Semicarbazone and Thiosemicarbazone of 5-acetyl-3-(methylsulfanyl)-1,2,4-triazine

Mariusz Mojzych

Institute of Chemistry, University of Podlasie, ul. 3 Maja 54, 08-110 Siedlce, Poland
e-mail: mojzych@ap.siedlce.pl

Received: 11 May 2004 / Accepted: 30 May 2004 / Published: 1 October 2005

Keywords: semicarbazone, thiosemicarbazone, 5-acetyl-1,2,4-triazine.

Semicarbazone as well as thiosemicarbazone of 5-acetyl-3-(methylsulfanyl)-1,2,4-triazine were synthesised as reactive intermediates for the synthesis of 1H-pyrazolo[4,3-e][1,2,4]triazine derivatives via acid promoted ring closure [1-3].

Starting ketone 1 was prepared according to a reported procedure [4]. To a solution of ketone 1 (169 mg, 1 mmol) and semicarbazone or thiosemicarbazone (1.2 mmol) in ethanol (20 ml) 10% HCl (0.5 ml) was added. The resulting reaction mixture was heated at reflux for 5 min and then stirred at room temperature for additional 30 min. The precipitated solid was filtered off, recrystallized from dioxane and dried under vacuum.

**Semicarbazone of 5-acetyl-3-(methylsulfanyl)-1,2,4-triazine (2)**

Yield 89%.

Melting Point: 260 °C.

$^1$H-NMR (200 MHz, DMSO-$d_6$): $\delta = 2.19$ (s, 3H); 2.62 (s, 3H); 6.96 (s, 2H, NH$_2$); 9.98 (s, 1H); 10.13 (s, 1H, NH).

IR (CHCl$_3$ film, cm$^{-1}$): 3339; 3106; 1693; 1520; 1486; 1426; 1312; 1256; 1183; 1133; 1058; 871; 713.

MS- EI (m/z, %): 226 (18) [M$^+$]; 209 (7); 184 (11); 183 (100); 182 (62); 155 (12); 154 (39); 140 (32); 82 (49); 81 (30); 74 (18); 53 (19).

HR-MS: Calculated for C$_7$H$_{10}$N$_6$OS: 226.0636. Found: 226.06314.

**Thiosemicarbazone of 5-acetyl-3-(methylsulfanyl)-1,2,4-triazine (3)**

Yield 87%.
Melting Point: 229°C.

IR (CHCl₃ film, cm⁻¹): 3304; 3158; 2969; 1587; 1495; 1423; 1243; 1127; 1057; 876.

¹H-NMR (200 MHz, DMSO-d₆): δ= 2.31 (s, 3H); 2.63 (s, 3H); 8.53 (s, 1H); 8.70 (s, 1H); 10.08 (s, 1H); 10.81 (s, 1H).

MS-EI (m/z, %): 242 (80) [M⁺]; 199 (21); 167 (7); 154 (6); 141 (5); 140 (22); 128 (27); 116 (100); 82 (12); 81 (12); 74 (14); 60 (24); 53 (13).

HR-MS: Calculated for C₇H₁₀N₆S₂: 242.04084; Found: 242.04129.

References:

Sample Availability: Available from MDPI.

© 2005 MDPI. All rights reserved.