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TECHNICAL MEMORANDUM No. 5/M/49

✓ Cyanamide from Urea and Thionyl Chloride
or Sulphuryl Chloride

A. W. H. Pryde and W. J. Watkinson

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Technical Representative for Artillery
OFFICERS GROUP NO. 1
U.S. Navy 100, P.P.O.
New York, N.Y.

Ref: ORD-TRANT-113

6 October 1949

SUBJECT: Cyanamide from Urea and Thionyl Chloride
or Sulphuryl Chloride.

TO : The Chief of Ordnance
Department of the Army
Washington 25, D.C.

Att: ORDTM

1. References:

None.

2. Purpose of this Report:

Forwarded herewith as Enclosure 1 is E.R.D.E. (Explosives Research and Development Establishment) Technical Memorandum No. 5/N/49 - "Cyanamide from Urea and Thionyl Chloride or Sulphuryl Chloride." by A.W.H. Pryde and W.J. Watkinson, dated June 1949.

3. Comments:

The introduction of the Memorandum is repeated for convenience herewith as follows:-

"It is stated (Bull de la Soc. Chem. de France (3) 11, 1069) that cyanamide is formed by the reaction of thionyl chloride and urea according to the equation



There is no yield quoted and conditions are not specifically stated. As urea is readily manufactured and comparatively cheap this reaction seemed worthy of investigation.

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MINISTRY OF SUPPLY.

EXPLOSIVES RESEARCH AND DEVELOPMENT ESTABLISHMENT.

E.R.D.E. TECHNICAL MEMORANDUM NO.5/M/49.

Cyanamide from Urea and Thionyl Chloride or
Sulphuryl Chloride.

by

A.W.H. Pryde

and

W.J. Watkinson.

—
This memorandum does not contain information of
overseas origin.
—

Submitted by

A. Foster
S.E.I.

Approved by

H. H. H. H.
C.S./E.R.D.E.

Waltham Abbey,
Essex.

June, 1949.

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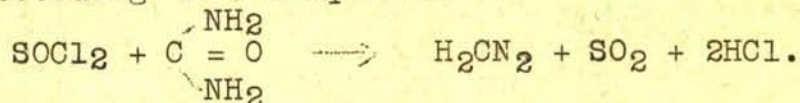
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Introduction.

It is stated (B1 de la Soc.Chem.de France (3) 11, 1069) that cyanamide is formed by the reaction of thionyl chloride and urea according to the equation

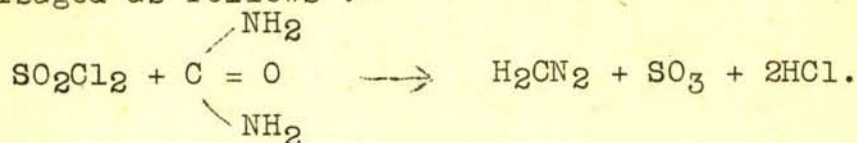


There is no yield quoted and conditions are not specifically stated. As urea is readily manufactured and comparatively cheap this reaction seemed worthy of investigation.

Experimental.

Numerous experiments with urea and thionyl chloride with and without excess of thionyl chloride and at various temperatures up to the boiling point of thionyl chloride were completely negative, no cyanamide being detected in any of the experiments.

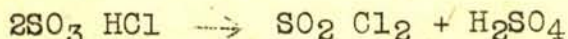
It seemed possible that sulphuryl chloride might give better results and would have the advantage over thionyl chloride that the sulphuryl chloride could readily be regenerated from the chlorosulphonic acid formed by distillation in the presence of mercuric sulphate as catalyst. The mechanism of the reaction was envisaged as follows :-



It was hoped that the SO_3 and HCl would be evolved as gases which, with the addition of another molecule of SO_3 would condense to chlorosulphonic acid



This chlorosulphonic acid, on distilling with 1% mercuric oxide as catalyst, would regenerate the sulphuryl chloride for re-use.



The H_2SO_4 could then be cracked to SO_3 to close the cycle and give a reasonably attractive process.

Experimental.

Urea and excess sulphuryl chloride were mixed at room temperature. A reaction set in and the temperature of the mixture rose to about 40°C . and was held at this till there was no further sign of reaction when a syrupy mass was left with the excess sulphuryl chloride floating in it in globules. The excess sulphuryl chloride was removed under reduced pressure, the mixture extracted with water and cyanamide estimated.

Yields were in all cases poor, 5% of theory being the maximum attained. The addition of anhydrous aluminium chloride to the reaction mixture seemed beneficial and resulted in very slightly

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greater yield.

A possible explanation of the low yields obtained seemed to be the fact that SO_2 and HCl were not evolved during the reaction but probably combine to form chlorosulphonic acid which is not removed from the reaction mixture and probably results in undesirable side reactions. For this reason a number of experiments were carried out in which sulphuryl chloride was made to react with a mixture of urea and lime when it was hoped that the lime might neutralise the chlorosulphonic acid as it was formed. The product however gave cyanamide in poor yield only in heating to a dull red heat.

This reaction seemed to offer very little promise as a process so no further work was done on it.

S.No.78.

M.No.198/49.

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