X-RAY DIFFRACTION OF HNS

C. C. Blair

DEVELOPMENT DIVISION

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Normal Process Development
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ABSTRACT

This report presents X-ray diffraction data obtained from HNS (hexanitrostilbene) recrystallized from various solvents. The same X-ray diffraction patterns were observed for all of the HNS samples except dioxane.

DISCUSSION

The data reported in this report are in degrees of two theta angle, using a copper target tube and graphite crystal monochromator.

Table I contains X-ray data obtained from Chemtronics Lot Nos. 66-48 HNS-I and 66-47 HNS-II. The only difference observed between the two samples by X-ray diffraction is the weak peak at 22.66 in the HNS-II sample. This peak may be due to an impurity in the solvent which aided in the growth of the HNS-II crystals.

Table II contains data obtained from Lot Nos. EB-5737-01 (HNS-I) and EB-5737 (HNS-II), both from Ensign-Bickford. There are several extra lines observed in the HNS-I sample.

Table III lists the twenty values obtained from HNS recrystallized from nitric acid. Patterns obtained from HNS recrystallized, respectively, from DMF, toluene, acetonitrile, and dioxane, are shown in Table IV. Apparently the dioxane was complexed with the HNS. Table V is the HNS/dioxane material after being heated for four hours at 100 C and 550 mm pressure. This material yields basically the same pattern as the HNS from the other solvents.

CONCLUSION

All of the solvents appear to yield HNS of the same crystal structure. The only basic difference appears to be in the crystal size. HNS-I and HNS-II are not polymorphs. The morphology of HNS-I and -II is different, but this habit variation is caused by something external.

Table I. Comparison of Chemtronics HNS-I and HNS-II
by X-Ray Diffraction

<u>Line No.</u>	<u>HNS-II Lot 66-47</u>	<u>HNS-I Lot 66-48</u>
1	14.78	14.78
2	16.90	16.95
3	18.08	18.07
4	20.04	20.05
5	20.70	20.71
6	21.00	21.02
7	21.80	21.80
8		22.66
9	23.30	23.32
10	23.80	23.75
11	24.35	24.30
12	24.57	24.60
13	25.51	25.53
14	26.32	26.25
15	27.35	27.33
16	27.63	27.68
17	28.20	28.15
18	28.62	28.60
19	29.02	29.10
20	30.10	30.18
21	30.80	30.82
22	31.10	31.18
23	32.15	32.12
24	32.40	32.45
25	34.15	34.22
26	36.25	36.20
27	36.50	36.56
28	37.80	37.90
29	40.50	40.50
30	41.95	41.90
31	42.37	42.35
32	44.86	44.80
33	49.18	49.20
34	52.15	52.35
35	58.15	58.15

Table II. Comparison of Ensign-Bickford HNS-I
and HNS-II by X-Ray Diffraction

<u>Line No.</u>	<u>HNS-II Lot No. 5737</u>	<u>HNS-I Lot No. 5737-01</u>
1		12.75
2	14.75	14.78
3	16.85	17.00
4	18.02	18.06
5	20.02	20.05
6	20.72	20.72
7		21.00
8		21.22
9	21.75	21.78
10		22.62
11	23.27	23.32
12	23.71	23.76
13	24.30	24.30
14		24.60
15	25.45	25.55
16	26.25	26.30
17	27.45	27.35
18		27.68
19		28.10
20	28.62	28.62
21	29.00	29.08
22		29.45
23		29.85
24	30.05	30.18
25	31.18	31.18
26	32.18	32.12
27	34.20	34.22
28	36.18	36.19
29		36.55
30	37.78	37.90
31		39.25
32	40.40	40.45
33	41.90	41.87
34	42.30	42.40
35	44.75	44.75
36	49.10	49.15

Table III. X-Ray Diffraction Data of HNS-II
Recrystallized from Nitric Acid

<u>Line No.</u>	<u>Recrystallized Nitric Acid</u>
1	14.75
2	16.93
3	18.02
4	20.00
5	20.65
6	21.06
7	21.75
8	22.60
9	23.25
10	23.68
11	24.40
12	25.48
13	26.18
14	27.25
15	27.60
16	28.58
17	28.98
18	30.12
19	30.75
20	31.10
21	32.30
22	34.15
23	35.10
24	36.10
25	36.50
26	37.86
27	40.42
28	41.80
29	42.35
30	44.65
31	45.08
32	46.15
33	46.60
34	47.73
35	49.10
36	49.50
37	52.30
38	58.15
39	58.70

Table IV. X-Ray Diffraction Data of HNS-II Recrystallized from Various Solvents

Line No.	DMF	Toluene	Acetonitrile	Dioxane
1	14.78	14.75	14.75	13.80
2	16.97	16.92	16.90	14.23
3	18.03	18.05	18.00	17.58
4	20.02	20.05	20.00	19.05
5	20.70	20.80	20.65	20.18
6	21.00	21.80	20.97	20.72
7	21.80		21.75	22.12
8	22.60	23.30	22.55	23.05
9	23.28	23.80	23.25	23.58
10	23.70	24.32	23.75	24.02
11	24.35	24.65	24.30	24.70
12	25.55	25.50	24.53	25.48
13	26.25	26.25	25.48	26.80
14	27.30	27.48	26.20	27.08
15	27.65	28.65	27.27	27.72
16	28.60	29.00	27.60	28.63
17	29.03	30.15	28.55	29.02
18	30.15	30.82	29.00	30.10
19	30.82	31.15	30.12	30.55
20	31.13	32.30	30.75	31.04
21	32.32	34.10	31.12	32.25
22	34.18	36.25	32.10	33.05
23	35.11	37.85	32.42	34.82
24	36.11	40.50	34.18	36.32
25	36.55	41.97	36.20	37.72
26	37.90	42.42	37.90	39.40
27	40.48	44.85	40.48	40.90
28	41.90	46.65	41.86	42.00
29	42.40	49.15	42.40	42.70
30	44.75	52.20	44.72	46.20
31			46.65	
32	46.22		49.18	
33	46.63		52.30	
34	47.72			
35	49.20			
36	49.60			
37	52.32			
38	58.12			
39	58.72			

Table V. X-Ray Diffraction Data for HNS-II
Recrystallized from Dioxane After
Heated for 4 Hours at 100 C and
550. mm

<u>Line No.</u>	<u>HNS/Dioxane</u>	
1	14.76	
2	16.84	
3	18.04	
4	20.06	
5	20.78	
6	21.80	
7	23.26	
8	23.74	
9	24.34	
10	Missing	
11	25.60	
12	27.55	Doublet
13	28.70	
14	29.20	
15	30.08	
16	32.15	
17	32.45	
18	34.00	
19	36.18	
20	37.75	
21	40.60	
22	42.20	
23	44.65	
24	46.70	