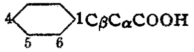
and acetate, acetate carbon is extensively utilized in the synthesis of glutamic and aspartic acids, leucine and lysine, but not for the synthesis of phenylalanine and tyrosine. Hence neither acetate nor intermediates derived from acetate (α-ketoglutarate, oxaloacetate) could have been precursors for the benzenoid amino acids. On the other hand, carbon from 1-C^{14} glucose appears in the benzene rings and in the β positions of the side chains of the two amino acids (Table II). Carbon atoms 1, 3 and 5 of the ring obtained as bromopicrin after conversion of tyrosine to picric acid[3] contained no significant radioactivity. Carbons 2 and 6 of the ring, obtained as bromopicrin after conversion of phenylalanine to *p*-bromo, *o*-nitroacetanilide had a specific activity which accounted for about three-fourths of the total activity in the benzene ring.

TABLE II

DISTRIBUTION OF C^{14} IN PHENYLALANINE AND TYROSINE IN PROTEIN FROM YEAST GROWN ON 1-C^{14} GLUCOSE

 1CβCαCOOH	Tyrosine[c] c.p.m. C^{14d}	Phenylalanine[c] c.p.m. C^{14d}
Total	26 ± 3	22 ± 2
COOH[a]	3 ± 1	1 ± 1
β-Carbon[b]	77 ± 8	97 ± 10
Picric acid	23 ± 8	.....
2,4-Dinitroaniline	....	17 ± 2
$C_{1,3,5}$	2 ± 1	.....
$C_{2,6}$	....	37 ± 4
C_2 or C_6 calcd.	....	74 ± 7

[a] By tyrosine decarboxylase and ninhydrin respectively. [b] By decarboxylation of *p*-hydroxybenzoic acid and of benzoic acid respectively. [c] Degradations were performed after dilution of the isolated amino acids by non-isotopic phenylalanine and tyrosine. [d] All samples counted after conversion to $BaCO_3$ and corrected to infinite thickness.

If two isotopically equilibrated 3-carbon intermediates from 1-C^{14} glucose condensed, any resulting six-membered ring would contain C^{14} at adjacent carbon atoms or in para position. The observed isotope distribution rules out this possibility. If, on the other hand, benzene rings were formed by direct cyclization of 1-C^{14} glucose, C^{14} should be present in one position only. Our data indicate that at least three-fourths of the radioactivity in the benzene rings is present in positions 2 and 6. Since no reactions of glucose other than formation of two-carbon units are known which would lead to the appearance of C^{14} in meta positions, and since acetate is excluded as an intermediate it is likely that only C_2 or C_6 is labeled. The data are therefore best explained by assuming a cyclization of glucose to form the benzene rings of phenylalanine and tyrosine. This conclusion is in accord with data of Baddiley, Ehrensvärd, *et al.*,[3] on tyrosine synthesis in *Torulopsis utilis* grown on acetate as the sole substrate, assuming that under their conditions acetate was utilized by way of glucose.

It can be calculated from the data in the tables that the specific activity in the benzene rings was less than that of the glucose. The possibility that the inositol present in the nutrient medium is responsible for this isotope dilution is being investigated.

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UNIVERSITY OF CHICAGO CHARLES GILVARG[4]
CHICAGO, ILLINOIS KONRAD BLOCH

RECEIVED NOVEMBER 11, 1950

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[3] F. Baddiley, G. Ehrensvärd, E. Klein, L. Reio and E. Saluste, *J. Biol. Chem.*, **183**, 777 (1950).

[4] Public Health Pre-doctoral fellow.

ADSORPTION OF IRON BY ANION EXCHANGE RESINS FROM HYDROCHLORIC ACID SOLUTIONS[1]

Sir:

As part of a more general study of the anion exchange behavior of metal ions in chloride and fluoride solutions it was found that iron(III) can be strongly adsorbed from relatively concentrated hydrochloric acid solutions.

The results of a series of elution experiments with tracer Fe^{59} and 0.023 cm.^{2} columns of Dowex-1 are shown in Table I. The elution constants $E = dA/V$ (where d = distance in cm. an adsorption band moves when V ml. of eluent pass through a column of cross sectional area A sq. cm.) were found to decrease rapidly with increasing hydrochloric acid concentrations above *ca.* 1 M and to reach the very small values of 2×10^{-4} in *ca.* 9 M HCl. In this respect Fe(III) is quite similar to Pa(V) which was previously discussed.[2] Adsorption is probably due to the formation of a strongly adsorbable negatively charged iron complex (or complexes) *e.g.*, $FeCl_4^-$ of probable negative charge minus one, the concentration of which increases with increasing hydrochloric acid concentration.

TABLE I

ADSORPTION OF Fe(III) BY DOWEX-1 FROM HYDROCHLORIC ACID SOLUTIONS

M HCl	E	D[a]	M HCl	E	D
0.5	1.74	0.16	4.0	0.0148	67
1.0	1.34	0.33	5.0	.0037	270
2.0	0.42	1.94	6.0	.00218	460
3.0	0.038	26	9.0	.00017	5900

[a] Calculated from equation $E = 1/(i + D)$ where i is the fractional interstitial space (*ca.* 0.42) and D is the volume distribution coefficient (amount per ml. of resin/amount per ml. of solution).

In view of this strong adsorbability of iron it can easily be separated from metal ions which do not form negatively charged complexes (*e.g.*, alkali metals, alkaline earths, etc.). In this con-

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[1] This document is based on work performed for the Atomic Energy Commission at the Oak Ridge National Laboratory. Part of this work was previously reported in report ONRL-286 (June, 1949).

[2] K. A. Kraus and G. E. Moore, THIS JOURNAL, **72**, 4293 (1950).

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 (*hk*0) 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 two-fold 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 (0*kl*) 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 (*hk*0), (*hk*1), (*hk*2), and (*hk*3) Weissenberg intensities (CuKα), including a prediction of weak (*hk*0) 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 $D_{2d}^{8} - P\bar{4}n2$ (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

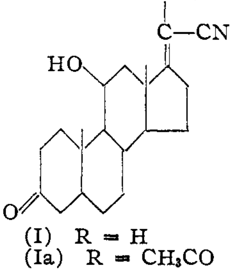
SYNTHESIS OF 11-HYDROXYLATED CORTICAL STEROIDS. 17(α)-HYDROXYCORTICOSTERONE

Sir:

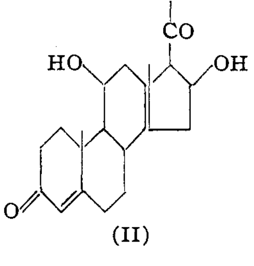
We wish to report the synthesis of 17(α)-hydroxycorticosterone otherwise known as Reichstein's Compound M[1] or Kendall's Compound F,[2] a substance found by preliminary studies[3] to have therapeutic activity similar to Cortisone.

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$_3$CO



(II)

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[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).