Synthesis of L-Rhamnose derived chiral bicyclic triazoles as novel sodium-glucose transporter (SGLT) inhibitors

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Supporting Information

Contents

Single crystal X-ray analysis: X-ray data for the compounds **13a** and **13b** were collected at room temperature using a Bruker Smart Apex CCD diffractometer with graphite monochromated MoKα radiation (λ=0.71073 Å) with ω-scan method [1]. Preliminary lattice parameters and orientation matrices were obtained from four sets of frames.

Integration and scaling of intensity data were accomplished using SAINT program [1]. The structure was solved by direct methods using SHELXS97 [2] and refinement was carried out by full-matrix least-squares technique using SHELXL97 [2]. Anisotropic displacement parameters were included for all non-hydrogen atoms. All the O-bound H atoms were located in difference Fourier maps, and their positions and isotropic displacement parameters were refined. All other H atoms were located in difference Fourier maps and subsequently geometrically optimized and allowed for as riding atoms, with C-H = 0.93- 0.97 Å, with $U_{iso}(H) = 1.5U_{eq}(C)$ for methyl H or $1.2U_{eq}(C)$. The methyl groups were allowed to rotate but not to tip. The absolute configuration of the procured material was known in advance and in the absence of significant anomalous scattering efforts, Friedel pairs were merged.

Crystal data for compounds **13a** and **13b** can be obtained free of charge at www.ccdc.cam.ac.uk/conts/retrieving.html [or from the Cambridge Crystallographic Data Centre (CCDC), 12 Union Road, Cambridge CB2 1EZ, UK; fax: +44(0) 1223 336 033; email: deposit@ccdc.cam.ac.uk].

1. Bruker (2001). SAINT (Version 6.28a) & SMART (Version 5.625). Bruker AXS Inc., Madison, Wisconsin, USA.
2. Sheldrick GM. (2008**)** Acta Crystallogr A64: 112-122.

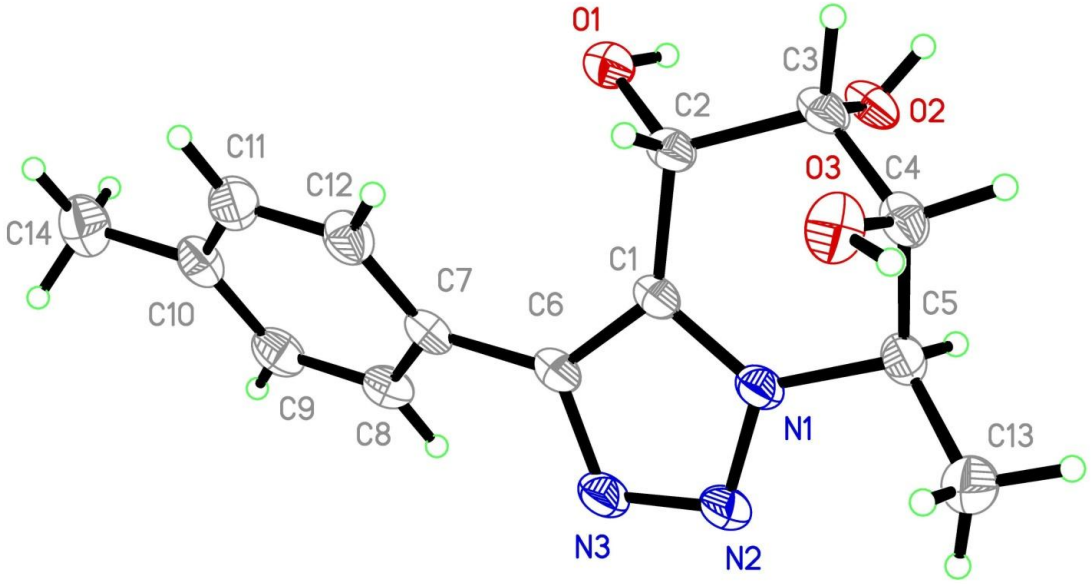


Figure S1: A view of **13b**, showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level and H atoms are represented by circles of arbitrary radii.

Experimental data:

(*S*)-1-((4*S*,5*S*)-5-((*R*)-1-Azidoethyl)-2,2-dimethyl-1,3-dioxolan-4-yl)-3-(4-methoxyphenyl) prop-2-yn-1-ol (12c). Yield: 85%; $[\alpha]_D^{27}$ -77.0 (c 1.0, $CHCl_3$). ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.32(d, J=8.8 Hz, 2H), 6.84(d, J=8.2 Hz, 2H), 4.79(d, J=3.6 Hz, 1H), 4.26(dd, J=3.6, 7.7 Hz, 1H), 4.17(dd, J=2.7, 7.7 Hz, 1H), 3.81 (s, 3H), 3.58(dq, J=2.5, 6.8 Hz, 1H), 1.52(s, 3H), 1.49(s, 3H), 1.46(d, J=6.8 Hz, 3H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 159.9, 133.1, 113.9, 113.8, 110.2, 87.1, 84.0, 80.0, 79.3, 62.3, 56.7, 55.2, 27.1, 27.0, 16.1. IR (neat) 3429, 2986, 2927, 2112, 1378, 1245, 1067, 767, 694 cm^{-1}. MS (ESI) *m/z* 332$[M+H]^+$, HRMS (ESI) Calcd for $C_{17}H_{22}N_3O_4[M+H]^+$: 332.16048, found: 332.15916**.**

(*S*)-1-((4*S*,5*S*)-5-((*R*)-1-Azidoethyl)-2,2-dimethyl-1,3-dioxolan-4-yl)-3-(4-(tert-butyl) phenyl) prop-2-yn-1-ol (12d). Yield: 94%; $[\alpha]_D^{27}$ -157.1 (c 1.0, $CHCl_3$). ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.35(d, J=8.3 Hz, 4H), 4.81(d, J=3.7 Hz, 1H), 4.27(dd, J=3.7, 8.3 Hz, 1H), 4.17(dd, J=3.0, 7.5 Hz, 1H), 3.58(dq, J=2.2, 6.7 Hz, 1H), 1.52(s, 3H), 1.49(s, 3H), 1.46(d, J=7.5 Hz, 3H), 1.30 (s, 9H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 152.2, 131.4, 125.3, 118.7, 110.2, 87.3, 84.6, 79.9, 79.2, 62.2, 56.7, 34.7, 31.0, 27.1, 27.0, 16.1. IR (neat) 3424, 2927, 2855, 2111, 1458, 1377, 1246, 1168, 1066, 874 cm^{-1}. MS (ESI) *m/z* 358$[M+H]^+$.

(*S*)-1-((4*S*,5*S*)-5-((*R*)-1-Azidoethyl)-2,2-dimethyl-1,3-dioxolan-4-yl)-3-(4-pentylphenyl) prop-2-yn-1-ol (12e). Yield: 96%; $[\alpha]_D^{27}$ -39.4 (c 1.0, $CHCl_3$). ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.33(d, J=8.2 Hz, 2H), 7.13(d, J=8.3 Hz, 2H), 4.80(d, J=3.2 Hz, 1H), 4.26(dd, J=3.8,7.9 Hz, 1H), 4.17(dd, J=2.8, 7.9 Hz, 1H), 3.58 (dq, J=2.7, 6.8 Hz, 1H), 2.59(t, J=7.9 Hz, 2H), 1.62-1.56(m, 2H), 1.52(s, 3H), 1.49(s, 3H), 1.46(d, J=6.8 Hz, 3H), 1.35-1.25(m,4H), 0.89(t, J=7.0 Hz, 3H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 144.0, 131.5, 128.4, 118.8, 110.2, 87.2, 84.8, 80.0, 79.3, 62.3, 56.7, 35.7, 31.3, 30.7, 27.1, 26.9, 22.4, 16.0, 13.9. IR (neat) 3424, 2927, 2855, 2111, 1458, 1377, 1246, 1168, 1066, 874 cm^{-1}. MS (ESI) *m/z* 372$[M+H]^+$, HRMS (ESI) Calcd for $C_{21}H_{30}N_3O_3[M+H]^+$: 372.22817, found: 372.22660.

(*S*)-3-([1,1'-Biphenyl]-4-yl)-1-((4*S*,5*S*)-5-((*R*)-1-azidoethyl)-2,2-dimethyl-1,3-dioxolan-4-yl) prop-2-yn-1-ol (12f). Yield: 91%; $[\alpha]_D^{27}$ -40.4 (c 1.0, $CHCl_3$). ^1H NMR (300 MHz, $CDCl_3$) δ 7.60-7.54(m, 4H), 7.51-7.48(m, 2H), 7.47-7.43(m, 2H), 7.39-7.35(m, 1H), 4.83(d, J=3.7 Hz, 1H), 4.29(dd, J=3.7, 8.3 Hz, 1H), 4.19(dd, J=3.0, 7.5 Hz, 1H), 3.59(dq, J=2.2, 6.7 Hz, 1H), 1.53(s, 3H), 1.50(s, 3H), 1.48(d, J=7.5 Hz, 3H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 141.6, 140.0, 132.1, 128.8, 127.7, 126.9, 120.5, 110.3, 86.9, 86.0, 80.0, 79.2, 62.4, 56.7, 27.1, 27.0, 16.1. IR (neat) 3429, 2986, 2927, 2112, 1378, 1245, 1067, 767, 694 cm^{-1}. MS (ESI) *m/z* 378$[M+H]^+$, HRMS (ESI) Calcd for $C_{22}H_{24}N_3O_3[M+H]^+$: 378.18122, found: 378.18071.

(*S*)-1-((4*S*,5*S*)-5-((*R*)-1-Azidoethyl)-2,2-dimethyl-1,3-dioxolan-4-yl)-3-(4-(trifluoro methoxy)phenyl) prop-2-yn-1-ol (12g). Yield: 82%; $[\alpha]_D^{27}$ -1.3 (c 1.0, $CHCl_3$). ^1H NMR (300 MHz, $CDCl_3$) δ 7.46(d, J=8.8 Hz, 2H), 7.18(d, J=8.8 Hz, 2H), 4.79(d, J=3.9 Hz, 1H), 4.26(dd, J=3.9, 7.9 Hz, 1H), 4.15(dd, J=2.8, 7.7 Hz, 1H), 3.55(dq, J=2.8, 6.8 Hz, 1H), 1.52(s, 3H), 1.48(s, 3H), 1.46(d, J=6.8 Hz, 3H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 149.2, 133.6, 121.3, 120.7, 119.3, 110.1, 84.5, 82.1, 80.9, 68.5, 68.0, 26.3, 25.0, 19.5. IR (neat) 3448, 2928, 2853, 2113, 1637, 1383, 1258, 1167, 569 cm^{-1}. MS (ESI) *m/z* 386$[M+H]^+$, HRMS (ESI) Calcd for $C_{17}H_{19}N_3O_4F_3[M+H]^+$: 386.13222, found: 386.13161.

(*S*)-1-((4*S*,5*S*)-5-((*R*)-1-Azidoethyl)-2,2-dimethyl-1,3-dioxolan-4-yl)-3-(4-(trifluoro methyl)phenyl) prop-2-yn-1-ol (12h). Yield: 87%; $[\alpha]_D^{27}$ -76.0 (c 1.0, $CHCl_3$). ^1H NMR (300 MHz, $CDCl_3$) δ 7.59(d, J=8.2 Hz, 2H), 7.54(d, J=8.0 Hz, 2H), 4.80(d, J=3.8 Hz, 1H), 4.27(dd, J=3.8, 7.7 Hz, 1H), 4.15(dd, J=2.8, 7.7 Hz, 1H), 3.54(dq, J=2.7, 6.8 Hz, 1H), 1.52(s, 3H), 1.48(s, 3H), 1.46(d, J=6.8 Hz, 3H). ^{13}C NMR (125MHz, $CDCl_3$) δ 131.9, 125.3, 110.4, 87.9, 85.6, 801, 79.1, 62.5, 56.7, 27.1, 27.0, 16.0. IR (neat) 3422, 2988, 2923, 2853, 2112, 1619, 1325, 1167, 1128, 1066, 847, 807 cm^{-1}. MS (ESI) *m/z* 370$[M+H]^+$, HRMS (ESI) Calcd for $C_{17}H_{19}N_3O_3F_3[M+H]^+$: 370.13730, found: 370.13578.

(*S*)-1-((4*S*,5*S*)-5-((*R*)-1-Azidoethyl)-2,2-dimethyl-1,3-dioxolan-4-yl)-3-(2-(trifluoro methyl) phenyl) prop-2-yn-1-ol (12i). Yield: 85%; $[\alpha]_D^{27}$ -57.1 (c 1.0, $CHCl_3$). ^1H NMR (300 MHz, $CDCl_3$) δ 7.67(d, J=7.7 Hz, 1H), 7.60(d, J=7.4 Hz, 1H), 7.51(t, J=7.4 Hz, 1H), 7.45(t, J=7.6 Hz, 1H), 4.84(d, J=3.8 Hz, 1H), 4.28(dd, J=3.9, 7.9 Hz, 1H), 4.17(dd, J=2.7, 7.7 Hz, 1H), 3.54(dq, J=2.5, 6.8 Hz, 1H), 1.52(s, 3H), 1.47(s, 3H), 1.45(d, J=6.8 Hz, 3H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 134.6, 131.3, 128.5, 125.7, 110.1, 84.5, 82.8, 80.8, 79.3, 68.5, 67.9, 26.3, 25.0, 19.4. IR (neat) 3422, 2988, 2923, 2853, 2112, 1619, 1325, 1167, 1128, 1066, 847, 807 cm^{-1}. MS (ESI) *m/z* 370$[M+H]^+$, HRMS (ESI) Calcd for $C_{17}H_{19}N_3O_3F_3[M+H]^+$: 370.13730, found: 370.13578.

(*S*)-1-((4*S*,5*S*)-5-((*R*)-1-Azidoethyl)-2,2-dimethyl-1,3-dioxolan-4-yl)-3-(3,4-dichloro phenyl) prop-2-yn-1-ol (12j). Yield: 80%; $[\alpha]_D^{27}$ -9.6 (c 1.0, $CHCl_3$). ^1H NMR (300 MHz, $CDCl_3$) δ 7.51(d, J=1.5 Hz, 1H), 7.40(d, J=8.2 Hz, 1H), 7.25(d, J=9.1 Hz, 1H), 4.76(d, J=3.9 Hz, 1H), 4.24(dd, J=4.1, 7.7 Hz, 1H), 4.12(dd, J=2.7, 7.7 Hz, 1H), 3.52(dq, J=2.7, 6.8 Hz, 1H), 1.52(s, 3H), 1.47(s, 3H), 1.46(d, J=6.8 Hz, 3H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 133.7, 133.3, 132.5, 131.1, 130.3, 110.1, 84.4, 80.8, 79.2, 68.5, 67.9, 26.3, 25.0, 19.4. IR (neat) 3445, 2986, 2922, 2852, 2112, 1466, 1378, 1225, 1132, 1086, 1028, 825, 771 cm^{-1}. MS (ESI) *m/z* 370$[M+H]^+$, HRMS (ESI) Calcd for $C_{16}H_{18}N_3O_3Cl_2[M+H]^+$: 370.07197, found: 370.07115.

(*S*)-1-((4*S*,5*S*)-5-((*R*)-1-Azidoethyl)-2,2-dimethyl-1,3-dioxolan-4-yl)-3-(3,4-difluoro phenyl) prop-2-yn-1-ol (12k). Yield: 83%; $[\alpha]_D^{27}$ -53.7 (c 1.0, $CHCl_3$). ^1H NMR (300 MHz, $CDCl_3$) δ 7.29-7.07(m, 3H), 4.76(d, J=4.5 Hz, 1H), 4.25(dd, J=3.7, 7.5 Hz, 1H), 4.13(dd, J=3.0, 7.5 Hz, 1H), 3.53(dq, J=2.2, 6.7 Hz, 1H), 1.52(s, 3H), 1.48(s, 3H), 1.46(d, J=6.7 Hz, 3H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 128.3, 120.8, 120.5, 117.7, 117.5, 110.4, 86.1, 84.9, 80.1, 79.1, 62.5, 56.6, 27.1, 27.0, 16.0. IR (neat) 3424, 2988, 2924, 2854, 2112, 1619, 1502, 1379, 1226, 1141, 1092, 964, 853, 809 cm^{-1}. MS (ESI) *m/z* 338$[M+H]^+$, HRMS (ESI) Calcd for

$C_{16}H_{18}N_3O_3F_2[M+H]^+$: 338.13107, found: 338.12998.

(*S*)-1-((4*S*,5*S*)-5-((*R*)-1-Azidoethyl)-2,2-dimethyl-1,3-dioxolan-4-yl)-3-(2,4-difluoro phenyl) prop-2-yn-1-ol (12l). Yield: 85%; $[\alpha]_D^{27}$ -138.4 (c 1.0, $CHCl_3$). ^1H NMR (300 MHz, $CDCl_3$) δ 7.44-7.39(m, 1H), 6.89-6.82(m, 2H), 4.83 (d, J=3.6 Hz, 1H), 4.27(dd, J=3.8, 7.9 Hz, 1H), 4.17(dd, J=2.7, 7.7 Hz, 1H), 3.61(dq, J=2.5, 6.8 Hz, 1H), 1.51(s, 3H), 1.48(s, 3H), 1.47(d, J=6.7 Hz, 3H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 128.4, 120.8, 120.5, 117.7, 117.4, 110.4, 86.4, 84.9, 80.1, 79.0, 62.4, 56.6, 27.1, 26.9, 16.0. IR (neat) 3424, 2988, 2924, 2854, 2112, 1619, 1502, 1379, 1226, 1141, 1092, 964, 853, 809 cm^{-1}. MS (ESI) *m/z* 337$[M+H]^+$, HRMS (ESI) Calcd for $C_{16}H_{18}N_3O_3F_2[M+H]^+$: 338.13107, found: 338.12998.

(*S*)-1-((4*S*,5*S*)-5-((*R*)-1-Azidoethyl)-2,2-dimethyl-1,3-dioxolan-4-yl)-3-(2,4,5-trimethyl phenyl)prop-2-yn-1-ol (12m). Yield: 90%; $[\alpha]_D^{27}$ -53.0 (c 1.0, $CHCl_3$). ^1H NMR (300 MHz, $CDCl_3$) δ 7.15(s, 1H), 6.96(s, 2H), 4.84(d, J=3.7 Hz, 1H), 4.27(dd, J=3.0, 7.5 Hz, 1H), 4.17(dd, J=2.2, 7.5 Hz, 1H), 3.58(dq, J=2.2, 6.7 Hz, 1H), 2.34(s, 3H), 2.22 (s, 3H), 2.18(s, 3H), 1.51(s, 3H), 1.47(s, 3H), 1.45(d, J=6.7 Hz, 3H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 137.7, 137.5, 133.7, 133.0, 130.8, 118.6, 110.2, 88.0, 86.4, 80.0, 79.3, 62.4, 56.9, 27.2, 27.0, 19.9, 19.6, 19.0, 16.1. IR (neat) 3539, 3282, 2926, 2852, 2696, 1463, 1098, 1061, 994 cm^{-1}. MS (ESI) *m/z* 344$[M+H]^+$**,** HRMS (ESI) Calcd for $C_{19}H_{26}N_3O_3[M+H]^+$: 344.19687, found: 344.19536**.**

(*S*)-1-((4*S*,5*S*)-5-((*R*)-1-Azidoethyl)-2,2-dimethyl-1,3-dioxolan-4-yl)-3-(phenanthren-9-yl) prop-2-yn-1-ol (12n). Yield: 94%; $[\alpha]_D^{27}$ -210.3 (c 1.0, $CHCl_3$). ^1H NMR (300 MHz, $CDCl_3$) δ 8.68(brd, J=7.4 Hz, 1H), 8.65(d, J=8.3 Hz, 1H), 8.38(dd, J=1.9, 6.7 Hz, 1H), 7.98(s, 1H), 7.84(d, J=7.9 Hz, 1H), 7.72-7.64 (m, 3H), 7.60(dt, J=1.0, 7.9 Hz, 1H), 4.97(d, J=3.9 Hz, 1H), 4.39(dd, J=3.9, 7.7 Hz, 1H), 4.26(dd, J=2.7, 7.7 Hz, 1H), 3.65(dq, J=2.7, 6.8 Hz, 1H), 1,55(s, 3H), 1.52(s, 3H), 1.46(d, J=6.8 Hz, 3H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 132.8, 131.0, 130.9, 130.4, 129.9, 128.5, 127.6, 127.0, 126.9, 126.7, 122.5, 110.1, 84.5, 81.5, 80.8, 68.5, 67.9,

26.3, 25.0, 19.4. IR (neat) 3445, 2987, 2934, 2111, 1454, 1383, 1219, 1065, 768, 725 cm^{-1}. MS (ESI) *m/z* 402$[M+H]^+$, HRMS (ESI) Calcd for $C_{24}H_{24}N_3O_3[M+H]^+$: 402.18122, found: 402.17978**.**

(*S*)-1-((4*S*,5*S*)-5-((*R*)-1-Azidoethyl)-2,2-dimethyl-1,3-dioxolan-4-yl)-3-(thiophen-3-yl) prop-2-yn-1-ol (12o). Yield: 81%; $[\alpha]_D^{27}$ -51.2 (c 1.0, $CHCl_3$). ^1H NMR (300 MHz, $CDCl_3$) δ 7.47(dd, J=1.5, 3.0 Hz, 1H), 7.28(dd, J=3.0, 5.2 Hz, 1H), 7.10(dd, J=1.5, 5.2 Hz, 1H), 4.78(d, J=3.7 Hz, 1H), 4.25(dd, J=3.7, 7.5 Hz, 1H), 4.15(dd, J=3.0, 7.5 Hz, 1H), 3.56(dq, J=3.0, 6.7 Hz, 1H), 1,52(s, 3H), 1.48(s, 3H), 1.46(d, J=6.7 Hz, 3H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 129.9, 125.2, 121.0, 110.0, 84.4, 80.7, 78.7, 76.9, 68.4, 67.9, 26.3, 25.0, 19.4. IR (neat) 3424, 2988, 2924, 2854, 2112, 1619, 1502, 1379, 1226, 1141, 1092, 964, 853, 809 cm^{-1}. MS (ESI) *m/z* 308$[M+H]^+$**,** HRMS (ESI) Calcd for $C_{14}H_{18}N_3O_3S[M+H]^+$: 308.10634, found: 308.10529**.**

(*S*)-1-((4*S*,5*S*)-5-((*R*)-1-Azidoethyl)-2,2-dimethyl-1,3-dioxolan-4-yl)dec-2-yn-1-ol (12p). Yield: 92%; $[\alpha]_D^{27}$ -53.0 (c 1.0, $CHCl_3$). ^1H NMR (300 MHz, $CDCl_3$) δ 4.58(qt, J=1.6 Hz, 1H), 4.14(dd, J=3.6, 7.7 Hz, 1H), 4.08(dd, J=2.5, 7.7 Hz, 1H), 3.52(dq, J=2.5, 6.8 Hz, 1H), 2.22(dt, J=1.9, 7.1 Hz, 2H), 1.52-1.47(m, 5H), 1.4(d, J=6.7 Hz, 3H), 1.44(s, 3H), 1.39-1.23(m, 6H), 0.89(t, J=7.3 Hz, 3H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 110.0, 88.3, 79.9, 79.3, 61.8, 56.8, 31.2, 28.5, 28.3, 27.1, 27.0, 22.5, 18.6, 16.1, 14.0. IR (neat) 3438, 2932, 2859, 2112, 1456, 1380, 1246, 1099, 879 cm^{-1}. MS (ESI) *m/z* 310$[M+H]^+$, HRMS (ESI) Calcd for $C_{16}H_{28}N_3O_3[M+H]^+$: 310.21252, found: 310.21097**.**

(*S*)-1-((4*S*,5*S*)-5-((*R*)-1-Azidoethyl)-2,2-dimethyl-1,3-dioxolan-4-yl)pentadec-2-yn-1-ol (12q).

Yield: 93%; $[\alpha]_D^{27}$ -77.2 (c 1.0, $CHCl_3$). ^1H NMR (300 MHz, $CDCl_3$) δ 4.58(m, 1H), 4.14(dd, J=3.6, 7.7 Hz, 1H), 4.08(dd, J=2.5, 7.7 Hz, 1H), 3.52(dq, J=2.5, 6.8 Hz, 1H), 2.21(dt, J=1.9, 7.1 Hz, 2H), 1.54-1.42(m, 10H), 1.39-1.20(m, 18H), 0.93-0.83(m, 5H). ^{13}C NMR (75 MHz,

$CDCl_3$) δ 110.0, 88.3, 79.8, 79.2, 61.8, 56.7, 31.8, 29.6, 29.4, 29.3, 29.1, 28.9, 28.3, 27.1, 26.9, 25.7, 22.6, 18.6, 16.1, 14.1. IR (neat) 3425, 2925, 2854, 2111, 1641, 1376, 1245, 1067, 1028, 878 cm^{-1}. MS (ESI) *m/z* 394$[M+H]^+$, HRMS (ESI) Calcd for $C_{22}H_{40}N_3O_3[M+H]^+$: 394.30642, found: 394.30482**.**

(4*S*,5*R*,6*S*,7*R*)-3-(4-Methoxyphenyl)-7-methyl-4,5,6,7-tetra hydro-[1,2,3]triazolo[1,5-a] pyridine-4,5,6-triol (13c). Yield: 75%; MP= 224°C; $[\alpha]_D^{27}$ -103.6 (c 1.0, CH_3OH). ^1H NMR (300 MHz, DMSO+$CDCl_3$) δ 7.83(d, J=8.6 Hz, 2H), 6.89(d, J=8.6 Hz, 2H), 5.44(d, J=4.5 Hz, 1H), 5.23(d, J=3.3 Hz, 1H), 5.10-4.98(m, 2H), 4.68-4.10(m, 1H), 4.12-3.98(m, 2H), 3.76(s, 3H), 1.59(d, J=6.7 Hz, 3H). ^{13}C NMR (75 MHz, DMSO+$CDCl_3$) δ 158.2, 143.5, 130.1, 128.1, 123.9, 113.1, 69.1, 68.1, 61.9, 54.6, 52.7, 14.9 . IR (neat) 3483, 3405, 3097, 2929, 2842, 1618, 1507, 1252, 1103, 1047, 991, 822 cm^{-1}. MS (ESI) *m/z* 292$[M+H]^+$; HR-MS (ESI) Calcd for $C_{14}H_{18}N_3O_4M+H]^+$: 292.1297, found: 292.1306.

(4*S*,5*R*,6*S*,7*R*)-3-(4-(tert-Butyl)phenyl)-7-methyl-4,5,6,7-tetra hydro-[1,2,3]triazolo[1,5-a]pyridine-4,5,6-triol (13d). Yield: 91%; MP= 185°C; $[\alpha]_D^{27}$ 14.0 (c 1.0, CH_3OH). ^1H NMR (300 MHz, DMSO+$CDCl_3$) δ 7.77(d, J=8.4 Hz, 2H), 7.34(d, J=8.4 Hz, 2H), 5.65(d, J=4.5 Hz, 1H), 5.34(d, J=4.1 Hz, 1H), 5.10-5.03(m, 2H), 4.66(dd, J=3.3, 6.7 Hz, 1H), 4.15-4.00(m, 2H), 1.58(d, J=6.7 Hz, 3H), 1.26(s, 3H). ^{13}C NMR (75 MHz, DMSO+$CDCl_3$) δ 149.8, 144.1, 131.1, 128.6, 127.0, 124.9, 69.3, 68.4, 62.2, 53.3, 34.3, 31.2, 15.3. IR (neat) 3483, 3405, 3097, 2929, 2842, 1618, 1507, 1252, 1103, 1047, 991, 822 cm^{-1}. MS (ESI) *m/z* 318$[M+H]^+$; HR-MS (ESI) Calcd for $C_{17}H_{24}N_3O_3[M+H]^+$: 318.1817, found: 318.1830.

(4*S*,5*R*,6*S*,7*R*)-7-Methyl-3-(4-pentylphenyl)-4,5,6,7-tetrahydro-[1,2,3]triazolo[1,5-a] pyridine-4,5,6-triol (13e). Yield: 93%; MP= 176°C; $[\alpha]_D^{27}$ -25.4 (c 1.0, CH_3OH). ^1H NMR (300 MHz, DMSO+$CDCl_3$) δ 7.80(d, J=8.1 Hz, 2H), 7.17(d, J=8.1 Hz, 2H), 5.49(d, J=4.7 Hz, 1H), 5.31(d, J=4.3 Hz, 1H), 5.16(d, J=8.4 Hz, 1H), 5.04(dd, J=3.9, 4.7 Hz, 1H), 4.12-3.97(m, 2H), , 1.64-1.53(m, 5H), 1.36-1.24(m, 4H), 0.86(t, J=6.7 Hz, 3H). ^{13}C NMR (75 MHz,

DMSO+$CDCl_3$) δ 143.6, 141.0, 11130.0, 128.8, 127.6, 126.9, 69.2, 68.2, 61.9, 52.7, 34.8, 30.7, 30.4, 21.8, 14.9, 13.6. IR (neat) 3543, 3265, 3929, 2856, 2702, 1450, 1351, 1100, 1062, 995, 858 cm^{-1}. MS (ESI) *m/z* 332$[M+H]^+$; HR-MS (ESI) Calcd for $C_{18}H_{26}N_3O_3[M+H]^+$: 332.1974, found: 332.1990.

(4*S*,5*R*,6*S*,7*R*)-3-([1,1'-Biphenyl]-4-yl)-7-methyl-4,5,6,7-tetrahydro-[1,2,3]triazolo[1,5-a] pyridine-4,5,6-triol (13f). Yield: 85%; MP= 185°C; $[\alpha]_D^{27}$ -50.8 (c 1.0, CH_3OH). ^1H NMR (300 MHz, DMSO+$CDCl_3$) δ 8.02(d, J=8.4 Hz, 2H), 7.64(d, J=8.4 Hz, 4H), 7.43(t, J=7.1 Hz, 2H), 7.31(t, J=7.3 Hz, 1H), 5.52(d, J=4.5 Hz, 1H), 5.35(d, J=4.1 Hz,1H), 5.23(d, J=8.8 Hz, 1H), 5.11(dd, J=3.9, 4.9 Hz, 1H), 4.69(m, 1H), 4.14-4.03(m, 2H), 1.63(d, J=6.7 Hz, 3H). ^{13}C NMR (75 MHz, DMSO+$CDCl_3$) δ 143.3, 139.7, 138.7, 131.4, 130.6, 128.5, 127.5, 127.0, 126.2, 126.1, 69.4, 68.2, 62.0, 52.7, 14.9 . IR (neat) 3390, 3245, 2931, 1545, 1448, 1219, 1100, 1040, 996, 771, 730, 697 cm^{-1}. MS (ESI) *m/z* 338$[M+H]^+$; HR-MS (ESI) Calcd for $C_{19}H_{20}N_3O_3$ $[M+H]^+$: 338.1504, found: 338.1524.

(4*S*,5*R*,6*S*,7*R*)-7-Methyl-3-(4-(trifluoromethoxy)phenyl)-4,5,6,7-tetrahydro[1,2,3] triazole[1,5-a]pyridine-4,5,6-triol (13g). Yield: 81%; MP= 165°C; $[\alpha]_D^{27}$ -5.0 (c 1.0, CH_3OH). ^1H NMR (300 MHz, DMSO+$CDCl_3$) δ 8.06(d, J=7.1 Hz, 2H), 7.28(d, J=8.3 Hz, 2H), 5.50(d, J=4.5 Hz, 1H), 5.31(d, J=3.9 Hz, 1H), 5.19-5.09 (m, 2H), 4.75-4.68(m, 1H), 4.18-4.09(m, 2H), 1.68(d, J=6.7 Hz, 3H). ^{13}C NMR (75 MHz, DMSO+$CDCl_3$) δ 147.4, 142.6, 131.1, 130.2, 128.4, 119.9, 69.9, 68.1, 61.9, 52.7, 14.6. IR (neat) 3549, 3447, 3201, 2979, 1495, 1368, 1267, 1226, 1098, 1038, 994, 695, 648 cm^{-1}. MS (ESI) *m/z* 346$[M+H]^+$; HR-MS (ESI) Calcd for $C_{14}H_{15}N_3O_4F_3[M+H]^+$: 346.1014, found: 346.1026**.**

(4*S*,5*R*,6*S*,7*R*)-7-Methyl-3-(4-(trifluoromethyl)phenyl)-4,5,6,7-tetrahydro-[1,2,3]triazolo [1,5-a]pyridine-4,5,6-triol (13h). Yield: 89%; MP= 192°C; $[\alpha]_D^{27}$ -54.1 (c 1.0, CH_3OH). ^1H NMR (300 MHz, DMSO+$CDCl_3$) δ 8.12(d, J=8.3 Hz, 2H), 7.65(d, J=8.0 Hz, 2H), 5.54(d, J=2.5, 1H), 5.40(d, J=2.4 Hz, 1H), 5.27(dd, J=7.3, 1.6 Hz, 1H), 5.11(dd, J=5.4, 3.3 Hz, 1H),

4.69-4.64(m, 1H), 4.10-4.04(m, 2H), 1.63 (d, J=6.7 Hz, 3H). ^{13}C NMR (75 MHz, DMSO+$CDCl_3$) δ 142.3, 135.3, 132.5, 127.4, 124.4, 69.7, 68.8, 62.0, 52.6, 14.8. IR (neat) 3378, 2915, 1623, 1327, 1170, 1107, 1063, 1008, 853, 643 cm^{-1} MS (ESI) *m/z* 330$[M+H]^+$; HR-MS (ESI) Calcd for $C_{14}H_{15}N_3O_3F_3[M+H]^+$: 330.1065, found: 330.1075**.**

(4*S*,5*R*,6*S*,7*R*)-7-Methyl-3-(2-(trifluoromethyl)phenyl)-4,5,6,7-tetrahydro-[1,2,3]triazolo [1,5-a]pyridine-4,5,6-triol (13i). Yield: 81%; MP= 140°C; $[\alpha]_D^{27}$ -12.2 (c 1.0, CH_3OH). ^{1}H NMR (300 MHz, DMSO+$CDCl_3$) δ 7.68(d, J=7.1 Hz, 1H), 7.58-7.42(m, 3H), 5.40(brs, 1H), 5.07(brs, 1H), 5.93(d, J=8.6 Hz, 1H), 4.59(brs, 1H), 4.44(d, J=8.3 Hz, 1H), 4.10-3.95(m, 2H), 1.67(d, J=5.8 Hz, 3H). ^{13}C NMR (125 MHz, DMSO+$CDCl_3$) δ 141.7, 132.8, 130.7, 127.7, 125.3, 70.0, 68.7, 62.0, 52.4, 14.0. IR (neat) 3378, 2915, 1623, 1327, 1170, 1107, 1063, 1008, 853, 643 cm^{-1}. MS (ESI) *m/z* 330$[M+H]^+$; HR-MS (ESI) Calcd for $C_{14}H_{15}N_3O_3F_3[M+H]^+$: 330.1065, found: 330.1075**.**

(4*S*,5*R*,6*S*,7*R*)-3-(3,4-Dichlorophenyl)-7-methyl-4,5,6,7-tetra hydro-[1,2,3]triazolo[1,5-a] pyridine-4,5,6-triol (13j). Yield: 73%; MP= 102°C; $[\alpha]_D^{27}$ 2.5 (c 1.0, CH_3OH). ^{1}H NMR (300 MHz, DMSO+$CDCl_3$) δ 8.11(d, J=1.8 Hz, 1H), 7.86(dd, J=1.8, 6.4 Hz, 1H), 7.47(d, J=8.3 Hz, 1H), 5.06(d, J=8.2 Hz, 1H), 4.69-4.60(m, 1H), 4.11-4.05(m, 2H), 1.61(d, J=6.7 Hz, 3H). ^{13}C NMR (75 MHz, DMSO+$CDCl_3$) δ 141.7, 132.0, 131.8, 131.1, 129.9, 129.8, 128.6, 126.8, 69.6, 68.2, 62.1, 52.8, 14.8. IR (neat) 3379, 2932, 1478, 1386, 1344, 1100, 1017, 989, 823 cm^{-1}. MS (ESI) *m/z* 330$[M+H]^+$; HR-MS (ESI) Calcd for $C_{13}H_{14}N_3O_3Cl_2[M+H]^+$: 330.0412, found: 330.0425**.**

(4*S*,5*R*,6*S*,7*R*)-3-(3,4-Difluorophenyl)-7-methyl-4,5,6,7-tetra hydro-[1,2,3]triazolo[1,5-a] pyridine-4,5,6-triol (13k). Yield: 79%; MP= 194°C; $[\alpha]_D^{27}$ -29.8 (c 1.0, CH_3OH). ^{1}H NMR (300 MHz, DMSO+$CDCl_3$) δ 7.95-7.89(m, 1H), 7.80-7.76(m, 1H), 7.35-7.29 (m, 1H), 5.05(d, J=3.8 Hz, 1H), 4.64(dd, J=2.7, 4.1 Hz, 1H), 4.09-4.03(m, 2H), 1.62(d, J=6.7 Hz, 3H). ^{13}C NMR (75 MHz, DMSO+$CDCl_3$) δ 142.0, 131.5, 128.8, 123.4, 116.6, 116.5, 115.9, 115.8,

69.6, 68.2, 62.0, 52.6, 14.8. IR (neat) 3542, 3250, 3068, 2906, 2710, 1517, 1280, 1100, 1061, 771 cm^{-1}. MS (ESI) *m/z* 298$[M+H]^+$; HR-MS (ESI) Calcd for $C_{13}H_{14}N_3O_3F_2$ $[M+H]^+$: 298.1003, found: 298.1020**.**

(4*S*,5*R*,6*S*,7*R*)-3-(2,4-Difluorophenyl)-7-methyl-4,5,6,7-tetra hydro-[1,2,3]triazolo[1,5-a] pyridine-4,5,6-triol (13l). Yield: 75%; MP= 264°C; $[\alpha]_D^{27}$ -89.3 (c 1.0, CH_3OH). ^1H NMR (300 MHz, DMSO+$CDCl_3$) δ 7.71-7.61(m, 1H), 7.16-7.00(m, 2H), 5.04(d, J=2.4 Hz, 1H), 4.62-4.54(m, 1H), 4.08-4.00(m, 2H), 1.66(d, J=6.7 Hz, 3H). ^{13}C NMR (75 MHz, DMSO+$CDCl_3$) δ 137.4, 133.3, 131.3, 116.7, 110.9, 110.6, 103.8, 103.4, 103.1, 70.3, 68.7, 62.7, 52.2, 14.6. IR (neat) 3542, 3250, 3068, 2906, 2710, 1517, 1280, 1100, 1061, 771 cm^{-1}. MS (ESI) *m/z* 298 $[M+H]^+$; HR-MS (ESI) Calcd for $C_{13}H_{14}N_3O_3F_2$ $[M+H]^+$: 298.1003, found: 298.1020**.**

(4*S*,5*R*,6*S*,7*R*)-7-Methyl-3-(2,4,5-trimethylphenyl)-4,5,6,7-tetrahydro-[1,2,3]triazolo[1,5-a]pyridine-4,5,6-triol (13m). Yield: 83%; MP= 152°C; $[\alpha]_D^{27}$ -32.1 (c 1.0, CH_3OH). ^1H NMR (300 MHz, DMSO+$CDCl_3$) δ 7.10(s, 1H), 6.94(s, 1H), 5.48(d, J=4.7 Hz, 1H), 5.14(d, J=3.7 Hz, 1H), 4.94(dd, J=5.0, 3.3 Hz, 1H), 4.68(d, J=8.6 Hz, 1H), 4.62-5.54(m, 1H), 4.10-3.93(m, 1H), 2.19(s, 3H), 2.17(s, 6H), 1.63(d, J=6.6 Hz, 3H). ^{13}C NMR (75 MHz, DMSO+$CDCl_3$) δ 143.9, 134.9, 133.5, 132.1, 131.7, 131.1, 130.7, 128.6, 69.7, 68.8, 62.4, 52.4, 19.2, 18.8, 18.6, 14.7. IR (neat) 3380, 2921, 2723, 1449, 1336, 1240, 1097, 1057, 990, 820, 625 cm^{-1}. MS (ESI) *m/z* 304 $[M+H]^+$; HR-MS (ESI) Calcd for $C_{16}H_{22}N_3O_3$ $[M+H]^+$: 304.1661, found: 304.1675**.**

(4*S*,5*R*,6*S*,7*R*)-7-Methyl-3-(phenanthren-9-yl)-4,5,6,7-tetrahydro-[1,2,3]triazolo[1,5-a] pyridine-4,5,6-triol (13n). Yield: 92%; MP= 305°C; $[\alpha]_D^{27}$ -97.8 (c 1.0, CH_3OH). ^1H NMR (300 MHz, DMSO+$CDCl_3$) δ 8.85-8.74(m, 2H), 8.03-7.89(m, 3H), 7.71-7.53(m, 4H), 5.63(d, J=4.9 Hz, 1H), 5.30(d, J=3.7 Hz, 1H), 5.10(dd, J=3.7, 4.7 Hz, 1H), 4.84(d, J=8.6 Hz, 1H), 4.74-4.65 (m, 1H), 4.15-3.97(m, 2H), 1.73(d, J=6.7 Hz, 3H). ^{13}C NMR (75 MHz,

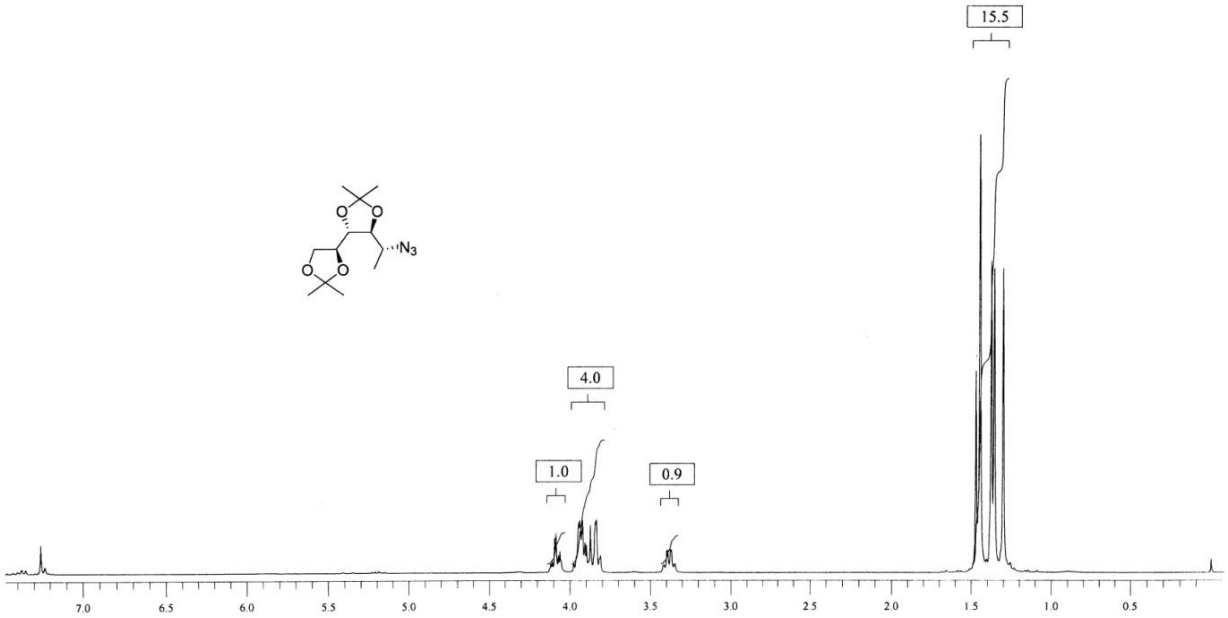
DMSO+$CDCl_3$) δ 142.8, 133.3, 130.7, 130.6, 129.6, 129.4, 128.4, 127.8, 126.5, 126.4, 126.1, 122.4, 122.2, 69.9, 68.8, 62.3, 52.5, 14.7. IR (neat) 3385, 3065, 2937, 2711, 1445, 1230, 1095, 1056, 986, 747, 725 cm^{-1}. MS (ESI) *m/z* 362 $[M+H]^+$; HR-MS (ESI) Calcd for $C_{21}H_{20}N_3O_3$ $[M+H]^+$: 362.1504, found: 362.1525**.**

(4*S*,5*R*,6*S*,7*R*)-7-Methyl-3-(thiophen-3-yl)-4,5,6,7-tetrahydro-[1,2,3]triazolo[1,5-a] pyridine-4,5,6-triol (13o). Yield: 72%; MP= 195°C; $[\alpha]_D^{27}$ 21.3 (c 1.0, CH_3OH). ^1H NMR (300 MHz, DMSO+$CDCl_3$) δ 7.89(d, J=2.2 Hz, 1H), 7.66(d, J=4.7 Hz, 1H), 7.36(dd, J=2.9, 4.7, 1H), 5.47(brs, 1H), 5.36-5.15(m, 1H), 5.00(brs, 1H), 4.64(dq, J=1.8, 6.6 Hz, 1H), 4.07(brs, 2H), 1.60(d, J=6.6 Hz, 3H). ^{13}C NMR (75 MHz, DMSO+$CDCl_3$) δ 140.7, 132.0, 130.2, 126.8, 124.6, 121.7, 69.5, 68.1, 61.9, 52.6, 14.7. IR (neat) 3379, 2932, 1478, 1386, 1344, 1100, 1017, 989, 823 cm^{-1}. MS (ESI) *m/z* 268$[M+H]^+$; HR-MS (ESI) Calcd for $C_{11}H_{14}N_3O_3S[M+H]^+$: 268.0755, found: 268.0770**.**

(4*S*,5*R*,6*S*,7*R*)-3-Hexyl-7-methyl-4,5,6,7-tetrahydro-[1,2,3]triazolo[1,5-a]pyridine-4,5,6-triol (13p). Yield: 87%; MP= 152°C; $[\alpha]_D^{27}$ -48.8 (c 1.0, CH_3OH). ^1H NMR (300 MHz, DMSO+$CDCl_3$) δ 5.44(brs, 1H), 5.21(brs, 1H), 5.01(d, J=9.0 Hz, 1H), 4.88(d, J=9.0 Hz, 1H), 4.48(q, J=6.7 Hz, 1H), 4.02(brs, 2H), 2.80-2.63(m, 2H), 1.68-1.54(m, 5H), 1.38-1.23(m, 6H), 0.88(t, J=6.9 Hz, 3H). ^{13}C NMR (125 MHz, DMSO+$CDCl_3$) δ 144.7, 130.6, 70.4, 68.8, 62.4, 51.7, 31.0, 28.8, 28.5, 24.8 , 21.9, 14.7, 13.7. IR (neat) 3544, 3331, 2932, 2857, 2699, 1700, 1643, 1354, 1242, 1098, 1060, 994 cm^{-1}. MS (ESI) *m/z* 270 $[M+H]^+$; HR-MS (ESI) Calcd for $C_{13}H_{24}N_3O_3$ $[M+H]^+$: 270.1817, found: 270.1805**.**

(4*S*,5*R*,6*S*,7*R*)-3-Dodecyl-7-methyl-4,5,6,7-tetrahydro-[1,2,3]triazolo[1,5-a]pyridine-4,5,6-triol (13q). Yield: 90%; MP= 165°C; $[\alpha]_D^{27}$ 24.2 (c 1.0, CH_3OH). ^1H NMR (300 MHz, DMSO+$CDCl_3$) δ 5.35(d, J=3.9 Hz, 1H), 5.03(d, J=2.6 Hz, 1H), 4.96(dd, J=6.7, 2.4 Hz, 1H), 4.76(d, J=9.2 Hz, 1H), 4.60-4.50(m, 1H), 4.09(brs, 2H), 2.76 (brt, J=4.7 Hz, 2H), 1.73-1.60(m, 5H), 1.41-1.18(m, 18H), 0.88(t, J=6.9 Hz, 3H). ^{13}C NMR (75 MHz, DMSO+$CDCl_3$)

δ 70.4, 68.8, 62.4, 51.8, 31.1, 28.9, 28.8, 28.7, 28.5, 24.8, 21.9, 14.3, 13.6. IR (neat) 3539, 3282, 2926, 2852, 2696, 1463, 1098, 1061, 994 cm^{-1}. MS (ESI) *m/z* 479$[M+H]^+$; HR-MS (ESI) Calcd for $C_{19}H_{36}N_3O_3$ $[M+H]^+$: 354.2756, found: 354.2762.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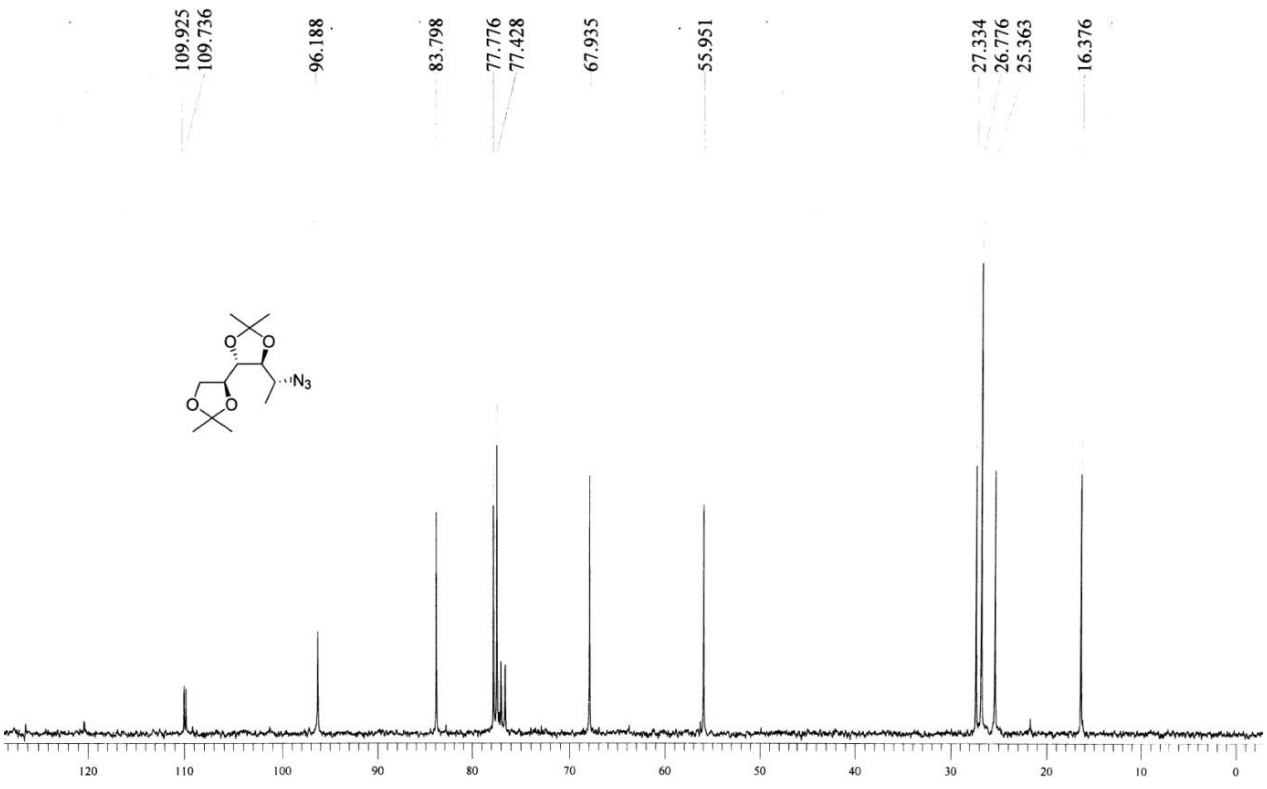
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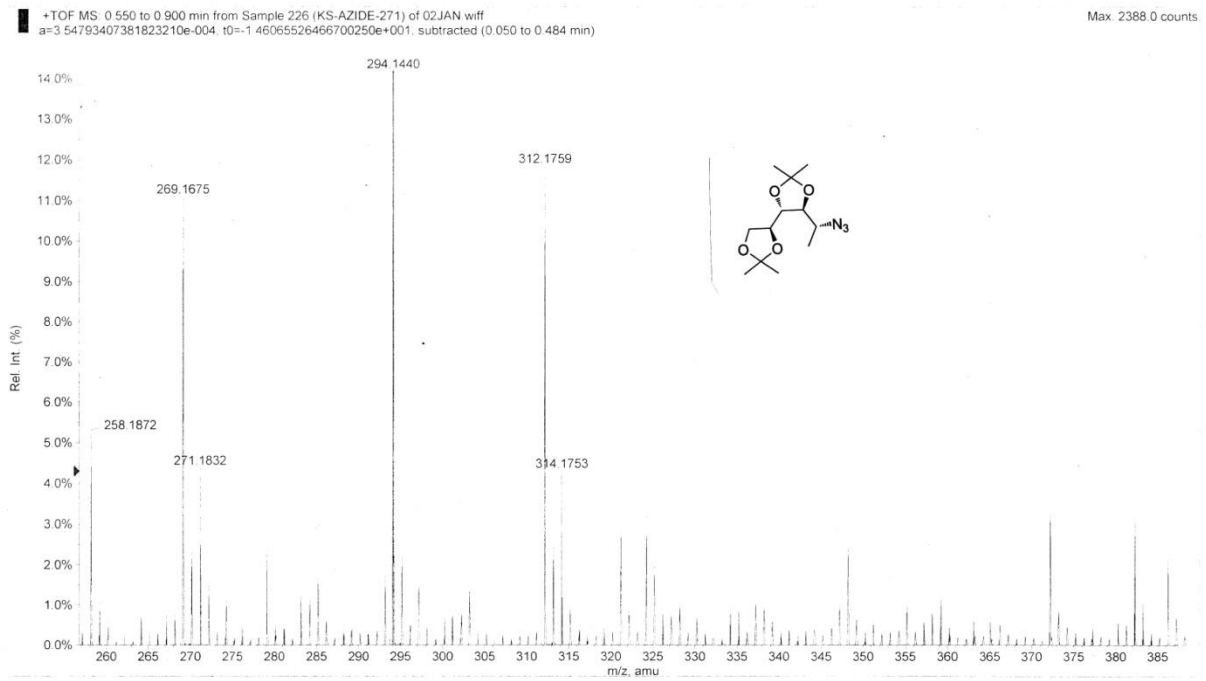
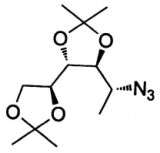


Elemental composition calculator



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Add proton:	N/A		
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	Formula	Calculated m/z (amu)	mDa Error	PPM Error	DBE
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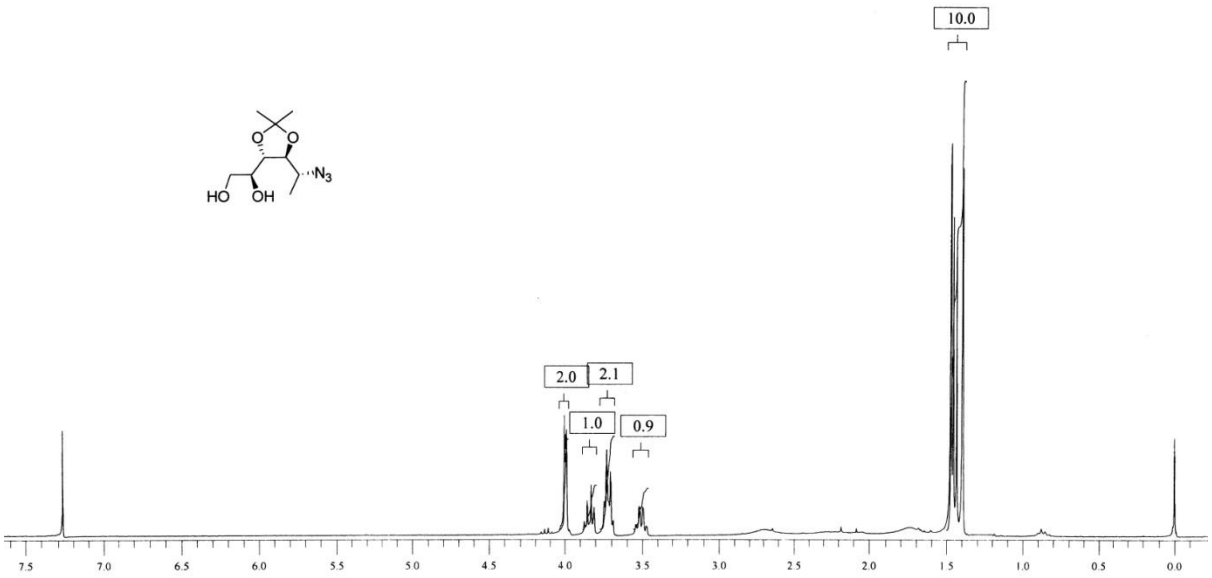
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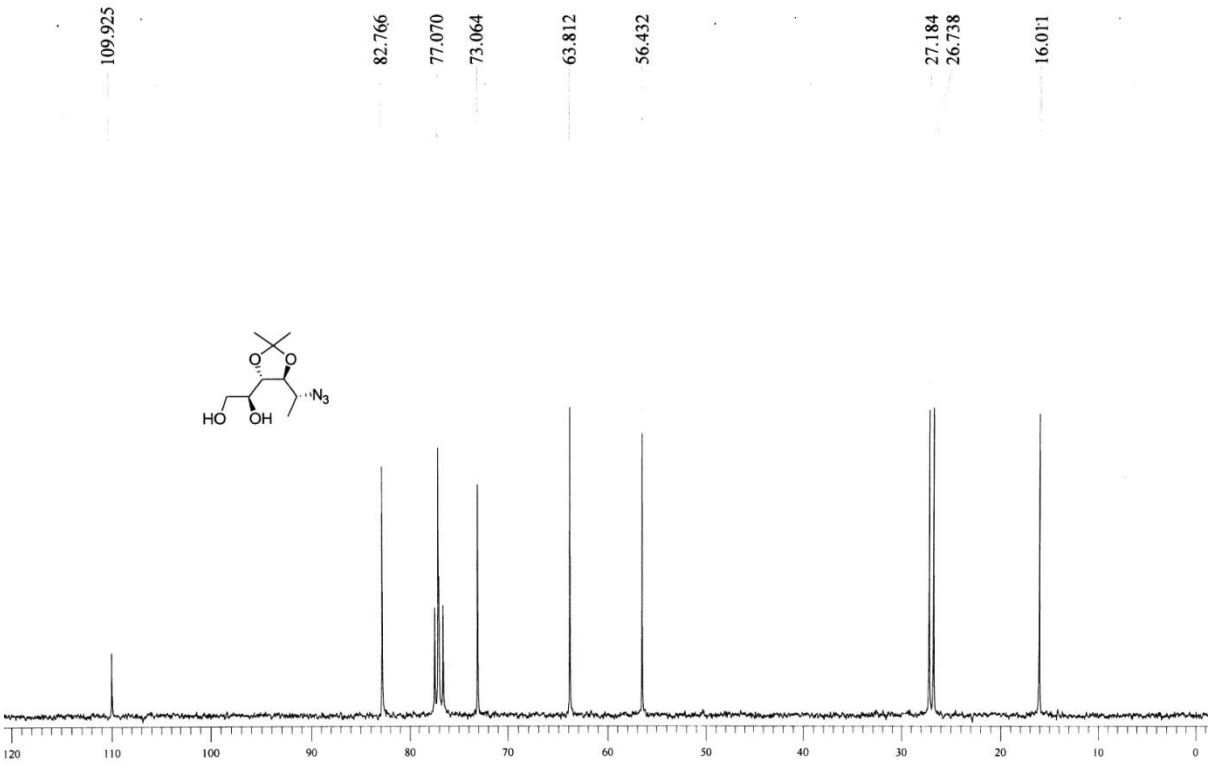
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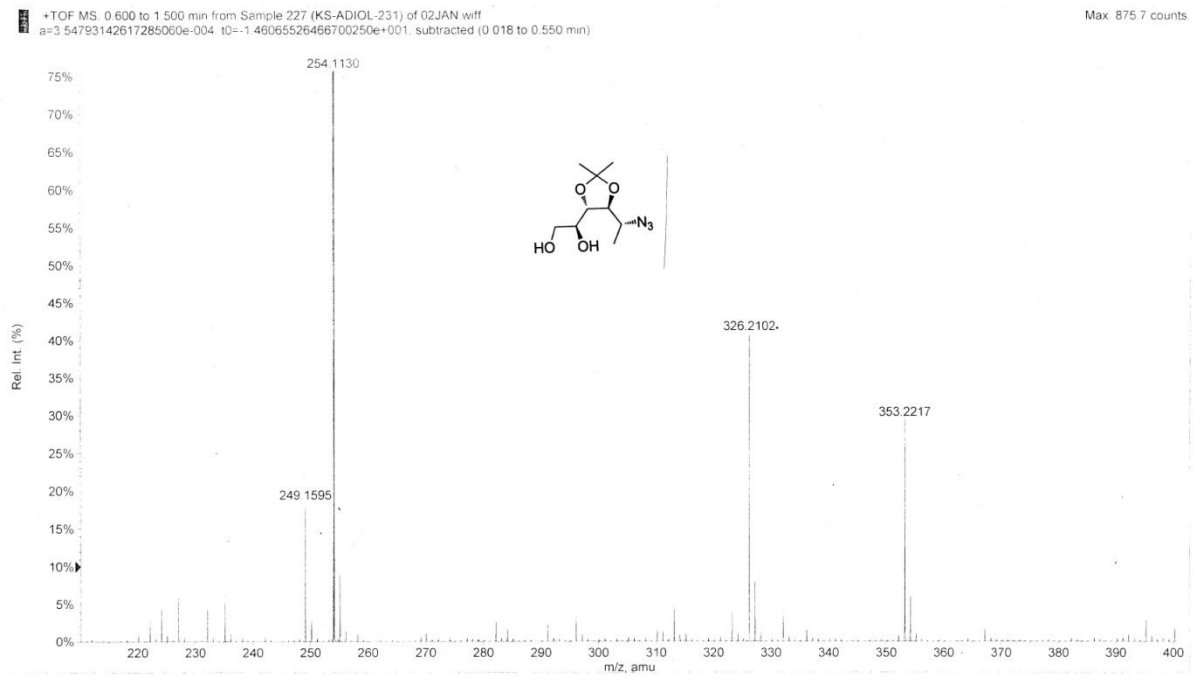
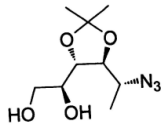


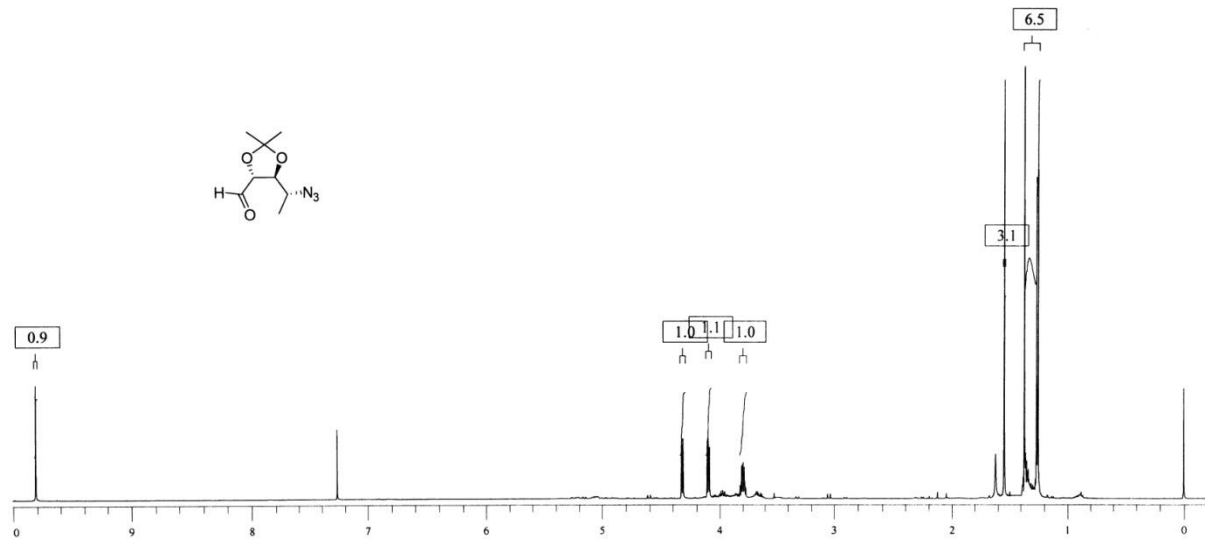
Elemental composition calculator

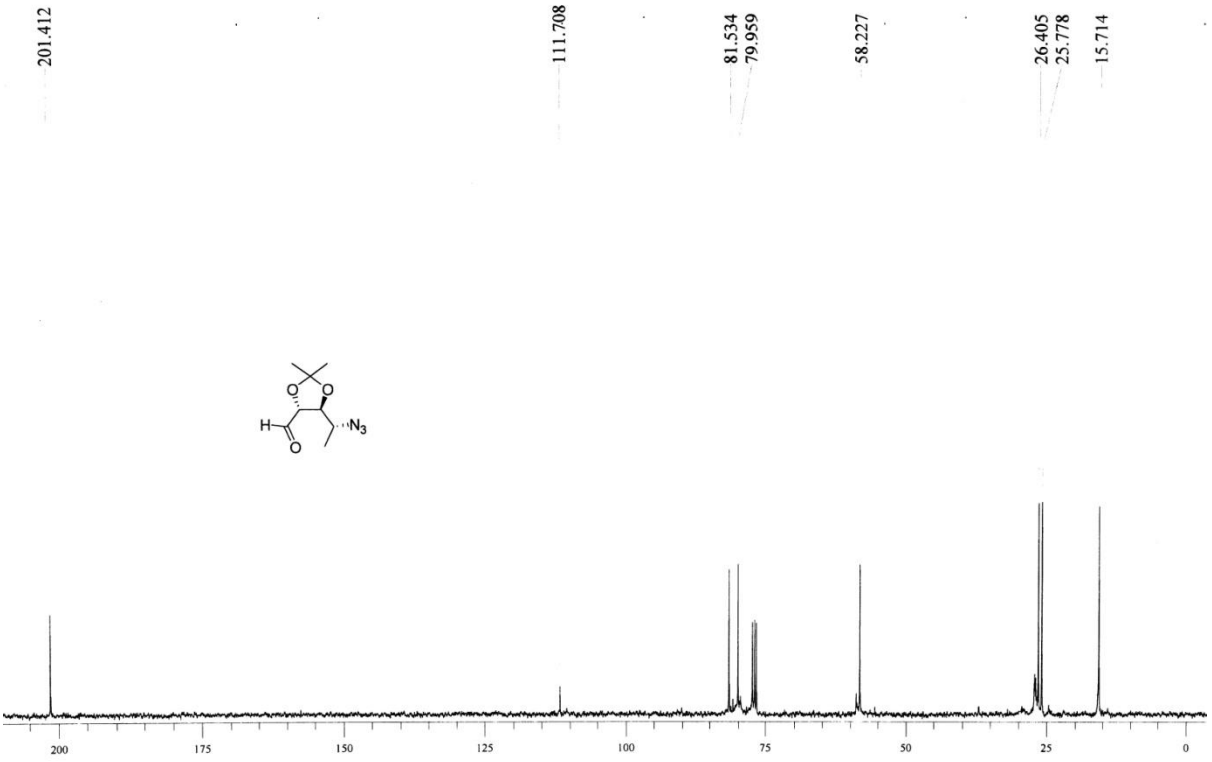


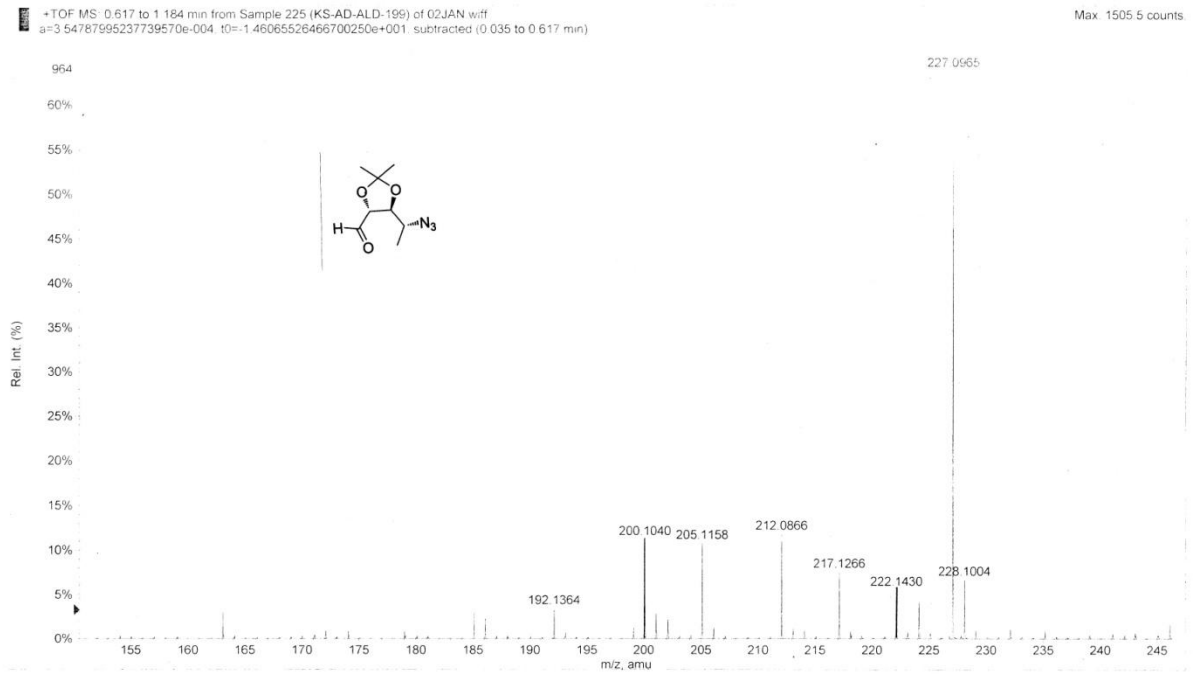
Target m/z:	+254.1130	amu	
Tolerance:	+6.0000	ppm	
Result type:	Elemental		
Max num of results:	100		
Min DBE:	-0.5000	Max DBE:	+50.000
Electron state:	OddAndEven		
Num of charges:	0		
Add water:	N/A		
Add proton:	N/A		
File Name:	02JAN.wiff		

	Elements	Min Number	Max Number
1	C	0	9
2	H	0	18
3	N	0	3
4	Na	0	1
5	O	0	4

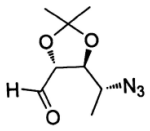
	Formula	Calculated m/z (amu)	mDa Error	PPM Error	DBE
1	$C_9H_{17}N_3O_4Na$	254.1116	1.3240	5.2104	2.5


6.5
3.1
0.9
1.0
1.1
1.0
H
O
O
O
N3
0
9
8
7
6
5
4
3
2
1
0


201.412
111.708
81.534
79.959
58.227
26.405
25.778
15.714
H
O
O
O
N3
200
175
150
125
100
75
50
25
0



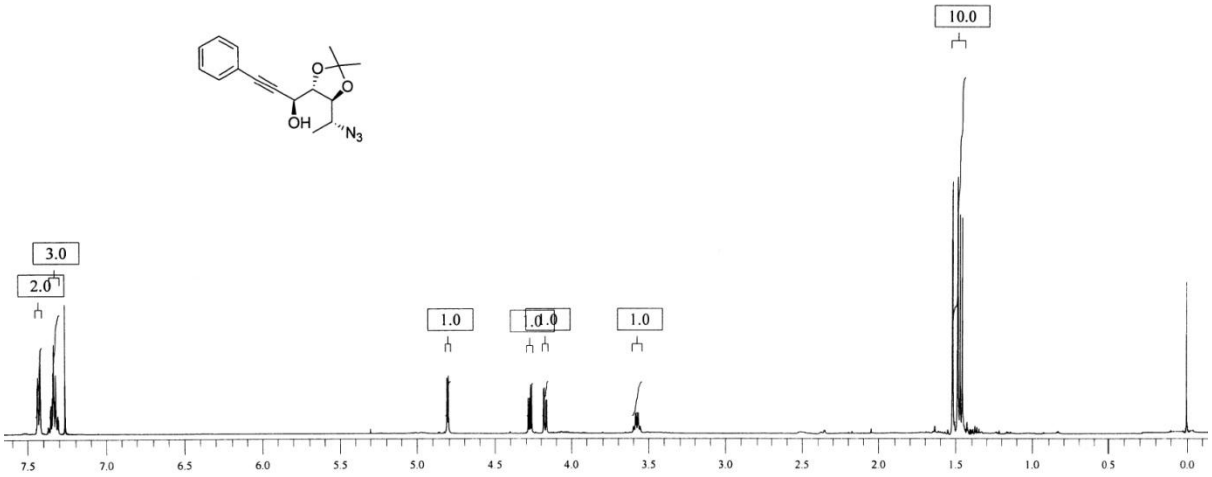
Elemental composition calculator

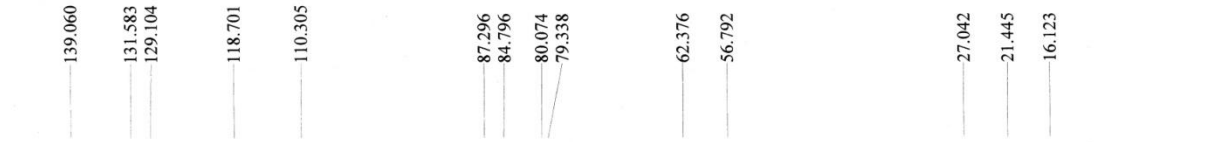


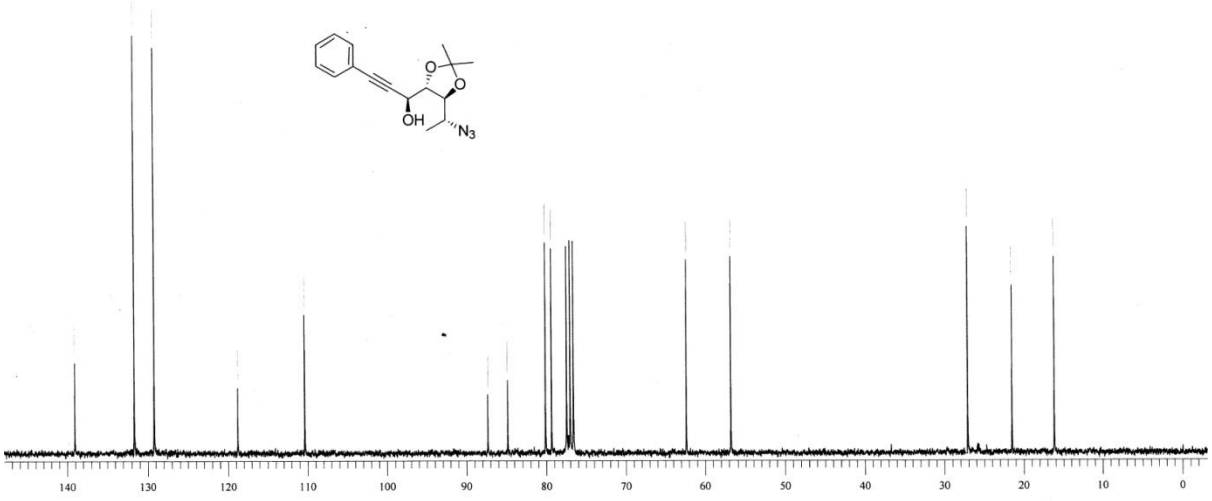
Target m/z:	+200.1040	amu	
Tolerance:	+5.0000	ppm	
Result type:	Elemental		
Max num of results:	100		
Min DBE:	-0.5000	Max DBE:	+50.000
Electron state:	OddAndEven		
Num of charges:	0		
Add water:	N/A		
Add proton:	N/A		
File Name:	02JAN.wiff		

	Elements	Min Number	Max Number
1	C	0	8
2	H	0	14
3	N	0	3
4	O	0	3

	Formula	Calculated m/z (amu)	mDa Error	PPM Error	DBE
1	$C_8H_{14}N_3O_3$	200.1035	0.4834	2.4162	3.5


10.0
3.0
2.0
1.0
1.0
1.0
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
1.0
0.5
0.0

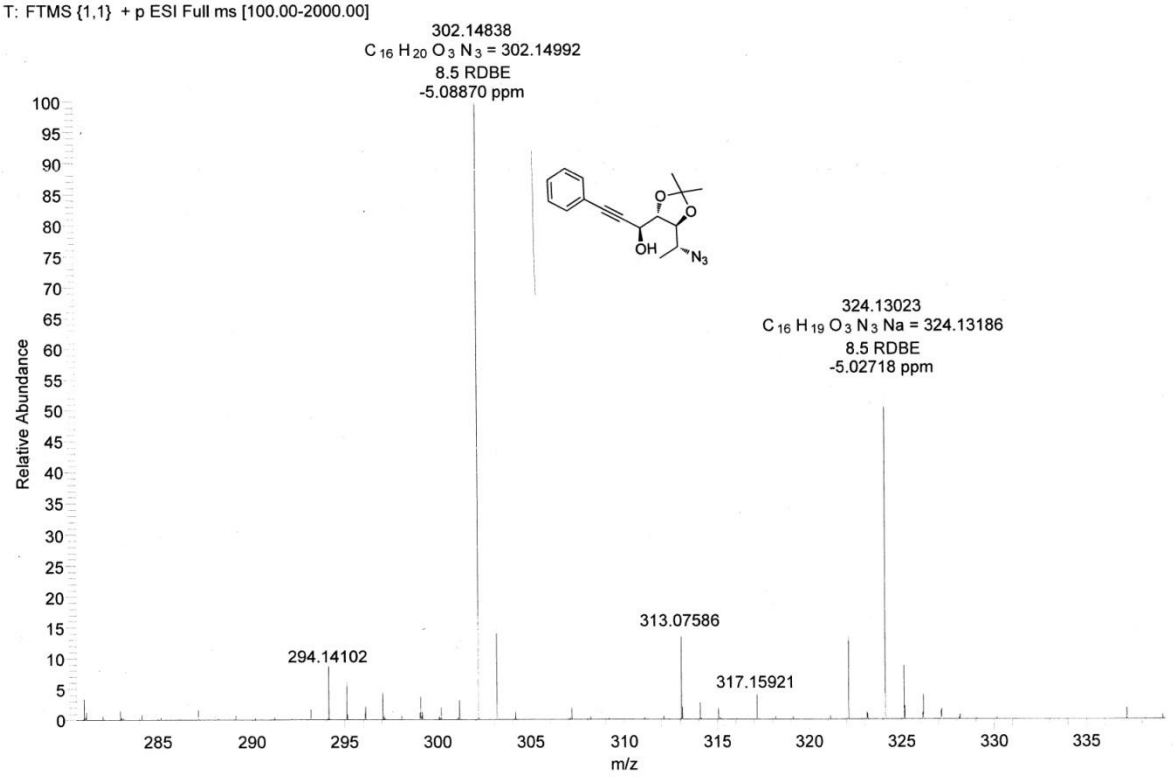

139.060
131.583
129.104
118.701
110.305
87.296
84.796
80.074
79.338
62.376
56.792
27.042
21.445
16.123

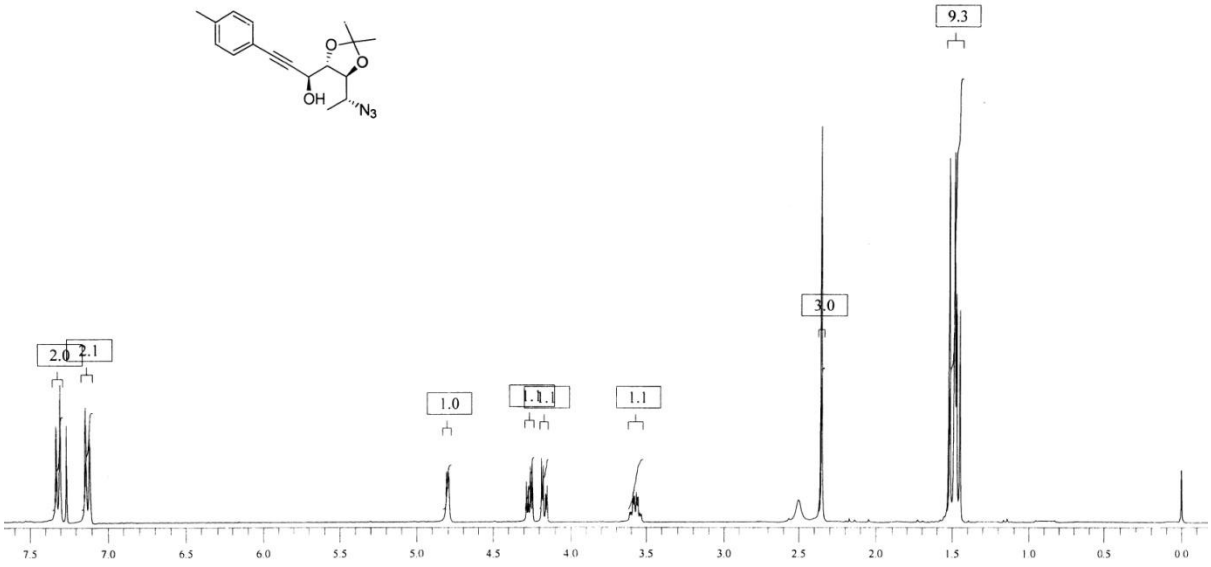

140
130
120
110
100
90
80
70
60
50
40
30
20
10
0

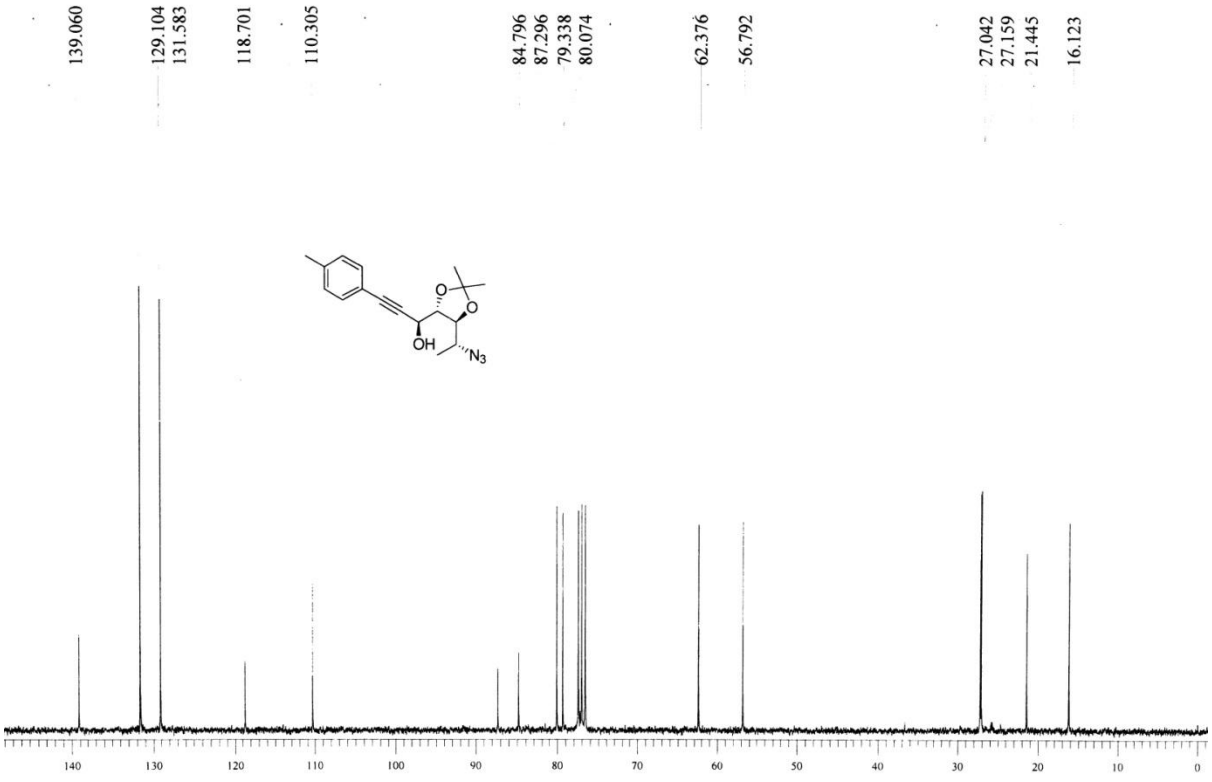
P-S-REDDY NATIONAL CENTRE FOR MASS SPECTROMETRY

KS-LPH #3-24 RT: 0.03-0.10 AV: 22 NL: 4.25E6

T: FTMS {1,1} + p ESI Full ms [100.00-2000.00]



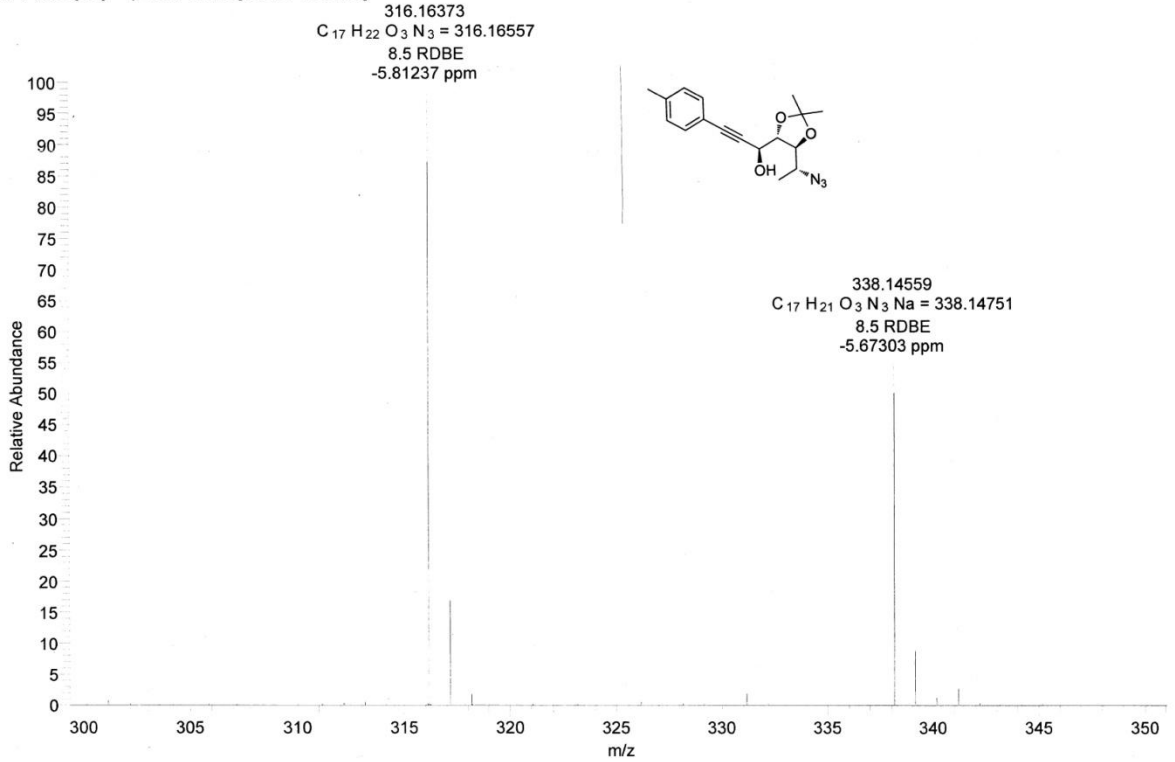


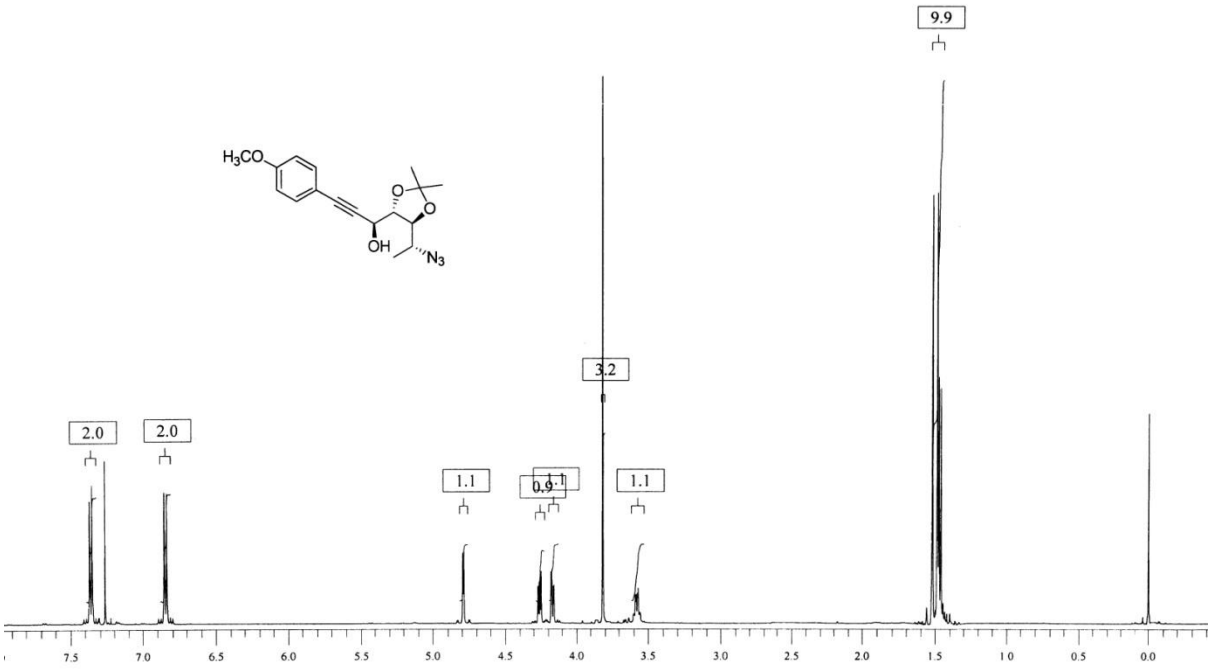


P-S-REDDY NATIONAL CENTRE FOR MASS SPECTROMETRY

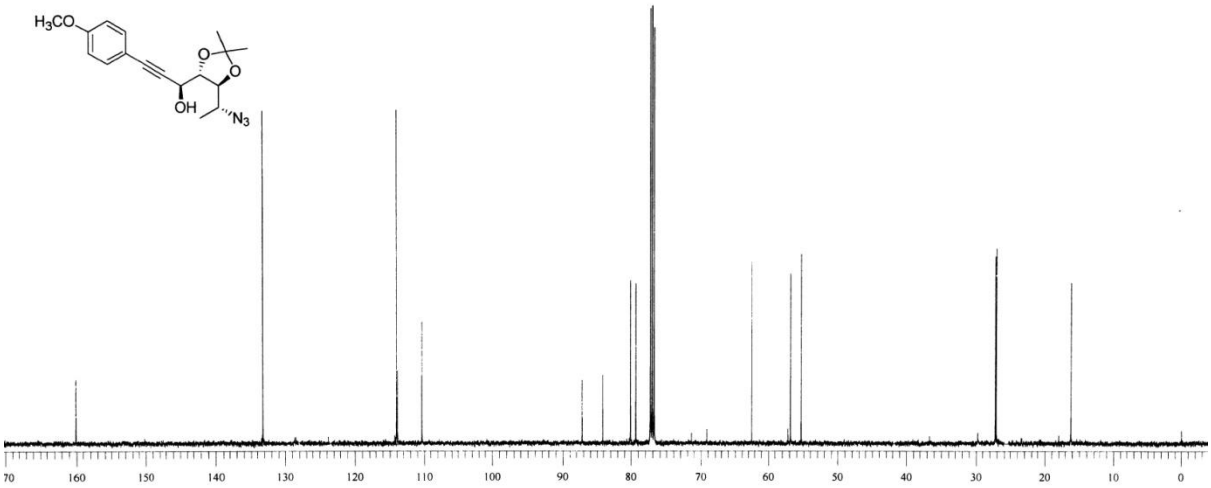
KS-L-PTOL #2-51 RT: 0.02-0.19 AV: 50 NL: 1.44E7

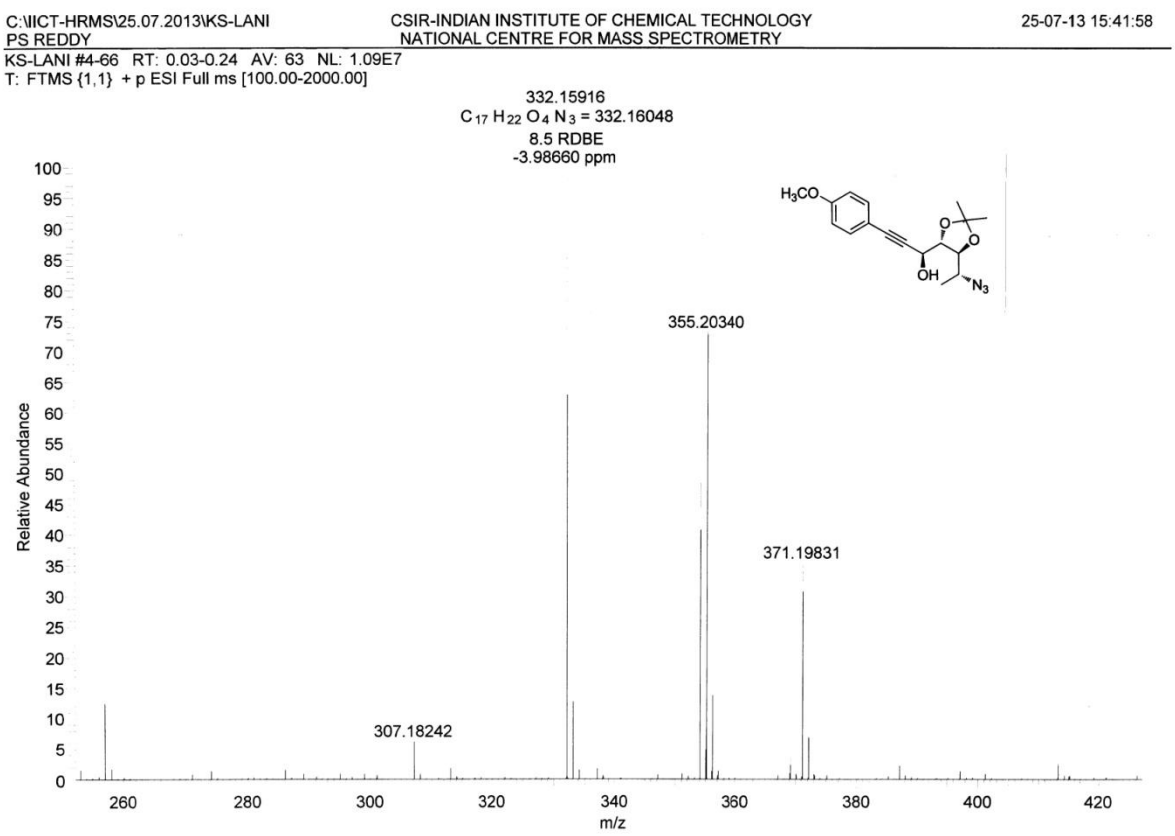
T: FTMS {1,1} + p ESI Full ms [100.00-2000.00]

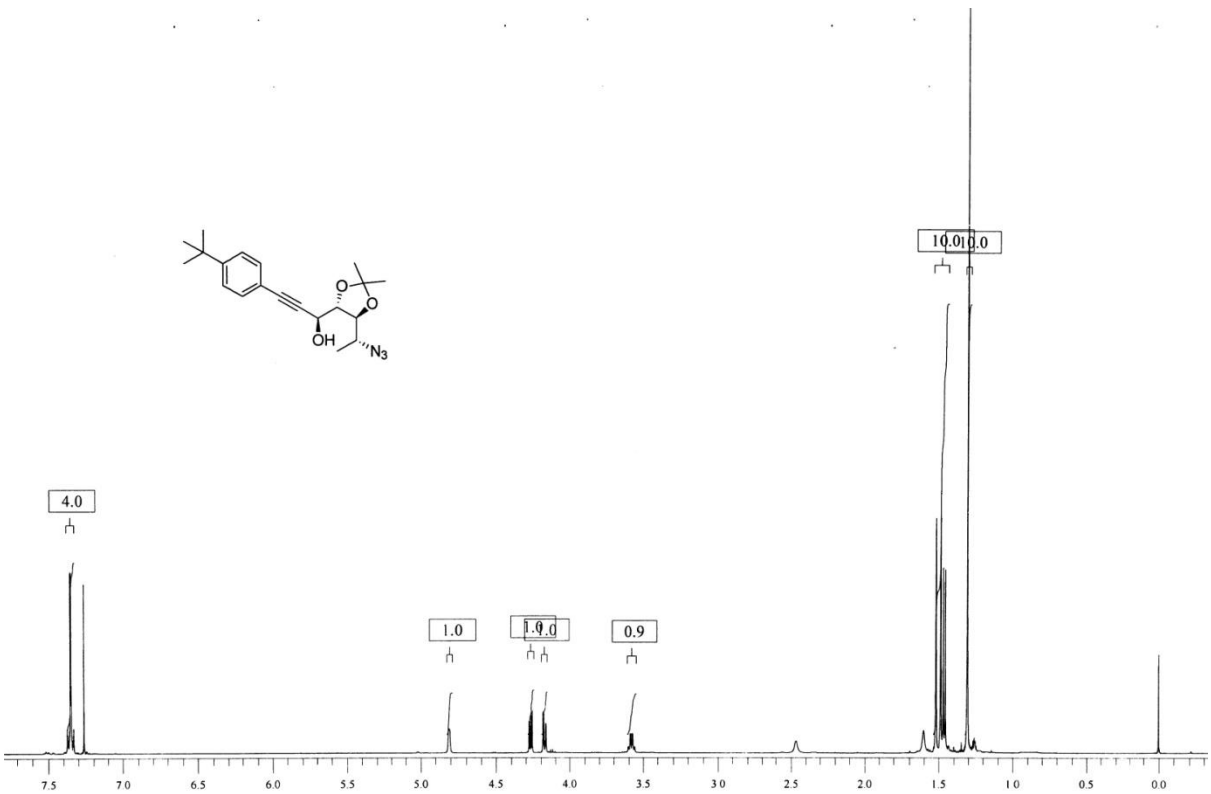


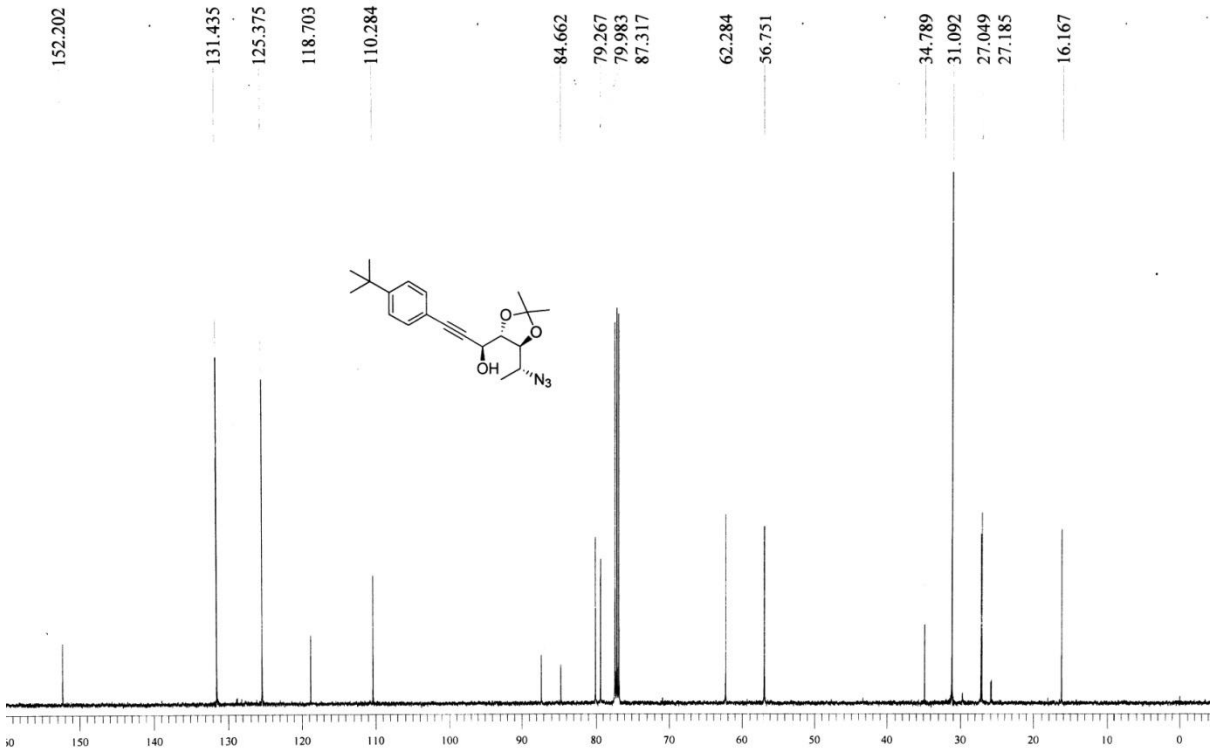


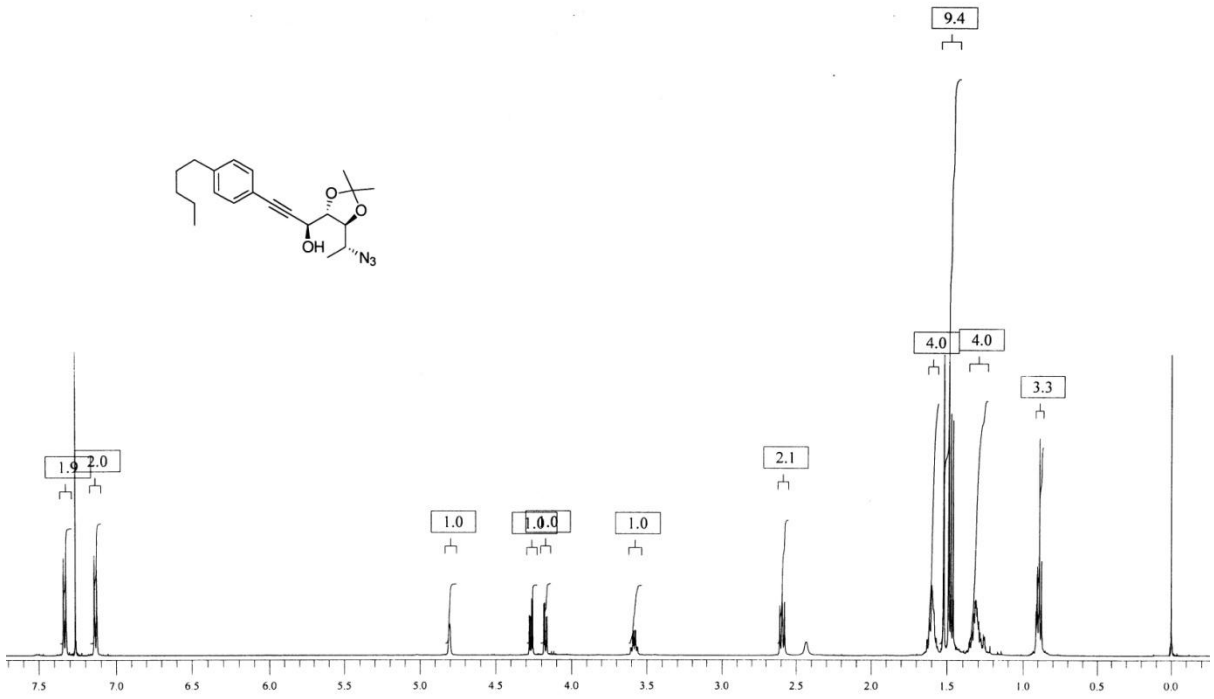
159.980
133.196
113.994
113.816
110.277
87.146
84.085
80.075
79.309
62.384
56.794
55.264
27.180
27.044
16.159

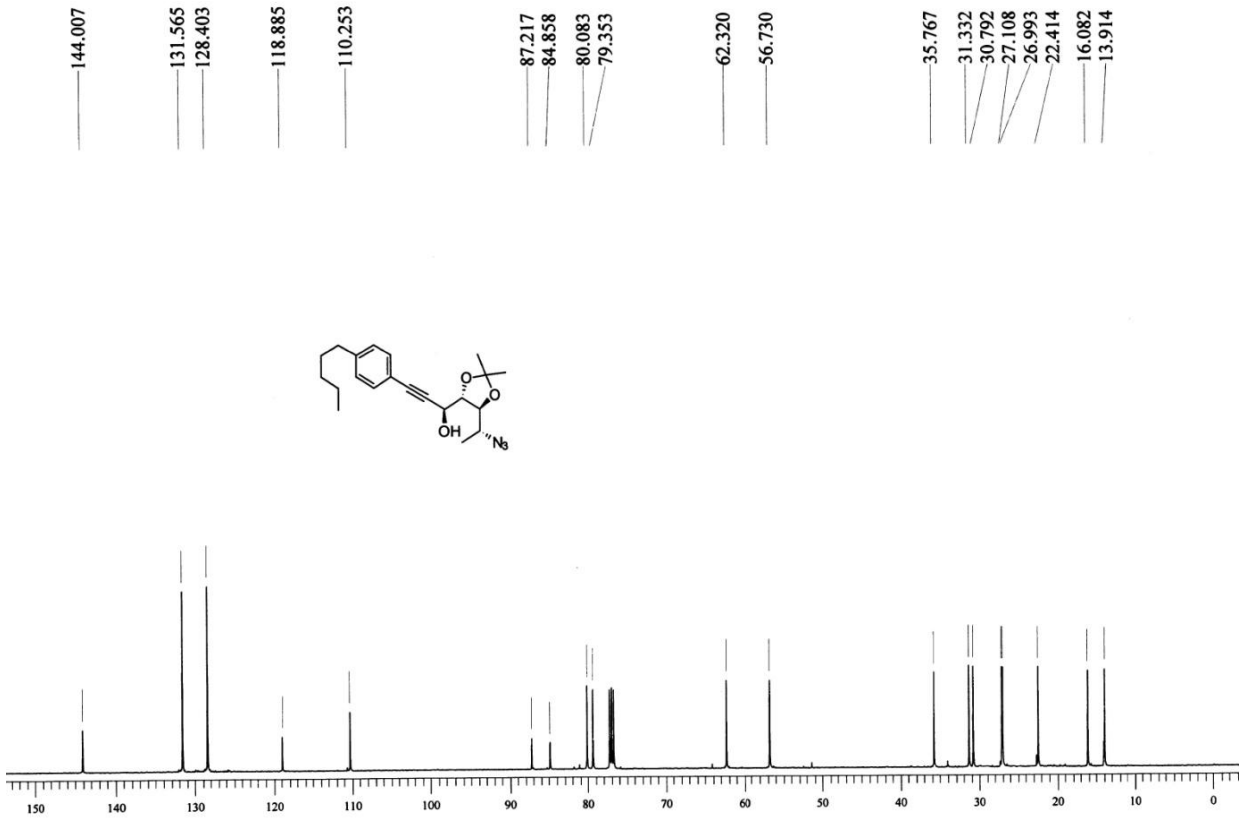


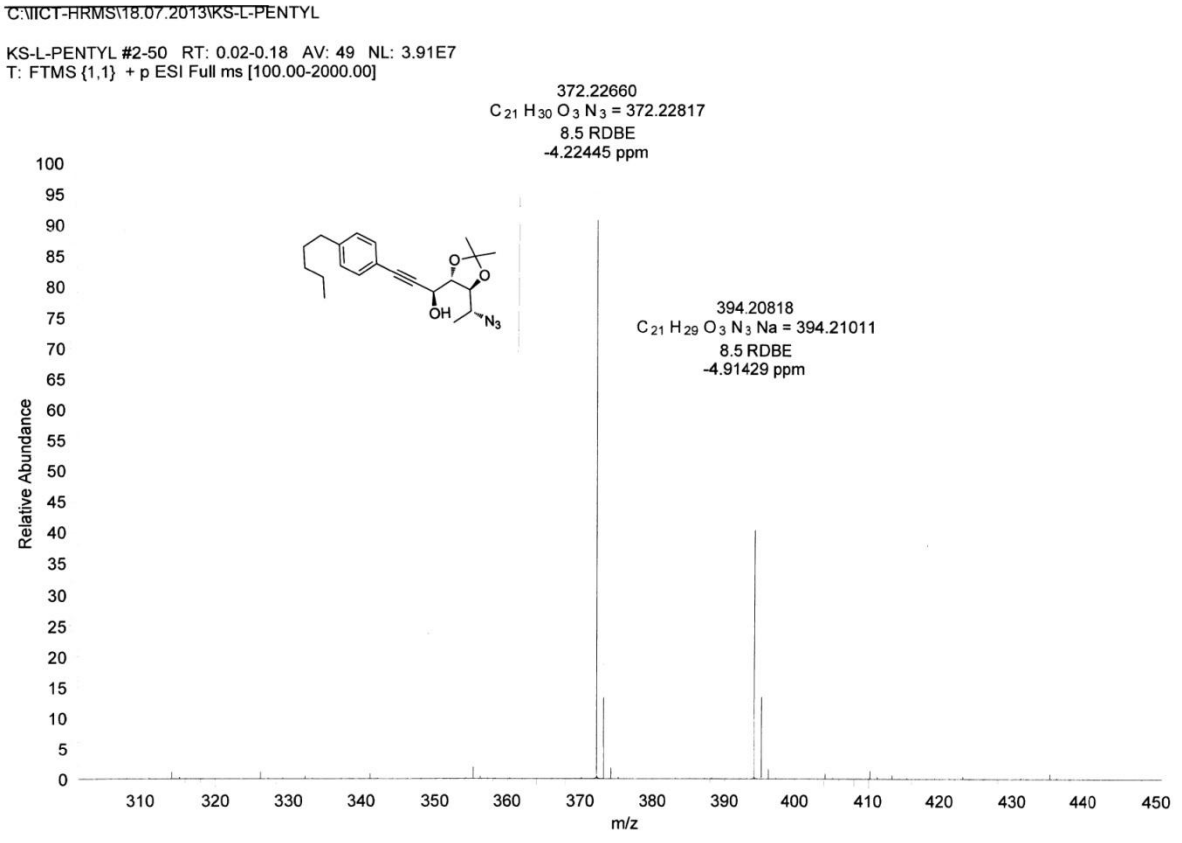

C:\IICT-HRMS\25.07.2013\KS-LANI
PS REDDY
CSIR-INDIAN INSTITUTE OF CHEMICAL TECHNOLOGY
NATIONAL CENTRE FOR MASS SPECTROMETRY
25-07-13 15:41:58
KS-LANI #4-66 RT: 0.03-0.24 AV: 63 NL: 1.09E7
T: FTMS {1,1} + p ESI Full ms [100.00-2000.00]
332.15916
C17 H22 O4 N3 = 332.16048
8.5 RDBE
-3.98660 ppm
355.20340
371.19831
307.18242
Relative Abundance
m/z

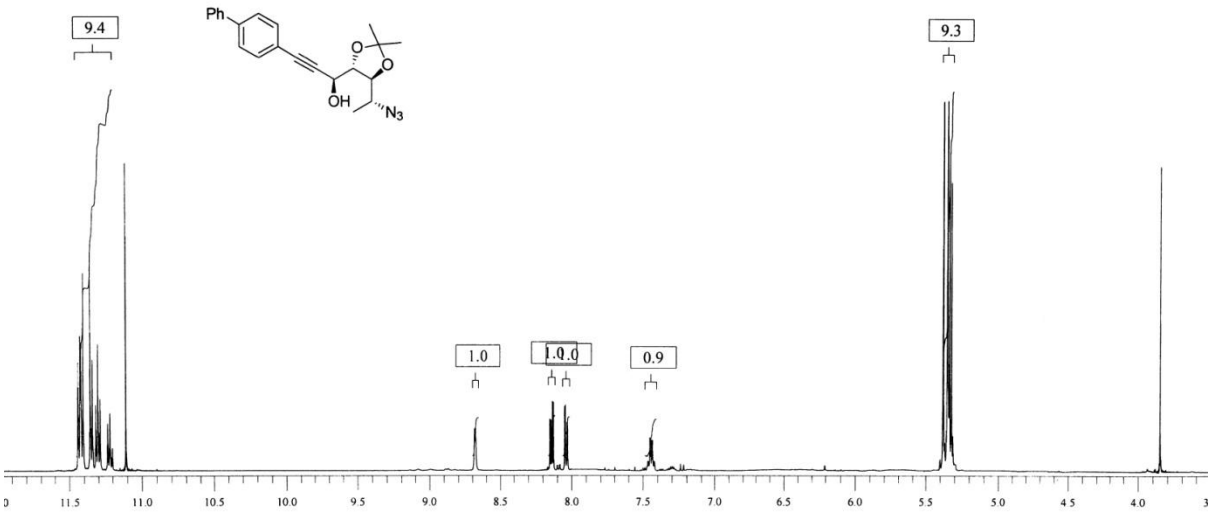

4.0
1.0
1.0.0
0.9
10.0
10.0


152.202
131.435
125.375
118.703
110.284
84.662
79.267
79.983
87.317
62.284
56.751
34.789
31.092
27.049
27.185
16.167
OH
N3

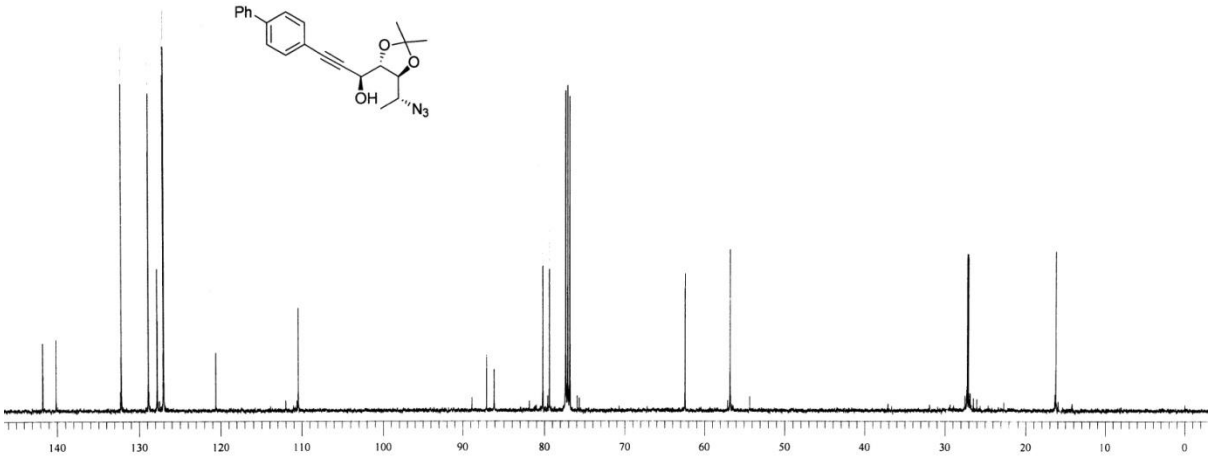

9.4
4.0
4.0
3.3
2.1
1.9
2.0
1.0
1.0
1.0
OH
N3

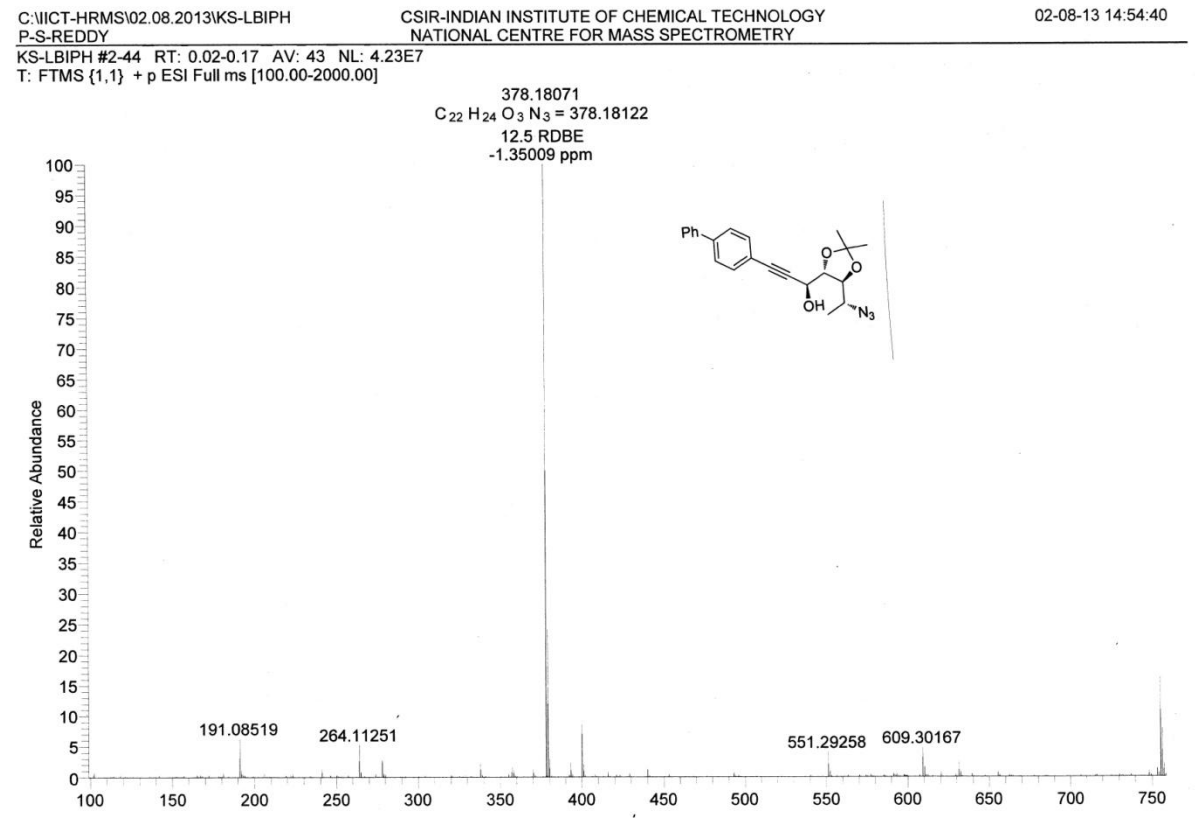

144.007
131.565
128.403
118.885
110.253
87.217
84.858
80.083
79.353
62.320
56.730
35.767
31.332
30.792
27.108
26.993
22.414
16.082
13.914
OH
N3
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0

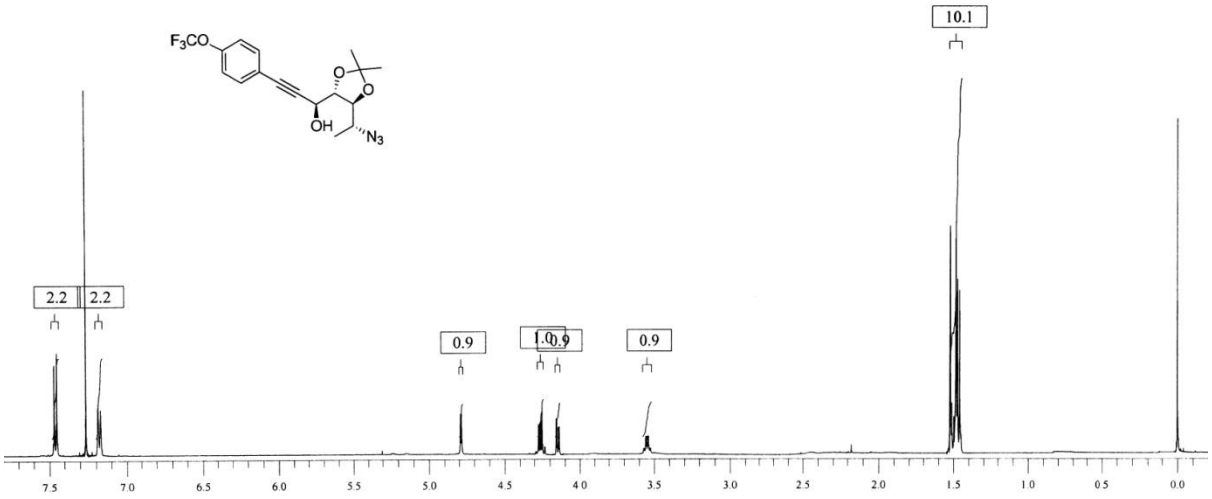

C:\IICT-HRMS\18.07.2013\KS-L-PENTYL
KS-L-PENTYL #2-50 RT: 0.02-0.18 AV: 49 NL: 3.91E7
T: FTMS {1,1} + p ESI Full ms [100.00-2000.00]
372.22660
C21 H30 O3 N3 = 372.22817
8.5 RDBE
-4.22445 ppm
394.20818
C21 H29 O3 N3 Na = 394.21011
8.5 RDBE
-4.91429 ppm
OH
N3
Relative Abundance
m/z
100
95
90
85
80
75
70
65
60
55
50
45
40
35
30
25
20
15
10
5
0
310
320
330
340
350
360
370
380
390
400
410
420
430
440
450

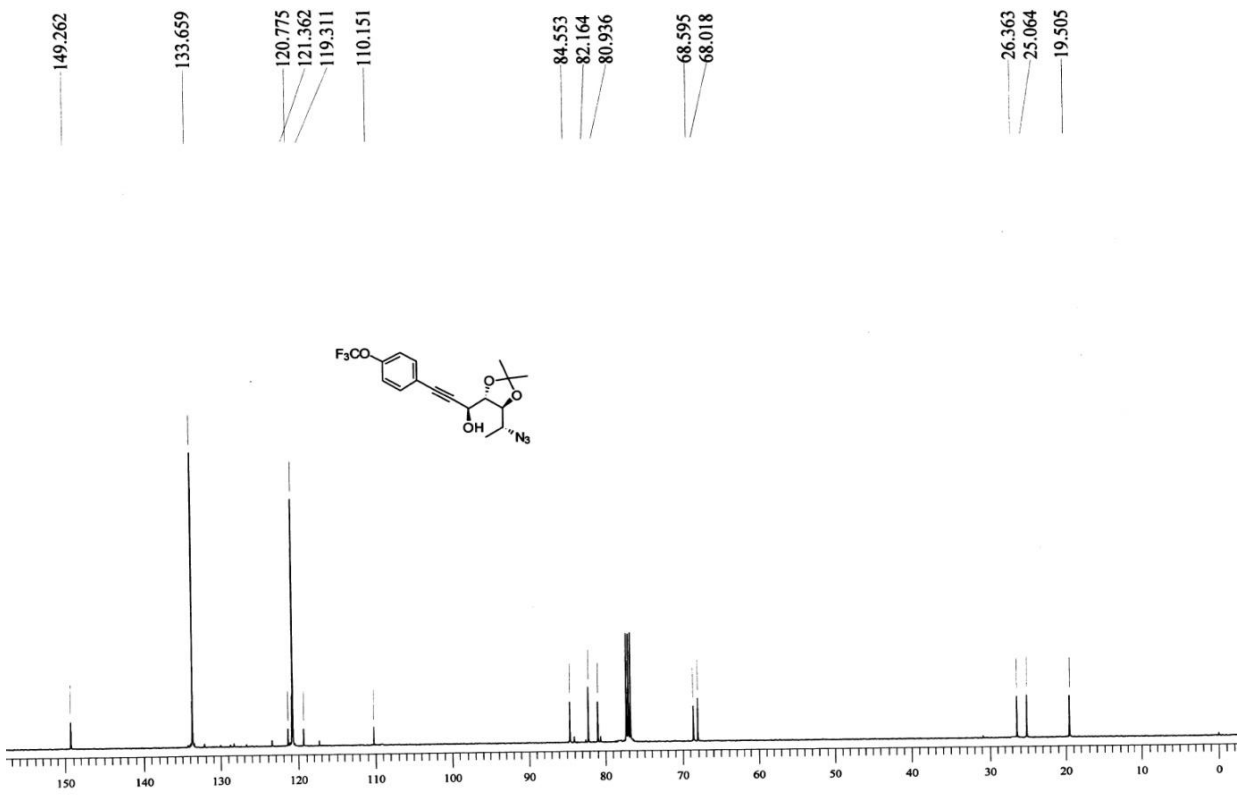

9.4
9.3
1.0
1.0.0
0.9
Ph
O
O
OH
N3

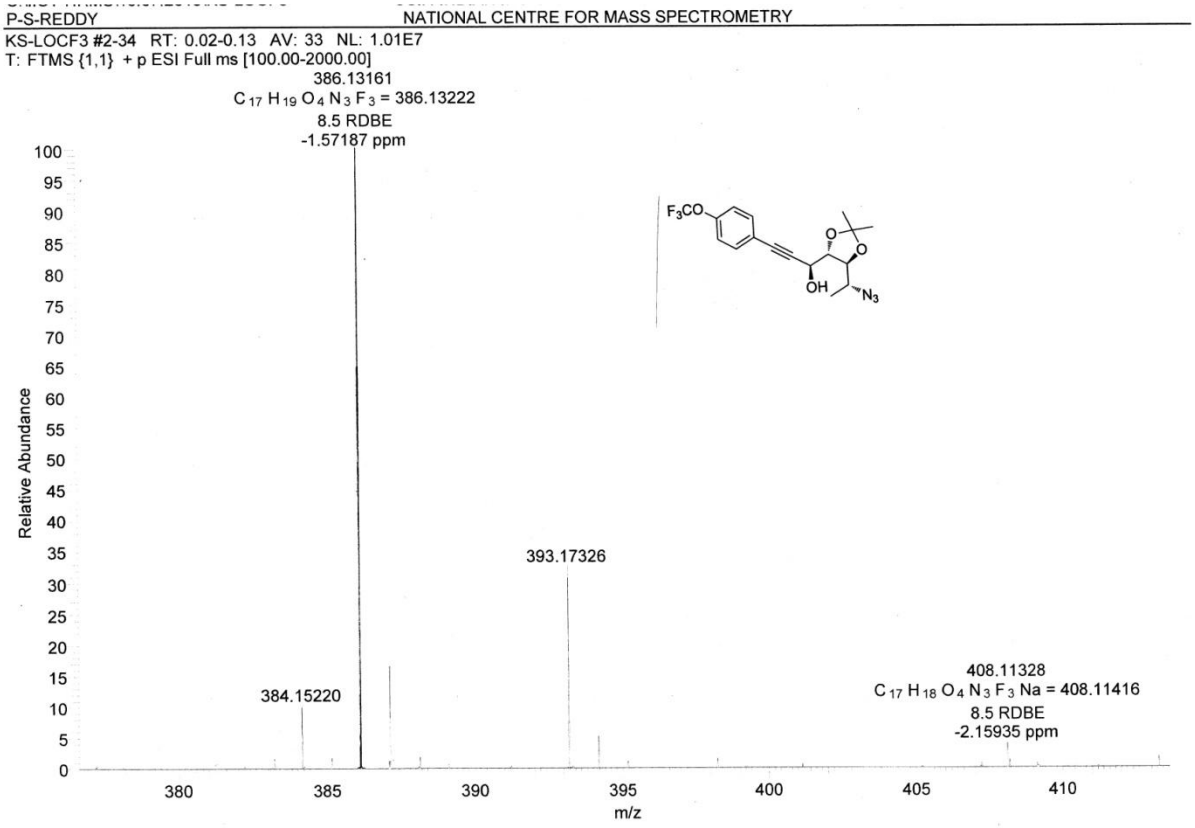

141.604
140.043
132.106
128.827
127.731
127.023
126.966
120.579
110.332
86.995
86.068
80.096
79.268
62.419
56.752
27.171
27.041
16.146

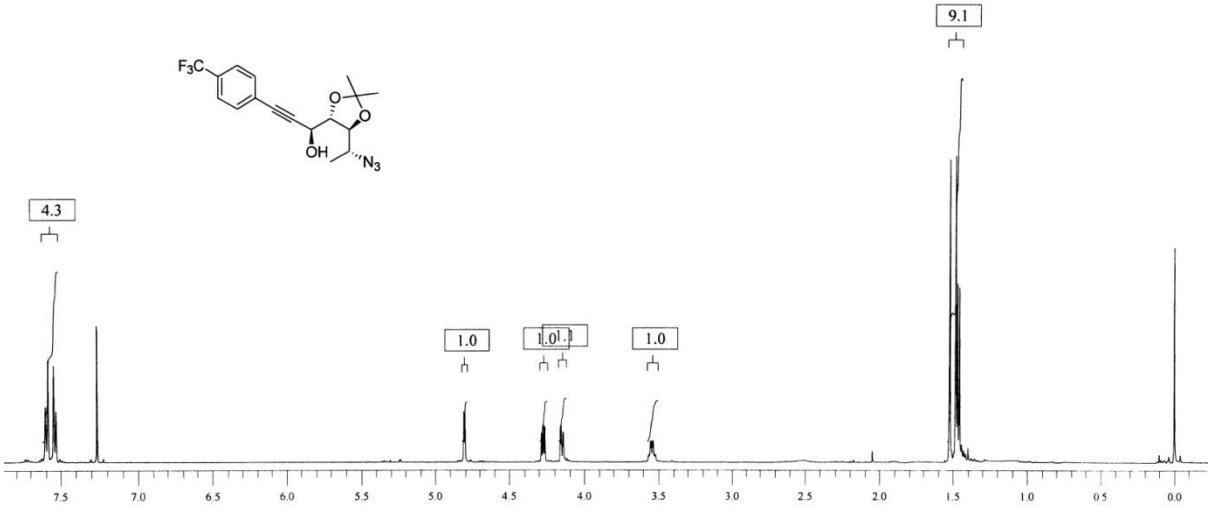

Ph
O
O
OH
N3


C:\IICT-HRMS\02.08.2013\KS-LBIPH
P-S-REDDY
CSIR-INDIAN INSTITUTE OF CHEMICAL TECHNOLOGY
NATIONAL CENTRE FOR MASS SPECTROMETRY
02-08-13 14:54:40
KS-LBIPH #2-44 RT: 0.02-0.17 AV: 43 NL: 4.23E7
T: FTMS {1,1} + p ESI Full ms [100.00-2000.00]
378.18071
C22 H24 O3 N3 = 378.18122
12.5 RDBE
-1.35009 ppm
Relative Abundance
191.08519
264.11251
551.29258
609.30167
Ph
OH
N3

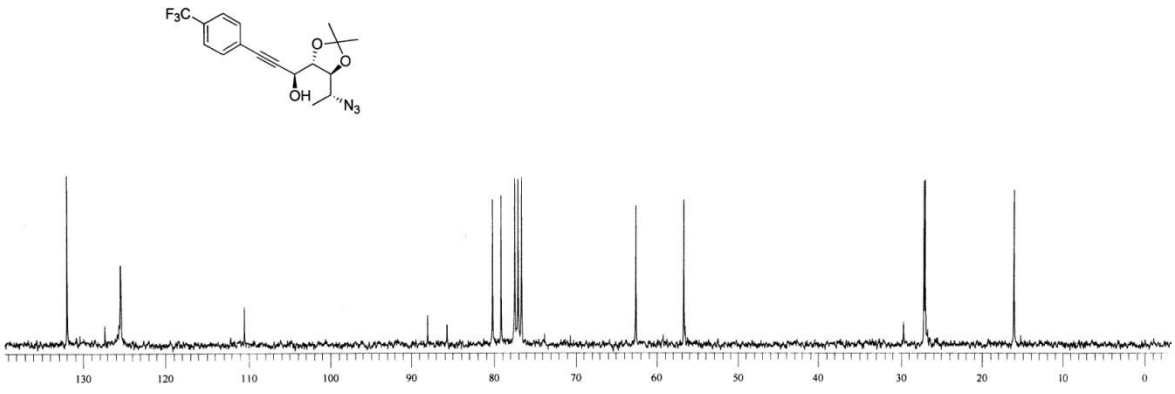

F3CO
OH
N3
10.1
2.2
2.2
0.9
1.0
0.9
0.9


149.262
133.659
120.775
121.362
119.311
110.151
84.553
82.164
80.936
68.595
68.018
26.363
25.064
19.505
F3CO
OH
N3
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0


P-S-REDDY
NATIONAL CENTRE FOR MASS SPECTROMETRY
KS-LOCF3 #2-34 RT: 0.02-0.13 AV: 33 NL: 1.01E7
T: FTMS {1,1} + p ESI Full ms [100.00-2000.00]
386.13161
C 17 H 19 O 4 N 3 F 3 = 386.13222
8.5 RDBE
-1.57187 ppm
F3CO
OH
N3
Relative Abundance
393.17326
384.15220
408.11328
C 17 H 18 O 4 N 3 F 3 Na = 408.11416
8.5 RDBE
-2.15935 ppm
m/z


F3C
O
O
OH
N3
4.3
1.0
1.0
1.0
9.1
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
1.0
0.5
0.0


131.977
125.321
110.451
87.979
85.666
80.183
79.114
62.538
56.707
27.169
27.023
16.086


F3C
O
O
OH
N3
130
120
110
100
90
80
70
60
50
40
30
20
10
0

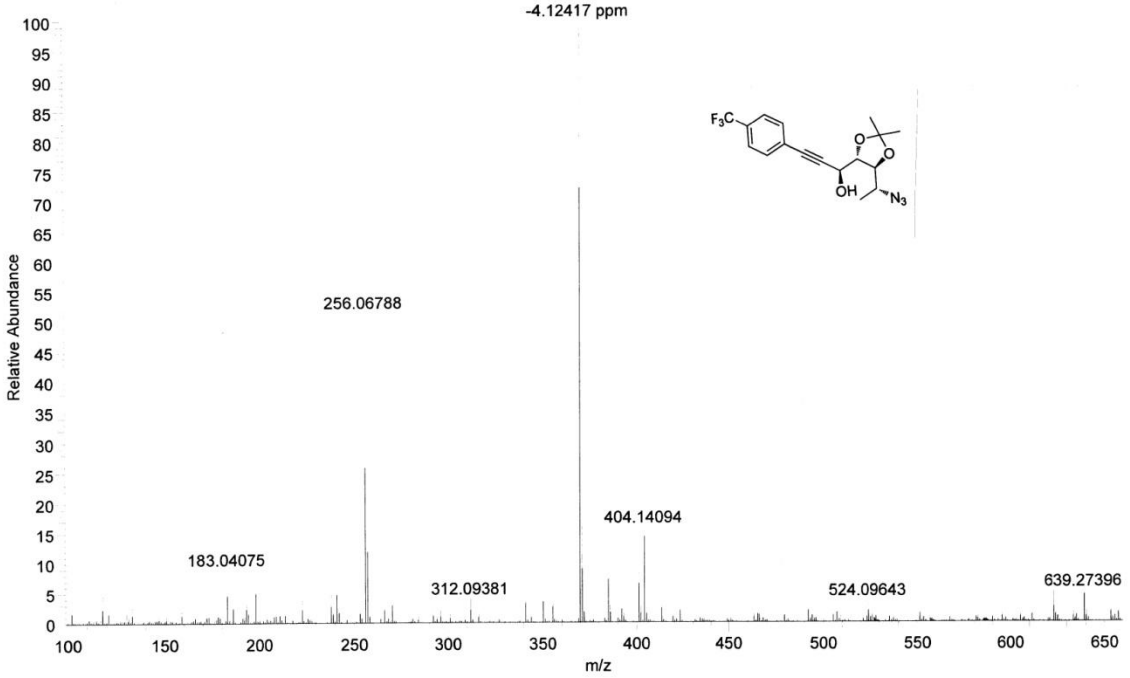
C:\IICT-HRMS\18.07.2013\KS-L4-CF3
P-S-REDDY

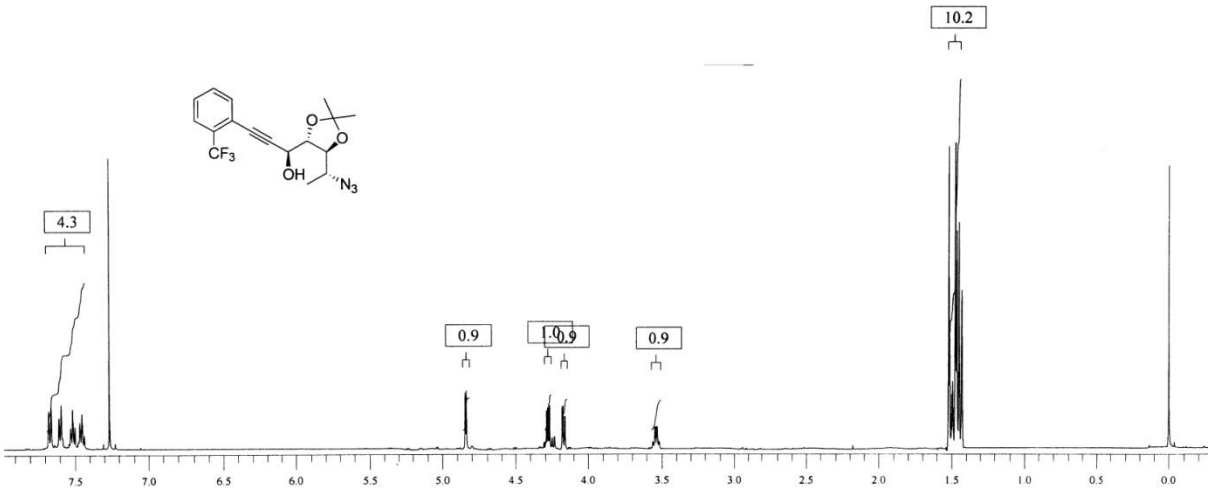
CSIR-INDIAN INSTITUTE OF CHEMICAL TECHNOLOGY
NATIONAL CENTRE FOR MASS SPECTROMETRY

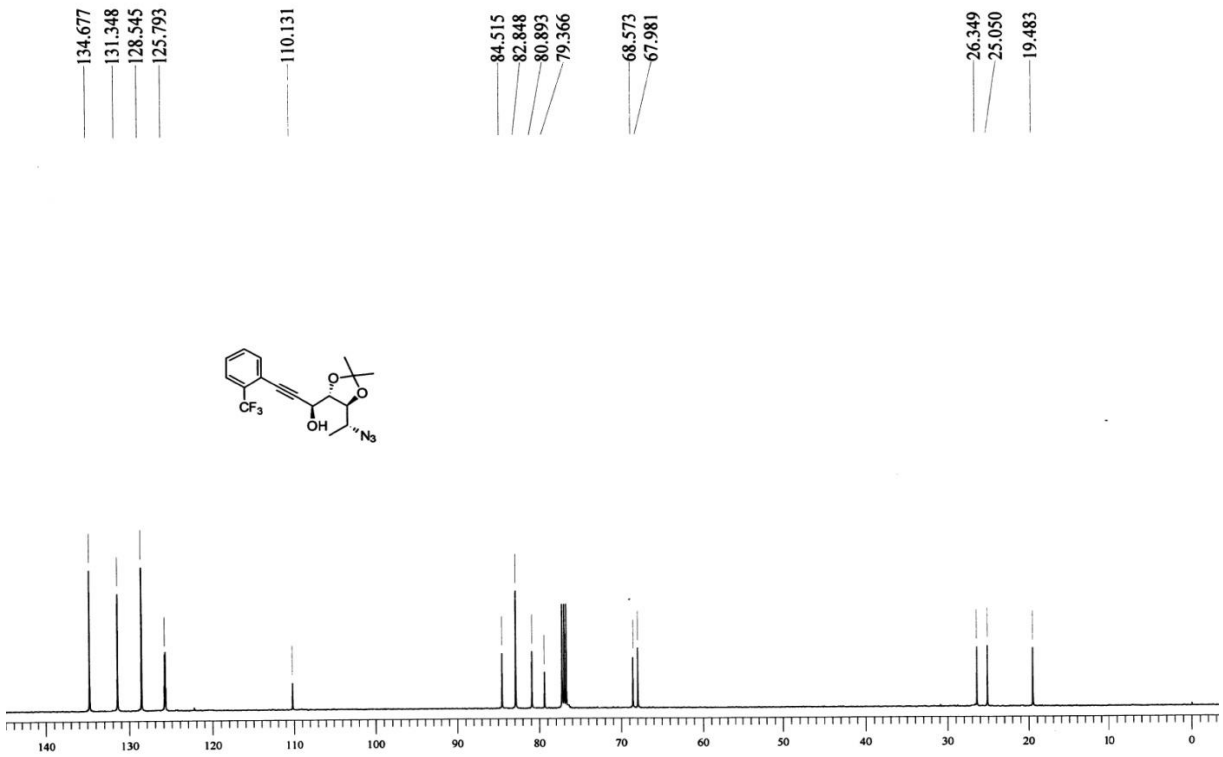
18-07-13 16:55:34

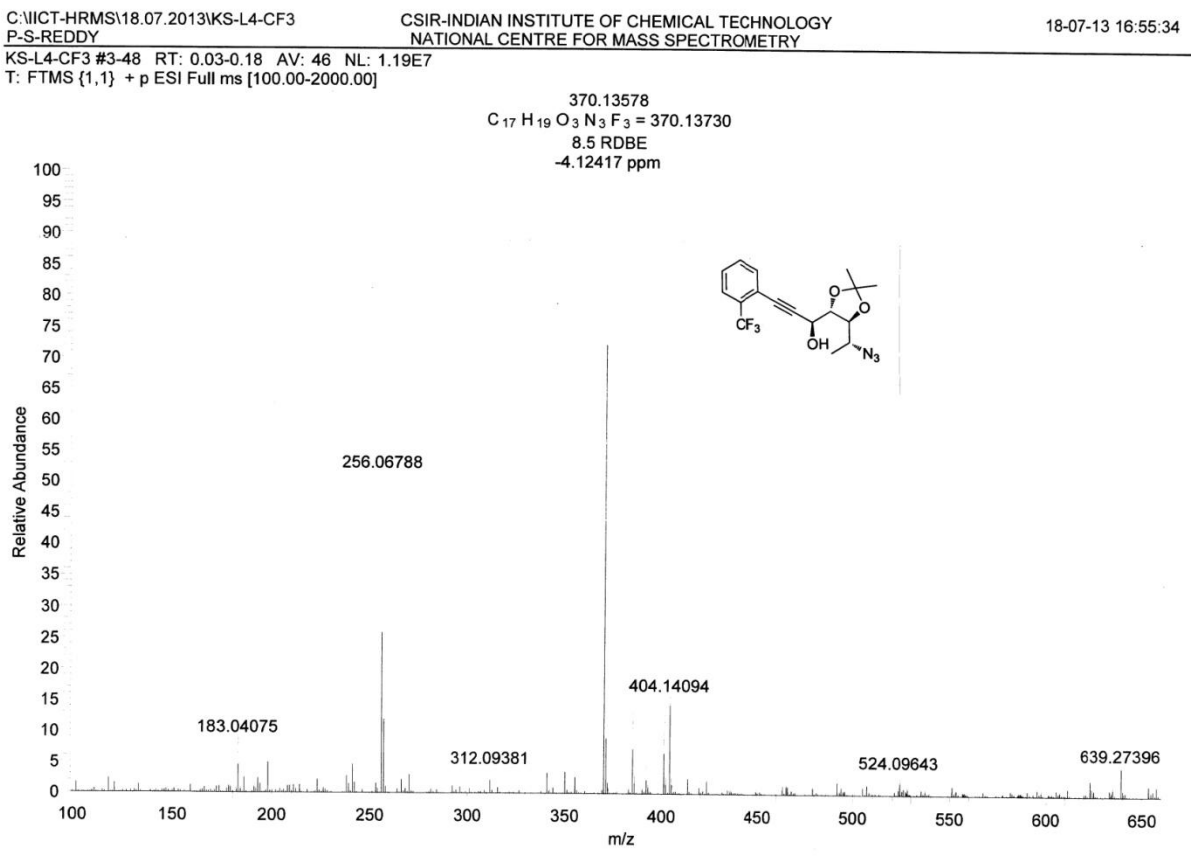
KS-L4-CF3 #3-48 RT: 0.03-0.18 AV: 46 NL: 1.19E7
T: FTMS {1,1} + p ESI Full ms [100.00-2000.00]

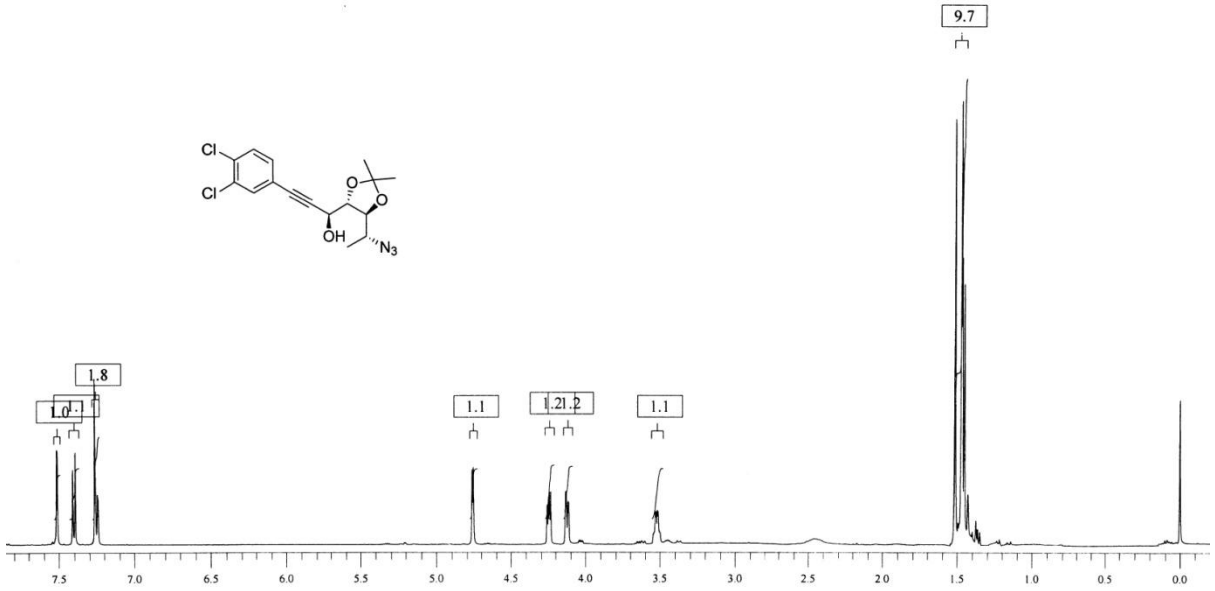
370.13578
$C_{17}H_{19}O_3N_3F_3$ = 370.13730
8.5 RDBE
-4.12417 ppm

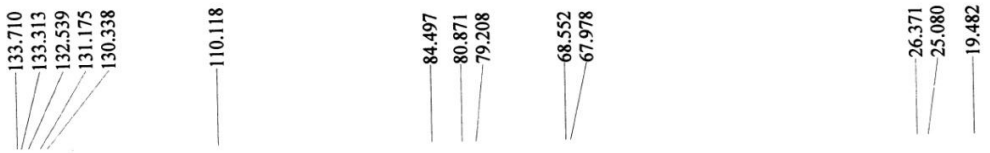


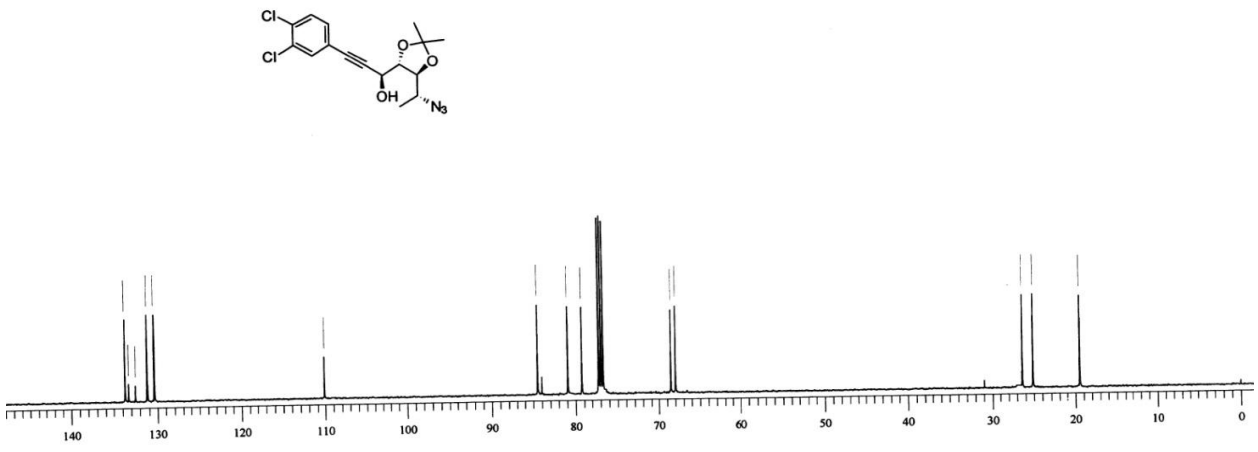


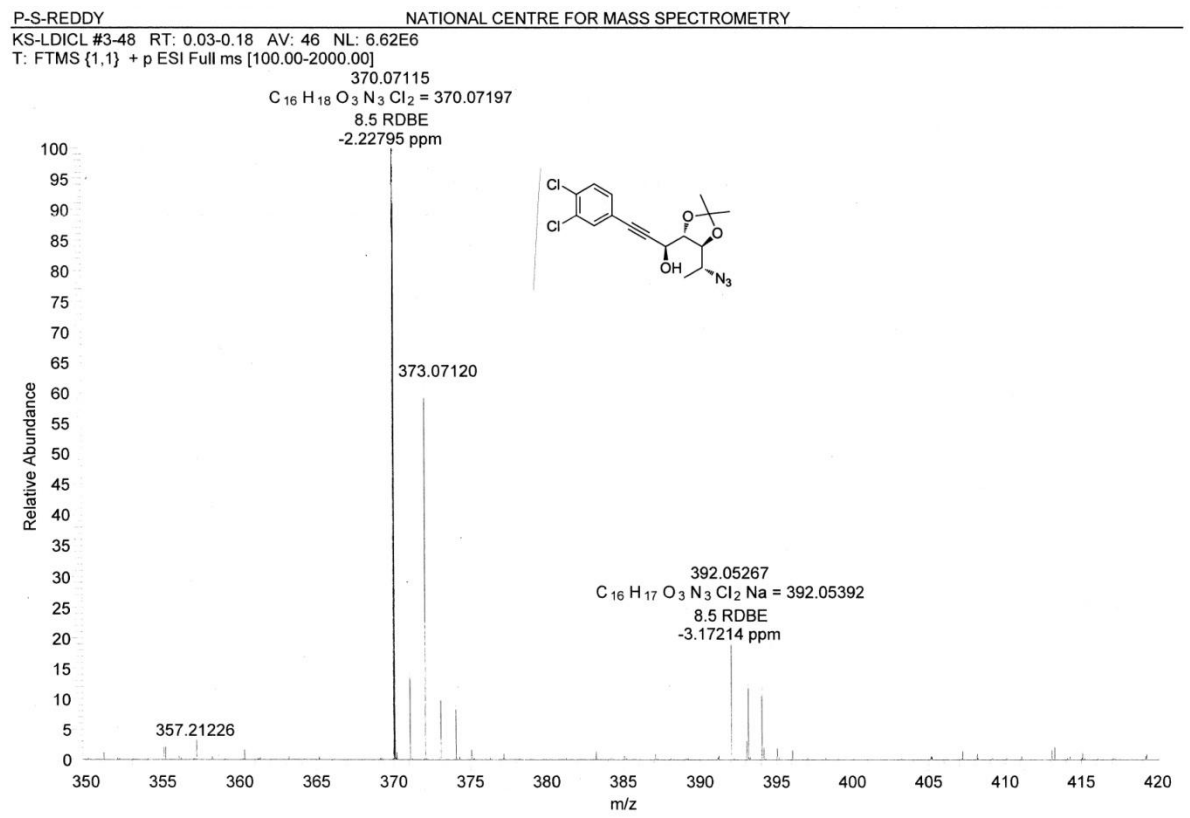

134.677
131.348
128.545
125.793
110.131
84.515
82.848
80.893
79.366
68.573
67.981
26.349
25.050
19.483
CF3
OH
N3
O
O
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0

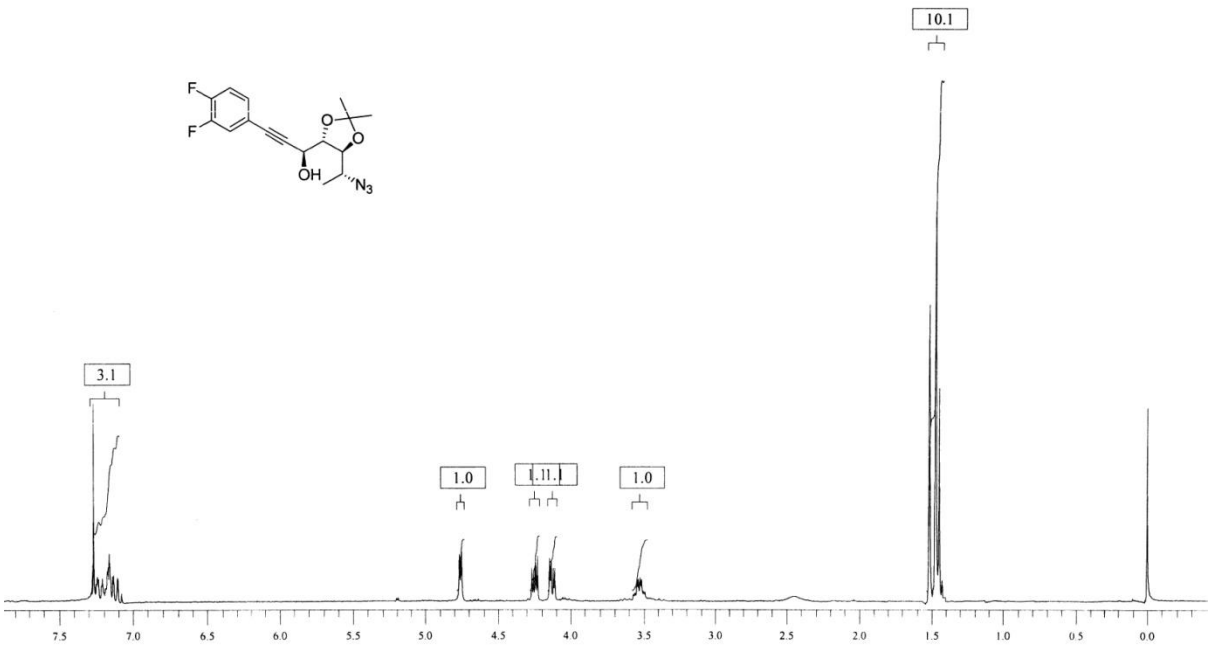

C:\IICT-HRMS\18.07.2013\KS-L4-CF3
P-S-REDDY
CSIR-INDIAN INSTITUTE OF CHEMICAL TECHNOLOGY
NATIONAL CENTRE FOR MASS SPECTROMETRY
18-07-13 16:55:34
KS-L4-CF3 #3-48 RT: 0.03-0.18 AV: 46 NL: 1.19E7
T: FTMS {1,1} + p ESI Full ms [100.00-2000.00]
370.13578
C17 H19 O3 N3 F3 = 370.13730
8.5 RDBE
-4.12417 ppm
Relative Abundance
m/z
256.06788
404.14094
183.04075
312.09381
524.09643
639.27396
CF3
OH
N3
O
O

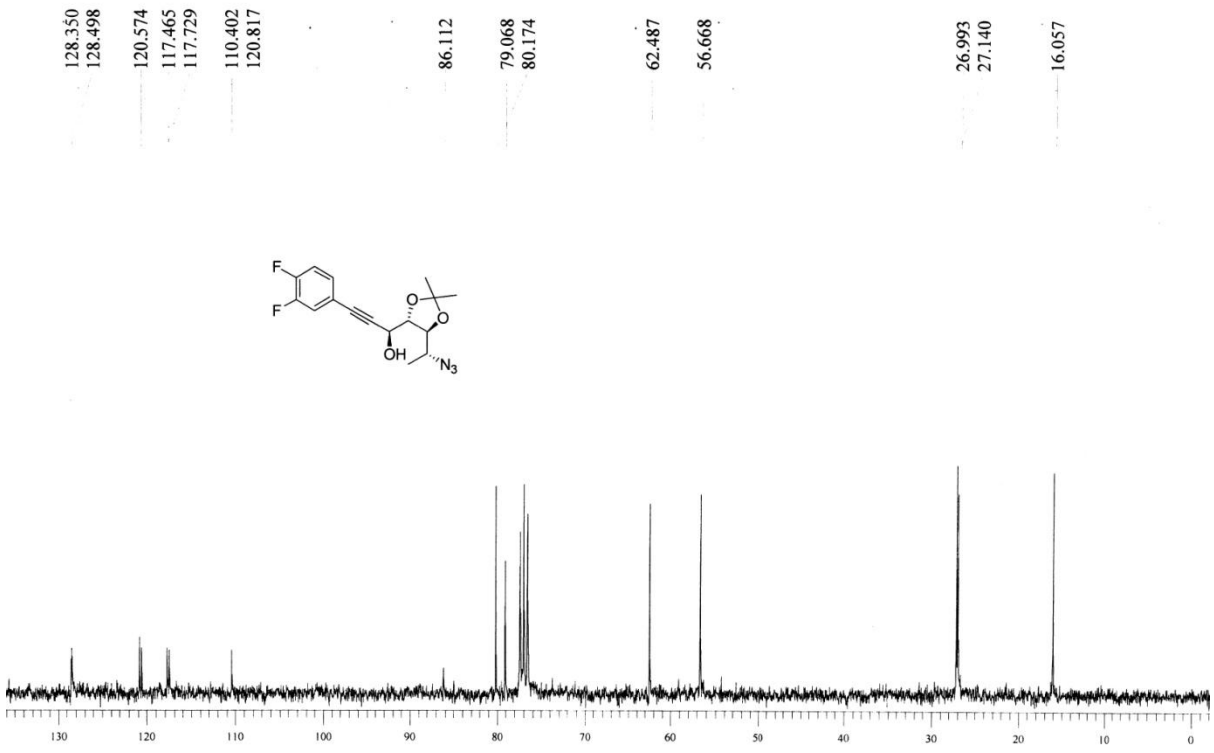

Cl
Cl
O
O
OH
N3
9.7
1.8
1.0
1.1
1.2
1.2
1.1
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0

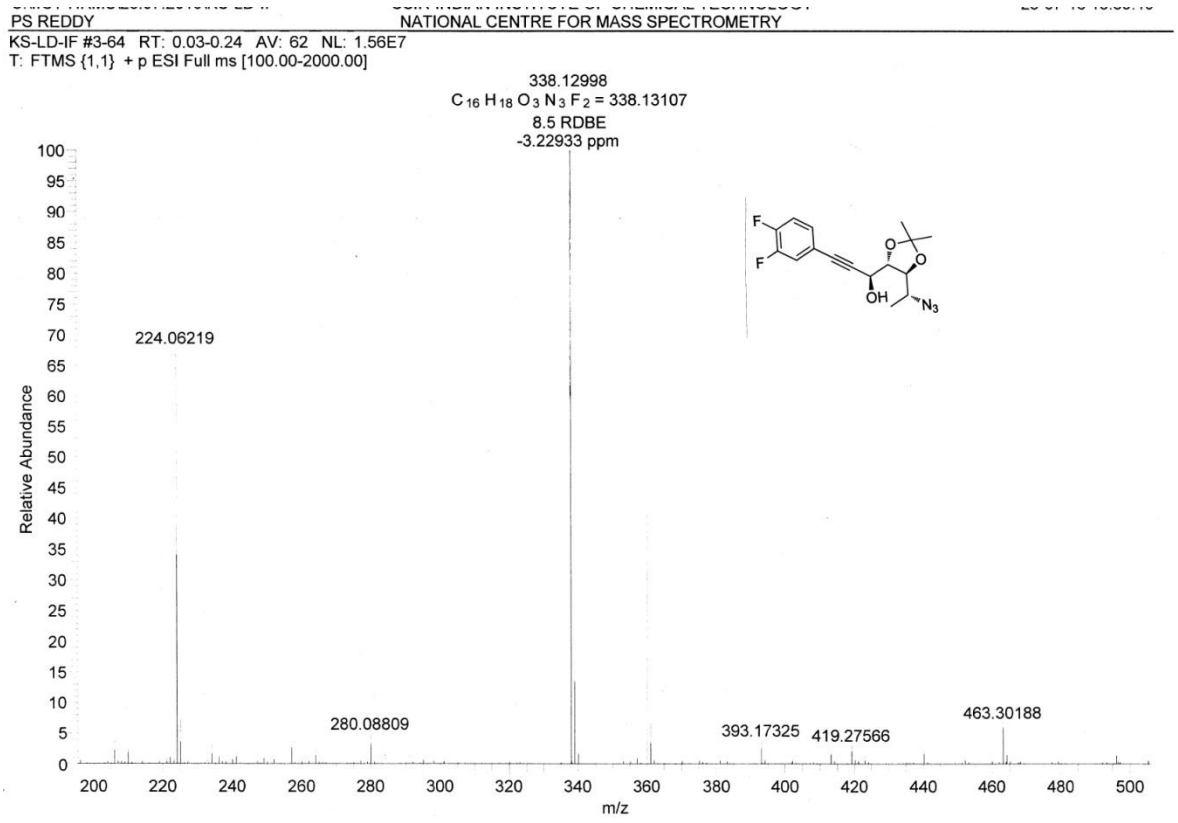

133.710
133.313
132.539
131.175
130.338
110.118
84.497
80.871
79.208
68.552
67.978
26.371
25.080
19.482

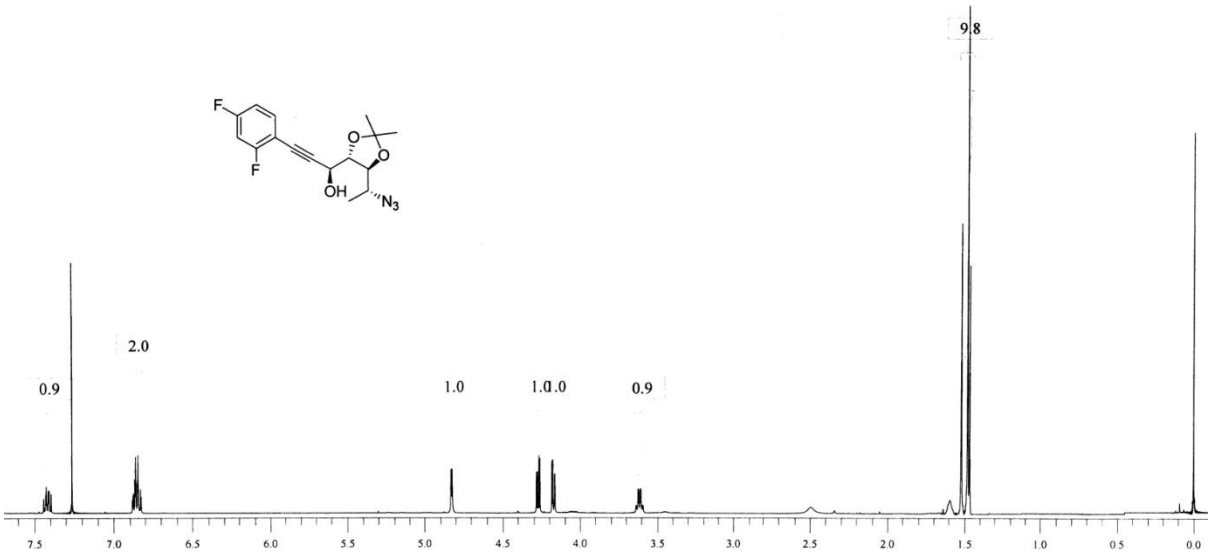

Cl
Cl
O
O
OH
N3
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0

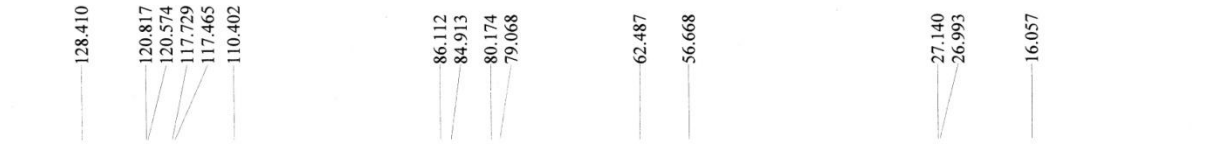

P-S-REDDY
NATIONAL CENTRE FOR MASS SPECTROMETRY
KS-LDICL #3-48 RT: 0.03-0.18 AV: 46 NL: 6.62E6
T: FTMS {1,1} + p ESI Full ms [100.00-2000.00]
370.07115
C 16 H 18 O 3 N 3 Cl 2 = 370.07197
8.5 RDBE
-2.22795 ppm
373.07120
392.05267
C 16 H 17 O 3 N 3 Cl 2 Na = 392.05392
8.5 RDBE
-3.17214 ppm
357.21226
Relative Abundance
m/z

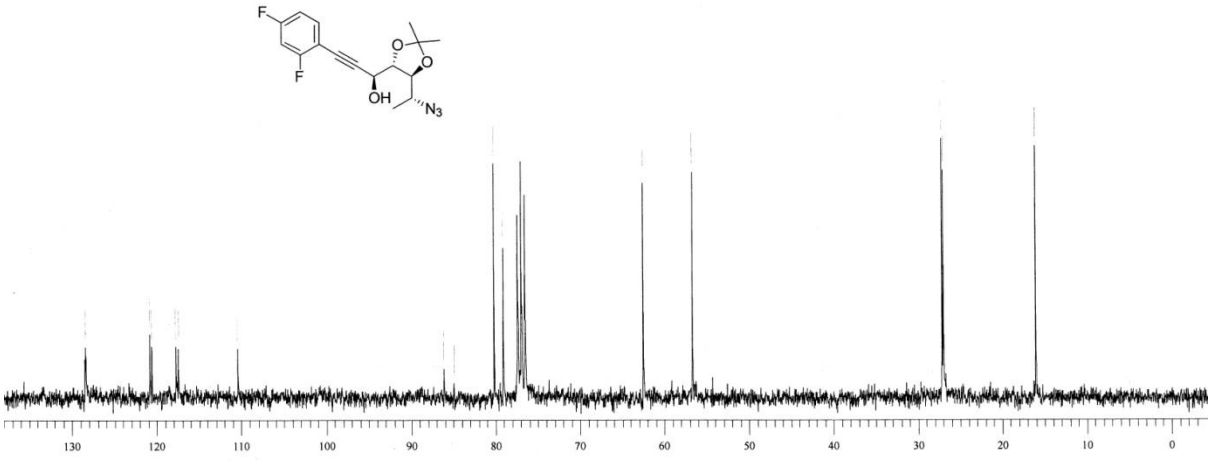

10.1
3.1
1.0
1.11
1.0

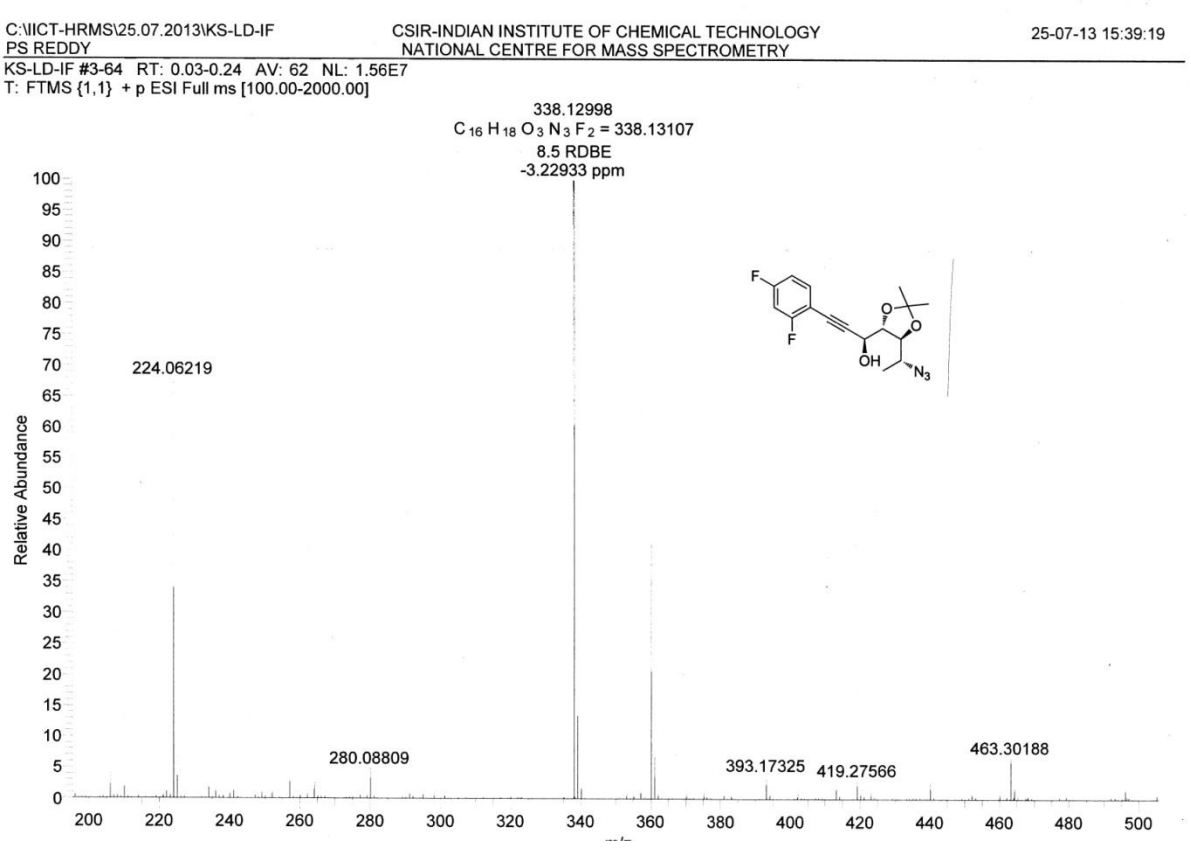

128.350
128.498
120.574
117.465
117.729
110.402
120.817
86.112
79.068
80.174
62.487
56.668
26.993
27.140
16.057
F
F
O
O
OH
N3
130
120
110
100
90
80
70
60
50
40
30
20
10
0

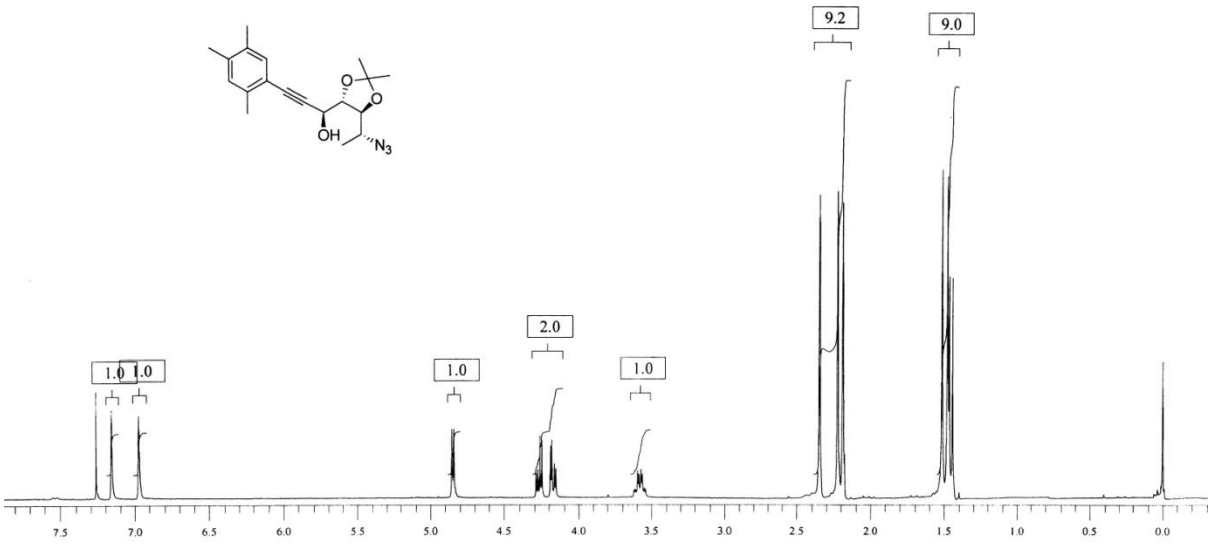

PS REDDY
NATIONAL CENTRE FOR MASS SPECTROMETRY
KS-LD-IF #3-64 RT: 0.03-0.24 AV: 62 NL: 1.56E7
T: FTMS {1,1} + p ESI Full ms [100.00-2000.00]
338.12998
C16 H18 O3 N3 F2 = 338.13107
8.5 RDBE
-3.22933 ppm
224.06219
280.08809
393.17325
419.27566
463.30188
Relative Abundance
m/z

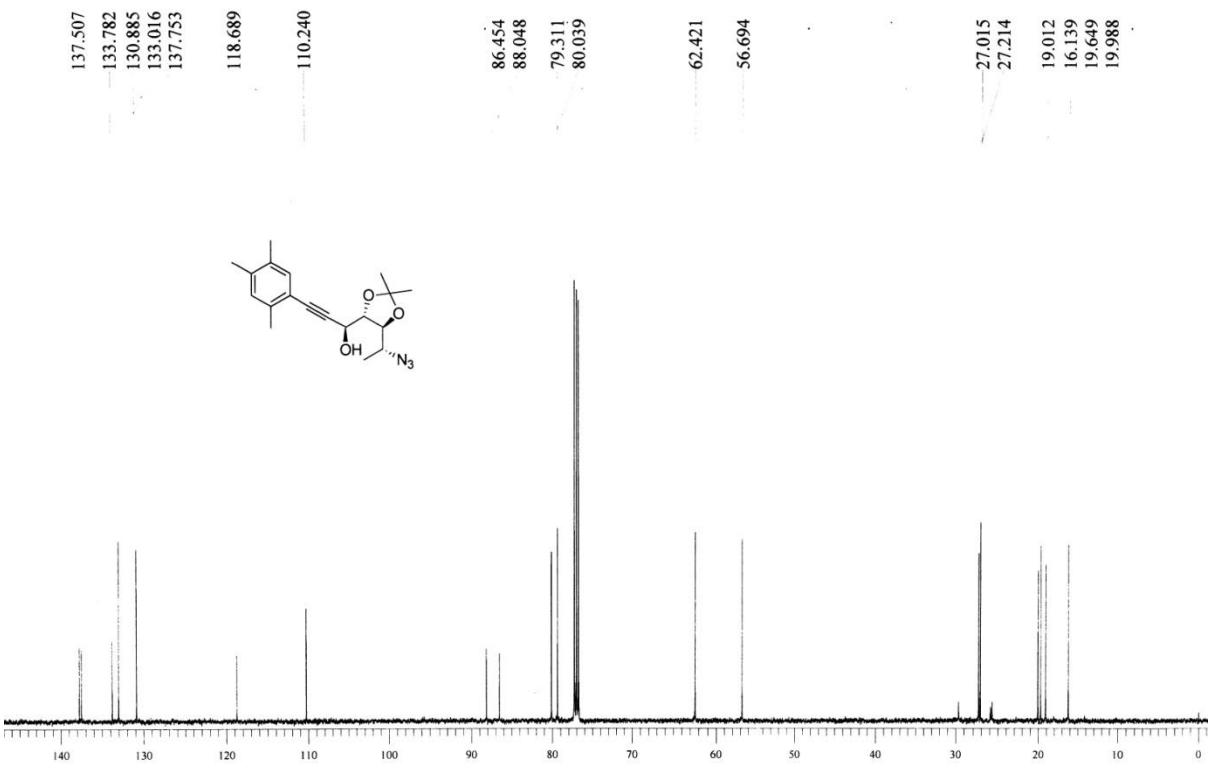

F
F
O
O
OH
N3
9.8
2.0
0.9
1.0
1.0.0
0.9
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0

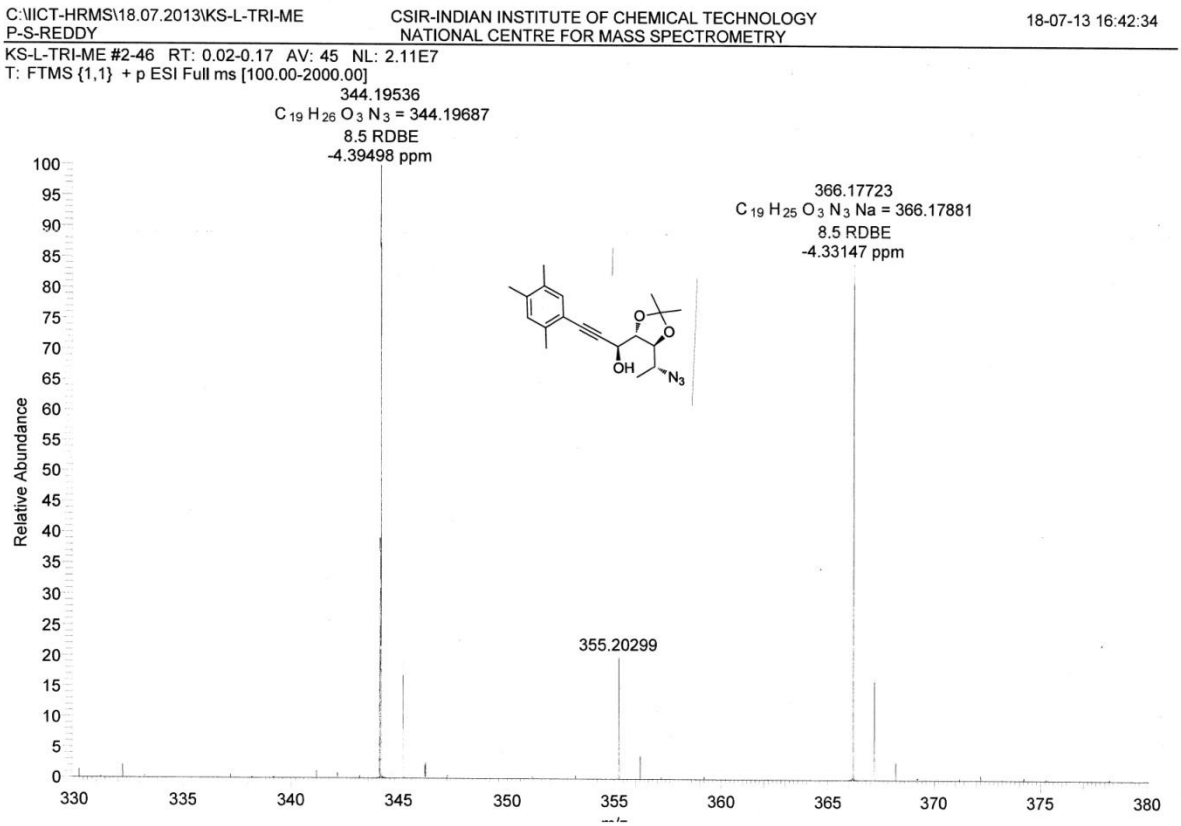

128.410
120.817
120.574
117.729
117.465
110.402
86.112
84.913
80.174
79.068
62.487
56.668
27.140
26.993
16.057

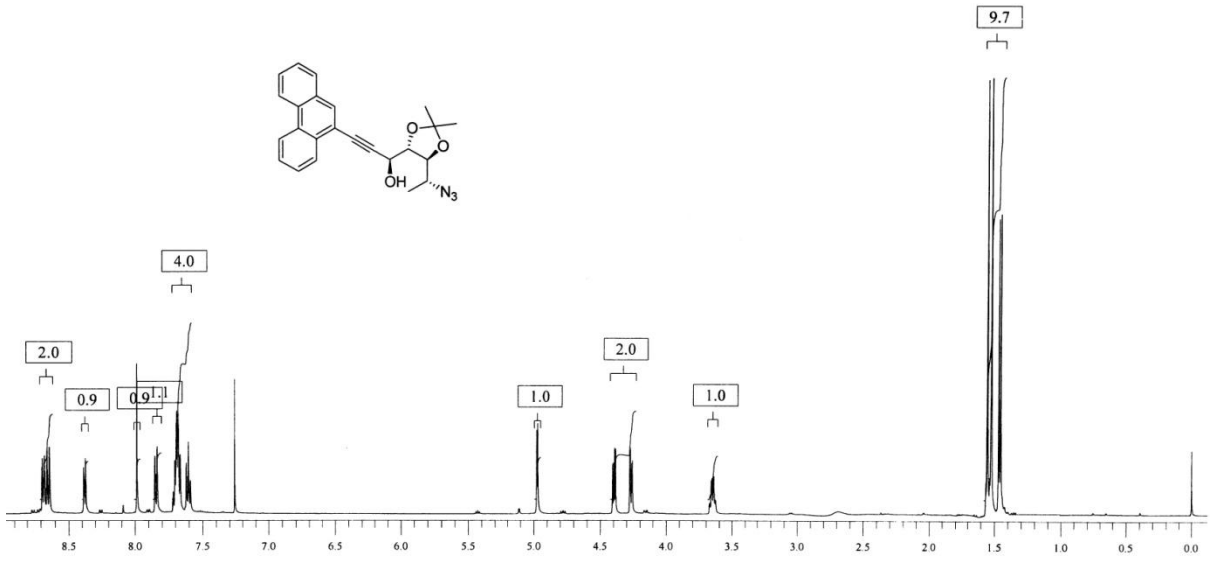

F
F
O
O
OH
N3
130
120
110
100
90
80
70
60
50
40
30
20
10
0

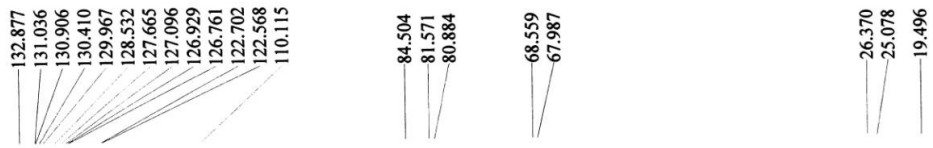

C:\IICT-HRMS\25.07.2013\KS-LD-IF
PS REDDY
CSIR-INDIAN INSTITUTE OF CHEMICAL TECHNOLOGY
NATIONAL CENTRE FOR MASS SPECTROMETRY
25-07-13 15:39:19
KS-LD-IF #3-64 RT: 0.03-0.24 AV: 62 NL: 1.56E7
T: FTMS {1,1} + p ESI Full ms [100.00-2000.00]
338.12998
C16 H18 O3 N3 F2 = 338.13107
8.5 RDBE
-3.22933 ppm
224.06219
280.08809
393.17325
419.27566
463.30188
Relative Abundance
F
F
O
O
OH
N3

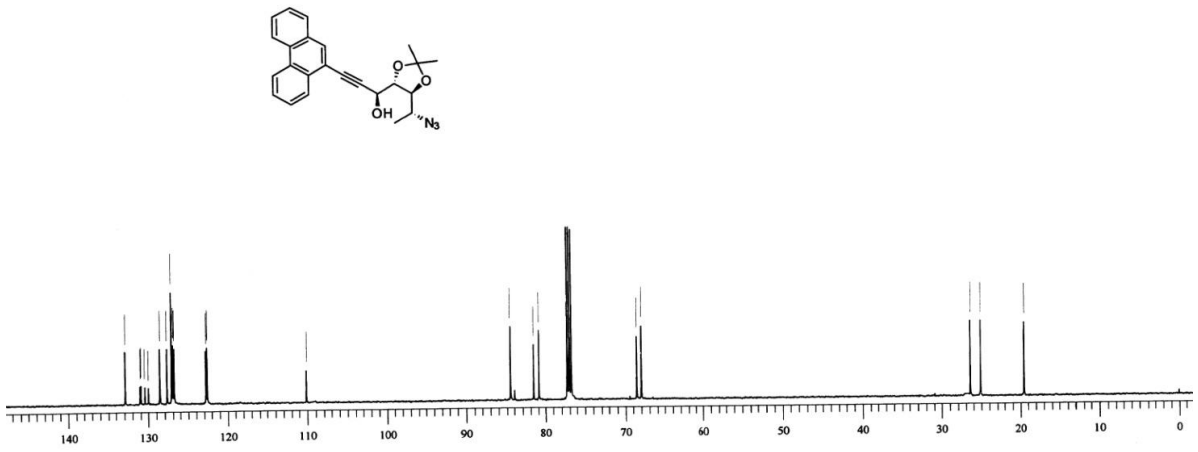

9.2
9.0
2.0
1.0
1.0
1.0
1.0
OH
N3


137.507
133.782
130.885
133.016
137.753
118.689
110.240
86.454
88.048
79.311
80.039
62.421
56.694
27.015
27.214
19.012
16.139
19.649
19.988
O
O
OH
N3
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0


C:\IICT-HRMS\18.07.2013\KS-L-TRI-ME P-S-REDDY
CSIR-INDIAN INSTITUTE OF CHEMICAL TECHNOLOGY
NATIONAL CENTRE FOR MASS SPECTROMETRY
18-07-13 16:42:34
KS-L-TRI-ME #2-46 RT: 0.02-0.17 AV: 45 NL: 2.11E7
T: FTMS {1,1} + p ESI Full ms [100.00-2000.00]
344.19536
C 19 H 26 O 3 N 3 = 344.19687
8.5 RDBE
-4.39498 ppm
366.17723
C 19 H 25 O 3 N 3 Na = 366.17881
8.5 RDBE
-4.33147 ppm
355.20299
Relative Abundance
O
O
OH
N3
100
95
90
85
80
75
70
65
60
55
50
45
40
35
30
25
20
15
10
5
0
330
335
340
345
350
355
360
365
370
375
380

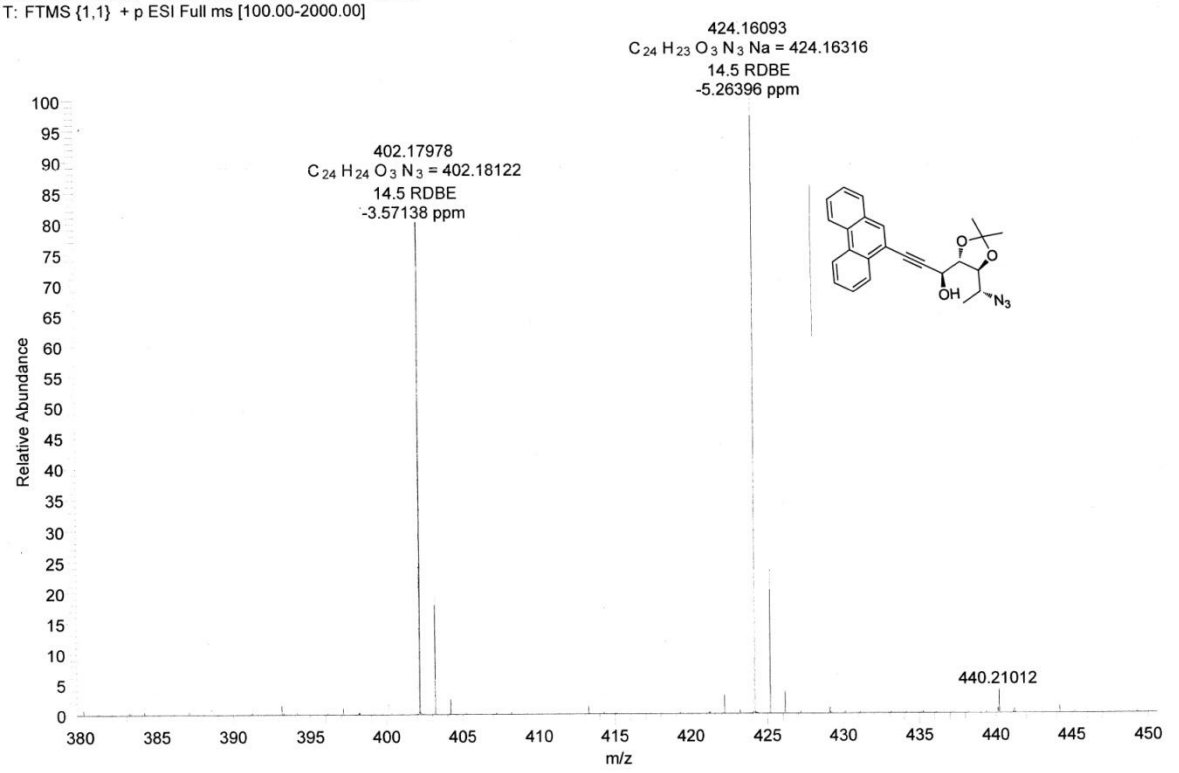

9.7
4.0
2.0
0.9
0.9
1.1
1.0
2.0
1.0
OH
N3
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0

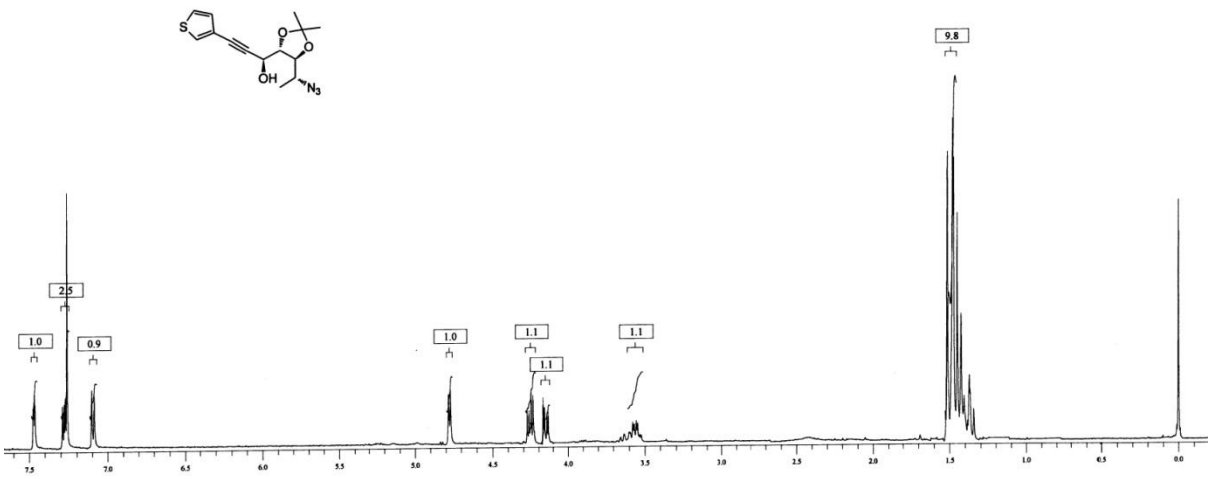

132.877
131.036
130.906
130.410
129.967
128.532
127.665
127.096
126.929
126.761
122.702
122.568
110.115
84.504
81.571
80.884
68.559
67.987
26.370
25.078
19.496

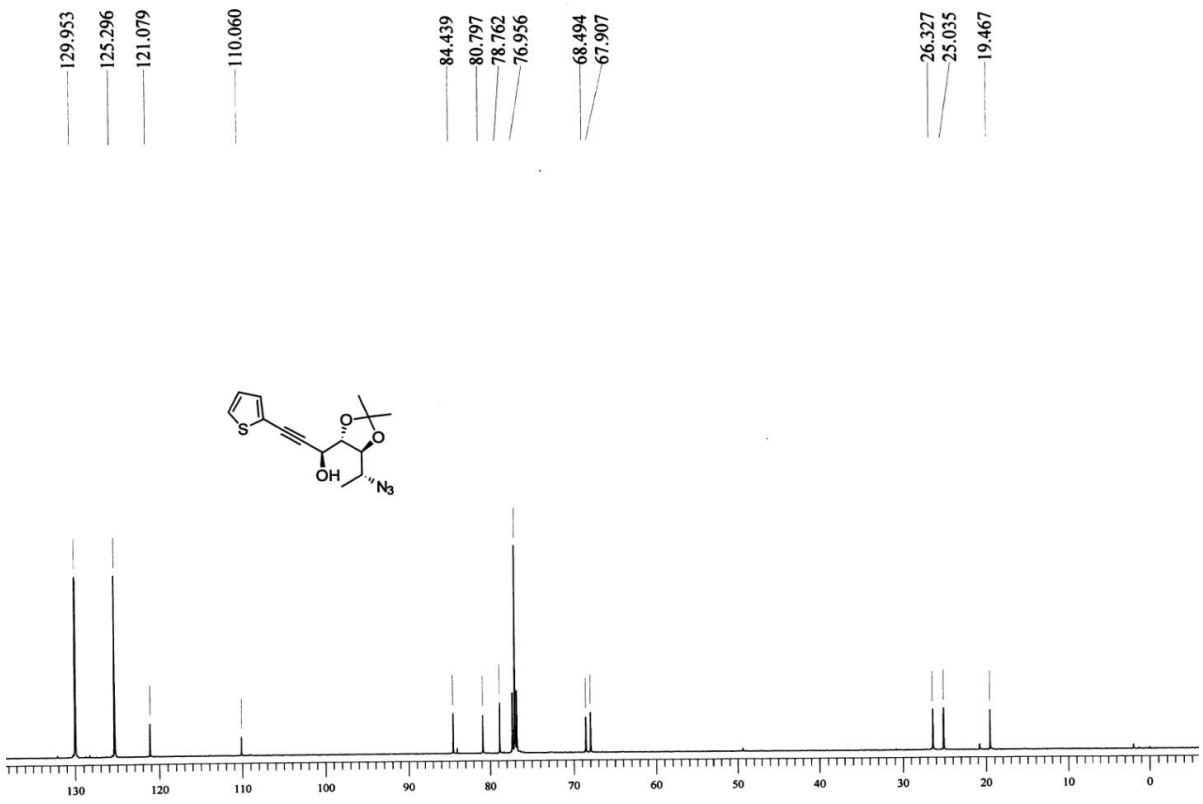

OH
N3
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0

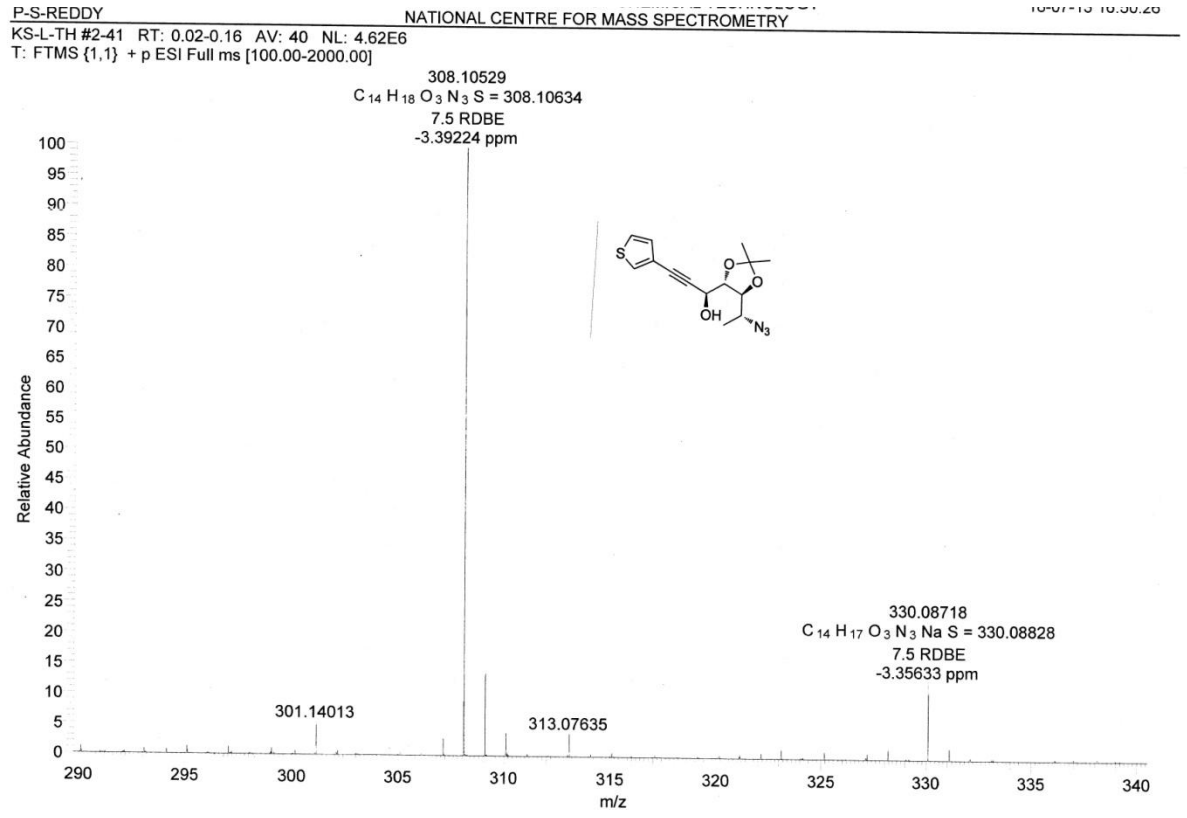
P-S-REDDY NATIONAL CENTRE FOR MASS SPECTROMETRY

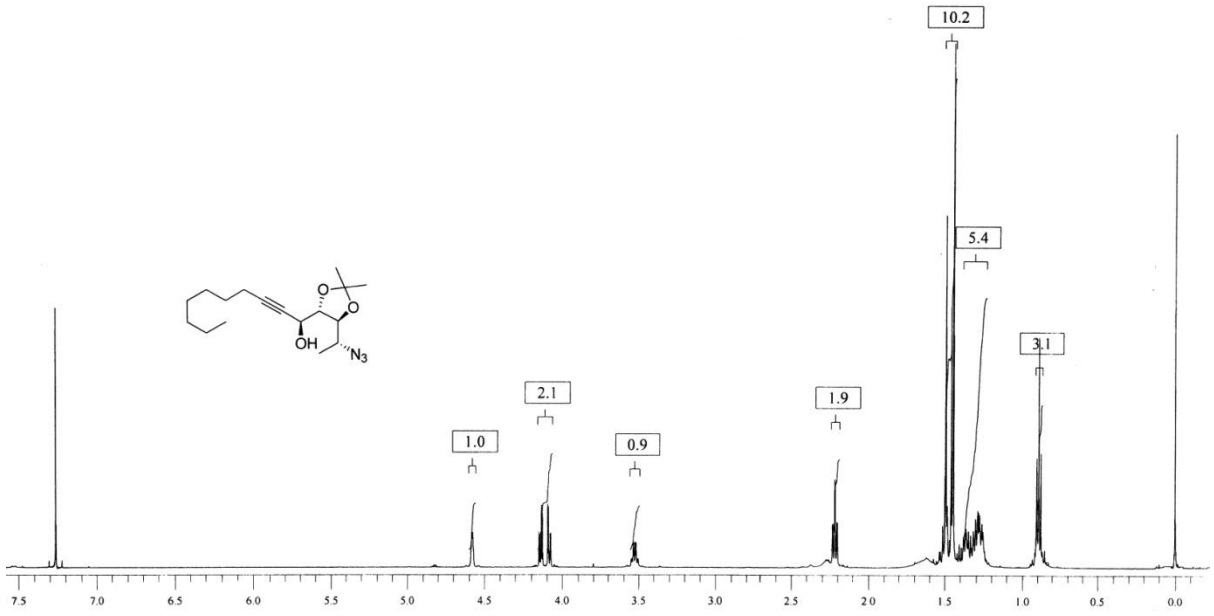
KS-L-ANTH #2-46 RT: 0.02-0.17 AV: 45 NL: 1.51E7
T: FTMS {1,1} + p ESI Full ms [100.00-2000.00]

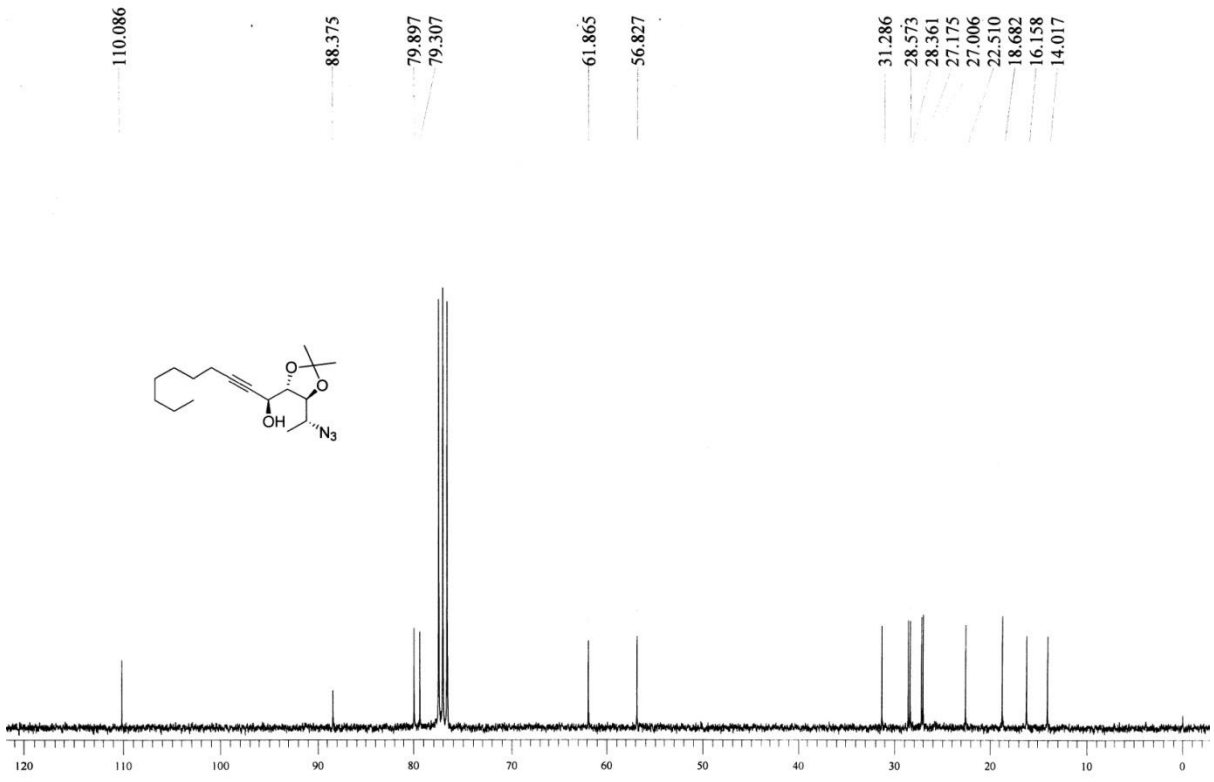


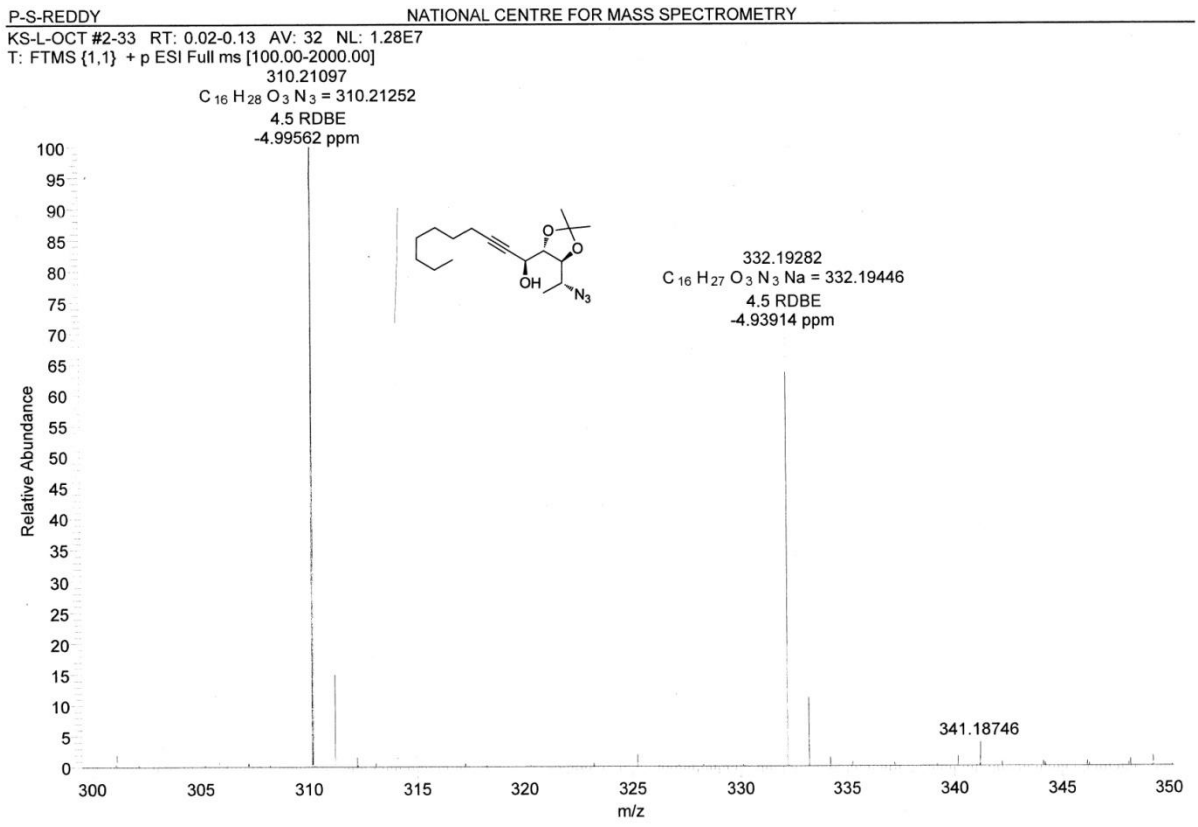


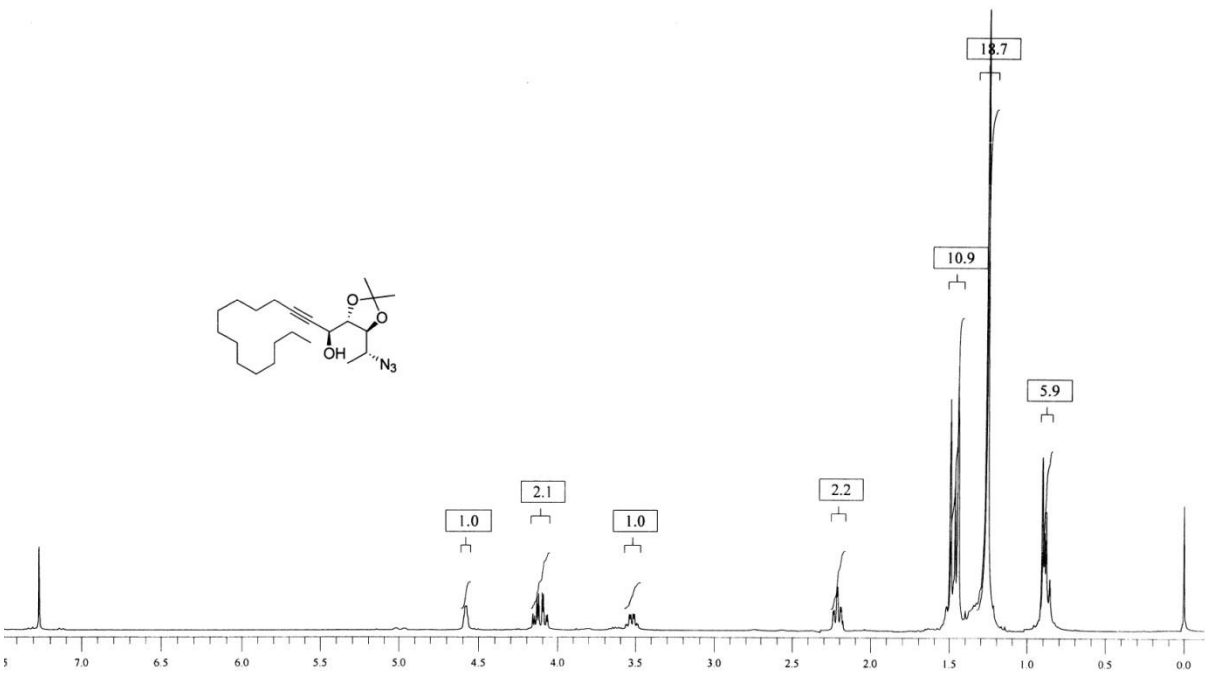

129.953
125.296
121.079
110.060
84.439
80.797
78.762
76.956
68.494
67.907
26.327
25.035
19.467
S
O
O
OH
N3
130
120
110
100
90
80
70
60
50
40
30
20
10
0

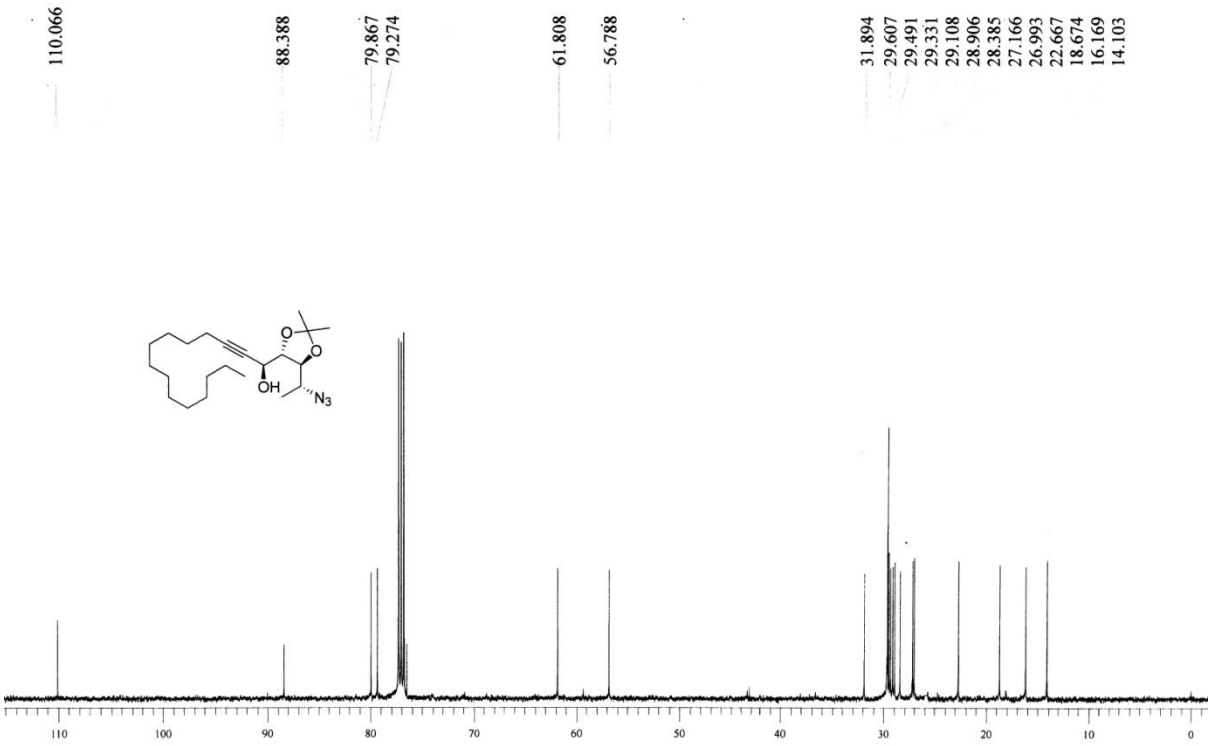

P-S-REDDY
NATIONAL CENTRE FOR MASS SPECTROMETRY
KS-L-TH #2-41 RT: 0.02-0.16 AV: 40 NL: 4.62E6
T: FTMS {1,1} + p ESI Full ms [100.00-2000.00]
308.10529
C 14 H 18 O 3 N 3 S = 308.10634
7.5 RDBE
-3.39224 ppm
330.08718
C 14 H 17 O 3 N 3 Na S = 330.08828
7.5 RDBE
-3.35633 ppm
301.14013
313.07635
Relative Abundance
m/z
S
O
O
OH
N3

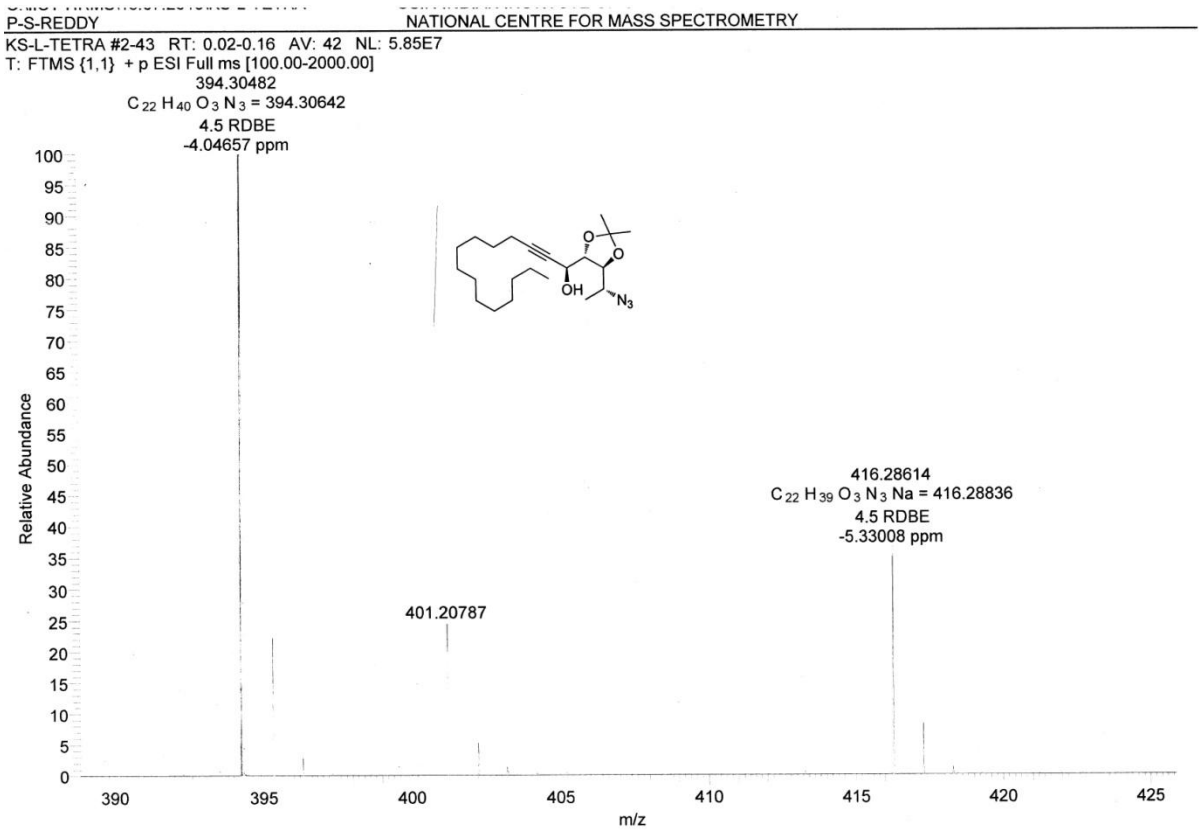

10.2
5.4
3.1
2.1
1.9
1.0
0.9
OH
N3
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0

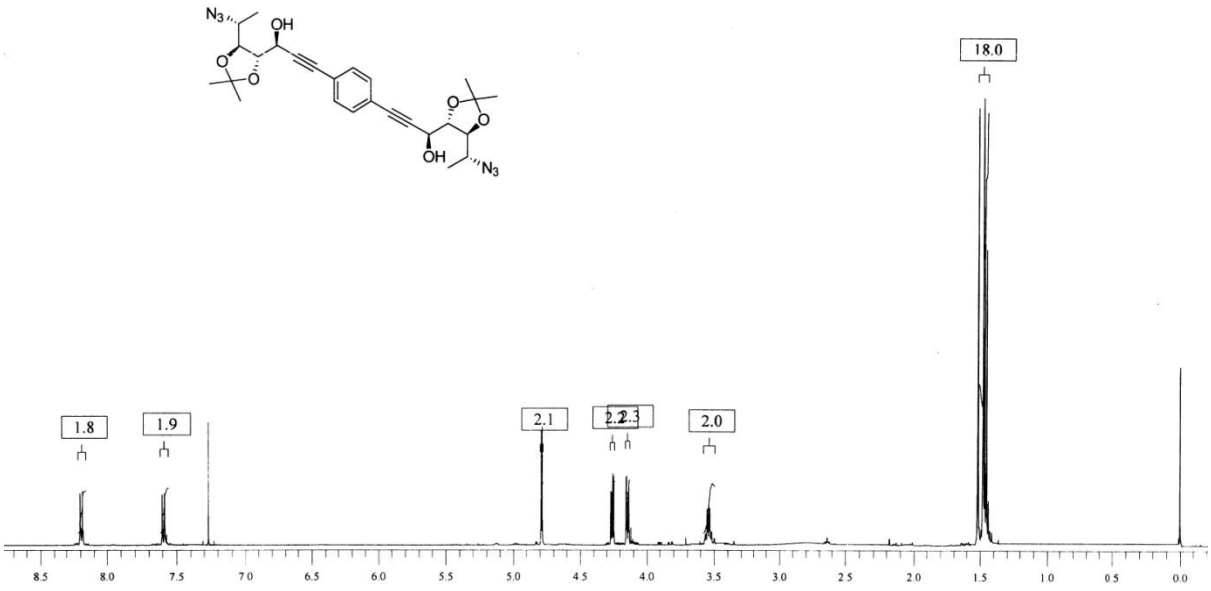

110.086
88.375
79.897
79.307
61.865
56.827
31.286
28.573
28.361
27.175
27.006
22.510
18.682
16.158
14.017
OH
N3
120
110
100
90
80
70
60
50
40
30
20
10
0

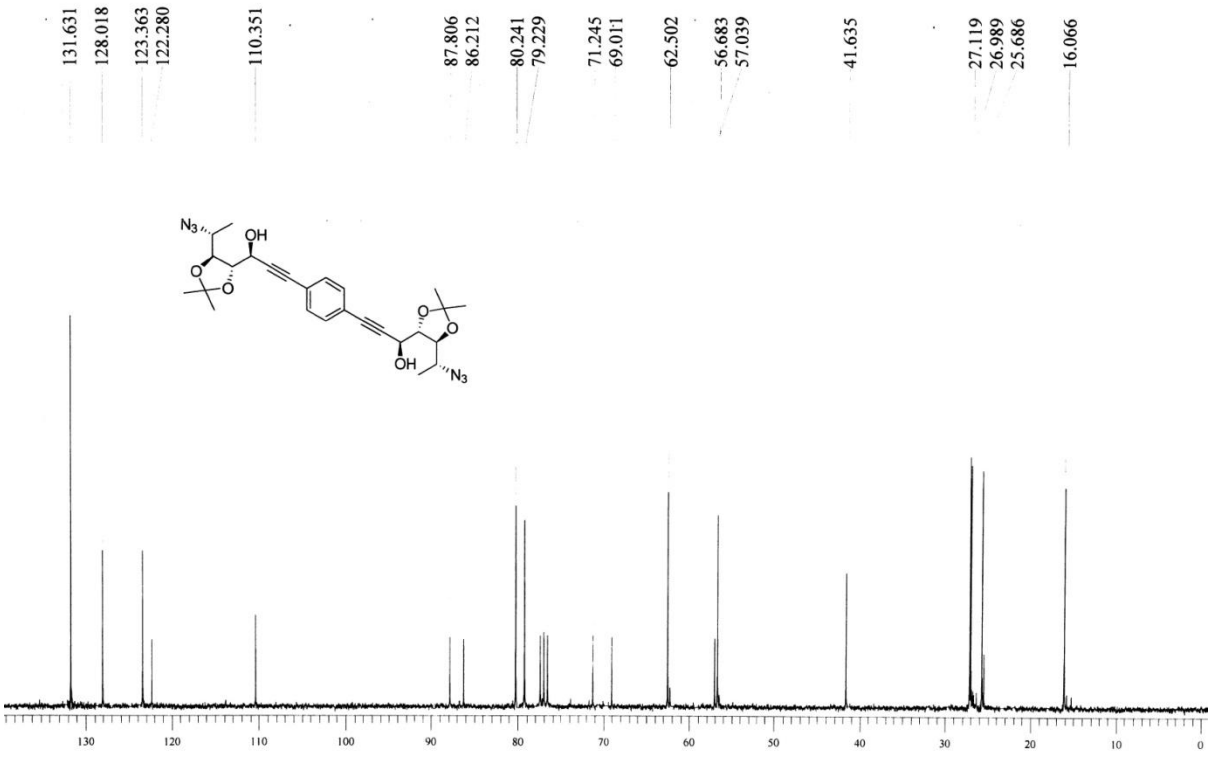

P-S-REDDY
NATIONAL CENTRE FOR MASS SPECTROMETRY
KS-L-OCT #2-33 RT: 0.02-0.13 AV: 32 NL: 1.28E7
T: FTMS {1,1} + p ESI Full ms [100.00-2000.00]
310.21097
C 16 H 28 O 3 N 3 = 310.21252
4.5 RDBE
-4.99562 ppm
332.19282
C 16 H 27 O 3 N 3 Na = 332.19446
4.5 RDBE
-4.93914 ppm
341.18746
Relative Abundance
m/z


18.7
10.9
5.9
2.2
2.1
1.0
1.0


110.066
88.388
79.867
79.274
61.808
56.788
31.894
29.607
29.491
29.331
29.108
28.906
28.385
27.166
26.993
22.667
18.674
16.169
14.103
O
O
OH
N3
110
100
90
80
70
60
50
40
30
20
10
0

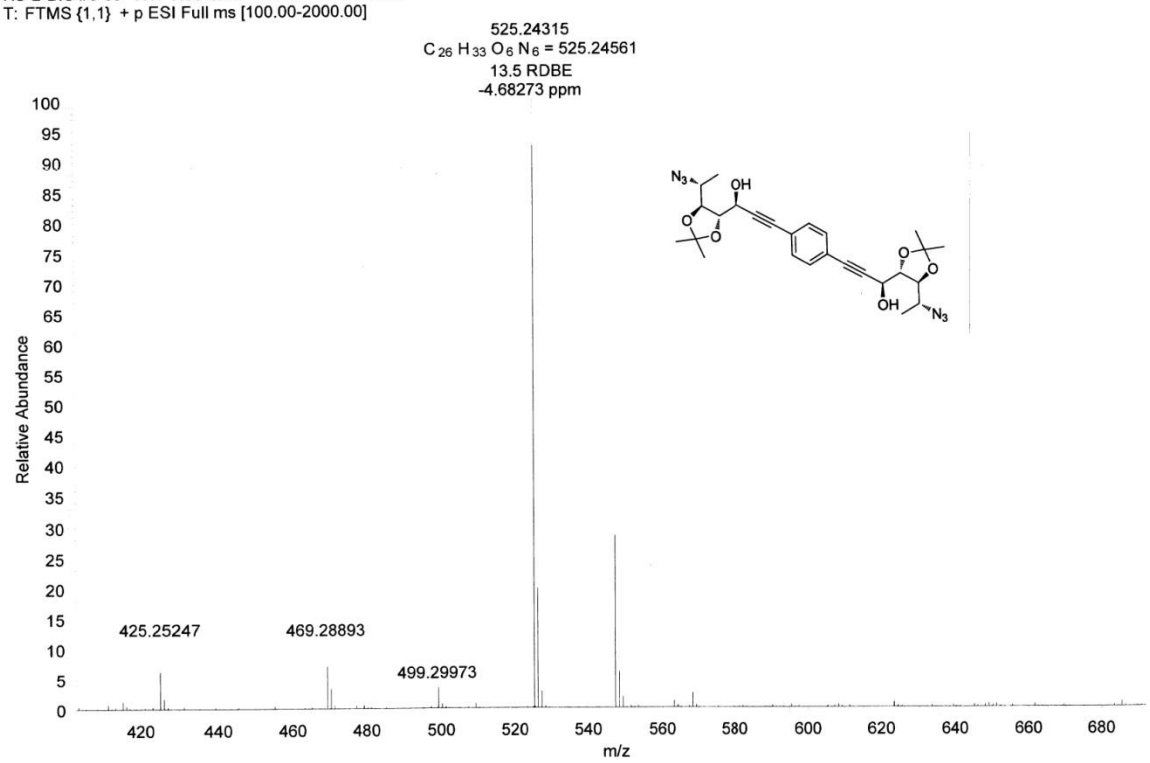

P-S-REDDY
NATIONAL CENTRE FOR MASS SPECTROMETRY
KS-L-TETRA #2-43 RT: 0.02-0.16 AV: 42 NL: 5.85E7
T: FTMS {1,1} + p ESI Full ms [100.00-2000.00]
394.30482
C22 H40 O3 N3 = 394.30642
4.5 RDBE
-4.04657 ppm
O
O
OH
N3
416.28614
C22 H39 O3 N3 Na = 416.28836
4.5 RDBE
-5.33008 ppm
401.20787
Relative Abundance
m/z
100
95
90
85
80
75
70
65
60
55
50
45
40
35
30
25
20
15
10
5
0
390
395
400
405
410
415
420
425

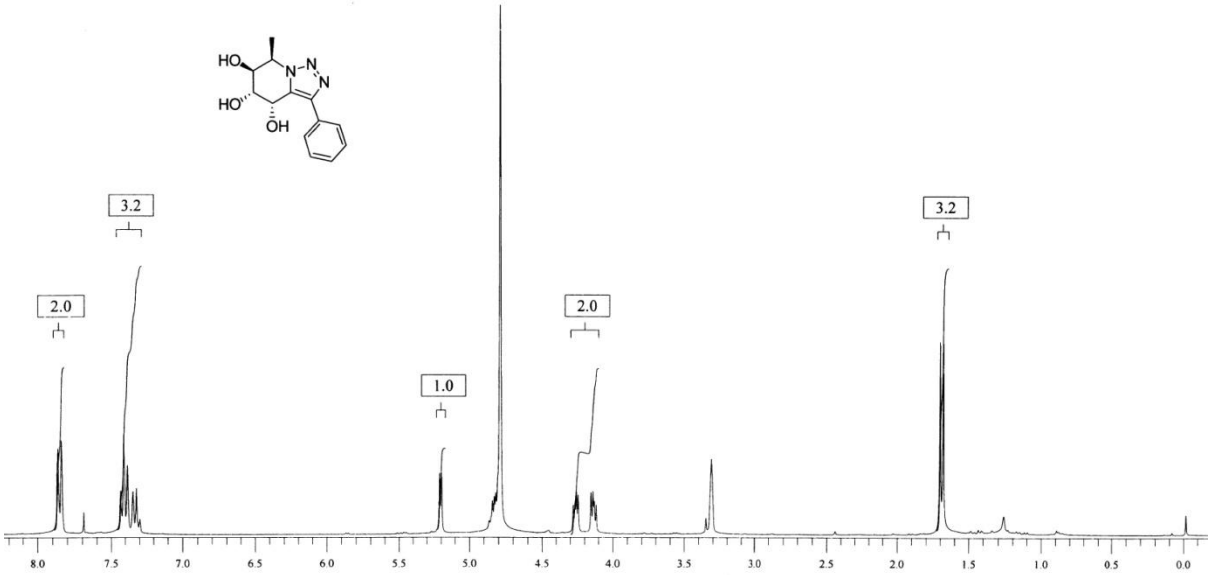

18.0
1.8
1.9
2.1
2.2
2.3
2.0

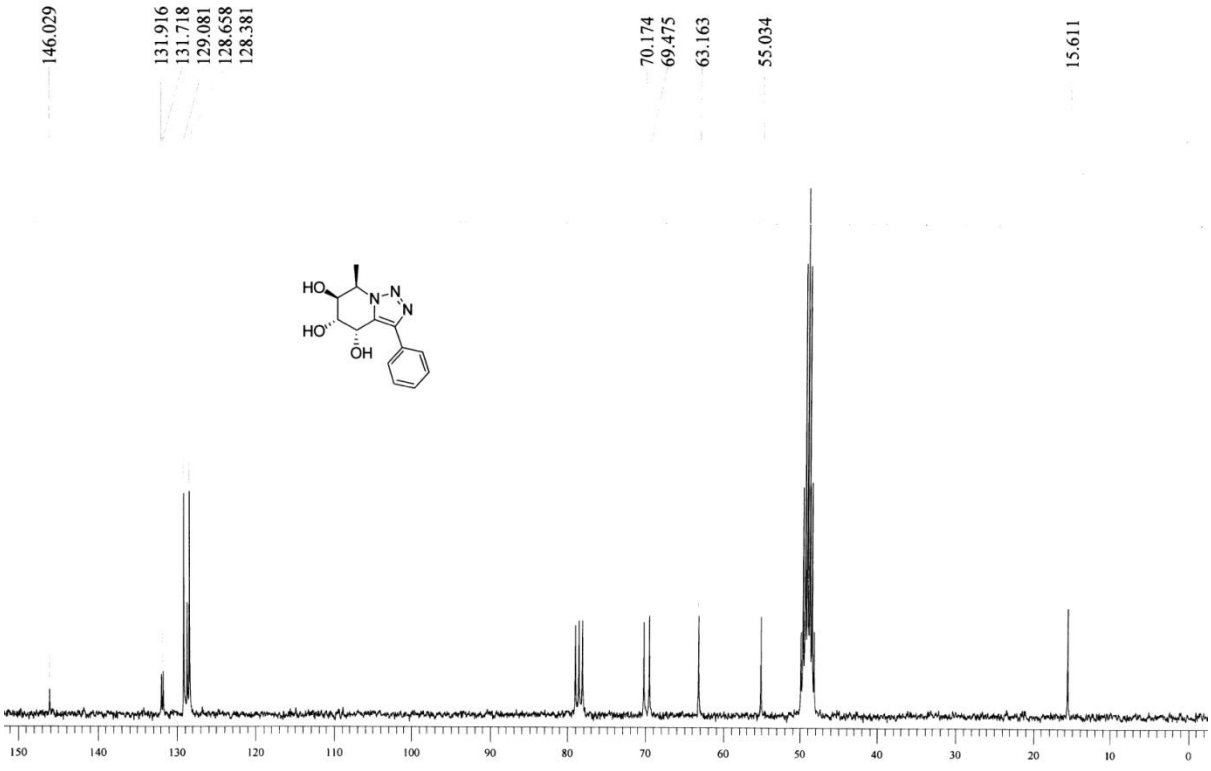

131.631
128.018
123.363
122.280
110.351
87.806
86.212
80.241
79.229
71.245
69.011
62.502
56.683
57.039
41.635
27.119
26.989
25.686
16.066

C:\IICT-HRMS\18.07.2013\KS-L-BIS

KS-L-BIS #3-68 RT: 0.03-0.25 AV: 66 NL: 8.32E6
T: FTMS {1,1} + p ESI Full ms [100.00-2000.00]

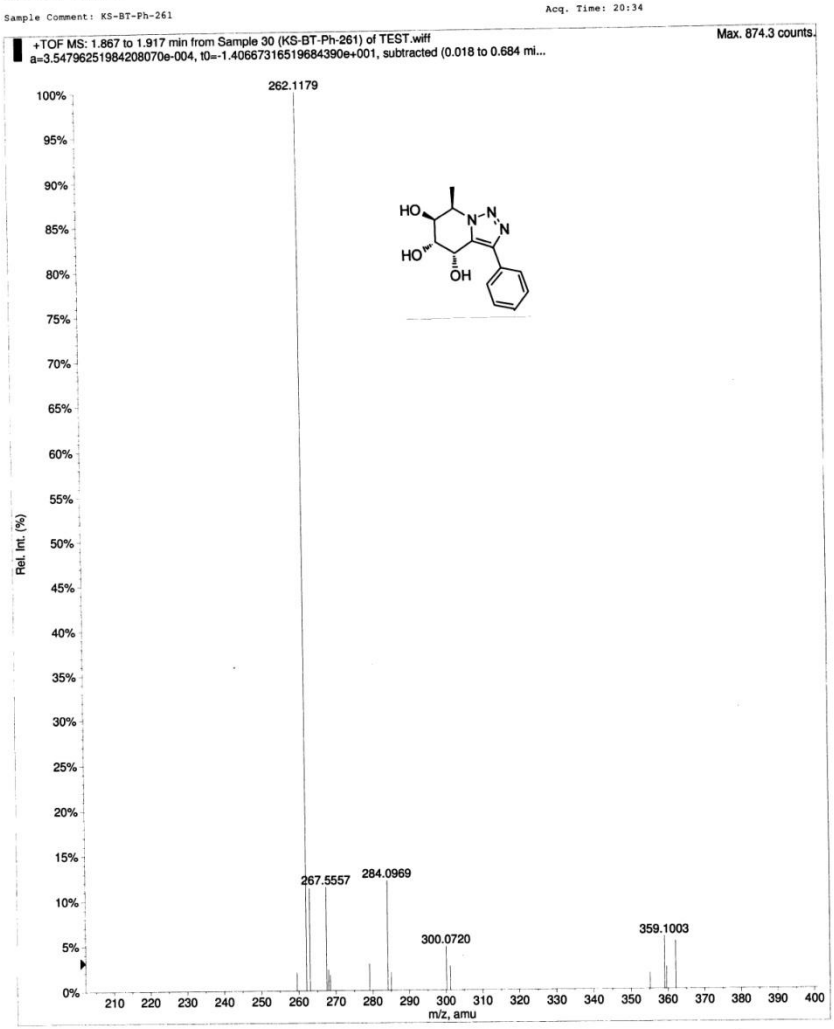



HO
HO
OH
N
N
N
3.2
2.0
1.0
2.0
3.2
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0


146.029
131.916
131.718
129.081
128.658
128.381
70.174
69.475
63.163
55.034
15.611
HO
HO
OH
N
N
N
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0

Acq. File: TEST.wiff Sample ID: TuneSampleID Acq. Date: Thursday, June 20, 2013

Sample Comment: KS-BT-Ph-261 Acq. Time: 20:34



Acq. File: n/a Sample ID: n/a Acq. Date: n/a

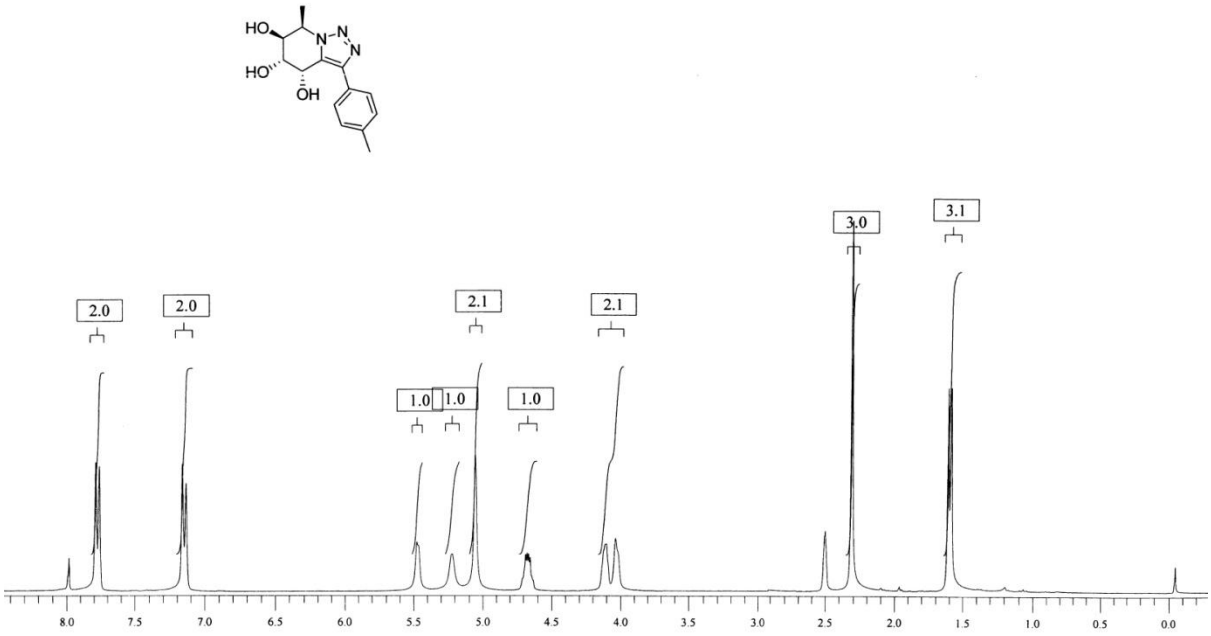
Sample Comment: n/a Acq. Time: n/a

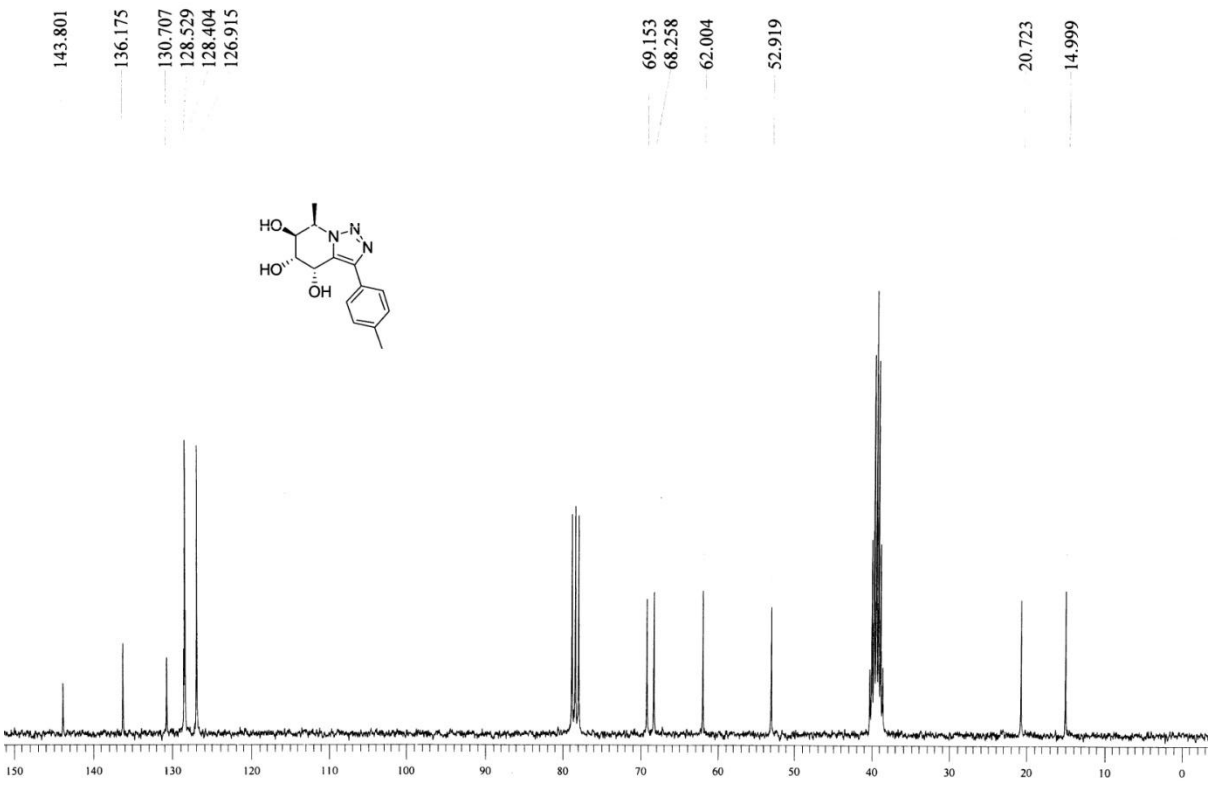
Elemental composition calculator

Target m/z: +262.1179 amu
Tolerance: +5.0000 ppm
Result type: Elemental
Max num of results: 100
Min DBE: -0.5000 Max DBE: +50.0000
Electron state: OddAndEven
Num of charges: 0
Add water: N/A
Add proton: N/A
File Name: TEST.wiff

	Elements	Min Number	Max Number
1	C	0	13
2	H	0	16
3	N	0	3
4	Na	0	1
5	O	0	3

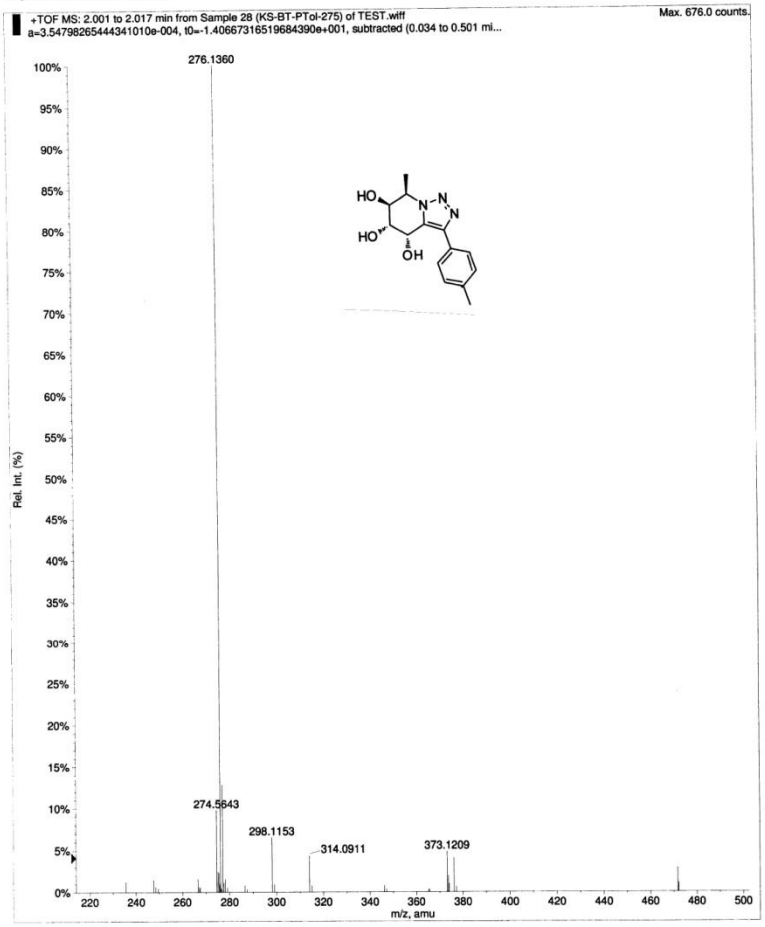
	Formula	Calculated m/z (amu)	mDa Error	PPM Error	DBE
1	C13 H16 N3 O3	262.1191	-1.2665	-4.8321	7.5


HO
HO
OH
N
N
N
2.0
2.0
2.1
2.1
3.0
3.1
1.0
1.0
1.0
8.0
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
1.0
0.5
0.0


143.801
136.175
130.707
128.529
128.404
126.915
69.153
68.258
62.004
52.919
20.723
14.999
HO
HO
OH
N
N
N
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0

Acq. File: TEST.wiff Sample ID: TuneSampleID Acq. Date: Thursday, June 20, 2013

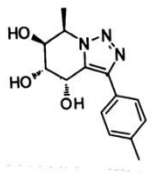
Sample Comment: KS-BT-PTol-275 Acq. Time: 20:23



Acq. File: n/a Sample ID: n/a Acq. Date: n/a

Sample Comment: n/a Acq. Time: n/a

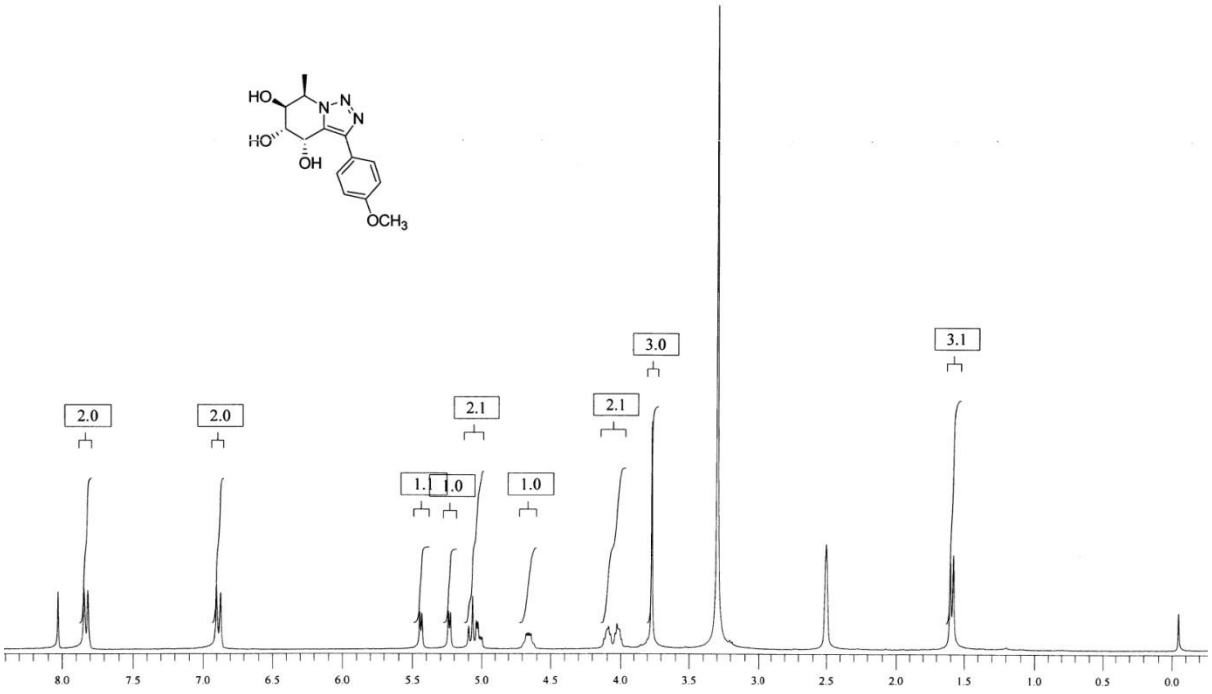
Elemental composition calculator

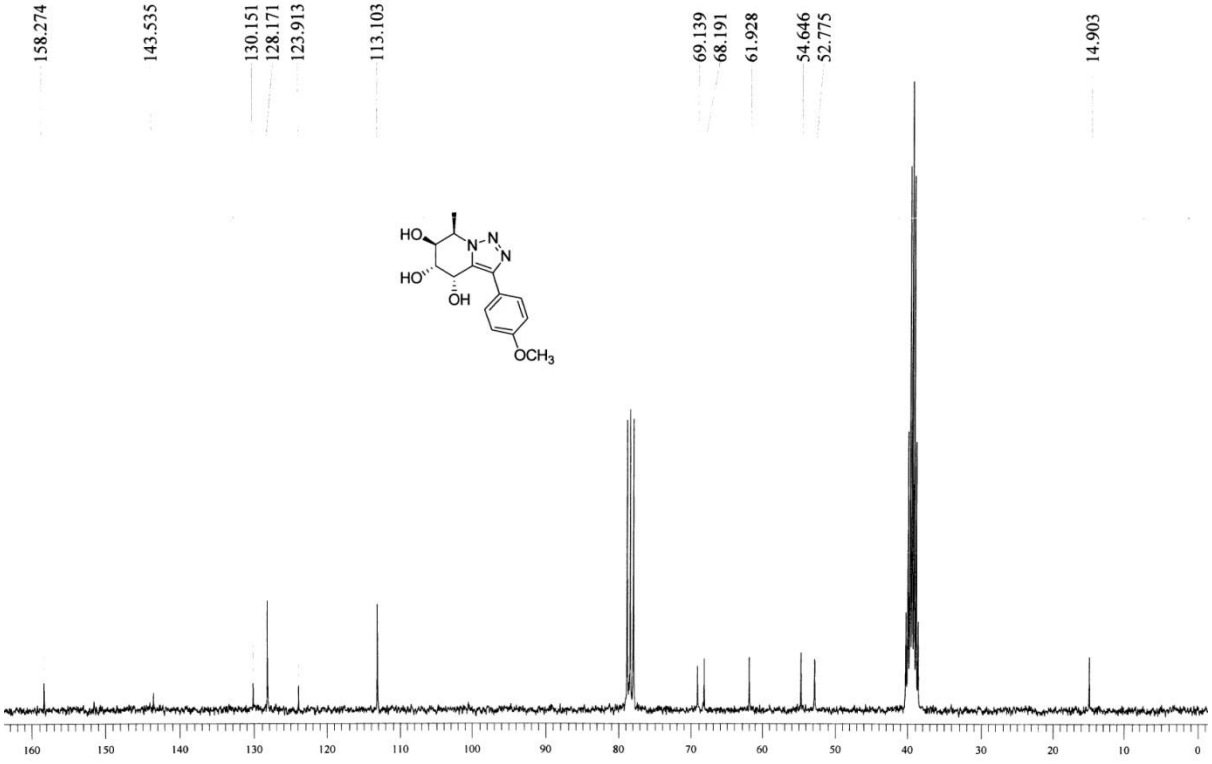


Target m/z: +276.1360 amu
Tolerance: +5.0000 ppm
Result type: Elemental
Max num of results: 100
Min DBE: -0.5000 Max DBE: +50.0000
Electron state: OddAndEven
Num of charges: 0
Add water: N/A
Add proton: N/A
File Name: TEST.wiff

	Elements	Min Number	Max Number
1	C	0	14
2	H	0	18
3	N	0	3
4	O	0	3

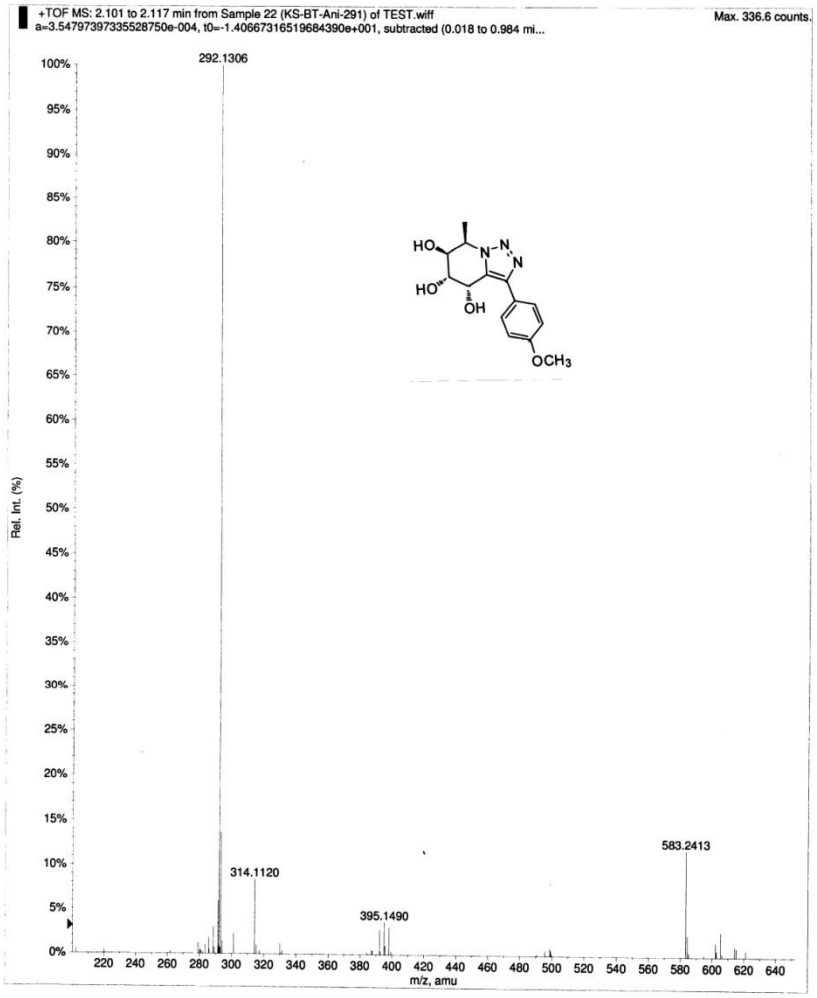
	Formula	Calculated m/z (amu)	mDa Error	PPM Error	DBE
1	C14 H18 N3 O3	276.1348	1.1833	4.2853	7.5


HO
HO
OH
N
N
N
OCH3
2.0
2.0
2.1
1.1
1.0
1.0
2.1
3.0
3.1
8.0
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
1.0
0.5
0.0


158.274
143.535
130.151
128.171
123.913
113.103
69.139
68.191
61.928
54.646
52.775
14.903
HO
HO
OH
N
N
N
OCH3
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0

Acq. File: TEST.wiff Sample ID: TuneSampleID Acq. Date: Thursday, June 20, 2013

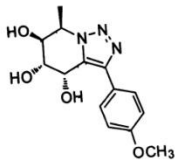
Sample Comment: KS-BT-Ani-291 Acq. Time: 19:56



Acq. File: n/a Sample ID: n/a Acq. Date: n/a

Sample Comment: n/a Acq. Time: n/a

Elemental composition calculator



Target m/z: +292.1306 amu

Tolerance: +5.0000 ppm

Result type: Elemental

Max num of results: 100

Min DBE: -0.5000 Max DBE: +50.0000

Electron state: OddAndEven

Num of charges: 0

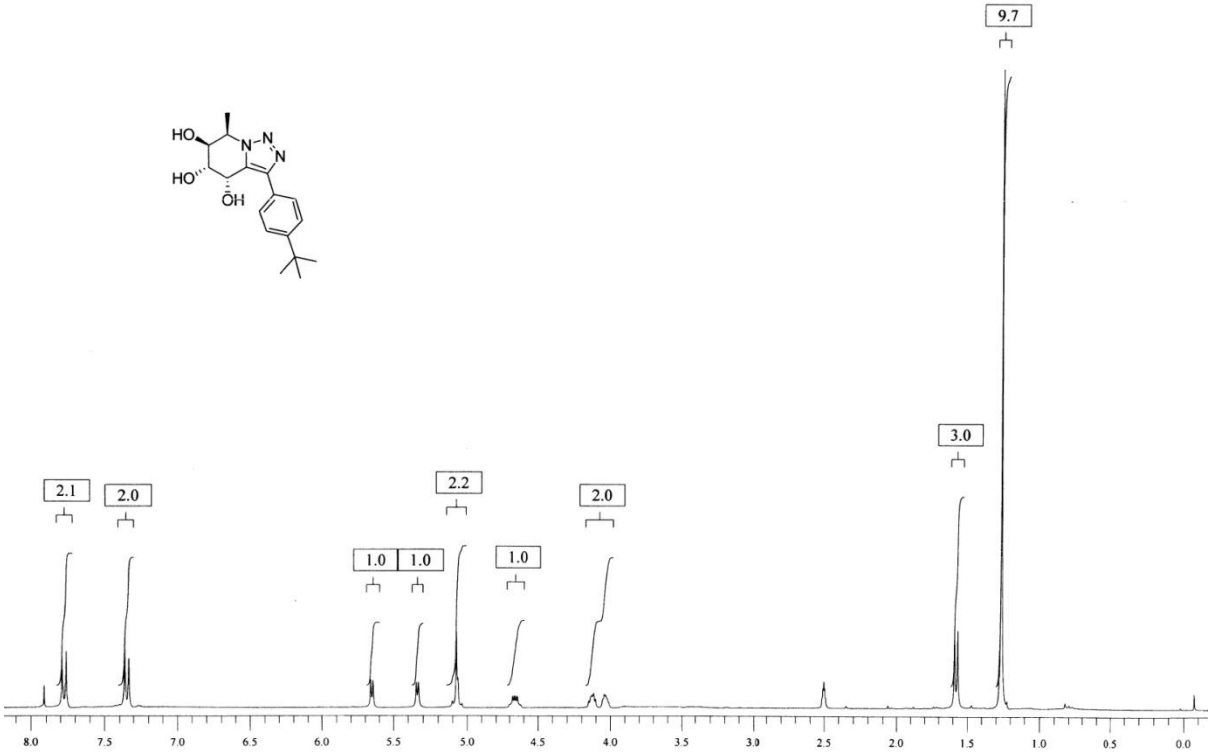
Add water: N/A

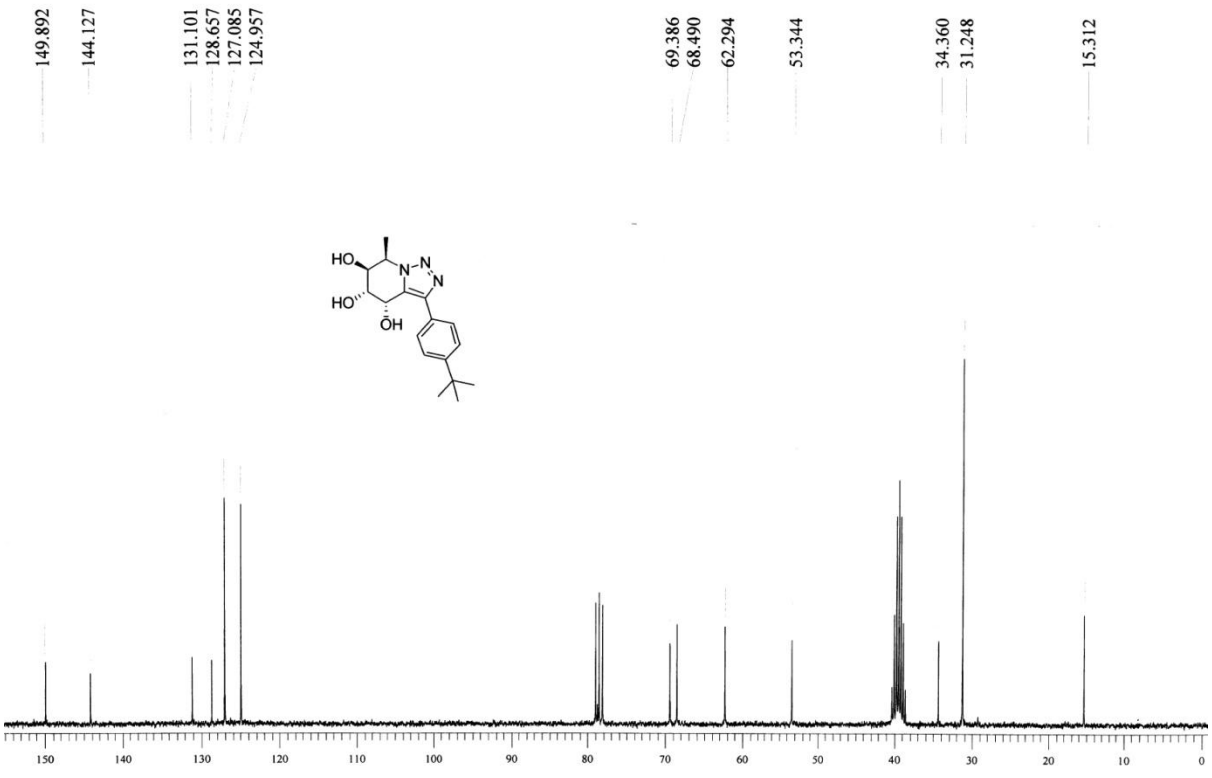
Add proton: N/A

File Name: TEST.wiff

	Elements	Min Number	Max Number
1	C	0	14
2	H	0	18
3	N	0	3
4	O	0	4

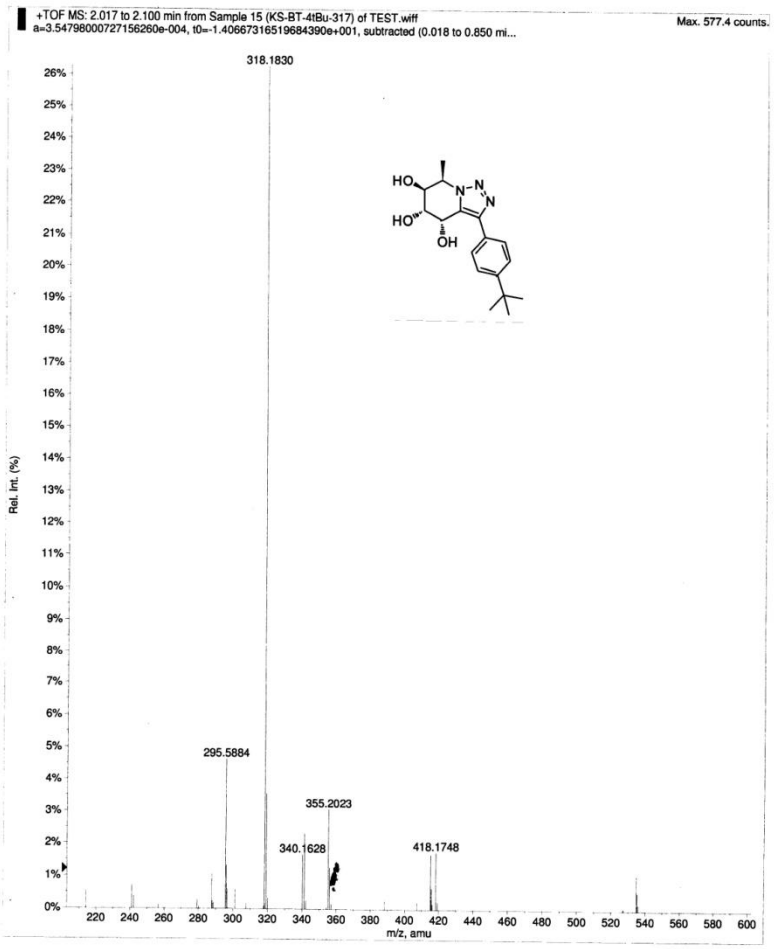
	Formula	Calculated m/z (amu)	mDa Error	PPM Error	DBE
1	C14 H18 N3 O4	292.1297	0.8686	2.9736	7.5


HO
HO
OH
N
N
N
9.7
3.0
2.1
2.0
2.2
2.0
1.0
1.0
1.0
8.0
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
1.0
0.5
0.0


149.892
144.127
131.101
128.657
127.085
124.957
69.386
68.490
62.294
53.344
34.360
31.248
15.312
HO
HO
OH
N
N
N
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0

Acq. File: TEST.wiff Sample ID: TuneSampleID Acq. Date: Thursday, June 20, 2013

Sample Comment: KS-BT-4tBu-317 Acq. Time: 19:12



Acq. File: n/a Sample ID: n/a Acq. Date: n/a

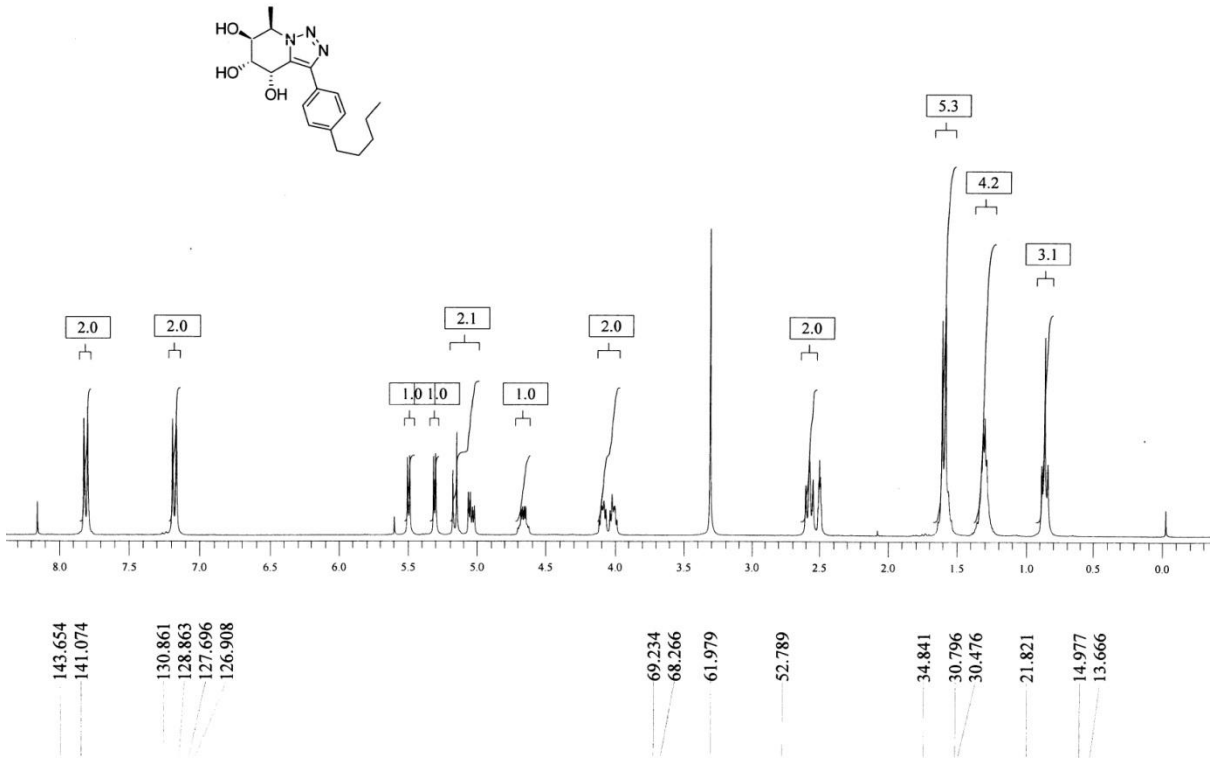
Sample Comment: n/a Acq. Time: n/a

Elemental composition calculator

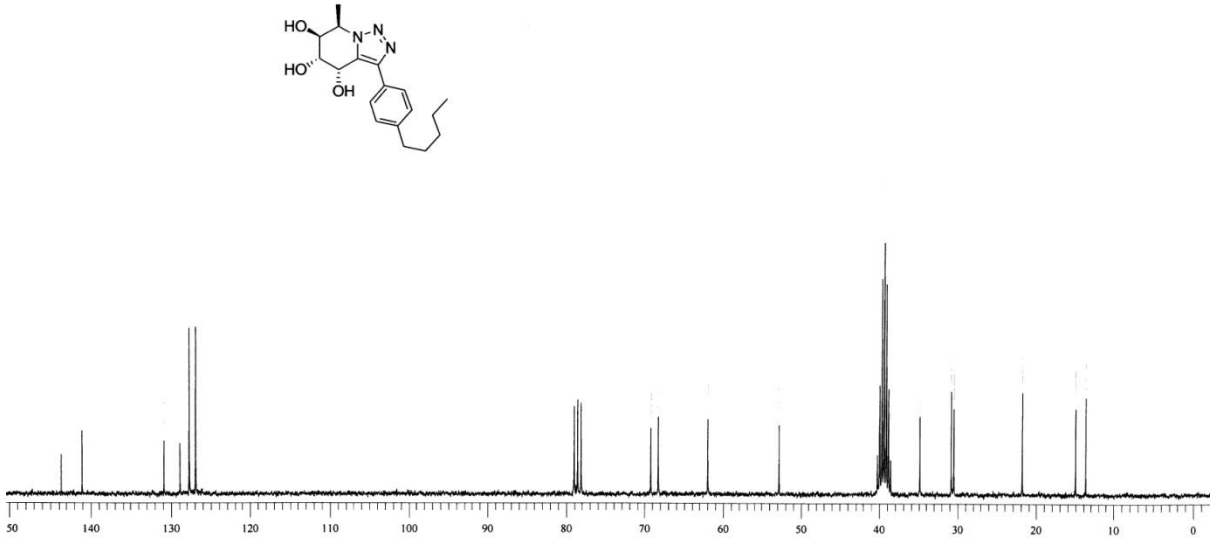
Target m/z: +318.1830 amu
Tolerance: +5.0000 ppm
Result type: Elemental
Max num of results: 100
Min DBE: -0.5000 Max DBE: +50.0000
Electron state: OddAndEven
Num of charges: 0
Add water: N/A
Add proton: N/A
File Name: TEST.wiff

	Elements	Min Number	Max Number
1	C	0	17
2	H	0	24
3	N	0	3
4	Na	0	1
5	O	0	3

	Formula	Calculated m/z (amu)	mDa Error	PPM Error	DBE
1	C17 H24 N3 O3	318.1817	1.2330	3.8754	7.5

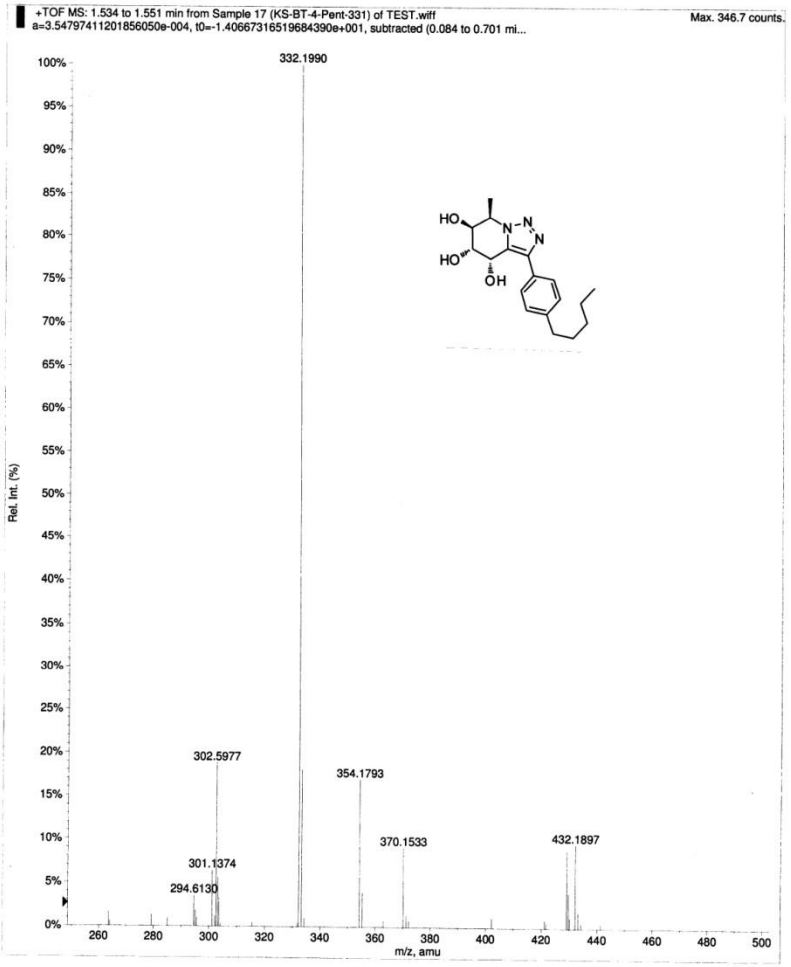

HO
HO
OH
N
N
N
5.3
4.2
3.1
2.0
2.0
2.1
2.0
2.0
1.0
1.0
1.0
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0

143.654
141.074
130.861
128.863
127.696
126.908
69.234
68.266
61.979
52.789
34.841
30.796
30.476
21.821
14.977
13.666


HO
HO
OH
N
N
N
50
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0

Acq. File: TEST.wiff Sample ID: TuneSampleID Acq. Date: Thursday, June 20, 2013

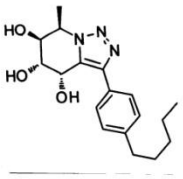
Sample Comment: KS-BT-4-Pent-331 Acq. Time: 19:32



Acq. File: n/a Sample ID: n/a Acq. Date: n/a

Sample Comment: n/a Acq. Time: n/a

Elemental composition calculator



Target m/z: +332.1990 amu

Tolerance: +5.0000 ppm

Result type: Elemental

Max num of results: 100

Min DBE: -0.5000 Max DBE: +50.0000

Electron state: OddAndEven

Num of charges: 0

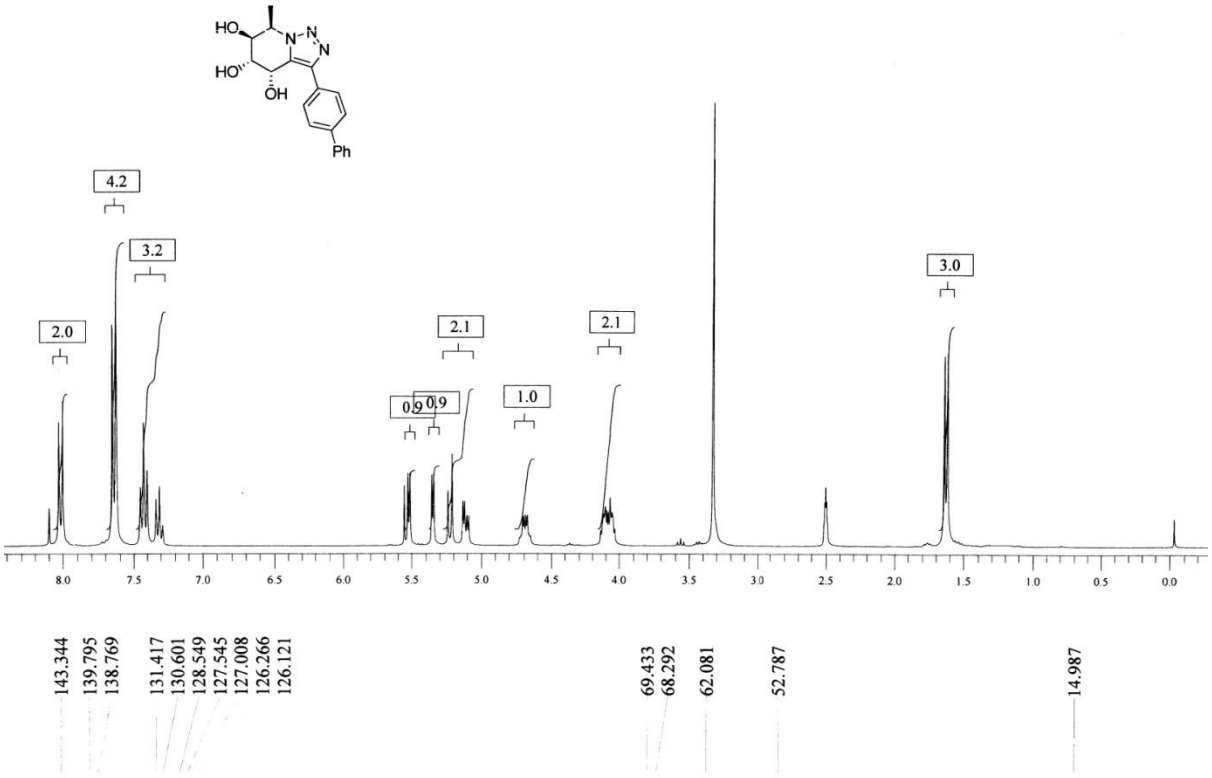
Add water: N/A

Add proton: N/A

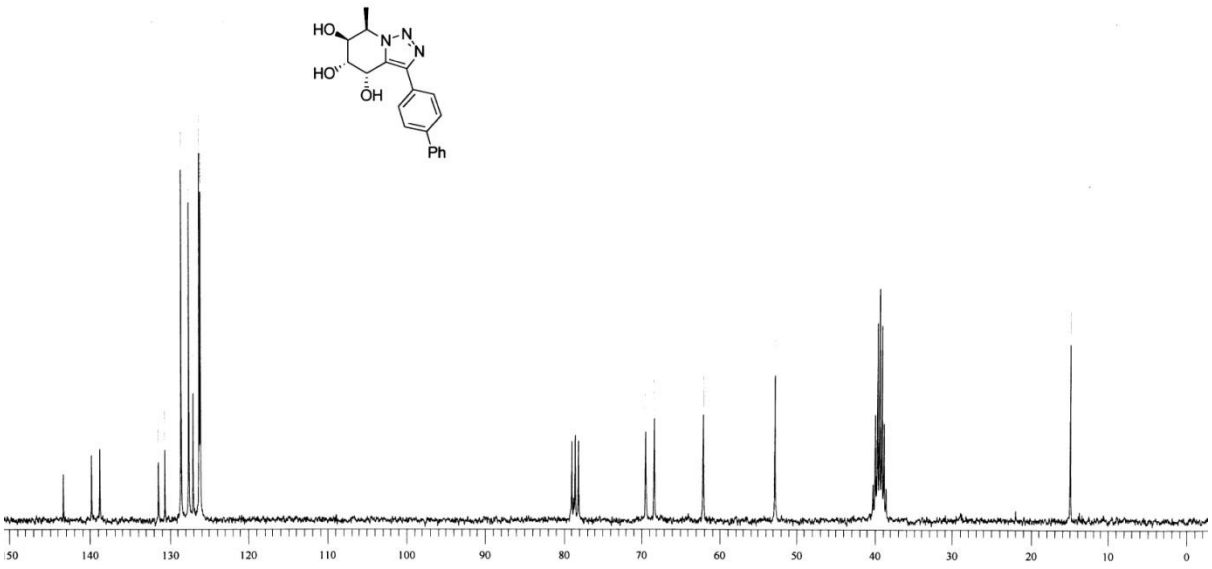
File Name: TEST.wiff

	Elements	Min Number	Max Number
1	C	0	18
2	H	0	26
3	N	0	3
4	O	0	3

	Formula	Calculated m/z (amu)	mDa Error	PPM Error	DBE
1	C18 H26 N3 O3	332.1974	1.5830	4.7652	7.5

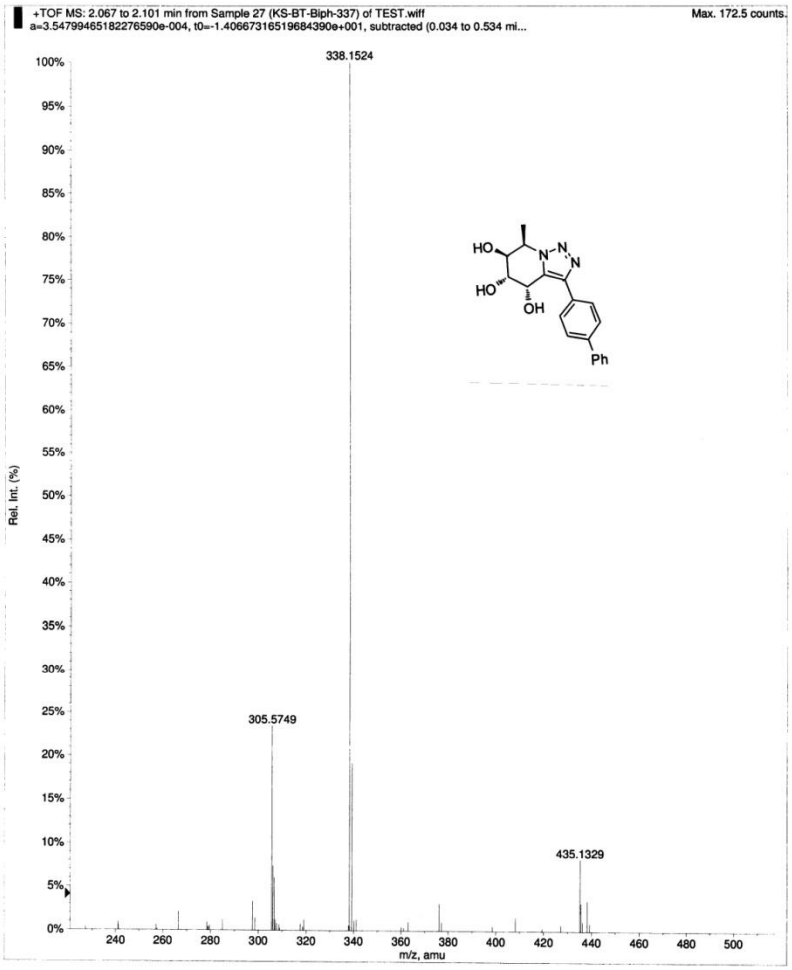


143.344
139.795
138.769
131.417
130.601
128.549
127.545
127.008
126.266
126.121
69.433
68.292
62.081
52.787
14.987



Acq. File: TEST.wiff Sample ID: TuneSampleID Acq. Date: Thursday, June 20, 2013

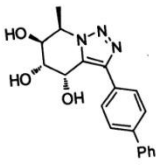
Sample Comment: KS-BT-Biph-337 Acq. Time: 20:19



Acq. File: n/a Sample ID: n/a Acq. Date: n/a

Sample Comment: n/a Acq. Time: n/a

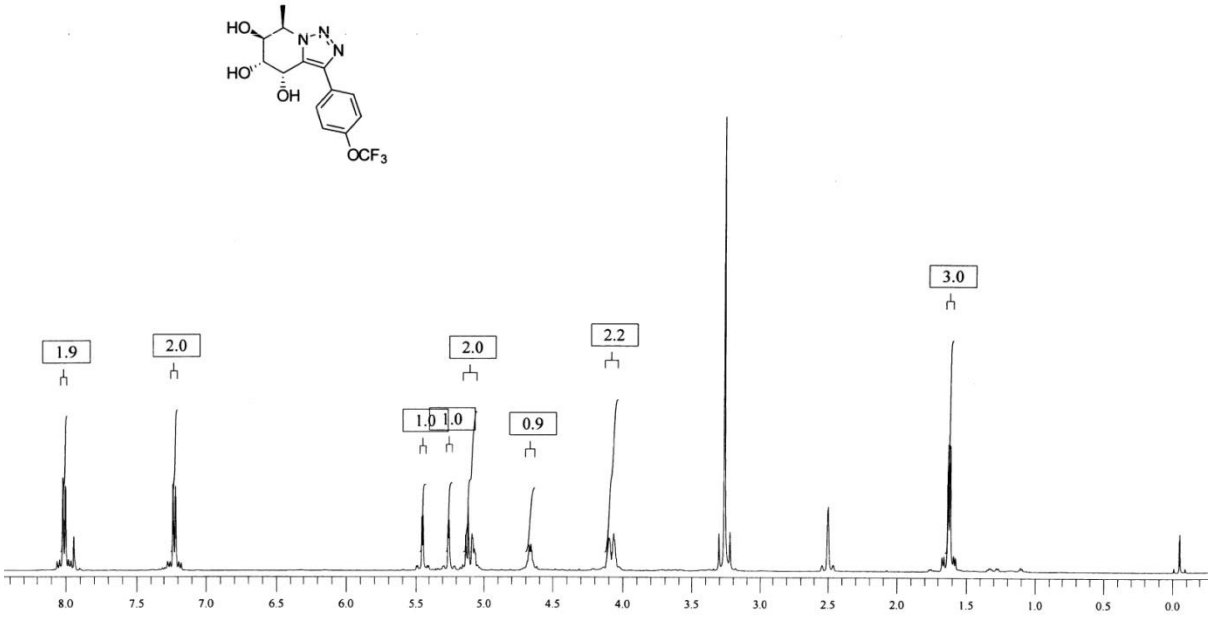
Elemental composition calculator



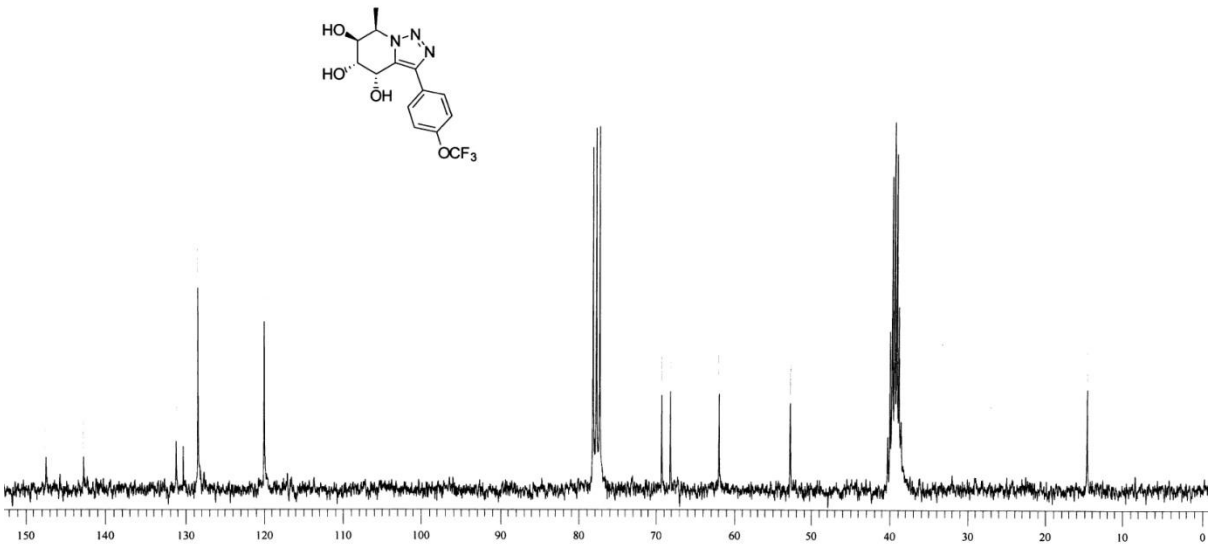
Target m/z: +338.1524 amu
Tolerance: +6.0000 ppm
Result type: Elemental
Max num of results: 100
Min DBE: -0.5000 Max DBE: +50.0000
Electron state: OddAndEven
Num of charges: 0
Add water: N/A
Add proton: N/A
File Name: TEST.wiff

	Elements	Min Number	Max Number
1	C	0	19
2	H	0	20
3	N	0	6
4	O	0	6

	Formula	Calculated m/z (amu)	mDa Error	PPM Error	DBE
1	C19 H20 N3 O3	338.1504	1.9332	5.7171	11.5

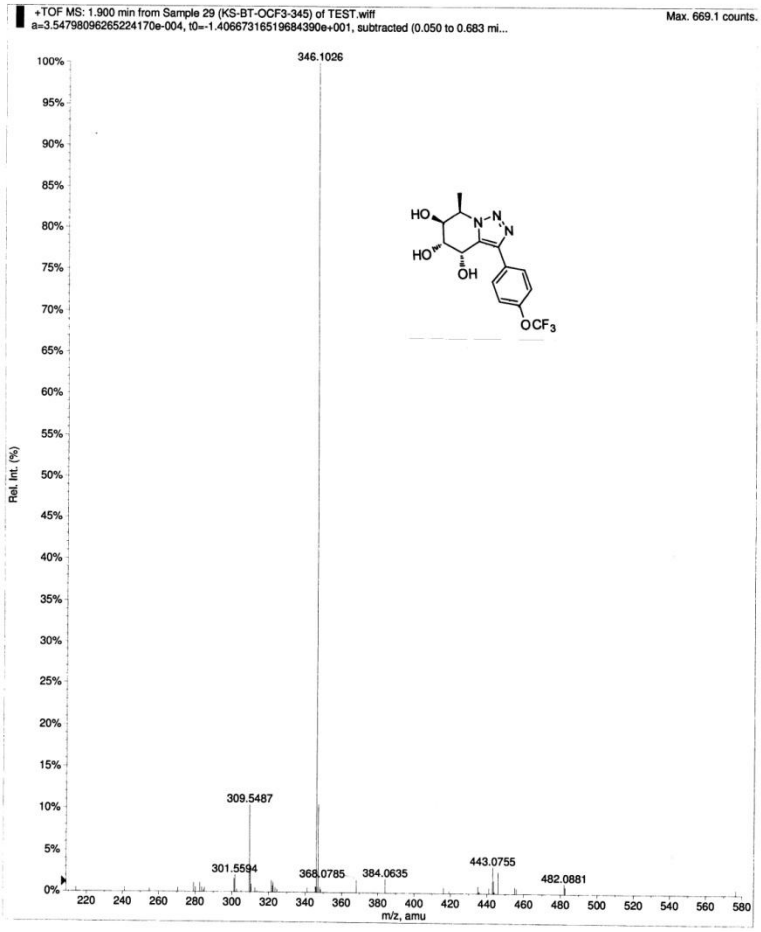

HO
HO
OH
N
N
N
OCF3
1.9
2.0
2.0
1.0
1.0
0.9
2.2
3.0
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0


147.422
142.610
131.143
130.269
128.402
119.933
69.272
68.147
61.930
52.767
14.678


HO
HO
OH
N
N
N
OCF3
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0

Acq. File: TEST.wiff Sample ID: TuneSampleID Acq. Date: Thursday, June 20, 2013

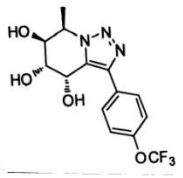
Sample Comment: KS-BT-OCF3-345 Acq. Time: 20:29



Acq. File: n/a Sample ID: n/a Acq. Date: n/a

Sample Comment: n/a Acq. Time: n/a

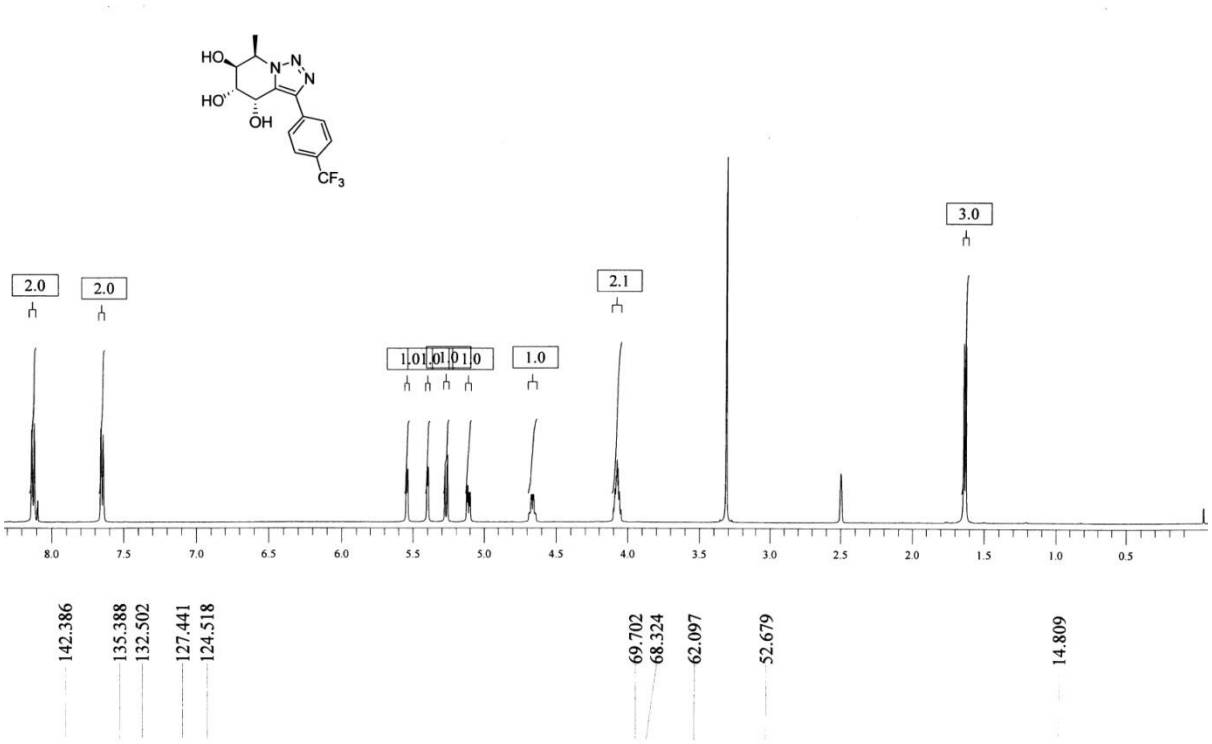
Elemental composition calculator



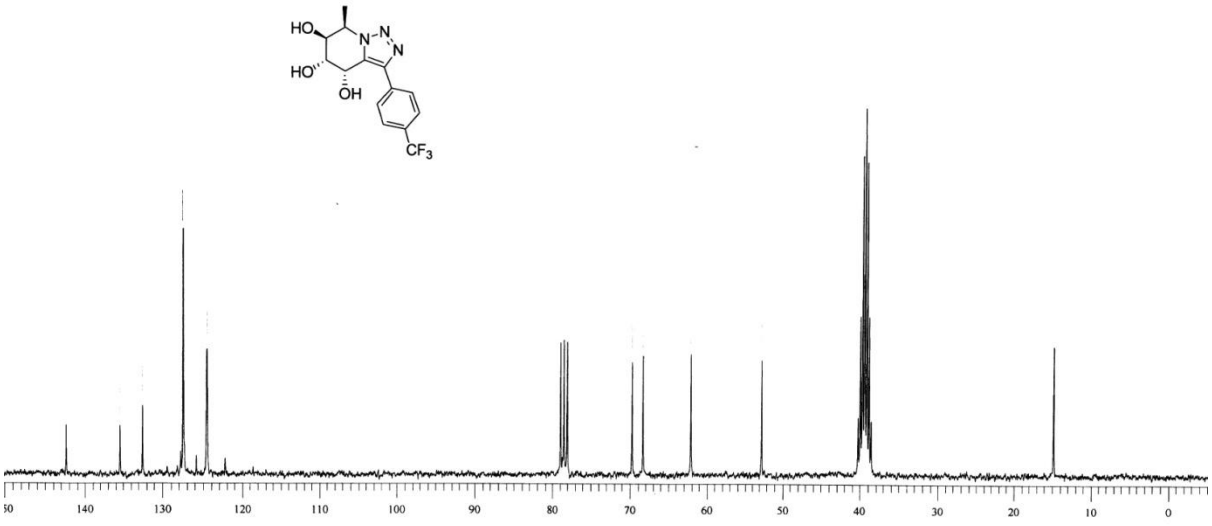
Target m/z: +346.1026 amu
Tolerance: +5.0000 ppm
Result type: Elemental
Max num of results: 100
Min DBE: -0.5000 Max DBE: +50.0000
Electron state: OddAndEven
Num of charges: 0
Add water: N/A
Add proton: N/A
File Name: TEST.wiff

	Elements	Min Number	Max Number
1	C	0	14
2	F	0	3
3	H	0	15
4	N	0	3
5	O	0	4

	Formula	Calculated m/z (amu)	mDa Error	PPM Error	DBE
1	C14 H15 N3 O4 F3	346.1014	1.1340	3.2766	7.5


HO
HO
OH
N
N
N
CF3
2.0
2.0
1.0
1.0
1.0
1.0
2.1
3.0
8.0
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
1.0
0.5

142.386
135.388
132.502
127.441
124.518
69.702
68.324
62.097
52.679
14.809

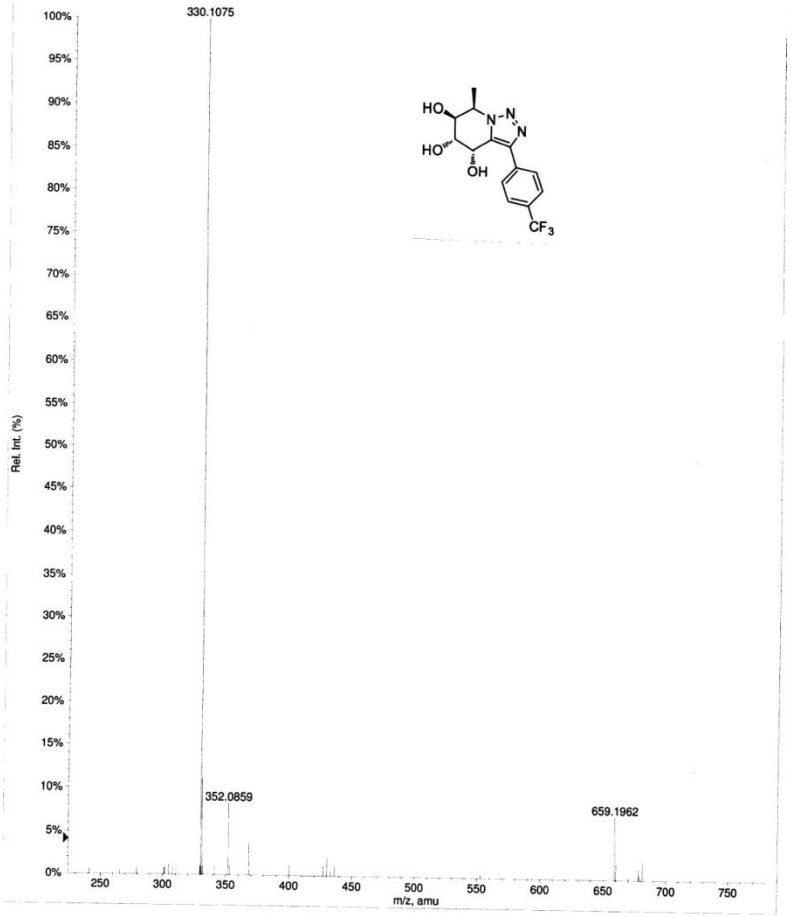

HO
HO
OH
N
N
N
CF3
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0

Acq. File: TEST.wiff Sample ID: TuneSampleID Acq. Date: Thursday, June 20, 2013

Sample Comment: KS-BT-4CF3-329 Acq. Time: 19:46

+TOF MS: 2.151 to 2.184 min from Sample 20 (KS-BT-4CF3-329) of TEST.wiff
a=3.54798103974653030e-004, t0=-1.40667316519684390e+001, subtracted (0.051 to 0.951 mi...

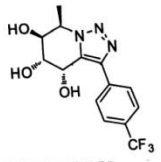
Max. 916.9 counts.



Acq. File: n/a Sample ID: n/a Acq. Date: n/a

Sample Comment: n/a Acq. Time: n/a

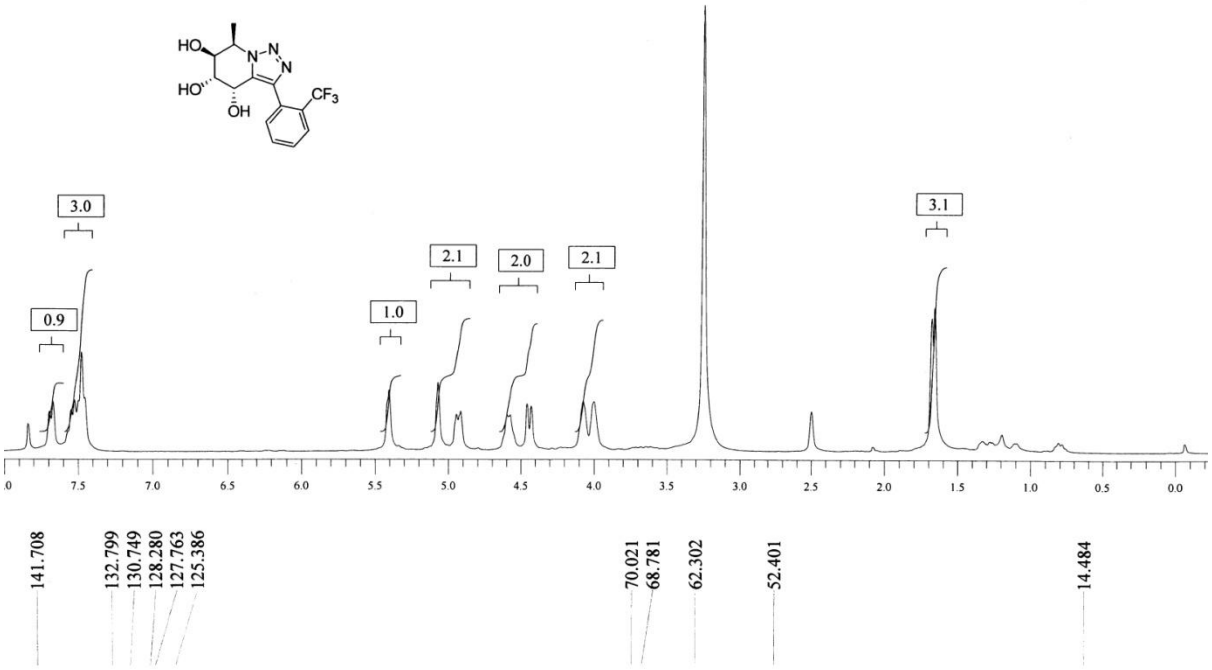
Elemental composition calculator



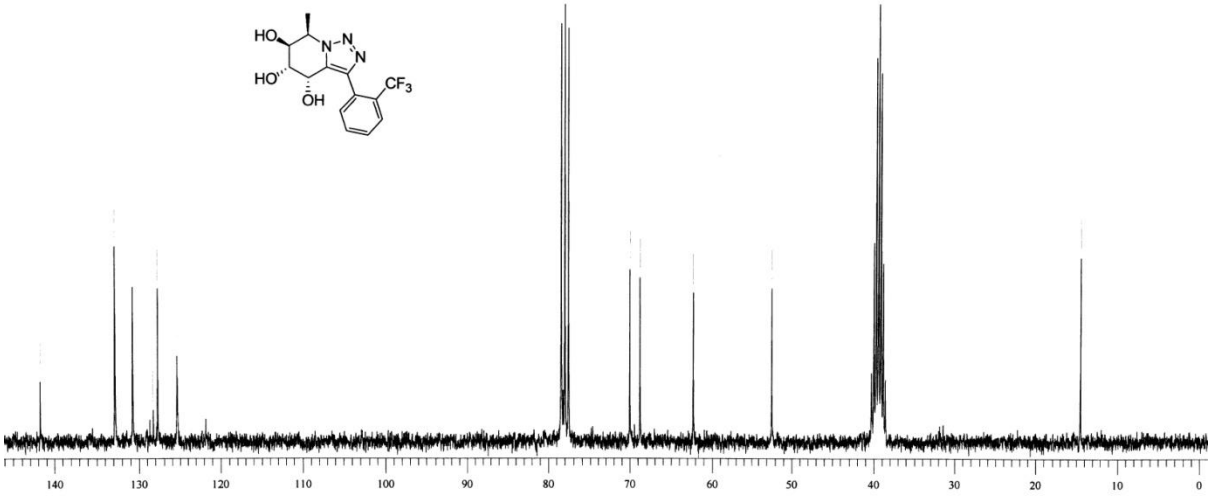
Target m/z: +330.1075 amu
Tolerance: +5.0000 ppm
Result type: Elemental
Max num of results: 100
Min DBE: -0.5000 Max DBE: +50.0000
Electron state: OddAndEven
Num of charges: 0
Add water: N/A
Add proton: N/A
File Name: TEST.wiff

	Elements	Min Number	Max Number
1	C	0	14
2	F	0	3
3	H	0	15
4	N	0	3
5	O	0	3

	Formula	Calculated m/z (amu)	mDa Error	PPM Error	DBE
1	C14 H15 N3 O3 F3	330.1065	0.9487	2.8739	7.5

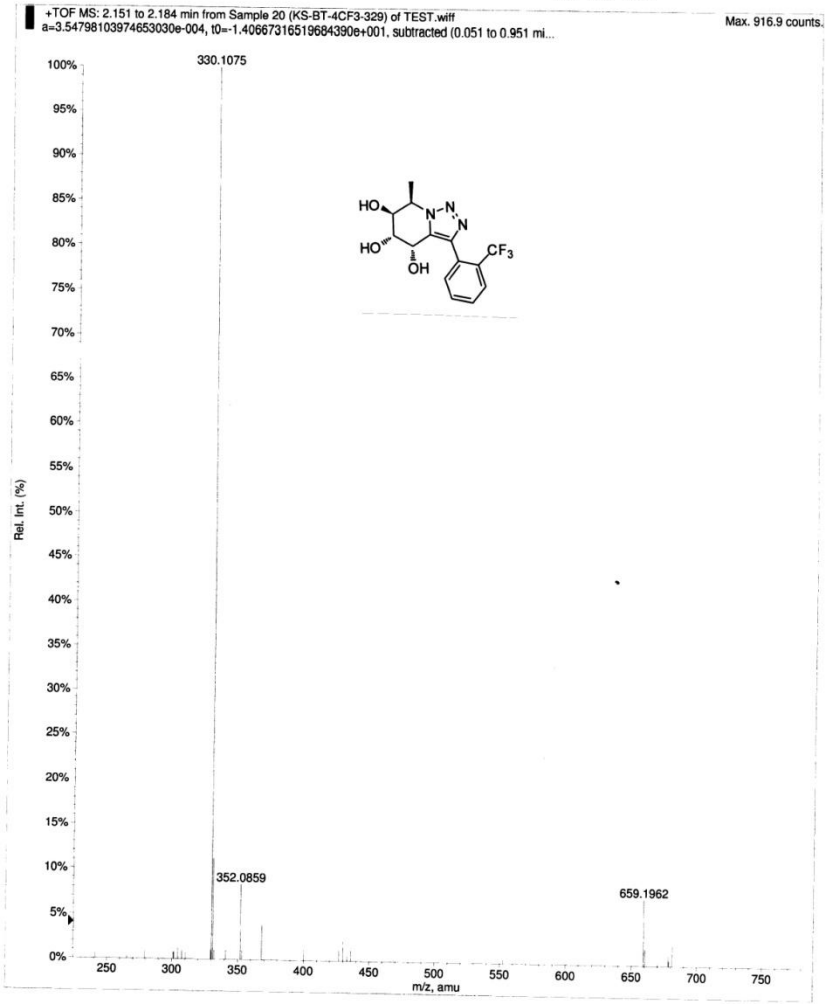

HO
HO
OH
N
N
N
CF3
3.0
0.9
1.0
2.1
2.0
2.1
3.1
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0

141.708
132.799
130.749
128.280
127.763
125.386
70.021
68.781
62.302
52.401
14.484


HO
HO
OH
N
N
N
CF3
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0

Acq. File: TEST.wiff Sample ID: TuneSampleID Acq. Date: Thursday, June 20, 2013

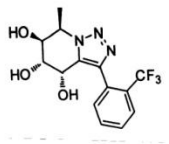
Sample Comment: KS-BT-4CF3-329 Acq. Time: 19:46



Acq. File: n/a Sample ID: n/a Acq. Date: n/a

Sample Comment: n/a Acq. Time: n/a

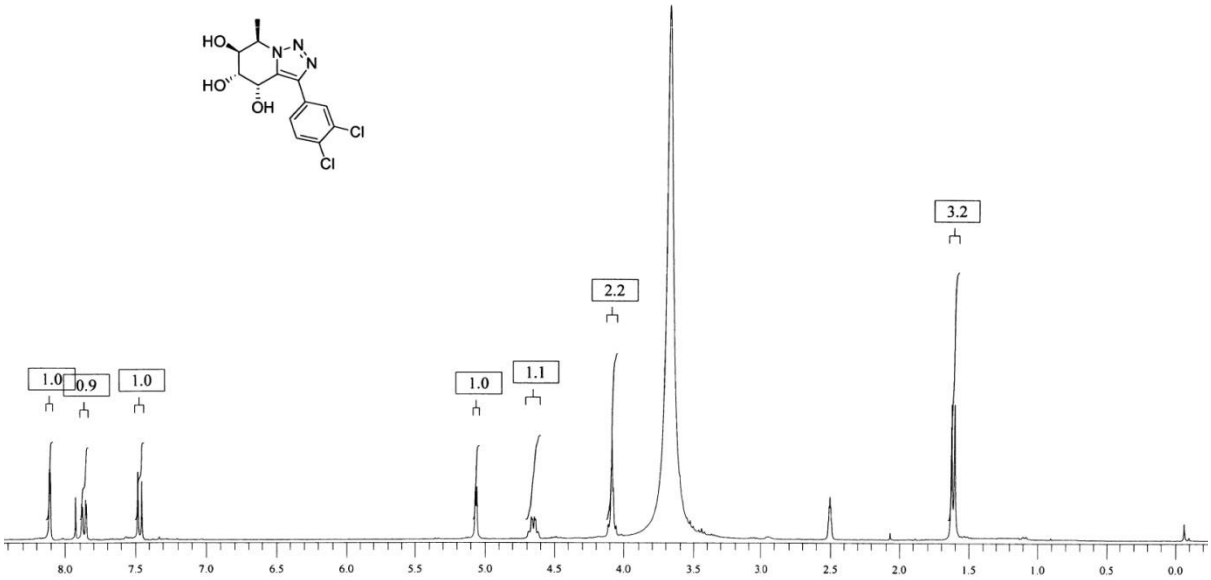
Elemental composition calculator

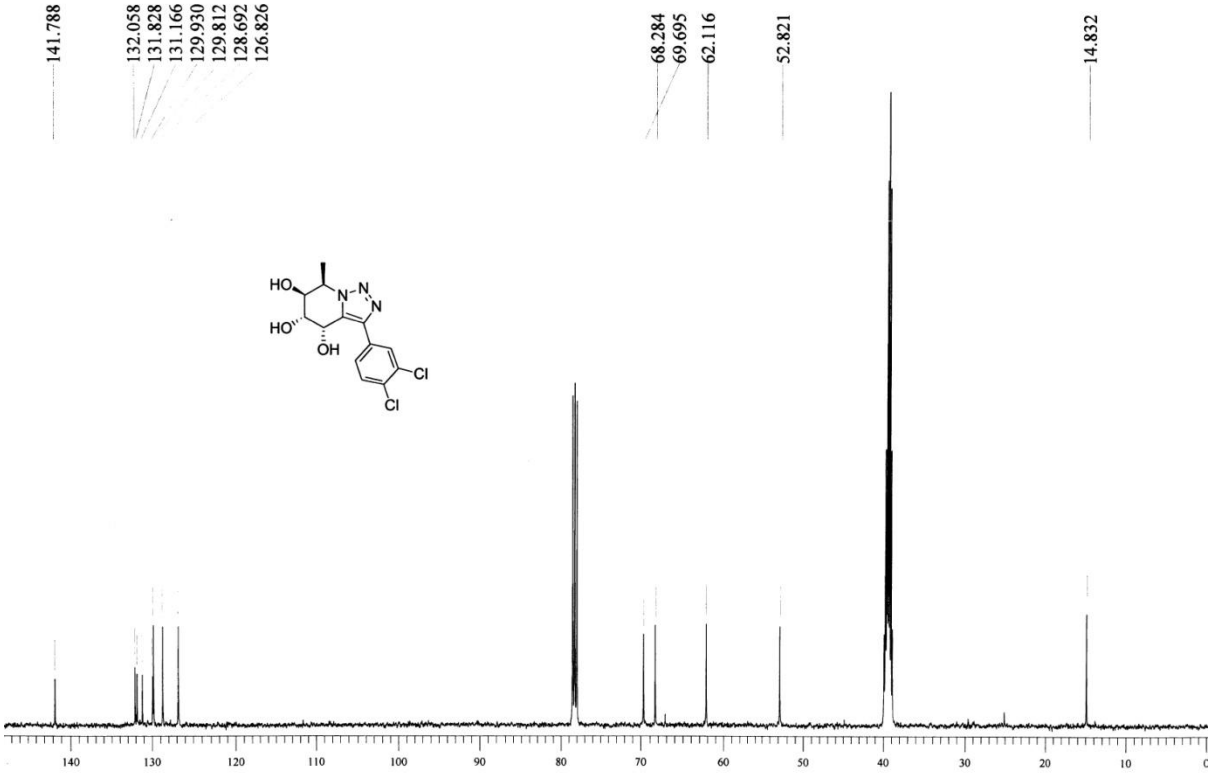


Target m/z: +330.1075 amu
Tolerance: +5.0000 ppm
Result type: Elemental
Max num of results: 100
Min DBE: -0.5000 Max DBE: +50.0000
Electron state: OddAndEven
Num of charges: 0
Add water: N/A
Add proton: N/A
File Name: TEST.wiff

	Elements	Min Number	Max Number
1	C	0	14
2	F	0	3
3	H	0	15
4	N	0	3
5	O	0	3

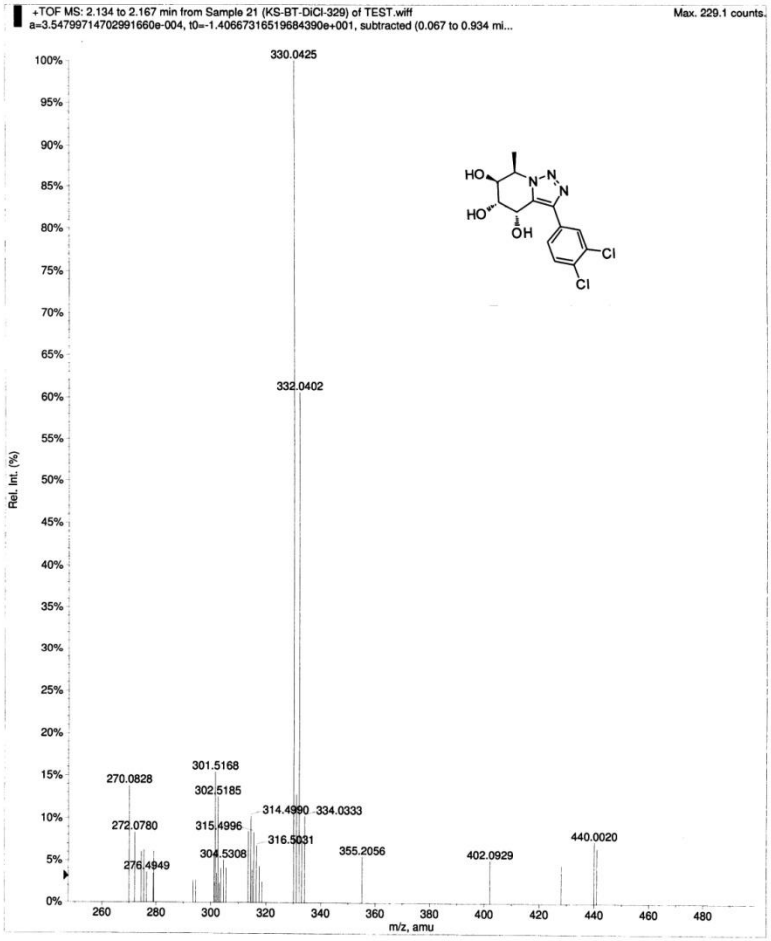
	Formula	Calculated m/z (amu)	mDa Error	PPM Error	DBE
1	C14 H15 N3 O3 F3	330.1065	0.9487	2.8739	7.5


HO
HO
OH
N
N
N
Cl
Cl
1.0
0.9
1.0
1.0
1.1
2.2
3.2
8.0
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
1.0
0.5
0.0


141.788
132.058
131.828
131.166
129.930
129.812
128.692
126.826
68.284
69.695
62.116
52.821
14.832
HO
HO
OH
N
N
N
Cl
Cl
140
130
120
110
100
90
80
70
60
50
40
30
20
10

Acq. File: TEST.wiff Sample ID: TuneSampleID Acq. Date: Thursday, June 20, 2013

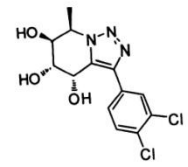
Sample Comment: KS-BT-DiCl-329 Acq. Time: 19:51



Acq. File: n/a Sample ID: n/a Acq. Date: n/a

Sample Comment: n/a Acq. Time: n/a

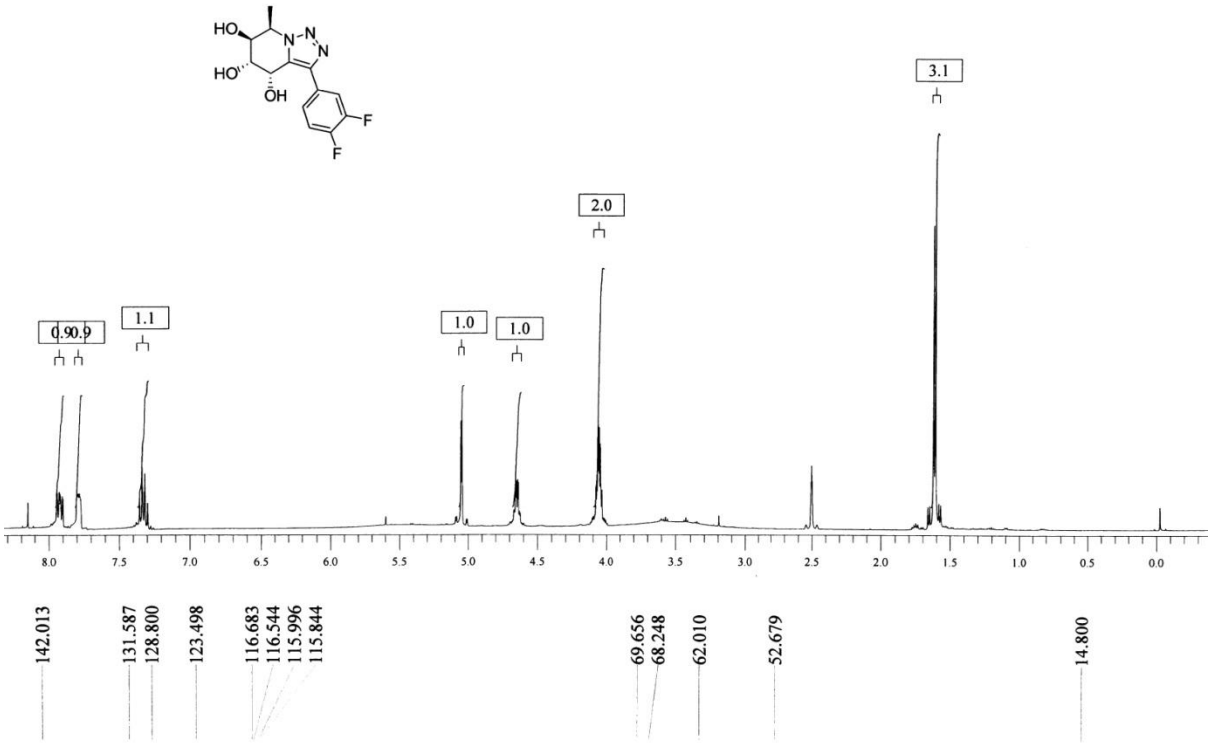
Elemental composition calculator



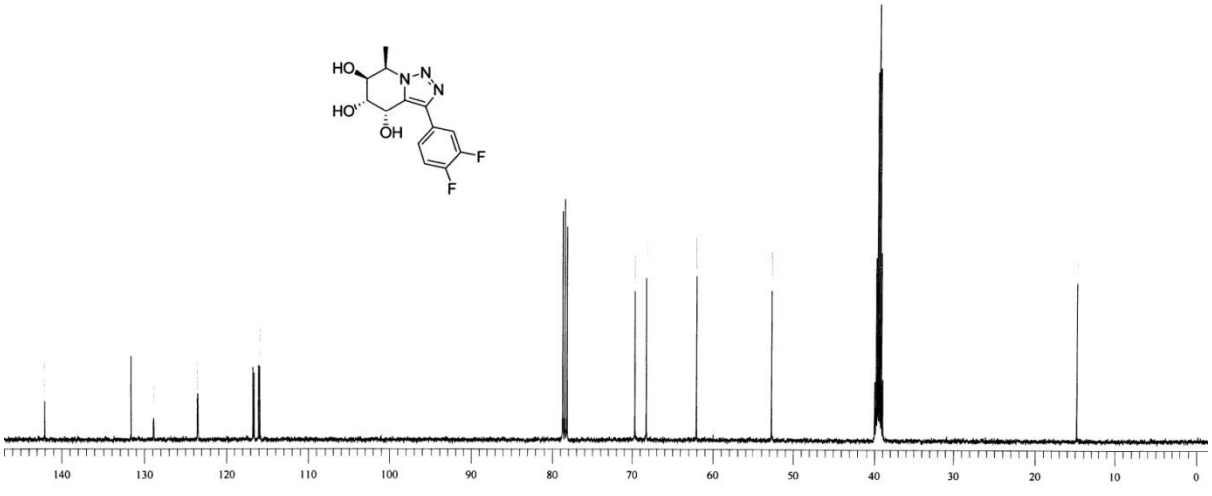
Target m/z: +330.0425 amu
Tolerance: +5.0000 ppm
Result type: Elemental
Max num of results: 100
Min DBE: -0.5000 Max DBE: +50.0000
Electron state: OddAndEven
Num of charges: 0
Add water: N/A
Add proton: N/A
File Name: TEST.wiff

	Elements	Min Number	Max Number
1	C	0	13
2	Cl	0	2
3	H	0	14
4	N	0	3
5	O	0	3

	Formula	Calculated m/z (amu)	mDa Error	PPM Error	DBE
1	C13 H14 N3 O3 Cl2	330.0412	1.2780	3.8723	7.5

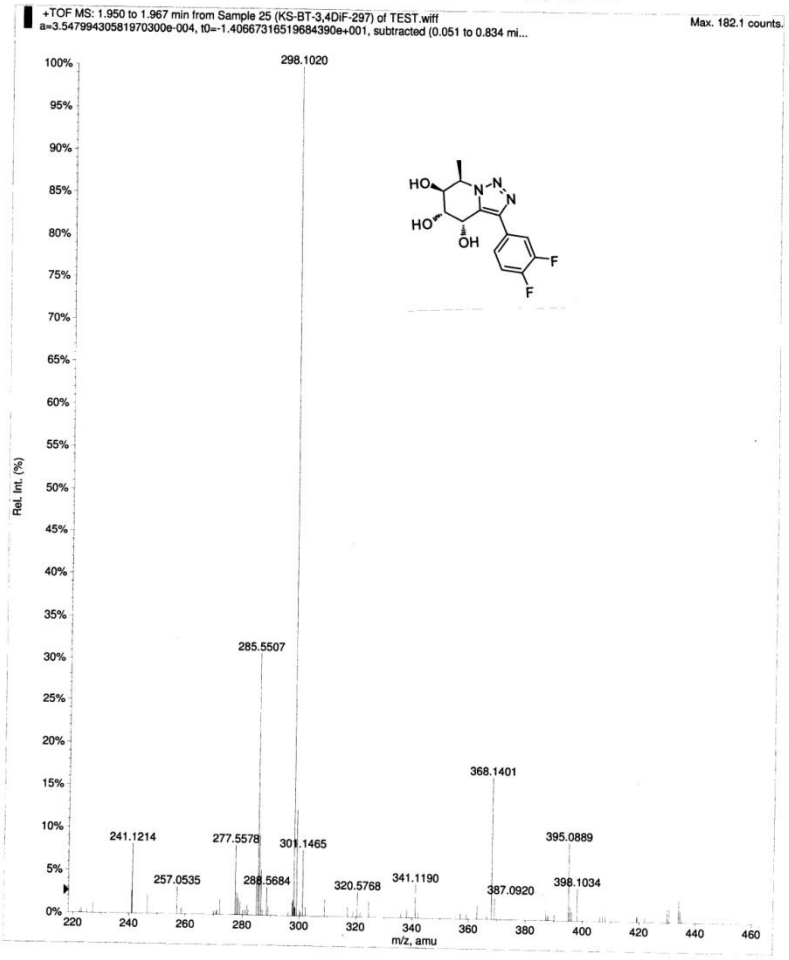


142.013
131.587
128.800
123.498
116.683
116.544
115.996
115.844
69.656
68.248
62.010
52.679
14.800



Acq. File: TEST.wiff Sample ID: TuneSampleID Acq. Date: Thursday, June 20, 2013

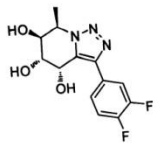
Sample Comment: KS-BT-3,4DiF-297 Acq. Time: 20:11



Acq. File: n/a Sample ID: n/a Acq. Date: n/a

Sample Comment: n/a Acq. Time: n/a

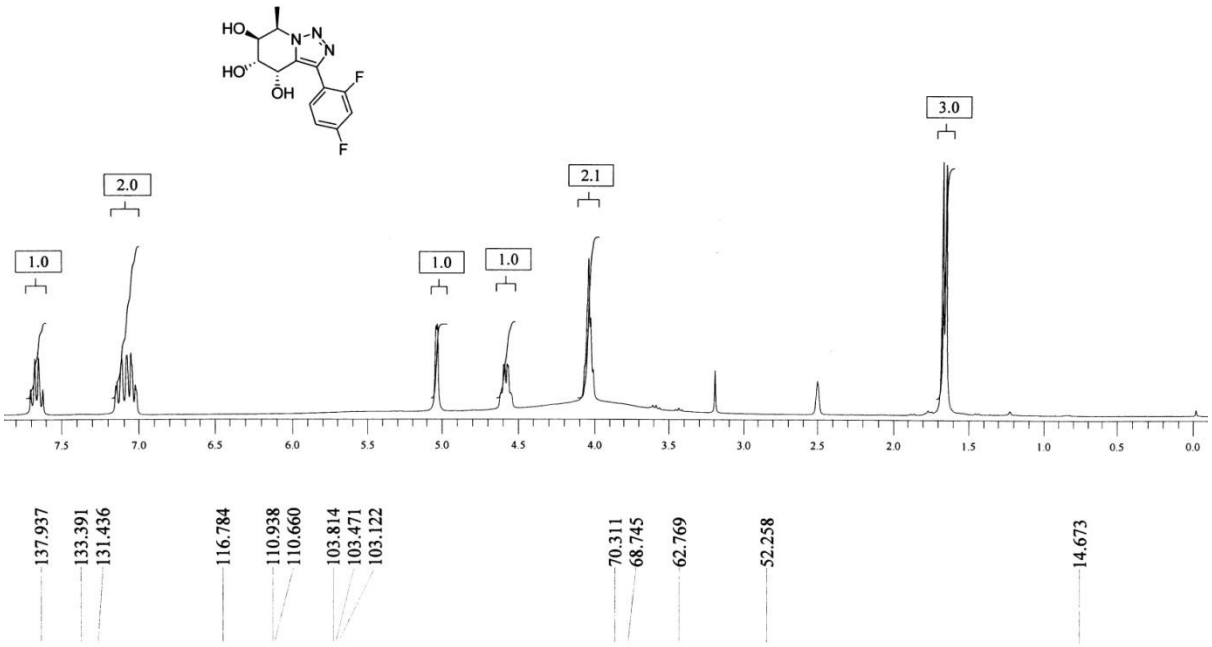
Elemental composition calculator



Target m/z: +298.1020 amu
Tolerance: +6.0000 ppm
Result type: Elemental
Max num of results: 100
Min DBE: -0.5000 Max DBE: +50.0000
Electron state: OddAndEven
Num of charges: 0
Add water: N/A
Add proton: N/A
File Name: TEST.wiff

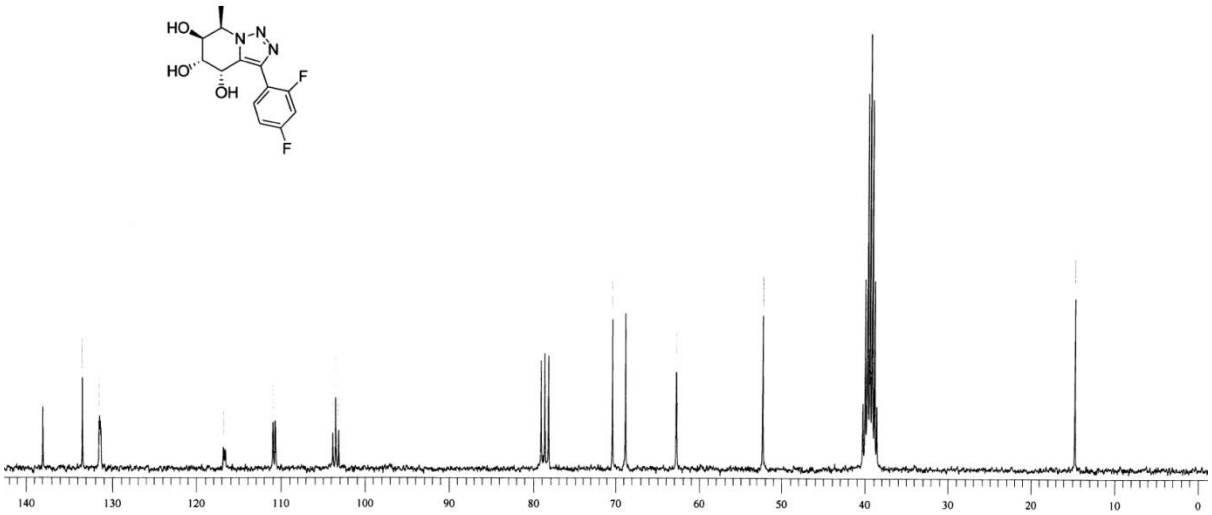
	Elements	Min Number	Max Number
1	C	0	13
2	F	0	2
3	H	0	14
4	N	0	3
5	O	0	4

	Formula	Calculated m/z (amu)	mDa Error	PPM Error	DBE
1	C13 H14 N3 O3 F2	298.1003	1.6769	5.6255	7.5


HO
HO
OH
N
N
N
F
F
1.0
2.0
1.0
1.0
2.1
3.0
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
1.0
0.5
0.0

137.937
133.391
131.436
116.784
110.938
110.660
103.814
103.471
103.122

70.311
68.745
62.769
52.258
14.673

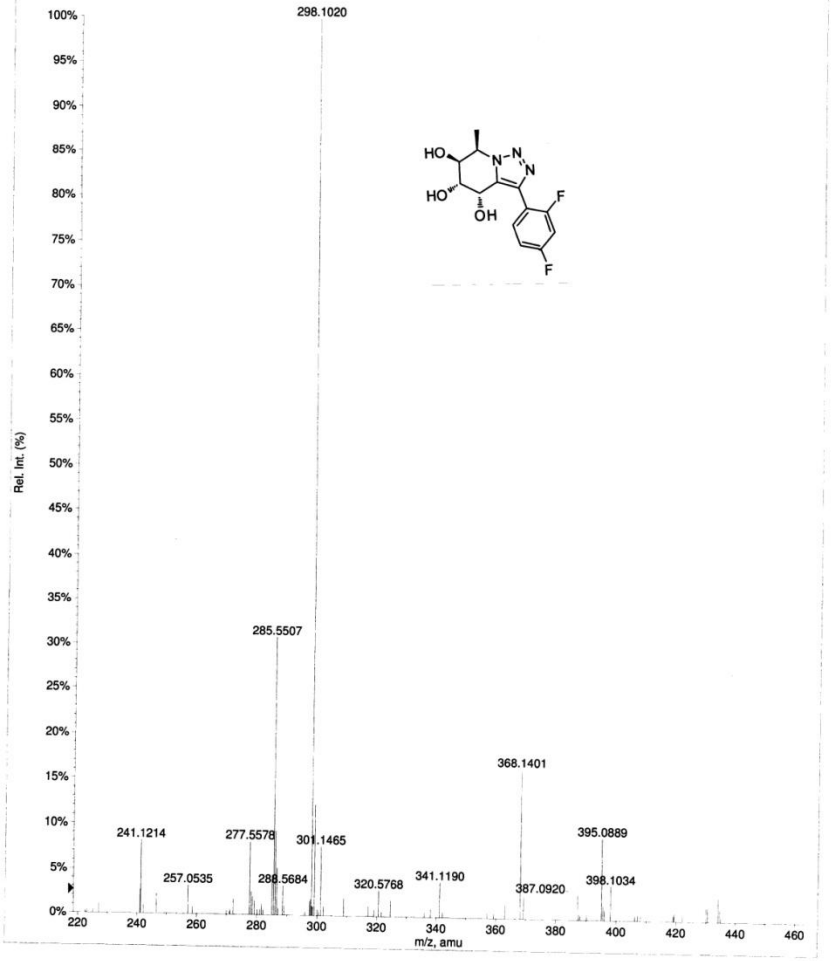

HO
HO
OH
N
N
N
F
F
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0

Acq. File: TEST.wiff Sample ID: TuneSampleID Acq. Date: Thursday, June 20, 2013

Sample Comment: KS-BT-3,4DiF-297 Acq. Time: 20:11

+TOF MS: 1.950 to 1.967 min from Sample 25 (KS-BT-3,4DiF-297) of TEST.wiff
a=3.54799430581970300e-004, t0=-1.40667316519684390e+001, subtracted (0.051 to 0.834 mi...

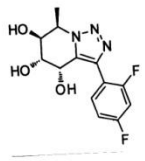
Max. 182.1 counts.



Acq. File: n/a Sample ID: n/a Acq. Date: n/a

Sample Comment: n/a Acq. Time: n/a

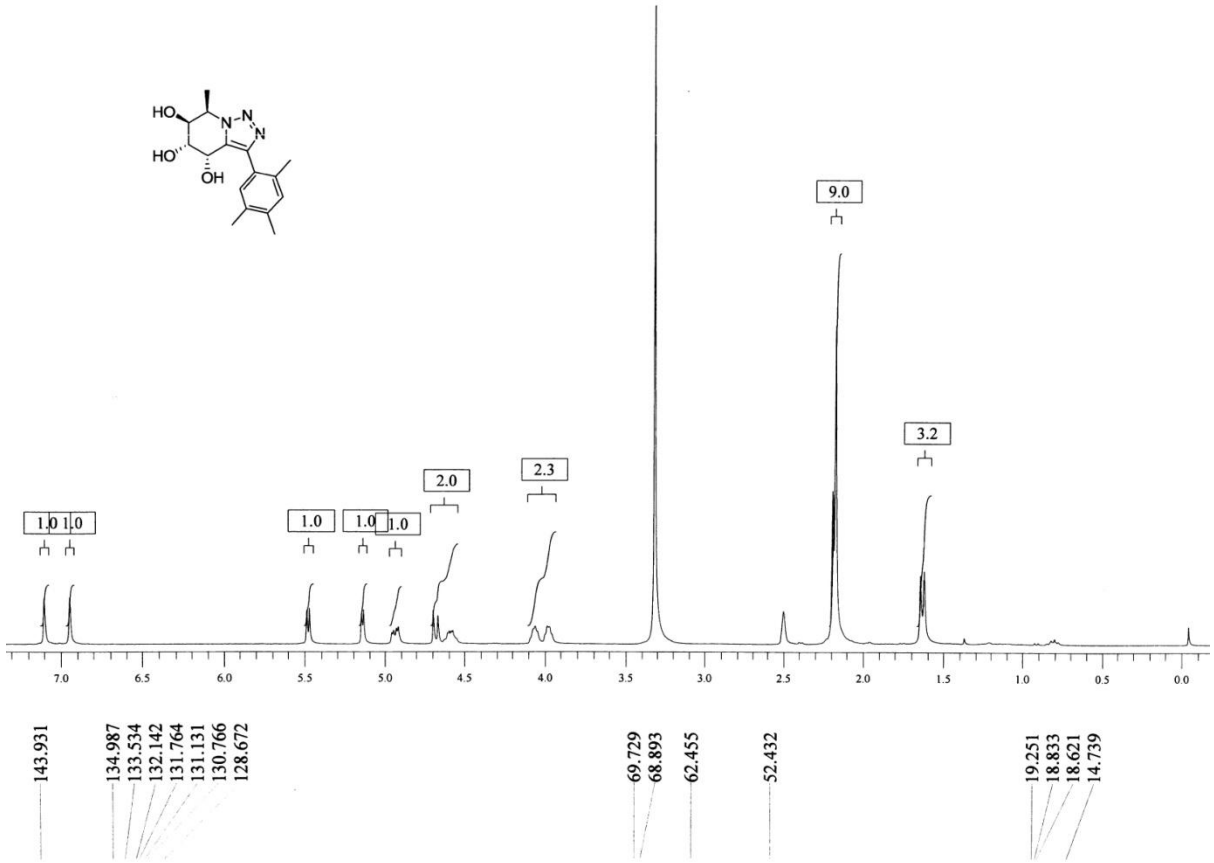
Elemental composition calculator



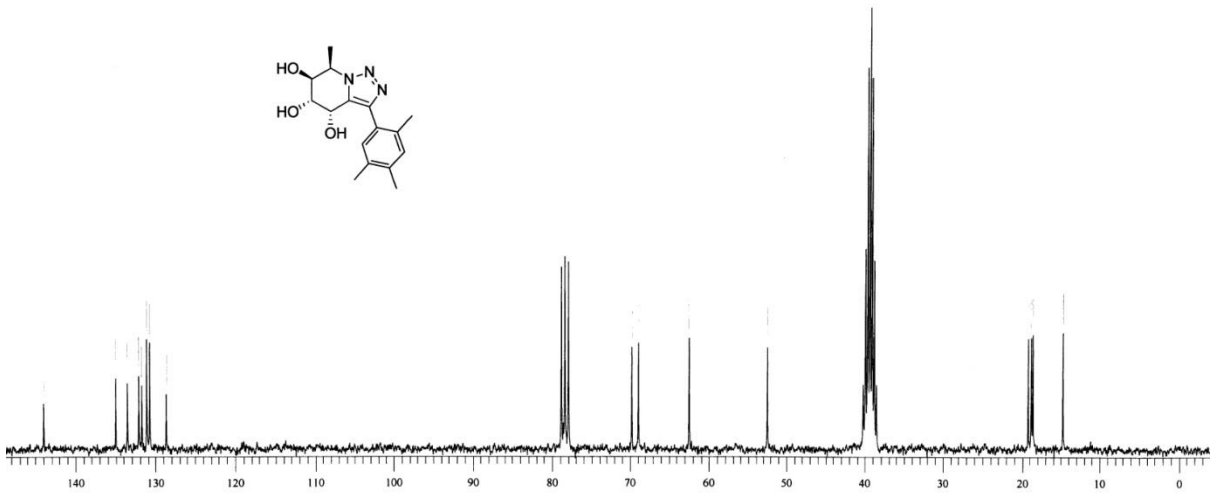
Target m/z: +298.1020 amu
Tolerance: +6.0000 ppm
Result type: Elemental
Max num of results: 100
Min DBE: -0.5000 Max DBE: +50.0000
Electron state: OddAndEven
Num of charges: 0
Add water: N/A
Add proton: N/A
File Name: TEST.wiff

	Elements	Min Number	Max Number
1	C	0	13
2	F	0	2
3	H	0	14
4	N	0	3
5	O	0	4

	Formula	Calculated m/z (amu)	mDa Error	PPM Error	DBE
1	C13 H14 N3 O3 F2	298.1003	1.6769	5.6255	7.5

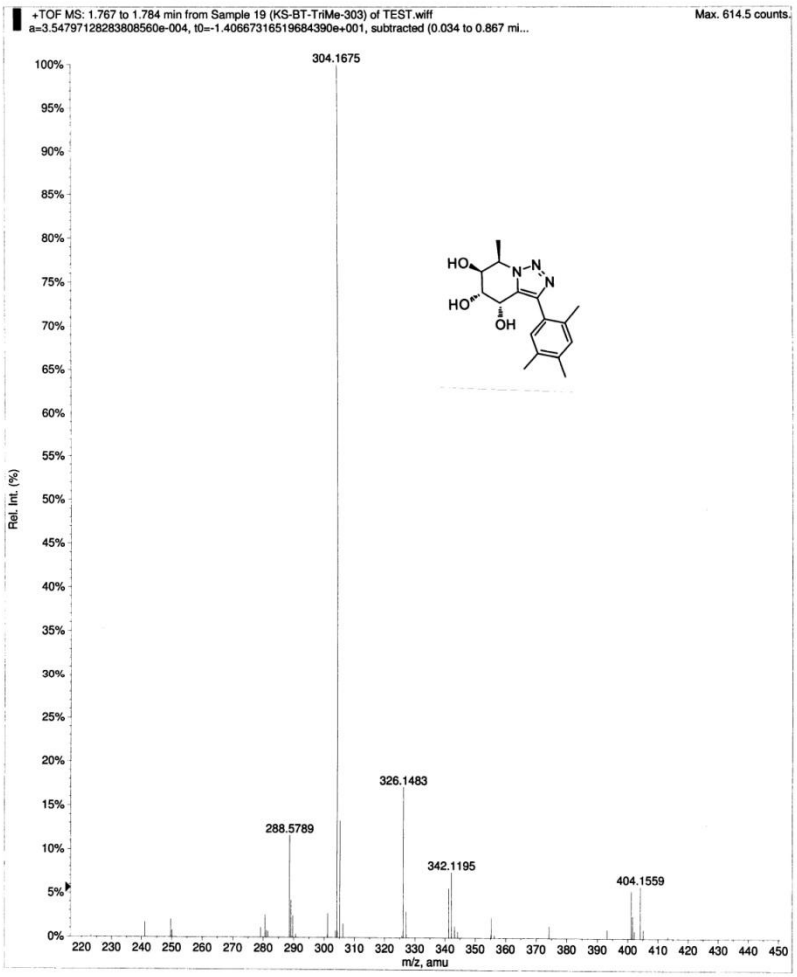

HO
HO
OH
N
N
N
1.0
1.0
1.0
1.0
1.0
2.0
2.3
9.0
3.2
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0

143.931
134.987
133.534
132.142
131.764
131.131
130.766
128.672
69.729
68.893
62.455
52.432
19.251
18.833
18.621
14.739


HO
HO
OH
N
N
N
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0

Acq. File: TEST.wiff Sample ID: TuneSampleID Acq. Date: Thursday, June 20, 2013

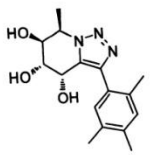
Sample Comment: KS-BT-TriMe-303 Acq. Time: 19:41



Acq. File: n/a Sample ID: n/a Acq. Date: n/a

Sample Comment: n/a Acq. Time: n/a

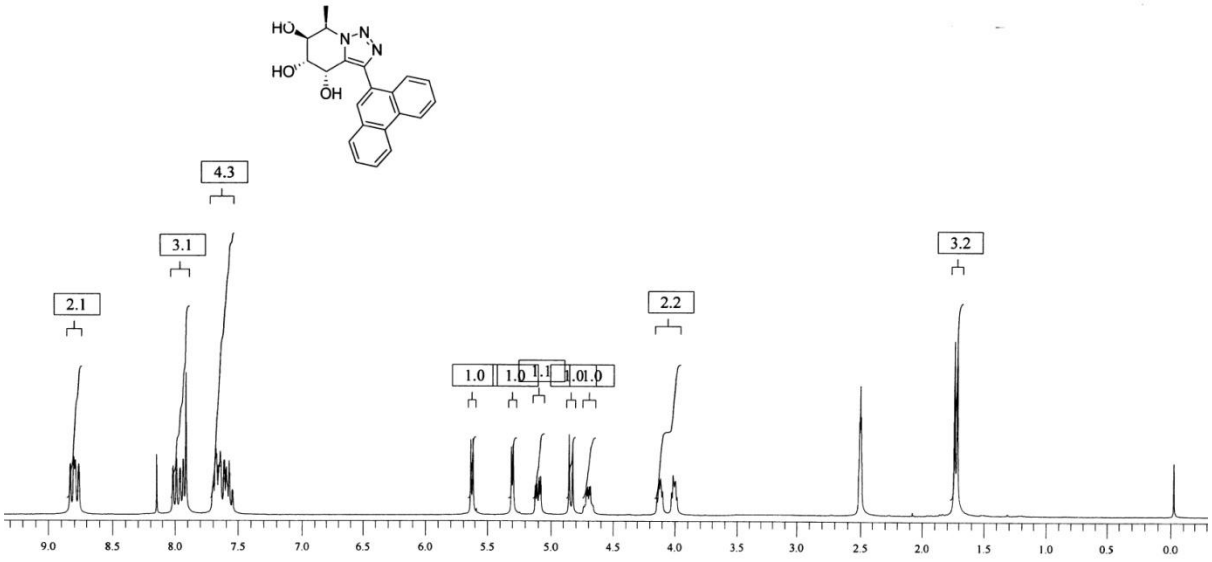
Elemental composition calculator

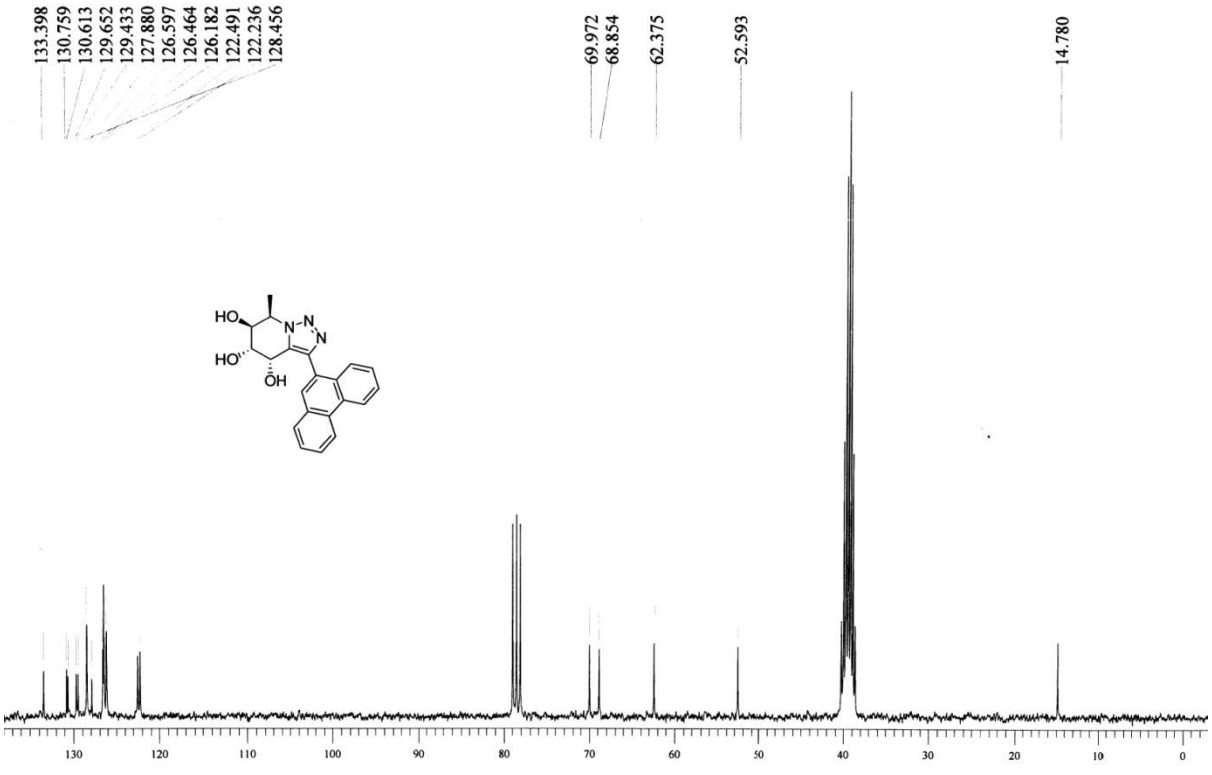


Target m/z: +304.1675 amu
Tolerance: +5.0000 ppm
Result type: Elemental
Max num of results: 100
Min DBE: -0.5000 Max DBE: +50.0000
Electron state: OddAndEven
Num of charges: 0
Add water: N/A
Add proton: N/A
File Name: TEST.wiff

	Elements	Min Number	Max Number
1	C	0	16
2	H	0	22
3	N	0	3
4	O	0	3

	Formula	Calculated m/z (amu)	mDa Error	PPM Error	DBE
1	C16 H22 N3 O3	304.1661	1.3831	4.5474	7.5


HO
HO
OH
N
N
N
4.3
3.1
2.1
2.2
3.2
1.0
1.0
1.1
1.01.0
9.0
8.5
8.0
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
1.0
0.5
0.0

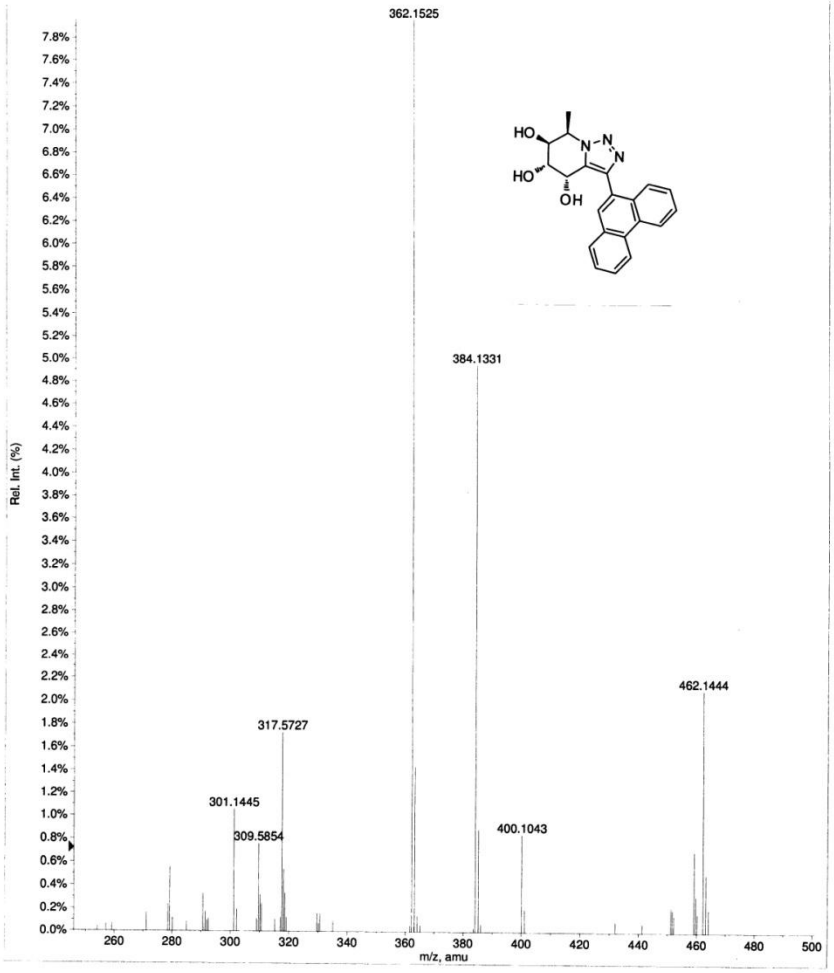

133.398
130.759
130.613
129.652
129.433
127.880
126.597
126.464
126.182
122.491
122.236
128.456
69.972
68.854
62.375
52.593
14.780
HO
HO
OH
N
N
N
130
120
110
100
90
80
70
60
50
40
30
20
10
0

Acq. File: TEST.wiff Sample ID: TuneSampleID Acq. Date: Thursday, June 20, 2013

Sample Comment: KS-BT-9Anth-361 Acq. Time: 19:27

+TOF MS: 1.501 to 1.517 min from Sample 16 (KS-BT-9Anth-361) of TEST.wiff
a=3.5479936410093212 0e-004, t0=-1.4066731651968439 0e+001, subtracted (0.051 to 0.784 m...

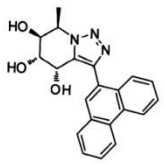
Max. 4674.5 counts.



Acq. File: n/a Sample ID: n/a Acq. Date: n/a

Sample Comment: n/a Acq. Time: n/a

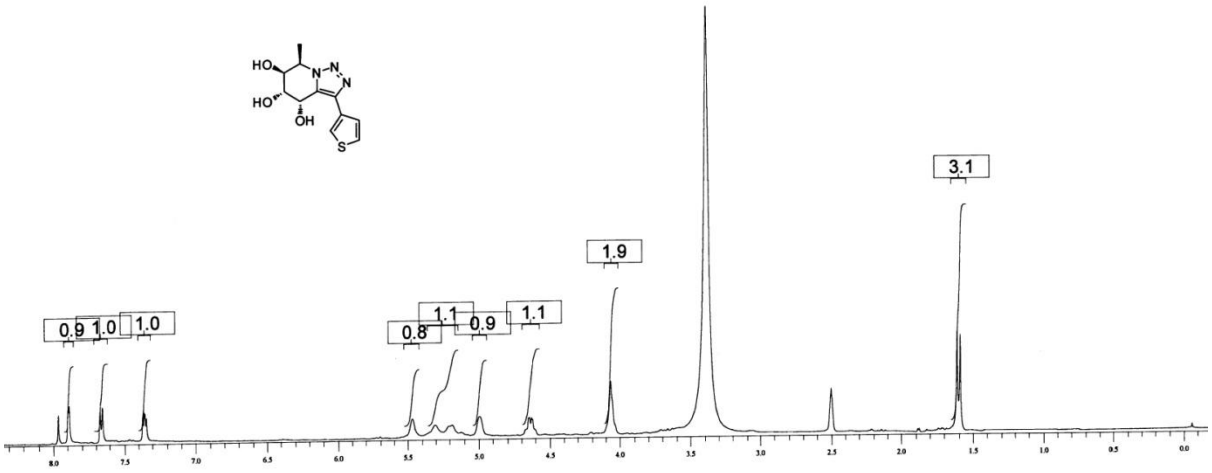
Elemental composition calculator

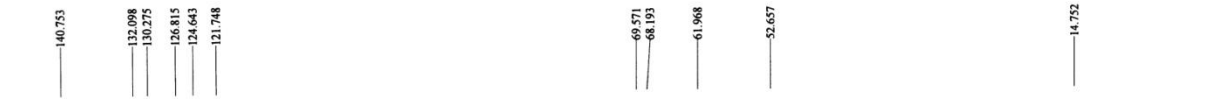


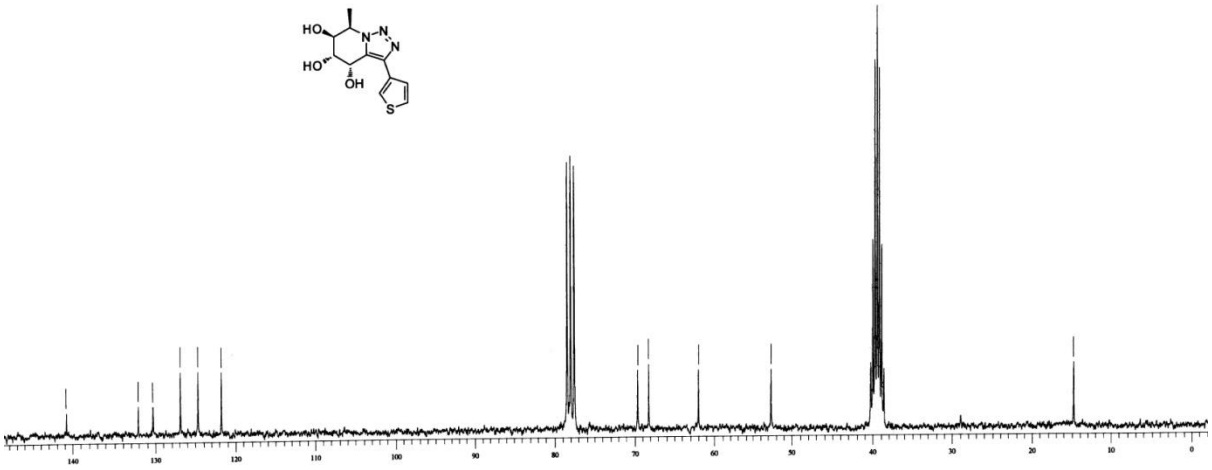
Target m/z: +362.1525 amu
Tolerance: +6.0000 ppm
Result type: Elemental
Max num of results: 100
Min DBE: -0.5000 Max DBE: +50.0000
Electron state: OddAndEven
Num of charges: 0
Add water: N/A
Add proton: N/A
File Name: TEST.wiff

	Elements	Min Number	Max Number
1	C	0	22
2	H	0	20
3	N	0	3
4	Na	0	1
5	O	0	3

	Formula	Calculated m/z (amu)	mDa Error	PPM Error	DBE
1	C21 H20 N3 O3	362.1504	2.0332	5.6143	13.

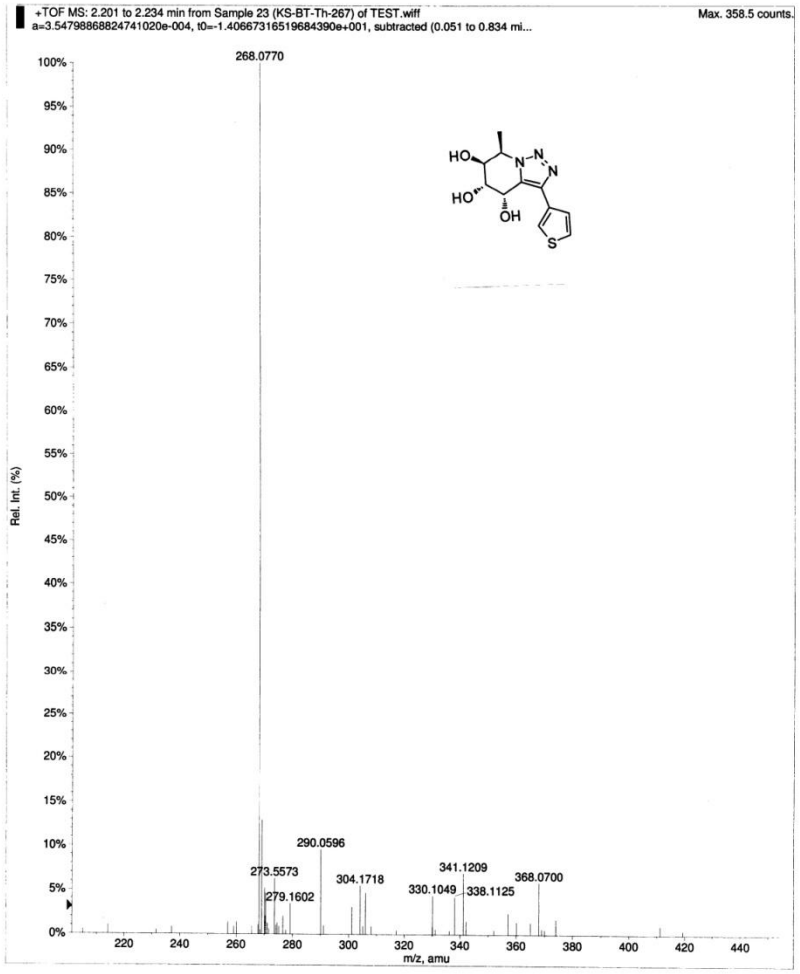

HO
HO
OH
N
N
N
S
0.9
1.0
1.0
0.8
1.1
0.9
1.1
1.9
3.1


140.753
132.098
130.275
126.815
124.643
121.748
69.571
68.193
61.968
52.657
14.752


HO
HO
OH
N
N
N
S

Acq. File: TEST.wiff Sample ID: TuneSampleID Acq. Date: Thursday, June 20, 2013

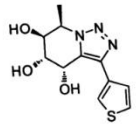
Sample Comment: KS-BT-Th-267 Acq. Time: 20:01



Acq. File: n/a Sample ID: n/a Acq. Date: n/a

Sample Comment: n/a Acq. Time: n/a

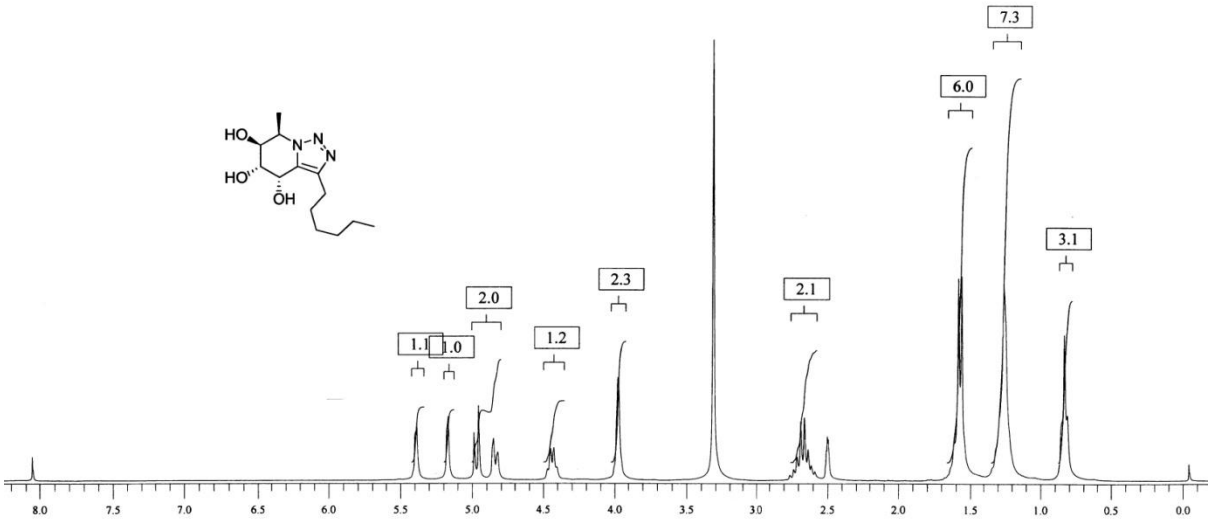
Elemental composition calculator

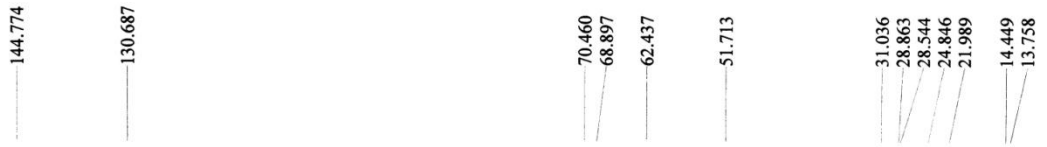


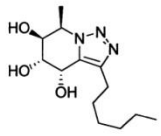
Target m/z: +268.0770 amu
Tolerance: +6.0000 ppm
Result type: Elemental
Max num of results: 100
Min DBE: -0.5000 Max DBE: +50.0000
Electron state: OddAndEven
Num of charges: 0
Add water: N/A
Add proton: N/A
File Name: TEST.wiff

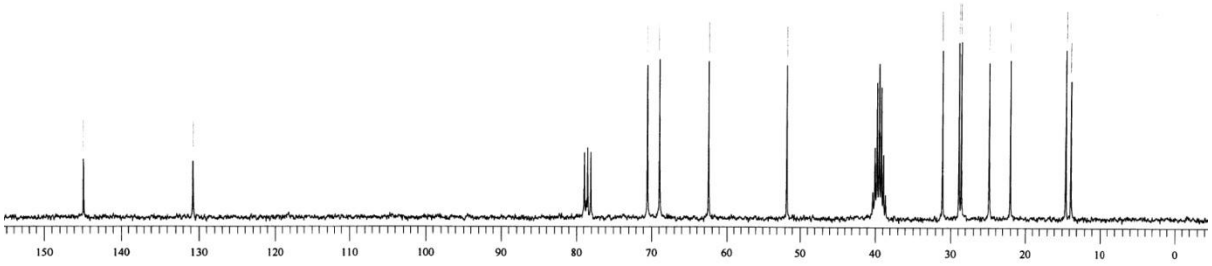
	Elements	Min Number	Max Number
1	C	0	11
2	H	0	14
3	N	0	3
4	O	0	4
5	S	0	1

	Formula	Calculated m/z (amu)	mDa Error	PPM Error	DBE
1	C11 H14 N3 O3 S	268.0755	1.4116	5.2660	6.5


HO
HO
OH
N
N
N
7.3
6.0
3.1
2.3
2.1
2.0
1.2
1.1
1.0
8.0
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
1.0
0.5
0.0

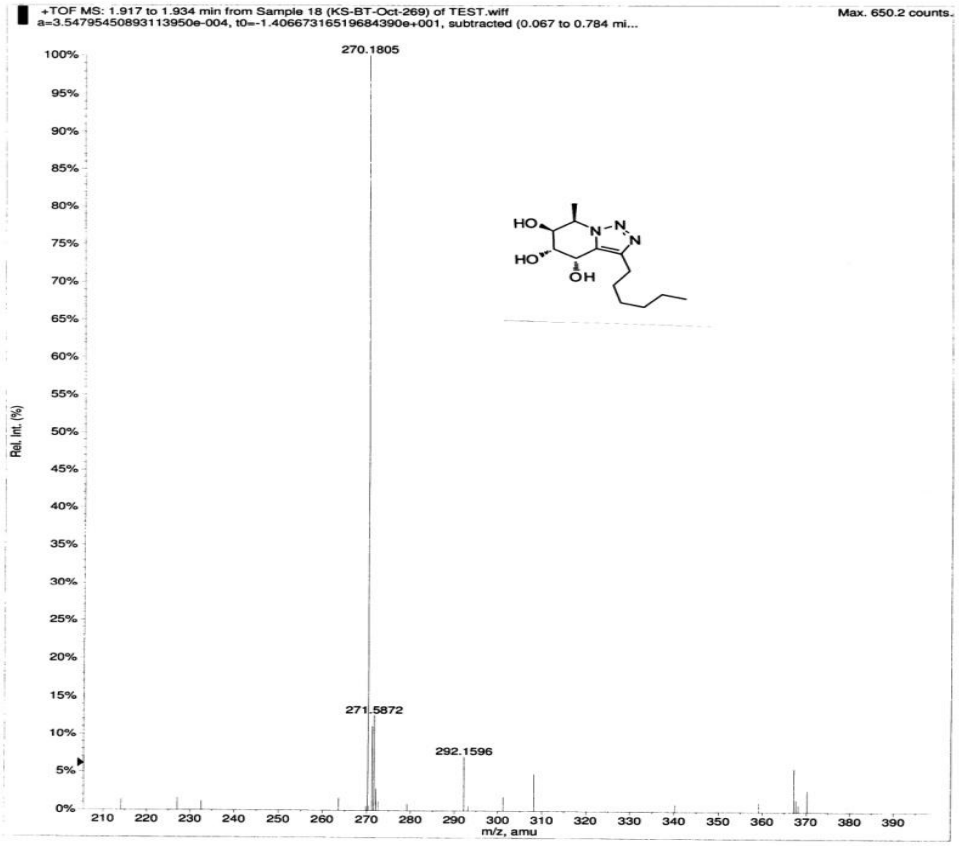

144.774
130.687
70.460
68.897
62.437
51.713
31.036
28.863
28.544
24.846
21.989
14.449
13.758


HO
HO
OH
N
N
N


150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0

Acq. File: TEST.wiff Sample ID: TuneSampleID Acq. Date: Thursday, June 20, 2013

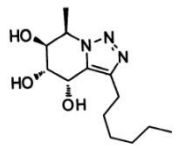
Sample Comment: KS-BT-Oct-269 Acq. Time: 19:37



Acq. File: n/a Sample ID: n/a Acq. Date: n/a

Sample Comment: n/a Acq. Time: n/a

Elemental composition calculator



Target m/z: +270.1805 amu

Tolerance: +5.0000 ppm

Result type: Elemental

Max num of results: 100

Min DBE: -0.5000 Max DBE: +50.0000

Electron state: OddAndEven

Num of charges: 0

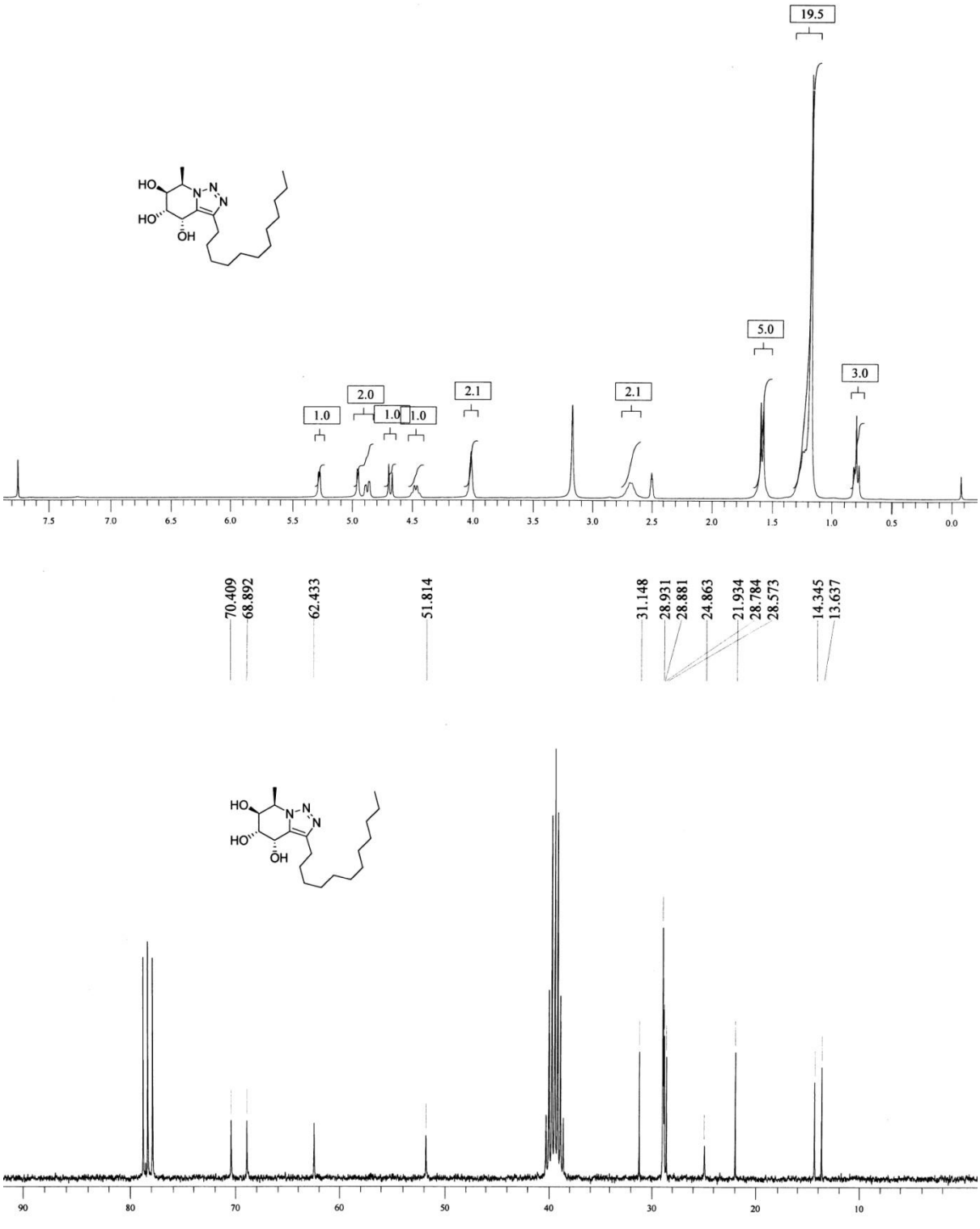
Add water: N/A

Add proton: N/A

File Name: TEST.wiff

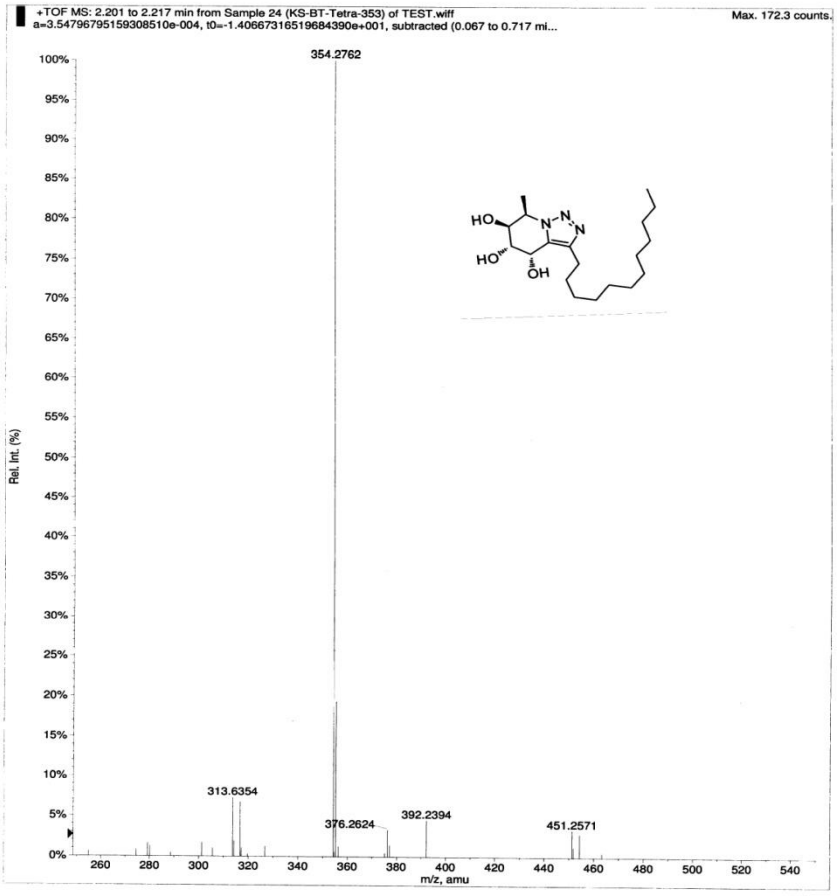
	Elements	Min Number	Max Number
1	C	0	13
2	H	0	24
3	N	0	3
4	O	0	3

	Formula	Calculated m/z (amu)	mDa Error	PPM Error	DBE
1	C13 H24 N3 O3	270.1817	-1.2669	-4.6891	3.5


HO
HO
OH
N
N
N
19.5
5.0
3.0
2.1
2.1
2.0
1.0
1.0
1.0
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
1.0
0.5
0.0
70.409
68.892
62.433
51.814
31.148
28.931
28.881
24.863
21.934
28.784
28.573
14.345
13.637
90
80
70
60
50
40
30
20
10

Acq. File: TEST.wiff Sample ID: TuneSampleID Acq. Date: Thursday, June 20, 2013

Sample Comment: KS-BT-Tetra-353 Acq. Time: 20:06



Acq. File: n/a Sample ID: n/a Acq. Date: n/a

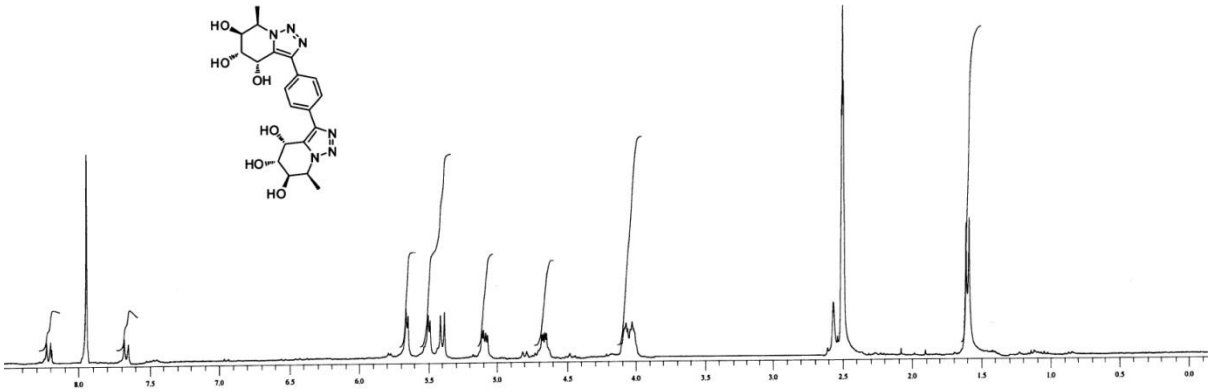
Sample Comment: n/a Acq. Time: n/a

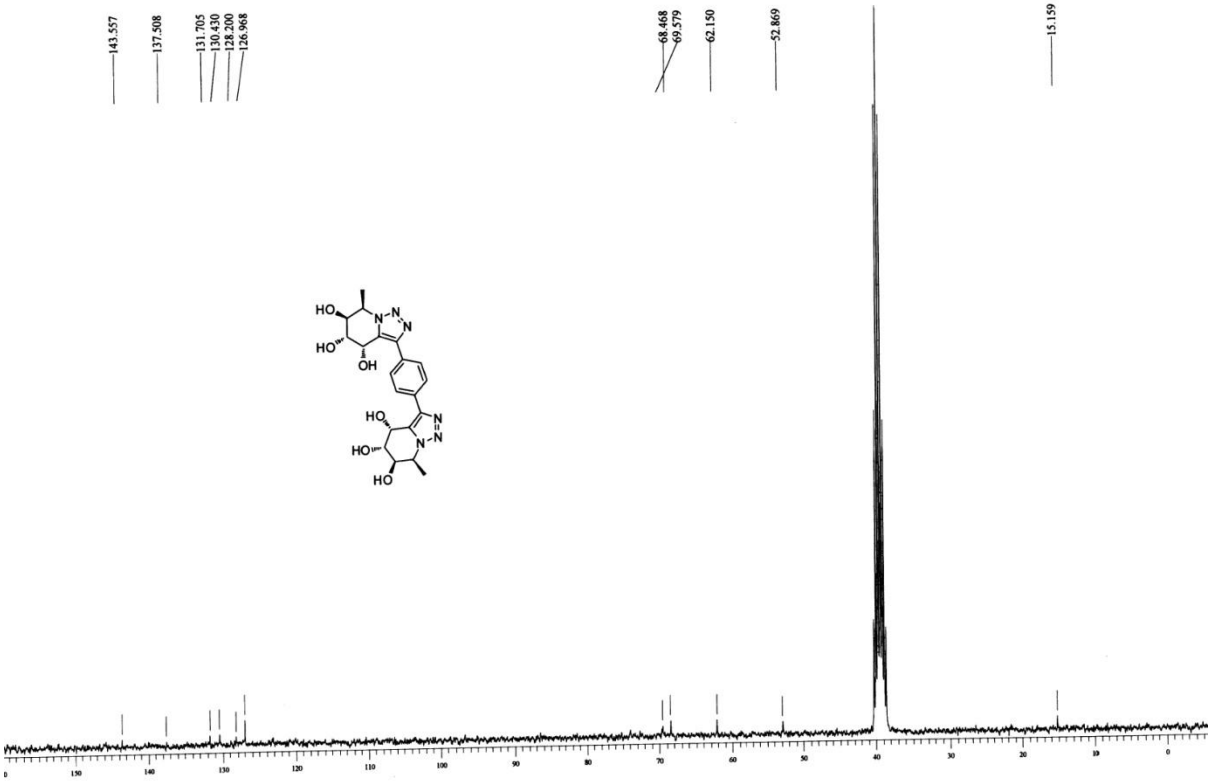
Elemental composition calculator

Target m/z: +354.2762 amu
Tolerance: +5.0000 ppm
Result type: Elemental
Max num of results: 100
Min DBE: -0.5000 Max DBE: +50.0000
Electron state: OddAndEven
Num of charges: 0
Add water: N/A
Add proton: N/A
File Name: TEST.wiff

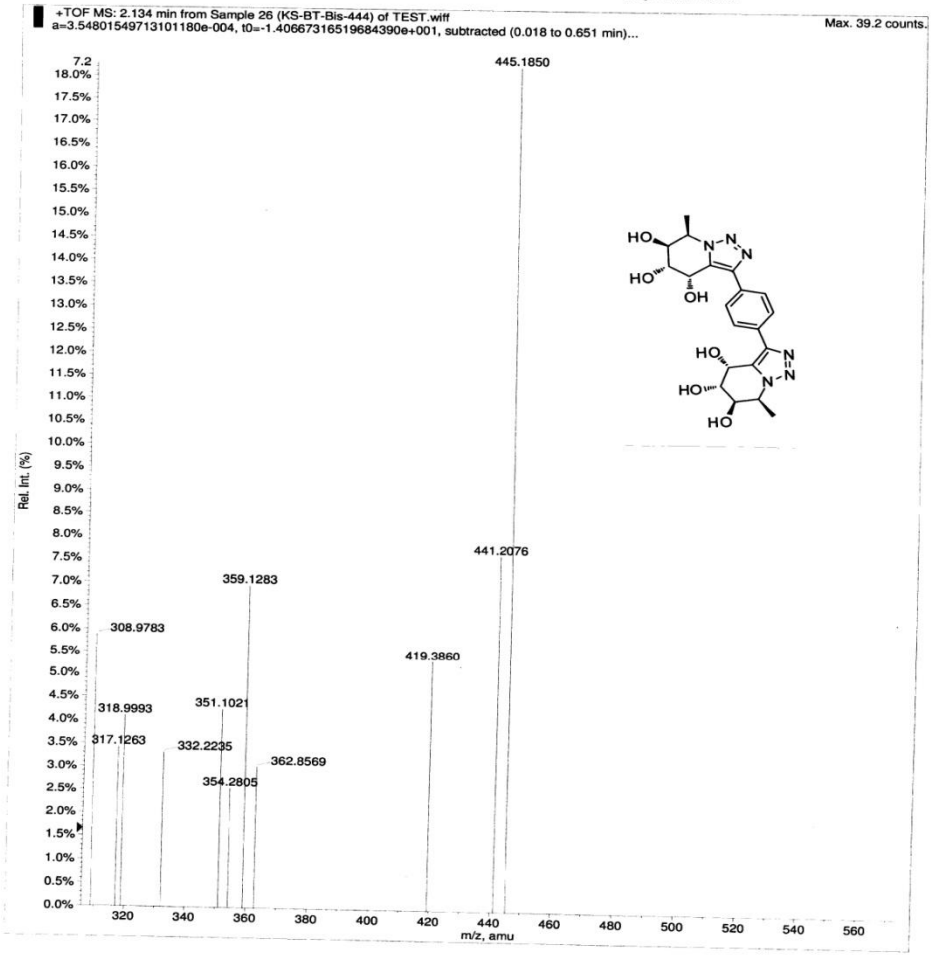
	Elements	Min Number	Max Number:
1	C	0	19
2	H	0	36
3	N	0	3
4	O	0	4

	Formula	Calculated m/z (amu)	mDa Error	PPM Error	DBE
1	C19 H36 N3 O3	354.2756	0.5326	1.5033	3.5



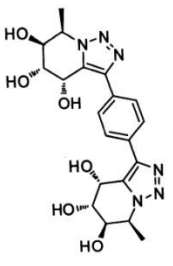

143.557
137.508
131.705
130.430
128.200
126.968
68.468
69.579
62.150
52.869
15.159

Acq. File: TEST.wiff
Sample ID: TuneSampleID
Acq. Date: Thursday, June 20, 2013
Acq. Time: 20:15
Sample Comment: KS-BT-Bis-444



Acq. File: n/a
Sample ID: n/a
Acq. Date: n/a
Acq. Time: n/a
Sample Comment: n/a

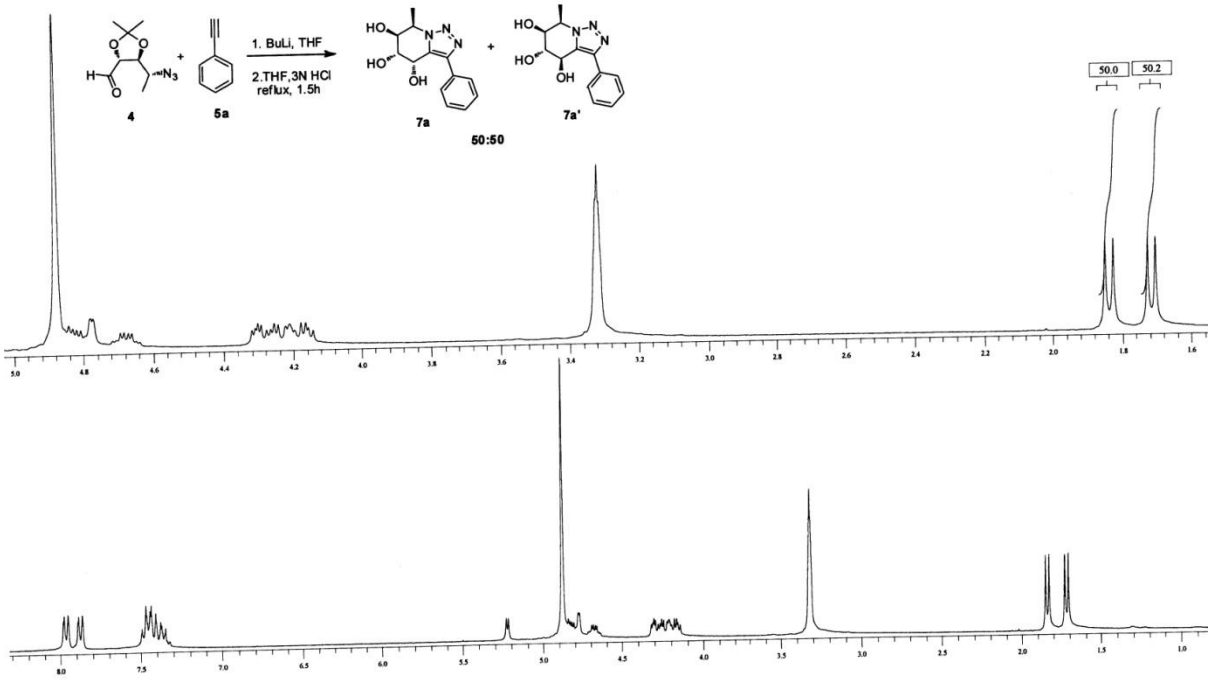
Elemental composition calculator

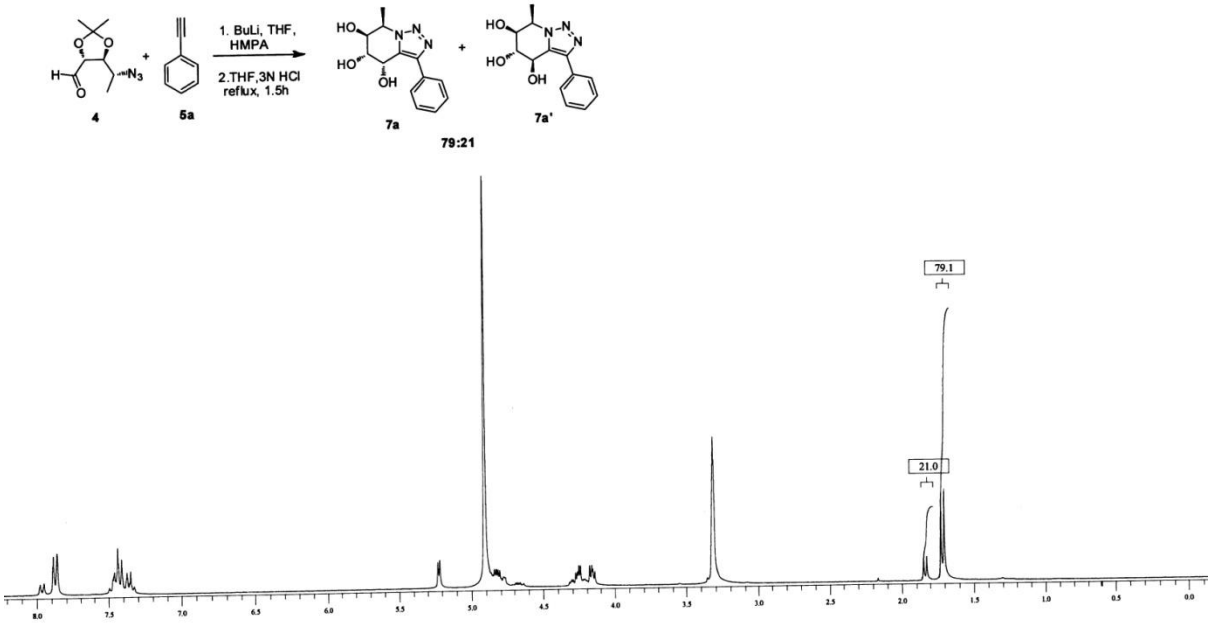


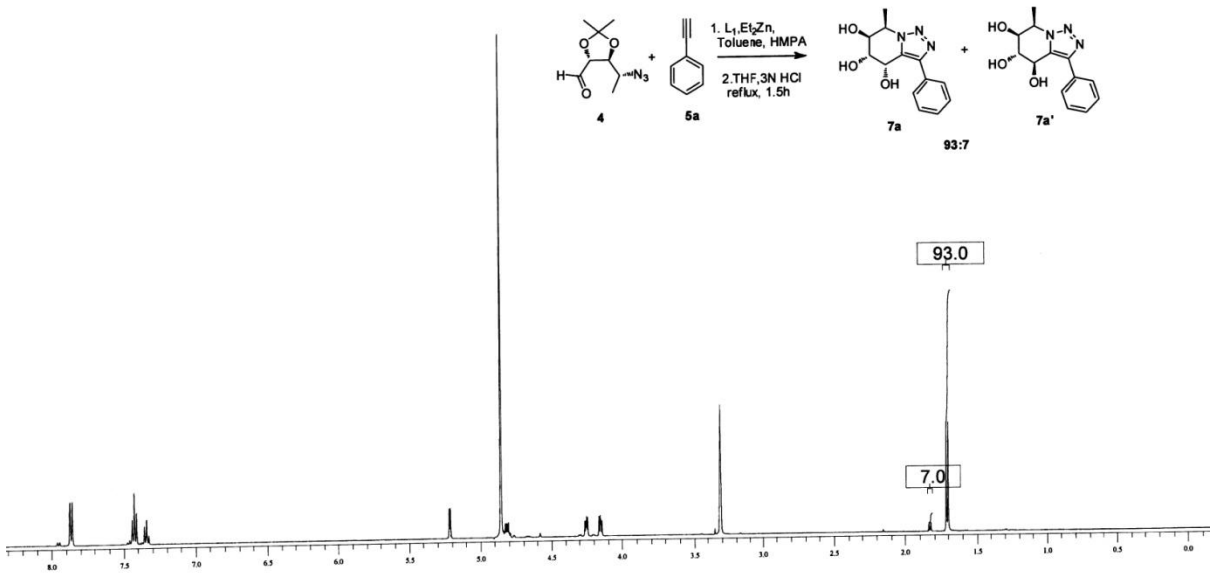
Target m/z: +445.1850 amu
Tolerance: +5.0000 ppm
Result type: Elemental
Max num of results: 100
Min DBE: -0.5000 Max DBE: +50.0000
Electron state: OddAndEven
Num of charges: 0
Add water: N/A
Add proton: N/A
File Name: TEST.wiff

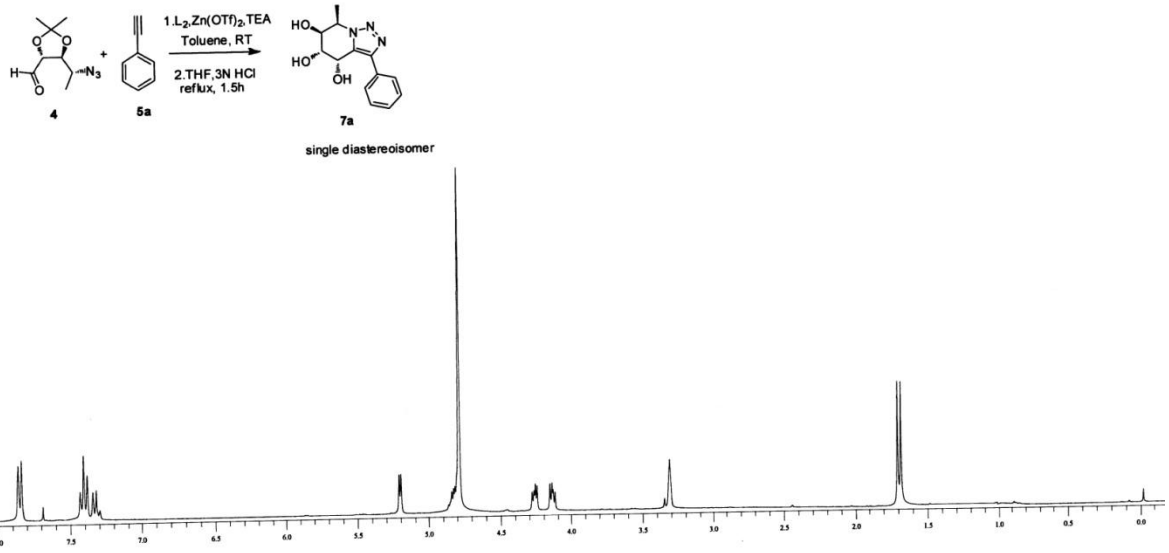
	Elements	Min Number	Max Number
1	C	0	20
2	H	0	25
3	N	0	6
4	O	0	6

	Formula	Calculated m/z (amu)	mDa Error	PPM Error	DBE
1	C20 H25 N6 O6	445.1835	1.4421	3.2393	11.5


1. BuLi, THF
2.THF,3N HCl
reflux, 1.5h
4
5a
7a
7a'
50:50
50.0
50.2


1. BuLi, THF,
HMPA
2.THF,3N HCl
reflux, 1.5h
4
5a
7a
7a'
79:21
79.1
21.0


1. L1,Et2Zn,
Toluene, HMPA
2.THF,3N HCl
reflux, 1.5h
4
5a
7a
7a'
93:7
93.0
7.0


1.L2,Zn(OTf)2,TEA
Toluene, RT
2.THF,3N HCl
reflux, 1.5h
4
5a
7a
single diastereoisomer