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SUMMARY OF CHLORINE FREE SYNTHESIS OF TATB

Zach Estes

DEVELOPMENT DIVISION

OCTOBER 1977

Process Development
Endeavor No. 106

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

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ABSTRACT

This report provides a summary of the Mason & Hanger efforts to synthesize s-triaminotrinitrobenzene (TATB) which is free from inorganic chlorides and other detrimental impurities.

DISCUSSION

The principal laboratory effort has been devoted to the synthesis of a chlorine free intermediate compound from TCTNB (s-trichlorotrinitrobenzene) which can readily be converted to TATB since TCTNB is a relatively

inexpensive starting material and the highly reactive chlorine atoms provided the possibility of replacement by other functional groups.

The most successful attempt to produce chlorine free TATB via TCTNB was by the following method:

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Although the TATB produced was free of chlorine, it lacked the required thermal stability. This is probably due to entrapment of the liberated alcohol or side reaction products. This method should perhaps be investigated further under different reaction conditions of amination, e.g. NH_4OH rather than dry ammonia gas in toluene.

Listed in Fig. 1 are a few of the synthesis routes which were tried without success.

Considering that the alkoxide groups of trinitrotrimethoxybenzene are easily replaced by ammonia to give TATB, it was decided to attempt the direct nitration of phloroglucinol:

This, however, was unsuccessful under the conditions tried, although other workers have reported success with this reaction. Nitration of phloroglucinol and nitration of the s-trimethoxybenzene were also unsuccessful.

Other unsuccessful attempts were made to produce TATB by the following methods:

This reaction failed in the oxidation stage. Low temperatures failed to oxidize the methyl groups while higher temperatures cause the loss of carbon dioxide.

In this scheme, analogous to the synthesis of trinitrobenzaldehyde from TNT, the N-bromosuccinimide failed to react.

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CONCLUSIONS

Probably the best methods of reducing the halogen content of TATB found thus far are by recrystallization and the emulsion processing(1). Emulsion amination of TCTNB in ammonium hydroxide appears to reduce

the inorganic chloride content by 1 or 2 orders of magnitude in laboratory scale experiments.

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- (1) Z. L. Estes, *Chlorine Free Synthesis of TATB*, MHSMP-77-24 (July 1977).

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