UDC 547. 859 + 542. 95

SYNTHESIS OF IMIDAZO[1, 2-*f*]PURINE DERIVATIVES

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Apparently the heterocyclic system imidazo[1, 2-*f*]purine is hitherto undescribed. Synthesis of a number of derivatives of the tricyclic system A has now been effected by applying imidazole ring closure of heterocyclic 2-amino compounds [1-3], using methods described in the literature. Reaction of 8-aminotheophylline with α-halogenoketones gives 7-β-ketoalkyl(aryl)-8-aminotheophyllines, cyclized to derivatives of A by heating with acids or treatment with dehydrating agents.

A

7-Phenacyl-8-aminotheophylline, mp 248-150° (decomp, from alcohol). Found: C 57. 52; H 4. 71; N 22. 45%. Calculated for $C_{15}H_{15}N_5O_3$: C 57. 50; H 4. 83; N 22. 35%.

2-Phenyl-6, 8-dimethylimidazo[1, 2-*f*]xanthine, decomp >320° (from AcOH). Found: C 60. 97; H 4. 41; N 23. 90%. Calculated for $C_{15}H_{13}N_5O_2$: C 61. 01; H 4. 44; N 23. 72%.

7-p-Bromophenacyl-8-aminotheophylline, mp 270-275° (decomp, from alcohol). Found: C 45. 66; H 3. 84; Br 20. 32; N 17. 97%. Calculated for $C_{15}H_{14}BrN_5O_3$: C 45. 91; H 3. 60; Br 20. 37; N 17. 86%.

2-p-Bromophenyl-6, 8-dimethylimidazo[1, 2-*f*]xanthine, decomp >325° (from AcOH). Found: C 47. 95; H 3. 39; N 18. 91%. Calculated for $C_{15}H_{12}BrN_5O_2$: C 48. 14; H 3. 23; N 18. 72%.

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SYNTHESIS AND ACIDOCHROMIC CONDENSATION OF BENZYL-β-ANILINOETHYLAMIDE

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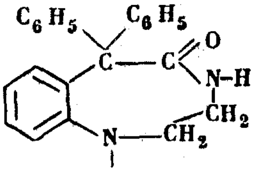
It has been shown that treatment of ethyl β-anilinoethyloxaminate (I) with phenylmagnesium bromide gives benzyl-β-(N-anilino)ethylamide (II). Treatment of the latter with acetic anhydride give benzyl-β-(N-acetylamino)ethylamide (III), which on treatment with concentrated sulfuric acid undergoes the acidochromic condensation. From the equation for the reaction and the analytical results, the product is assigned the structure 2-[N-(β-aminoethyl)acetamide]-triphenylacetic acid lactam (IV).

$C_6H_5NH(CH_2)_2NHCOCOOC_2H_5 \rightarrow C_6H_5NH(CH_2)_2NHCOC(OH)(C_6H_5)_2 \rightarrow$

I II

$C_6H_5N(COCH_3)(CH_2)_2NHCOC(OH)(C_6H_5)_2 \rightarrow$

III



IV

I was obtained in 94.5% yield from N-phenylethylenediemaine and diethyloxalate [1]. Plates, mp 85-86° (from aqueous ethanol). Found: N 11.95; 12.17%. Calculated for $C_{12}H_{16}O_3N_2$: N 11.87%.

II was prepared by reacting 0.1 mole phenylmagnesium bromide in 60 ml ether with 0.02 mole I. The organomagnesium complex was decomposed with a saturated solution of NH_4Cl, the ethereal layer steam distilled, and the solidified residue crystallized from alcohol. Needles mp 133-134°. Yield 6.5 g (98%). With conc. H_2SO_4 it gave a greenish brown color, which changed to brown. Found: N 8.25, 8.35%. Calculated for $C_{22}H_{22}O_2N_2$: N 8.09%.

To synthesize III, 8 g amide II was boiled for 10 min with 20 ml acetic anhydride, then diluted with 50 ml water, and after the anhydride had been hydrolyzed, the precipitate was filtered off and crystallized from dilute AcOH. Plates mp 189-190°, yield 5 g (55.8%). With conc. H_2SO_4 it gave a bluish-green color, changing to brown. Found: C 74.15, 73.97; H 6.31, 6.41; N 7.00, 7.18%. Calculated for $C_{24}H_{24}O_3N_2$: C 74.26; H 6.23; N 7.21%.

To prepare IV, 4 g III was dissolved in 7 ml glacial AcOH, 20 ml conc. H_2SO_4 added, and the whole allowed to stand till a persistent brown color appeared. It was then diluted with 100 ml water, the precipitate filtered off, and crystallized from alcohol. Plates mp 178-180°, yield 3.6 g (94.2%). Found: C 77.76, 77.87; H 6.25, 6.13; N 7.47, 7.61%. Calculated for $C_{24}H_{22}O_2N_2$: C 77.81; H 5.99; N 7.56%.

We intend to carry out similar syntheses using other arylamines.

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