

Phytochemistry, 1972, Vol. 11, p. 2351. Pergamon Press. Printed in England.

PENDULETIN 4'-O-METHYL ETHER FROM *PERITYLE VASEYI*

L. SOUTHWICK and T. J. MABRY

The Cell Research Institute and Department of Botany, University of Texas at Austin,
TX 78712, U.S.A.

J. AVERETT

Department of Biology, University of Missouri, St. Louis, MO 63121, U.S.A.

and

A. M. POWELL

Department of Botany, Sul Ross State University, Alpine, TX 79830, U.S.A.

(Received 24 January 1972)

Key Word Index—*Perityle vaseyi*; Compositae; penduletin 4'-methyl ether; 5-hydroxy-3,6,7,4'-tetramethoxyflavone.

Chloroform extraction of dried, ground leaves of *Perityle vaseyi* Coult. gave, after column chromatography of the extract, a flavonoid whose NMR spectrum (CDCl_3) indicated the presence of four methoxyl groups [δ 3.88 (s), 3.92 (s), 3.96 (s), 3.98 (s)], a 4'-oxygenated B-ring [H-3',5': 7.04 (d, $J = 9.0$); H-2',6': 8.11 (d, $J = 9.0$)], and a free 5-hydroxyl group [12.77 (s)]. The spectrum also exhibited a one-proton singlet at 6.53, typical for an H-8 proton. The NMR spectrum of the new compound in benzene- d_6 was in accord with the presence of 3-, 6-, 7- and 4'-methoxyl groups:^{1,2} 3.30 (7-OMe, 4'-OMe), 3.77 (3-OMe), and 3.92 (6-OMe). The NMR data therefore indicated that the flavonoid was penduletin 4'-O-methyl ether.

In addition, the color of the compound in UV light when spotted on paper (purple under UV and UV/ NH_3) was consistent with a flavonol containing a 5-hydroxyl group and substituted 3- and 4'-hydroxyl groups. The UV spectra—a set of five were recorded under standard conditions—were almost identical with those for penduletin 4'-O- β -D-glucoside (pendulin).³ The spectral data thus establish that the new flavonol is penduletin 4'-O-methyl ether (5-hydroxy-3,6,7,4'-tetramethoxyflavone).

EXPERIMENTAL

P. vaseyi Coult. plant material was collected in Big Bend National Park just north of Castolon, Brewster Co., Texas, on 29 July 1967 (A. M. Powell, J. Averett and T. Watson 1544). A CHCl_3 extraction of the air-dried, ground leaves was worked up in the usual way.⁴ Chromatography of the extract over silica gel (CHCl_3 elution) afforded penduletin 4'-O-methyl ether as a yellow solid; m.p. 170–172° (acetone water; softening at about 165°); MS: molecular ion at m/e 358.1050; calcd. for $\text{C}_{19}\text{H}_{18}\text{O}_7$, 358.1052; R_f (TBA) 0.87; UV: λ_{max} (MeOH): 255sh, 273, 335 nm; λ_{max} (NaOMe): 290, 325sh, 362sh nm; λ_{max} (AlCl_3): 265sh, 283, 305sh, 364, 400sh nm; λ_{max} (AlCl_3 -HCl): 263sh, 284, 305sh, 362, 400sh nm; λ_{max} (NaOAc): 273, 338 nm.

Acknowledgements—The mass spectrum was obtained on a DuPont (CEC) 21-110 high resolution mass spectrometer, provided by a National Science Foundation grant (NSF GP-8509) to the Chemistry Department, The University of Texas, Austin. We thank the Robert A. Welch (Grant F-130) and the National Science (GB-29576X) Foundations and the National Institutes of Health (Grants HD-04488 and 5-TO1-GM-03789 for financial support.

¹ R. G. WILSON, J. H. BOWIE and D. H. WILLIAMS, *Tetrahedron* **24**, 1407 (1968).

² E. RODRIGUEZ, N. J. CARMAN and T. J. MABRY, *Phytochem.* **11**, 409 (1972).

³ T. J. MABRY, K. R. MARKHAM and M. B. THOMAS, *The Systematic Identification of Flavonoids*, Springer, Heidelberg-New York (1970).

⁴ T. J. MABRY, H. E. MILLER, H. B. KAGAN and W. RENOLD, *Tetrahedron* **22**, 1144 (1966).