colorless platelets [11.1 g., m.p. 105.5–107.5°, λ_{max} 281 mμ (ϵ = 3,160) and 287 mμ (ϵ = 3,140), 5.82, 6.18, 6.37, and 7.37 μ (chloroform): $[\alpha]^{25}D$ +122°; calcd. for $C_{22}H_{30}O_4$: C, 73.71; H, 8.44; OCH_3, 17.32. Found: C, 73.93; H, 8.43; OCH_3, 17.05]. Compound II formed a bright yellow 2,4-dinitrophenylhydrazone [m.p. 203.5–207.5°, λ_{max} 3.04, 5.83, 6.23, 6.33, and 7.61 μ; calcd. for $C_{28}H_{34}N_4O_7$: C, 62.44; H, 6.36. Found: C, 62.64; H, 6.46].

To confirm the structure assigned to II, methyl O-methyl-7-acetonylpodocarpate was synthesized in the following manner: methyl O-methyl-7-acetylpodocarpate[3] (IV) was converted by a Willgerodt reaction to the corresponding thiomorpholide [V, m.p. 177–180.5°, λ_{max} 281 mμ (ϵ = 17,600), 5.81, 6.20, 6.37 and 6.71 μ (chloroform); $[\alpha]^{25}D$ +101° (ethanol); calcd. for $C_{25}H_{35}NO_4S$: S, 7.20; N, 3.14. Found: S, 7.20; N, 3.17]. Hydrolysis of V with sulfuric acid in acetic acid gave the corresponding monobasic acid [VI, m.p. 144.5–147°, presoftened, λ max 281 mμ (ϵ = 2,690) and 287 mμ (ϵ = 2,630), 5.82, 6.18, 6.35, and 6.66 μ (KBr); calcd. for $C_{21}H_{28}O_5$: C, 69.97; H, 7.83; OCH_3, 17.22; COOH, 12.49. Found: C, 70.23; H, 8.03; OCH_3, 16.87; COOH, 12.33]. Addition of an ether solution of VI to an excess of methylmagnesium bromide gave II which was identical [melting point, no depression on mixing: ultraviolet and infrared spectra] with the compound derived from the Willgerodt reaction on methyl O-methyl-7-propionylpodocarpate.

When heated with sulfur and morpholine, methyl O-methyl-7-acetonylpodocarpate (II) was converted to III (41% yield).

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(3) W. P. Campbell and D. Todd, THIS JOURNAL, **62**, 1287 (1940).

DIVISION OF CHEMICAL RESEARCH
G. D. SEARLE AND COMPANY ROY H. BIBLE, JR.
CHICAGO 80, ILLINOIS

RECEIVED JUNE 3, 1957

THE PREPARATION OF BERYLLIUM AMALGAM[1]

Sir:

The preparation of a beryllium amalgam by electrolysis of concentrated solutions of $BeCl_2$ into a mercury cathode has been claimed by Prytz[2] but this method is not confirmed elsewhere. A new and novel method, involving electrolysis of beryllium from a NaCl–$BeCl_2$ fused salt mixture into a mercury cathode has been developed in this laboratory and applied to the preparation of beryllium metal.[3]

Procedure.—Equimolar quantities of NaCl and $BeCl_2$ are melted together (224°)[4] to form an electrolyte. Beryllium chloride can be prepared by hydrochlorination of beryllium metal at 500°.

The electrolysis cell consists of a 4-liter resin reaction flask with a cylindrical inner sleeve to facilitate removal of the electrolyte at the conclusion of the electrolysis. Heat is supplied by an electric heating mantle.

Mercury (5000 g.) is placed in the cell and chunks of electrolyte are added quickly. The cell is closed and the electrolyte is melted down in an atmosphere of argon. A carbon anode is then inserted and the electrolysis commenced at about 5 volts and 6 amperes at temperatures of 300–350°.

Amalgam tends to climb through the salt to the anode. Therefore it is desirable to stir the cathode during electrolysis to avoid premature termination of the experiment. At the conclusion of the electrolysis (50–100 amp.-hr.) the cell is opened and the inner sleeve removed with the electrolyte within it. The amalgam, which varies in viscosity from a free flowing liquid to a semi-solid paste, depending on beryllium concentration, is found floating on the mercury at the bottom of the cell. The current efficiency of the electrolysis is approximately 65%.

The formation of amalgam evidently is sensitive to small traces of oxygen. Rigorous purification of the argon cover is required and it is essential that the cell be leak tight if amalgam is to be formed.

It is evident from this description that it also might be possible to prepare a beryllium amalgam by a displacement reaction with sodium amalgam and the fused-salt mixture used in electrolysis.

Properties of the Amalgam.—The amalgam is unstable in air, decomposing spontaneously with the formation of a black powder which is approximately 75% Be by weight. Dilute amalgams can be concentrated to about 2% beryllium by pinholing. The 2% amalgam is a thick pasty material from which additional mercury can be removed by such methods as pressing in a die or heating *in vacuo*.

Surprisingly, the room temperature X-ray diffraction pattern of the amalgam is the same as solid (−78°) mercury. The exact nature of the amalgam is not known. By analogy with the Mg–Hg system the compound $BeHg_2$ (2.2% Be) is suspected. It is hoped this information will assist in further investigation of the Be–Hg system.

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(1) Presented at Miami meeting of American Chemical Society April 7–12, 1957. Research supported by the Atomic Energy Commission.

(2) M. Prytz, *Z. anorg. allgem. Chem.*, **193**, 113 (1930).

(3) M. C. Kells, R. B. Holden, C. I. Whitman, AEC report SEP-207 (1956).

(4) J. M. Schmidt, *Bull. soc. chim.*, **39**, 1686–1703 (1926).

(5) Stanford Research Institute, Menlo Park, California.

SYLVANIA-CORNING M. C. KELLS[5]
NUCLEAR CORPORATION R. B. HOLDEN
BAYSIDE, NEW YORK C. I. WHITMAN

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TOTAL SYNTHESIS OF PENTACYCLOSQUALENE[1]

Sir:

Despite the probable genesis of the pentacyclic hydropicene triterpenes[2] from squalene by cation-olefin cyclization with subsequent rearrangement of carbon and hydrogen,[3,4,5] the pentacyclic substance I which would be formed from squalene by

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(1) This investigation was supported by a fellowship (AF-6570) from the National Institute of Arthritis and Metabolic Diseases, Public Health Service.

(2) α-Amyrin, β-amyrin, taraxerol, taraxasterol, friedelin, alnusenol and their oxygenated derivatives.

(3) A. Eschenmoser, L. Ruzicka, O. Jeger and D. Arigoni, *Helv. Chim. Acta*, **38**, 1890 (1955).

(4) E. J. Corey and J. J. Ursprung, THIS JOURNAL, **78**, 183 (1956).

(5) T. T. Tchen and K. Bloch, *ibid.*, **78**, 1516 (1956).