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Parallel Synthesis and Biological Evolution of Quinic Acid Derivatives as Immuno-suppressing Agents against T-cell Receptors

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General experimental methods:

Methanol, dichloromethane and acetone were distilled before use. All reactions were performed under an inert atmosphere with unpurified reagents and dry solvents. Analytical thin-layer chromatography (TLC) was performed using 0.25 mm silica gel coated plates. Flash chromatography was performed using the indicated solvent and silica gel 60 (230-400 mesh). All the microwave experiments were performed in CEM microwave reactor under optimized reaction conditions of power and pressure. ^{1}H NMR (300 MHz) and ^{13}C NMR (75 MHz) spectra were recorded on a 300 MHz spectrometer. Chemical shifts are reported in parts per million (ppm) on the scale from an internal standard.

Biological Methods:

Materials- Jurkat human acute T-cell leukemia line was obtained from Bioresource Collection and Research Center (BCRC, Hsinchu, Taiwan). RPMI-1640 medium was purchased from HyClone (Logan, UT, USA). Five-week-old female BALB/c mice were purchased from BioLASCO (Yilan, Taiwan). Fetal bovine serum (FBS) was purchased from Biological Industries (Haemek, Israel). Penicillin–Streptomycin–Neomycin (PSN) Antibiotic Mixture was purchased from Gibco BRL (Rockville, MD, USA). ELISA kits for human IL-2 and mouse IgG/IgE were obtained from R&D Systems (Minneapolis, MN, USA) and Bethyl (Montgomery, TX, USA), respectively. Anti-IL-2 mAb was supplied by R&D Systems (Minneapolis, MN, USA). Anti-human CD28 antibody and anti-human CD3 antibody were purchased from BioLegend (San Diego, CA, USA). 3-[4,5-Dimethylthiazol-2-yl]-2,5-diphenyl tetrazolium bromide (MTT), ovalbulmin (OVA; albumin from chicken egg white), complete Freund's adjuvant, heparin sodium salt from porcine intestinal mucosa and

3,3',5,5' tetramethylbenzidine (TMB) liquid substrate system for ELISA were purchased from Sigma (St. Louis, MO, USA). [3-(2-aminoethylamino)propyl] trimethoxy silane (3-APTMS) (99%), glutaraldehyde (25%) and bovine serum albumin (BSA) were obtained from Sigma Aldrich (St. Louis, MO, USA). Both CD28 and CD80 receptors were from ID Labs (London, ON, Canada). Cynarin was purchased from AppliChem (Darmstadt, Germany). Silicon-made tips for AFM (NanoWizard, JPK Instruments, Berlin, Germany) imaging and force measurement were ordered from Nanosensor (Neuchatel, Switzerland). Water used in this study was de-ionized and distilled.

MTT colorimetric assay- Cytotoxicity of Cyn and Cyn-1324 on T-cells were investigated by MTT colorimetric assay. Jurkat T-cells (5×10^5 cells/ml; 100 μL) were incubated with different concentrations of compounds for 24 h at 37℃. Cell solutions were then centrifuged at 200g for 10 min and the supernatants were removed. 200 μL MTT (0.5 mg/mL in culture medium) was then added and the cell solutions were incubated again for 4 h at 37 ℃. 200 μL DMSO lysis buffer was added into the cell mediums and the concentrations of dissolved MTT crystals were measured by plate reader (Dynatech, Chantilly, VA, USA) at 560 nm. The survival rate (%) were determined as follows: OD_{560nm} of testing sample (cells with derivatives)/OD_{560nm} of control (cells without derivatives)×100%.

Stimulation of T-Cells- Jurkat T-cells were maintained in a humidified atmosphere of 5% CO_2/95% air at 37 ℃ in RPMI-1640 medium including penicillin, streptomycin and 10% heat-inactivated FBS. In present experiments, Signal 1 and Signal 2 were stimulated *via* addition of anti-CD3 and anti-CD28, respectively. Flat-bottom 96-well plates were coated with 1 μg/mL of anti-CD3 for 24 h at 4 ℃. Wells including

anti-CD3 were then washed twice with phosphate-buffered saline (PBS) to remove unbound anti-CD3. Jurkat T-cells (200 μL, 2×10^6 cells/mL) with or without Cyn-1324 (PBS buffer only; control group) were then added to the wells. Cells were activated by anti-CD3 in wells for 15 min (Signal 1 stimulation). Consequently, anti-CD28 (1 μg/mL) was then added into the wells (for Signal 2 stimulation) or PBS buffer only (for Signal 1 stimulation only) for 24 h at 37 ℃. IL-2 release from stimulated T-cells (100 μL) was then measured by enzyme-linked immunosorbent assay (ELISA; see below section for detail).

IL-2 measurement- A 96-well flat-bottom plate was coated with anti-IL-2 mAb (100 μL at 4 μg/mL) in PBS (pH 7.3) at room temperature overnight. The plate was washed three times with 300 μL of PBS containing 0.05% Tween 20 (PBST) and was incubated for more than 1 h with a blocking solution containing 1% bovine serum albumin in PBS. After plates were washed again with PBS-T, 100 μL of testing sample (T-cell incubation medium) and 100 μL of biotinylated anti-IL-2 detection antibodies (400 ng/mL) were added and incubated for 2 h at room temperature. Finally, 100 μL of streptavidin horseradish peroxidase (1/2,000 dilution of a 1.25 mg/mL solution) and 100 μL of substrate solution containing H_2O_2 and tetramethylbenzidine (1:1 v/v) were added for 20 min in the dark at room temperature. The reaction was terminated by the addition of 50 μL stop solution (1.0 M H_2SO_4). The optical density of each well at 450 nm was determined using a microplate reader. Inhibited amount of IL-2 expressed from Signal 2 of T-cells was normalized by relative percentage as follows: OD_{450nm} of tested sample (OD_{450nm} of Signal 1 plus Signal 2 stimulation with Cyn-1324 minus OD_{450nm} of Signal 1 stimulation only)/ OD_{450nm} of control (OD_{450nm} of Signal 1 plus Signal 2 stimulation without Cyn-1324

minus OD_{450nm} of Signal 1 stimulation only)×100%.

Immobilization of proteins on tip and chip- Protein (CD28)-protein (CD80) interaction and interruption (by Cyn-1324) can be *in vitro* investigated by using atomic force spectroscopy. Binding force measurement is established by immobilizing one protein (CD80) on AFM tip (called afm-CD8) and another protein (CD28) on silicon chip (called chip-CD28). The procedures were followed: (a) afm-CD80. AFM tips were cleaned by oxygen plasma cleaner (PCD 150, Taiwan) under 250 mTorr and 80 W for 2 min and immersed in piranha solution (H_2SO_4 : H_2O_2 = 3 : 1 (v/v)) for 10 min to remove micro-particles, metal ions, and other organic materials. A self-assembled monolayer (SAM) on the tips was formed by incubation with 3-APTMS (1%) in ethanol for 1 h. After washing the 3-APTMS-coated tips with ethanol/water several times, 2.5% glutaraldehyde (a common cross-linking protein) solution was added and the tips were incubated for 1 h. The unbounded glutaraldehyde was removed by rinsing with water. The treated tips were then inserted into CD80 solution (100 μg/mL) and incubated overnight at 4 ^{o}C. (b) chip-CD28, SiO_2 wafer was heated in a horizontal furnace to 1,050 ^{o}C. After cutting heated wafer into pieces, similar steps (treating 3-APTMs until adding glutaraldehyde) as shown above were done on chips. These treated chips were then incubated with CD28 solution (25 μg/mL) for 30 min at room temperature. A 40 μg/mL BSA solution was added to both treated tips (afm-CD80) and chips (chip-CD28) to fill-up the vacant space on their surfaces. Washing with NaOH (0.05M) was performed before experiments.

Atomic force spectroscopy (AFM) measurement- Current unbinding force (F_u) measurement *via* AFM between chip-CD28 and afm-CD80 can be empirically

described as: $F_u = (k_B T / x_\beta) \ln(r x_\beta / k_{off} k_B T)$ (Eq. 1), where k_B, T, x_β, r and k_{off} indicate Boltzmann constant, absolute temperature (K), distance between energy potential minimum and energy barrier maximum, loading rate and dissociation rate (off rate), respectively (19-21). The loading rate (r) can be defined as the rate of force applied to the bond between proteins (e.g., r = pulling velocity × effective spring constant). Due to Eq. 1, F_u is proportional to the logarithm of r. Therefore, selection of an optimum loading rate for measurement of the unbinding forces among proteins may be essential. Upon the loading rate being determined, F_u was measured for the interaction between afm-CD80 and chip-CD28 at more than 200 different locations. To measure the blocking effect of Cyn-1324 on the binding between chip-CD28 and afm-CD80, procedures followed: (a) Cyn-1324 solution (500 μg/mL) was added to the treated chip (including chip-CD28) and incubated for 30 min. The chip including chip-CD28/Cyn-1324 was then washed with PBS buffer; (b) The treated chip (including chip-CD28/Cyn-1324) was then examined by binding with afm-CD80 on AFM tip. Both force curves and unbinding forces were recorded and calculated by using AFM.

Animal immunization and treatment- (a) *efficacy test*- BALB/c mice (twelve-week-old; n=20) were divided by four groups (5/per group): n1 group (PBS buffer only; control group), n2 group (OVA only), n3 group (OVA/Cyn-1324) and n4 group (OVA/CSA). For n2 group, mice were immunized with 50 μg OVA (in emulsion of a complete Freund's adjuvant) at day_0 and 50 μg OVA (in PBS buffer) from day_7 to day_35 by intraperitoneally injecting weekly. Concurrently, n1 (PBS only), n3 (Cyn-1324; 14mg/kg) and n4 (CSA, 50mg/kg) groups were intraperitoneally injected weekly from day_0 to day_35. After immunization, mice bloods were collected weekly from tail

veins and analyzed by ELISA (see section below for detail). (b) *toxicity test*- BALB/c mice (4 groups; 5/per group) were intraperitoneally injected three times/per week (group_1: treated with PBS after OVA-sentitization; group_2: treated with Cyn-1324 after OVA-sentilization; group_3: treated with CSA after OVA-sentilization and group_4: control group without immmunization). Doses used were increasingly changed every two-week from 50 to 400 mg/kg. Mice body weight and mortality were recorded weekly and daily, respectively.

Serum sample preparation and test- (a) *sample preparation*- Blood collected from mice above were transferred to heparinized tubes and centrifuged at 4,000g for 5 min. Supernatant serums were collected and stored at −80 ^{o}C for later experiments. For IgG detection, serum samples were melted and diluted to 10^5-fold by diluent buffer (1% BSA with 0.05% Tween 20 in PBS buffer). For IgE test, the sample medium was diluted to only 10-fold. (b) *ELISA test*- Flat-bottomed microtitre plates were coated overnight with 100 μL diluted coating antibody (1 μL anitbody plus PBS buffer to total 100 μL) to each well and washed five times with PBS containing 0.05% Tween 20 (PBS/T). Blocking was done by using block solution (1% BSA in PBS buffer). The plates were then incubated at room temperature for 1 h and washed again by PBS/T buffer. Afterward, serum samples above were added into plates and incubated at room temperature for 1 h. After washing, 100 μL HP-conjugated mouse IgG (or IgE) detection antibody (x75,000) in sample diluent was added. Plates were again incubated for 1 h and washed. 100 μL TMB substrate solution was added. After 15 min, the reaction was stopped by addition of 50 μL stop solution (1M H_2SO_4). The absorbance was determined at 450 nm. Standard curves (IgG/IgE) were obtained by using known concentrations of IgG (9–500 ng/mL) and IgE (4−250 ng/mL). By using

these standard curves, quantities of IgG and IgE from unknown samples can be obtained.

Experimental Section

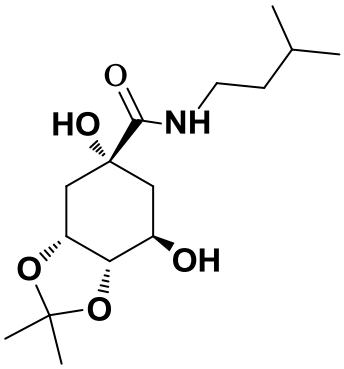
General procedure for the synthesis of (3a*R*,5*R*,7*R*,7a*S*)-5,7-dihydroxy-2,2-dimethyl-*N*-(2-phenylethyl)hexahydro-1,3-benzodioxole-5-carboxamide (3a).

Spectral data

(3a*R*,5*R*,7*R*,7a*S*)-5,7-dihydroxy-2,2-dimethyl-*N*-(2-phenylethyl)hexahydro-1,3-benzodioxole-5-carboxamide (3a)

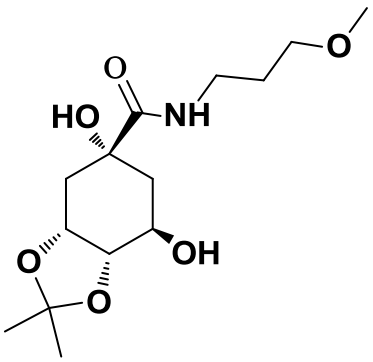
^{1}H NMR (300 MHz, $CDCl_3$) δ 7.32 – 7.19 (m, 5H), 7.07 (t, *J* = 5.6 Hz, 1H), 4.86 (s, 1H), 4.60 – 4.43 (m, 1H), 4.18 – 4.07 (m, 1H), 3.83 – 3.79 (m, 1H), 3.52 (dd, *J* = 13.2, 6.8 Hz, 2H), 3.39 (s, 1H), 2.90 – 2.73 (m, 1H), 2.44 – 2.31 (m, 1H), 2.23 – 2.11 (m, 1H), 2.05 – 1.88 (m, 2H), 1.48 (s, 3H), 1.33 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 176.6, 138.5, 128.8, 128.7, 126.7, 108.7, 76.1, 72.9, 72.1, 65.9, 40.5, 37.0, 35.6, 34.4, 27.1, 24.4; MS (EI) 335.2; HRMS (EI) calcd. for $C_{18}H_{25}NO_5$ 335.1733; found 335.1729; IR (cm^{-1}, neat) 3359, 1644.

(3a*R*,5*R*,7*R*,7a*S*)-5,7-dihydroxy-2,2-dimethyl-*N*-(3-methylbutyl)hexahydro-1,3-benzodioxole-5-carboxamide (3b)



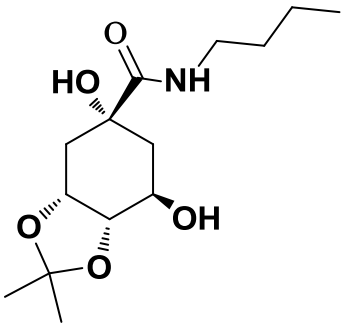
^{1}H NMR (300 MHz, $CDCl_3$) δ 6.97 (s, 1H), 4.97 (s, 1H), 4.63 – 4.34 (m, 1H), 4.10 (d, *J* = 3.9 Hz, 1H), 3.78 (s, 1H), 3.46 (s, 1H), 3.31 – 3.15 (m, 2H), 2.38 (dd, *J* = 15.6, 2.1 Hz, 1H), 2.18 (dd, *J* = 14.8, 5.1 Hz, 1H), 1.98 (ddd, *J* = 14.6, 8.9, 2.7 Hz, 2H), 1.65 – 1.49 (m, 1H), 1.46 (s, 3H), 1.38 (dd, *J* = 14.5, 7.2 Hz, 2H), 1.30 (s, 3H), 0.87 (d, *J* = 6.6 Hz, 6H); ^{13}C NMR (75 MHz, $CDCl_3$); δ 176.4, 108.6, 76.1, 72.8, 72.1, 65.9, 38.2, 37.7, 37.1, 34.4, 27.1, 25.9, 24.4, 22.5; MS (EI) 301.3; HRMS (EI) calcd. for $C_{15}H_{27}NO_5$ 301.1889; found 301.1887; IR (cm^{-1}, neat) 3399, 1646.

(3a*R*,5*R*,7*R*,7a*S*)-5,7-dihydroxy-*N*-(3-methoxypropyl)-2,2-dimethylhexahydro-1,3-benzodioxole-5-carboxamide (3c)



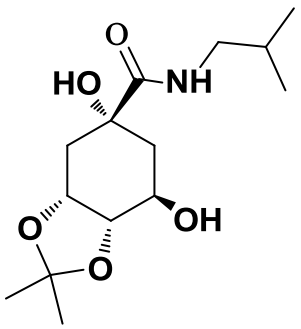
^{1}H NMR (300 MHz, $CDCl_3$) δ 7.43 (s, 1H), 4.96 (s, 1H), 4.64 – 4.52 (m, 1H), 4.21 – 4.10 (m, 1H), 3.80 (s, 1H), 3.47 (t, *J* = 5.8 Hz, 2H), 3.43 – 3.35 (m, 2H), 3.34 (s, 3H), 2.43 (dd, *J* = 15.6, 2.4 Hz, 1H), 2.26 (ddd, *J* = 15.0, 5.1, 1.5 Hz, 1H), 2.03 (ddd, *J* = 14.9, 9.0, 3.0 Hz, 2H), 1.84 – 1.76 (m, 2H), 1.50 (s, 3H), 1.34 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 176.6, 108.7, 76.0, 72.8, 72.1, 71.3, 65.9, 58.9, 37.8, 37.1, 34.4, 29.0, 27.1, 24.4; MS (EI) 303.2; HRMS (EI) calcd. for $C_{14}H_{25}NO_6$ 303.1682; found 303.1687; IR (cm^{-1}, neat) 3399, 1646.

(3a*R*,5*R*,7*R*,7a*S*)-*N*-butyl-5,7-dihydroxy-2,2-dimethylhexahydro-1,3-benzodioxole-5-carboxamide (3d)



^{1}H NMR (300 MHz, $CDCl_3$) δ 7.00 (s, 1H), 4.95 (d, *J* = 3.7 Hz, 1H), 4.58 – 4.49 (m, 1H), 4.15 – 4.07 (m, 1H), 3.83 – 3.75 (m, 1H), 3.43 (s, 1H), 3.34 – 3.14 (m, 2H), 2.40 (dd, *J* = 15.6, 2.5 Hz, 1H), 2.21 (ddd, *J* = 15.0, 5.3, 1.4 Hz, 1H), 1.99 (ddd, *J* = 14.8, 8.6, 3.1 Hz, 2H), 1.56 – 1.40 (m, 2H), 1.47 (s, 3H), 1.39 – 1.23 (m, 2H), 1.31 (s, 3H), 0.89 (t, *J* = 7.3 Hz, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 176.6, 108.7, 76.1, 72.8, 72.1, 65.9, 39.2, 37.1, 34.5, 31.5, 27.1, 24.4, 20.0, 13.8; MS (EI) 287.2; HRMS (EI) calcd. for $C_{14}H_{25}NO_5$ 287.1733; found 287.1725; IR (cm^{-1}, neat) 3739, 3370, 1646.

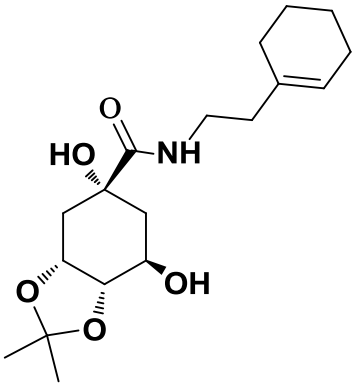
(3a*R*,5*R*,7*R*,7a*S*)-5,7-dihydroxy-2,2-dimethyl-*N*-(2-methylpropyl)hexahydro-1,3-benzodioxole-5-carboxamide (3e)



Chemical Formula: $C_{14}H_{25}NO_5$
Exact Mass: 287.1733

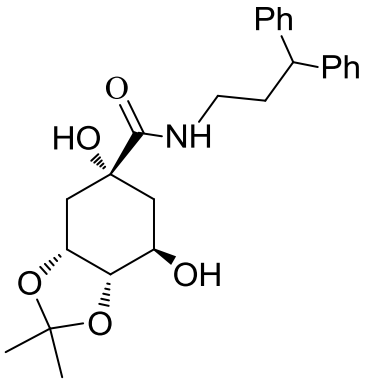
^{1}H NMR (300 MHz, $CDCl_3$) δ 7.06 (s, 1H), 4.63 – 4.52 (m, 1H), 4.15 (ddd, *J* = 6.9, 2.9, 1.5 Hz, 1H), 3.86 – 3.75 (m, 1H), 3.10 (td, *J* = 6.7, 2.1 Hz, 2H), 2.44 (dd, *J* = 15.6, 2.5 Hz, 1H), 2.25 (ddd, *J* = 15.0, 5.1, 1.5 Hz, 1H), 2.03 (ddd, *J* = 14.9, 7.3, 3.1 Hz, 2H), 1.91 – 1.68 (m, 1H), 1.49 (s, 1H), 1.34 (s, 1H), 0.91 (d, *J* = 6.7 Hz, 6H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 176.7, 108.7, 76.0, 72.9, 72.1, 65.8, 46.7, 37.1, 34.5, 28.6, 27.1, 24.4, 20.1; MS (EI) 287.2; HRMS (EI) calcd. for $C_{14}H_{25}NO_5$ 287.1733; found 287.1726; IR (cm^{-1}, neat) 3737, 3399, 1646.

(3a*R*,5*R*,7*R*,7a*S*)-*N*-[2-(cyclohex-1-en-1-yl)ethyl]-5,7-dihydroxy-2,2-dimethylhexahydro-1,3-benzodioxole-5-carboxamide (3f)



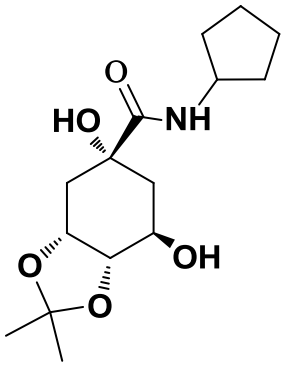
^{1}H NMR (300 MHz, $CDCl_3$) δ 6.97 (s, 1H), 5.45 (s, 1H), 4.96 (s, 1H), 4.62 – 4.52 (m, 1H), 4.14 (d, *J* = 6.9 Hz, 1H), 3.80 (s, 1H), 3.45 – 3.23 (m, 3H), 2.42 (dd, *J* = 15.6, 2.1 Hz, 1H), 2.24 (dd, *J* = 15.0, 5.0 Hz, 1H), 2.14 (t, *J* = 6.7 Hz, 2H), 2.09 – 1.82 (m, 6H), 1.69 – 1.50 (m, 4H), 1.49 (s, 3H), 1.33 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 176.5, 134.3, 124.0, 108.7, 76.0, 72.8, 72.1, 65.8, 37.6, 37.1, 37.0, 34.5, 27.9, 27.1, 25.3, 24.4, 22.9, 22.4; MS (EI) 339.2; HRMS (EI) calcd. for $C_{18}H_{29}NO_5$ 339.2046; found 339.2055; IR (cm^{-1}, neat) 3739, 3396, 1646.

(3a*R*,5*R*,7*R*,7a*S*)-*N*-(3,3-diphenylpropyl)-5,7-dihydroxy-2,2-dimethylhexahydro-1,3-benzodioxole-5-carboxamide (3g)



^{1}H NMR (300 MHz, $CDCl_3$) δ 7.40 – 7.15 (m, 10H), 7.09 (t, *J* = 5.9 Hz, 1H), 4.59 – 4.51 (m, 1H), 4.15 (dd, *J* = 6.8, 2.2 Hz, 1H), 4.02 – 3.90 (m, 1H), 3.86 (dt, *J* = 5.9, 3.1 Hz, 1H), 3.24 (dd, *J* = 14.2, 6.3 Hz, 2H), 2.48 – 2.16 (m, 4H), 2.10 – 1.96 (m, 2H), 1.52 (s, 3H), 1.35 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 176.5, 143.9, 128.6, 127.7, 126.4, 108.6, 76.2, 72.9, 72.1, 65.9, 48.9, 38.2, 37.0, 35.0, 34.3, 27.1, 24.4; MS (EI) 425.0; HRMS (EI) calcd. for $C_{25}H_{31}NO_5$ 425.2202; found 425.2202; IR (cm^{-1}, neat) 3401, 1648.

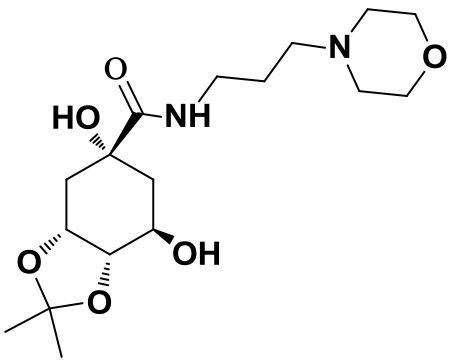
(3a*R*,5*R*,7*R*,7a*S*)-*N*-cyclopentyl-5,7-dihydroxy-2,2-dimethylhexahydro-1,3-benzo dioxole-5-carboxamide (3h)



^{1}H NMR (300 MHz, $CDCl_3$) δ 6.92 (d, *J* = 6.5 Hz, 1H), 4.64 – 4.52 (m, 1H), 4.28 – 4.01 (m, 2H), 3.84 – 3.76 (m, 1H), 2.44 (dd, *J* = 15.6, 2.5 Hz, 1H), 2.26 (ddd, *J* = 15.0,

5.0, 1.6 Hz, 1H), 2.07 – 1.94 (m, 4H), 1.75 – 1.52 (m, 4H), 1.50 (s, 3H), 1.47 – 1.39 (m, 2H), 1.34 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 176.2, 108.7, 75.9, 72.6, 72.1, 65.8, 51.1, 37.0, 34.4, 33.2, 33.1, 27.1, 24.4, 23.9; MS (EI) 299.2; HRMS (EI) calcd. for $C_{15}H_{25}NO_5$ 299.1733; found 299.1735; IR (cm^{-1}, neat) 3743, 3401, 1648.

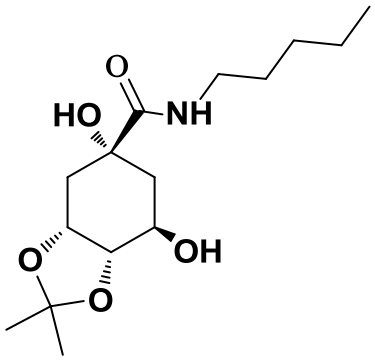
(3a*R*,5*R*,7*R*,7a*S*)-5,7-dihydroxy-2,2-dimethyl-*N*-[3-(morpholin-4-yl)propyl]hexahydro-1,3-benzodioxole-5-carboxamide (3i)



^{1}H NMR (300 MHz, $CDCl_3$) δ 8.10 (s, 1H), 4.62 – 4.45 (m, 1H), 4.11 (dd, *J* = 6.6, 1.7 Hz, 1H), 3.79 – 3.75 (m, 1H), 3.75 – 3.62 (m, 4H), 3.36 – 3.30 (m, 2H), 2.53 – 2.30 (m, 7H), 2.20 (dd, *J* = 14.9, 5.3 Hz, 1H), 1.97 (ddd, *J* = 14.8, 8.4, 3.0 Hz, 2H), 1.77-1.59 (m, 2H), 1.48 (s, 3H), 1.31 (s, 3H); ^{3}C NMR (75 MHz, $CDCl_3$) δ 176.5, 108.6, 76.0, 72.8, 72.1, 66.8, 65.8, 57.8, 53.8, 39.3, 37.2, 34.6, 27.1, 24.9, 24.4; MS

(ESI) 359; $(M+H)^+$ HRMS (ESI) calcd. for $C_{17}H_{30}N_2O_6$ 358.2104; found 359.2184 (M+H);IR (cm^{-1}, neat) 3741, 3318, 1637.

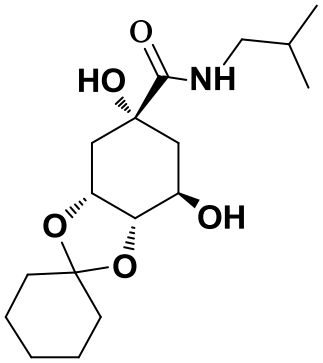
(3a*R*,5*R*,7*R*,7a*S*)-5,7-dihydroxy-2,2-dimethyl-*N*-pentylhexahydro-1,3-benzodioxole-5-carboxamide (3j)



^{1}H NMR (300 MHz, $CDCl_3$) δ 7.00 (s, 1H), 4.62 – 4.48 (m, 1H), 4.24 – 4.01 (m, 1H), 3.81 – 3.77 (m, 1H), 3.37 – 3.13 (m, 2H), 2.41 (dd, *J* = 15.6, 2.5 Hz, 1H), 2.32 – 2.12 (m, 1H), 2.00 (ddd, *J* = 14.9, 8.4, 3.1 Hz, 2H), 1.60 – 1.40 (m, 2H), 1.48 (s, 3H), 1.33 (s, 3H), 1.40 – 1.22 (m, 4H), 0.87 (t, *J* = 6.8 Hz, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 176.6, 108.7, 76.1, 72.8, 72.1, 65.9, 39.4, 37.1, 34.5, 29.2, 29.0, 27.1, 24.4, 22.4, 14.0; MS (EI) 301.2; HRMS (EI) calcd. for $C_{15}H_{27}NO_5$ 301.1889; found 301.1897; IR (cm^{-1}, neat) 3739, 3399, 1646.

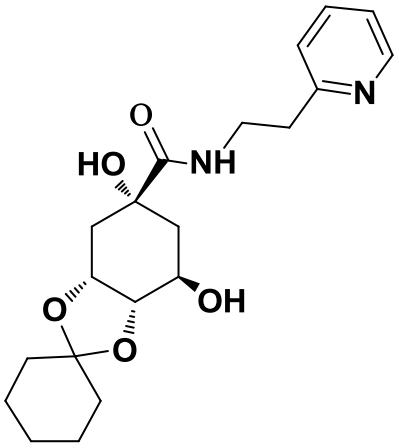
(3a*R*,5*R*,7*R*,7a*S*)-5,7-dihydroxy-*N*-(2-methylpropyl)hexahydrospiro[1,3-benzodio

xole-2,1'-cyclohexane]-5-carboxamide (3k)



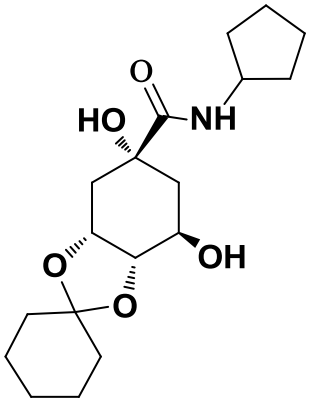
^{1}H NMR (300 MHz, $CDCl_3$) δ 7.06 (s, 1H), 4.82 (s, 1H), 4.61 – 4.43 (m, 1H), 4.18 – 4.07 (m, 1H), 3.86 – 3.72 (m, 1H), 3.53 (s, 1H), 3.06 (t, *J* = 6.5 Hz, 2H), 2.38 (dd, *J* = 15.6, 2.5 Hz, 1H), 2.18 (ddd, *J* = 14.7, 5.6, 1.0 Hz, 1H), 2.01 (ddd, *J* = 12.1, 11.0, 3.1 Hz, 2H), 1.84 – 1.70 (m, 1H), 1.70 – 1.26 (m, 10H), 0.88 (d, *J* = 6.7 Hz, 6H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 176.5, 109.4, 75.9, 73.1, 71.8, 66.2, 46.6, 37.2, 37.0, 34.5, 33.6, 28.5, 25.1, 24.0, 23.5, 20.0; MS (ESI) 328 (M+H); HRMS (ESI) calcd. for $C_{17}H_{29}NO_5$ 327.2046; found 328.2126 (M+H); IR (cm^{-1}, neat) 3741, 3401, 1648.

(3a*R*,5*R*,7*R*,7a*S*)-5,7-dihydroxy-*N*-[2-(pyridin-2-yl)ethyl]hexahydrospiro[1,3-ben zodioxole-2,1'-cyclohexane]-5-carboxamide (3l)



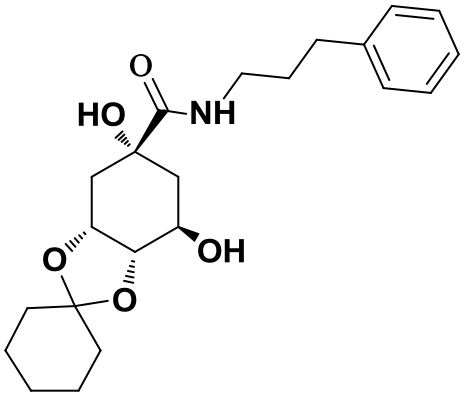
^{1}H NMR (300 MHz, $CDCl_3$) δ 8.52 (d, *J* = 4.7 Hz, 1H), 7.66 (t, *J* = 5.4 Hz, 1H), 7.60 (td, *J* = 7.7, 1.8 Hz, 1H), 7.16 – 7.11 (m, 2H), 4.87 (s, 1H), 4.56 – 4.43 (m, 1H), 4.15 – 4.06 (m, 1H), 3.81 (s, 1H), 3.73 – 3.60 (m, 2H), 3.49 (s, 1H), 3.00 (t, *J* = 6.5 Hz, 2H), 2.36 (dd, *J* = 15.6, 2.5 Hz, 1H), 2.17 (ddd, *J* = 14.8, 5.6, 1.2 Hz, 1H), 2.09 – 1.92 (m, 2H), 1.73 – 1.29 (m, 10H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 176.6, 159.1, 149.4, 136.7, 123.5, 121.8, 109.4, 75.9, 73.0, 71.8, 66.2, 38.7, 37.2, 37.1, 37.0, 34.4, 33.7, 25.1, 24.1, 23.6; MS (EI) 376.0; HRMS (EI) calcd. for $C_{20}H_{28}N_2O_5$ 376.1998; found 376.2001; IR (cm^{-1}, neat) 3737, 3338, 1648.

(3a*R*,5*R*,7*R*,7a*S*)-*N*-cyclopentyl-5,7-dihydroxyhexahydrospiro[1,3-benzodioxole-2,1'-cyclohexane]-5-carboxamide (3m)



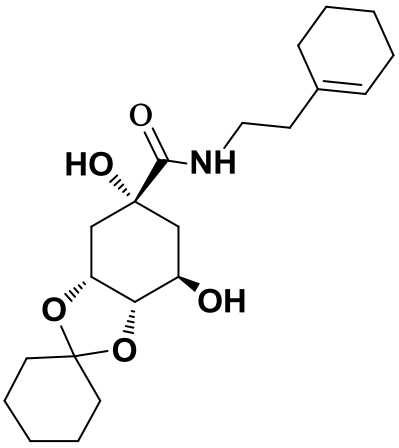
^{1}H NMR (300 MHz, $CDCl_3$) δ 6.90 (d, *J* = 7.6 Hz, 1H), 4.83 (s, 1H), 4.60 – 4.47 (m, 1H), 4.20 – 4.02 (m, 2H), 3.81 – 3.78 (m, 1H), 3.48 (s, 1H), 2.39 (dd, *J* = 15.6, 2.5 Hz, 1H), 2.19 (ddd, *J* = 14.9, 5.4, 1.2 Hz, 1H), 2.07 – 1.84 (m, 4H), 1.76 – 1.28 (m, 16H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 176.0, 109.4, 75.8, 72.8, 71.8, 66.1, 51.0, 37.1, 37.0, 34.4, 33.6, 33.1, 33.1, 25.1, 24.1, 23.8, 23.5; MS (EI) 339.0; HRMS (EI) calcd. for $C_{18}H_{29}NO_5$ 339.2046; found 339.2044; IR (cm^{-1}, neat) 3743, 3401, 1644.

(3a*R*,5*R*,7*R*,7a*S*)-5,7-dihydroxy-*N*-(3-phenylpropyl)hexahydrospiro[1,3-benzodioxole-2,1'-cyclohexane]-5-carboxamide (3n)



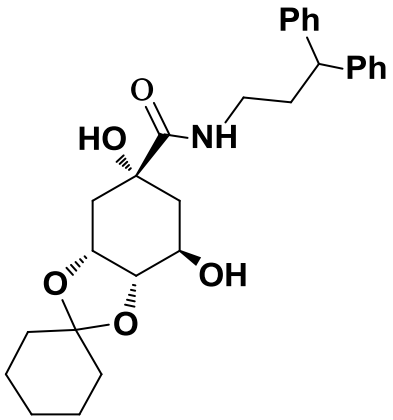
^{1}H NMR (300 MHz, $CDCl_3$) δ 7.30 – 7.15 (m, 5H), 7.06 (t, *J* = 5.4 Hz, 1H), 4.80 (s, 1H), 4.62 – 4.45 (m, 1H), 4.23 – 4.06 (m, 1H), 3.86 – 3.82 (m, 1H), 3.51 (s, 1H), 3.29 (dd, *J* = 13.5, 6.8 Hz, 2H), 2.64 (t, *J* = 7.7 Hz, 2H), 2.41 (dd, *J* = 15.6, 2.4 Hz, 1H), 2.21 (dd, *J* = 14.8, 5.1 Hz, 1H), 2.03 (ddd, *J* = 14.6, 8.2, 3.0 Hz, 2H), 1.94 – 1.79 (m, 2H), 1.79 – 1.32 (m, 10H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 176.5, 141.2, 128.5, 128.4, 126.1, 109.4, 75.9, 73.0, 71.8, 66.2, 38.9, 37.2, 37.0, 34.4, 33.6, 33.1, 31.1, 25.1, 24.0, 23.5; MS (EI) 389.0; HRMS (EI) calcd. for $C_{22}H_{31}NO_5$ 389.2202; found 389.2201; IR (cm^{-1}, neat) 3743, 3401, 1646.

(3a*R*,5*R*,7*R*,7a*S*)-*N*-[2-(cyclohex-1-en-1-yl)ethyl]-5,7-dihydroxyhexahydrospiro[1,3-benzodioxole-2,1'-cyclohexane]-5-carboxamide (3o)



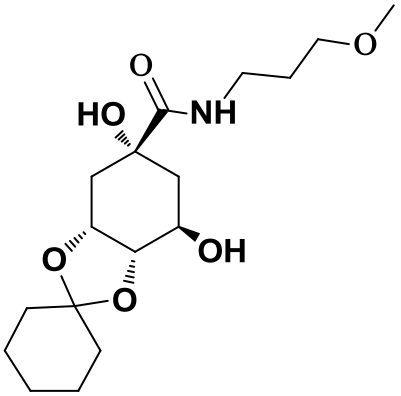
^{1}H NMR (300 MHz, $CDCl_3$) δ 6.96 (t, *J* = 5.3 Hz, 1H), 5.42 (s, 1H), 4.86 (s, 1H), 4.61 – 4.38 (m, 1H), 4.21 – 4.02 (m, 1H), 3.88 – 3.74 (m, 1H), 3.41 (d, *J* = 14.3 Hz, 1H), 3.38 – 3.24 (m, 2H), 2.37 (dd, *J* = 15.6, 2.5 Hz, 1H), 2.23 – 2.06 (m, 3H), 2.06 – 1.83 (m, 6H), 1.72 – 1.28 (m, 14H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 176.4, 134.2, 123.9, 109.4, 75.8, 72.9, 71.7, 66.1, 37.6, 37.1, 37.0, 36.9, 34.5, 33.6, 27.9, 25.3, 25.1, 24.0, 23.5, 22.9, 22.4; MS (EI) 379.0; HRMS (EI) calcd. for $C_{21}H_{33}NO_5$ 379.2359; found 379.2361; IR (cm^{-1}, neat) 3739, 3394, 1646.

(3a*R*,5*R*,7*R*,7a*S*)-*N*-(3,3-diphenylpropyl)-5,7-dihydroxyhexahydrospiro[1,3-benzodioxole-2,1'-cyclohexane]-5-carboxamide (3p)



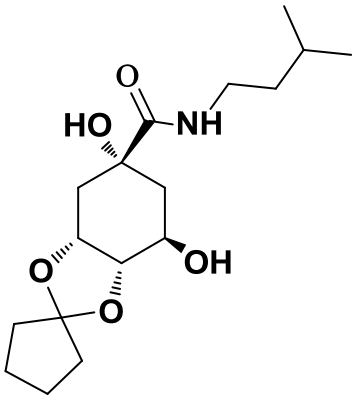
^{1}H NMR (300 MHz, $CDCl_3$) δ 7.29 – 7.02 (m, 10H), 6.95 (t, *J* = 5.7 Hz, 1H), 4.71 (s, 1H), 4.51 – 4.40 (m, 1H), 4.05 (dd, *J* = 6.3, 2.9 Hz, 1H), 3.86 (t, *J* = 7.8 Hz, 1H), 3.80 – 3.71 (m, 1H), 3.40 (s, 1H), 3.14 (dd, *J* = 13.9, 6.5 Hz, 2H), 2.40 – 2.05 (m, 4H), 2.01 – 1.83 (m, 2H), 1.61 – 1.32 (m, 10H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 176.5, 144.0, 128.7, 127.8, 126.5, 109.4, 75.9, 73.0, 71.8, 66.2, 49.0, 38.2, 37.2, 37.0, 35.1, 34.4, 33.6, 25.1, 24.0, 23.5; MS (EI) 465.0; HRMS (EI) calcd. for $C_{28}H_{35}NO_5$ 465.2515; found 465.2313; IR (cm^{-1}, neat) 3401, 1644.

(3a*R*,5*R*,7*R*,7a*S*)-5,7-dihydroxy-*N*-(3-methoxypropyl)hexahydrospiro[1,3-benzodioxole-2,1'-cyclohexane]-5-carboxamide (3q)



^{1}H NMR (300 MHz, $CDCl_3$) δ 7.40 (s, 1H), 4.85 (s, 1H), 4.59 – 4.49 (m, 1H), 4.12 (dd, *J* = 6.6, 1.9 Hz, 1H), 3.81 (s, 1H), 3.49 – 3.41 (m, 2H), 3.41 – 3.34 (m, 2H), 3.32 (s, 3H), 2.40 (dd, *J* = 15.6, 2.2 Hz, 1H), 2.21 (dd, *J* = 14.9, 4.3 Hz, 1H), 2.10 – 1.95 (m, 2H), 1.84 – 1.72 (m, 2H), 1.71 – 1.32 (m, 10H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 176.5, 109.4, 75.8, 72.9, 71.7, 71.3, 66.1, 58.9, 37.8, 37.2, 37.0, 34.5, 33.6, 29.1, 25.1, 24.1, 23.6; MS (EI) 343.2; HRMS (EI) calcd. for $C_{17}H_{29}NO_6$ 343.1995; found 343.1990; IR (cm^{-1}, neat) 3365, 1646.

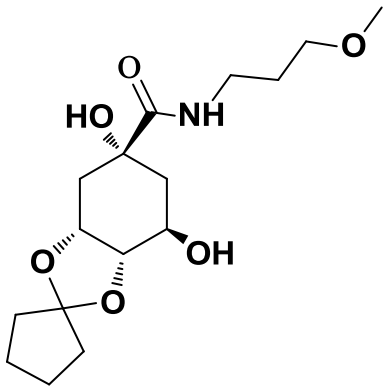
(3a*R*,5*R*,7*R*,7a*S*)-5,7-dihydroxy-*N*-(3-methylbutyl)hexahydrospiro[1,3-benzodioxole-2,1'-cyclopentane]-5-carboxamide (3r)



^{1}H NMR (300 MHz, $CDCl_3$) δ 6.95 (s, 1H), 4.84 (s, 1H), 4.41 (s, 1H), 4.07 (s, 1H), 3.80 (s, 1H), 3.39 (s, 1H), 3.27 (d, *J* = 7.1 Hz, 2H), 2.42 (d, *J* = 15.5 Hz, 1H), 2.29 – 2.13 (m, 1H), 2.11 – 1.51 (m, 11H), 1.48 – 1.34 (m, 2H), 0.91 (d, *J* = 6.4 Hz, 6H); ^{13}C NMR (75 MHz, $CDCl_3$)

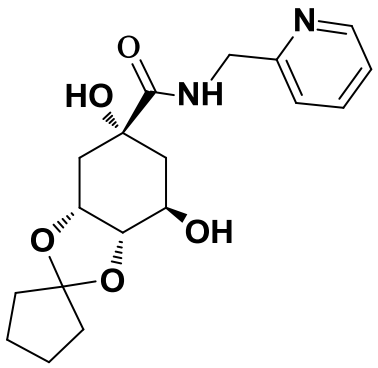
δ 176.4, 118.8, 75.9, 73.1, 72.6, 66.0, 38.3, 37.8, 37.2, 36.7, 36.2, 34.4, 26.0, 24.1, 23.2, 22.5; MS (EI) 327.0; HRMS (EI) calcd. for $C_{17}H_{29}NO_5$ 327.2046; found 327.2048; IR (cm^{-1}, neat) 3401, 1646.

(3a*R*,5*R*,7*R*,7a*S*)-5,7-dihydroxy-*N*-(3-methoxypropyl)hexahydrospiro[1,3-benzodioxole-2,1'-cyclopentane]-5-carboxamide (3s)

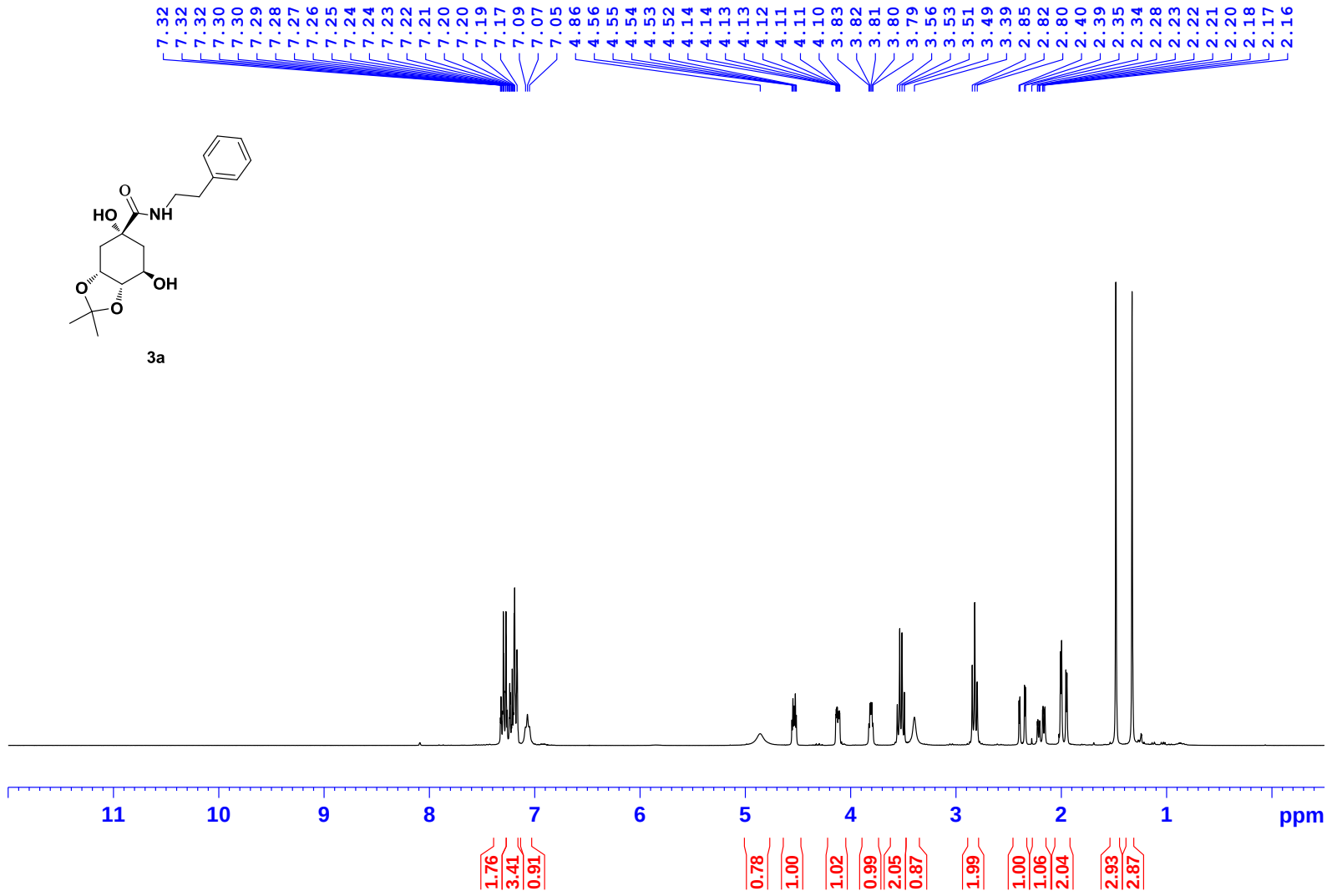


^{1}H NMR (300 MHz, $CDCl_3$) δ 7.41 (s, 1H), 4.86 (d, *J* = 3.1 Hz, 1H), 4.45 – 4.39 (m, 1H), 4.06 (dd, *J* = 6.6, 3.0 Hz, 1H), 3.80 (d, *J* = 2.3 Hz, 1H), 3.45 (t, *J* = 5.8 Hz, 2H), 3.40 – 3.34 (m, 2H), 3.32 (s, 1H), 2.41 (dd, *J* = 15.6, 2.2 Hz, 1H), 2.21 (dd, *J* = 14.8, 5.6 Hz, 1H), 2.09 – 1.91 (m, 3H), 1.90 – 1.59 (m, 9H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 176.4, 118.8, 76.0, 73.1, 72.6, 71.3, 66.0, 58.9, 37.8, 37.3, 36.7, 36.2, 34.4, 29.1, 24.0, 23.2; MS (EI) 329.2; HRMS (EI) calcd. for $C_{16}H_{27}NO_6$ 329.1838; found 329.1829; IR (cm^{-1}, neat) 3725, 3363, 1648.

(3a*R*,5*R*,7*R*,7a*S*)-5,7-dihydroxy-*N*-(pyridin-2-ylmethyl)hexahydrospiro[1,3-benzodioxole-2,1'-cyclopentane]-5-carboxamide (3t)



^{1}H NMR (300 MHz, $CDCl_3$) δ 8.53 (d, *J* = 4.6 Hz, 1H), 8.09 (s, 1H), 7.65 (td, *J* = 7.7, 1.7 Hz, 1H), 7.34 – 7.11 (m, 2H), 4.76 (s, 1H), 4.59 – 4.53 (m, 2H), 4.43 – 4.39 (m, 1H), 4.06 (dd, *J* = 6.3, 3.9 Hz, 1H), 3.88 – 3.83 (m, 1H), 3.69 (s, 1H), 2.43 (dd, *J* = 15.6, 2.7 Hz, 1H), 2.22 (dd, *J* = 14.7, 6.4 Hz, 1H), 2.08 (ddd, *J* = 14.7, 13.6, 2.7 Hz, 2H), 2.00 – 1.57 (m, 8H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 176.5, 156.2, 149.3, 137.0, 122.6, 122.0, 118.8, 76.5, 73.6, 72.9, 66.3, 44.4, 37.6, 36.8, 36.4, 34.4, 24.0, 23.2;MS (EI) 348.2; HRMS (EI) calcd. for $C_{18}H_{24}N_2O_5$ 348.1685; found 348.1695; IR (cm^{-1}, neat) 3390, 1656.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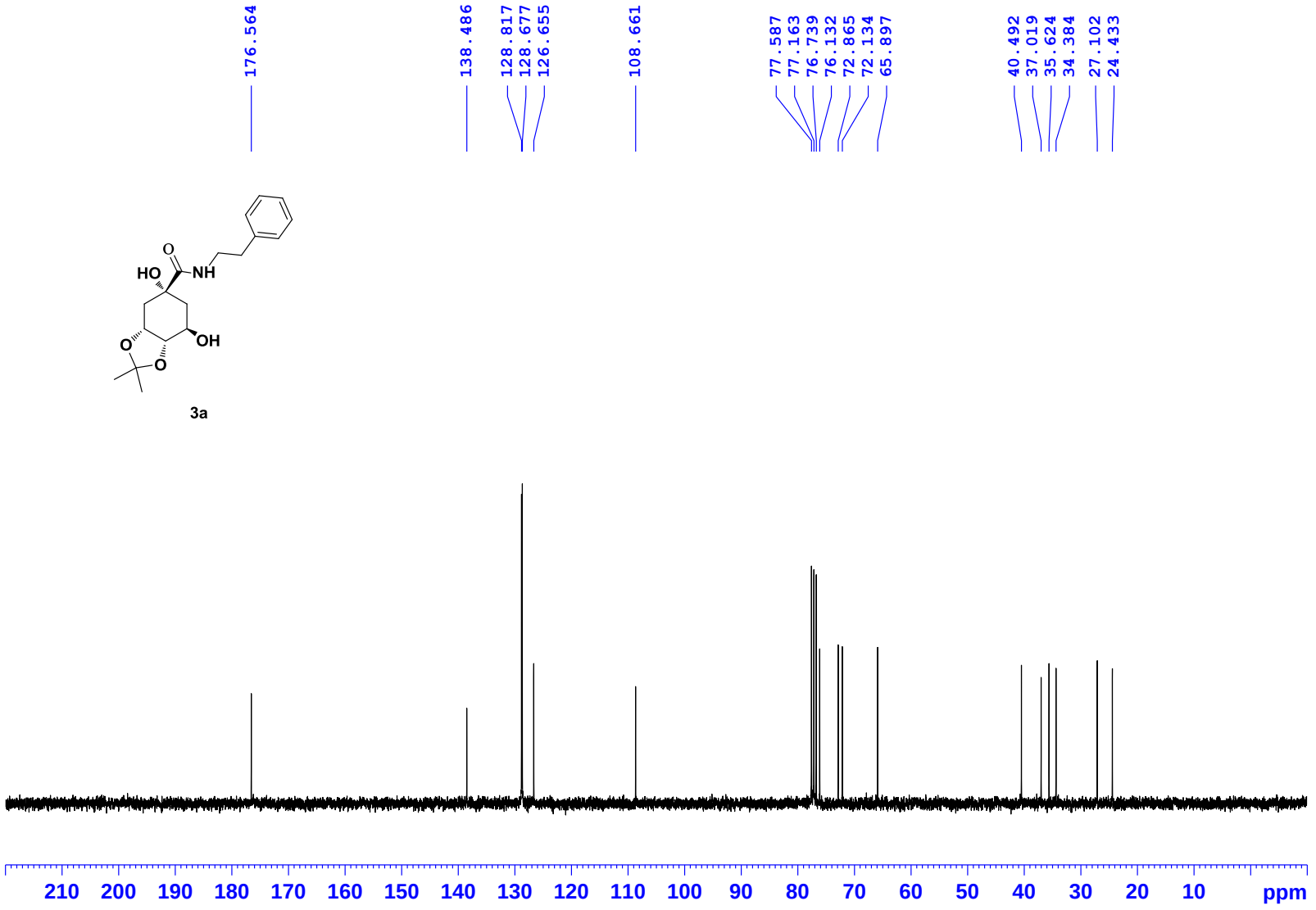
4.86 4.56 4.55 4.54 4.53 4.52 4.14 4.14 4.13 4.13 4.12 4.11 4.11 4.10
3.83 3.82 3.81 3.80 3.79 3.56 3.53 3.51 3.49 3.39
2.85 2.82 2.80 2.40 2.39 2.35 2.34 2.28 2.23 2.22 2.21 2.20 2.18 2.17 2.16
HO
O
NH
OH
O
O
3a
11 10 9 8 7 6 5 4 3 2 1 ppm
1.76 3.41 0.91 0.78 1.00 1.02 0.99 2.05 0.87 1.99 1.00 1.06 2.04 2.93 2.87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37.019
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HO
O
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OH
O
O
3a
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10
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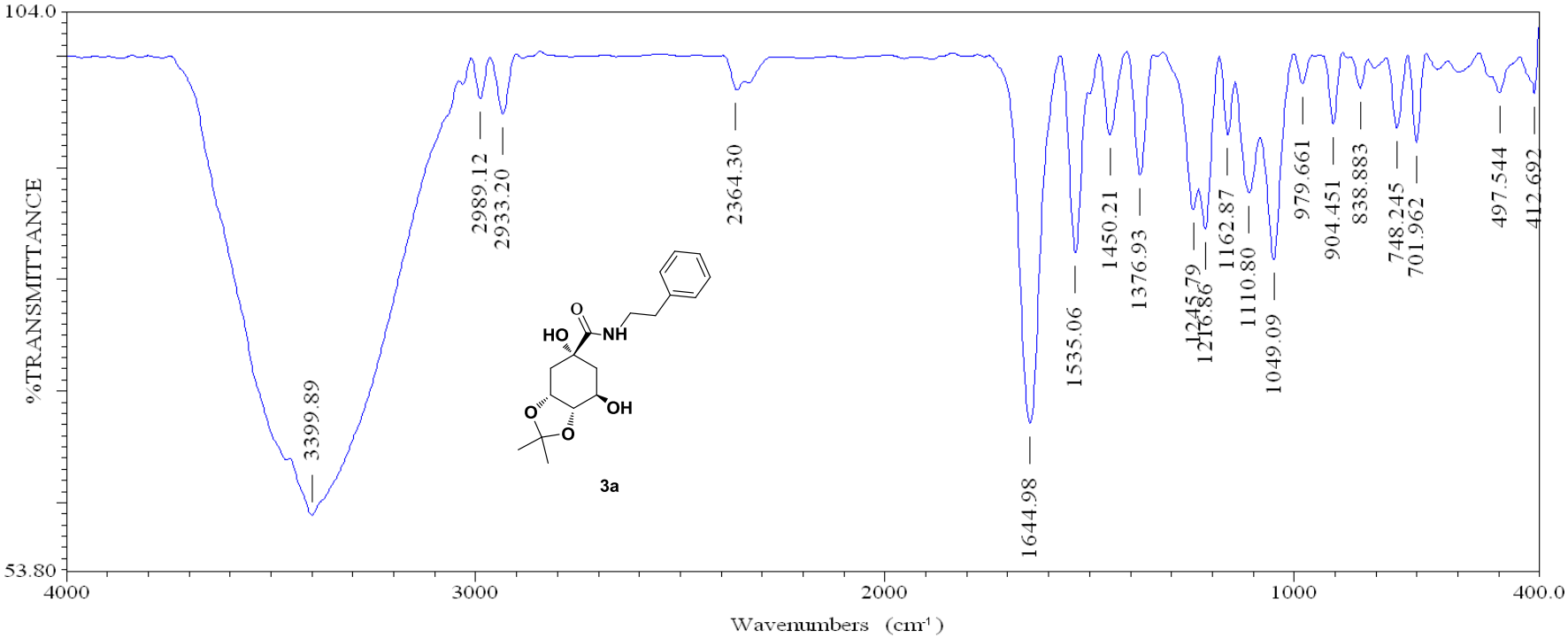
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53.80
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2000
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1049.09
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HO
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NH
OH
O
O
3a

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Samp: 1315-leeut-D01 Start : 16:02:47 109
Mode: EI +VE +LMR BSCAN (EXP) UP LR NRM
Oper: Inlet :
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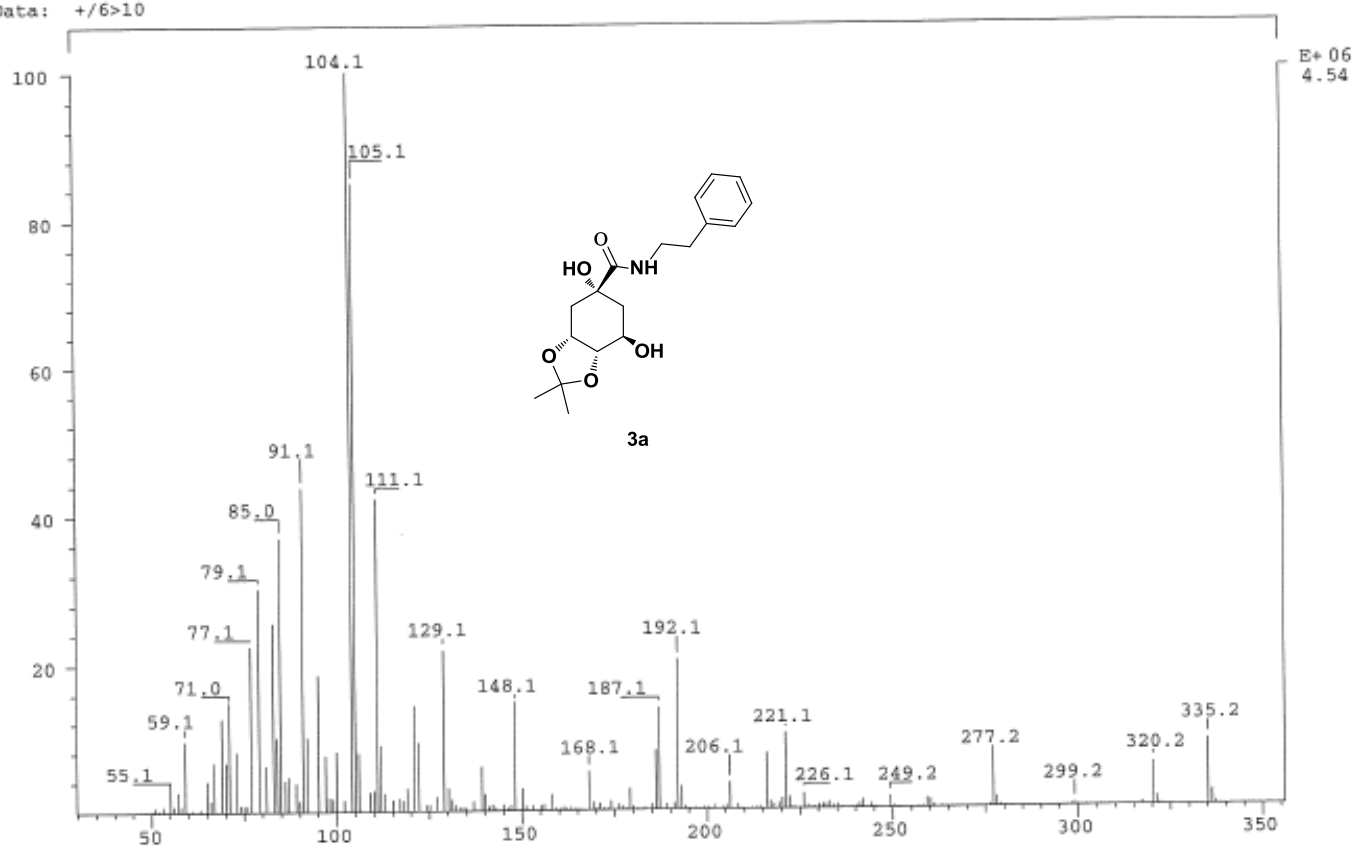
Norm: 104.1 RIC : 36581099 #peaks: 407
Peak: 1000.00 mmu
Data: +/6>10



3a

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Oper:                                                   Inlet  :
Limt: (  0)     .    . .
    : (419) C25.H25.N.O5
Peak: 1000.00 mmu   R+D: -2.0 > 60.0
Data: +/322>460  (CMASS : converted |CMASS : converted |CMASS : conve
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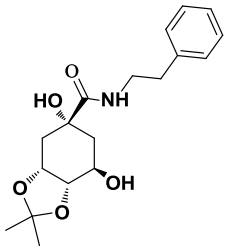
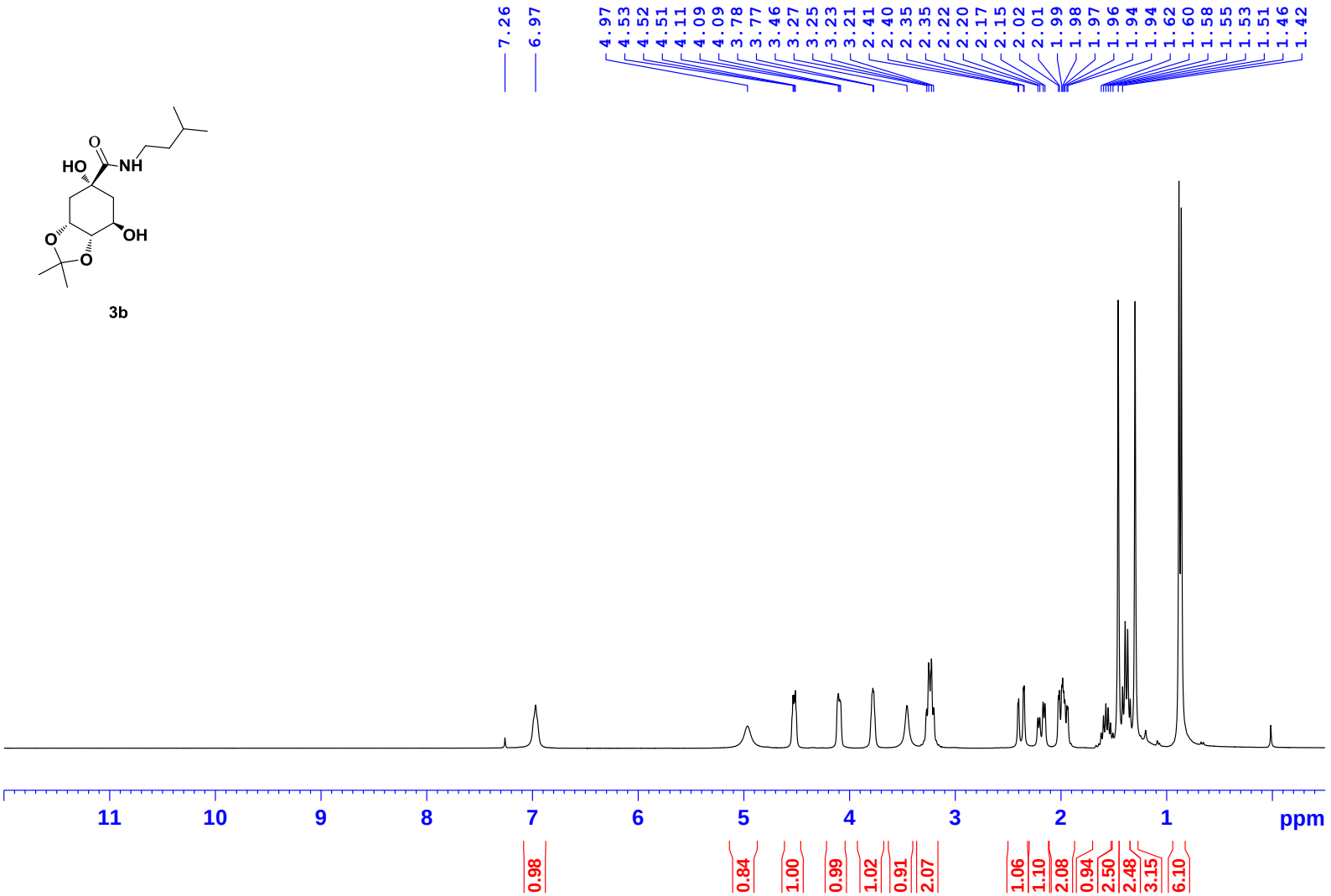


3a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3.21
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2.22
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1.97
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1.94
1.94
1.62
1.60
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1.55
1.53
1.51
1.46
1.42
HO
O
NH
OH
O
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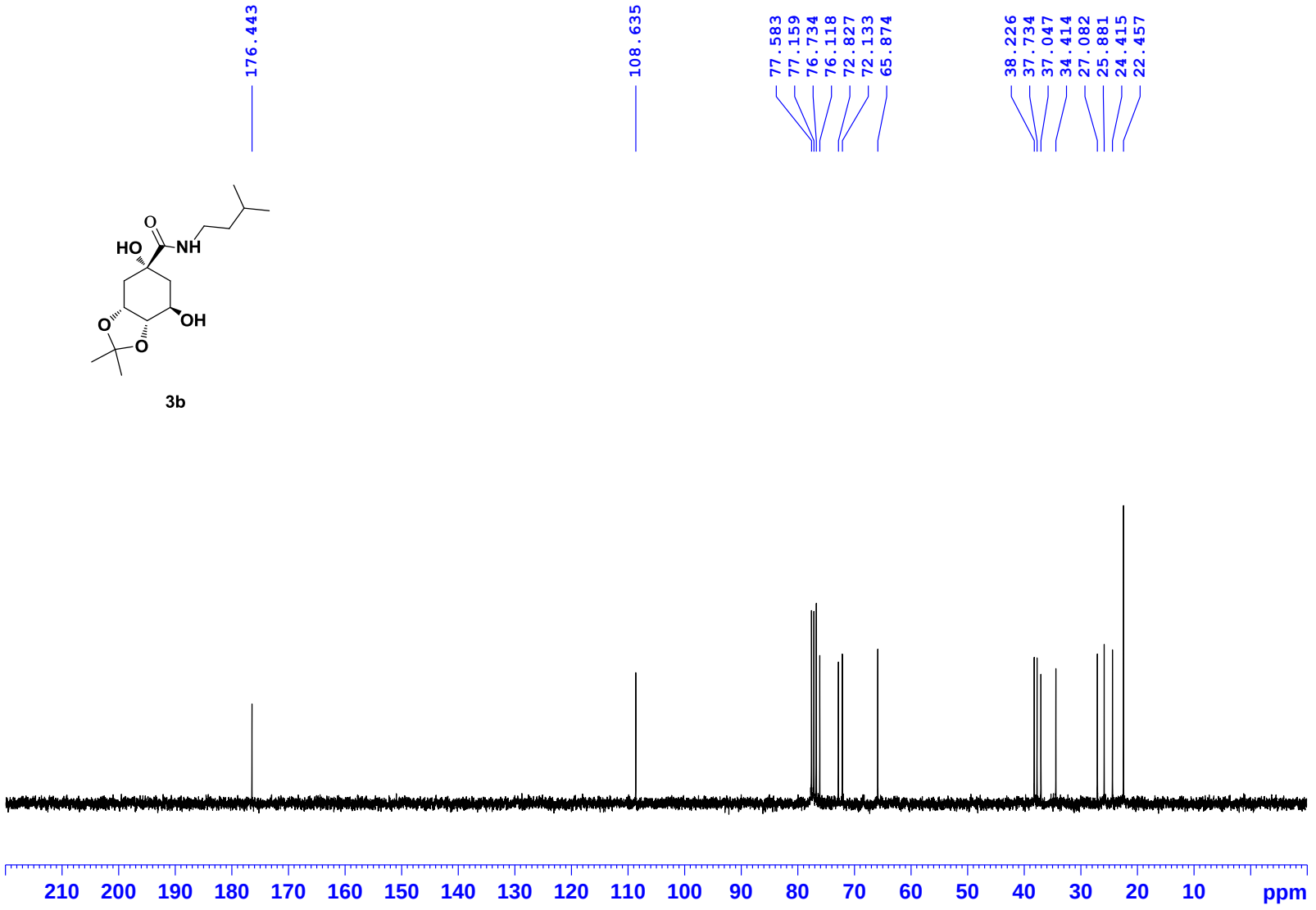
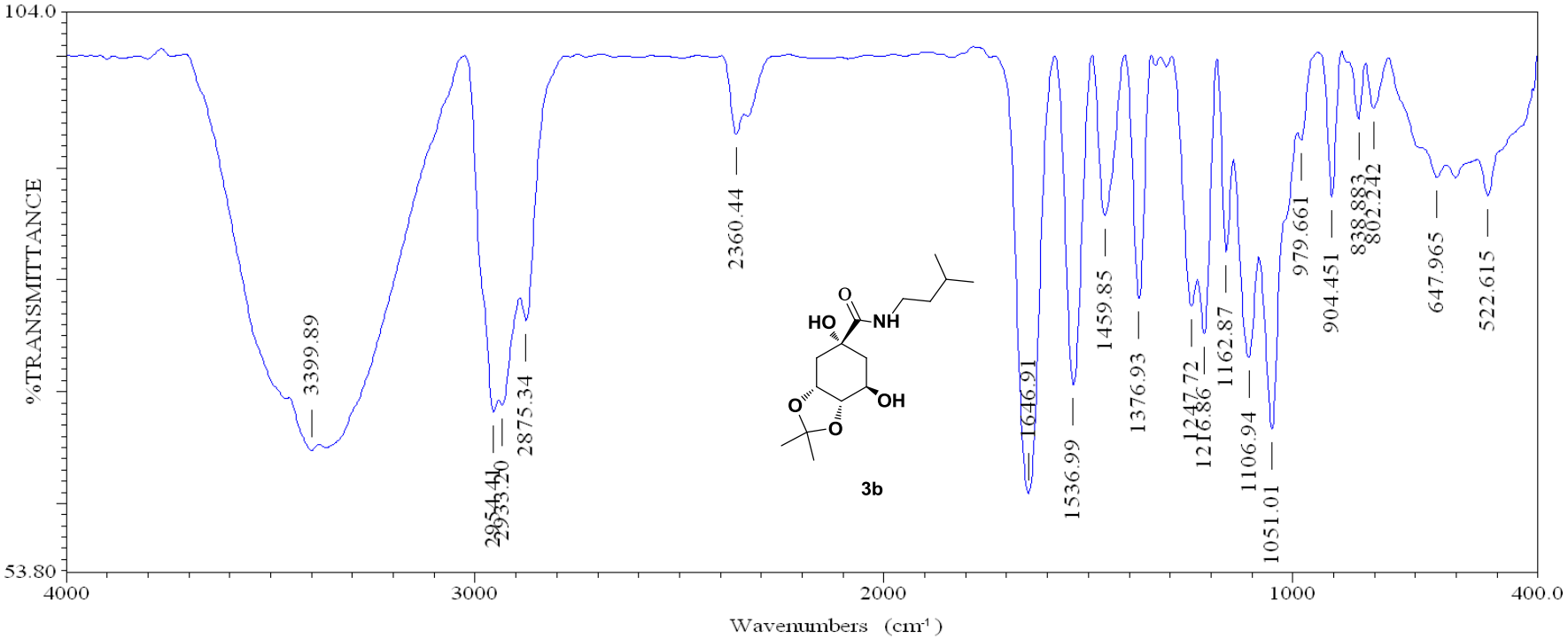
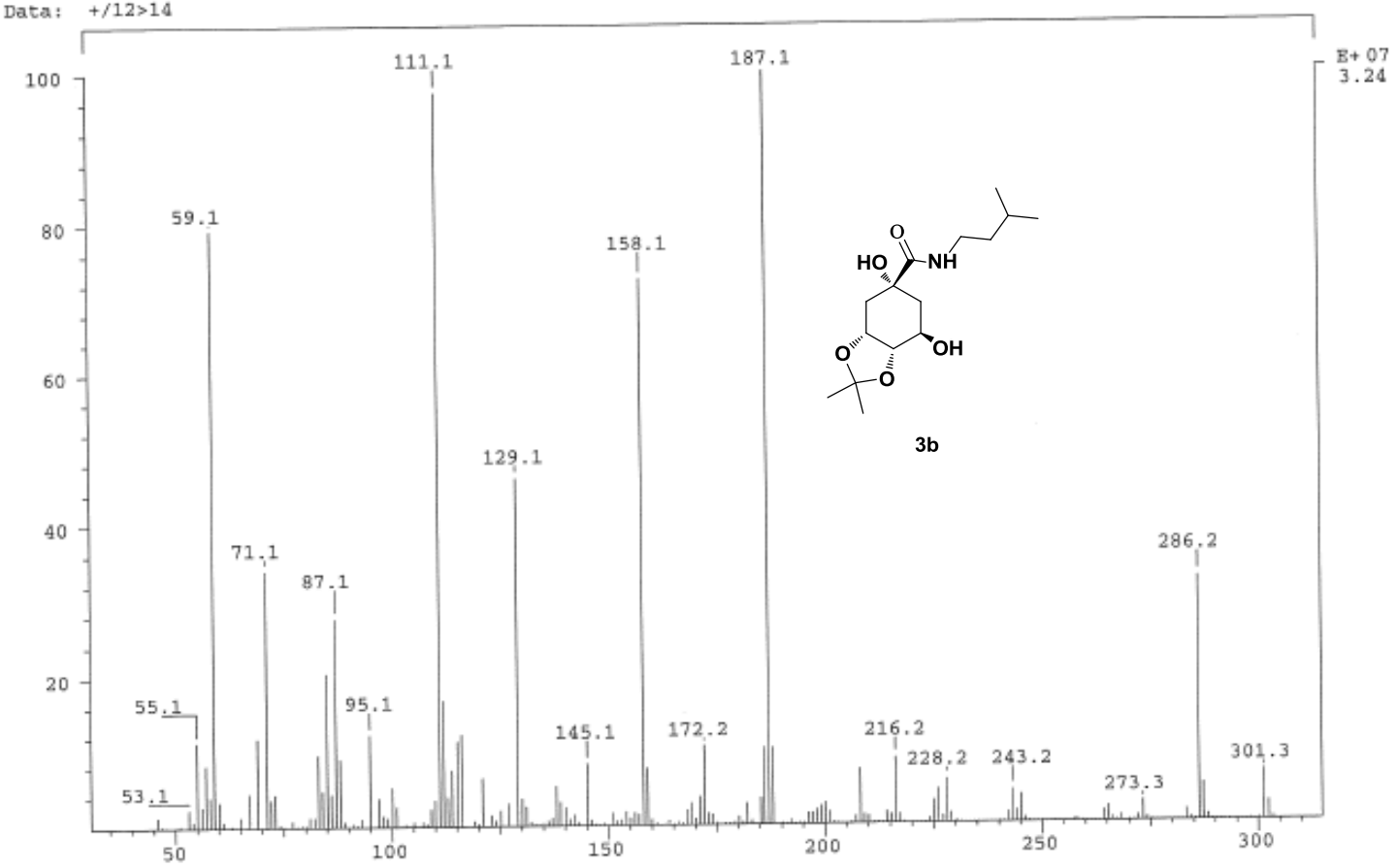
3b
11
10
9
8
7
6
5
4
3
2
1
ppm
0.98
0.84
1.00
0.99
1.02
0.91
2.07
1.06
1.10
2.08
0.94
2.50
2.48
3.15
6.10


176.443
108.635
77.583
77.159
76.734
76.118
72.827
72.133
65.874
38.226
37.734
37.047
34.414
27.082
25.881
24.415
22.457
HO
O
NH
OH
O
O
3b
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm


104.0
53.80
%TRANSMITTANCE
4000
3000
2000
1000
400.0
Wavenumbers (cm-1)
3399.89
2954.40
2933.20
2875.34
2360.44
1646.91
1536.99
1459.85
1376.93
1247.72
1216.86
1162.87
1106.94
1051.01
979.661
904.451
838.883
802.242
647.965
522.615
HO
NH
O
OH
O
O
3b

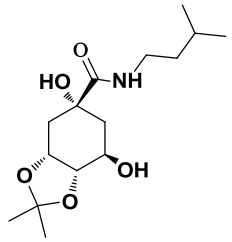
SPEC: lei2824 29-Feb-12 REG : 01:10.9 #9
Samp: 1316-leeut-D02 Start : 16:17:15 102
Mode: EI +VE +LMR BSCAN (EXP) UP LR NRM
Oper: Inlet :
Base: 187.1 Inten : 32359596 Masses: 45 > 500
Norm: 187.1 RIC : 293626709 #peaks: 471
Peak: 1000.00 mmu
Data: +/12>14



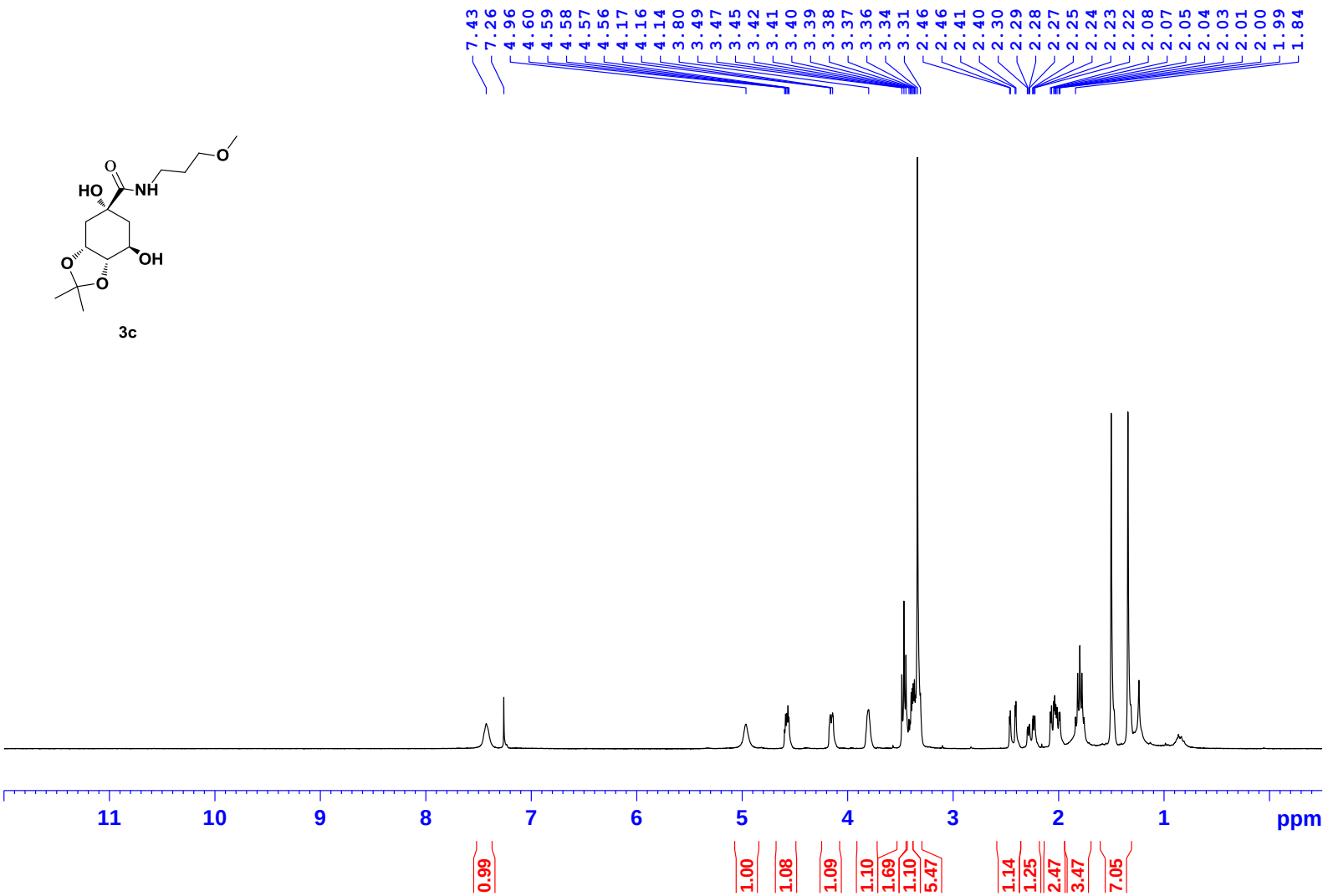
3b

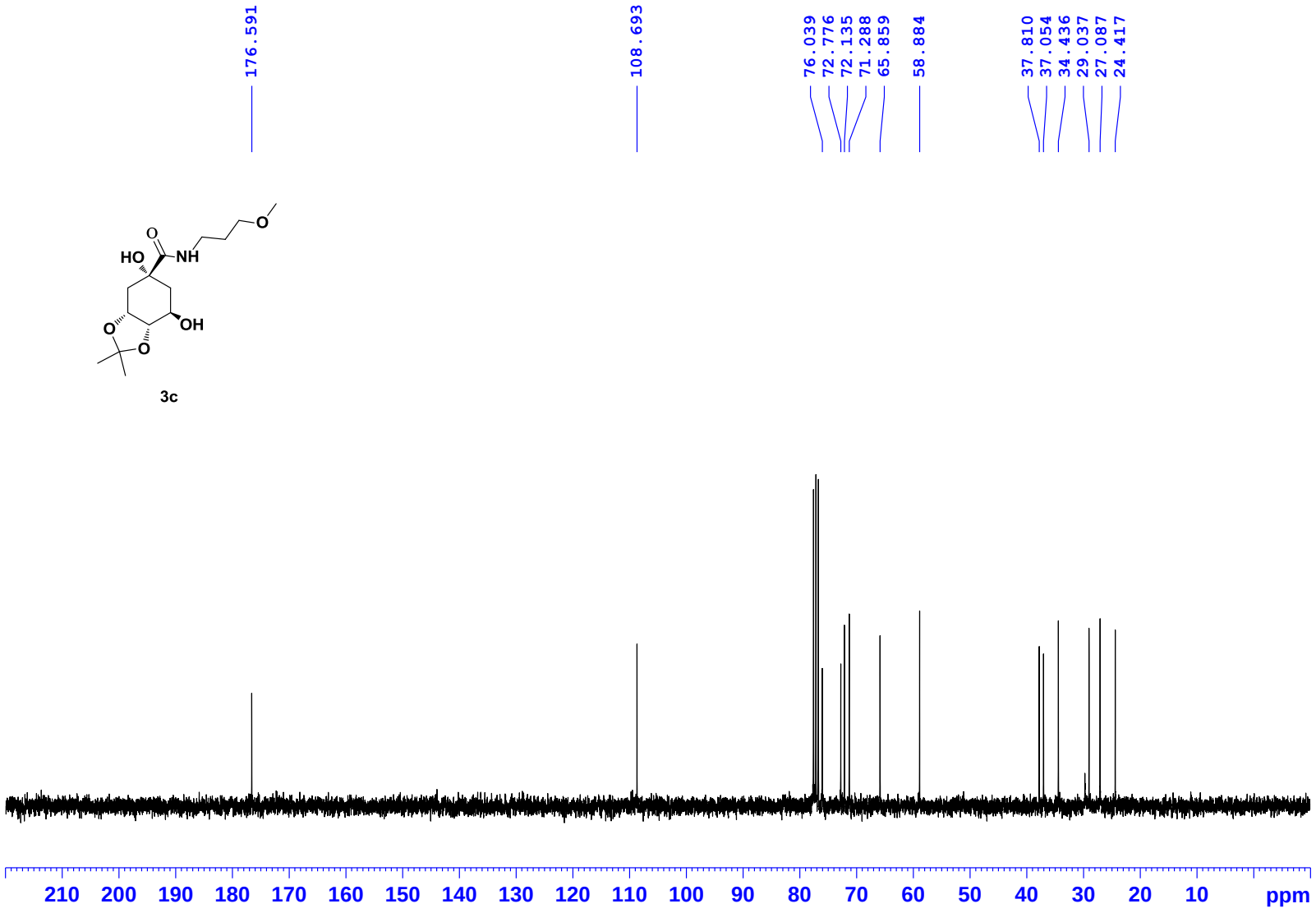
```
LIST: hei1435-c2                              05-Mar-12 REG    : 01:49.1      #9
Samp: 1316leeutd02                                      Start  : 23:33:50    839
Mode: EI +VE +LMR  ESCAN (EXP) UP HR NRM
Oper:                                                   Inlet  :
Limt: (  0)     .     .  .
    : (474) C28.H28.N.O6
Peak: 1000.00 mmu   R+D: -2.0 > 60.0
Data: +/344>441  (CMASS : converted; CMASS : converted |CMASS : conve
```

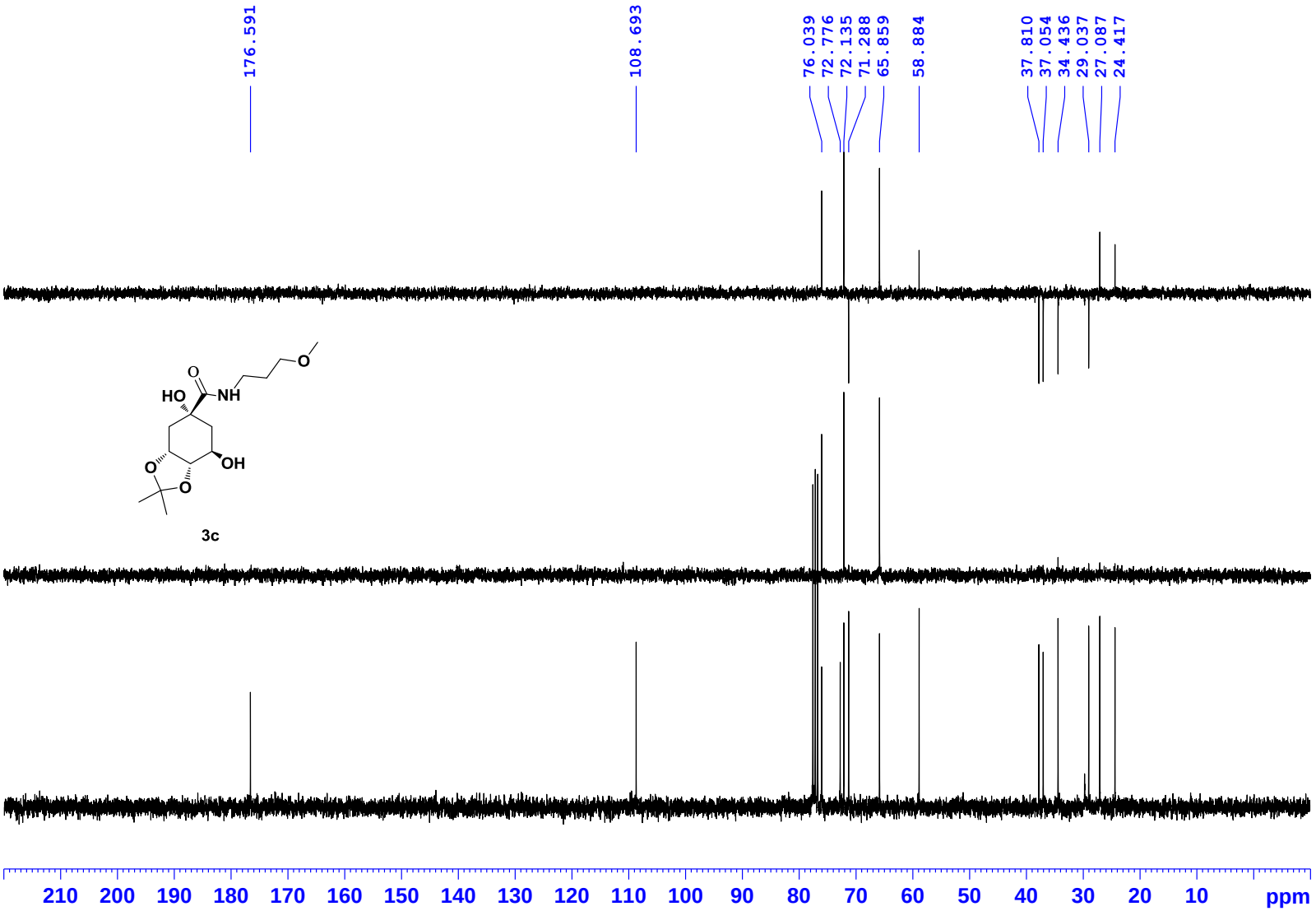
Mass	Intensity	%RA	Flags	(mmu) Delta	R+D	Composition
301.1887	646232	100.00	#	0.3	3.0	C15.H27.N.O5

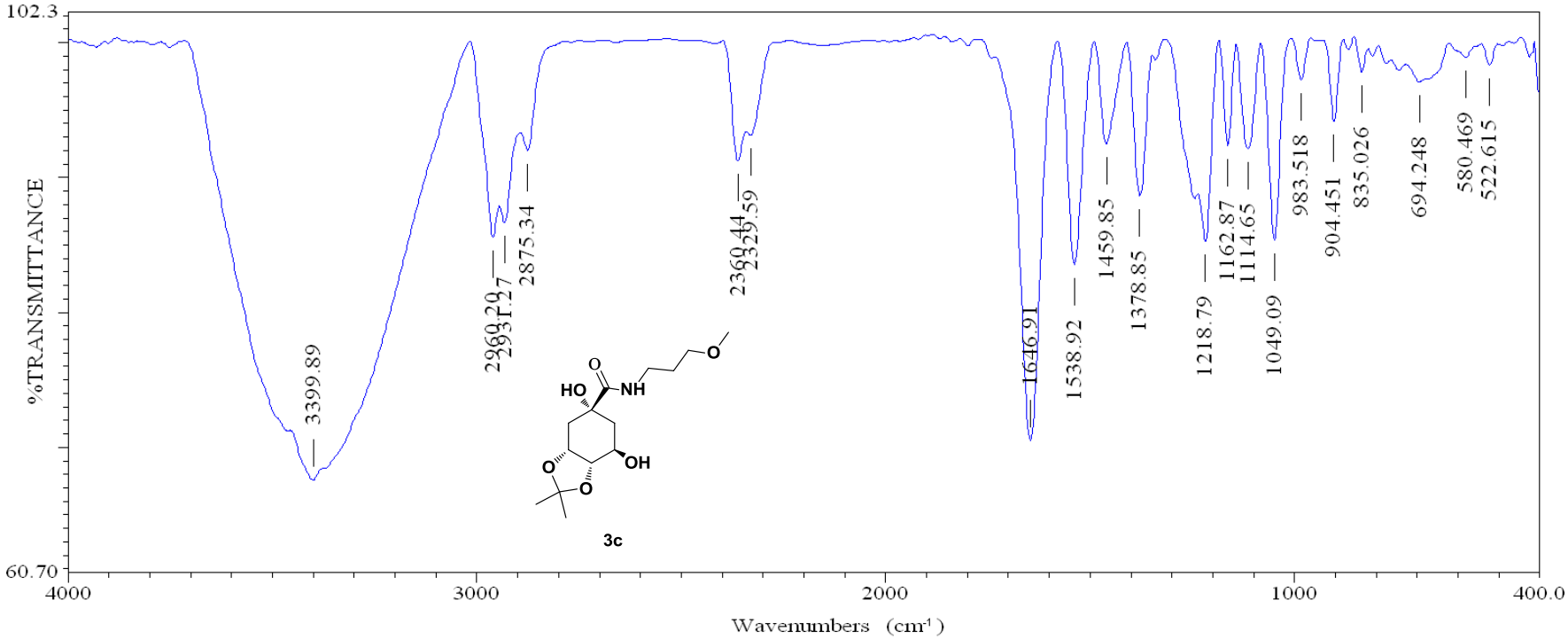


3b

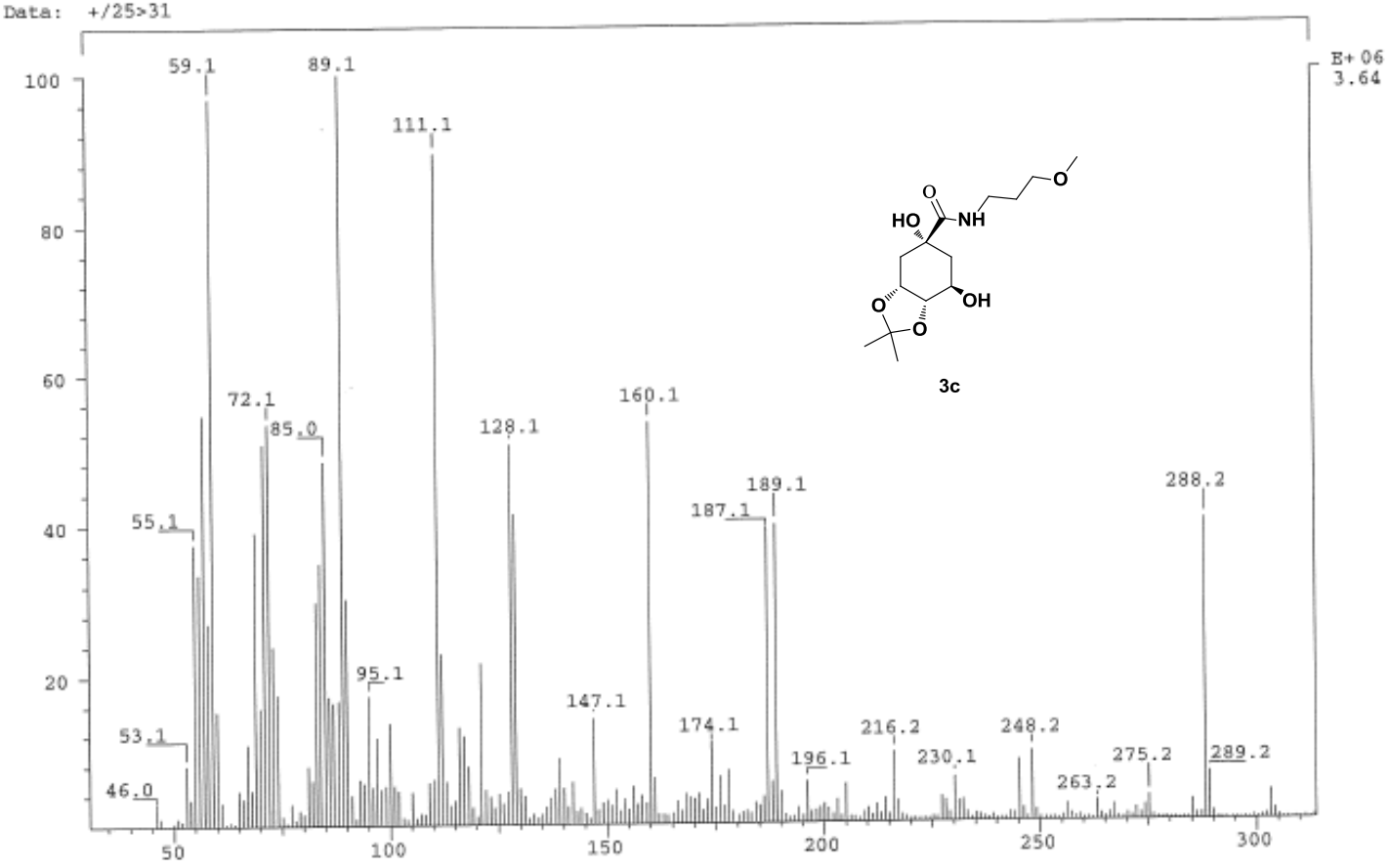

7.43
7.26
4.96
4.60
4.59
4.58
4.57
4.56
4.17
4.16
4.14
3.80
3.49
3.47
3.45
3.42
3.41
3.40
3.39
3.38
3.37
3.36
3.34
3.31
2.46
2.41
2.40
2.30
2.29
2.28
2.27
2.25
2.24
2.23
2.22
2.08
2.07
2.05
2.04
2.03
2.01
2.00
1.99
1.84
HO
NH
O
O
OH
O
O
3c
11
10
9
8
7
6
5
4
3
2
1
ppm
0.99
1.00
1.08
1.09
1.10
1.69
1.10
5.47
1.14
1.25
2.47
3.47
7.05


176.591
108.693
76.039
72.776
72.135
71.288
65.859
58.884
37.810
37.054
34.436
29.037
27.087
24.417
HO
NH
O
O
OH
O
O
3c
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm


176.591
108.693
76.039
72.776
72.135
71.288
65.859
58.884
37.810
37.054
34.436
29.037
27.087
24.417
HO
NH
O
OH
O
O
3c
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm


102.3
60.70
%TRANSMITTANCE
4000
3000
2000
1000
400.0
Wavenumbers (cm-1)
3399.89
2960.20
2931.27
2875.34
2360.44
2329.59
1646.91
1538.92
1459.85
1378.85
1218.79
1162.87
1114.65
1049.09
983.518
904.451
835.026
694.248
580.469
522.615
HO
NH
O
OH
O
O
O
3c

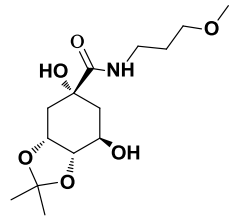
SPEC: lei2825 29-Feb-12 REG : 02:25.8 #9
Samp: 1317-leeut-D03 Start : 16:28:40 55
Mode: EI +VE +LMR BSCAN (EXP) UP LR NRM
Oper: Inlet :
Base: 89.1 Inten : 3636902 Masses: 45 > 500
Norm: 89.1 RIC : 65233216 #peaks: 474
Peak: 1000.00 mmu
Data: +/25>31



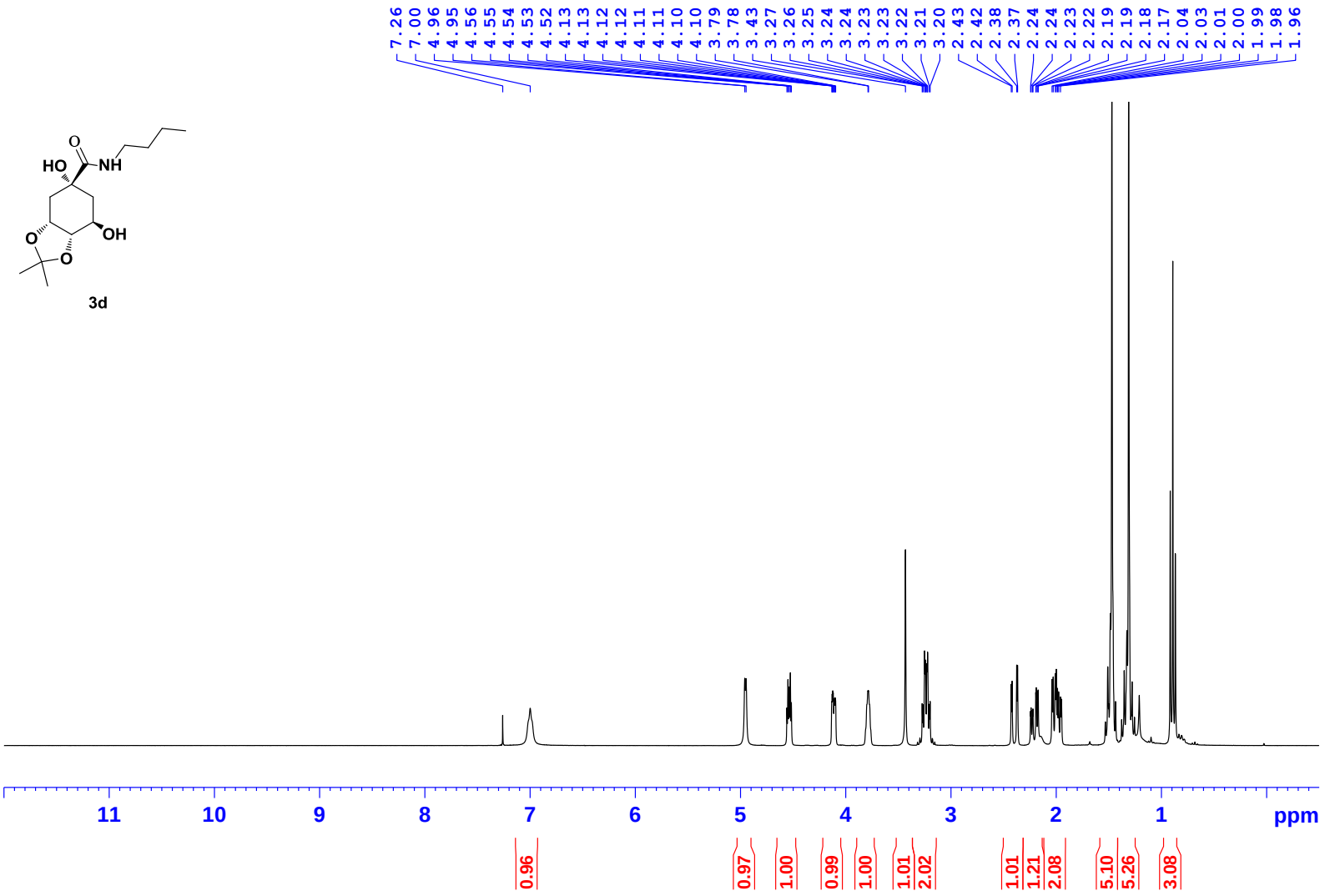
3c

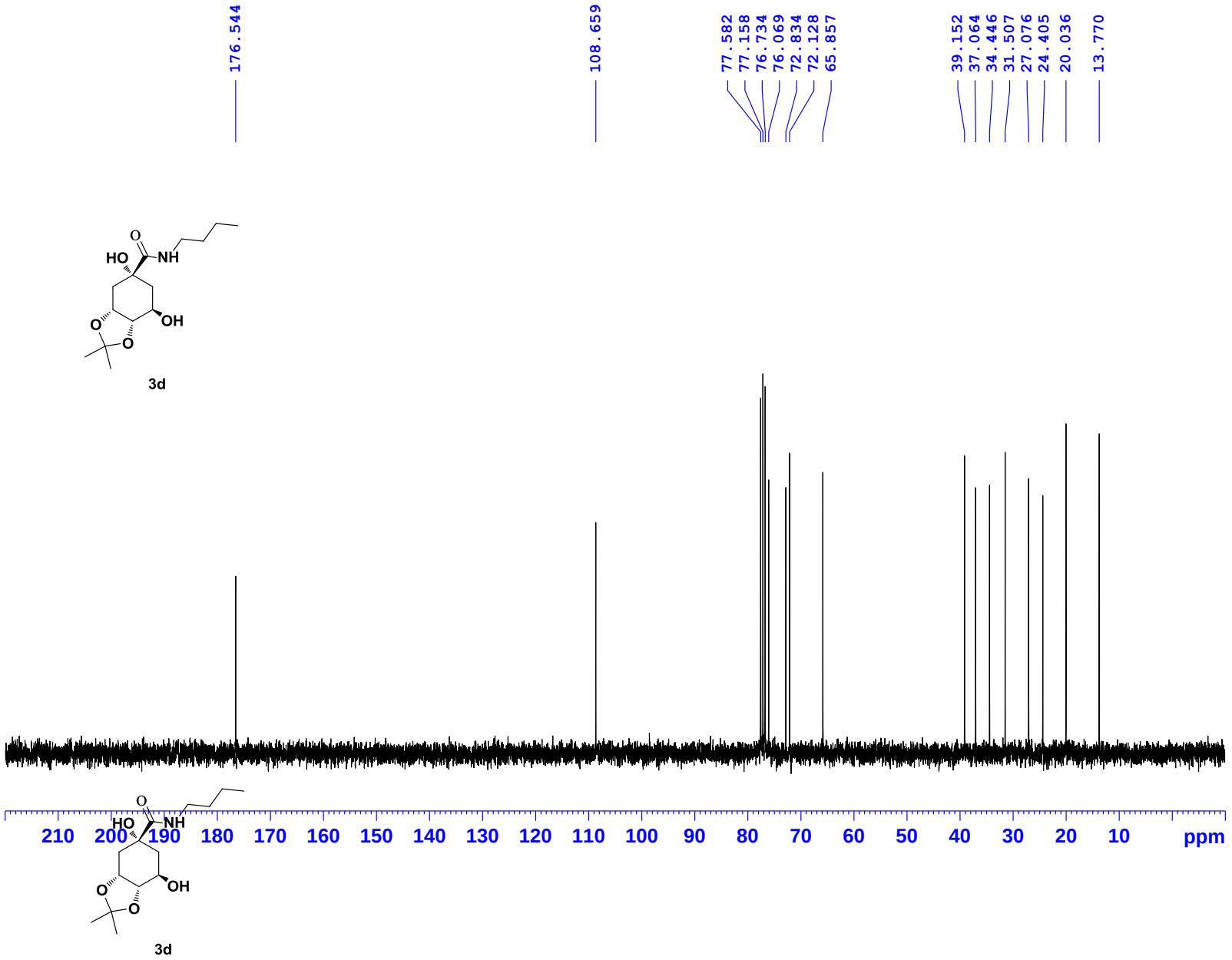
```
LIST: hei1434-c3                                  05-Mar-12 REG    : 04:32.7     #9
Samp: 13171eeutd03                                          Start  : 23:19:22  1045
Mode: EI +VE +LMR  ESCAN (EXP) UP HR NRM
                                                            Inlet  :
Oper:
Limt: (  0)     .    . .
    : (456) C25.H30.N.O7
Peak: 1000.00 mmu   R+D: -2.0 > 60.0
Data: +/860>1019  (CMASS : converted; CMASS : converted; CMASS : conv
```

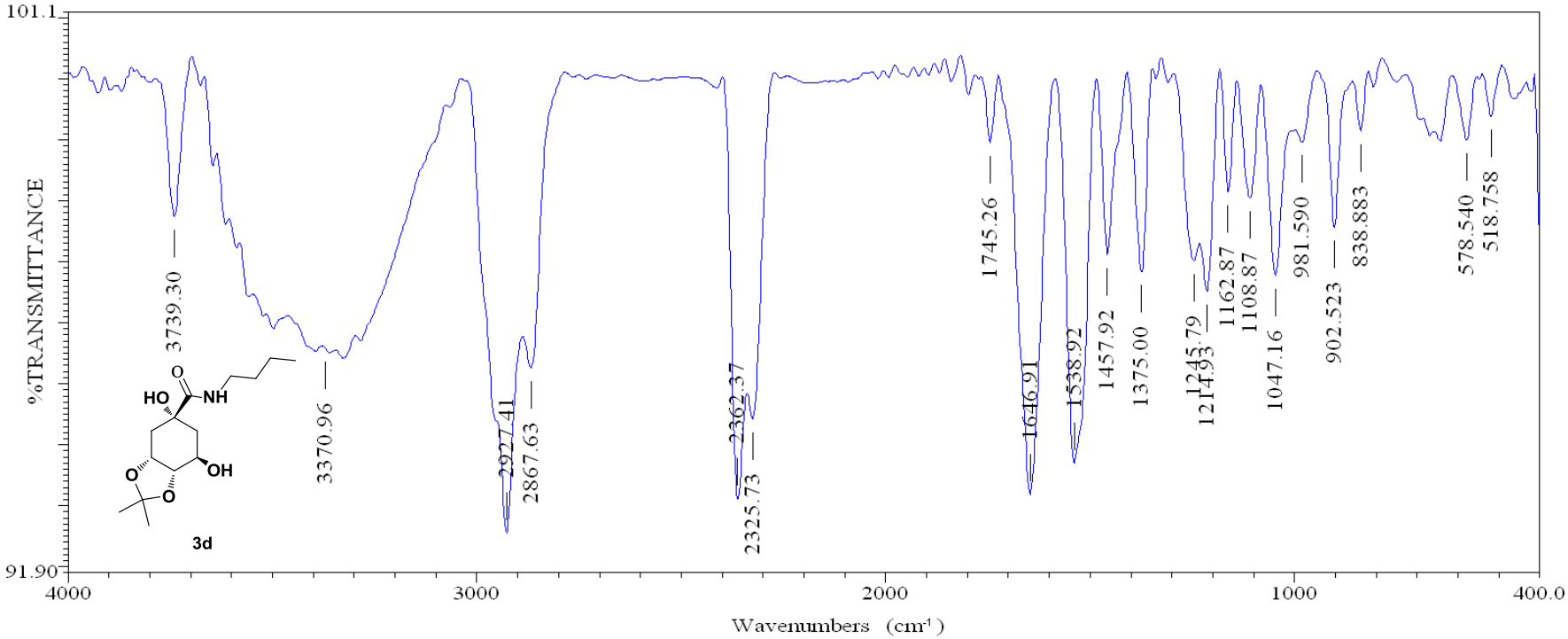
Mass	Intensity 0	%RA	Flags	(mmu) Delta	R+D	Composition
303.1687	46831	18.41	#	-0.5	3.0	C14.H25.N.O6



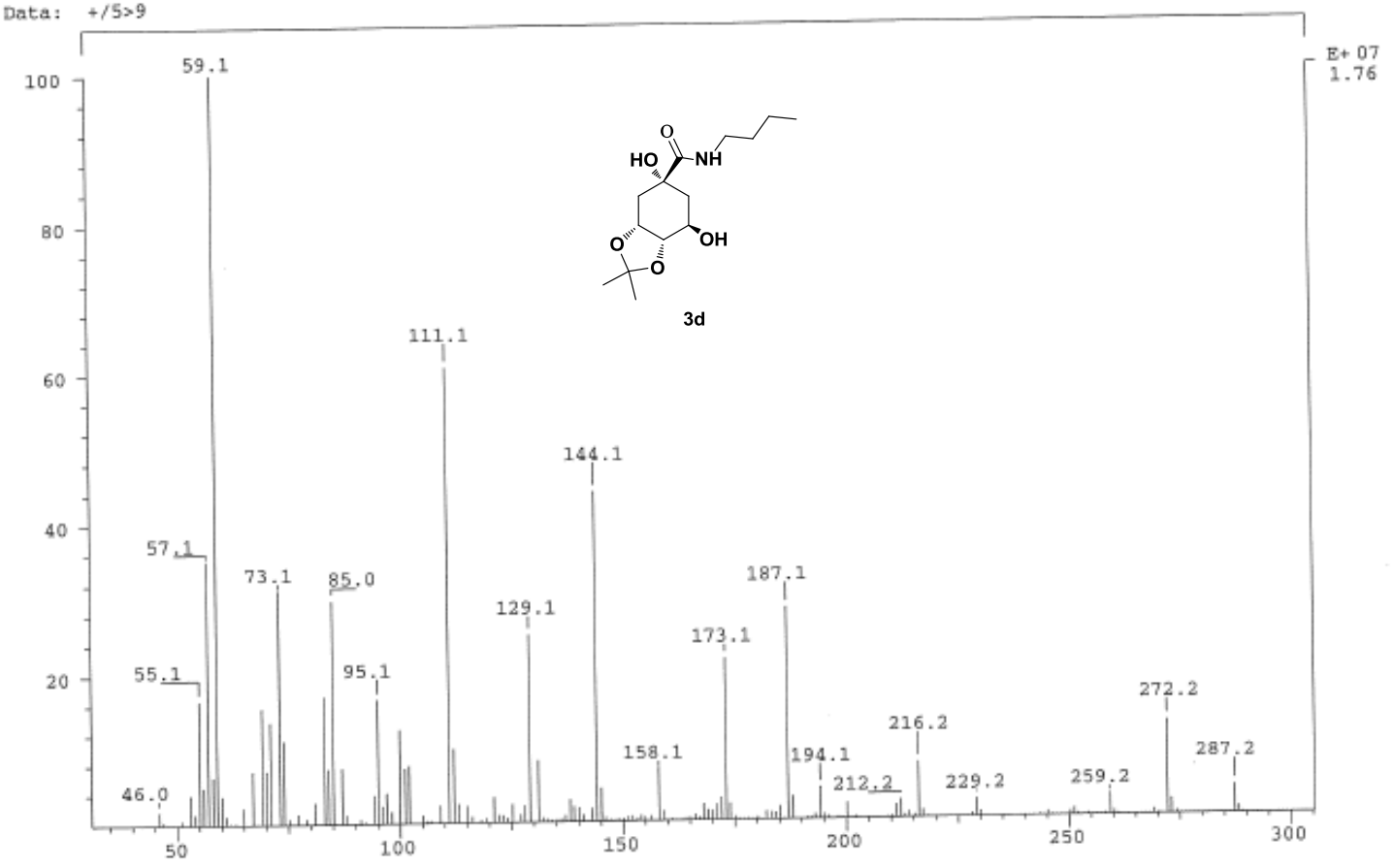
3c


7.26
7.00
4.96
4.95
4.56
4.55
4.54
4.53
4.52
4.13
4.13
4.12
4.12
4.11
4.11
4.10
4.10
3.79
3.78
3.43
3.27
3.26
3.25
3.24
3.24
3.23
3.23
3.22
3.21
3.20
2.43
2.42
2.38
2.37
2.24
2.24
2.23
2.22
2.19
2.19
2.18
2.17
2.04
2.03
2.01
2.00
1.99
1.98
1.96
HO
NH
O
OH
O
O
3d
11
10
9
8
7
6
5
4
3
2
1
ppm
0.96
0.97
1.00
0.99
1.00
1.01
2.02
1.01
1.21
2.08
5.10
5.26
3.08


176.544
108.659
77.582
77.158
76.734
76.069
72.834
72.128
65.857
39.152
37.064
34.446
31.507
27.076
24.405
20.036
13.770
HO
NH
O
OH
O
O
3d
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm
HO
NH
O
OH
O
O
3d


101.1
91.90
%TRANSMITTANCE
4000
3000
2000
1000
400.0
Wavenumbers (cm-1)
3739.30
3370.96
2927.41
2867.63
2362.37
2325.73
1745.26
1646.91
1538.92
1457.92
1375.00
1245.79
1214.93
1162.87
1108.87
1047.16
981.590
902.523
838.883
578.540
518.758
HO
NH
O
OH
O
O
3d

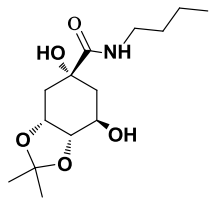
SPEC: lei2826 29-Feb-12 REG : 00:36.3 #9
Samp: 1318-leeut-D04 Start : 16:38:00 61
Mode: EI +VE +LMR BSCAN (EXP) UP LR NRM Inlet :
Oper: Masses: 45 > 500
Base: 59.1 Inten : 17629092 #peaks: 452
Norm: 59.1 RIC : 127014707
Peak: 1000.00 mmu
Data: +/5>9



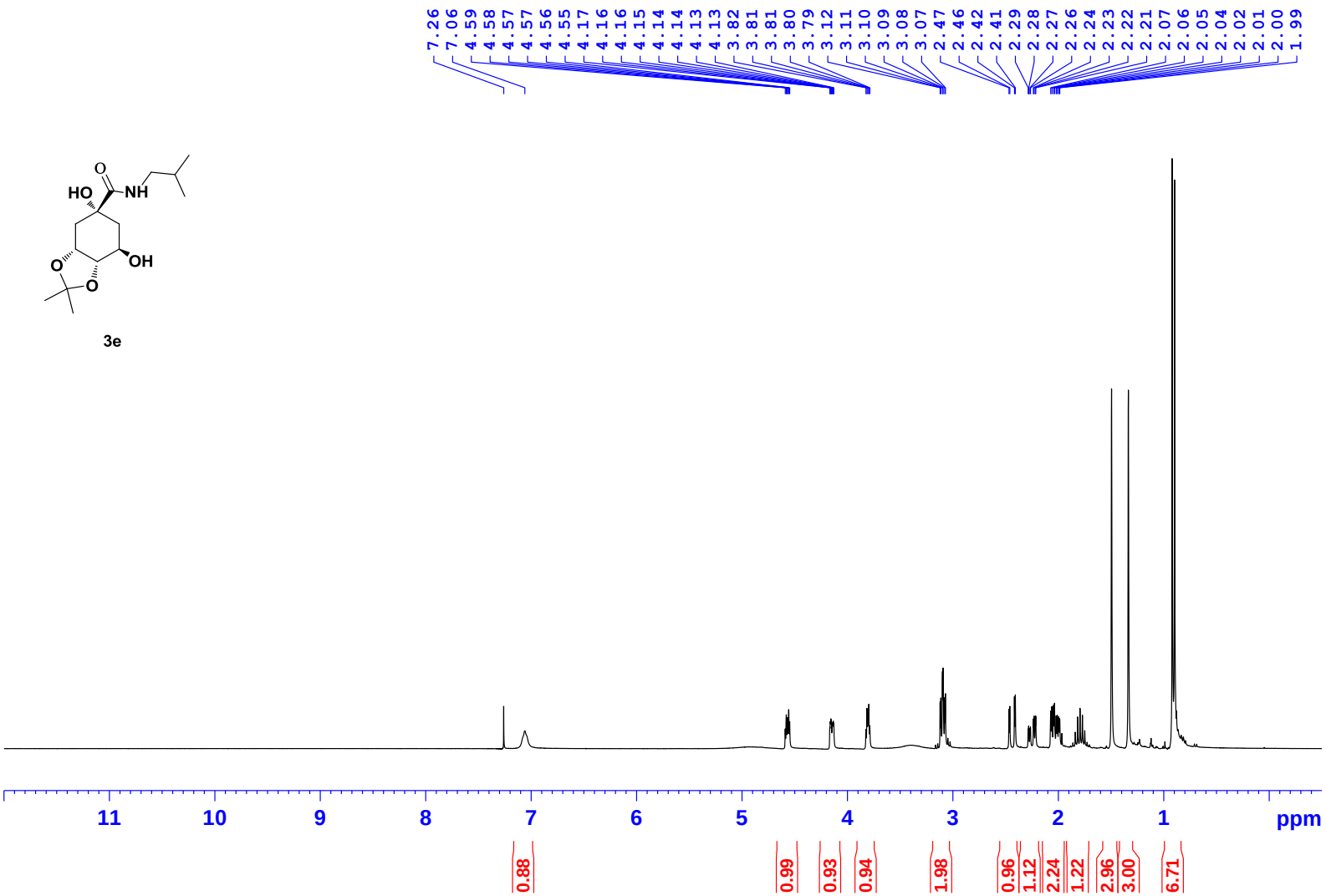
3d

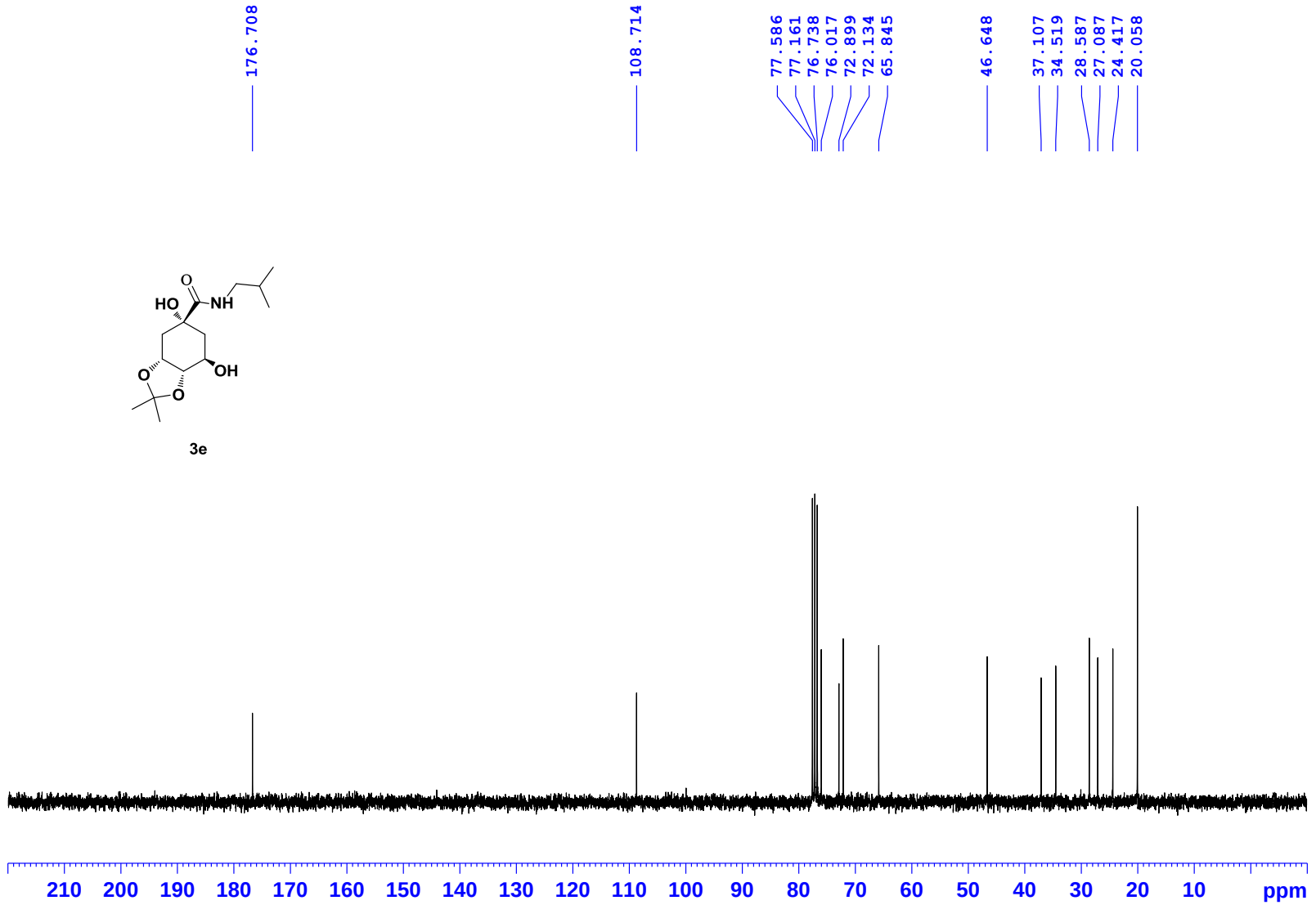
```
LIST: hei1433-c1                                  05-Mar-12 REG    : 02:32.7     #9
Samp: 131@leeutD04                                          Start  : 22:37:30   595
Mode: EI +VE +LMR  ESCAN (EXP) UP HR NRM
Oper:                                                       Inlet  :
Limt: (  0)     .     .     .
    : (456) C25.H30.N.O7
Peak: 1000.00 mmu   R+D: -2.0 > 60.0
Data: +/453>552  (CMASS : converted |CMASS : converted |CMASS : conve
```

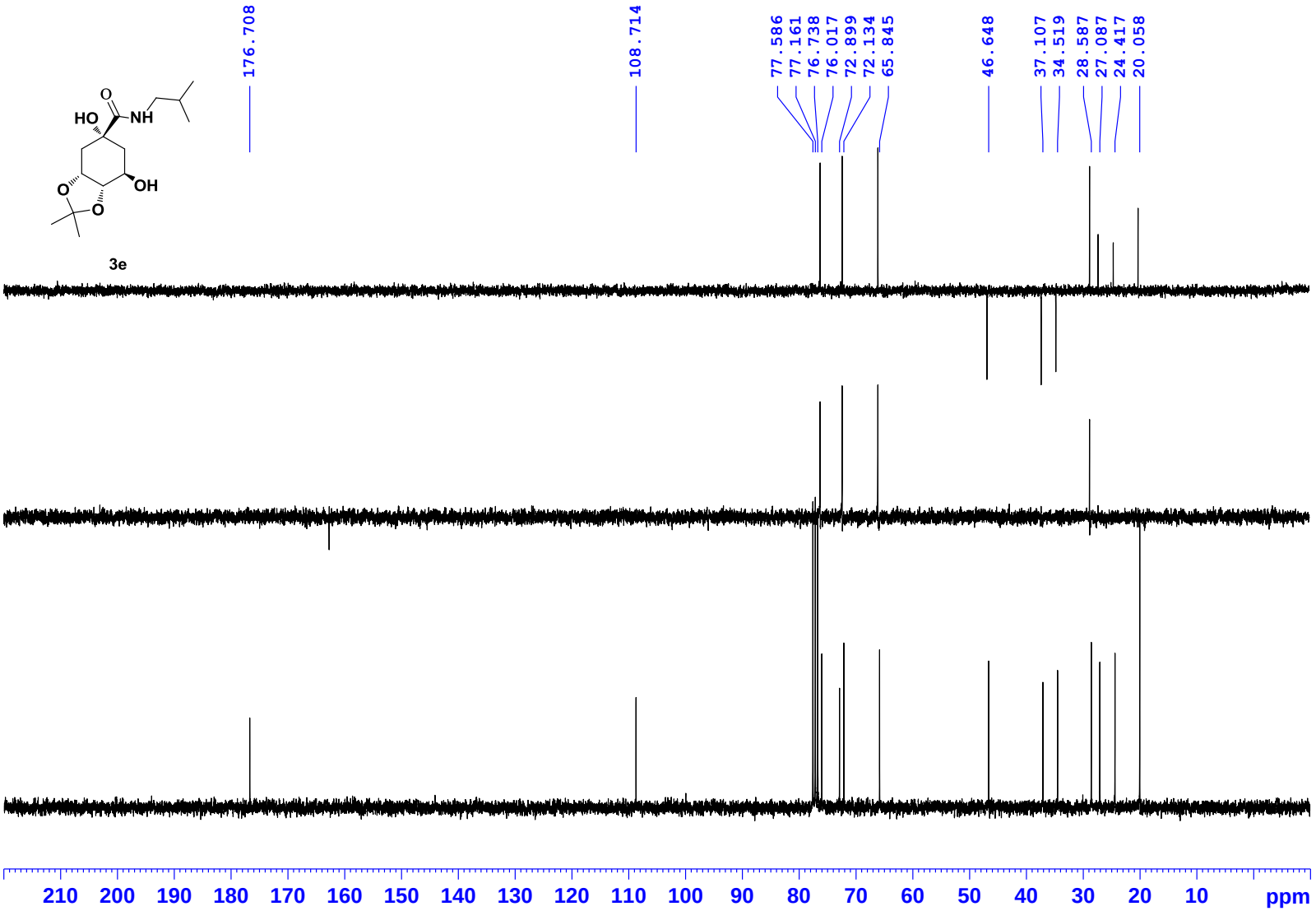
Mass	Intensity	%RA	Flags	(mmu) Delta	R+D	Composition
287.1725	1596276	100.00	#	0.8	3.0	C14.H25.N.O5

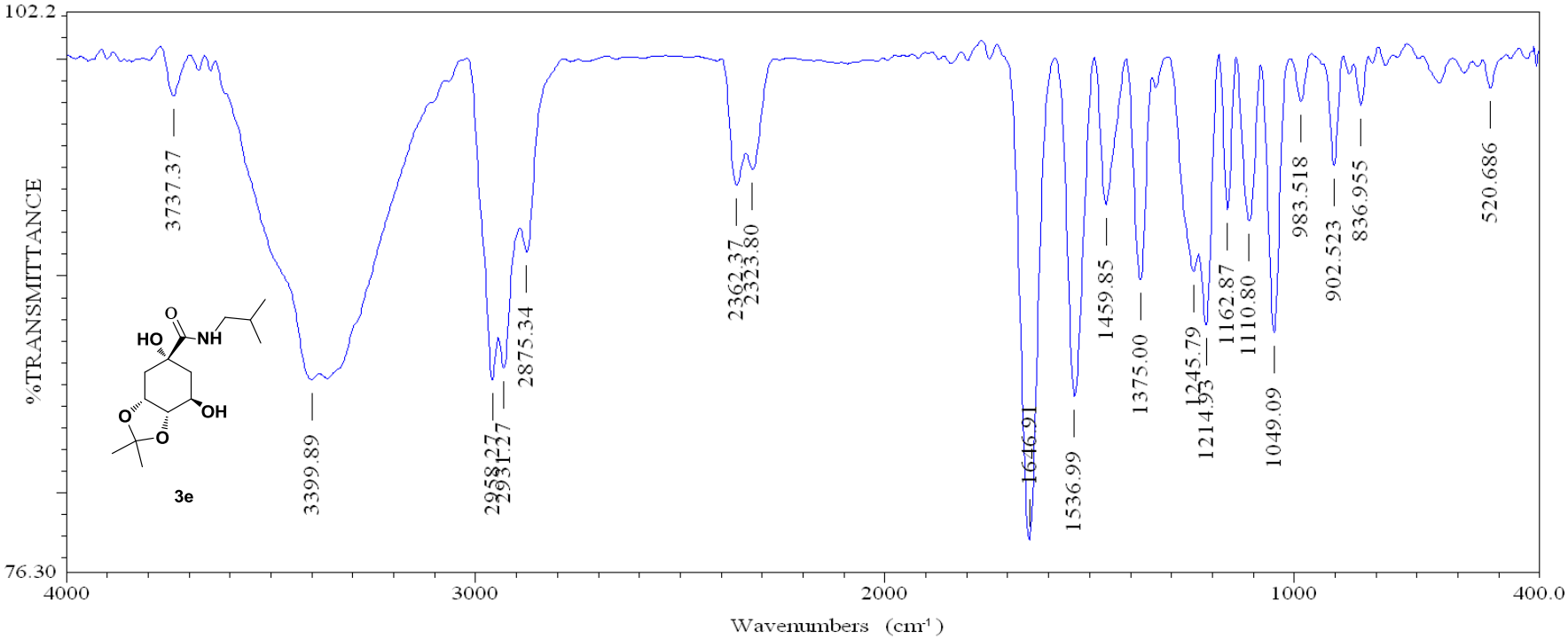


3d

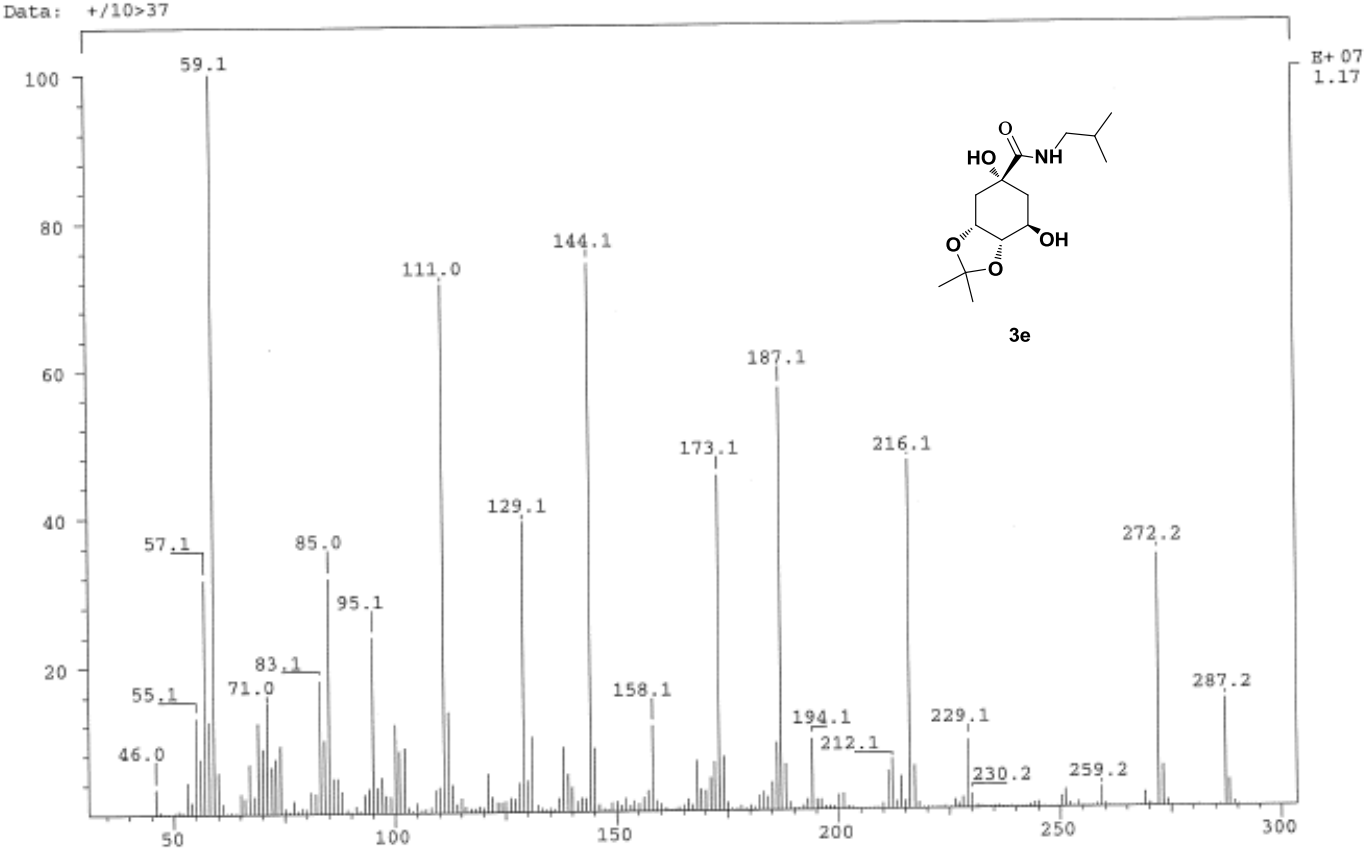

7.26
7.06
4.59
4.58
4.57
4.57
4.56
4.55
4.17
4.16
4.16
4.15
4.14
4.14
4.13
4.13
3.82
3.81
3.81
3.80
3.79
3.12
3.11
3.10
3.09
3.08
3.07
2.47
2.46
2.42
2.41
2.29
2.28
2.27
2.26
2.24
2.23
2.22
2.21
2.07
2.06
2.05
2.04
2.02
2.01
2.00
1.99
HO
O
NH
OH
O
O
3e
11
10
9
8
7
6
5
4
3
2
1
ppm
0.88
0.99
0.93
0.94
1.98
0.96
1.12
2.24
1.22
2.96
3.00
6.71


176.708
108.714
77.586
77.161
76.738
76.017
72.899
72.134
65.845
46.648
37.107
34.519
28.587
27.087
24.417
20.058
HO
O
NH
OH
O
O
3e
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm


176.708
108.714
77.586
77.161
76.738
76.017
72.899
72.134
65.845
46.648
37.107
34.519
28.587
27.087
24.417
20.058
HO
O
NH
OH
O
O
3e
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm


102.2
76.30
%TRANSMITTANCE
4000
3000
2000
1000
400.0
Wavenumbers (cm-1)
3737.37
3399.89
2958.27
2931.27
2875.34
2362.37
2323.80
1646.91
1536.99
1459.85
1375.00
1245.79
1214.93
1162.87
1110.80
1049.09
983.518
902.523
836.955
520.686
HO
NH
O
OH
O
O
3e

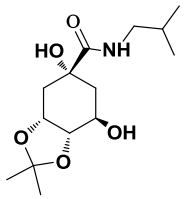
SPEC: lei2827 01-Mar-12 REG : 00:55.6 #9
Samp: 1319-leeut-D05 Start : 07:30:06 133
Mode: EI +VE +LMR BSCAN (EXP) UP LR NRM
Oper: Inlet :
Base: 59.1 Inten : 11732574 Masses: 45 > 500
Norm: 59.1 RIC : 130626270 #peaks: 517
Peak: 1000.00 mmu
Data: +/10>37



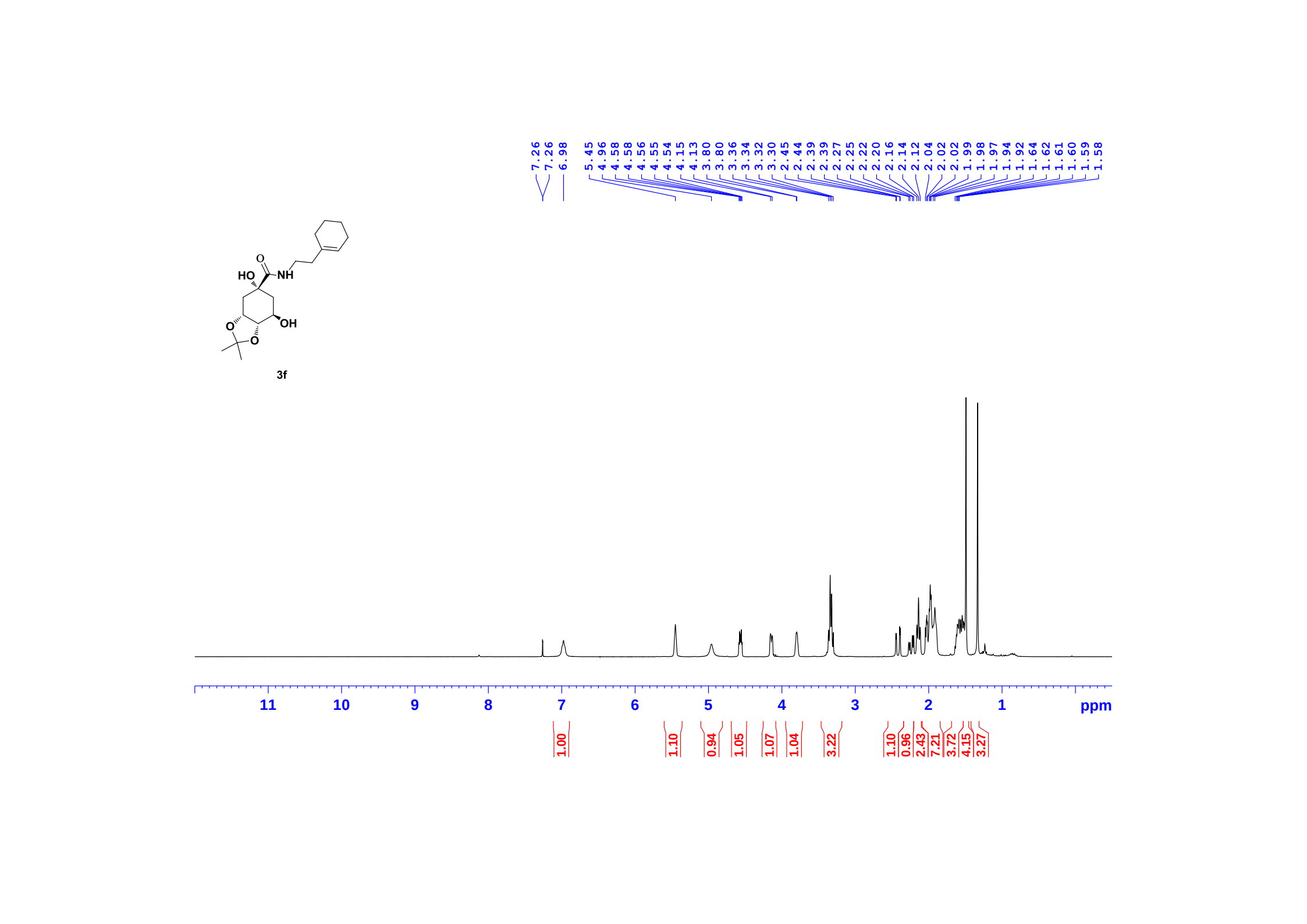
3e

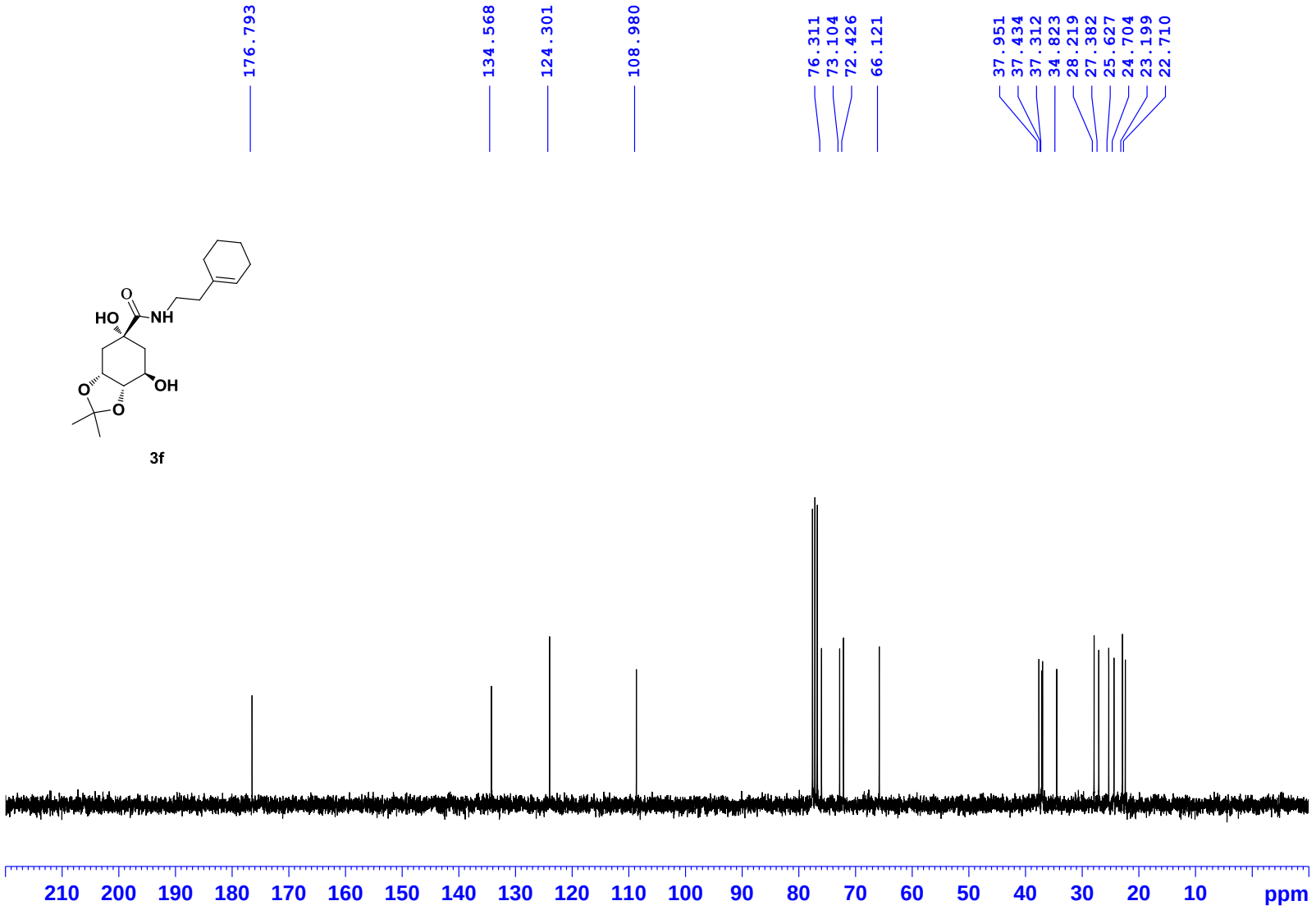
```
LIST: hei1432-c2                              05-Mar-12 REG    : 01:07.9      #9
Samp: 13191eeutD05                                      Start  : 22:18:12    637
Mode: EI +VE +LMR  ESCAN (EXP) UP HR NRM
Oper:                                                   Inlet  :
Limt: (  0)    .    . .
    : (456) C25.H30.N.O7
Peak: 1000.00 mmu   R+D: -2.0 > 60.0
Data: +/197>307  (CMASS : converted; CMASS : converted |CMASS : conve
```

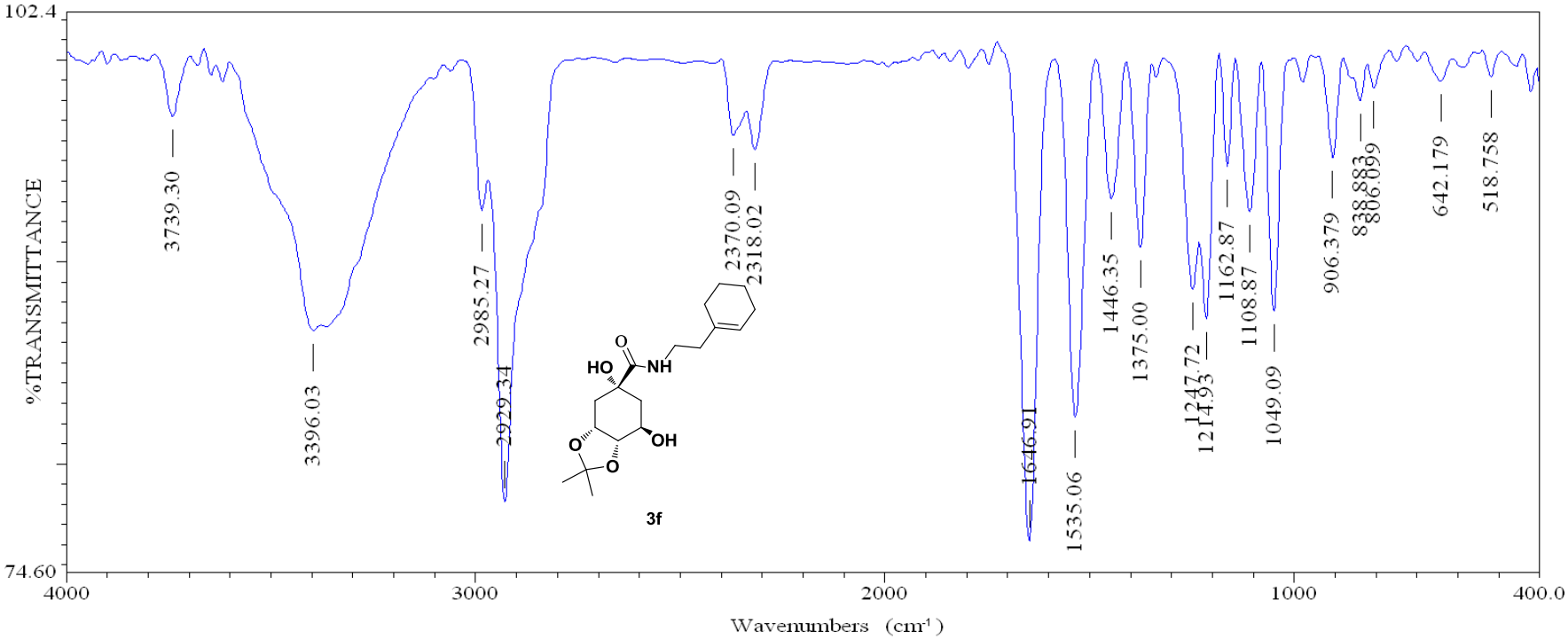
Mass	Intensity	%RA	Flags	(mmu) Delta	R+D	Composition
287.1726	2544634	100.00	#	0.7	3.0	C14.H25.N.O5



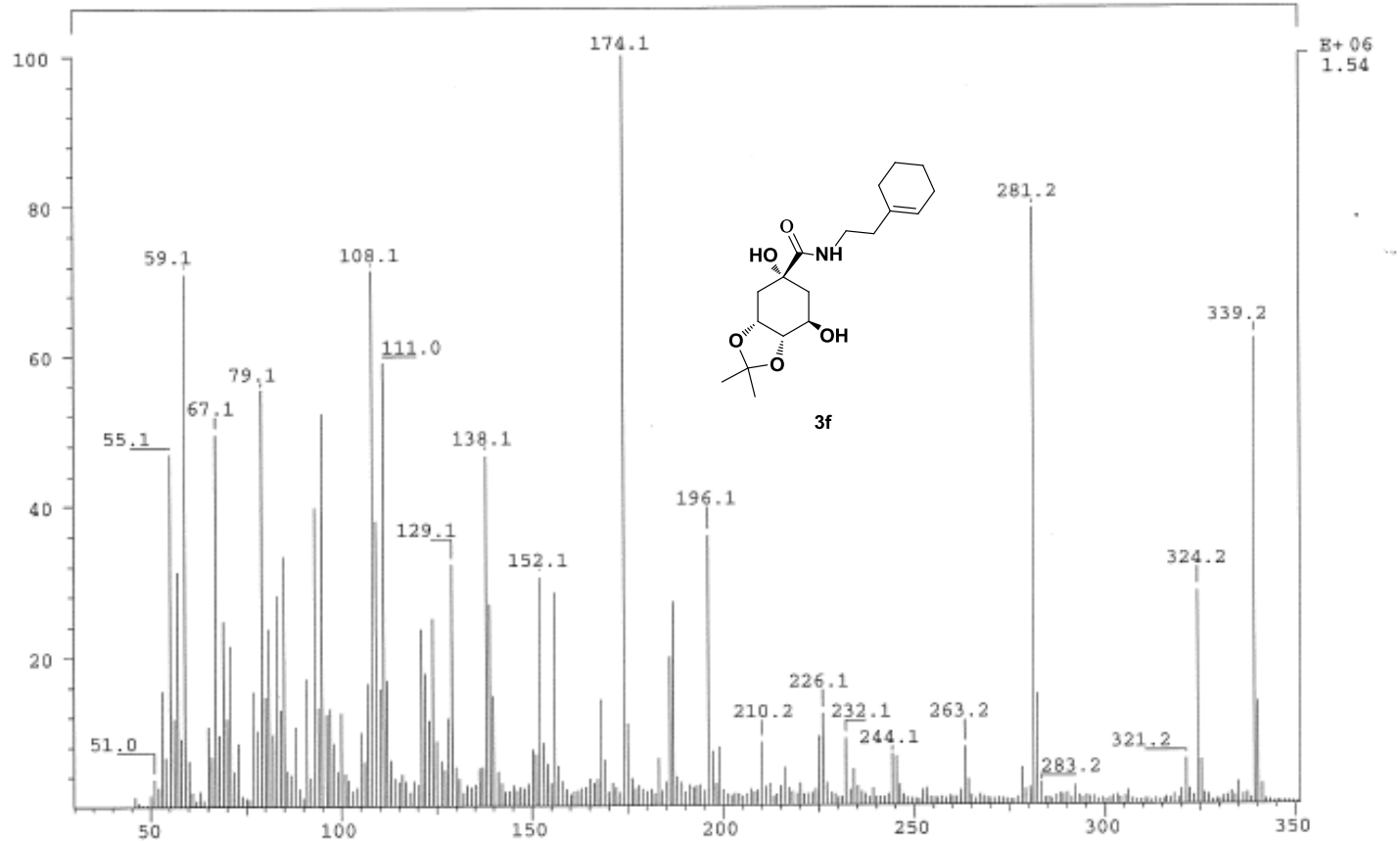
3e


7.26
7.26
6.98
5.45
4.96
4.58
4.58
4.56
4.55
4.54
4.15
4.13
3.80
3.80
3.36
3.34
3.32
3.30
2.45
2.44
2.39
2.39
2.27
2.25
2.22
2.20
2.16
2.14
2.12
2.04
2.02
2.02
1.99
1.98
1.97
1.94
1.92
1.64
1.62
1.61
1.60
1.59
1.58
HO
NH
O
OH
O
O
3f
11
10
9
8
7
6
5
4
3
2
1
ppm
1.00
1.10
0.94
1.05
1.07
1.04
3.22
1.10
0.96
2.43
7.21
3.72
4.15
3.27


176.793
134.568
124.301
108.980
76.311
73.104
72.426
66.121
37.951
37.434
37.312
34.823
28.219
27.382
25.627
24.704
23.199
22.710
HO
NH
O
OH
O
O
3f
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm


102.4
74.60
%TRANSMITTANCE
4000
3000
2000
1000
400.0
Wavenumbers (cm-1)
3739.30
3396.03
2985.27
2929.34
2370.09
2318.02
1646.91
1535.06
1446.35
1375.00
1247.72
1214.93
1162.87
1108.87
1049.09
906.379
838.883
806.099
642.179
518.758
HO
NH
O
OH
O
O
3f

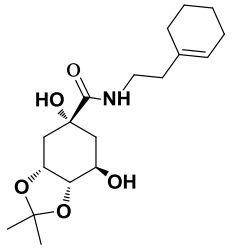
SPEC: 1ei2828 01-Mar-12 REG : 01:22.2 #9
Samp: 1320-leeut-D06 Start : 07:58:47 105
Mode: EI +VE +LMR BSCAN (EXP) UP LR NRM
Oper: Inlet :
Base: 174.1 Inten : 1541023 Masses: 45 > 500
Norm: 174.1 RIC : 34943518 #peaks: 506
Peak: 1000.00 mmu
Data: +/14>41



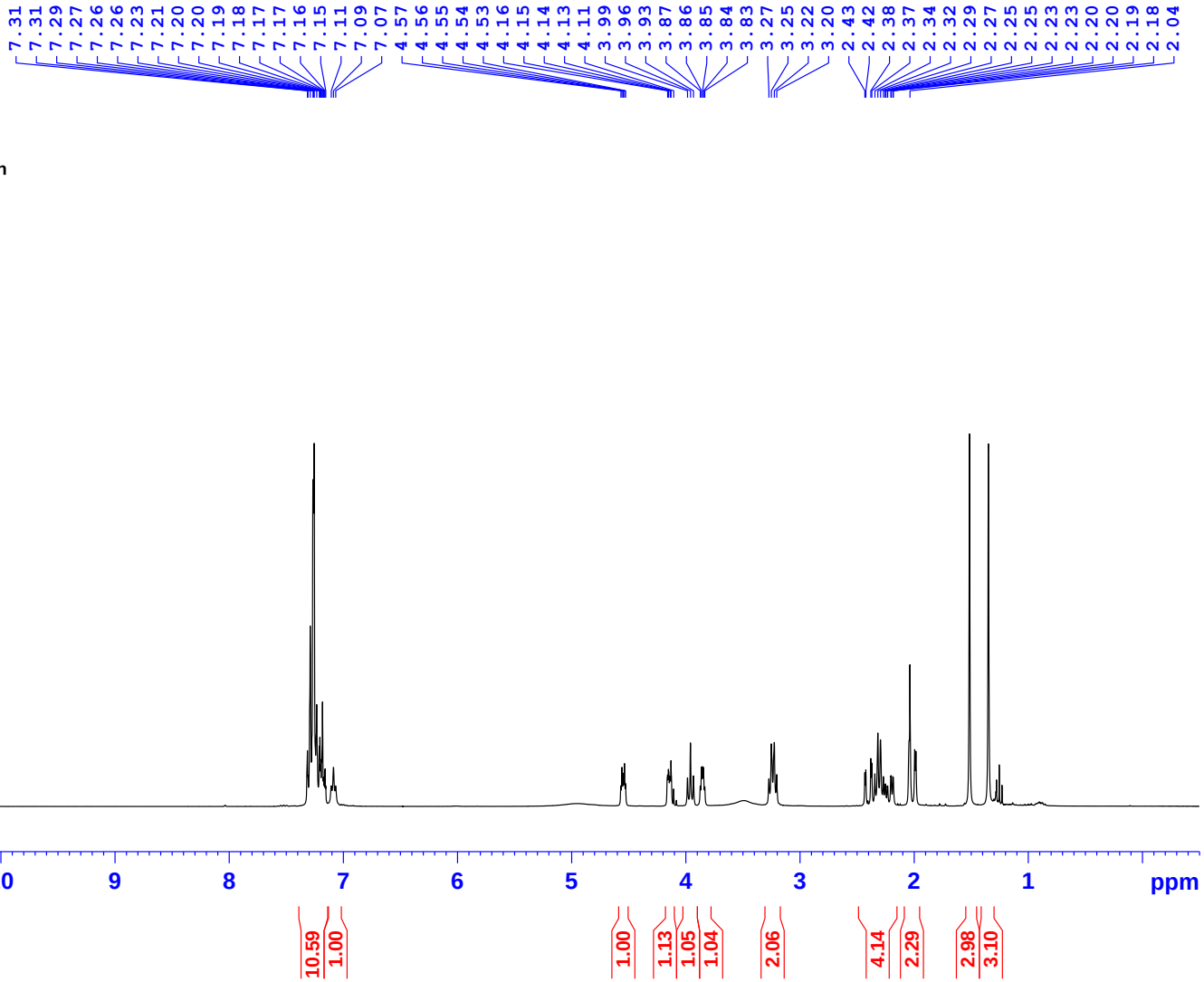
3f

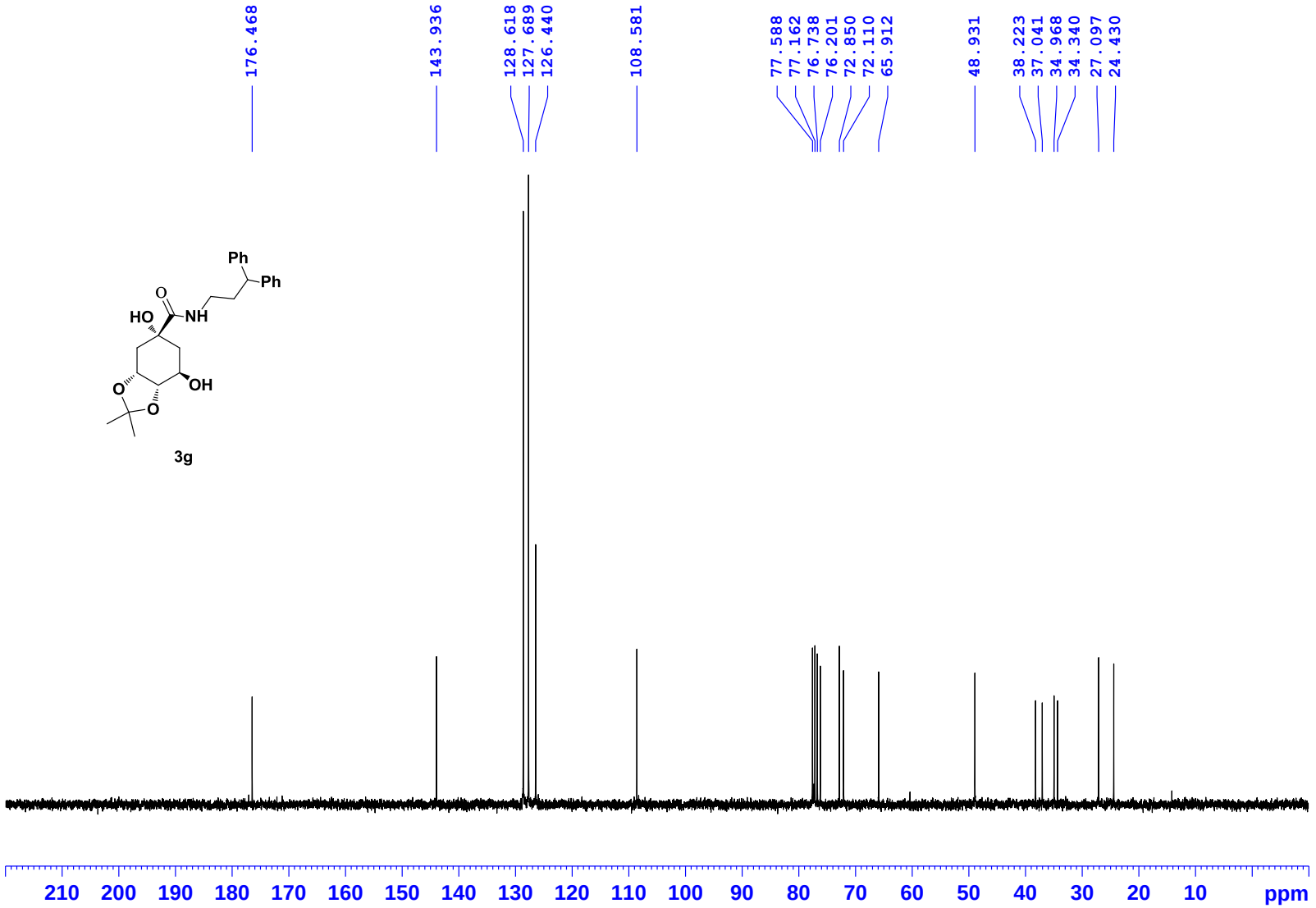
```
LIST: hei1430-c4                              05-Mar-12 REG   : 01:13.4     #9
Samp: 1320leeutD6                                       Start : 21:13:32  2832
Mode: EI +VE +LMR  ESCAN (EXP) UP HR NRM
Oper:                                                   Inlet :
Limt: (  0)     .    . .
    : (440) C25.H30.N.O6
Peak: 1000.00 mmu   R+D: -2.0 > 60.0
Data: +/217>499  (CMASS : converted |CMASS : converted |CMASS : conve
```

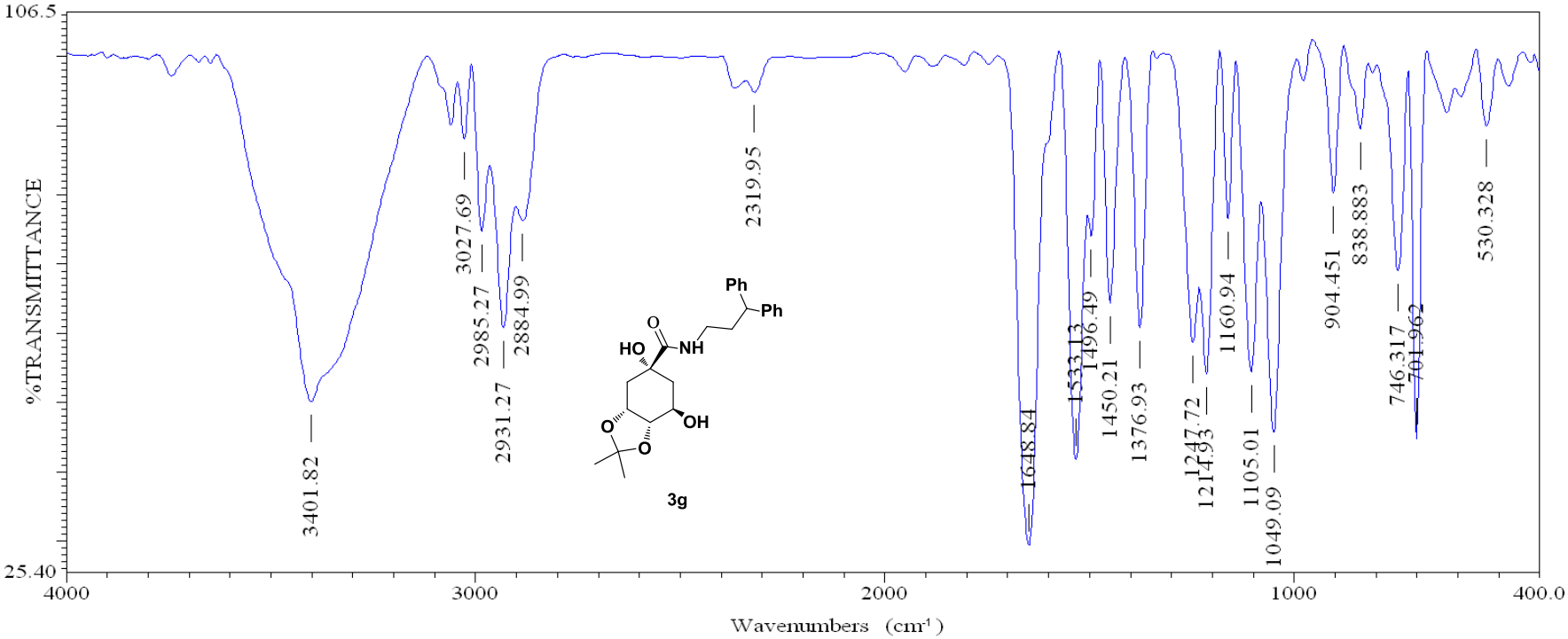
Mass	Intensity	%RA	Flags	(mmu) Delta	R+D	Composition
339.2055	7855517	100.00	#	-0.9	5.0	C18.H29.N.O5

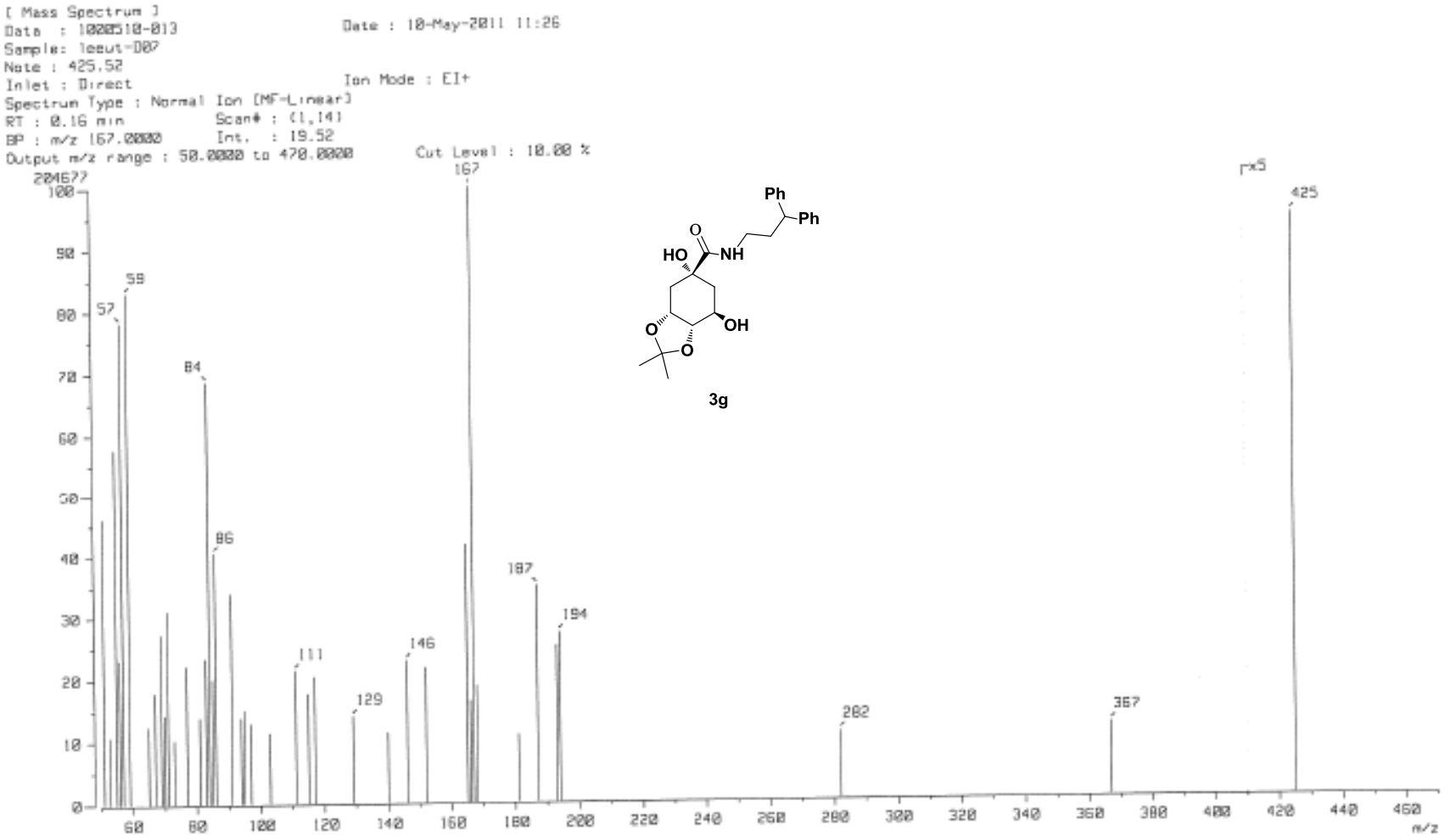


3f


7.31
7.31
7.29
7.27
7.26
7.26
7.23
7.21
7.20
7.20
7.19
7.18
7.17
7.17
7.16
7.15
7.11
7.09
7.07
4.57
4.56
4.55
4.54
4.53
4.16
4.15
4.14
4.13
4.11
3.99
3.96
3.93
3.87
3.86
3.85
3.84
3.83
3.27
3.25
3.22
3.20
2.43
2.42
2.38
2.37
2.34
2.32
2.29
2.27
2.25
2.25
2.23
2.23
2.20
2.20
2.19
2.18
2.04
Ph
Ph
O
HO
NH
OH
O
O
3g
11
10
9
8
7
6
5
4
3
2
1
ppm
10.59
1.00
1.00
1.13
1.05
1.04
2.06
4.14
2.29
2.98
3.10


176.468
143.936
128.618
127.689
126.440
108.581
77.588
77.162
76.738
76.201
72.850
72.110
65.912
48.931
38.223
37.041
34.968
34.340
27.097
24.430
Ph
Ph
O
HO
NH
OH
O
O
3g
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

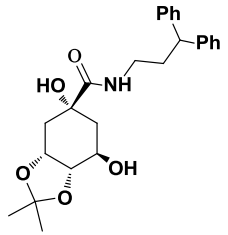

106.5
25.40
%TRANSMITTANCE
4000
3000
2000
1000
400.0
Wavenumbers (cm-1)
3401.82
3027.69
2985.27
2931.27
2884.99
2319.95
1648.84
1533.13
1496.49
1450.21
1376.93
1247.72
1214.93
1160.94
1105.01
1049.09
904.451
838.883
746.317
701.962
530.328
HO
O
NH
Ph
Ph
OH
O
O
3g


[Mass Spectrum]
Data : 1000510-013
Sample: 1esut-007
Note : 425.52
Inlet : Direct
Spectrum Type : Normal Ion (MF-Linear)
RT : 0.16 min
BP : m/z 167.0000
Output m/z range : 50.0000 to 470.0000
Date : 10-May-2011 11:26
Ion Mode : EI+
Scan# : (1,14)
Int. : 19.52
Cut Level : 10.00 %
204677
167
425
59
57
84
86
187
194
111
146
129
282
367
x5
Ph
Ph
O
HO
NH
OH
O
O
3g
m/z

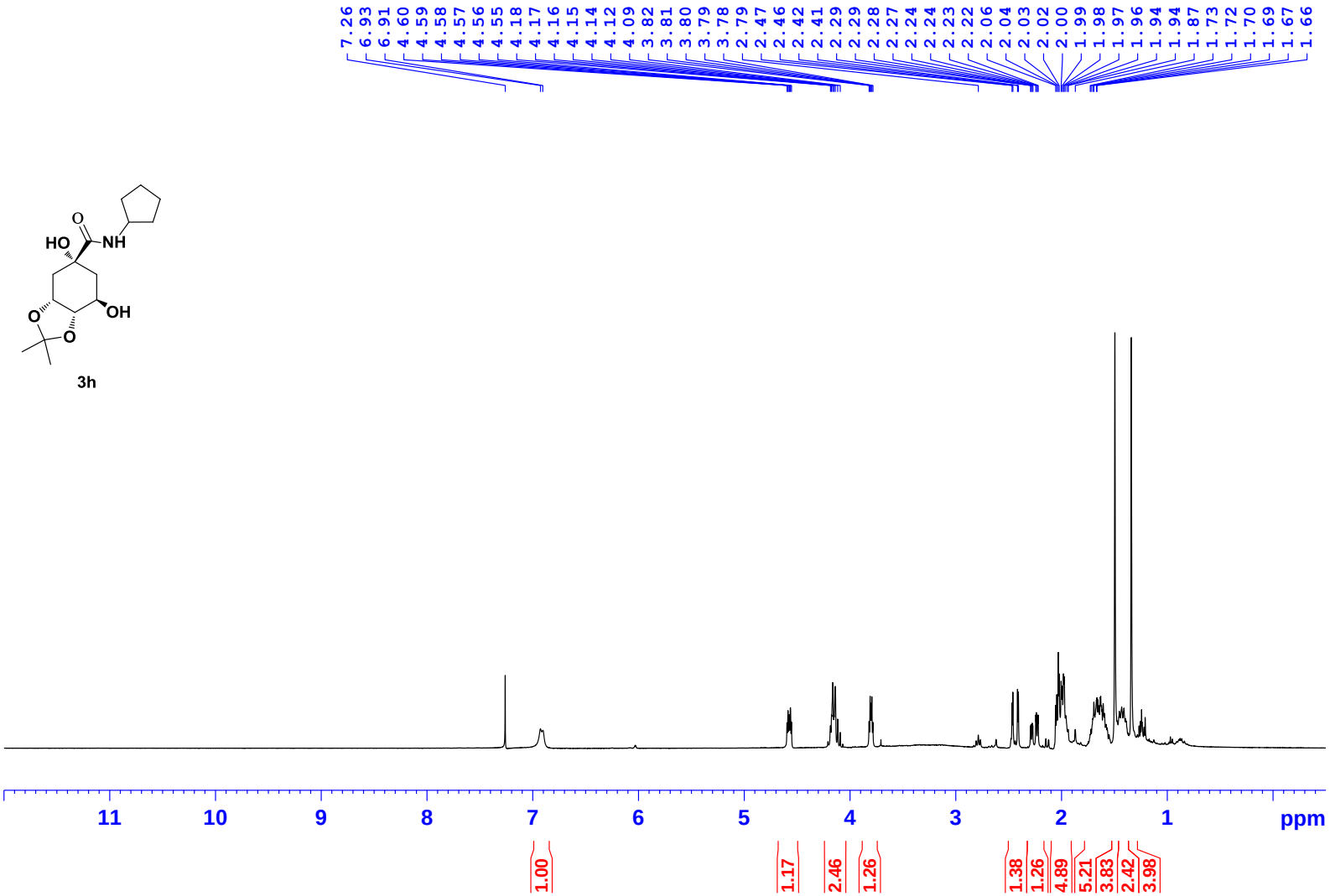
[Elemental Composition]

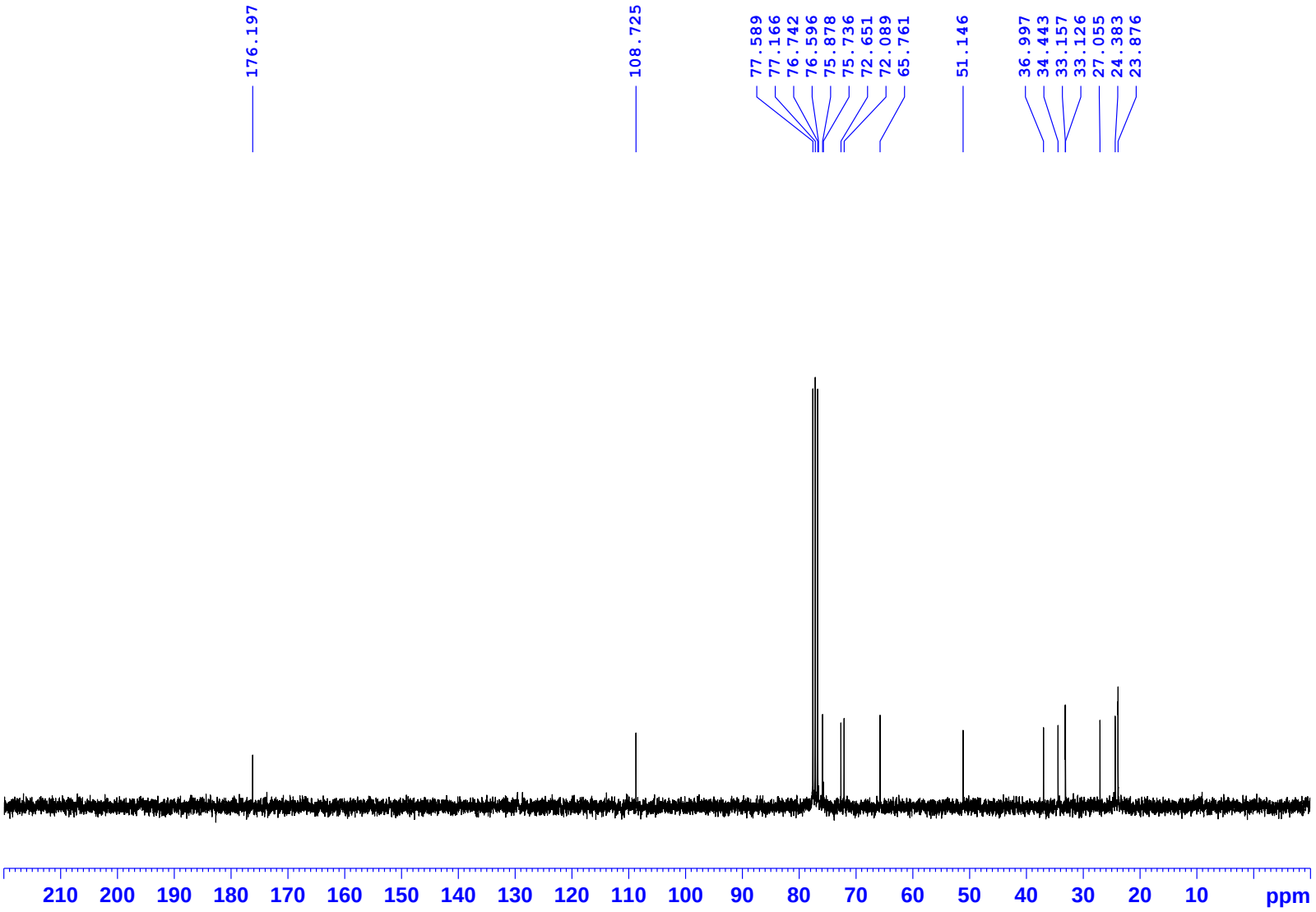
Data : 1000512-010
Date : 12-May-2011 11:39
Sample: Leeut-D07
Note : 425.52
Inlet : Direct
Ion Mode : EI+
RT : 0.52 min
Scan#: 34
Elements : C 25/0, H 36/0, N 1/0, O 5/0
Mass Tolerance : 10ppm, 3mmu if m/z < 300, 20mmu if m/z > 2000
Unsaturation (U.S.) : -0.5 - 200.0

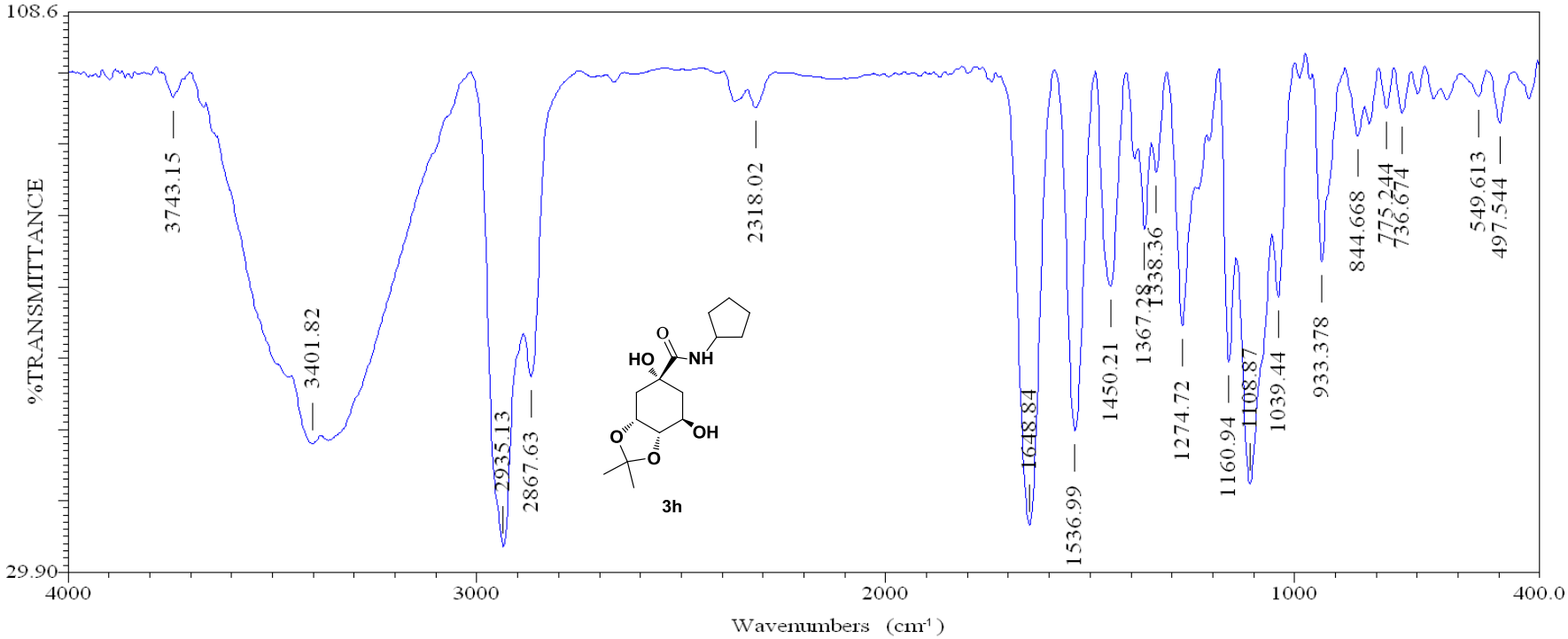
Observed m/z	Int%	Err[ppm / mmu]	U.S.	Composition
425.2202	100.0	-0.1 / +0.0	11.0	C 25 H 31 N O 5

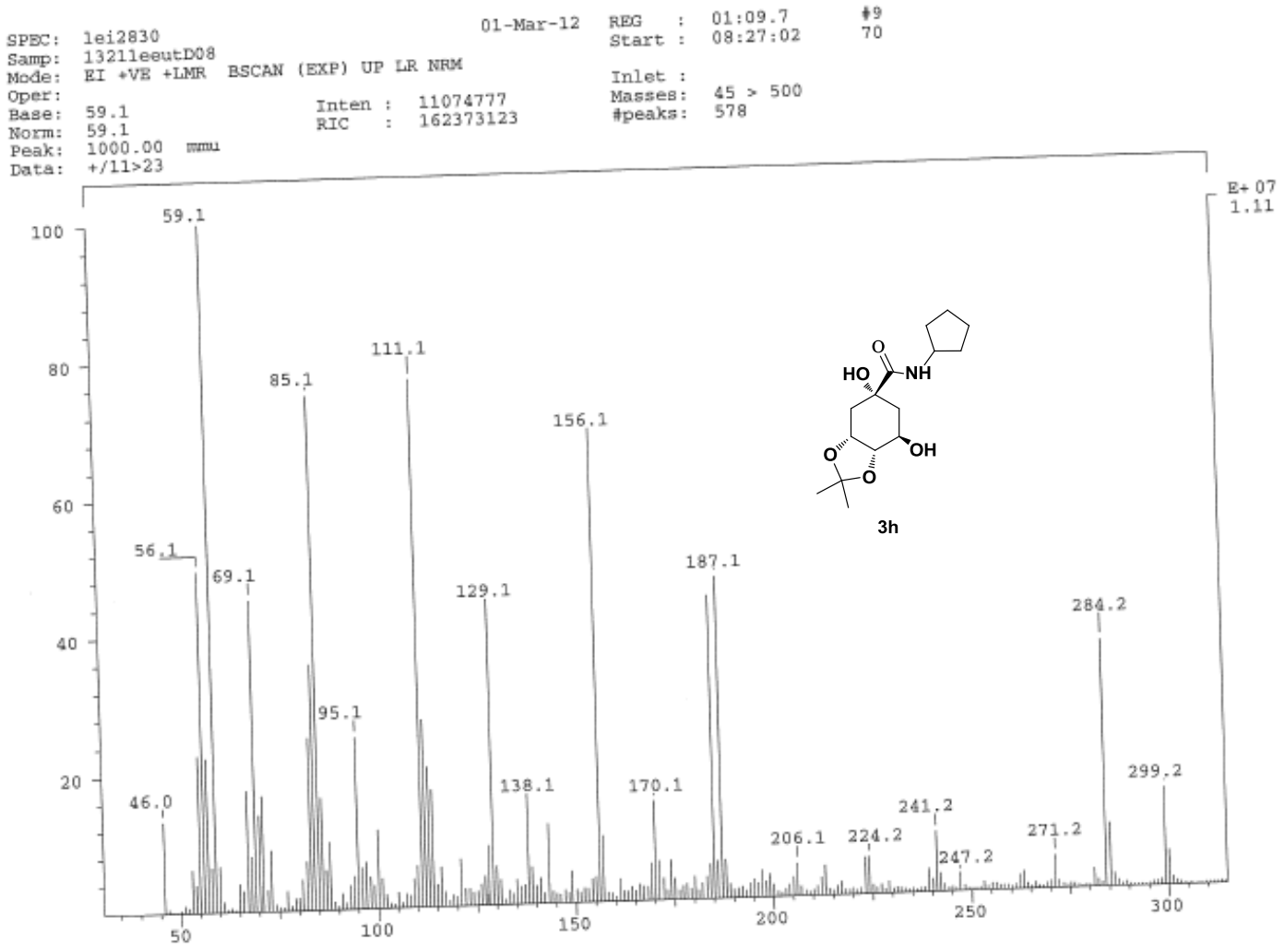


3g


7.26
6.93
6.91
4.60
4.59
4.58
4.57
4.56
4.55
4.18
4.17
4.16
4.15
4.14
4.12
4.09
3.82
3.81
3.80
3.79
3.78
2.79
2.47
2.46
2.42
2.41
2.29
2.29
2.28
2.27
2.24
2.24
2.23
2.22
2.06
2.04
2.03
2.02
2.00
1.99
1.98
1.97
1.96
1.94
1.94
1.87
1.73
1.72
1.70
1.69
1.67
1.66
HO
O
NH
OH
O
O
3h
11
10
9
8
7
6
5
4
3
2
1
ppm
1.00
1.17
2.46
1.26
1.38
1.26
4.89
5.21
3.83
2.42
3.98

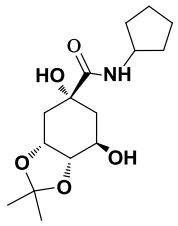

176.197
108.725
77.589
77.166
76.742
76.596
75.878
75.736
72.651
72.089
65.761
51.146
36.997
34.443
33.157
33.126
27.055
24.383
23.876
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm


108.6
29.90
%TRANSMITTANCE
4000
3000
2000
1000
400.0
Wavenumbers (cm-1)
3743.15
3401.82
2935.13
2867.63
2318.02
1648.84
1536.99
1450.21
1367.28
1338.36
1274.72
1160.94
1108.87
1039.44
933.378
844.668
775.244
736.674
549.613
497.544
HO
NH
O
OH
O
O
3h

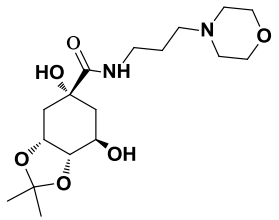

SPEC: 1ei2830
Samp: 1321leeutD08
Mode: EI +VE +LMR BSCAN (EXP) UP LR NRM
Oper:
Base: 59.1
Norm: 59.1
Peak: 1000.00 mmu
Data: +/11>23
01-Mar-12
Inten : 11074777
RIC : 162373123
REG : 01:09.7
Start : 08:27:02
#9
70
Inlet :
Masses: 45 > 500
#peaks: 578
E+07
1.11
59.1
111.1
85.1
156.1
56.1
69.1
129.1
187.1
284.2
95.1
138.1
170.1
299.2
46.0
206.1
224.2
241.2
247.2
271.2
HO
NH
O
OH
O
O
3h

```
LIST: hei1428-c1                                  05-Mar-12 REG    : 02:10.7     #9
Samp: 1321leeutd08                                          Start  : 20:43:59  2166
Mode: EI +VE +LMR  ESCAN (EXP) UP HR NRM
                                                            Inlet  :
Oper:
Limt: (  0)     .     . .
    : (435) C25.H25.N.O6
Peak: 1000.00 mmu   R+D: -2.0 > 60.0
Data: +/402>681  (CMASS : converted |CMASS : converted |CMASS : conve
```

Mass	Intensity	%RA	Flags	(mmu) Delta	R+D	Composition
299.1735	916654	100.00	#	-0.3	4.0	C15.H25.N.O5

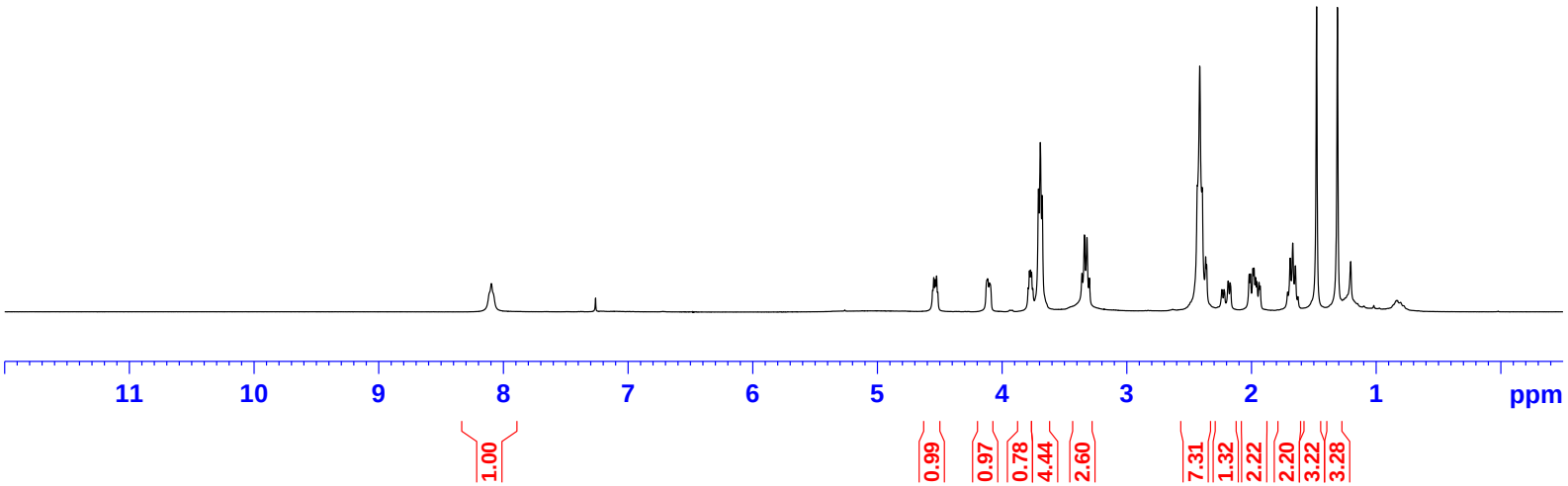


3h

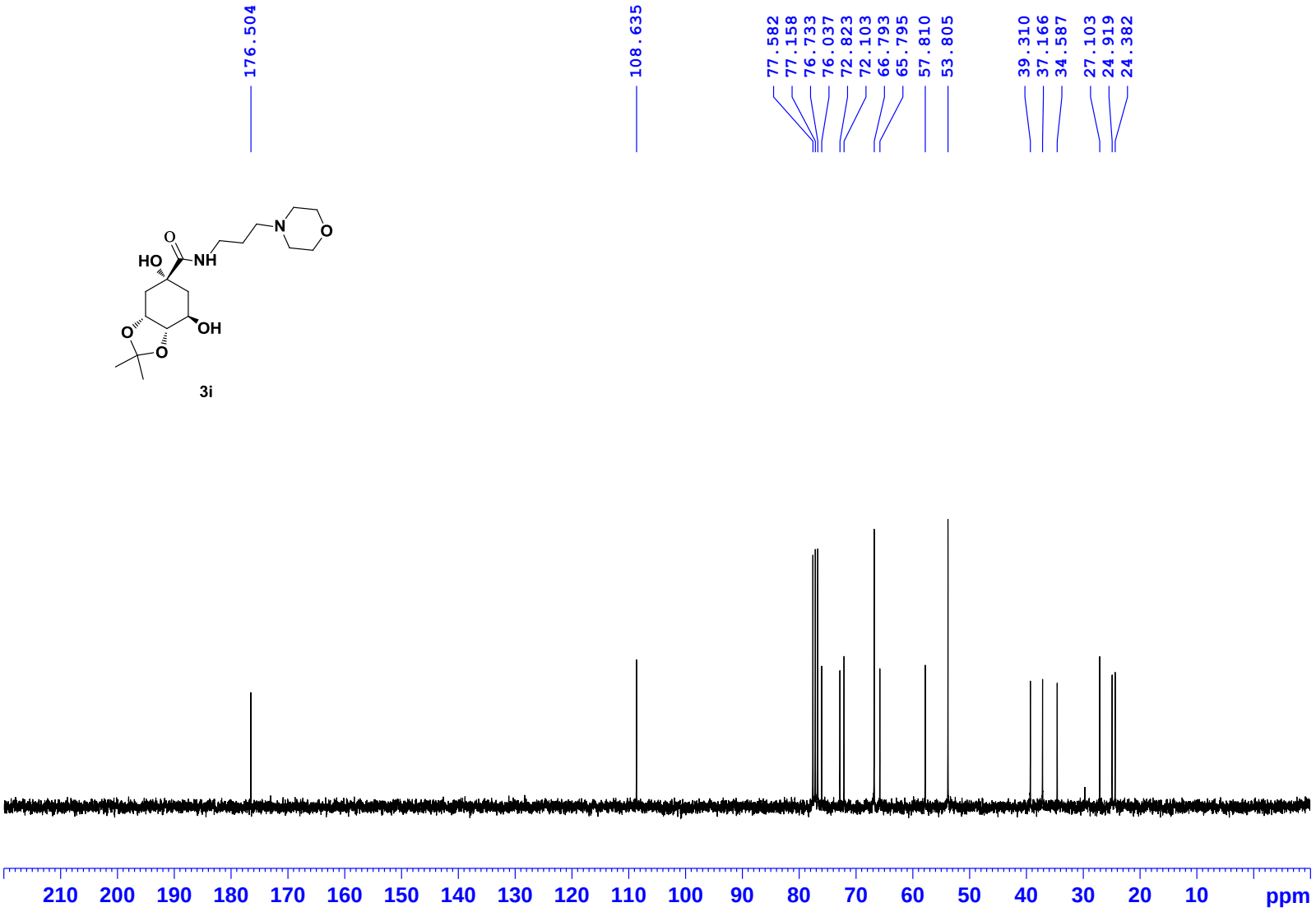


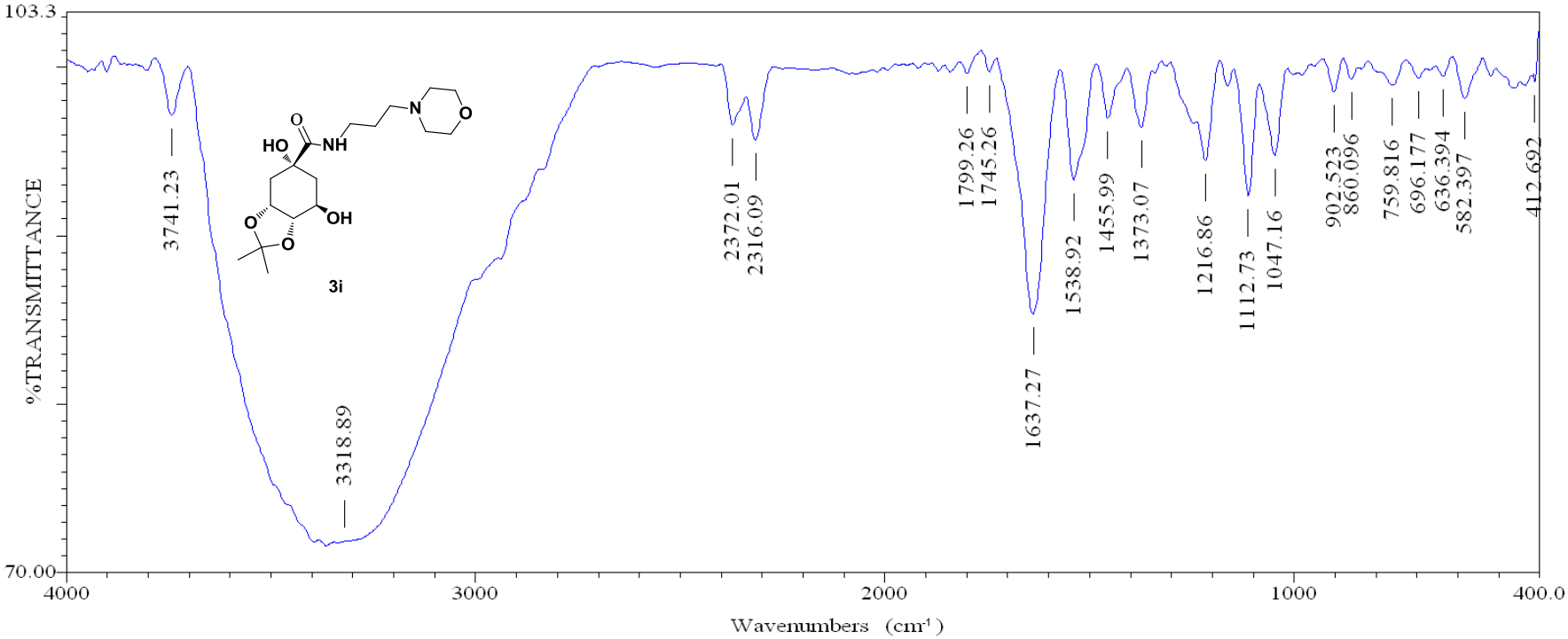
3i

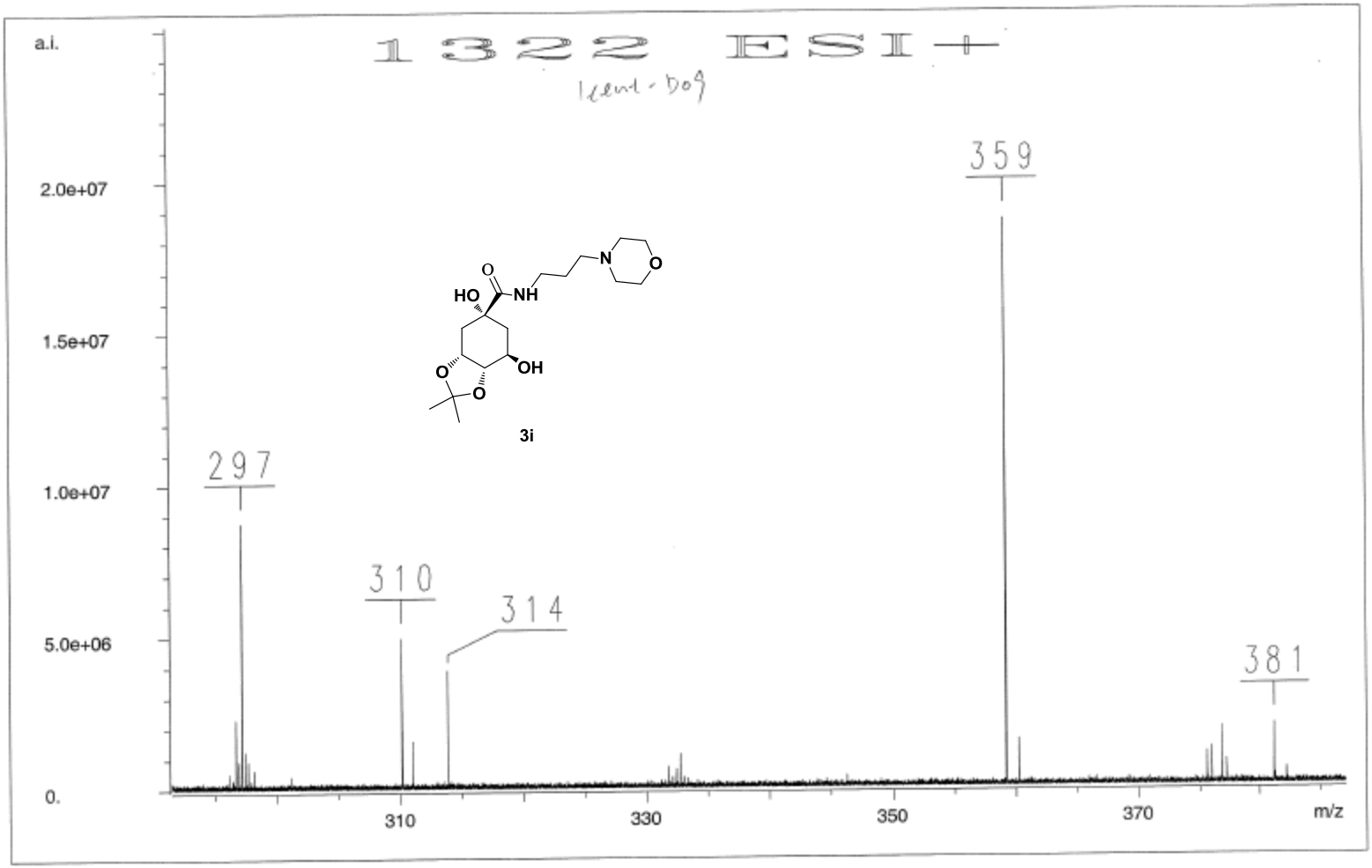
8.10
7.26
4.56
4.55
4.54
4.53
4.52
4.12
4.10
4.09
3.79
3.78
3.77
3.76
3.75
3.71
3.69
3.68
3.36
3.34
3.32
3.30
2.43
2.41
2.39
2.37
2.36
2.24
2.22
2.19
2.17
2.02
2.01
1.99
1.98
1.97
1.96
1.94
1.93
1.71
1.69
1.67
1.65



1.00
0.99
0.97
0.78
4.44
2.60
7.31
1.32
2.22
2.20
3.22
3.28

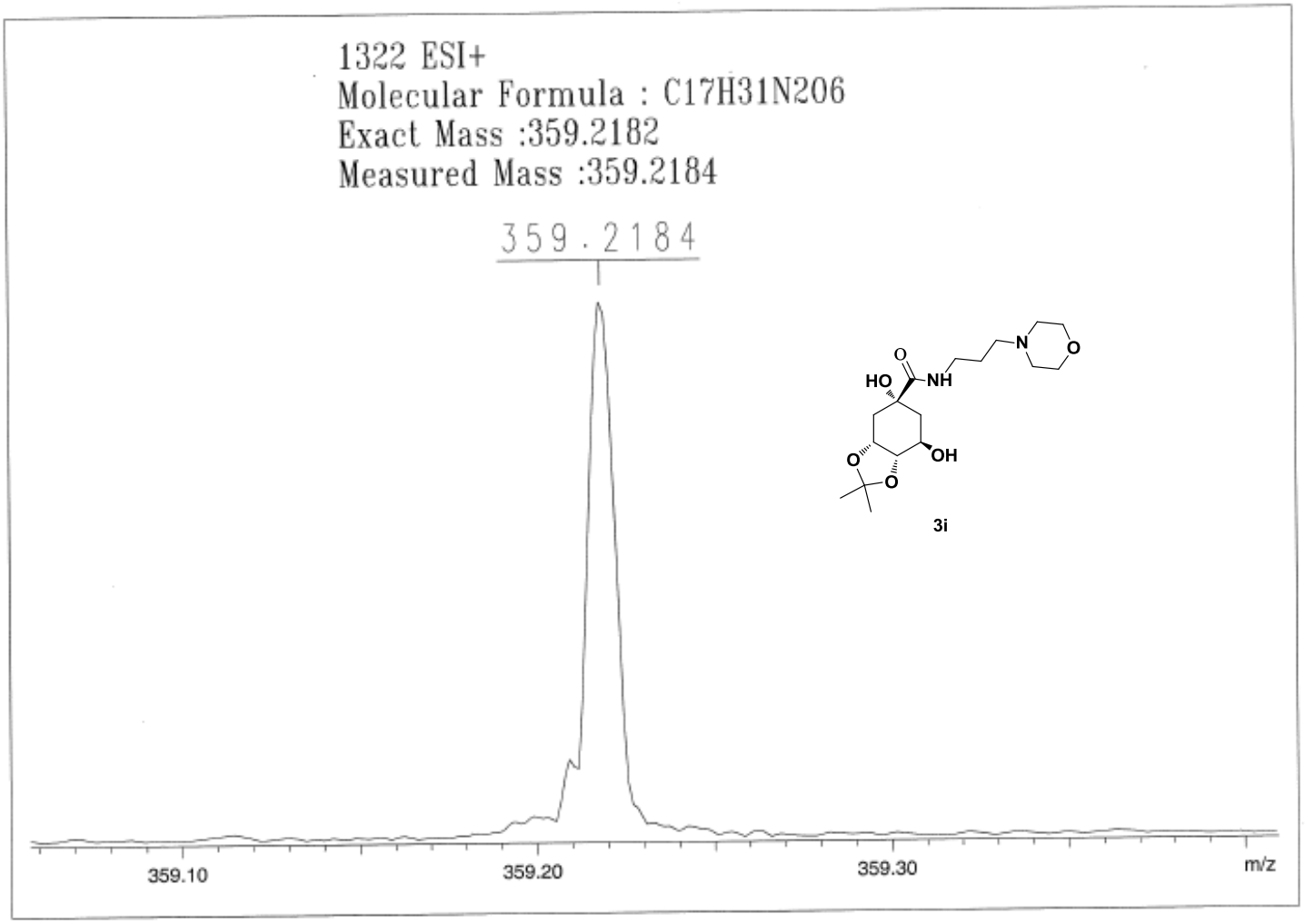

176.504
108.635
77.582
77.158
76.733
76.037
72.823
72.103
66.793
65.795
57.810
53.805
39.310
37.166
34.587
27.103
24.919
24.382
HO
NH
O
N
O
OH
O
O
3i
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm


103.3
70.00
%TRANSMITTANCE
4000
3000
2000
1000
400.0
Wavenumbers (cm-1)
3741.23
3318.89
2372.01
2316.09
1799.26
1745.26
1637.27
1538.92
1455.99
1373.07
1216.86
1112.73
1047.16
902.523
860.096
759.816
696.177
636.394
582.397
412.692
HO
NH
O
N
O
OH
O
O
3i



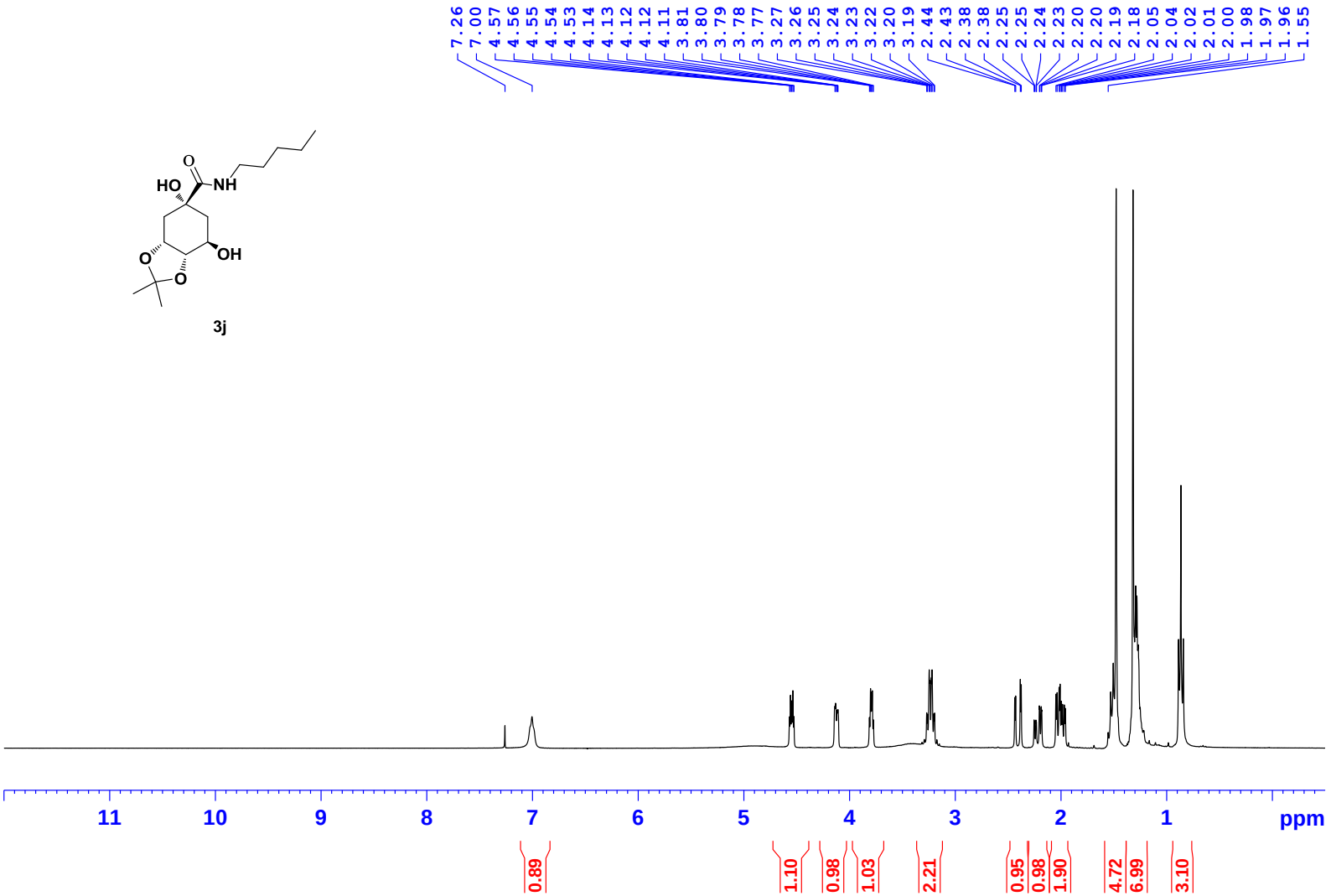
/d=/Data/yu/1322/2/pdata/1 Administrator Wed May 25 15:19:57 2011

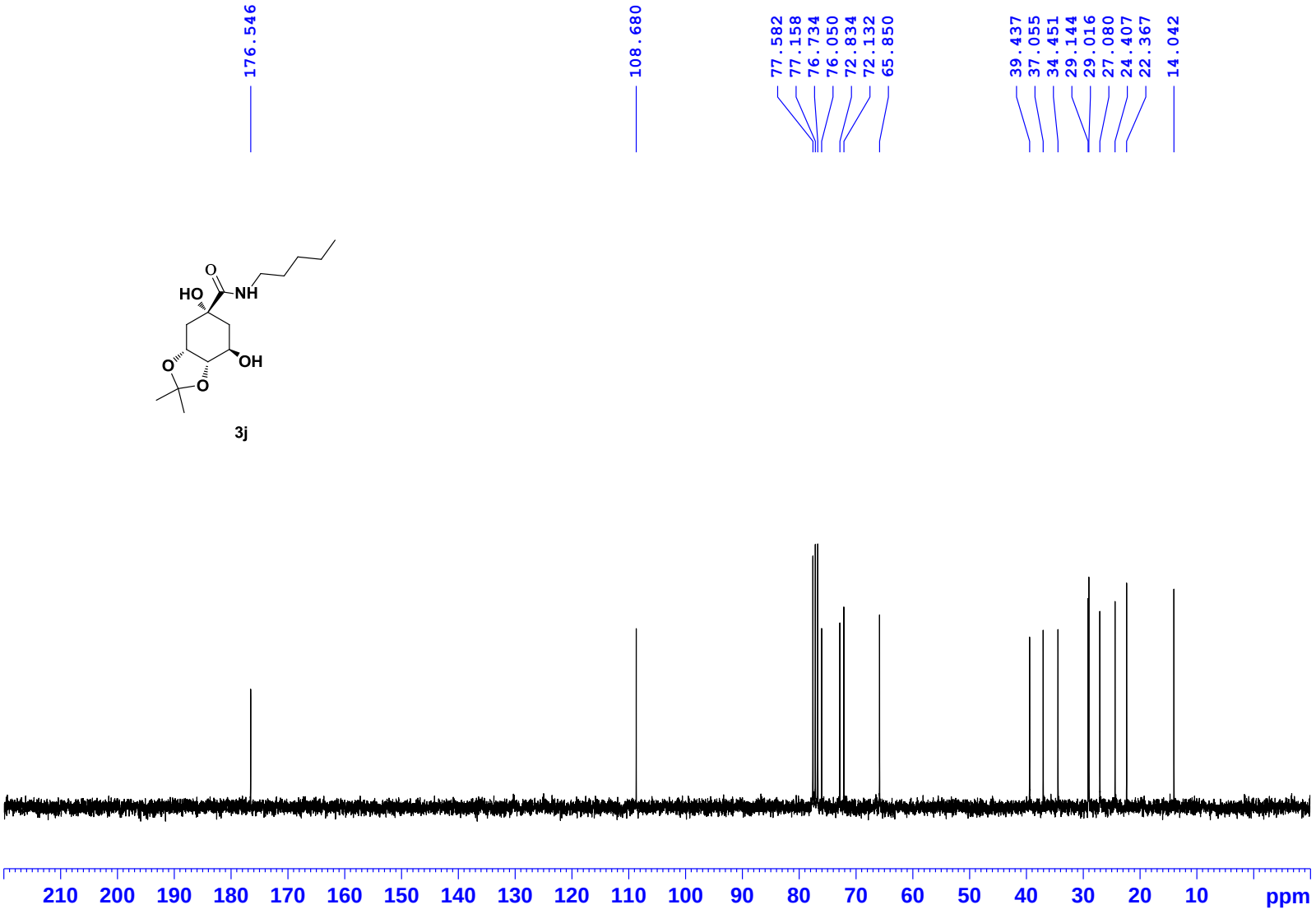
1322 ESI+
Molecular Formula : C17H31N2O6
Exact Mass :359.2182
Measured Mass :359.2184

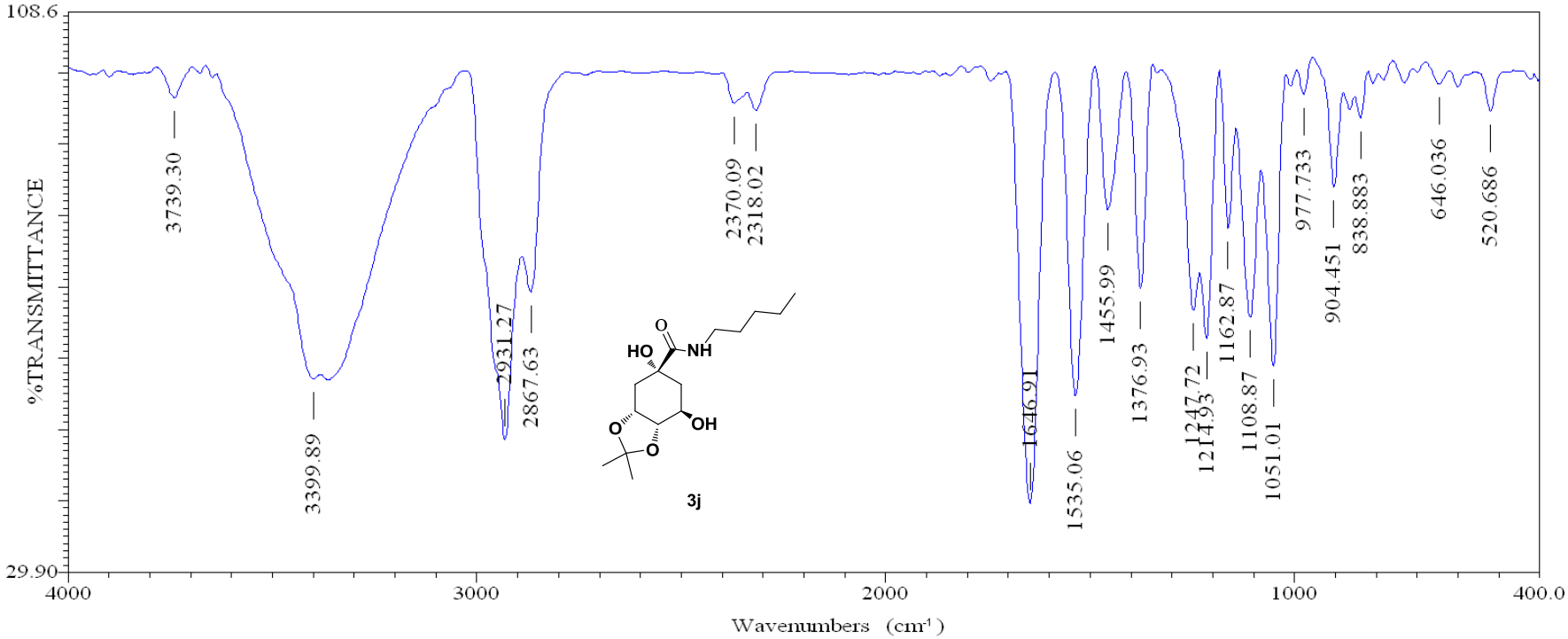


3i

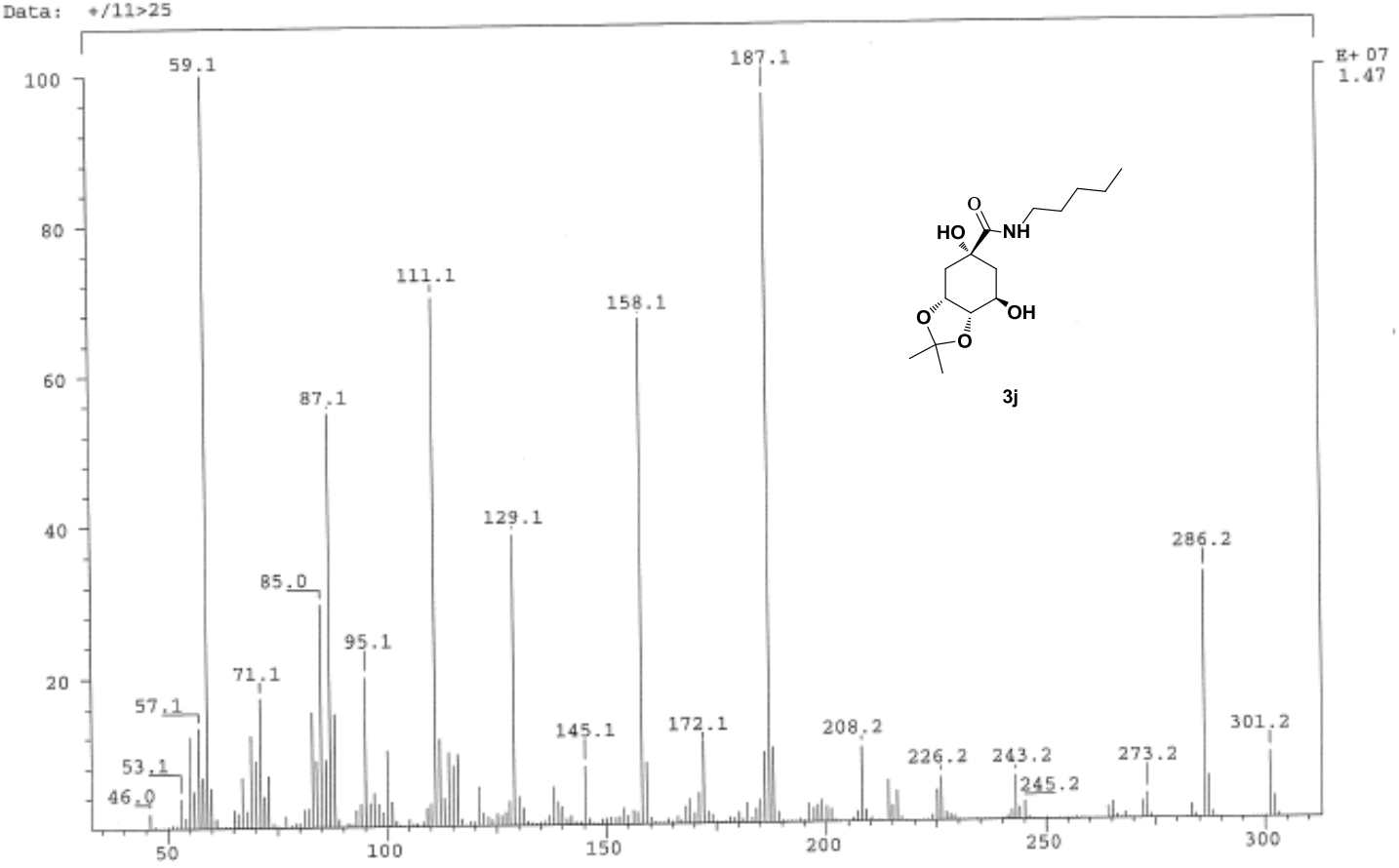
/d=/Data/yu/1322/1/pdata/1 Administrator Wed May 25 15:21:28 2011


7.26
7.00
4.57
4.56
4.55
4.54
4.53
4.14
4.13
4.12
4.11
3.81
3.80
3.79
3.78
3.77
3.27
3.26
3.25
3.24
3.23
3.22
3.20
3.19
2.44
2.43
2.38
2.38
2.25
2.25
2.24
2.23
2.20
2.20
2.19
2.18
2.05
2.04
2.02
2.01
2.00
1.98
1.97
1.96
1.55
HO
O
NH
OH
O
O
3j
11
10
9
8
7
6
5
4
3
2
1
ppm
0.89
1.10
0.98
1.03
2.21
0.95
0.98
1.90
4.72
6.99
3.10


176.546
108.680
77.582
77.158
76.734
76.050
72.834
72.132
65.850
39.437
37.055
34.451
29.144
29.016
27.080
24.407
22.367
14.042
HO
NH
O
OH
O
O
3j
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm


108.6
29.90
%TRANSMITTANCE
3739.30
3399.89
2931.27
2867.63
2370.09
2318.02
1646.91
1535.06
1455.99
1376.93
1247.72
1214.93
1162.87
1108.87
1051.01
977.733
904.451
838.883
646.036
520.686
HO
NH
O
OH
O
O
3j
4000
3000
2000
1000
400.0
Wavenumbers (cm-1)

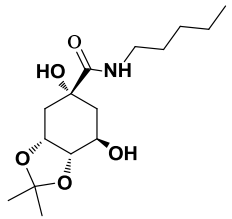
SPEC: lei2846 01-Mar-12 REG : 00:55.2 #9
Samp: 13231eeutD10 Start : 16:12:05 102
Mode: EI +VE +LMR BSCAN (EXP) UP LR NRM
Oper: Inlet :
Base: 59.1 Inten : 14741448 Masses: 45 > 400
Norm: 59.1 RIC : 149894209 #peaks: 431
Peak: 1000.00 mmu
Data: */11>25



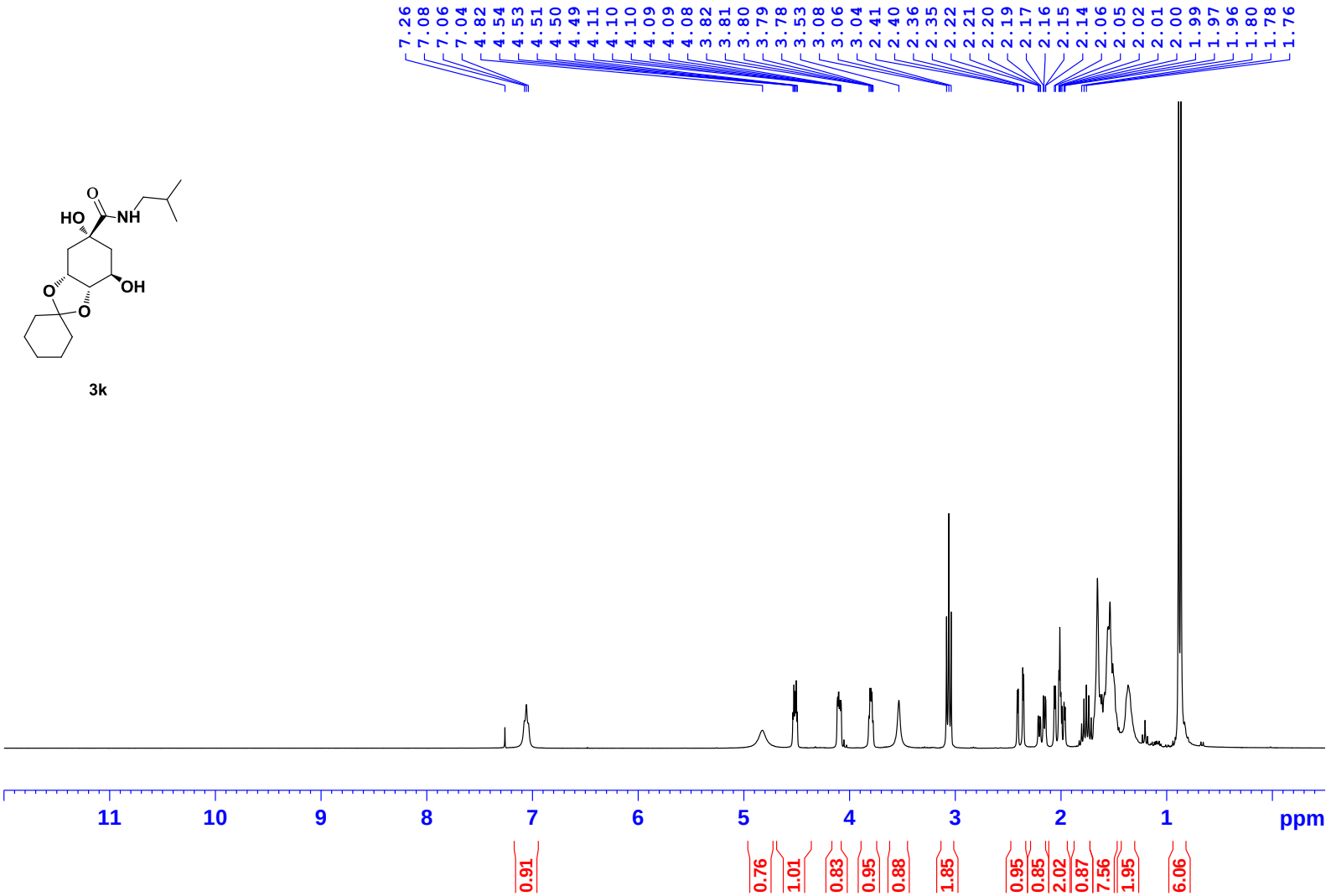
3j

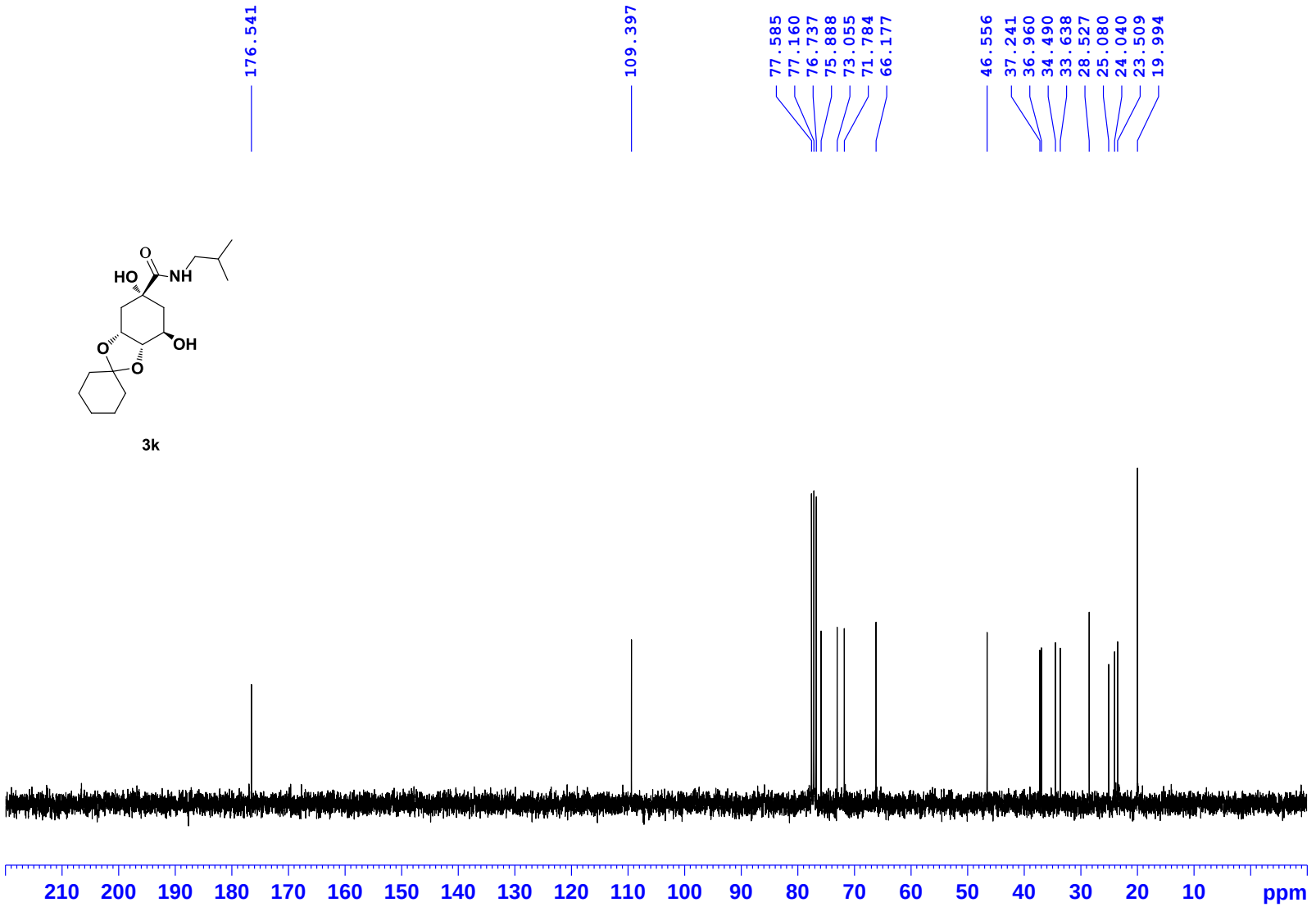
```
LIST: hei1424-c2                              05-Mar-12 REG    : 00:54.6      #9
Samp: 1323leeutd10                                      Start  : 19:31:20    814
Mode: EI +VE +LMR  ESCAN (EXP) UP HR NRM
Oper:                                                   Inlet  :
Limt: (  0)     .     . .
    : (371) C19.H33.N.O6
Peak: 1000.00 mmu   R+D: -2.0 > 60.0
Data: +/163>287  (CMASS : converted |CMASS : converted |CMASS : conve
```

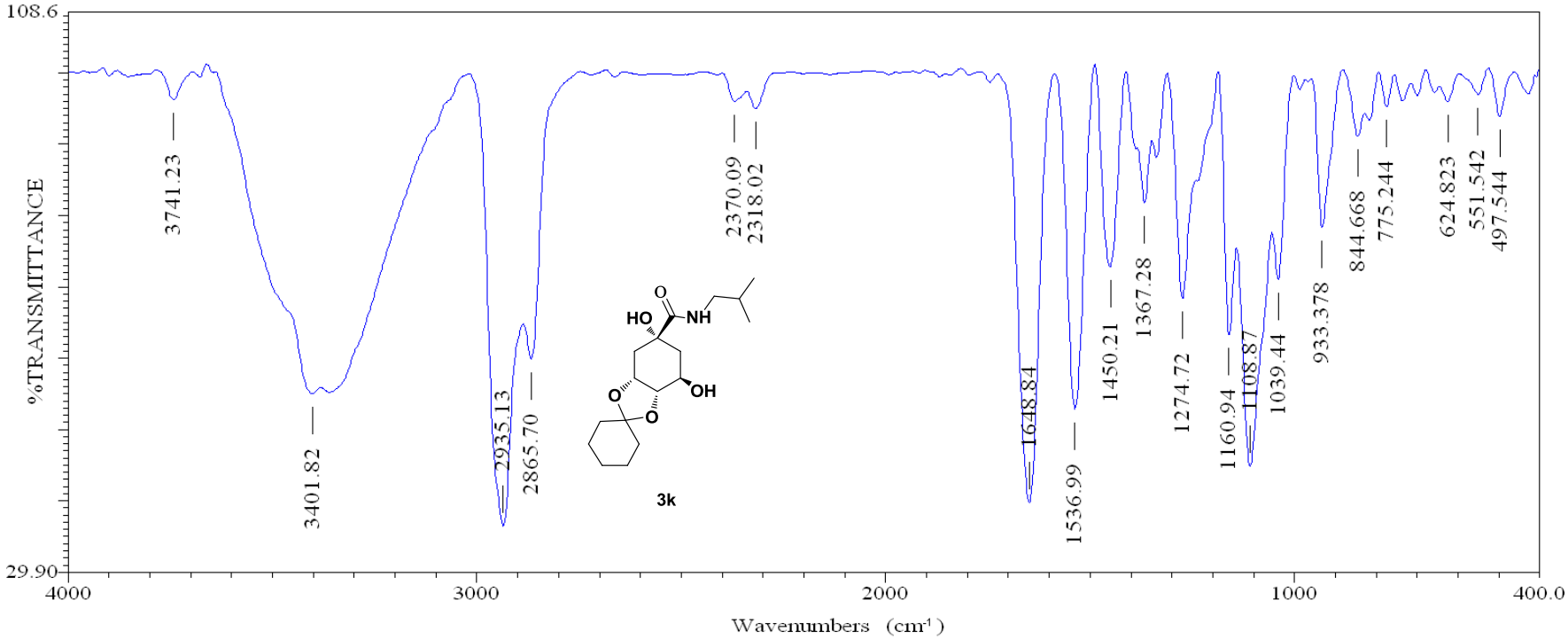
Mass	Intensity	%RA	Flags	(mmu) Delta	R+D	Composition
301.1897	1013276	100.00	#	-0.7	3.0	C15.H27.N.O5

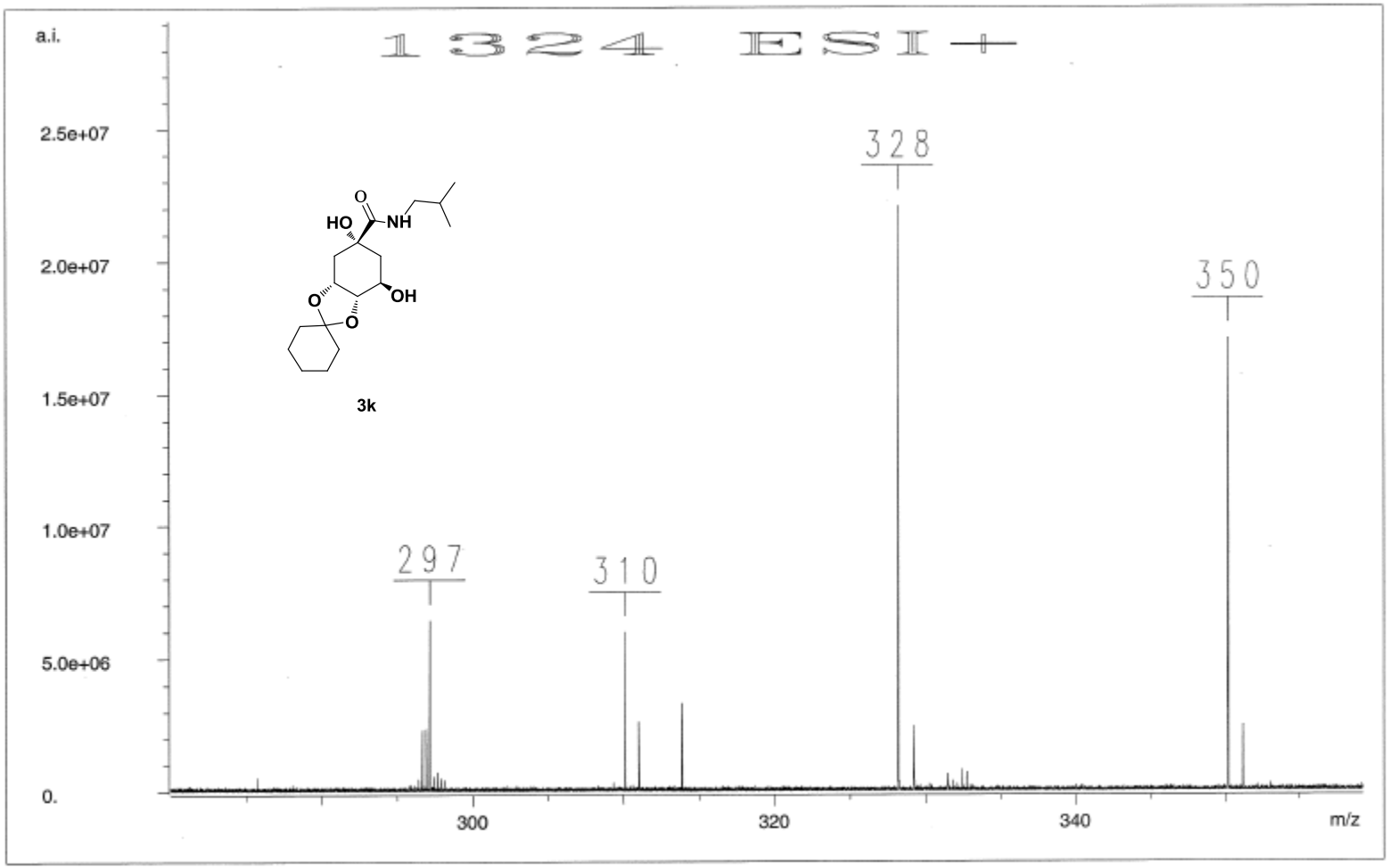


3j

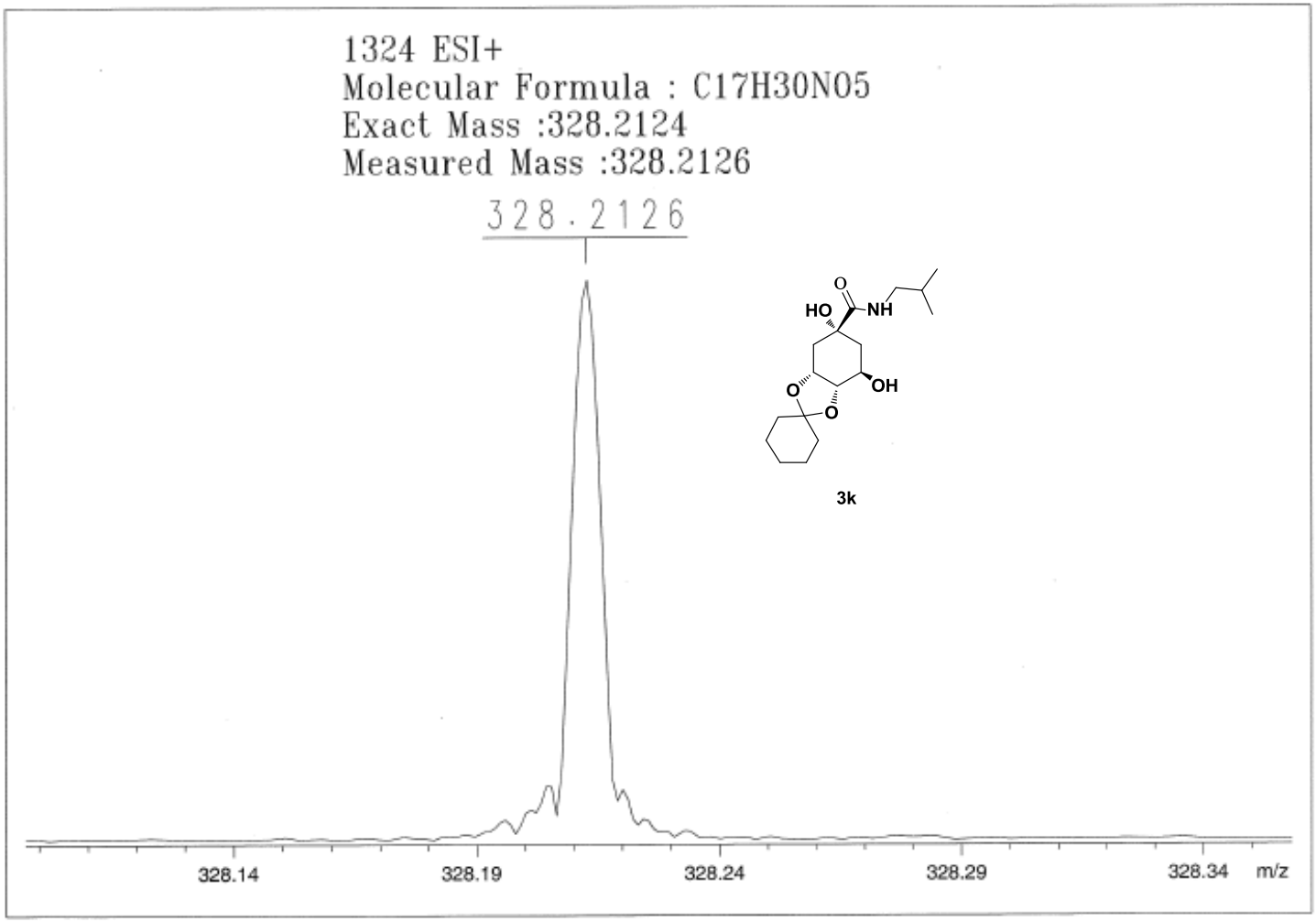

7.26
7.08
7.06
7.04
4.82
4.54
4.53
4.51
4.50
4.49
4.11
4.10
4.10
4.09
4.09
4.08
3.82
3.81
3.80
3.79
3.78
3.53
3.08
3.06
3.04
2.41
2.40
2.36
2.35
2.22
2.21
2.20
2.19
2.17
2.16
2.15
2.14
2.06
2.05
2.02
2.01
2.00
1.99
1.97
1.96
1.80
1.78
1.76
HO
O
NH
OH
O
O
3k
11
10
9
8
7
6
5
4
3
2
1
ppm
0.91
0.76
1.01
0.83
0.95
0.88
1.85
0.95
0.85
2.02
0.87
7.56
1.95
6.06


176.541
109.397
77.585
77.160
76.737
75.888
73.055
71.784
66.177
46.556
37.241
36.960
34.490
33.638
28.527
25.080
24.040
23.509
19.994
HO
O
NH
OH
O
O
3k
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

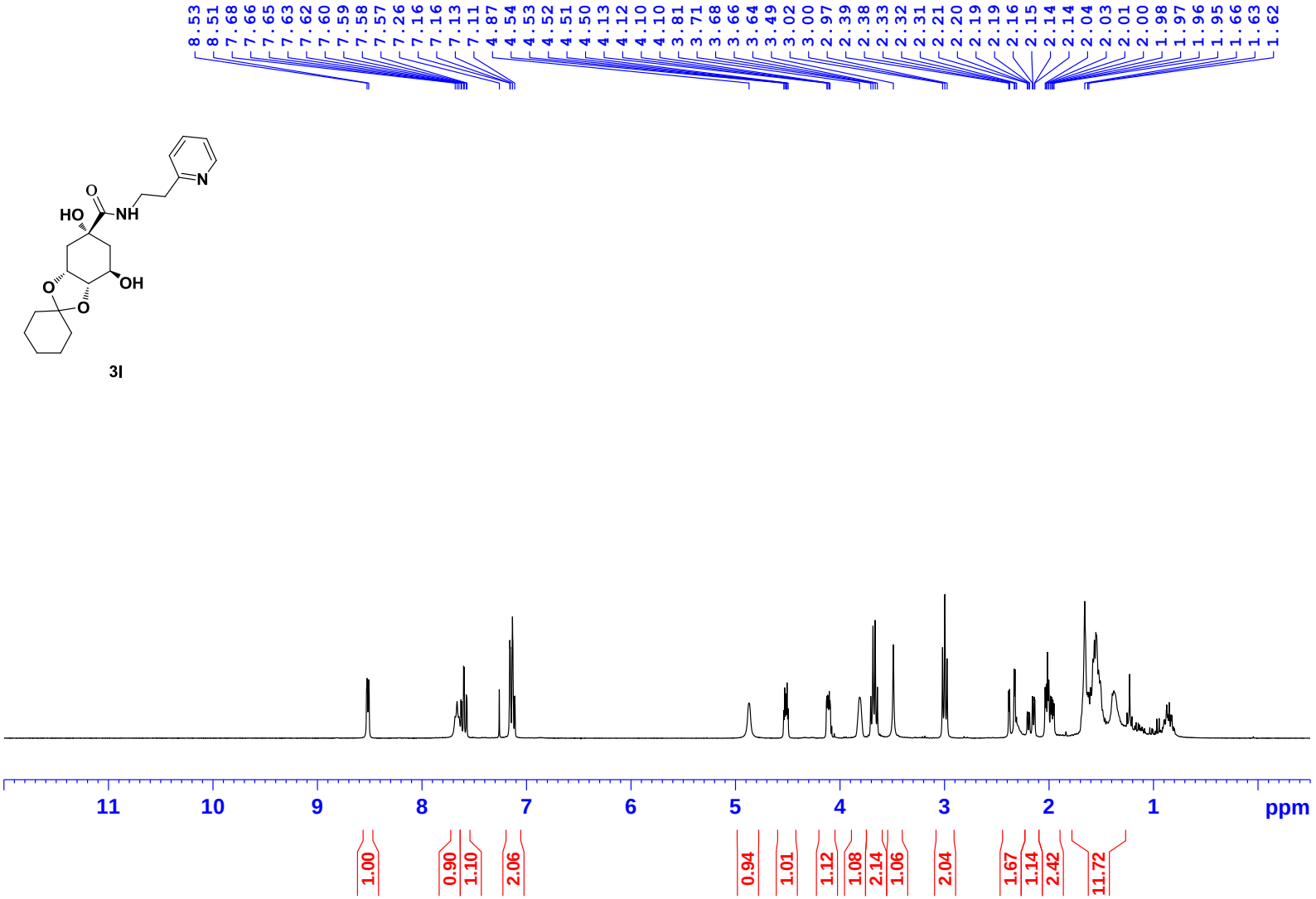

108.6
29.90
%TRANSMITTANCE
3741.23
3401.82
2935.13
2865.70
2370.09
2318.02
1648.84
1536.99
1450.21
1367.28
1274.72
1160.94
1108.87
1039.44
933.378
844.668
775.244
624.823
551.542
497.544
HO
NH
O
OH
O
O
3k
4000
3000
2000
1000
400.0
Wavenumbers (cm-1)



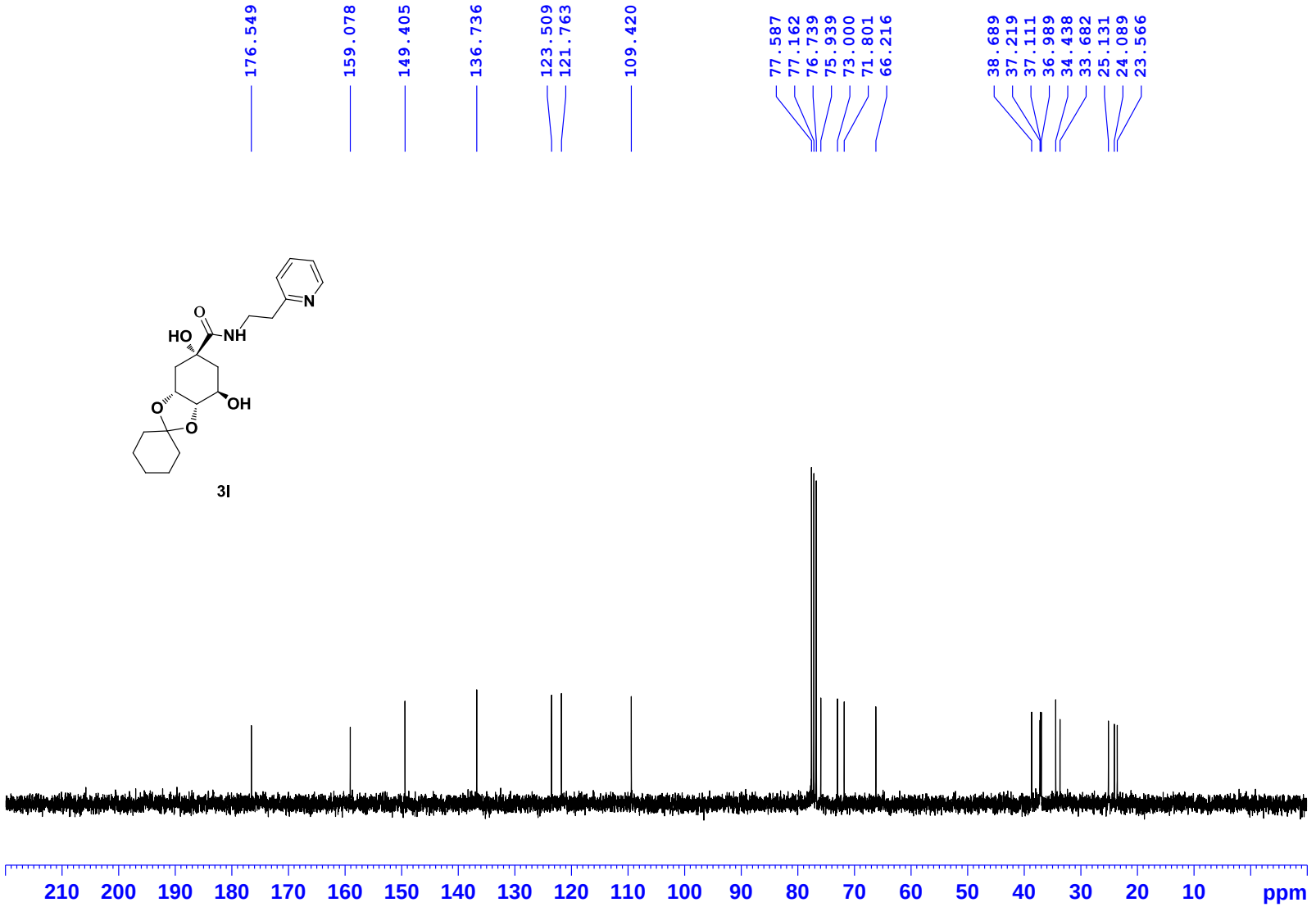
/d=/Data/yu/1324/1/pdata/1 Administrator Wed May 25 15:30:32 2011

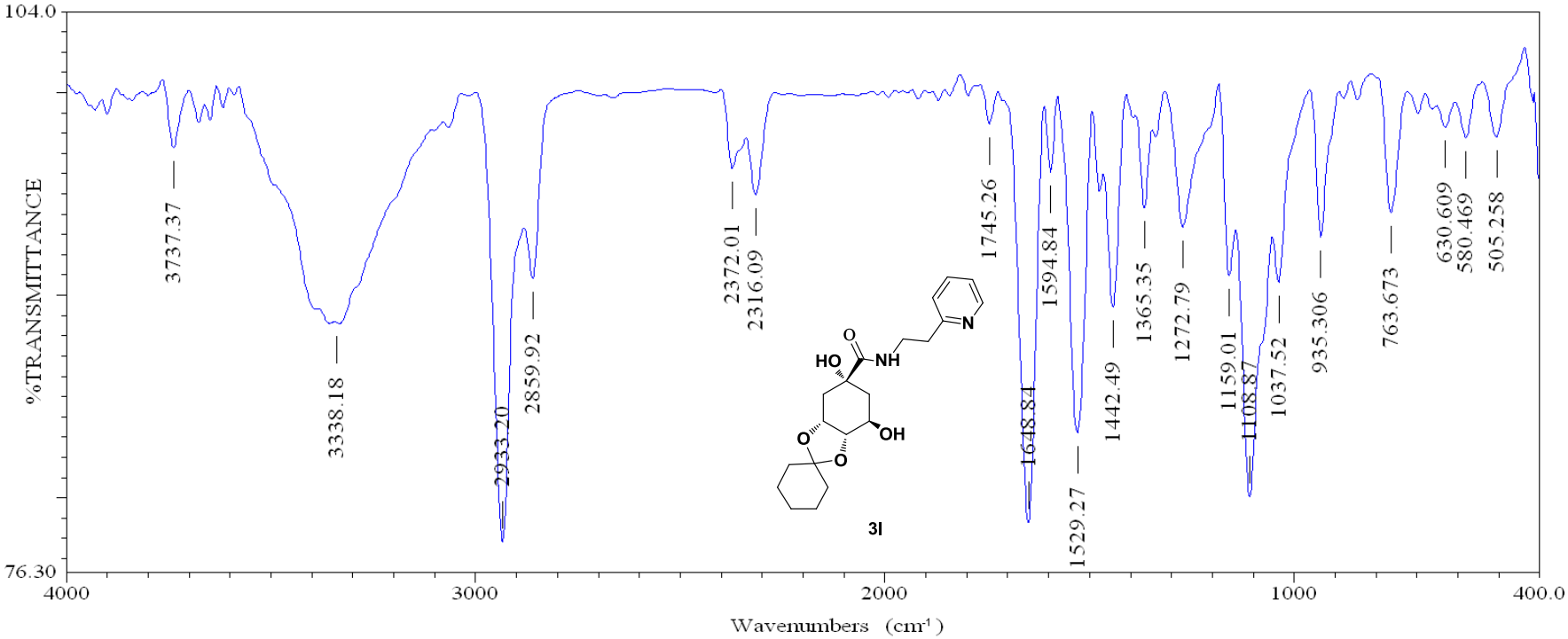


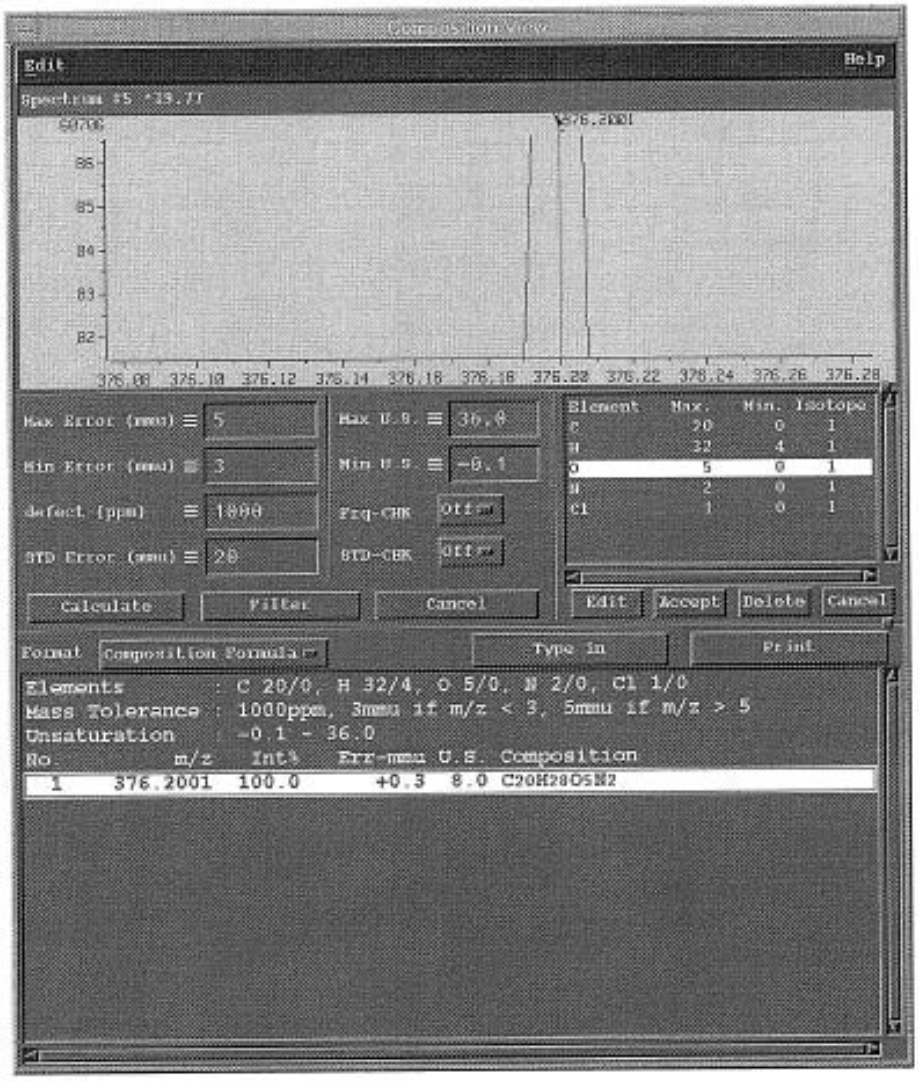
/d=/Data/yu/1324/2/pdata/1 Administrator Wed May 25 15:29:56 2011

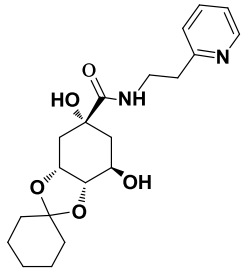

8.53
8.51
7.68
7.66
7.65
7.63
7.62
7.60
7.59
7.58
7.57
7.26
7.16
7.16
7.13
7.11
4.87
4.54
4.53
4.52
4.51
4.50
4.13
4.12
4.10
4.10
3.81
3.71
3.68
3.66
3.64
3.49
3.02
3.00
2.97
2.39
2.38
2.33
2.32
2.31
2.21
2.20
2.19
2.19
2.16
2.15
2.14
2.14
2.04
2.03
2.01
2.00
1.98
1.97
1.96
1.95
1.66
1.63
1.62
HO
O
NH
N
OH
O
O
11
10
9
8
7
6
5
4
3
2
1
ppm
1.00
0.90
1.10
2.06
0.94
1.01
1.12
1.08
2.14
1.06
2.04
1.67
1.14
2.42
11.72

3l


176.549
159.078
149.405
136.736
123.509
121.763
109.420
77.587
77.162
76.739
75.939
73.000
71.801
66.216
38.689
37.219
37.111
36.989
34.438
33.682
25.131
24.089
23.566
HO
O
NH
N
OH
O
O
3l
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm


104.0
76.30
%TRANSMITTANCE
3737.37
3338.18
2933.20
2859.92
2372.01
2316.09
1745.26
1648.84
1594.84
1529.27
1442.49
1365.35
1272.79
1159.01
1108.87
1037.52
935.306
763.673
630.609
580.469
505.258
HO
NH
O
N
OH
O
O
3I
4000
3000
2000
1000
400.0
Wavenumbers (cm-1)

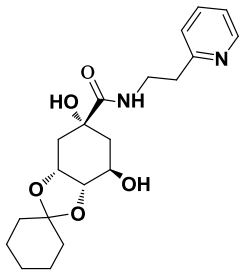

Composition View
Edit
Help
Spectrum #5 *13.77
376.2001
Max Error (mmu) 5
Min Error (mmu) 3
defect (ppm) 1000
STD Error (mmu) 20
Max U.S. 36.0
Min U.S. -0.1
Frq-CHK Off
STD-CHK Off
Element Max. Min. Isotope
C 20 0 1
H 32 4 1
O 5 0 1
N 2 0 1
Cl 1 0 1
Calculate
Filter
Cancel
Edit
Accept
Delete
Cancel
Format Composition Formula
Type In
Print
Elements : C 20/0, H 32/4, O 5/0, N 2/0, Cl 1/0
Mass Tolerance : 1000ppm, 3mmu if m/z < 3, 5mmu if m/z > 5
Unsaturation : -0.1 - 36.0
No. m/z Int% Err-mmu U.S. Composition
1 376.2001 100.0 +0.3 8.0 C20H28O5N2


HO
O
NH
N
OH
O
O

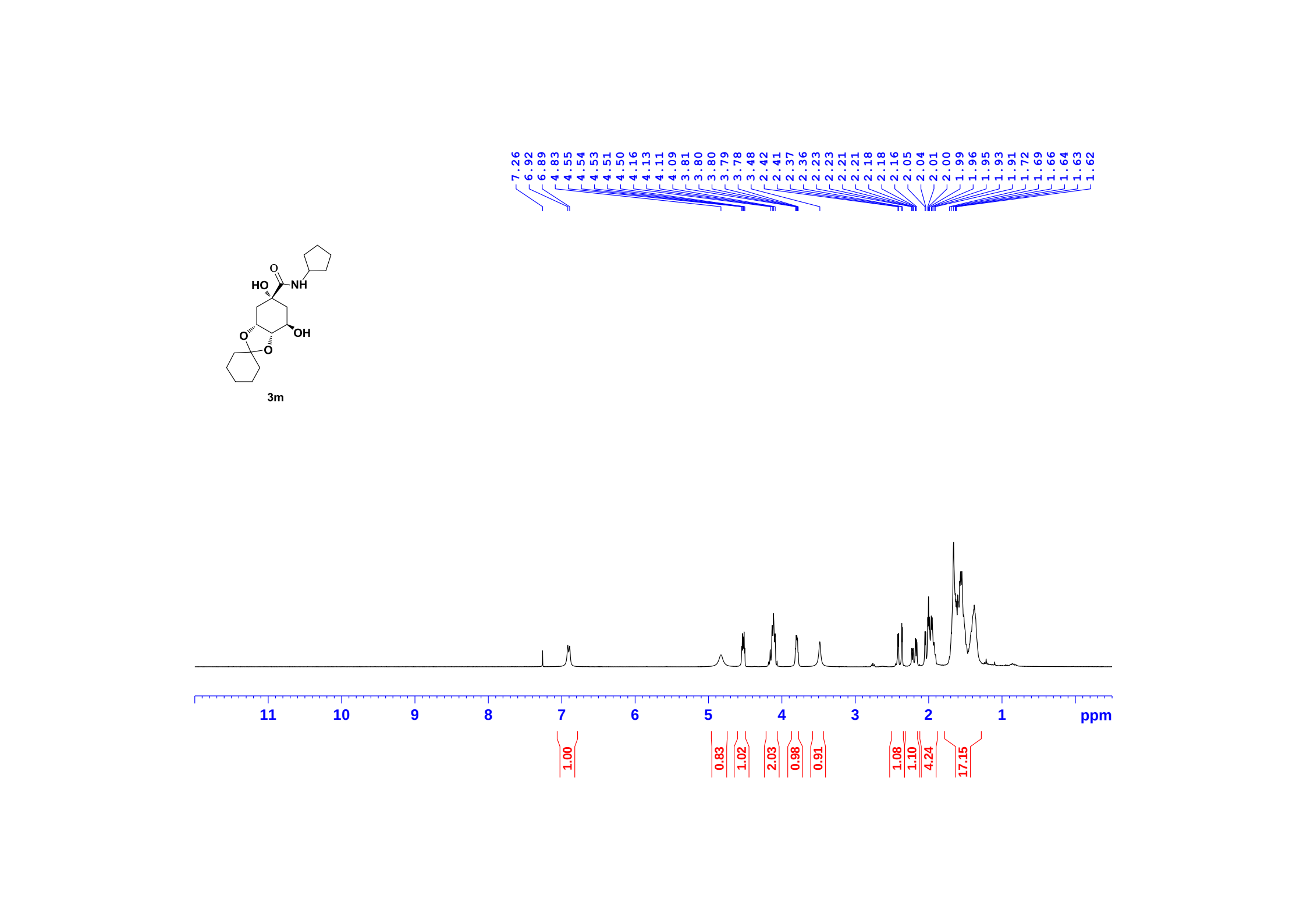
3l

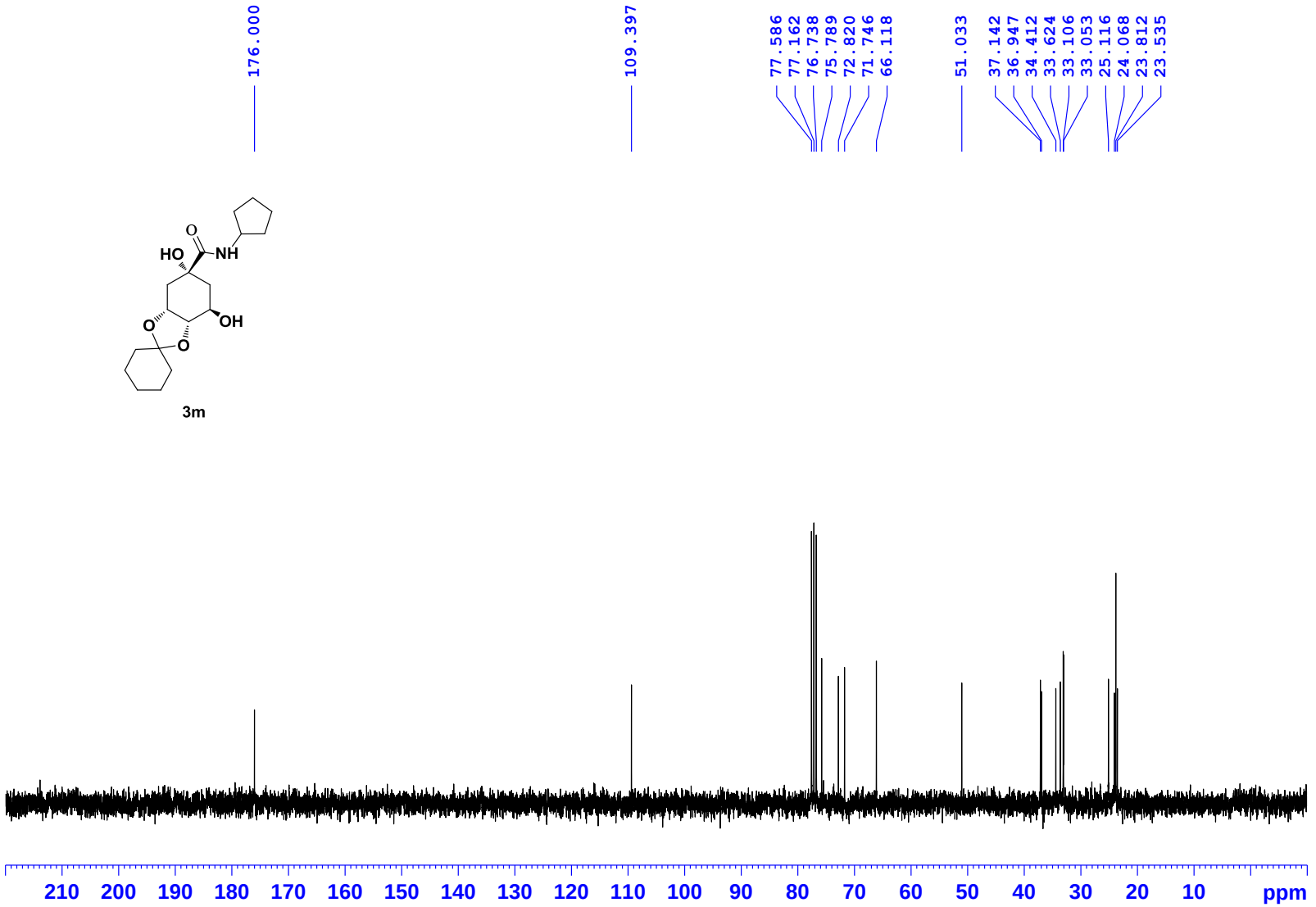
[Elemental Composition]
Data : 201105148 Date : 31-May-2011 17:22
Sample: leeut D-12
Note : -
Inlet : Direct Ion Mode : EI+
RT : 0.40 min Scan#: 17
Elements : C 20/0, H 32/4, O 5/0, N 2/0, Cl 1/0
Mass Tolerance : 1000ppm, 3mmu if m/z < 3, 5mmu if m/z > 5
Unsaturation (U.S.) : -0.1 - 36.0

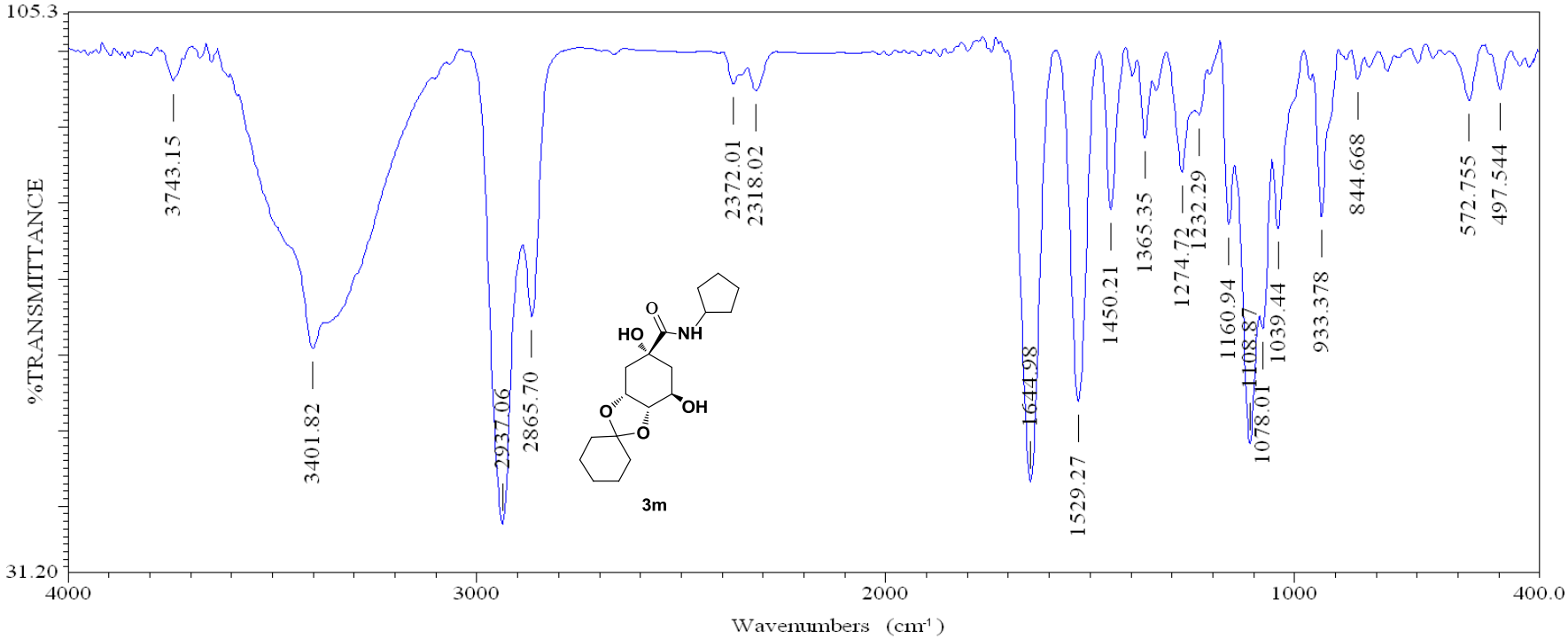
Observed m/z	Int%	Err[ppm / mmu]	U.S.	Composition
376.2001	100.0	+0.8 / +0.3	8.0	C 20 H 28 O 5 N 2

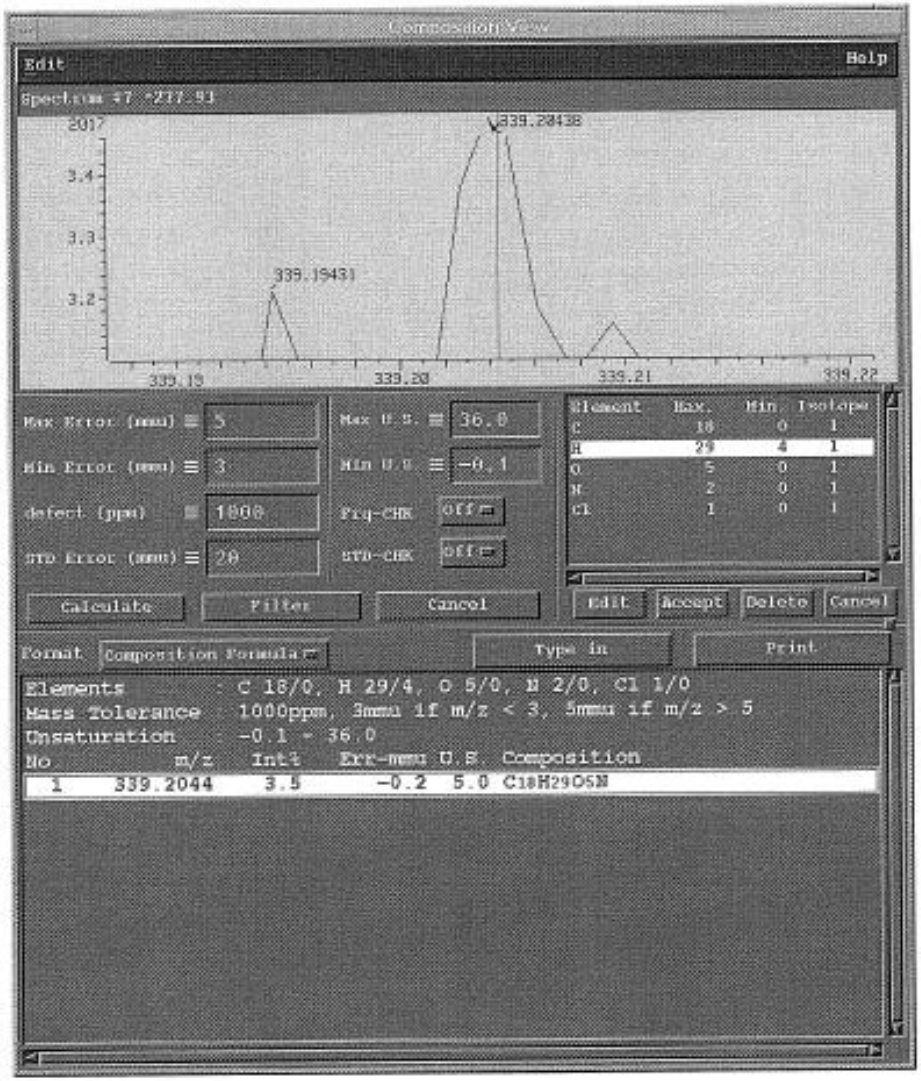


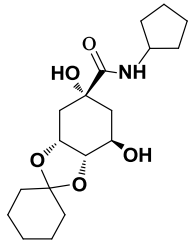
3l


7.26
6.92
6.89
4.83
4.55
4.54
4.53
4.51
4.50
4.16
4.13
4.11
4.09
3.81
3.80
3.80
3.79
3.78
3.48
2.42
2.41
2.37
2.36
2.23
2.23
2.21
2.21
2.18
2.16
2.05
2.04
2.01
2.00
1.99
1.96
1.95
1.93
1.91
1.72
1.69
1.66
1.64
1.63
1.62
HO
NH
O
OH
O
O
3m
11
10
9
8
7
6
5
4
3
2
1
ppm
1.00
0.83
1.02
2.03
0.98
0.91
1.08
1.10
4.24
17.15


176.000
109.397
77.586
77.162
76.738
75.789
72.820
71.746
66.118
51.033
37.142
36.947
34.412
33.624
33.106
33.053
25.116
24.068
23.812
23.535
HO
O
NH
OH
O
O
3m
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm


105.3
31.20
%TRANSMITTANCE
3743.15
3401.82
2937.06
2865.70
2372.01
2318.02
1644.98
1529.27
1450.21
1365.35
1274.72
1232.29
1160.94
1108.87
1078.01
1039.44
933.378
844.668
572.755
497.544
HO
NH
O
OH
O
O
3m
4000
3000
2000
1000
400.0
Wavenumbers (cm-1)

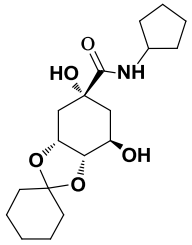

Composition View
Edit
Help
Spectrum #7 *217.93
2017
3.4
3.3
3.2
339.20438
339.19431
339.19
339.20
339.21
339.22
Max Error (mmu) 5
Min Error (mmu) 3
defect (ppm) 1000
STD Error (mmu) 20
Max U.S. 36.0
Min U.S. -0.1
Frq-CHK Off
STD-CHK Off
Element Max. Min. Isotope
C 18 0 1
H 29 4 1
O 5 0 1
N 2 0 1
Cl 1 0 1
Calculate
Filter
Cancel
Edit
Accept
Delete
Cancel
Format Composition Formula
Type in
Print
Elements : C 18/0, H 29/4, O 5/0, N 2/0, Cl 1/0
Mass Tolerance : 1000ppm, 3mmu if m/z < 3, 5mmu if m/z > 5
Unsaturation : -0.1 - 36.0
No m/z Int% Err-mmu U.S. Composition
1 339.2044 3.5 -0.2 5.0 C18H29O5N


O
HO
NH
O
OH
O

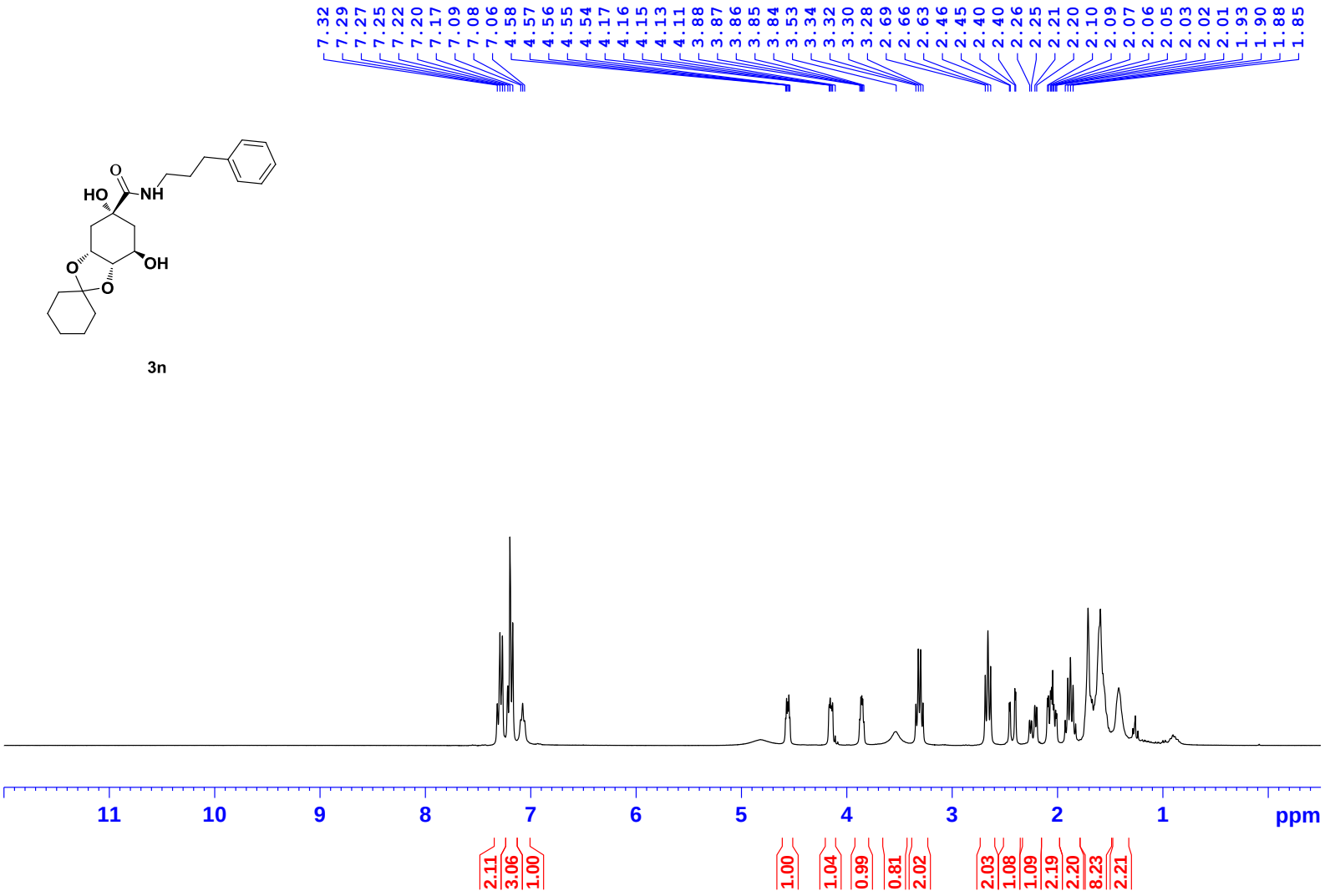
3m

[Elemental Composition]
Data : 201105150 Date : 31-May-2011 17:29
Sample: leeut D-13
Note : -
Inlet : Direct Ion Mode : EI+
RT : 0.77 min Scan#: 29
Elements : C 18/0, H 29/4, O 5/0, N 2/0, Cl 1/0
Mass Tolerance : 1000ppm, 3mmu if m/z < 3, 5mmu if m/z > 5
Unsaturation (U.S.) : -0.1 - 36.0

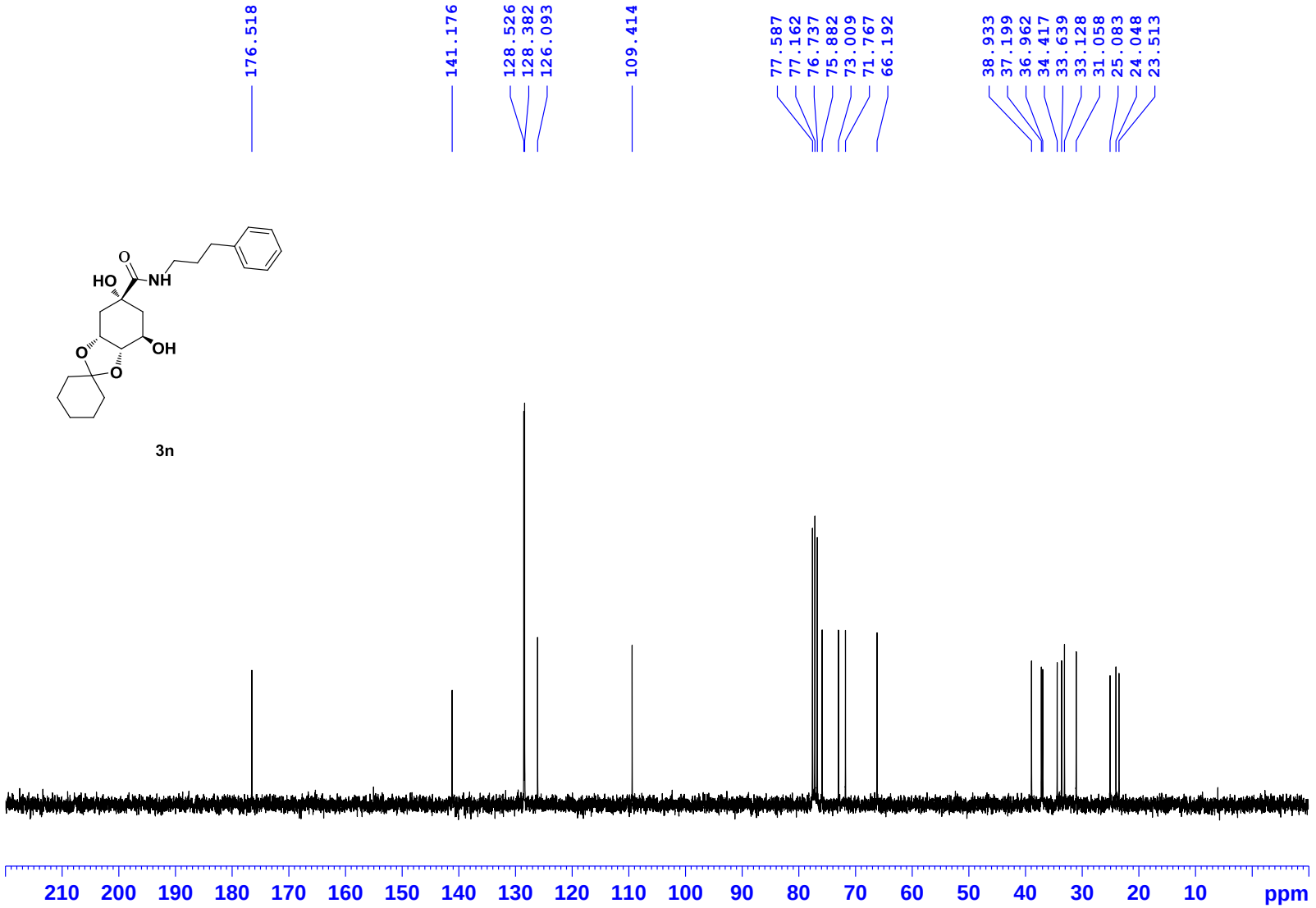
Observed m/z	Int%	Err[ppm / mmu]	U.S.	Composition
339.2044	3.5	-0.6 / -0.2	5.0	C 18 H 29 O 5 N

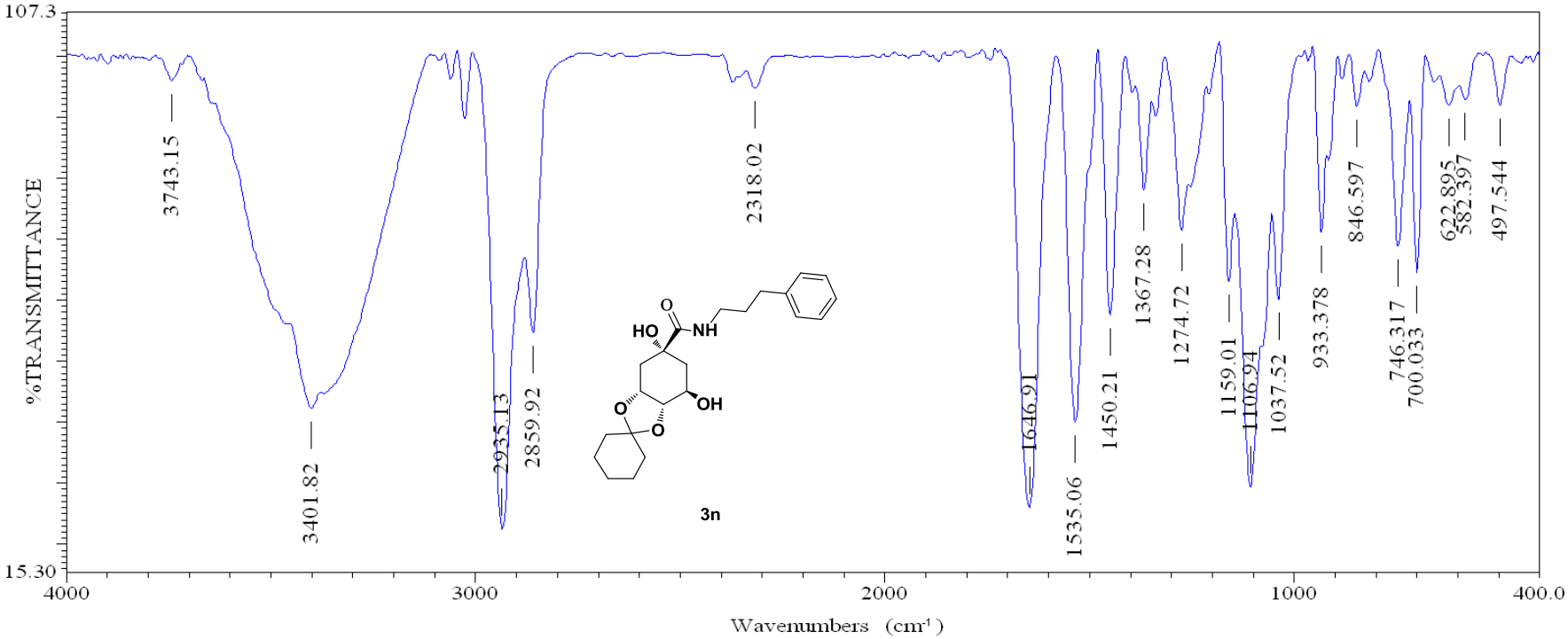


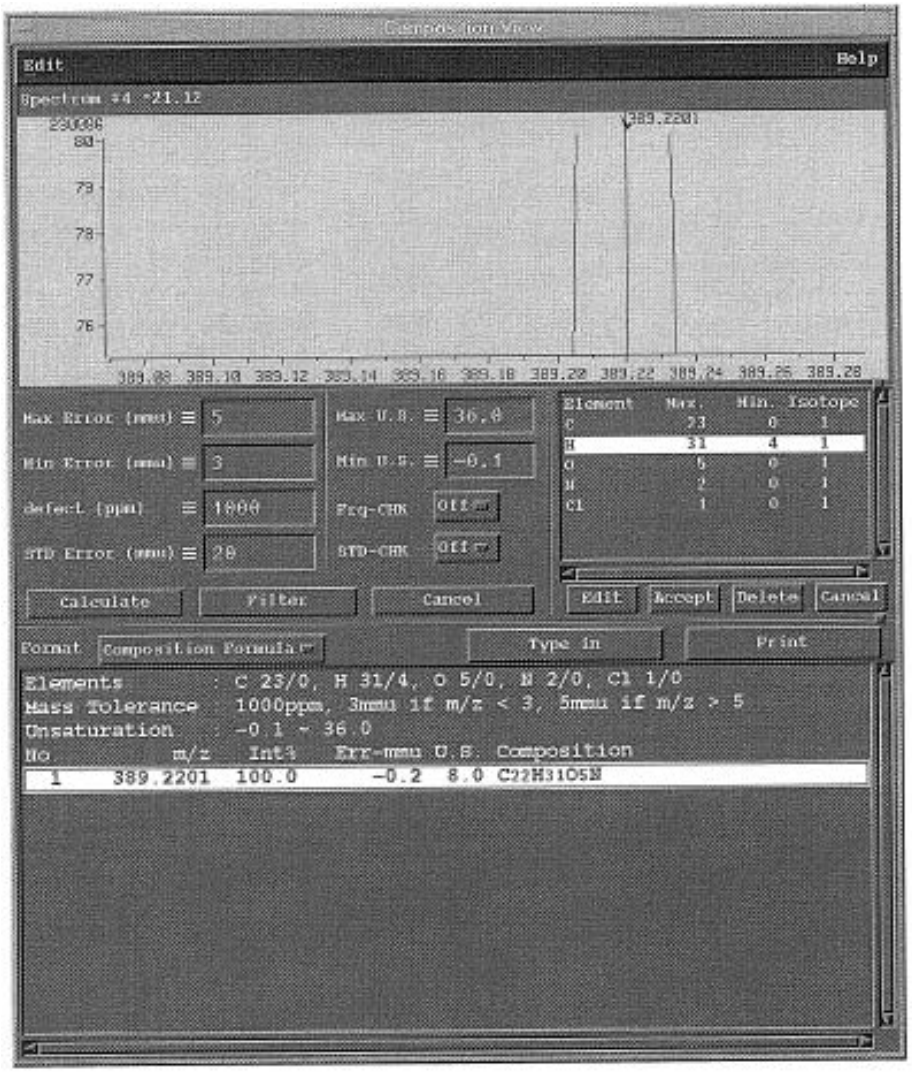
3m

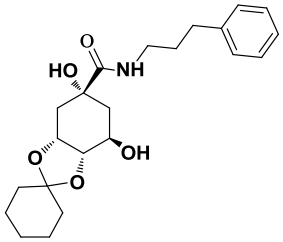

7.32
7.29
7.27
7.25
7.22
7.20
7.17
7.09
7.08
7.06
4.58
4.57
4.56
4.55
4.54
4.17
4.16
4.15
4.13
4.11
3.88
3.87
3.86
3.85
3.84
3.53
3.34
3.32
3.30
3.28
2.69
2.66
2.63
2.46
2.45
2.40
2.40
2.26
2.25
2.21
2.20
2.10
2.09
2.07
2.06
2.05
2.03
2.02
2.01
1.93
1.90
1.88
1.85
HO
O
NH
OH
O
O
11
10
9
8
7
6
5
4
3
2
1
ppm
2.11
3.06
1.00
1.00
1.04
0.99
0.81
2.02
2.03
1.08
1.09
2.19
2.20
8.23
2.21

3n


176.518
141.176
128.526
128.382
126.093
109.414
77.587
77.162
76.737
75.882
73.009
71.767
66.192
38.933
37.199
36.962
34.417
33.639
33.128
31.058
25.083
24.048
23.513
HO
O
NH
OH
O
O
3n
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm


107.3
15.30
%TRANSMITTANCE
4000
3000
2000
1000
400.0
Wavenumbers (cm-1)
3743.15
3401.82
2935.13
2859.92
2318.02
1646.91
1535.06
1450.21
1367.28
1274.72
1159.01
1106.94
1037.52
933.378
846.597
746.317
700.033
622.895
582.397
497.544
HO
NH
O
OH
O
O
3n

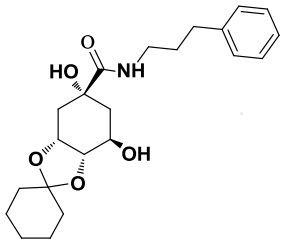

Composition View
Edit
Help
Spectrum #4 -21.12
389.2201
389.08 389.10 389.12 389.14 389.16 389.18 389.20 389.22 389.24 389.26 389.28
Max Error (mmu) 5
Min Error (mmu) 3
Defect (ppm) 1000
STD Error (mmu) 20
Max U.S. 36.0
Min U.S. -0.1
Freq-CHK Off
STD-CHK Off
Element Max. Min. Isotope
C 23 0 1
H 31 4 1
O 5 0 1
N 2 0 1
Cl 1 0 1
Calculate
Filter
Cancel
Edit
Accept
Delete
Cancel
Format Composition Formula
Type in
Print
Elements : C 23/0, H 31/4, O 5/0, N 2/0, Cl 1/0
Mass Tolerance : 1000ppm, 3mmu if m/z < 3, 5mmu if m/z > 5
Unsaturation : -0.1 ~ 36.0
No m/z Int% Err-mmu U.S. Composition
1 389.2201 100.0 -0.2 8.0 C22H31O5N


O
HO
NH
O
O
OH

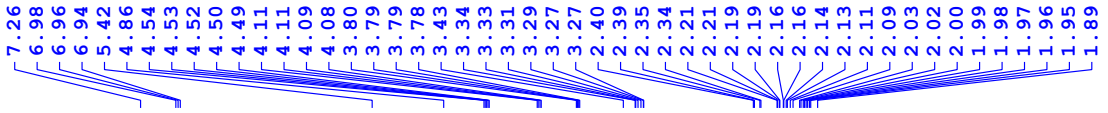
3n

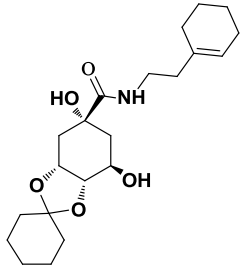
[Elemental Composition]
Data : 201105151 Date : 31-May-2011 17:38
Sample: leeut D-14
Note : -
Inlet : Direct Ion Mode : EI+
RT : 0.05 min Scan#: 3
Elements : C 23/0, H 31/4, O 5/0, N 2/0, Cl 1/0
Mass Tolerance : 1000ppm, 3mmu if m/z < 3, 5mmu if m/z > 5
Unsaturation (U.S.) : -0.1 - 36.0

Observed m/z	Int%	Err[ppm / mmu]	U.S.	Composition
389.2201	100.0	-0.4 / -0.2	8.0	C 22 H 31 O 5 N

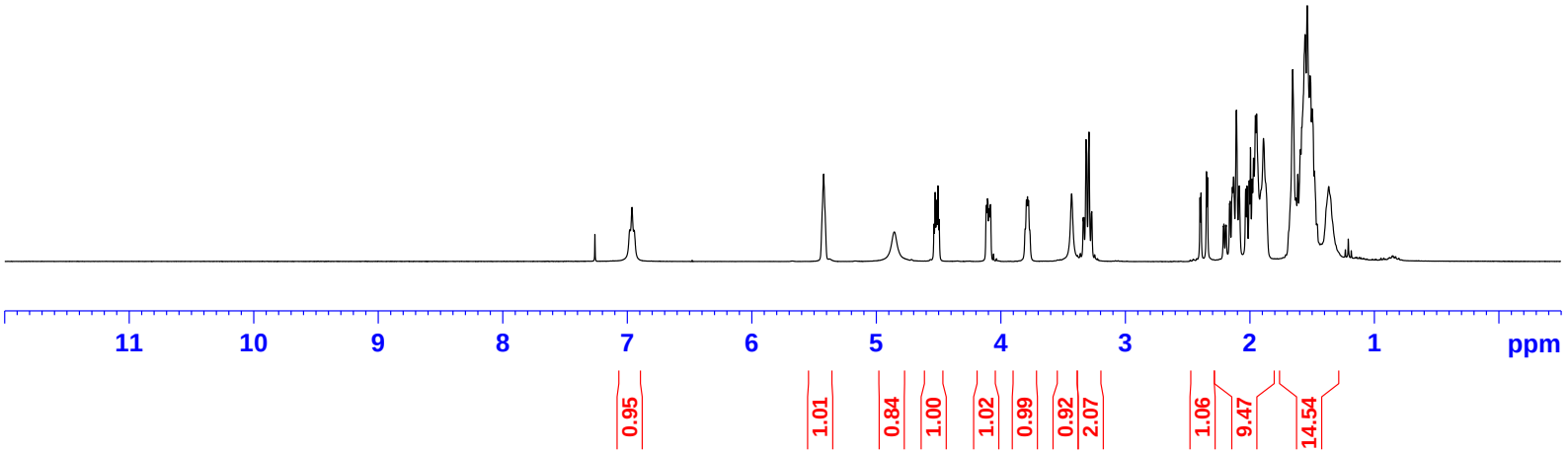


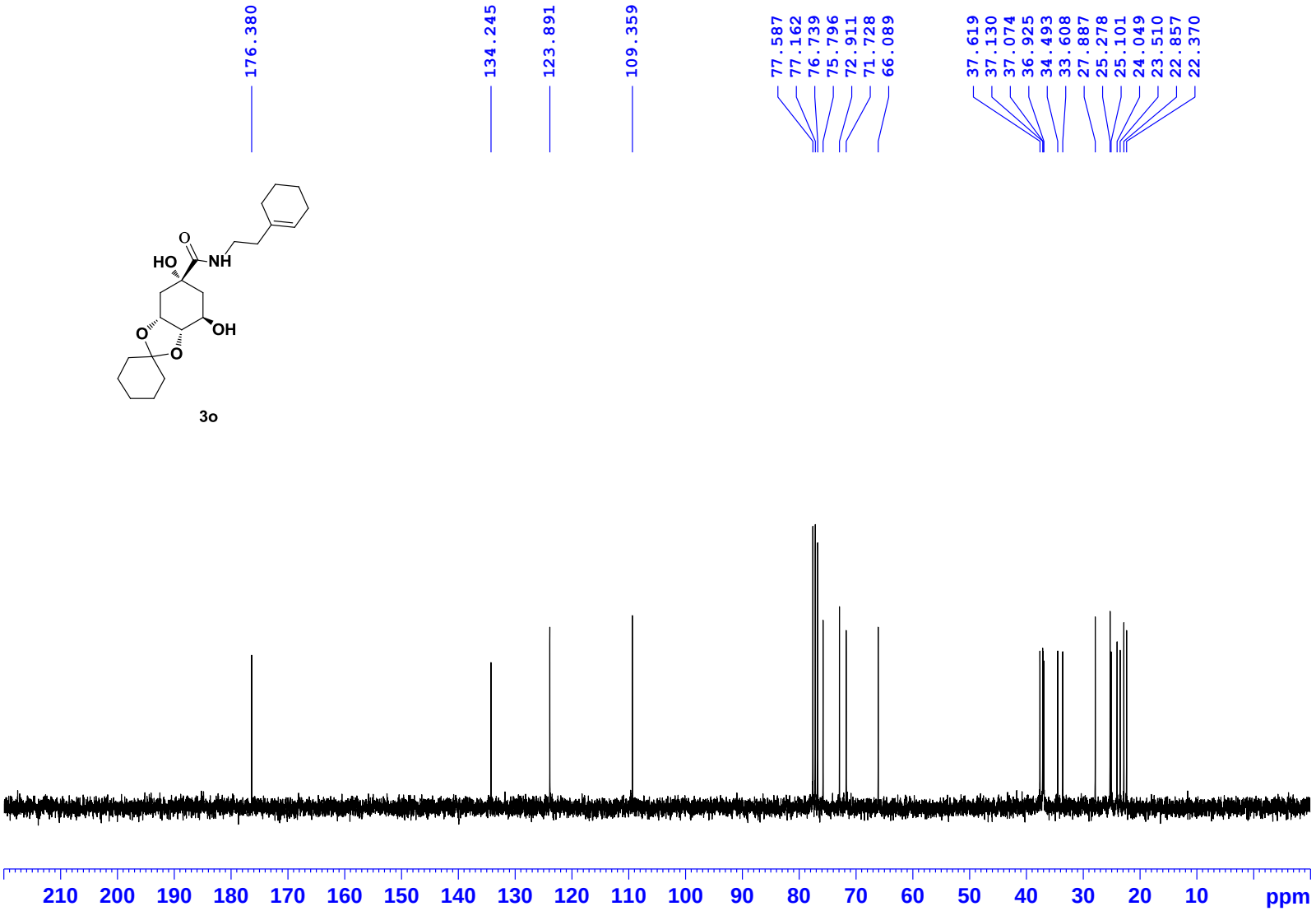
3n

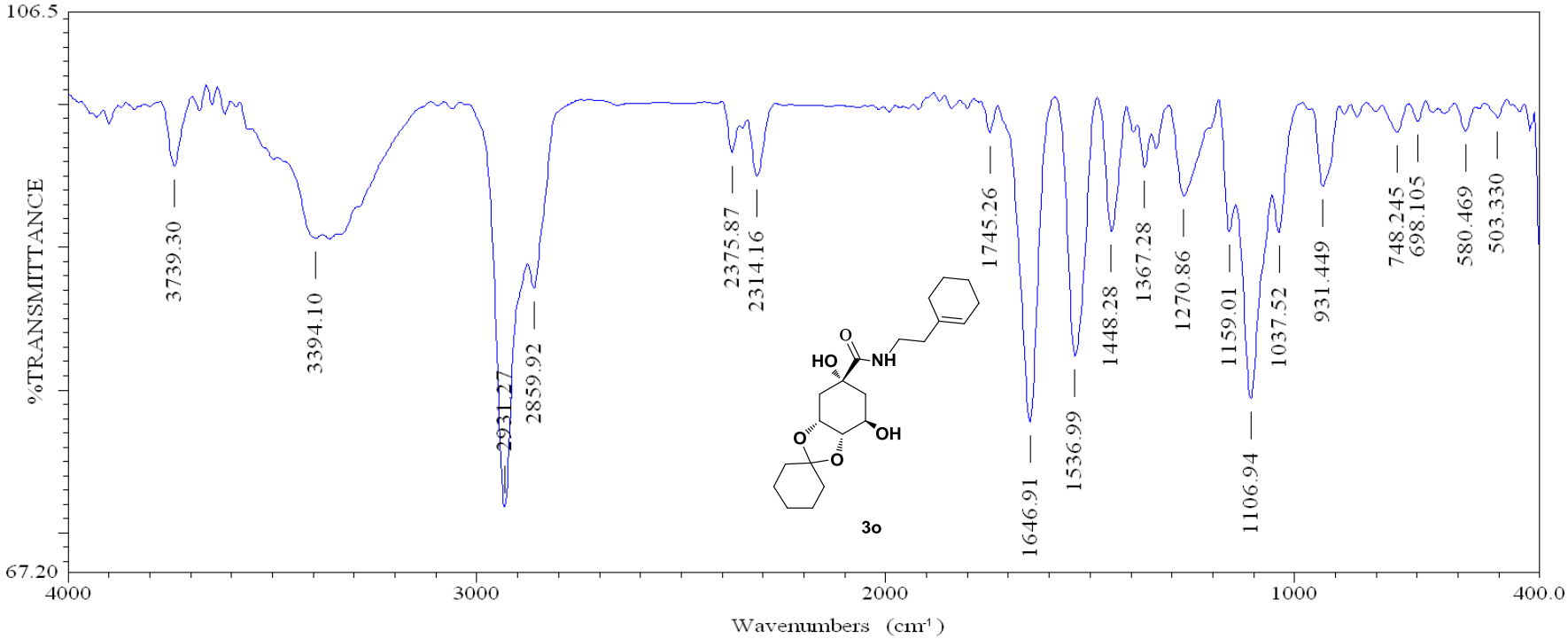

7.26
6.98
6.96
6.94
5.42
4.86
4.54
4.53
4.52
4.50
4.49
4.11
4.11
4.09
4.08
3.80
3.79
3.79
3.78
3.43
3.34
3.33
3.31
3.29
3.27
3.27
2.40
2.39
2.35
2.34
2.21
2.21
2.19
2.19
2.16
2.16
2.14
2.13
2.11
2.09
2.03
2.02
2.00
1.99
1.98
1.97
1.96
1.95
1.89

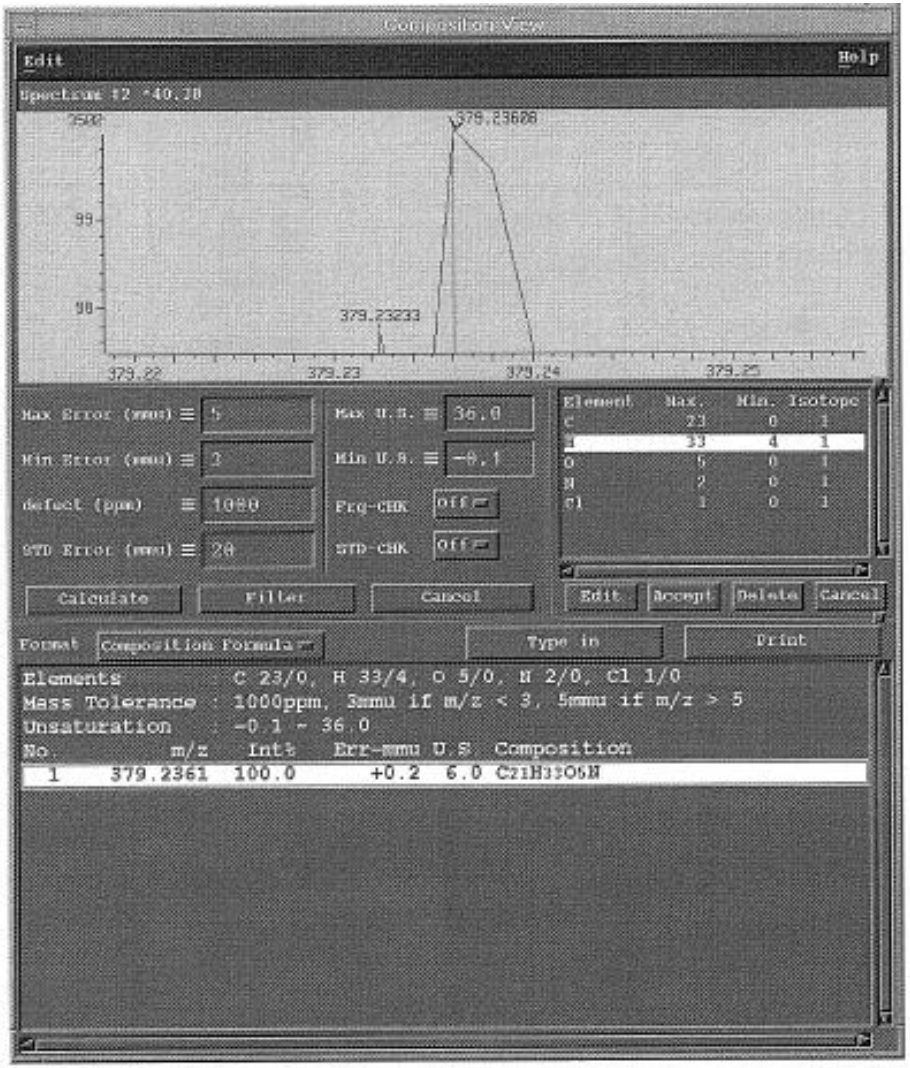

O
HO
NH
O
OH
O

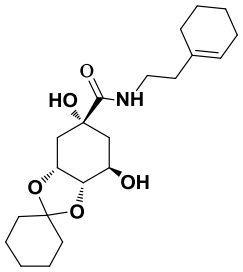
3o


11
10
9
8
7
6
5
4
3
2
1
ppm
0.95
1.01
0.84
1.00
1.02
0.99
0.92
2.07
1.06
9.47
14.54


176.380
134.245
123.891
109.359
77.587
77.162
76.739
75.796
72.911
71.728
66.089
37.619
37.130
37.074
36.925
34.493
33.608
27.887
25.278
25.101
24.049
23.510
22.857
22.370
HO
NH
O
OH
O
O
3o
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm


106.5
67.20
%TRANSMITTANCE
3739.30
3394.10
2931.27
2859.92
2375.87
2314.16
1745.26
1646.91
1536.99
1448.28
1367.28
1270.86
1159.01
1106.94
1037.52
931.449
748.245
698.105
580.469
503.330
HO
NH
O
OH
O
O
3o
4000
3000
2000
1000
400.0
Wavenumbers (cm-1)


Composition View
Edit
Help
Spectrum #2 *40.20
2500
99
98
379.23608
379.23233
379.22
379.23
379.24
379.25
Max Error (mmu) ≡ 5
Min Error (mmu) ≡ 3
defect (ppm) ≡ 1000
STD Error (mmu) ≡ 20
Max U.S. ≡ 36.0
Min U.S. ≡ -0.1
Frq-CHK Off
STD-CHK Off
Element Max. Min. Isotope
C 23 0 1
H 33 4 1
O 5 0 1
N 2 0 1
Cl 1 0 1
Calculate
Filter
Cancel
Edit
Accept
Delete
Cancel
Format Composition Formula
Type in
Print
Elements : C 23/0, H 33/4, O 5/0, N 2/0, Cl 1/0
Mass Tolerance : 1000ppm, 3mmu if m/z < 3, 5mmu if m/z > 5
Unsaturation : -0.1 ~ 36.0
No. m/z Int% Err-mmu U.S Composition
1 379.2361 100.0 +0.2 6.0 C21H33O6N


HO
O
NH
OH
O
O

3o

[Elemental Composition]

Data : 201105152 Date : 01-Jun-2011 10:39

Sample: leeut D-15

Note : -

Inlet : Direct Ion Mode : EI+

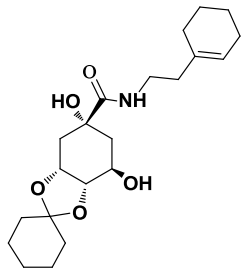
RT : 1.59 min Scan#: 70

Elements : C 23/0, H 33/4, O 5/0, N 2/0, Cl 1/0

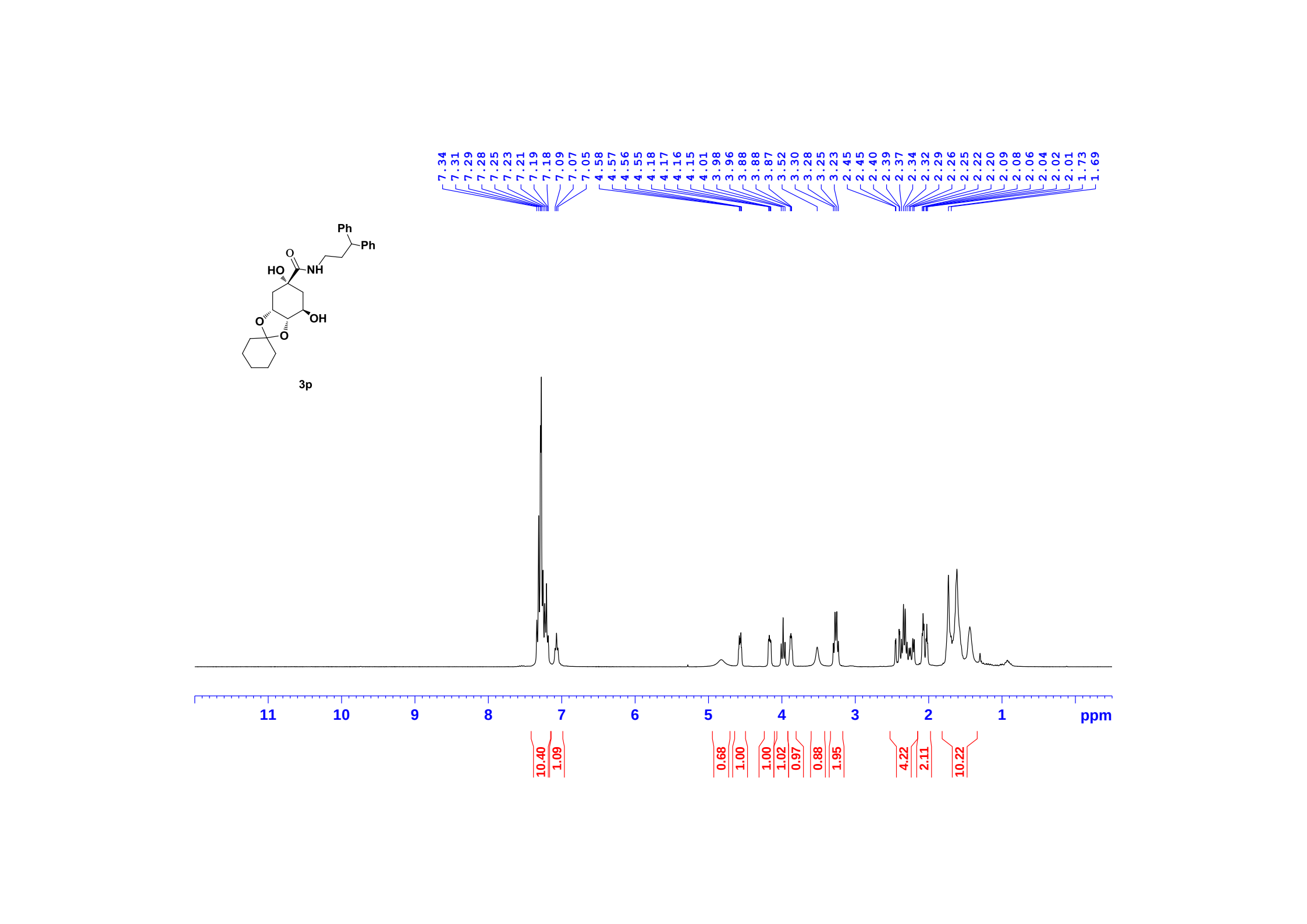
Mass Tolerance : 1000ppm, 3mmu if m/z < 3, 5mmu if m/z > 5

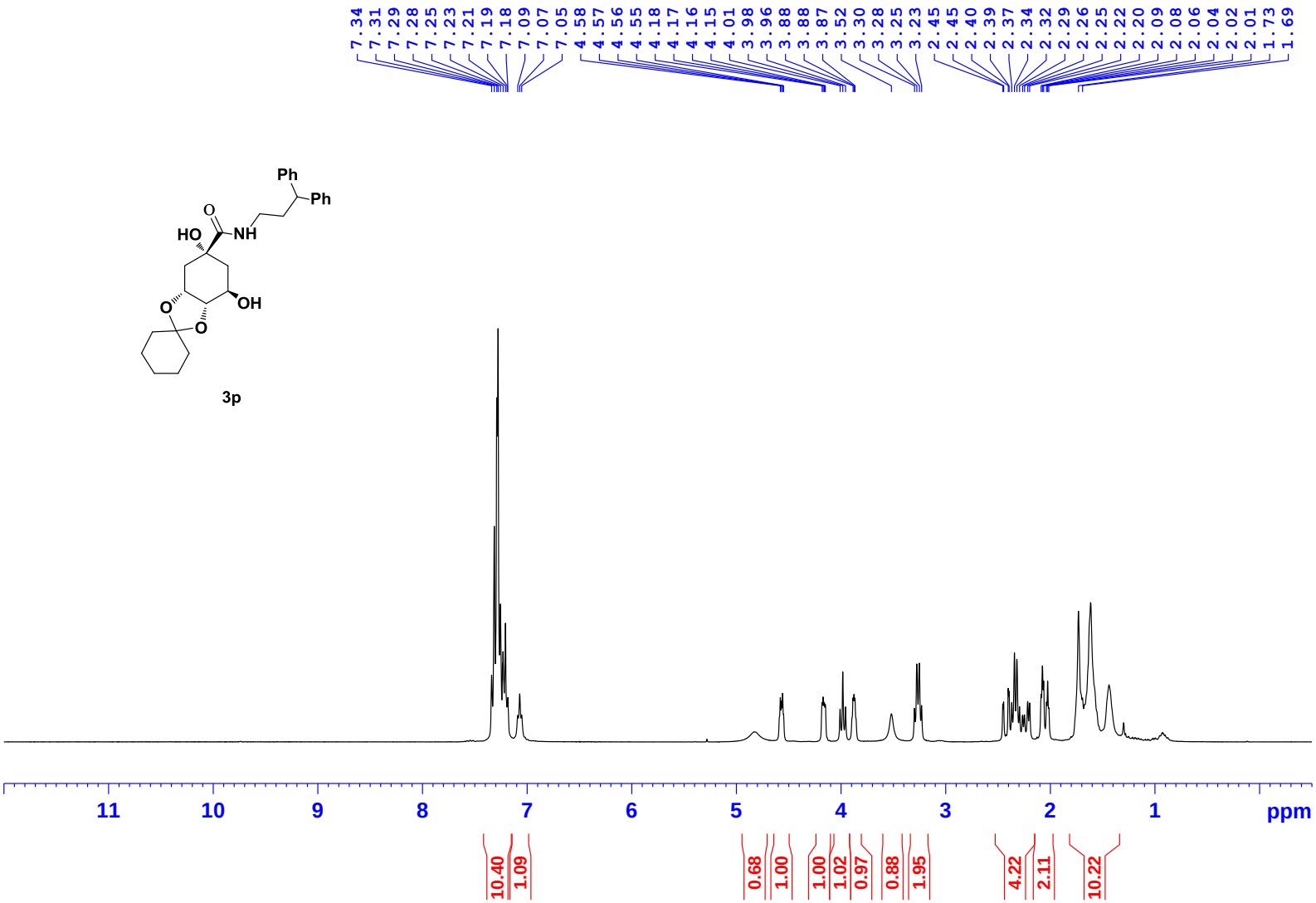
Unsaturation (U.S.) : -0.1 - 36.0

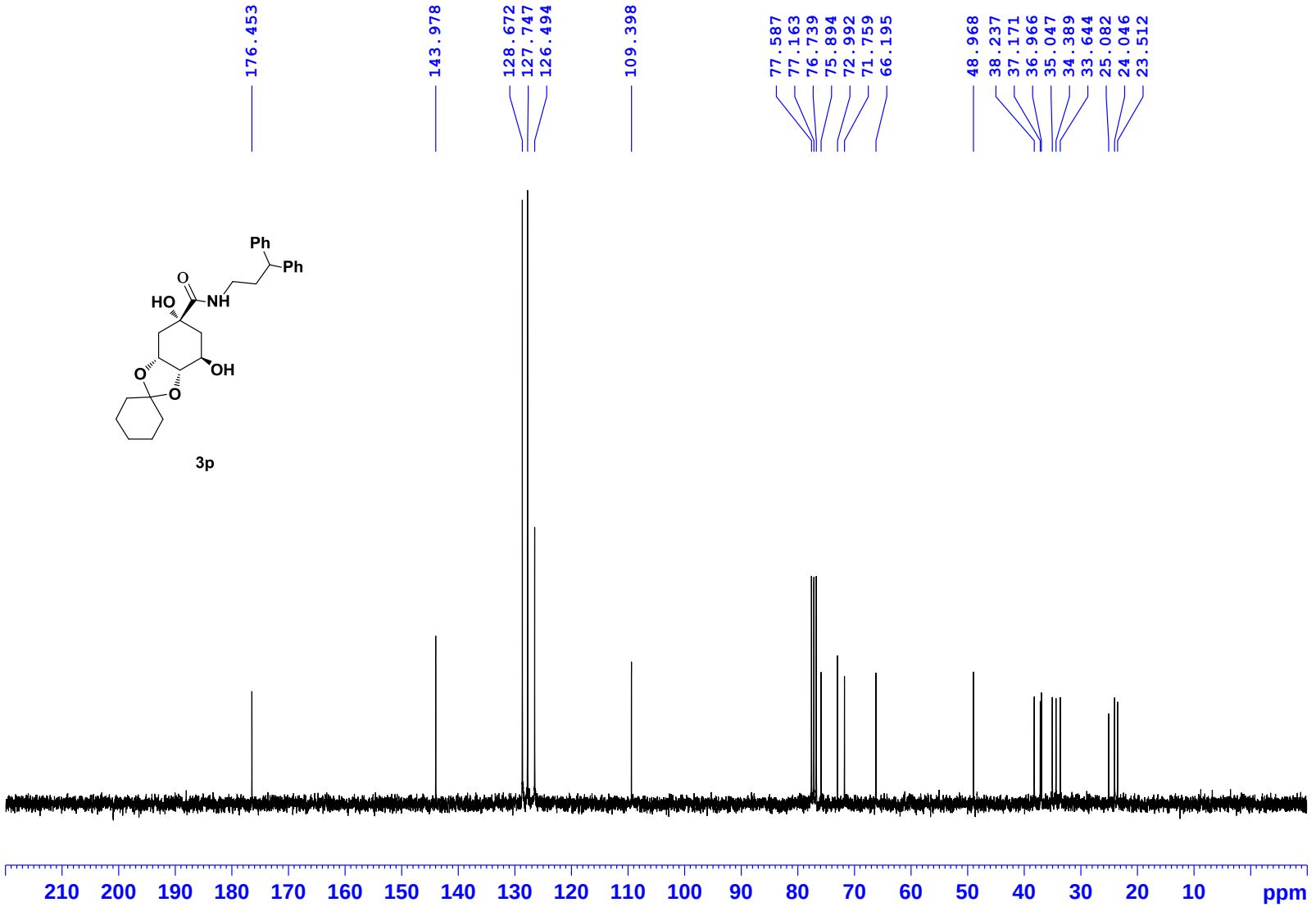
Observed m/z	Int%	Err[ppm / mmu]	U.S.	Composition
379.2361	100.0	+0.6 / +0.2	6.0	C 21 H 33 O 5 N

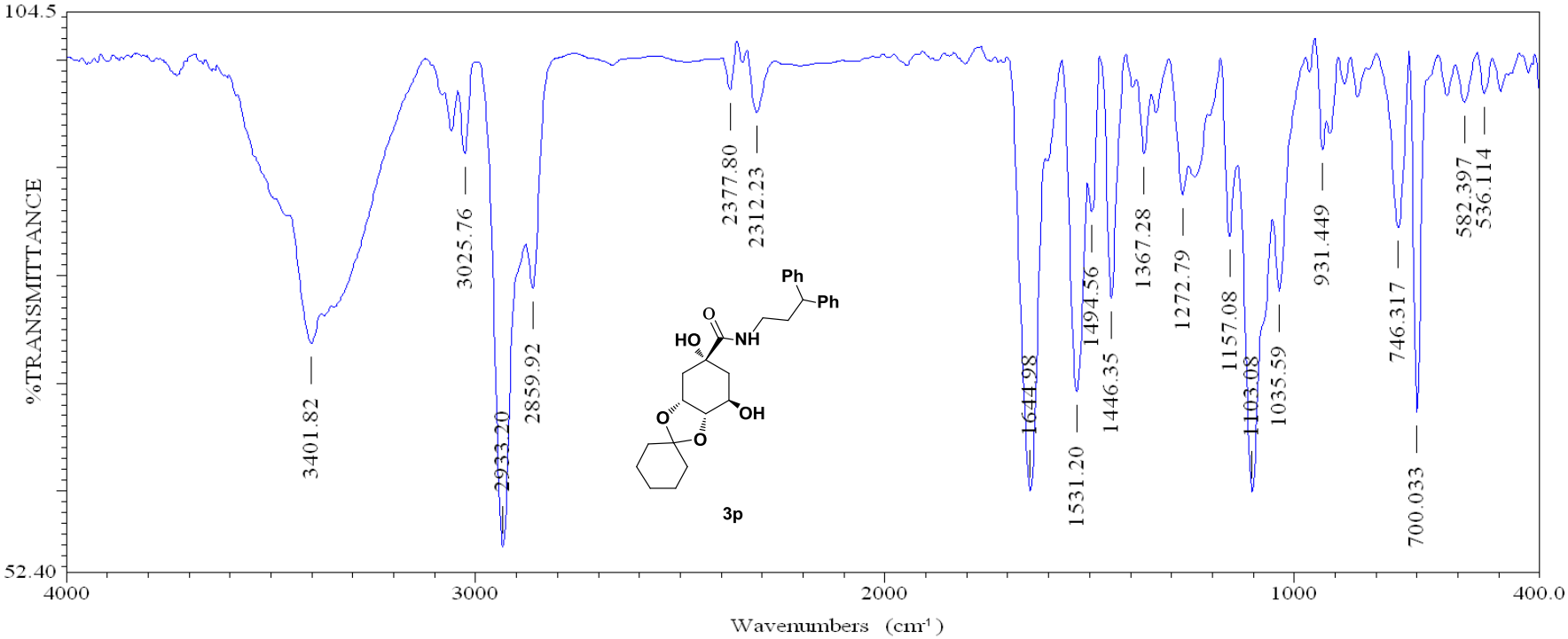


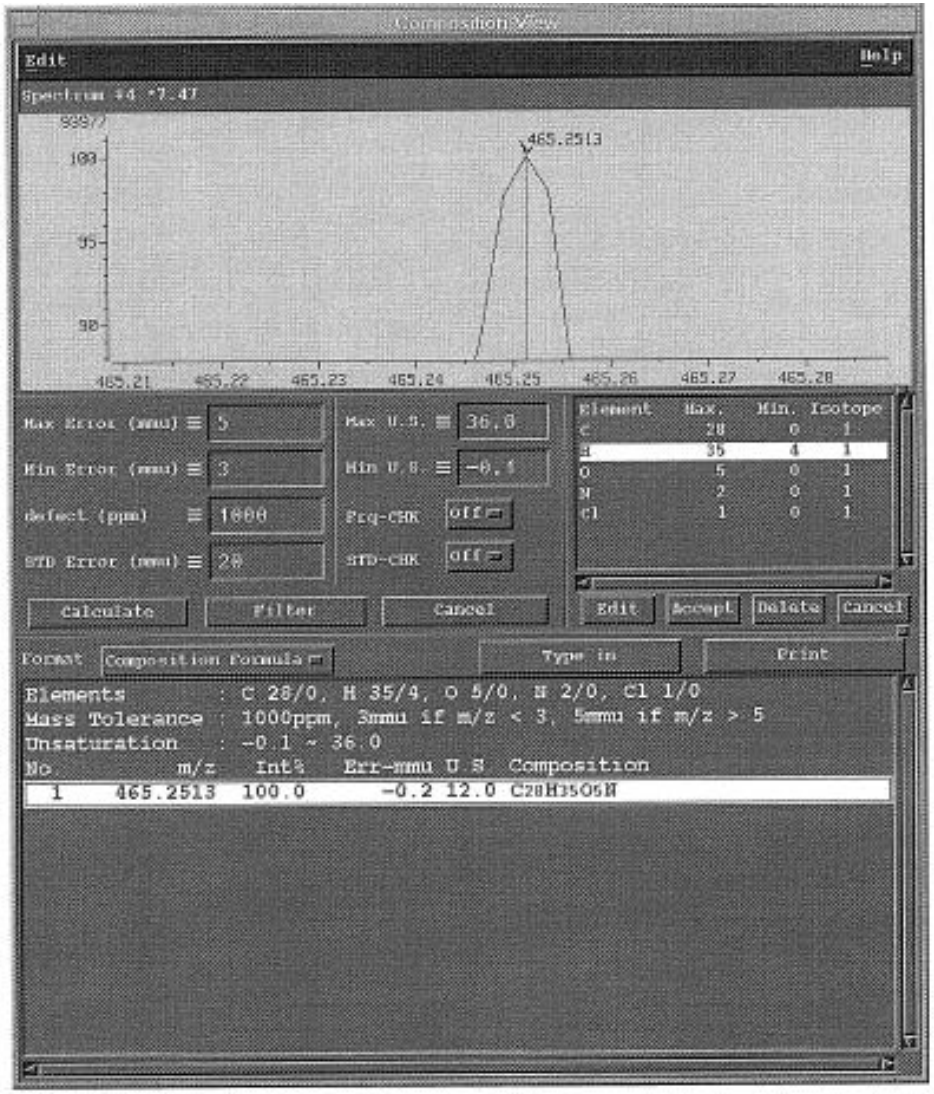
3o

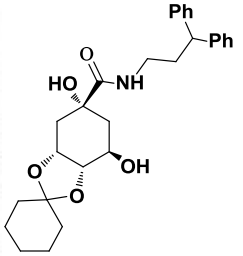

7.34
7.31
7.29
7.28
7.25
7.23
7.21
7.19
7.18
7.09
7.07
7.05
4.58
4.57
4.56
4.55
4.18
4.17
4.16
4.15
4.01
3.98
3.96
3.88
3.88
3.87
3.52
3.30
3.28
3.25
3.23
2.45
2.45
2.40
2.39
2.37
2.34
2.32
2.29
2.26
2.25
2.22
2.20
2.09
2.08
2.06
2.04
2.02
2.01
1.73
1.69
Ph
Ph
O
HO
NH
OH
O
O
3p
11
10
9
8
7
6
5
4
3
2
1
ppm
10.40
1.09
0.68
1.00
1.00
1.02
0.97
0.88
1.95
4.22
2.11
10.22


7.34
7.31
7.29
7.28
7.25
7.23
7.21
7.19
7.18
7.09
7.07
7.05
4.58
4.57
4.56
4.55
4.18
4.17
4.16
4.15
4.01
3.98
3.96
3.88
3.88
3.87
3.52
3.30
3.28
3.25
3.23
2.45
2.45
2.40
2.39
2.37
2.34
2.32
2.29
2.26
2.25
2.22
2.20
2.09
2.08
2.06
2.04
2.02
2.01
1.73
1.69
Ph
Ph
O
HO
NH
O
OH
O
3p
11
10
9
8
7
6
5
4
3
2
1
ppm
10.40
1.09
0.68
1.00
1.00
1.02
0.97
0.88
1.95
4.22
2.11
10.22


176.453
143.978
128.672
127.747
126.494
109.398
77.587
77.163
76.739
75.894
72.992
71.759
66.195
48.968
38.237
37.171
36.966
35.047
34.389
33.644
25.082
24.046
23.512
Ph
Ph
O
HO
NH
OH
O
O
3p
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm


104.5
52.40
%TRANSMITTANCE
4000
3000
2000
1000
400.0
Wavenumbers (cm-1)
3401.82
3025.76
2933.20
2859.92
2377.80
2312.23
1644.98
1531.20
1494.56
1446.35
1367.28
1272.79
1157.08
1103.08
1035.59
931.449
746.317
700.033
582.397
536.114
HO
O
NH
Ph
Ph
OH
O
O
3p


Composition View
Edit
Help
Spectrum #4 *7.47
465.2513
100
95
90
465.21
465.22
465.23
465.24
465.25
465.26
465.27
465.28
Max Error (mmu) 5
Min Error (mmu) 3
defect (ppm) 1000
STD Error (mmu) 20
Max U.S. 36.0
Min U.S. -0.1
Frq-CHK off
STD-CHK off
Element Max. Min. Isotope
C 28 0 1
H 35 4 1
O 5 0 1
N 2 0 1
Cl 1 0 1
Calculate
Filter
Cancel
Edit
Accept
Delete
Cancel
Format Composition Formula
Type in
Print
Elements : C 28/0, H 35/4, O 5/0, N 2/0, Cl 1/0
Mass Tolerance : 1000ppm, 3mmu if m/z < 3, 5mmu if m/z > 5
Unsaturation : -0.1 ~ 36.0
No. m/z Int% Err-mmu U.S Composition
1 465.2513 100.0 -0.2 12.0 C28H35O5N

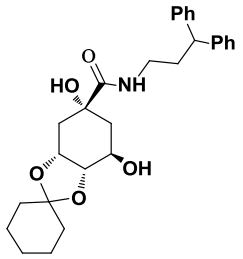

Ph
Ph
O
HO
NH
OH
O
O

3p

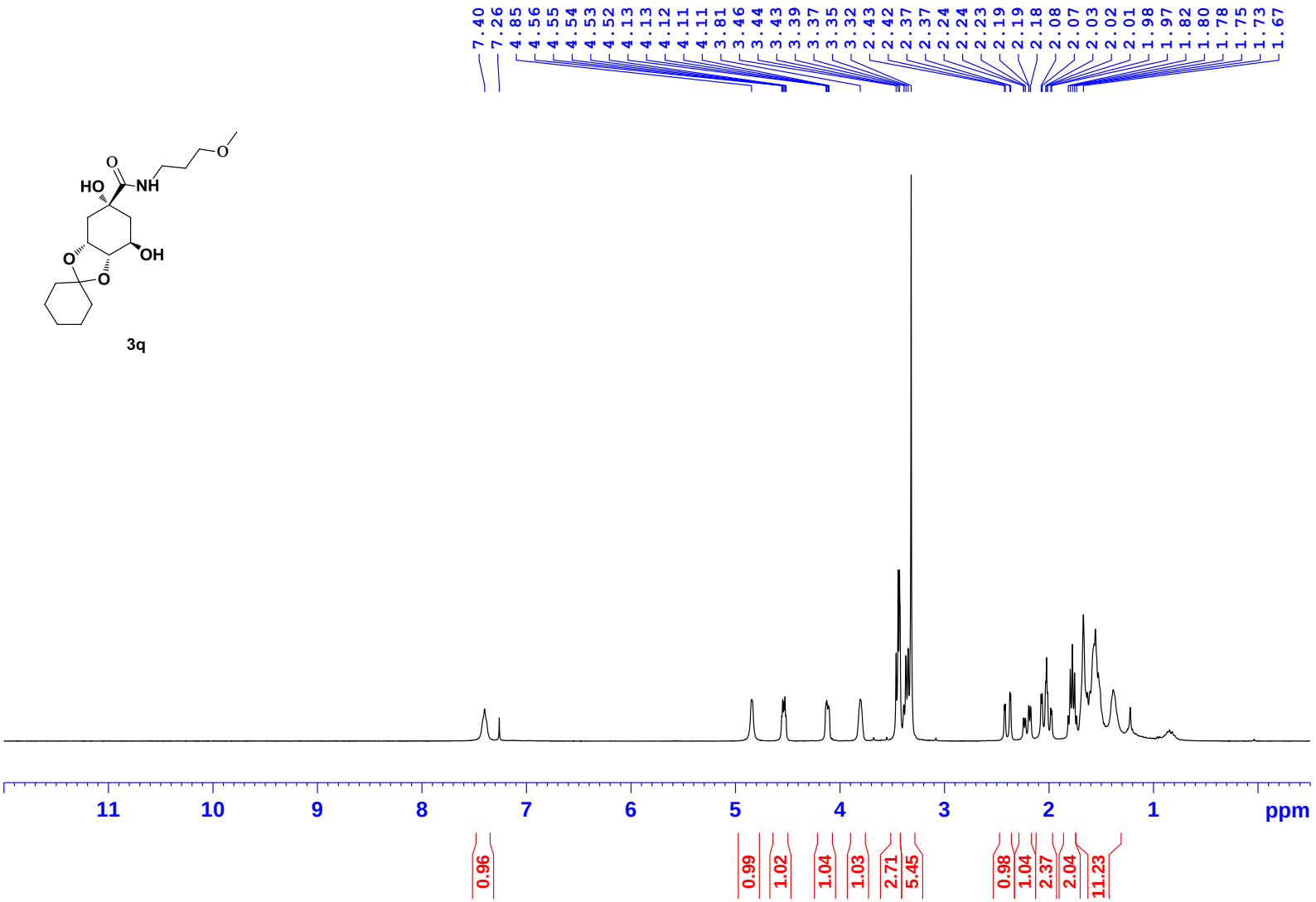
[Elemental Composition]

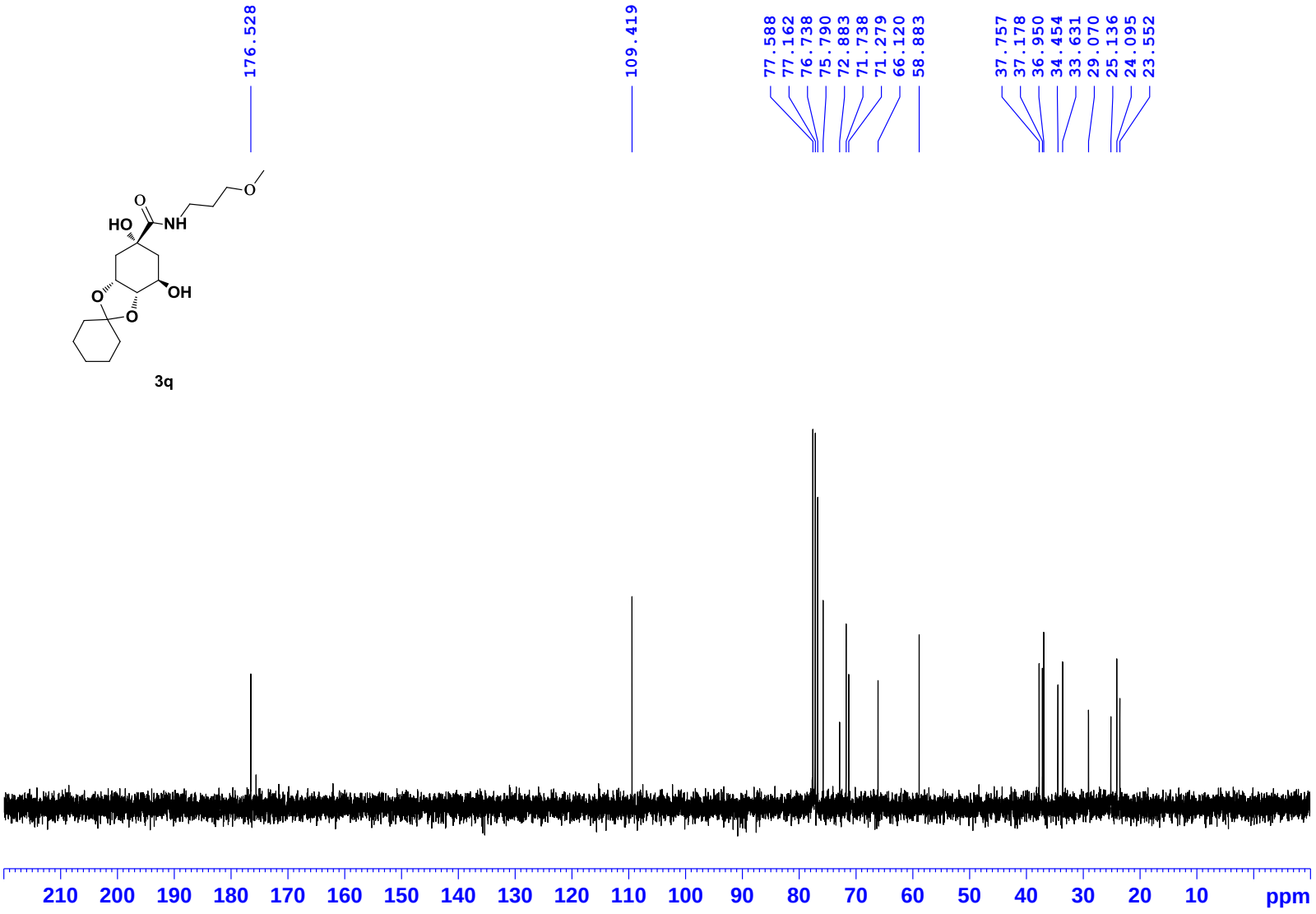
Data : 201105153 Date : 01-Jun-2011 10:46
Sample: leeut D-16
Note : -
Inlet : Direct Ion Mode : EI+
RT : 0.99 min Scan#: 50
Elements : C 28/0, H 35/4, O 5/0, N 2/0, Cl 1/0
Mass Tolerance : 1000ppm, 3mmu if m/z < 3, 5mmu if m/z > 5
Unsaturation (U.S.) : -0.1 - 36.0

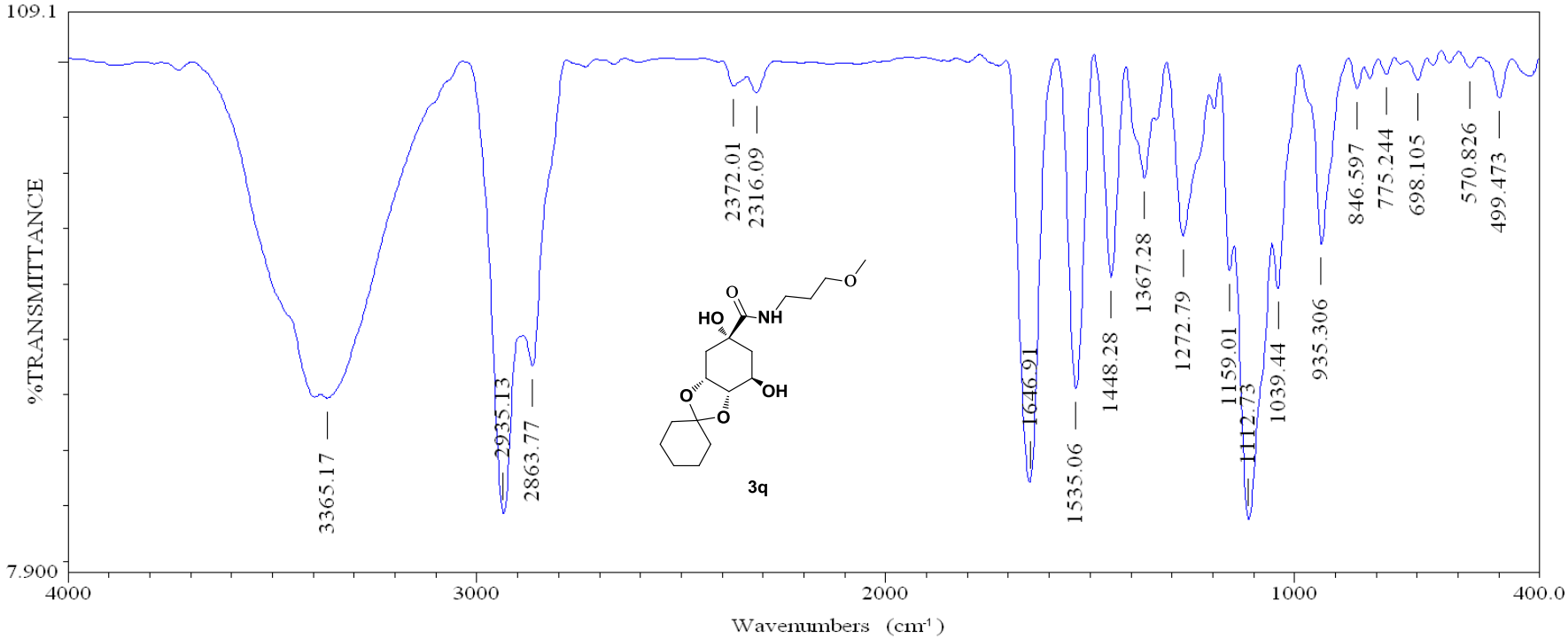
Observed m/z	Int%	Err[ppm / mmu]	U.S.	Composition
465.2513	100.0	-0.4 / -0.2	12.0	C 28 H 35 O 5 N



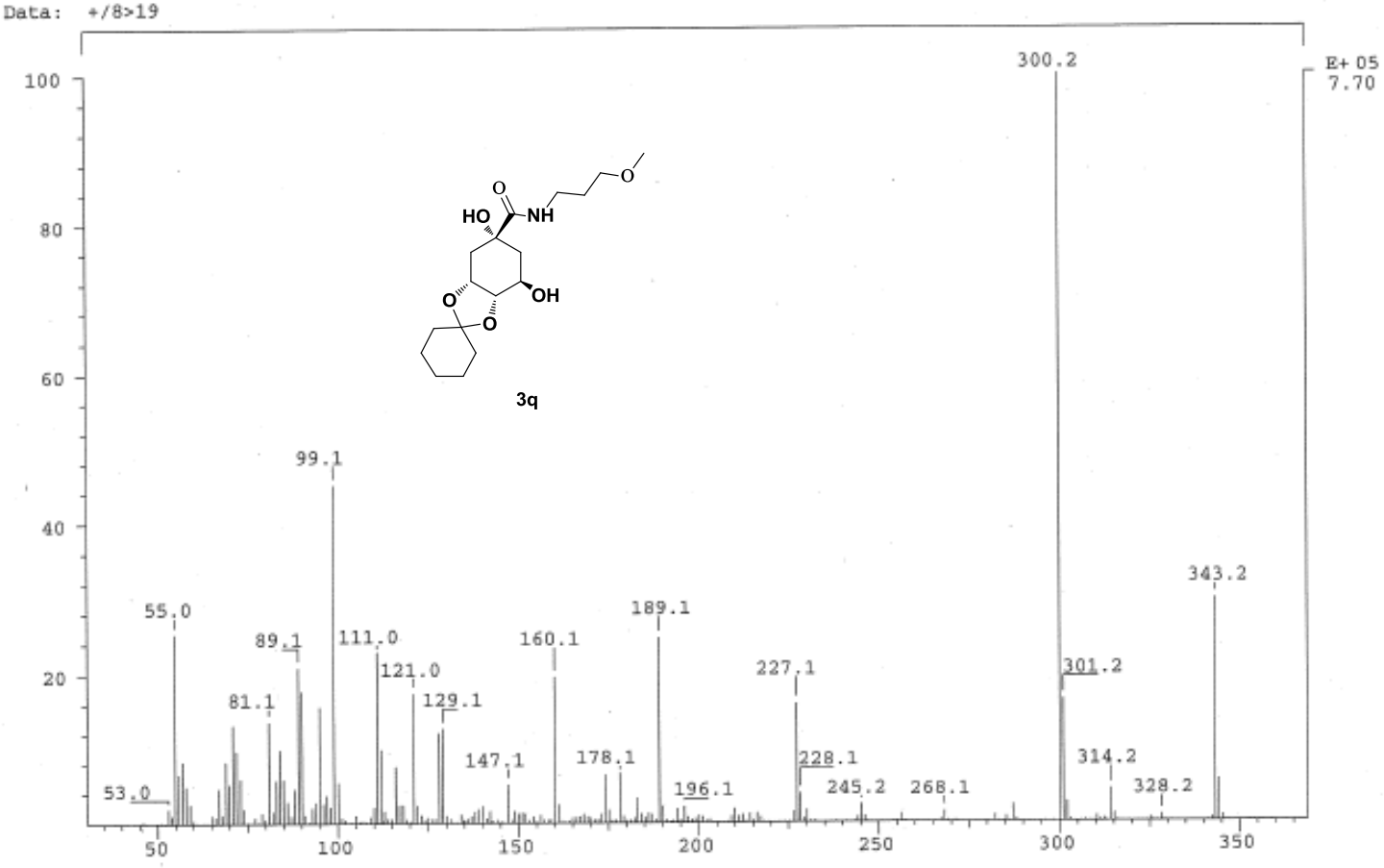
3p


7.40
7.26
4.85
4.56
4.55
4.54
4.53
4.52
4.13
4.13
4.12
4.11
4.11
3.81
3.46
3.44
3.43
3.39
3.37
3.35
3.32
2.43
2.42
2.37
2.37
2.24
2.24
2.23
2.19
2.19
2.18
2.08
2.07
2.03
2.02
2.01
1.98
1.97
1.82
1.80
1.78
1.75
1.73
1.67
HO
O
NH
O
OH
O
O
3q
11
10
9
8
7
6
5
4
3
2
1
ppm
0.96
0.99
1.02
1.04
1.03
2.71
5.45
0.98
1.04
2.37
2.04
11.23


176.528
109.419
77.588
77.162
76.738
75.790
72.883
71.738
71.279
66.120
58.883
37.757
37.178
36.950
34.454
33.631
29.070
25.136
24.095
23.552
HO
NH
O
O
OH
O
O
3q
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm


109.1
7.900
%TRANSMITTANCE
4000
3000
2000
1000
400.0
Wavenumbers (cm-1)
3365.17
2935.13
2863.77
2372.01
2316.09
1646.91
1535.06
1448.28
1367.28
1272.79
1159.01
1112.73
1039.44
935.306
846.597
775.244
698.105
570.826
499.473
HO
NH
O
OH
3q

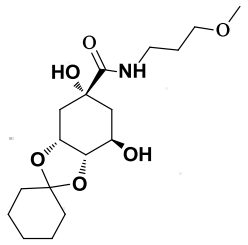
SPEC: lei5125(leeutd17) 15-Sep-11 REG : 00:49.5 #9
Samp: Start : 12:39:17 90
Mode: EI +VE +LMR BSCAN (EXP) UP LR NRM
Oper: Inlet :
Base: 300.2 Inten : 770126 Masses: 45 > 450
Norm: 300.2 RIC : 5523135 #peaks: 328
Peak: 1000.00 mmu
Data: +/8>19



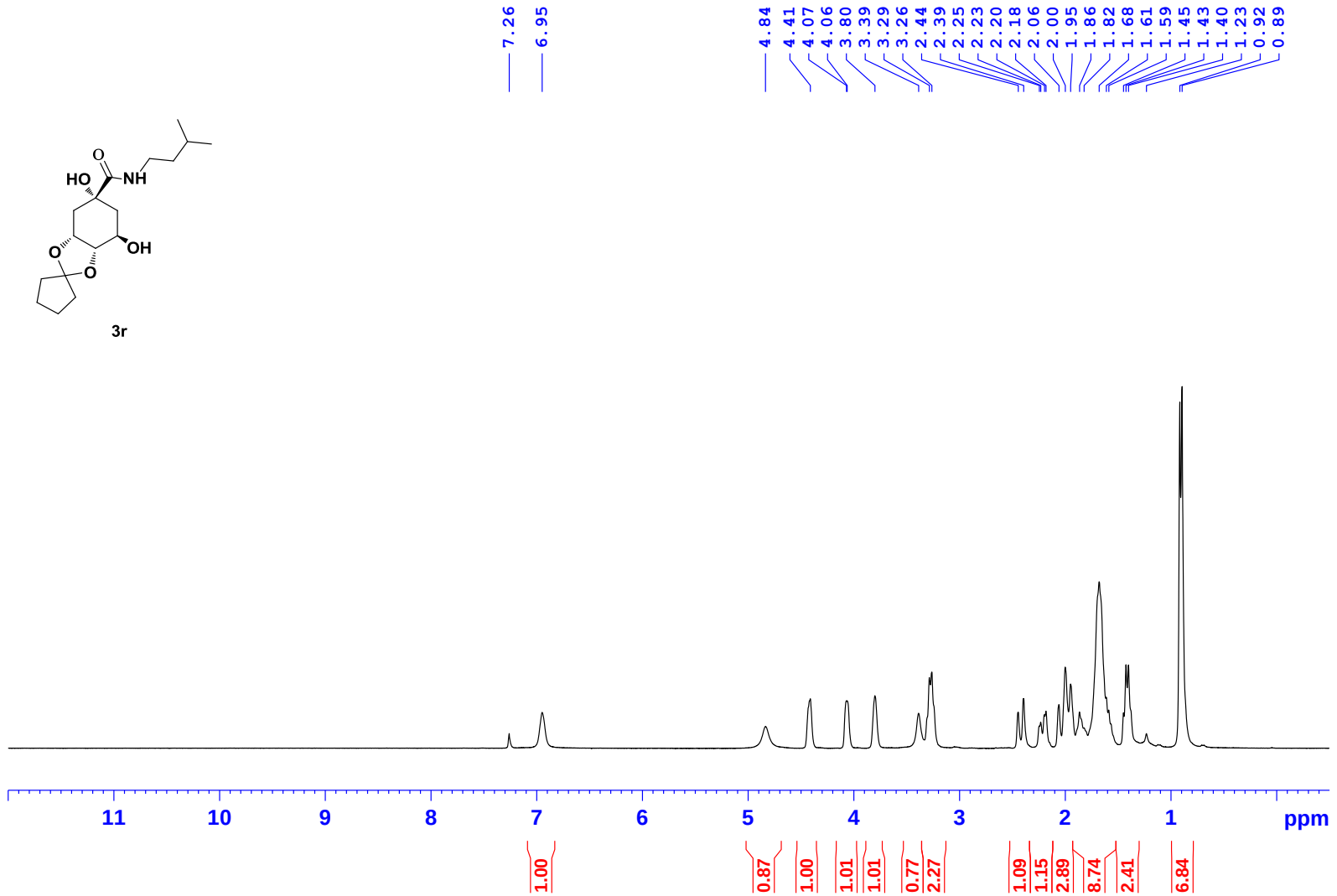
3q

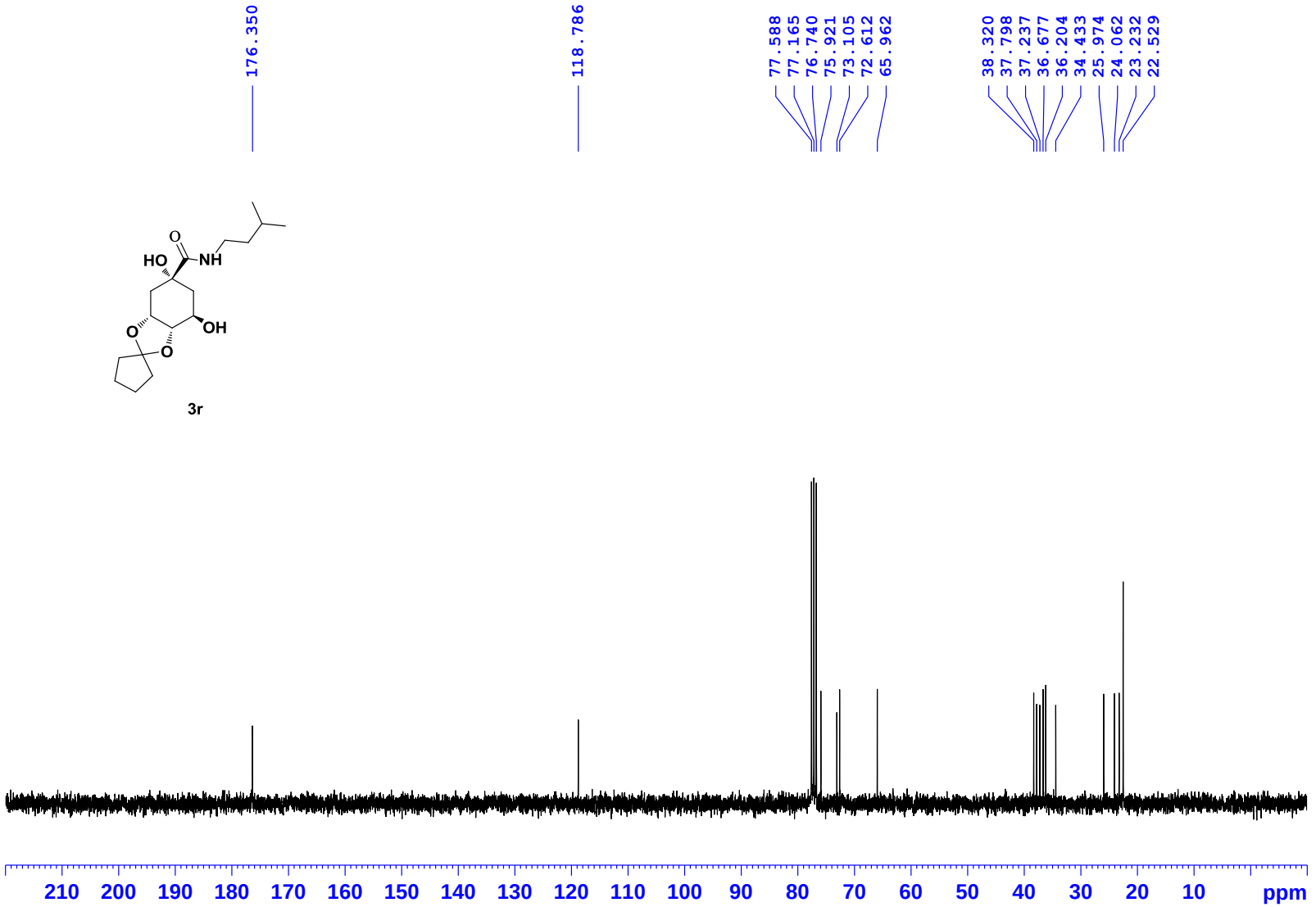
```
LIST: hei2529(leeutd17)-c1                    29-Sep-11 REG   : 01:36.0     #9
                                                        Start : 14:34:59    793
Samp:
Mode: EI +VE +LMR  ESCAN (EXP) UP HR NRM                Inlet :
Oper:
Limt: (  0)     .     .     .
    : (398) C19.H44.N.O7
Peak: 1000.00 mmu   R+D: -2.0 > 60.0
Data: +/295>407  (CMASS : converted |CMASS : converted |CMASS : conve
```

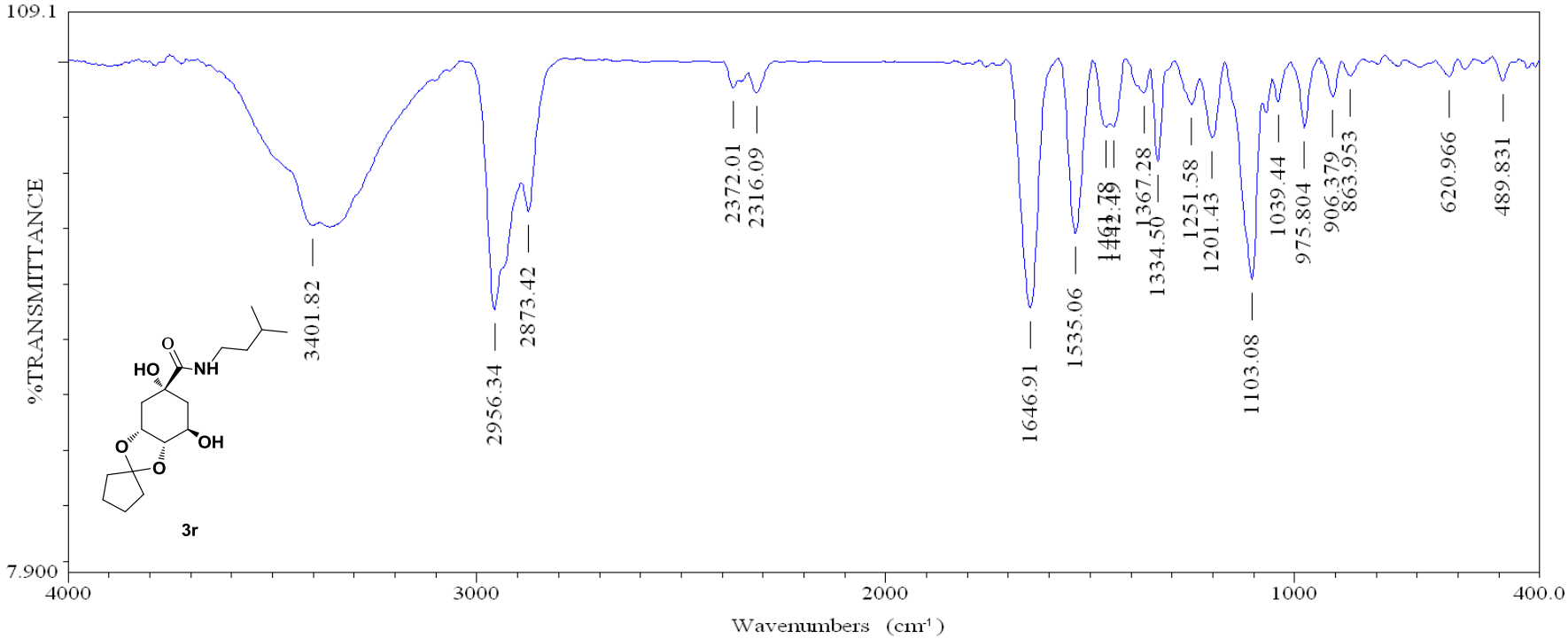
Mass	Intensity 2	%RA	Flags	(mmu) Delta	R+D	Composition
343.1990	11539	100.00	#	0.5	4.0	C17.H29.N.O6

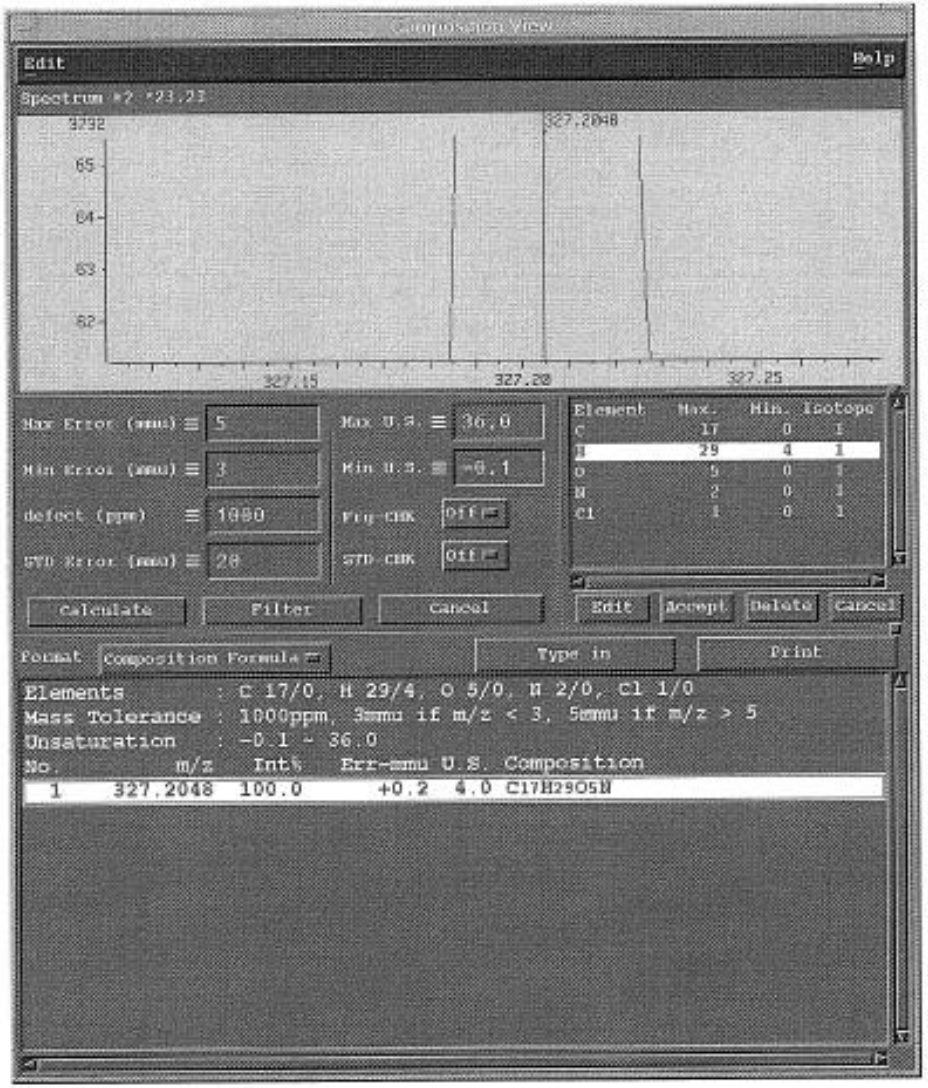


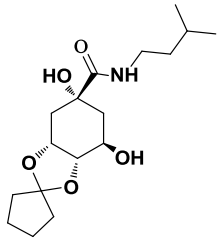
3q


7.26
6.95
4.84
4.41
4.07
4.06
3.80
3.39
3.29
3.26
2.44
2.39
2.25
2.23
2.20
2.18
2.06
2.00
1.95
1.86
1.82
1.68
1.61
1.59
1.45
1.43
1.40
1.23
0.92
0.89
HO
NH
O
OH
O
O
3r
11
10
9
8
7
6
5
4
3
2
1
ppm
1.00
0.87
1.00
1.01
1.01
0.77
2.27
1.09
1.15
2.89
8.74
2.41
6.84


176.350
118.786
77.588
77.165
76.740
75.921
73.105
72.612
65.962
38.320
37.798
37.237
36.677
36.204
34.433
25.974
24.062
23.232
22.529
HO
NH
O
OH
O
O
3r
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm


109.1
%TRANSMITTANCE
7.900
4000
3000
2000
1000
400.0
Wavenumbers (cm-1)
3401.82
2956.34
2873.42
2372.01
2316.09
1646.91
1535.06
1442.48
1367.28
1334.50
1251.58
1201.43
1103.08
1039.44
975.804
906.379
863.953
620.966
489.831
HO
O
NH
OH
O
O
3r

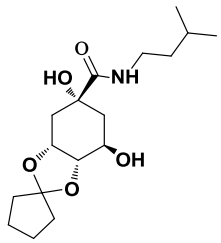

Composition View
Edit
Help
Spectrum #2 *23.23
3732
65
64
63
62
327.2048
327.15
327.20
327.25
Max Error (mmu) 5
Min Error (mmu) 3
defect (ppm) 1000
STD Error (mmu) 20
Max U.S. 36.0
Min U.S. -0.1
Fig-Chk Off
STD-Chk Off
Element Max. Min. Isotope
C 17 0 1
H 29 4 1
O 5 0 1
N 2 0 1
Cl 1 0 1
Calculate
Filter
Cancel
Edit
Accept
Delete
Cancel
Format Composition Formula
Type in
Print
Elements : C 17/0, H 29/4, O 5/0, N 2/0, Cl 1/0
Mass Tolerance : 1000ppm, 3mmu if m/z < 3, 5mmu if m/z > 5
Unsaturation : -0.1 ~ 36.0
No. m/z Int% Err-mmu U.S. Composition
1 327.2048 100.0 +0.2 4.0 C17H29O5N


O
HO
NH
OH
O
O

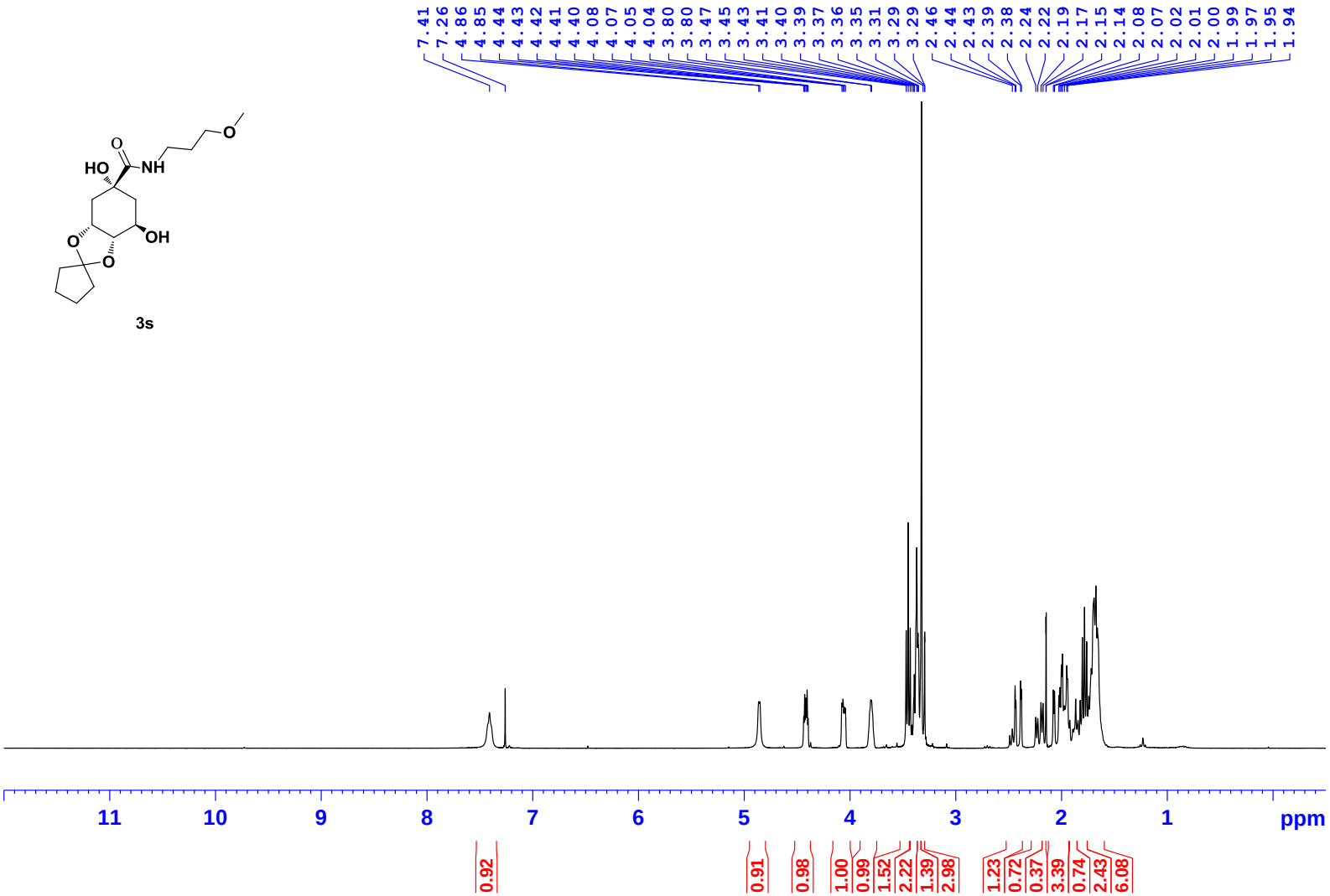
3r

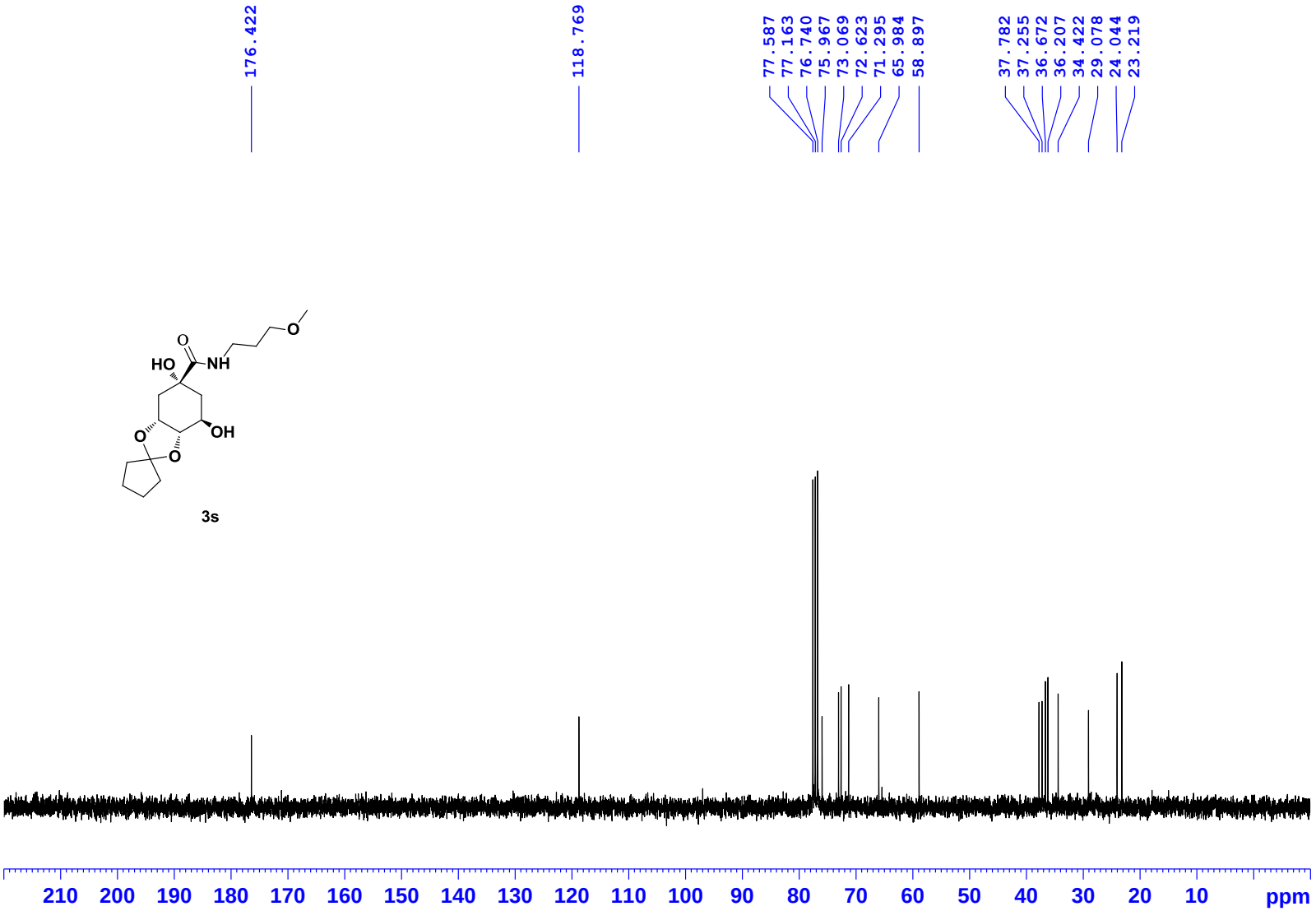
[Elemental Composition]
Data : 201105157 Date : 01-Jun-2011 15:11
Sample: leeut D-18
Note : -
Inlet : Direct Ion Mode : EI+
RT : 0.46 min Scan#: 17
Elements : C 17/0, H 29/4, O 5/0, N 2/0, Cl 1/0
Mass Tolerance : 1000ppm, 3mmu if m/z < 3, 5mmu if m/z > 5
Unsaturation (U.S.) : -0.1 - 36.0

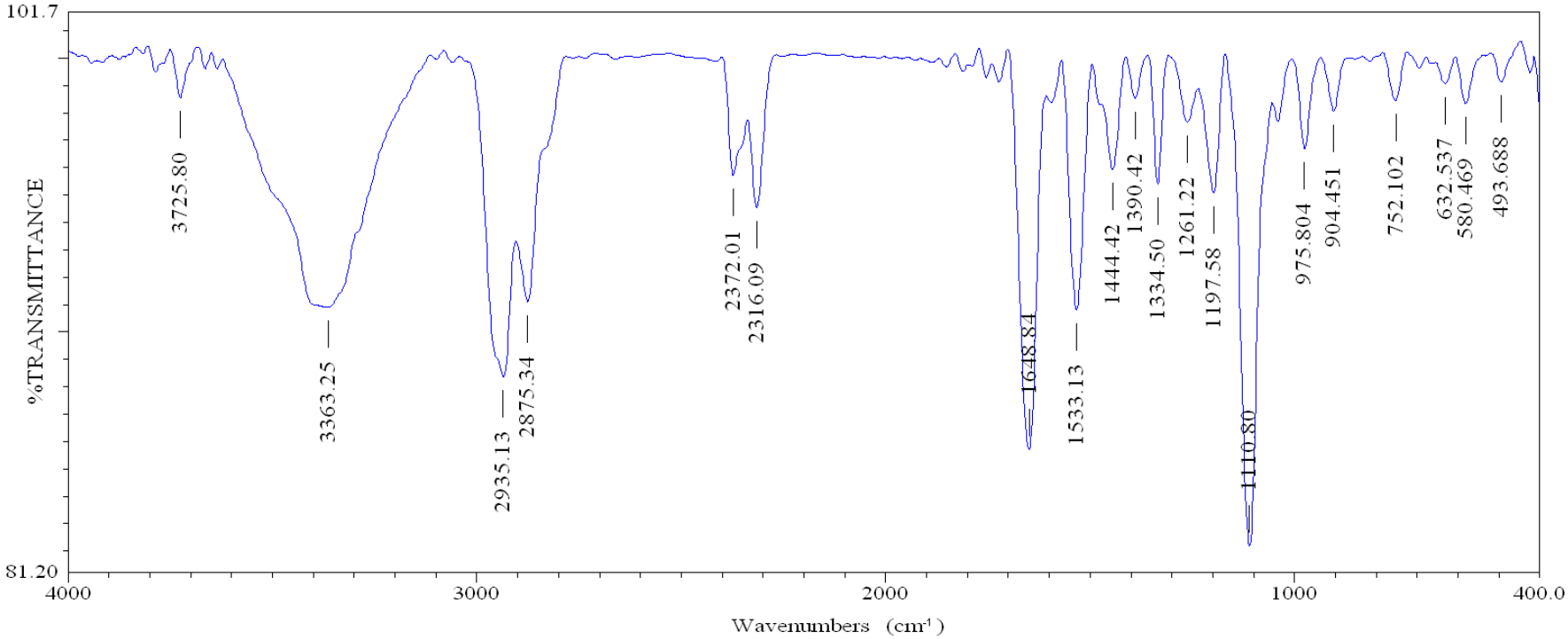
Observed m/z	Int%	Err[ppm / mmu]	U.S.	Composition
327.2048	100.0	+0.6 / +0.2	4.0	C 17 H 29 O 5 N



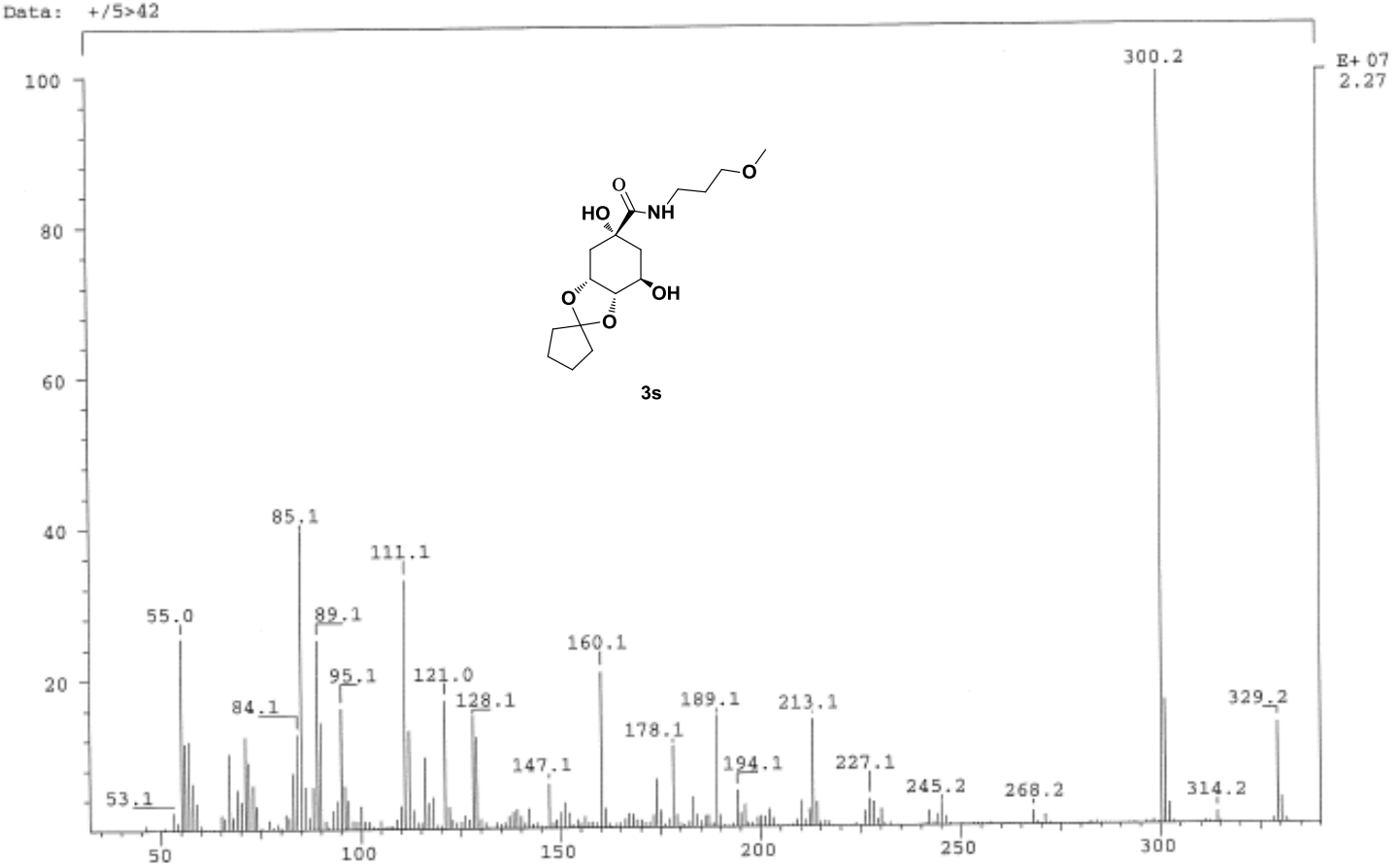
3r


7.41
7.26
4.86
4.85
4.44
4.43
4.42
4.41
4.40
4.08
4.07
4.05
4.04
3.80
3.47
3.45
3.43
3.41
3.40
3.39
3.37
3.36
3.35
3.31
3.29
3.29
2.46
2.44
2.43
2.39
2.38
2.24
2.22
2.19
2.17
2.15
2.14
2.08
2.07
2.02
2.01
2.00
1.99
1.97
1.95
1.94
HO
NH
O
O
OH
O
O
3s
11
10
9
8
7
6
5
4
3
2
1
ppm
0.92
0.91
0.98
1.00
0.99
1.52
2.22
1.39
2.98
1.23
0.72
0.37
3.39
0.74
2.43
6.08


176.422
118.769
77.587
77.163
76.740
75.967
73.069
72.623
71.295
65.984
58.897
37.782
37.255
36.672
36.207
34.422
29.078
24.044
23.219
HO
NH
O
OH
O
O
O
3s
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm


101.7
81.20
%TRANSMITTANCE
3725.80
3363.25
2935.13
2875.34
2372.01
2316.09
1648.84
1533.13
1444.42
1390.42
1334.50
1261.22
1197.58
1110.80
975.804
904.451
752.102
632.537
580.469
493.688
4000
3000
2000
1000
400.0
Wavenumbers (cm-1)

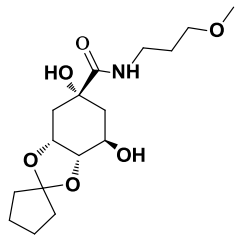
SPEC: lei2837 01-Mar-12 REG : 00:31.7 #9
Samp: 1333leeutD19 Start : 10:05:53 212
Mode: EI +VE +LMR BSCAN (EXP) UP LR NRM
Oper: Inlet :
Base: 300.2 Inten : 22666186 Masses: 45 > 400
Norm: 300.2 RIC : 169837677 #peaks: 354
Peak: 1000.00 mmu
Data: +/5>42



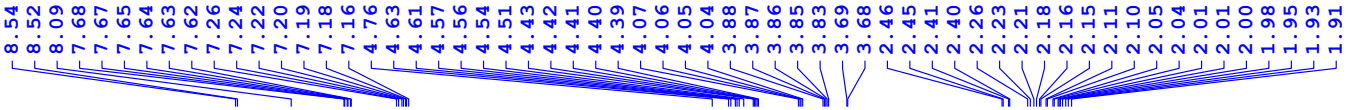
3s

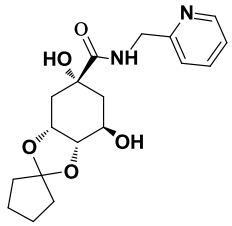
```
LIST: hei1422-c3                                  05-Mar-12 REG    : 05:38.3      #9
Samp: 13331eeutd19                                          Start  : 18:51:48   2225
Mode: EI +VE +LMR  ESCAN (EXP) UP HR NRM
Oper:                                                       Inlet  :
Limt: (  0)     .    . .
    : (371) C19.H33.N.O6
Peak: 1000.00 mmu   R+D: -2.0 > 60.0
Data: +/1013>1340  (CMASS : converted |CMASS : converted |CMASS : con
```

Mass	Intensity	%RA	Flags	(mmu) Delta	R+D	Composition
329.1829	48872	89.13	#	0.9	4.0	C16.H27.N.O6

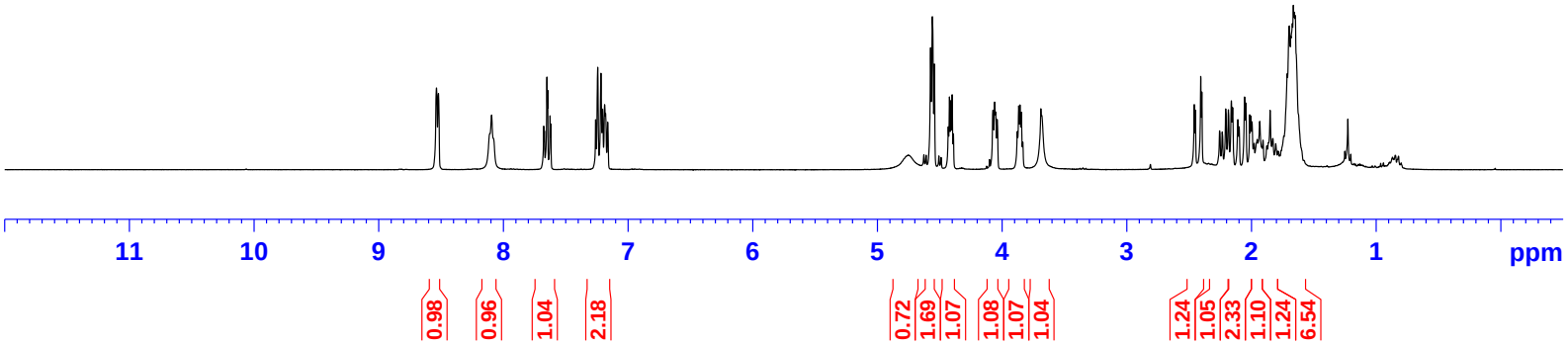


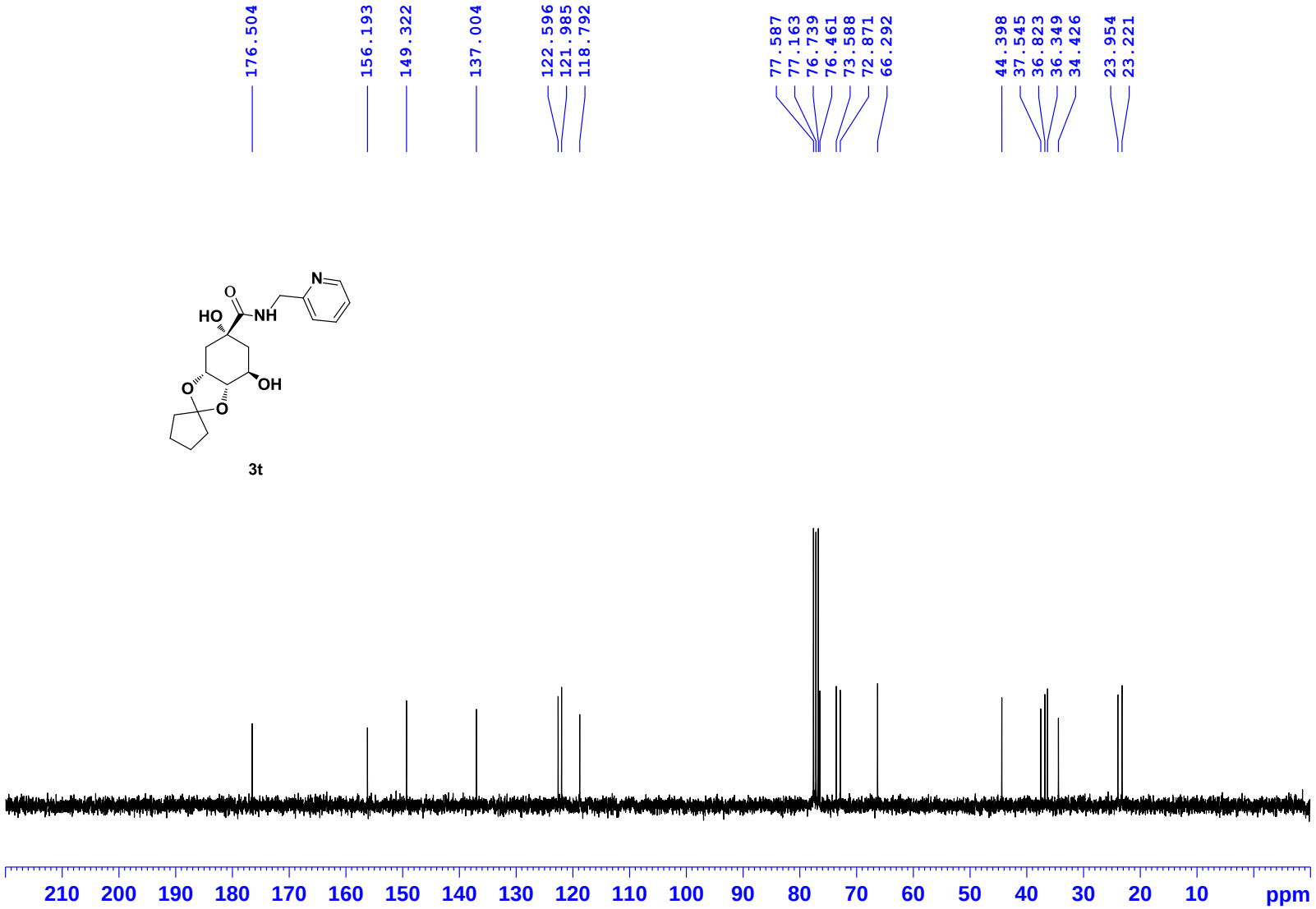
3s

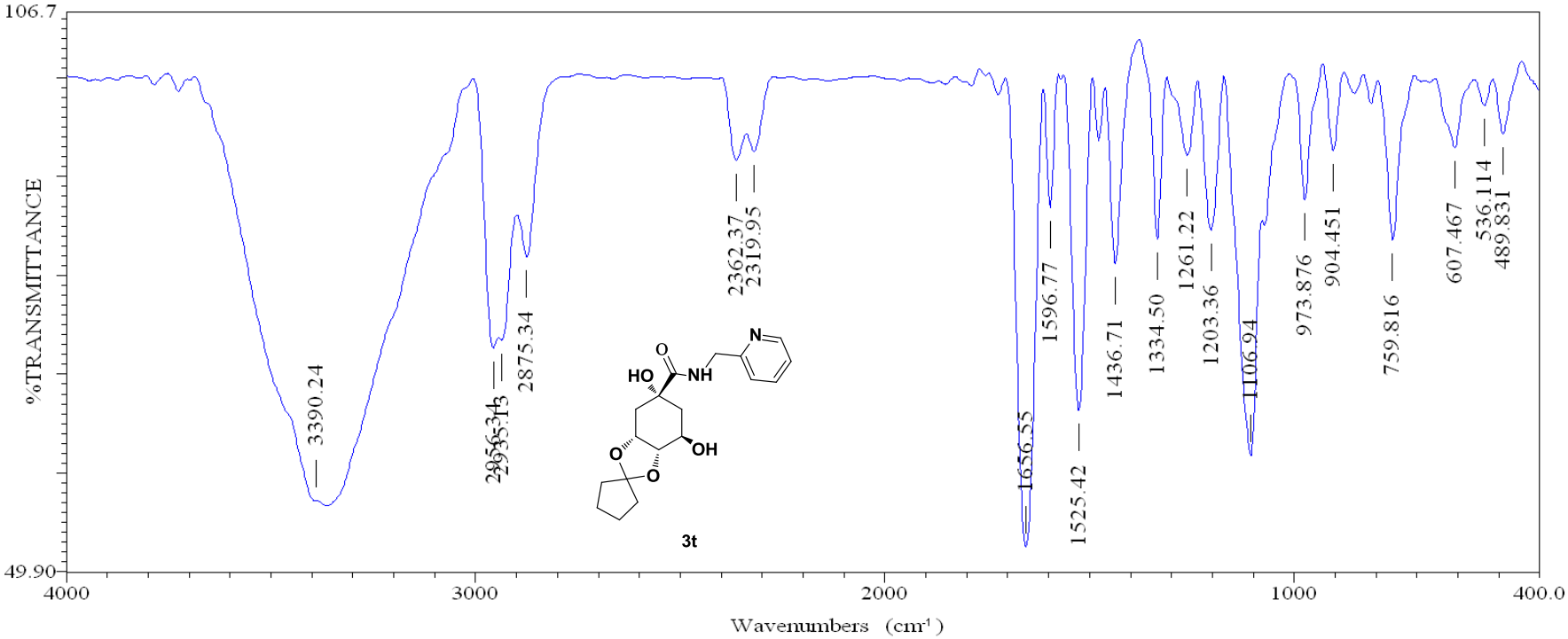

8.54
8.52
8.09
7.68
7.67
7.65
7.64
7.63
7.62
7.26
7.24
7.22
7.20
7.19
7.18
7.16
4.76
4.63
4.61
4.57
4.56
4.54
4.51
4.43
4.42
4.41
4.40
4.39
4.07
4.06
4.05
4.04
3.88
3.87
3.86
3.85
3.83
3.69
3.68
2.46
2.45
2.41
2.40
2.26
2.23
2.21
2.18
2.16
2.15
2.11
2.10
2.05
2.04
2.01
2.01
2.00
1.98
1.95
1.93
1.91


HO
O
NH
N
OH
O
O

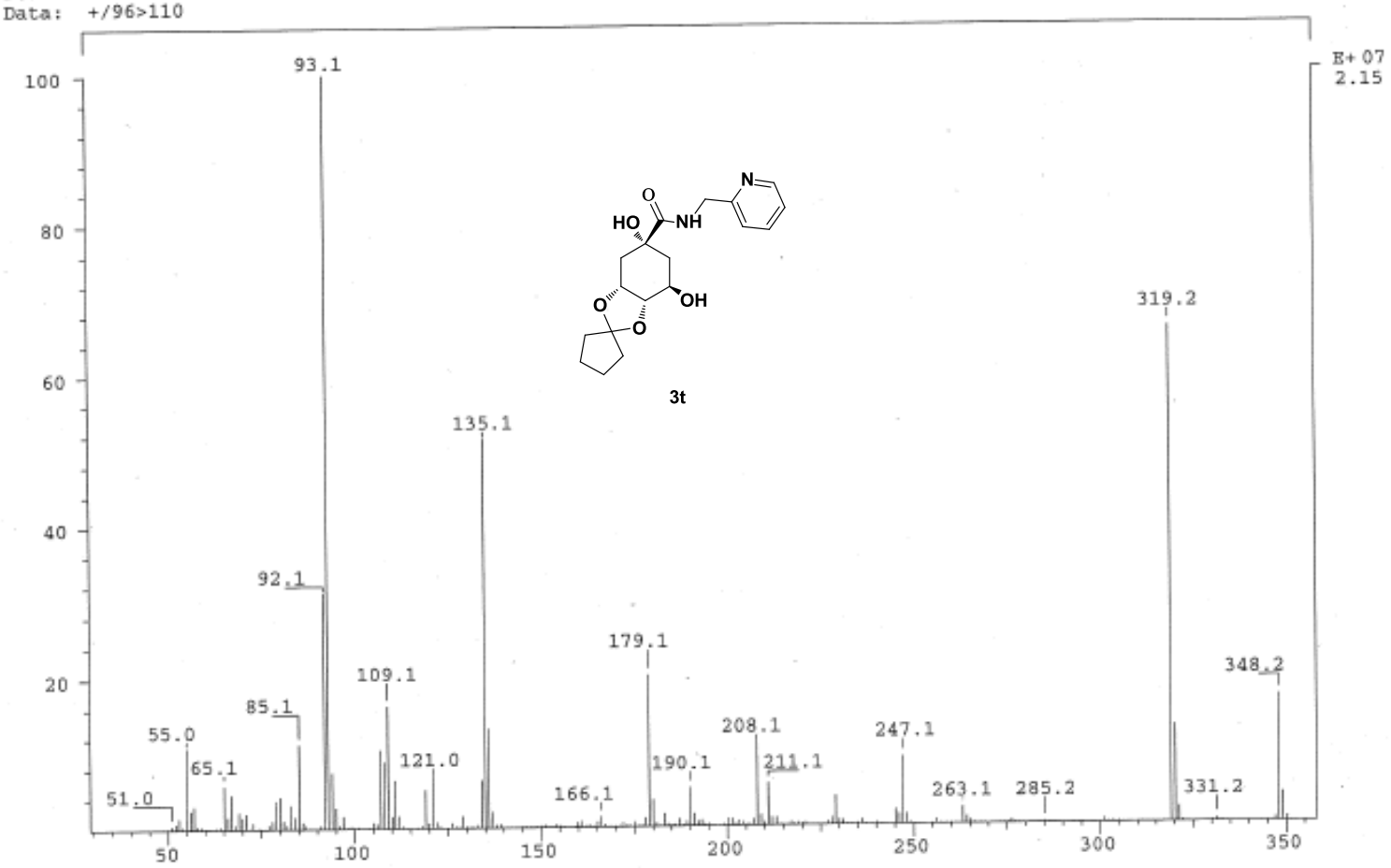
3t


11
10
9
8
7
6
5
4
3
2
1
ppm
0.98
0.96
1.04
2.18
0.72
1.69
1.07
1.08
1.07
1.04
1.24
1.05
2.33
1.10
1.24
6.54


176.504
156.193
149.322
137.004
122.596
121.985
118.792
77.587
77.163
76.739
76.461
73.588
72.871
66.292
44.398
37.545
36.823
36.349
34.426
23.954
23.221
HO
O
NH
N
OH
O
O
3t
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm


106.7
%TRANSMITTANCE
49.90
3390.24
2956.34
2935.13
2875.34
2362.37
2319.95
1656.55
1596.77
1525.42
1436.71
1334.50
1261.22
1203.36
1106.94
973.876
904.451
759.816
607.467
536.114
489.831
HO
NH
N
O
OH
O
O
3t
4000
3000
2000
1000
400.0
Wavenumbers (cm-1)

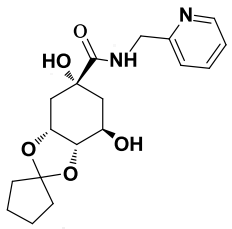
SPEC: lei5119(leeut-d20) 15-Sep-11 REG : 09:01.2 #9
Samp: Start : 10:07:13 200
Mode: EI +VE +LMR BSCAN (EXP) UP LR NRM
Oper: Inlet :
Base: 93.1 Inten : 21473074 Masses: 45 > 530
Norm: 93.1 RIC : 121254407 #peaks: 421
Peak: 1000.00 mmu
Data: +/96>110



3t

```
LIST: hei2527(leeutd20)-c1                    29-Sep-11 REG   : 04:03.6     #9
Samp:                                                   Start : 14:01:18  1153
Mode: EI +VE +LMR  ESCAN (EXP) UP µR NRM                Inlet :
Oper:
Limt: (  0)    .    .    .
    : (392) C20.H44.N2.O5
Peak: 1000.00 mmu   R+D: -2.0 > 60.0
Data: +/752>944  (CMASS : converted |CMASS : converted |CMASS : conve
```

Mass	Intensity	%RA	Flags	(mmu) Delta	R+D	Composition
348.1695	171096	100.00	#	-1.0	8.0	C18.H24.N2.O5



3t

ORTEP representation of compound 3k (cyn 1324)

CCDC number 844606

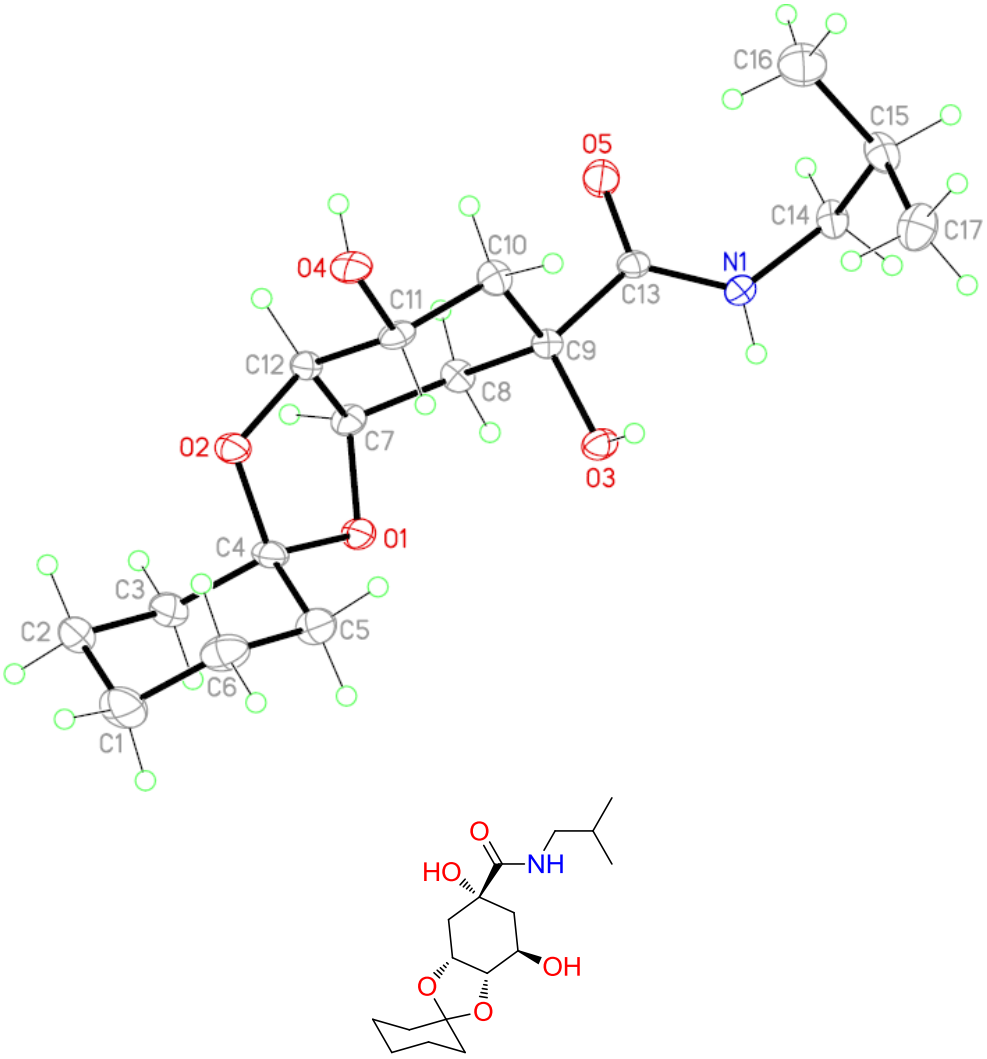


Table 1. Crystal data and structure refinement for 101129LTB.

Identification code	101129ltb	
Empirical formula	C17 H29 N O5	
Formula weight	327.41	
Temperature	100(2) K	
Wavelength	0.71073 Å	
Crystal system	Triclinic	
Space group	P 1	
Unit cell dimensions	a = 11.0822(7) Å	α= 96.815(4)°.
	b = 11.5824(7) Å	β= 99.359(4)°.
	c = 15.3798(14) Å	γ = 115.128(3)°.
Volume	1724.1(2) $Å^3$	
Z	4	
Density (calculated)	1.261 Mg/m^3	
Absorption coefficient	0.092 mm^{-1}	
F(000)	712	
Crystal size	0.22 x 0.20 x 0.12 mm^3	
Theta range for data collection	1.37 to 26.47°.	
Index ranges	-13<=h<=13, -14<=k<=13, -18<=l<=19	
Reflections collected	28034	
Independent reflections	12032 [R(int) = 0.1095]	
Completeness to theta = 26.47°	98.3 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	0.9486 and 0.7706	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	12032 / 3 / 846	
Goodness-of-fit on F^2	1.069	
Final R indices [I>2sigma(I)]	R1 = 0.0599, wR2 = 0.1379	
R indices (all data)	R1 = 0.0777, wR2 = 0.1859	
Absolute structure parameter	-1.5(9)	
Extinction coefficient	0.066(4)	
Largest diff. peak and hole	0.565 and -0.510 e.$Å^{-3}$	

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3)
for 101129LTB. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
C(1)	5940(5)	9670(5)	4630(3)	32(1)
C(2)	4743(5)	8262(5)	4415(3)	26(1)
C(3)	4876(5)	7417(4)	3631(3)	23(1)
C(4)	4929(4)	8016(4)	2788(3)	17(1)
C(5)	6112(5)	9420(4)	3006(3)	24(1)
C(6)	6000(5)	10266(4)	3791(3)	26(1)
C(7)	3756(4)	6466(4)	1478(3)	16(1)
C(8)	3830(4)	6003(4)	532(3)	14(1)
C(9)	4112(4)	6999(4)	-77(3)	16(1)
C(10)	3229(4)	7705(4)	1(3)	17(1)
C(11)	3370(4)	8329(4)	955(3)	16(1)
C(12)	3070(4)	7367(4)	1570(3)	15(1)
C(13)	3662(4)	6295(4)	-1079(3)	14(1)
C(14)	4279(4)	6291(4)	-2540(3)	17(1)
C(15)	3972(5)	7208(4)	-3078(3)	21(1)
C(16)	2612(5)	7159(5)	-3019(3)	33(1)
C(17)	5122(5)	8591(4)	-2788(3)	27(1)
C(18)	9524(4)	3288(4)	2675(3)	21(1)
C(19)	10068(5)	2844(5)	3478(3)	26(1)
C(20)	11421(5)	3897(5)	4037(3)	31(1)
C(21)	11330(5)	5149(5)	4327(3)	30(1)
C(22)	10809(5)	5586(5)	3533(3)	31(1)
C(23)	9453(5)	4532(5)	2969(3)	28(1)
C(24)	8364(5)	1529(4)	1425(3)	21(1)
C(25)	7460(4)	1539(4)	556(3)	18(1)
C(26)	8196(4)	2769(4)	222(3)	18(1)
C(27)	9569(4)	2996(4)	11(3)	16(1)
C(28)	10267(4)	2341(4)	589(3)	19(1)
C(29)	9905(4)	2209(4)	1487(3)	19(1)
C(30)	9362(4)	2391(4)	-973(3)	16(1)
C(31)	10027(4)	2717(4)	-2421(3)	17(1)
C(32)	9193(5)	3166(4)	-3049(3)	23(1)

C(33)	7692(5)	2547(5)	-2986(3)	31(1)
C(34)	9327(6)	2883(5)	-4012(3)	32(1)
C(35)	5652(5)	4649(5)	4627(3)	29(1)
C(36)	6908(5)	4440(5)	4961(3)	28(1)
C(37)	8164(5)	5693(5)	5415(3)	24(1)
C(38)	7869(5)	6455(4)	6167(3)	22(1)
C(39)	6621(5)	6668(4)	5830(3)	22(1)
C(40)	5348(5)	5387(5)	5372(3)	25(1)
C(41)	9594(4)	7760(4)	7472(3)	17(1)
C(42)	9140(4)	8542(4)	8105(3)	15(1)
C(43)	9409(4)	8304(4)	9056(3)	16(1)
C(44)	8498(4)	6878(4)	9056(3)	15(1)
C(45)	8845(5)	5990(4)	8429(3)	18(1)
C(46)	8969(4)	6312(4)	7508(3)	18(1)
C(47)	8780(4)	6664(4)	10025(3)	16(1)
C(48)	7996(5)	6510(4)	11420(3)	22(1)
C(49)	8305(5)	7735(5)	12077(3)	22(1)
C(50)	8317(5)	7421(5)	13014(3)	29(1)
C(51)	9647(5)	8849(5)	12050(3)	34(1)
C(52)	1571(5)	452(5)	4508(3)	33(1)
C(53)	847(5)	715(5)	5203(3)	29(1)
C(54)	1205(5)	259(5)	6065(3)	25(1)
C(55)	2728(5)	858(4)	6449(3)	18(1)
C(56)	3473(5)	629(5)	5752(3)	25(1)
C(57)	3131(5)	1095(5)	4903(3)	33(1)
C(58)	4335(4)	1310(4)	7729(3)	18(1)
C(59)	4513(5)	1250(4)	8712(3)	18(1)
C(60)	3620(4)	1645(4)	9221(3)	15(1)
C(61)	3217(4)	2623(4)	8790(3)	16(1)
C(62)	4258(4)	3460(4)	8302(3)	20(1)
C(63)	4395(4)	2602(4)	7523(3)	19(1)
C(64)	4458(4)	2267(4)	10190(3)	16(1)
C(65)	4951(5)	2018(4)	11746(3)	20(1)
C(66)	4337(4)	2571(4)	12401(3)	20(1)
C(67)	2935(5)	1541(5)	12467(3)	25(1)
C(68)	4225(5)	3769(4)	12174(3)	26(1)
N(1)	4574(4)	6747(3)	-1569(2)	16(1)
N(2)	10107(4)	3129(3)	-1469(2)	17(1)

N(3)	7880(4)	6681(3)	10490(2)	18(1)
N(4)	4140(4)	1580(3)	10813(2)	17(1)
O(1)	5101(3)	7275(3)	2061(2)	19(1)
O(2)	3618(3)	8011(3)	2500(2)	18(1)
O(3)	5559(3)	7852(3)	179(2)	18(1)
O(4)	2494(3)	8947(3)	973(2)	19(1)
O(5)	2477(3)	5427(3)	-1392(2)	22(1)
O(6)	8206(3)	2299(3)	2174(2)	26(1)
O(7)	10369(3)	3478(3)	2044(2)	20(1)
O(8)	7039(3)	366(3)	-89(2)	23(1)
O(9)	10505(3)	4359(3)	185(2)	18(1)
O(10)	8532(3)	1210(3)	-1280(2)	20(1)
O(11)	7640(3)	5800(3)	6899(2)	19(1)
O(12)	9049(3)	7698(3)	6541(2)	22(1)
O(13)	9925(3)	9884(3)	8097(2)	19(1)
O(14)	7134(3)	6683(3)	8771(2)	18(1)
O(15)	9804(3)	6515(3)	10326(2)	22(1)
O(16)	3006(3)	373(3)	7225(2)	21(1)
O(17)	3233(3)	2235(3)	6784(2)	22(1)
O(18)	2415(3)	540(3)	9262(2)	25(1)
O(19)	5582(3)	4249(3)	8877(2)	30(1)
O(20)	5456(3)	3397(3)	10364(2)	23(1)

Table 3. Bond lengths [Å] and angles [°] for 101129LTB.

C(1)-C(6)	1.533(7)
C(1)-C(2)	1.556(7)
C(1)-H(1A)	0.9900
C(1)-H(1B)	0.9900
C(2)-C(3)	1.528(7)
C(2)-H(2A)	0.9900
C(2)-H(2B)	0.9900
C(3)-C(4)	1.538(6)
C(3)-H(3A)	0.9900
C(3)-H(3B)	0.9900
C(4)-O(1)	1.416(5)
C(4)-O(2)	1.446(5)
C(4)-C(5)	1.547(6)
C(5)-C(6)	1.518(6)
C(5)-H(5A)	0.9900
C(5)-H(5B)	0.9900
C(6)-H(6A)	0.9900
C(6)-H(6B)	0.9900
C(7)-O(1)	1.444(5)
C(7)-C(8)	1.516(6)
C(7)-C(12)	1.536(6)
C(7)-H(7)	1.0000
C(8)-C(9)	1.534(5)
C(8)-H(8A)	0.9900
C(8)-H(8B)	0.9900
C(9)-O(3)	1.435(5)
C(9)-C(10)	1.527(6)
C(9)-C(13)	1.549(6)
C(10)-C(11)	1.510(6)
C(10)-H(10A)	0.9900
C(10)-H(10B)	0.9900
C(11)-O(4)	1.432(5)
C(11)-C(12)	1.512(5)
C(11)-H(11)	1.0000
C(12)-O(2)	1.431(5)
C(12)-H(12)	1.0000

C(13)-O(5)	1.235(5)
C(13)-N(1)	1.324(5)
C(14)-N(1)	1.458(5)
C(14)-C(15)	1.533(5)
C(14)-H(14A)	0.9900
C(14)-H(14B)	0.9900
C(15)-C(16)	1.501(7)
C(15)-C(17)	1.517(6)
C(15)-H(15)	1.0000
C(16)-H(16A)	0.9800
C(16)-H(16B)	0.9800
C(16)-H(16C)	0.9800
C(17)-H(17A)	0.9800
C(17)-H(17B)	0.9800
C(17)-H(17C)	0.9800
C(18)-O(7)	1.428(5)
C(18)-O(6)	1.430(5)
C(18)-C(23)	1.497(7)
C(18)-C(19)	1.525(6)
C(19)-C(20)	1.502(7)
C(19)-H(19A)	0.9900
C(19)-H(19B)	0.9900
C(20)-C(21)	1.515(7)
C(20)-H(20A)	0.9900
C(20)-H(20B)	0.9900
C(21)-C(22)	1.498(7)
C(21)-H(21A)	0.9900
C(21)-H(21B)	0.9900
C(22)-C(23)	1.506(7)
C(22)-H(22A)	0.9900
C(22)-H(22B)	0.9900
C(23)-H(23A)	0.9900
C(23)-H(23B)	0.9900
C(24)-O(6)	1.451(5)
C(24)-C(29)	1.527(6)
C(24)-C(25)	1.539(6)
C(24)-H(24)	1.0000
C(25)-O(8)	1.428(5)

C(25)-C(26)	1.511(5)
C(25)-H(25)	1.0000
C(26)-C(27)	1.526(6)
C(26)-H(26A)	0.9900
C(26)-H(26B)	0.9900
C(27)-O(9)	1.435(5)
C(27)-C(30)	1.528(6)
C(27)-C(28)	1.550(6)
C(28)-C(29)	1.507(6)
C(28)-H(28A)	0.9900
C(28)-H(28B)	0.9900
C(29)-O(7)	1.440(5)
C(29)-H(29)	1.0000
C(30)-O(10)	1.255(5)
C(30)-N(2)	1.324(5)
C(31)-N(2)	1.461(5)
C(31)-C(32)	1.516(6)
C(31)-H(31A)	0.9900
C(31)-H(31B)	0.9900
C(32)-C(34)	1.524(7)
C(32)-C(33)	1.532(7)
C(32)-H(32)	1.0000
C(33)-H(33A)	0.9800
C(33)-H(33B)	0.9800
C(33)-H(33C)	0.9800
C(34)-H(34A)	0.9800
C(34)-H(34B)	0.9800
C(34)-H(34C)	0.9800
C(35)-C(40)	1.516(7)
C(35)-C(36)	1.530(7)
C(35)-H(35A)	0.9900
C(35)-H(35B)	0.9900
C(36)-C(37)	1.503(7)
C(36)-H(36A)	0.9900
C(36)-H(36B)	0.9900
C(37)-C(38)	1.533(6)
C(37)-H(37A)	0.9900
C(37)-H(37B)	0.9900

C(38)-O(11)	1.428(5)
C(38)-O(12)	1.440(5)
C(38)-C(39)	1.526(7)
C(39)-C(40)	1.529(6)
C(39)-H(39A)	0.9900
C(39)-H(39B)	0.9900
C(40)-H(40A)	0.9900
C(40)-H(40B)	0.9900
C(41)-O(12)	1.441(5)
C(41)-C(46)	1.531(6)
C(41)-C(42)	1.536(6)
C(41)-H(41)	1.0000
C(42)-O(13)	1.427(4)
C(42)-C(43)	1.526(5)
C(42)-H(42)	1.0000
C(43)-C(44)	1.527(5)
C(43)-H(43A)	0.9900
C(43)-H(43B)	0.9900
C(44)-O(14)	1.414(5)
C(44)-C(45)	1.536(6)
C(44)-C(47)	1.544(6)
C(45)-C(46)	1.520(6)
C(45)-H(45A)	0.9900
C(45)-H(45B)	0.9900
C(46)-O(11)	1.441(5)
C(46)-H(46)	1.0000
C(47)-O(15)	1.243(5)
C(47)-N(3)	1.322(5)
C(48)-N(3)	1.460(5)
C(48)-C(49)	1.513(6)
C(48)-H(48A)	0.9900
C(48)-H(48B)	0.9900
C(49)-C(51)	1.510(7)
C(49)-C(50)	1.526(6)
C(49)-H(49)	1.0000
C(50)-H(50A)	0.9800
C(50)-H(50B)	0.9800
C(50)-H(50C)	0.9800

C(51)-H(51A)	0.9800
C(51)-H(51B)	0.9800
C(51)-H(51C)	0.9800
C(52)-C(53)	1.513(7)
C(52)-C(57)	1.539(7)
C(52)-H(52A)	0.9900
C(52)-H(52B)	0.9900
C(53)-C(54)	1.539(6)
C(53)-H(53A)	0.9900
C(53)-H(53B)	0.9900
C(54)-C(55)	1.504(6)
C(54)-H(54A)	0.9900
C(54)-H(54B)	0.9900
C(55)-O(16)	1.421(5)
C(55)-O(17)	1.441(5)
C(55)-C(56)	1.520(6)
C(56)-C(57)	1.524(7)
C(56)-H(56A)	0.9900
C(56)-H(56B)	0.9900
C(57)-H(57A)	0.9900
C(57)-H(57B)	0.9900
C(58)-O(16)	1.421(5)
C(58)-C(59)	1.506(6)
C(58)-C(63)	1.544(6)
C(58)-H(58)	1.0000
C(59)-C(60)	1.534(6)
C(59)-H(59A)	0.9900
C(59)-H(59B)	0.9900
C(60)-O(18)	1.422(5)
C(60)-C(64)	1.526(6)
C(60)-C(61)	1.560(6)
C(61)-C(62)	1.529(6)
C(61)-H(61A)	0.9900
C(61)-H(61B)	0.9900
C(62)-O(19)	1.421(5)
C(62)-C(63)	1.533(6)
C(62)-H(62)	1.0000
C(63)-O(17)	1.439(5)

C(63)-H(63)	1.0000
C(64)-O(20)	1.264(5)
C(64)-N(4)	1.315(5)
C(65)-N(4)	1.465(5)
C(65)-C(66)	1.525(6)
C(65)-H(65A)	0.9900
C(65)-H(65B)	0.9900
C(66)-C(68)	1.520(6)
C(66)-C(67)	1.534(6)
C(66)-H(66)	1.0000
C(67)-H(67A)	0.9800
C(67)-H(67B)	0.9800
C(67)-H(67C)	0.9800
C(68)-H(68A)	0.9800
C(68)-H(68B)	0.9800
C(68)-H(68C)	0.9800
N(1)-H(1N)	0.8800
N(2)-H(2N)	0.8800
N(3)-H(3N)	0.8800
N(4)-H(4N)	0.8800
O(3)-H(3)	0.8400
O(4)-H(4)	0.8400
O(8)-H(8)	0.8400
O(9)-H(9)	0.8400
O(13)-H(13)	0.8400
O(14)-H(14)	0.8400
O(18)-H(18)	0.8400
O(19)-H(19)	0.8400

C(6)-C(1)-C(2)	110.9(4)
C(6)-C(1)-H(1A)	109.5
C(2)-C(1)-H(1A)	109.5
C(6)-C(1)-H(1B)	109.5
C(2)-C(1)-H(1B)	109.5
H(1A)-C(1)-H(1B)	108.0
C(3)-C(2)-C(1)	110.3(4)
C(3)-C(2)-H(2A)	109.6
C(1)-C(2)-H(2A)	109.6

C(3)-C(2)-H(2B)	109.6
C(1)-C(2)-H(2B)	109.6
H(2A)-C(2)-H(2B)	108.1
C(2)-C(3)-C(4)	111.6(4)
C(2)-C(3)-H(3A)	109.3
C(4)-C(3)-H(3A)	109.3
C(2)-C(3)-H(3B)	109.3
C(4)-C(3)-H(3B)	109.3
H(3A)-C(3)-H(3B)	108.0
O(1)-C(4)-O(2)	107.1(3)
O(1)-C(4)-C(3)	112.7(3)
O(2)-C(4)-C(3)	106.9(3)
O(1)-C(4)-C(5)	108.5(3)
O(2)-C(4)-C(5)	110.8(3)
C(3)-C(4)-C(5)	110.7(3)
C(6)-C(5)-C(4)	111.3(4)
C(6)-C(5)-H(5A)	109.4
C(4)-C(5)-H(5A)	109.4
C(6)-C(5)-H(5B)	109.4
C(4)-C(5)-H(5B)	109.4
H(5A)-C(5)-H(5B)	108.0
C(5)-C(6)-C(1)	111.9(4)
C(5)-C(6)-H(6A)	109.2
C(1)-C(6)-H(6A)	109.2
C(5)-C(6)-H(6B)	109.2
C(1)-C(6)-H(6B)	109.2
H(6A)-C(6)-H(6B)	107.9
O(1)-C(7)-C(8)	112.0(3)
O(1)-C(7)-C(12)	101.3(3)
C(8)-C(7)-C(12)	116.3(3)
O(1)-C(7)-H(7)	109.0
C(8)-C(7)-H(7)	109.0
C(12)-C(7)-H(7)	109.0
C(7)-C(8)-C(9)	116.2(3)
C(7)-C(8)-H(8A)	108.2
C(9)-C(8)-H(8A)	108.2
C(7)-C(8)-H(8B)	108.2
C(9)-C(8)-H(8B)	108.2

H(8A)-C(8)-H(8B)	107.4
O(3)-C(9)-C(10)	113.7(3)
O(3)-C(9)-C(8)	106.8(3)
C(10)-C(9)-C(8)	110.4(3)
O(3)-C(9)-C(13)	111.0(3)
C(10)-C(9)-C(13)	104.8(3)
C(8)-C(9)-C(13)	110.3(3)
C(11)-C(10)-C(9)	114.0(3)
C(11)-C(10)-H(10A)	108.8
C(9)-C(10)-H(10A)	108.8
C(11)-C(10)-H(10B)	108.8
C(9)-C(10)-H(10B)	108.8
H(10A)-C(10)-H(10B)	107.6
O(4)-C(11)-C(10)	110.6(3)
O(4)-C(11)-C(12)	109.8(3)
C(10)-C(11)-C(12)	112.5(3)
O(4)-C(11)-H(11)	107.9
C(10)-C(11)-H(11)	107.9
C(12)-C(11)-H(11)	107.9
O(2)-C(12)-C(11)	111.7(3)
O(2)-C(12)-C(7)	102.2(3)
C(11)-C(12)-C(7)	114.4(3)
O(2)-C(12)-H(12)	109.4
C(11)-C(12)-H(12)	109.4
C(7)-C(12)-H(12)	109.4
O(5)-C(13)-N(1)	123.8(4)
O(5)-C(13)-C(9)	119.7(3)
N(1)-C(13)-C(9)	116.2(3)
N(1)-C(14)-C(15)	112.8(3)
N(1)-C(14)-H(14A)	109.0
C(15)-C(14)-H(14A)	109.0
N(1)-C(14)-H(14B)	109.0
C(15)-C(14)-H(14B)	109.0
H(14A)-C(14)-H(14B)	107.8
C(16)-C(15)-C(17)	111.6(4)
C(16)-C(15)-C(14)	111.2(4)
C(17)-C(15)-C(14)	111.5(4)
C(16)-C(15)-H(15)	107.4

C(17)-C(15)-H(15)	107.4
C(14)-C(15)-H(15)	107.4
C(15)-C(16)-H(16A)	109.5
C(15)-C(16)-H(16B)	109.5
H(16A)-C(16)-H(16B)	109.5
C(15)-C(16)-H(16C)	109.5
H(16A)-C(16)-H(16C)	109.5
H(16B)-C(16)-H(16C)	109.5
C(15)-C(17)-H(17A)	109.5
C(15)-C(17)-H(17B)	109.5
H(17A)-C(17)-H(17B)	109.5
C(15)-C(17)-H(17C)	109.5
H(17A)-C(17)-H(17C)	109.5
H(17B)-C(17)-H(17C)	109.5
O(7)-C(18)-O(6)	104.3(3)
O(7)-C(18)-C(23)	108.8(4)
O(6)-C(18)-C(23)	110.2(4)
O(7)-C(18)-C(19)	111.1(4)
O(6)-C(18)-C(19)	110.9(3)
C(23)-C(18)-C(19)	111.3(4)
C(20)-C(19)-C(18)	111.6(4)
C(20)-C(19)-H(19A)	109.3
C(18)-C(19)-H(19A)	109.3
C(20)-C(19)-H(19B)	109.3
C(18)-C(19)-H(19B)	109.3
H(19A)-C(19)-H(19B)	108.0
C(19)-C(20)-C(21)	111.2(4)
C(19)-C(20)-H(20A)	109.4
C(21)-C(20)-H(20A)	109.4
C(19)-C(20)-H(20B)	109.4
C(21)-C(20)-H(20B)	109.4
H(20A)-C(20)-H(20B)	108.0
C(22)-C(21)-C(20)	111.4(4)
C(22)-C(21)-H(21A)	109.3
C(20)-C(21)-H(21A)	109.3
C(22)-C(21)-H(21B)	109.3
C(20)-C(21)-H(21B)	109.3
H(21A)-C(21)-H(21B)	108.0

C(21)-C(22)-C(23)	111.5(4)
C(21)-C(22)-H(22A)	109.3
C(23)-C(22)-H(22A)	109.3
C(21)-C(22)-H(22B)	109.3
C(23)-C(22)-H(22B)	109.3
H(22A)-C(22)-H(22B)	108.0
C(18)-C(23)-C(22)	111.9(4)
C(18)-C(23)-H(23A)	109.2
C(22)-C(23)-H(23A)	109.2
C(18)-C(23)-H(23B)	109.2
C(22)-C(23)-H(23B)	109.2
H(23A)-C(23)-H(23B)	107.9
O(6)-C(24)-C(29)	103.3(3)
O(6)-C(24)-C(25)	107.8(3)
C(29)-C(24)-C(25)	115.0(4)
O(6)-C(24)-H(24)	110.2
C(29)-C(24)-H(24)	110.2
C(25)-C(24)-H(24)	110.2
O(8)-C(25)-C(26)	114.0(3)
O(8)-C(25)-C(24)	109.3(4)
C(26)-C(25)-C(24)	110.3(3)
O(8)-C(25)-H(25)	107.7
C(26)-C(25)-H(25)	107.7
C(24)-C(25)-H(25)	107.7
C(25)-C(26)-C(27)	116.0(3)
C(25)-C(26)-H(26A)	108.3
C(27)-C(26)-H(26A)	108.3
C(25)-C(26)-H(26B)	108.3
C(27)-C(26)-H(26B)	108.3
H(26A)-C(26)-H(26B)	107.4
O(9)-C(27)-C(26)	111.8(3)
O(9)-C(27)-C(30)	109.1(3)
C(26)-C(27)-C(30)	110.6(3)
O(9)-C(27)-C(28)	107.5(3)
C(26)-C(27)-C(28)	111.3(3)
C(30)-C(27)-C(28)	106.5(3)
C(29)-C(28)-C(27)	114.7(4)
C(29)-C(28)-H(28A)	108.6

C(27)-C(28)-H(28A)	108.6
C(29)-C(28)-H(28B)	108.6
C(27)-C(28)-H(28B)	108.6
H(28A)-C(28)-H(28B)	107.6
O(7)-C(29)-C(28)	110.2(3)
O(7)-C(29)-C(24)	104.2(3)
C(28)-C(29)-C(24)	113.9(4)
O(7)-C(29)-H(29)	109.5
C(28)-C(29)-H(29)	109.5
C(24)-C(29)-H(29)	109.5
O(10)-C(30)-N(2)	122.0(4)
O(10)-C(30)-C(27)	119.6(4)
N(2)-C(30)-C(27)	118.3(4)
N(2)-C(31)-C(32)	113.5(4)
N(2)-C(31)-H(31A)	108.9
C(32)-C(31)-H(31A)	108.9
N(2)-C(31)-H(31B)	108.9
C(32)-C(31)-H(31B)	108.9
H(31A)-C(31)-H(31B)	107.7
C(31)-C(32)-C(34)	110.7(4)
C(31)-C(32)-C(33)	110.8(4)
C(34)-C(32)-C(33)	111.8(4)
C(31)-C(32)-H(32)	107.8
C(34)-C(32)-H(32)	107.8
C(33)-C(32)-H(32)	107.8
C(32)-C(33)-H(33A)	109.5
C(32)-C(33)-H(33B)	109.5
H(33A)-C(33)-H(33B)	109.5
C(32)-C(33)-H(33C)	109.5
H(33A)-C(33)-H(33C)	109.5
H(33B)-C(33)-H(33C)	109.5
C(32)-C(34)-H(34A)	109.5
C(32)-C(34)-H(34B)	109.5
H(34A)-C(34)-H(34B)	109.5
C(32)-C(34)-H(34C)	109.5
H(34A)-C(34)-H(34C)	109.5
H(34B)-C(34)-H(34C)	109.5
C(40)-C(35)-C(36)	112.3(4)

C(40)-C(35)-H(35A)	109.1
C(36)-C(35)-H(35A)	109.1
C(40)-C(35)-H(35B)	109.1
C(36)-C(35)-H(35B)	109.1
H(35A)-C(35)-H(35B)	107.9
C(37)-C(36)-C(35)	112.7(4)
C(37)-C(36)-H(36A)	109.0
C(35)-C(36)-H(36A)	109.0
C(37)-C(36)-H(36B)	109.0
C(35)-C(36)-H(36B)	109.0
H(36A)-C(36)-H(36B)	107.8
C(36)-C(37)-C(38)	110.8(4)
C(36)-C(37)-H(37A)	109.5
C(38)-C(37)-H(37A)	109.5
C(36)-C(37)-H(37B)	109.5
C(38)-C(37)-H(37B)	109.5
H(37A)-C(37)-H(37B)	108.1
O(11)-C(38)-O(12)	105.7(3)
O(11)-C(38)-C(39)	108.3(4)
O(12)-C(38)-C(39)	109.4(4)
O(11)-C(38)-C(37)	111.4(3)
O(12)-C(38)-C(37)	109.7(4)
C(39)-C(38)-C(37)	112.1(3)
C(38)-C(39)-C(40)	112.1(4)
C(38)-C(39)-H(39A)	109.2
C(40)-C(39)-H(39A)	109.2
C(38)-C(39)-H(39B)	109.2
C(40)-C(39)-H(39B)	109.2
H(39A)-C(39)-H(39B)	107.9
C(35)-C(40)-C(39)	110.2(4)
C(35)-C(40)-H(40A)	109.6
C(39)-C(40)-H(40A)	109.6
C(35)-C(40)-H(40B)	109.6
C(39)-C(40)-H(40B)	109.6
H(40A)-C(40)-H(40B)	108.1
O(12)-C(41)-C(46)	101.3(3)
O(12)-C(41)-C(42)	111.1(3)
C(46)-C(41)-C(42)	112.7(3)

O(12)-C(41)-H(41)	110.5
C(46)-C(41)-H(41)	110.5
C(42)-C(41)-H(41)	110.5
O(13)-C(42)-C(43)	110.4(3)
O(13)-C(42)-C(41)	106.9(3)
C(43)-C(42)-C(41)	109.9(3)
O(13)-C(42)-H(42)	109.8
C(43)-C(42)-H(42)	109.8
C(41)-C(42)-H(42)	109.8
C(42)-C(43)-C(44)	109.3(3)
C(42)-C(43)-H(43A)	109.8
C(44)-C(43)-H(43A)	109.8
C(42)-C(43)-H(43B)	109.8
C(44)-C(43)-H(43B)	109.8
H(43A)-C(43)-H(43B)	108.3
O(14)-C(44)-C(43)	105.7(3)
O(14)-C(44)-C(45)	112.7(3)
C(43)-C(44)-C(45)	109.7(3)
O(14)-C(44)-C(47)	111.0(3)
C(43)-C(44)-C(47)	108.4(3)
C(45)-C(44)-C(47)	109.1(3)
C(46)-C(45)-C(44)	115.8(3)
C(46)-C(45)-H(45A)	108.3
C(44)-C(45)-H(45A)	108.3
C(46)-C(45)-H(45B)	108.3
C(44)-C(45)-H(45B)	108.3
H(45A)-C(45)-H(45B)	107.4
O(11)-C(46)-C(45)	111.3(3)
O(11)-C(46)-C(41)	102.1(3)
C(45)-C(46)-C(41)	116.4(3)
O(11)-C(46)-H(46)	108.9
C(45)-C(46)-H(46)	108.9
C(41)-C(46)-H(46)	108.9
O(15)-C(47)-N(3)	124.5(4)
O(15)-C(47)-C(44)	120.5(4)
N(3)-C(47)-C(44)	115.0(4)
N(3)-C(48)-C(49)	112.4(4)
N(3)-C(48)-H(48A)	109.1

C(49)-C(48)-H(48A)	109.1
N(3)-C(48)-H(48B)	109.1
C(49)-C(48)-H(48B)	109.1
H(48A)-C(48)-H(48B)	107.9
C(51)-C(49)-C(48)	111.2(4)
C(51)-C(49)-C(50)	111.9(4)
C(48)-C(49)-C(50)	107.9(4)
C(51)-C(49)-H(49)	108.6
C(48)-C(49)-H(49)	108.6
C(50)-C(49)-H(49)	108.6
C(49)-C(50)-H(50A)	109.5
C(49)-C(50)-H(50B)	109.5
H(50A)-C(50)-H(50B)	109.5
C(49)-C(50)-H(50C)	109.5
H(50A)-C(50)-H(50C)	109.5
H(50B)-C(50)-H(50C)	109.5
C(49)-C(51)-H(51A)	109.5
C(49)-C(51)-H(51B)	109.5
H(51A)-C(51)-H(51B)	109.5
C(49)-C(51)-H(51C)	109.5
H(51A)-C(51)-H(51C)	109.5
H(51B)-C(51)-H(51C)	109.5
C(53)-C(52)-C(57)	110.6(4)
C(53)-C(52)-H(52A)	109.5
C(57)-C(52)-H(52A)	109.5
C(53)-C(52)-H(52B)	109.5
C(57)-C(52)-H(52B)	109.5
H(52A)-C(52)-H(52B)	108.1
C(52)-C(53)-C(54)	110.9(4)
C(52)-C(53)-H(53A)	109.5
C(54)-C(53)-H(53A)	109.5
C(52)-C(53)-H(53B)	109.5
C(54)-C(53)-H(53B)	109.5
H(53A)-C(53)-H(53B)	108.1
C(55)-C(54)-C(53)	112.2(4)
C(55)-C(54)-H(54A)	109.2
C(53)-C(54)-H(54A)	109.2
C(55)-C(54)-H(54B)	109.2

C(53)-C(54)-H(54B)	109.2
H(54A)-C(54)-H(54B)	107.9
O(16)-C(55)-O(17)	104.4(3)
O(16)-C(55)-C(54)	110.0(3)
O(17)-C(55)-C(54)	108.8(4)
O(16)-C(55)-C(56)	111.3(4)
O(17)-C(55)-C(56)	110.4(4)
C(54)-C(55)-C(56)	111.7(3)
C(55)-C(56)-C(57)	111.5(4)
C(55)-C(56)-H(56A)	109.3
C(57)-C(56)-H(56A)	109.3
C(55)-C(56)-H(56B)	109.3
C(57)-C(56)-H(56B)	109.3
H(56A)-C(56)-H(56B)	108.0
C(56)-C(57)-C(52)	111.0(4)
C(56)-C(57)-H(57A)	109.4
C(52)-C(57)-H(57A)	109.4
C(56)-C(57)-H(57B)	109.4
C(52)-C(57)-H(57B)	109.4
H(57A)-C(57)-H(57B)	108.0
O(16)-C(58)-C(59)	111.0(3)
O(16)-C(58)-C(63)	102.6(3)
C(59)-C(58)-C(63)	114.7(3)
O(16)-C(58)-H(58)	109.5
C(59)-C(58)-H(58)	109.5
C(63)-C(58)-H(58)	109.5
C(58)-C(59)-C(60)	116.5(4)
C(58)-C(59)-H(59A)	108.2
C(60)-C(59)-H(59A)	108.2
C(58)-C(59)-H(59B)	108.2
C(60)-C(59)-H(59B)	108.2
H(59A)-C(59)-H(59B)	107.3
O(18)-C(60)-C(64)	106.6(3)
O(18)-C(60)-C(59)	111.6(3)
C(64)-C(60)-C(59)	106.7(3)
O(18)-C(60)-C(61)	109.6(3)
C(64)-C(60)-C(61)	110.5(3)
C(59)-C(60)-C(61)	111.7(3)

C(62)-C(61)-C(60)	113.7(4)
C(62)-C(61)-H(61A)	108.8
C(60)-C(61)-H(61A)	108.8
C(62)-C(61)-H(61B)	108.8
C(60)-C(61)-H(61B)	108.8
H(61A)-C(61)-H(61B)	107.7
O(19)-C(62)-C(61)	113.2(3)
O(19)-C(62)-C(63)	108.4(4)
C(61)-C(62)-C(63)	110.7(3)
O(19)-C(62)-H(62)	108.2
C(61)-C(62)-H(62)	108.2
C(63)-C(62)-H(62)	108.2
O(17)-C(63)-C(62)	107.3(3)
O(17)-C(63)-C(58)	105.7(3)
C(62)-C(63)-C(58)	114.2(3)
O(17)-C(63)-H(63)	109.9
C(62)-C(63)-H(63)	109.9
C(58)-C(63)-H(63)	109.9
O(20)-C(64)-N(4)	122.2(4)
O(20)-C(64)-C(60)	119.6(3)
N(4)-C(64)-C(60)	118.2(4)
N(4)-C(65)-C(66)	113.8(4)
N(4)-C(65)-H(65A)	108.8
C(66)-C(65)-H(65A)	108.8
N(4)-C(65)-H(65B)	108.8
C(66)-C(65)-H(65B)	108.8
H(65A)-C(65)-H(65B)	107.7
C(68)-C(66)-C(65)	112.8(3)
C(68)-C(66)-C(67)	110.0(4)
C(65)-C(66)-C(67)	112.0(4)
C(68)-C(66)-H(66)	107.3
C(65)-C(66)-H(66)	107.3
C(67)-C(66)-H(66)	107.3
C(66)-C(67)-H(67A)	109.5
C(66)-C(67)-H(67B)	109.5
H(67A)-C(67)-H(67B)	109.5
C(66)-C(67)-H(67C)	109.5
H(67A)-C(67)-H(67C)	109.5

H(67B)-C(67)-H(67C)	109.5
C(66)-C(68)-H(68A)	109.5
C(66)-C(68)-H(68B)	109.5
H(68A)-C(68)-H(68B)	109.5
C(66)-C(68)-H(68C)	109.5
H(68A)-C(68)-H(68C)	109.5
H(68B)-C(68)-H(68C)	109.5
C(13)-N(1)-C(14)	123.6(4)
C(13)-N(1)-H(1N)	118.2
C(14)-N(1)-H(1N)	118.2
C(30)-N(2)-C(31)	125.6(3)
C(30)-N(2)-H(2N)	117.2
C(31)-N(2)-H(2N)	117.2
C(47)-N(3)-C(48)	122.4(4)
C(47)-N(3)-H(3N)	118.8
C(48)-N(3)-H(3N)	118.8
C(64)-N(4)-C(65)	123.2(4)
C(64)-N(4)-H(4N)	118.4
C(65)-N(4)-H(4N)	118.4
C(4)-O(1)-C(7)	107.0(3)
C(12)-O(2)-C(4)	107.9(3)
C(9)-O(3)-H(3)	109.5
C(11)-O(4)-H(4)	109.5
C(18)-O(6)-C(24)	109.6(3)
C(18)-O(7)-C(29)	105.8(3)
C(25)-O(8)-H(8)	109.5
C(27)-O(9)-H(9)	109.5
C(38)-O(11)-C(46)	105.3(3)
C(38)-O(12)-C(41)	109.6(3)
C(42)-O(13)-H(13)	109.5
C(44)-O(14)-H(14)	109.5
C(55)-O(16)-C(58)	105.9(3)
C(63)-O(17)-C(55)	105.7(3)
C(60)-O(18)-H(18)	109.5
C(62)-O(19)-H(19)	109.5

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters ($Å^2 x 10^3$) for 101129LTB. The anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2 a^{*2} U^{11} + ... + 2 h k a^* b^* U^{12}]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
C(1)	32(3)	35(3)	22(2)	-8(2)	0(2)	14(2)
C(2)	24(3)	38(3)	16(2)	4(2)	4(2)	16(2)
C(3)	22(3)	29(2)	19(2)	6(2)	2(2)	13(2)
C(4)	15(2)	22(2)	15(2)	1(2)	-1(2)	11(2)
C(5)	19(2)	28(2)	26(2)	7(2)	4(2)	10(2)
C(6)	20(3)	22(2)	31(3)	-4(2)	0(2)	8(2)
C(7)	15(2)	17(2)	20(2)	5(2)	4(2)	12(2)
C(8)	18(2)	11(2)	16(2)	4(2)	4(2)	9(2)
C(9)	13(2)	19(2)	14(2)	3(2)	2(2)	6(2)
C(10)	20(2)	17(2)	18(2)	5(2)	5(2)	10(2)
C(11)	12(2)	17(2)	22(2)	6(2)	2(2)	9(2)
C(12)	14(2)	19(2)	13(2)	3(2)	2(2)	8(2)
C(13)	13(2)	14(2)	15(2)	4(2)	2(2)	7(2)
C(14)	23(2)	18(2)	18(2)	8(2)	9(2)	15(2)
C(15)	30(3)	25(2)	18(2)	8(2)	8(2)	20(2)
C(16)	33(3)	42(3)	34(3)	14(2)	7(2)	26(2)
C(17)	29(3)	25(2)	33(3)	9(2)	13(2)	15(2)
C(18)	16(2)	31(2)	18(2)	6(2)	3(2)	13(2)
C(19)	25(3)	33(3)	24(2)	12(2)	9(2)	16(2)
C(20)	27(3)	46(3)	20(2)	8(2)	2(2)	17(2)
C(21)	28(3)	36(3)	24(3)	-1(2)	2(2)	16(2)
C(22)	34(3)	33(3)	26(2)	0(2)	6(2)	18(2)
C(23)	26(3)	41(3)	21(2)	5(2)	4(2)	21(2)
C(24)	19(2)	22(2)	27(2)	11(2)	8(2)	12(2)
C(25)	12(2)	20(2)	21(2)	4(2)	8(2)	6(2)
C(26)	19(2)	17(2)	21(2)	3(2)	4(2)	12(2)
C(27)	13(2)	16(2)	19(2)	5(2)	3(2)	8(2)
C(28)	13(2)	23(2)	25(2)	8(2)	4(2)	11(2)
C(29)	18(2)	22(2)	23(2)	8(2)	5(2)	13(2)
C(30)	11(2)	18(2)	22(2)	3(2)	2(2)	10(2)
C(31)	16(2)	19(2)	21(2)	3(2)	6(2)	12(2)
C(32)	30(3)	24(2)	24(2)	6(2)	8(2)	19(2)
C(33)	27(3)	50(3)	23(2)	4(2)	4(2)	25(2)

C(34)	40(3)	45(3)	23(2)	9(2)	7(2)	29(3)
C(35)	38(3)	26(2)	14(2)	0(2)	2(2)	11(2)
C(36)	42(3)	27(2)	14(2)	-2(2)	7(2)	17(2)
C(37)	31(3)	29(2)	22(2)	5(2)	8(2)	22(2)
C(38)	28(3)	19(2)	14(2)	3(2)	6(2)	6(2)
C(39)	28(3)	24(2)	14(2)	3(2)	2(2)	14(2)
C(40)	27(3)	30(2)	21(2)	2(2)	3(2)	16(2)
C(41)	16(2)	18(2)	19(2)	7(2)	5(2)	9(2)
C(42)	14(2)	10(2)	20(2)	2(2)	4(2)	5(2)
C(43)	12(2)	11(2)	18(2)	3(2)	3(2)	0(2)
C(44)	10(2)	12(2)	20(2)	3(2)	1(2)	5(2)
C(45)	23(2)	15(2)	20(2)	4(2)	4(2)	13(2)
C(46)	20(2)	17(2)	20(2)	1(2)	7(2)	11(2)
C(47)	15(2)	10(2)	20(2)	0(2)	3(2)	4(2)
C(48)	24(3)	19(2)	18(2)	5(2)	3(2)	4(2)
C(49)	18(2)	32(2)	20(2)	2(2)	3(2)	14(2)
C(50)	28(3)	38(3)	19(2)	-1(2)	3(2)	15(2)
C(51)	35(3)	28(3)	28(3)	1(2)	4(2)	8(2)
C(52)	33(3)	44(3)	18(2)	5(2)	3(2)	16(2)
C(53)	25(3)	39(3)	21(2)	5(2)	0(2)	16(2)
C(54)	22(3)	29(2)	18(2)	4(2)	7(2)	6(2)
C(55)	26(3)	15(2)	14(2)	2(2)	5(2)	11(2)
C(56)	19(2)	39(3)	21(2)	2(2)	5(2)	17(2)
C(57)	33(3)	46(3)	20(2)	7(2)	12(2)	16(2)
C(58)	16(2)	22(2)	19(2)	0(2)	3(2)	11(2)
C(59)	21(2)	17(2)	19(2)	2(2)	2(2)	11(2)
C(60)	10(2)	15(2)	18(2)	3(2)	1(2)	5(2)
C(61)	16(2)	19(2)	16(2)	1(2)	2(2)	11(2)
C(62)	19(2)	16(2)	21(2)	4(2)	0(2)	6(2)
C(63)	13(2)	24(2)	19(2)	7(2)	3(2)	6(2)
C(64)	13(2)	21(2)	17(2)	3(2)	3(2)	10(2)
C(65)	19(2)	26(2)	18(2)	3(2)	2(2)	13(2)
C(66)	24(3)	22(2)	17(2)	4(2)	2(2)	15(2)
C(67)	26(3)	27(2)	27(2)	6(2)	12(2)	14(2)
C(68)	34(3)	25(2)	24(2)	4(2)	9(2)	17(2)
N(1)	13(2)	21(2)	16(2)	4(1)	4(1)	8(2)
N(2)	16(2)	15(2)	21(2)	3(1)	4(2)	6(2)
N(3)	17(2)	19(2)	16(2)	3(1)	1(2)	7(2)

N(4)	18(2)	16(2)	15(2)	1(1)	3(1)	7(2)
O(1)	16(2)	27(2)	15(1)	0(1)	3(1)	14(1)
O(2)	17(2)	25(2)	15(1)	0(1)	2(1)	14(1)
O(3)	13(2)	16(1)	20(2)	5(1)	3(1)	4(1)
O(4)	18(2)	21(2)	23(2)	4(1)	2(1)	15(1)
O(5)	19(2)	22(2)	19(2)	1(1)	5(1)	5(1)
O(6)	19(2)	38(2)	21(2)	5(1)	7(1)	13(2)
O(7)	19(2)	27(2)	18(2)	6(1)	8(1)	12(1)
O(8)	25(2)	16(2)	23(2)	2(1)	7(1)	5(1)
O(9)	15(2)	14(1)	24(2)	-2(1)	6(1)	7(1)
O(10)	20(2)	14(1)	24(2)	0(1)	6(1)	6(1)
O(11)	24(2)	17(1)	14(1)	3(1)	3(1)	8(1)
O(12)	24(2)	19(2)	16(2)	5(1)	4(1)	4(1)
O(13)	18(2)	12(1)	25(2)	5(1)	6(1)	5(1)
O(14)	13(2)	15(1)	23(2)	7(1)	3(1)	4(1)
O(15)	21(2)	26(2)	23(2)	6(1)	0(1)	15(1)
O(16)	25(2)	19(2)	16(2)	2(1)	0(1)	9(1)
O(17)	23(2)	21(2)	17(2)	3(1)	-1(1)	9(1)
O(18)	18(2)	19(2)	24(2)	9(1)	-2(1)	-2(1)
O(19)	30(2)	17(2)	22(2)	6(1)	-1(1)	-5(1)
O(20)	17(2)	20(2)	19(2)	3(1)	-1(1)	-1(1)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters (Å ^{2}x 10^3) for 101129LTB.

	x	y	z	U(eq)
H(1A)	5809	10220	5113	38
H(1B)	6820	9649	4851	38
H(2A)	4760	7874	4954	31
H(2B)	3856	8291	4258	31
H(3A)	4085	6537	3484	27
H(3B)	5722	7320	3812	27
H(5A)	7001	9392	3156	29
H(5B)	6088	9809	2468	29
H(6A)	5163	10378	3613	32
H(6B)	6801	11140	3936	32
H(7)	3272	5690	1736	19
H(8A)	2950	5228	234	17
H(8B)	4560	5719	580	17
H(10A)	3482	8388	-360	21
H(10B)	2255	7073	-261	21
H(11)	4340	9018	1194	19
H(12)	2055	6827	1460	18
H(14A)	3480	5415	-2719	20
H(14B)	5075	6207	-2694	20
H(15)	3910	6890	-3725	25
H(16A)	2625	7427	-2388	49
H(16B)	1884	6267	-3261	49
H(16C)	2441	7750	-3370	49
H(17A)	4952	9125	-3194	41
H(17B)	5997	8583	-2814	41
H(17C)	5161	8957	-2171	41
H(19A)	9394	2595	3857	31
H(19B)	10178	2064	3258	31
H(20A)	12126	4063	3683	37
H(20B)	11706	3603	4578	37
H(21A)	10709	5010	4741	36
H(21B)	12250	5840	4657	36

H(22A)	11488	5828	3157	37
H(22B)	10703	6370	3748	37
H(23A)	8747	4368	3323	33
H(23B)	9171	4833	2431	33
H(24)	8074	614	1509	25
H(25)	6617	1543	707	22
H(26A)	8353	3524	683	21
H(26B)	7583	2752	-330	21
H(28A)	10011	1461	242	23
H(28B)	11274	2856	695	23
H(29)	10348	1727	1797	23
H(31A)	9614	1753	-2576	21
H(31B)	10968	3064	-2518	21
H(32)	9576	4132	-2850	28
H(33A)	7182	2886	-3372	47
H(33B)	7645	2762	-2361	47
H(33C)	7288	1598	-3186	47
H(34A)	8953	1939	-4226	49
H(34B)	10297	3310	-4028	49
H(34C)	8818	3219	-4402	49
H(35A)	5811	5140	4138	34
H(35B)	4845	3790	4374	34
H(36A)	6701	3850	5390	33
H(36B)	7100	4010	4442	33
H(37A)	8463	6232	4965	29
H(37B)	8918	5497	5673	29
H(39A)	6829	7253	5397	26
H(39B)	6428	7103	6346	26
H(40A)	5059	4846	5823	30
H(40B)	4586	5573	5117	30
H(41)	10617	8148	7608	20
H(42)	8141	8275	7881	18
H(43A)	9209	8879	9476	19
H(43B)	10386	8510	9261	19
H(45A)	9723	6028	8735	21
H(45B)	8126	5081	8340	21
H(46)	9516	5918	7251	22
H(48A)	8735	6253	11585	27

H(48B)	7126	5794	11467	27
H(49)	7553	7985	11908	27
H(50A)	7456	6660	12996	44
H(50B)	8413	8171	13441	44
H(50C)	9090	7232	13206	44
H(51A)	10395	8620	12221	51
H(51B)	9813	9634	12472	51
H(51C)	9603	9017	11438	51
H(52A)	1372	800	3975	39
H(52B)	1225	-501	4311	39
H(53A)	-157	252	4950	35
H(53B)	1122	1661	5353	35
H(54A)	768	495	6522	30
H(54B)	827	-703	5927	30
H(56A)	3211	-314	5593	30
H(56B)	4475	1099	6013	30
H(57A)	3583	879	4448	39
H(57B)	3490	2055	5048	39
H(58)	5044	1140	7489	22
H(59A)	5487	1822	9020	22
H(59B)	4320	347	8761	22
H(61A)	2314	2127	8356	19
H(61B)	3118	3204	9269	19
H(62)	3915	4048	8044	24
H(63)	5263	3106	7337	23
H(65A)	5884	2696	11766	24
H(65B)	5042	1273	11949	24
H(66)	4971	2846	13011	23
H(67A)	2288	1244	11878	38
H(67B)	2593	1923	12911	38
H(67C)	3030	798	12656	38
H(68A)	3601	3531	11581	39
H(68B)	5134	4439	12165	39
H(68C)	3869	4108	12631	39
H(1N)	5404	7356	-1292	19
H(2N)	10700	3937	-1202	21
H(3N)	7187	6800	10228	22
H(4N)	3405	819	10662	20

H(3)	5720	8536	-17	26
H(4)	1693	8417	685	28
H(8)	7348	550	-545	35
H(9)	10187	4791	464	27
H(13)	9570	10322	8325	29
H(14)	6595	5882	8662	27
H(18)	1815	318	8780	37
H(19)	5754	3834	9251	44