



Supporting Information

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**for Highly Enantioselective Phase-Transfer Catalytic Alkylation
of 2-Phenyl-2-oxazoline-4-carboxylic acid *tert*-butyl ester
for the Asymmetric Synthesis of α -Alkylserine**

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2-Phenyl-2-oxazoline-4-carboxylic acid *tert*-butyl ester (7).

To a methylenechloride solution (60 mL) of ethyl benzimidate hydrochloride (3.71g, 20.0 mmol) and L-serine *tert*-butyl ester hydrochloride (3.52g, 20.0 mmol) in dichloromethane was added triethylamine (5.58 mL, 40.0 mmol). The reaction mixture was refluxed for 2 h and stirred for 10 h at room temperature. The reaction mixture was diluted with methylenechloride (200 mL), washed with water (2 x 50 mL), dried over anhydrous MgSO₄, filtered, and concentrated *in vacuo*. The residue was purified by column chromatography (silica gel, hexane:EtOAc = 9:1) to afford **7** (5.16g, 98% yield) as a white solid: ¹H NMR (300 MHz, CDCl₃) δ 7.99 (d, *J* = 7.1 Hz, 2 H), 7.53-7.36 (m, 3 H), 4.84 (dd, *J* = 8.0, 10.5 Hz, 1 H), 4.62-4.53 (m, 2 H), 1.51 (s, 9 H) ppm; ¹³C NMR (100 MHz,

CDCl₃) δ 170.3, 165.9, 131.6, 128.5, 128.1, 127.0, 82.0, 69.7, 69.3, 27.9 ppm; IR (KBr) 2979, 1734, 1644, 1453, 1364, 1298, 1222, 1156, 1089, 1026, 967, 846, 777, 696 cm⁻¹. MS (FAB) m/z 248 [M+H]⁺. Mp 41.7-45.3 °C.

Representative Procedure for Enantioselective Catalytic Alkylation of 2-phenyl-2-oxazoline-4-carboxylic acid *tert*-butyl ester **7 under Phase-Transfer Conditions (Benzylation).**

To a toluene solution (0.80 mL) of 2-Phenyl-2-oxazoline-4-carboxylic acid *tert*-butyl ester **7** (50.0 mg, 0.200 mmol), chiral catalyst **4b** (9.55 mg, 0.005 mmol) (purchased from Aldrich Co.) and KOH (56.1 mg, 1.00 mmol) was added benzyl bromide (0.12 mL, 1.00 mmol) at 0 °C. The reaction mixture was stirred (5 h). After complete the reaction, the reaction mixture was diluted with ethyl acetate (20 mL), washed with water (2 x 5 mL), dried over anhydrous MgSO₄, filtered, and concentrated *in vacuo*. The residue was purified by column chromatography (silica gel, hexane:EtOAc = 20:1) to afford **8f** (65 mg, 98% yield) as a pale yellow oil. The enantioselectivity was determined by chiral HPLC analysis (DAICEL Chiralcel OD-H, hexane:2-propanol = 500:3.0, flow rate = 1.0 mL/min, 23 °C, λ = 254 nm, retention times; *S* (major) 9.5 min, *R* (minor) 16.1 min, over 99% ee). Absolute configuration was determined by comparison of the optical rotation of α -benzylserine from the acid hydrolysis of **8e** with the reported value.^{4m}: ¹H NMR (300 MHz, CDCl₃) δ 7.94 (d, *J* = 7.1 Hz, 2 H), 7.50-7.15 (m, 8 H), 4.66 (d, *J* = 8.8 Hz, 1 H), 4.31 (d, *J* = 8.8 Hz, 1 H), 3.25 (dd, *J* = 13.8, 46.7 Hz, 2 H), 1.46 (s, 9 H) ppm; ¹³C NMR (100 MHz, CDCl₃) δ 171.3, 164.6, 135.6, 131.5, 130.3, 128.5, 128.1, 128.0, 127.2, 126.8, 82.1, 78.7, 72.8,

43.1, 27.8 ppm; IR (KBr) 2978, 1726, 1644, 1453, 1366, 1280, 1160, 1095, 978, 847, 697 cm^{-1} . MS (FAB) m/z 338 $[\text{M}+\text{H}]^+$.

Characterization of Alkylation Products.

4-Ethyl-2-phenyl-2-oxazoline-4-carboxylic acid *tert*-butyl ester (8a); [R = ethyl]

The enantioselectivity was determined by chiral HPLC analysis (DAICEL Chiralcel OD-H, hexane:2-propanol = 500:3.0, flow rate = 1.0 mL/min, 23 °C, λ = 254 nm, retention times; *S* (major) 7.3 min, *R* (minor) 11.8 min, 93% ee): ^1H NMR (300 MHz, CDCl_3) δ 7.99 (d, J = 8.3 Hz, 2 H), 7.37-7.51 (m, 3 H), 4.71 (d, J = 8.9 Hz, 1 H), 4.21 (d, J = 8.9 Hz, 1 H), 1.95 (qd, J = 1.4, 7.5 Hz, 2 H), 1.50 (s, 9 H), 0.94 (t, J = 7.5 Hz, 3 H) ppm; ^{13}C NMR (100 MHz, CDCl_3) δ 172.0, 164.1, 131.5, 128.5, 128.2, 127.4, 81.7, 78.9, 73.4, 31.0, 27.9, 8.0 ppm; IR (KBr): 2973, 2926, 1726, 1646, 1455, 1364, 1252, 1146, 1067, 1026, 971, 845, 778, 694 cm^{-1} . MS (ESI) m/z 276 $[\text{M}+\text{H}]^+$, 298 $[\text{M}+\text{Na}]^+$.

4-Allyl-2-phenyl-2-oxazoline-4-carboxylic acid *tert*-butyl ester (8b); [R = allyl]

The enantioselectivity was determined by chiral HPLC analysis (DAICEL Chiralcel OD-H, hexane:2-propanol = 500:3.0, flow rate = 1.0 mL/min, 23 °C, λ = 254 nm, retention times; *S* (major) 6.2 min, *R* (minor) 7.8 min, 97% ee): ^1H NMR (300MHz, CDCl_3) δ 7.98 (d, J = 6.8 Hz, 2 H), 7.52-7.38 (m, 3 H), 5.80-5.66 (m, 1 H), 5.20-5.13 (m, 2 H), 4.70 (d, J = 8.8 Hz, 1 H), 4.27 (d, J = 8.8 Hz, 1 H), 2.76-2.60 (m, 2 H), 1.50 (s, 9 H) ppm; ^{13}C NMR (100 MHz, CDCl_3) δ 171.3,

164.6, 131.9, 131.6, 128.6, 128.3, 127.2, 119.5, 82.0, 77.8, 73.1, 42.1, 27.9 ppm; IR (KBr) 2979, 1728, 1644, 1452, 1365, 1275, 1147, 1089, 1027, 978, 922, 846, 777, 696 cm^{-1} . MS (FAB) m/z 288 $[\text{M}+\text{H}]^+$.

4-(2-Methyl-allyl)-2-phenyl-2-oxazoline-4-carboxylic acid *tert*-butyl ester (8c); [R = 2-Methyl-allyl]

The enantioselectivity was determined by chiral HPLC analysis (DAICEL Chiralcel OD-H, hexane:2-propanol = 500:2.5, flow rate = 1.0 mL/min, 23 °C, λ = 254 nm, retention times; *S* (major) 7.3 min, *R* (minor) 8.4 min, 96% ee): ^1H NMR (300 MHz, CDCl_3) δ 7.95 (d, J = 7.0 Hz, 2 H), 7.45-7.34 (m, 3 H), 4.85 (s, 1 H), 4.82 (d, J = 8.8 Hz, 1 H), 4.68 (s, 1 H), 4.29 (d, J = 8.8 Hz, 1 H), 2.83 (d, J = 14.6 Hz, 1 H), 2.53 (d, J = 14.6 Hz, 1 H), 1.74 (s, 3 H), 1.45 (s, 9 H) ppm; ^{13}C NMR (100 MHz, CDCl_3) δ 171.2, 164.2, 140.9, 131.5, 128.5, 128.2, 127.3, 114.3, 81.9, 77.9, 73.3, 45.5, 27.8, 23.9 ppm; IR (KBr) 2976, 2926, 1727, 1644, 1453, 1365, 1286, 1160, 1102, 978, 898, 846, 776, 695 cm^{-1} . MS (ESI) m/z 302 $[\text{M}+\text{H}]^+$, 324 $[\text{M}+\text{Na}]^+$.

2-phenyl-4-prop-2-ynyl-2-oxazoline-4-carboxylic acid *tert*-butyl ester (8d); [R = propargyl]

The enantioselectivity was determined by chiral HPLC analysis (DAICEL Chiralcel OD-H, hexane:2-propanol = 500:3.0, flow rate = 1.0 mL/min, 23 °C, λ = 254 nm, retention times; *S* (major) 11.6 min, *R* (minor) 9.5 min, 97% ee): ^1H NMR (300 MHz, CDCl_3) δ 7.97 (d, J = 6.8 Hz, 2 H), 7.51-7.36 (m, 3 H), 4.85 (d, J = 8.8 Hz, 1 H), 4.47 (d, J = 8.8 Hz, 1 H), 2.95 (dd, J = 2.7, 16.7 Hz, 1 H), 2.69

(dd, $J=2.7, 16.7$ Hz, 1 H), 1.95 (t, $J = 2.7$ Hz, 1 H), 1.49 (s, 9 H) ^{13}C NMR (100 MHz, CDCl_3) δ 170.0, 165.5, 131.8, 128.6, 128.2, 126.9, 82.5, 78.7, 77.4, 73.5, 71.0, 27.9, 27.8. IR (KBr): 2979, 1731, 1641, 1453, 1366, 1279, 1161, 1101, 978, 845, 695 cm^{-1} . MS (FAB) m/z 286 $[\text{M}+\text{H}]^+$.

4-(4-Cyano-benzyl)-2-phenyl-2-oxazoline-4-carboxylic acid *tert*-butyl ester (8f); [R = 4-cyanobenzyl]

The enantioselectivity was determined by chiral HPLC analysis (DAICEL Chiralcel OD-H, hexane:2-propanol = 500:3.5, flow rate = 1.0 mL/min, $23\text{ }^\circ\text{C}$, $\lambda = 254\text{ nm}$, retention times; *S* (major) 25.3 min, *R* (minor) 43.6 min, 92% ee): ^1H NMR (300 MHz, CDCl_3) δ 7.91 (d, $J = 8.5$ Hz, 2 H), 7.58-7.37 (m, 7 H), 4.63 (d, $J = 9.1$ Hz, 1 H), 4.23 (d, $J = 9.1$ Hz, 1 H), 3.35 (d, $J = 13.8$ Hz, 1 H), 3.18 (d, $J = 13.8$ Hz, 1 H), 1.43 (s, 9 H) ppm; ^{13}C NMR (100 MHz, CDCl_3) δ 170.8, 165.0, 141.4, 131.8, 131.7, 131.1, 128.4, 128.2, 126.8, 118.7, 110.7, 82.5, 78.2, 73.3, 43.2, 27.8 ppm; IR(KBr) 2978, 2228, 1728, 1644, 1452, 1366, 1281, 1159, 1092, 979, 847, 759, 697 cm^{-1} . MS (FAB) m/z 363 $[\text{M}+\text{H}]^+$.

2-Phenyl-4-(4-trifluoromethyl-benzyl)-2-oxazoline-4-carboxylic acid *tert*-butyl ester (8g); [R = 4-trifluoromethylbenzyl]

The enantioselectivity was determined by chiral HPLC analysis (DAICEL Chiralcel OD-H, hexane:2-propanol = 500:3.0, flow rate = 1.0 mL/min, $23\text{ }^\circ\text{C}$, $\lambda = 254\text{ nm}$, retention times; *S* (major) 9.6 min, *R* (minor) 13.7 min, 94% ee): ^1H NMR (300 MHz, CDCl_3) δ 7.94 (d, $J = 7.1$ Hz, 2 H), 7.51-7.38 (m, 7 H), 4.66 (d, $J = 8.9$ Hz, 1 H), 4.26 (d, $J = 8.9$ Hz, 1 H), 3.29 (dd, $J=13.8, 25.1$ Hz, 2 H), 1.46 (s,

9 H) ppm; ^{13}C NMR (100 MHz, CDCl_3) δ 171.0, 165.0, 140.0, 131.8, 130.7, 129.3, 128.9, 128.5, 128.3, 127.0, 125.0, 82.5, 78.5, 73.2, 42.9, 27.8 ppm; IR (KBr) 2979, 1728, 1645, 1452, 1366, 1327, 1281, 1163, 1121, 1066, 1023, 980, 849, 695 cm^{-1} . MS (FAB) m/z 406 $[\text{M}+\text{H}]^+$.

4-(4-*tert*-Butyl-benzyl)-2-phenyl-2-oxazoline-4-carboxylic acid *tert*-butyl ester (8h); [R = 4-*tert*-butylbenzyl]

The enantioselectivity was determined by chiral HPLC analysis (DAICEL Chiralcel OD-H, hexane:2-propanol = 500:2.0, flow rate = 1.0 mL/min, $23\text{ }^\circ\text{C}$, λ = 254 nm, retention times; *S* (major) 9.3 min, *R* (minor) 10.3 min, >99% ee): ^1H NMR (300 MHz, CDCl_3) δ 8.05 (d, J = 7.1 Hz, 2 H), 7.51-7.36 (m, 3 H), 7.26-7.16 (m, 4 H), 4.67 (d, J = 8.9 Hz, 1 H), 4.31 (d, J = 8.9 Hz, 1 H), 3.22 (dd, J = 13.8, 38.6 Hz, 2 H), 1.46 (s, 9 H), 1.26 (s, 9 H) ppm; ^{13}C NMR (100 MHz, CDCl_3) δ 171.3, 164.5, 149.5, 132.5, 131.5, 129.9, 128.5, 128.1, 127.3, 125.0, 82.0, 78.9, 73.0, 42.8, 34.3, 31.2, 27.9 ppm; IR (KBr) 2964, 1723, 1644, 1454, 1365, 1278, 1161, 1091, 1025, 978, 847, 695 cm^{-1} . MS (FAB) m/z 394 $[\text{M}+\text{H}]^+$.

4-(4-Fluoro-benzyl)-2-phenyl-2-oxazoline-4-carboxylic acid *tert*-butyl ester (8i); [R = 4-fluorobenzyl]

The enantioselectivity was determined by chiral HPLC analysis (DAICEL Chiralcel OD-H, hexane:2-propanol = 500:2.0, flow rate = 1.0 mL/min, $23\text{ }^\circ\text{C}$, λ = 254 nm, retention times; *S* (major) 11.8 min, *R* (minor) 17.1 min, >99% ee): ^1H NMR (300 MHz, CDCl_3) δ 7.93 (d, J = 7.1 Hz, 2 H), 7.51-7.37 (m, 3 H), 7.26-7.21 (m, 2H), 6.96-6.88 (m, 2 H), 4.63 (d, J = 8.8 Hz, 1 H), 4.27 (d, J = 8.8 Hz, 1

H) 3.20 (dd, $J = 13.9, 21.2$ Hz, 2 H), 1.47 (s, 9 H) ppm; ^{13}C NMR (100 MHz, CDCl_3) δ 171.3, 164.7, 164.1 160.7, 131.9, 131.8, 131.6, 131.3, 128.5, 128.2, 127.1, 115.0, 114.8, 82.2, 78.6, 72.9, 42.3, 27.9 ppm; IR (KBr) 2978, 1726, 1644, 1510, 1366, 1279, 1224, 1160, 1101, 979, 846, 695 cm^{-1} . MS (FAB) m/z 356 $[\text{M}+\text{H}]^+$.

4-(4-Methoxy-benzyl)-2-phenyl-2-oxazoline-4-carboxylic acid *tert*-butyl ester (8j); [R = 4-methoxybenzyl]

The enantioselectivity was determined by chiral HPLC analysis (DAICEL Chiralcel OD-H, hexane:2-propanol = 500:3.0, flow rate = 1.0 mL/min, 23 °C, λ = 254 nm, retention times; *S* (major) 13.7 min, *R* (minor) 22.4 min, >99% ee): ^1H NMR (300 MHz, CDCl_3) δ 7.95 (d, $J = 7.3$ Hz, 2 H), 7.50-7.36 (m, 3 H), 7.17 (d, $J = 8.5$ Hz, 2 H), 6.75 (d, $J = 8.5$ Hz, 2 H), 4.65 (d, $J = 8.8$ Hz, 1 H), 4.30 (d, $J = 8.8$ Hz, 1 H), 3.72 (s, 3 H), 3.19 (dd, $J = 13.9, 17.8$ Hz, 2 H), 1.47 (s, 9 H) ppm; ^{13}C NMR (100 MHz, CDCl_3) δ 171.5, 164.5, 158.4, 131.5, 131.3, 128.6, 128.5, 128.2, 127.5, 113.5, 82.1, 78.9, 72.7, 55.1, 42.2, 27.9 ppm; IR (KBr) 2929, 1726, 1643, 1513, 1455, 1365, 1252, 1160, 1091, 1033, 978, 846, 695 cm^{-1} . MS (FAB) m/z 368 $[\text{M}+\text{H}]^+$.

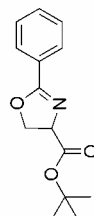
4-Naphthalene-2-ylmethyl-2-phenyl-2-oxazoline-4-carboxylic acid *tert*-butyl ester (8k); [R = 2-naphthylmethyl]

The enantioselectivity was determined by chiral HPLC analysis (DAICEL Chiralcel OD-H, hexane:2-propanol = 500:2.0, flow rate = 1.0 mL/min, 23 °C, λ = 254 nm, retention times; *S* (major) 24.8 min, *R* (minor) 31.6 min, 97% ee)

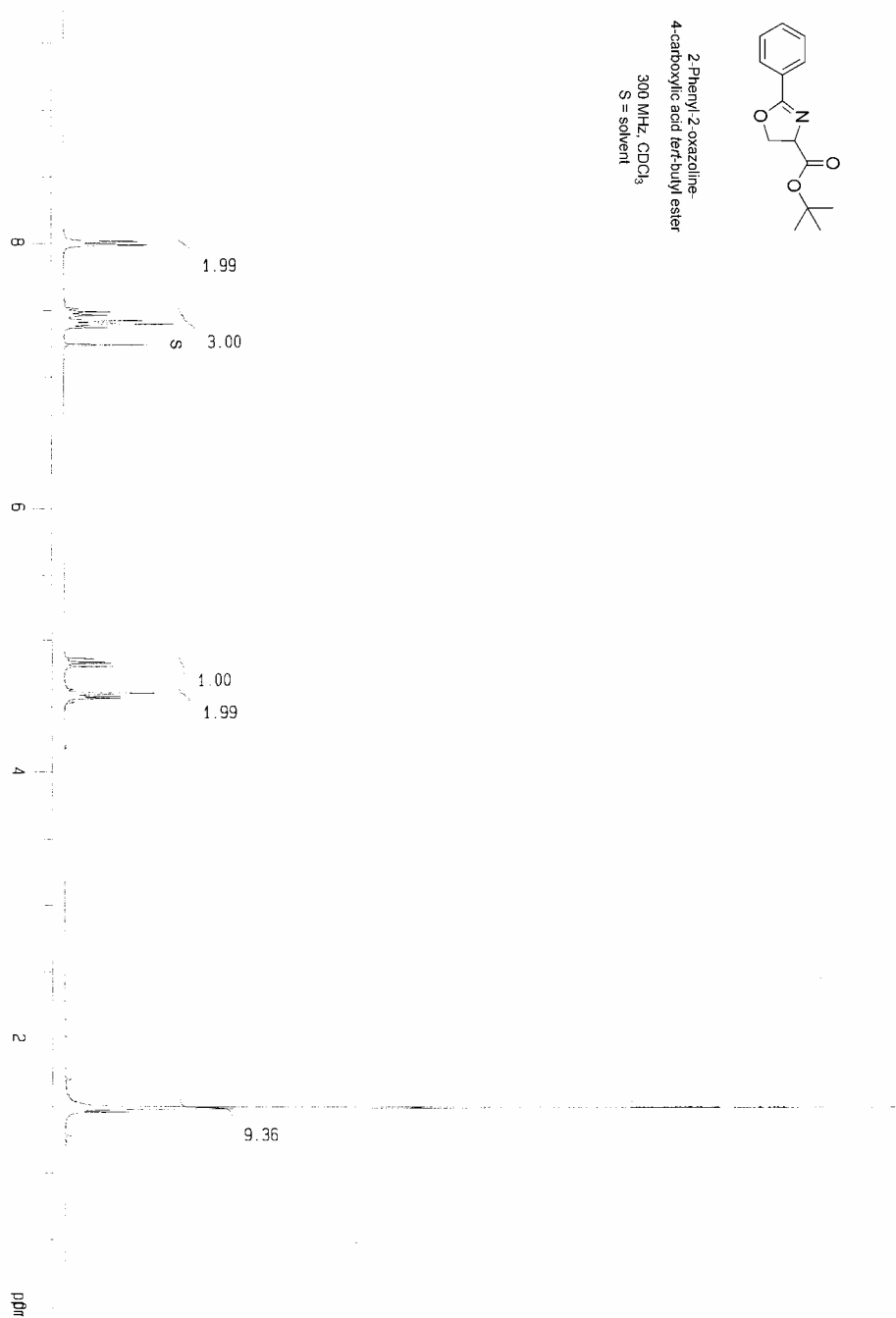
ppm: ^1H NMR (300 MHz, CDCl_3) δ 7.95 (d, J = 7.3 Hz, 2 H), 7.76-7.67 (m, 4 H), 7.50-7.35 (m, 6 H), 4.70 (d, J = 8.9 Hz, 1 H), 4.38 (d, J = 8.9 Hz, 1 H), 3.43 (s, 2 H), 1.48 (s, 9 H) ppm; ^{13}C NMR (100 MHz, CDCl_3) δ 171.4, 164.7, 133.3, 133.2, 132.3, 131.5, 129.0, 128.6, 128.5, 128.2, 127.6, 127.5, 127.4, 127.2, 125.8, 125.5, 82.2, 78.9, 72.8, 43.2, 27.9 ppm; IR (KBr) 2977, 1726, 1643, 1452, 1365, 1280, 1159, 1092, 978, 846, 752, 696 cm^{-1} . MS (FAB) m/z 388 $[\text{M}+\text{H}]^+$.

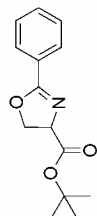
Procedure for deprotection. (S)-(+)- α -Benzylserine.

To an ethanol solution (1.5 mL) of 4-Benzyl-2-phenyl-2-oxazoline-4-carboxylic acid *tert*-butyl ester **8e** (500 mg, 1.48 mmol) was added 6N-HCl (1.5 mL) and the reaction mixture was refluxed for 24 h. After the solvent was removed *in vacuo*, the residue was purified by column chromatography (5% aq.- NH_4OH) using ion-exchange resin (Dowex[®] 50WX8-100) to give (S)-(+)- α -benzylserine as a white solid (365 mg, 98%). $[\alpha]_{\text{D}}^{20} = +16.4$ (c 0.89, H_2O) [lit. $[\alpha]_{\text{D}}^{20} = +16.4$ (c 0.81, H_2O)]. Physical and spectral properties were consistent with the literature values.^{4m,17}



2-Phenyl-2-oxazoline-
4-carboxylic acid tert-butyl ester
300 MHz, CDCl₃
S = solvent

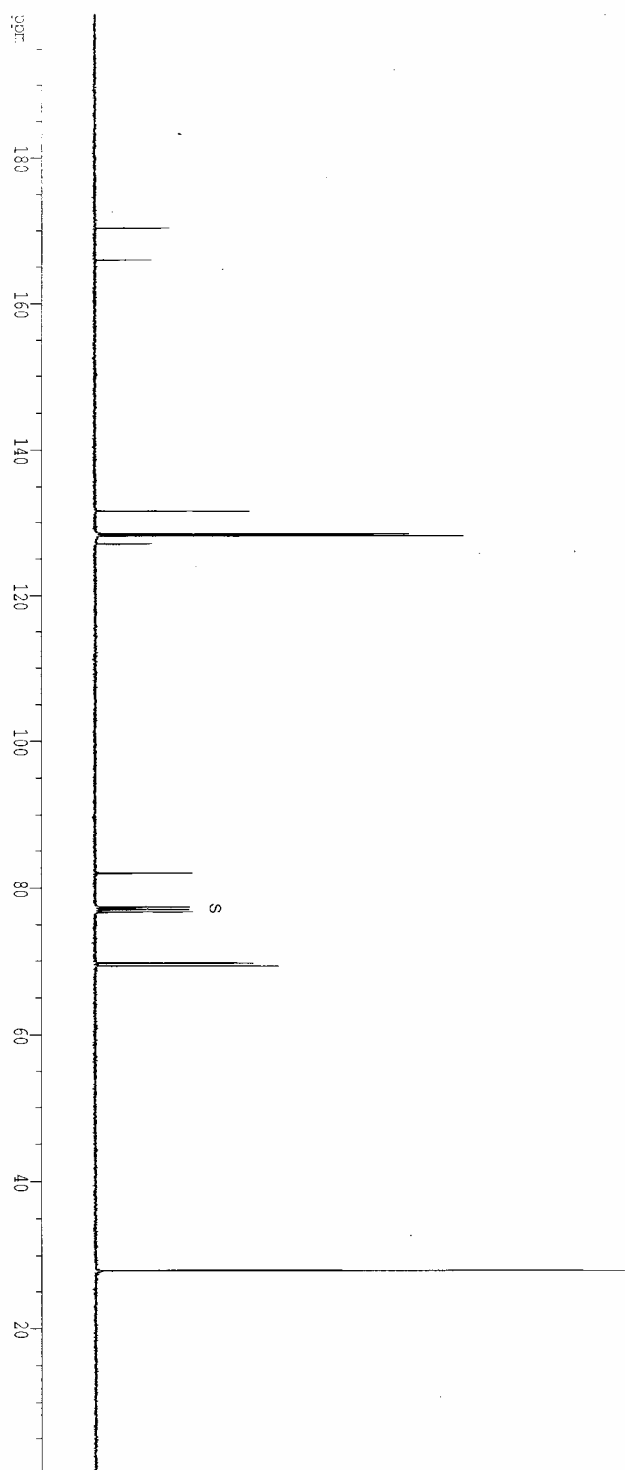


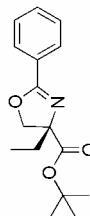


2-Phenyl-2-oxazoline-
4-carboxylic acid tert-butyl ester

400 MHz, CDCl₃

S = solvent



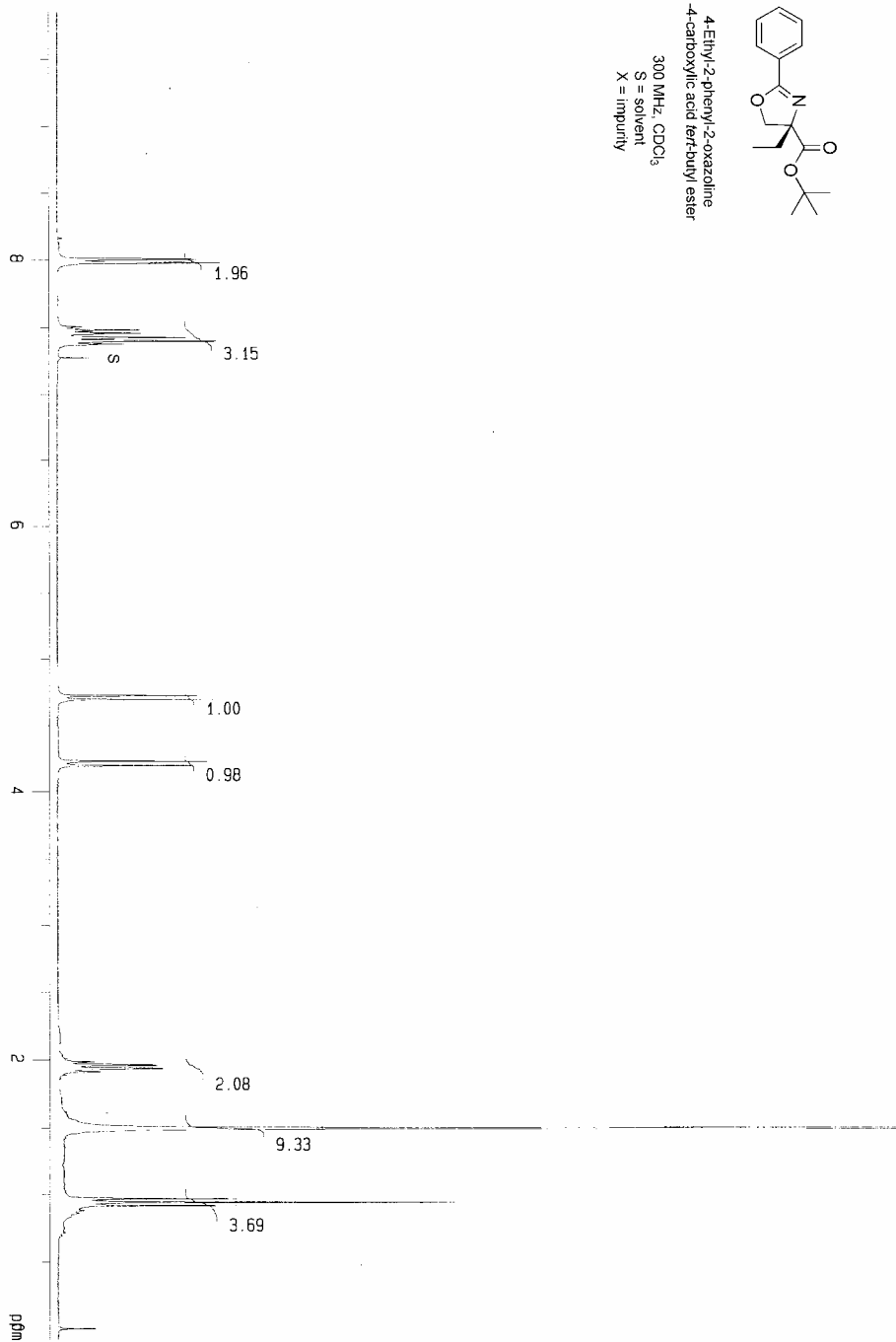


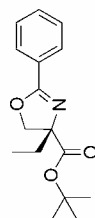
4-Ethyl-2-phenyl-2-oxazoline-4-carboxylic acid tert-butyl ester

300 MHz, CDCl₃

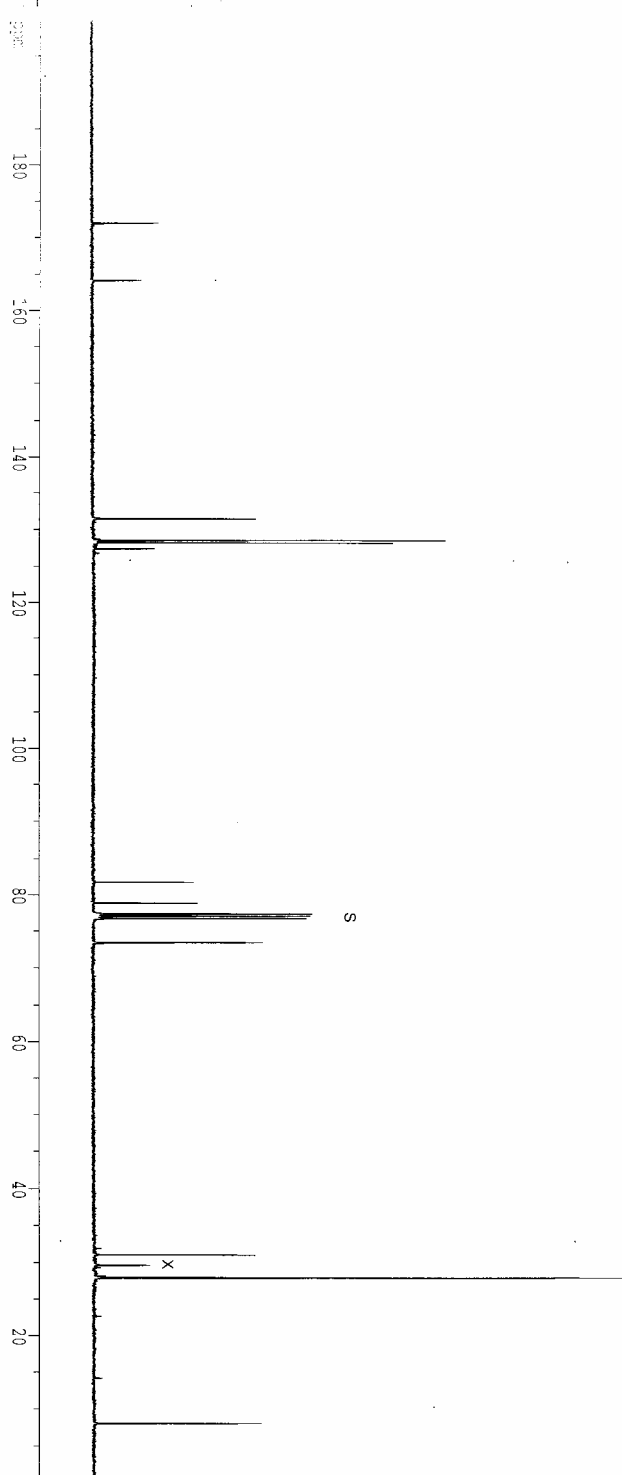
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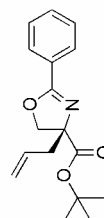
X = impurity





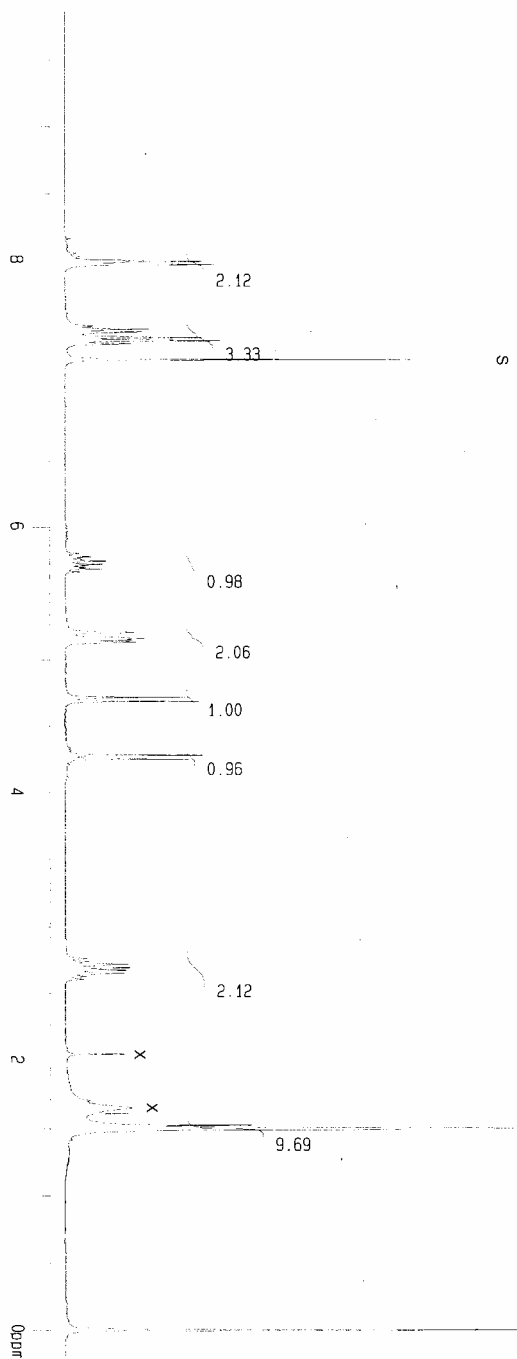
4-Ethyl-2-phenyl-2-oxazoline
4-carboxylic acid *tert*-butyl ester
400 MHz, CDCl₃
S = solvent
X = impurity

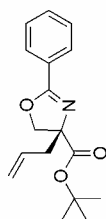




4-allyl-2-phenyl-2-oxazoline-
4-carboxylic acid tert-butyl ester

300 MHz, CDCl₃
S = solvent
X = impurity



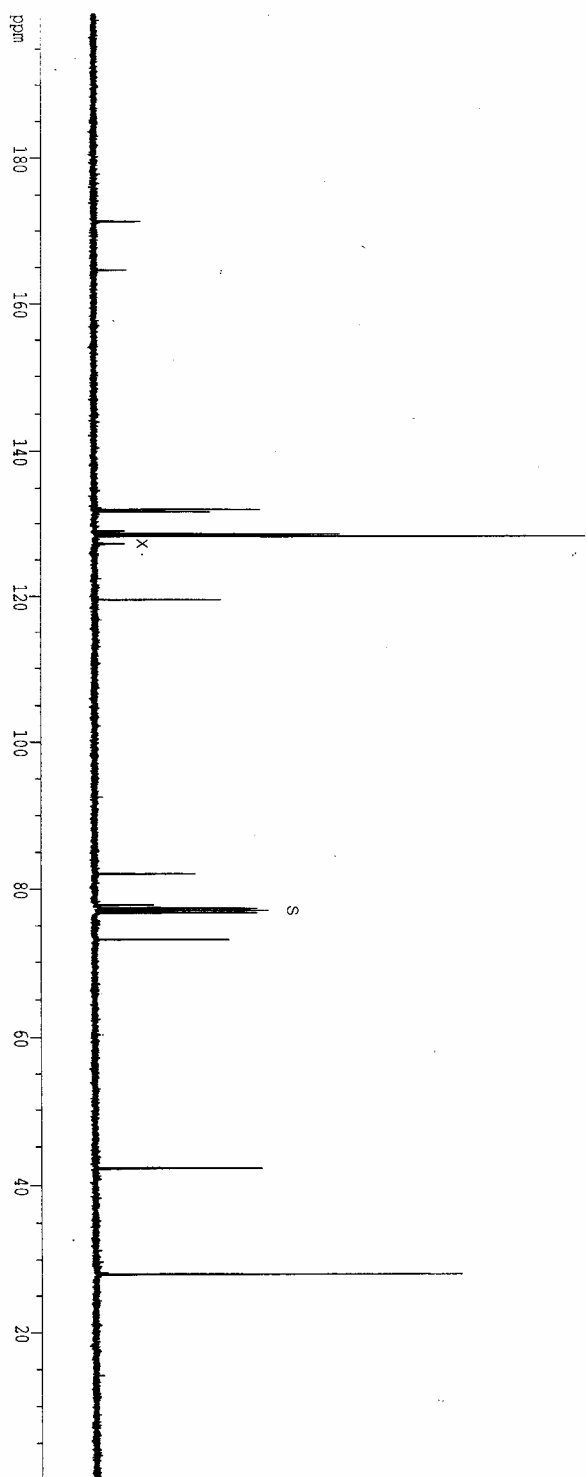


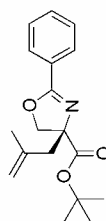
4-Allyl-2-phenyl-2-oxazoline-
4-carboxylic acid *tert*-butyl ester

400 MHz, CDCl₃

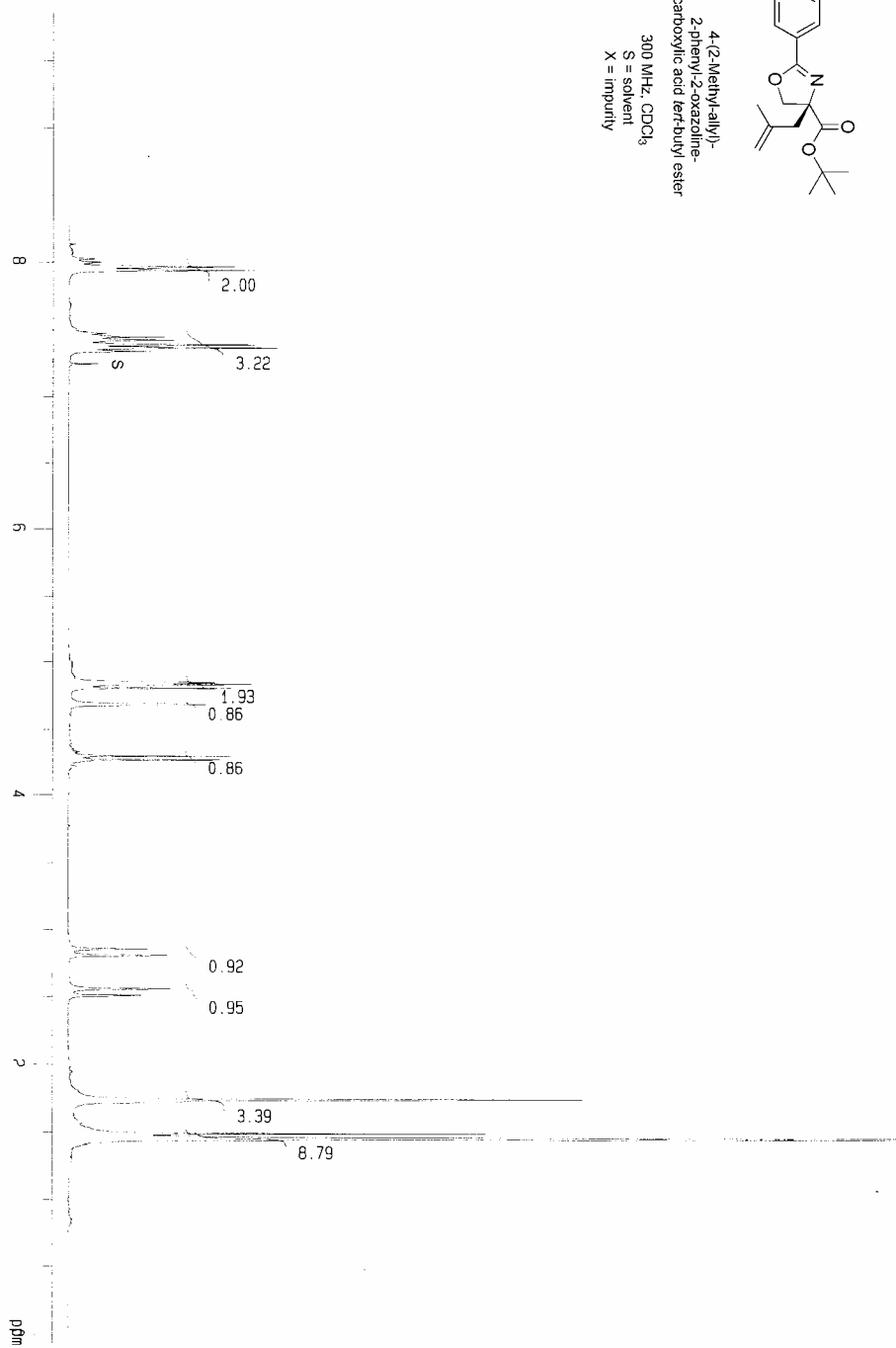
S = solvent

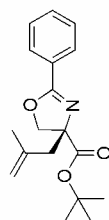
X = impurity





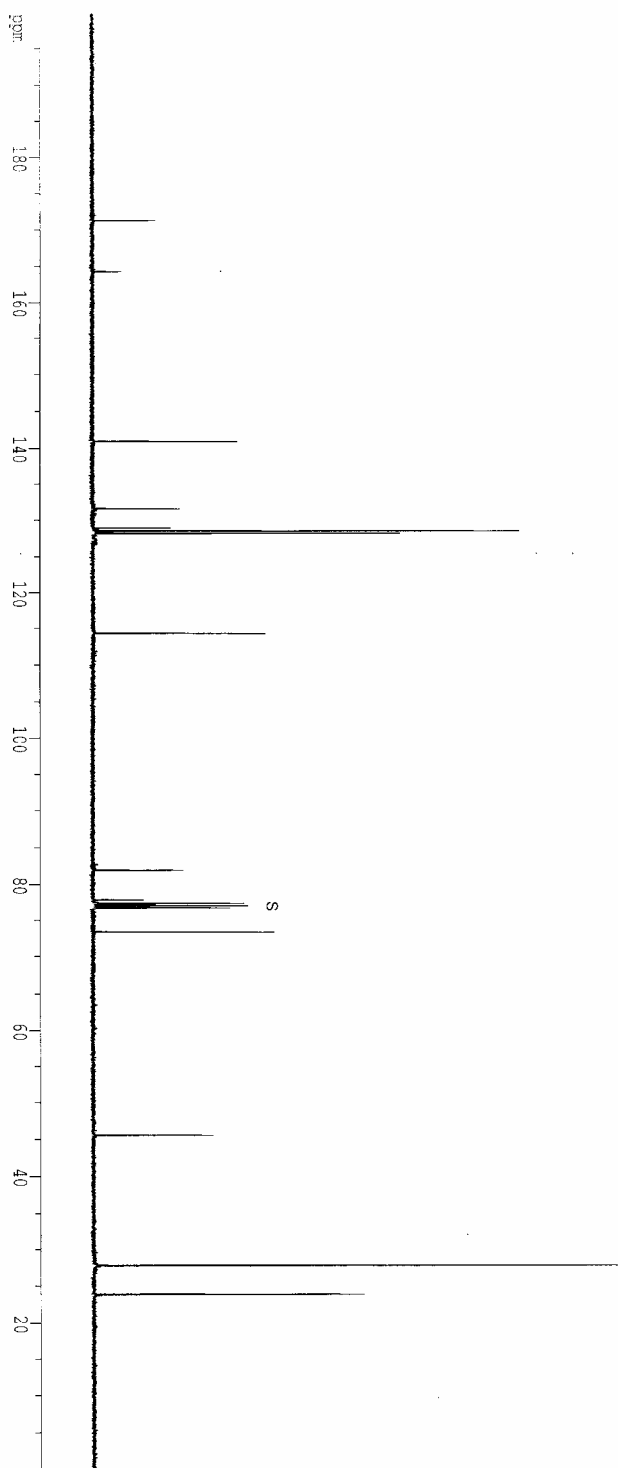
4-(2-Methyl-allyl)-
2-phenyl-2-oxazoline-
4-carboxylic acid tert-butyl ester
300 MHz, CDCl₃
S = solvent
X = impurity

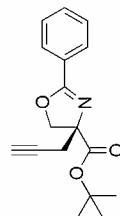




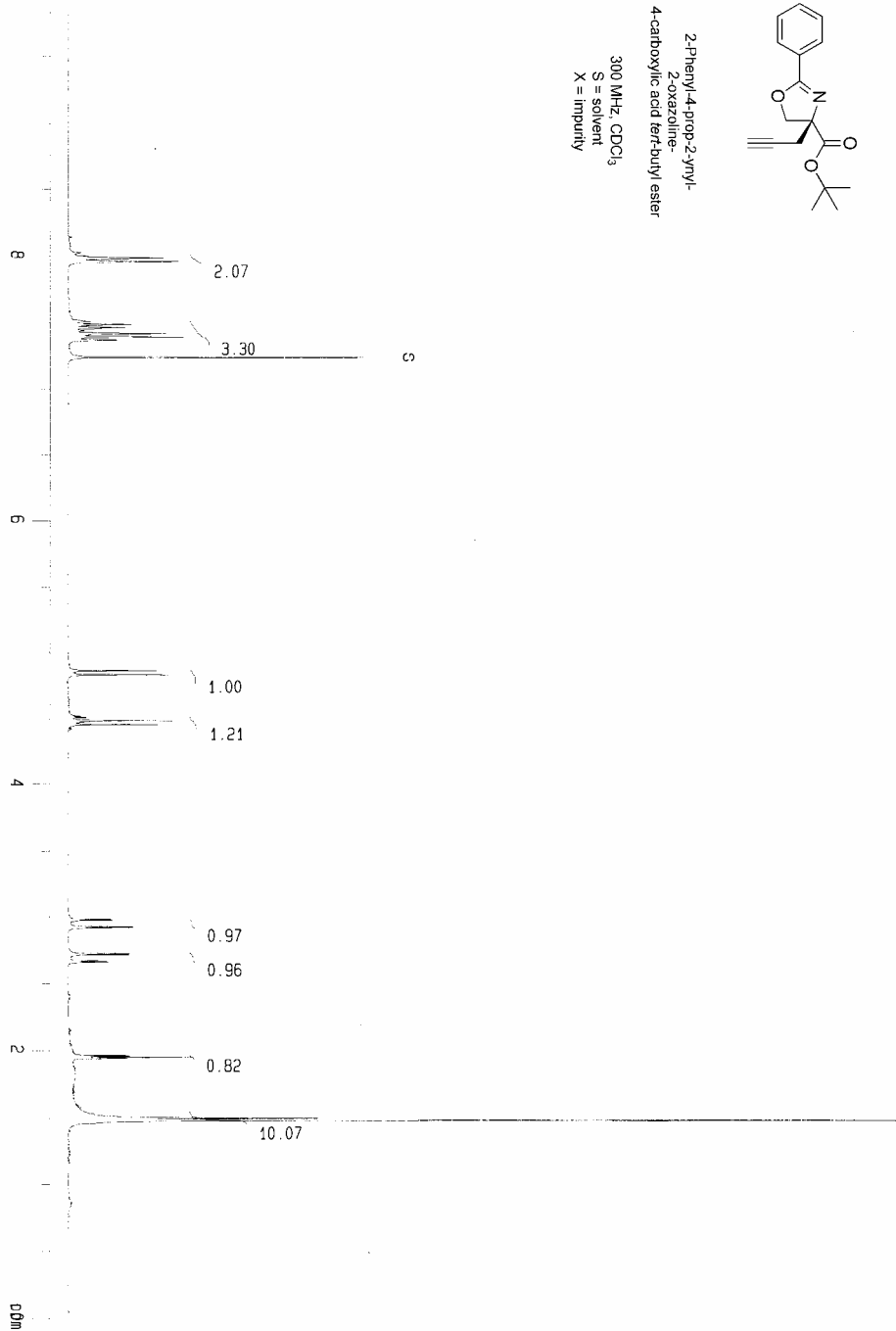
4-(2-Methyl-allyl)-
2-phenyl-2-oxazoline-
4-carboxylic acid *tert*-butyl ester

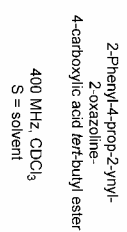
400 MHz, CDCl₃
S = solvent

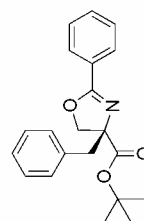




2-phenyl-4-prop-2-ynyl-
2-oxazoline-
4-carboxylic acid tert-butyl ester
300 MHz, CDCl₃
S = solvent
X = impurity

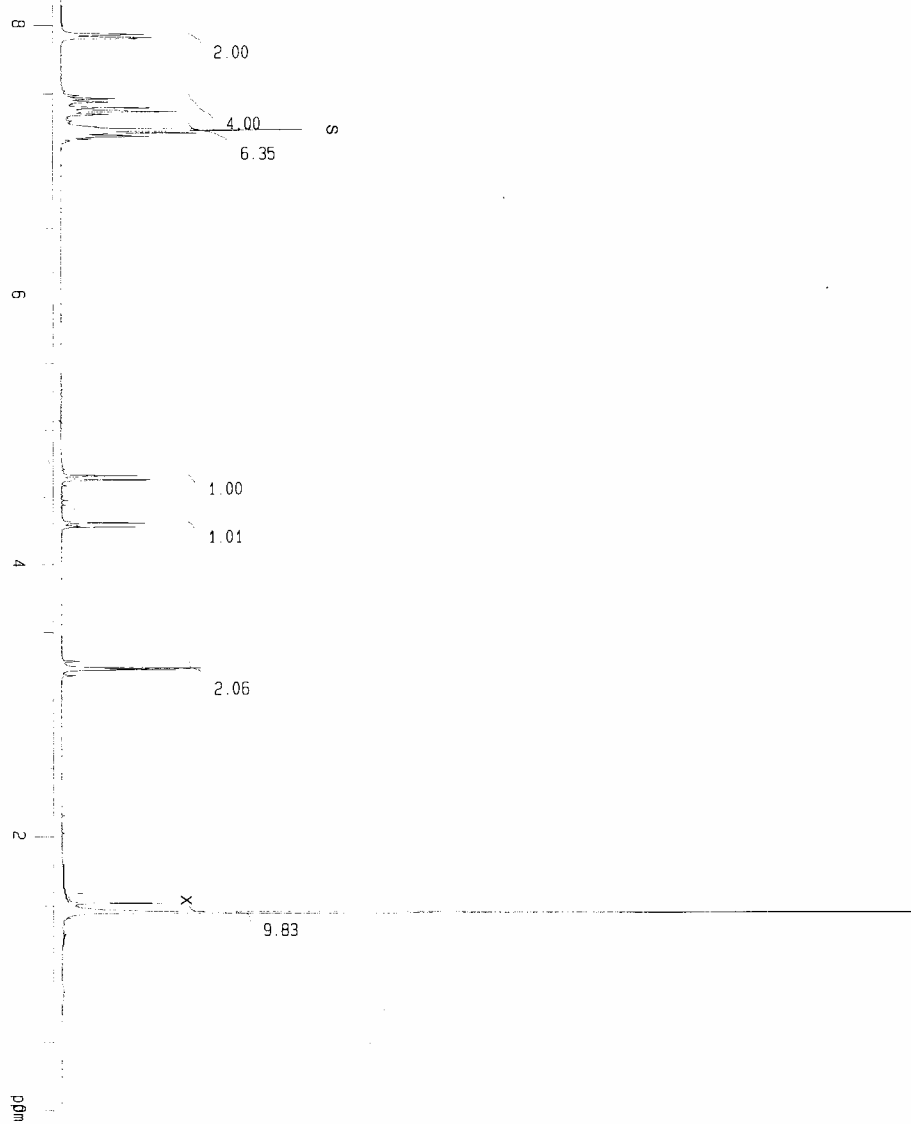


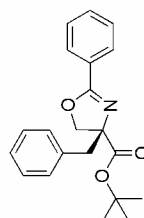




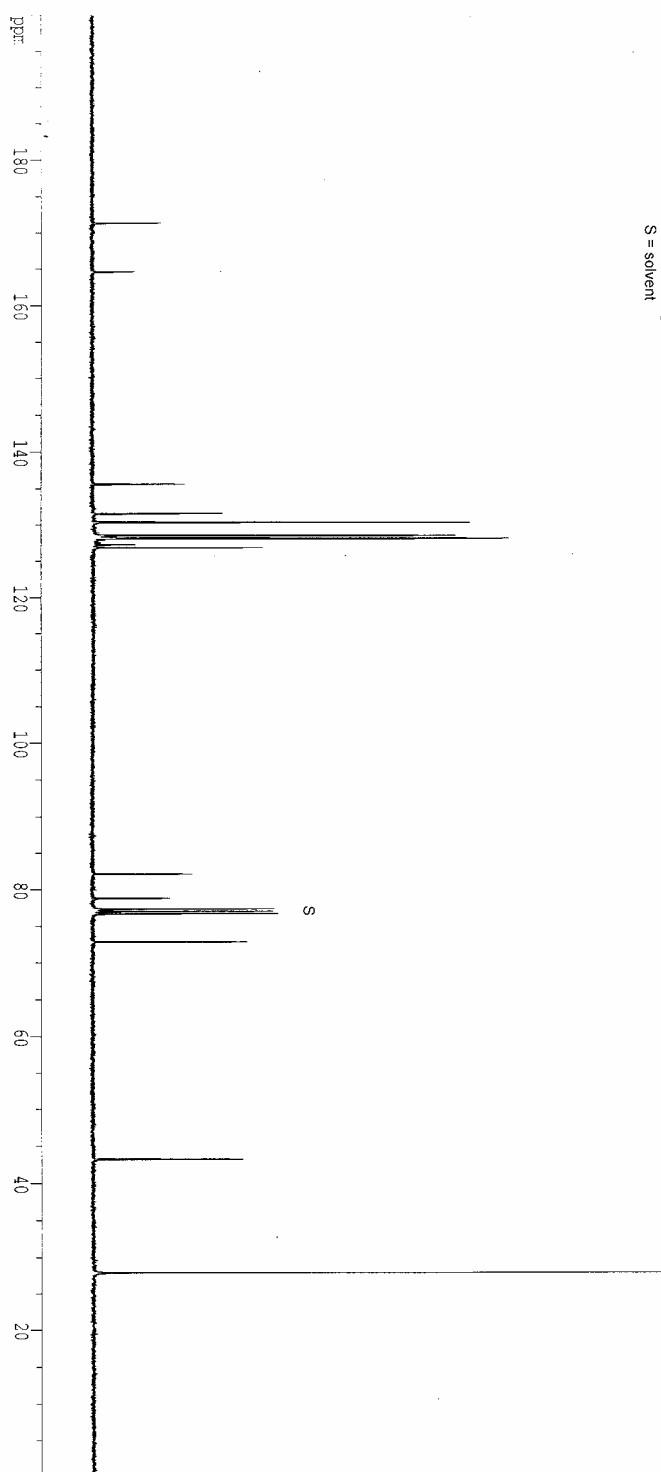
4-Benzyl-2-phenyl-2-oxazoline-4-carboxylic acid tert-butyl ester

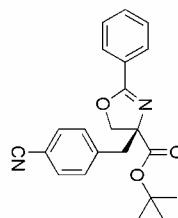
300 MHz, CDCl₃
 S = solvent
 X = impurity





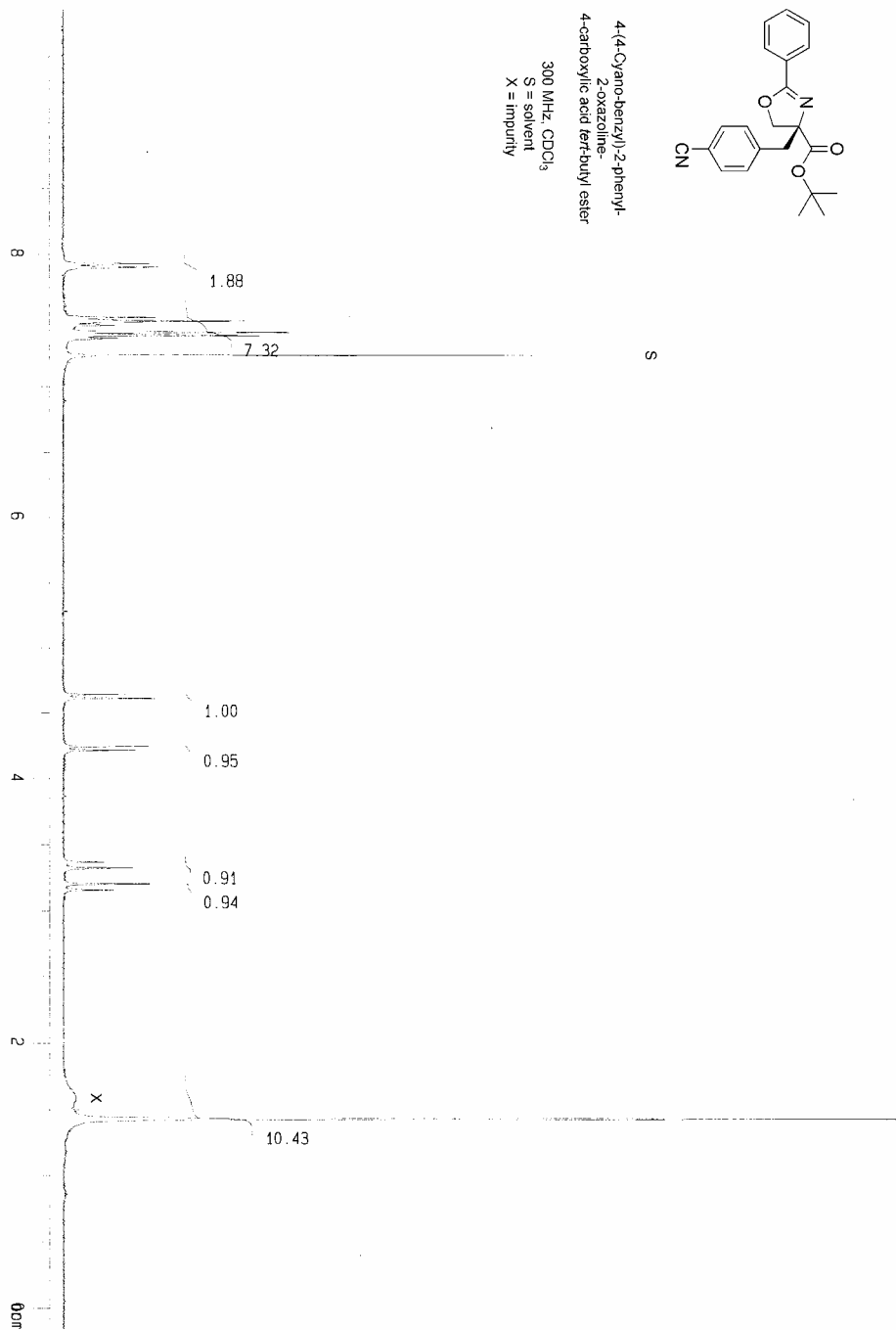
4-Benzyl-2-phenyl-2-oxazoline-
4-carboxylic acid *tert*-butyl ester
400 MHz, CDCl₃
S = solvent

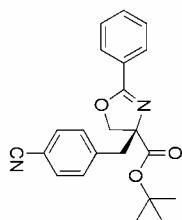




4-(4-Cyano-benzyl)-2-phenyl-
2-oxazoline-
4-carboxylic acid tert-butyl ester

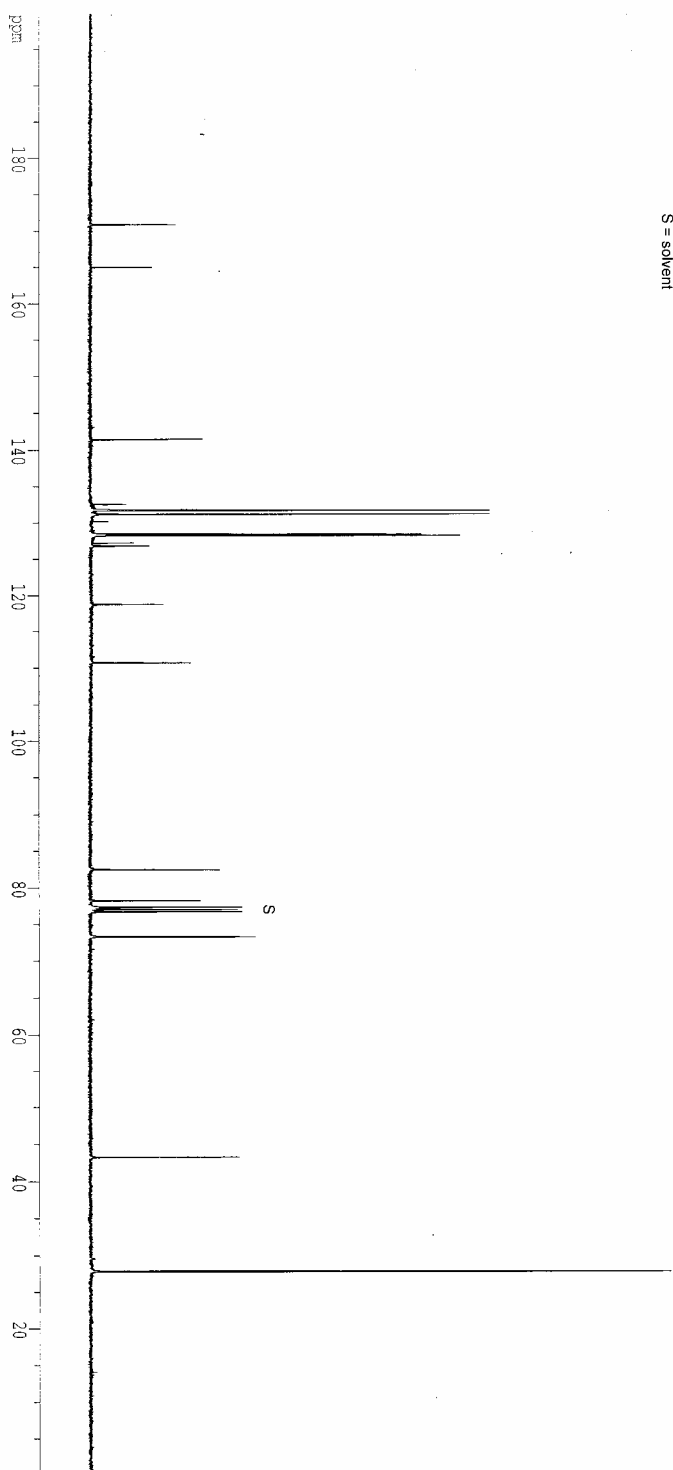
300 MHz, CDCl₃
S = solvent
X = impurity

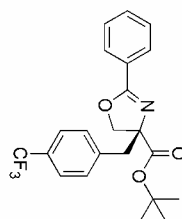




4-(4-Cyano-benzyl)-2-phenyl-
2-oxazoline-
4-carboxylic acid tert-butyl ester

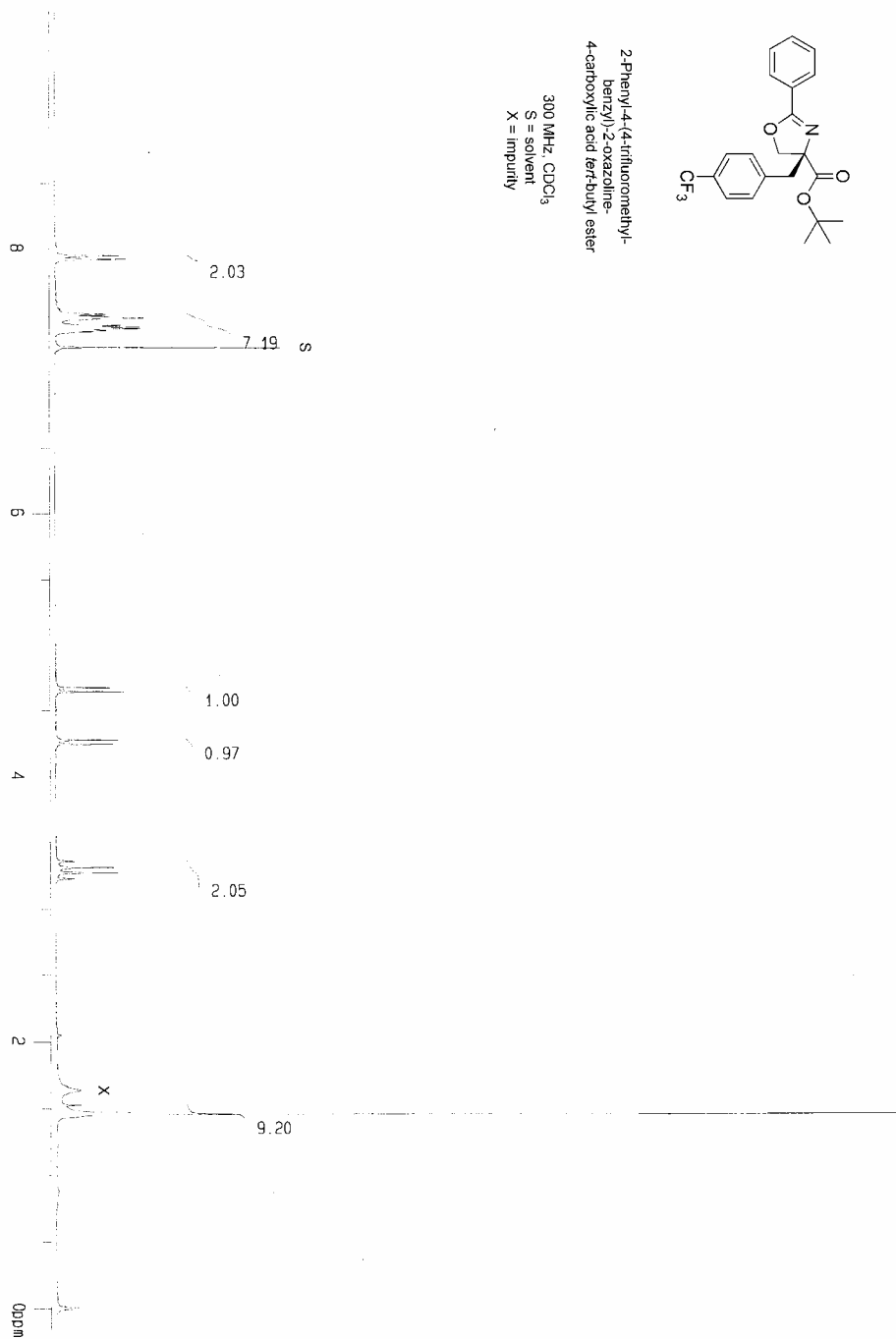
400 MHz, CDCl₃
S = solvent

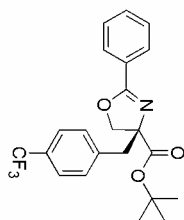




2-Phenyl-4-(4-trifluoromethyl-
benzyl)-2-oxazoline-
4-carboxylic acid tert-butyl ester

300 MHz, CDCl₃
S = solvent
X = impurity

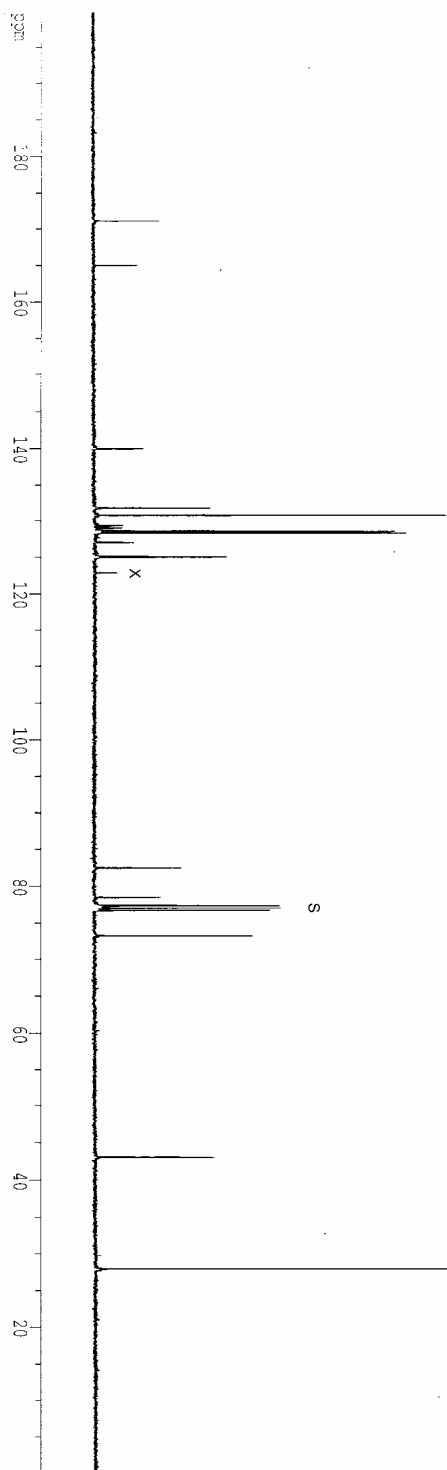


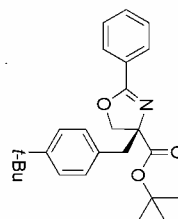


2-Phenyl-4-(4-trifluoromethyl-
benzyl)-2-oxazoline-
4-carboxylic acid *tert*-butyl ester

400 MHz, CDCl₃

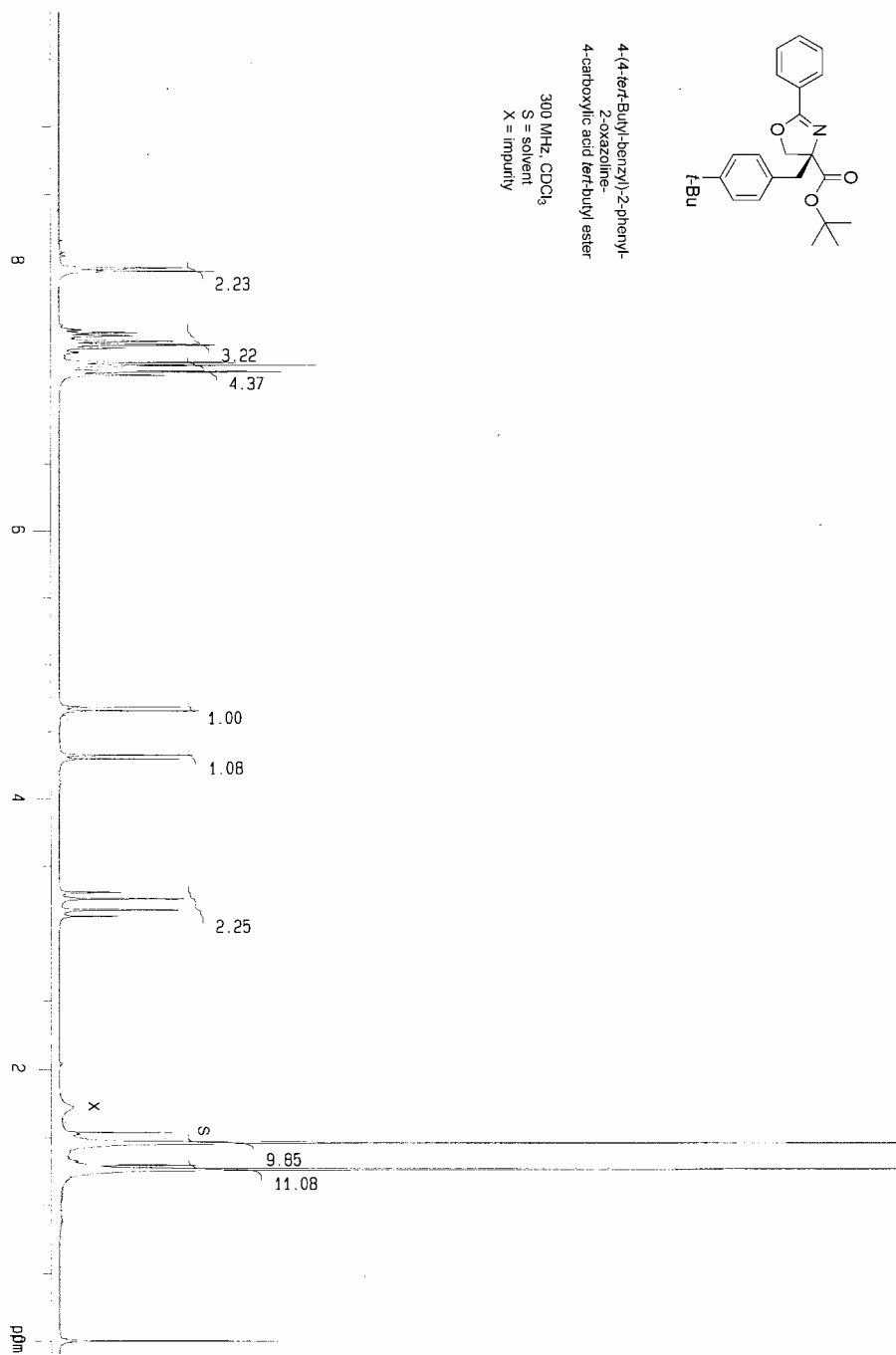
S = solvent
X = impurity

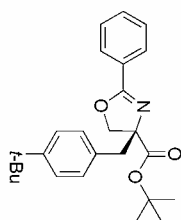




4-(4-(*tert*-Bu)-benzyl)-2-phenyl-
2-oxazoline-
4-carboxylic acid *tert*-butyl ester

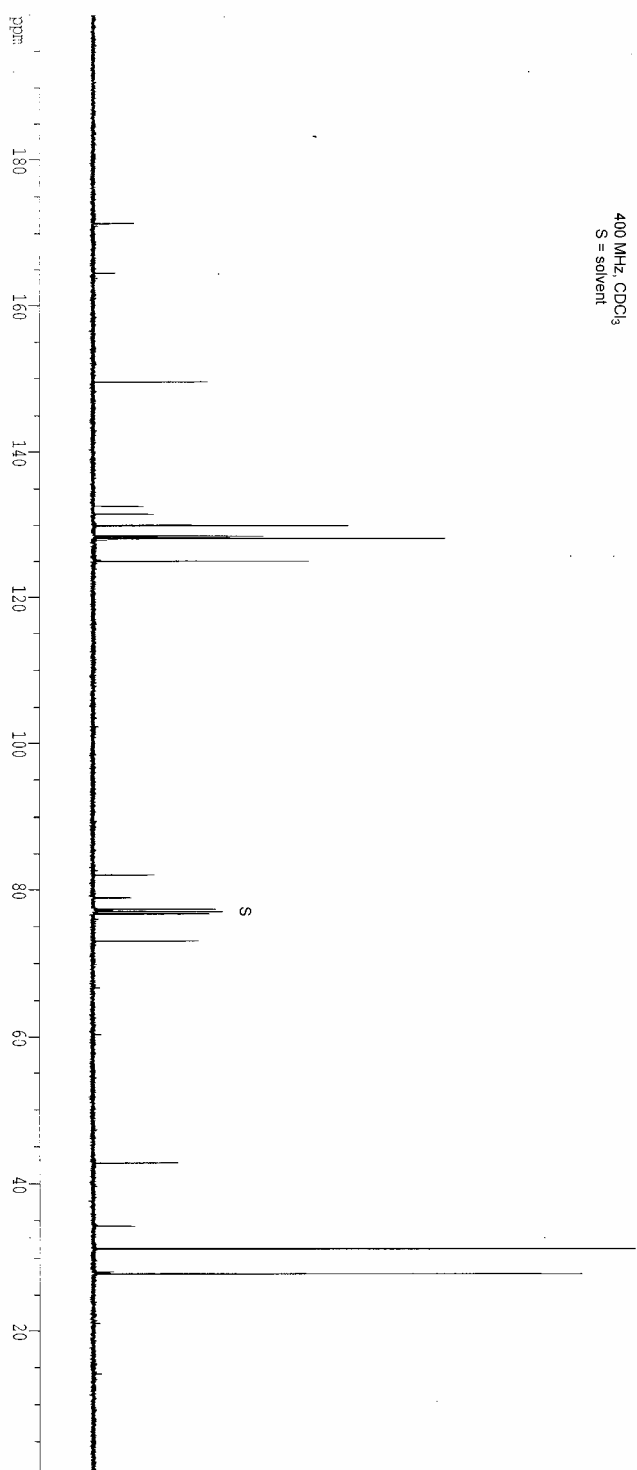
300 MHz, CDCl₃
S = solvent
X = impurity

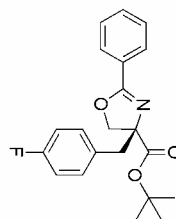




4-(4-*tert*-butyl-benzyl)-2-phenyl-
2-oxazoline-
4-carboxylic acid *tert*-butyl ester

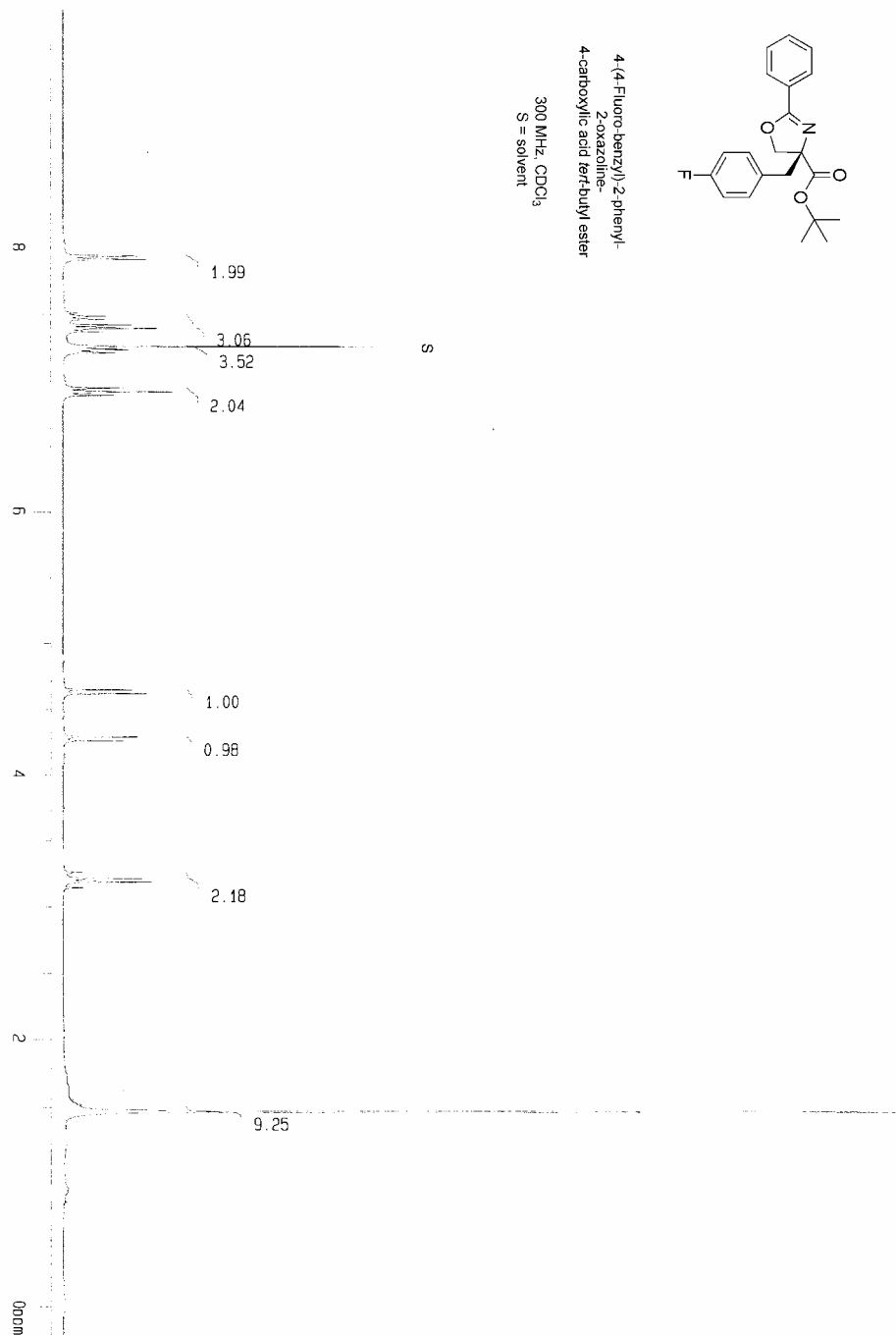
400 MHz, CDCl₃
S = solvent

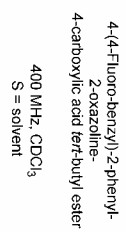


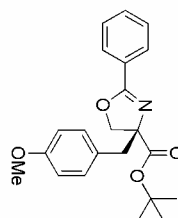


4-(4-fluoro-benzyl)-2-phenyl-
2-oxazoline-
4-carboxylic acid *tert*-butyl ester

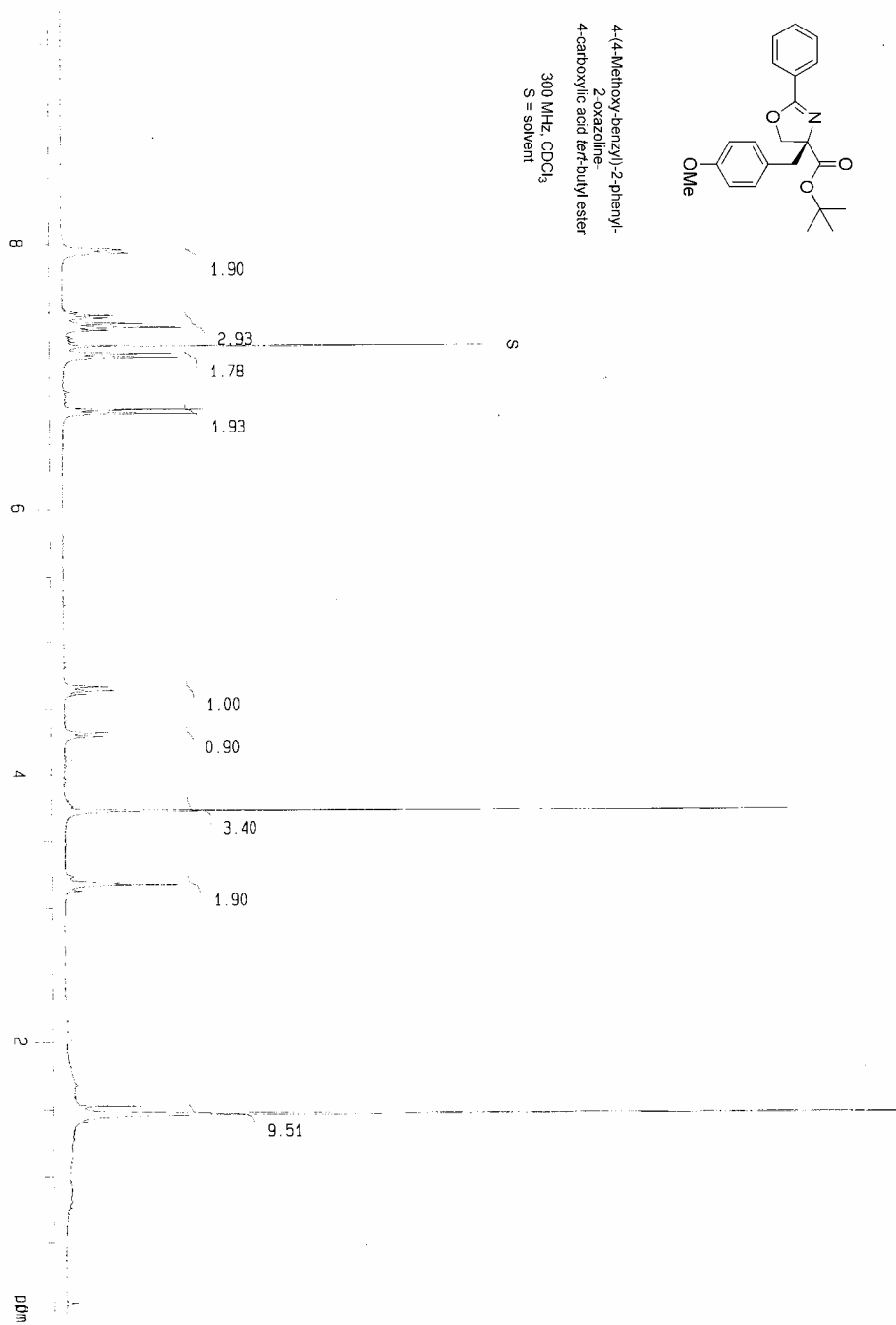
300 MHz, CDCl₃
S = solvent

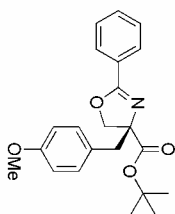






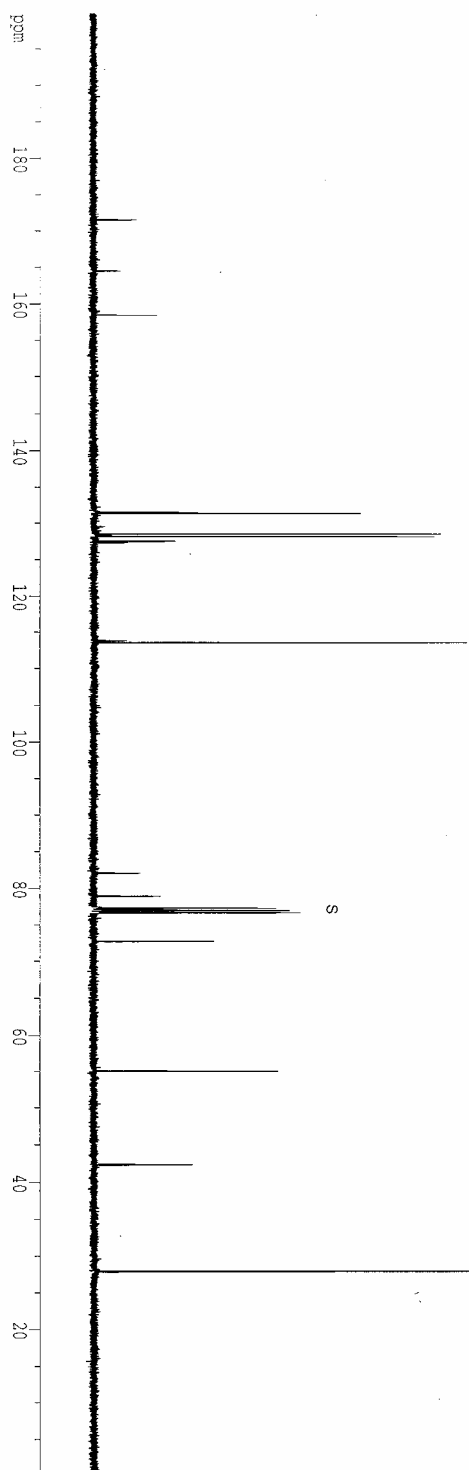
4-(4-Methoxy-benzyl)-2-phenyl-
2-oxazoline-
4-carboxylic acid tert-butyl ester
300 MHz, CDCl₃
S = solvent

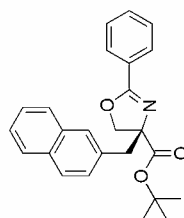




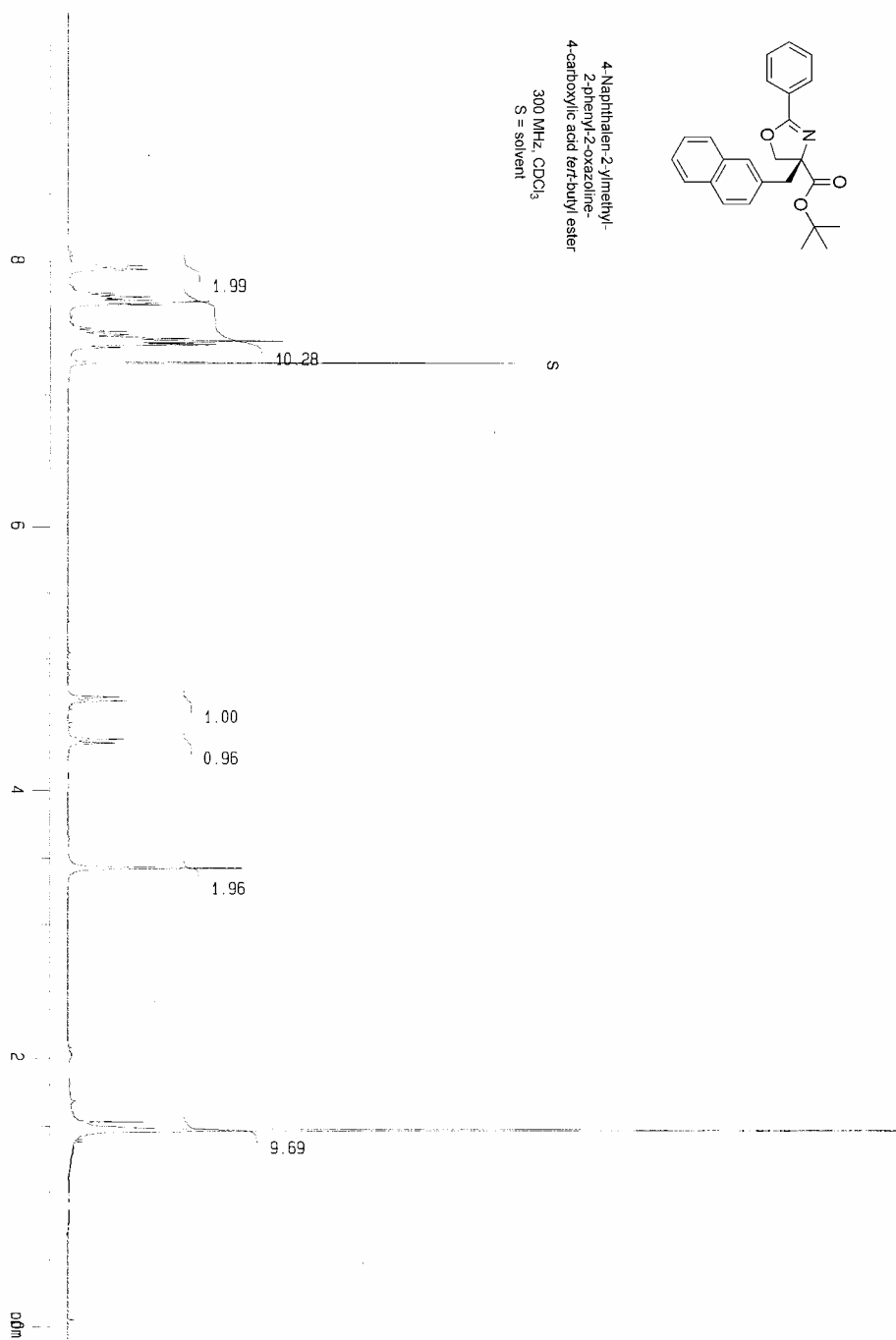
4-(4-Methoxy-benzyl)-2-phenyl-
4-carboxylic acid tert-butyl ester-
2-oxazoline

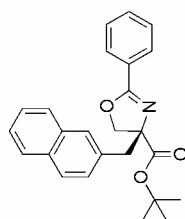
400 MHz, CDCl₃
S = solvent





4-Naphthalen-2-ylmethyl-
2-phenyl-2-oxazoline-
4-carboxylic acid tert-butyl ester
300 MHz, CDCl₃
S = solvent





4-Naphthalen-2-ylmethyl-
2-phenyl-2-oxazoline-
4-carboxylic acid tert-butyl ester
400 MHz, CDCl_3
S = solvent

