

run. Improvement might have been effected by the use of coated ribbons to increase the area pyrometered.

At temperatures above 2400°K., relatively large losses of tungsten were noted. These losses were more than could be accounted for by evaporation, and were assumed to be the result of a reaction between thorium oxide and tungsten in which volatile tungsten oxides were formed.

Acknowledgment.—Mr. F. H. Morgan's help in obtaining the calibration curve with the W-Mo thermocouple and Mr. H. Blecher's technical assistance are gratefully acknowledged.

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Ethyl β -Morpholinocrotonate

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Monoalkylation of acetoacetic ester is seldom clean and sometimes difficult. Alkylation of β -aminocrotonic ester¹ stops cleanly after a single alkyl group. A most satisfactory member of this series is the one derived from morpholine, using directions for the preparation of β -anilinoacetonate.²

Ethyl β -Morpholinocrotonate.—A mixture of 511 g. (3.93 moles) of ethyl acetoacetate, 400 cc. (4.60 moles) of morpholine, 10 cc. of 85% formic acid, and 2.5 l. of benzene was refluxed, and the water separated.² In 8 hours 75 cc. had been collected. The solution was then distilled, and

(1) (a) R. Robinson, *J. Chem. Soc.*, **109**, 1043 (1916); (b) W. M. Lauer and G. W. Jones, *This Journal*, **59**, 232 (1937).

(2) C. S. Hamilton, ed., *Org. Syntheses*, **29**, 42 (1949).

yielded 592 g., 76%, of the product, b.p. 120–124° (0.5 mm.), d_{25}^{25} 1.099, n_D^{25} 1.5162. A sample was redistilled (b.p. 116° (0.35 mm.)) and analyzed.

Anal. Calcd. for $C_{10}H_{17}NO_3$ (199.24): C, 60.28; H, 8.60. Found: C, 60.20; H, 8.65.

Treatment of the aminoester with either propargyl bromide or propargyl *p*-toluenesulfonate, followed by treatment with hot water,¹ yielded **3-carbethoxy-5-hexynone-2**, b.p. 69–73° (1 mm.), in excellent yield. A single redistillation gave a sample of analytical purity (b.p. 71° (1 mm.)).

Anal. Calcd. for $C_9H_{12}O_3$ (168.19): C, 64.27; H, 7.19. Found: C, 63.60; H, 7.20.

3-Carbethoxy-5-hexynone-2, 2,4-dinitrophenylhydrazone, m.p. 97–98°.³

Anal. Calcd. for $C_{15}H_{16}N_4O_8$ (348.31): C, 51.72; H, 4.63; N, 16.09. Found: C, 51.74; H, 4.69; N, 15.94.

Acid hydrolysis yields **5-hexynone-2**,⁴ b.p. 62–64° (12 mm.). The **2,4-dinitrophenylhydrazone** of **5-hexynone-2** melts at 138–139°.

Anal. Calcd. for $C_{12}H_{12}N_4O_4$ (276.25): N, 20.28. Found: N, 20.34.

Treatment of ethyl β -morpholinocrotonate with 2,3-dichloropropene, followed by refluxing with water, yielded directly **5-chloro-5-hexenone-2**, b.p. 74–77° (16 mm.) in 86% yield. The **2,4-dinitrophenylhydrazone** has m.p. 84–85°.

Anal. Calcd. for $C_{12}H_{13}ClN_4O_4$ (312.71): C, 46.09; H, 4.19; N, 17.92. Found: C, 46.12; H, 4.31; N, 18.34.

Treatment of the chloroketone with sodium amide in liquid ammonia, in an attempt to effect the condensation of two molecules to a derivative of 1,6-cyclodecadiyne, yielded no detectable tertiary alcohol.

Acknowledgment.—We wish to thank Dr. M. C. Whiting for informative discussions during the course of this work.

(3) All melting points taken with Anschütz thermometers in a Hershberg apparatus.

(4) T. E. Gardner and W. H. Perkin, *J. Chem. Soc.*, **91**, 851 (1907).

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COMMUNICATIONS TO THE EDITOR

THREE NEW NEUTRON DEFICIENT ISOTOPES OF YTTRIUM

Sir:

Three new yttrium isotopes have been discovered by proton bombardment of spectroscopically pure yttrium oxide¹ in the Rochester 130-inch cyclotron. Targets of yttrium oxide were placed in an aluminum foil envelope which was mounted on the end of a probe and placed inside the tank of the cyclotron. The target was bombarded with both 240 and 130 Mev. protons for exposure times of one and two hours.

The yttrium oxide was dissolved in hot nitric acid and inert carriers of zirconium, strontium, rubidium and bromine were added to the solution. The yttrium fraction was purified from all the spallation products and the sample was followed under a Geiger-Müller tube for gross decay. The activities found had half lives of: 2–4 hours, 14 hours, 38 hours, 80 hours, 25.5 days and finally 65 days.

(1) The yttrium oxide was supplied through the courtesy of Ames Laboratory, Iowa State College.

In one experiment 105-day Y^{88} was also observed. The half-lives of the known² yttrium isotopes in this region are: Y^{88} 2 hours, 105 days; Y^{87} 14 hours, 80 hours; Y^{86} 14.6 hours; and Y^{84} 3.7 hours. The 38-hour, 25.5-day, and 65-day activities found in the yttrium gross decay curve can be identified with Sr^{83} , Sr^{82} , and Sr^{85} , respectively. This means that these strontium isotopes found in the purified yttrium sample are the daughters of their respective yttrium parents which are previously unreported yttrium isotopes. Thus, Y^{82} decayed to 25.5-day Sr^{82} , Y^{83} decayed to 38-hour Sr^{83} , and Y^{85} decayed to 65-day Sr^{85} .

Since the yttrium gross decay curve was so complex, it was impossible to resolve the half lives of these new isotopes directly. Therefore, a series of isolation experiments were performed in which the strontium daughter isotopes were removed from the yttrium at periodic intervals. These strontium

(2) Nuclear Data by K. Way, et al., Circular of the National Bureau of Standards 499; General Electric Research Laboratory Chart of the Nuclides.