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NEW METHOD FOR THE DIRECT CYANOETHYLATION OF PYRAZOLE DERIVATIVES

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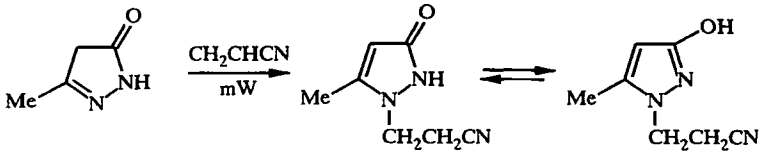
Pyrazol-5-ones unsubstituted at $N_{(1)}$ atom can be cyanoethylated at the oxygen atom or in position 4 in accordance with their structures; 1-cyanoethyl compounds are obtained in a different way [1]. We have shown that acrylonitrile (used in a ratio 1:2) cyanoethylates 3-methylpyrazol-5-one on the surface of neutral aluminum oxide without any solvent when the process is activated by microwave radiation (MO785VT domestic microwave oven, power 850 W, 30 min). The process leads to formation of the previously unknown 1-cyanoethyl-5-methylpyrazol-3-one (yield 59%), i.e., the attack is at the imine nitrogen atom. The compound exists mainly in the hydroxypyrazole form. The same reaction occurs when one uses aluminum oxide modified with caustic soda.



The direct interaction of the same pyrazol-5-one with acrylonitrile, as activated by microwaves, leads to 1-cyanoethyl-3-methylpyrazol-5-one with a low yield. Cyanoethylation of 3,5-dimethylpyrazole under the same conditions leads to a mixture of 1- and 4-cyanoethyl derivatives in a ratio of about 2:1.

1-Cyanoethyl-5-methylpyrazol-3-one, mp 166°C, IR spectrum 3200, 2270, 1710 cm^{-1}. PMR spectrum (DMSO-d_6): 2,25 (3H, s, 5-CH_3); 2,77 (2H, t, N—CH_2); 4,08 (2H, t, CH_2—CN); 5,45 ppm (1H, s, 4-H). ^{13}C NMR spectrum ($CDCl_3$): 11,14; 18,91 (CH_3); 37,16 (N—CH_2); 57,04 (CH_2—CN); 91,82 ($C_{(4)}$); 116,69 (CN); 144,80 ($C_{(5)}$); 177,79 ppm ($C_{(3)}$). Mass spectrum, *m/z* (*I*, %): 151 (30) [M^+]; 111 (100); 98 (5); 82 (27); 67 (7); 54 (12).

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