

EXTRACTION AND ISOLATION OF FLAVONOIDS.—Dried material (500 g) was extracted sequentially with 85% and 50% aqueous MeOH. After filtration the extracts were combined and concentrated in vacuo. The aqueous syrup was partitioned first against CH₂Cl₂ and then EtOAc. The CH₂Cl₂ extract was concentrated and adsorbed onto Polyclar (Polycar AT, GAF Corp.). After drying, the resulting powder was charged onto a Polyclar column packed in H₂O-MeOEt-MeOH-Me₂CO (13:3:3:1). Flavonoids were eluted with the same solvent system. The EtOAc extract was chromatographed over a Polyclar column using the same procedure described for the CH₂Cl₂ extract. For both columns, fractions were collected on the basis of monitoring the bands with uv light. All bands were further separated by paper chromatography (Whatman 3MM) using 15% HOAc and TBA (t-BuOH-HOAc-H₂O, 3:1:1). Final purification of each compound for spectral analysis was by standard procedures (7) using 80% or 100% MeOH over Sephadex LH-20 columns. Compounds were identified by uv, ¹H nmr, ms (following acid hydrolysis for the glycosides), color reactions (7) and authentic sample comparisons. Previously unreported data for kaempferol 3-O-β-glucuronide: uv λ max (MeOH) 265, 300sh, 350; λ max (MeOH+NaOMe) 271, 330sh, 397; λ max (MeOH+AlCl₃) 268, 305sh, 375; λ max (MeOH+AlCl₃/HCl) 267, 300, 355, 400sh; λ max (MeOH+NaOAc) 369, 325sh, 378; λ max (MeOH+NaOAc/H₃BO₃) 261, 300sh, 372; ¹H nmr (as trimethylsilyl ether, CCl₄, TMS) δ 3.40-3.80 (4H, m), 5.67 (1H, m), 6.12 (1H, d, J=2.5 Hz), 6.23 (1H, d, J=2.5 Hz), 6.83 (2H, d, J=8.5 Hz), 7.93 (2H, d, J=8.5 Hz).

Details of identification are available upon request to the senior author.

TRIMETHYLSILYLATION.—This was performed as described by Mabry *et al.* (7).

HYDROLYSIS.—Hydrolysis of the glycosides with 0.1 N TFA (45 minutes) yielded the expected aglycones and sugar residues except for kaempferol 3-O-β-glucuronide. The hydrolysis of kaempferol 3-O-β-glucuronide was carried out by both β-glucuronidase and 1 N HCl (1 h).

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FLAVONOID AGLYCONES FROM *AGERATINA TOMENTELLA*

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As a part of our chemosystematic survey of the tribe Eupatorieae (Compositae) (1-4), we investigated the flavonoid aglycones of *Ageratina tomentella* (Schard.) R.M. King & H. Robinson. Six 6-methoxyflavones were isolated, namely: 6-methoxyluteolin and its 3'-methyl ether, 7,3'-dimethyl ether, 7,4'-dimethyl ether, and 7,3',4'-trimethyl ether, and 6-methoxyapigenin. The 6-methoxylation, 7-methoxylation, and 6,7-dimethoxylation appear to be characteristic of the main evolutionary line in the genus *Ageratina* (3-6). However, other *Ageratina* species produce flavonols alone or mixed with flavones (3-6).

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EXPERIMENTAL

PLANT MATERIAL.—Aerial parts of *A. tomentella* were collected 7 mi south of the border with Puebla along Hwy. 190 from Huajuapán to Izúcar, Oaxaca, Mexico, on October 5, 1984. Voucher material (Sundberg and Lavin 3035) is deposited in the Plant Resources Center at the University of Texas at Austin, Austin, Texas.

EXTRACTION AND ISOLATION OF FLAVONOIDS.—Ground, dried leaves and flowers (1.6 kg) were extracted with CH_2Cl_2 . The CH_2Cl_2 extracts were evaporated in vacuo until dry. The concentrated CH_2Cl_2 extract (126.8 g) was dissolved in CH_2Cl_2 and adsorbed onto a minimum amount of silica gel. After drying, the resulting powder was charged onto a silica gel column packed in hexane. The column was eluted with hexane and gradually increased to pure EtOAc in 10% increments. All fractions were further separated over Sephadex LH-20 using hexane- CH_2Cl_2 -MeOH (7:4:1). All compounds were purified over Sephadex LH-20 in 100% MeOH prior to analysis by uv, ^1H nmr, ms, color reactions on paper under uv light [7], and comparisons with authentic samples.

Details of the isolation and identification are available upon request to the major author.

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FLAVONOIDS FROM *GUTIERREZIA WRIGHTII*

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In our earlier chemotaxonomic studies in the *Gutierrezia-Xanthocephalum* complex (Compositae, tribe Astereae) (1-5), we reported that the two woody species, *Gutierrezia grandis* and *Gutierrezia microcephala*, produce large quantities of flavonoids, mostly with 6,8-oxygenation. In contrast, *Gutierrezia alamanii* var. *megalcephala*, a perennial herbaceous species, yields only a few flavonoids, all without 6,8-oxygenation and all in relatively low amounts. In our present study, we found that *Gutierrezia wrightii* A. Gray, an annual herbaceous species, contains mainly flavonoids without 6,8-oxygenation and, like *G. alamanii* var. *megalcephala*, does not produce them in large amounts. Fifteen flavonoids were isolated from *G. wrightii* including kaempferol and its 3-O- β -D-glucoside, quercetin and its 3-O- β -D-glucoside and 3-O- β -D-glucuronide, isorhamnetin and its 3-O- β -D-glucoside, 5,7,4'-trihydroxy-3,6-dimethoxyflavone, 3,5,7,3',4'-pentahydroxy-6-methoxyflavone, 3,5,7,4'-tetrahydroxy-6,3'-dimethoxyflavone, 5,7,4'-trihydroxy-3,6,8-trimethoxyflavone, 5,7-dihydroxy-3,6,8,4'-tetramethoxyflavone, 5,7,4'-trihydroxy-3,6,8,3'-tetramethoxyflavone, luteolin, and chrysoeriol. We present previously unreported spectral data for quercetin 3-O- β -D-glucuronide (6), 5,7,4'-trihydroxy-3,6,8-trimethoxyflavone (1), and 5,7-dihydroxy-3,6,8,4'-tetramethoxyflavone (7).

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