EXPERIMENTAL

Dried *B. officinalis* was purchased from a local store and from the Indiana Botanical Gardens, Hammond, Indiana. Seed was purchased from Stokes Nursery, and plants were grown to maturity for identification (CSU Herbarium No. 53478; D. Wilken, Department of Botany). Alkaloid isolations from garden-grown leaves and stems indicated similar alkaloid content whether fresh or dried. Roots contained the alkaloids as the free base, while fresh leaves had mainly *N*-oxides. A purchased, dried, bulk sample of plant fragments (496 g) yielded 48 mg of a crude alkaloid mixture (Zn reduction procedure). Tlc [with *o*-chloranil-Ehrlich's reagent visualization (5)] showed two major and four trace alkaloids. One major alkaloid was identified as lycopsamine by 360 MHz pmr, ms, and tlc comparison with a standard and with literature (6) values. The second was tentatively identified as supinidine viridiflorate (either cynaustine or amabiline) (6,7) by 360 MHz pmr and ms. A standard sample was not available for this identification.

Full details of isolation and identification are available from the senior author.

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SESQUITERPENE LACTONES OF ONE CHEMICAL RACE OF *HELIANTHUS MAXIMILIANI*

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Three distinct sesquiterpene lactone chemical races of the widely distributed perennial sunflower *Helianthus maximiliani* Schrader (Asteraceae, Heliantheae) have been established by tlc surveys (1). As part of an extended study of *H. maximiliani*, we report here three *trans trans*-$\Delta^{1,10}$, $\Delta^{4,5}$-germacradienolides from a north-central Texas population that represents one of these races whose chemistry has not previously been reported. Earlier studies have described a race producing heliangolides (2) and a race which produces guaianolides and germacrolides (3).

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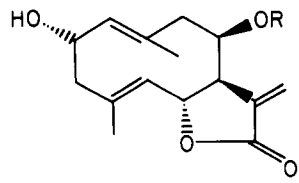

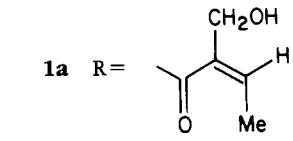

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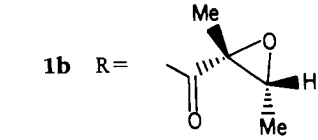

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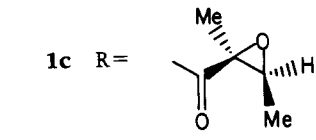

1c R=

The compounds reported here are the germacrolides desacetyleupaserrin (**1a**), the principle constituent, and two diastereoisomers of 8β-epoxyangeloyloxy-2α-hydroxycostunolide, namely, mollisorin B (**1b**), which has the 2′S,3′S conformation, and its 2′R,3′R diastereoisomer (**1c**). Desacetyleupasserrin was previously reported from several species of both *Helianthus* (4-7) and *Eupatorium* (8-10). Mollisorin B has been reported from several species of *Helianthus* (3, 5, 7, 11), while its 2′R,3′R diastereoisomer was found in *Helianthus resinosus* (7), *Helianthus pumilus* (11), and *Eupatorium mikanioides* (8). The assignment of the absolute configurations of the diastereoisomers follows the arguments of Herz (8).

EXPERIMENTAL

PLANT MATERIAL.—Leaves and flowers of *H. maximiliani* were collected by J. Gershenzon and E.S. Stewart on September 4, 1981, 5 miles north of Caradan (Mills Co.), Texas. Voucher specimen (J.G. #200) is on deposit in the Herbarium of the University of Texas at Austin.

EXTRACTION AND SEPARATION.—Air-dried aerial parts (3.45 kg dry weight) were extracted with CH_2Cl_2 and worked up in the usual manner (12). The resulting extract (91 g) was charged on a silica gel column eluted with a CH_2Cl_2-*iso*-PrOH gradient beginning with 100% CH_2Cl_2. Desacetyleupaserrin (15 g) was obtained from fractions eluted with CH_2Cl_2-*iso*-PrOH (95:5). It was further purified on a second silica gel column (hexane-EtOAc, 1:1 with increasing amounts of EtOAc up to 100%) and by preparative tlc (2 mm, silica gel; CH_2Cl_2-*iso*-PrOH, 10:1, and toulene-EtOAc, 5:6); it crystallized from CH_2Cl_2-EtOAc [mp 132-134°, uncorr.; lit 134-135° (4)]. Hplc analysis of the crude extract indicated that desacetyleupaserrin represents more than 90% of the sesquiterpene lactone material in this population (C-18, ultrasphere ODS; 25 cm×4.6 mm id; uv detector 254 nm and 215 nm; H_2O-MeOH-acetonitrile, 50:35:15).

Mollisorin B (43 mg) was isolated from fractions eluted from the primary column with CH_2Cl_2-*iso*-PrOH (99:1). It was crystallized from CH_2Cl_2-EtOAc, 1:1 [mp 164-167°, uncorr.; lit. 165-166° (5)]. Pmr indicated the presence of the 2′R,3′R diastereoisomer mixed with the crystalline mollisorin B. The two diastereoisomers were separated and purified from combined primary column fractions using reverse phase hplc (C-18, ultrasphere ODS; 25 cm× 10 mm id; uv detector 254 nm; H_2O-MeOH-acetonitrile, 50:35:15). This procedure yielded 16 mg of the 2′R,3′R diastereoisomer, which was obtained as a colorless gum.

IDENTIFICATION.—The reported compounds were identified by comparison of spectral data and mp with published values.

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