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SYNTHESIS OF S-(3-CHLOROQUINOXALIN-2-YL) ESTERS OF ALIPHATIC AND AROMATIC THIOSULFONIC ACIDS

V. I. Lubenets, S. V. Vasylyuk, and V. P. Novikov

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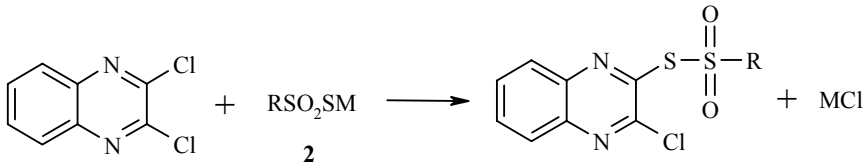
Despite the high biological activity of 2,3-quinoxaline derivatives [1, 2], no data are available on synthesis of their derivatives containing a thiosulfonate moiety. Earlier [3] in order to obtain the S-(3-chloroquinoxalin-2-yl) ester of 4-acetylaminobenzenethiosulfonic acid, we studied the reaction of sodium 4-acetylaminobenzenethiosulfonate with 2,3-dichloroquinoxaline in DMF at 65-70°C, but instead of the target product, under these conditions 5a,13a-dihydro-1,4-dithiinodi[2,3-*b*]quinoxaline was formed.

While continuing studies of the reaction of salts of aliphatic and aromatic thiosulfonic acids in aprotic solvents (acetone, dioxane) at temperatures of 20-45°C, for the first time we have obtained S-(3-chloroquinoxalin-2-yl) esters of aliphatic and aromatic thiosulfonic acids in 20%-52% yields. The structure of compounds **3a-c** has been confirmed by spectral data and elemental analysis.



1 **3a–c**

3 a R = *i*-Bu, **b** R = Ph, **c** R = $MeCONHC_6H_4$; M = Na, K

The ^{1}H NMR spectra were taken on a Varian VXR spectrometer (300 MHz) in DMSO-d_6, internal standard TMS.

S-(3-Chloroquinoxalin-2-yl) Ester of Isobutylthiosulfonic Acid (3a). Yield 20%; mp 130-131°C (ethanol). IR spectrum (KBr), ν, cm^{-1}: 1120, 1320 (SO_2). ^{1}H NMR spectrum, δ, ppm (*J*, Hz): 0.98 (3H, s, CH_3); 1.00 (3H, s, CH_3); 2.58 (1H, m, CH); 3.51-3.52 (2H, d, *J* = 3.0, CH_2); 7.56-8.27 (4H, m, H-5,6,7,8). Found, %: C 45.08; H 4.37; Cl 10.83; N 8.67; S 20.45. $C_{12}H_{13}ClN_2O_2S_2$. Calculated, %: C 45.49; H 4.14; Cl 11.19; N 8.84; S 20.24.

S-(3-Chloroquinoxalin-2-yl) Ester of Phenylthiosulfonic Acid (3b). Yield 48%; mp 121-122°C (ethanol). IR spectrum (KBr), ν, cm^{-1}: 1114, 1320 (SO_2), 1586, 1600 (Ar). ^{1}H NMR spectrum, δ, ppm: 7.44-8.87 (9H, m, Ar). Found, %: C 49.21; H 2.85; Cl 10.06; N 7.91; S 18.52. $C_{14}H_9ClN_2O_2S_2$. Calculated, %: C 49.93; H 2.68; Cl 10.55; N 8.32; S 19.02.

Lvov Polytechnical National University, Lvov 79013, Ukraine; e-mail: vnovikov@polynet.lviv.ua.
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S-(3-Chloroquinoxalin-2-yl) Ester of 4-Acetylaminobenzenethiosulfonic Acid (3c). Yield 52%; mp 174°C (methanol). IR spectrum (KBr), ν, cm^{-1}: 1140, 1336 (SO_2), 1578, 1592, 1600 (Ar), 1616 (NH), 1674 (C=O), 3304 (NH). ^1H NMR spectrum, δ, ppm: 2.12 (3H, s, $COCH_3$), 7.59-8.59 (8H, m, Ar). Found, %: C 48.28; H 3.52; Cl 8.61; N 10.23; S 15.85. $C_{16}H_{12}ClN_3O_3S_2$. Calculated, %: C 48.79; H 3.05; Cl 9.02; N 10.67; S 16.26.

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