nection other ions of oxidation number three are of particular interest. Of these, aluminum, chromium and rare earths have been tested to date and they all were found to be practically not adsorbed from strong HCl solutions. To illustrate the effectiveness of the method an iron impurity (30 mg./l.) was separated from a 2 *M* aluminum chloride solution in 3 *M* HCl using a 10 cm. column of 0.024 sq. cm. cross-section. After passage of 30 ml. of solution (flow rate *ca.* 0.25 ml. $cm.^{-2}$ $min.^{-1}$), when the experiment was interrupted, iron could be detected (visually) only in the first 0.6 cm. of the column. Thus large volumes of solution could be processed with small amounts of resin.

OAK RIDGE NATIONAL LABORATORY GEORGE E. MOORE
OAK RIDGE, TENNESSEE KURT A. KRAUS

RECEIVED NOVEMBER 6, 1950

THE CRYSTAL STRUCTURE OF A SIGMA PHASE, FeCr[1]

Sir:

Despite the importance of the sigma phase in transition group alloys, the crystal structure of the phase has not heretofore been determined, primarily because the materials, prepared by solid-state transition, are microcrystalline and not well suited for single crystal work, while powder photographs have proved too complicated for satisfactory interpretation.

However, we have succeeded in isolating from a specimen of σ-FeCr (46.5 at. % Cr) two single crystals roughly 0.1 mm. in size. Single crystal and powder photography gave a 30-atom primitive tetragonal cell (Laue symmetry D_{4h}), with $a_0 = 8.799$ Å. and $c_0 = 4.546$ Å.

The only observable ($hk0$) reflections (aside from a few faint ones at large Bragg angles) within the CuKα limit are uniformly strong, and are those ({410}, {330}, {550}, {720}, {820}, {660}, {960}, {11·1·0}, and {10·5·0}) which would result from fifteen atoms at the points of a slightly distorted hexagonal net (with the following typical (x,y) coördinates with respect to a vertical twofold axis: I (1) (0, 0); II (2) (1/5, 1/5); III (2) (2/5, 2/5); IV (2) (2/3, 1/3); V (4) (7/15, 2/15); VI (4) (11/15, 1/15)) plus fifteen others in an equivalent net rotated 90° with respect to the first. The relative positions of the two nets are as in an "ideal" structure with space group $D_{4h}^{14} - P4/mnm$, as indicated by *n*-glide extinctions in ($0kl$) Weissenberg data (CuKα).

That "ideal" structure is ruled out by the general (hkl) intensities. However, atoms IV in the second layer have (x,y) coördinates (1/6, 1/6, etc.) very close to those of II in the first layer, and if the eight atoms II and IV are moved to new positions 8(j) with $x = 11/60$, $z = 1/4$, a structure with space group D_{4h}^{14} is obtained which gives a satisfactory qualitative accounting for the ($hk0$), ($hk1$), ($hk2$), and ($hk3$) Weissenberg intensities (CuKα), including a prediction of weak ($hk0$) reflections which are perhaps not altogether inconsistent with those observed. Until intensity work (now in progress) has yielded quantitative data, a choice between this space group and $D_{2d}^{3} - P\bar{4}n2$ (obtainable with small distortions of the above structure) or $C_{4v}^{4} - P4nm$ (permitting a closely related structure derived from the "ideal" structure by shifting II and IV about 1/4 in z) cannot be made. All other space groups have been ruled out.

The identities of the atoms are not yet known. They may be very difficult to determine because iron and chromium have nearly the same scattering factors.

This work is being continued. We are indebted to Professor Pol Duwez and Mr. Paul Pietrokowsky of this Institute for the sample of σ-FeCr. We are grateful to Professor Linus Pauling for helpful discussions, and to Miss Linda Pauling and Mrs. Nan Arp for computational assistance.

GATES AND CRELLIN LABORATORIES OF CHEMISTRY
CALIFORNIA INSTITUTE OF TECHNOLOGY
PASADENA, CALIFORNIA DAVID P. SHOEMAKER
CONTRIBUTION NO. 1488 BROR GUNNAR BERGMAN

RECEIVED NOVEMBER 11, 1950

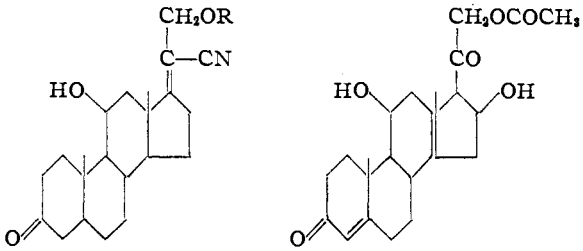
SYNTHESIS OF 11-HYDROXYLATED CORTICAL STEROIDS. 17(α)-HYDROXYCORTICOSTERONE

Sir:

We wish to report the synthesis of 17(α)-hydroxycorticosterone otherwise known as Reichstein's Compound M[1] or Kendall's Compound F,[2] a substance found by preliminary studies[3] to have therapeutic activity similar to Cortisone.

The biosynthesis of 17(α)-hydroxycorticosterone from 11-desoxy-17(α)-hydroxycorticosterone has been demonstrated using techniques of perfusion in the isolated beef adrenal gland[4] and of incubation with adrenal homogenates.[5]

We have synthesized 17(α)-hydroxycorticosterone starting with 20-cyano-17-pregnene-21-ol-3,-



(I) R = H
(Ia) R = CH_3CO

(II)

(1) Work done in part under a contract with the Office of Naval Research and in part under a program sponsored by the Carbide and Carbon Chemicals Corporation.

(1) Reichstein, *Helv. Chim. Acta*, **20**, 953 (1937).

(2) Mason, Hoehn and Kendall, *J. Biol. Chem.*, **124**, 459 (1938).

(3) Hench, Kendall, Slocumb and Polley, *Arch. Int. Med.*, **85**, 545 (1950).

(4) Hechter, Jacobsen, Jeanloz, Levy, Marshall, Pincus and Schenker, *Arch. Biochem.*, **25**, 457 (1950).

(5) McGinty, Smith, Wilson and Worrel, *Science*, **112**, 506 (1950).