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## SPECTRAL STUDIES ON ALKALOIDS

## Part IV.—The Identification of Berbericine Hydroiodide as Palmatine Iodide

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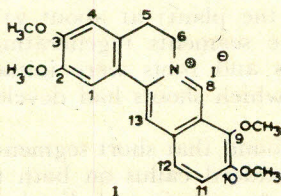
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In continuation<sup>1</sup> of our studies on the alkaloids of *Berberis lycium* Royle, evidence is presented in this paper which indicates that berbericine hydroiodide,<sup>2</sup> C<sub>21</sub>H<sub>22</sub>NO<sub>4</sub>I, m. p. 205–206°C (dec.), is identical with palmatine iodide (i). (lit.<sup>3</sup> m.p. 238–239°C(dec.).



The reported berbericine hydroiodide<sup>2</sup> was found impure and was further purified to give berberine iodide and berbericine hydroiodide.

Berbericine hydroiodide was reported<sup>2</sup> to contain three methoxyl and one *N*-methyl groups. The fourth oxygen atom was assumed to be present as hydroxyl group.

The UV spectrum of berbericine hydroiodide in methanol showed absorption bands at  $\lambda_{\max}$  225 (log  $\epsilon$  4.65), 265 (4.53) and sh. 272 m $\mu$  (4.50), which is characteristic<sup>4</sup> of protoberberine type of alkaloids. Moreover, the UV and IR spectra of berbericine hydroiodide resembled very closely the characteristic features of palmatine chloride,<sup>5</sup> suggesting that both the bases might be identical. This was in turn unequivocally established by NMR evidence;<sup>6</sup> in trifluoroacetic acid, the following peaks were obtained for both the substances—0.35 (C<sub>8</sub>—H), 1.42 (C<sub>13</sub>—H), 1.99 (2H at C<sub>11</sub> and C<sub>12</sub>), 2.40 (C<sub>1</sub>—H), 3.00 (C<sub>4</sub>—H), 5.75 and 5.89 (2 OCH<sub>3</sub> at C<sub>9</sub> and C<sub>10</sub>), 5.94 and 6.0 (2 OCH<sub>3</sub> at C<sub>2</sub> and C<sub>3</sub>), a triplet at 5.09 (H<sub>2</sub>—C<sub>6</sub>) and another triplet at 6.66  $\tau$  (H<sub>2</sub>—C<sub>5</sub>), the unresolved portions of spectra being virtually identical, too.

On the basis of above studies, it is confirmed that berbericine hydroiodide is identical with palmatine iodide I.

## Experimental

*Purification of Berbericine Hydroiodide.*—Berbericine hydroiodide<sup>2</sup> still showed two spots on thin layer chromatogram (on alumina, irrigation with ethanol). The fast-moving compound was shown to be berberine iodide by comparison of *R<sub>f</sub>* values of the mixture and an authentic sample of berberine iodide prepared from berberine chloride by usual methods.

Berbericine hydroiodide (1.0 g) was chromatographed on alumina (50 g, May and Baker) prepared in ethanol. Ten fractions of 200 ml each were collected. The purity of each fraction was monitored by thin layer chromatography. First four fractions were found to contain berberine iodide and the last three berbericine hydroiodide. The last 3 fractions were combined and concentrated to give yellow needles of berbericine iodide, m.p. 205–206°C(dec); palmatine iodide, (lit<sup>3</sup> m.p. 238–239°C(dec.).

The IR, UV and NMR spectra of berbericine hydroiodide and palmatine chloride were essentially identical and showed main absorption bands. UV:  $\lambda_{\max}^{\text{MeOH}}$  225, 265, sh 272 m $\mu$  (log  $\epsilon$  4.65, 4.53 and 4.50). NMR in trifluoroacetic acid showed peaks at  $\tau$  0.35(1H), 1.42(1H), 1.99(2H), 2.40(1H), 3.00(1H), 5.75 and 5.89(6H), 5.94 and 6.0(6H), 5.09(2H) triplet, 6.66(2H, triplet) and some unidentified peaks due to some minor impurity.

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## References

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