Bis[(3-methoxycarbonyl-5-methyl pyrazol)-1-yl Thiocarbonyl] Disulfide

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This experiment is performed according to literature method [1-4]. 3(5)-Methoxycarbonyl-5(3)-pyrazole 1 (3.35 g, 0.024 mol) and triethylamine (6,65 g, 0.048 mol) in ethanol solution were cooled to 5°C under stirring, carbon disulfide (3,65 g, 0.048 mol) was added to the solution. After 1 hour of stirring, solid iodine (2,8 g, 0.022 mol) was added in portions and stirred until the colour disappeared completely. Then a methanolic solution of iodine was added dropwise until a faint colour persists. Excess of iodine was neutralized with Na$_2$S$_2$O$_3$ solution. The product was extracted with diethyl ether, washed thrice with water, dried over Na$_2$SO$_4$, filtered, and diethylether was evaporated at room temperature to give liquid compound 2.

Yield: (90%).

$^1$H-NMR(CDCl$_3$) d (ppm): 2,30 (s, 6H, CH$_3$); 4,86 (s, 6H, CO$_2$CH$_3$); 6,53 (s, 2H, Hpy).

$^{13}$C-NMR(CDCl$_3$) d (ppm): 166 (CO$_2$), 126 (C3), 52 (O-CH$_3$)

IR (KBr , cm$^{-1}$): 3200 (-S-S-); 1720 (C=O); 1240 (C=S), 166 (CO$_2$), 126 (C$_3$), 52 (O-CH$_3$).

MS (m/z): 430 [M]$^+$.  

U.V.: $l_{max}$ = 290 nm (-C=S).

References


Sample Availability: Available from the authors and from MDPI.

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