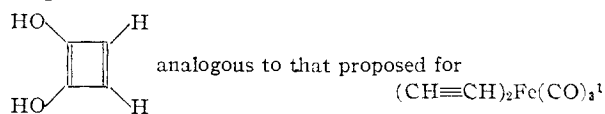


that the complex  $\text{Fe}_2\text{C}_{10}\text{H}_4\text{O}_8$ , formed<sup>10,11</sup> by the interaction of acetylene with an alkaline solution of iron carbonyl anion, contains a four-carbon chain. The experimental evidence presented<sup>9</sup> is equally compatible with the presence of a cyclobutadiene ring such as



The principle of carbon skeleton synthesis during complex formation offers a new route to the formation of organic compounds which may have practical value in synthetic organic chemistry. Indeed, we succeeded in synthesizing durohydroquinone without isolation of an intermediate complex by merely adding methanolic HCl to the mixture of dimethylacetylene and  $\text{Fe}(\text{CO})_5$  during irradiation.

A complex similar to I may conceivably be an intermediate in Reppe's synthesis of hydroquinone from iron pentacarbonyl and acetylene at elevated pressure and temperature.<sup>10</sup>

(10) W. Reppe and H. Vetter, *Ann. Chem. Justus Liebig*, **582**, 133 (1953).

(11) H. W. Sternberg, R. A. Friedel, R. Markby and I. Wender, *THIS JOURNAL*, **78**, 3621 (1956).

CENTRAL EXPERIMENT STATION      HEINZ W. STERNBERG  
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RECEIVED JANUARY 24, 1958

#### A CRYSTALLINE PENTAPOLYPHOSPHATE

Sir:

In the sodium<sup>1</sup> and potassium<sup>2</sup> phosphate systems, the largest chain oligophosphate which appears in the phase diagram is the tripolyphosphate. Because of this, there was considerable discussion in the scientific literature of the 40's that higher members of the homologous series of chain phosphates, such as the tetra- and pentapolyphosphates, do not exist. This notion has, of course, been disproved by modern tools such as ion exchange and paper chromatography.<sup>3</sup> Nevertheless, it is of considerable interest to point out here that pentapolyphosphate appears as a crystalline entity in a phase diagram.

In 1955, it was shown<sup>4</sup> that a crystalline salt of the composition  $3\text{PbO}\cdot 2\text{P}_2\text{O}_5$  appeared in the lead phosphate phase diagram. Paper chromatographic studies<sup>5</sup> in this Laboratory demonstrated that this compound and a similar barium compound were truly tetrapolyphosphates. We have now found that the crystalline calcium phosphate called trömelite which appears in the calcium phosphate phase diagram<sup>6</sup> is a pentapolyphosphate—the anion of which consists of a chain of five phosphorus

(1) E. P. Partridge, V. Hicks and G. V. Smith, *THIS JOURNAL*, **63**, 454 (1941); G. W. Morey and E. Ingerson, *Am. J. Sci.*, **242**, 1 (1944).

(2) G. W. Morey, *THIS JOURNAL*, **76**, 4724 (1954); G. W. Morey, F. R. Boyd, J. R. England and W. C. Chen, *ibid.*, **77**, 5003 (1955).

(3) E.g., E. Karl-Kroupa, *Anal. Chem.*, **28**, 1091 (1956).

(4) R. K. Osterheld and R. P. Langguth, *J. Phys. Chem.*, **59**, 76 (1955).

(5) R. P. Langguth, R. K. Osterheld and E. Karl-Kroupa, *ibid.*, **60**, 1335 (1956).

(6) W. L. Hill, G. F. Faust and D. S. Reynolds, *Am. J. Sci.*, **242**, 457 (1944).

atoms alternating with oxygen atoms. This has been demonstrated by paper chromatography<sup>3</sup> of solutions made by dissolving the trömelite in various solutions containing ethylenediaminetetraacetate. These results will be reported in detail, along with data on the molecular constitution of the other crystalline calcium phosphates, in a forthcoming paper to be submitted to *This Journal*.

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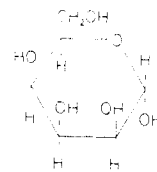
SHIGERU OHASHI

RECEIVED JANUARY 8, 1958

#### ISOLATION OF D-TALOSE FROM A NATURAL SOURCE

Sir:

Although D-talose (I) has been well-known for many years, it has not been isolated previously from natural sources. This communication reports the isolation of D-talose from the antibiotic, hygro-



mycin B.<sup>1</sup> The antibiotic was hydrolyzed with 0.5 N sulfuric acid, and the sulfate was removed as barium sulfate. The resulting solution was passed over IR-120, and the effluent was concentrated to dryness under reduced pressure. The residue was triturated twice with methanol. The methanolic solution thus obtained yielded crystalline D-talose. The crude talose was purified by crystallization of the impurities from water, concentration of the aqueous, trituration of the residue with methanol and again crystallizing from the methanolic extract. The crystals thus formed had the following properties: m.p. 128–132°;  $[\alpha]^{26}_D +16.9^\circ$  at equilibrium (c 1, H<sub>2</sub>O). *Anal.* Calcd. for C<sub>6</sub>H<sub>12</sub>O<sub>6</sub>: C, 39.99; H, 6.72; mol. wt., 180. Found: 39.96; H, 6.92; mol. wt. (crystallographic), 178. Melting points reported for α-D-talose are 133–134°,<sup>2</sup> 130–135°,<sup>3</sup> and 127–129°.<sup>4</sup> Reported rotations are, after mutarotation, +20.8°,<sup>2</sup> +19.7°,<sup>3</sup> and +20.6°.<sup>4</sup> Comparison of the product isolated from hygromycin B with synthetic α-D-talose<sup>5</sup> by means of X-ray diffraction patterns and paper chromatography indicated that the two were identical. The methylphenylhydrazine of the natural D-talose was prepared according to the procedure of Levene and Tipson.<sup>3</sup> This derivative melted at 153–154° (lit.<sup>3</sup> 154°) and upon admixture with an authentic sample showed no depression in melting point. The X-ray diffraction pattern of the derivative from natural material was identical with that from synthetic material.

More D-talose was obtained by concentration of the filtrate after removal of the first crop of crystalline material and trituration of the residue with methanol. This material was identified by melting

(1) R. L. Mann and W. W. Bromer, *THIS JOURNAL*, **80**, May (1958).

(2) W. W. Pigman and H. S. Isbell, *J. Research Natl. Bur. Standards*, **19**, 189 (1937).

(3) P. A. Levene and R. S. Tipson, *J. Biol. Chem.*, **93**, 631 (1931).

(4) W. Bosshard, *Helv. Chim. Acta*, **18**, 482 (1935).

(5) Supplied by General Biochemicals, Inc.