27
10-25-77
25ing to NTIS

MHSMP-77-24
Dist. Category UC-45

MASTER

CHLORINE FREE SYNTHESIS OF TATB

Zach L. Estes

DEVELOPMENT DIVISION

JULY 1977

For
Lawrence Livermore Laboratory
Livermore, California
SANL No. 500-017

Mason & Hanger-Silas Mason Co., Inc.
Parlex Plant

P. O. BOX 30020
AMARILLO, TEXAS 79177
806-335-1581

operated for the
ENERGY RESEARCH AND DEVELOPMENT ADMINISTRATION
under
U. S. GOVERNMENT Contract EY 76-C-04-0487

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.

Printed in the United States of America
Available from

National Technical Information Service

U. S. Department of Commerce

5285 Port Royal Road

Springfield, VA 22161

Price: Printed Copy \$~~3.50~~; Microfiche \$2.25

4. 2

CHLORINE FREE SYNTHESIS OF TATB

Zach L. Estes

DEVELOPMENT DIVISION

For
Lawrence Livermore Laboratory
Livermore, California
SANL No. 500-017

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.

INTRODUCTION

The principal objective of this endeavor is to produce TATB that is free of the potentially corrosive ammonium chloride. Previous attempts to synthesize a chlorine free precursor to TATB have resulted in difficult to separate mixtures which are the result of side reactions. A new variation of the present process has been tried on a laboratory scale which appears to produce TATB with one to two orders of magnitude less ammonium chloride than the standard process. Several other possible synthesis routes to totally chlorine free TATB were tried without success.

DISCUSSION

It has been previously shown that the inorganic chloride content of TATB can be reduced from an average of 0.6% to 0.1% by adding water to the reaction mixture of TCTNB dissolved in toluene and aminating in the usual way with ammonia gas. Pilot plant runs containing 5, 10, 15 and 20% water failed, however, to show a decrease below the 0.1% chloride content with

increasing water content. Considering that the water/toluene contact area remains relatively constant with increasing water content, and that the chloride decrease may be related to this two-phase contact area, it was decided to try aminations in which an emulsifying agent was added to the reaction mixture. The results of a few of these laboratory trials are shown in Table I. The best results seemed to be obtained using dilute ammonium hydroxide as the aminating agent rather than ammonia gas. The emulsifying agent used was a anionic-nonionic surfactant mixture at approximately 0.25% to 2% concentration in the reaction mixture. Thus far, too few trials have been made to optimize all of the process variables of concentrations, temperature, reaction time, etc. A typical DTA of the TATB produced is shown in Fig 1 (Run No. 17). SEM photomicrographs (Fig. 2) indicates the absence of the usual voids in the TATB particles. These particles are small as well as all past chloride eliminations run in the laboratory. Larger particles have generally been obtained at the pilot plant scale.

Table I. Trial Results

Run No.	TCTNB (g)	Toluene (ml)	NH ₄ OH (ml)	Temperature (C)	Time (hr)	Special Conditions	Inorganic Chloride %
10	10	50	200	23	20	200 ml water added	0.0052
11	10	50	300	0-23	20	3 hr at 0 C, then 23 C	0.0011
12	10	50	300	0-23	20	5 hr at 0 C, then 23 C	0.0053
13	10	300	100	23	20	No surfactant added	0.063
14	10	300	100	23	20		0.0025
15*							0.0058
16	20	100	400	25	24	150 ml water; NH ₄ OH drop-wise	0.0026
17	15	100	150	50	24	150 ml water; NH ₄ OH drop-wise	0.0077
18	15	75	100	80	22	2 hr at 80 C; NH ₄ OH drop-wise	0.0020
19	10	75	150	50-60	24	High purity TCTNB; NH ₄ OH drop-wise	0.0020

*Repeat of Run No. 10.

Fig. 1. Emulsion Processed TATB (Run No. 17)

Mag. 270

Mag. 575

Mag. 1350

Mag. 2700

Mag. 6800

Mag. 6800

Fig. 2. TATB Using Ammonium Hydroxide as the Aminating Agent (Run No. 17)

CONCLUSIONS

Assuming the analytical data obtained for inorganic chloride are reasonably accurate, the results obtained thus far appear encouraging toward reduction of ammonium chlo-

ride. Further work in the area of optimizing reaction conditions, particularly in the selection of an emulsifier, is indicated. Additional tests of other properties of the TATB are needed as well as pilot plant scale up work.