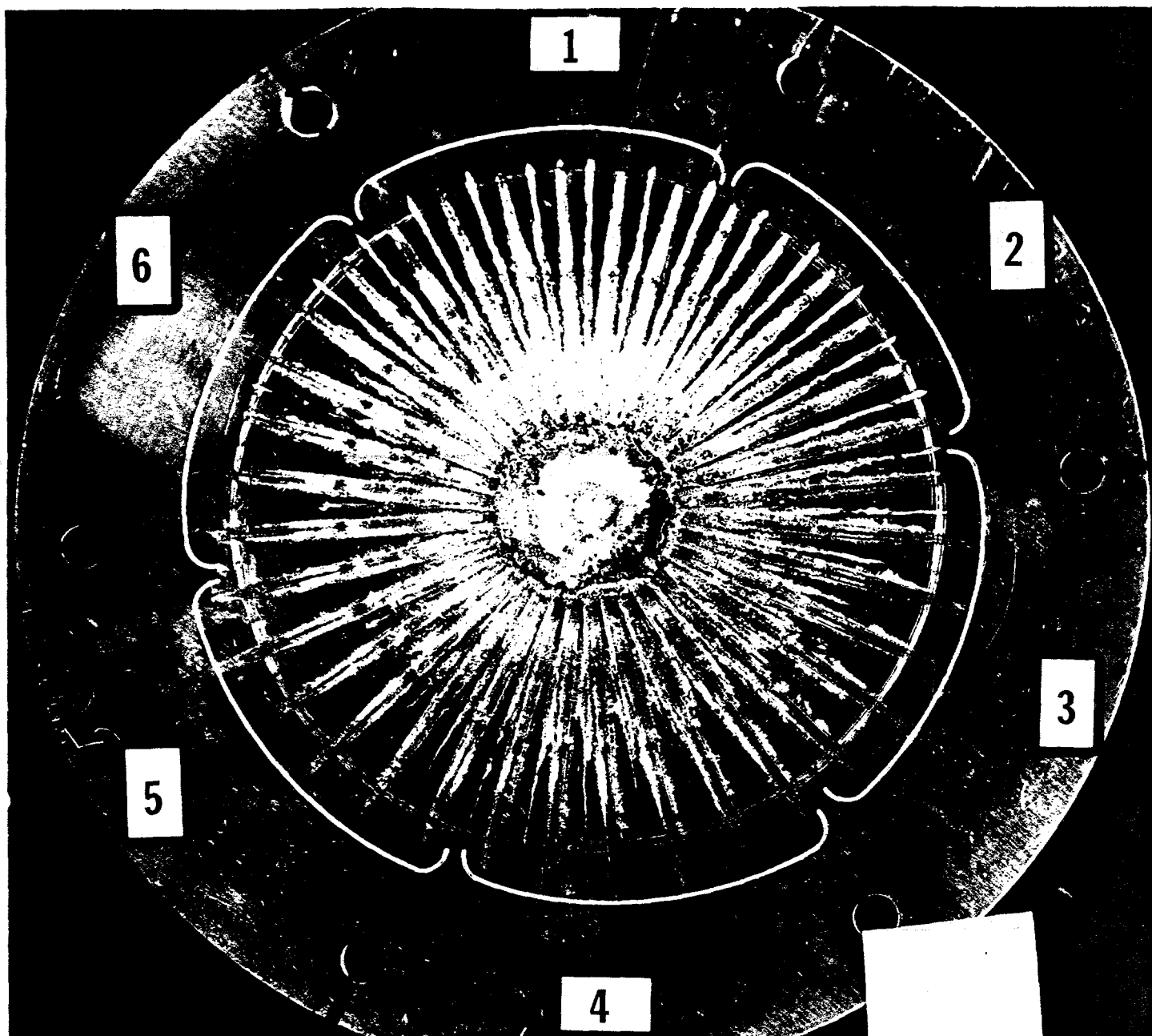
No. 8, 1340-301 PETN - Du Pont Mil Spec

No. 9, 1340-322 RDX - Micronized

No. 10, 1341-322 RDX - Class E

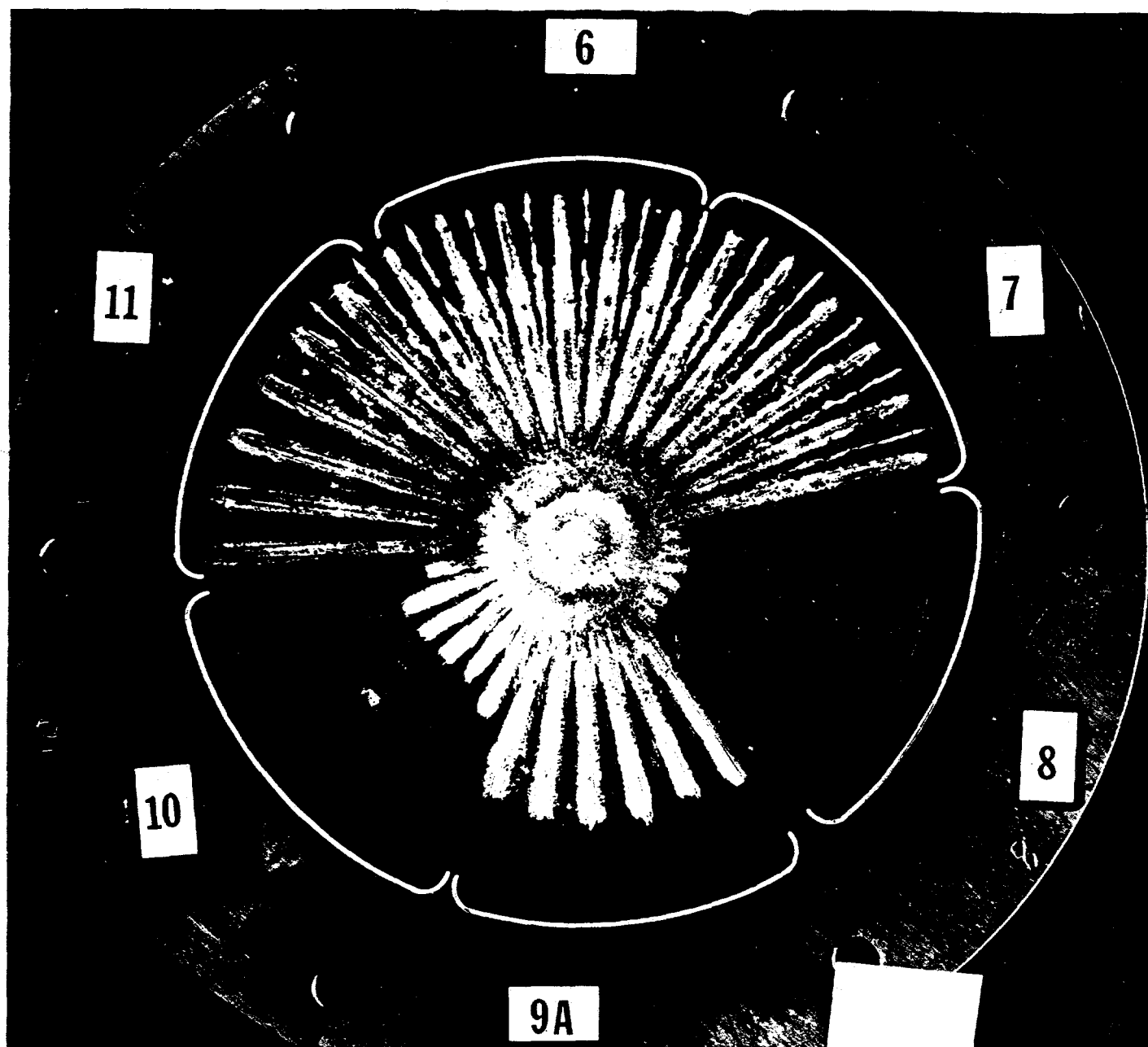
No. 11, 1341-301 PETN - Micronized MRL 22A, 8 hrs/116 C

FIG. 13. ELL Sunburst No. 17, 30 hrs/100 C Witness Plate



- No. 1, 1288 PETN - Development Extex
- No. 2, 1286-2 PETN - Micronized MRL 22A, 6 hrs/116 C
- No. 3, 1288-1 PETN - Micronized MRL 22A
- No. 4, 1281 PETN - Micronized
- No. 5, 1328 PETN - Semi-continuous, Batch Extex
- No. 6, 393 PETN - Production Extex

Fig. 14. LLL Sunburst No. VII, 200 hrs/100 C
Witness Plate

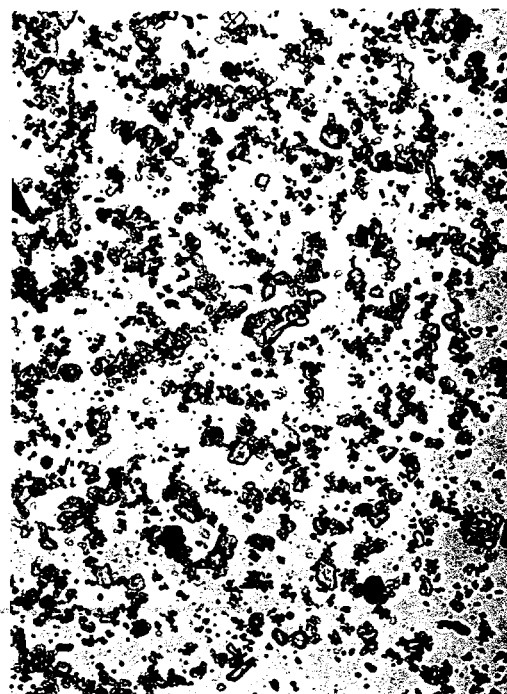


No. 6, 393 PETN - Production Extex
 No. 7, 9255 PETN - Micronized (Low Surface Area)
 No. 8, 1340-301 PETN - Du Pont Mil Spec
 No. 9A, 1356 RDX - Micronized
 No. 10, 1341-322 RDX - Class E
 No. 11, 1341-301 PETN - Micronized NRL 22A, 8 hrs/116 C

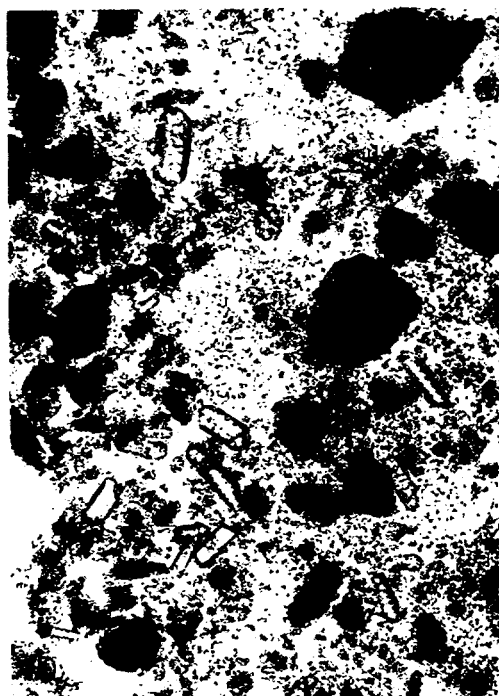
Fig. 15. LLL Sunburst No. VIII, 300 hrs/100 C
 Witness Plate



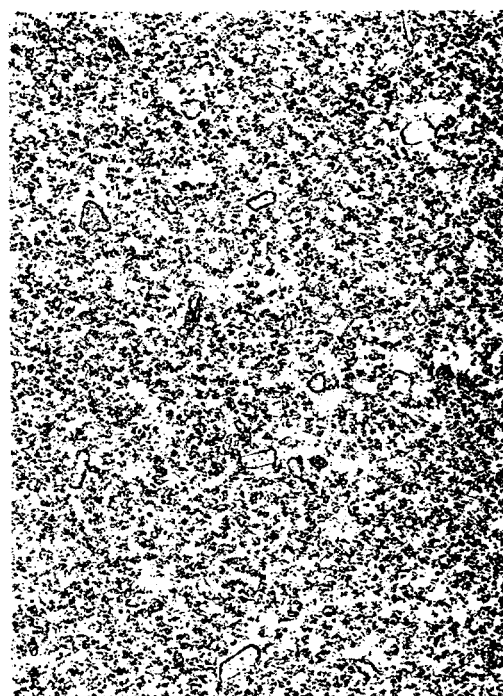
No. 1 PETN 1288 160X
Dev Extex Type So(P)
5,250 cm²/g



No. 1 PETN 1288 160X
Dev Extex Type, w/Syl-
gard

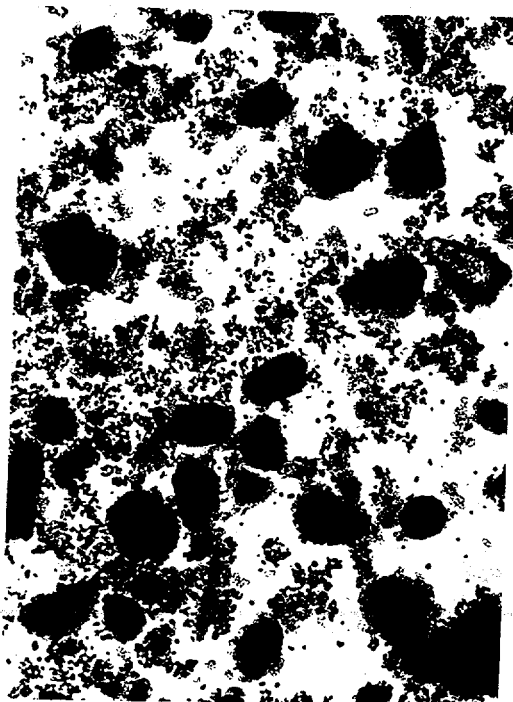


No. 2 PETN 1286-2 160X
Mic - Treated w/MRL 22A,
Heat Treated, 8 hrs @ 116 C
So(P) - 18,600 cm²/g



No. 2 PETN 1286-2 160X
Mic - Treated w/MRL 22A,
Heat Treated, 8 hrs @ 116 C
w/Sylgard

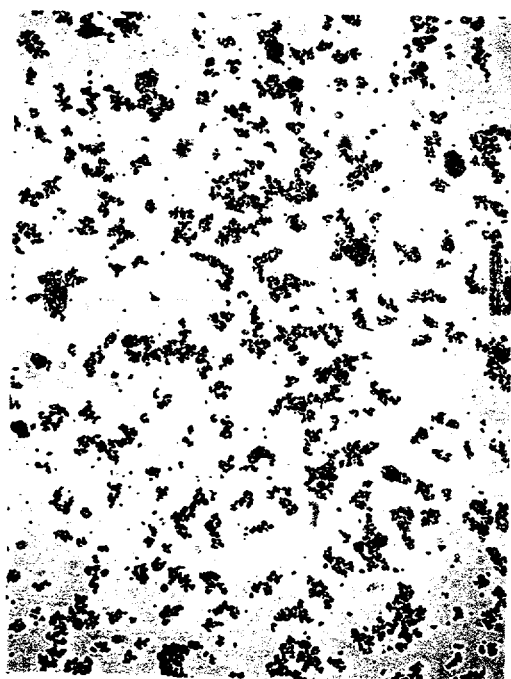
Fig. 16



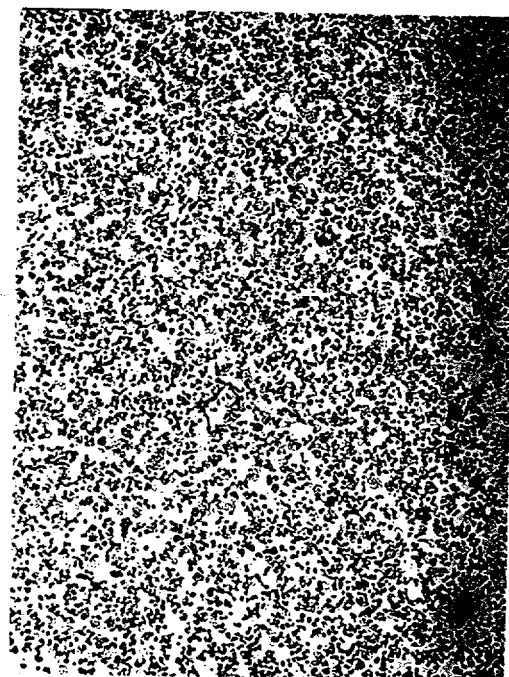
No. 3 PETN 1286-1 160X,
Mic - Treated w/MRL 22A
So(P) - 20,700 cm²/g



No. 3 PETN 1286-1 160X,
Mic - Treated w/MRL 22A
w/Sylgard

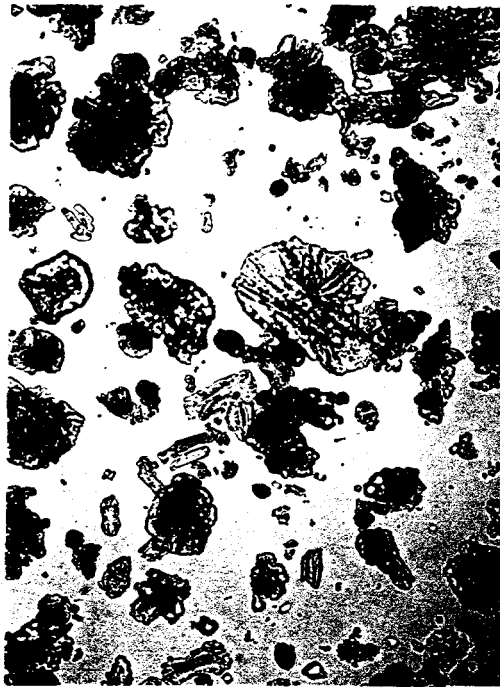


No. 4 PETN 1281 160X
Mic
So(P) - 19,400 cm²/g

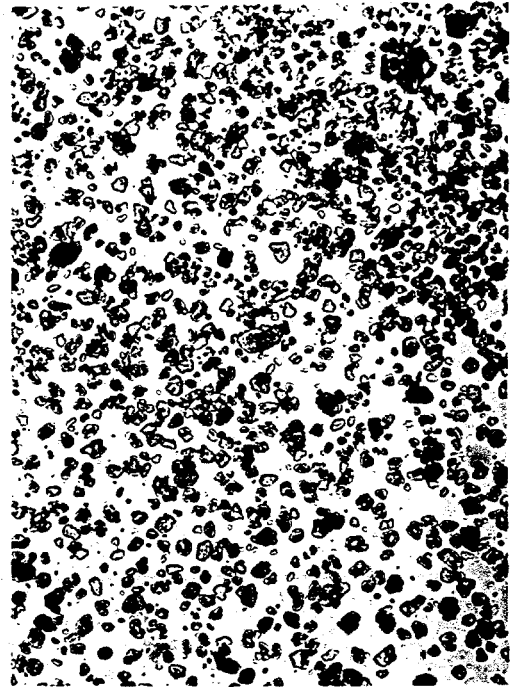


No. 4 PETN 1281 160X, Mic -
Mic
w/Sylgard

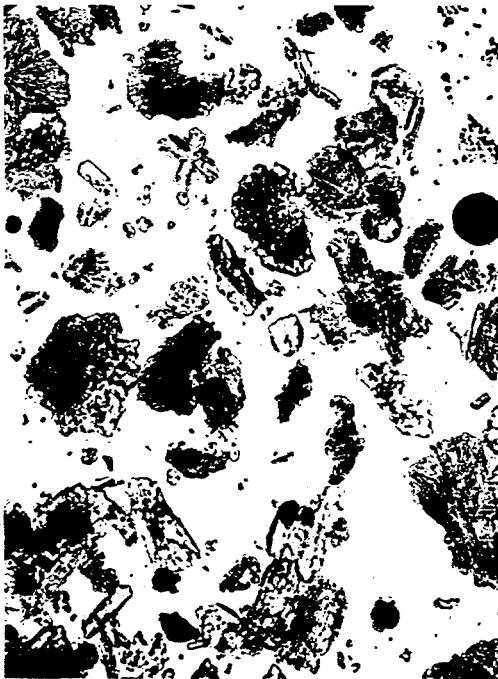
Fig. 17



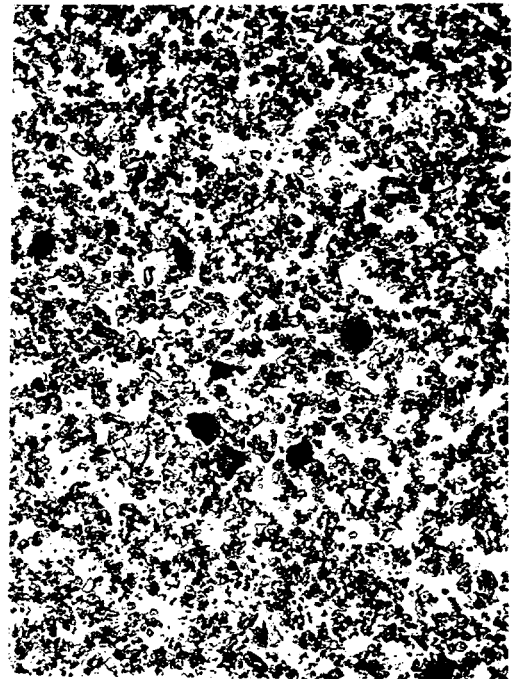
No. 5 PETN 1328 160X, Extex
Type, Semi-continuous, So(P)
4,150 cm²/g



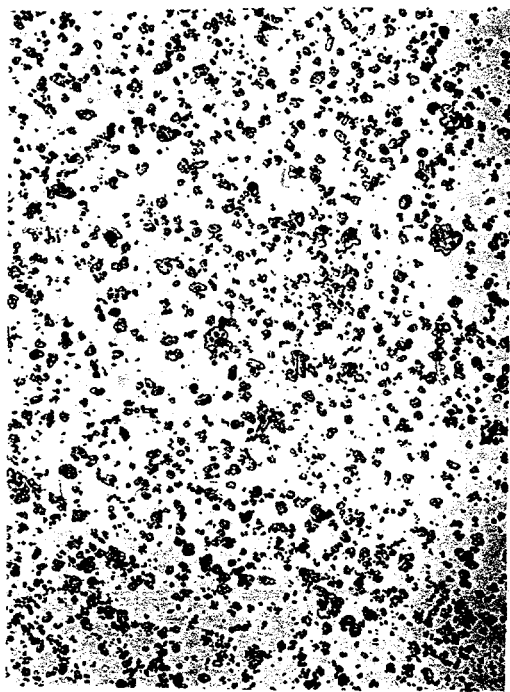
No. 5 PETN 1328 160X, Extex
Type, Semi-continuous, w/Syl-
gard



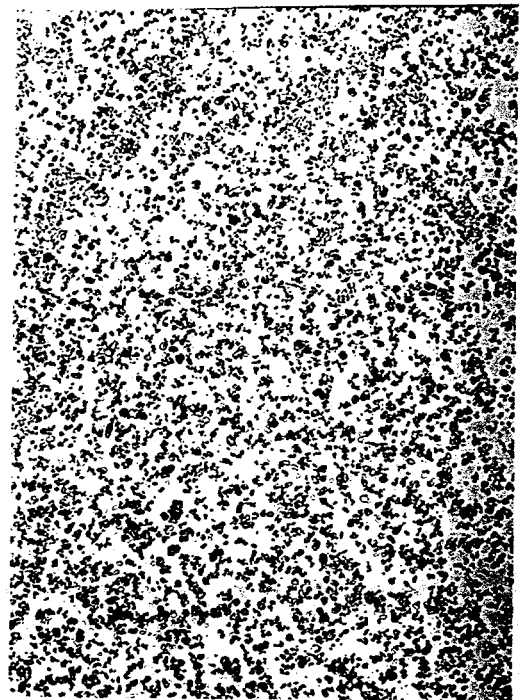
No. 6 PETN 393 160X, Pro-
duction - Extex Type, So(P)
4,850 cm²/g



No. 6 PETN 393 160X, Pro-
duction - Extex Type, w/
Sylgard



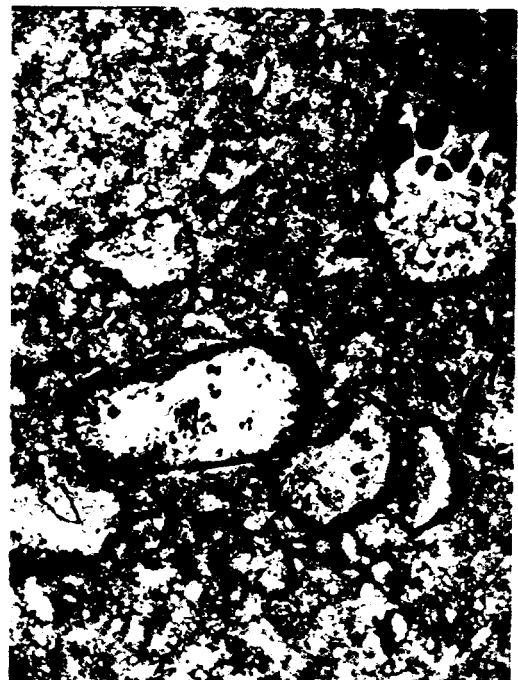
No. 7 PETN 9255 160X, Mic
So(P) - 9,200 cm²/g



No. 7 PETN 9255 160X, Mic
w/Sylgard

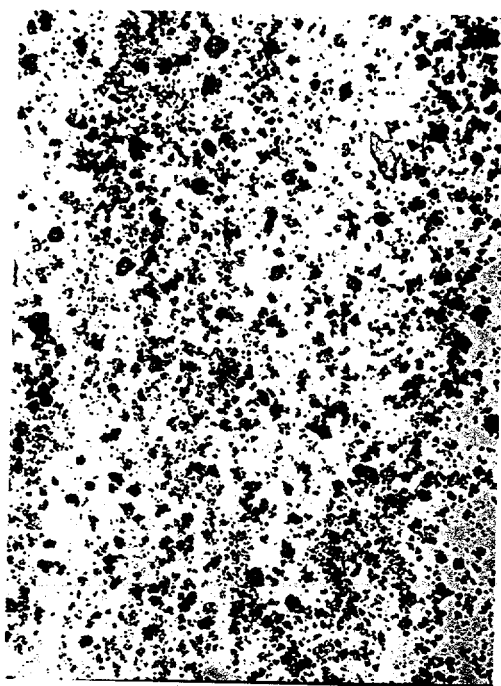


No. 8 PETN 1340-301 160X
Du Pont Mil Spec, So(P) -
600 cm²/g

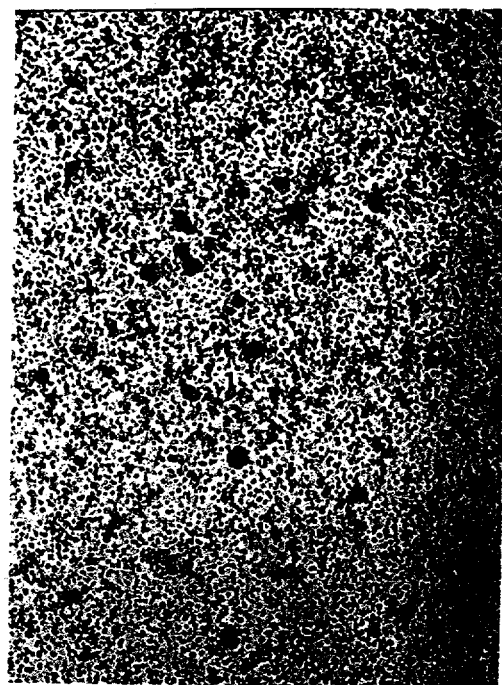


No 8 PETN 1340-301 160X
Du Pont Mil Spec, w/Syl-
gard

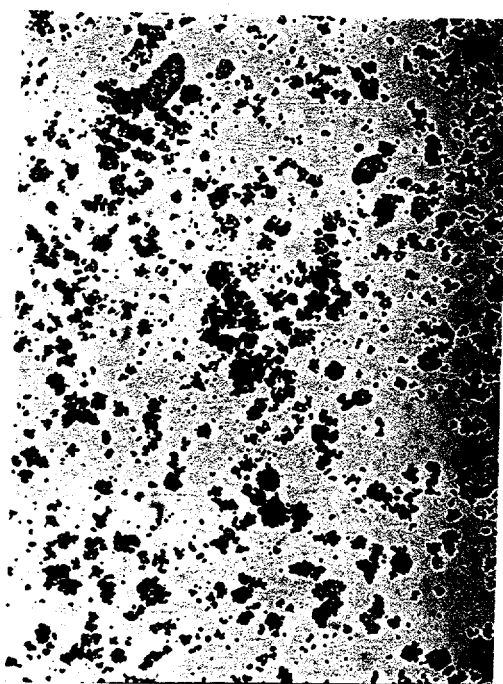
Fig. 19



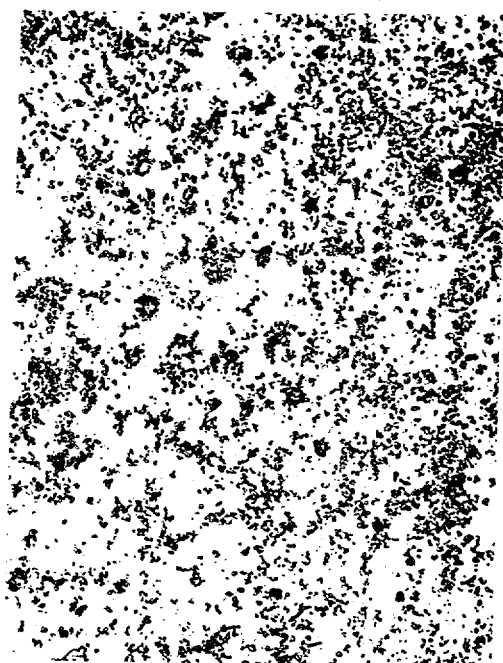
No. 9 RDX 1340-322 160X,
Mic - 5% in Acetone, So(P)
13,900 cm²/g



No. 9 RDX 1340-322 160X,
Mic - 5% in Acetone, w/
Sylgard



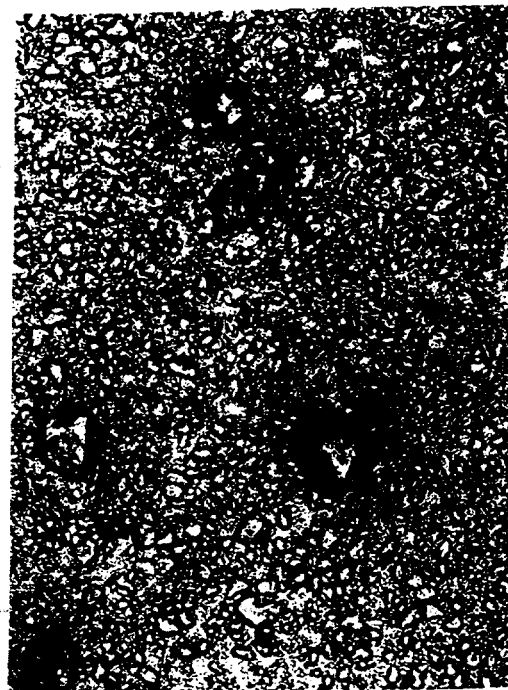
No. 9A RDX 1356 160X, Mic -
2% in Acetone, So(P) -
12,000 cm²/g



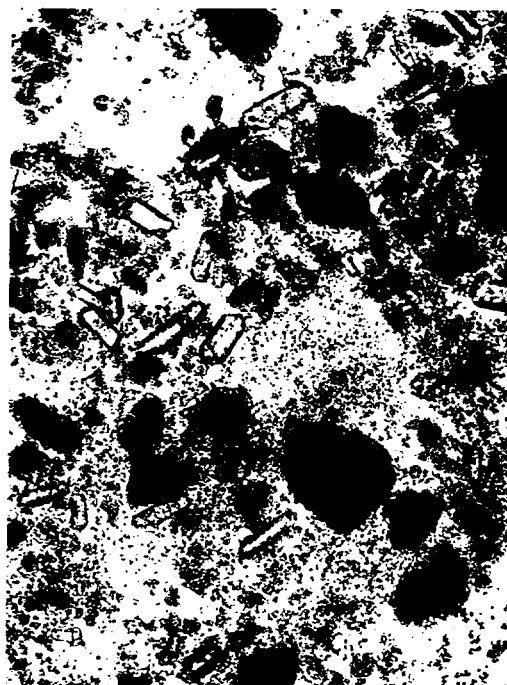
No. 9A RDX 1356 160X, Mic -
2% in Acetone w/Sylgard



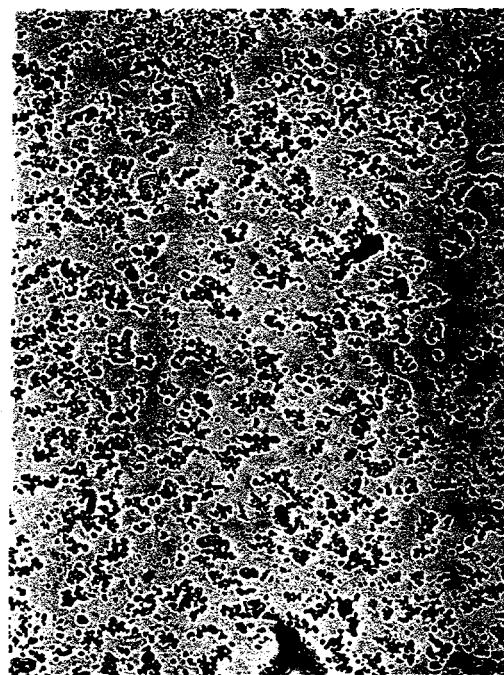
No. 10 RDX 1341 160X, Class
E (42-57), So(P) - 900 cm²/g



No. 10 RDX 1341 160X, Class
E, w/Sylgard



No. 11 PETN 1341 160X, Mic -
Treated w/MRL 22A, Heat
Treated, 8 hrs @ 116 C So(P)
18,600 cm²/g



No. 11 PETN 1341 160X, Mic -
Treated w/MRL 22A, Heat Treated,
8 hrs @ 116 C, w/Sylgard