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DEVELOPMENT DIVISION

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Process Development
Endeavor No. 107

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ABSTRACT

This report details the analytical results of six lots of high purity 2,2´,4,4´,6,6´-hexanitrostilbene (HNS I) synthesized by the Development Division, Mason & Hanger, Pantex Plant. A total of approximately 130 ~~kilograms~~ (kg) was produced in lot sizes ranging from 5.2 to 46.4 kg. The results clearly indicate the excellent reproducibility of the process and high purity of the final product.

DISCUSSION

The continuous process used for synthesizing high purity HNS I has been described in an earlier report(*1*). Basically, the system described in the above report was scaled-up to 3 kg of raw HNS I produced in approximately 4 hours. The filtered raw product from two such runs were then combined and processed to "crude" HNS I. Laboratory tests were then performed on the crude batches before their combination for subsequent processing to "finished" HNS I lots. The amount of crude material combined for further processing is dependent on two factors. These factors are: (1) the desired lot size, and (2) the size of the slurrying equipment which presently limits the "finished" lot size to 20 kg. When the lot size was 20 kg or less, the "finished" lot was considered final material and tested per Sandia Specification SLA 5003C. If the desired final lot size exceeded 20 kg, the "finished" lots were sampled and in process tests made prior to wet blending the finished HNS I into a blended lot.

After blending (final lot size limited to 50 kg) the material was sampled and tested using the above Sandia specification. The number of samples analyzed per lot of material was determined by lot size, dictated by specification and were as follows:

Lot Size (lbs)	Lot Size (kg)	Number of Samples
2-16	0.9- 7.3	2
17-54	7.7-24.5	3
55-128	25.0-58.2	4

(1) *T. W. Stull, Synthesis of High Purity Hexanitrostilbene, MHSMP-75-37 (September 1975).*

Two specifications available for HNS I are the Navy WS 5003F and the previously mentioned Sandia document. The Sandia specification test requirements differed from the Navy document in that: Allowable water soluble maximum was reduced from 0.20 to 0.03 weight percent. Allowable dimethyl formamide (DMF) insoluble maximum was reduced from 0.10 to 0.03 weight percent.

Additional tests included in the Sandia specification for DMF insoluble particles, conductivity and pressure density were adopted. The analytical results of the six lots in Table I are listed in order of date of manufacture with Lot 7157-07C-001 being the first lot produced after equipment installation. The major deviation in the results occurred in this lot. This deviation is attributed to first efforts with the process equipment. The learning curve is apparent and close inspection of the remaining data indicates the curve levels out with Lot 7231-07C-003.

All analytical test methods are straightforward and well defined in the Sandia specification. The melting point (MP), MP range, vacuum stability, conductivity and liquid chromatography (LC) data indicate a general product improvement up to and including Lot 7312-07C-003. Water soluble, DMF insolubles, pressure density, DMF insoluble particles and output tests are well within specification limits and indicate a very uniform material.

Further discussion of tests and test methods will be limited to areas where it is felt clarification will benefit.

Table I. Analytical Results on Six Lots of High Purity HNS I

Analytical Tests	Lot Numbers 7157-07C-001	7231-07C-002	7237-07C-001	7312-07C-003	8017-07C-001	8191-07C-001	Specification Limits
Lot Size (kg)	10.9	46.4	5.2	33.6	9.1	25.0	N/A
Melting Point (C)	316.0	316.6	316.7	317.9	317.9	317.4	Not less than -2 C of melting point of SCS*
Melting Point Range (C)	1.2	1.2	0.7	0.9	0.7	0.5	Shall not exceed that of SCS.
Vacuum Stability (ml/g)							
First 20 Minutes	0.53	0.41	0.41	0.30	0.40	0.35	3.0 Maximum
Additional 2 Hours	0.28	0.26	0.29	0.24	0.27	0.18	1.1 Maximum
Water Solubles (Wt. %)	0.01	0.01	0.01	0.02	0.01	0.02	0.03 Maximum
DMF Insolubles (Wt. %)	0.01	0.01	0.02	0.01	0.01	0.01	0.03 Maximum
Liquid Chromatography % HNS	99.6	99.9	99.9	100.0	99.9	99.9	Information Only
Pressure Density (g/cc)							
3 Kpsi	1.16	1.10	-	1.16	1.15	-	Information Only
16 Kpsi	1.50	1.45	-	1.48	1.48	-	
32 Kpsi	1.60	1.58	-	1.60	1.57	-	
DMF Insoluble Particles Number Retained on							
USS 40 Mesh Sieve	0.66	0.0	0.0	0.25	0.0	0.25	Average less than 1 Max
USS 60 Mesh Sieve	1.3	0.25	0.0	1.8	2.0	0.25	Average 5 Max
Conductivity (Mhos x 10^{-6})	2.2	1.2	0.5	0.5	1.6	1.4	Less than 1 ppm NaCl**
Output Test Plate Dent (In)	0.049	0.049	-	0.047	-	-	0.042 Min.

N/A = Not Applicable
- = Not Analyzed
SCS = Standard Comparison Sample

**Standard comparison sample supplied by the Quality Evaluation Laboratory (QEL) Naval Ammunition Laboratory (NAD), Crane, Ind. (SCS melting point - 310.6 C, melting point range - 1.9 C).*

***Conductivity of 1 ppm NaCl approximately 3.2 x 10^{-6} mhos.*

NOTE: The results listed above are the averaged values of the number of samples taken per lot.

The liquid chromatography (LC) test(2), while not required by specification, is presently the best means of assessing the purity of HNS I. The LC column used provides good separation of the major known impurities (dipicrylethane, trinitrobenzyl chloride and trinitrotoluene). The results, however, should not be considered absolute as, there is a remote possibility that other impurities are present and not separated. When tested using this method, the average purity of the six lots was 99.87 percent with the last five lots being 99.9 percent pure and above. (Commercially available HNS purity ranges from 93 to 98 percent.)

Of all tests required, the most difficult requirement is the DMF insoluble particle limits. A 25 g sample is dissolved in DMF and the solution passed through a two sieve nest (USS 40 and 60 mesh). All foreign particles trapped on those sieves are counted and photographed. To pass the specification requirements of (1) less than an average of one particle on the USS 40, and (2) a maximum average of five on the USS 60, is difficult. Accomplishment of this requires extreme cleanliness in all process operations, special filtering techniques and very clean laboratory conditions when performing the analysis.

The only analysis performed and not listed in Table I is gap sensitivity. This was performed on four lots of material only and all met specification requirements. All six lots of HNS I passed every Sandia specification test to which they were subjected without any type of rework employed.

CONCLUSIONS

The Pantex process for synthesizing hexanitrostilbene yields a very reproducible product of high purity, meets all specification requirements and is superior to commercially available material.

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(2) *C. L. Schaffer, HNS Assay by Liquid Chromatography, MHSMP-77-51 (July-September 1977).*

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