(IX) is identical with that of a known sample of 17α-hydroxycorticosterone acetate. 1,3-Propanediol may be substituted for ethylene glycol in the over-all process with success. Compound F acetate was prepared by this method in approximately 15% over-all yield from 11α-hydroxyprogesterone, the synthesis of which by the bio-oxygenation of progesterone was recently described in a communication from these laboratories.[4] Similarly treatment of VI as given for VIII above, gave the hitherto unreported 11β,17α-dihydroxy-4-pregnene-3,20-dione (21-desoxy Compound F), m.p. 225–228°, $[\alpha]^{24}\mathrm{D}$ $+136°$ (acetone); $\lambda_{\max.}^{\mathrm{EtOH}}$ 241 mμ, $E = 15{,}500$. (*Anal.* Calcd. for $C_{21}H_{30}O_4$: C, 72.80; H, 8.73. Found: C, 72.72; H, 8.70). Further details of this work will be published shortly.

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[4] D. H. Peterson and H. C. Murray, This Journal, **74**, 1871 (1952); D. H. Peterson, H. C. Murray, S. H. Eppstein, L. M. Reineke, A. Weintraub, P. D. Meister and H. M. Leigh, *ibid.*, **74**, 5933 (1952); U. S. Patent 2,602,769, July 8, 1952.

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ELECTRON DISTRIBUTION IN TRIPHENYLMETHYL:[1] HYPERFINE STRUCTURE OF THE PARAMAGNETIC RESONANCE ABSORPTION OF $(C_6H_5)_3C^{13}$*

Sir:

Hyperfine structure in the paramagnetic resonance absorption of free radicals is associated with interactions between electronic and nuclear magnetic moments.[2] From the magnitudes of the hyperfine splittings, deductions concerning the electronic distribution may be made.

The abundant isotope of carbon, C^{12}, has zero magnetic moment and leads to no hyperfine splittings. C^{13}, on the other hand, possesses spin $^1/_2$ and a magnetic moment of 0.7021 nuclear magneton. One C^{13} nucleus in a free radical molecule should split the paramagnetic resonance absorption spectrum into two components. The magnitude of the splitting is dependent on the average magnetic field at the nucleus contributed by the spin and orbital motion of the electron.[3] This field is determined in part by the average of the reciprocal of the cube of the distance between electron and nucleus.

We have examined the paramagnetic resonance absorption of triphenylmethyl containing C^{13} in the methyl position. The sample contained 53 atom per cent. of C^{13} in this position. The spectrum in dilute solution (10^{-3} M in hexaphenylethane) consists of three lines, equally spaced.[4] The interval between the low field line and the high field line is 22 ± 5 oersteds. The central line arises from molecules containing C^{12} in the methyl position. The intensities are consistent with the isotopic abundance of the original sample. Triphenylmethyl prepared from materials of normal isotopic abundance by a procedure identical with the one used for the C^{13} containing compound gave a single line.

The splitting which we have observed arises from an average field at the C^{13} nucleus of about 3×10^4 oersteds.[5] This field corresponds to a separation of 0.7 ångström units between the unpaired electron and the nucleus of the methyl carbon atom. This interpretation must be viewed with caution. Further work, now being pursued, on the anisotropies in the spectra of single crystals dilute in the free radical constituent, is essential for a more quantitative description of the electronic distribution.

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[1] Assisted by the joint program of O.N.R. and A.E.C.

[2] G. E. Pake, J. Townsend and S. I. Weissman, *Phys. Rev.*, **85**, 682 (1952); C. A. Hutchison, Jr., R. C. Pastor and A. G. Kowalsky, *J. Chem. Phys.*, **20**, 534 (1952).

[3] In the case here considered the orbital contribution is unimportant.

[4] The measurements were made at 9000 mc. in fields close to 3200 oersteds.

[5] This number may be compared with a field of 6×10^4 oersteds at the nitrogen nucleus in nitric oxide.

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STUDIES ON ADRENOCORTICOTROPIN. V. THE ISOLATION OF CORTICOTROPIN-A

Sirs:

By means of a 200-plate counter-current distribution of a fraction derived from unhydrolyzed hog pituitary extract by means of an ion exchange column[1] we have obtained a product which appears to be pure.

In this work we have started with the slow-moving fraction from Amberlite XE-97 columns, referred to as ID in a previous publication.[1] The system *s*-butyl alcohol/0.2% trichloroacetic acid has been used for all the distribution studies reported here.

A preliminary 24-plate run showed that fraction ID gave a single peak with a distribution coefficient of about 1.7. Comparison with the theoretical curve suggested a purity of 80–85% with most of the impurities running slower than the main peak and imperfectly separated from it. On the basis of this information, a 200-plate run was made on a large batch of material. Because of the tendency of ACTH to give broad peaks in concentrated solution, the 300-mg. sample was scattered over six tubes. As before, a single peak was obtained, broader than theoretical and with a distribution coefficient of about 1.75. Material from the center of this peak, falling well within the limits of the theoretical curve for $k = 1.75$ was recovered as the trichloracetate and used for the remainder of this study.

Figure 1 shows a re-distribution of this material

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[1] W. F. White and W. L. Fierce, This Journal, **75**, 245 (1953).