Jatropham in *Lilium candidum* L.

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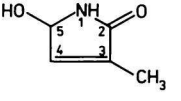
A nitrogen-containing compound — jatropham — was isolated from petals of *Lilium candidum* L. (family *Liliaceae*); its presence in this plant species has not been reported as yet. Jatropham was obtained by chromatography on silica gel and identified by spectral means.

Азотсодержащее вещество — иатрофам — было выделено из лепестков *Lilium candidum* L. (семейство *Liliaceae*); о его нахождении в растениях этого вида до сих пор не сообщалось. Иатрофам был выделен путем хроматографии на силикагеле и идентифицирован с помощью спектральных методов.

Our preceding papers on *Lilium candidum* L. [1, 2] dealt with the isolation and identification of succinic, R-(+)-methylsuccinic, itaconic, and fumaric acids, a mixture of unbranched *C*-20—*C*-30 acids, the flavone kaempherol, and a nitrogen-containing compound $C_{20}H_{17}NO_7$.

This paper concerns the isolation of another nitrogen-containing compound, $C_5H_7NO_2$, which was shown to be identical with jatropham isolated for the first time from *Jatropha macrorrhiza* (*Euphorbiaceae*) by *Wiedhopf* and coworkers [3]. These authors proposed the structure of 5-hydroxy-4-methyl-3-pyrrolin-2--one for it from the NMR, UV, IR, ORD, and mass spectral data and mentioned also its antitumour activity. Japanese authors [4] located the methyl group from C-4 to C-3 and correctness of the revised structure verified by synthesis [5, 6]. The spectral data of jatropham isolated from petals of *Lilium candidum* L. are consistent with those of the corrected structure.



Experimental

The melting point was measured on a Kofler micro hot-stage, the UV spectra of methanolic solutions and the IR spectra of KBr pellets were recorded with UV VIS (Zeiss, Jena) and Perkin—Elmer, model 477 spectrophotometers, respectively. The mass spectra were taken with an AEI MS 902 apparatus and the ^{1}H NMR spectra of deuterioacetone solutions with tetramethylsilane as an internal standard were determined with an AM-300 (Bruker) instrument. Silica gel No. 4 (Silpearl) modified according to [7] and silica gel G according to Stahl, type 60 and Silufol UV_{254} and UV_{365} were employed for column and thin-layer chromatography, respectively.

The dried petals of *Lilium candidum* L. (3500 g) were stepwise macerated with 95 % and 70 % ethanol. The combined extract (1370 g) was concentrated under diminished pressure, the residue was dissolved in hydrochloric acid ($c = 0.15$ mol dm^{-3}) and the solution was exhaustively extracted with light petroleum, ether, and chloroform. The residue was made alkaline to pH = 11 and taken into chloroform and chloroform—ethanol (volume ratio (φ_r) = 2 : 1).

Compounds present in the latter solvent (8.6 g) were separated by chromatography on a silica gel-packed column (500 g) using benzene and acetone in various ratios as eluent. Fractions (128 total) amounting 150 cm^3 were worked up and the content was checked by thin-layer chromatography using following solvent systems: benzene—acetone (φ_r = 8.5 : 1.5; 8 : 2), chloroform—methanol (9 : 1; 8 : 2; 7 : 3). Chromatograms were sprayed with concentrated sulfuric acid and visualized by UV light. Fractions with the same product were collected; the 34th fraction afforded a uniform compound (70 mg), m.p. = 115—117 °C. Mass spectrum for $C_5H_7NO_2$ M_r(M^+, found) = 113.0455, M_r(M^+, calc.) = 113.0476; m/z (I_r/%): 98 (100), 85, 69, 68. UV spectrum (methanol), λ_{max}/nm: 241. IR spectrum (KBr), $\tilde{\nu}$/cm^{-1}: 3298 (ν(O—H)), 1686 (ν(C=O)), 1647 (ν(C=C)), 1450, 1380 (δ(C—H)) in CH_3, 1408 (ν(C—N)), 1304 (δ(N—H)), 1220 (ν(O—C—N)), 1059 (ν(C—O)). ^{1}H NMR spectrum, δ_r/ppm: 7.4 (bs, 1H, NH), 6.59 (ddq, 1H, $J_{4,5}$ = 1.9 Hz, $J_{1,4}$ = 1.2 Hz, J_{4,CH_3} = 1.8 Hz, C-4—H), 5.46 (dddq, 1H, $J_{5,OH}$ = 9.0 Hz, $J_{1,5}$ = 1.3 Hz, J_{5,CH_3} = 1.3 Hz, C-5—H), 4.78 (d, 1H, OH), 1.76 (dd, 3H, CH_3). ^{13}C NMR spectrum, δ_r/ppm: 172.9 (C-2), 141.9 (C-4), 136.4 (C-3), 79.2 (C-5), 10.4 (CH_3).

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