Supporting Information

"O-Alkenylhydroxylamines: a New Concept for Functionalization"

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General Procedure for the Preparation of *O*-Homoallyl Hydroxylamines

A solution of DEAD (1.2 eq.) in THF (0.25 M) was added dropwise to a solution of the homoallylic alcohol, triphenylphosphine (1.2 eq.) and N-hydroxyphthalimide (1.2 eq.) in THF (0.25 M) under nitrogen at 0°C. The mixture was allowed to warm to room temperature and stirred for four hours. THF was then evaporated and the residue was dissolved in dichloromethane (*ca* 0.3 M). Hydrazine hydrate (3 eq.) was added and the mixture was stirred at room temperature for two hours. The mixture was filtered through celite, washing with dichloromethane, and the dichloromethane was removed under reduced pressure. The residue was preabsorbed on silica gel and purified by flash chromatography on silica gel, eluting with 5 - 15% ethyl acetate/ hexane.

hydroxylamine 3a

Colorless oil, R_f = 0.27 (25% EtOAc : Hexane)

m/z (EI) 131 (30), 91 (100)

IR (Neat) 3316, 3031, 2893, 1641, 1494, 1455, 1360, 1178

^{1}H (200 MHz, $CDCl_3$) 2.42 (dt, J 14.3, 7.3, 1H, CH_2), 2.60 (dt, J 14.3, 7.3, 1H, CH_2), 4.56 (dd, J 6.2, 7.7, 1H, OCH), 5.08 (m, 2H, $=CH_2$), 5.25 (s, 2H, NH_2), 5.76 (ddt, J 7, 10.2, 17.2, 1H, =CH), 7.35 (m, 5H, Ph)

^{13}C (50 MHz, $CDCl_3$) 40.6, 86.6, 117.1, 126.7, 127.8, 128.5, 134.4, 141.0.

hydroxylamine 3b

Colorless oil, R_f = 0.41 (25% EtOAc: Hexane)

m/z (EI) 130 (M^++H) (19), 79 (45)

IR (Neat) 3363, 2947, 1557, 1420, 1396

^{1}H (200 MHz, $CDCl_3$) 0.89 (d, J 7, 3H, Me), 0.91 (d, J 7, 3H, Me), 1.9 (m, 1H, Me_2CH), 2.27 (m, 2H, CH_2), 3.35 (dt, J 5.5, 6.6, 1H, OCH), 5.08 (m, 2H, $=CH_2$), 5.26 (brs, 2H, ONH_2), 5.87 (ddt, 17.2, 10.2, 7, 1H, =CH)

^{13}C (50 MHz, $CDCl_3$) 18.0, 18.3, 29.1, 33.6, 88.0, 115.8, 135.4.

hydroxylamine 3c

Colorless oil, R_f = 0.46 (25% EtOAc: Hexane)

IR (Neat) 3315, 2929, 1477, 1372, 1256, 1112

^{1}H (200 MHz, $CDCl_3$) 0.07 (s, 6H, Me_2Si), 0.91 (s, 9H, *t*-Bu), 2.31 (m, 2H, CH_2), 3.68 (m, 3H, OCH_2, OCH), 4.82 (brs, 2H, ONH_2), 5.10 (m, 2H, $=CH_2$), 5.84 (ddt, 17.2, 10.3, 7, 1H, =CH)

^{13}C (50 MHz, $CDCl_3$) -5.16, 13.71, 17.81, 25.4, 34.0, 63.2, 83.5, 116.3, 134.4

hydroxylamine 4a

Yellow oil , R_f = 0.27 (25% EtOAc : Hexane)

m/z (EI) 222 (14) (M^+ +H), 131 (100) , 91 (22)

IR (Neat) 3279, 2956 , 1736, 1643 cm^{-1}

^{1}H (200 MHz, $CDCl_3$) 2.4 - 2.8 (m, 2H, CH_2), 3.70 (s, 3H, OMe), 4.81 (t, J 7.0, 1H, PhCH), 5.08 (m, 2H, $=CH_2$), 5.78 (m, 1H, CH=), 7.10 (s, 1H, NH), 7.30 (m, 5H, Ph)

^{13}C (50 MHz, $CDCl_3$) 39.5, 52.6, 87.4, 117.5, 127.2, 128.1, 128.4, 128.5, 139.5, 133.6, 157.6.

Preparation of Ns- hydroxylamine 5a

p-Nosyl chloride (204 mg, 0.92 mmole) was added to a solution of the hydroxylamine **3a** (100 mg, 0.61 mmole) in 1:1 dichloromethane/ water (4 ml). Sodium carbonate (97 mg, 0.92 mmole) was added and the mixture was stirred

overnight. The layers were separated and the organic layer was washed with brine, dried (Na_2SO_4) and evaporated. The residue was purified by flash chromatography on silica gel (1.5 g) eluting with 5 and 10% ethyl acetate/hexane to give the sulfonamide (150 mg, 70%).

Hydroxylamine 5a

Yellow solid, mp 132 - 133 °C, R_f = 0.29 (25% EtOAc : Hexane)

m/z (EI) 131 (60), 91 (100)

IR (KBr) 3236, 3112, 2931, 1606, 1348, 1170

^1H (200 MHz, $CDCl_3$) 2.55 (m, 2H, CH_2), 4.99 (m, 2H, $=CH_2$), 5.78 (m, 1H, =CH), 6.98 (s, 1H, NH), 7.30 (m, 5H, Ph), 8.08 (d, J 8.4, 2H, $SO_2C_6H_4NO_2$), 8.35 (d, J 8.4, 2H, $SO_2C_6H_4NO_2$)

^{13}C (50 MHz, $CDCl_3$) 39.1, 88.2, 117.5, 123.6, 126.6, 128.1, 129.6, 138, 133.1.

General Procedure for the Preparation of CBZ derivatives

N-Benzyloxycarbonyloxysuccinimide (1.2 eq.) was added to a solution (ca 0.05 M) of the hydroxylamine in dichloromethane/ water (1:1). Sodium hydrogen carbonate (2 eq.) was added and the mixture was stirred overnight. The layers were separated and the organic layer was washed with brine, dried (Na_2SO_4) and evaporated. The residue was purified by flash chromatography on silica gel eluting with 5 and 10% ethyl acetate/hexane.

hydroxylamine 6a

Yellow Oil, R_f = 0.31 (25% EtOAc : Hexane)

m/z (HFAB) found: 298.1466 (M^++ H) $C_{18}H_{20}NO_3$ requires: 298.1365

(FAB) 298 (7), (M^++ H), 131 (70), 91 (100)

IR (Neat) 3280, 3033, 2945, 1724 cm^{-1}

1H (200 MHz, $CDCl_3$) 2.48 (dtt, J 14.3, 7, 1.5, 1H, CH_2), 2.76 (dtt, J 14.3, 7, 1.5, 1H, CH_2), 4.81 (t, J 6.7, 1H, PhCH), 5.02 (m, 2H, $=CH_2$), 5.14 (2d, J 12, 2H, CH_2Ph), 5.78 (ddt, J 17.2, 10.3, 7, 1H, =CH), 7.15 (s, 1H, NH), 7.33 (m, 10H, Ph)

^{13}C (50 MHz, $CDCl_3$) 39.6, 67.4, 87.4, 117.5, 127.2, 128.2 128.4, 128.5, 135.5, 139.4, 133.6, 156.9.

hydroxylamine 6b

Yellow oil, R_f = 0.39 (25% EtOAc : Hexane)

m/z (HFAB) found: 264.1600 (M^++ H) $C_{15}H_{21}NO_3$ requires: 263.1521

(FAB) 264.1 (M^++ H), 91.0 (100)

IR (Neat) 3288, 3068, 2962, 1718 cm^{-1}

^1H (200 MHz, $CDCl_3$) 0.92 (d, J 7, 3H, Me), 0.95 (d, J 7, 3H, Me), 1.94 (m, 1H, Me_2CH), 2.32 (m, 2H, CH_2), 3.62 (q, J 5.6, 1H, OCH), 5.04 - 5.14 (m, 2H, $=CH_2$), 5.16 (s, 2H, CH_2Ph), 5.89 (ddt, J 17.2, 10.2, 7, 1H, =CH), 7.30 (m, 5H, Ph, NH)

^{13}C (50 MHz, $CDCl_3$) 18.0, 18.3, 29.6, 33.9, 67.6, 90.4, 117.0, 128.5, 128.7, 135.8, 133.8, 157.1.

hydroxylamine 6c

Colorless oil, R_f = 0.6 (25% EtOAc : Hexane)

m/z (EI) 178 (15), 165 (18), 117 (43), 91 (100)

IR (Neat) 3296, 3016, 2947, 1735, 1506, 1471, 1406, 1272, 1117, 1043 cm^{-1}.

^1H (200 MHz, $CDCl_3$) 0.08 (s, 6H, Me_2Si), 0.90 (s, 9H, t-Bu), 2.37 (t, 2H, J 6.6, CH_2), 3.75 (d, 2H, J 5, CH_2), 3.88 (sextet, 1H, J 6, OCH), 5.06 (brd, 1H, J 9, $=CH_2$), 5.12 (brd, 1H, J 17, $=CH_2$), 5.18 (s, 2H, CH_2Ph), 5.87 (ddt, 1H, J 17, 9, 7, =CH), 7.36 (5H, m, Ph)

^{13}C (50 MHz, $CDCl_3$) -3.4, 8.1, 25.3, 33.4, 46.2, 61.2, 67.6, 86.2, 117.2, 127.9, 128.1, 128.8, 133.3, 158.6

General Procedure for the Preparation of *t*-Boc derivatives

Di-*t*-butyldicarbonate (1.1 eq.) was added to a solution (0.05 M) of the alcohol in dichloromethane/ water (1:1). Solid sodium hydroxide (2 eq.) was added and the mixture was stirred overnight. The organic layer was separated, dried (Na_2SO_4) and evaporated. The residue was purified by flash chromatography on silica gel eluting with hexane and 5% ethyl acetate/ hexane.

Hydroxylamine 7a

Colorless oil, R_f = 0.38 (25% EtOAc : Hexane)

m/z (EI) 263 (13) (M^+), 131 (93), 115 (43), 91 (100)

IR (Neat) 3289, 3066, 2979, 1718, 1643 cm^{-1}

^1H (200 MHz, $CDCl_3$) 1.44 (s, 9H, *t*-Bu), 2.46 (dt, J 14.6, 6.6, 2H, CH_2), 2.73 (dt, J 14.6, 6.6, 2H, CH_2), 4.79 (t, J 7.0, 1H, PhCH), 5.05 (m, 2H, $=CH_2$), 5.13 (m, 2H, $CH=CH_2$), 5.80 (ddt, J 7.0, 10.3, 17.2, 1H, =CH), 6.90 (s, 1H, NH), 7.35 (m, 5H, Ph)

^{13}C (50 MHz, $CDCl_3$) 28.0, 39.5, 81.3, 86.9, 117.3, 127.1, 128.0, 128.3, 139.7, 133.7, 139.7.

hydroxylamine 7b

Colorless oil, R_f = 0.41 (50% EtOAc : Hexane)

m/z (CI) 230 (87), 229 (100), 202 (68), 174 (50), 170 (26)

IR (Neat) 3300, 3079, 2975, 1750, 1671, 1471, 1391, 1369, 1250, 1170 cm^{-1}.

^1H (200 MHz, $CDCl_3$) 0.78 (d, J 6, Me), 0.82 (d, J 6, Me), 1.35 (s, 9H, *t*-Bu), 1.80 (m, 1H, Me_2CH), 2.19 (m, 2H, CH_2), 3.45 (q, J 6, OCH), 4.93 (m, 1H, $=CH_2$), 5.02 (m, 1H, $=CH_2$), 5.76 (m, 1H, =CH), 6.8 (s, 1H, NH)

^{13}C (50 MHz, $CDCl_3$) 18.2, 28.2, 29.3, 33.6, 81.5, 89.8, 116.6, 135.2, 156.9.

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White semi-solid, mp <40°C; R_f = 0.63 (25% EtOAc : Hexane)

m/z (EI) 145 (9) (M^+-ONHBoc), 91 (100)

IR (KBr) 3251, 2985, 1713, 1644 cm^{-1}

^1H (200 MHz, $CDCl_3$) 1.46 (s, 9H, *t*-Bu), 1.8 - 2.20 (m, 4H, $(CH_2)_2$), 4.74 (t, J 6.8, 2H, PhCH),4.97 (brd, 1H, J 9, $=CH_2$), 5.03 (brd, 1H, J 18, =CH), 5.82 (ddt, J 18, 9, 6.3, 1H, $CH=CH_2$), 6.87 (s, 1H, NH), 7.35 (m, 5H, Ar).

^{13}C (50 MHz, $CDCl_3$) 28.3, 29.8, 34.5, 81.5, 87.2, 117.6, 127.3, 128.4, 128.6, 140.0, 133.9, 156.5.

The corresponding unprotected hydroxylamine shows the following data:

Colorless oil, R_f = 0.45 (25% EtOAc : Hexane)

m/z (FAB) 178.0 (5) (M^++H), 145.1 (30), 91 (100)

IR (Neat) 3319, 3050, 2937, 1641, 1186 cm^{-1}

^1H (200 MHz, $CDCl_3$) 1.80 - 2.10 (m, 4H, $(CH_2)_2$), 4.50 (t, J 7, 1H, PhCH), 5.0 (m, 2H, $=CH_2$), 5.20 (s, 2H, NH_2), 5.83 (ddt, J 17.2, 10.3, 7, =CH), 7.30 (m, 5H, Ar)

^{13}C (50 MHz, $CDCl_3$) 29.6, 35.0, 86.5, 114.6, 126.5, 127.5, 128.3, 141.5, 138.8.

General Procedure for the Cyclofunctionalization Reaction

A mixture of palladium (II) chloride (10 mole%) and copper (II) acetate hydrate (3 eq.) in acetonitrile (*ca* 0.1 M) was cooled to 0°C under nitrogen. A solution of the hydroxylamine in methanol (*ca* 0.1 M) was added. The atmosphere was changed to carbon monoxide (1 atm) and tetramethylguanidine (3 eq.) was added. The mixture was allowed to warm to room temperature and stirred overnight. Precipitated solids were removed by filtration through a pad of silica gel, washing with ethyl acetate. The solvents were evaporated and the residue was purified by flash column chromatography on silica gel, eluting with 5-15% ethyl acetate/ hexane.

Isoxazolidine8a

Yellow oil, R_f = 0.14 (25% EtOAc : Hexane)

m/z (EI) 280 (100) (M^++H), 129 (22)

IR (Neat) 2955, 1737 cm^{-1}

^1H (200 MHz, $CDCl_3$) 2.05 (ddd, J 6.2, 10.3, 12.6, 1H, H4), 2.63 (dd, J 8.6, 16.2, 1H, CH_2CO_2), 2.97 (m, 2H, H4, CH_2CO_2), 3.68 (s, 3H, OMe), 3.80 (s, 3H, OMe), 4.73 (tt, J 8.6, 6.2, 1H, H3), 4.91 (dd, J 6.2, 9.9, 1H, H5), 7.30 (m, 5H, PH)

^{13}C (50 MHz, $CDCl_3$) 40.6, 43.2, 51.8, 53.5, 83.4, 126.4, 126.6, 128.5, 136.5, 158.5, 170.9.

Isoxazolidine *cis*-9a

Yellow solid, mp 99 - 100 °C, Rf = 0.22 (25% EtOAc : Hexane);

Found: C (53.23), H (4.26), N (6.89) $C_{18}H_{18}N_2SO_7$ requires: C (53.20), H (4.46), N (6.89)

IR (Neat) 3105, 2955, 1739, 1608, 1349, 1014 cm^{-1}

^1H (200 MHz, $CDCl_3$) 2.08 (ddd, J 7.3, 10, 12.5, 1H, H4), 2.68 (dd, J 8.2, 15.8, 1H, H4), 3.08 (m, 2H, CH_2CO_2), 3.66 (s, 3H, OMe), 4.76 (ddd, J 5.9, 7.3, 15.8, 1H, H3), 5.24 (dd, J 5.9, 10.2, 1H, H5), 7.24 (m, 5H, Ph), 8.10 (d, 2H, J 8.9, Ns), 8.30 (d, 2H, J 8.9, Ns)

^{13}C (50 MHz, $CDCl_3$) 40.7, 43.1, 52.21, 57.2, 84.0, 124.3, 126.9, 128.9, 129.2, 130.7, 135.9, 142.1, 160.0, 170.6

Isoxazolidine *trans*-9a

Yellow solid, mp 108 - 110 °C, R_f = 0.15 (25% EtOAc : Hexane)

m/z (FAB) 185 (90), 93 (100)

IR (Neat) 3107, 2955, 1733, 1608, 1362, 1015 cm^{-1}

^{1}H (200 MHz, $CDCl_3$) 2.33 (m, 2H, H4), 2.62 (dd, 1H, J 6.5, 16.5, CH_2CO_2), 2.86 (dd, 1H, J 6.5, 16.5, 1H, CH_2CO_2), 3.63 (s, 3H, OMe), 4.65 (m, 1H, H3), 5.11 (t, J 8.4, 1H, H5), 7.21 (m, 5H, Ph), 7.94 (d, 2H, J 8.8, Ns), 8.15 (d, 2H, J 8.8, Ns)

^{13}C (50 MHz, $CDCl_3$) 39.2, 41.0, 52.1, 58.3, 83.0, 123.9, 127.1, 128.7, 129.1, 136.3, 140.8, 170.3.

Isoxazolidine 10a

Yellow oil, R_f = 0.28 (25% EtOAc : Hexane)

m/z (HFAB) found: 356.1497 [M^++ H] $C_{20}H_{22}NO_5$ requires: 356.1420

(FAB) 356.1 (78) (M^+ +H), 312.1 (30), 91.0 (100)

IR (Neat) 3031, 2958, 1738 cm^{-1}

^{1}H (200 MHz, $CDCl_3$) 2.06 (ddd, J 6.2, 10.3, 12.4, 1H, H4), 2.64 (dd, J 8.6, 16.1, 1H, CH_2CO_2), 2.98 (m, 2H, H4, CH_2CO_2), 3.66 (s, 3H, OMe), 4.76 (tt, J 6.2, 8.6, 1H, H3), 4.90 (dd, J 6.2, 10.3, 1H, H5), 5.21 (d, 1H, J 12, OCH_2Ph), 5.27 (d, 1H, J 12, OCH_2Ph), 7.40 (m, 10H, Ar)

^{13}C (50 MHz, $CDCl_3$) 40.4, 43.1, 51.7, 57.5, 68.0, 83.4, 126.6, 128.1, 128.3, 128.5, 128.6, 128.7, 135.8, 136.6, 161.0, 170.9.

Isoxazolidine 11a

White solid, mp 74 - 76 °C, R_f = 0.28 (25% EtOAc : Hexane)

m/z (EI) 321 (1) (M^+) 221 (62), 189 (51), 129 (100)

IR (KBr) 3325, 2982, 1733, 1693 cm^{-1}

^{1}H (200 MHz, $CDCl_3$) 1.52 (s, 9H, *t*-Bu), 2.05 (m, 1H, CH_2CO_2Me), 2.63 (dd, 1H, J 5.9, 15.8, CH_2CO_2Me), 2.96 (m, 2H, H4), 3.69 (s, 3H, OMe), 4.71 (m, 1H, H3), 4.90 (dd, 1H, J 6.3, 9.8, H5), 7.38 (m, 5H, Ph)

^{13}C (50 MHz, $CDCl_3$) 28.4, 40.9, 43.4, 51.9, 57.6, 82.4, 83.0, 126.8, 128.6, 128.7,137.3, 157.5, 171.4.

Isoxazolidine 10b

Colorless oil, R_f = 0.34 (25% EtOAc : Hexane)

m/z (HFAB) found: 322.1649 (M^++ H) $C_{17}H_{24}NO_5$ requires: 322.1576

m/z (FAB) 322, 278, 204, 172, 91(100)

IR (Neat) 3046, 2976, 1755, 1701, 1451, 1396, 1317, 1218, 1087

^{1}H (200 MHz, $CDCl_3$) 0.91 (d, J 7, 3H, Me), 1.10 (d, J 7, 3H, Me), 1.65 (ddd, 1H, J 12.4, 9.9, 3.9, H4), 1.85 (m, 1H, Me_2CH), 2.51 (dd, 1H, J 8.8, 15.8, CH_2CO_2). 2.54 (m, 1H, H4), 2.58 (dd, 1H, J 5.9, 15.8, CH_2CO_2), 3.63 (m, 1H, H5), 3.66 (s, 3H, OMe), 4.63 (tt, 1H, J 5.9, 8.8, H3), 5.15 (d, 1H, J 12.5, CH_2Ph), 5.25 (d, 1H, J 12.5, CH_2Ph), 7.19 (m, 5H, Ph)

^{13}C (50 MHz, $CDCl_3$) 19.0, 19.3, 29.6, 33.9, 42.1, 50.1, 57.8, 67.6, 80.3, 128.3, 128.7, 135.8, 156.2, 168.8.

Isoxazolidine 11b

Colorless oil, R_f = 0.24 (50% EtOAc : Hexane)

m/z (EI) 288 (M^++H) (100), 232 30), 188 (16)

IR (Neat) 2975, 1741, 1717, 1438, 1369, 1333, 1256, 1167 cm^{-1}

^{1}H (200 MHz, $CDCl_3$) 0.90 (d, J 6.6, 3H, Me), 0.96 (d, J 6.6, 3H, Me), 1.47 (s, 9H, *t*-Bu), 1.59 (ddd, 1H, J 12.5, 9.9, 6, H4), 1.90 (m, 1H, Me_2CH), 2.47 (dd, J 9, 16, 1H, CH_2CO_2) 2.52 (ddd, 1H, J 6.2, 7, 13, H4), 2.81 (dd, J 5.5, 16, 1H, CH_2CO_2), 3.58 (ddd, 1H, J 13.9, 9.9, 6.6, H3), 3.68 (s, 3H, OMe), 4.55 (dt, J 8.6, 6, 1H, H5)

^{13}C (50 MHz, $CDCl_3$) 18.2, 19.2, 28.1, 30.8, 38.3, 40.6, 51.6, 56.9, 81.6, 86.6, 157.4, 171.3.

Isoxazolidine 10c

Colorless oil, R_f = 0.66 (25% EtOAc : Hexane)

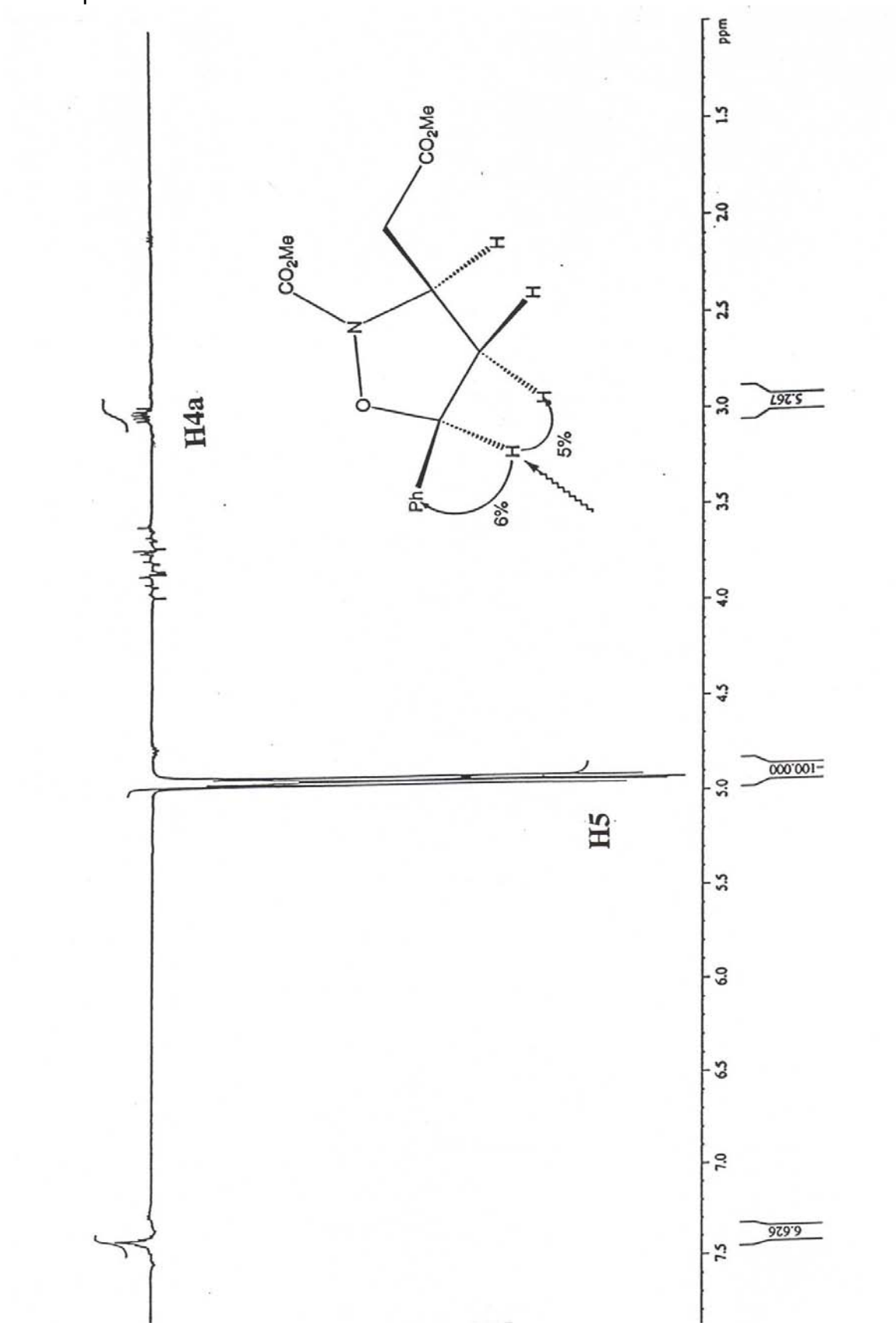
m/z (EI) 192 (66), 160 (40), 150 (21), 91 (100)

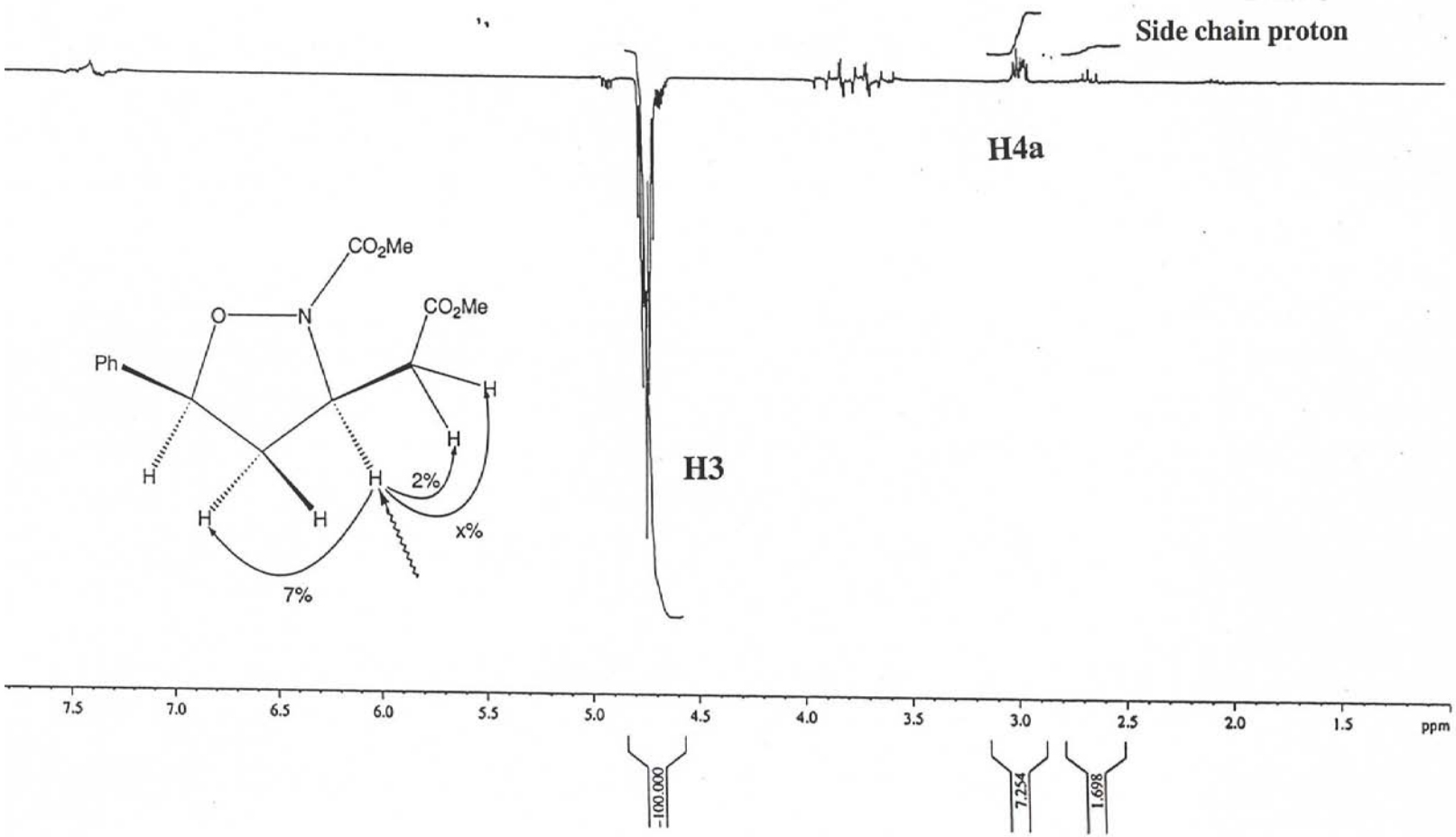
IR (Neat) 2954, 2863, 1741, 1720, 1258 cm^{-1}

^{1}H (200 MHz, $CDCl_3$) 0.15 (s, 6H, Me_2Si), 0.78 (s, 9H, *t*-Bu), 1.92 (ddd, 1H, J 12, 8.1, 4 H4), 2.43 (dt, 1H, J 12.5, 8.4, 1H, H4), 2.46 (dd, 1H, J 16.1, 8.4, CH_2CO_2), 2.70 (dd, 1H, J 16.1, 6.2, CH_2CO_2), 3.60 (s, 3H, OMe), 3.63 (dd, 1H, J 11.7, 4, OCH_2), 3.75 (dd, 1H, J 11.7, 2.9, OCH_2), 4.05 (m, 1H, H5). 4.64 (m, 1H, H3), 5.02 (d, J 12.5, CH_2Ph), 5.10 (d, J 12.5, CH_2Ph), 7.3 (m, 5H, Ph).

^{13}C (50 MHz, $CDCl_3$) -5.3, 18.5, 26.0, 35.5, 39.8, 51.8, 57.0, 62.0, 68.1, 82.0, 128.24, 128.4, 128.7, 135.9, 158.3, 171.5.

nOe experiments for isoxazolidine **8a**

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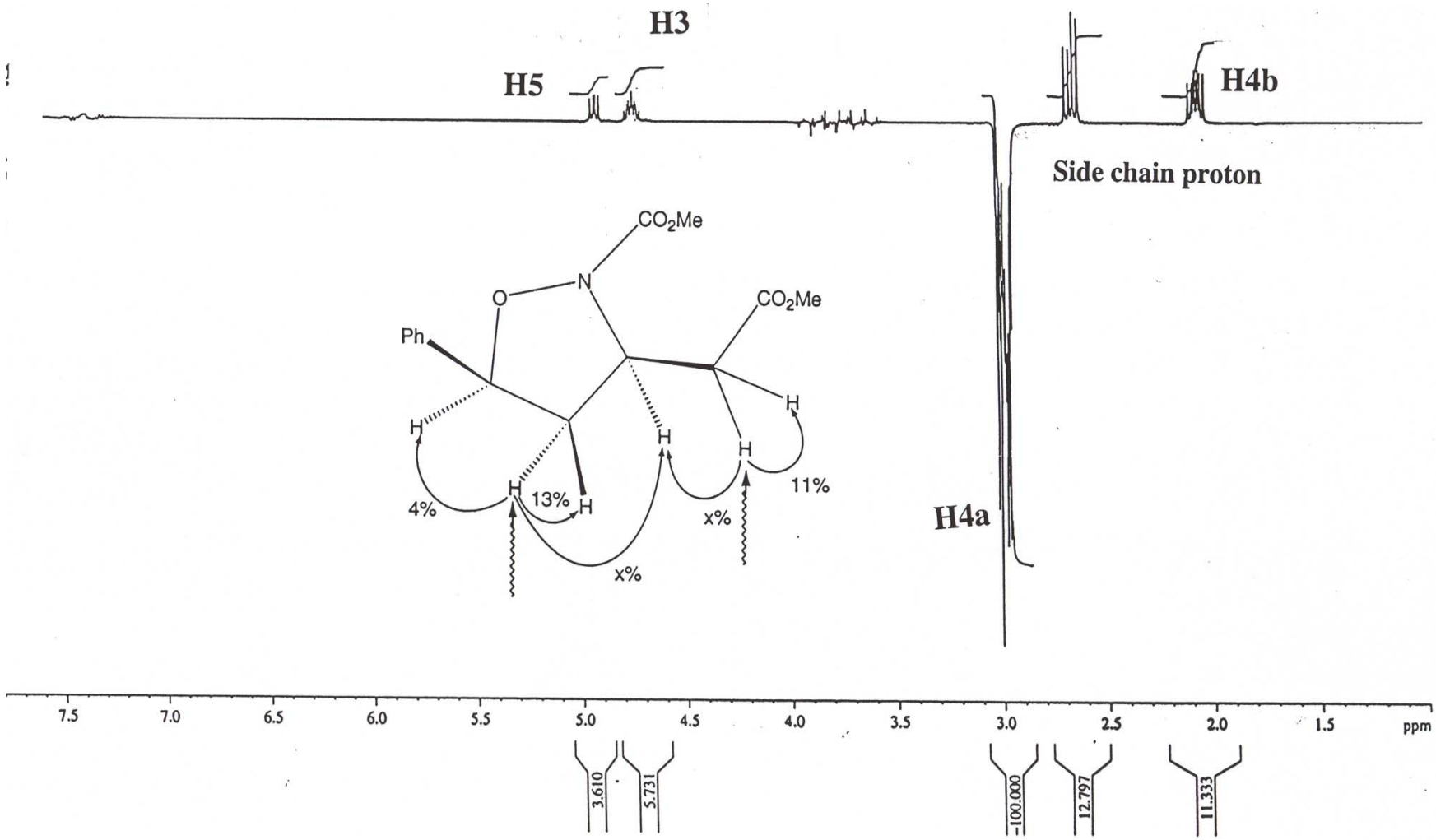
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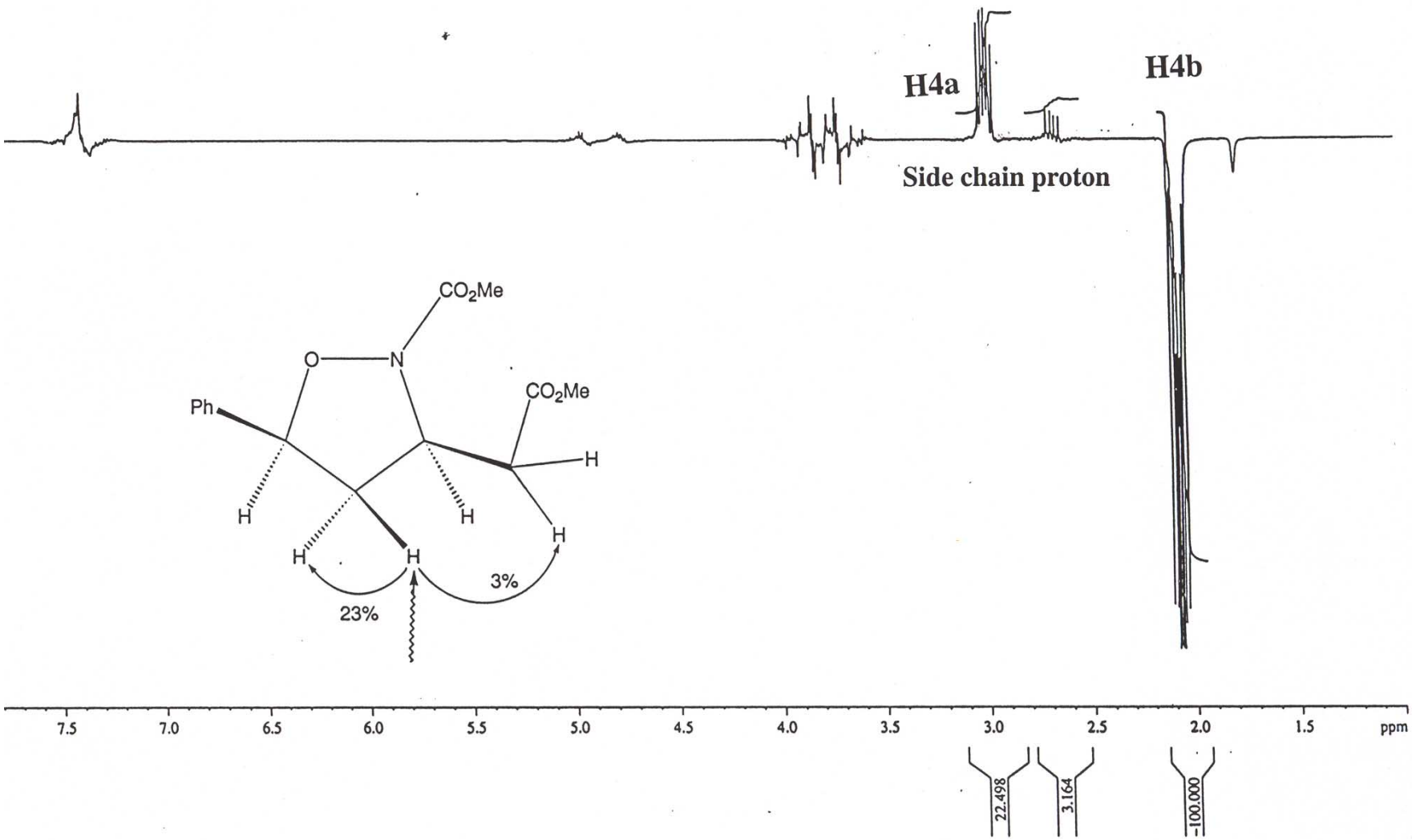
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ppm
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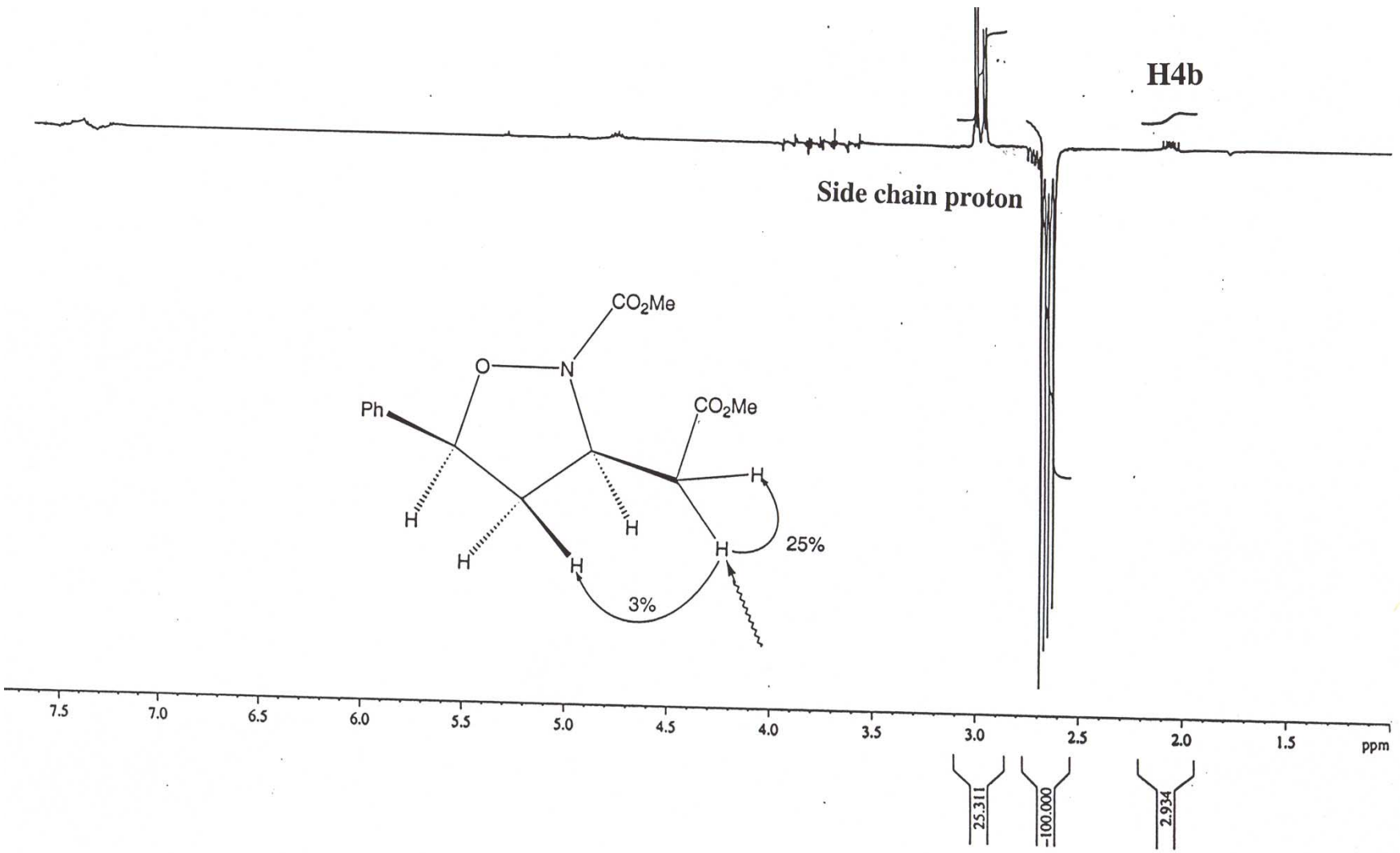
3.164
100.000


H4b
Side chain proton
CO2Me
O
N
Ph
CO2Me
H
H
H
H
H
H
25%
3%
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
ppm
25.311
100.000
2.934