Chemistry of Heterocyclic Compounds, Vol. 30, No. 6, 1994

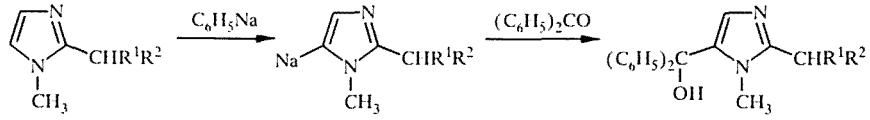
SODIUM DERIVATIVES OF 1-METHYL-2-ALKYLIMIDAZOLES

Yu. V. Koshchienko, G. P. Shapkina and B. A. Tertov

It is known that 1,2-dimethyl- and 1-methyl-2-butylimidazole are metallated at the alkyl group at position 2 by phenylsodium and 2-lithio-1-methylimidazole respectively [1, 2].

We have established that 1-methyl-2-ethyl- (Ia), 1-methyl-2-propyl- (Ib) and 1-methyl-2-isopropylimidazole (Ic) are converted exclusively to the 5-sodio-1-methyl-2-alkylimidazoles (IIa-c) by phenylsodium.

The organosodium compounds IIa-c were converted to carbinols IIIa-c to elucidate their structures and to determine their yields.



Ia–в IIa–в IIIa–в

I—III a $R^1 = H$, $R^2 = CH_3$; b $R^1 = H$, $R^2 = C_2H_5$; c $R^1 = R^2 = CH_3$

Since the 5-sodioimidazoles IIa-c are formed in good yield (48-72%) they may be of interest as key intermediates for the synthesis of various 5-substituted imidazoles.

For example, a solution of a 1-methyl-2-alkylimidazole Ia-c (10 mmole) in toluene (10 cm^3) was added at 30-35°C with stirring over 10 min to a solution of phenylsodium prepared from sodium (1.1 g, 48 mmole) and chlorobenzene (2.6 g, 23 mmole) in absolute toluene (25 cm^3 under argon. The mixture was stirred at this temperature for 5 h, then a solution of benzophenone (4.19 g, 23 mmol) in toluene (20 cm^3) was added and stirring continued for 1.5 h. Unreacted sodium was destroyed with ethanol (5 cm^3) and the diphenyl(1-methyl-2-alkylimidazolyl-5)carbinols (IIIa-c) were isolated by standard methods.

The isomeric 1-methyl-2-(1-alkyl-2,2-diphenyl-2-hydroxyethyl)imidazoles and 1-methyl-2-(1,1-dimethyl-2,2-diphenyl-2-hydroxyethyl)imidazoles were not observed among the reaction products.

Diphenyl(1-methyl-2-ethylimidazolyl-5)carbinol (IIIa). Yield 48%. mp 183-184°C (from ethyl acetate). ^1H NMR spectrum ($CDCl_3$): 1.10 (3H, t, CH_3), 2.38 (2H, q, CH_2), 3.11 (3H, s, NCH_3), 5.12 (1H, br. s, OH), 5.86 (1H, s, 4-H), 7.12 ppm (10H, s, $2C_6H_5$).

Diphenyl(1-methyl-2-propylimidazolyl-5)carbinol (IIIb). Yield 72%. mp 152-153°C (from ethyl acetate). ^1H NMR spectrum ($CDCl_3$): 0.85 (3H, t, CH_3), 1.59 (2H, q, CH_2), 2.42 (2H, t, CH_2), 2.18 (3H, s, NCH_3), 5.20 (1H, br. s, OH), 5.91 (1H, s, 4-H), 7.15 ppm (10H, s, $2C_6H_5$).

Diphenyl(1-methyl-2-isopropylimidazolyl-5)carbinol (IIIc). Yield 58%. mp 190-191°C (from ethyl acetate). ^1H NMR spectrum ($CDCl_3$): 1.12 (6H, d, $2CH_3$), 2.82 (H, m, CH), 3.19 (3H, s, NCH_3), 4.71 (1H, br. s, OH), 5.90 (1H, s, 4-H), 7.11 ppm (10H, s, $2C_6H_5$).

The elemental analysis results for compounds IIIa-c agreed with the calculated values.

Evidently other 1,2-dialkylimidazoles in which the substituent at carbon $C_{(2)}$ is not a methyl group will be alkylated by phenylsodium analogously to compounds Ia-c.

Physical and Organic Chemistry Research Institute, Rostov State University, Rostov-on-Don 344104. Translated from Khimiya Geterotsiklicheskikh Soedinenii, No. 6, pp. 847-848, June, 1994. Original article submitted May 25, 1994.

0009-3122/94/3006-0743$12.50

REFERENCES

1. B. Iddon and B. L. Lim, J. Chem. Soc. Perkin I., No. 2, 271 (1983).
2. L. Brandsma and H. D. Verkujsse, Preparative Polar Organometallic Chemistry, Springer, Berlin (1987), p. 128.