

LETTERS
TO THE EDITOR

Synthesis of 5-Alkyl(benzyl)idene-3-hydroxymethyl-2-thioxothiazolidin-4-ones

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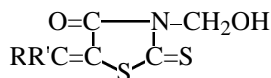
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Received December 24, 2002

3-Hydroxymethylthiazolidinones are practically important compounds which are usually obtained by reaction of thiazolidin-4-ones with formaldehyde in organic solvents in the presence of a catalytic amount of an amine. The reaction is reversible, and in some cases the yields of the target products are low [1].

With the goal of extending the synthetic potential of solid-phase reactions we made an attempt to prepare 3-hydroxymethylthiazolidin-4-ones by reaction of gaseous formaldehyde with crystalline salts derived from thiazolidin-4-ones. As examples we selected ammonium salts of 2-thioxothiazolidin-4-one.

Initial 2-thioxothiazolidin-4-one alkylammonium salt was placed in a glass tube, and gaseous formaldehyde was passed through the tube until the substrate was converted completely into final product I–X. The progress of the reaction was monitored by TLC [2], following the disappearance of the spot belonging to the initial compound.



I, R = Me, R' = Me; II, R = Me, R' = Et; III, R = Me, R' = Pr; IV, R = Me, R' = Bu; V, RR' = (CH₂)₅; VI, R = Me, R' = C₅H₁₁; VII, R = Me, R' = C₆H₁₃; VIII, R = Me, R' = Ph; IX, R = H, R' = Ph; X, R = H, R' = *p*-MeC₆H₄.

3-Hydroxymethyl-5-isopropylidene-2-thioxothiazolidin-4-one (I). Yield 98%, mp 114°C, *R_f* 0.34. Found, %: N 7.11; S 31.72. C₇H₉NO₂S₂. Calculated, %: N 6.89; S 31.55.

3-Hydroxymethyl-5-(1-methylpropylidene)-2-thioxothiazolidin-4-one (II). Yield 98%, mp 65°C, *R_f* 0.30. Found, %: N 6.70; S 29.80. C₈H₁₁NO₂S₂. Calculated, %: N 6.45; S 29.51.

3-Hydroxymethyl-5-(1-methylbutylidene)-2-

thioxothiazolidin-4-one (III). Yield 97%, mp 75°C, *R_f* 0.37. Found, %: N 6.27; S 27.82. C₉H₁₃NO₂S₂. Calculated, %: N 6.06; S 27.72.

3-Hydroxymethyl-5-(1-methylpentylidene)-2-thioxothiazolidin-4-one (IV). Yield 97%, mp 55°C, *R_f* 0.39. Found, %: N 5.62; S 25.80. C₁₀H₁₅NO₂S₂. Calculated, %: N 5.71; S 26.14.

3-Hydroxymethyl-5-cyclohexylidene-2-thioxothiazolidin-4-one (V). Yield 98%, mp 122°C, *R_f* 0.32. Found, %: N 6.02; S 26.22. C₁₀H₁₃NO₂S₂. Calculated, %: N 5.76; S 26.35.

3-Hydroxymethyl-5-(1-methylhexylidene)-2-thioxothiazolidin-4-one (VI). Yield 97%, mp 54°C, *R_f* 0.35. Found, %: N 5.41; S 24.70. C₁₁H₁₇NO₂S₂. Calculated, %: N 5.40; S 24.72.

3-Hydroxymethyl-5-(1-methylheptylidene)-2-thioxothiazolidin-4-one (VII). Yield 98%, mp 40°C, *R_f* 0.47. Found, %: N 5.13; S 23.41. C₁₂H₁₉NO₂S₂. Calculated, %: N 5.12; S 23.45.

3-Hydroxymethyl-5-(α -methylbenzylidene)-2-thioxothiazolidin-4-one (VIII). Yield 97%, mp 127°C, *R_f* 0.45. Found, %: N 5.52; S 23.88. C₁₂H₉NO₂S₂. Calculated, %: N 5.28; S 24.17.

5-Benzylidene-3-hydroxymethyl-2-thioxothiazolidin-4-one (IX). Yield 98%, mp 153°C, *R_f* 0.18. Found, %: N 5.29; S 25.24. C₁₁H₉NO₂S₂. Calculated, %: N 5.57; S 25.52.

3-Hydroxymethyl-5-(*p*-methylbenzylidene)-2-thioxothiazolidin-4-one (X). Yield 98%, mp 162°C, *R_f* 0.18. Found, %: N 5.19; S 23.78. C₁₂H₁₁NO₂S₂. Calculated, %: N 5.28; S 24.19.

ACKNOWLEDGMENTS

This study was financially supported by the Ministry of Education of the Russian Federation and by the St. Petersburg Authorities (project for young candidates of sciences, no. PD 02-1.3-359).

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