

reaction with sodamide was complete, 7.0 g. of *o*-methylbenzyl chloride⁸ was added dropwise. This reaction mixture was boiled under reflux for two hours, cooled, and washed several times with water. After removal of the toluene *in vacuo*, the residual oil was distilled yielding 7.1 g. (60%) of a colorless oil; b. p. 116–117° at 1.2 mm.; n_D^{25} 1.4984.

Anal. Calcd. for $C_{14}H_{20}O_3$: C, 71.15; H, 8.65. Found: C, 71.25; H, 8.74.

Hydrolysis of a sample of 2,2-dimethyl-4-(*o*-methylbenzyloxymethyl)-1,3-dioxolane (2.0 g.) was accomplished by boiling it for one hour with a solution containing 14 ml. of alcohol, 5 ml. of water and 0.2 ml. of concen-

dosages muscular paralysis was incomplete.

DEPARTMENT OF CHEMISTRY
UNIVERSITY OF ROCHESTER
ROCHESTER, NEW YORK

G. L. SAUVAGE
V. BOBKELHEIDE

RECEIVED FEBRUARY 21, 1949

Five New Tertiary Carbinols and Four New Aliphatic Hydrocarbons

In the course of work on the mechanism of anomalous Grignard reactions,¹ five tertiary carbinols were obtained, which are believed to be new. Four of these were converted to the corresponding hydrocarbons.

| Compound | °C. | B. p. ^a Mm. | n_D^{20} | Yield, % | Formula | Analyses, % | | | |
|--|-----------|---------------------------|------------|-------------|-----------------|-------------|-------|----------|-------|
| | | | | | | Carbon | | Hydrogen | |
| | | | | | | Calcd. | Found | Calcd. | Found |
| 2,4,5-Trimethylheptan-4-ol ^b | 54–55 | 3 | 1.4382 | 15 | $C_{10}H_{22}O$ | 75.9 | 76.3 | 13.9 | 13.9 |
| 3,4-Dimethyloctan-4-ol ^c | 64–65 | 3 | 1.4418 | 20 | $C_{10}H_{22}O$ | 75.9 | 75.8 | 13.9 | 14.0 |
| 3,4-Dimethylnonan-4-ol ^d | 68–69 | 2 | 1.4430 | 15 | $C_{11}H_{24}O$ | 76.7 | 76.2 | 13.9 | 14.1 |
| 2,6-Dimethyl-4-isopropylheptan-4-ol ^e | 48–49 | 1 | 1.4448 | 20 | $C_{12}H_{26}O$ | 77.4 | 77.6 | 14.0 | 13.9 |
| 2,6-Dimethyl-4-isobutylheptan-4-ol ^f | 72–73 | 2 | 1.4390 | 15 | $C_{12}H_{26}O$ | 78.0 | 77.6 | 14.0 | 13.9 |
| 2,4,5-Trimethylheptane ^g | 49.5 | 18 | 1.4160 | | $C_{10}H_{22}$ | 84.5 | 84.6 | 15.5 | 15.1 |
| 3,4-Dimethyloctane ^g | 58.0–58.4 | 20 | 1.4182 | | $C_{10}H_{22}$ | 84.5 | 84.6 | 15.5 | 15.0 |
| 3,4-Dimethylnonane ^g | 75.0–75.2 | 18 | 1.4223 | | $C_{11}H_{24}$ | 84.8 | 84.9 | 15.2 | 14.8 |
| 2,6-Dimethyl-4-isobutylheptane | 81.9–82.5 | 20 | 1.4238 | | $C_{12}H_{26}$ | 84.6 | 84.3 | 15.4 | 15.0 |

^a Uncorrected. By the interaction of *s*-BuMgBr and MeCOBu-*i*. ^c By the interaction of *s*-BuMgBr and MeCOBu-*n*. ^d By the interaction of *s*-BuMgBr and MeCOAm-*n*. ^e By the interaction of *i*-PrMgBr and *i*-BuCOBu-*i*. ^f By the interaction of *i*-BuMgBr and *i*-BuCOBu-*i*. Skraup and Freundlich, *Ber.*, **55**, 1080 (1922), report that they made this by the action of *i*-BuMgBr on isovaleric ester, but say that the pure carbinol could not be obtained, since it decomposed when distilling in a high vacuum. No physical constants were given. ^g Calculations of the physical constants of this are given by Francis, *Ind. Eng. Chem.*, **35**, 442–448 (1943).

trated sulfuric acid. The mixture was neutralized with sodium carbonate, and the organic layer was separated and distilled. There was obtained 0.75 g. (45%) of a colorless oil; b. p. 149–151° at 0.4 mm.; n_D^{25} 1.5330.

Anal. Calcd. for $C_{11}H_{16}O_3$: C, 67.32; H, 8.22. Found: C, 67.52; H, 8.11.

Attempts to prepare 3-(*o*-methylbenzyloxy)-1,2-propanediol by the reaction of the sodium salt of *o*-methylbenzyl alcohol with epichlorohydrin or glycerol α -monochlorohydrin were unsuccessful.

Since 3-(*o*-methylbenzyloxy)-1,2-propanediol is an analog of myanesin (3-(*o*-toloxy)-1,2-propanediol), it was tested for physiological action by intraperitoneal injection into mice.⁴ It was found that even at lethal

(3) Newman, *THIS JOURNAL*, **62**, 2295 (1940).

(4) Physiological tests were made by F. M. Berger, M.D., Department of Pediatrics, University of Rochester School of Medicine, Rochester, New York.

In the conversion of the carbinols to the hydrocarbons, the former were dehydrated to the olefins with naphthalene-2-sulfonic acid, and the olefins were fractionated, the column used being packed to a length of 12 inches with Fenske helices, and fitted with a total-reflux-variable-take-off still head. They were then reduced in glacial acetic acid with Adams catalyst and hydrogen at 50 p. s. i. pressure until no unsaturation could be detected with a solution of bromine in carbon tetrachloride. The hydrocarbons were fractionated through the column described above.

(1) Shine and Turner, "The Anomalous Reactions of Grignard Reagents (I)," submitted for publication in *THIS JOURNAL*.

DEPARTMENT OF CHEMISTRY
BEDFORD COLLEGE
UNIVERSITY OF LONDON
LONDON, ENGLAND

H. J. SHINE

E. E. TURNER

RECEIVED NOVEMBER 5, 1948

COMMUNICATIONS TO THE EDITOR

EXCHANGE REACTIONS BETWEEN CERIUM(III) AND CERIUM(IV) AND BETWEEN IRON(II) AND IRON(III)

Sir:

We have studied the exchange reaction between cerium(III) and cerium(IV) in perchloric acid and in sulfuric acid solutions. Using electrical migration methods to partially separate the reactants we have found no evidence that the exchange is measurably slow. The 30-day Ce^{141} used as cerium(III) tracer was obtained from the Clinton

Laboratories on allocation from the U. S. Atomic Energy Commission. It was purified by precipitation of cerium(III) fluoride and ammonium hexanitratocerate(IV). The Ce^{141} content of solutions was determined by gamma-counting. Cerium was determined by titration with iron(II) sulfate. In sulfuric acid solutions cerium(IV) migrated toward the anode, cerium(III) toward the cathode. In perchloric acid solutions both ions migrated toward the cathode, cerium(III) being the more mobile. Samples enriched in each