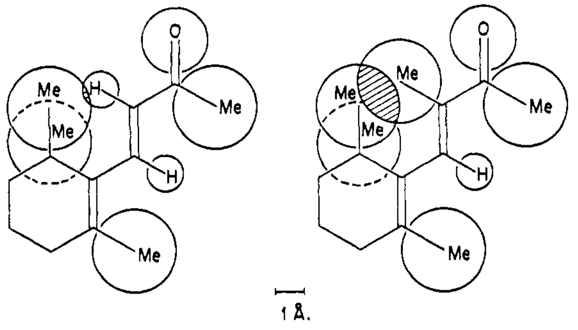

β-Ionone *β-i-Methylionone*

Fig. 1.

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SOME CHARACTERISTICS OF A CRYSTALLINE COMPOUND DERIVED FROM VITAMIN B_{12}

Sir:

Vitamin B_{12},[1] 23.7 mg., was stirred with 80 mg. of platinum catalyst in an atmosphere of hydrogen for twenty hours at atmospheric pressure as described by Kaczka and co-workers.[2,3] During the period, 1.12 ml. of hydrogen was taken up, corresponding to about 3 mols. The color changes described elsewhere[2] were noted. About 18 mg. of red crystals (I) was obtained from acetone–water solutions of the resultant product. Further purification, accompanied by the removal of brown material, was accomplished by silicic acid chromatography, using the same procedure employed for vitamin B_{12b},[4] followed by recrystallization. The absorption spectrum maxima following this procedure and after drying at 110° for twenty hours at 1 mm. over phosphorus pentoxide, were as follows: 273 mμ, $E^{1\%}_{1\,\mathrm{cm.}}$ 132; 351 mμ, 159; 525 mμ, 54. These maxima and extinction coefficients are characteristic of vitamin B_{12b}. Upon adding sodium hydroxide to a concentration of 0.01 *N*, the absorption spectra of I and of vitamin B_{12b} were found to undergo identical bathochromic shifts of the two main absorption bands to 357 mμ and 536 mμ, respectively. No such shifts were observed with vitamin B_{12}. The infrared absorption spectrum[5] of I was similar to that of vitamin B_{12b} and both showed the absence of a band at 2140 cm.$^{-1}$. The presence of a band at this point was observed with vitamin B_{12}.[6] The biological activity of I was compared with those of vitamins B_{12} and B_{12b} using *L. leichmannii*[7] and chicks.[8] All three preparations had the same activity within the limits of error of the assay methods. In contrast, vitamin B_{12a}, also produced by hydrogenation of vitamin B_{12},[2] was reported to have only 20% of the activity of vitamin B_{12} for *L. leichmannii*[2,9] and 30 ± 15% of the activity vitamin B_{12} for chicks.[2] A band in the absorption spectrum of vitamin B_{12a} was reported at 315 mμ ($E^{1\%}_{1\,\mathrm{cm.}}$ 80).[2] This band was absent from the absorption spectra of both I and vitamin B_{12b}.[6] The present experimental results indicate that, under our conditions, vitamin B_{12b} was produced by hydrogenation of vitamin B_{12}.

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(1) Purchased from Merck and Co., Rahway, New Jersey.

(2) E. Kaczka, D. E. Wolf and K. Folkers, THIS JOURNAL, **71**, 1514 (1949).

(3) The hydrogenation was carried out by Mr. W. Fulmor.

(4) J. V. Pierce, A. C. Page, Jr., E. L. R. Stokstad and T. H. Jukes, THIS JOURNAL, **71**, 2952 (1949).

(5) Kindly measured by Dr. R. C. Gore, Stamford Research Laboratories, American Cyanamid Company.

(6) J. V. Pierce, A. C. Page, Jr., E. L. R. Stokstad and T. H. Jukes, THIS JOURNAL, **72**, in press (1950).

(7) C. E. Hoffmann, E. L. R. Stokstad, B. L. Hutchings, A. C. Dornbush and T. H. Jukes, *J. Biol. Chem.*, **181**, 635 (1949).

(8) E. L. R. Stokstad, T. H. Jukes, J. V. Pierce, A. C. Page, Jr., and A. L. Franklin, *ibid.*, **180**, 647 (1947).

(9) D. Hendlin and H. B. Woodruff, paper presented at 116th meeting, American Chemical Society, Atlantic City, N. J., September, 1949.

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A NEW IRON CARBIDE IN HYDROCARBON SYNTHESIS CATALYSTS

Sir:

During the course of hydrocarbon synthesis investigations in this laboratory, X-ray diffraction patterns of certain catalyst samples have indicated the presence of a new iron carbide phase. Iron catalysts in the hydrocarbon synthesis process using CO and H_2 have been characterized by the presence of iron carbide either as Fe_2C (Hägg) or Fe_2C (hexagonal).[1] There has been great interest in these carbides because of the role they may play in the catalytic process.[2]

The catalyst used in these investigations was obtained from reduced, promoted mill scale which analyzes approximately 97.0% total iron, with minor amounts of Mn, Cu, Ni, Al, S and P, and 0.7% K_2CO_3 as promoter. The new iron carbide appeared along with Fe_3O_4 and Fe_2C (Hägg) during the course of a fluidized synthesis run at 27 atm. pressure and 360°, and eventually it constituted approximately 90% of the total catalyst charge.

The X-ray diffraction pattern of this carbide phase is shown in Fig. 1(b). In this pattern some faint lines are attributed to Fe_3O_4 and Fe_2C (Hägg).[3] For comparison, the diffraction pattern for Fe_2C (Hägg) is shown in Fig. 1(a), and that for α-Fe is shown in Fig. 1c. These diffraction patterns were obtained with a 0.6-mm. extruded sample in a 71.6-mm. powder camera using radia-

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(1) Hofer, Cohn and Peebles, THIS JOURNAL, **71**, 189 (1949).

(2) Storch, "Advances in Catalysis and Related Subjects," Vol. I, Academic Press, Inc., New York, N. Y., 1948, pp. 115–156.

(3) Jack, *Proc. Roy. Soc.* (*London*), **195A**, 56 (1948).