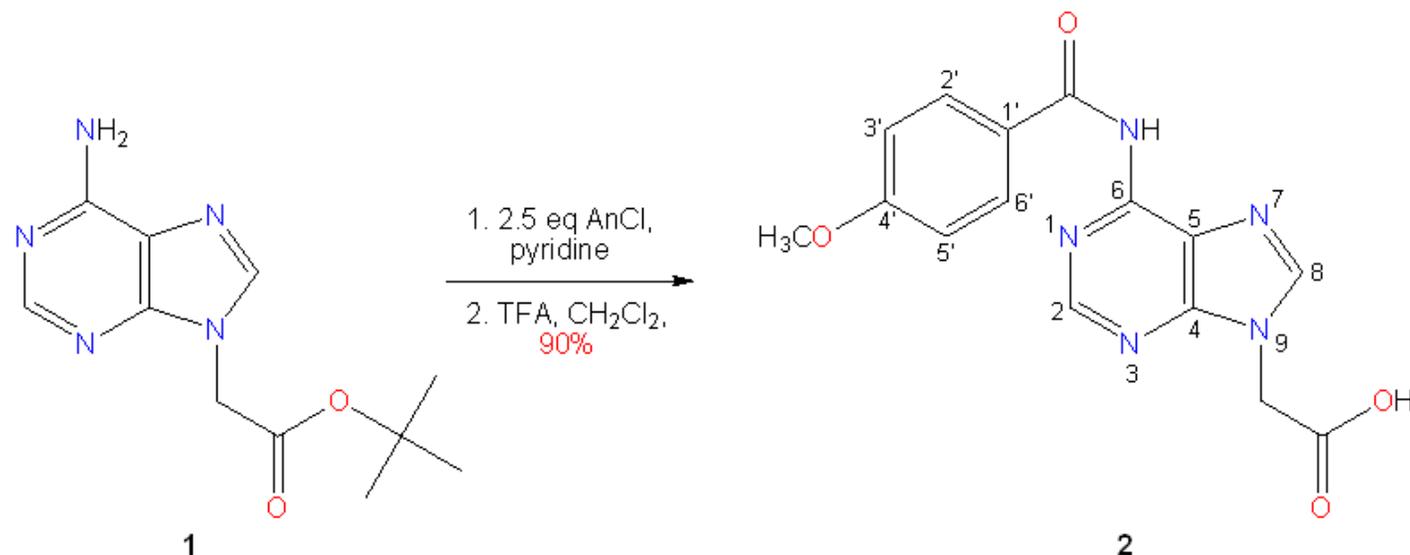


{6-[(4-Methoxybenzoyl)amino]-9H-purin-9-yl}acetic acid**Petra Pádár, Györgyi Kovács and Lajos Kovács***

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{6-[(4-Methoxybenzoyl)amino]-9H-purin-9-yl}acetic acid **2** is one of the substances used in the synthesis of peptide nucleic acid (PNA) adenine monomer and oligomers as well. In our previous work [1] the title acid **2** was prepared by controlled basic hydrolysis of the corresponding N^6, N^6 -dianisoylated ethyl ester derivative. The pH of this reaction solution was carefully maintained to prevent undesired hydrolysis of the N^6 -(4-methoxybenzoyl) (anisoyl) protecting group which reduced the yield. In our present approach we have chosen the *tert*-butyl ester **1** as starting material [2] for anisoylation and the resulting N^6, N^6 -dianisoylated substance was subjected to acidic hydrolysis to afford compound **2** in an excellent overall yield. In this latter step the simultaneous removal of one anisoyl and *tert*-butyl groups happened.

Experimental: *tert*-Butyl (6-amino-9H-purin-9-yl)acetate **1** (10.00 g, 40.1 mmol) [2] was suspended in anhydrous pyridine (120 mL), heated to 80 °C for 30 min, then cooled to room temperature. 4-Methoxybenzoyl chloride (17.90 g, 100 mmol) was added in portions and the mixture was stirred for 18 h at room temperature, then evaporated *in vacuo* and the residue was coevaporated with toluene (3 x 50 mL). The residue was dissolved in dichloromethane (160 mL) and the solution was washed with 10% (w/v) citric acid (2 x 70 mL), dried (MgSO₄) and evaporated *in vacuo*. The crude product was dissolved in dichloromethane (150 mL), trifluoroacetic acid (32 mL) was added, followed by water (1.6 mL) as a scavenger, and the mixture was stirred for 2 days at room temperature. The solution was evaporated *in vacuo*, coevaporated with toluene (2 x 50 mL), and acetonitrile (3 x 50 mL). The residue was thoroughly triturated with diethyl ether (5 x 50 mL), and filtered to give a white powder **2** (11.80 g, 90%), mp. 220 °C (darkens), 226 °C (decomp.) TLC: single spot. An analytically pure sample was obtained by recrystallisation from methanol (1500 mL of solvent was required for the above amount).

Mp. 231-236 °C. (lit. [3] 222-226 °C).

TLC: *n*-butanol-acetic acid-water: 4:1:1, R_f: 0.37.

¹H NMR (DMSO-*d*₆, 500 MHz, ppm, asterisks denote interchangeable assignments): 3.84 (s, 3H, OCH₃), 5.12 (s, 2H, CH₂), 7.07 (d, *J*=8.3 Hz, 2H, H-3', H-5'), 8.06 (d, *J*=8.3 Hz, 2H, H-2', H-6'), 8.45 (s, 1H, H-8*), 8.71 (s, 1H, H-2*).

^{13}C NMR (DMSO- d_6 , 125 MHz, ppm, assignment based on decoupled and J -modulated spin echo experiments): 44.18 (CH₂), 55.37 (OCH₃), 113.59 (C-3', C-5'), 125.00 (C-5), 125.49 (C-1'), 130.44 (C-2', C-6'), 144.84 (C-8), 150.23 (C-4), 151.40 (C-2), 152.39 (C-6), 162.47, 165.00, 168.92 (2xCO, C-4').

ESI-MS (m/z): 328 ([M+H]⁺).

Anal. calcd. for C₁₅H₁₃N₅O₄ (327.295): C, 55.05; H, 4.00; N, 21.40; found: C, 55.23; H, 4.09; N, 21.31%.

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Sample availability: sample available from the authors.

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