Crystal Structure of (*E*)-2,3-Dichloro-1-(pyrrolidin-2-ylidene)-Δ^2-pyrrolinium Bromide

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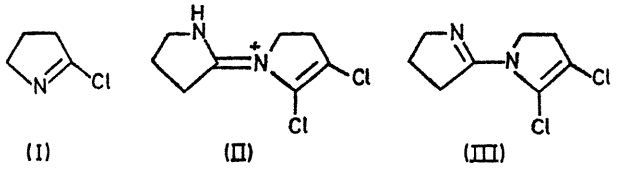
Summary The reaction product of pyrrolidin-2-one with hydrogen chloride and phosphorous pentachloride has been shown to be 2-(2,3-dichloro-Δ^2-pyrrolin-1-yl)-Δ^2-pyrroline, rather than 2-chloro-Δ^1-pyrroline as previously reported, by a single crystal *X*-ray structure determination of the hydrobromide salt of the product.

TAFEL and WASSMUTH reported the preparation of 2-chloro-Δ^1-pyrroline (I) (C_4H_6NCl) by reaction of pyrrolidin-



(I) (II) (III)

2-one with HCl and PCl_5.[1] Etienne and Correia reported the preparation of (I) using similar reaction conditions.[2] We repeated these procedures; the product obtained had physical properties in agreeemnt with those reported for (I).

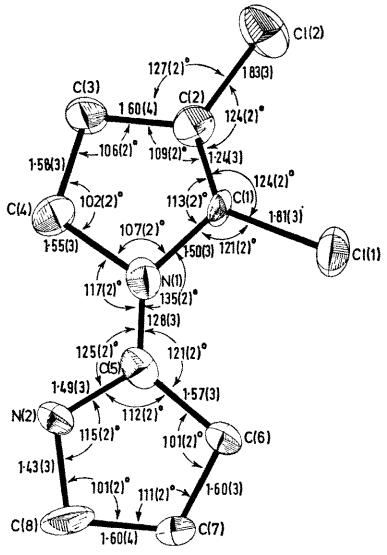
The mass spectrum of the product showed a molecular ion *m/e* 204·02, which is consistent with the formula $C_8H_{10}N_2Cl_2$. Its n.m.r. spectrum (CCl_4) showed absorptions at τ 8·10 (m, 2H) 7·31 (t, 2H, *J* 8 Hz), 6·45 (t, 2H, *J* 7 Hz), and 6·00 (t, 2H, *J* 9 Hz). Spin decoupling experiments were performed at 100 MHz. Irradiation at τ 6·45 (8·10 collapsed to t, *J* 8 Hz), at 6·00 (7·30 collapsed to s), and at 7·10 (8·10 collapsed to t, *J* 7 Hz) indicated the groups $-CH_2-CH_2-$ and $-CH_2-CH_2-CH_2-$.

The product formed stable mineral acid salts. The hydrobromide (II) was obtained as crystals suitable for *X*-ray examination. The crystals analysed as $C_8H_{10}N_2Cl_2$,HBr,$2H_2O$.

Crystal data: Monoclinic, $P2_1/c$; $a = 9{\cdot}193(5)$, $b = 21{\cdot}901(9)$, $c = 9{\cdot}428(5)$ Å, $\beta = 138{\cdot}16(2)°$; $D_c = 1{\cdot}688$, $D_o = 1{\cdot}681$ g cm^{-3}; $Z = 4$. Cell constants were measured on a Picker diffractometer. Intensity data were collected with Zr-filtered Mo-K_α radiation using the θ–2θ scan mode. A total of 604 unique, non-zero reflections were used for the structure refinement.

The structure was solved by the heavy-atom method and refined with anisotropic temperature factors for all of the non-hydrogen atoms. Hydrogen atoms were not included in the calculation; the final *R* value was 0·082 for the 604 observed reflections.

The Figure shows the bond angles and bond lengths for structure (II). Thus, the compound is (*E*)-2,3-dichloro-1-



FIGURE

(pyrrolidin-2-ylidene)-Δ^2-pyrrolinium bromide, and the product originally obtained by Tafel and Wassmuth must be 2-(2,3-dichloro-Δ^2-pyrrolin-1-yl)-Δ^1-pyrroline.[4]

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