Supporting Information

Isocyanide based multicomponent click reaction: Green and an improved synthesis of 1-substituted 1H-1,2,3,4-tetrazoles

Shrikant G. Pharande, Manuel A. Rentería-Gómez and Rocío Gámez-Montaño*

Departamento de Química, División de Ciencias Naturales y Exactas, Universidad de Guanajuato, Noria S/N, Col. Noria Alta, Guanajuato, C.P. 36050, Gto., México.

e-mail (R.G.-M.): rociogm@ugto.mx

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General information

All reactions were carried out in 10 ml stoppered glass tube. Reactions were monitored by silica gel TLC plates, using mixture of hexane and ethyl acetate as eluents. Melting points were determined on a Fisher-Johns apparatus and were uncorrected. Isocyanides were synthesized using literature procedure\textsuperscript{1} and TMSN\textsubscript{3} was purchased from Sigma Aldrich. \textsuperscript{1}H NMR spectra were recorded on Bruker Advance III spectrometer (500 MHz) at 295 K in CDCl\textsubscript{3}; chemical shifts (\(\delta\) in ppm) and coupling constants (\(J\) in Hz) are reported in standard fashion with reference to either internal standard tetramethylsilane (TMS) (\(\delta_H = 0\) ppm) or CHCl\textsubscript{3} (\(\delta_H = 7.26\) ppm). \textsuperscript{13}C NMR spectra were recorded on Bruker Advance III spectrometer (126 MHz) at room temperature in CDCl\textsubscript{3}; chemical shifts (\(\delta\) in ppm) are reported relative to CHCl\textsubscript{3} (\(\delta_C = 77.00\) ppm). In \textsuperscript{1}H NMR, the following abbreviations were used throughout: s = singlet, d = doublet, dd = doublet of doublet, m = multiplet and bs = broad singlet. The assignment of signals was confirmed by \textsuperscript{1}H and \textsuperscript{13}C spectral data. IR spectra were recorded by ATR method using neat compounds. The wavelengths are reported in reciprocal centimeters (\(\nu_{\text{max}}/\text{cm}^{-1}\)). HRMS spectra were acquired via electrospray ionization ESI (+) and recorded via the TOF method on Bruker mass spectrometer. Ultrasound irradiated reactions were performed in 10 ml stoppered glass tube placed into a water bath of a Branson 1510 sonicator cleaner working at 42 kHz \pm 6\% frequencies.

Reference:

General procedure for the synthesis of 1-substituted 1H-1,2,3,4-tetrazoles (7)

A dry 10 ml tube was charged with an isocyanide (1 mmol), TMSN₃ (2 mmol) and H₂O (2 mmol). The reaction was then sonicated in an ultrasonic cleaner water bath (45 kHz) at room temperature for 1 hr. The resulting crude products were diluted with DCM (2 ml) and filtered through small Na₂SO₄ bed to remove traces of water. The resulting filtrate kept as it is at room temperature for evaporation to afford pure products. In some cases, to remove the traces of isocyanide, wash of 2ml EtOAc: Hex (0.5/9.5 V/V) was given.

Spectral data

1-(4-methoxyphenyl)-1H-tetrazole (7a)

Based on GP, 50 mg 4-methoxy phenyl isocyanide (0.37 mmol), 0.099 cm³ TMSN₃ (0.75 mmol) and 0.013 cm³ H₂O (0.75 mmol) were reacted together to afford 62 mg (94%) as a light brown solid. Melting range 118-119°C, ¹H NMR (500 MHz, CDCl₃) 8.91 (s, 1H), 7.59 (d, J = 10 Hz, 2H), 7.06 (d, J = 10 Hz, 2H), 3.88 (s, 3H); ¹³C NMR (126 MHz, CDCl₃) δ 160.88, 140.72, 127.08, 123.12, 115.34, 55.87; FT-IR (ATR) νmax/cm⁻¹ 3051.0, 2942.8, 1603.1, 1280.5; HRMS (ESI⁺): m/z calcd. for C₈H₉N₄O 177.0770, found 177.0782.

¹H NMR spectra of the compound 7a
1-(3,4-dimethoxyphenyl)-1H-tetrazole (7b)

Based on GP, 50 mg 3,4-dimethoxy phenyl isocyanide (0.30 mmol), 0.081 cm$^3$ TMSN$_3$ (0.61 mmol) and 0.011 cm$^3$ H$_2$O (0.61 mmol) were reacted together to afford 60 mg (95%) as a creamy white solid. Melting range 172-174°C; $^1$H NMR (500 MHz, CDCl$_3$) $\delta$ 8.93 (s, 1H), 7.25 (d, $J$ = 2.5 Hz, 1H), 7.17 (dd, $J$ = 8.6, 2.5 Hz, 1H), 6.99 (d, $J$ = 8.6 Hz, 1H), 3.96 (s, 3H), 3.95 (s, 3H); $^{13}$C NMR (126 MHz, CDCl$_3$) $\delta$ 150.53, 150.31, 140.78, 127.21, 113.57, 111.53, 105.66, 56.50, 56.42; FT-IR (ATR) $\nu_{max}$/cm$^{-1}$ 3040.7, 2944.5, 1594.0, 1283.9; HRMS (ESI$^+$): m/z calcd. for C$_9$H$_{11}$N$_4$O$_2$+ 207.0876, found 207.0881.
1-(3,4,5-trimethoxyphenyl)-1H-tetrazole (7c)

Based on GP, 50 mg 3,4,5-trimethoxy phenyl isocyanide (0.25 mmol), 0.068 cm$^3$ TMSN$_3$ (0.51 mmol) and 0.009 cm$^3$ H$_2$O (0.51 mmol) were reacted together to afford 54 mg (88%) as a light white solid. Melting range 169-171°C; $^1$H NMR (500 MHz, CDCl$_3$) δ 8.96 (s, 1H), 6.89 (s, 2H), 3.94 (s, 6H), 3.90 (s, 3H); $^{13}$C NMR (126 MHz, CDCl$_3$) δ 154.39, 140.81, 139.47, 129.57, 99.39, 61.23, 56.72; FT-IR (ATR) $\nu_{\text{max}}$/cm$^{-1}$ 3050.0, 2941.3, 1601.7, 1282.5; HRMS (ESI$^+$): m/z calcd. for C$_{10}$H$_{13}$N$_4$O$_3$+ 237.0982, found 237.0990.

$^{13}$C NMR spectra of the compound 7b

$^1$H NMR spectra of the compound 7c
1-(2,6-dimethylphenyl)-1H-tetrazole (7d)

Based on GP, 50 mg 2,6-dimethyl phenyl isocyanide (0.38 mmol), 0.101 cm$^3$ TMSN$_3$ (0.76 mmol) and 0.013 cm$^3$ H$_2$O (0.76 mmol) were reacted together to afford 52 mg (78%) as a brown white solid. Melting range 98-100°C; $^1$H NMR (500 MHz, CDCl$_3$) δ 8.69 (s, 1H), 7.38 (d, $J = 7.6$, 1H), 7.23 (d, $J = 7.6$ Hz, 2H), 1.98 (s, 6H); $^{13}$C NMR (126 MHz, CDCl$_3$) δ 143.67, 135.50, 132.43, 131.08, 129.02, 17.57; FT-IR (ATR) $\nu_{\text{max}}$/cm$^{-1}$ 3031.4, 2942.0, 1612.3, 1281.5; HRMS (ESI$^+$): m/z calcd. for C$_9$H$_{11}$N$_4$ 175.0978, found 175.0988.

$^{13}$C NMR spectra of the compound 7c

$^1$H NMR spectra of the compound 7d
1-(4-chlorophenyl)-1H-tetrazole (7e)

Based on GP, 50 mg 4-chloro phenyl isocyanide (0.36 mmol), 0.096 cm³ TMSN₃ (0.72 mmol) and 0.013 cm³ H₂O (0.72 mmol) were reacted together to afford 51 mg (78%) as a brown solid. Melting range 98-100°C; ¹H NMR (500 MHz, CDCl₃) δ 8.99 (s, 1H), 7.67 (d, J = 9 Hz, 1H, 2H), 7.60 – 7.57 (d, J = 9 Hz, 2H); ¹³C NMR (126 MHz, CDCl₃) δ 140.51, 136.27, 132.43, 130.62, 122.60; FT-IR (ATR) υₘₐₓ/cm⁻¹ 3050.4, 2942.1, 1601.0, 1281.5; HRMS (ESI⁺): m/z calcd. for C₇H₆N₄Cl⁺ 181.0276, found 181.0280.

¹H NMR spectra of the compound 7e

¹³C NMR spectra of the compound 7d
Based on GP, 50 mg 2-bromo phenyl isocyanide (0.27 mmol), 0.073 cm³ TMSN₃ (0.54 mmol) and 0.010 cm³ H₂O (0.54 mmol) were reacted together to afford 52 mg (85%) as a light brown solid. Melting range 96-98°C; ¹H NMR (500 MHz, CDCl₃) δ 9.00 (s, 1H), 7.82 (d, J = 8.0 Hz, 1H), 7.57 – 7.53 (m, 2H), 7.51 – 7.45 (m, 1H); ¹³C NMR (126 MHz, CDCl₃) δ 143.45, 143.43, 134.37, 132.35, 129.04, 128.17, 118.77; FT-IR (ATR) νmax/cm⁻¹ 3051.9, 2942.8, 1600.0, 1281.0; HRMS (ESI⁺): m/z calcd. for C₇H₆BrN₄⁺ 224.9770, found 224.9782.
1-phenyl-1H-tetrazole (7g)

Based on GP, 50 mg phenyl isocyanide (0.48 mmol), 0.128 cm$^3$ TMSN$_3$ (0.96 mmol) and 0.017 cm$^3$ H$_2$O (0.96 mmol) were reacted together to afford 57 mg (81%) as a light brown solid. Melting range 64-66°C; $^1$H NMR (500 MHz, CDCl$_3$) δ 9.01 (s, 1H), 7.71 (d, J = 7.6 Hz, 2H), 7.62 - 7.57 (m, 2H), 7.56 - 7.52 (m, 1H); $^{13}$C NMR (126 MHz, CDCl$_3$) δ 140.62, 133.99, 130.36, 130.20, 121.38; FT-IR (ATR) $\nu_{\text{max}}$/cm$^{-1}$ 3057.9, 2921.1, 1603.2, 1263.5; HRMS (ESI+): m/z calcd. for C$_7$H$_7$N$_4^+$ 147.0665, found 147.0670.

$^1$H NMR spectra of the compound 7g
1-benzyl-1H-tetrazole (7h)

Based on GP, 50 mg benzyl isocyanide (0.42 mmol), 0.113 cm$^3$ TMSN$_3$ (0.85 mmol) and 0.015 cm$^3$ H$_2$O (0.85 mmol) were reacted together to afford 66 mg (97%) as a light brown solid. Melting range 59-61°C; $^1$H NMR (500 MHz, CDCl$_3$) δ 8.51 (s, 1H), 7.44 – 7.36 (m, 3H), 7.33 – 7.27 (m, 2H), 5.59 (s, 2H); $^{13}$C NMR (126 MHz, CDCl$_3$) δ 142.58, 133.02, 129.55, 129.50, 128.44, 52.31; FT-IR (ATR) $\nu_{\text{max}}$/cm$^{-1}$ 3053.5, 2943.1, 1601.5, 1281.9; HRMS (ESI$^+$): m/z calcd. for C$_8$H$_9$N$_4^+$ 161.0821, found 161.0827.
1-{4-methoxybenzyl}-1H-tetrazole (7i)

Based on GP, 50 mg 4-methoxy benzyl isocyanide (0.33 mmol), 0.090 cm$^3$ TMSN$_3$ (0.67 mmol) and 0.012 cm$^3$ H$_2$O (0.67 mmol) were reacted together to afford 56 mg (87%) as a light brown solid. Melting range 70-72$^\circ$C; $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 8.39 (s, 1H), 7.19 (d, $J$ = 8.8 Hz, 2H), 6.85 (d, $J$ = 8.6 Hz, 2H), 5.45 (s, 2H), 3.75 (s, 3H); $^{13}$C NMR (101 MHz, CDCl$_3$) $\delta$ 160.53, 142.29, 130.11, 124.75, 114.95, 55.52, 51.99; FT-IR (ATR) $\nu_{\text{max}}$/cm$^{-1}$ 3050.0, 2940.1, 1602.1, 1282.0; HRMS (ESI$^+$): m/z calcd. for C$_9$H$_{11}$N$_4$O$^+$ 191.0927, found 191.0935.
1-(3,4-dimethoxybenzyl)-1H-tetrazole (7j)

Based on GP, 50 mg 3,4-dimethoxy benzyl isocyanide (0.28 mmol), 0.074 cm³ TMSN₃ (0.56 mmol) and 0.010 cm³ H₂O (0.56 mmol) were reacted together to afford 56 mg (90%) as a light brown solid. Melting range 63-65°C; ¹H NMR (500 MHz, CDCl₃) δ 8.43 (s, 1H), 7.28 – 7.07 (m, 1H), 6.40 (d, J = 10.3 Hz, 2H), 5.40 (s, 2H), 3.74 (s, 3H), 3.72 (s, 3H); ¹³C NMR (126 MHz, CDCl₃) δ 162.35, 158.58, 142.62, 131.95, 113.69, 104.87, 99.01, 55.67, 55.63, 47.55; FT-IR (ATR) νmax/cm⁻¹ 3045.4, 2931.7, 1588.2, 1285.4; HRMS (ESI⁺): m/z calcd. for C₁₀H₁₃N₄O₂⁺ 243.0852, found 243.0861.

¹H NMR spectra of the compound 7j
$^{13}$C NMR spectra of the compound 7j

1-(2,3-dimethoxybenzyl)-1H-tetrazole (7k)

Based on GP, 50 mg 2,3-dimethoxy benzyl isocyanide (0.28 mmol), 0.074 cm$^3$ TMSN$_3$ (0.56 mmol) and 0.010 cm$^3$ H$_2$O (0.56 mmol) were reacted together to afford 55 mg (88%) as a light brown solid. Melting range 49-51°C; $^1$H NMR (500 MHz, CDCl$_3$) δ 8.61 (s, 1H), 7.08 – 7.04 (m, 1H), 6.97 (dd, $J = 8.3, 1.3$ Hz, 1H), 6.90 – 6.87 (m, 1H), 5.57 (s, 2H), 3.87 (s, 3H), 3.84 (s, 3H); $^{13}$C NMR (126 MHz, CDCl$_3$) δ 152.91, 147.34, 142.75, 126.61, 124.72, 122.03, 114.21, 61.02, 56.00, 47.53; FT-IR (ATR) $\nu_{\text{max}}$/cm$^{-1}$ 3055.2, 2940.4, 1605.0, 1284.6; HRMS (ESI$^+$): m/z calcd. for C$_{10}$H$_{13}$N$_2$O$_2^+$ 221.1033, found 221.1042.

$^1$H NMR spectra of the compound 7k
Based on GP, 50 mg 2-methoxy benzyl isocyanide (0.33 mmol), 0.090 cm³ TMSN₃ (0.67 mmol) and 0.012 cm³ H₂O (0.67 mmol) were reacted together to afford 54 mg (84%) as a colorless liquid. **¹H NMR** (500 MHz, CDCl₃) δ 8.56 (s, 1H), 7.41 - 7.37 (m, 1H), 7.33 – 7.30 (dd, J = 7.5, 1.6 Hz, 1H), 6.99 (t, J = 7.5 Hz, 1H), 6.93 (d, J = 8.3 Hz, 1H), 5.57 (s, 2H), 3.86 (s, 3H); **¹³C NMR** (126 MHz, CDCl₃) δ 157.44, 142.74, 131.39, 131.02, 121.34, 121.33, 111.11, 55.66, 47.84; **FT-IR (ATR)** νmax/cm⁻¹ 3050.8, 2941.1, 1601.8, 1282.5; **HRMS (ESI⁺)**: m/z calcd. for C₉H₁₁N₄O⁺ 191.0927, found 191.0937.
1-(2,4-dimethoxybenzyl)-1H-tetrazole (7m)

Based on GP, 50 mg 2,4-dimethoxy benzyl isocyanide (0.28 mmol), 0.074 cm$^3$ TMSN$_3$ (0.56 mmol) and 0.010 cm$^3$ H$_2$O (0.56 mmol) were reacted together to afford 55 mg (88%) as a light brown solid. Melting point 119-121°C; $^1$H NMR (500 MHz, CDCl$_3$) δ 8.47 (s, 1H), 6.92 – 6.85 (m, 2H), 6.80 (s, 1H), 5.52 (s, 2H), 3.89 (s, 3H), 3.85 (s, 3H); $^{13}$C NMR (126 MHz, CDCl$_3$) δ 149.84, 142.34, 131.51, 125.07, 121.36, 111.61, 111.37, 56.15, 56.12, 52.34; FT-IR (ATR) $\nu_{\text{max}}$/cm$^{-1}$ 3053.0, 2942.1, 1603.0, 1280.3; HRMS (ESI$^+$): m/z calcd. for C$_{10}$H$_{13}$N$_4$O$_2^+$ 221.1033, found 221.1044.

$^{1}$H NMR spectra of the compound 7m
Based on GP, 50 mg phenethyl isocyanide (0.38 mmol), 0.101 cm$^3$ TMSN$_3$ (0.76 mmol) and 0.013 cm$^3$ H$_2$O (0.76 mmol) were reacted together to afford 63 mg (95%) as a light brown solid. Melting range 67-69°C; $^1$H NMR (500 MHz, CDCl$_3$) δ 8.12 (s, 1H), 7.25 – 7.19 (m, 3H), 6.97 (d, $J = 6.7$ Hz, 2H), 4.61 (t, $J = 6.9$ Hz, 2H), 3.16 (t, $J = 6.9$ Hz, 2H); $^{13}$C NMR (126 MHz, CDCl$_3$) δ 142.68, 136.13, 129.24, 128.73, 127.67, 49.85, 36.27; FT-IR (ATR) $\nu_{\text{max}}$/cm$^{-1}$ 3051.2, 2945.1, 1613.2, 1275.3; HRMS (ESI$^+$): m/z calcd. for C$_9$H$_{11}$N$_4^+$ 175.0978, found 175.0982.
Based on GP, 50 mg 4-methoxy phenethyl isocyanide (0.31 mmol), 0.082 cm³ TMSN₃ (0.62 mmol) and 0.011 cm³ H₂O (0.62 mmol) were reacted together to afford 55 mg (87%) as a light brown solid. Melting range 60-62°C; ¹H NMR (500 MHz, CDCl₃) δ 8.19 (s, 1H), 6.95 – 6.91 (m, 2H), 6.83 – 6.78 (m, 2H), 4.63 (t, J = 6.9 Hz, 2H), 3.76 (s, 3H), 3.15 (t, J = 6.9 Hz, 2H); ¹³C NMR (126 MHz, CDCl₃) δ 159.05, 142.72, 129.77, 128.01, 114.59, 55.38, 50.08, 35.42; FT-IR (ATR) ν max/cm⁻¹ 3042.1, 2940.1, 1590.2, 1290.3; HRMS (ESI⁺): m/z calcd. for C₁₀H₁₃N₄O⁺ 205.1083, found 205.1091.
1-(3-methoxyphenethyl)-1H-tetrazole (7p)

Based on GP, 50 mg 3-methoxy phenethyl isocyanide (0.31 mmol), 0.082 cm³ TMSN₃ (0.62 mmol) and 0.011 cm³ H₂O (0.62 mmol) were reacted together to afford 58 mg (92%) as a light brown solid. Melting range 59-61°C; ¹H NMR (500 MHz, CDCl₃) δ 8.20 (s, 1H), 7.23 – 7.18 (m, 1H), 6.79 (dd, J = 8.3, 2.4 Hz, 1H), 6.62 (d, J = 7.5 Hz, 1H), 6.56 – 6.53 (m, 1H), 4.67 (t, J = 6.9 Hz, 2H), 3.75 (s, 3H), 3.19 (t, J = 6.9 Hz, 2H); ¹³C NMR (126 MHz, CDCl₃) δ 160.08, 142.59, 137.53, 130.16, 120.76, 114.38, 112.80, 55.21, 49.63, 36.15; FT-IR (ATR) νmax/cm⁻¹ 3033.0, 2951.1, 1608.0, 1278.5; HRMS (ESI⁺): m/z calcd. for C₁₀H₁₃N₄O⁺ 205.1083, found 205.1091.
1-(tert-butyl)-1H-tetrazole (7q)

Based on GP, 0.068 cm$^3$ tert. butyl isocyanide (0.60 mmol), 0.159 cm$^3$ TMSN$_3$ (1.20 mmol) and 0.021 cm$^3$ H$_2$O (1.20 mmol) were reacted together to afford 70 mg (92%) as a white solid. Melting range 42-44°C; $^1$H NMR (500 MHz, CDCl$_3$) δ 8.61 (s, 1H), 1.72 (s, 9H); $^{13}$C NMR (126 MHz, CDCl$_3$) δ 139.91, 59.75, 29.93; FT-IR (ATR) ν$\text{max}$/cm$^{-1}$ 1583.0, 1273.3; HRMS (ESI$^+$): m/z calcd. for C$_5$H$_{11}$N$_4^+$ 149.0797, found 149.0802.
Based on GP, 50 mg N,N-diethyl-2-isocyano-3-phenylpropanamide (0.21 mmol), 0.057 cm³ TMSN₃ (0.43 mmol) and 0.008 cm³ H₂O (0.43 mmol) were reacted together to afford 43 mg (71%) as a white solid. Melting range 109-111°C; **¹H NMR** (500 MHz, CDCl₃) δ 9.13 (s, 1H), 7.32 – 7.27 (m, 3H), 7.19 – 7.16 (m, 2H), 5.89 (dd, J = 9.6, 6.0 Hz, 1H), 3.55 (dd, J = 13.0, 9.6 Hz, 1H), 3.47 (dq, J = 14.2, 7.1 Hz, 1H), 3.26 (dd, J = 13.0, 6.0 Hz, 1H), 3.14 (dq, J = 14.2, 7.1 Hz, 1H), 3.07 (q, J = 7.3 Hz, 2H), 1.03 (t, J = 7.1 Hz, 3H), 0.99 (t, J = 7.2 Hz, 3H); **¹³C NMR** (126 MHz, CDCl₃) δ 166.08, 142.24, 134.10, 129.41, 129.15, 128.11, 59.34, 42.54, 41.64, 40.76, 14.55, 12.73; **FT-IR** (ATR) ν max/cm⁻¹ 3030.4, 2938.1, 1660.9, 1597.0, 1289.2; **HRMS** (ESI⁺): m/z calcd. for C₁₄H₂₀N₅O⁺ 274.1662, found 274.1671.
Based on GP, 50 mg 2-isocyno-3-phenyl-1-(pyrrolidin-1-yl)propan-1-one (0.22 mmol), 0.058 cm$^3$ TMSN$_3$ (0.44 mmol) and 0.008 cm$^3$ H$_2$O (0.44 mmol) were reacted together to afford 46 mg (76%) as a white solid. Melting range 179-181$^\circ$C; $^1$H NMR (500 MHz, CDCl$_3$) $\delta$ 9.17 (s, 1H), 7.33 – 7.27 (m, 3H), 7.18 (dd, $J$ = 7.5, 1.7 Hz, 2H), 5.79 (dd, $J$ = 9.6, 6.2 Hz, 1H), 3.49 (dd, $J$ = 13.1, 9.6 Hz, 1H), 3.45 – 3.37 (m, 3H), 3.29 (dd, $J$ = 13.1, 6.2 Hz, 1H), 2.81 – 2.74 (m, 1H), 1.86 – 1.74 (m, 2H), 1.73 – 1.62 (m, 2H); $^{13}$C NMR (126 MHz, CDCl$_3$) $\delta$ 164.92, 142.28, 134.09, 129.31, 129.10, 128.11, 126.37, 46.90, 46.45, 40.64, 25.96, 24.12; FT-IR (ATR) $\nu_{\text{max}}$/cm$^{-1}$ 3046.4, 2950.5, 1653.0, 1605.2, 1272.2; HRMS (ESI$^+$): m/z calcd. for C$_{14}$H$_{18}$N$_3$O$^+$ 272.1505, found 272.1518.
$^{13}$C NMR spectra of the compound 7s
Gram-scale experiment for product 7a

A dry 25 ml RBF was charged with an isocyanide (1.1 gm, 8.2 mmol), TMSN$_3$ (2.19 mL, 16.5 mmol) and H$_2$O (0.297 mL, 16.5 mmol). The reaction was then sonicated in an ultrasonic cleaner water bath (45 kHz) at room temperature for 1 hr. The resulting crude products were diluted with DCM (5 ml) and filtered through small Na$_2$SO$_4$ bed to remove traces of water. The resulting filtrate kept as it is at room temperature for evaporation to afford 1.31 gm of pure product 7a in 89% yield.