242 mμ, log ϵ 4.23). Mild alkaline hydrolysis of V, VI and VII afforded 6α-fluoro compound "S" (VII) (m.p. 203–205°, $[\alpha]_D$ +135°, λ_{max} 236 mμ, log ϵ 4.21), 6β-fluoro compound "S" (IX) (m.p. 222–224°, $[\alpha]_D$ +14°, λ_{max} 234 mμ, log ϵ 4.09) and Δ^1-dehydro-6α-fluoro compound "S" (X) (m.p. 210–212°, $[\alpha]_D$ +66°, λ_{max} 241 mμ, log ϵ 4.22), respectively.

Adrenal incubation[6] of both VIII and X followed by monoacetylation at C-21 gave, in good yield, 6α-fluorohydrocortisone acetate (XI) (m.p. 215–217°, $[\alpha]_D$ +149° (diox.), λ_{max} 237 mμ, log ϵ 4.23) and 6α-fluoroprednisolone acetate (XII) (m.p. 235–237°, $[\alpha]_D$ +114° (diox.), λ_{max} 242 mμ, log ϵ 4.25), respectively. Selenium dioxide oxidation[5] of XI gave XII. Chromic acid oxidation of XI and XII provided 6α-fluorocortisone acetate (XIII) (m.p. 215–216°, $[\alpha]_D$ +190°, λ_{max} 233 mμ, log ϵ 4.21) and 6α-fluoroprednisone acetate (XIV) (m.p. 228–230°, $[\alpha]_D$ +142° (diox.), λ_{max} 237 mμ, log ϵ 4.23).

An alternative completely chemical route to the 6-fluoro corticoids proceeded from cortisone acetate 3-monoketal (XV).[7] Acetylation of XV at C-17 and peracid epoxidation of the resulting 17,21-diacetate (XVI) (m.p. 180–182°, $[\alpha]_D$ −20°) afforded the 5α,6α-epoxide (XVII) (m.p. 233–234°, $[\alpha]_D$ −18°). Boron trifluoride opening of XVII gave the corresponding 5α-hydroxy-6β-fluoro-3-ketal (XVIII) (m.p. 231–232°, $[\alpha]_D$ −9°) which was directly converted by acid treatment to 6α-fluorocortisone 17,21-diacetate (XIX) (m.p. 270–272°, $[\alpha]_D$ +108°, λ_{max} 233 mμ, log ϵ 4.20). Alkaline hydrolysis of XIX and subsequent monoacetylation gave XIII. Milder acid treatment of XVIII afforded 6β-fluorocortisone 17,21-diacetate (XX) (m.p. 212–213°, $[\alpha]_D$ +62°, λ_{max} 230 mμ, log ϵ 4.09).

In the anti-inflammatory and thymolytic assays,[8] 6α-fluorocortisone acetate (XIII), 6α-fluorohydrocortisone acetate (XI), 6α-fluoro-prednisolone acetate (XII) and 6α-fluoroprednisone acetate (XIV) had respective activities of (10,6), (10,8), (20,62), (20,23) times that of hydrocortisone acetate. In the adrenalectomized nonsalt loaded rat XI, XII and XIV promoted sodium excretion.[9]

The details of this work together with the syntheses of other 6-fluoro steroid hormones[4b,10] will be described in future publications.

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(6) A. Zaffaroni, H. J. Ringold, G. Rosenkranz, F. Sondheimer, G. H. Thomas and C. Djerassi, THIS JOURNAL, **80**, October (1958).

(7) R. Antonucci, S. Bernstein, M. Heller, R. Lenhard, R. Littell and J. H. Williams, *J. Org. Chem.*, **18**, 70 (1953).

(8) Anti-inflammatory (cotton pellet implant) and thymolytic activity in adrenalectomized rat, oral route, hydrocortisone acetate = 1. We wish to thank Dr. R. Dorfman of the Worcester Foundation for these assays.

(9) Mineral assays by Dr. R. Dorfman and the Endocrine Labs., Madison, Wisconsin.

(10) By a similar series of reactions there was prepared 6α-fluoro-17α-acetoxy-progesterone (m.p. 249–250°, $[\alpha]_D$ +56°, λ_{max} 236 mμ, log ϵ 4.21) and 1-dehydro-6α-fluoro-17α-acetoxyprogesterone (m.p. 258–261°, $[\alpha]_D$ +24°, λ_{max} 241 mμ, log ϵ 4.18) which possess high progestational activity. Thus the former in the Clauberg assay exhibits oral activity equal to 17α-ethynyl-19-nortestosterone (Norlutin).

TRANSPARENT $BaFe_{12}O_{19}$ AND $SrFe_{12}O_{19}$

Sir:

By a solid state reaction of barium fluoride and ferric oxide in the ratio 1:6, under oxygen, a red, transparent, single crystalline form of $BaFe_{12}O_{19}$ was obtained. The hexagonal platelets, approximately 50 microns thick and up to 2 mm. in diameter, are perfectly suitable for the observation and study of Weiss domains, which in the virgin state are about 1 micron wide. As an alternate technique, the reaction of ferric oxide in molten barium chloride or strontium chloride under oxygen, also yielded transparent platelets of $BaFe_{12}O_{19}$ and $SrFe_{12}O_{19}$. The compositions of the reaction products were established by chemical analysis and single crystal X-ray studies. An apparently similar form of $BaFe_{12}O_{19}$ was described recently by C. Kooy,[1] without indicating the method of preparation.

(1) C. Kooy, *Philips Technical Review*, **19**, 286 (1958).

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THE RADIOLYTIC SYNTHESIS OF THE *cis*- AND *trans*-ISOMERS OF 1,2-DICHLOROETHYLENE OXIDE

Sir:

In the course of some investigations on the radiation chemistry of the symmetrical dichloroethylenes, the *cis* and *trans* forms of 1,2-dichloroethylene oxide have been produced. These compounds have not been reported previously, and in view of the current interest in radiation utilization, we are presenting preliminary data on the radiolytic synthesis and physical properties of these compounds.

The *cis* and *trans* forms of 1,2-dichloroethylene were irradiated in glass cells *in vacuo* with 40-Mev. helium ions impingent on the liquid. After irradiation, the low-boiling products plus the bulk of the 1,2-dichloroethylene were stored in glass-stoppered bottles in the presence of air after preliminary analysis for high-boiling products. In samples that had stood several months, two peaks were found in GLP chromatograms (nonyl phthalate column) that were not present in samples chromatogrammed immediately after irradiation. These peaks were concentrated in the pot liquid by distillation through a small Vigreux column and were further concentrated and purified by running repeat GLP chromatograms of the pot liquid, collecting the respective peaks each time. By this means samples of 1 g. and 0.2 g. of the two respective peak materials were isolated in relatively pure form. These are referred to as dichloroethylene oxide I and dichloroethylene oxide II according to their respective GLP chromatographic emergence times.

The relative yield of oxide I to oxide II from *trans*-1,2-dichloroethylene was about 4.6:1. The ratio from irradiated *cis*-1,2-dichloroethylene was smaller but has not been well determined because of the much lower yield from this isomer.

The compounds isolated have been characterized as the *cis* and *trans* isomers of 1,2-dichloroethylene