absolute ethanol as the solvent. The compounds examined were purified by fractional distillation immediately before determination of the spectra. In the case of mesityl methyl ether, the original sample was redistilled and the spectrum redetermined; identical results were obtained with both samples. The wave lengths and logarithms of the molar extinction coefficients at the absorption maxima and at prominent points of inflection are listed in Table I.

TABLE I

ULTRAVIOLET ABSORPTION DATA

Compound	Source[a]	Maxima[b] λ, (mμ)	Log ε
Mesityl methyl ether	1	315	1.50
		278	2.88
		274	2.83
		270	2.80
		215	3.90
p-Tolyl methyl ether	2	286	3.21
		279	3.27
		223	3.88
Mesitylene[c]	2	272	2.26
		268	2.26
		265	2.34
		(262)	2.24
		259	2.21
		(256)	2.17
		215	3.87

[a] Source references: (1) Biedermann and Ledoux, *Ber.*, 8, 57 (1875); (2) Eastman Kodak Co., redistilled. [b] The wave lengths in parentheses refer to inflection points. [c] *Cf.* Conrad-Billroth, *Z. physik. Chem.*, B29, 170 (1935); Pickett, Walter and France, THIS JOURNAL, 58, 2296 (1936).

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UNIVERSITY OF PENNSYLVANIA, PHILADELPHIA, PA.
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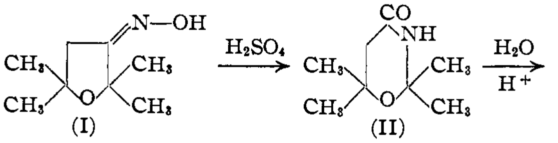
Beckmann Rearrangement of 2,2,5,5-Tetramethyltetrahydro-3-furanone Oxime

BY G. F. HENNION AND JOSEPH L. O'BRIEN

Previous work made available a liberal sample of 2,2,5,5-tetramethyltetrahydro-3-furanone.[1] It was thought that the Beckmann rearrangement of its oxime[2] (I) would prove of interest because the anticipated lactam (II) should undergo ring cleavage in the process, leading ultimately to acetone, ammonia and β,β-dimethylacrylic acid. These are indeed the products of the reaction with moderately strong sulfuric acid.



$$CH_3COCH_3 + (CH_3)_2C{=}CH{-}COOH + NH_3$$

This confirms the instability of (II) and indicates that the oxime (I) has the *trans* structure, perhaps on account of the steric effect of the neighboring *gem*-dimethyl group.

Experimental

A solution of 5 g. of the oxime (m. p. 122–123°)[3] in 10 ml. of approximately 77% sulfuric acid in a Claisen flask was heated while nitrogen was bubbled through it. Vigorous reaction occurred and about 3 ml. of distillate was recovered in the receiver cooled in a salt–ice mixture. After drying with calcium chloride (0.95 g. recovered), the boiling point (micro) was 62° and the n^{25}D 1.381. Qualitative tests indicated that it was chiefly acetone. In a duplicate run the distillate was treated with 2,4-dinitrophenylhydrazine; the derivative was obtained in 64% yield based on the oxime used and melted at 123–124°.

Ammonia liberated from one of the above sulfuric acid solutions, collected and titrated in the usual way, showed a 55% yield.

The alkaline residue from the ammonia distillation was filtered from a small amount of tar and extracted with ether (discarded). The aqueous solution was then acidified with sulfuric acid and extracted twice with 20-ml. portions of ether. The ether extract was dried with calcium chloride and the ether evaporated, leaving 0.75 g. of residue which soon crystallized. This material was purified by solution with potassium carbonate, reprecipitation with hydrochloric acid and crystallization from petroleum ether. Small needles melting at 66–67° were thus obtained. The bromine addition product melted at 105–106°. Ustinoff[4] gives 69.5–70° and 105–106° as the melting points of β,β-dimethylacrylic acid and its dibromide, respectively.

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[1] Froning and Hennion, THIS JOURNAL, **62**, 653 (1940).

[2] Dupont, *Ann. chim.*, [8] **30**, 536 (1913).

[3] Dupont[2] gives 128°.

[4] Ustinoff, *J. prakt. Chem.*, [2] **34**, 478 (1886).

DEPARTMENT OF CHEMISTRY
UNIVERSITY OF NOTRE DAME
NOTRE DAME, INDIANA
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Halogen–Metal Interconversions with Halogenated Anilines and Anilides

BY HENRY GILMAN AND C. G. STUCKWISCH[1]

In connection with studies concerned with the introduction of variously substituted aryl groups into organolead compounds, it was desirable to prepare organolithium compounds containing functional groups such as amino or alkylamino. A method of choice for the synthesis of such types is the halogen–metal interconversion reaction.[2]

$$RX + R'Li \longrightarrow RLi + R'X.$$

The halogen compounds studied in the present investigation are *o*-bromoaniline, *p*-bromoaniline, *p*-bromo-N-methylaniline and N-(*p*-iodophenyl)-phthalimide. In each instance a diethyl ether solution of *n*-butyllithium was added to the halogen compound dissolved in diethyl ether. The reaction mixture was carbonated and the resulting acid isolated as such or as the benzenesulfonamide. Details are given in the experimental section.

The previously reported reaction of *n*-butyl-

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[2] (a) Gilman and Stuckwisch, THIS JOURNAL, **64**, 1007 (1942); (b) Gilman and Arntzen, *ibid.*, **69**, 1537 (1947); (c) Gilman and Melstrom, *ibid.*, **70**, 4177 (1948); (d) for general references, see pp. 538–539 of Gilman, "Organic Chemistry," John Wiley and Sons, Inc., New York, N. Y., 1943.