

(Z)-2-Methyl-3-(1-phenyl-ethylamino)-but-2-enoic acid ethyl ester

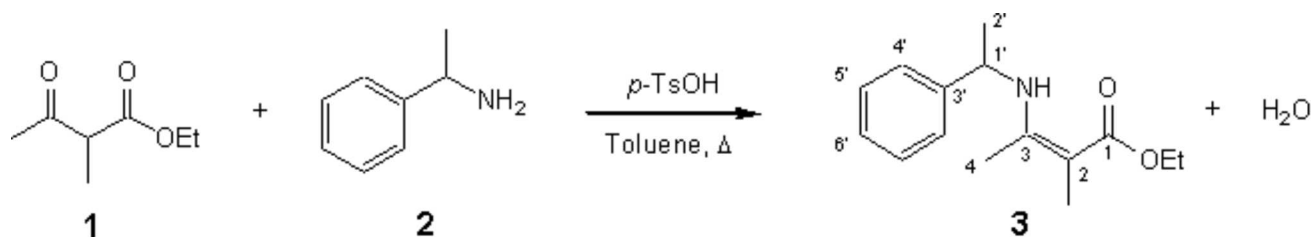
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p-Toluenesulfonic acid (48 mg, 0.25 mmol) was added to a stirred mixture of ethyl 2-methyl-acetoacetate (**1**) (346 mg, 2.40 mmol) and α -methylbenzylamine (**2**) (358 mg, 2.95 mmol) in toluene (13 mL) [1]. Then a Dean-Stark trap device was fit and the reaction refluxed overnight. The mixture was allowed to reach room temperature and percolated over a silica gel pad and the solvent evaporated under reduced pressure to yield the title compound **3** (540 mg, 2.32 mmol, 96%).

IR (neat, cm^{-1}): 3250 (N-H), 3062, 3027, 780, 702 (Ph), 1646, 1250, 1121 (COOEt), 1598 (C=C-N).

^1H NMR (300 MHz, CDCl_3): δ = 1.30 (3H, *t*, J =7.1 Hz, $\text{CH}_3\text{CH}_2\text{O}$); 1.50 (3H, *d*, J =6.9 Hz, H-2'); 1.75* (3H, *s*, Me-2); 1.78* (3H, *s*, H-4); 4.16 (2H, *dq*, J =7.1, 1.0 Hz, $\text{CH}_3\text{CH}_2\text{O}$); 4.64 (1H, *quint*, J =6.9 Hz, H-1'); 7.18–7.35 (5H, *m*, Ph); 9.66 (1H, *br d*, J =6.9 Hz, NH).

*These signals may be interchanged.

^{13}C NMR (75 MHz, CDCl_3): δ = 171.10 (C-1); 87.70 (C-2); 159.00 (C-3); 15.75 (C-4); 12.54 (Me-2); 14.63 ($\text{CH}_3\text{CH}_2\text{O}$); 58.73 ($\text{CH}_3\text{CH}_2\text{O}$); 53.17 (C-1'); 25.17 (C-2'); 145.75 (C-3'); 125.44 (C-4', C-8'); 128.65 (C-5', C-7'); 126.80 (C-6').

EI-MS (70 eV, m/z): 248 ($\text{M}^+\text{+H}$, 7%); 247 (M^+ , 14); 232 ($\text{M}^+\text{--Me}$, 2); 218 ($\text{M}^+\text{--Et}$, 0.7); 202 ($\text{M}^+\text{--OEt}$, 3); 186 (4); 174 ($\text{M}^+\text{--COOEt}$, 5); 145 (15); 105 (C_8H_9^+ , 100); 79 (10); 77 (10); 42 (13).

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References

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