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3-METHOXYCHRYSAZIN, A NEW ANTHRAQUINONE FROM *XYRIS SEMIFUSCATA**

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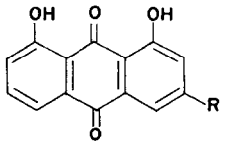
Key Word Index—*Xyris semifuscata*; Xyridaceae; anthraquinones; chrysazin; 3-methoxychrysazin.

From *Xyris semifuscata* Baker, harvested in Madagascar, the two major anthraquinones have been isolated by means of steam distillation followed by column chromatography on silica. The less polar of the two quinones was identified as chrysazin (**1**) (mmp, PMR, IR, UV and mass spectrum) [1–5]. The other gave a MW 270 (mass spectrum). The PMR spectrum indicated, after comparison with that of chrysazin, a monomethylated 1,3,8-trihydroxy-9,10-anthraquinone. From the IR spectrum it was concluded, that it was the 3-méthoxy-compound (**2**) [6]. This compound has been synthesized by Eder and Hauser [7] and they observed a m.p. very close to that we found (180°) for the isolated compound. Although known as a synthetic product, this is the first report of the natural occurrence of 3-méthoxy-chrysazin. Chrysazin is a well known natural compound [8].



(**1**) R = H
(**2**) R = OMe

EXPERIMENTAL

Isolation. Dried leaves and stems (30 g) were steam distilled from 1 N H_2SO_4. The fraction obtained (300 mg) was chromatographed on a column of silica gel (Mallinckrodt 100 mesh), using hexane–EtOAc (99:1) as the eluent. The two major quinones were collected separately.

Chrysazin (**1**): recrystallized from *n*-hexane as dark orange crystals (50 mg). mp, mmp: 191°. TLC: R_f 0·60 on precoated silica layers "Merck" with *n*-hexane-EtOAc (4:1) as the eluent. MS (70 eV): 241 (16%), 240 (100%), 212 (13%), 92 (12·5%), 184 (12%). 100 MHz PMR-spectrum (in C_6D_6): 6·85 (*d*, *J* 3·5 Hz) (H_4 and H_5), 6·86 (*d*, *J* 5·3 Hz) (H_2 and H_7), 7·64 (q, *J* 5·3 and 3·5 Hz) (H_3 and H_6) and 11·85 (2 OH's) ppm. IR (KBr): 1675 (*m*), 1625 (*s*), 1600 (sh), 1570 (w) cm^{-1}. UV (C_2H_5OH): 223, 250, 270, 280, 426 nm.

3-methoxychrysazine (**2**): recrystallized from 25% benzene in hexane as brilliant yellow needles (20 mg). TLC: R_f 0·50. MS (70 eV): 271 (16%), 270 (100%), 241 (12·5%), 212 (10%), 121 (10%). 100 MHz PMR-spectrum (in C_6D_6): 3·02 (*s*) (OCH_3), 6·44 (*d*, *J* 2·5 Hz) (H_4), 6·88 (*d*, *J* 5·3 Hz) (H_5), 6·89 (*d*, *J* 3·5 Hz) (H_7), 7·41 (*d*, *J* 2·5 Hz) (H_2), 7·67 (*q*, *J* 5·3 and 3·5 Hz) (H_6) and 12·16 and 12·26 (2 OH's) ppm. IR (KBr): 1675 (*m*), 1625 (*s*), 1610 (*s*), 1595 (sh), 1575 (w), 1560 (w), UV (EtOH): 222, 245, 263, 280, 428 nm.

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