2,2,5-Trimethylhexa-3,4-dienal Azine

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The homoallenylaldehyde 1 was prepared according to the literature [1,2]. Half an equivalent of hydrazine monohydrate [3] was added dropwise to a solution of 1 with a catalytic amount of p-TSA in diethylether [4]. The mixture was stirred at r.t. for 2 h, evaporated under reduced pressure to remove the solvent. The product was purified by column chromatography on silica gel using CH\(_2\)Cl\(_2\) to give the title compound (2). Liquid, 38% yield.

\(n_D^{20} = 1.4910\).

TLC (CHCl\(_3\)): \(R_f = 0.52\).

MS (EI): 257 ([M-CH\(_3\)]\(^+\)), 163, 148, 136, 122, 79, 67

IR (neat): 740 (s), 810 (s), 900 (w), 1010 (w), 1100 (w), 1195 (w), 1255 (m), 1370 (w), 1460 (w), 1630 (C=\(\equiv\)C), 1950 (C=C=C), 2860 (w), 2920 (w), 2960 (s).

\(^1\)H NMR (acetone-d\(_6\)): 1.14 (12H, s, (CH\(_3\))\(_2\)C), 1.63 (12H, d, (CH\(_3\))\(_2\)C=C=C, \(^5\)J=2.8 Hz), 4.89-4.97 (2H, sept, HC=C=C, \(^5\)J=2.8 Hz), 7.50 (2H, s, HC=N).

\(^13\)C NMR (acetone-d\(_6\)): 20.66 (C6, C7), 25.51 (C8, C9), 38.93 (C2), 96.38 (C3), 98.02 (C5), 168.31 (C1), 200.61 (C4).

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References


Sample Availability: Available from the author.