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A NEW SYNTHESIS OF CYANOGEN CHLORIDE

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Pure cyanogen chloride has been prepared in about 95% yield (calculated on the amount of Cl_2O used) by the reaction of Cl_2O with solid AgCN at room temperature.

Introduction

Cyanogen chloride, CNCl, can be prepared (1,2) by passing chlorine gas into aqueous solutions of HCN, NaCN, $Na_2Zn(CN)_4$ or $K_2Zn(CN)_4$. We wish to report here the preparation of CNCl by the reaction of a purified sample of chlorine monoxide, Cl_2O, with dry AgCN at room temperature. The purification and the characterization of CNCl are discussed.

Experimental

The work was carried out in a high vacuum system. Chlorine monoxide was prepared (3) by allowing Cl_2 gas to react with yellow mercuric oxide powder at -78° C for 24 hours. It was purified by fractional distillation and condensation in a high vacuum line and its purity checked by infrared spec-

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trum (4). The chlorine monoxide sample gas was repeatedly passed through a U-trap, packed loosely with anahydrous silver cyanide and glass wool. The products of reaction were collected in a trap cooled in liquid nitrogen. Cyanogen chloride was retained in traps cooled in isopropyl alcohol--dry ice slush (-78° C), when the reaction products were allowed to distill through two traps cooled at -78° C into traps cooled in liquid nitrogen. Elemental analysis (calcd. for CNCl, C = 19.52; N = 22.78; Cl = 57.68, found C = 19.45; N = 22.79) showed that the sample was reasonably pure. Complete characterization was made by comparing the observed infrared spectrum (ν_{max} observed cm^{-1}: 2200, 2219, 788, 770, 720, 700 and 380) with that reported in the literature (5) and by analyzing the observed mass spectrum as shown in the table.

TABLE

m/e	Relative Intensity	Positively Charged Ions
61	100	$^{12}C^{14}N^{35}Cl^{+}$
63	30.4	$^{12}C^{14}N^{37}Cl^{+}$
30.5	3.7	$^{12}C^{14}N^{35}Cl^{++}$
31.5	1.4	$^{12}C^{14}N^{37}Cl^{++}$
35	10.8	$^{35}Cl^{+}$
37	4.3	$^{37}Cl^{+}$
26	8.7	$^{12}C^{14}N^{+}$
14	6.4	$^{14}N^{+}$ or N_2^{++}
12	10.6	$^{12}C^{+}$

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