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TATB - Water Amination Process

W. T. Quinlin

DEVELOPMENT DIVISION

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MASTER

Process Development
Endeavor No. 106

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ABSTRACT

A pilot-plant scale process for amination of sym-trichlorotrinitrobenzene (TCTNB) in the presence of water to produce sym-triaminotrinitrobenzene (TATB) has been developed. With this process the chlorine content from trapped impurities is reduced from the typical 0.5 to 1.0% range down to about 0.1%. This reduction in chlorine is accompanied by a decrease in size of the median particle by 5 to 10 μm.

DISCUSSION

The present production process(*1*) for TATB includes a final amination reaction of TCTNB to produce TATB. The reaction is conducted in dry toluene at 150 C with mild agitation. Ammonia gas is added to the reactor ullage and the reaction takes place at the liquid vapor interface. Particle growth occurs in the reaction zone. The growth process ceases after the particles settle below the reaction zone. The TATB is isolated by cooling the reactor contents, adding water, and filtering the product from the mixture following several minutes of rapid agitation. The product is then washed with hot water, steamed and dried at 115 C.

TATB produced in this manner has been found to contain 0.5% or more chlorine. There are two sources for chlorine; the major source being ammonium chloride which is a by-product of the amination and is found as inclusions in the TATB crystals (Fig. 1). The other source is from organic chlorides which result from the incomplete amination of TCTNB and its impurities.

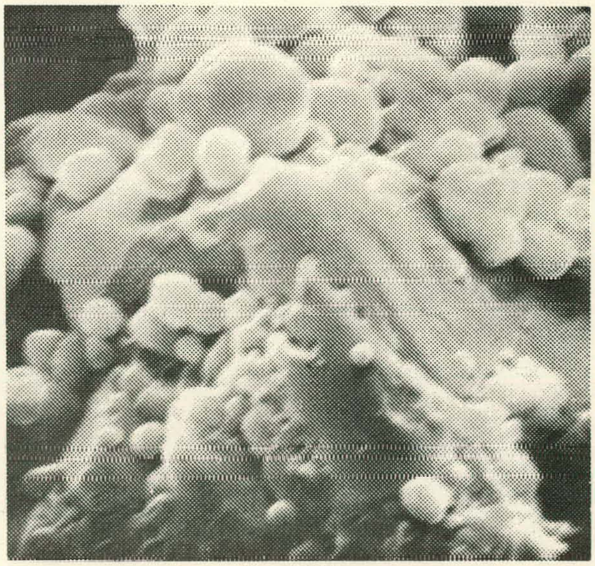

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Fig. 1. TATB From Dry Amination (Unwashed)

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(1) W. T. Quinlin, Z. L. Estes, V. H. Evans, C. L. Schaffer, Pilot Scale Synthesis of TATB, MHSMP-76-20 (July 1976).

An amination process which reduces the solid ammonium chloride available for entrapment in the TATB particles was first suggested and evaluated by Ted Benziger(2) at LASL. His work indicated that amination in the presence of several percent water in the toluene substantially lowers the chlorine content in the TATB.

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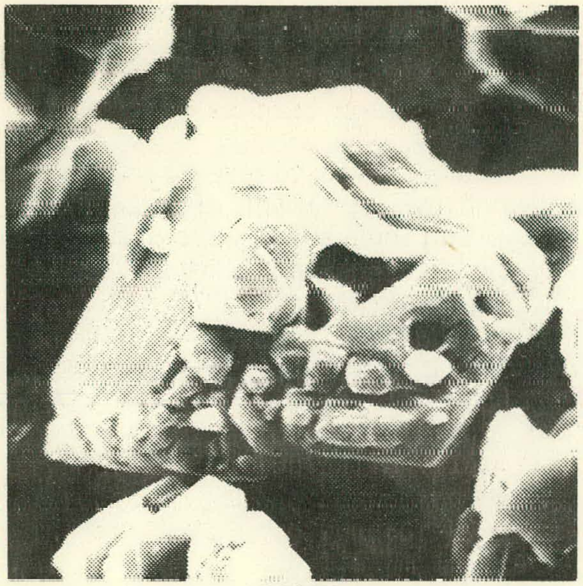
(2) C. D. Alley, Private Communication, Pantex Plant (6/12/74).

The present work had three objectives for the development of a water amination process; the production of TATB with an inorganic chloride content of 0.1% or less, a TATB particle size similar that from the standard process and a process which could be conducted efficiently in existing equipment. The process that was developed met the chlorine content objective (Table I). The TATB particle size was smaller than that from the standard process. The crystals from the water amination process (Fig. 2) are more solid and more regular in shape than those from the standard amination process.

Table I. Water Amination Batch Data

Batch No.	kg (TCTNB) / ℓ (Toluene)	Volume Percent (H_2O)	Total Chlorine (Wt %)	Inorganic Chloride (Wt %)	% < 20 (μm)	% < 44 (μm)	Median Particle (μm)
7194-01[a]	0.075	0	1.07	0.86	10	56	42
7213-02	0.075	0	0.96	0.80	17	67	40
7196-01	↓	5	0.10	0.10	22	75	35
7272-02	↓	10	0.07	0.02	21	76	34
7206-01	↓	20	0.18	0.02	34	95	25
7206-02	↓	30	0.11	0.02	19	81	34
7273-01	↓	40	0.05	0.01	21	90	32
7217-01	↓	50	0.08	0.01	25	90	30
7276-01	0.095	0	0.88	1.00	16	85	34
7277-01	↓	5	0.29	0.02	32	86	29
7277-02	↓	10	0.09	0.06	50	91	26
7200-01	↓	20	0.12	0.01	29	83	31
7210-02	↓	30	0.03	0.01	22	92	30
7210-01	↓	40	0.07	0.01	16	85	34
7209-01	↓	50	0.02	0.01	24	89	30
7209-02	↓	60	0.05	0.01	29	85	27
7243-01	↓	70	0.08	0.05	20	92	31
7243-02	0.054	30	0.05	0.01	25	92	29
7206-02	0.075	↓	0.11	0.02	19	81	34
7210-01	0.095	↓	0.07	0.05	17	80	34
7201-01	0.108	↓	0.10	0.05	23	79	34
7279-02	0.126	↓	0.08	0.02	16	82	32

[a]Aminated at 150 C. All other runs at 125 C



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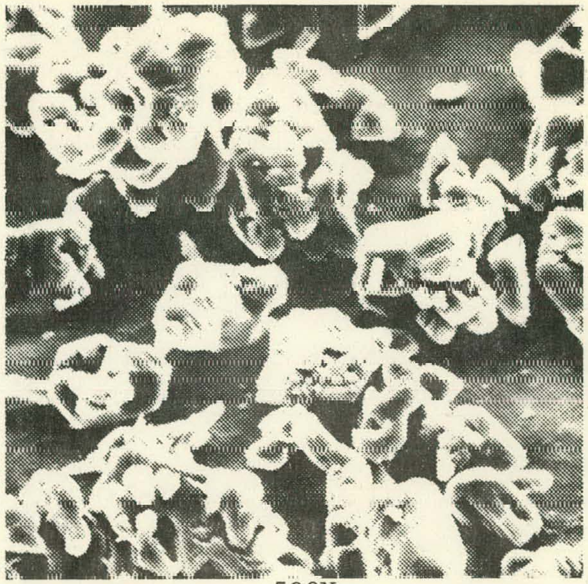

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Fig. 2. TATB From Water Amination

EXPERIMENTAL

General Procedure for Amination of TCTNB

The aminations were conducted in a 100-gallon stainless steel reactor fitted with nitrogen and ammonia inlet lines, solid addition port, 2 hp agitation unit, vent line and bottom port for solution inlet and slurry outlet. A 100-gallon glassed steel feed vessel was connected to the inlet port of the reactor by a transfer line through 1.5 μm in-line filter unit. The products were isolated in a plate and frame filter press.

The total volume of liquid used for all reactions was 100 ℓ, which was either toluene or a toluene/water mixture. The toluene was pumped into the feed vessel and TCTNB was then added. After solution was complete it was transferred through the in-line filter to the reactor. For wet aminations the water was then metered into the reactor.

The reaction mixture was then brought to 125 $\pm$ 2 C by steam heat to the vessel jacket. The vessel pressure at 125 C was found to be about 0.276 MPa which corresponds to the pressure from 150 C dry amination. An initial ammonia flow of 0.03 kg/min was established with the ammonia regulator set at 0.414 MPa. The agitator rate was 200 rpm. The reaction was continued until the vessel pressure approached the regulator pressure and the amount of ammonia added was in excess of one-third by weight of the TCTNB weight which is in excess of the 6:1 mole ratio required.

The reaction mixtures were cooled to 100 C and the water/toluene ratio brought to 1:1. The mixture was then agitated at 200 rpm and 100 C for 30 minutes. The TATB was then isolated in the filter press, washed with hot water for a minimum of 30 minutes, and steamed for a minimum of 1 hour. The product was dried for 16 hours in a forced-air oven at 125 C and analytical samples taken.

Variations to the General Procedure

Dry amination batches were made at the standard 150 C (7194-01) and at 125 C (7276-01 and 7213-02). The analytical data from these batches were used for comparison to the results from the water amination batches.

All concentrations were calculated as kg of TCTNB per liter of toluene. Two amination series were made varying the water toluene ratio in each, the first series at 0.075 kg (TCTNB)/ℓ (toluene) (Fig. 3) and the second at 0.094 kg (TCTNB)/ℓ (toluene) (Fig. 4). Additional reactions were made holding the water/toluene ratio constant and varying the TCTNB/toluene concentration (Fig. 5).

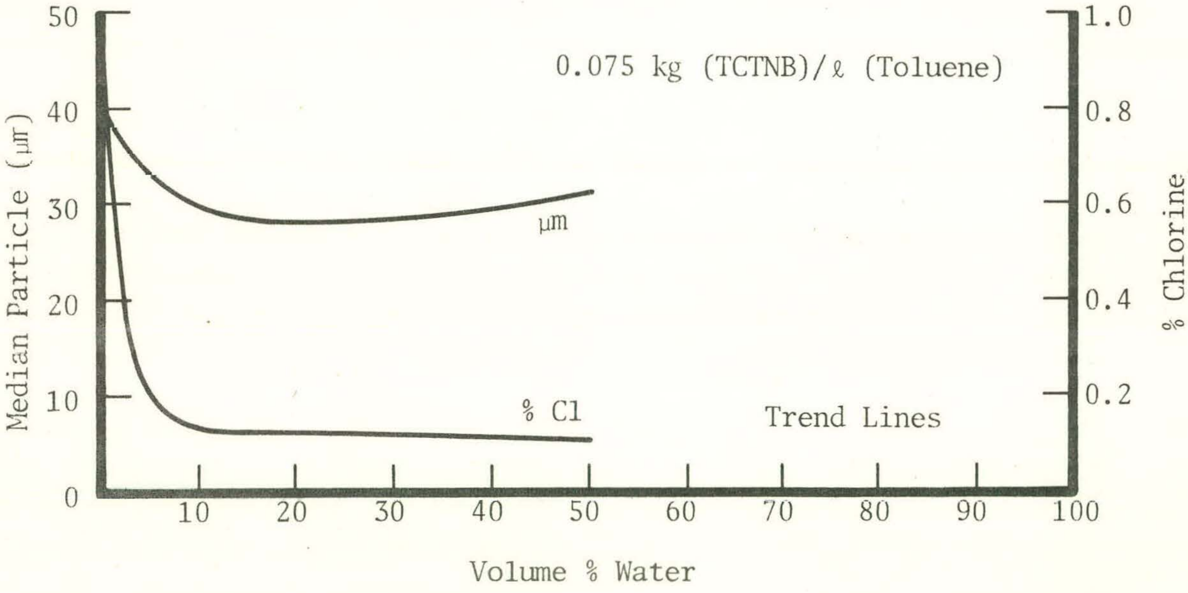

Fig. 3. Effect of Water on TATB Particle Size and Chlorine Content

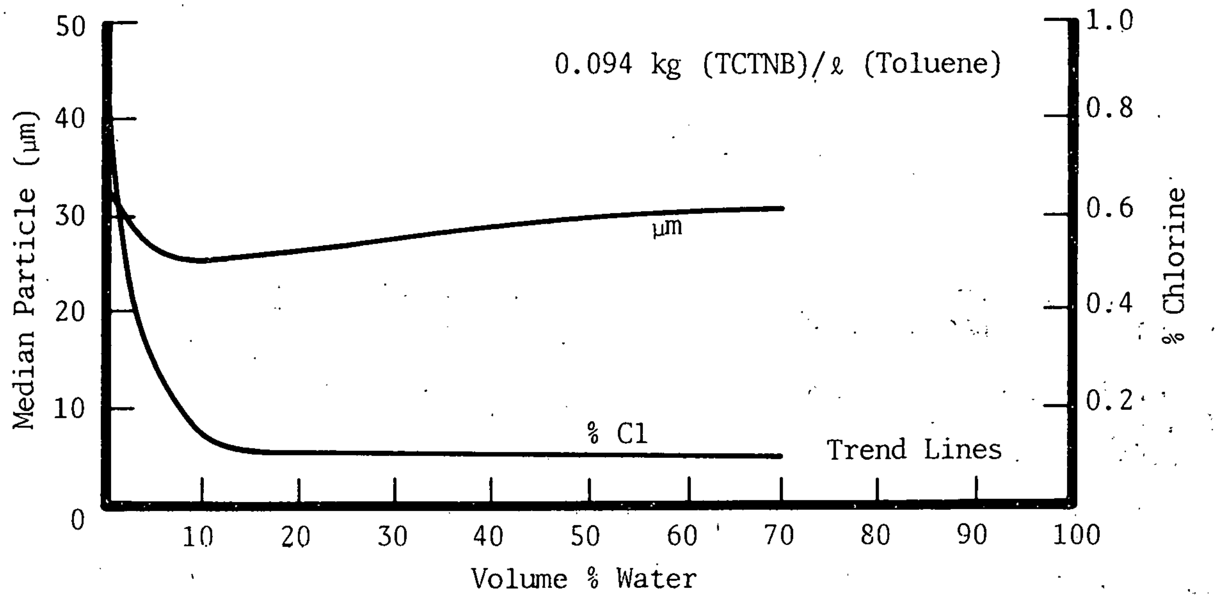

Fig. 4. Effect of Water on TATB Particle Size and Chlorine Content

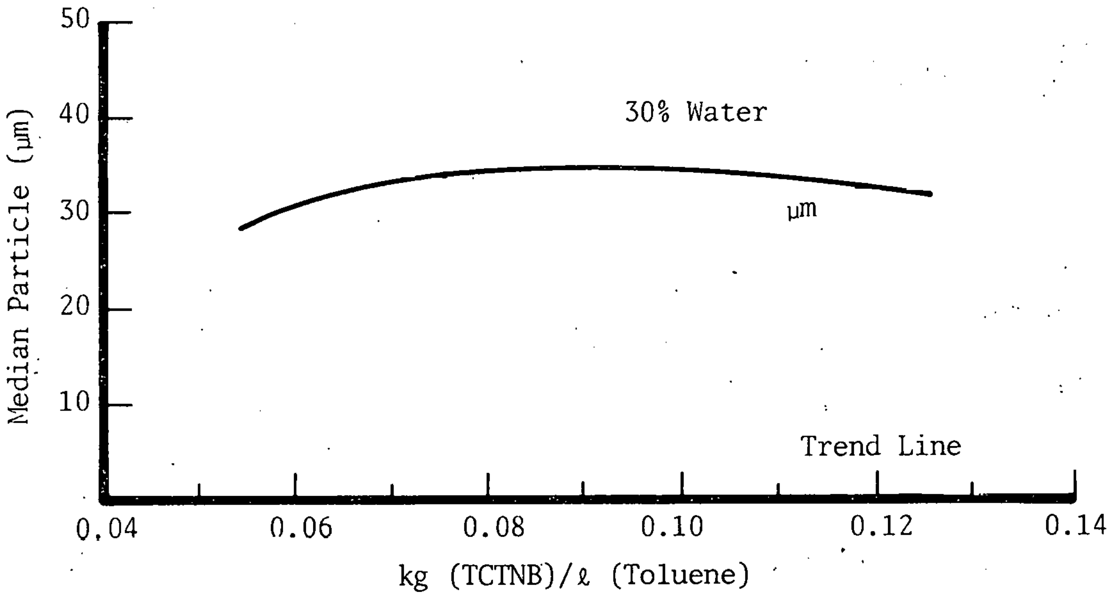

Fig. 5. Effect of TCTNB Concentration on Particle Size

SUMMARY

The developed water amination process is adaptable to the existing amination equipment used for standard aminations. Water aminations at 125 C give the same system pressures as standard aminations conducted at 150 C. A vigorously agitated water-toluene mixture of 10 to 30% water by volume is adequate for solution of the TCTNB and removal of ammonium chloride as it is formed. The throughput is only slightly lowered for the water amination process compared to that of the standard process.

Product analysis shows the total chlorine content reduced to about 0.1% and the inorganic chloride to 0.05% or less with 30% water. The particles are somewhat smaller than from standard amination. Further work is being conducted to increase the particle size of the water aminated product.

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