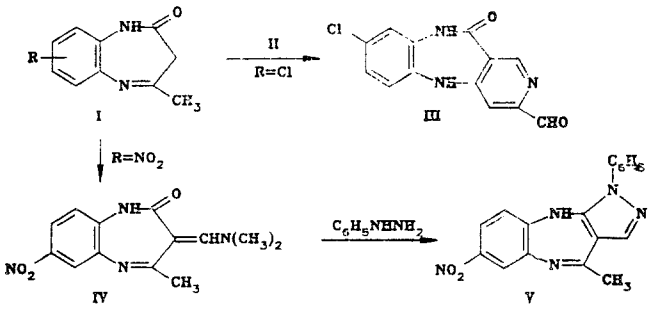
FORMYLATION OF 2,3-DIHYDRO-4-METHYL-1H-1,5-BENZODIAZEPIN-2-ONES

V. N. Proshkina, Z. F. Solomko,
and N. Ya. Bozhanova

UDC 547.891.2.07:543.51

Treatment of 2,3-dihydro-4-methyl-8-chloro-1H-1,5-benzodiazepin-2-one (Ia) with DMF and phosphorus oxychloride (II) in the ratio Ia:II of 1:4 and subsequent neutralization of the product with aqueous ammonia gives 10,11-dihydro-3-formyl-8-chloro-5H-pyrido[4,3-b]-[1,5]benzodiazepin-11-one (III). A similar scheme has been reported for pyrazolones [1]. Apparently the methyl and methylene groups are formylated at the same time and subsequent reaction of the intermediate with ammonia leads to aldehyde III with mp 301°C in 38% of yield. IR spectrum (KBr): 1680 (NHCO), 1707 (CHO), 3200-3067 cm^{-1} (NH); PMR spectrum (CF_3COOH), δ: 9.77 (1H, s, NH), 8.85 (1H, s, NH), 8.57 (1H, s, COH), 7.06 (1H, s, CH), 6.92 (1H, s, CH), 6.8-7.1 ppm (3H, m, Ar); mass spectrum, m/z (relative intensity, %): 275 (33), 274 (16), 273 (100), 247 (13), 246 (9), 245 (37), 210 (14), 182 (14)



Reaction of 2,3-dihydro-4-methyl-7-nitro-1H-1,5-benzodiazepin-2-one with the Vilsmeier reagent (1:4) permits the separation of the product formylated only at the methylene group. Further reaction of the diazepinone IV [2] with phenylhydrazine and subsequent cyclization of the phenylhydrazone in acetic acid leads to 1,10-dihydro-4-methyl-7-nitro-1-phenylpyrazolo[5,4-b][1,5]benzodiazepine (V) with mp 247°C in 98% yield. IR spectrum: 3293-3220 (NH), 1613-1493 (C=N and C=C), 1367, 1500 cm^{-1} (NO_2); mass spectrum, m/z (relative intensity, %): 319 (27), 318 (18), 316 (2), 287 (4), 274 (4), 273 (100), 272 (2), 262 (6), 245 (6) 245 (2), 225 (2), 224 (6), 211 (2), 210 (12), 184 (8).

Elemental analytical data agreed with that calculated.

LITERATURE CITED

1. S. B. Barnela, R. S. Pandit, and S. Seshadri, Indian J. Chem., 14B, 668 (1976).
2. Z. F. Solomko, V. N. Proshkina, N. Ya. Bozhanova, S. V. Loban', and L. N. Babichenko, Khim. Geterotsikl. Soedin., No. 2, 223 (1984).

Three-Hundred-Year-Union-of-the-Ukraine-with-Russia, State University, Dnepropetrovsk 320625. Translated from Khimiya Geterotsiklicheskikh Soedinenii, No. 9, p. 1288, September, 1988. Original article submitted February 3, 1988.

0009-3122/88/2409-1071$12.50