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Ethyl-(*R*,*S*)-5-acetyl-4,5-dihydro-3-isoxazole Acetate

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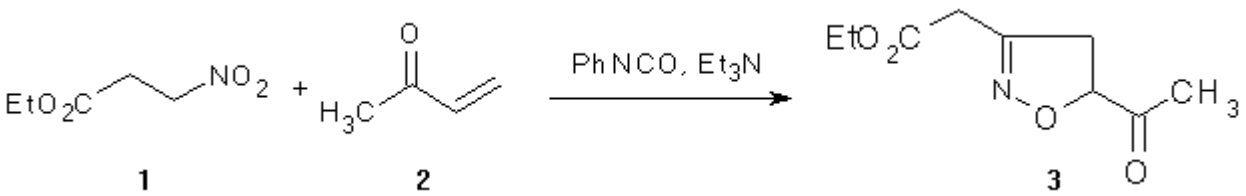

The isoxazoline **3** was prepared by the [3+2] cycloaddition of the nitroderivative **1** [1] with methyl vinyl ketone **2** under *Mukaiyama* conditions [2].

Phenyl isocyanate (2.5 ml, 23 mmol) was slowly added (4 h) to a mixture of ethyl-nitropropionate **1** (1.46 g, 10 mmol) and methyl vinyl ketone **2** (1.6 ml, 20 mmol) in dry benzene (60 ml) containing a few drops of Et_3N. The mixture was stirred at room temperature for two days. The suspension was filtered to eliminate the precipitate of diphenylurea and to the filtrate was added H_2O (100 ml). The resulting biphasic system was stirred for two hours at room temperature. After separation, the organic phase was dried and concentrated in vacuo. The crude residue was purified by flash chromatography (ether/light petroleum = 7:3) to give the desired compound **3** as a yellow oil (1 g, 50%).

TLC (ether/light petroleum = 6:4) R_f 0.42

IR (neat, cm^{-1}): 2985, 1737, 1722, 1599, 1555, 1503, 1444, 1400, 1381, 1260, 1203, 1096, 1027.

^{1}HNMR ($CDCl_3$) d: 4.9 (dd, 1H, J = 9.4 Hz, 4.2 Hz, CH_2CHO); 4.18 (q, 2H, J = 7.2 Hz, $CO_2CH_2CH_3$); 3.07 (dd, 1H, J = 17.2 Hz, J = 9.4 Hz, CH*H*CHO); 2.76 (dd, 1H, J = 17.2 Hz, 4.2 Hz, C*H*HCHO); 2.54 (d, 2H, J = 7 Hz, CH_2CO_2); 2.17 (s, 3H, $COCH_3$); 1.25 (t, 3H, J = 7.2 Hz, $CO_2CH_2CH_3$)

Anal. calc. for $C_9H_{13}NO_4$ (199.20): C 54.26, H 6.58, N 7.03; found: C 54.61, H 6.65, N 7.10

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Sample availability: Available from the authors.