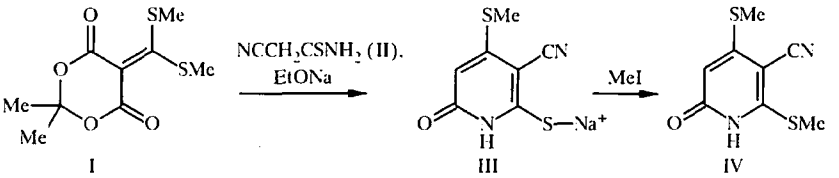
Chemistry of Heterocyclic Compounds, Vol. 35, No. 9, 1999

SYNTHESIS OF SODIUM 5-CYANO-4-METHYLTHIO-2-OXOPYRIDINE-6(1H)-THIOLATE AND ITS ALKYLATION

S. G. Krivokolysko, V. V. Dotsenko, V. D. Dyachenko, and V. P. Litvinov

We have previously used Meldrum's acid to synthesize sulfur-containing partially hydrogenated pyridones [1-3]. In this note we report the possibility of preparing non-hydrogenated pyridones by the reaction of di(methylthio)methylene-substituted Meldrum's acid I with cyanothioacetamide (II) by boiling in ethanol in the presence of sodium ethoxide. The synthesized sodium pyridine-2-thiolate (III) was converted into the corresponding sulfide IV by alkylation with methyl iodide.



I III IV

Sodium 5-Cyano-4-methylthio-2-oxopyridin-6(1H)-thiolate (III). Yield 61%; mp 340°C (dec.). IR spectrum: 3390-3540 (NH), 2200 (CN), 1620-1680 cm^{-1} (CO). ^1H NMR spectrum (DMSO-d_6): 2.32 (3H, s, SMe); 5.37 (1H, s, $C_{(3)}H$); 10.61 ppm (1H, br. s., NH). Found, %: C 38.33; H 2.11; N 12.94; S 29.02. $C_7H_5NaN_2OS_2$. Calculated, %: C 38.17; H 2.29; N 12.72; S 29.12.

5-Cyano-4,6-dimethylthiopyridin-2(1H)-one (IV). Yield 77%; mp 270-273°C. IR spectrum: 3390-3540 (NH), 2200 (CN), 1620-1710 cm^{-1} (CO). ^1H NMR spectrum (DMSO-d_6): 2.54 and 2.58 (6H, s, 2SMe); 6.26 (1H, s, $C_{(3)}H$); 12.0 ppm (1H, br. s., NH). Found, %: C 45.11; H 3.95; N 13.42; S 30.13. $C_8H_8N_2OS_2$. Calculated, %: C 45.26; H 3.80; N 13.20; S 30.21.

REFERENCES

1. V. N. Nesterov, S. G. Krivokolysko, V. D. Dyachenko, V. V. Dotsenko, and V. P. Litvinov, *Izv. Akad. Nauk, Ser. Khim.*, No. 5, 1029 (1997).
2. V. D. Dyachenko, S. G. Krivokolysko, and V. P. Litvinov, *Izv. Akad. Nauk, Ser. Khim.*, No. 10, 1852 (1997).
3. V. D. Dyachenko, S. G. Krivokolysko, and V. P. Litvinov, *Izv. Akad. Nauk, Ser. Khim.*, No. 11, 2016 (1997).

Taras Shevchenko Lugansk State Pedagogical University, Lugansk 348011, Ukraine; e-mail: kgb@lgpi.lugansk.ua; N. D. Zelinsky Institute of Organic Chemistry, Russian Academy of Sciences, Moscow 117913, Russia; e-mail ope@cacr.ioch.ac.ru. Translated from Khimiya Geterotsiklicheskikh Soedinenii, No. 9, 1256-1257, September, 1999. Original article submitted May 18, 1999.

0009-3122/99/3509-1111$22.00©1999 KluwerAcademic/Plenum Publishers