

isolated by filtration and dried overnight (20°, 1 mm). The yield is 0.88 g (64 %) of V. [Found: C 48.3; H 5.5; N 24.9. Calc. for  $C_9H_{12}N_4O_3$  (224.2): C 48.2; H 5.4; N 25.0].

The products obtained in the above synthesis do not always behave in the same way, when heated in a tube. They may soften and melt completely at temperatures between 115° and 152°, depending upon the particular product, as well as upon the rate of heating. The yields obtained in the synthesis are also not reproducible, being either around 65 % or around 85 %. However, all products are always analytically pure, give a red ferric chloride reaction, and have identical infrared absorption spectra in a chloroform solution. It has not been investigated, whether the different products obtained in the crystalline state are polymorphic or isomeric (e.g. tautomeric).

*3-Hydroxypicolylcarbamie acid methyl ester* (VI). I (1.00 g) is added to a solution of sodium methoxide in methanol [from sodium (0.15 g) and methanol (5 ml)] and the mixture heated under reflux (5 min). The reaction mixture is cooled and acetic acid (0.37 ml) and then water (10 ml) added. The resulting suspension of white crystals is stirred for half an hour. The crystals are isolated by filtration, washed with water (10 ml) and dried (2 h, 50°, 1 mm). The yield is 1.07 g (89 %) of VI. [Found: C 48.8; H 4.2; N 14.7;  $OCH_3$  15.1. Calc. for  $C_8H_8N_2O_4$  (196.2): C 49.0; H 4.1; N 14.3; one  $OCH_3$  15.8]. The product gives a red ferric chloride reaction.

The above analyses indicate, that the product contains about 4 % of pyridoöxazine as an impurity. Thin-layer chromatography also shows the presence of pyridoöxazine. We believe that the pyridoöxazine was not present in the wet crystals, but has been formed during drying.

When heated in a tube the product melts around 170° and resolidifies around 220° (formation of I). 1.00 g of the product was heated in an open tube to 190–210° for 7 min. During this time it first gave off methanol and then solidified. The resulting brown solid was crystallized from acetic acid (12 ml) with carbon black to give 0.64 g (76 %) of I, m.p. 265–270° with decomposition.<sup>1</sup> [Found: C 51.3; H 2.7; N 16.9. Calc. for  $C_7H_4N_2O_3$  (164.1): C 51.2; H 2.5; N 17.1].

1. Clauson-Kaas, N., Brammer Petersen, J., Sørensen, G. O., Olsen, G. and Jansen, G. *Acta Chem. Scand.* **19** (1965) 1147.

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## Studies on Orchidaceae Alkaloids

### V.\* A New Alkaloid from *Phalaenopsis amabilis* Bl.

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A new alkaloid phalaenopsin (I) has been isolated from *Phalaenopsis amabilis* Bl., m.p. 99°–101°, elementary formula  $C_{20}H_{27}NO_5$ , MW 361 which was verified by its mass spectrum. Prominent peaks in the mass spectrum are  $m/e$  361 ( $M^+$ ), 330, 288, 124 (base peak), 83, and a metastable ion at  $m/e$  230 corresponding to a breakdown of  $m/e$  361 to  $m/e$  288.

The hydrochloride of I ( $C_{20}H_{28}ClNO_5$ ) had m.p. 155°–156°. The UV absorption of the base has maxima at 264, 258, 254, 248, and 243  $m\mu$ , the IR spectrum shows a strong band at 1735  $cm^{-1}$ , but no NH band. The molecule contains one  $O-CH_3$  but no  $N-CH_3$  or  $C-CH_3$ . The hydrochloride of I shows the following distinct bands in the NMR spectrum (DMSO  $d_6$ ):  $\tau = 2.55$  5H singlet,  $\tau = 4.3$  1H singlet ( $N^+ - H$ ),  $\tau = 5.72$  2H triplet ( $J = 6$  cps),  $\tau = 6.24$  3H singlet,  $\tau = 6.84$  2H singlet,  $\tau = 6.86$  and 7.33 2H A–B ( $J = 16$  cps).

The data suggest that the alkaloid is an ester containing a *mono*-substituted benzene nucleus. All elementary analyses gave the correct values. The  $LD_{50}$  of the hydrochloride of I in mice (intravenous) is 75 mg/kg. A full report on the isolation and structure of I will be published later.

The mass spectra were measured by Dr Ryhage and the NMR spectrum of the hydrochloride by Dr. Melera, Varian AG, Zürich, on a HA 100 spectrometer. The work has been supported by grants from the *Swedish Natural Science Research Council*.

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