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ISOLATION AND IDENTIFICATION OF THE HYPOGLYCEMIC AGENT, CARBOXYATRACTYLATE, FROM *XANTHIUM STRUMARIUM*

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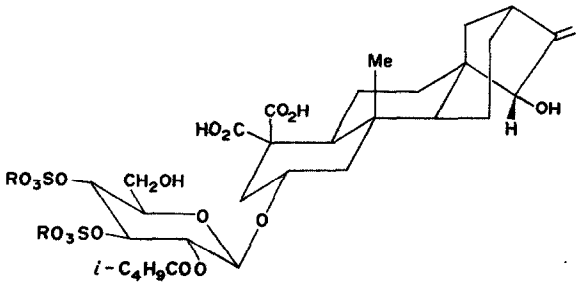
Key Word Index—*Xanthium strumarium*; Compositae; cocklebur; hypoglycemia; potassium carboxyatractylate; carboxyatractyloside.

Plant. Xanthium strumarium burrs (cocklebur). *Source.* Oxford, Mississippi. *Previous work.* Sesquiterpenes [1], essential oils [2], sitosterol, stigmasterol, and campesterol [3,4] and a hypoglycemic agent [5,6]. *Toxicity.* Potassium carboxyatractylate is a very toxic hypoglycemic agent ($LD_{50} = 10.7$ mg/kg; IP in mice) which strongly inhibits translocation of adenine nucleotides across the mitochondrial membrane [7].

Present work. The whole, ground burrs were extracted by successive percolation with hexane, $CHCl_3$, EtOH, and H_2O. The H_2O extract was flash evaporated *in vacuo* at 50°, and the residue was treated with tetrahydrofuran:H_2O; 7:3. This solvent mixture was decanted and removed *in vacuo* at 50°. This residue was triturated with cold MeOH to yield 0.02% of an insoluble glycoside.

The glycoside was purified by forming the potassium salt and recrystallizing from H_2O; ν_{max}^{KBr} 3480, 2980, 1735, 1640, 1270, 1040, 1000, and 800 cm^{-1}. Hydrolysis of the glycoside in aq KOH (20%, reflux for 8 hr.) yielded a sugar, sulfate, isovalarate, and, upon neutralization, an acidic aglycone. Treatment of the aglycone with CH_2N_2 yielded a compound with a MI of *m/e* 392. Subsequent silylation of this derivative yielded a compound with a MI of *m/e* 536.

Direct comparison (mmp, IR, NMR, $[\alpha]_D$, and TCL) of the potassium salt of the glycoside with an authentic sample of potassium carboxyatractylate (**1b**) [8] confirmed the identity of the isolated glycoside as carboxyatractyloside (**1a**).



(1a) R = H
(1b) R = K

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REFERENCES

1. Winters, T. E., Geissman, T. A. and Safir, D. (1969) *J. Org. Chem.* **34,** 153.
2. Baslas, R. K. (1970) *Flavour Ind.* **1,** 474.
3. Mukerje, S. (1970) *Indian J. Pharm.* **32,** 48.
4. Khafagy, S. M. and Metwally, A. M. (1971) *Planta Med.* **19,** 234.
5. Dhar, M. L., Dhar, M. M., Dhawan, B. N., Mehrotra, B. N. and Ray, C. (1968) *Indian J. Exp. Biol.* **6,** 232.
6. Jupiecki, F. P., Ogzewalla, C. D. and Schell, F. M. (1974) *J. Pharm. Sci.* **63,** 1166.
7. Luciani, S., Martini, N. and Santi, R. (1971) *Life Sci.* **10,** 961.
8. Danieli, B., Bombardelli, E., Bonait, A. and Gabetta, B. (1972) *Phytochemistry* **11,** 3501.

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TRITERPENOIDS AND FATTY ACIDS FROM SOME MOSSES. OBTUSIFOLIOL FROM *RACOMITRIUM LANUGINOSUM**

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Key Word Index—Musci; alkanes; tetra- and pentacyclic triterpenoids; obtusifoliol; phytosterols; fatty acids.

Mosses. Anomodon viticulosus Hook. and Tayl; *Brachythecium rivulare* B.S.G.; *Campylopus introflexus* (Hedw.) Brid.; *Ctenidium molluscum* (Hedw.) Mitt.; *Raco-*

*Part 5 in the series "Triterpenes from Mosses". For Part 4 see Marsili, A., Morelli, I., Bernardini, C. and Pacchiani, M. (1972) *Phytochemistry* **11,** 2003.