Zn(pybox)-Complex Catalyzed Asymmetric Aqueous Mukaiyama-Aldol Reactions

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Contents

Experimental	page S-2
Characterization of compounds 6 , 7a-h , 11a-e , 12 , 13	page S-2
References	page S-8
HPLC data	page S-10

Experimental

General: Optical rotations were measured with a Digital Polarimeter at room temperature. ^1H NMR spectra were recorded on 400 and 500 MHz spectrometers in CDCl_3 with Me_4Si as internal standard. High resolution mass spectra were taken on a mass spectrometer with time-of-flight (TOF) detector. Reactions were controlled using TLC on silica [alu-plates (0.2 mm)]. All reagents and solvents were purified and dried according to common methods. All organic solutions were dried over Na_2SO_4 . Reaction products were purified by flash chromatography using silica gel 60 (240-400 mesh). HPLC analysis were performed on HPLC system equipped with chiral stationary phase columns, detection at 254 nm.

(2*S*,3*S*)-3-Hydroxy-2-methyl-1,3-diphenyl-propan-1-one (6)^{1,2}

^1H NMR (200 MHz) δ (*syn*): 1.19 (d, 3H, J 7.2 Hz), 3.67 (d, 1H, J 1.7 Hz), 4.11 (dq, 1H, J 3.0, 7.3 Hz), 5.23 (brs, 1H), 7.20 - 7.65 (m, 8H), 7.85-8.20 (m, 2H).

^{13}C NMR (50 MHz) δ : 11.1, 47.1, 73.0, 125.9, 127.3, 128.2, 128.4, 128.7, 133.5, 135.6, 141.7, 205.8.

$[\alpha]_{\text{D}} +4.0$ (c 1.10 in CHCl_3 , 70 %ee), lit.[2]: $[\alpha]_{\text{D}} +11.7$ (c 1.03 in CHCl_3 , 95% ee) for (2*S*,3*S*)-isomer;

HPLC (Daicel Chiralpak AD, hexane/*i*-PrOH = 9/1, flow rate = 1 mL/min; *syn* isomer: t_1 = 9.9 min, t_2 = 12.2 min (major), *anti* isomer: t_1 = 14.6 min, t_2 = 16.0 min (major).

3-Hydroxy-3-(4-methoxy-phenyl)-2-methyl-1-phenyl-propan-1-one (7a)³

^1H NMR (200 MHz) δ (*syn*): 1.27 (d, 3H, J 7.2 Hz), 3.57 (d, 1H, J 2.0 Hz), 3.67 (dq, 1H, J 3.6, 7.1 Hz), 3.78 (s, 3H), 5.16 (m, 1H), 6.80-6.84 (m, 2H), 7.24-7.38 (m, 2H), 7.40-7.64 (m, 3H), 7.84-8.20 (m, 2H).

^{13}C NMR (50 MHz) δ : 11.5, 47.2, 55.2, 72.9, 113.6, 127.1, 127.8, 128.4, 128.7, 133.4, 133.9, 135.7, 205.6.

HPLC (Daicel Chiralpak AD, hexane/*i*-PrOH = 9/1, flow rate = 1 mL/min; *syn* isomer: t_1 = 15.9 min, t_2 = 17.9 min (major), *anti* isomer: t_1 = 25.0 min (major), t_2 = 25.8 min.

Hydroxy-3-(2-methoxy-phenyl)-2-methyl-1-phenyl-propan-1-one (7b)³

¹H NMR (200 MHz) δ (*syn*): 1.15 (d, 3H, *J* 7.2 Hz), 3.58 (s, 3H), 3.94 (dq, 1H, *J* 3.0, 7.1 Hz), 5.41 (brs, 1H), 6.85 (dd, 1H, *J* 1.0, 8.2 Hz), 6.92-7.20 (m, 1H), 7.18-7.30 (m, 1H), 7.40-7.63 (m, 4H), 7.94-8.04 (m, 2H).

¹³C NMR (50 MHz) δ : 10.8, 44.1, 55.1, 69.2, 109.9, 120.5, 127.6, 128.1, 128.4, 128.5, 129.5, 133.2, 135.9, 155.4, 205.7.

HPLC (Daicel Chiralpak AD-H, hexane/*i*-PrOH = 9/1, flow rate = 1 mL/min; *syn* isomer: t_1 = 12.5 min, t_2 = 18.9 min (major).

3-Hydroxy-3-(4-methyl-phenyl)-2-methyl-1-phenyl-propan-1-one (7c)¹

¹H NMR (200 MHz) δ (*syn*): 1.19 (d, 3H, *J* 7.2 Hz), 2.32 (s, 3H), 3.60 (d, 1H, *J* 1.7 Hz), 3.68 (dq, 1H, *J* 3.3, 7.1 Hz), 5.19 (d, 1H, *J* 0.8 Hz), 7.08-8.00 (m, 9H).

¹³C NMR (50 MHz) δ : 11.3, 21.0, 47.1, 73.0, 125.9, 128.4, 128.7, 128.8, 133.4, 135.6, 136.8, 138.8, 205.5.

HPLC (Daicel Chiralpak AD-H, hexane/*i*-PrOH = 9/1, flow rate = 1 mL/min; *syn* isomer: t_1 = 10.7 min, t_2 = 12.2 min (major), *anti* isomer: t_1 = 16.6 min (major), t_2 = 17.8 min.

3-(2-Chlorophenyl)-3-hydroxy-2-methyl-1-phenyl-propan-1-one (7d)³

¹H NMR (200 MHz) δ (*syn*): 1.10 (d, 3H, *J* 7.2 Hz), 3.94 (dq, 1H, *J* 5.5, 7.4 Hz), 4.03 (d, 1H, OH, *J* 1.9 Hz), 5.55 (s, 1H), 7.20-7.72 (m, 7H), 7.98-8.03 (m, 2H).

¹³C NMR (50 MHz) δ : 10.4, 42.9, 69.7, 126.7, 128.4, 128.6, 128.8, 129.2, 131.0, 133.7, 135.5, 138.6, 206.3.

HPLC (Daicel Chiralpak AD-H, hexane/*i*-PrOH = 9/1, flow rate = 1 mL/min; *syn* isomer: t_1 = 7.9 min, t_2 = 11.0 min (major), *anti* isomer: t_1 = 14.0 min, t_2 = 16.6 min (major).

3-(4-Chlorophenyl)-3-hydroxy-2-methyl-1-phenyl- propan-1-one (7e)¹

¹H NMR (200 MHz) δ (*syn*): 1.17 (d, 3H, *J* 7.2 Hz), 3.64 (dq, 1H, *J* 3.2, 7.3 Hz), 3.82 (d, 1H, *J* 1.9 Hz), 5.20 (t, 1H, *J* 3.0 Hz), 7.25-7.65 (m, 8H), 7.88-8.10 (m, 2H).

¹³C NMR (50 MHz) δ : 11.1, 46.8, 72.4, 127.4, 127.9, 128.6, 128.8, 132.9, 133.6, 135.4, 140.3, 205.4.

HPLC (Daicel Chiralpak AD-H, hexane/*i*-PrOH = 9/1, flow rate = 1 mL/min; *syn* isomer: *t*₁ = 11.1 min, *t*₂ = 12.5 min (major), *anti* isomer: *t*₁ = 15.7 min (major), *t*₂ = 20.6 min.

3-Hydroxy-2-methyl-1,5-diphenyl-pent-4-en-1-one (7f)³

¹H NMR (200 MHz) δ (*syn*): 1.36 (d, 3H, *J* 7.4 Hz), 3.40 (brs, 1H, OH), 3.64 (dq, 1H, *J* 3.5, 7.3 Hz), 4.72-4.81 (m, 1H), 6.24 (dd, 1H, *J* 5.7, 16.0 Hz), 6.71 (dd, 1H, *J* 1.4, 16.0 Hz), 7.10-7.70 (m, 8H), 7.80-8.10 (m, 2H).

¹³C NMR (50 MHz) δ : 11.9, 45.7, 72.2, 126.4, 127.5, 128.4, 128.8, 128.7, 129.1, 130.9, 133.4, 135.8, 136.6, 205.0.

HPLC (Daicel Chiralpak AD-H, hexane/*i*-PrOH = 9/1, flow rate = 1 mL/min; *syn* isomer: *t*₁ = 17.8 min (major), *t*₂ = 21.8 min, *anti* isomer: *t*₁ = 20.0 min, *t*₂ = 26.9 min.

3-Hydroxy-2,4,4-trimethyl-1-phenyl-pentan-1-one (7g)⁴

¹H NMR (400 MHz) δ (*syn*): 0.99 (s, 9H), 1.28 (d, 3H, *J* 7.2 Hz), 2.62 (s, 1H, OH), 3.74-3.80 (m, 2H), 7.45-7.51 (m, 2H), 7.58-7.61 (m, 1H), 7.95-8.00 (m, 2H).

¹³C NMR (50 MHz) δ : 13.1, 26.9, 35.7, 40.9, 77.6, 128.5, 128.8, 133.3, 135.7, 205.3.

HPLC (Daicel Chiralpak AD-H, hexane/*i*-PrOH = 9/1, flow rate = 1 mL/min; *syn* isomer: *t*₁ = 5.7 min, *t*₂ = 6.8 min (major).

3-Hydroxy-2,4-dimethyl-1-phenyl-pentan-1-one (7h)¹

¹H NMR (400 MHz) δ (*syn*): 0.96 (d, 3H, *J* 6.7 Hz), 1.04 (d, 3H, *J* 6.6 Hz), 1.25 (d, 3H, *J* 7.0 Hz), 3.11 (d, 1H, OH, *J* 1.9 Hz), 3.60 (m, 2H), 7.47-7.65 (m, 1H), 7.90-7.97 (m, 2H).

¹³C NMR (50 MHz) δ : 10.7, 19.0, 19.1, 30.7, 41.8, 128.4, 128.8, 133.4, 135.9, 205.9.

HPLC (Daicel Chiralpak AD-H, hexane/*i*-PrOH = 9/1, flow rate = 1 mL/min; *syn* isomer: t_1 = 6.5 min, t_2 = 7.9 min (major), *anti* isomer was separated using flash chromatography: t_1 = 8.3 min, t_2 = 10.2 min (major).

3-Hydroxy-2-methyl-1-phenyl-nonan-1-one (7i)¹

¹H NMR (200 MHz) δ (*syn*): 0.89 (t, 3H, J 6.2 Hz), 1.26 (d, 3H, J 5.2 Hz), 1.00-1.80 (m, 10H), 3.17 (s, 1H, OH), 3.48 (dq, 1H, J 3.0, 7.3 Hz), 4.04 (dd, 1H, J 3.8 Hz), 7.42-7.66 (m, 4H), 7.90-8.20 (m, 2H).

¹³C NMR (50 MHz) δ : 11.0, 14.0, 22.6, 26.0, 29.3, 31.8, 34.3, 44.5, 71.3, 128.4, 128.7, 133.4, 135.9, 205.8.

HPLC (Daicel Chiralpak AS-H, hexane/*i*-PrOH = 95/5, flow rate = 1 mL/min; *syn* isomer: t_1 = 6.7 min, t_2 = 8.7 min (major), *anti* isomer: t_1 = 7.1 min, t_2 = 15.3 min.

3-Hydroxy-2-methyl-1-phenyl-heksan-1-one (7j)⁵

¹H NMR (200 Hz) δ (*syn*): 0.95 (t, 3H, J 6.9 Hz), 1.25 (d, 3H, J 7.1 Hz), 1.32-1.70 (m, 4H), 3.10 (s, 1H, OH), 3.45 (dq, 2H, J 2.8, 7.1 Hz), 4.10-4.20 (m, 2H), 7.42-7.64 (m, 4H), 7.88-8.04 (m, 2H).

¹³C NMR (50 Hz) δ : 11.1, 14.1, 19.3, 36.5, 44.5, 71.0, 128.4, 128.7, 133.4, 135.9, 205.8.

HPLC (Daicel Chiralpak AS-H, hexane/*i*-PrOH = 95/5, flow rate = 1 mL/min; *syn* isomer: t_1 = 8.1 min, t_2 = 11.3 min (major), *anti* isomer: t_1 = 8.3 min, t_2 = 17.5 min.

3-Hydroxy-2-methyl-1,5-diphenyl-pentan-1-one (7k)³

¹H NMR (200MHz) δ (*syn*): 1.27 (d, 3H, J 7.2 Hz), 1.62-2.04 (m, 2H), 2.62-2.79 (m, 1H), 2.82-2.98 (m, 1H), 3.16 (d, 1H, OH, J 1.6 Hz), 3.44 (dq, 1H, J 7.2, 2.8 Hz), 4.08 (m, 1H), 7.12-7.34 (m, 5H), 7.41-7.52 (m, 2H), 7.50-7.63 (m, 3H), 7.86-7.97 (m, 2H).

¹³C NMR (50 MHz) δ : 11.1, 32.3, 36.1, 44.6, 70.6, 125.8, 128.3, 128.4(2C), 128.5, 128.7, 133.4, 141.9, 205.7;

HPLC (Daicel Chiralpak AS-H, hexane/*i*-PrOH = 97/3, flow rate = 1 mL/min; *syn* isomer: t_1 = 12.0 min, t_2 = 18.8 min (major), *anti* isomer: t_1 = 13.4 min (major), t_2 = 32.4 min.

4-(Benzyloxy)-3-hydroxy-2-methyl-1-phenyl-butan-1-one (7l)⁶

¹H NMR (200 MHz) δ (*syn*): 1.26 (d, 3H, *J* 7.12 Hz), 2.89 (d, 1H, OH, *J* 4.27 Hz), 3.44-3.62 (m, 2H), 4.16-4.26 (m, 1H), 4.49 (s, 2H), 7.20-7.65 (m, 8H), 7.85-8.15 (m, 2H).

HPLC (Daicel Chiralpak AS, hexane/*i*-PrOH = 9/1, flow rate = 1 mL/min; *syn* isomer: *t*₁ = 8.3 min, *t*₂ = 10.5 min (major), *anti* isomer: *t*₁ = 12.3 min, *t*₂ = 14.7 min (major).

2-Hydroxy-3-methyl-4-oxo-4-phenyl-ethyl-butanoate (7m)⁷

¹H NMR (200 MHz) δ (*syn*): 1.27 (t, 3H, *J* 7.1 Hz), 1.30 (d, 3H, *J* 7.2 Hz), 3.23 (d, 1H, *J* 4.4 Hz), 3.86-4.10 (m, 1H), 4.26 (q, 2H, *J* 7.2, 14.4 Hz), 4.60 (t, 1H, *J* 4.2 Hz), 7.40-7.65 (m, 4H).

HPLC (Daicel Chiralpak AS-H, hexane/*i*-PrOH = 9/1, flow rate = 1 mL/min; *syn* isomer: *t*₁ = 14.8 min, *t*₂ = 18.8 min (major), *anti* isomer: *t*₁ = 21.8 min, *t*₂ = 32.7 min.

3-Hydroxy-2-methyl-1-phenyl-propan-1-one (7n)⁸

¹H NMR (200 Hz) δ : 1.24 (d, 3H, *J* 7.2 Hz), 2.42 (brs, 1H, OH), 3.45-4.10 (m, 3H), 7.25-7.65 (m, 4H), 7.80-8.15 (m, 1H).

¹³C NMR (50 MHz) δ : 14.5, 42.9, 64.5, 128.4, 128.7, 133.3, 136.1, 204.4;

HPLC (Daicel Chiralpak AD-H, hexane/*i*-PrOH = 9/1, flow rate = 1 mL/min: *t*₁ = 8.6 min, *t*₂ = 9.8 min

(3*R*)-Hydroxy-1,3-diphenyl-propan-1-one (11a)¹

¹H NMR (200 MHz) δ : 3.38 (d, 2H, *J* 6.2 Hz), 3.60 (brs, 1H, OH), 5.35 (t, 1H, *J* 6.2 Hz), 7.25-7.63 (m, 8H), 7.93-7.98 (m, 2H).

¹³C NMR (50 MHz) δ : 47.4, 70.0, 125.7, 127.6, 128.1, 128.5, 128.7, 133.6, 200.1.

for ligand 2: $[\alpha]_D^{+27}$ (*c* 1.00 in MeOH, 60 %ee), lit.[7]: $[\alpha]_D^{+31.97}$ (*c* 0.76 in MeOH, 98% ee); HPLC (Daicel Chiralpak AS, hexane/*i*-PrOH = 9/1, flow rate = 1 mL/min: *t*₁ = 11.9 min, *t*₂ = 13.5 min (major).

3-(4-Chlorophenyl)-3-hydroxy-1-phenyl-propan-1-one (11b)⁹

¹H NMR (200 MHz) δ : 3.32 (d, 2H, J 6.2 Hz), 3.74 (d, 1H, J 2.8 Hz), 5.30 (td, 1H, J 2.8, 6.0 Hz), 7.25-7.66 (m, 7H), 7.88-7.98 (m, 2H).

¹³C NMR (50 MHz) δ : 47.2, 69.3, 127.1, 128.0, 128.6, 128.7, 133.2, 133.7, 136.3, 141.4, 199.8.

HPLC (Daicel Chiralpak AD-H, hexane/*i*-PrOH = 9/1, flow rate = 1 mL/min: t_1 = 16.8 min, t_2 = 20.2 min (major).

3-(2-Chlorophenyl)-3-hydroxy-1-phenyl-propan-1-one (11c)¹⁰

¹H NMR (200 MHz) δ : 3.14 (dd, 1H, J 9.5, 17.7 Hz), 3.54 (dd, 1H, J 2.2, 17.7 Hz), 3.90 (d, 1H, OH, J 2.5 Hz), 5.68 (d, 1H, J 9.5 Hz), 7.16-7.64 (m, 6H), 7.65-7.76 (m, 1H), 7.90-8.00 (m, 2H).

¹³C NMR (50 MHz) δ : 45.3, 66.7, 127.2, 128.1, 128.5, 128.6, 129.2, 131.1, 133.6, 136.3, 140.3, 200.1.

HPLC (Daicel Chiralpak AS-H, hexane/*i*-PrOH = 9/1, flow rate = 1 mL/min: t_1 = 8.8 min, t_2 = 10.8 min (major).

3-Hydroxy-3-(4-methoxy-phenyl)-1-phenyl-propan-1-one (11d)¹¹

¹H NMR (200 MHz) δ : 3.34 (s, 1H), 3.36 (d, 1H, J 3.3 Hz), 3.53 (brs, 1H), 3.80 (s, 3H), 5.36-5.41 (m, 1H), 6.88-6.92 (m, 2H), 7.43-7.37 (m, 2H), 7.42-7.48 (m, 2H), 7.54-7.60 (m, 1H), 7.93-7.96 (m, 2H).

HPLC (Daicel Chiralpak AS-H, hexane/*i*-PrOH = 9/1, flow rate = 1 mL/min: t_1 = 27.3 min (major), t_2 = 28.9 min; and Daicel Chiralpak AD-H, hexane/*i*-PrOH = 9/1, flow rate = 1 mL/min: t_1 = 27.2 min, t_2 = 28.4 min (major).

3-Hydroxy-3-(2-methoxy-phenyl)-1-phenyl-propan-1-one (11e)¹²

¹H NMR (200 MHz) δ : 3.24 (dd, 1H, *J* 9.0, 17.0 Hz), 3.50 (dd, 1H, *J* 4.0, 17.0 Hz), 3.68 (d, 1H, OH, *J* 4.0 Hz), 3.83 (s, 3H), 5.60 (d, 1H, *J* 8.0 Hz), 6.88 (d, 2H, *J* 8.0 Hz), 7.00 (td, 1H, *J* 2.0, 7.4 Hz), 7.27 (td, 1H, *J* 2.0, 7.4 Hz), 7.38-7.62 (m, 4H), 7.90-8.02 (m, 2H).

¹³C NMR (50 MHz) δ : 45.7, 55.2, 65.5, 110.1, 120.8, 126.4, 128.1, 128.3, 128.5, 131.0, 133.3, 136.7, 155.6, 200.5.

HPLC (Daicel Chiralpak AS-H, hexane/*i*-PrOH = 9/1, flow rate = 1 mL/min: *t*₁ = 14.1 min, *t*₂ = 19.7 min (major).

1-Hydroxy-2-methyl-1-phenylpentan-3-one (12)¹

¹H NMR (200 Hz) δ (*syn*): 1.00 (t, 3H, *J* 7.2 Hz), 1.08 (d, 3H, *J* 7.2 Hz), 2.26-2.62 (m, 2H), 2.78-2.99 (m, 1H), 3.15 (d, 3H, *J* 2.4 Hz), 5.05 (dd, 1H, *J* 3.9, 1.8 Hz), 7.19-7.40 (m, 5H);

HPLC (Daicel Chiralpak AS-H, hexane/*i*-PrOH = 9/1, flow rate = 1 mL/min; *syn* isomer: *t*₁ = 11.2 min (major), *t*₂ = 14.0 min, *anti* isomer: *t*₁ = 12.3 min (major), *t*₂ = 19.6 min.

2-[Hydroxy(phenyl)methyl]cyclohexanone (13)¹³

For ligand **2**: ***Syn* isomer:**

¹H NMR (200 Hz) δ : 1.40-1.94 (m, 5H), 2.00-2.20 (m, 1H), 2.26-2.72 (m, 3H), 3.15 (d, 1H, OH, *J* 3.16 Hz), 5.39 (t, 1H, *J* 2.85 Hz), 7.18-7.44 (m, 5H).

¹³C NMR (50 MHz) δ : 24.8, 25.9, 27.9, 42.6, 57.1, 70.5, 125.7, 126.9, 128.1, 141.4, 214.7.

HPLC (Daicel Chiralpak AS-H, hexane/*i*-PrOH = 95/5, flow rate = 1 mL/min: *t*₁ = 13.7 min (major), *t*₂ = 16.4 min. ***Anti* isomer:** ¹H NMR (200 Hz) δ : 1.20-1.88 (m, 5H), 2.00-2.20 (m, 1H), 2.24-2.72 (m, 3H), 3.12 (d, 1H, OH, *J* 3.2 Hz), 3.96 (d, 1H, *J* 2.7 Hz), 4.78 (dd, 1H, *J* 8.8, 2.7 Hz), 7.18-7.42 (m, 5H).

¹³C NMR (50 MHz) δ : 24.7, 27.8, 30.8, 42.7, 57.4, 74.7, 126.9, 127.8, 128.1, 128.3, 215.4.

HPLC (Daicel Chiralpak AS-H, hexane/*i*-PrOH = 95/5, flow rate = 1 mL/min: *t*₁ = 17.4 min (major), *t*₂ = 18.7 min.

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Analiza: 211 ChromaX wersja1.2c

mly-md-035-a

Chiralpak AD-H

Heksan-iPrOH (9:1)

1ml/min

NUMER PIKU	CZAS RETENCJI [min]	UDZIAŁ PROCENTOWY
A/1	9,847	14,153
A/2	12,227	80,921
A/3	14,507	1,787
A/4	15,890	3,139
SUMA

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Nazwa analizy: md035-b.chr / gc1.met
Data utworzenia: 28-09-04 10:15 /
Analiza: 212 ChromaX wersja 1.2c

mly-md-035-b
Chiralpak AD-H
Heksan-iPrOH (9:1)
1ml/min

NUMER PIKU	CZAS RETENCJI [min]	UDZIAŁ PROCENTOWY
A/1	15,950	15,409
A/2	17,967	73,722
A/3	25,015	6,387
A/4	25,827	4,482
SUMA

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Nazwa analizy: md035-c.chr / gc1.met

Data utworzenia: 05-10-04 08:51 /

Analiza: 228 ChromaX wersja1.2c

mly-md-035-c1

Chiralpak AD-H

Heksan-iPrOH (9:1)

1ml/min

NUMER PIKU	CZAS RETENCJI [min]	UDZIAŁ PROCENTOWY
A/1	12,595	15,810
A/2	18,960	84,190
SUMA

C₁₇H₁₈O₃
Mol. Wt.: 270,32

Raport wydrukowano: 27-07-2005 12:47

| ja054a.chr |

Nazwa analizy: ja054a.chr / gc1.met

Data utworzenia: 30-09-04 10:57 /

Analiza: 217 ChromaX wersja 1.2c

jj-ja-054a
chiralpak ad-h
Heksan-iPrOH 9:1
1 ml/min

NUMER PIKU	CZAS RETENCJI [min]	UDZIAŁ PROCENTOWY
A/1	10,737	14,295
A/2	12,210	80,318
A/3	16,657	3,764
A/4	17,765	1,623
SUMA

$C_{17}H_{18}O_2$
Mol. Wt.: 254,32

Raport wydrukowano: 27-07-2005 12:50

| ja061a1.chr |

Nazwa analizy: ja061a1.chr / gc1.met
Data utworzenia: 14-10-04 15:04 /
Analiza: 246 ChromaX wersja1.2c

jj-ja-61-a1
chiralpak ad-h
Heksan-iPrOH 9:1
1 ml/min

NUMER PIKU	CZAS RETENCJI [min]	UDZIAŁ PROCENTOWY
A/1	7,897	17,483
A/2	10,960	82,096
A/3	13,998	0,178
A/4	16,597	0,243
SUMA

Raport wydrukowano: 27-07-2005 12:53

| ja059a.chr |

Nazwa analizy: ja059a.chr / gc1.met
Data utworzenia: 07-10-04 14:14 /
Analiza: 233 ChromaX wersja1.2c

jj-ja-059a
chiralpak AD-H
Heksan-iPrOH 9:1
1 ml/min

NUMER PIKU	CZAS RETENCJI [min]	UDZIAŁ PROCENTOWY
A/1	11,185	14,707
A/2	12,535	78,378
A/3	15,755	4,257
A/4	20,643	2,658
SUMA

Nazwa analizy: md035-e.chr / gc1.met
Data utworzenia: 05-10-04 09:53 /
Analiza: 229 ChromaX wersja1.2c

mly-md-035-e
Chiralpak AD-H
Heksan-iPrOH (9:1)
1ml/min

NUMER PIKU	CZAS RETENCJI [min]	UDZIAŁ PROCENTOWY
A/1	17,765	74,196
A/2	19,997	6,309
A/3	21,763	14,227
A/4	26,908	5,268
SUMA

Nazwa analizy: md035-e.chr

Raport wydrukowano: 27-07-2005 12:54

| ja059b2.chr |

Nazwa analizy: ja059b2.chr / gc1.met
Data utworzenia: 12-10-04 14:24 /
Analiza: 244 ChromaX wersja1.2c

jj-ja-059b2
chiralpak ad-h
Heksan-iPrOH 9:1
1 ml/min

NUMER PIKU	CZAS RETENCJI [min]	UDZIAŁ PROCENTOWY
A/1	6,493	14,284
A/2	7,865	85,716
SUMA

Operator: Joanna

Object name: ja58-ash_Det1 - Created: 16/02/2005, 11:52

Path: >Joanna>Pomiary>ASH

	Ret.time [min]	Start [min]	End [min]	Area [mAU*min]	Height [mAU]	% Area	Width [min]
1	6.770	6.46	6.98	72.1991	465.717	16.2846	0.141
2	7.087	6.98	7.33	15.8222	99.1131	3.5687	0.135
3	8.721	8.42	9.46	339.346	1219.15	76.5399	0.268
4	15.340	14.87	15.90	15.9914	39.5883	3.6069	0.381

Object name: Time table - m_uv(95:5) - Created: 31/01/2005, 12:37

Path:

	Time min	Flow ml/min	Valve %A	Valve %B	D.In	WLength 1
1	0.00	1.000	95	5	-	254
2	0.02	1.000	95	5	1	254
3	60.00	1.000	95	5	-	254

Operator: Joanna

Object name: ja58-ash_Det1 - Created: 16/02/2005, 11:29

Path: >Joanna>Pomiary>ASH

Operator: Joanna

Object name: ja101A-ash_Det1 - Created: 16/02/2005, 13:15

Path: >Joanna>Pomiary>ASH

	Ret.time [min]	Start [min]	End [min]	Area [mAU*min]	Height [mAU]	% Area	Width [min]
1	8.068	7.78	8.22	61.3896	361.294	12.7097	0.146
2	8.318	8.22	8.59	20.8092	113.777	4.3082	0.135
3	11.285	11.02	12.04	388.988	1119.61	80.5338	0.340
4	17.752	17.32	18.10	11.8252	34.5424	2.4482	0.339

Object name: Time table - m_uv(95:5) - Created: 31/01/2005, 12:37

Path:

	Time min	Flow ml/min	Valve %A	Valve %B	D.In	WLength 1
1	0.00	1.000	95	5	-	254
2	0.02	1.000	95	5	1	254
3	60.00	1.000	95	5	-	254

7j

C₁₃H₁₈O₂
Mol. Wt.: 206,28

Operator: Joanna

Object name: ja101A-ash_Det1 - Created: 16/02/2005, 12:32

Path: >Joanna>Pomiary>ASH

Object name: Time table - m_uv(97:3) - Created: 15-02-2005, 15:29
Path:

	Time min	Flow ml/min	Valve %A	Valve %B	D.In	WLength 1
1	0.00	1.000	97	3	-	254
2	0.02	1.000	97	3	1	254
3	60.00	1.000	97	3	-	254

Operator: Joanna

Object name: jb-05b_Det1 - Created: 17-05-2005, 13:15

Path: >Joanna>Pomiary>ASH

	Ret.time [min]	Start [min]	End [min]	Area [mAU*min]	Height [mAU]	% Area	Width [min]
1	12.051	11.42	12.88	33.5691	109.106	12.9384	0.283
2	13.417	12.88	15.08	26.5833	83.2739	10.2459	0.270
3	18.868	18.27	20.60	192.107	364.85	74.0429	0.492
4	32.452	31.61	33.42	7.19437	8.84702	2.7729	0.781

$C_{18}H_{20}O_2$
Mol. Wt.: 268,35

Operator: Joanna

Object name: jb-05b_Det1 - Created: 17-05-2005, 12:24

Path: >Joanna>Pomiary>ASH

Object name: Time table - m_uv - Created: 17/01/2005, 22:19

Path:

	Time min	Flow ml/min	Valve %A	Valve %B	D.In	WLength 1
1	0.00	1.000	90	10	-	254
2	0.02	1.000	90	10	1	254
3	60.00	1.000	90	10	-	254

Operator: Joanna

Object name: ja67A-2_Det1_Det1 - Created: 18/02/2005, 15:14

Path: >Joanna>Pomiary>ASH

	Ret.time [min]	Start [min]	End [min]	Area [mAU*min]	Height [mAU]	% Area	Width [min]
1	14.802	14.25	16.55	311.415	618.321	41.2906	0.477
2	18.853	18.32	21.12	205.847	274.894	27.2933	0.695
3	21.886	21.12	24.24	96.0811	123.326	12.7394	0.731
4	32.705	31.54	35.30	140.861	127	18.6767	1.032

C₁₃H₁₆O₄
Mol. Wt.: 236,26

Operator: Joanna

Object name: ja67A-2_Det1_Det1 - Created: 18/02/2005, 14:34

Path: >Joanna>Pomiary>ASH

Raport wydrukowano: 27-07-2005 13:47

| ja021.chr |

Nazwa analizy: ja021.chr / gc1.met

Data utworzenia: 09-07-04 12:20 /

Analiza: 111 ChromaX wersja1.2c

jj-ja-021

chiralpak as

Heksan-iPrOH 9:1

1 ml/min

NUMER PIKU	CZAS RETENCJI [min]	UDZIAŁ PROCENTOWY
A/1	11,900	23,705
A/2	13,565	76,295
SUMA

$C_{15}H_{14}O_2$
Mol. Wt.: 226,27

Raport wydrukowano: 27-07-2005 13:24

| ja040a.chr |

Nazwa analizy: ja040a.chr / gc1.met
Data utworzenia: 30-07-04 13:30 /
Analiza: 158 ChromaX wersja1.2c

jj-ja-040a
chiralpak ad-h
Heksan-iPrOH 9:1
1 ml/min

NUMER PIKU	CZAS RETENCJI [min]	UDZIAŁ PROCENTOWY
A/1	16,795	32,145
A/2	20,255	67,855
SUMA

C₁₅H₁₃ClO₂
Mol. Wt.: 260,72

Object name: Time table - m_uv - Created: 17-01-2005, 22:19
Path:

	Time min	Flow ml/min	Valve %A	Valve %B	D.In	WLength 1
1	0.00	1.000	90	10	-	254
2	0.02	1.000	90	10	1	254
3	60.00	1.000	90	10	-	254

Operator: Joanna
Object name: md-37a_Det1 - Created: 27-07-2005, 14:43
Path: >Joanna>Pomiary>ASH

	Ret.time [min]	Start [min]	End [min]	Area [mAU*min]	Height [mAU]	% Area	Width [min]
1	8.822	8.52	9.31	128.883	541.006	27.6213	0.225
2	10.890	10.36	12.49	337.725	991.217	72.3787	0.326

11c

C₁₅H₁₃ClO₂
Mol. Wt.: 260,72

Operator: Joanna
Object name: md-37a_Det1 - Created: 27-07-2005, 14:24
Path: >Joanna>Pomiary>ASH

Raport wydrukowano: 27-07-2005 13:43

| ja056a.chr |

Nazwa analizy: ja056a.chr / gc1.met
Data utworzenia: 01-10-04 15:12 /
Analiza: 221 ChromaX wersja1.2c

jj-ja-056a
chiralpak as
Heksan-iPrOH 9:1
1 ml/min

NUMER PIKU	CZAS RETENCJI [min]	UDZIAŁ PROCENTOWY
A/1	14,783	13,957
A/2	18,633	86,043
SUMA

$C_{16}H_{16}O_3$
Mol. Wt.: 256,30