

1940

Direct Syntheses of Amides From Long-Chained Aliphatic Amines

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Recommended Citation

Hunter, Byron A.; Harber, William I.; and Gilman, H. (1940) "Direct Syntheses of Amides From Long-Chained Aliphatic Amines," *Proceedings of the Iowa Academy of Science*, 47(1), 263-264.

Available at: <https://scholarworks.uni.edu/pias/vol47/iss1/52>

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These organolithium compounds of the nitrogen heterocycles are of a relatively high order of reactivity with other functional groups.

Some of the types reported on in the pyridine series are:

1. 3—pyridyllithium in 70 per cent yield from 3—bromopyridine and *n*—butyllithium,
2. 3—quinollythium in 52 per cent yield from 3—bromopyridine,
3. 5—bromo—3—pyridyllithium in 41 per cent yield from 3, 5—dibromopyridine and slightly more than two equivalents of *n*—butyllithium.

In the carbazole series, some of the organolithium compounds reported are:

1. N—ethyl—2, 8—dilithiocarbazole in 84-91 per cent yield from N—ethyl—2, 8—dibromocarbazole, and in 79-92 per cent yield from the corresponding di-iodo-compound.
2. N—ethyl—2—lithiocarbazole¹ from the corresponding iodo-carbazole in 68 per cent yield.

¹ Studies by Irving Banner.

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DIRECT SYNTHESSES OF AMIDES FROM LONG-CHAINED ALIPHATIC AMINES

BYRON A. HUNTER, WILLIAM I. HARBER AND H. GILMAN

An examination has been made of direct procedures for the synthesis of amides. Dodecylamine, tetradecylamine, hexadecylamine and octadecylamine have been treated with a variety of aliphatic and aromatic acids. Direct heating of the amine and acid, in open containers and at elevated temperatures, gave quite satisfactory yields of amides.

This direct procedure has been resolved into two stages. First, a mixture of amine and acid, dissolved or suspended in warm petroleum ether, gives promptly and in good yield the corresponding salt. These salts can be used for the characterization of amine and acid. Second, when the salts are heated they are converted to the amides.

As an example, *n*-octadecyl amine and acetic acid gives a 24 per cent yield of salt melting at 84.5-85°. Upon heating this salt at 225° for fifteen minutes there is a quantitative conversion to *n*-octadecylacetamide (m.p., 78-78.5°).

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PYRROLIDINES FROM PRIMARY AMINES

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HAROLD A. HOPPENS

Pyrrolidine has been prepared from *n*-butylamine by heating *N*-chloro-*N*-acyl derivatives of the amine with concentrated sulfuric acid.

N-chloro-*N*-butylacetamide underwent ring closure with the formation of pyrrolidine in 50 per cent yields when heated with 95 per cent sulfuric acid for one hour at 130-140°. With 99.5 per cent acid the percentage yield was much lower.

N-chloro-*N*-*n*-butyl-*p*-toluenesulfonamide when heated with 95 per cent sulfuric acid for thirty minutes at 140° formed pyrrolidine in 50 per cent yields.

In both cases considerable amounts of *n*-butylamine were also formed.

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BEHAVIOR OF SOME *p*-HYDROXYBENZALDEHYDE DERIVATIVES TOWARD OXIDIZING AGENTS

R. P. PERRY AND L. CHARLES RAIFORD

Benzaldehyde¹ and its alkyl, alkoxy and halogen substitution products are readily oxidized to the corresponding acids. Contact with air often brings about the change.² Bücking,³ and Fittig and

¹ Wöhler and Liebig, *Ann.*, 3,250 (1832).

² Bornemann, *Ber.*, 17, 1466 (1884).

³ Bücking, *Ber.*, 9,529 (1876).