

Supporting Information

Diastereoselective synthesis of tetrahydrofurans *via* reaction of γ,δ -epoxycarbanions with aldehydes

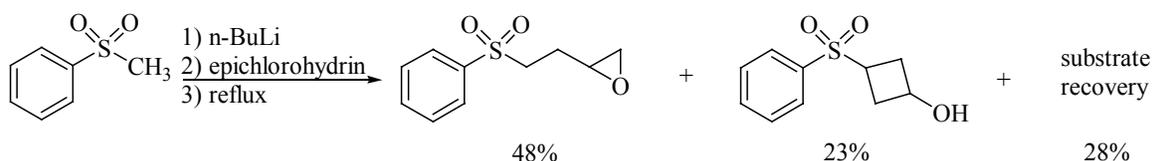
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General. Unless otherwise noted, all reactions were carried out under atmosphere of argon in dried glassware using standard Schlenk techniques. THF was distilled from K / benzophenone ketyl. Lithium *tert*-butoxide (1M solution in THF) was purchased from Aldrich, potassium *tert*-butoxide solution (\approx 1M solution in THF) was prepared by dissolving of commercial material. BuLi was used as a 2.5M solution in hexanes, purchased from Aldrich.

Analytical thin layer chromatography (TLC) was performed on 0.25 mm Merck silica gel 60 F₂₅₄ plates. Visualization was accomplished with UV light or after development in anisaldehyde stain¹. Preparative thin layer chromatography was performed on 2 mm silica gel plates. Preparative column chromatography was performed on Silica gel 60 (0.040 - 0.063 mm, 230 - 400 mesh ASTM) Merck. Enantiomeric excesses were determined using Knauer HPLC chromatograph (Diode Array Detector) with column Chiracel OD-H with hexane : *iso*-propanol (9 : 1; 1mL / min). ¹H and ¹³C NMR spectra were recorded on Bruker 500 and Varian 200 spectrometers. Chemical shifts are reported in ppm from the solvent resonance (CDCl₃ 7.26 ppm). Data are reported as follows: chemical shift, multiplicity, (s = singlet, d = doublet, t = triplet, q = quartet, br = broad, m = multiplet), coupling constants and number of protons. Mass spectra were obtained on AMD 604 Intectra GmbH spectrometer in electron ionization mode or on Mariner™ in electrospray mode. IR spectra were taken on a FT-IR Perkin Elmer Spectrum 2000 using a film (for oils) or in KBr pellets (for solids). Melting points were uncorrected.



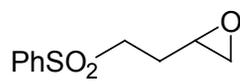
Synthesis of 1. Methyl phenyl sulfone (39g; 0.25 mol) in THF (500 mL) was purged with argon and cooled to 0°C. n-BuLi (105 mL, 0.263 mol) was added dropwise as a solution in hexanes. Mixture was left

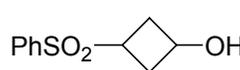
¹ See for example Leonard, J., Lygo, B., Procter, G., *Advanced Practical Organic Chemistry*, 2nd Ed.; Stanley Thornes (Publishers) Ltd 1998, p. 149.

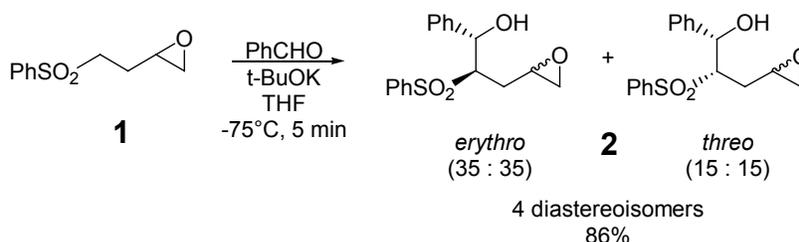
for 1h and then cooled to -45°C. Epichlorohydrine (25.3g; 0.275 mol) was added slowly as a solution in THF (150 mL) and mixture was allowed to warm to rt². Then it was refluxed for 1.5 h, cooled to rt, aqueous solution of NH₄Cl (10% w/w, 500 mL) was added and mixture was concentrated *in vacuo*. Extraction with ethyl acetate (3×250 mL), washing with brine and drying with MgSO₄ gave crude material, which was separated chromatographically with hexane : ethyl acetate (3 : 1) as an eluent.

Products in order of separated fractions:

PhSO₂CH₃ ▶ recovered **methyl phenyl sulfone** (26%, 10g), mp: 86-87°C (Lit.³ 88°C).

 ▶ **3,4-epoxybutyl phenyl sulfone** (48%, 22.5g)
Oil. IR (neat): 3616, 3062, 2997, 2927, 1585, 1447, 1411, 1307, 1147, 1086, 915, 799, 743, 690, 592, 563, 538 cm⁻¹. ¹H NMR (200 MHz, CDCl₃): δ 7.85-7.95 (m, 2H), 7.51-7.72 (m, 3H), 3.13-3.30 (m, 2H), 2.94-3.04 (m, 1H), 2.72-2.79 (m, 1H), 2.44-2.51 (m, 1H), 2.05-2.24 (m, 1H), 1.66-1.91 (m, 1H). ¹³C NMR (50 MHz, CDCl₃): δ 138.8, 133.9, 129.3, 128.0, 52.6, 50.0, 47.0, 25.8. HRMS (ESI): calcd for C₁₀H₁₂SO₃Na 235.0399, found 235.0410. Anal. calcd for C₁₀H₁₂SO₃ C, 56.58; H, 5.70; S, 15.11. Found C, 56.37; H, 5.60; S, 15.22.

 ▶ **3-hydroxycyclobutyl phenyl sulfone** (23%, 12.2g)
Oil. ¹H NMR (200 MHz, CDCl₃): δ 7.83-7.95 (m, 2H), 7.51-7.75 (m, 3H), 4.04-4.27 (m, 1H), 3.24-3.44 (m, 1H), 2.20-2.90 (m, 5H). ¹³C NMR (50 MHz, CDCl₃): δ 138.0, 133.8, 129.3, 128.1, 61.7, 49.0, 34.6. MS (ESI): calcd for C₁₀H₁₂SO₃Na 235.0399, found 235.1.



Reaction of 1 with benzaldehyde and protonation at low temperature (scheme 1 in the article).

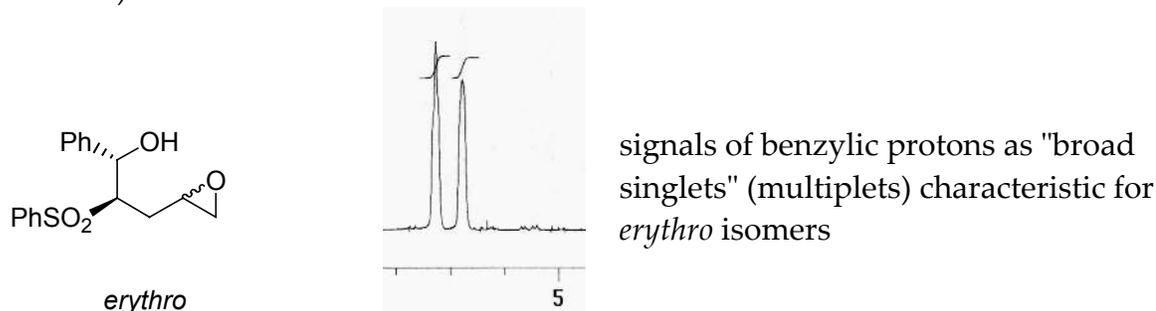
To a solution of **1** (212mg, 1 mmol) and benzaldehyde (135 mg, 1.25 mmol) in THF (4mL) at -75°C under argon solutions of *t*-BuOK (2mL, 1M in THF) was added. After 5 minutes aqueous NH₄Cl was added

² When reaction was quenched below 0°C 1-chloro-4-(phenylsulfonyl)-2-butanol was isolated Mp: 75-76°C. IR (neat): 3511, 3446, 3064, 2927, 1585, 1446, 1420, 1262, 1153, 1084, 1024, 915, 857, 813, 746, 685, 602, 576, 536 cm⁻¹. ¹H NMR (200 MHz, CDCl₃): δ 7.87-7.96 (m, 2H), 7.52-7.73 (m, 3H), 3.87-4.02 (m, 1H), 3.14-3.63 (m, 4H), 2.50 (s br, 1H), 1.77-2.15 (m, 2H). ¹³C NMR (50 MHz, CDCl₃): δ 138.8, 133.9, 129.4, 128.0, 69.4, 52.7, 49.4, 27.2. MS (ESI): calcd for C₁₀H₁₃SO₃³⁵ClNa 271.0, found 271.0. Anal. calcd for C₁₀H₁₃SO₃Cl C, 48.29; H, 5.27; S, 12.89; Cl 14.25. Found C, 48.20; H, 5.41; S, 12.74; Cl 14.15.

³ C. C. Price, J. J. Hydock, *J. Am Chem. Soc.* **1952**, *74*, 1943.

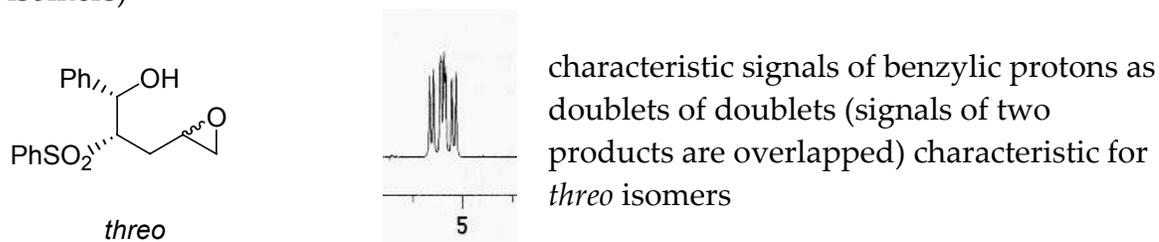
and mixture was extracted with ethyl acetate, washed with brine and dried MgSO_4 . Chromatographic separation with hexane : ethyl acetate (3 : 1 to 1 : 1) gave adduct **2** as a mixture of diastereoisomers of **2** (273 mg, 86% yield). This mixture was analyzed with ^1H NMR (35 : 35 : 15 : 15, diastereoselectivity was based on integration of signals at 5 - 5.5 ppm) and separated at preparative thin layer chromatography with hexane : ethyl acetate (2 : 1) to give two pairs of diastereoisomers *erythro* and *threo* as equimolar mixtures of products differing by configuration at the oxirane ring.

erythro-3-(oxiran-2-yl)-1-phenyl-2-(phenylsulfonyl)propan-1-ol (**2** as equimolar mixture of *erythro* isomers)



Mp: 127-130°C. IR (KBr): 3463, 3068, 3005, 2895, 1585, 1493, 1456, 1446, 1403, 1303, 1284, 1147, 1085, 1059, 954, 837, 759, 734, 700, 683, 632, 579, 548, 511, 456 cm^{-1} . ^1H NMR (500 MHz, CDCl_3): δ 7.88-7.95 (m, 4H), 7.60-7.66 (m, 2H), 7.51-7.57 (m, 4H), 7.19-7.24 (m, 4H), 7.10-7.18 (m, 6H), 5.37-5.39 (m, 1H), 5.27-5.29 (m, 1H), 3.44 (d, $J = 2.5$ Hz, 1H), 3.43 (d, $J = 2.7$ Hz, 1H), 3.36 (ddd, $J = 7.5, 4.0, 1.5$ Hz, 1H), 3.33 (ddd, $J = 5.4, 5.4, 1.8$ Hz, 1H), 2.63-2.68 (m, 1H), 2.43 (dd, $J = 4.8, 4.0$ Hz), 2.38-2.42 (m, 1H), 2.31 (dd, $J = 4.4, 4.4$ Hz, 1H), 1.94-2.15 (m, 4H), 1.90 (ddd, $J = 15.6, 6.4, 4.0$ Hz), 1.85 (dd, $J = 5.0, 2.6$ Hz, 1H). ^{13}C NMR (50 MHz, CDCl_3): δ 139.3, 139.2, 137.4, 137.2, 134.3, 134.3, 129.5, 129.5, 128.7, 128.5, 127.8, 125.3, 69.3, 68.9, 67.9, 67.5, 50.1, 50.0, 48.1, 47.8, 25.0, 25.0. MS (ESI): calcd for $\text{C}_{17}\text{H}_{18}\text{SO}_4$ 341.0818 found 341.1. Anal. calcd for $\text{C}_{17}\text{H}_{18}\text{SO}_4$ C, 64.13; H, 5.70; S, 10.07. Found C, 64.10; H, 5.81; S, 10.10.

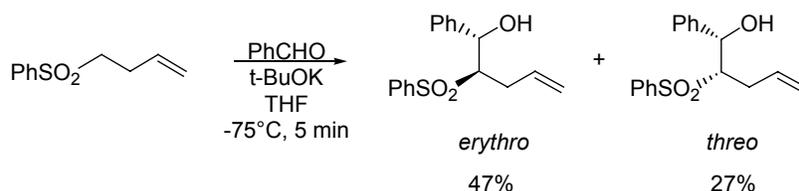
threo-3-(oxiran-2-yl)-1-phenyl-2-(phenylsulfonyl)propan-1-ol (**2** as an equimolar mixture of *threo* isomers)



Oil. IR (neat): 3502, 3062, 2925, 1603, 1585, 1495, 1480, 1447, 1410, 1305, 1145, 1084, 832, 757, 736, 703, 690, 628, 586, 564, 543 cm^{-1} . ^1H NMR (200 MHz, CDCl_3): δ 7.85-7.93 (m, 4H), 7.50-7.72 (m, 6H), 7.20-7.37 (m, 10H), 5.10 (dd, $J = 8.6, 3.0$ Hz, 1H, isomer a), 5.06 (dd, $J = 8.3, 3.3$ Hz, 1H, isomer b), 4.32 (d, $J = 3.2$ Hz, 1H, isomer a), 4.23 (d, $J = 3.3$ Hz, 1H, isomer b), 3.53-3.67 (m, 2H), 2.22-2.53 (m, 4H), 2.01 (dd, $J = 4.9, 2.7$ Hz, 1H, isomer b), 1.85 (dd, $J = 4.9, 2.7$ Hz, 1H, isomer a), 1.73-1.91 (m, 2H), 1.49-1.70 (m, 2H). ^{13}C NMR (50 MHz, CDCl_3): δ 139.3 (isomer a), 139.1 (isomer b), 138.2 (isomer b), 137.8 (isomer a), 134.0, 129.2, 128.8, 128.7, 128.7, 128.6, 127.2 (isomer a), 127.1 (isomer b), 73.5 (isomer b), 73.1 (isomer a), 68.2 (isomer b), 67.8 (isomer a), 49.6 (isomer b), 49.3 (isomer a), 48.0 (isomer a), 47.7 (isomer b), 30.4 (isomer b), 29.7 (isomer a).

a). HRMS (ESI): calcd for C₁₇H₁₈SO₄ 341.0818 found 341.0822. Anal. calcd for C₁₇H₁₈SO₄ C, 64.13; H, 5.70; S, 10.07. Found C, 64.06; H, 5.86; S, 9.87.

To confirm assignment of product as pairs of *erythro* and *threo* diastereoisomers independent synthesis of these compounds was realized.



To a solution of 3-butenyl phenyl sulfone⁴ (584 mg, 2.98 mmol) and benzaldehyde (404 mg, 3.81 mmol) in THF (7mL) at -75°C under argon solution of *t*-BuOK (3.5mL, 1M in THF) was added. After 5 minutes aqueous NH₄Cl was added and mixture was extracted with ethyl acetate, washed with brine and dried MgSO₄. Chromatographic separation with hexane : ethyl acetate (3 : 1 to 1 : 1) gave in order of separated fractions:

▶ recovered 3-butenyl phenyl sulfone (100 mg, 17% yield)

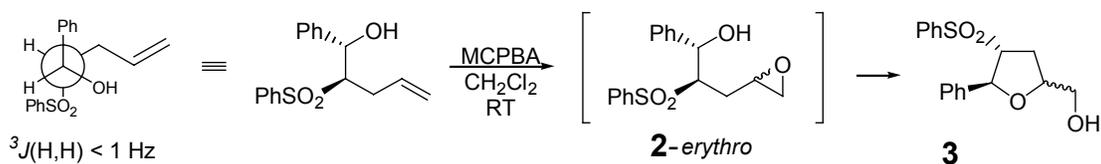
▶ ***erythro*-1-phenyl-2-(phenylsulfonyl)-4-penten-1-ol** (420 mg, 47% yield)
 Oil. IR (neat): 3507, 3065, 2923, 1641, 1585, 1496, 1448, 1304, 1143, 1083, 919, 758, 737, 702, 690, 630, 582, 562, 539 cm⁻¹. ¹H NMR (200 MHz, CDCl₃): δ 7.91-7.99 (m, 2H), 7.53-7.74 (m, 3H), 7.17-7.36 (m, 5H), 5.48-5.51 (m, 1H), 5.27 (dddd, *J* = 16.8, 10.3, 7.0, 7.0 Hz, 1H), 4.66-4.71 (m, 1H), 4.56-4.66 (m, 1H), 3.42 (s br, 1H), 3.21-3.30 (m, 1H), 2.52-2.63 (m, 2H). ¹³C NMR (50 MHz, CDCl₃): δ 139.5, 137.6, 134.6, 134.1, 129.3, 128.7, 128.3, 127.6, 125.5, 116.8, 70.5, 69.4, 25.9. MS (ESI): calcd for C₁₇H₁₈SO₃Na 325.38 found 325.1. Anal. calcd for C₁₇H₁₈SO₃ C, 67.52; H, 6.00; S, 10.60. Found C, 67.19; H, 6.25; S, 10.52.

▶ ***threo*-1-phenyl-2-(phenylsulfonyl)-4-penten-1-ol** (240 mg, 27% yield)
 Mp: 103-104°C. IR (neat): 3492, 3076, 2920, 1640, 1448, 1281, 1226, 1140, 1085, 1057, 1010, 913, 761, 737, 699, 682, 577, 555, 526, 514 cm⁻¹. ¹H NMR (200 MHz, CDCl₃): δ 7.86-7.93 (m, 2H), 7.50-7.71 (m, 3H), 7.27-7.33 (m, 5H), 5.12-5.28 (m, 1H), 5.09 (d, *J* = 8.1 Hz, 1H), 4.70-4.79 (m, 1H), 4.54-4.66 (m, 1H), 4.26 (s br, 1H), 3.40-3.52 (m, 1H), 3.21-3.30 (m, 1H), 2.04-2.41 (m, 2H). ¹³C NMR (50 MHz, CDCl₃): δ 139.3,

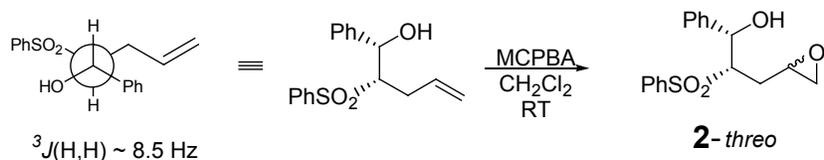
⁴ Synthesized from 4-bromo-1-butene and phenylsulfinic acid sodium salt in DMSO: Oil. IR (neat): 3624, 3069, 2982, 2924, 1642, 1585, 1479, 1447, 1406, 1307, 1234, 1146, 1086, 998, 922, 800, 746, 690, 633, 591, 556, 533, 441 cm⁻¹. ¹H NMR (200 MHz, CDCl₃): δ 7.88-7.96 (m, 2H), 7.52-7.72 (m, 3H), 5.62-5.84 (m, 1H), 5.04-5.11 (m, 1H), 4.99-5.03 (m, 1H), 3.11-3.22 (m, 2H), 2.39-2.54 (m, 2H). ¹³C NMR (50 MHz, CDCl₃): δ 139.0, 133.7, 129.3, 128.1, 117.1, 55.4, 26.8. Anal. calcd for C₁₀H₁₂SO₂ C, 61.20; H, 6.16; S, 16.34. Found C, 61.48; H, 6.11; S, 16.60.

138.4, 133.9, 132.9, 129.1, 128.7, 128.5, 127.2, 117.7, 73.0, 70.3, 31.0. MS (ESI): calcd for C₁₇H₁₈SO₃Na 325.38 found 325.1. Anal. calcd for C₁₇H₁₈SO₃ C, 67.52; H, 6.00; S, 10.60. Found C, 67.40; H, 6.09; S, 10.59.

Both diastereoisomers of adducts were subjected to oxidation with *m*-chloroperbenzoic acid (MCPBA).



To a solution of *erythro*-1-phenyl-2-(phenylsulfonyl)-4-penten-1-ol (255mg, 0.84 mmol) and in CH₂Cl₂ (10mL) at rt MCPBA (254 mg, 85% w/w, 1.25 mmol) was added. Flask was left at rt for 3 days, then solution was washed with aqueous solutions of Na₂CO₃ and NaCl and dried with MgSO₄. Chromatographic separation with hexane : ethyl acetate (3 : 1 to 1 : 1) gave **3** as an equimolar mixture of diastereoisomers (227mg, 85%). This mixture was analyzed with ¹H NMR (diastereoselectivity was based on integration of doublets at 5 - 5.5 ppm).

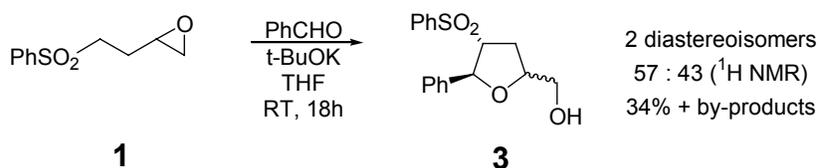


To a solution of *threo*-1-phenyl-2-(phenylsulfonyl)-4-penten-1-ol (106 mg, 0.35 mmol) and in CH₂Cl₂ (5mL) at rt MCPBA (117 mg, 85% w/w, 0.58 mmol) was added. Flask was left at rt for 3 days, then solution was washed with aqueous solutions of Na₂CO₃ and NaCl and dried with MgSO₄. Chromatographic separation with hexane : ethyl acetate (3 : 1 to 1 : 1) gave **2-threo** as an equimolar mixture of diastereoisomers (99mg, 89%). This mixture was analyzed with ¹H NMR (diastereoselectivity was based on integration of signals at 5 - 5.5 ppm).

Distinctive reactivity of diastereoisomers was attributed to structure of preferred conformations according to ¹H NMR spectra⁵. Both isomers are oxidized to oxiranes, which favour *anti* orientation of sterically demanding phenyl and phenylsulfonyl groups, but second step - acid catalysed cyclization of epoxyalcohol⁶ - is possible only for *erythro* isomer, where reacting centers are synclinal. Antiperiplanar orientation in *threo* adduct preclude cyclization to 5-membered ring.

⁵ Truce, W. E.; Klingler, T. C. *J. Org. Chem.* **1970**, *35*, 1834.

⁶ Coxon, J. M.; Hartshorn, M. P.; Swallow, W. H. *Aust. J. Chem.* **1973**, *26*, 2521.

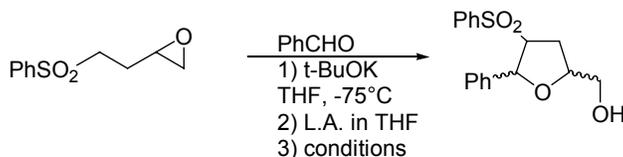


Reaction of 1 with benzaldehyde and protonation at RT (scheme 1 in the article).

To a solution of **1** (212mg, 1 mmol) and benzaldehyde (135 mg, 1.25 mmol) in THF (4mL) at -75°C under argon solutions of *t*-BuOK (1mL, 1M in THF) was added. Flask was allowed to warm to rt and left for 18h, then aqueous NH_4Cl was added and mixture was extracted with ethyl acetate, washed with brine and dried MgSO_4 . Chromatographic separation with hexane : ethyl acetate (3 : 1 to 1 : 1) gave tetrahydrofuran derivative as a mixture of diastereoisomers (117mg, 34%). Small amounts of some unidentified byproducts were also isolated. Mixture of diastereoisomers of **3** was analyzed with ^1H NMR (diastereoselectivity was based on integration of doublets at 5 - 5.5 ppm).

Optimizations

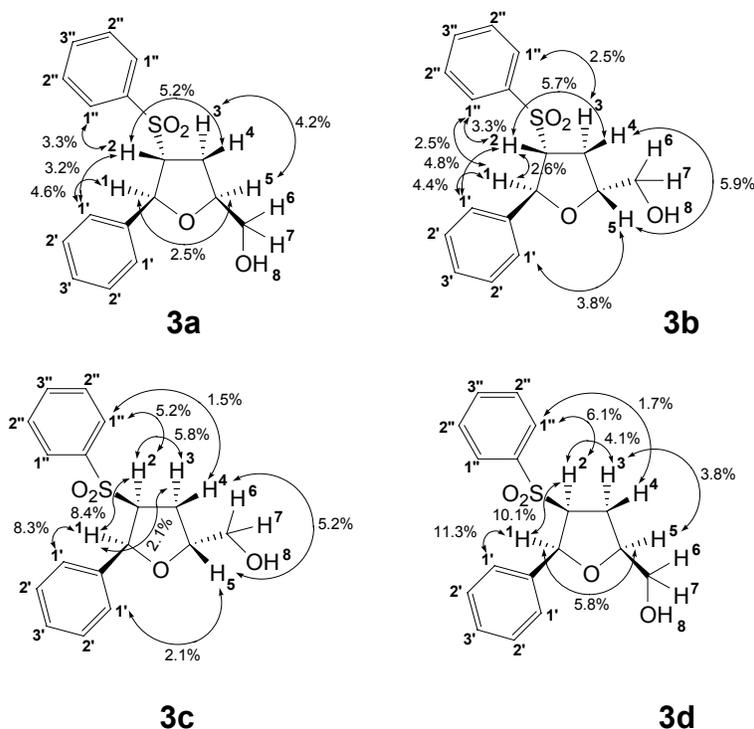
Lewis acid additives were tested under conditions of reaction carried out at room temperature.



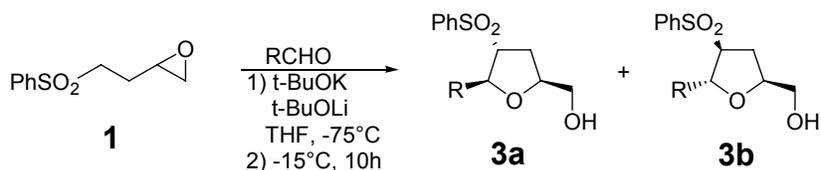
Entry	Lewis acid	Base <i>t</i> -BuOK	Conditions	Yield	Diastereoselectivity 3a : 3b : 3c : 3d
a	LiBr (2eq.)	1eq.	RT, 10h	54%	30:30:10:10
b	LiBr (1eq.)	1eq.	RT, 10h	31%	73:27:0:0
c	LiBr (1eq.)	2eq.	RT, 10h	56%	87:13:0:0
d	<i>t</i> -BuOLi (1eq.)	1eq.	RT, 10h	52%	84:16:0:0
e	<i>t</i> -BuOLi (1eq.)	1eq.	-15°C , 10h	87%	77:23:0:0

Lithium bromide used in excess favored cyclization in good yield, however with poor diastereoselectivity (entry a). Decreasing its amount to 1 equivalent improved diastereoselectivity (entry b) and increasing amount of base also increased yield (entry c). Finally mixture of lithium and potassium tert-butoxides was found superior (entry d) ensuring good yield and diastereoselectivity at lower temperature (entry e).

Reaction in entry "a" was performed on 2.5 mmol scale and all diastereoisomers were separated with consecutive chromatography purifications. Stereochemistry was established on ^1H NMR COSY (^1H - ^1H) and NOE spectra (see characterization data).



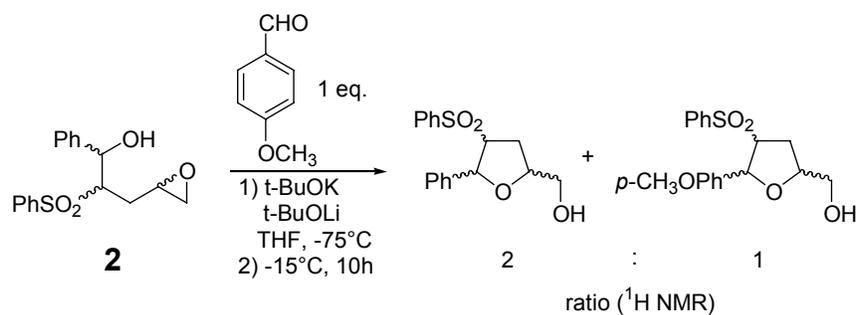
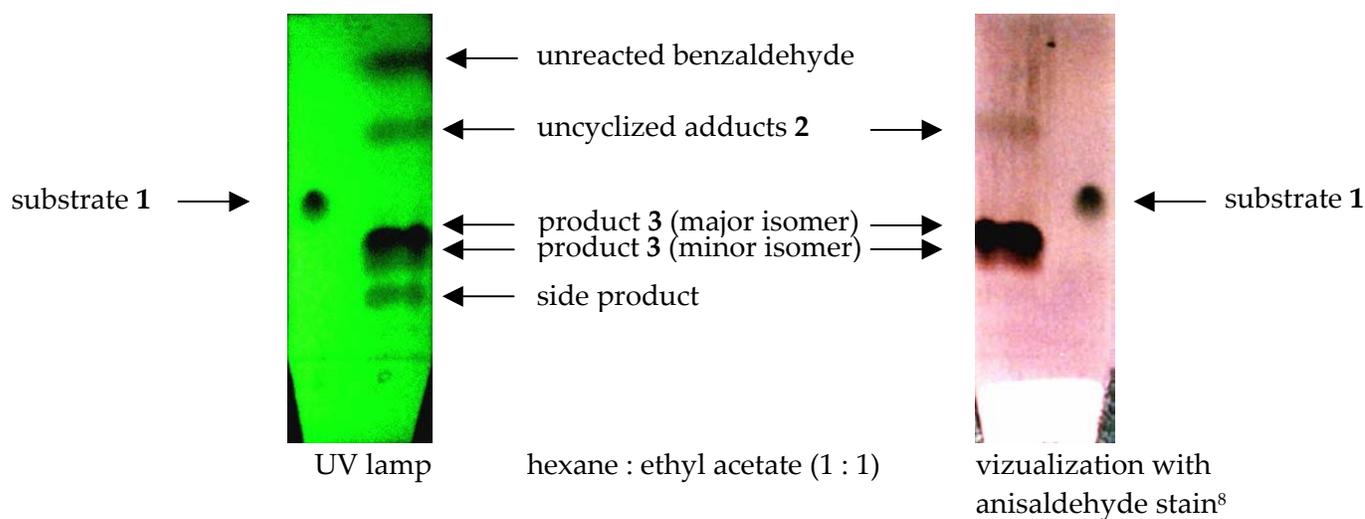
General procedure of synthesis and isolation of tetrahydrofuranes (table 1 in the article).



To a solution of **1** (212mg, 1 mmol) and aldehyde (1.25 mmol) in THF (4mL) at -75°C under argon solutions of *t*-BuOK (1mL, 1M in THF) and *t*-BuOLi (1mL, 1M in THF, Aldrich) were added consecutively. Flask was left at $-20 \sim -15^{\circ}\text{C}$ for 10h, then aqueous NH_4Cl was added and mixture was extracted with ethyl acetate, washed with brine and dried MgSO_4 . Chromatographic separation with hexane : ethyl acetate (6 : 1 to 1 : 2)⁷ gave tetrahydrofuran derivative as a mixture of diastereoisomers. This mixture was analyzed with ^1H NMR (diastereoselectivity was based on integration of doublets at 5 - 5.5 ppm) and separated at preparative thin layer chromatography with hexane : diethyl ether (1 : 3). Solid compounds were additionally crystalized form hexane : ethyl acetate mixture.

⁷ For reaction in entry 7 (R = *tert*-butyl; table 1 in the article) hexane : chloroform (3 : 1) was used as eluent.

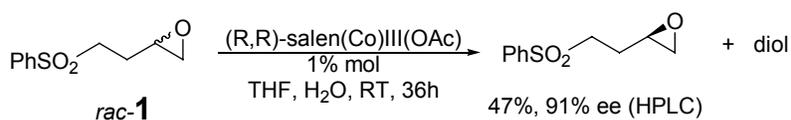
TLC analyses of reaction mixture (1 with benzaldehyde) before chromatographic purification



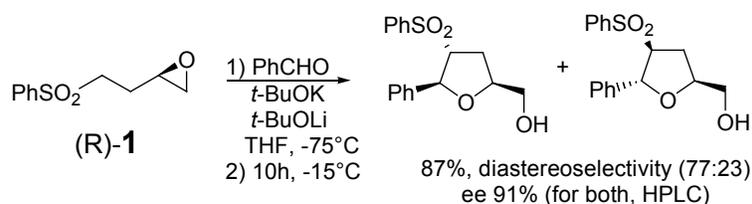
Reaction of aldol adducts of benzaldehyde with 1 and *p*-methoxybenzaldehyde under optimized reaction conditions (scheme 3 in the article).

Mixture of diastereoisomers of **2** (243 mg, 0.76 mmol) and *p*-methoxybenzaldehyde (105 mg, 0.77 mmol) were subjected to optimized reaction conditions. Flash chromatography separation of tetrahydrofuran derivatives gave mixture of products as colorless oil (193 mg). This mixture was analyzed with ¹H NMR. All four possible products, major and minor diastereoisomers of products from benzaldehyde and *p*-methoxybenzaldehyde gave characteristic doublets around 5 - 5.5 ppm, which were undoubtedly assigned and integrated.

⁸ See for example Leonard, J., Lygo, B., Procter, G., *Advanced Practical Organic Chemistry*, 2nd Ed.; Stanley Thornes (Publishers) Ltd 1998, p. 149.



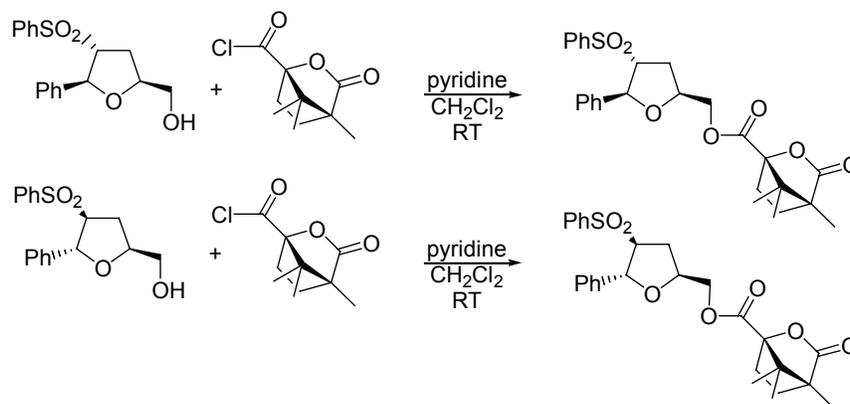
Synthesis of enantiomerically enriched 1 (scheme 5 in the article) was performed according to procedure described in literature⁹ (colorless oil, yield 47%; ee 91% of (R)-1)



Reaction of enantiomerically enriched 1 (scheme 5 in the article) with benzaldehyde was performed according to optimized procedure. Products were analyzed on HPLC with chiral column (91% ee for both isomers).

Derivatization of chiral tetrahydrofuranes

Products of reaction of enantiomerically enriched 1 with benzaldehyde were derivatized by esterification with (-)- ω -camphanic acid chloride to establish absolute configuration by X-Ray analyses.

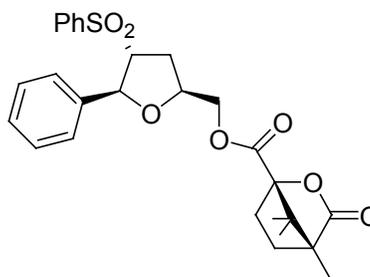
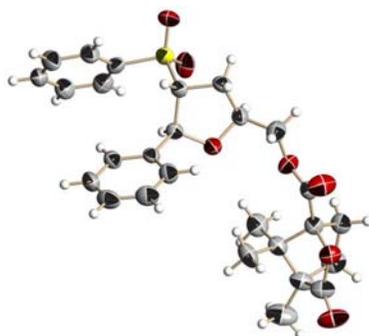


To solution of (-)- ω -camphanic acid chloride (61mg, 0.28 mmol) in CH_2Cl_2 (4 mL) pyridine (100mg, 1.27 mmol) and **3** (60 mg, 0.19 mmol) were added consecutively. Mixture was stirred at rt for 1h and separated by flash chromatography (hexane : ethyl acetate, 3 : 1). Crystallization from hexane : ethyl

⁹ Jin C., Ramirez R. D., Gopalan A. S., *Tetrahedron Lett.* **2001**, 42, 4747.

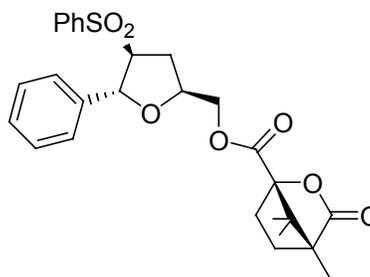
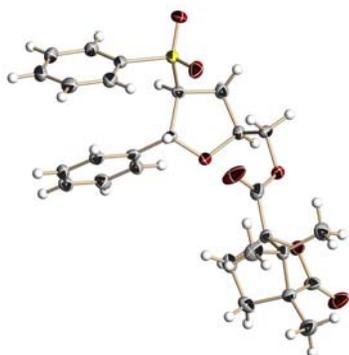
acetate mixture gave product (60mg, 64% yield). Second crystallization from the same mixture formed crystals appropriate for X-Ray analysis.

[(2S,4R,5S)-5-phenyl-4-(phenylsulfonyl)tetrahydro-2-furanyl]methyl (1S)-4,7,7-trimethyl-3-oxo-2-oxabicyclo[2.2.1]heptane-1-carboxylate (derivative of 3a, CCDC 269770)



Mp: 172-174°C. ¹H NMR (200 MHz, CDCl₃): δ 7.82-7.89 (m, 2H), 7.46-7.69 (m, 3H), 7.10-7.25 (m, 5H), 5.33 (d, *J* = 5.4 Hz, 1H), 4.49-4.63 (m, 1H), 4.34-4.52 (m, 2H), 3.71-3.82 (m, 1H), 2.56 (ddd, *J* = 13.6, 5.9, 3.5 Hz, 1H), 2.26-2.48 (m, 2H), 1.84-2.12 (m, 2H), 1.64-1.78 (m, 1H), 1.12 (s, 3H), 1.05 (s, 3H), 0.94 (s, 3H). ¹³C NMR (50 MHz, CDCl₃): δ 178.0, 167.4, 139.6, 137.8, 134.0, 129.4, 128.5, 128.2, 125.9, 91.0, 80.4, 76.6, 70.6, 65.3, 54.8, 54.3, 30.8, 29.7, 28.9, 16.7, 16.7, 9.7.

[(2S,4S,5R)-5-phenyl-4-(phenylsulfonyl)tetrahydro-2-furanyl]methyl (1S)-4,7,7-trimethyl-3-oxo-2-oxabicyclo[2.2.1]heptane-1-carboxylate (derivative of 3b, CCDC 269769)

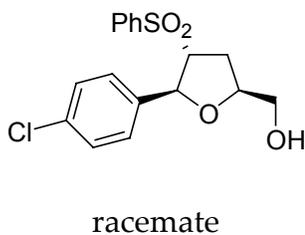


Mp: 133-135°C. ¹H NMR (200 MHz, CDCl₃): δ 7.81-7.90 (m, 2H), 7.46-7.68 (m, 3H), 7.10-7.27 (m, 5H), 5.46 (d, *J* = 6.1 Hz, 1H), 4.30-4.59 (m, 3H), 3.89 (dt, *J* = 8.5, 6.2 Hz, 1H), 2.34-2.56 (m, 3H), 1.83-2.14 (m, 2H), 1.63-1.78 (m, 1H), 1.12 (s, 3H), 1.08 (s, 3H), 0.97 (s, 3H). ¹³C NMR (50 MHz, CDCl₃): δ 178.0, 167.2, 139.5, 137.9, 134.1, 129.4, 128.6, 128.5, 128.1, 125.7, 91.1, 79.8, 76.5, 70.7, 65.5, 54.8, 54.3, 30.6, 29.0, 16.8, 16.7, 9.7.

Crystallographic data (excluding structural factors) for the structures reported in this paper has been deposited with the Cambridge Crystallographic Data Center and allocated the deposition numbers CCDC 269769 and 269770. Copies of the data can be obtained free of charge on application to CCDC, 12 Union Road, Cambridge CB2 1EW, UK (Fax: Int code + (1223)336-033; E-mail:deposit@ccdc.cam.ac.uk).

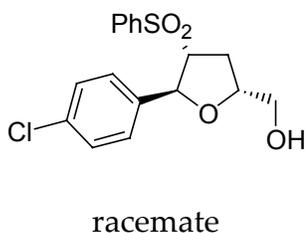
Characterization data of tetrahydrofuranes

Entry 1, major isomer **[(2S*,4R*,5S*)-5-(4-chlorophenyl)-4-(phenylsulfonyl)tetrahydro-2-furanyl] methanol**



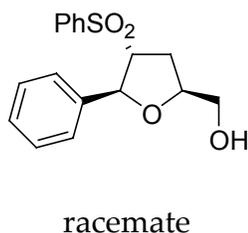
Mp: 106-107°C. IR (KBr): 3307, 2900, 1585, 1492, 1448, 1306, 1143, 1089, 1058, 1014, 940, 822, 750, 724, 685, 604, 567, 504 cm⁻¹. ¹H NMR (200 MHz, CDCl₃): δ 7.79-7.90 (m, 2H), 7.47-7.72 (m, 3H), 7.06-7.26 (m, 4H), 5.29 (d, *J* = 5.9 Hz, 1H), 4.25-4.35 (m, 1H), 3.84-3.98 (m, 1H), 3.58-3.73 (m, 2H), 2.47 (ddd, *J* = 13.6, 6.0, 3.5 Hz, 1H), 2.15-2.35 (m, 1H), 1.85-1.96 (m, 1H). ¹³C NMR (50 MHz, CDCl₃): δ 138.3, 138.0, 134.1, 129.5, 128.7, 128.5, 127.6, 79.9, 79.7, 70.9, 63.2, 29.5. HRMS (ESI): calcd for C₁₇H₁₇SO₄Cl³⁵Na 375.0428, found 375.0447. Anal. calcd for C₁₇H₁₈SO₄Cl C, 57.87; H, 4.86; S, 9.09; Cl, 10.05. Found C, 58.04; H, 4.97; S, 9.17; Cl, 9.95.

Entry 1, minor isomer **[(2R*,4R*,5S*)-5-(4-chlorophenyl)-4-(phenylsulfonyl)tetrahydro-2-furanyl] methanol**



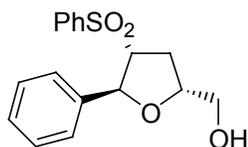
Mp: 97-98°C. IR (KBr): 3390, 2870, 1584, 1490, 1448, 1306, 1146, 1087, 1013, 804, 756, 725, 686, 590, 557, 503 cm⁻¹. ¹H NMR (200 MHz, CDCl₃): δ 7.85-7.93 (m, 2H), 7.50-7.73 (m, 3H), 7.15-7.31 (m, 4H), 5.52 (d, *J* = 5.7 Hz, 1H), 4.29-4.43 (m, 1H), 3.76-3.92 (m, 2H), 3.60-3.75 (m, 1H), 2.36-2.54 (m, 1H), 2.12-2.29 (m, 1H), 1.97-2.06 (m, 1H). ¹³C NMR (50 MHz, CDCl₃): δ 138.7, 137.8, 134.1, 133.8, 129.4, 128.7, 128.5, 127.1, 79.8, 78.9, 71.3, 63.4, 29.7. HRMS (ESI): calcd for C₁₇H₁₇SO₄Cl³⁵Na 375.0428, found 375.0438. Anal. calcd for C₁₇H₁₈SO₄Cl C, 57.87; H, 4.86; S, 9.09; Cl, 10.05. Found C, 57.76; H, 4.91; S, 9.18; Cl, 9.90.

Entry 2, major isomer, **3a**
[(2S*,4R*,5S*)-5-phenyl-4-(phenylsulfonyl)tetrahydro-2-furanyl]methanol



Mp: 118-119°C. IR (KBr): 3532, 2879, 1446, 1287, 1189, 1147, 1108, 1084, 1026, 937, 770, 749, 719, 690, 618, 602, 534, 518 cm⁻¹. ¹H NMR (500 MHz, CDCl₃): δ 7.82-7.86 (m, 2H), 7.60-7.65 (m, 1H), 7.48-7.53 (m, 2H), 7.21-7.25 (m, 3H), 7.11-7.16 (m, 2H), 5.30 (d, *J* = 5.8 Hz, 1H), 4.31-4.37 (m, 1H), 3.87-3.93 (m, 1H), 3.70-3.75 (m, 1H), 3.64-3.69 (m, 1H), 2.59 (ddd, *J* = 13.8, 6.0, 3.3 Hz, 1H), 2.25-2.33 (m, 1H), 1.91-1.96 (m, 1H). ¹³C NMR (50 MHz, CDCl₃): δ 139.7, 138.1, 134.0, 129.7, 128.6, 128.3, 126.1, 80.5, 79.8, 71.0, 63.2, 29.4. HRMS (ESI): calcd for C₁₇H₁₈SO₄Na 341.0818, found 341.0838. Anal. calcd for C₁₇H₁₈SO₄ C, 64.13; H, 5.70; S, 10.07. Found C, 64.27; H, 5.88; S, 10.16.

Entry 2, minor isomer,
3b

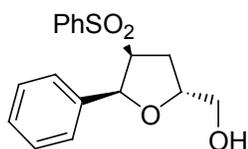


racemate

[(2R*,4R*,5S*)-5-phenyl-4-(phenylsulfonyl)tetrahydro-2-furanyl]methanol

Mp: 91-92°C. IR (KBr): 3508, 2935, 1447, 1322, 1291, 1148, 1085, 1036, 832, 763, 741, 721, 699, 605, 583, 529 cm⁻¹. ¹H NMR (500 MHz, CDCl₃): δ 7.87-7.90 (m, 2H), 7.61-7.65 (m, 1H), 7.50-7.55 (m, 2H), 7.20-7.30 (m, 5H), 5.54 (d, *J* = 5.3 Hz, 1H), 4.35-4.41 (m, 1H), 3.88-3.94 (m, 1H), 3.81-3.86 (m, 1H), 3.65-3.71 (m, 1H), 2.42-2.46 (m, 1H), 2.22-2.27 (m, 1H), 2.17-2.21 (m, 1H). ¹³C NMR (50 MHz, CDCl₃): δ 140.1, 138.0, 134.1, 129.4, 128.7, 128.6, 128.0, 125.6, 79.7, 79.6, 71.3, 63.5, 29.3. HRMS (ESI): calcd for C₁₇H₁₈SO₄Na 341.0818, found 341.0834. Anal. calcd for C₁₇H₁₈SO₄ C, 64.13; H, 5.70; S, 10.07. Found C, 64.13; H, 5.86; S, 9.96.

Isolated in optimization
experiment only, **3d**

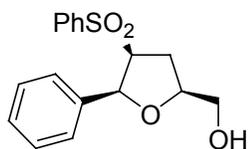


racemate

[(2R*,4S*,5S*)-5-phenyl-4-(phenylsulfonyl)tetrahydro-2-furanyl]methanol

Mp: 93°C (dec.). IR (KBr): 3256, 2886, 1447, 1307, 1193, 1143, 1115, 1085, 939, 732, 686, 557, 512 cm⁻¹. ¹H NMR (500 MHz, CDCl₃): δ 7.47-7.49 (m, 1H), 7.35-7.38 (m, 2H), 7.27-7.31 (m, 2H), 7.19-7.25 (m, 3H), 7.14-7.18 (m, 2H), 5.36 (d, *J* = 6.0 Hz, 1H), 4.72-4.77 (m, 1H), 4.15-4.20 (m, 1H), 3.81-3.87 (m, 1H), 3.58-3.65 (m, 1H), 2.59 (ddd, *J* = 14.0, 7.3, 3.9 Hz, 1H), 2.39-2.46 (m, 1H), 2.08-2.12 (m, 1H), 1.88-1.93 (m, 1H). ¹³C NMR (125 MHz, CDCl₃): δ 139.2, 135.8, 132.8, 128.8, 128.0, 127.8, 127.7, 127.3, 81.3, 78.6, 67.9, 64.5, 29.2. MS (ESI): calcd for C₁₇H₁₈SO₄Na 341.0818, found 341.1. Anal. calcd for C₁₇H₁₈SO₄ C, 64.13; H, 5.70; S, 10.07. Found C, 62.91; H, 5.43; S, 9.11.

Isolated in optimization
experiment only, **3c**

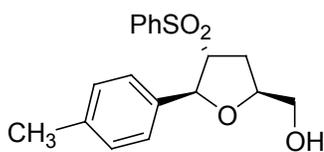


racemate

[(2S*,4S*,5S*)-5-phenyl-4-(phenylsulfonyl)tetrahydro-2-furanyl]methanol

Mp: 149-150°C. IR (KBr): 3521, 2889, 1446, 1305, 1290, 1140, 1114, 1083, 1057, 757, 732, 687, 575, 508 cm⁻¹. ¹H NMR (500 MHz, CDCl₃): δ 7.42-7.46 (m, 1H), 7.31-7.35 (m, 2H), 7.17-7.29 (m, 5H), 5.07 (d, *J* = 6.5 Hz, 1H), 4.17-4.22 (m, 1H), 4.10-4.15 (m, 1H), 4.04 (dd, *J* = 12.1, 2.7 Hz, 1H), 3.81 (dd, *J* = 12.1, 4.5 Hz, 1H), 2.77-2.84 (m, 1H), 2.39-2.47 (m, 1H), 2.50 (s br, 1H). ¹³C NMR (125 MHz, CDCl₃): δ 139.2, 134.6, 132.8, 128.8, 128.1, 127.8, 127.7, 127.7, 127.6, 127.6, 127.5, 81.6, 78.1, 67.1, 63.5, 28.5. MS (ESI): calcd for C₁₇H₁₈SO₄Na 341.0818, found 341.1. Anal. calcd for C₁₇H₁₈SO₄ C, 64.13; H, 5.70; S, 10.07. Found C, 64.11; H, 5.65; S, 10.00.

Entry 3, major isomer

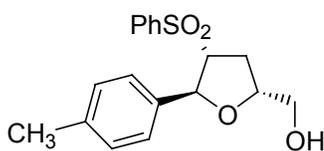


racemate

[(2S*,4R*,5S*)-5-(4-methylphenyl)-4-(phenylsulfonyl)tetrahydro-2-furanyl] methanol

Mp: 100-102°C. IR (KBr): 3497, 3306, 2917, 2877, 1516, 1446, 1306, 1144, 1083, 1036, 938, 818, 754, 724, 688, 612, 583, 561, 543, 516 cm⁻¹. ¹H NMR (500 MHz, CDCl₃): δ 7.82-7.85 (m, 2H), 7.59-7.64 (m, 1H), 7.47-7.53 (m, 2H), 6.99-7.05 (m, 4H), 5.26 (d, *J* = 5.8 Hz, 1H), 4.28-4.34 (m, 1H), 3.85-3.91 (m, 1H), 3.68-3.74 (m, 1H), 3.61-3.67 (m, 1H), 2.59 (ddd, *J* = 13.8, 6.0, 3.3 Hz, 1H), 2.29 (s, 3H), 2.24-2.32 (m, 1H), 1.95-2.00 (m, 1H). ¹³C NMR (125 MHz, CDCl₃): δ 138.2, 138.1, 136.7, 133.9, 129.3, 129.2, 128.6, 126.1, 80.4, 79.7, 70.9, 63.2, 29.5, 21.2. HRMS (ESI): calcd for C₁₈H₂₀SO₄Na 355.0975, found 355.0955. Anal. calcd for C₁₈H₂₀SO₄ C, 65.04; H, 6.06; S, 9.65. Found C, 64.83; H, 5.93; S, 9.51.

Entry 3, minor isomer

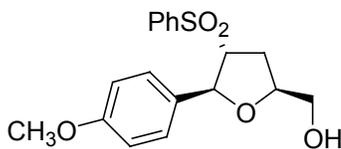


racemate

[(2R*,4R*,5S*)-5-(4-methylphenyl)-4-(phenylsulfonyl)tetrahydro-2-furanyl] methanol

Mp: 116-117°C. IR (KBr): 3359, 2956, 1516, 1450, 1305, 1290, 1241, 1148, 1084, 1042, 973, 827, 774, 755, 717, 691, 624, 596, 565, 536, 519 cm⁻¹. ¹H NMR (500 MHz, CDCl₃): δ 7.87-7.90 (m, 2H), 7.61-7.65 (m, 1H), 7.50-7.55 (m, 2H), 7.06-7.11 (m, 4H), 5.50 (d, *J* = 5.3 Hz, 1H), 4.33-4.39 (m, 1H), 3.86-3.92 (m, 1H), 3.79-3.85 (m, 1H), 3.63-3.69 (m, 1H), 2.41-2.48 (m, 1H), 2.30 (s, 3H), 2.20-2.27 (m, 1H), 2.14-2.20 (m, 1H). ¹³C NMR (125 MHz, CDCl₃): δ 138.0, 137.7, 137.0, 134.0, 129.3, 129.3, 128.6, 125.6, 79.5, 79.5, 71.2, 63.6, 29.4, 21.0. HRMS (ESI): calcd for C₁₈H₂₀SO₄Na 355.0975, found 355.0992. Anal. calcd for C₁₈H₂₀SO₄ C, 65.04; H, 6.06; S, 9.65. Found C, 65.15; H, 6.19; S, 9.71.

Entry 4, major isomer

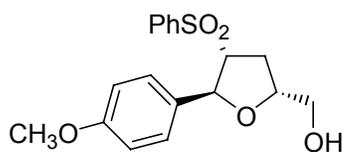


racemate

[(2S*,4R*,5S*)-5-(4-methoxyphenyl)-4-(phenylsulfonyl)tetrahydro-2-furanyl]methanol

Mp: 87-89°C. IR (KBr): 3261, 2958, 1613, 1585, 1515, 1448, 1305, 1252, 1172, 1147, 1084, 1034, 831, 752, 718, 690, 638, 622, 607, 586, 544, 517 cm⁻¹. ¹H NMR (200 MHz, CDCl₃): δ 7.78-7.87 (m, 2H), 7.44-7.67 (m, 3H), 7.00-7.09 (m, 2H), 6.70-6.80 (m, 2H), 5.22 (d, *J* = 6.2 Hz, 1H), 4.23-4.37 (m, 1H), 3.84-3.94 (m, 1H), 3.76 (s, 3H), 3.58-3.75 (m, 2H), 2.50 (ddd, *J* = 13.6, 6.2, 3.7 Hz, 1H), 2.20-2.39 (m, 1H), 1.88 (s br, 1H). ¹³C NMR (50 MHz, CDCl₃): δ 159.6, 138.1, 133.9, 131.6, 129.3, 128.5, 127.5, 113.9, 80.4, 79.5, 70.7, 63.2, 55.3, 29.4. HRMS (ESI): calcd for C₁₈H₂₀SO₅Na 371.0924, found 371.0944. Anal. calcd for C₁₈H₂₀SO₅ C, 62.05; H, 5.79; S, 9.20. Found C, 62.26; H, 5.74; S, 9.03.

Entry 4, minor isomer

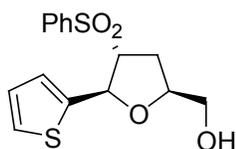


racemate

[(2R*,4R*,5S*)-5-(4-methoxyphenyl)-4-(phenylsulfonyl)tetrahydro-2-furanyl]methanol

Mp: 93-94°C. IR (KBr): 3340, 2938, 1611, 1584, 1512, 1451, 1304, 1247, 1177, 1147, 1085, 1033, 972, 841, 777, 758, 690, 573, 546, 528 cm⁻¹. ¹H NMR (200 MHz, CDCl₃): δ 7.83-7.91 (m, 2H), 7.46-7.69 (m, 3H), 7.07-7.19 (m, 2H), 6.75-6.85 (m, 2H), 5.46 (d, *J* = 5.7 Hz, 1H), 4.30-4.44 (m, 1H), 3.76-3.94 (m, 2H), 3.77 (s, 3H), 3.61-3.72 (m, 1H), 2.38-2.54 (m, 1H), 2.17-2.34 (m, 1H), 1.85 (s br, 1H). ¹³C NMR (50 MHz, CDCl₃): δ 159.4, 138.0, 134.0, 131.9, 129.3, 128.6, 127.1, 114.0, 79.5, 79.4, 71.0, 63.6, 55.3, 29.4. HRMS (ESI): calcd for C₁₈H₂₀SO₅Na 371.0924, found 371.0942. Anal. calcd for C₁₈H₂₀SO₅ C, 62.05; H, 5.79; S, 9.20. Found C, 62.19; H, 5.75; S, 9.18.

Entry 5, major isomer

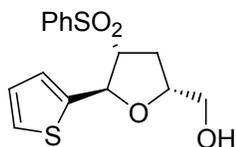


racemate

[(2S*,4R*,5R*)-4-(phenylsulfonyl)-5-(2-thienyl)tetrahydro-2-furanyl]methanol

Mp: 102-103°C. IR (KBr): 3534, 3106, 2974, 2933, 2878, 1582, 1478, 1446, 1386, 1288, 1274, 1190, 1147, 1108, 1147, 1108, 1082, 1014, 950, 936, 852, 839, 783, 752, 719, 689, 609, 552, 527 cm⁻¹. ¹H NMR (200 MHz, CDCl₃): δ 7.83-7.92 (m, 2H), 7.46-7.70 (m, 3H), 7.17 (dd, *J* = 5.1, 1.1 Hz, 1H), 6.82 (dd, *J* = 5.1, 3.6 Hz, 1H), 6.64 (d, *J* = 3.6 Hz, 1H), 5.56 (d, *J* = 6.0 Hz, 1H), 4.26-4.40 (m, 1H), 3.73-3.94 (m, 2H), 3.53-3.68 (m, 1H), 2.48-2.63 (m, 1H), 2.27-2.46 (m, 1H), 1.91-2.02 (m, 1H). ¹³C NMR (50 MHz, CDCl₃): δ 143.0, 137.8, 134.1, 129.4, 128.5, 126.8, 125.5, 125.2, 79.9, 76.9, 71.2, 63.1, 28.9. HRMS (ESI): calcd for C₁₅H₁₆S₂O₄Na 347.0382, found 347.0379. Anal. calcd for C₁₅H₁₆S₂O₄ C, 55.53; H, 4.97; S, 19.77. Found C, 55.35; H, 4.97; S, 20.04.

Entry 5, minor isomer

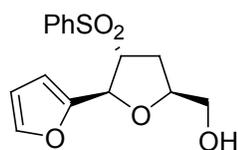


racemate

[(2R*,4R*,5R*)-4-(phenylsulfonyl)-5-(2-thienyl)tetrahydro-2-furanyl]methanol

Mp: 92-93°C. IR (KBr): 3516, 3071, 2914, 1584, 1448, 1395, 1322, 1292, 1150, 1086, 1071, 1025, 831, 764, 722, 688, 605, 582, 527 cm⁻¹. ¹H NMR (200 MHz, CDCl₃): δ 7.86-7.94 (m, 2H), 7.48-7.71 (m, 3H), 7.20 (dd, *J* = 5.1, 1.3 Hz, 1H), 6.86 (dd, *J* = 5.1, 3.6 Hz, 1H), 6.75-6.80 (m, 1H), 5.71 (d, *J* = 5.4 Hz, 1H), 4.32-4.46 (m, 1H), 3.91-4.05 (m, 1H), 3.78-3.91 (m, 1H), 3.59-3.73 (m, 1H), 2.25-2.61 (m, 2H), 2.02-2.12 (m, 1H). ¹³C NMR (50 MHz, CDCl₃): δ 143.4, 137.8, 134.1, 129.4, 128.6, 127.0, 125.6, 125.2, 79.4, 76.5, 71.2, 63.3, 28.9. HRMS (ESI): calcd for C₁₅H₁₆S₂O₄Na 347.0382, found 347.0398. Anal. calcd for C₁₅H₁₆S₂O₄ C, 55.53; H, 4.97; S, 19.77. Found C, 55.53; H, 5.06; S, 20.11.

Entry 6, major isomer

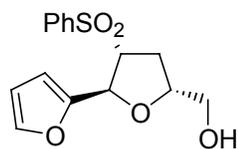


racemate

[(2S*,4R*,5S*)-5-(2-furyl)-4-(phenylsulfonyl)-tetrahydro-2-furanyl]methanol

Mp: 69-70°C. IR (KBr): 3350, 3263, 2932, 2876, 1586, 1504, 1449, 1348, 1306, 1147, 1110, 1086, 1045, 1015, 925, 784, 746, 721, 685, 599, 566 cm⁻¹. ¹H NMR (200 MHz, CDCl₃): δ 7.76-7.87 (m, 2H), 7.42-7.65 (m, 3H), 7.21-7.25 (m, 1H), 6.17 (dd, *J* = 3.3, 1.9 Hz, 1H), 6.08 (d, *J* = 3.3 Hz, 1H), 5.23 (d, *J* = 6.8 Hz, 1H), 4.23-4.37 (m, 1H), 4.01-4.14 (m, 1H), 3.74-3.87 (m, 1H), 3.48-3.63 (m, 1H), 2.54-2.70 (m, 1H), 2.31-2.49 (m, 1H), 2.00-2.12 (m, 1H). ¹³C NMR (50 MHz, CDCl₃): δ 150.3, 143.0, 137.8, 133.9, 129.2, 128.3, 110.4, 109.3, 79.7, 74.3, 67.0, 63.6, 28.9. HRMS (ESI): calcd for C₁₅H₁₆SO₅Na 331.0611, found 331.0625. Anal. calcd for C₁₅H₁₆SO₅ C, 58.43; H, 5.23; S, 10.40. Found C, 58.40; H, 5.42; S, 10.88.

Entry 6, minor isomer

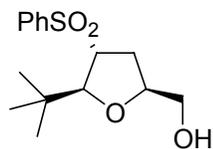


racemate

[(2R*,4R*,5S*)-5-phenyl-4-(phenylsulfonyl)tetrahydro-2-furanyl]methanol

Mp: 77-78°C. IR (KBr): 3467, 3133, 2932, 1584, 1504, 1448, 1301, 1246, 1149, 1103, 1086, 1069, 1046, 1016, 967, 915, 883, 834, 751, 723, 687, 599, 562 cm⁻¹. ¹H NMR (200 MHz, CDCl₃): δ 7.82-7.90 (m, 2H), 7.45-7.68 (m, 3H), 7.24-7.28 (m, 1H), 6.21 (dd, *J* = 3.3, 1.8 Hz, 1H), 6.13 (d, *J* = 3.3 Hz, 1H), 5.41 (d, *J* = 5.7 Hz, 1H), 4.28-4.42 (m, 1H), 4.08-4.22 (m, 1H), 3.74-3.87 (m, 1H), 3.54-3.69 (m, 1H), 2.28-2.58 (m, 2H), 2.10-2.21 (m, 1H). ¹³C NMR (50 MHz, CDCl₃): δ 151.3, 143.0, 137.7, 134.0, 129.3, 128.4, 110.4, 108.8, 79.5, 73.6, 67.2, 63.3, 28.6. HRMS (ESI): calcd for C₁₅H₁₆SO₅Na 331.0611, found 331.0626. Anal. calcd for C₁₅H₁₆SO₅ C, 58.43; H, 5.23; S, 10.40. Found C, 58.63; H, 5.24; S, 10.41.

Entry 7, major isomer

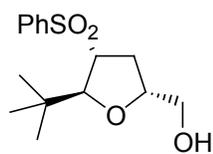


racemate

[(2S*,4R*,5S*)-5-(tert-butyl)-4-(phenylsulfonyl)-tetrahydro-2-furanyl]methanol

Mp: 100-101°C. IR (KBr): 3462, 3067, 2954, 2898, 1585, 1481, 1447, 1397, 1366, 1305, 1149, 1085, 1038, 998, 961, 850, 781, 752, 720, 690, 648, 616, 583, 553, 530 cm⁻¹. ¹H NMR (200 MHz, CDCl₃): δ 7.87-7.96 (m, 2H), 7.53-7.73 (m, 3H), 4.11-4.28 (m, 1H), 4.20 (d, *J* = 4.9 Hz, 1H), 3.74-3.87 (m, 1H), 3.41-3.59 (m, 2H), 2.25 (ddd, *J* = 13.9, 4.9, 0.8 Hz, 1H), 1.77-2.04 (m, 2H), 0.79 (s, 9H). ¹³C NMR (50 MHz, CDCl₃): δ 138.0, 134.0, 129.4, 128.9, 85.4, 79.1, 66.0, 63.3, 34.5, 30.1, 25.5. HRMS (ESI): calcd for C₁₅H₂₂SO₄Na 321.1131, found 321.1138. Anal. calcd for C₁₅H₂₂SO₄ C, 60.38; H, 7.43; S, 10.75. Found C, 60.23; H, 7.49; S, 10.25.

Entry 7, minor isomer

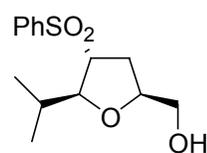


racemate

[(2R*,4R*,5S*)-5-(*tert*-butyl)-4-(phenylsulfonyl)-tetrahydro-2-furanyl]methanol

Mp: 104-106°C. IR (KBr): 3528, 2968, 2922, 2875, 1584, 1478, 1451, 1398, 1370, 1305, 1212, 1148, 1117, 1087, 1059, 1030, 998, 951, 880, 819, 768, 730, 691, 649, 588, 551, 511 cm⁻¹. ¹H NMR (200 MHz, CDCl₃): δ 7.89-7.97 (m, 2H), 7.52-7.73 (m, 3H), 4.18-4.36 (m, 1H), 4.26 (d, *J* = 3.2 Hz, 1H), 3.45-3.80 (m, 3H), 2.33-2.50 (m, 1H), 2.09-2.33 (m, 2H), 0.81 (s, 9H). ¹³C NMR (50 MHz, CDCl₃): δ 137.8, 134.0, 129.4, 128.9, 87.2, 80.8, 65.7, 64.1, 36.1, 29.1, 25.9. HRMS (ESI): calcd for C₁₅H₂₂SO₄Na 321.1131, found 321.1123. Anal. calcd for C₁₅H₂₂SO₄ C, 60.38; H, 7.43; S, 10.75. Found C, 60.19; H, 7.11; S, 10.62.

Entry 8, major isomer

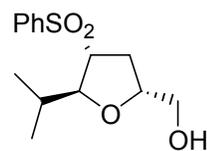


racemate

[(2S*4R*,5S*)-5-(isopropyl)-4-(phenylsulfonyl)-tetrahydro-2-furanyl]methanol

Oil. IR (neat): 3502, 2962, 1585, 1447, 1305, 1146, 1085, 1046, 853, 752, 720, 690, 636, 612, 581, 559 cm⁻¹. ¹H NMR (200 MHz, CDCl₃): δ 7.88-7.95 (m, 2H), 7.53-7.73 (m, 3H), 4.24 (dd, *J* = 4.9, 4.9 Hz, 1H), 4.07-4.20 (m, 1H), 3.74-3.85 (m, 1H), 3.43-3.57 (m, 2H), 2.31 (ddd, *J* = 13.8, 5.6, 2.3 Hz, 1H), 1.93-2.17 (m, 1H), 1.77-1.88 (m, 1H), 1.55-1.77 (m, 1H), 0.78-0.87 (m, 6H). ¹³C NMR (50 MHz, CDCl₃): δ 138.1, 134.0, 129.4, 128.7, 83.0, 78.9, 66.5, 63.2, 32.2, 29.5, 18.8, 16.7. HRMS (ESI): calcd for C₁₄H₂₀SO₄Na 307.0975, found 307.0974. Anal. calcd for C₁₄H₂₀SO₄ C, 59.13; H, 7.09; S, 11.28. Found C, 57.59; H, 7.21; S, 11.06.

Entry 8, minor isomer



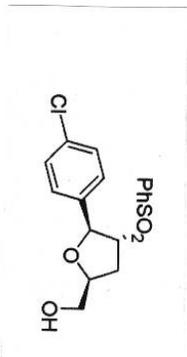
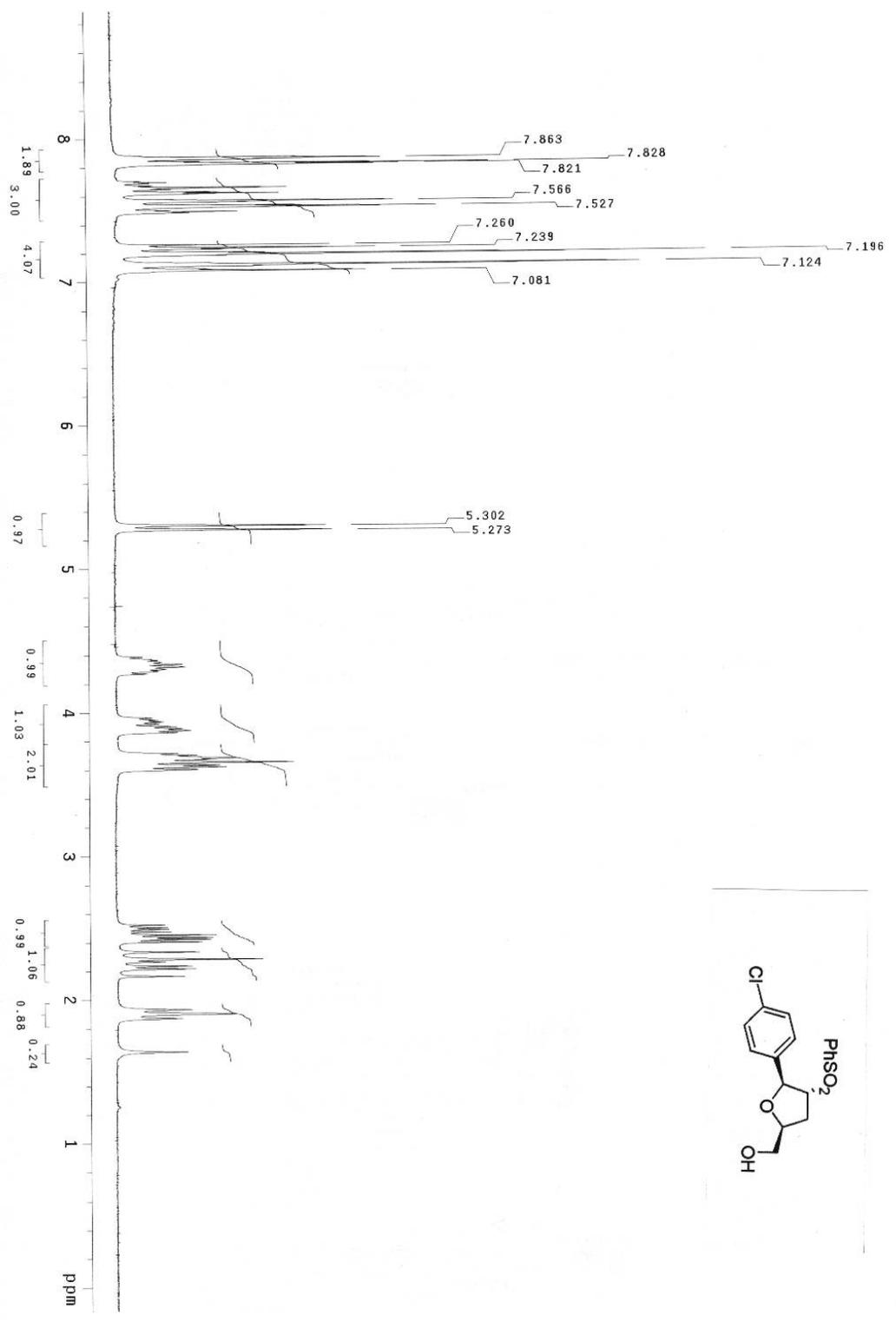
racemate

[(2R*,4R*,5S*)-5-(isopropyl)-4-(phenylsulfonyl)-tetrahydro-2-furanyl]methanol

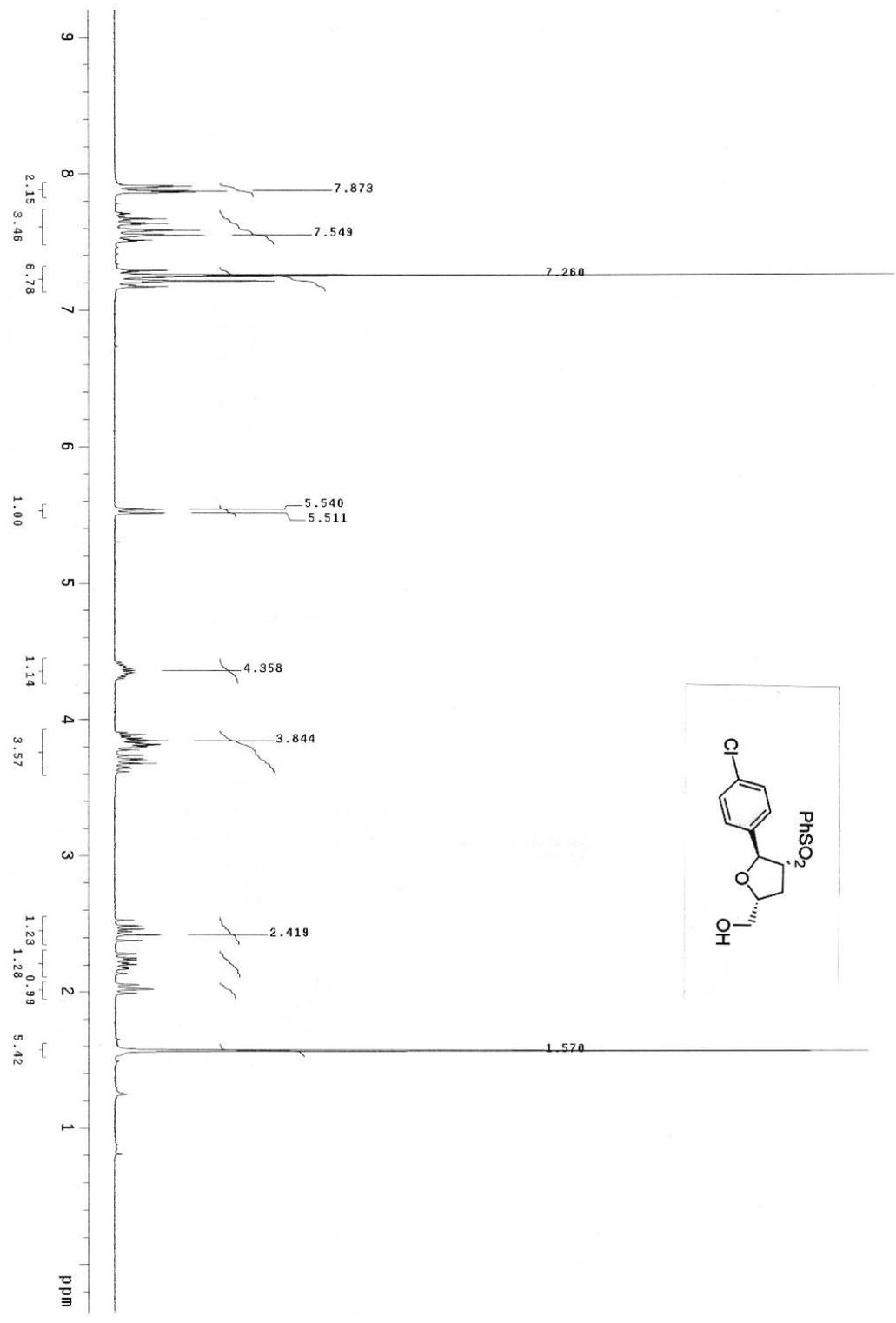
Oil. IR (neat): 3502, 2962, 2876, 1585, 1468, 1447, 1390, 1305, 1148, 1086, 1036, 948, 856, 758, 722, 691, 594, 557 cm⁻¹. ¹H NMR (200 MHz, CDCl₃): δ 7.88-7.96 (m, 2H), 7.53-7.73 (m, 3H), 4.13-4.27 (m, 1H), 4.02-4.16 (m, 1H), 3.68-3.81 (m, 1H), 3.41-3.65 (m, 2H), 2.25-2.42 (m, 1H), 2.00-2.20 (m, 2H), 1.54-1.76 (m, 1H), 0.78-0.93 (m, 6H). ¹³C NMR (50 MHz, CDCl₃): δ 138.0, 134.0, 129.4, 128.7, 83.8, 78.6, 66.9, 63.5, 31.7, 28.8, 19.0, 17.3. HRMS (ESI): calcd for C₁₄H₂₀SO₄Na 307.0975, found 307.0987. Anal. calcd for C₁₄H₂₀SO₄ C, 59.13; H, 7.09; S, 11.28. Found C, 55.56; H, 7.08; S, 11.57.

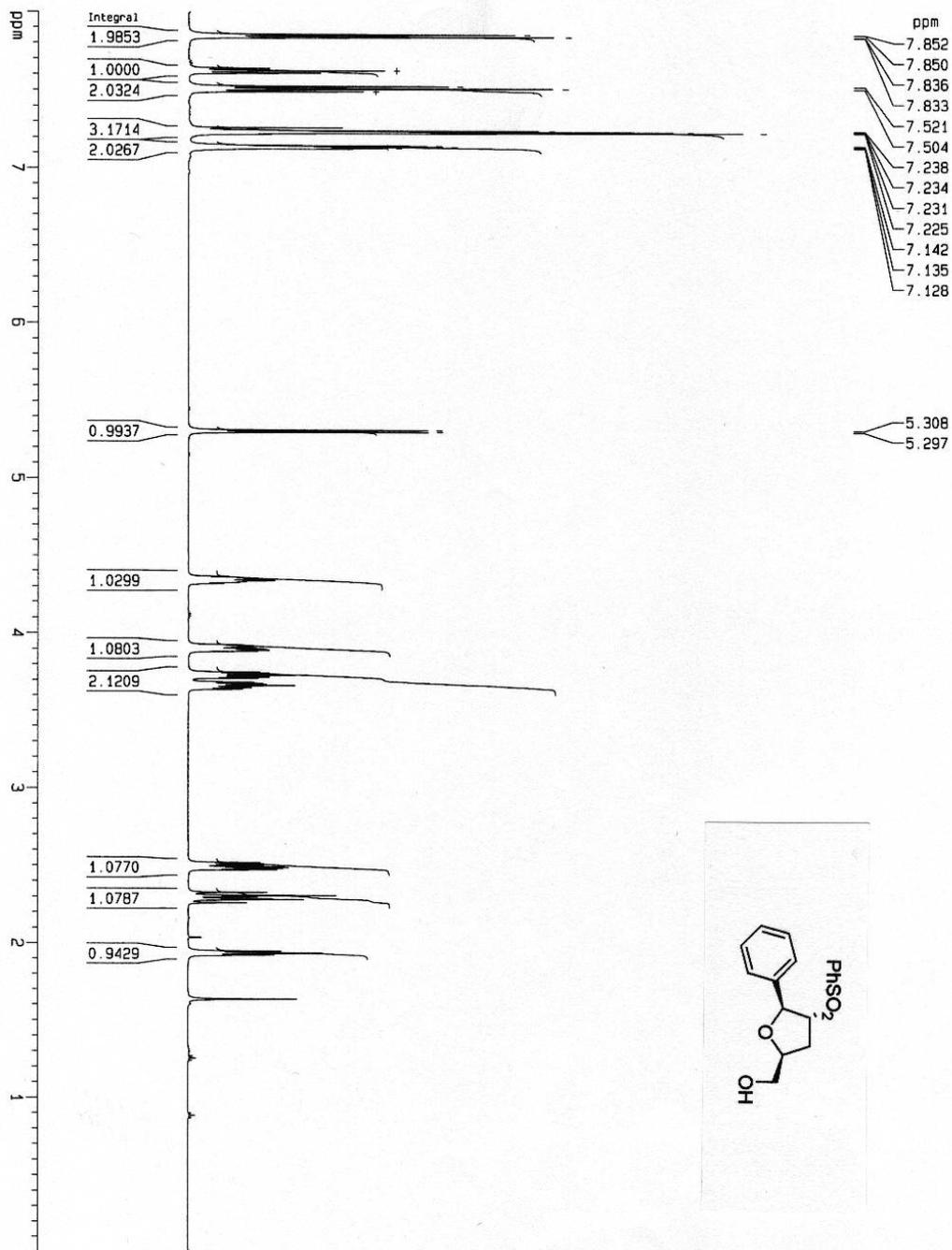
Reproductions of ¹H NMR spectra of tetrahydrofuranes

DK-91aH
Pulse Sequence: szpu1



-DK-91bh
Pulse Sequence: s2pu1





Current Data Parameters
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 PROCNO 1

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 Time 17.31

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 SOLVENT CDCl3
 NS 32
 DS 0
 SMH 6510.417 Hz
 FIDRES 0.198962 Hz
 AQ 2.516523 sec
 RG 128
 DM 76.800 usec
 DE 6.78 usec
 TE 303.0 K
 D1 0.00100000 sec

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 PL1 3.00 dB
 SF01 500.1328887 MHz

F2 - Processing parameters
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 SF 500.1300140 MHz
 NDM no
 SSB 0
 LB 0.00 Hz
 GB 0
 PC 0.30

10 NMR plot parameters
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 F1 4001.04 Hz
 F2P 0.000 ppm
 F2 0.00 Hz
 PPM0K 0.36364 ppm/cm
 HZCM 181.86546 Hz/cm



Current Data Parameters

NAME MB-5838
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters

Date_ 20040302
 Time 10:24
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 PROCBH 5 mm TBI 1H/1
 PULPROG zg
 TO 32768
 SOLVENT CDCl3
 NS 8
 DS 0
 SMH 4495.403 Hz
 FIDRES 0.137219 Hz
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 RG 80.6
 DW 111.200 usec
 DE 6.78 usec
 TE 303.0 K
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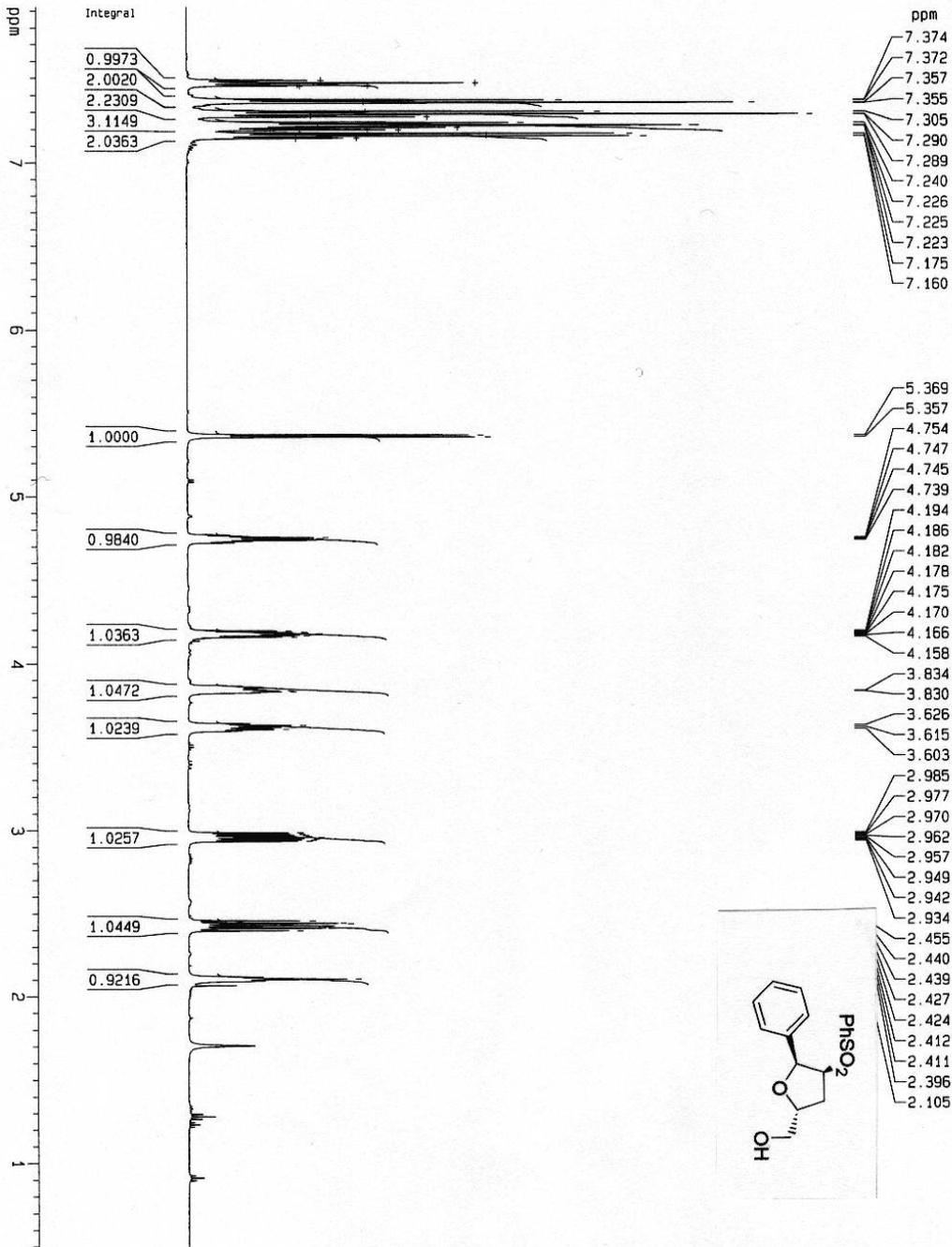
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F2 - Processing parameters

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 MDW EM
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 LB 0.00 Hz
 GB 0
 PC 1.00

10 NMR plot parameters

CX 22.00 cm
 F1P 9.118 ppm
 F1 4080.07 Hz
 F2P -76.99 Hz
 F2 0.37600 ppm/cm
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Current Data Parameters
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 EXPNO 1
 PROCNO 1

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 Time 17.36

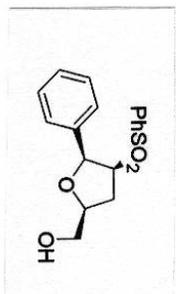
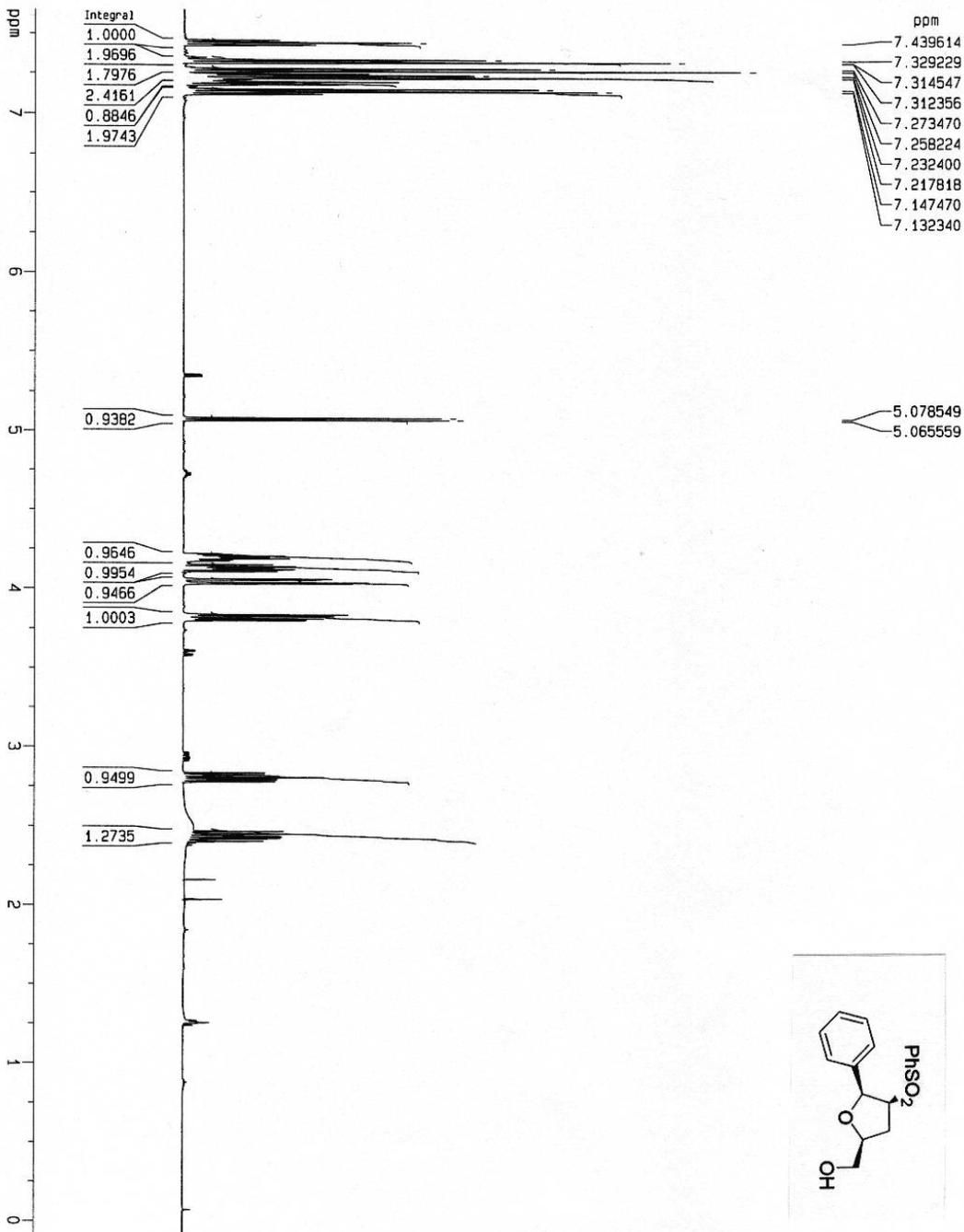
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 PULPROG zg
 TD 32768
 SOLVENT CDCl3
 NS 32
 DS 0

SMH 6510.417 Hz
 FIDRES 0.198682 Hz
 AQ 2.5165323 sec
 RG 64
 DW 76.800 usec
 DE 6.78 usec
 TE 303.0 K
 D1 0.00100000 sec

***** CHANNEL f1 *****
 NUC1 1H
 P1 2.50 usec
 PL1 3.00 dB
 SF01 500.1328897 MHz

F2 - Processing parameters
 SI 32768
 SF 500.1299987 MHz
 RGW no
 SSB 0
 LB 0.00 Hz
 GB 0
 PC 0.30

10 MHz plot parameters
 CX 22.00 cm
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 F1 3963.99 Hz
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 F2 248.51 Hz
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 HZCM 168.88515 Hz/cm



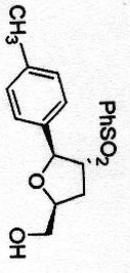
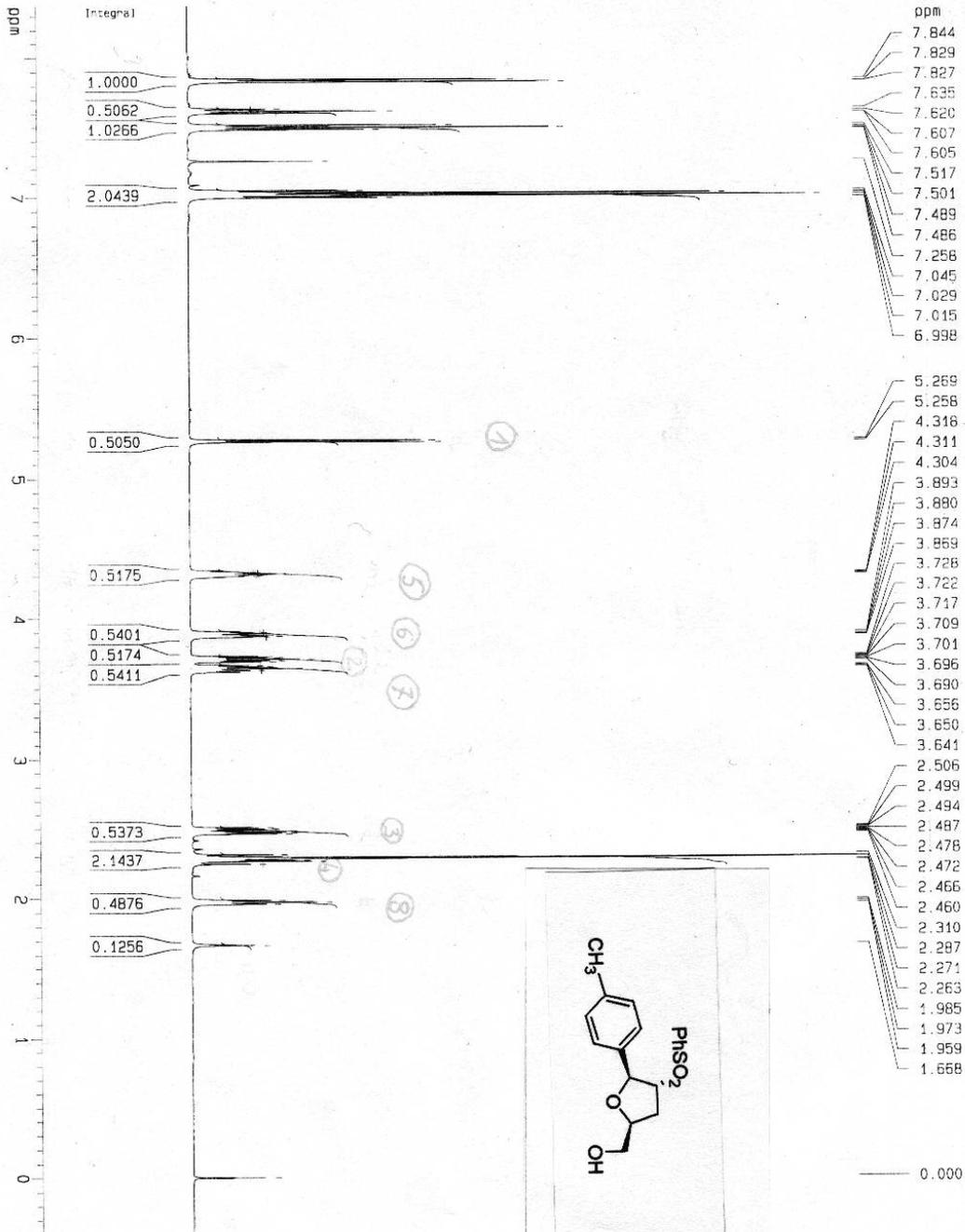
Current Data Parameters
 NAME: M9-5830
 EXPNO: 1
 PROCNO: 1

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 DS: 0
 SWH: 4496.403 Hz
 FIDRES: 0.137219 Hz
 AQ: 3.6438515 sec
 RG: 64
 DM: 111.200 usec
 DE: 6.78 usec
 TE: 303.0 K
 D1: 0.00100000 sec

***** CHANNEL f1 *****
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 P1: 2.50 usec
 PL1: 3.00 dB
 SF01: 500.1320005 MHz

F2 - Processing parameters
 SI: 32768
 SF: 500.1300142 MHz
 MDW: 6M
 SSB: 0
 LB: -1.20 Hz
 GB: 0.2
 PC: 1.00

10 NMR plot parameters
 CX: 22.00 cm
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 F1: 3825.79 Hz
 F2: -0.106 ppm
 F2: -52.82 Hz
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 HZ0H: 176.30054 Hz/cm



Current Data Parameters
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 EXPNO: 1
 PROCNO: 1

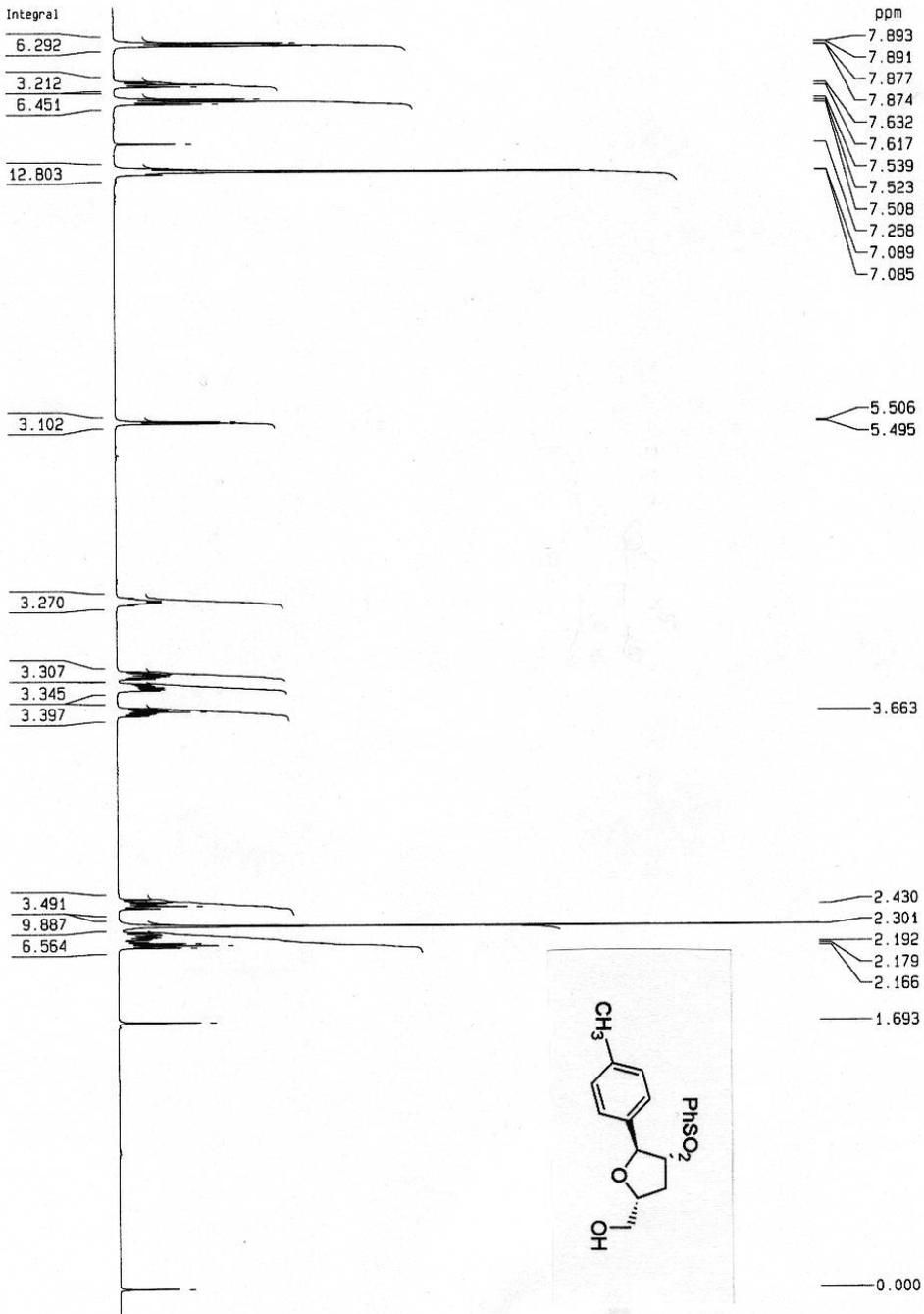
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 SFO: 327.86
 CQ: 13
 NS: 32
 DS: 0
 SWH: 5810.417 Hz
 FIDRES: 0.198882 Hz
 AQ: 2.5186323 sec
 RG: 64
 DW: 76.800 usec
 DE: 6.78 usec
 TE: 303.0 K
 D1: 0.00100000 sec

===== CHANNEL f1 =====
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 P1: 2.50 usec
 PL1: 3.00 dB
 SF01: 500.1328897 MHz

F2 - Processing parameters
 SI: 32768
 SF: 500.1300144 MHz
 KW: no
 SSB: 0
 LB: 0.00 Hz
 GB: 0
 PC: 0.30

13 NMR plot parameters
 CX: 22.00 cm
 F1P: 9.374 dBm
 F1: 4187.59 Hz
 F2P: -0.390 dBm
 F2: -195.04 Hz
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 HZCM: 199.22714 Hz/cm

ppm



Current Data Parameters
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 EXPNO 1
 PROCNO 1

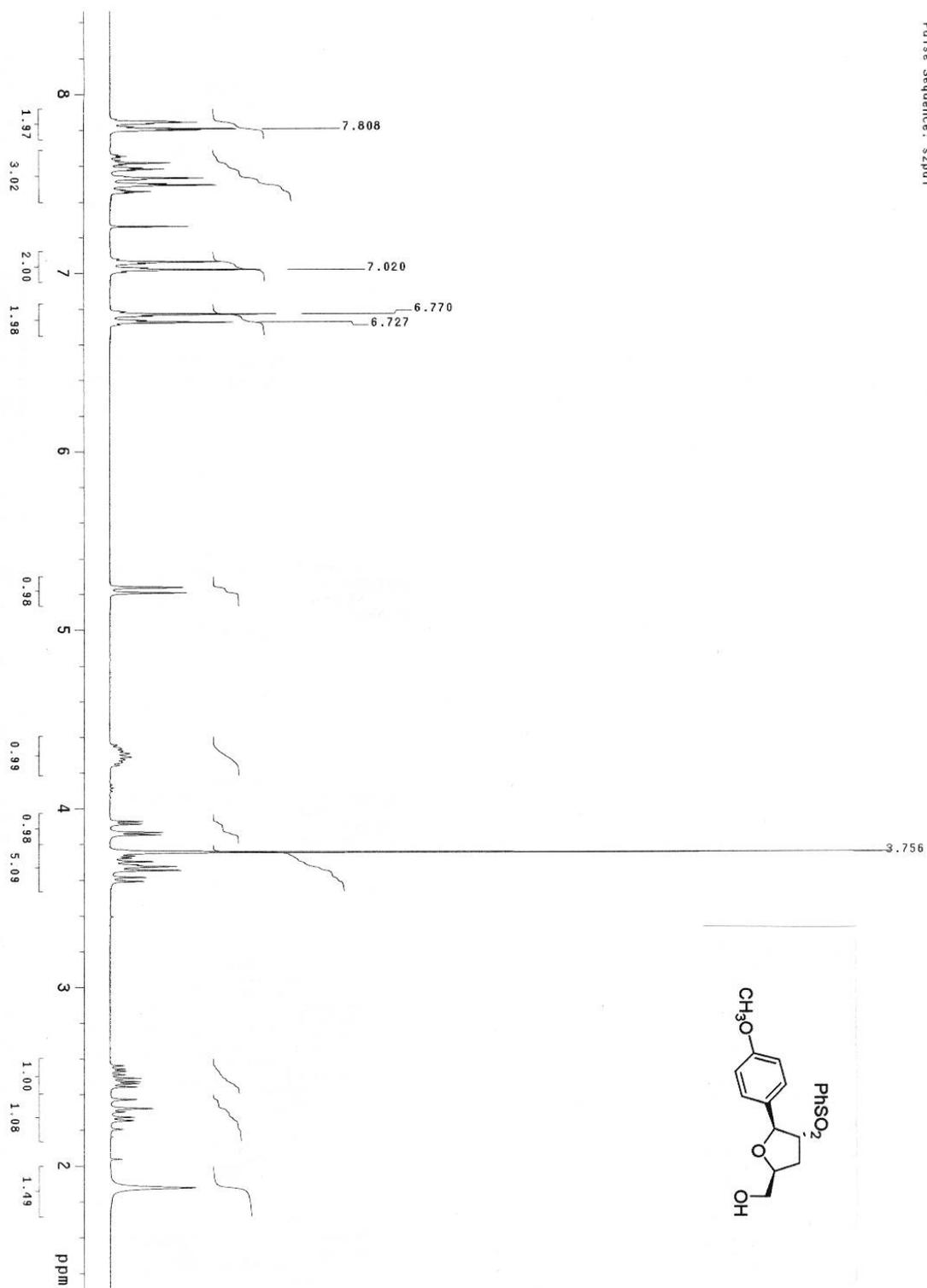
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 SOLVENT CDCl3
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 FIDRES 0.198682 Hz
 AQ 2.5166323 sec
 RG 57
 DM 76.800 usec
 DE 6.78 usec
 TE 303.0 K
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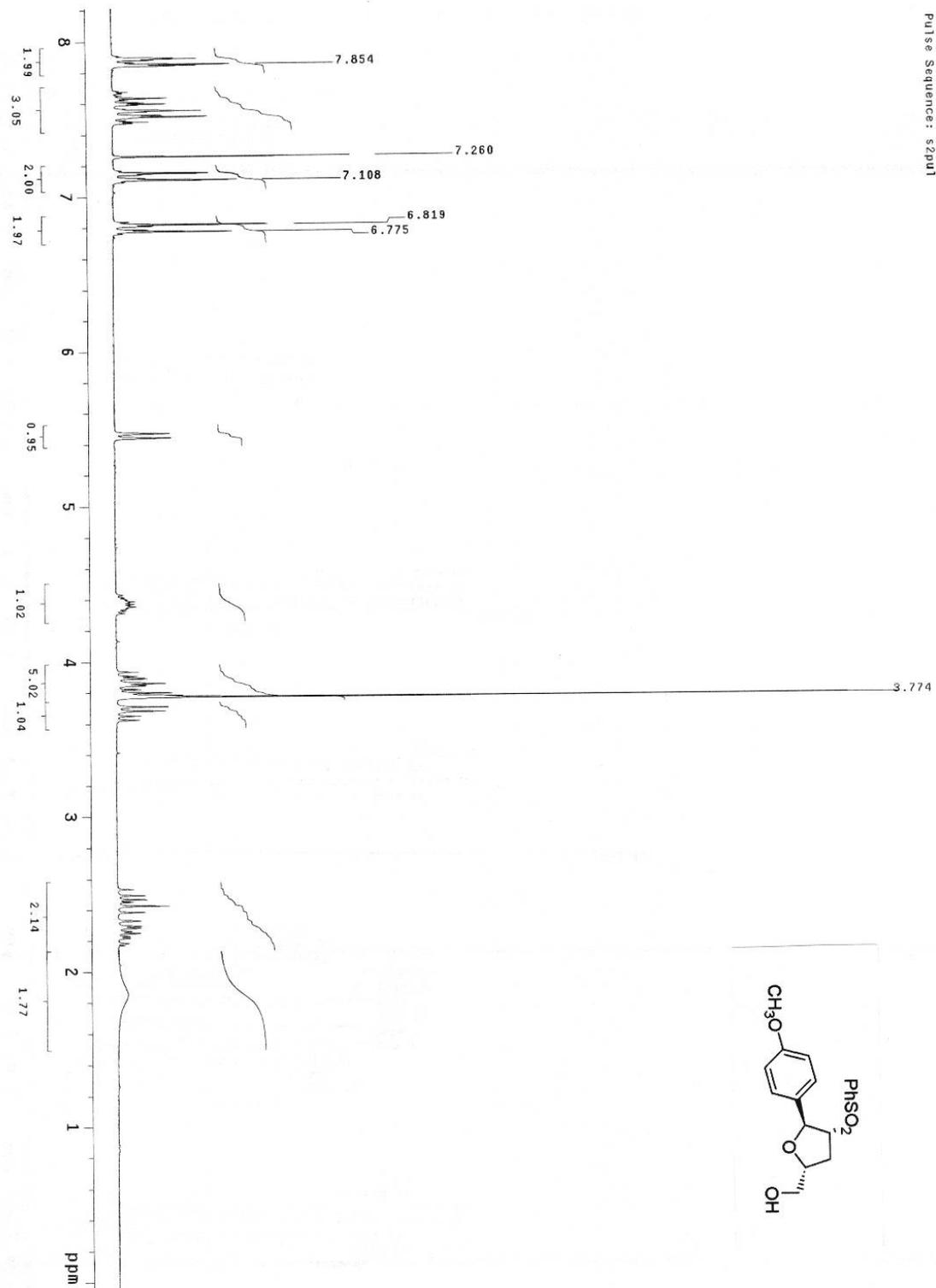
F2 - Processing parameters
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 SSB 0
 LB 0.00 Hz
 GB 0
 PC 1.00

1D NMR plot parameters
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 F1 4063.84 Hz
 F2P -75.10 Hz
 F2 0.37617 ppm/cm
 PPMVM 188.13394 Hz/cm
 HZCM

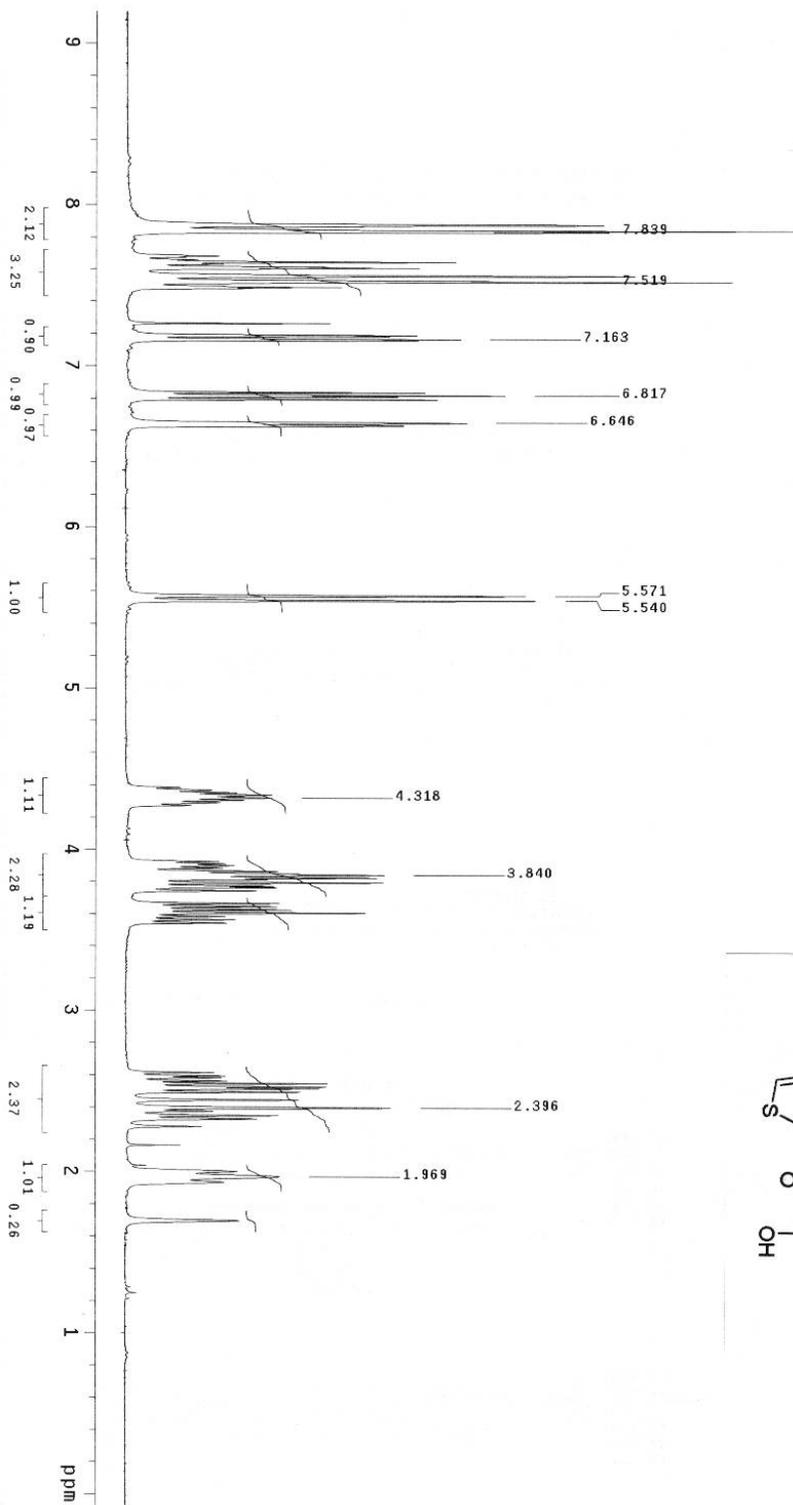
DK-90ah
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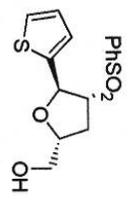
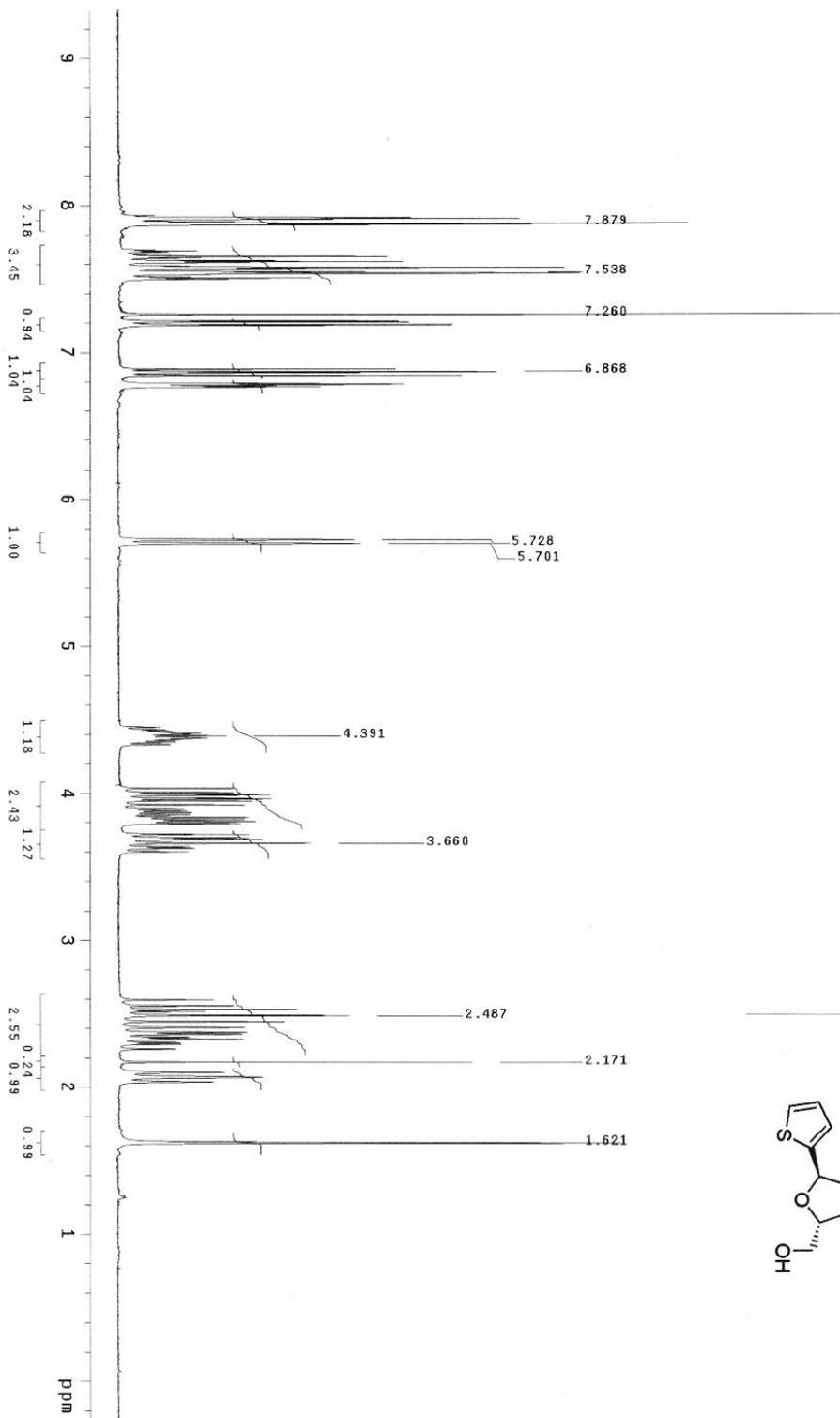
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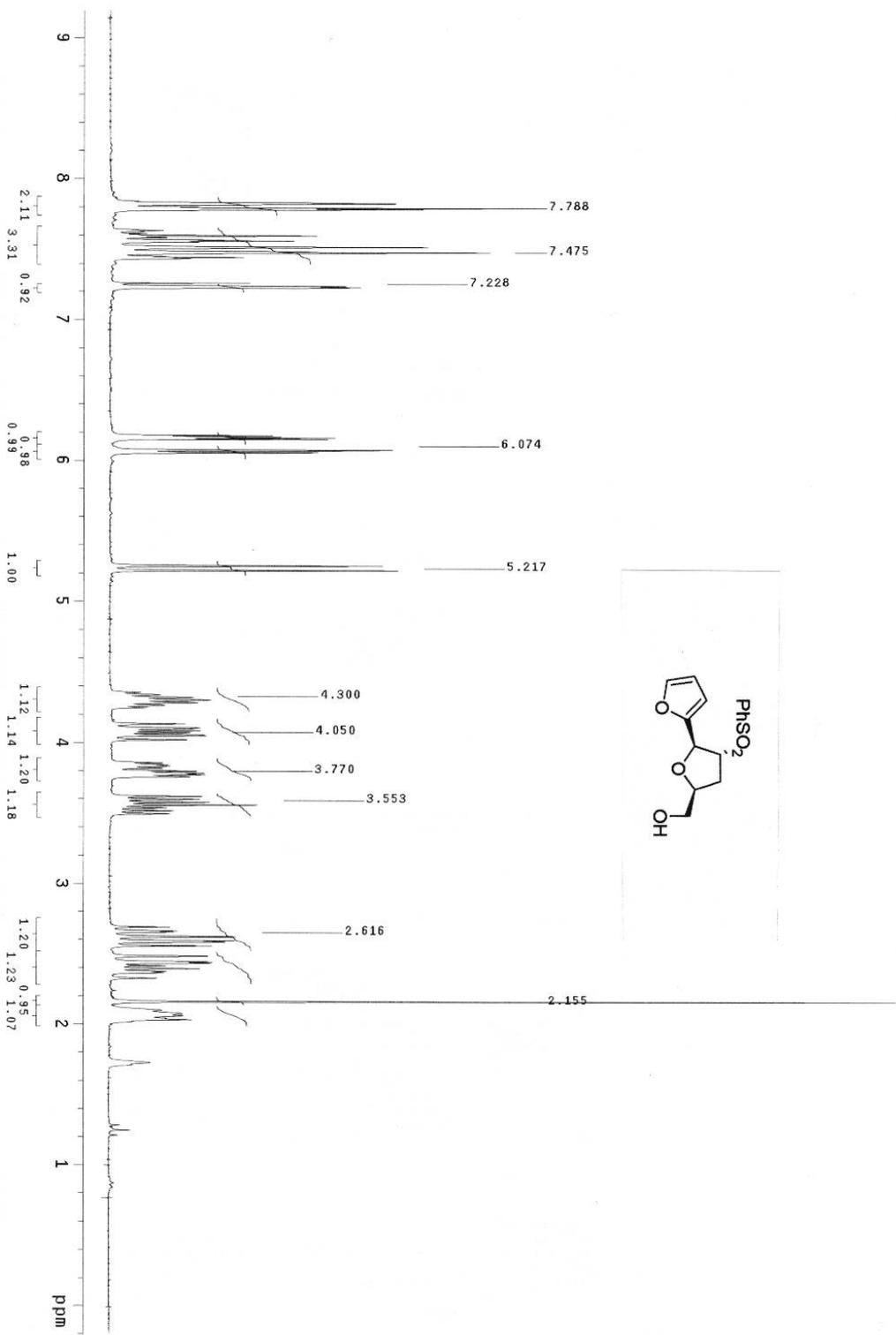
DK-110AH
Pulse Sequence: szpu1



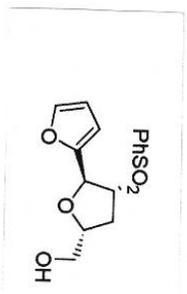
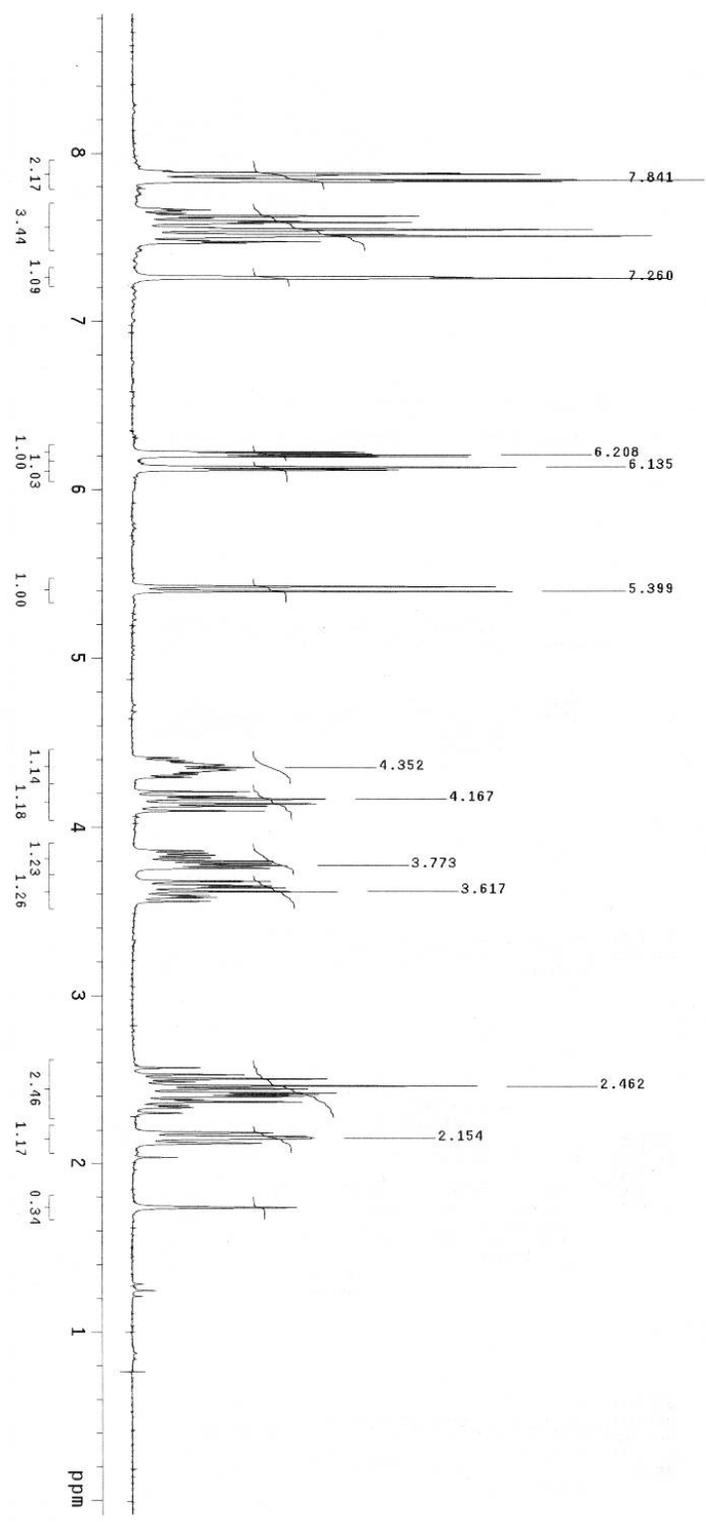
DK-110BH
Pulse Sequence: s2pu1



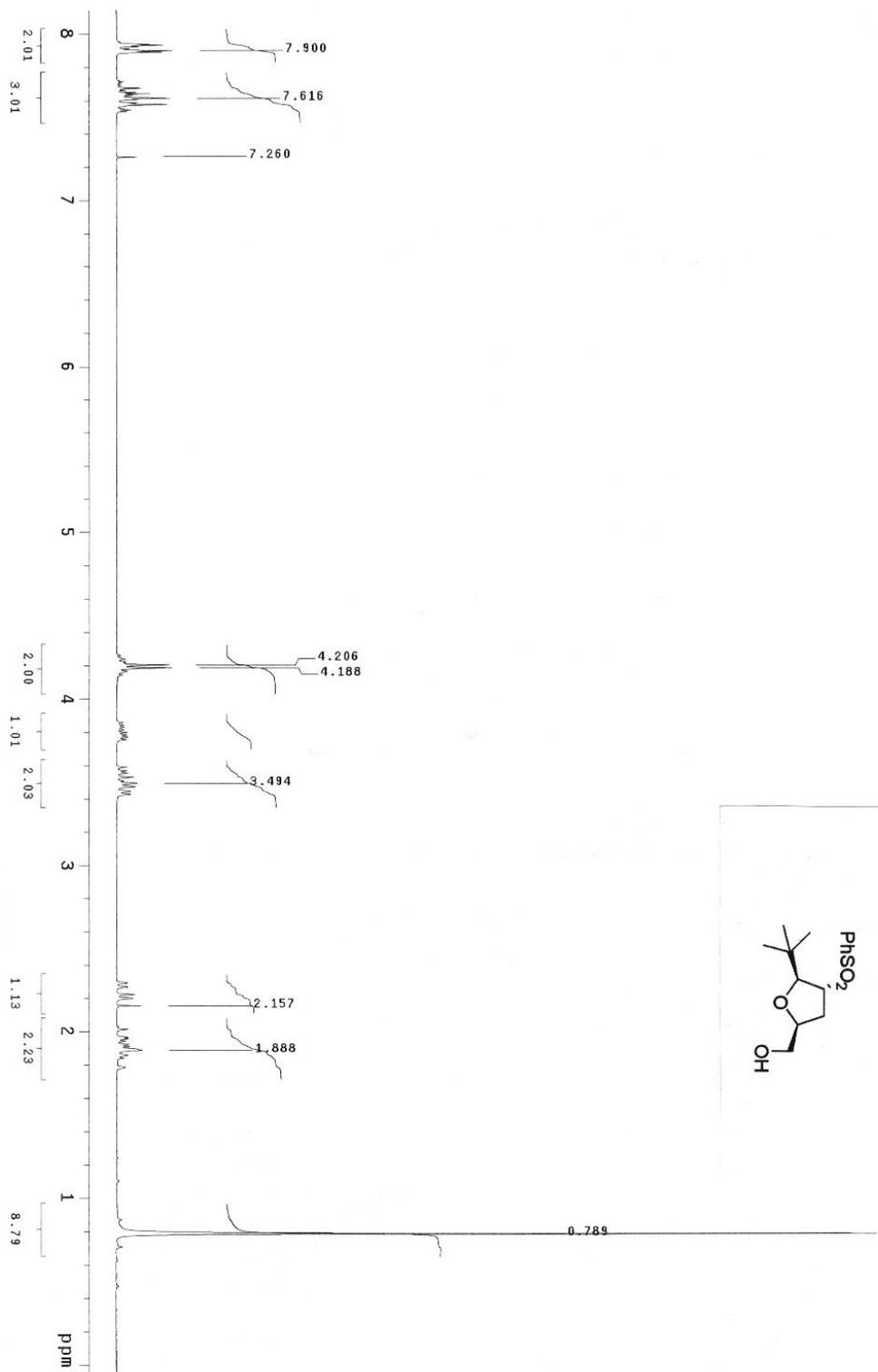
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Pulse Sequence: s2pu1



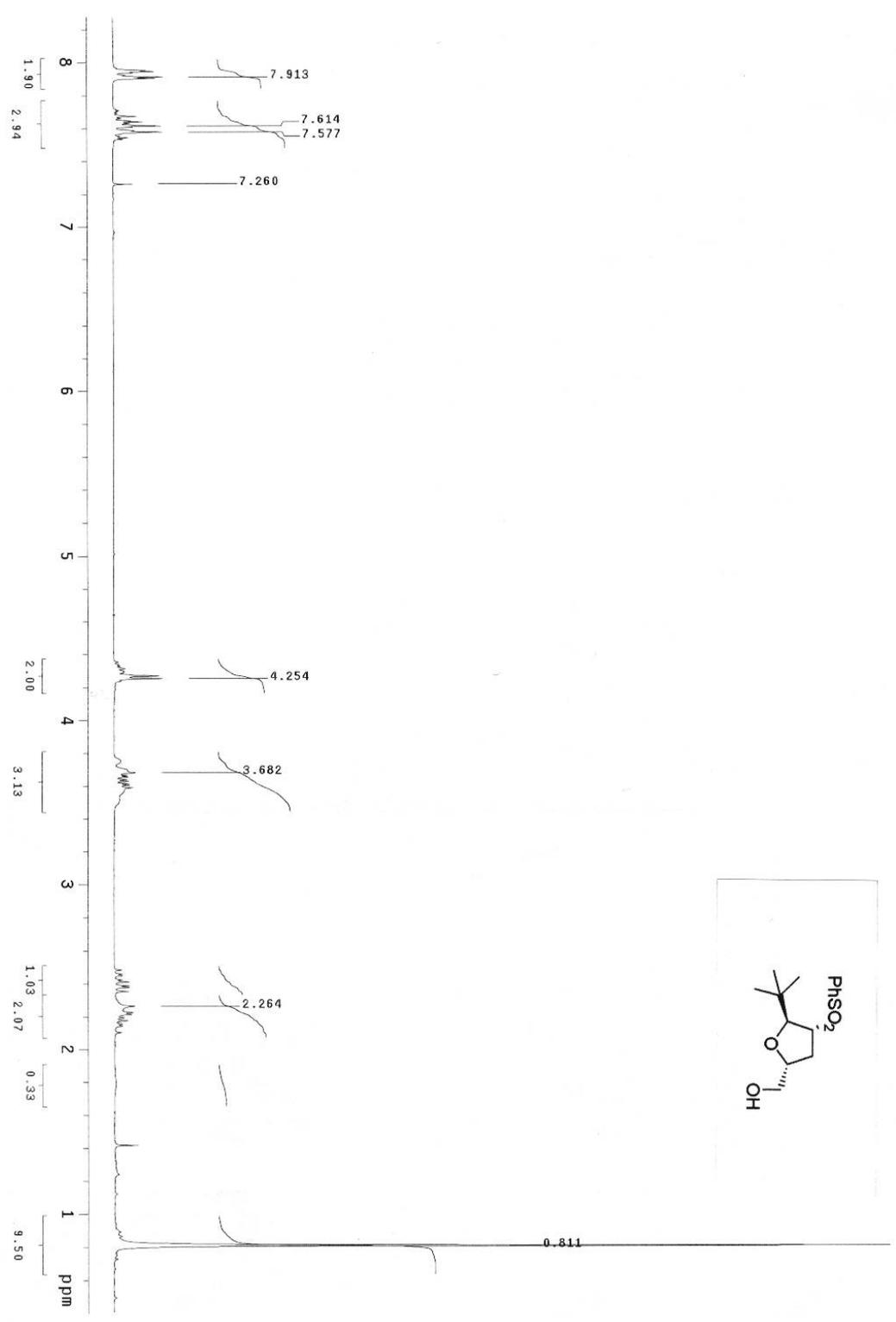
DK-114bH
Pulse Sequence: szpu1



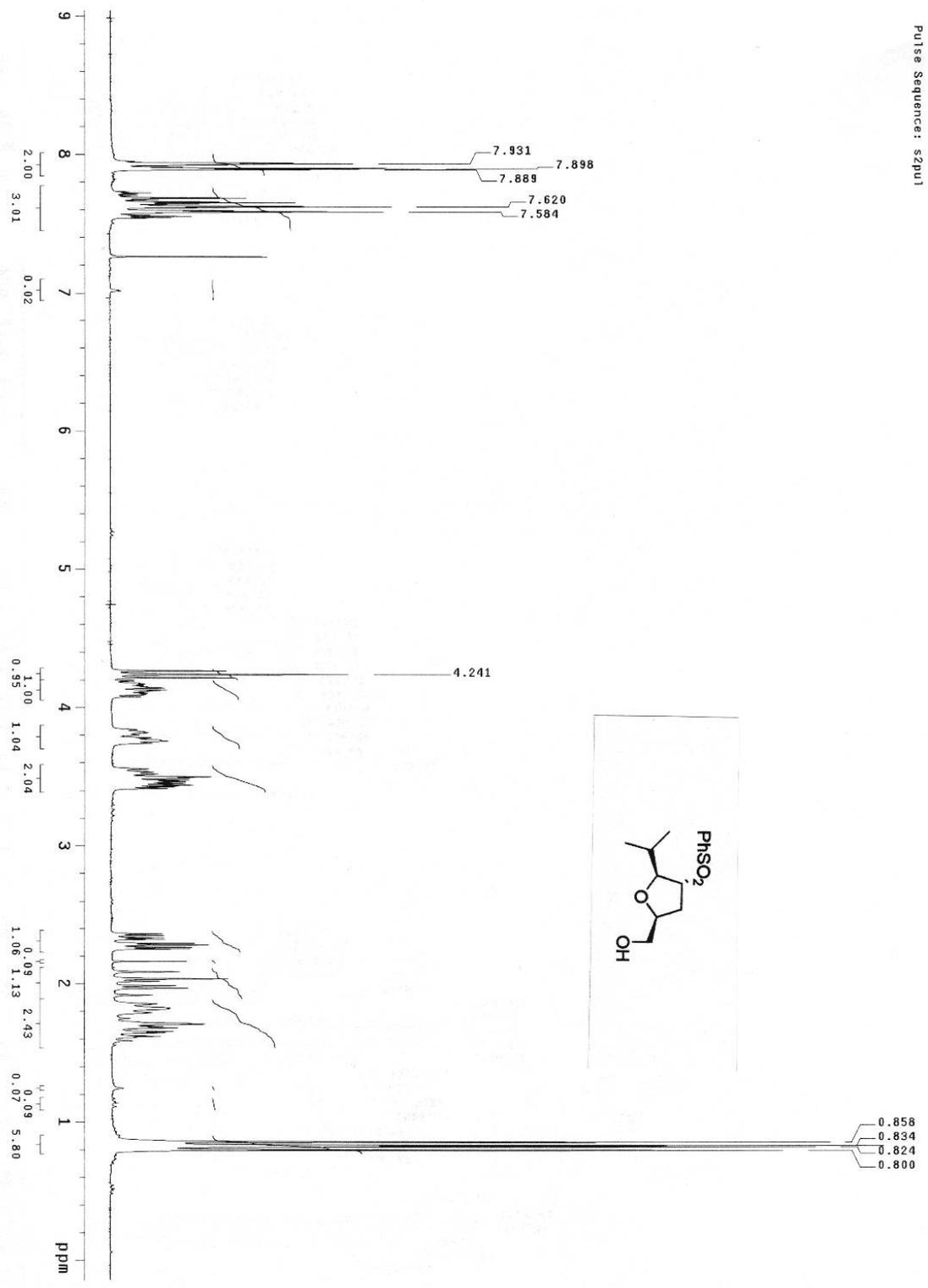
DK-9a4H
Pulse Sequence: s2pu1



DK-98BH
Pulse Sequence: s2pu1



NB-558AH
Pulse Sequence: szpu1



MB-5580H
Pulse Sequence: s2pu1

