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CYCLIC RECRYSTALLIZATION OF
SANDIA FURNISHED HNS I

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MASTER

DEVELOPMENT DIVISION

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For
Sandia Laboratories
Albuquerque, New Mexico
(P.O. No. 03-7008)

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ABSTRACT

The Pantex HNS recrystallization process followed by a crystal altering step was used to modify 4.77 kg of Sandia furnished HNS IB. The resulting HNS II (2.04 kg) was evaluated by the Navy specification WS-5003 as modified by Sandia Laboratories.

- The HNS II (Lot 6201-137-01) was shipped to Sandia.

DISCUSSION

A cyclic recrystallization of Sandia furnished HNS IB was conducted in the existing 200-gallon system. The 4.77 kg batch was approximately the minimum which could be processed. This resulted in an inefficient operation but the recovered product should have been subjected to as nearly as possible identical processing and handling environment as a standard cyclic recrystallized product.

The process involved complete solution of 4.77 kg of HNS in 64 litres of dimethylformamide (DMF) to 105 C. An additional 5 litres of DMF was heated to 105 C in a separate vessel and used to preheat the filtration system. The HNS solution was then passed through

the filtration system which had 1.5 μ m elements. The 74 litres of DMF containing the 4.77 kg of HNS was received in the 200-gallon recrystallization vessel. The solution was then heated to 105 C and temperature cycled. Fig. 1 shows the temperature time relationship for the cycles and cool down. After cooling the product was isolated in a plate and frame filter press, rinsed with 90 litres of acetone and dried at steam temperature for 2.5 hours.

The product was then transferred to a 30-gallon slurry mixer containing 55 litres of acetone. The slurry was continued for 30 minutes at 100 rpm. The product was again isolated in the filter press and dried at steam temperature for 2.5 hours.

The HNS crystals were then altered in a Cowles Dissolver using 1:1 water methanol as the working fluid. The process was run for 5 minutes at 2400 rpm. After oven drying a yield of

2.05 kg of HNS II was recovered. Two samples were analyzed. Table I lists the analytical results and a comparison with the Sandia modified Navy WS-5003 specifications.

Fig. 1. Cyclic Recrystallization of HNS

Table I. Analytical Data Lot 6201-137-01

Test	Data	Specification Limits
Melt Point	316.0 C	> 314.1 C
Melt Range	1.0 C	< 1.1 C
Vacuum Stability		
First 20 min	0.62 ml/g	< 0.6 ml/g
Additional 120 min	0.30 ml/g	< 0.6 ml/g
Surface Moisture	0.001 Wt. %	< 0.05 Wt. %
Water Solubles	0.026 Wt. %	< 0.03 Wt. %
DMF Insolubles	0.015 Wt. %	< 0.03 Wt. %
Insoluble Particle		
On 40 USS Mesh	7	< 1
On 60 USS Mesh	33	≤ 5
Conductivity	1.1×10^{-6} mho	< 3.0×10^{-6} mho
Bulk Density	0.56 g/cc	> 0.45 g/cc
Pressure Density		
At 3 Kpsi	1.36	Information Only
At 16 Kpsi	1.55	Information Only
At 32 Kpsi	1.63	Information Only