COMPARATIVE EFFICIENCY OF ACETONE
AND METHANOL FOR PURIFYING RAW SYNTHESIZED
2,2', 4,4', 6,6' HEXANITROSTILBENE

T. W. Stull

DEVELOPMENT DIVISION

APRIL - JUNE 1976

Normal Process Development
Endeavor No. 107

MASTER

Mason & Hanger-Silas Mason Co., Inc.
Parlex Plant
P. O. BOX 647
AMARILLO, TEXAS 79177
806-335-1581

operated for the
ENERGY RESEARCH AND DEVELOPMENT ADMINISTRATION
under
U. S. GOVERNMENT Contract DA-11-173-AMC-487(A)

DISTRIBUTION OF THIS DOCUMENT IS UNLIMITED

DISCLAIMER

This report was prepared as an account of work sponsored by an agency of the United States Government. Neither the United States Government nor any agency Thereof, nor any of their employees, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness, or usefulness of any information, apparatus, product, or process disclosed, or represents that its use would not infringe privately owned rights. Reference herein to any specific commercial product, process, or service by trade name, trademark, manufacturer, or otherwise does not necessarily constitute or imply its endorsement, recommendation, or favoring by the United States Government or any agency thereof. The views and opinions of authors expressed herein do not necessarily state or reflect those of the United States Government or any agency thereof.

DISCLAIMER

Portions of this document may be illegible in electronic image products. Images are produced from the best available original document.

NOTICE

This report was prepared as an account of work sponsored by the United States Government. Neither the United States nor the United States Energy Research and Development Administration, nor their employees, nor any of their contractors, subcontractors, or their employees, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness or usefulness of any information, apparatus, product or process disclosed, or represents that its use would not infringe privately-owned rights.

COMPARATIVE EFFICIENCY OF ACETONE AND METHANOL
FOR
PURIFYING RAW SYNTHESIZED 2,2', 4,4', 6,6' HEXANITROSTILBENE

T. W. Stull

DEVELOPMENT DIVISION

April - June 1976
Endeavor No. 107

NOTICE

This report was prepared as an account of work sponsored by the United States Government. Neither the United States nor the United States Energy Research and Development Administration, nor any of their employees, nor any of their contractors, subcontractors, or their employees, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness or usefulness of any information, apparatus, product or process disclosed, or represents that its use would not infringe privately owned rights.

MASTER

H-1

DISTRIBUTION OF THIS DOCUMENT IS UNLIMITED

ABSTRACT

The purpose of this effort was to determine the washing efficiency of methanol as compared to acetone when used for purification of raw synthesized 2,2', 4,4', 6,6' hexanitrostilbene (HNS I). This study revealed that methanol is slightly inferior to acetone when used for this purpose.

DISCUSSION

The present method used for purification of raw synthesized 2,2',4,4', 6,6' hexanitrostilbene (HNS) utilizes acetone, 95 C distilled water and dimethylformamide (DMF) for product cleanup. Methanol was proposed as a possible substitute for the acetone portion of the solvent mixture. This was suggested as a means of improving the safety aspects of the whole process as methanol has a higher flash point and a higher explosive threshold limit than that of acetone.

This endeavor was undertaken to compare the efficiency of the two solvents in question. The raw product to accomplish this comparison was obtained using a one kilogram continuous synthesis system described in a previous report(1). The raw product from this system was subdivided into four individual batches by collecting the effluent at four approximately 16-minute intervals. Two of these batches were processed normally (using acetone) and with the remaining two batches, methanol was substituted for the acetone. The normal process for purification involves initial filtering of the reaction mixture and the following wash and slurry cycles:

<u>Cycle No.</u>	<u>Wash Solvent</u>	<u>Volume/Wash (Litres)</u>
1, 2, 3 & 4	Acetone	0.85
5, 6 & 7	95 C Distilled Water	1.3
8, 9 & 10	Acetone	0.85

NOTE: At this point the material is labelled "Crude" HNS I, dried at 100 C for a minimum period of 16 hours, removed from the oven, cooled, blended and sampled. The remaining material is then slurried and rewashed as follows:

11 & 12	DMF 70 C*	0.5
13, 14 & 15	Acetone	0.85
16, 17, 18 & 19	95 C Distilled Water	1.3
20 & 21	Acetone	0.85

**The crude HNS is slurried with DMF at 70 C for a period of one to five minutes and allowed to cool to 30 - 35 C before filtering*

Note: At the end of the 21st cycle the material is again dried, cooled, blended and sampled.

(1) MHSMP-75-37, Synthesis of High Purity Hexanitrostilbene

Two methods for evaluation were employed, liquid chromatography (LC) and yield, both at the crude and finished HNS I levels. Results of these evaluations are listed in Tables I and II. The LC analysis method is in the process of being refined and as yet, results cannot be considered absolute. However, at this time it is the most accurate means available for determining the relative purity of HNS samples.

The LC results for crude HNS samples indicate that acetone is definitely superior to methanol as a washing medium. However, when viewing finished HNS I results and yield figures it is apparent that the margin of acetone's superiority is significantly reduced.

CONCLUSIONS

Results of this study show that the two solvents are very close to being equal in their ability to purify raw HNS I. Acetone has a slight advantage in that the final purity is slightly above that of the methanol washed material.

Table I. Yield Details.

	Acetone Washed		Methanol Washed	
	<u>Sample 1</u>	<u>Sample 3</u>	<u>Sample 2</u>	<u>Sample 4</u>
Synthesis Effluent Weight (kg)	12.601	12.743	12.474	12.729
Equivalent TNT Weight Supplied (kg)	0.511	0.517	0.506	0.517
Crude Yield (kg)	0.221	0.224	0.221	0.229
Crude Yield (%)	<u>42.2</u>	<u>43.3</u>	<u>43.7</u>	<u>44.3</u>
Finished Yield (kg)	0.177	0.178	0.177	0.181
Finished Yield (%)	34.6	34.4	35.0	35.0

Table II. Liquid Chromatography Results

	Acetone Washed		Methanol Washed	
	<u>Sample 1</u>	<u>Sample 3</u>	<u>Sample 2</u>	<u>Sample 4</u>
CRUDE HNS I				
% Trinitrobenzene	0.3	0.3	0.5	0.6
% Hexanitrobibenzyl	2.2	2.4	3.6	4.0
% HNS I	97.5	97.4	95.9	95.4
% HNS I Average	97.4		95.6	
FINISHED HNS I				
% Trinitrobenzene	0.6	0.7	0.6	0.8
% Hexanitrobibenzyl	1.2	0.7	1.3	0.8
% HNS I	98.2	98.6	98.0	98.5
% HNS I Average	98.4		98.2	