$Pb(OAc)_2$–H_2S, and adjustment of the final vol. to 350 ml, the betaines were precipitated as a globular mass of periodides (50 g) with hydrogen periodide (120 ml 16% I_2 in 14% HI plus 32 g I_2 in 35 ml conc. HI). This was transformed to a solution of iodides as above, which was shaken with an aqueous suspension of excess silver chloride and filtered. Picric acid (5·3 g) was added to the total filtrate (220 ml) at the boiling point, resulting in the recovery, after purification of combined fractions by recrystallization from water, of (±)-stachydrine picrate, m.p. 198–199° (199–200°)[9] (7·2 g, stachydrine representing 0·47% of dry wt extracted). (Found: C, 41·77; H, 4·18; N, 15·21. Calc. for $C_{13}H_{16}N_4O_9$: C, 41·94; H, 4·30; N, 15·05%). The identity was established by comparison with a synthetic sample and spectroscopically: IR (KBr) ν 1725 cm^{-1}; NMR (4:1 $CDCl_3/d_6$-DMSO) τ 7·2–8·1 m [$C(3)H_2 + C(4)H_2$]; 6·82 *s* [NMe]; 6·59 *s* [NMe]; 6·21 *t* [$C(5)H_2$]; 5·41 *t* [C(2)H]; 1·55 *s* [OH]; 1·39 [2 × aromatic CH].[12] The (±) stachydrine isolated represents only some 25% of the total betaines present in the rhizome.

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Phytochemistry, 1973, Vol. 12, pp. 1180 to 1181. Pergamon Press. Printed in England.

STEROL CONTENT OF *SPINACIA OLERACEA*

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Key Word Index—*Spinacia oleracea*; Chenopodiaceae; spinach; sterols; α-spinasterol; stigmastenol; stigmastanol.

Hart[1] reported the isolation of sterols from the spinach, *Spinacia oleraceae* L. fat which was subsequently identified as α-spinasterol.[2]

Processing fresh leaves of Egyptian spinach in the usual manner gave 0·003% unsaponifiable matter (on wet wt basis). This material was resolved by chromatography on an alumina column. Elution with *n*-hexane followed by C_6H_6 containing 15% $CHCl_3$ gave a crystalline material which exhibited a positive Liebermann–Burchardt test and a yellow colour reaction with the tetranitromethane reagent. The product was thought to be pure as judged from the constant m.p. (147–149°) and optical rotation, $[\alpha]_D$ −45°. The acetate derivative, m.p. 169°, $[\alpha]_D$ −8°, and the benzoate derivative, m.p. 187°, $[\alpha]_D$ −10·7° were also prepared. The acetylated material was inspected by TLC[3] and found to be homogenous,

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[1] HART, M. C. (1929) *J. Biol. Chem.* **82,** 1116.
[2] HART, M. C. and HEYL, F. H. (1932) *J. Biol. Chem.* **95,** 311.
[3] COPIUS-PEEREBOOM, J. W. (1964) *Z. Anal. Chem.* **205,** 325.

inseparable from and identical with α-spinasteryl acetate, R_f 0·58. Examination of the benzoate derivative, however, revealed that the material is actually composed of three sterols having R_f 0·34 (I), 0·53 (II), 0·65 (III).

The MS[4,5] of the isolated sterol mixture indicated that sterol I is a $C_{29}H_{48}O$ sterol (M^+, *m/e* 412), containing two double bonds, which exhibited the fragmentation pattern expectable for α-spinasterol. The principal features of this pattern include the loss of a monounsaturated $C_{10}H_{19}$ side chain alone and together with 42 m.u., and the expulsion of butadiene from ring *A* in dehydroxylated ion species. Sterol II is stigmastenol, $C_{29}H_{50}O$ (M^+, *m/e* 414) containing one double bond located in the steroid nucleus. This was evidenced by the total loss of the saturated side chain $C_{10}H_{21}$. Sterol III is stigmastanol, $C_{29}H_{52}O$ (M^+, *m/e* 416) which is fully saturated. The observed fragmentations are those expectable for saturated sterols and include products resulting from break down of ring *A* in the dehydroxylated ion species by a retro-Diels–Alder type of reaction.

The unsaponifiable fraction of the seed fat afforded a sterol mixture which was found to be identical with that isolated from the leaves.

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Phytochemistry, 1973, Vol. 12, pp. 1181 to 1182. Pergamon Press. Printed in England.

5,4′-DIHYDROXY-3,7-DIMETHOXYFLAVONE FROM *AMBROSIA ERIOCENTRA*

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Key Word Index—*Ambrosia eriocentra*; Compositae; flavonol; kaempferol 3,7-dimethyl ether.

Plant. *Ambrosia eriocentra* (Gray) Payne. *Source*. Collected by R. J. Barr on 3 May 1965, and on 3 May 1967, near Wickenburg, Maricopa County, Arizona. (Barr No. 65-191 and 67-143 on deposit in herbarium of Florida State University.) *Previous work*. No crystalline sesquiterpene lactones.[1]

Isolation and identification. The above-ground parts of Barr No. 65-143, wt 9·8 kg, were extracted with $CHCl_3$ and worked up in the usual fashion.[2] The crude gum, wt 31·9 g, was only sparingly soluble in benzene or $CHCl_3$. It was dissolved in 1:1 C_6H_6–$CHCl_3$ with the aid of some EtOH and chromatographed over 500 g of silicic acid in the usual manner. 24 g of gum was recovered from the first 4 l. of eluate; subsequent fractions consisted of gummy mixtures. The gum from the first 4 l. of eluate was redissolved in

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[2] HERZ, W. and HÖGENAUER, G. (1962) *J. Org. Chem.* **27**, 905.