

LETTERS  
TO THE EDITOR

## Reaction of 4-Dichloromethylene-*p*-tolyl-1,3-oxazol-5(4*H*)-one with *o*-Aminothiophenol

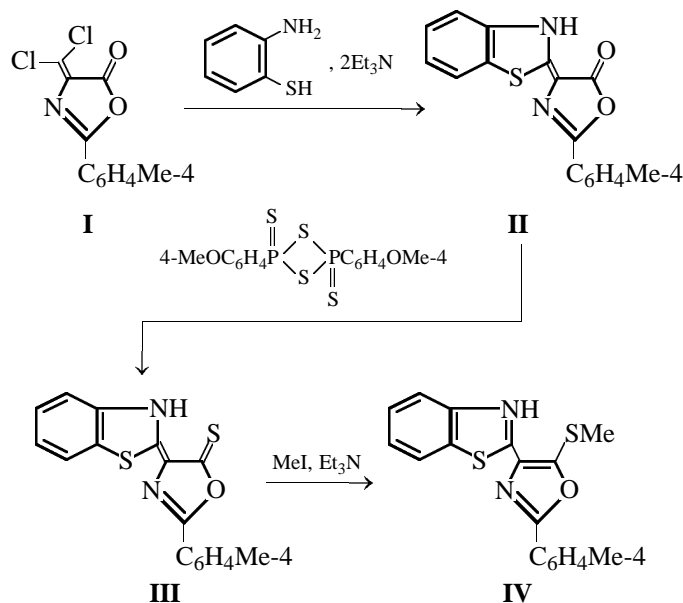
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Numerous substituted oxazoles and thiazoles have been prepared from accessible unsaturated azlactones containing a dichloromethylene group at the 4-position

[1–4]. Here we report on a new synthetic application of these agents: preparation of previously unknown 5-mercapto-1,3-oxazole derivatives (see scheme).



In the **I** → **II** transformation, the azlactone ring is undoubtedly preserved, as judged from the IR spectra in which the C=O stretching band in the 2-oxazolin-5-one moiety is readily identified. The structure of the final product of the reaction sequence **II** → **III** → **IV** is confirmed not only by IR, but also by <sup>1</sup>H NMR spectra confirming formation of the methylthio group. Other data indicating the structure of **IV** and its analogs will be considered in a forthcoming paper.

**2-(5-Oxo-2-*p*-tolyl-1,3-oxazol-5-ylidene)-2,3-dihydro-1,3-benzothiazole II.** To a solution of 0.008 mol of azlactone **I** [5] in 20 ml of tetrahydro-

furan, we added with stirring and cooling with ice-cold water 0.01 mol of triethylamine and a solution of 0.008 mol of *o*-aminothiophenol in 15 ml of tetrahydrofuran over a period of 30 min. The mixture was allowed to stand for 24 h at 20–25°C, the precipitate of triethylammonium chloride was filtered off, the solvent was removed in a vacuum, and the residue was treated with water, dried in a vacuum desiccator over phosphorus pentoxide, and recrystallized from dioxane–benzene, 1 : 1; yield 95%. IR spectrum (KBr),  $\nu$ , cm<sup>−1</sup>: 1720 (C=O), 3205 (NH). Found, %: N 9.20; S 10.35. C<sub>17</sub>H<sub>12</sub>N<sub>2</sub>O<sub>2</sub>S. Calculated, %: N 9.08; S 10.40.

**2-(5-Methylthio-2-*p*-tolyl-1,3-oxazol-4-yl)-1,3-benzothiazole IV.** A mixture of equimolar amounts of **II** and Lawesson's reagent (0.005 mol each) in 20 ml of dioxane was refluxed for 48 h, the solvent was removed in a vacuum, and the residue was treated with 20 ml of 10% NaOH solution for 2 h. The precipitate of **III** was filtered off, washed with water, dried in a vacuum desiccator over phosphorus pentoxide, and then suspended in 15 ml of ethanol. To this mixture, 0.006 mol of triethylamine and 0.006 mol of methyl iodide were added. The mixture was refluxed for 3 h and cooled with ice-cold water; the precipitate was filtered off and recrystallized from acetonitrile; yield of **IV** 70%. IR spectrum (KBr),  $\nu$ ,  $\text{cm}^{-1}$ : no bands at 1610–1800 and 3200–3600.  $^1\text{H}$  NMR spectrum,  $\delta$ , ppm: 2.41 s (3H,  $\text{C}_6\text{H}_4\text{CH}_3$ ), 2.76 s (3H,  $\text{SCH}_3$ ), 7.22–8.10 m (8H,  $2\text{C}_6\text{H}_4$ ). Found, %: C 63.75; H 4.08; N 8.59; S 18.70.  $\text{C}_{18}\text{H}_{14}\text{N}_2\text{OS}_2$ . Calculated, %: C 63.88; H 4.17; N 8.28; S 18.95.

The IR spectra were recorded on a Specord M-80 spectrophotometer in KBr. The  $^1\text{H}$  NMR spectra were taken on a Varian VXR-300 spectrometer (solvent  $\text{DMSO}-d_6$ , internal reference TMS).

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