α-Nitro Ketone Synthesis Using *N*-Acylbenzotriazoles

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SUPPORTING INFORMATION

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Experimental Section

Melting points were uncorrected. ^{1}H (300 MHz) and ^{13}C (75 MHz) NMR spectra were recorded on a 300 MHz NMR spectrometer in $CDCl_3$ unless stated. Column chromatography was performed on silica gel 200–425 mesh. THF was distilled from sodium-benzophenone ketyl and DMSO was dried over molecular sieves prior to use. *N*-acylbenzotriazoles were prepared according to literature procedures.[32]

General procedure for the preparation of α-Nitro Ketones 5a-p: A mixture of the appropriate nitroalkane **3** (2 mmol) and potassium *t*-butoxide (0.49 g, 4.4 mmol) in DMSO (10 mL) was stirred for 10 min. while the temperature was maintained at 10 ^{o}C. *N*-Acylbenzotriazole **2** (2 mmol) in DMSO (10 mL) was added dropwise to the resulting solution, and the mixture was stirred for 2 hrs at 10 ^{o}C and then at room temperature for 6 hrs. The mixture was poured into water (40 mL), acidified with acetic acid 10%, and then extracted with ethyl acetate (3x30 mL). The extracts were washed with water, dried over Na_2SO_4 and the solvent removed under reduced pressure. The residue was placed in a silica-gel column and eluted with hexanes/EtOAc 10:1 to give α-nitro ketones **5**.

Ethyl 5-methyl-2-nitro-3-oxohexanoate (5b): Ethyl nitroacetate (**3d**) (0.266 g, 2 mmol) was reacted with 1-benzotriazole-1-yl-3-methyl-butan-1-one (**2a**) (0.406 g, 2 mmol) according to the general procedure. Purification by column chromatography gave 0.313 g (72%) as colorless oil. ^{1}H NMR [keto + enol] δ 5.85 (s, 1H), 4.37 (q, *J* = 7.1 Hz, 2H), 2.57–2.54 (m, 1H), 2.38 (d, *J* = 7.1 Hz, 1H), 2.28–2.11 (m, 1H), 1.35 (t, *J* = 7.1 Hz, 3H), 0.96 (d, *J* = 6.7 Hz, 3H), 0.98 (d, *J* = 6.7 Hz, 3H). ^{13}C NMR [Keto + enol] δ 192.8, 178.1, 160.0, 93.4, 63.9, 62.8, 49.2, 40.7, 26.7, 24.2, 22.2, 22.1, 13.9, 13.7, (two carbons were

not observed). Anal. Calcd. For $C_9H_{15}NO_5$: C, 49.76; H, 6.96; N, 6.45. Found: C, 50.20; H, 7.14; N, 6.45.

2-Nitro-4-phenyl-3-butyn-2-one (5c): Nitromethane (**3a**) (0.122 g, 2 mmol) was reacted with 1-benzotriazol-1-yl)-3-phenyl-2-propyn-1-one (**2b**) (0.495 g, 2 mmol) according to the general procedure. Purification by column chromatography gave 0.182 g (48%) as lightly yellow microcrystals, mp 71–72 °C (lit.[34] 72.0–72.5 ^{0}C). ^{1}H NMR δ 7.63–7.57 (m, 2H), 7.57–7.52 (m, 1H), 7.46–7.41 (m, 2H), 5.42 (s, 2H). ^{13}C NMR δ 172.0, 133.6, 132.1, 128.9, 118.3, 97.7, 84.4, 84.1. Anal. Calcd. For $C_{10}H_7NO_3$: C, 63.49; H, 3.73; N, 7.40. Found: 63.49; H, 3.61; N, 7.26.

1-Nitro-1-phenyl-1-ethanone (5d): Nitromethane (**3a**) (0.122 g, 2 mmol) was reacted with benzotriazol-1-yl(phenyl)methanone (**2c**) (0.446 g, 2 mmol) according to the general procedure. Purification by column chromatography gave 0.267 g (81%) as colorless prisms, mp 103–105 ^{o}C (lit.[26a] 104–106 ^{o}C). ^{1}H NMR (DMSO-d_6) δ 7.97–7.94 (m, 2H), 7.78–7.73 (m, 1H), 7.63–7.58 (m, 2H), 6.56 (s, 3H). ^{13}C NMR (DMSO-d_6) δ 188.4, 134.9, 133.4, 129.1, 128.5, 82.8. Anal. Calcd. For $C_8H_7NO_3$: C, 58.18; H, 4.27; N, 8.48. Found: 58.04; H, 4.18; N, 8.42.

1–(3-Chlorophenyl)-2-nitro-1-ethanone (5e): Nitromethane (**3a**) (0.122 g, 2 mmol) was reacted with benzotriazol-1-yl(3-chlorophenyl)methanone (**2e**) (0.515 g, 2 mmol) according to the general procedure. Purification by column chromatography gave 0.331 g (83%) as colorless plates, mp 103–105 °C (lit.[26a] 164–166 ^{o}C). ^{1}H NMR (DMSO-d_6) δ 7.99 (t, J = 1.8 Hz, 1H), 7.91 (ddd, J = 7.9, 1.8, 0.6 Hz, 1H), 7.83 (ddd, J = 7.9, 1.8, 0.6 Hz, 1H), 7.65 (dt, J = 7.9 , 1.8, 0.6 Hz, 1H), 6.57 (s, 3H). ^{13}C NMR (DMSO-d_6) δ 187.7,

135.4, 134.6, 134.1, 131.2, 128.4, 127.2, 83.0. Anal. Calcd. For $C_8H_6ClNO_3$: C, 48.14; H, 3.03; N, 7.02. Found: C, 48.34; H, 2.92; N, 6.89.

1−(4-Chlorophenyl)-2-nitro-1-propanone (5f): Nitroethane (**3b**) (0.150 g, 2 mmol) was reacted with benzotriazol-1-yl(4-chlorophenyl)methanone (**2f**) (0.515 g, 2 mmol) according to the general procedure. Purification by column chromatography gave 0.265 g (62%) as colorless needles, mp 73–74 °C. ^{1}H NMR δ 7.90 (d, *J* = 8.7 Hz, 2H), 7.52 (d, *J* = 8.7 Hz, 2H), 6.11 (q, *J* = 7.0 Hz, 1H), 1.85 (d, *J* = 7.0 Hz, 3H). ^{13}C NMR δ 188.4, 141.5, 131.9, 130.1, 129.6, 84.6, 15.9. Anal. Calcd, For $C_9H_8ClNO_3$: C, 50.60; H, 3.77; N, 6.56. Found: C, 50.80; H, 3.58; N, 6.52.

2-Nitro-1-(*p*-tolyl)-1-butanone (5g): 1-Nitropropane (**3c**) (0.178 g, 2 mmol) was reacted with benzotriazol-1-yl(4-methylphenyl)methanone (**2d**) (0.475 g, 2 mmol) according to the general procedure. Purification by column chromatography gave 0.236 g (57%) as colorless oil. ^{1}H NMR δ 7.85 (d, *J* = 8.2 Hz, 2H), 7.32 (d, *J* = 8.1 Hz, 2H), 6.01 (dd, *J* = 9.2, 4.8 Hz, 1H), 2.43 (s, 3H), 2.43–2.30 (m, 1H), 2.26–2.14 (m, 1H), 1.08 (t, *J* = 7.4 Hz, 3H).^{13}C NMR δ 188.6, 145.9, 131.4, 129.8, 128.8, 91.0, 24.2, 21.7, 10.4. Anal. Calcd. For $C_{11}H_{13}NO_3$: C, 63.76; H, 6.32. Found: C, 63.61; H, 6.30.

1-(4-Chlorophenyl)-2-nitro-1-butanone (5h): 1-Nitropropane (**3c**) (0.178 g, 2 mmol) was reacted with benzotriazol-1-yl(4-chlorophenyl)methanone (**2f**) (0.515 g, 2 mmol) according to the general procedure. Purification by column chromatography gave 0.332 g (73%) as colorless plates, mp 68–70 ^{o}C. ^{1}H NMR δ 7.90 (d, *J* = 8.5, Hz, 2H), 7.51 (d, *J* = 8.5 Hz, 2H), 5.94 (dd, *J* = 9.2, 5.9 Hz, 1H), 2.49–2.13 (m, 2H), 1.08 (t, *J* = 7.4 Hz, 3H).^{13}C NMR δ 187.7, 141.4, 132.3, 130.1, 129.6, 90.9, 24.1, 10.4. Anal. Calcd. For $C_{10}H_{10}ClNO_3$: C, 52.76; H, 4.43; N, 6.15. Found: C, 52.96; H, 4.34; N, 6.05.

1-(4-Fluorophenyl)-2-nitro-1-butanone (5i): 1-Nitropropane (**3c**) (0.178 g, 2 mmol) was reacted with benzotriazol-1-yl(4-fluorophenyl)methanone (**2g**) (0.482 g, 2 mmol) according to the general procedure. Purification by column chromatography gave 0.321 g (76%) as a colorless oil. ^{1}H NMRR δ 8.04–7.99 (m, 2H), 7.24–7.17 (m, 2H), 6.01 (dd, *J* = 9.1, 4.8 Hz, 1H), 2.45–2.16 (m, 2H), 1.12 (dt, *J* = 7.4 Hz, 3H). ^{13}C NMR δ 187.5, 166.5 (d, *J* = 258.8 Hz), 131.5 (d, *J* = 9.7 Hz), 130.3, (d, *J* = 3.5 Hz), 116.4 (d, *J* = 22.3), 90.9, 24.1, 10.3. Anal. Calcd. For $C_{10}H_{10}FNO_3$: C, 56.87; H, 4.77; N, 6.63. Found: C, 57.27; H, 4.89; N, 6.86.

2-Nitro-1-(4-nitrophenyl)-1-butanone (5j): 1-Nitropropane (**3c**) (0.178 g, 2 mmol) was reacted with benzotriazol-1-yl(4-nitrophenyl)methanone (**2h**) (0.536 g, 2 mmol) according to the general procedure. Purification by column chromatography gave 0.372 g (78%) as yellow needles, mp 97–98 ^{o}C. ^{1}H NMR δ 8.38 (d, *J* = 8.7 Hz, 2H), 8.14 (d, *J* = 8.7 Hz, 2H), 5.98 (dd, *J* = 8.9, 5.1 Hz, 1H), 2.47–2.21 (m, 2H), 1.12 (t, *J* = 7.4 Hz, 3H). ^{13}C NMR δ 187.6, 151.0, 138.5, 129.8, 124.3, 91.1, 23.9, 10.3. Anal. Calcd. For $C_{10}H_{10}N_2O_5$: C, 50.42; H, 4.23; N, 11.76. Found: C, 50.53; H, 4.06; N, 11.53.

2-Nitro-1-(2-thienyl)-1-ethanone (5k): Nitromethane (**3a**) (0.122 g, 2 mmol) was reacted with benzotriazol-1-yl(2-thienyl)methanone (**2i**) (0.458 g, 2 mmol) according to the general procedure. Purification by column chromatography gave 0.291 g (85%) as colorless plates, mp 107–109 ^{o}C. ^{1}H NMR (DMSO-d_6) δ8.22 d, *J* = 4.8 Hz, 1H), 8.04 (d, *J* = 3.6 Hz, 1H), 7.35 (dd, *J* = 4.8, 4.3 Hz, 1H), 6.43 (s, 2H). ^{13}C NMR (DMSO-d_6) δ 181.0, 139.6, 137.6, 135.8, 129.3, 81.8. Anal. Calcd. For $C_6H_5NO_3S$: C, 42.10; H, 2.94; N, 8.18. Found: C, 42.15; H, 2.76; N, 8.05.

1-(2-Furyl)-2-nitro-1-ethanone (5l): Nitromethane (**3a**) (0.122 g, 2 mmol) was reacted with benzotriazol-1-yl(2-furyl)methanone (**2j**) (0.426 g, 2 mmol) according to the general procedure. Purification by column chromatography gave 0.245 g (79%) as pale yellow plates, mp 73–75 ^{o}C. ^{1}H NMR δ 7.68 (dd, *J* = 1.6, 0.7 Hz, 1H), 7.43 (dd, *J* = 3.7, 0.5 Hz, 1H), 6.67 (dd, *J* = 3.7, 1.6 Hz, 1H), 5.72 (s, 2H). ^{13}C NMR δ 174.4, 149.6, 148.2, 119.9, 113.4, 80.1. Anal. Calcd. For $C_6H_5NO_4$: C, 46.46; H, 3.25; N, 9.03. Found: C, 46.37; H, 3.11; N, 8.87.

1-(2-Furyl)-2-nitro-1-butanone (5m): 1-Nitropropane (**3c**) (0.178 g, 2 mmol) was reacted with benzotriazol-1-yl(2-furyl)methanone (**2j**) (0.426 g, 2 mmol) according to the general procedure. Purification by column chromatography gave 0.315 g (86%) as a pale yellow oil. ^{1}H NMR δ 7.69 (dd, *J* = 1.6, 0.8 Hz, 1H), 7.43 (dd, *J* = 3.7, 0.8 Hz, 1H), 6.65 (dd, *J* = 3.7, 1.7 Hz, 1H), 5.78 (dd, *J* = 8.7, 5.5 Hz, 1H), 2.46–2.17 (m, 2H), 1.08 (t, *J* = 7.4 Hz, 3H). ^{13}C NMR δ 177.3, 150.2, 148.1, 120.3, 11.3, 90.8, 23.6, 10.2. Anal. Calcd. For $C_8H_9N_2O_4$: C, 52.46; H, 4.95. Found: C, 52.78; H, 4.93.

Benzyl *N*-(1-benzyl-3-nitro-2-oxopropyl)carbamate (5n): Nitromethane (**3a**) (0.122 g, 2 mmol) was reacted with benzyl *N*-(2-benzotriazol-1-yl)-1-benzyl-2-oxoethyl]carbamate (**2k**) (0.801 g, 2 mmol) according to the general procedure. Purification by column chromatography gave 0.404 g (59%) as colorless plates, mp 107–108 ^{o}C. ^{1}H NMR δ 7.36–7.28 (m, 8H), 7.14–7.12 (m, 2H), 5.30 (d, AB system, *J* = 15.4 Hz, 1H), 5.25 (d, *J* = 6.8 Hz, 1H), 5.09 (d, AB system, *J* = 15.4Hz, 1H), 5.08 (s, 2H), 4.51 (q, *J* = 7.0 Hz, 1H), 3.17–3.01 (m, 2H) ^{13}C NMR δ 195.9, 156.0, 135.6, 134.7, 129.2, 129.1, 128.6, 128.5, 128.2, 127.7, 81.9, 67.7, 59.8, 36.6. Anal. Calcd. For $C_{18}H_{18}N_2O_5$: C, 63.15; H, 5.30; N, 8.18. Found: C, 63.25; H, 5.36; N, 8.11.

Benzyl *N*-(1-benzyl-3-nitro-2-oxobutyl)carbamate (5o): Nitroethane (**3b**) (0.150 g, 2 mmol) was reacted with benzyl *N*-(2-benzotriazol-1-yl)-1-benzyl-2-oxoethyl]carbamate (**2k**) (0.801 g, 2 mmol) according to the general procedure. Purification by column chromatography gave 0.278 g (39%) as colorless needles, mp 58–59 ^{o}C. ^{1}H NMR [2 stereoisomers in 2:1 ratio] δ 7.36–7.28 (m, 8H), 7.16–7.14 (m, 2H), 5.42–5.31 (m, 1H), 5.16 (d, *J* = 8.8 Hz, 1H), 5.08 (s, 2H), 4.77–4.64 (m, 1H), 3.24–3.18 (m, 1H), 3.04–2.97 (m, 1H), 1.70 (d, *J* = 7.0 Hz, 1H), 1.57 (d, *J* = 6.7 Hz, 2H). ^{13}C NMR δ 198.5, 155.9, 135.7, 135.2, 135.1, 129.2, 129.0, 128.6, 128.4, 128.1, 127.5, 86.2, 85.6, 67.6, 67.5, 59.4, 59.2, 37.1, 36.6, 14.9, 14.5. Anal. Calcd. For $C_{19}H_{20}N_2O_5$: C, 64.04; H, 5.66; N, 7.86. Found: C, 64.33; H, 5.82; N, 7.80.

Benzyl *N*-{1-[2-(methylsulfanyl)ethyl]-3-nitro-2-oxopropyl}carbamate (5p) /benzotriazole (5:1): Nitromethane (**3a**) (0.122 g, 2 mmol) was reacted with benzyl *N*-(1-benzotriazol-1-ylcarbonyl)-3-(methylsulfanyl)propyl]carbamate (**2l**) (0.768 g, 2 mmol) according to the general procedure. Purification by column chromatography gave 0.463 g (71%) as colorless needles, mp 78–80 ^{o}C. ^{1}H NMR δ 7.36 (s, 5H), 5.58 (br d, *J* = 4.1 Hz, 1H), 5.56 (d, AB system, *J* = 15.1 Hz, 1H), 5.47 (d, AB system, *J* = 15.1 Hz, 1H), 5.14 (s, 2H), 4.57–4.50 (m, 1H), 2.60–2.54 (m, 2H), 2.27–2.18 (m, 1H), 2.10–1.98 (m, 4H). ^{13}C NMR δ 195.6, 156.2, 135.5, 128.7, 128.2, 126.1, 81.6, 67.8, 57.8, 29.8, 29.2, 15.3. Anal. Calcd. For $C_{14}H_{18}N_2O_5S.C_6H_5N_3$: C, 52.13; H, 5.47; N, 10.40. Found: C, 52.25; H, 5.47; N, 10.35.

General procedure for the preparation of (E)-1-(2-nitro-1-butenyl)-1*H*-benzotriazoles 6a–c: To a solution of **3c** in dry THF (5 mL), 1.1 equivalent of *n*-BuLi (1.4 mL, 1.6 M in pentane, 2.2 mmol) was added at –78 ^{o}C. The solution was stirred at -

78 ^{o}C for 1 h, and a solution of 2 (2 mmol) in THF (10 mL) was added. The reaction mixture was stirred for 10 h while the temperature was allowed to rise to 20 ^{o}C. After quenching with water (5 mL) and extraction with EtOAc (3 x 20 mL), the combined organic layers were washed with water (25 mL), dried over $MgSO_4$ and the solvent was removed in *vacuo*. The resultant oil was subjected to column chromatography (eluent: ethyl acetate/ hexanes = 1: 10 then 1: 5) to give the pure product.

1-[(E)-2-Nitro-1-phenyl-but-1-en-1-yl]-1H-benzotriazole (6b): 1-Nitropropane (**3c**) (0.178 g, 2 mmol) was reacted with benzotriazol-1-yl(phenyl)methanone (**2c**) (0.446 g, 2 mmol) according to the general procedure. Purification by column chromatography gave 0.353 g (60%) as colorless needles, mp 89–91 ^{o}C. ^{1}H NMR δ 8.59 (d, *J* = 8.4 Hz, 1H), 8.18–8.12 (m, 3H), 7.72–7.65 (m, 2H), 7.58–7.49 (m, 3H), 3.59 (q, *J* = 7.6 Hz, 2H), 1.53 (t, *J* = 7.6 Hz, 3H). ^{13}C NMR δ 163.1, 162.7, 146.5, 133.8, 130.9, 130.0, 129.7, 128.8, 128.3, 125.8, 119.9, 115.6, 21.0, 11.3. Anal. Calcd. For $C_{16}H_{14}N_4O_2$: C, 65.29; H, 4.79; N, 19.04. Found: C, 65.33; H, 4.90; N, 19.10.

1-[(E)-1-(3-Chlorophenyl)-2-nitro-but-1-en-1-yl]-1H-benzotriazole (6c): 1-Nitro-propane (**3c**) (0.178 g, 2 mmol) was reacted with benzotriazol-1-yl(3-chlorophenyl)methanone (**2e**) (0.515 g, 2 mmol) according to the general procedure. Purification by column chromatography gave 0.427 g (65%) as colorless plates, mp 148–150 ^{o}C. ^{1}H NMR δ 8.55 (d, *J* = 8.4 Hz, 1H), 8.15–8.03 (m, 3H), 7.72–7.63 (m, 2H), 7.54–7.47 (m, 3H), 3.58 (q, *J* = 7.7 Hz, 2H), 1.53 (t, *J* = 7.7 Hz, 3H). ^{13}C NMR δ 163.0, 162.0, 146.5, 135.0, 133.8, 130.8, 130.1, 129.9, 129.6, 127.7, 125.8, 119.9, 115.5, 21.0, 11.3. Anal. Calcd. For $C_{16}H_{13}ClN_4O_2$: C, 58.45; H, 3.99; N, 17.04. Found: C, 58.41; H, 3.97; N, 17.02.

General procedure for the preparation of *O*-acyl oximes 9a–d: To a stirred solution of secondary nitroalkane **7** (6 mmol) in dry THF (40 mL), *n*-BuLi (5.12 mL, 1.56 M in pentane, 8 mmol) was added at –78 ^{o}C. The solution was stirred at -78 ^{o}C for 1 h. Then the solution of **2** (4 mmol) in THF (10 mL) was added. The reaction mixture was allowed to warm up to room temperature, and stirred for 2h. After quenching with NH_4Cl and extraction with ether (3 x 30 mL), the combined organic layers were washed with brine (25 mL), dried over $MgSO_4$ and the solvent was removed in vacuo. The residue was subjected to column chromatography (eluent: ethyl acetate/ hexanes = 1: 19) to give the pure product.

1-[Benzoyloxy)imino]cyclohexane (9b): 1-Nitrocyclohexane (**7b**) (0.775 g, 6 mmol) was reacted with benzotriazol-1-yl(phenyl)methanone (**2c**) (0.893 g, 4 mmol) according to the general procedure. Purification by column chromatography gave 0.235 g (27%) as colorless microcrystals, mp 50–51 ^{o}C (lit.[35] 62–63 ^{o}C). ^{1}H NMR δ 8.08–8.05 (m, 2H), 7.60–7.55 (m, 1H), 7.48– (m, 2H), 2.70–2.65 (m, 2H), 2.49–2.45 (m, 2H), 1.84-1.62 (m, 6H). ^{13}C NMR δ 169.4, 164.1, 133.0, 129.4, 129.2, 128.3, 32.0, 27.0, 26.6, 25.7, 25.3. Anal. Calcd. For $C_{13}H_{15}NO_2$: C, 71.87; H, 6.96; N, 6.45. Found: C, 71.72; H, 7.07; N, 6.51.

1-{[(Cyclohexylideneamino)oxy]carbonyl}-4-methylbenzene (9c): 1-Nitrocyclohexane (**7b**) (0.775 g, 6 mmol) was reacted with benzotriazol-1-yl(4-methylphenyl)methanone (**2d**) (0.949 g, 4 mmol) according to the general procedure. Purification by column chromatography gave 0.185 g (20%) as colorless microcrystals, mp 69–70 ^{o}C. ^{1}H NMR δ 8.02 (d, *J* = 7.4 Hz, 2H), 7.30 (d, *J* = 7.4 Hz, 2H), 2.74–2.70 (m, 2H), 2.54–2.45 (m, 5H), 1.89–1.71 (m, 6H). ^{13}C NMR δ 169.3, 164.3, 144.5, 143.8, 130.2, 129.6, 129.1, 126.6,

32.2, 27.1, 26.7, 25.8, 25.4, 21.6. Anal. Calcd. For $C_{14}H_{17}NO_2$: C, 72.70; H, 7.41; N, 6.06. Found: C, 72.53; H, 7.52; N, 5.77.

2-{[(Cyclohexylideneamino)oxy]carbonyl}furan (9d): 1-Nitrocyclohexane (**7b**) (0.775 g, 6 mmol) was reacted with benzotriazol-1-yl(2-furyl)methanone (**2j**) (0.853 g, 4 mmol) according to the general procedure. Purification by column chromatography gave 0.489 g (59%) as colorless microcrystals, mp 70–71 ^{o}C. ^{1}H NMR δ 7.62 (dd, *J* = 1.7, 0.8 Hz, 1H), 7.24 (dd, *J* = 3.5, 0.8 Hz, 1H), 6.54 (dd, J = 3.5, 1.7 Hz, 1H), 2.68–2.64 (m, 2H), 2.48–2.43 (m, 2H), 1.83–1.63 (m, 6H). ^{13}C NMR δ 169.8, 156.6, 146.5, 143.4, 118.2, 111.8, 32.1, 27.0, 26.7, 25.8, 25.4. Anal. Calcd. For $C_{11}H_{13}NO_3$: C, 63.76; H, 6.32; N, 6.76. Found: C, 63.94; H, 6.65; N, 6.77.

Crystal data for (**9d**): $C_{11}H_{13}NO_3$, MW 207.22, orthorhombic, space group $Pna2_1$, *a* = 18.052(3), *b* = 5.7905(9), *c* = 10.111(2) Å, V = 1056.9(3) Å^3, F(000) = 440, Z = 4, T = -180 ^{o}C, μ (MoKα) = 0.095 mm^{-1}, D_{calcd} = 1.302 g.cm^{-3}, $2\theta_{max}$ 53^{o}, wR(F^2) = 0.0636 (all 2009 data), R = 0.0234 (1922 data with I > 2σI).

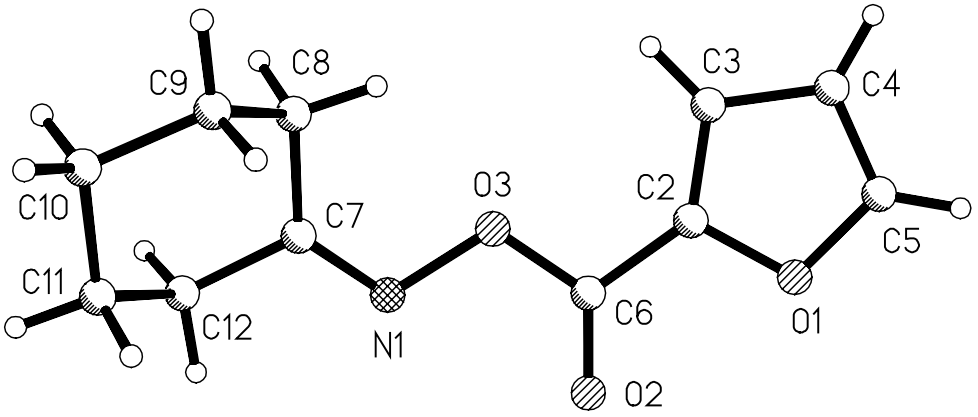

Figure 1. X-ray crystal structure of **9d**. Selected bond lengths (Å): C7 – N1 1.281(2), N1 – O3 1.459(1), O3 – C6 1.360(1), C6 – O2 1.203(1), C6 – C2 1.461(2).