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## COMPOSITAE

### 5-HYDROXY-6,7,4'-TRIMETHOXYFLAVONE FROM *AGERATINA GILBERTII*

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**Key Word Index**—*Ageratina gilbertii*; Compositae; 5-hydroxy-6,7,4'-trimethoxyflavone.

*Plant.* *Ageratina gilbertii* (B. L. Robins). King and H. Robins.\* *Source.* Collected by S. Gibaja and R. Ferreyra near Surco, Lima, Peru in July 1967 (Ferreyra voucher No. 16892, deposited in herbarium of Museo de Historia Natural Javier Prado, Lima, Peru). *Previous work.* None.

*Isolation and identification.* The above ground parts were extracted with  $\text{CHCl}_3$  and worked up in the usual fashion.<sup>1</sup> The crude gum, wt 150 g from about 8 kg of plant material, was chromatographed over 1 kg of silicic acid (Mallinckrodt 100 mesh), 2 l. fractions being collected in the following order: Fr. 1–25 benzene, 26–40 benzene– $\text{CHCl}_3$  (1:1), 41–100  $\text{CHCl}_3$ . The only solid material was obtained from Fr. 8–30. The yellow crystals were combined and recrystallized from EtOAc, yield 0.330 g, m.p. 188–189° (lit. 187–190°,<sup>2</sup> 187–188°,<sup>3</sup> 188°<sup>4</sup>), MW 328 (MS). Calc. for  $\text{C}_{18}\text{H}_{16}\text{O}_6$ : 328. It gave a green  $\text{FeCl}_3$  test and was identified as 5-hydroxy-6,7,4'-trimethoxyflavone (salvigenin<sup>4</sup>) spectroscopically; UV  $\lambda_{\text{max}}$  330 and 277 nm (unaltered after addition of NaOAc or NaOAc– $\text{H}_3\text{BO}_4$ ), 354, 302 and 290 nm after addition of  $\text{AlCl}_3$ , NMR signals ( $\text{CDCl}_3$ ) at 7.84d and 7.02d (2 protons each, AB system,  $J = 9$  Hz, H-2', H-3', H-5' and H-6'), 6.57 and 6.54 (H-3 and H-8), 3.97, 3.94 and 3.90 ppm (three methoxys). The monoacetate was recrystallized from acetone–light petroleum: m.p. 168–169°, MW (MS): 370. Calc. for  $\text{C}_{20}\text{H}_{18}\text{O}_7$ : 370.

Although 5-hydroxy-6,7,4'-trimethoxyflavone has been known for 25 yr, the present finding constitutes only the second report of its occurrence in nature. It has previously been prepared from scutellarein<sup>2</sup> and by total synthesis<sup>3</sup> and has been isolated from *Salvia triloba* L.f.<sup>4,†</sup>

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\* The name of this taxon, previously known as *Eupatorium gilbertii* Robins, has been revised recently, R. M. KING and H. ROBINSON, *Phytologia* **19**, 208 (1970).

† After this manuscript was submitted, there appeared a report of the isolation of salvigenin from buds of *Alnus japonica*, F. WOLLENWEBER and M. WASSUM, *Tetrahedron Letters* 797 (1972).

<sup>1</sup> W. HERZ and G. HÖGENAUER, *J. Org. Chem.* **27**, 905 (1962).

<sup>2</sup> K. WISWESWARA RAO and T. R. SESHADRI, *Proc. Ind. Acad. Sci.* **26A**, 183 (1947).

<sup>3</sup> K. FUKUI, T. MATSUMOTO and T. KIMASHITA, *Bull. Soc. Chim. Japan* **37**, 662 (1964).

<sup>4</sup> A. ULUBELEN, S. ÖZTÜRK and S. ISILDATICI, *J. Pharm. Sci.* **58**, 1037 (1968).