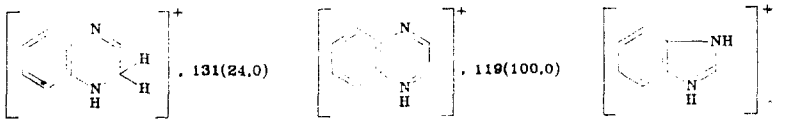


91 (32.9) $[p\text{-}CH_3M_6H_4]^+$, in agreement with the data in [6].

LITERATURE CITED

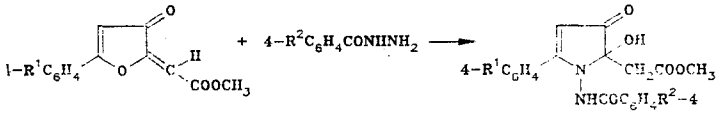
1. Yu. S. Andreichikov, V. O. Kozminych, N. M. Igidov, and E. N. Kozminych, Vth Int. Symp. on Furan Chemistry, Riga (1988), p. 127.
2. Yu. S. Andreichikov, V. O. Koz'miykh, and E. N. Manelova, Zh. Org. Khim., 21, 402 (1985).
3. Yu. S. Andreichikov and V. O. Koz'minykh, Zh. Org. Khim., 25, 618 (1989).
4. R. M. Acheson and W. R. Tully, J. Chem. Soc., No. 8, 1117 (1970).
5. A. N. Kost, Z. F. Solomko, V. A. Budylin, and T. S. Semenova, Khim. Geterotsikl. Soedin., No. 5, 696 (1972).
6. A. N. Kost, P. A. Sharbatyan, P. B. Terent'ev, Z. F. Solomko, V. S. Tkachenko, and L. G. Gergel', Zh. Org. Khim., 8, 2113 (1972).

SYNTHESIS OF 5-ARYL-1-(AROYLAMINO)-2-HYDROXY-2-(METHOXYCARBONYLMETHYL)-2,3-DIHYDROPYRROL-3-ONES

E. N. Koz'minykh, V. O. Koz'minykh, and Yu. S. Andreichikov

UDC 547.724'725+547.745'746+547.775

Previously it was determined that 5-aryl-2-(methoxycarbonylmethylene)-2,3-dihydrofuran-3-ones (I) react with a 70% aqueous solution of hydrazine in ethanol with the formation of 7-aryl-1,2,3,4-tetrahydropyridazino[4,3-c]pyridazin-3-ones [1]. In carrying out the reaction of compounds Ia and Ib with aroylhydrazines under similar conditions, we unexpectedly obtained 5-aryl-1-(aroylamino)-2-hydroxy-2-(methoxycarbonylmethyl)-2,3-dihydropyrrol-3-ones (IIa and IIb).



Ia,b IIa,b

Ia, IIa R^1=H, Ib, IIb R^1=CH_3; IIa R^2=CH_3, IIb R^2=CH_3O

Apparently, pyrrolones II are formed as a result of nucleophilic addition due to attack of the amino group of aroylhydrazine at the electrophilic center at the $C_{(5)}$ atom of the heterocycle with subsequent recyclization of the intermediate enehydrazine. The amide group NH does not participate in recyclization because of significantly decreased nucleophilicity.

To a solution of 0.01 mole of compounds Ia and Ib [2] in 150 ml of 96% ethanol is added 0.01 mole of aroylhydrazine, and the whole is boiled for 10-15 min. The solvent is evaporated, the residue is washed with acetonitrile and crystallized from ethanol or a chloroform–hexane mixture (1:1), and compounds IIa and IIb are obtained.

Compound IIa. The yield was 78%, with mp 118-119°C (with decomposition). IR spectrum (KBr): 3430-3420 (NH), 1740 (CO ester), 1712 (CO ring), 1625 (CO amide), 1610-1590 cm^{-1} (C=C). PMR spectrum ($CDCl_3$): 3.65 (3H, singlet, OCH_3); 3.70 (2H, double doublet, CH_2, J_{gem} = 21.0 Hz); 5.37 (2H, broadened singlet, 4-H, OH); 7.23-7.78 (10H, multiplet, $2C_6H_5$); 8.01 ppm (1H, broadened singlet, NH). Mass spectrum, m/z (relative intensity, %): 366 (1) $[M]^+$, 348 (5) $[M - H_2O]^+$, 334 (11) $[M - CH_3CH]^+$, 290 (9) $[M - COOCH_3 - OH]^+$, 266 (19) [M –

Perm Pharmaceutical Institute, Perm 614600. Translated from Khimiya Geterotsiklicheskikh Soedinenii, No. 2, pp. 278-279, February, 1990. Original article submitted May 12, 1989.

 0009-3122/90/2602-0242$12.50

$CH_3OCOCH{=}C{=}O]^+$, 103 (10) $[C_6H_5C{\equiv}N]^+$, 77 (44) $[C_6H_5]^+$. The spectral data agree with those available for 2-(alkoxycarbonylmethyl)-2-hydroxy-1,5-diaryl-2,3-dihydropyrrol-3-ones [3].

Compound IIb. The yield was 77%, with mp 149-150°C (with decomposition).

LITERATURE CITED

1. Yu. S. Andreichikov, E. N. Koz'minykh, L. O. Kon'shina, and V. O. Koz'minykh, Khim. Geterotsikl. Soedin., No. 10, 1428 (1985).
2. Yu. S. Andreichikov, V. O. Koz'minykh, and E. N. Manelova, Zh. Org. Khim., 21, 402 (1985).
3. Yu. S. Andreichikov and V. O. Koz'minykh, Zh. Org. Khim., 25, 618 (1989).

SYNTHESIS OF 4,2'-ANHYDRO-4-HYDROXY-3-(α-D-XYLOFURANOSYL)HEXAHYDRO-PYRIMIDINE-2-THIONES

A. D. Shutalev, L. A. Ignatova, and B. V. Unkovskii

UDC 547.455.5'854.07'963.32

It has been shown earlier [1-3] that in the reaction of 2,3-O-isopropylideneribofuranosylamine p-toluenesulfonate with β-isothiocyanatoaldehydes in the presence of bases, 4,5'-anhydro-4-hydroxy-3-(2,3-O-isopropylidene-β-D-ribofuranosyl)hexahydropyrimidine-2-thiones are formed, which when heated in an aqueous acetic acid solution, successively convert into 4,5'-anhydro-4-hydroxy-3-(β-D-ribofuranosyl)hexahydropyrimidine-2-thiones, 4-hydroxy-3-(D-ribosyl)hexahydropyrimidine-2-thiones, and finally, 4,2-anhydro-4-hydroxy-3-(α-D-ribofuranosyl)hexahydropyrimidine-2-thiones. The formation of 4,5'- and 4,2'-anhydroribosides possibly proceeds by nucleophilic substitution of the hydroxyl group at the $C_{(4)}$ carbon atom of the pyrimidine ring by the action of the free hydroxy group of the sugar residue in the molecules of the intermediately formed 4-hydroxyhexahydropyrimidine-2-thione $N_{(3)}$-ribosides. As an extension of the investigations carried out in [1-3], it was of interest to study the reaction of 3,5-O-isopropylidenexylofuranosylamine p-toluenesulfonate (I), containing a free hydroxyl group at the $C_{(2)}$ carbon atom of the sugar residue, with β-isothiocyanatoaldehydes.

We found that in the reaction of compound I with 3-isothiocyanatopropanal (II) in chloroform in the presence of triethylamine at 0°C, 4,2'-anhydro-4-hydroxy-3-(3',5'-O-isopropylidene-α-D-xylofuranosyl)hexahydropyrimidine-2-thione (IIIa) is formed in a yield of 59%, mp 220-220.5°C (from methanol), $[\alpha]_D^{21}$ +156° (c = 0.855, DMSO). The reaction proceeds stereoselectively, and compound IIIa is obtained in the form of a single diastereomer, having, according to the PMR spectroscopy data, an (R)-configuration of a new chiral center at the $C_{(4)}$ carbon atom.

We showed that the isopropylidene protection in xyloside IIIa is very labile and is readily removed by dilute acetic acid or by alcohols in the presence of hydrochloric acid, as a result of which (4R)-4,2'-anhydro-4-hydroxy-3-(α-D-xylofuranosyl)hexahydropyrimidine-2-thione (IIIb) is formed in 82-98% yields, mp 164-164.5°C (from alcohol), $[\alpha]_D^{21}$ +160° (c = 0.77, DMSO) (see top of following page)

M. V. Lomonosov Moscow Institute of Fine Chemical Technology, Moscow 119831.

Translated from Khimiya Geterotsiklicheskikh Soedinenii, No. 2, pp. 279-280, February, 1990. Original article submitted June 1, 1989.

0009-3122/90/2602-0243$12.50