*Chemistry of Heterocyclic Compounds, Vol. 44, No. 2, 2008*

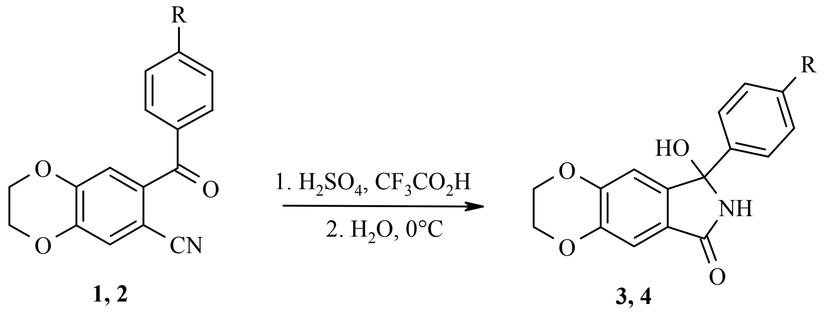
# 3-HYDROXYPHTHALIMIDINES FROM *o*-CYANOBENZOPHENONES

**S. S. Mochalov and M. I. Khasanov**

**Keywords:** 5,6-ethylenedioxy-3-hydroxyphthalimides, 2-cyano-4,5-ethylenedioxybenzophenones.

3-Hydroxyphthalimidines belong to a class of not readily available heterocyclic compounds. As species containing a γ-lactam ring, these compounds hold interest not only for biological research but also as starting materials for the synthesis of potential pharmaceutical agents [1]. Only a few individual 3-hydroxyphthalimidines have now been obtained by circuitous synthetic pathways [2-4] in the absence of a direct method of synthesis.

In a study of the hydrolysis of 2-cyano-4,5-ethylenedioxybenzophenones **1** and **2** by the action of a mixture of sulfuric and trifluoroacetic acids, we unexpectedly found that these compounds are directly converted in high yield to the corresponding 3-hydroxyphthalimides **3** and **4**.



**1, 2** **3, 4**

**1, 3** R = H; **2, 4** R = Me

The structure of initially obtained 3-hydroxyphthalimidines **3** and **4** was indicated by $^1$H NMR spectroscopy and mass spectrometry, while the structure of **3** was also established by X-ray diffraction structural analysis*.

We are presently determining whether this transformation can be used as a general method for the preparation of 3-hydroxyphthalimidines.

The $^1$H NMR spectra were taken on a Bruker ORX-500 spectrometer at 500 MHz in DMSO-$d_6$ with TMS as the internal standard. The mass spectra were taken on a Finnigan MAT INCOS 50 mass spectrometer.

---

* The X-ray diffraction structural analysis data for 5,6-ethylenedioxy-3-hydroxyphthalimidine (**3**) will be published in *Acta Crystallographica* (Section E) in 2008.

M. V. Lomonosov Moscow State University, 119992 Moscow, Russia; e-mail: ssmoch@org.chem.msu.ru. Translated from Khimiya Geterotsiklicheskikh Soedinenii, No. 2, pp. 294-296, February, 2008. Original article submitted January 16, 2008.

**5,6-Ethylenedioxy-3-hydroxy-3-phenylphthalimidine (3).** A solution of 2-cyano-4,5-ethylenedioxybenzophenone (**1**) (1.00 g, 3.8 mmol) in a mixture of concentrated sulfuric acid (1 ml, 8 g) and trifluoroacetic acid (4 ml, 4.7 g) was heated at reflux with stirring for 5 h. The reaction mixture was then poured into 1:1 water-ice (75 ml). The precipitate formed was filtered off, washed with water until the wash water was neutral, and recrystallized from acetone to give compound **3** (0.88 g, 82%), mp 212-213°C. $^1$H NMR spectrum, δ, ppm (*J*, Hz): 4.24 (4H, s, $OCH_2CH_2O$); 6.70 (1H, s, H-4), 6.80 (1H, s, OH); 7.05 (1H, s, H-7); 7.27 (1H, t, *J* = 7.2, H-4'); 7.33 (3H, t, *J* = 7.2, H-3', H-5'); 7.45 (2H, d, *J* = 7.4, ArH); 9.07 (1H, s, NH). Electron impact mass spectrum at 70 eV, *m/z* ($I_{rel}$, %): 283 $[M]^+$ (7), 265 (100), 237 (49), 223 (42), 206 (18), 209 (15), 188 (25), 181 (16), 162 (70), 153 (21), 149 (7), 134 (92), 119 (10), 105 (21), 77 (28), 62 (12), 58 (8), 50 (80), 43 (36), 39 (8). Found, %: C 67.55; H 4.41; N 4.72. $C_{16}H_{13}NO_4$. Calculated, %: C 67.84; H 4.62; N 4.95.

**5,6-ethylenedioxy-3-hydroxy-3-(*p*-tolyl) phthalimidine (4)** was obtained analogously from 2-cyano-4,5-ethylenedioxy-4'-methylbenzophenone (1.00 g, 3.6 mmol) in 86% yield (0.92 g). $^1$H NMR spectrum, δ, ppm (*J*, Hz): 2.27 (3H, s, $CH_3$); 4.24 (4H, s, $OCH_2CH_2O$); 6.68 (1H, s, H-4); 6.72 (1H, s, OH); 7.03 (1H, s, H-7); 7.13 (2H, d, *J* = 7.9) and 7.32 (2H, d, *J* = 7.9, ArH); 9.00 (1H, s, NH). Electron impact mass spectrum at 70 eV, *m/z* ($I_{rel}$, %): 297 $[M]^+$ (19), 279 (100), 264 (78), 251 (35), 237 (47), 223 (11), 206 (35), 195 (14), 188 (40), 162 (52), 139 (10), 134 (75), 119 (66), 104 (9), 91 (25), 78 (21), 65 (15), 50 (58), 39 (12). Found, %: C 68.18; H 5.14; N 4.54. $C_{17}H_{15}NO_4$. Calculated, %: C 68.67; H 5.09; N 4.71.

## REFERENCES

1. A. Eschenmoser, *Pure Appl. Chem.*, **7**, 297 (1963).
2. R. R. Schmidt and E. Schlipf, *Chem. Ber.*, **103**, 3783 (1970).
3. F. A. Golec and J. Auerbach, US Pat. 4331600; *http://www.espacenet.com.*
4. W. J. Houlihan and J. Nadelson, US Pat. 3959269; *http://www.espacenet.com.*