THE DETERMINATION OF MINIMUM SET
TIME AT AMBIENT TEMPERATURE FOR REACTING
TRINITROBENZYL CHLORIDE TO HEXANITROSTILBENE
IN THE CONTINUOUS HNS I SYNTHESIS PROCESS

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DEVELOPMENT DIVISION

APRIL - JUNE 1976

*Normal Process Development
Endeavor No. 107*

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ABSTRACT

The present continuous HNS I synthesis process defines a minimum period of 18 hours at ambient temperature for reacting trinitrobenzyl chloride to hexanitrostilbene. This effort was undertaken to determine the minimum time at ambient temperature for this reaction to be essentially complete. It was determined that the reaction is essentially complete at the end of a 6 hour time period.

DISCUSSION

The continuous synthesis process for the synthesis of 2,2'-, 4,4'-, 6,6'-hexanitrostilbene has been described in an earlier report(1). The two step reaction of the process is detailed in a Navy report(2). The process basically consists of metering two separate solutions, (1) trinitrotoluene (TNT) dissolved in a 2/1 mixture of tetrahydrofuran (THF) and methanol (MeOH), and (2) sodium hypochlorite (NaOCl) into a common junction and cooling coil held at a temperature of 0-5 C. The first reaction occurs in the cooling coil and converts TNT to trinitrobenzyl chloride. The effluent from the coil containing the product and excess solvent is allowed to react at ambient temperature for a minimum period of 18 hours in which time the trinitrobenzyl chloride is reacted to HNS.

This effort was undertaken to define at what point within the set time the second reaction was essentially complete. Samples for the evaluation were obtained by collecting the coil effluent at 6 minute time intervals. These 6 minute samples were then allowed to set at ambient temperature for 0.5, 1, 2, 4, 6 and 18 hour periods before filtering was initiated.

Duplicate samples were run at each time interval and all samples were processed identically to the "crude" HNS I stage. The crude samples were then dried at 100 C for 16 hours, cooled, weighed to determine yield and then sampled for liquid chromatography (LC) analysis.

(1) T. W. Stull, *A Continuous Process For The Synthesis of Hexanitrostilbene*, MHSMP-75-5F, Oct-Dec 1974

(2) K. G. Shipp, *Heat Resistant Explosive*, NOLTR 64-34 (April 22, 1964)

Table I lists the yield and LC results for all samples. Yield at the various time intervals is the most significant evaluation factor because at this time LC results cannot be considered absolute and have significant scatter due to the difficulty of sampling a "crude" product. Yield results show that the reaction is essentially complete at the 6 hour period.

CONCLUSIONS

This work has determined that the second reaction (conversion of trinitrobenzyl chloride to hexanitrostilbene) is approximately 99 percent complete at the end of 6 hour "set time" at ambient temperature. If time becomes an important factor, the now prescribed 18 hour set time could be reduced to 6 hours without significantly effecting the overall yield.

Table I. Yield and Purity of HNS I as a Function of Set Time in Reacting Trinitrobenzyl Chloride to Hexanitrostilbene

<u>Reaction Set Time (hrs)</u>	<u>Yield (%)</u>	<u>HNS I (%)</u>
0.5	32.5	92.3
1.0	42.0	92.0
2.0	43.2	93.8
4.0	43.4	92.2
6.0	43.6	93.5
18.0	43.8	92.4