ISSN 1070-3632, Russian Journal of General Chemistry, 2011, Vol. 81, No. 11, pp. 2385–2386. © Pleiades Publishing, Ltd., 2011.
Original Russian Text © V.D. Dyachenko, Yu.Yu. Pugach, O.V. Shishkin, 2011, published in Zhurnal Obshchei Khimii, 2011, Vol. 81, No. 11, pp. 1932–1933.

LETTERS
TO THE EDITOR

Unusual Formation of 2-(4-Morpholino-5*H*-chromeno-[2,3-*d*]pyrimidin-2-yl)phenol

V. D. Dyachenko[a], **Yu. Yu. Pugach**[a], **and O. V. Shishkin**[b]

[a] *Taras Shevchenko Lugansk National University, Oboronnaya ul. 2, Lugansk, 91011 Ukraine*
e-mail: dvd_lug@online.lg.ua

[b] *Institute of Single Crystals, National Academy of Sciences of Ukraine, Khar'kov, Ukraine*

Received February 15, 2011

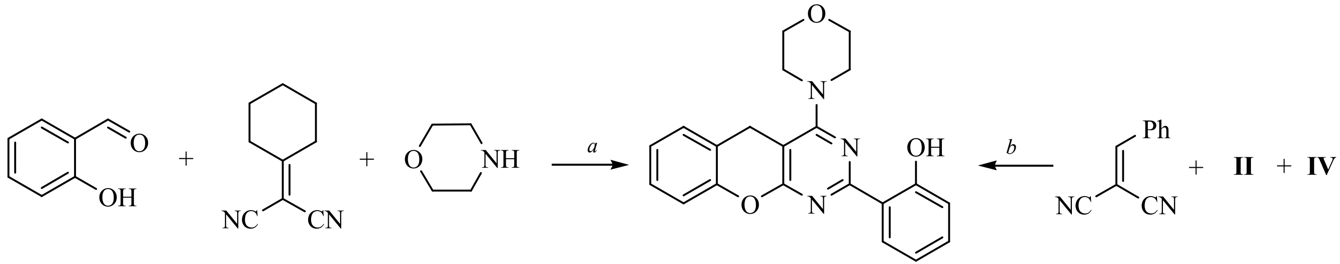
DOI: 10.1134/S1070363211110338

2-(4-Morpholino-5*H*-chromeno[2,3-*d*]pyrimidin-2-yl)phenol **I** has been recently synthesized via the three-component condensation of salicylic aldehyde **II**, malonic nitrile **III** and morpholine **IV** in ethanol in the presence of $LiClO_4$ [1], as well as under the microwave irradiation of the reactants **II**–**IV** at 100°C for 6.3 min [2].

The compound **I** was for the first time shown to be formed in the reaction of equimolar amounts of salicylic aldehyde **II**, cyclohexylidene malodinitrile **V** and morpholine **IV** in ethanol at 20°C (method *a*). Replacement of compound **V** with benzylidene malonodinitrile **VI** (method *b*) does not change the reaction direction: a heterocyclic compound **I** is formed. The structure of the latter was studied by the X-ray diffraction which results will be reported later.

The mechanism of this reaction and the limits of its applicability are under study.

2-(4-Morpholino-5*H*-chromeno[2,3-*d*]pyrimidin-2-yl)phenol (I). *a.* A mixture of 7.1 ml (10 mmol) of salicylal **II**, 0.87 ml (10 mmol) of morpholine **IV** and 1.46 g (10 mmol) of cyclohexylidene malonodinitrile **V** in 20 ml of ethanol at 20°C was stirred for 1 h and kept for 48 h. The resulting precipitate was filtered off, washed with ethanol and hexane. Yield 2.6 g (72%), yellow crystals, mp 205–208°C (EtOH), (210°C [2]). IR spectrum, ν, cm^{-1}: 3445 (OH). ^1H NMR spectrum, δ, ppm: 3.52 t (4H, CH_2NCH_2, *J* 4.0 Hz), 3.80 t (4H, CH_2OCH_2, *J* 4.0 Hz), 4.01 s (2H, CH_2), 6.93 t (2H, H_{Ar}, *J* 8.0 Hz), 7.11–7.20 m (2H, H_{Ar}), 7.27 t (1H, H_{Ar}, *J* 8.0 Hz), 7.33–7.41 m (2H, H_{Ar}), 8.27 d (1H, H_{Ar}, *J* 8.0 Hz), 13.11 br. s (1H, OH). ^{13}C NMR spectrum, δ_C, ppm: 24.99, 48.01, 65.88, 97.56, 116.24, 117.30, 117.99, 118.78, 119.82, 124.52, 128.05, 128.59, 128.96, 132.84, 159.69, 160.49, 163.98. Mass spectra, *m*/*z* (I_{rel}, %): 361 (100) $[M]^+$, 304 (28), 275 (14), 248 (5), 155 (11), 128 (13), 86 (24) $[\text{morpholinyl}]^+$. Found, %: C 69.68; H 5.24; N 11.52. $C_{21}H_{19}N_3O_3$. Calculated, %: C 69.79; H 5.30; N 11.63.



II **V** **IV** **I** **VI**

b. Similarly to method *a* using 1.54 g (10 mmol) of compound **VI**. Yield 2.78 g (77%).

The melting point was determined on a Kofler heating bench. The IR spectrum was registered on a FIR Spectrum One (Perkin Elmer) instrument from KBr pellets. The ^1H and ^{13}C NMR spectra were recorded on a Bruker Avance II 400 spectrometer (399.9601 MHz) in DMSO-d_6 with internal TMS. The mass spectrum was taken on a MX-1321 spectrometer (70 eV) using direct input of the sample into the ion source. The reaction progress was monitored by the TLC method on Silufol UV-254 plates eluting with acetone–hexane mixture (3:5) and detecting with iodine vapor and UV irradiation.

REFERENCES

1. Ghahremanzadeh, R., Amanpour, T., and Bazgir, A., *Tetrahedron Lett*., 2010, vol. 51, no. 31, p. 4202.
2. Zonouzi, A., Biniaz, M., Mirzazadeh, R., Talebi, M., and Ng, S.W., *Heterocycles*, 2010, vol. 81, no. 5, p. 1271.