

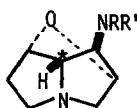
ABSOLUTE CONFIGURATIONS OF PYRROLIZIDINE ALKALOIDS OF
THE LOLINE GROUP

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Loline (=festucine, I),¹ norloline (II),^{1a} lolinine (III),^{1b} and de-corticasine (IV)² have been isolated from three genera of the families Gramineae and Leguminosae. The constitution and relative configurations of loline (I) were firmly established by an X-ray study on its dihydrochloride by Dickerson and McMillan,³ and as II-IV have been chemically interconverted^{1b,4} with loline (I), the structures of all four are established except for absolute configuration. As there was no evidence regarding the absolute configuration, and as both configurations at the starred center are known among the pyrrolizidine alkaloids,⁵ we undertook the determination of the absolute configuration of the dihydrochloride of loline (I) by anomalous dispersion.⁶



	<u>R</u>	<u>R'</u>
I	Me	H
II	H	H
III	Me	Ac
IV	H	Propionyl

Using the cell constants, atomic coordinates, and thermal parameters of Dickerson and McMillan,³ and the anomalous dispersion corrections 0.3 for $\Delta f'$ and 0.7 for $\Delta f''$ for scattering of Cu X-rays by Cl,⁷ structure factors were calculated for each enantiomer for all reflections below $2\theta=55^\circ$. The reflections were sorted according to percent difference in calculated structure factors for each enantiomer. Using a crystal

0.5x0.5x1.25 mm kindly provided by Tookey and Yates, ten of the more intense reflections with calculated differences between 9 and 22% were measured on a Picker automatic diffractometer along with their negatives in 2 θ . All ten showed differences of the expected magnitude, with nine of the ten in the direction favoring the configuration depicted in I; the tenth was the weakest by a factor of two. We conclude that the correct absolute configurations for these tricyclic alkaloids are those shown in I-IV.

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