

Two Pyrex tubes were evacuated and filled simultaneously from a reservoir containing a deuterium-hydrogen mixture. The first tube was fitted with a magnetic break at the lower end and, after filling, was sealed off at the upper end. The second tube was equipped with a stopcock and was coated on the inside with a layer of "Lubriscal." At the end of twenty-two days mass spectrographic analysis showed the gas in the first tube to contain 2.07% deuterium and that in the second 2.09%. It is evident, therefore, that an apparatus containing stopcocks may be used in the study of deuterium-hydrogen mixtures without fear of interchange between deuterium and hydrogen from the stopcock grease.

FRICK CHEMICAL LABORATORY      AUSTIN J. GOULD  
PALMER PHYSICAL LABORATORY      WALKER BLEAKNEY  
PRINCETON UNIVERSITY  
PRINCETON, NEW JERSEY

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THE SYNTHESIS OF POLYNUCLEAR  
HYDROCARBONS BY THE CYCLODEHYDRATION  
OF AROMATIC ALCOHOLS

Sir:

Since our preliminary publications in this field,<sup>1</sup> a number of articles have been published abroad<sup>2</sup> which show that these communications of ours have escaped notice there and which make it seem desirable to us to report the following additional results which have been obtained in our laboratories. This is done in the hope that it will save our fellow investigators the time and money which might otherwise be spent in the duplication of experiments.

Our studies in the tetralin field, for example, already have shown that 1-methyltetralin can be obtained by the action of sulfuric acid upon any one of the following:  $\text{PhCH}_2\text{CH}_2\text{CH}_2\text{CH}(\text{OH})\text{Me}$ ,  $\text{PhCH}_2\text{CH}_2\text{CH}_2\text{CH}=\text{CH}_2$ ,  $\text{PhCH}_2\text{CH}_2\text{CH}(\text{OH})\text{CH}_2\text{Me}$ , or  $\text{PhCH}_2\text{CH}=\text{CHCH}_2\text{Me}$ .

1-Ethyltetralin has been prepared similarly from both  $\text{Ph}(\text{CH}_2)_4\text{CH}(\text{OH})\text{Me}$  and  $\text{Ph}(\text{CH}_2)_4\text{CH}=\text{CH}_2$ .

In two of the above compounds, a shift in the location of the double bond is indicated in connection with the cyclization.

In the case of  $\text{Ph}(\text{CH}_2)_2\text{CMe}(\text{OH})\text{CH}_2\text{Me}$ ,

(1) (a) Bogert, *Science*, [N. S.] **76**, 475 (1932); (b) **77**, 197 (1933); (c) **77**, 289 (1933).

(2) (a) Schlenk and Bergmann, *Chemistry & Industry*, **52**, 207 (1933); (b) Cook, Hewett, Haslewood, *et al.*, *ibid.*, **52**, 949 (1933); (c) Ruzicka, Ehmman, Goldberg and Hösl, *Helv. Chim. Acta*, **16**, 833 (1933); (d) Kou, *J. Chem. Soc.*, 1081 (1933); (e) Cook and Hewett, *ibid.*, 1098 (1933); (f) Fulton and Robinson, *ibid.*, 1463 (1933).

both 1,2-dimethyltetralin and 1-methyl-1-ethylindane appear to be formed.

As stated in our initial announcements,<sup>1</sup> these studies are being pursued in various directions, especially with the object in view of preparing synthetically compounds of biological interest.

LABORATORIES OF ORGANIC      MARSTON TAYLOR BOGERT  
CHEMISTRY      DAVID DAVIDSON  
COLUMBIA UNIVERSITY      RICHARD O. ROBLIN, JR.  
NEW YORK, N. Y.

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FREEZING POINTS OF MIXTURES OF THE WATERS,  
 $\text{H}^1_2\text{O}$  AND  $\text{H}^2_2\text{O}$

Sir:

We have determined the freezing points and specific gravities at 25° on 15-cc. portions of mixtures of  $\text{H}^2_2\text{O}$  and  $\text{H}^1_2\text{O}$  using a Beckmann thermometer calibrated by the Bureau of Standards, and standard technique. The samples were prepared by electrolysis of potash solutions using nickel electrodes, followed by repeated distillations from alkaline permanganate to remove carbon dioxide and other impurities which would lower the freezing point. The freezing points were independent of the supercooling (1 to 2.8°) and were constant to within 0.001° for periods of at least ten minutes. The zero point of the thermometer was determined repeatedly against the freezing point of ordinary distilled water using precisely the same technique. Slow freezing of the 39.9% sample over a period of three hours with continuous stirring, until one-third had separated as ice, produced no change in equilibrium temperature greater than the experimental error of 0.002°. The specific gravities of the melted ice and unfrozen liquid, using a 1.3-cc. pycnometer, checked to 0.02%.

These observations indicate that the solid

Approx. % heavy component 100% =	Sp. gr. 25/25° °C.	F. p., °C.	Sp. gr. 25/25° (sp. g. -1)	F. P. (sp. g. -1)	Observer
1.23	0.053	1.001376	38.5	Washburn, Smith and Frandsen, <i>B. S. J.</i> <i>Res.</i> , <b>11</b> , 453 (1933)	
14.7	.632	1.01644	38.5	This investigation	
19.1	.824	1.02135	38.6	This investigation	
39.9	1.079	1.04456	37.7	This investigation	
39.5	1.070	1.04411	37.9	This investigation; re- distilled <i>in vacuo</i>	
94.6?	3.8	1.1056 <sup>a</sup>	36.	Lewis and Macdonald	

<sup>a</sup> The terms specific gravity and density are used interchangeably for this value [Lewis and Macdonald, *THIS JOURNAL*, **55**, 3058 (1933)].