

LETTERS TO THE EDITOR

Amidation of Ethyl *N*-Benzoyl-1-adamantylacetimidate

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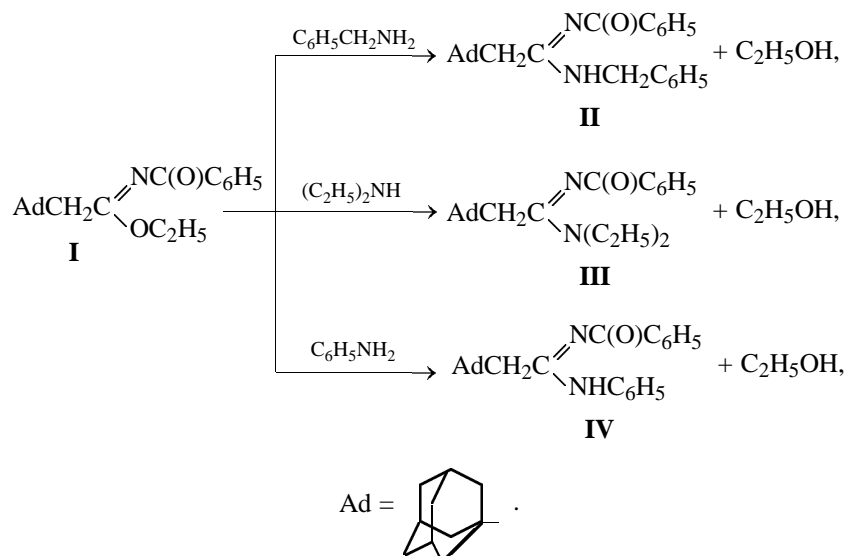
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We have for the first time prepared N^1, N^2 -di- and N^1, N^1, N^2 -trisubstituted amidines.

As object for study we chose ethyl *N*-benzoyl-1-

adamantylacetimidate (**I**). It was brought in reactions with primary alkylarylamines (benzylamine), secondary aliphatic amines (diethylamine), and primary aromatic amines (aniline) .



The syntheses were carried out in the media of the corresponding amines. At 50–60°C, the reactions were complete in one or, with aniline, two hours. The latter fact can be explained by the lower basicity of the aromatic amine.

The products are white crystalline substances with fairly high well-defined melting points. Due to the electron-donor effect of the amino groups, the C=N and C=O absorption bands in the IR spectra are shifted red by 10–20 cm⁻¹.

N^2 -Benzoyl- N^1 -benzyl-1-adamantylacetamidine (II). Compound **I**, 1.63 g, and 10 ml of benzylamine were placed in a moisture-proof four-necked reactor

equipped with a stirrer, a thermometer, and a reflux condenser. The reaction mixture was stirred for 1 h at 50–60°C. Excess amine was removed in a vacuum, and the residue was crystallized from ethanol to obtain 1.78 g (92%) of compound **II**, mp 128–129°C. IR spectrum, ν , cm⁻¹: 1650 (C=O), 1610 (C=N), 3460 (N–H), 1248 (C–N), 3080, 3040, 1595 (Ar), 1468, 1380, 1100, 985 (Ad). ¹H NMR spectrum, δ , ppm: 1.38 s (6H, Ad), 1.51 s (6H, Ad), 1.74 s (3H, Ad), 2.51 s (2H, CH₂), 3.44 t (1H, NH), 4.47 s (2H, CH₂), 7.56, m (10H, 2 C₆H₅). Found, %: C 80.56; H 8.12; N 7.30. C₂₆H₃₀N₂O. Calculated, %: C 80.79; H 7.82; N 7.25.

***N*²-Benzoyl-*N*¹,*N*¹-diethyl-1-adamantylacetamidine (III).** Compound **I**, 1.63 g, and 10 ml of diethylamine were placed in a moisture-proof four-necked reactor equipped with a stirrer, a thermometer, and a reflux condenser. The reaction mixture was stirred for 1 h under reflux. Excess amine was distilled off in a vacuum, and the residue was crystallized from ethanol to obtain 1.61 g (91%) of compound **III**, mp 153–154°C. IR spectrum, ν , cm^{-1} : 1640 (C=O), 1610 (C=N), 1245 (C–N), 3080, 3050, 1600, 1480 (Ar), 1460, 1373, 1100, 982 (Ad). ¹H NMR spectrum, δ , ppm: 1.20 t (6H, 2CH₃), 1.46 s (6H, Ad), 1.58 s (6H, Ad), 1.76 s (3H, Ad), 2.77 s (2H, CH₂), 3.49 d (4H, 2CH₂), 7.64 m (5H, C₆H₅). Found, %: C 78.49; H 9.42; N 8.12. C₂₃H₃₂N₂O. Calculated, %: C 78.37; H 9.15; N 7.95.

***N*²-Benzoyl-*N*¹-phenyl-1-adamantylacetamidine (IV).** Compound **I**, 1.63 g, and 10 ml of aniline were

placed in a moisture-proof four-necked reactor equipped with a stirrer, a thermometer, and a refluxed condenser. The reaction mixture was stirred for 2 h at 507360°C. Excess amine was removed in a vacuum, and the residue was crystallized from ethanol to obtain 1.79 g (96%) of compound **IV**, mp 126–127°C. IR spectrum, ν , cm^{-1} : 1645 (C=O), 1615 (C=N), 3470 (NH), 1265 (C–N), 3080, 3050, 1595, 1480 (Ar), 1460, 1100, 985 (Ad). ¹H NMR spectrum, δ , ppm: 1.45 s (6H, Ad), 1.58 s (6H, Ad), 1.80 s (3H, Ad), 2.09 s (1H, NH), 2.42 s (2H, CH₂), 7.44 m (10H, 2C₆H₅). Found, %: C 80.73; H 7.82; N 7.85. C₂₅H₂₈N₂O. Calculated, %: C 80.61; H 7.85; N 7.52.

The IR spectra were recorded on a Perkin-Elmer 882 spectrometer in Vaseline oil using NaCl or KBr prisms. The ¹H NMR spectra were measured on a Tesla BS-487 spectrometer (100 MHz) against HMDS in CCl₄, CDCl₃, and acetone-*d*₆.