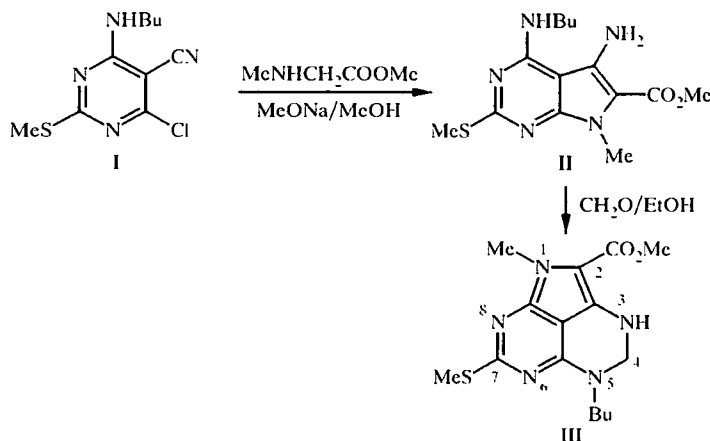


NEW HETEROCYCLIC SYSTEM, 1,3,5,6,8-PENTAAZAACENAPHTHENE

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This work continues our investigations on the synthesis of *peri*-condensed heterocycles based on the cyclocondensation of 4,5-diaminothieno[2,3-*d*]pyrimidines with certain one-carbon electrophilic reagents [1,2]. In the present communication we propose a synthesis of 5-butyl-1-methyl-7-methylthio-1,3,4,5-tetrahydro-1,3,5,6,8-pentaazaacenaphthene-2-carboxylic acid methyl ester (III), the first representative of a new heterocyclic system.

The synthesis of compound III was effected by the interaction of 4-butylamino-6-chloro-2-methylthio-pyrimidine-5-carbonitrile (I), obtained previously by us [3], with sarcosine methyl ester in the presence of sodium methylate, and subsequent cyclocondensation of the pyrrolo[2,3-*d*]pyrimidine derivative (II) with formaldehyde.



EXPERIMENTAL

4-Butylamino-7-methyl-2-methylthio-7H-pyrrolo[2,3-*d*]pyrimidine-6-carbonitrile (II). Mixture of compound I (2 g; 9.1 mmol), methanol (25 ml), sarcosine methyl ester hydrochloride (2.52 g; 18.1 mmol), and Na₂CO₃ (2 g; 18.9 mmol) was boiled for 5.5 h. The solution was filtered hot, and the filtrate evaporated to one third the initial volume. Water was added to precipitate the product completely, the solid was filtered off, and recrystallized. Compound II (1.39 g; 55%) was obtained; mp 135.5-136°C (2-propanol). ¹H NMR spectrum (DMSO-*d*₆): 1.0 (3H, t, CH₃); 1.4 (2H, m, CH₂); 1.65 (2H, m, CH₂); 2.5 (3H, s, SCH₃); 3.5 (2H, q, NCH₂); 3.7 (3H, s, NCH₃); 3.8 (3H, s, OCH₃); 6.1 (2H, s, NH₂); 7.0 ppm (1H, br. t, NH). IR spectrum: 3418, 3335, 3231 (NH, NH₂), 1693 cm⁻¹ (CO). Found, %: C 51.81; H 6.46; N 21.82. C₁₄H₂₁N₅O₂S. Calculated, %: C 51.95; H 6.54; N 21.64.

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5-Butyl-1-methyl-7-methylthio-1,3,4,5-tetrahydro-1,3,5,6,8-pentaazaacenaphthene-2-carboxylic Acid Methyl Ester (III). Formalin (32%) (0.081 g; 0.86 mmol) was added to solution of compound II (0.14 g; 0.43 mmol) in methanol (10 ml). The reaction mixture was boiled for 6 h, formalin (0.081 g) being added after each 2 h period (twice). After cooling the reaction mixture to room temperature, the solid was filtered off, and recrystallized. Compound III (0.07 g; 48%) was obtained; mp 149-150°C (methanol). ¹H NMR spectrum (DMSO-d₆): 0.9 (3H, t, CH₃); 1.41 (2H, m, CH₂); 1.73 (2H, m, CH₂); 2.5 (3H, s, SCH₃); 3.76 (3H, t, NCH₂); 3.83 (3H, s, NCH₃); 3.86 (3H, s, OCH₃); 4.85 (2H, d, CH₂); 6.97 ppm (1H, br. t, NH). IR spectrum: 3389 (NH), 1696 cm⁻¹ (CO). Found, %: C 53.74; H 6.36; N 20.48. C₁₅H₂₁N₅O₂S. Calculated, %: C 53.7; H 6.31; N 20.08.

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