Electronic Supplementary Material (ESI) for Organic & Biomolecular Chemistry

Supporting information

Organocatalyzed stereospecific C-C bond formation of β-lactams

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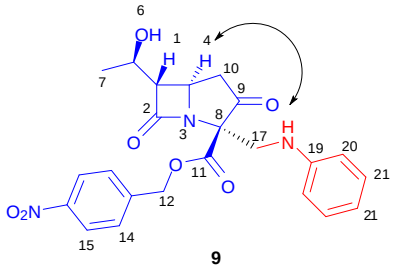
Contents:

1. NMR

The observed NOE interaction between H_4 and H_{18} in Mannich compound **9** that confirms the absolute configuration at C_8 is presented in Figure **1**. This long range interaction (black arrow) supports the presence of similar configuration at C_8 like Aldol reaction (See Figure **1**).



9

Figure 1. Long range interaction in compound **9**

2. Experimental

Reagents and solvents were purchased from Sigma Aldrich and Fluka Chemicals. All NMR spectra were recorded on Bruker AVANCE III 400 MHz instrument. The chemical shifts are expressed in ppm downfield from TMS as the internal standard and the coupling constants are reported in Hertz. Thin layer chromatography was performed using Merck Kieselgel 60 F254. Compounds were purified by column chromatography packed with 60-200 mesh Silica gel and Shimadzu C18 column prep HPLC. Optical rotations were recorded on a Perkin-Elmer Polarimeter (Model 341). High resolution spectrometric data were obtained using Bruker maxis 4G instrument operating at ambient temperatures.

1a. General Procedure for Aldol reaction

A mixture of compound **1** (1.0 eq) and aldehyde (10.0 eq) was stirred in the presence of catalyst (0.2 eq) and acetic acid (0.2 eq) at room temperature. Pyrrolidine, L-proline and D-proline were used in turns as catalysts in the reactions for all the substrates that were tested. The reaction progress was monitored using TLC and LCMS, on completion the reactions were quenched using water and extracted with DCM (30 mL x 3). The extracts were dried with Mg_2SO_4, which was subsequently removed by filtration. The solvent was removed under reduced pressure, and the crude product mixture was purified by column chromatography. The structure was confirmed using NMR and Mass.

(*2S,5R,6S*)-4-nitrobenzyl 6-((*R*)-1-hydroxyethyl)-2-(hydroxymethyl)-3,7-dioxo-1-azabicyclo [3.2.0]heptane-2-carboxylate (2)

The crude product was purified by column chromatography (ethyl acetate/hexane, 70:30; R_f = 0.3) to afford the product (76%) as a colorless oil. $[\alpha]_{20}^{D}$ = -26.6 (*c* = 0.1, $CHCl_3$) ^{1}HNMR (400 MHz, DMSO): δ 8.26 (d, *J* = 8.56 Hz, 2H), 7.69 (d, *J* = 8.56 Hz, 2H), 5.86 (s, 1H), 5.37 (s, 1H), 5.32 (dd, *J* = 9.37 Hz, 2H), 4.48 – 4.49 (m, 1H), 4.17 (p, 1H), 2.95 – 3.04 (m, 2H), 2.58 (q, *J* = 16.87, *J* = 9.62, 1H), 1.33 (d, *J* = 6.28 Hz, 3H); ^{13}C NMR (100 MHz DMSO): δ 171.9, 166.1, 161.9, 148.1, 143.8, 131.6, 128.3, 124.3, 114.1, 65.5, 63.5, 62.6, 52.9, 37.4, 21.6; HRMS (ESI-) *m/z* calcd. for $C_{17}H_{18}N_2O_8$: 377.0979; found [M-H] 377.1391

(*2S,5R,6S*)-4-nitrobenzyl 2-((*R*)-hydroxy(phenyl)methyl)-6-((*R*)-1-hydroxyethyl)-3,7-dioxo-1-azabicyclo[3.2.0]heptane-2-carboxylate (3)

The crude product was purified by column chromatography (ethyl acetate /hexane, 70:30; R_f = 0.2) to afford the product (60%) as a semisolid. $[\alpha]_{20}^{D}$ = +20.0 (*c* = 0.1, $CHCl_3$) ^{1}HNMR (600 MHz, DMSO): δ 8.25 (d, *J* = 8.52 Hz, 2H), 7.74 (d, *J* =8.52 Hz, 2H), 7.69 (m, 2H), 7.53 (s, 1H), 7.42 (m, 3H),, 5.41 (s, 2H), 4.35 (m, 1H), 3.96 (m, 1H), 3.02 (d.d, *J* = 2.58, 6.3 Hz, 1H), 2.61 (m, 2H), 1.13 (d, *J* = 8.52 Hz, 3H); ^{13}C NMR (150 MHz DMSO): δ 172.5, 167.2, 163.3, 147.2,143.6, 136.4, 132.3,130.3, 128.6, 128.6,

128.3, 122.6, 65.5, 63.9, 62.3, 53.5, 38.0, 22.3; HRMS (ESI-) *m/z* calcd. for $C_{23}H_{22}N_2O_8$: 453.1292; found [M-H] 453.1694

1b. General Procedure for Mannich reaction

To a stirred solution of formaldehyde (2.0 eq, 36% aqueous solution) in DMSO (3 mL), substituted amine (2.0 eq) was added at ambient temperature. After 2 h, the compound **1** (1.0 eq) and catalyst (0.3 eq) were added and the reaction mixture was stirred for 20 h while being monitored using TLC. The reaction mixture was then quenched by addition of PBS buffer (1 mL), water (3 mL) and the aqueous phase was extracted three times with EtOAc. The combined organic layers were dried with Mg_2SO_4, which was subsequently removed by filtration. Next, the solvent was removed under reduced pressure, and the crude product mixture was purified by column chromatography.

(2*S*,5*R*,6*S*)-4-nitrobenzyl 6-((*R*)-1-hydroxyethyl)-3,7-dioxo-2-((phenylamino)methyl)-1-azabicyclo [3.2.0]heptane-2-carboxylate (9)

The crude product was purified by column chromatography (EtOAc/hexane, 80:20; R_f = 0.4) to afford the product (55%) as a semisolid. $[\alpha]_{20}^{D}$ = -30.0 (*c* = 0.1, $CHCl_3$) ^{1}H NMR (400 MHz, $CDCl_3$):δ = 8.21 (d, *J* = 8.64 Hz, 2 H), 7.70 (s, 1 H), 7.51 (d, *J* = 8.60 Hz, 2H), 7.45 (d, *J* = 7.88 Hz, 2 H), 7.30 (t, *J* = 7.82 Hz, 2 H), 7.12 (t, *J* = 7.38 Hz, 1 H), 6.14 (s, 1 H), 6.04 (s, 1 H), 5.29 (d, *J* = 9.72 Hz, 2 H), 4.60 (m, 1 H), 4.17 (m, 1 H), 3.06 (m, 1 H), 3.04 (m, 1 H), 2.66 (d.d, *J* = 15.84 and 9.52 Hz, 1 H), 1.37 (d, *J* = 6.16 Hz, 3 H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ = 168.3, 165.5, 162.1, 148.0, 142.2, 137.2, 130.8, 129.2, 128.6, 125.1, 124.0, 120.2, 115.7, 66.9, 66.0, 64.8, 55.7, 40.2, 21.6 ppm. HRMS (ESI+) *m/z* calcd. for $C_{23}H_{23}N_3O_7$: 454.1608; found [M+H] 454.2985

(*2S,5R,6S*)-4-nitrobenzyl 6-((*R*)-1-hydroxyethyl)-2-((4-methoxyphenylamino)methyl)-3,7-dioxo-1-azabicyclo[3.2.0]heptane-2-carboxylate (10)

The crude product was purified by column chromatography (EtOAc/hexane, 80:20; R_f = 0.4) to afford the product (52%) as a semisolid. $[\alpha]_{20}^{D}$ = -50.0 (*c* = 0.1, $CHCl_3$) ^{1}H NMR (400 MHz, DMSO):δ = 9.87 (s, 1 H), 8.20 (d, *J* = 8.72 Hz, 2 H), 7.66 (d, *J* = 8.72 Hz, 2 H), 7.40 (d, *J* = 9.00 Hz, 2 H), 6.83 (d, *J* = 9.00 Hz, 2H), 5.83 (d, *J* = 9.96 Hz, 2 H), 5.31 (s, 2 H), 5.03 (d, *J* = 4.40 Hz, 2 H)), 4.53-4.49 (m, 1 H), 3.97 (q, *J* = 16.24, and 5.44 Hz, 1 H) 1 H), 3.69 (s, 3 H), 3.11 (dd, *J* = 5.30 and 2.38 Hz, 1 H), 2.77-2.69 (m, 2H), 1.10 (d, *J* = 6.32 Hz, 3 H) ppm. ^{13}C NMR (100 MHz, DMSO): δ = 167.5, 166.0, 161.8, 155.2, 147.1, 143.2,

131.9, 131.5, 128.5, 123.5, 120.7, 113.7, 113.5, 65.4, 63.4, 62.7, 55.0, 40.1, 21.6 ppm. HRMS (ESI+) *m/z* calcd. for $C_{24}H_{25}N_3O_8$: 484.1714; found [M+H] 484.1712

(*2S,5R,6S*)-4-nitrobenzyl 2-((4-bromophenylamino)methyl)-6-((*R*)-1-hydroxyethyl)-3,7-dioxo-1-azabicyclo[3.2.0]heptane-2-carboxylate (11)

The crude product was purified by column chromatography (EtOAc/hexane, 80:20; R_f = 0.4) to afford the product (50%) as a semisolid. $[\alpha]_{20}^{D}$ = -70.0 (*c* = 0.1, $CHCl_3$) ^{1}H NMR (400 MHz, $CDCl_3$):δ = 8.23 (d, *J* = 8.52 Hz, 2 H), 7.78 (s, 1 H), 7.52 (d, *J* = 8.52 Hz, 2 H),7.41-7.35(m, 4H), 6.17 (s,1H), 6.06 (s,1H), 5.29 (q, *J* = 19.74 and 47.06 Hz, 2 H), 4.61-4.59 (m, 1 H), 4.20-4.16 (m, 1 H), 3.06 (dd, *J* = 19.26 and 11.34 Hz, 1 H), 2.68-2.64 (m, 2H), 1.37 (d, *J* = 6.12 Hz, 3 H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ = 168.2, 165.4, 162.1, 148.0, 142.1, 136.3, 132.2, 130.7, 128.6, 124.1, 121.7, 117.7, 115.8, 66.9, 64.7, 55.6, 40.4, 21.6 ppm. HRMS (ESI-) *m/z* calcd. for $C_{23}H_{22}BrN_3O_7$: 530.0557; found [M-H] 530.0612

1c. General Procedure for Michael reaction

To a stirred solution of compound **1** (1.0 eq) and catalyst (0.2 eq) in DMSO (0.5 mL) at room temperature, was added nitrostyrene or cyclopentenone (2.0 eq). The mixture was stirred at ambient temperature for 24 h while being monitored by TLC. The reaction mixture was then quenched by adding water (5 mL) and the aqueous layer was extracted three times with DCM (30 mL). The combined organic layers were dried with Mg_2SO_4 which was subsequently removed by filtration. The concentrated extract was subjected to silica gel for purification to afford the desired product.

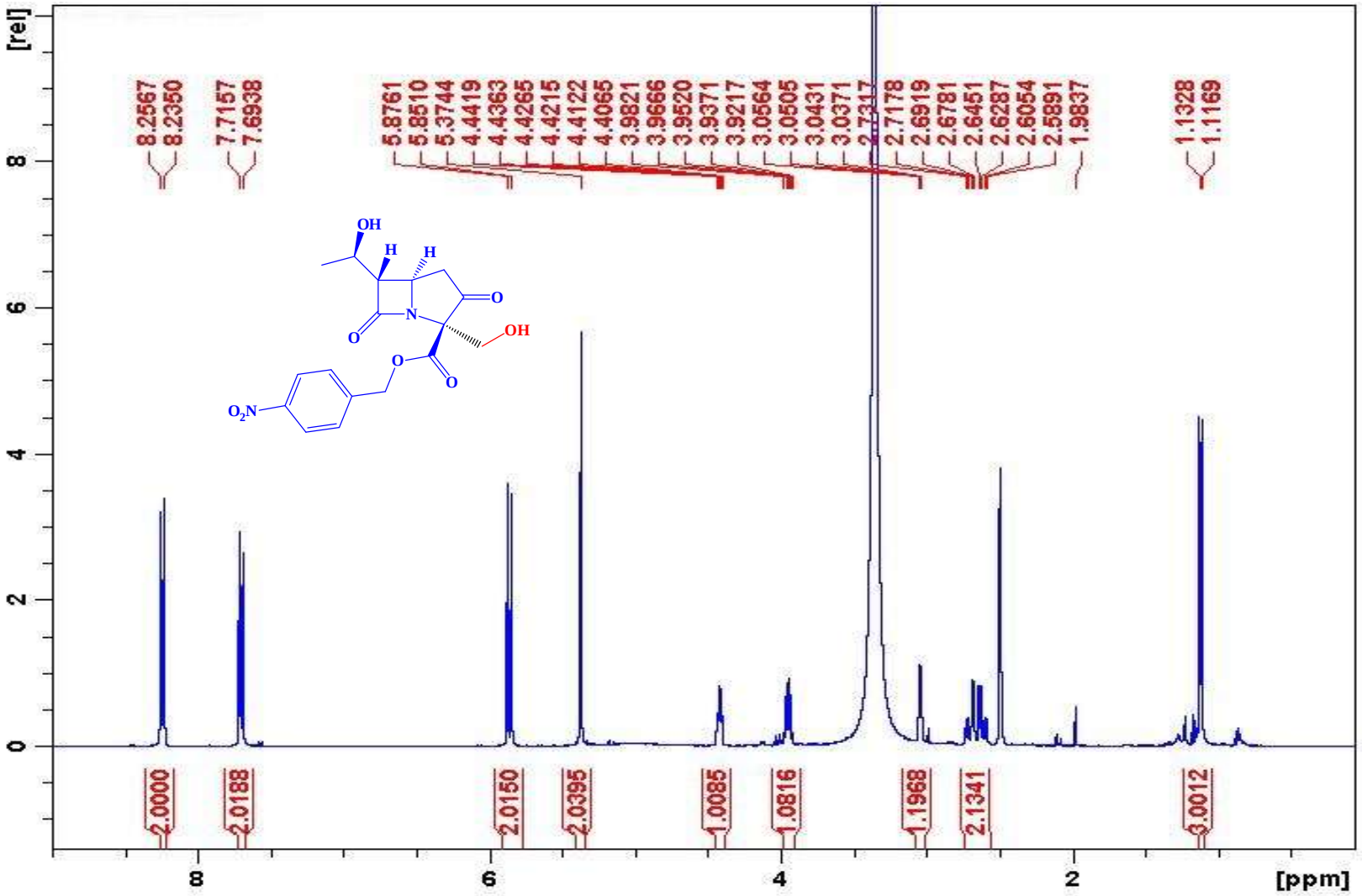
(*2S,5R,6S*)-4-nitrobenzyl 6-((*R*)-1-hydroxyethyl)-2-((*S*)-1-(4-methoxyphenyl)-2-nitroethyl)-3,7-dioxo-1-azabicyclo [3.2.0] heptane-2-carboxylate (12)

The crude product was purified by column chromatography (EtOAc/hexane, 50:50; R_f = 0.2) to afford the product (41%) as a Pale yellow powder. $[\alpha]_{20}^{D}$ = +156.7 (*c* = 0.1, $CHCl_3$) ^{1}HNMR (400 MHz, $CDCl_3$): δ 8.24 (d, *J* = 8.60 Hz, 2H), 7.48 (d, *J* = 8.60 Hz, 2H), 7.21 (d, *J* = 8.68 Hz, 2H), 6.82 (d, *J* = 8.72 Hz, 2H), 5.32 (m, 2H), 5.00 (dd, *J* = 13.17, 11.17 Hz, 2H), 4.36 (dd, *J* = 11.11, 4.10 Hz, 1H), 4.10 (m, 1H), 3.56 (m, 1H), 3.12 (dd, *J* = 5.20, 2.56 Hz, 1H), 2.43 (dd, *J* = 8.80, 8.72 Hz, 1H), 2.27 (dd, *J* = 6.88, 6.96 Hz, 1Hz) 1.29 (d, *J* = 6.28 Hz, 3H) ppm. ^{13}C NMR (100 MHz $CDCl_3$): δ 207.3, 171.5, 164.9, 160.9, 141.7, 131.7, 129.1, 124.1, 114.5, 94.5, 74.3, 67.3, 67.1, 55.4, 55.0, 51.1, 44.1, 40.5, 21.2 ppm. HRMS (ESI+) *m/z* calcd. for $C_{25}H_{25}N_3O_{10}$: 528.1612; found [M+H] 528.3154

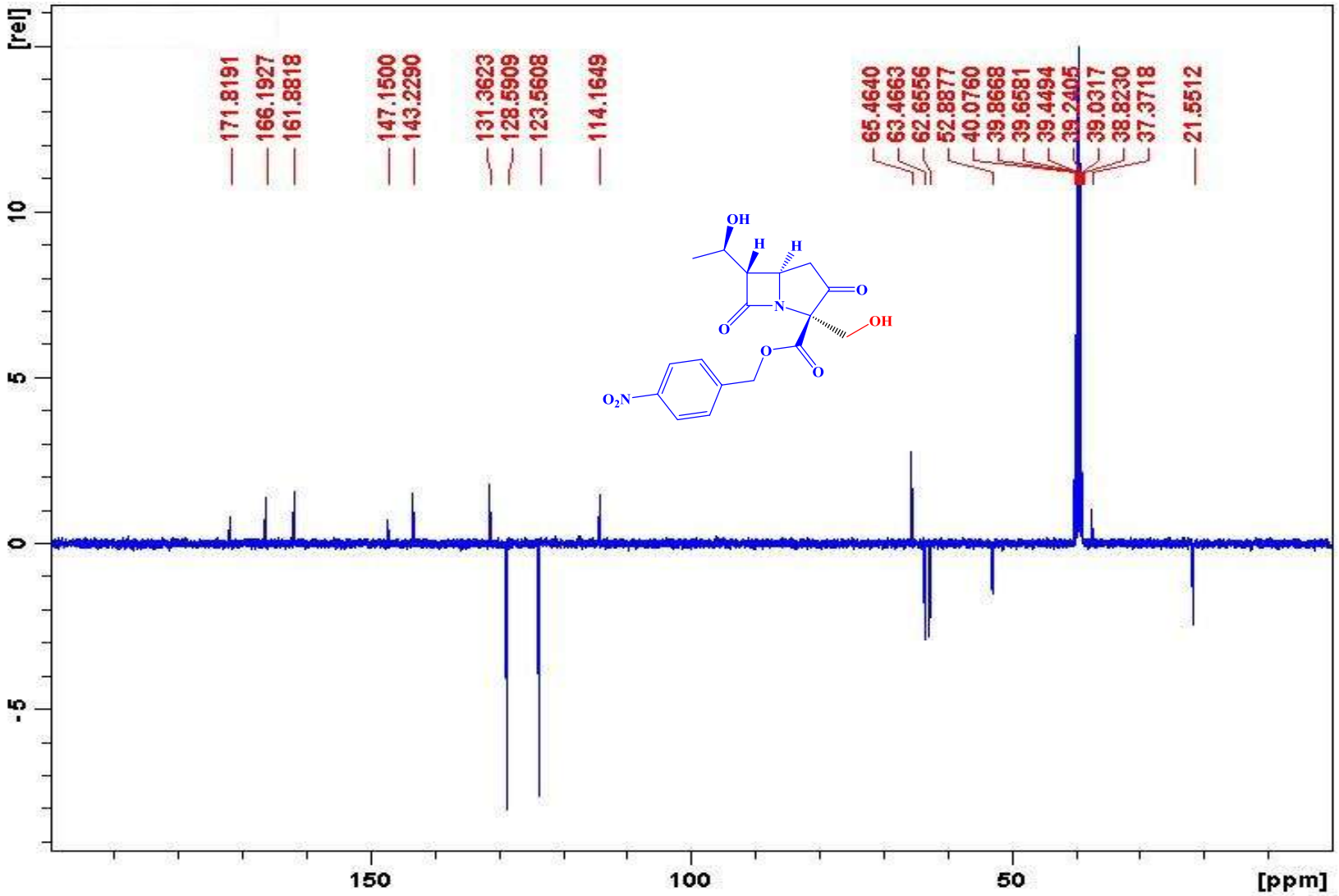
(*2S,5R,6S*)-4-nitrobenzyl 6-((*R*)-1-hydroxyethyl)-3,7-dioxo-2-((*R*)-3-oxocyclopentyl)-1-azabicyclo[3.2.0]heptane-2-carboxylate (13)

The crude product was purified by column chromatography (EtOAc/hexane, 50:50; R_f = 0.2) to afford the product (67%) as a White powder. $[\alpha]_{20}^{D}$ = +215.0 (*c* = 0.1, $CHCl_3$) ^{1}HNMR (400 MHz, $CDCl_3$): δ 8.22 (d, *J* = 7.16 Hz, 2H), 7.45 (d, *J* = 8.56 Hz, 2H), 5.27 (s,2H), 4.25 (s, 1H), 4.03 (s, 1H), 3.29 (t, 1H), 3.17 (d, 1H), 2.97- 2.95 (d, *J* = 11.05 Hz, 1H), 2.55- 2.39 (dd, *J* = 17.91, 8.58 Hz, 2H), 2.19 (d, *J* = 9.49 Hz, 2H), 2.04 (d, *J* = 7.08 Hz, 1H), 1.80 (d, *J* = 19.61 Hz, 1H), 1.34 (d, *J* = 4.80 Hz, 3H) ppm. ^{13}C NMR (100 MHz $CDCl_3$): δ 215.3, 208.0, 164.9, 161.0, 140.8, 128.7, 124.5, 76.6, 66.5, 66.2, 64.9, 50.2, 42.1, 40.9, 38.4, 38.0, 23.6, 21.9 1 ppm. HRMS (ESI-) *m/z* calcd. for $C_{21}H_{22}N_2O_8$: 429.1292; found [M-H] 429.1442

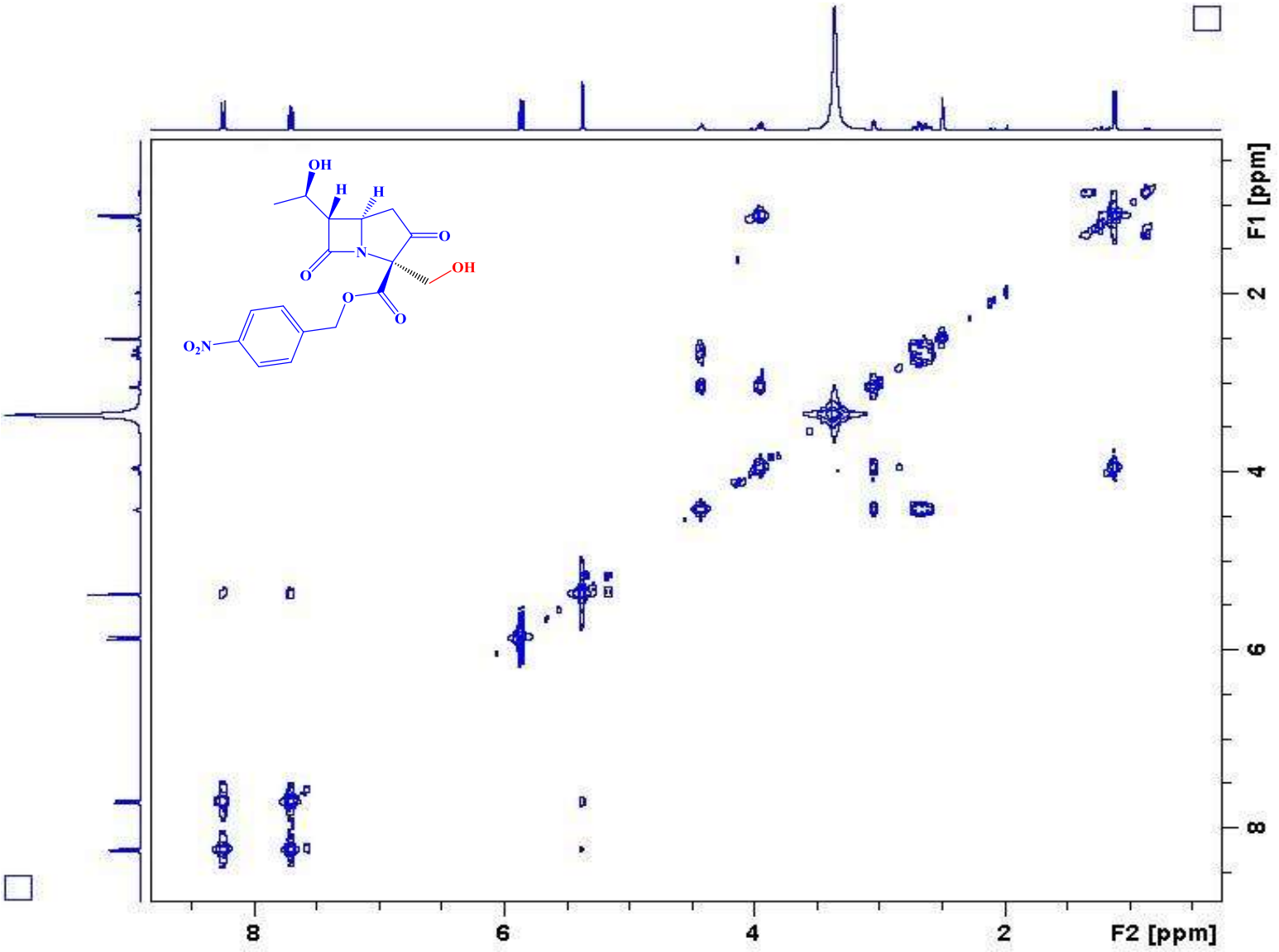
^{1}H-NMR spectra of ((*2S*)-4-nitrobenzyl-6-((*R*)-1-hydroxyerthyl)-2-(hydroxymethyl)-3,7-dioxo-1-azabicyclo[3.2.0]heptane-2-carboxylate)



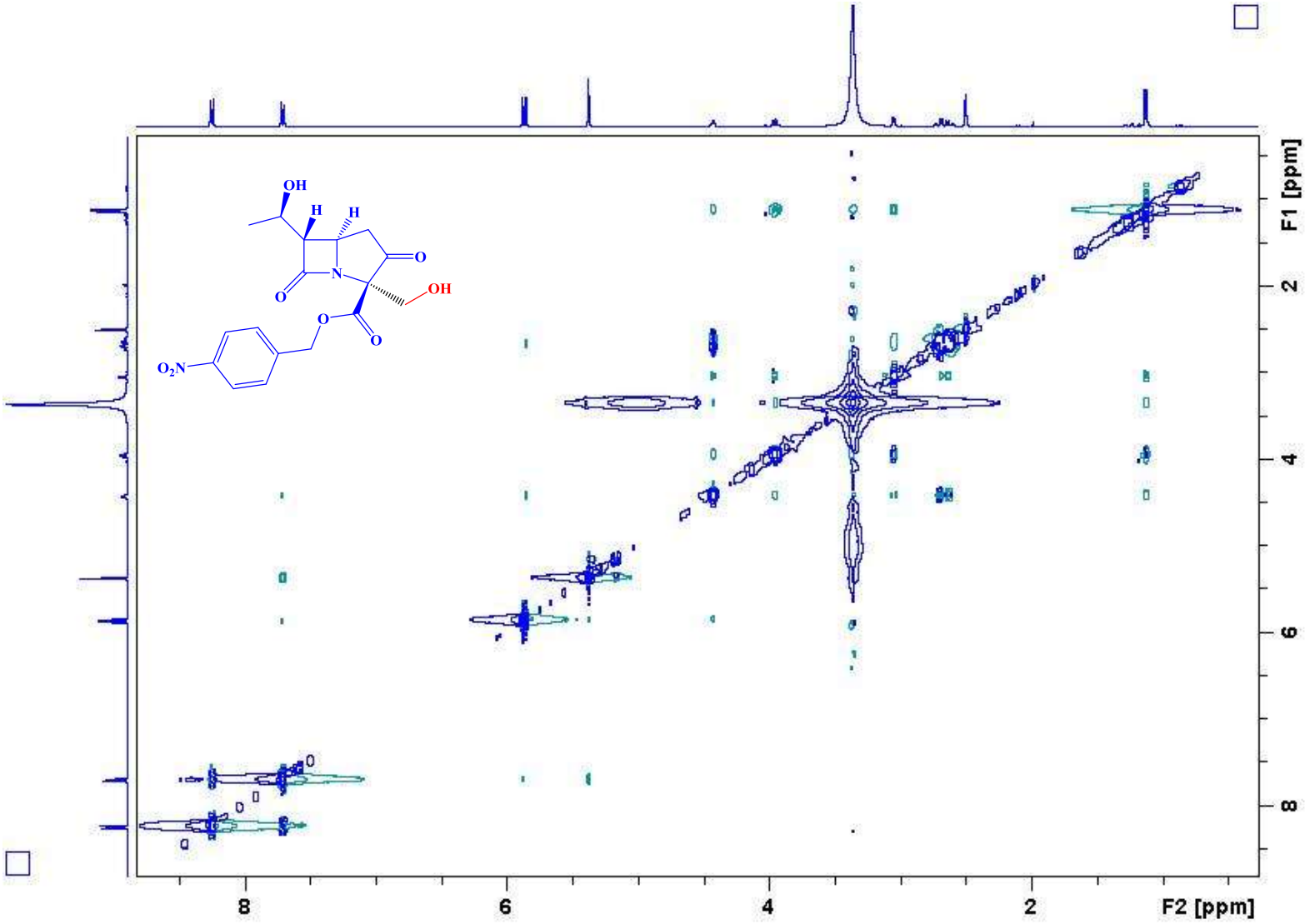
^{13}C-NMR spectra of ((*2S*)-4-nitrobenzyl-6-((*R*)-1-hydroxyerthyl)-2-(hydroxymethyl)-3,7-dioxo-1-azabicyclo[3.2.0]heptane-2-carboxylate



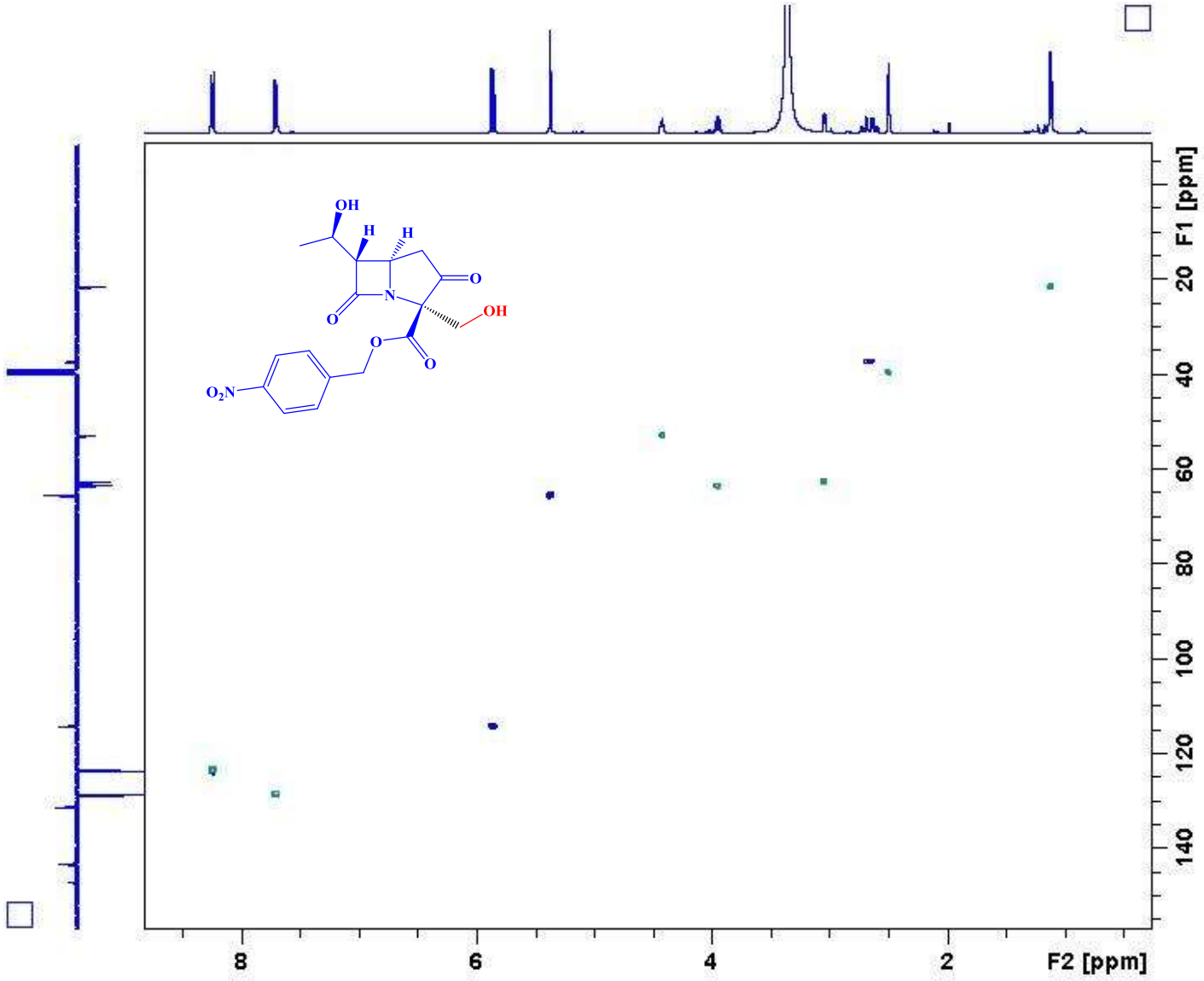
COSY of ((*2S*)-4-nitrobenzyl-6-((*R*)-1-hydroxyerthyl)-2-(hydroxymethyl)-3,7-dioxo-1-azabicyclo[3.2.0]heptane-2-carboxylate



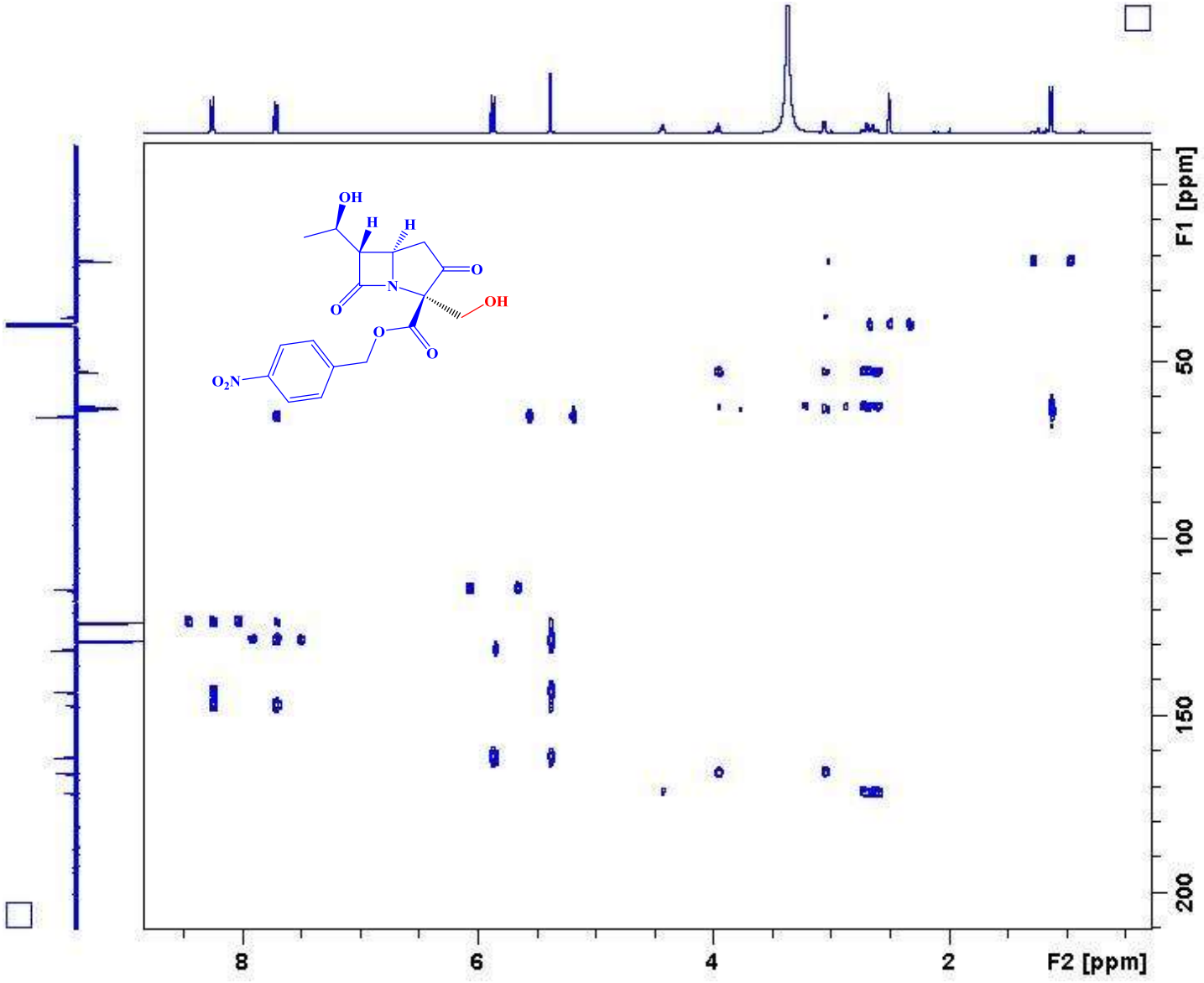
NOESY of ((*2S*)-4-nitrobenzyl-6-((*R*)-1-hydroxyerthyl)-2-(hydroxymethyl)-3,7-dioxo-1-azabicyclo[3.2.0]heptane-2-carboxylate



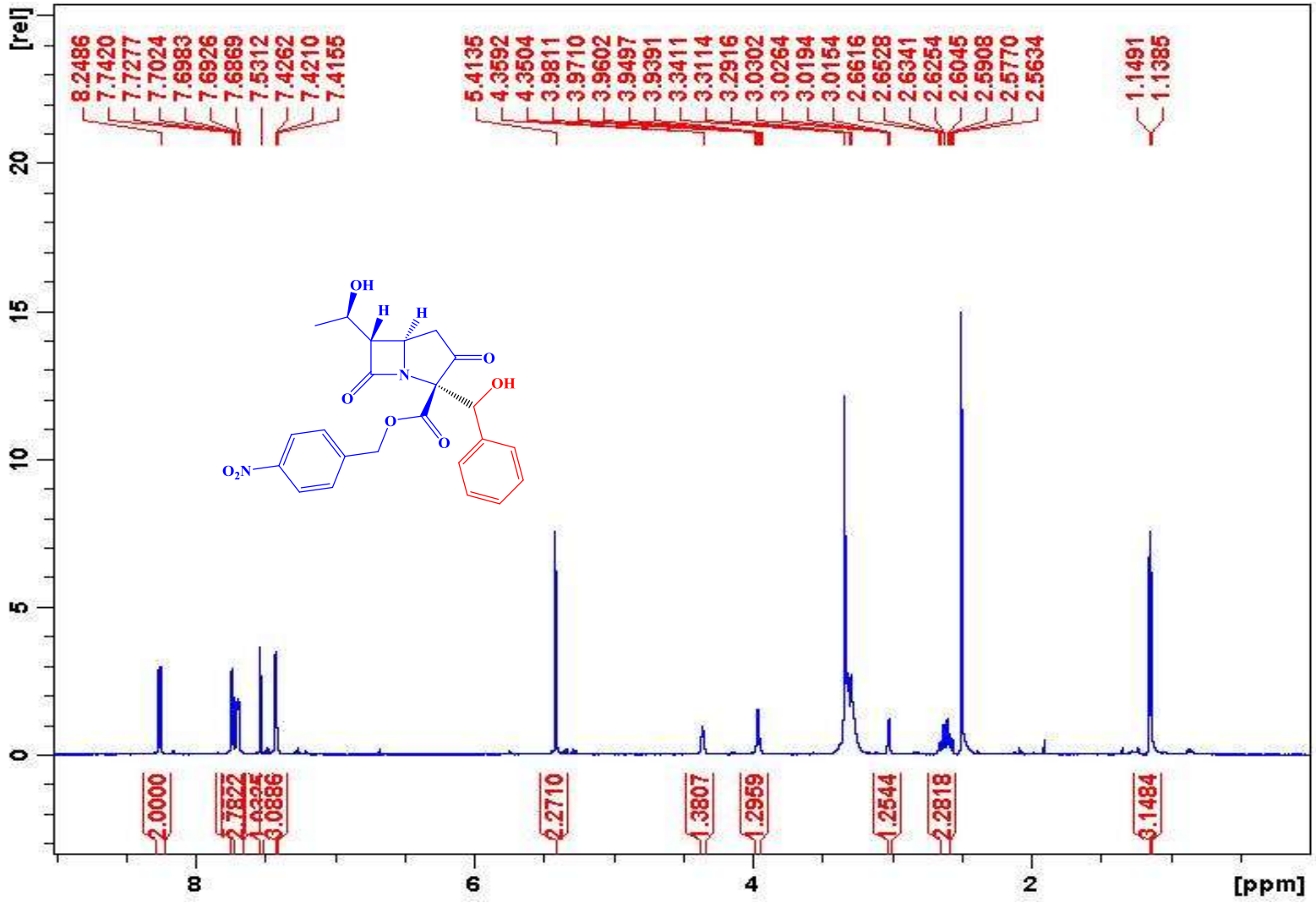
HSQC of ((*2S*)-4-nitrobenzyl-6-((*R*)-1-hydroxyerthyl)-2-(hydroxymethyl)-3,7-dioxo-1-azabicyclo[3.2.0]heptane-2-carboxylate



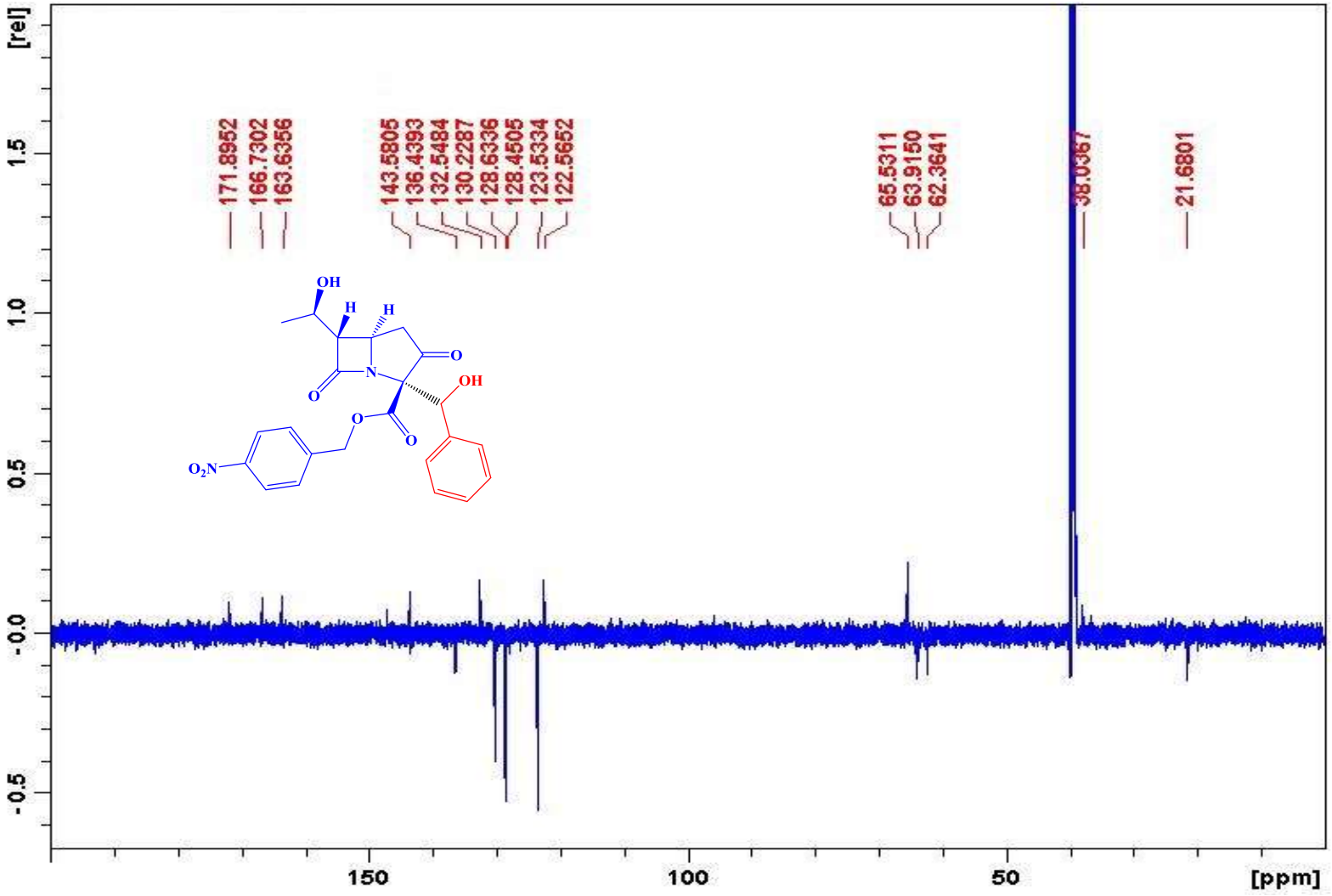
HMBC of ((*2S*)-4-nitrobenzyl-6-((*R*)-1-hydroxyerthyl)-2-(hydroxymethyl)-3,7-dioxo-1-azabicyclo[3.2.0]heptane-2-carboxylate



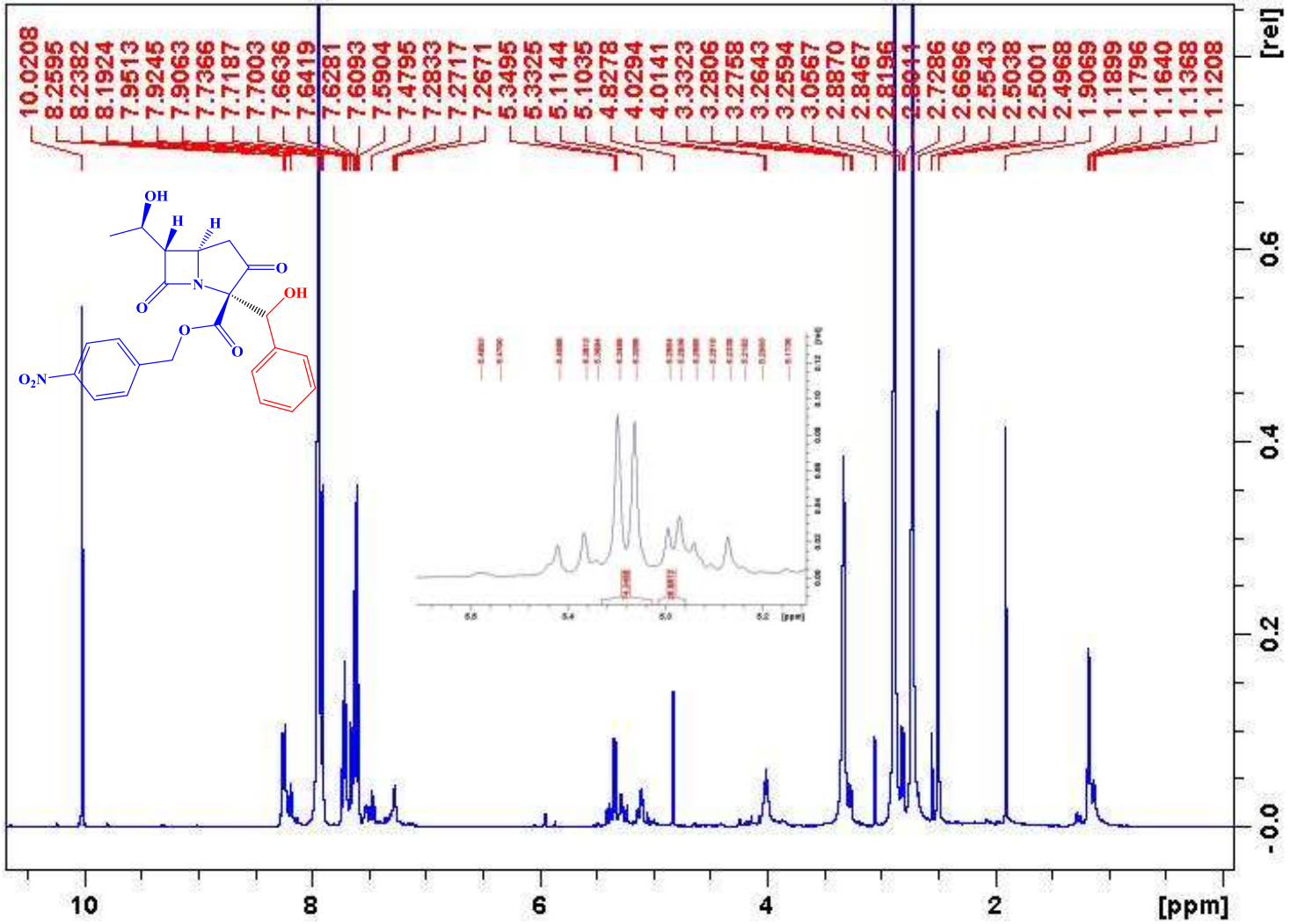
^{1}H-NMR spectra of (*2S*)-nitrobenzyl2-((*R*)-hydroxy(phenyl)methyl)-6-(1-hydroxyethyl)-3,7-dioxo-1-azabicyclo[3.2.0]heptane-2-carboxylate



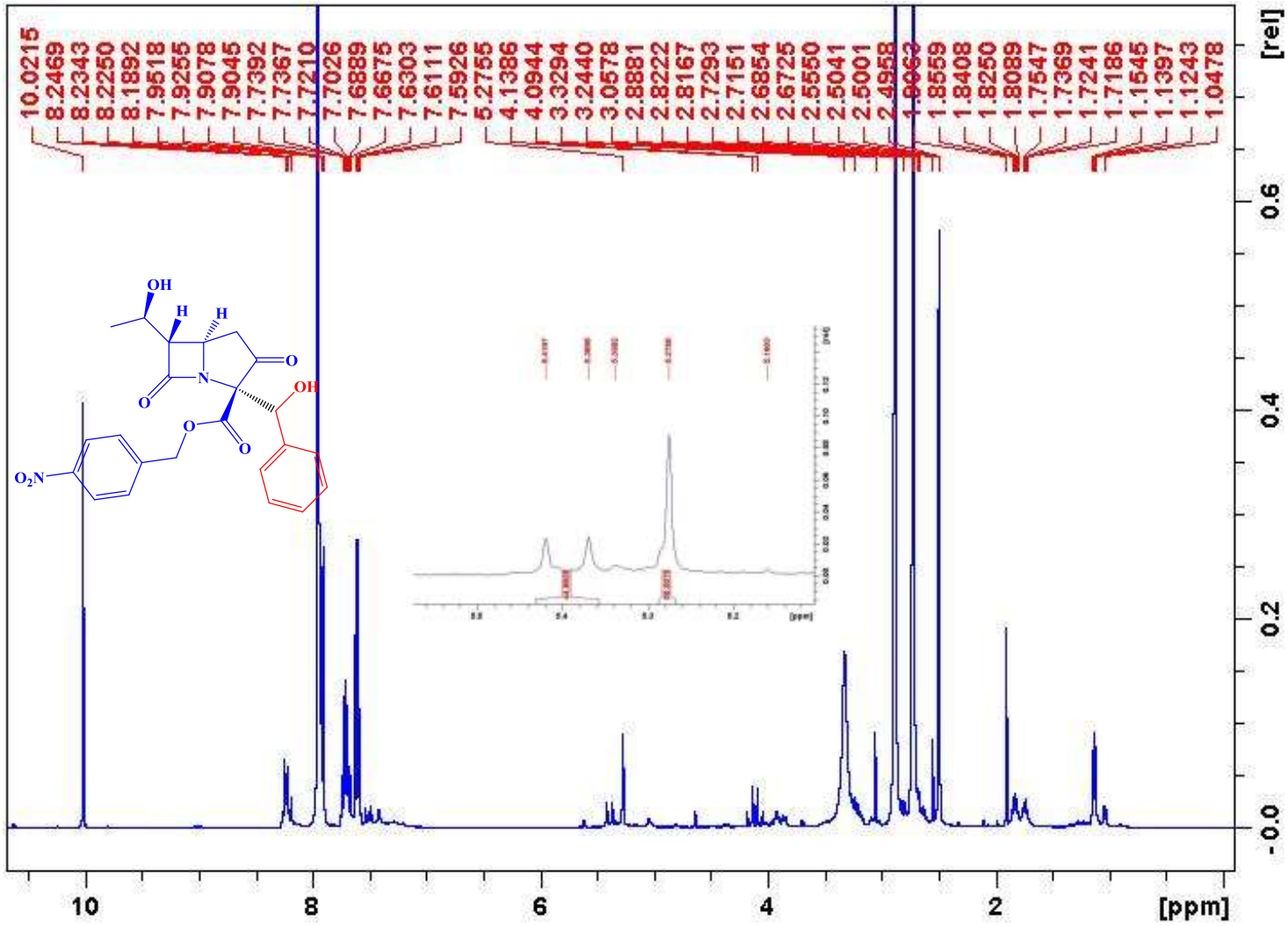
^{13}C-NMR spectra of (*2S*)-nitrobenzyl2-((*R*)-hydroxy(phenyl)methyl)-6-(1-hydroxyethyl)-3,7-dioxo-1-azabicyclo[3.2.0]heptane-2-carboxylate



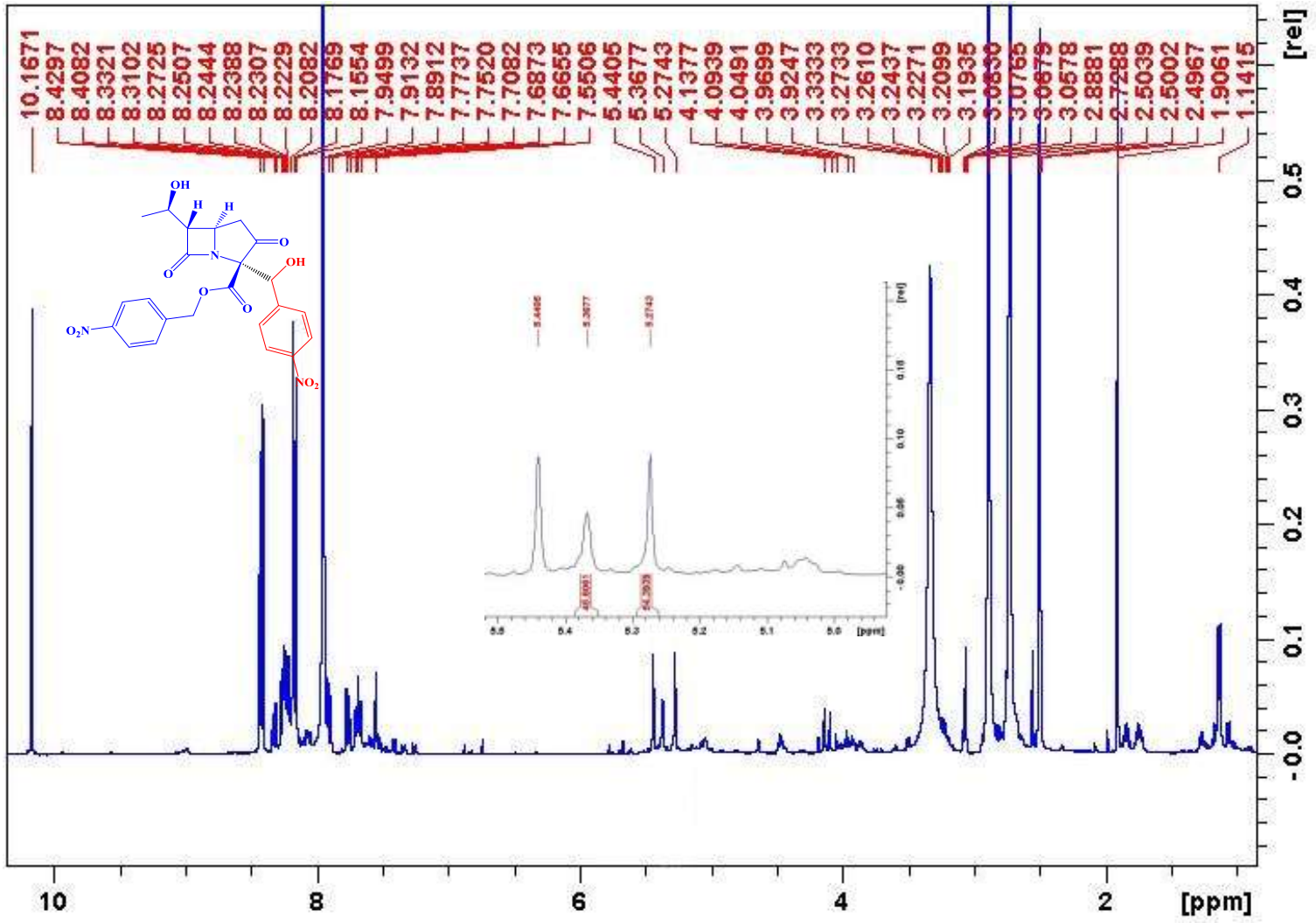
Crude ^{1}H-NMR spectra of (2S)-nitrobenzyl2-((R)-hydroxy(phenyl)methyl)-6-(1-hydroxyethyl)-3,7-dioxo-1-azabicyclo[3.2.0]heptane-2-carboxylate



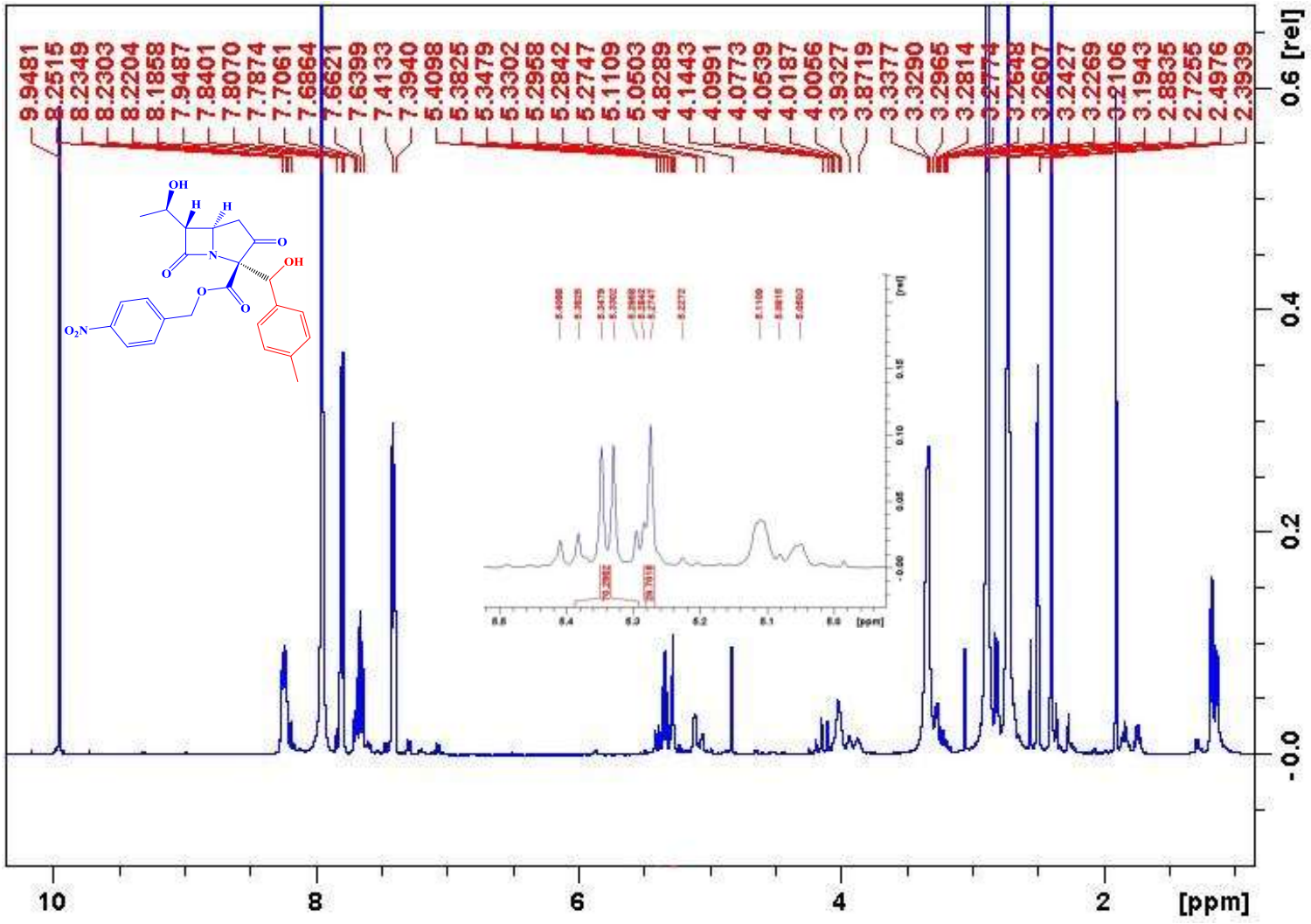
Crude ^{1}H-NMR spectra of (2S)-nitrobenzyl2-((R)-hydroxy(phenyl)methyl)-6-(1-hydroxyethyl)-3,7-dioxo-1-azabicyclo[3.2.0]heptane-2-carboxylate



Crude ^{1}H-NMR spectra of 4-nitrobenzyl (*2S,5R,6S*)-2-((*S*)-hydroxy(4-nitrophenyl)methyl)-6-((*R*)-1-hydroxyethyl)-3,7-dioxo-1-azabicyclo[3.2.0]heptane-2-carboxylate



Crude ^{1}H-NMR spectra of 4-nitrobenzyl (*2S,5R,6S*)-2-((*S*)-hydroxy(p-tolyl)methyl)-6-((*R*)-1-hydroxyethyl)-3,7-dioxo-1-azabicyclo[3.2.0]heptane-2-carboxylate



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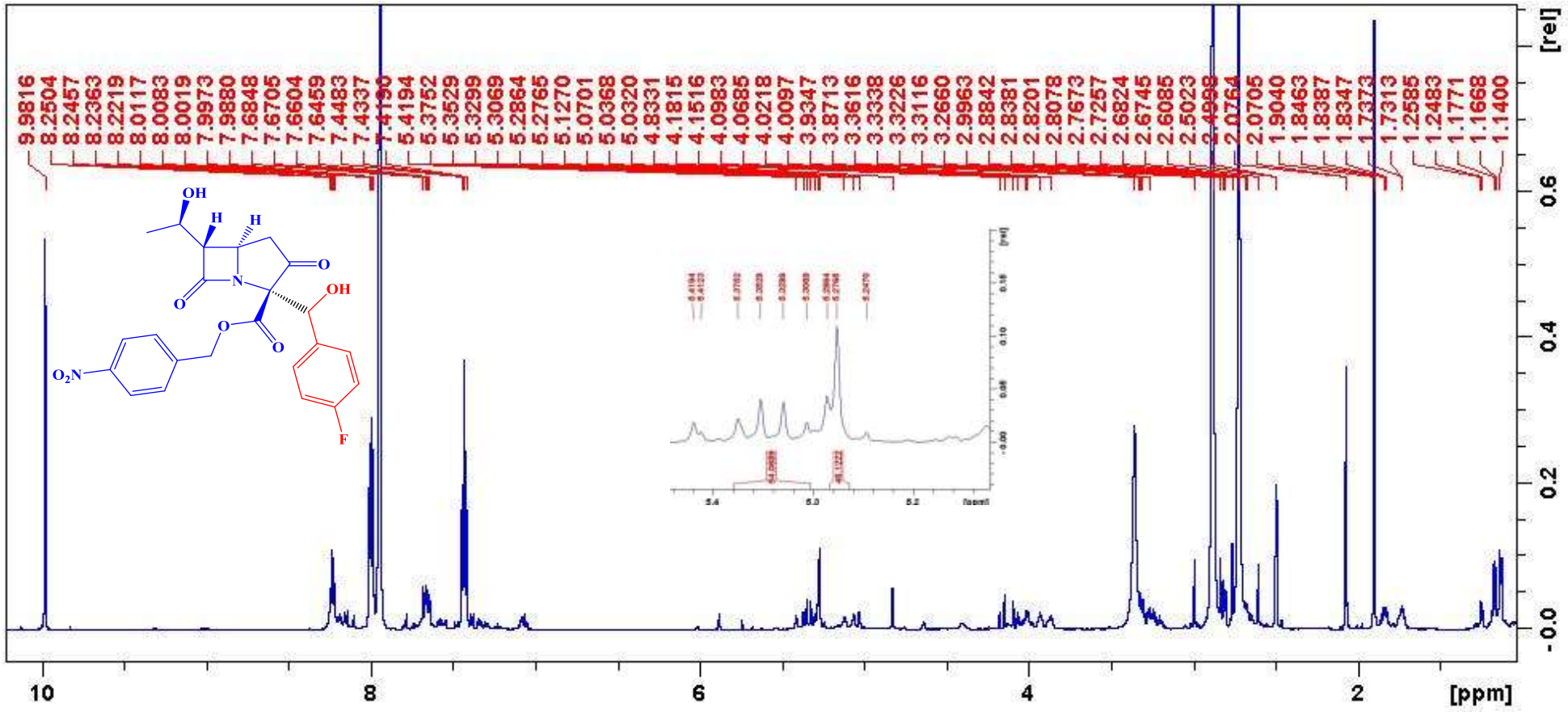
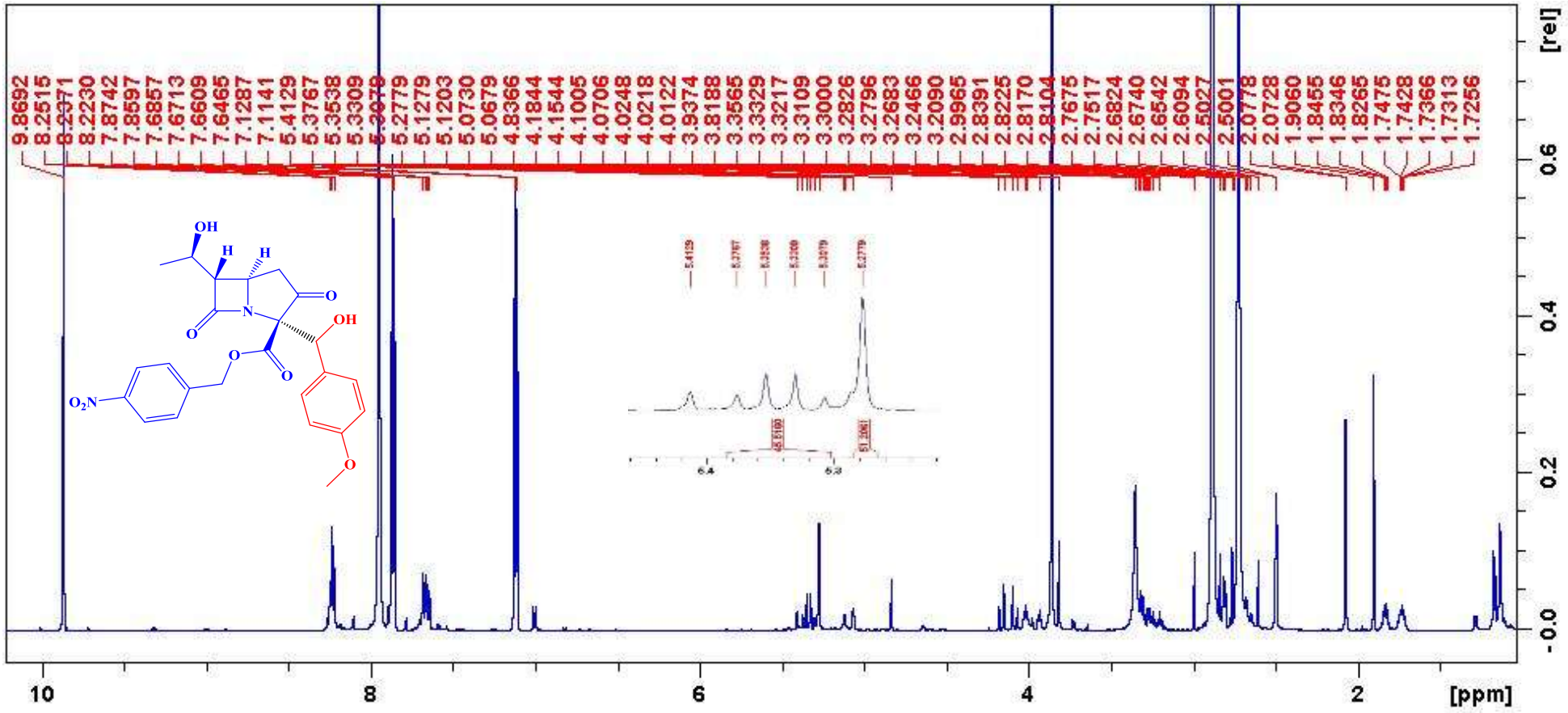
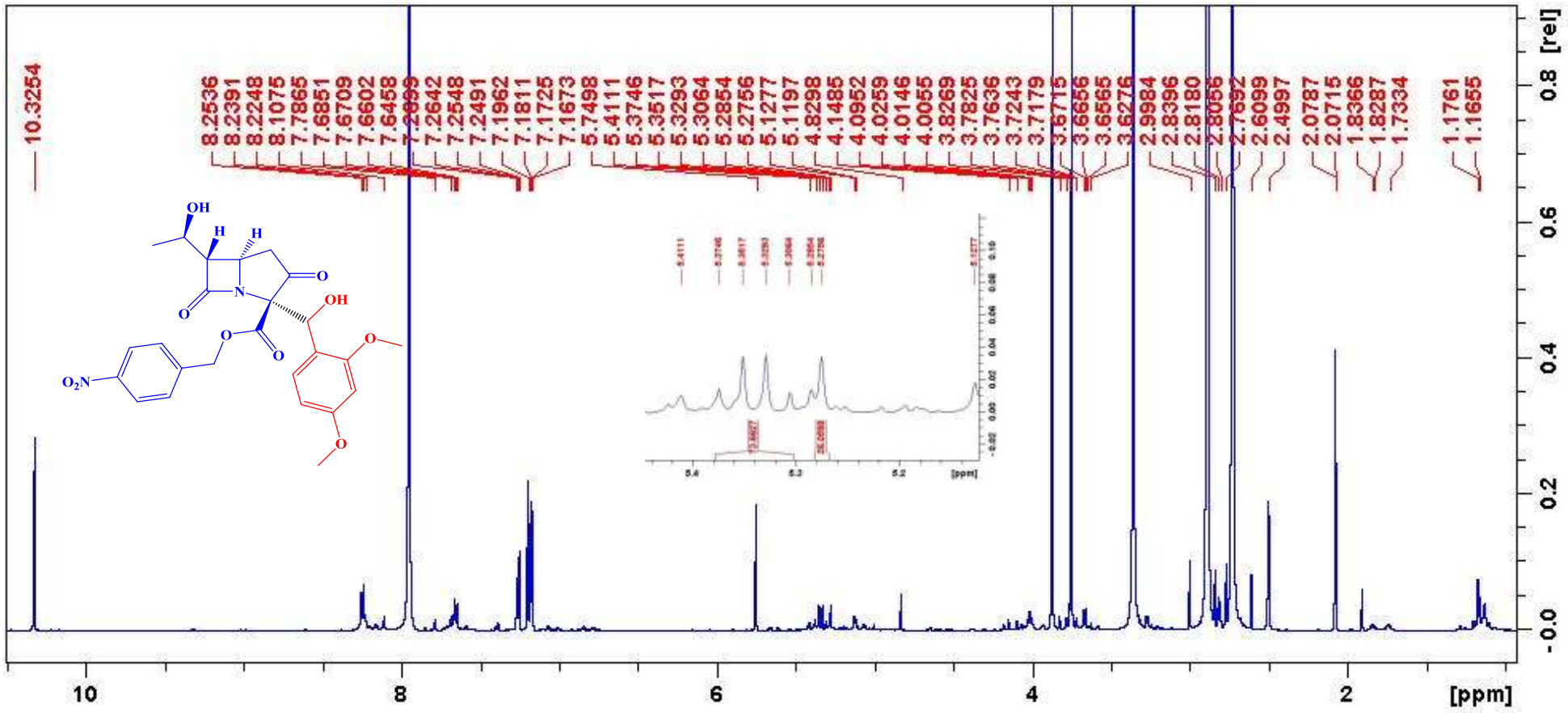


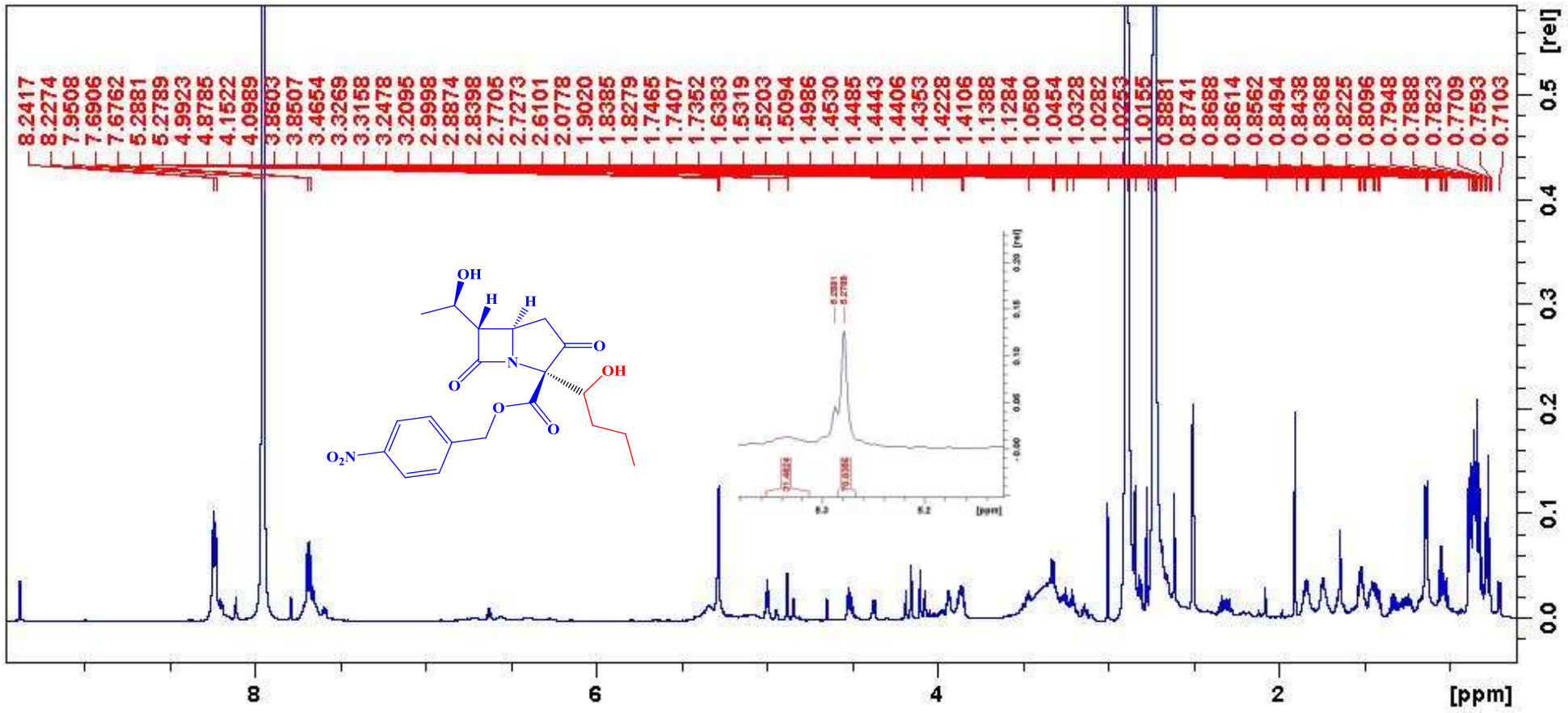
Crude ^{1}H-NMR spectra of 4-nitrobenzyl (*2S,5R,6S*)-2-((*S*)-hydroxy(4-methoxyphenyl)methyl)-6-((*R*)-1-hydroxyethyl)-3,7-dioxo-1-azabicyclo[3.2.0]heptane-2-carboxylate



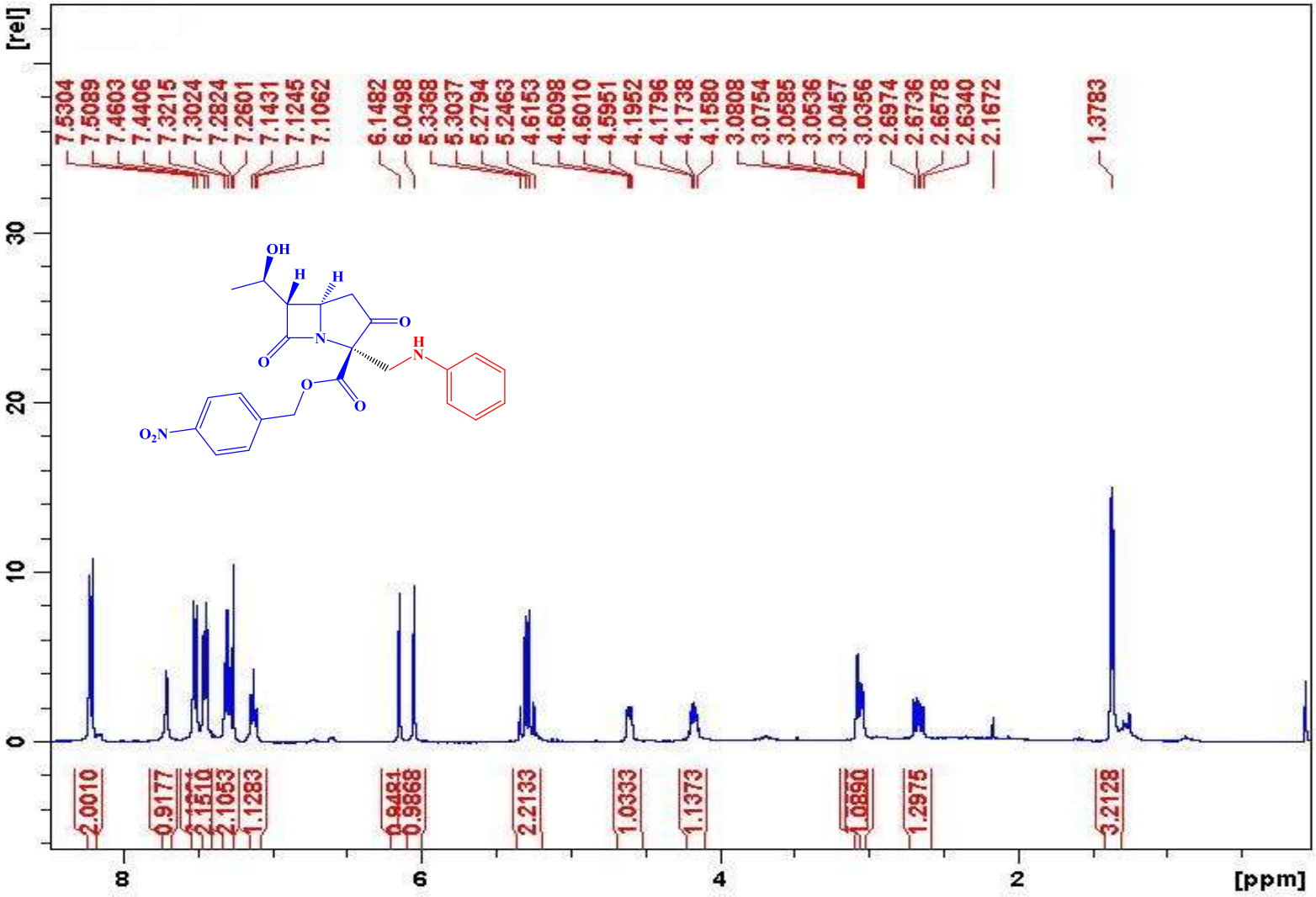
Crude ^{1}H-NMR spectra of 4-nitrobenzyl (*2S,5R,6S*)-2-((*S*)-(2,4-dimethoxyphenyl)(hydroxy)methyl)-6-((*R*)-1-hydroxyethyl)-3,7-dioxo-1-azabicyclo[3.2.0]heptane-2-carboxylate



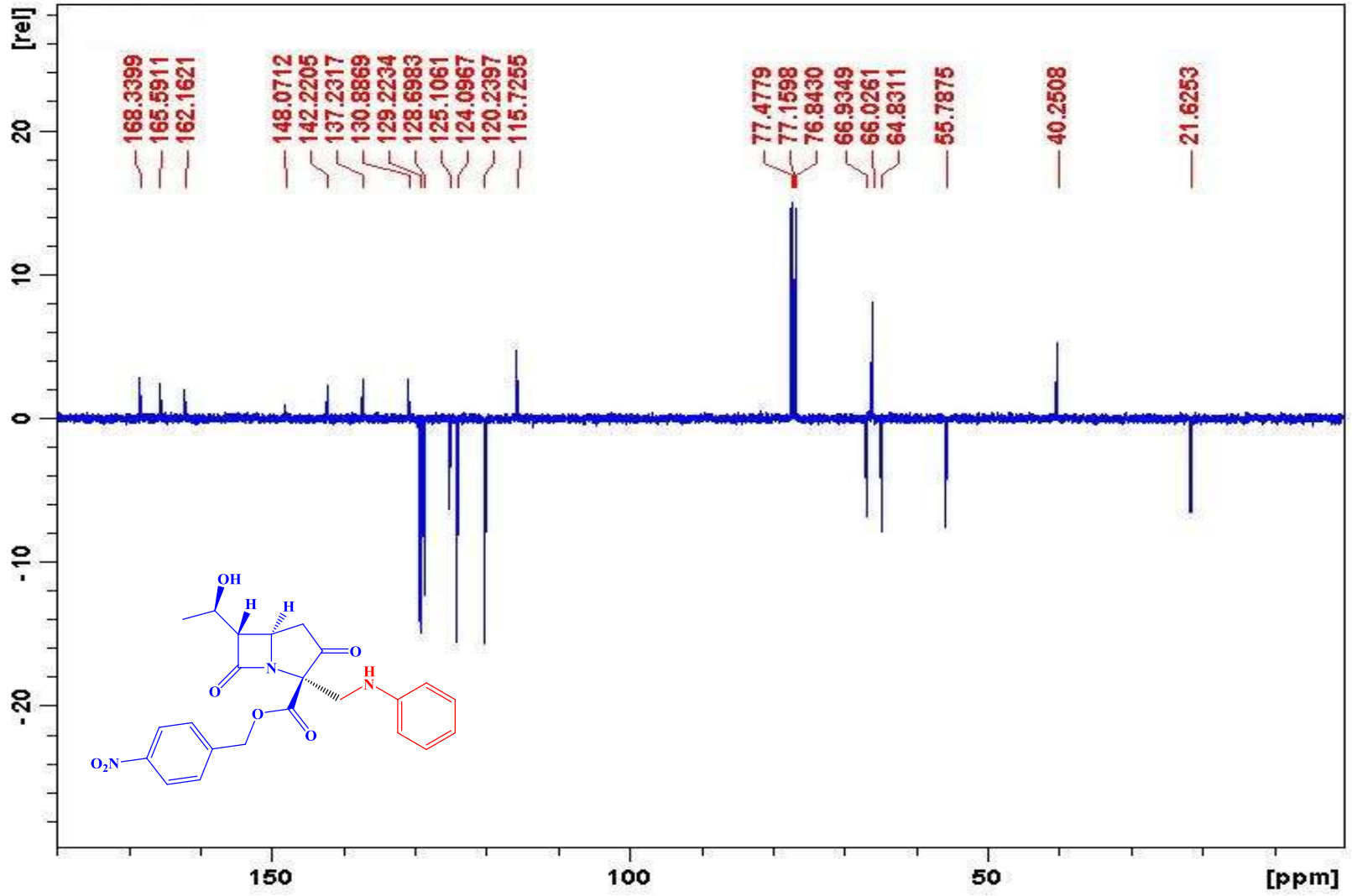
Crude ^{1}H-NMR spectra of 4-nitrobenzyl (*2S,5R,6S*)-2-((*S*)-1-hydroxybutyl)-6-((*R*)-1-hydroxyethyl)-3,7-dioxo-1-azabicyclo[3.2.0]heptane-2-carboxylate



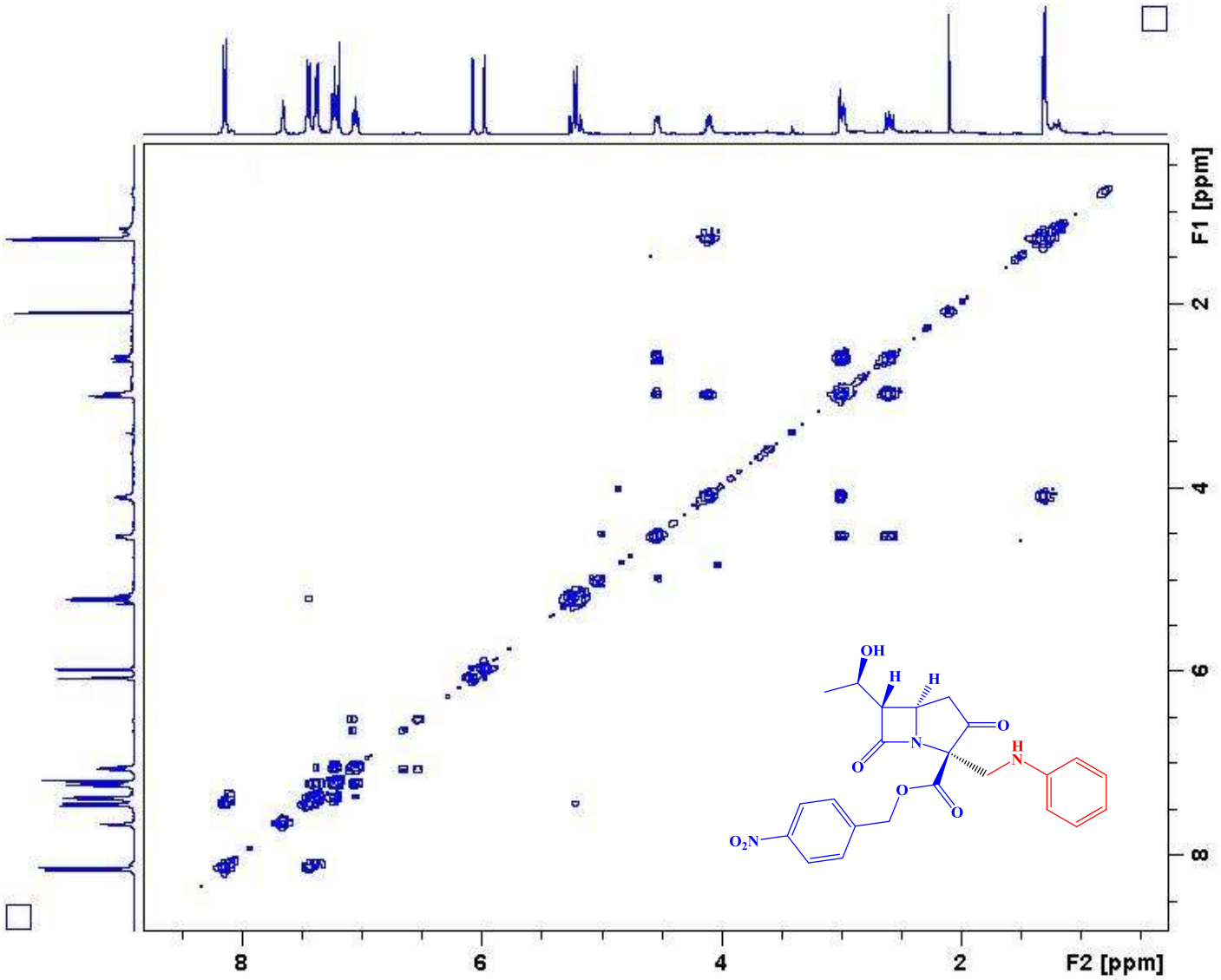
^{1}H-NMR spectra of (2*S*,5*R*,6*S*)-4-nitrobenzyl 6-((*R*)-1-hydroxyethyl)-3,7-dioxo-2-((phenylamino)methyl)-1-azabicyclo[3.2.0]heptane-2-carboxylate



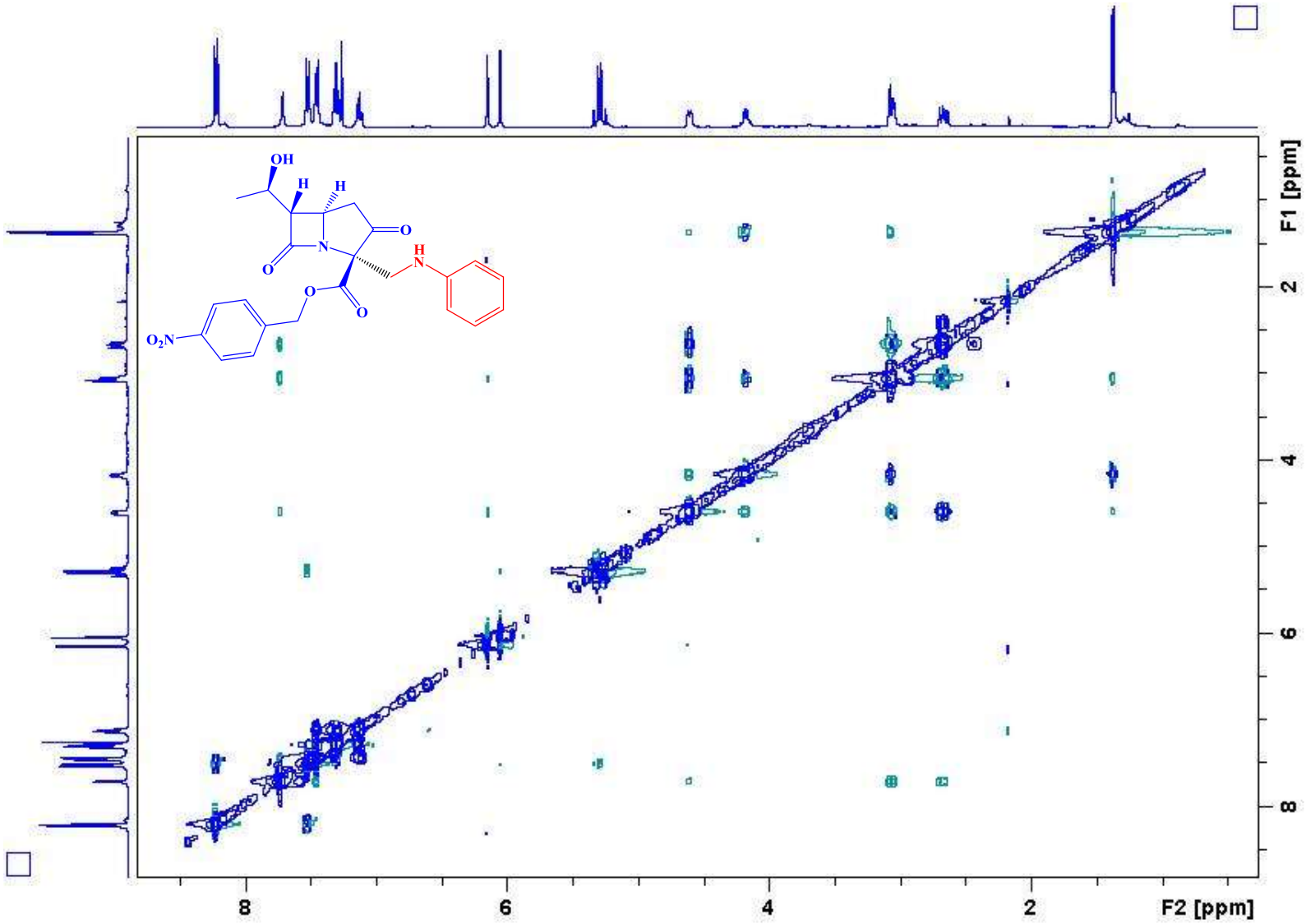
^{13}C-NMR spectra of (2*S*,5*R*,6*S*)-4-nitrobenzyl 6-((*R*)-1-hydroxyethyl)-3,7-dioxo-2-((phenylamino)methyl)-1-azabicyclo[3.2.0]heptane-2-carboxylate



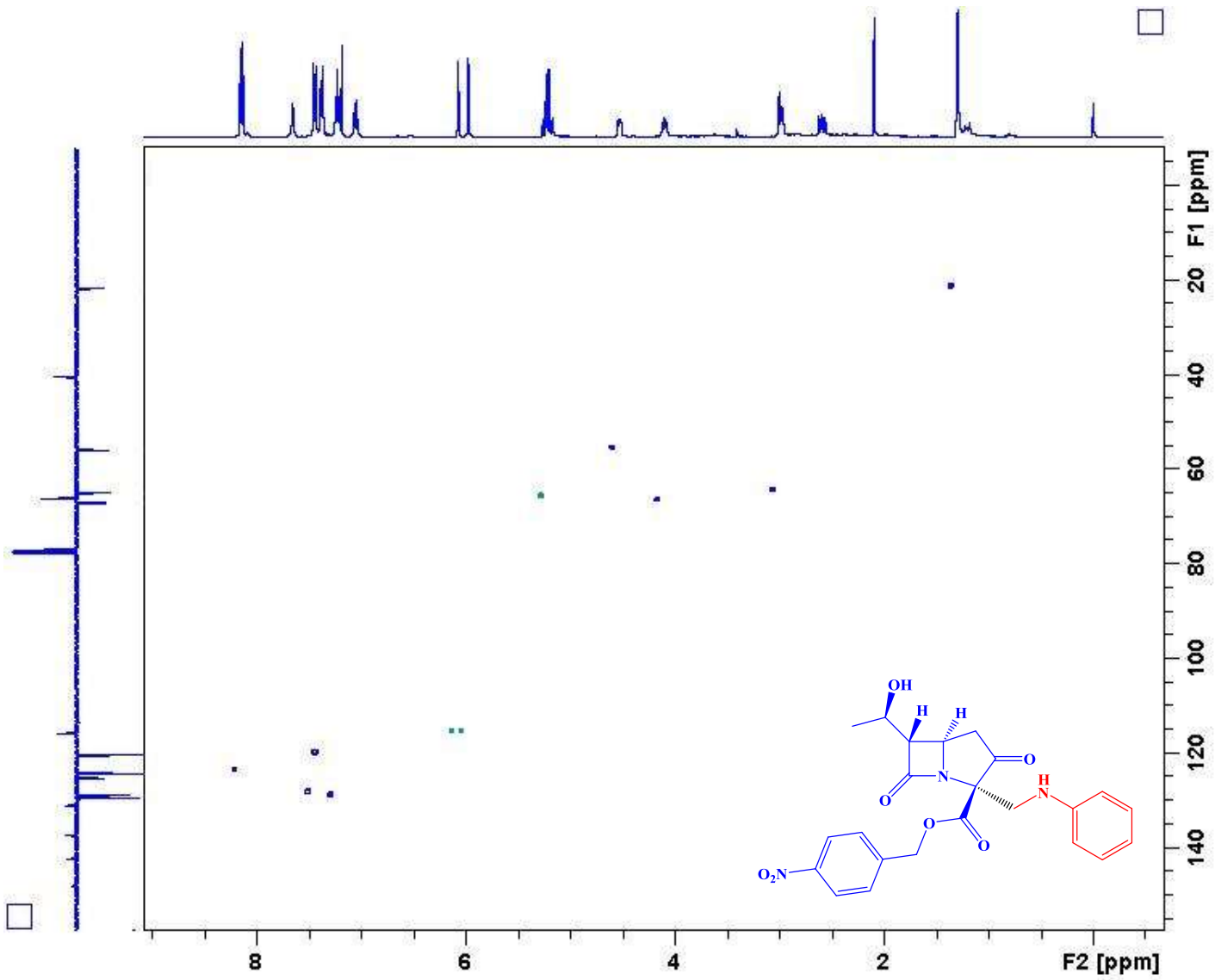
COSY of (2*S*,5*R*,6*S*)-4-nitrobenzyl 6-((*R*)-1-hydroxyethyl)-3,7-dioxo-2-((phenylamino)methyl)-1-azabicyclo[3.2.0]heptane-2-carboxylate



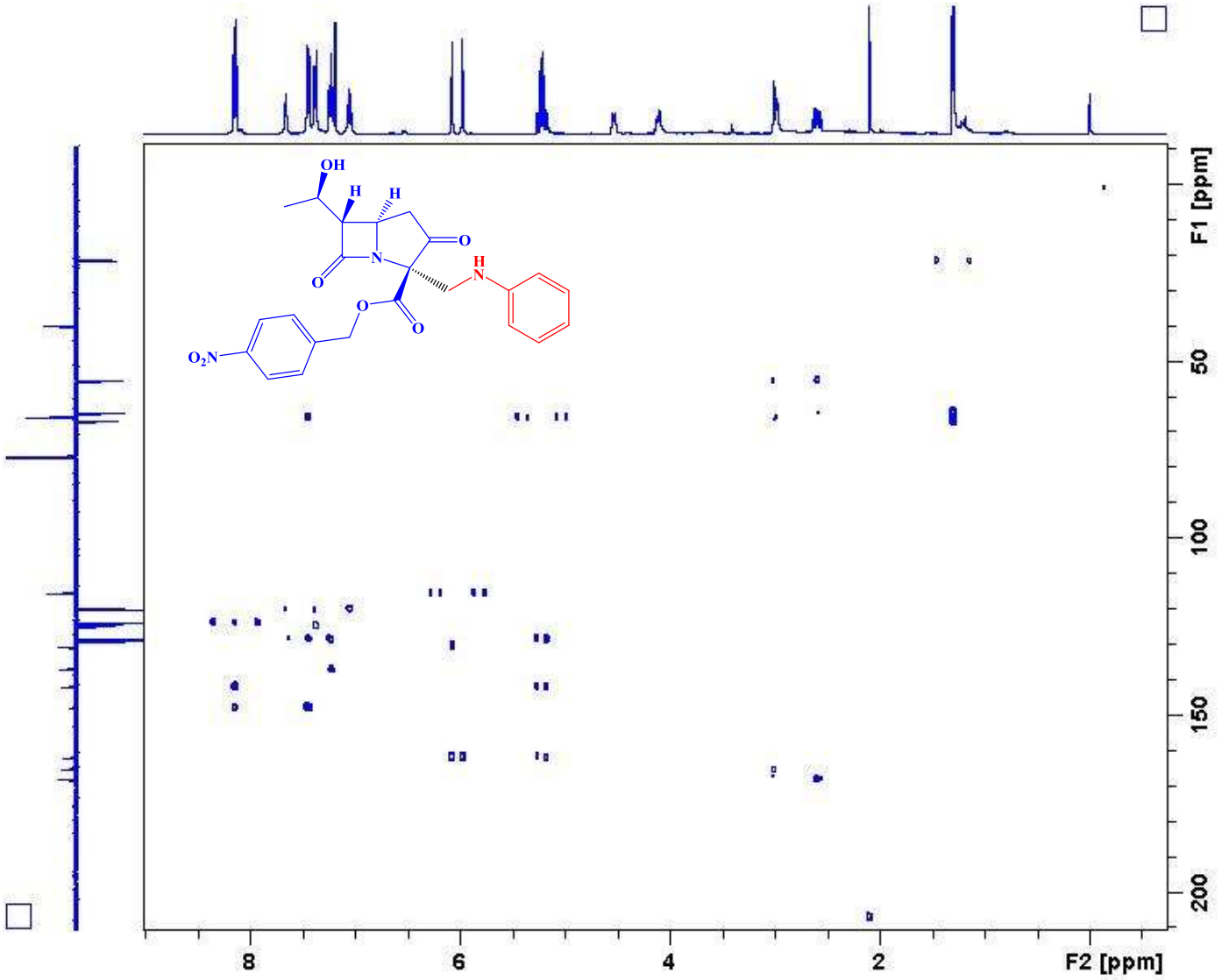
NOESY of (2*S*,5*R*,6*S*)-4-nitrobenzyl 6-((*R*)-1-hydroxyethyl)-3,7-dioxo-2-((phenylamino)methyl)-1-azabicyclo[3.2.0]heptane-2-carboxylate



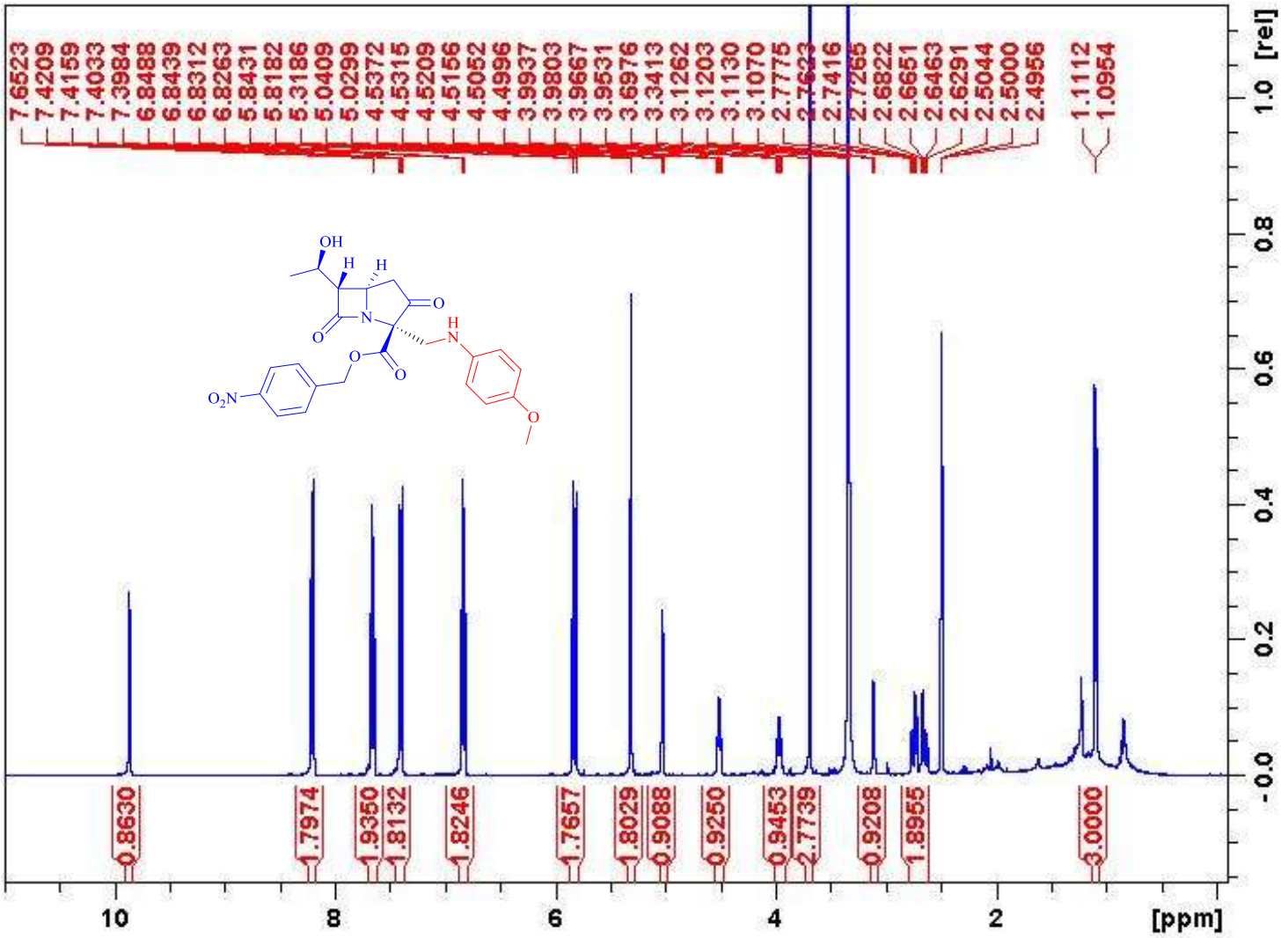
HSQC of (2*S*,5*R*,6*S*)-4-nitrobenzyl 6-((*R*)-1-hydroxyethyl)-3,7-dioxo-2-((phenylamino)methyl)-1-azabicyclo[3.2.0]heptane-2-carboxylate



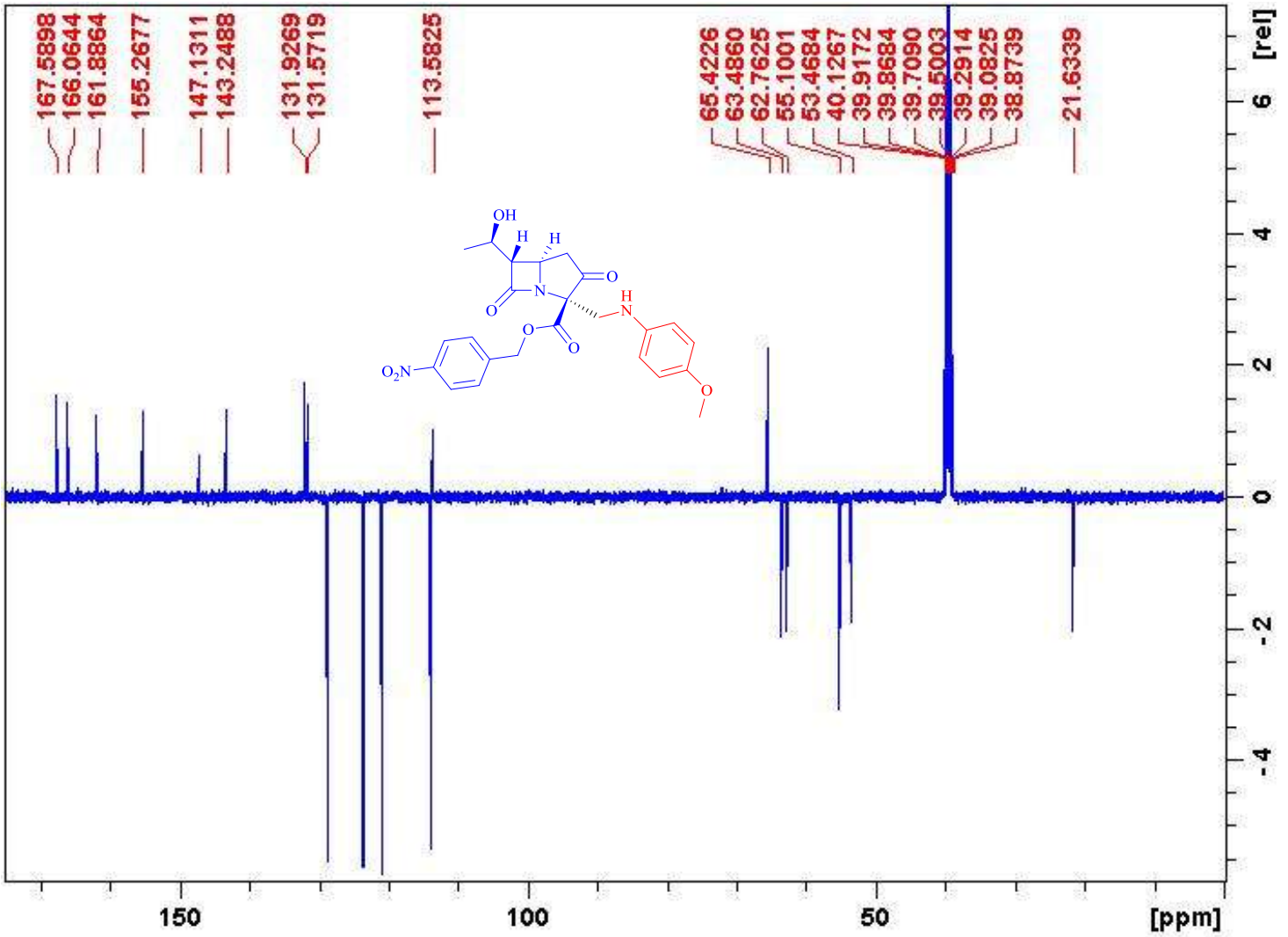
HMBC of (2*S*,5*R*,6*S*)-4-nitrobenzyl 6-((*R*)-1-hydroxyethyl)-3,7-dioxo-2-((phenylamino)methyl)-1-azabicyclo[3.2.0]heptane-2-carboxylate



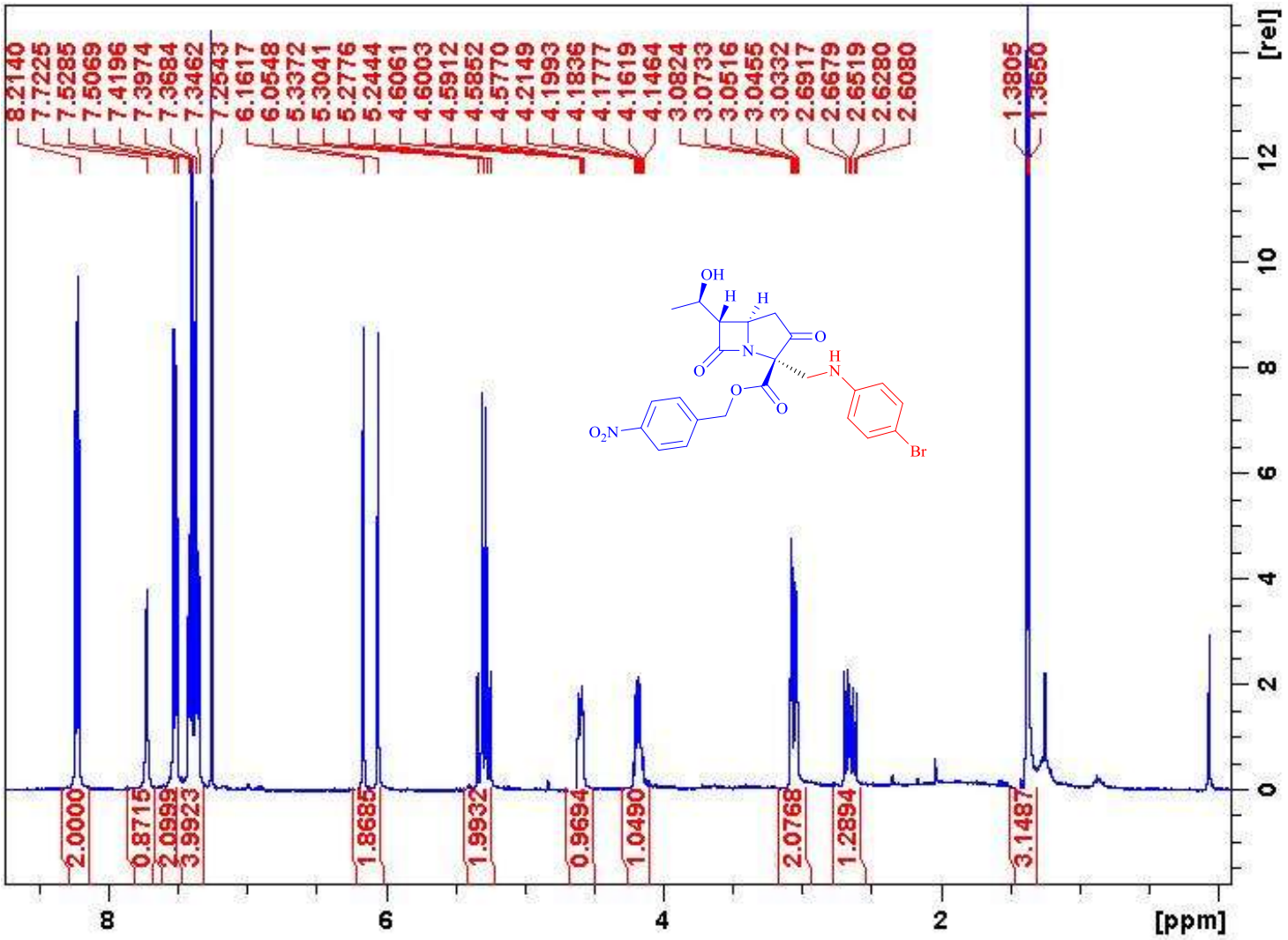
^{1}H-NMR spectra of (*2S,5R,6S*)-4-nitrobenzyl 6-((*R*)-1-hydroxyethyl)-2-((4-methoxyphenylamino)methyl)-3,7-dioxo-1-azabicyclo[3.2.0]heptane-2-carboxylate



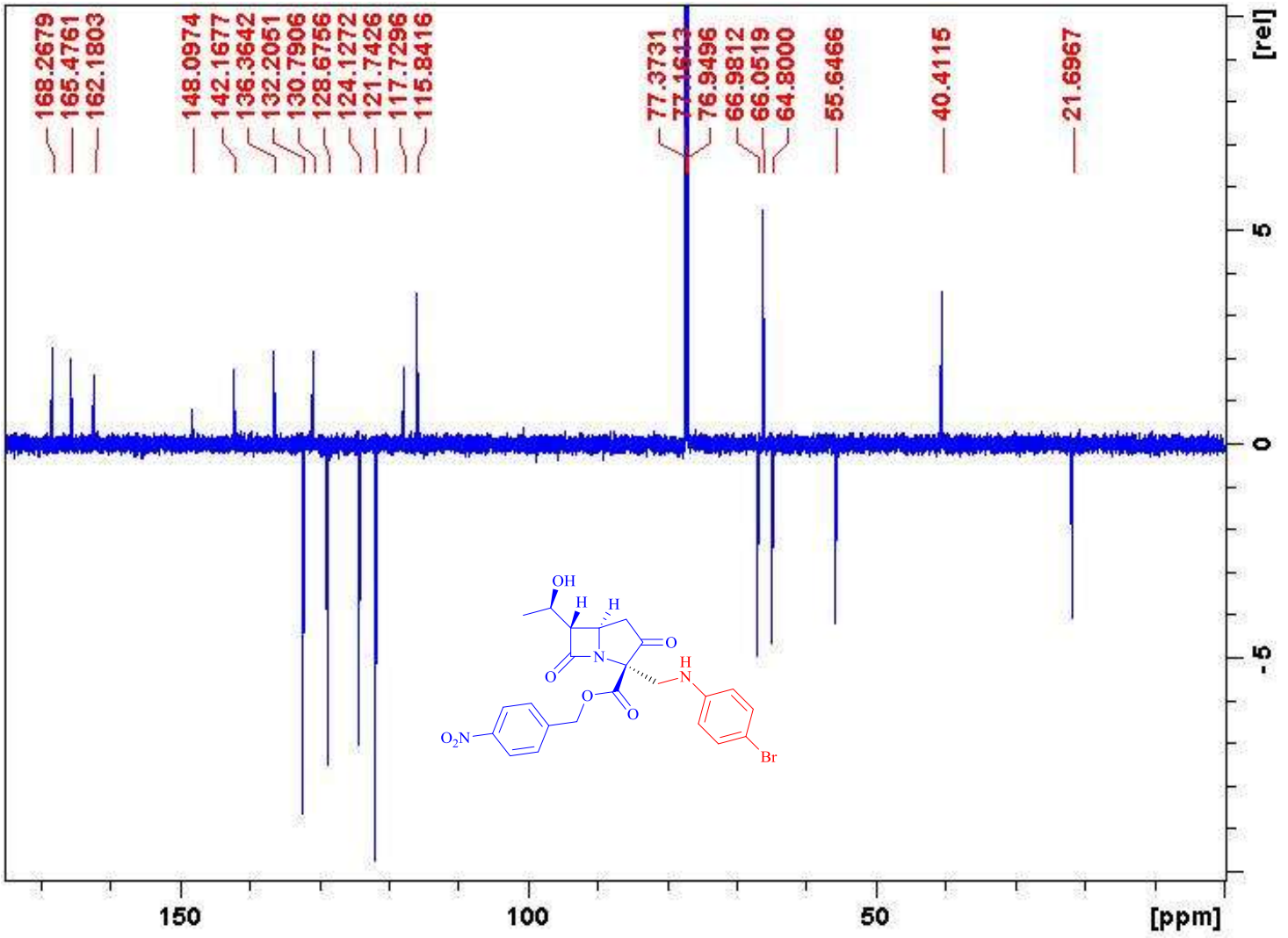
^{13}C-NMR spectra of (*2S,5R,6S*)-4-nitrobenzyl 6-((*R*)-1-hydroxyethyl)-2-((4-methoxyphenylamino)methyl)-3,7-dioxo-1-azabicyclo[3.2.0]heptane-2-carboxylate



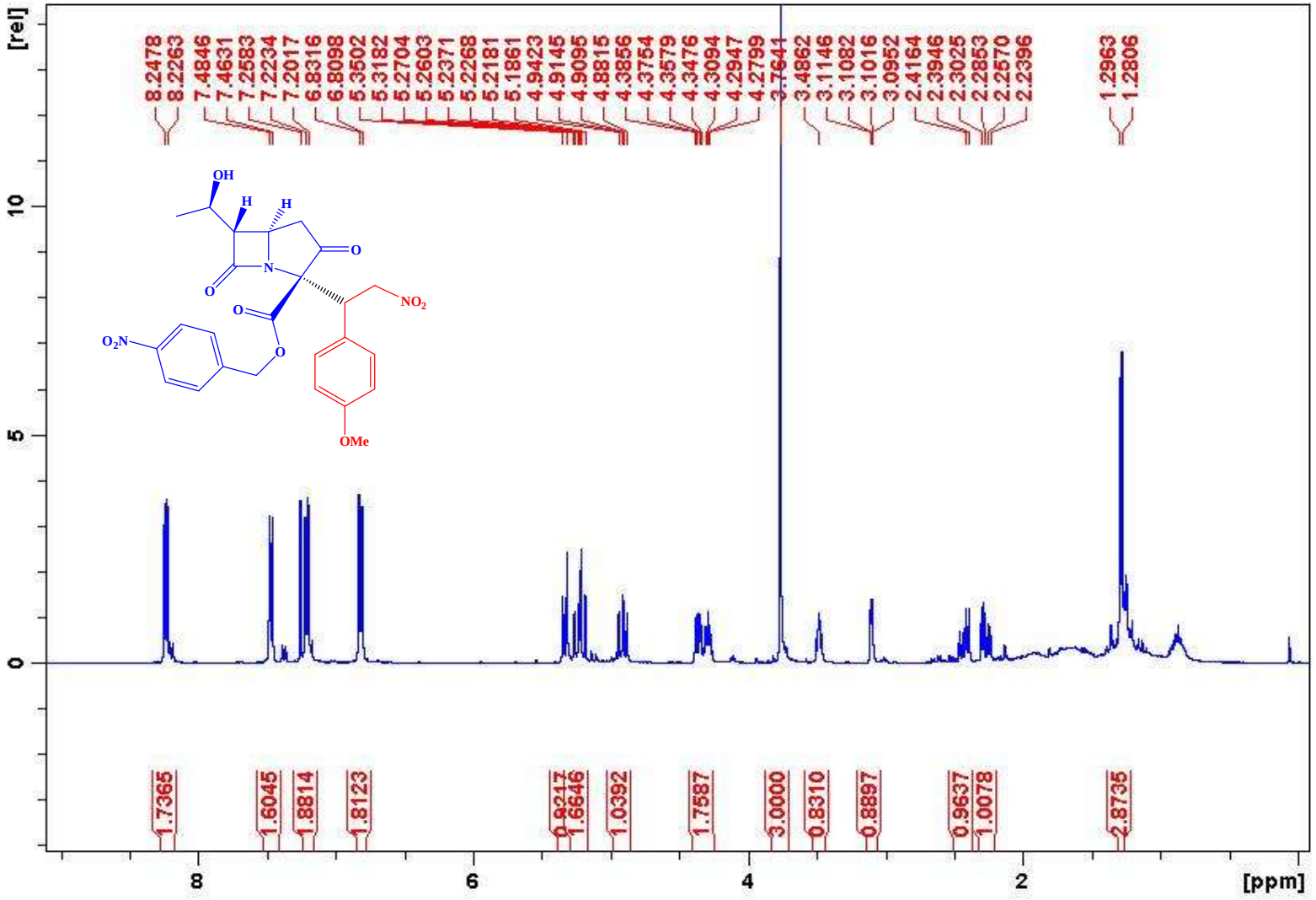
^{13}C-NMR spectra of (*2S,5R,6S*)-4-nitrobenzyl 2-((4-bromophenylamino)methyl)-6-((*R*)-1-hydroxyethyl)-3,7-dioxo-1-azabicyclo[3.2.0]heptane-2-carboxylate



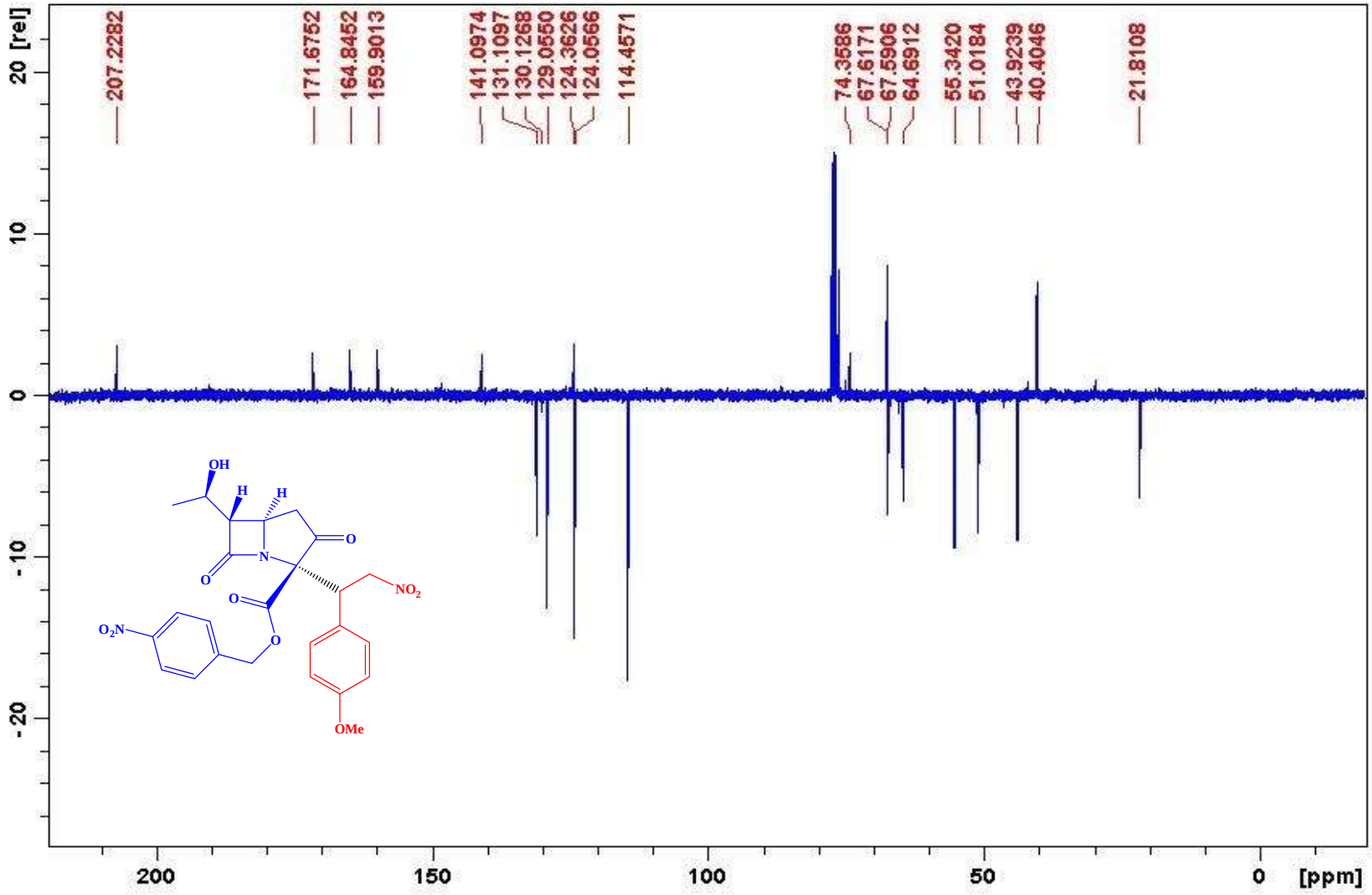
^{13}C-NMR spectra of (*2S,5R,6S*)-4-nitrobenzyl 2-((4-bromophenylamino)methyl)-6-((*R*)-1-hydroxyethyl)-3,7-dioxo-1-azabicyclo[3.2.0]heptane-2-carboxylate



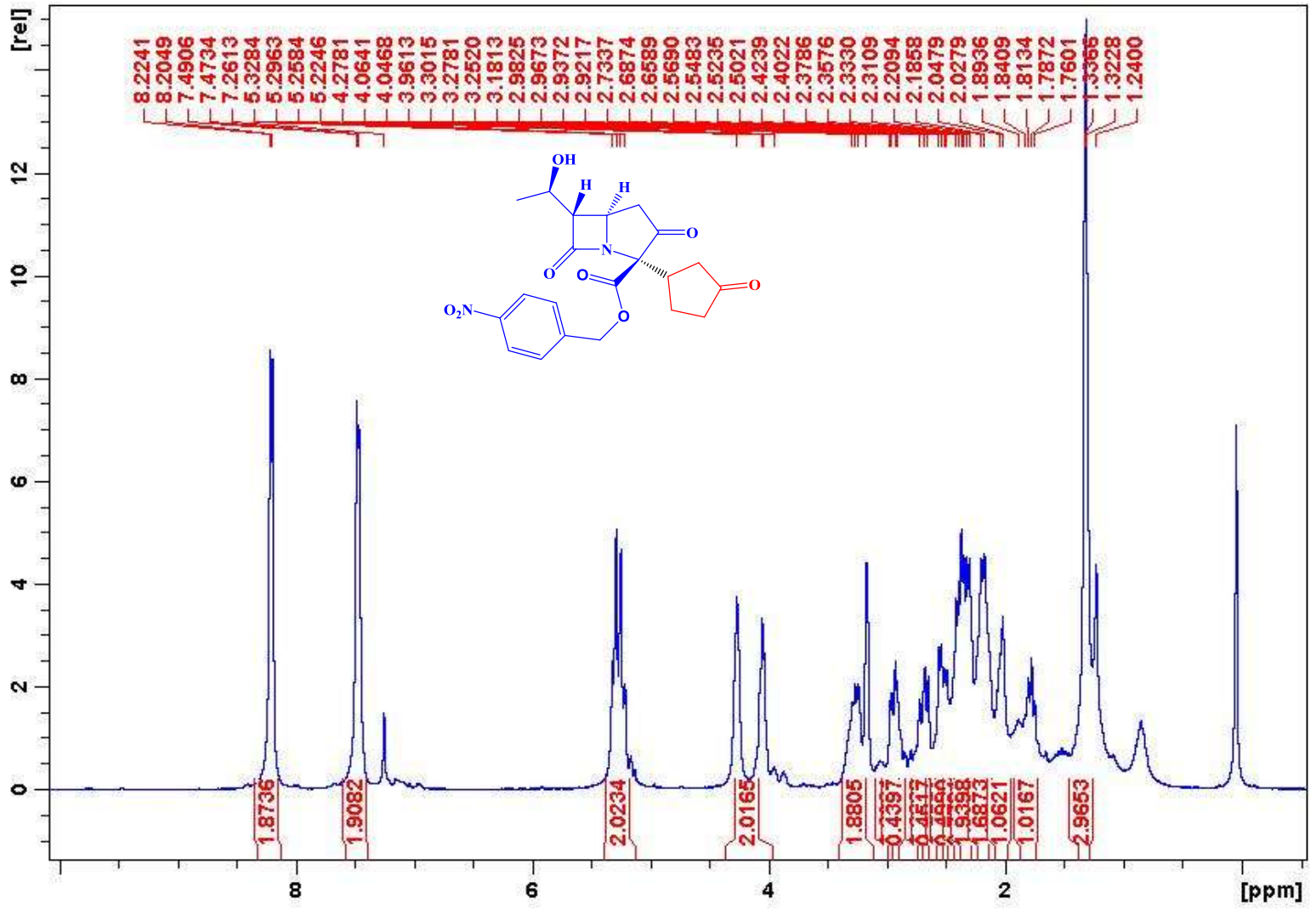
^{1}H-NMR spectra of (*2S,5R,6S*)-4-nitrobenzyl 6-((*R*)-1-hydroxyethyl)-2-((*S*)-1-(4-methoxyphenyl)-2-nitroethyl)-3,7-dioxo-1-azabicyclo [3.2.0] heptane-2-carboxylate



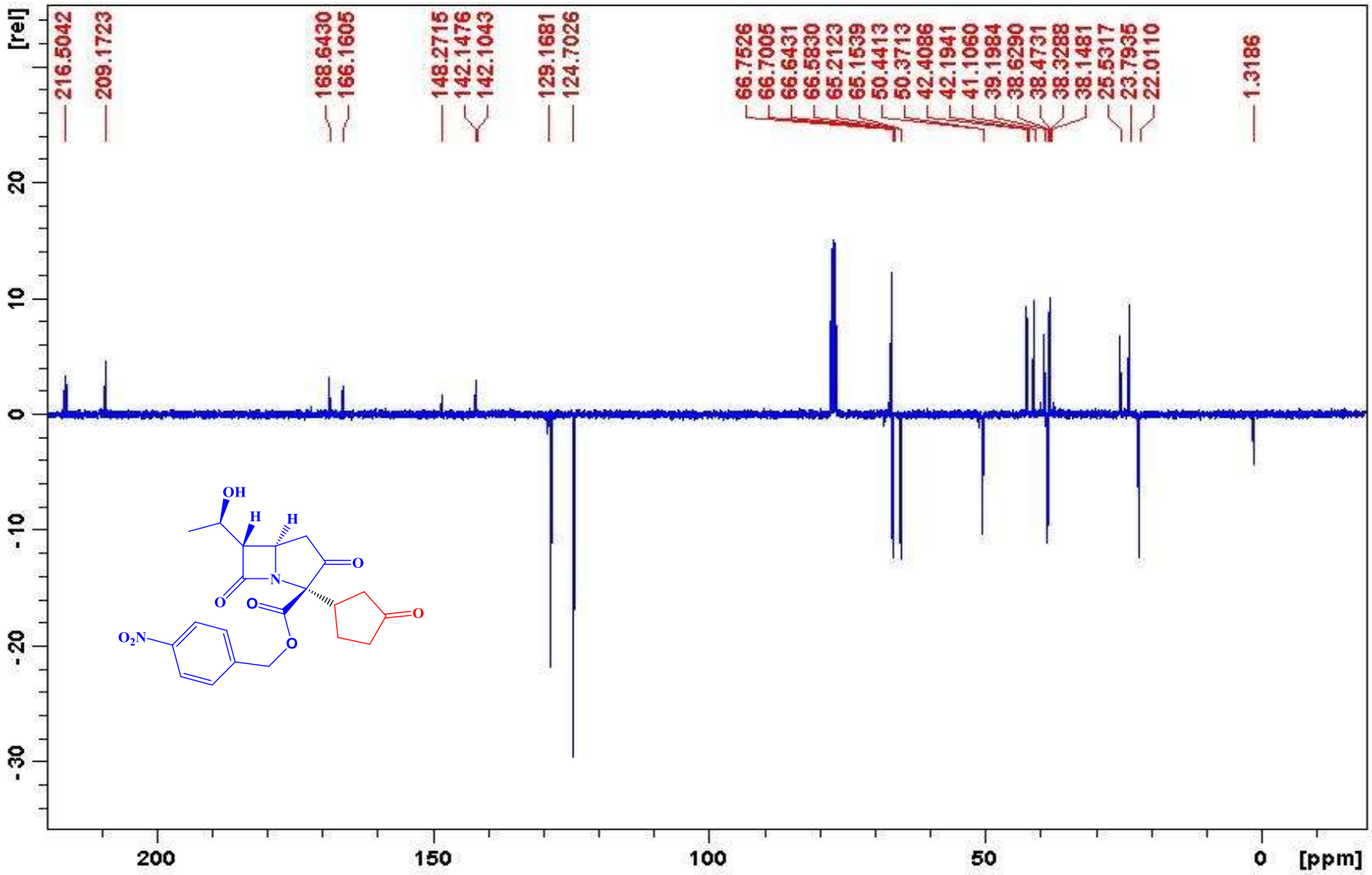
^{13}C-NMR spectra of (2S,*5R,6S*)-4-nitrobenzyl 6-((*R*)-1-hydroxyethyl)-2-((*S*)-1-(4-methoxyphenyl)-2-nitroethyl)-3,7-dioxo-1-azabicyclo [3.2.0] heptane-2- carboxylate



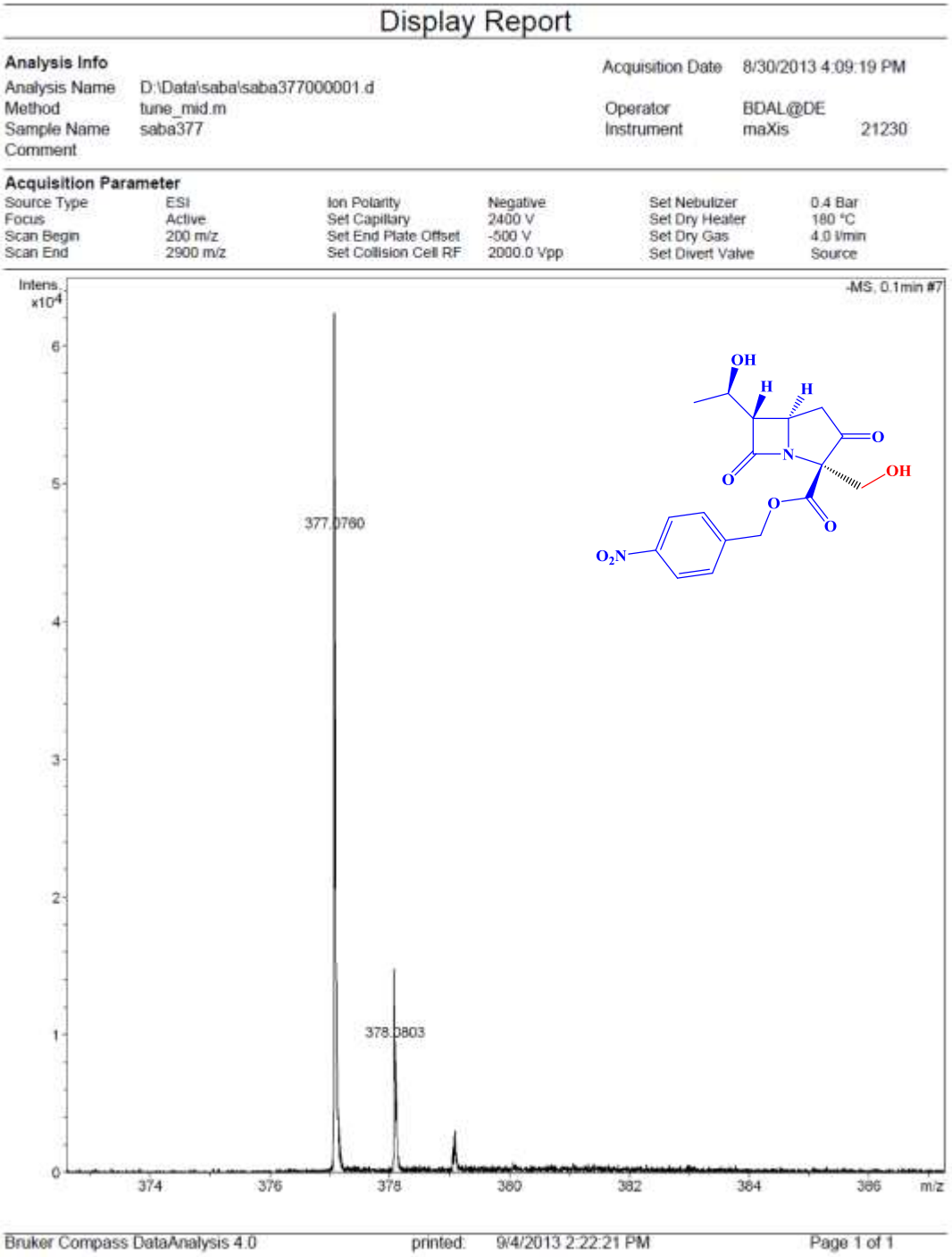
^{1}H-NMR (*2S,5R,6S*)-4-nitrobenzyl 6-((*R*)-1-hydroxyethyl)-3,7-dioxo-2-((*R*)-3-oxocyclopentyl)-1-azabicyclo[3.2.0]heptane-2-carboxylate



^{13}C-NMR (*2S,5R,6S*)-4-nitrobenzyl 6-((*R*)-1-hydroxyethyl)-3,7-dioxo-2-((*R*)-3-oxocyclopentyl)-1-azabicyclo[3.2.0]heptane-2-carboxylate



HRMS of ((*2S*)-4-nitrobenzyl-6-((*R*)-1-hydroxyerthyl)-2-(hydroxymethyl)-3,7-dioxo-1-azabicyclo[3.2.0]heptane-2-carboxylate)



HRMS of (*2S*)-nitrobenzyl2-((*R*)-hydroxy(phenyl)methyl)-6-(1-hydroxyethyl)-3,7-dioxo-1-azabicyclo[3.2.0]heptane-2-carboxylate

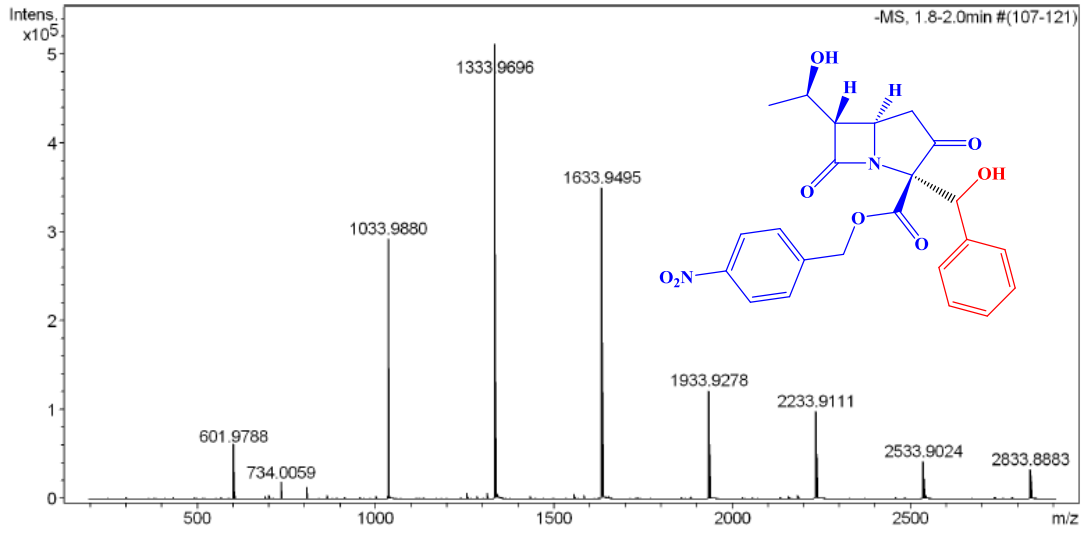
Display Report

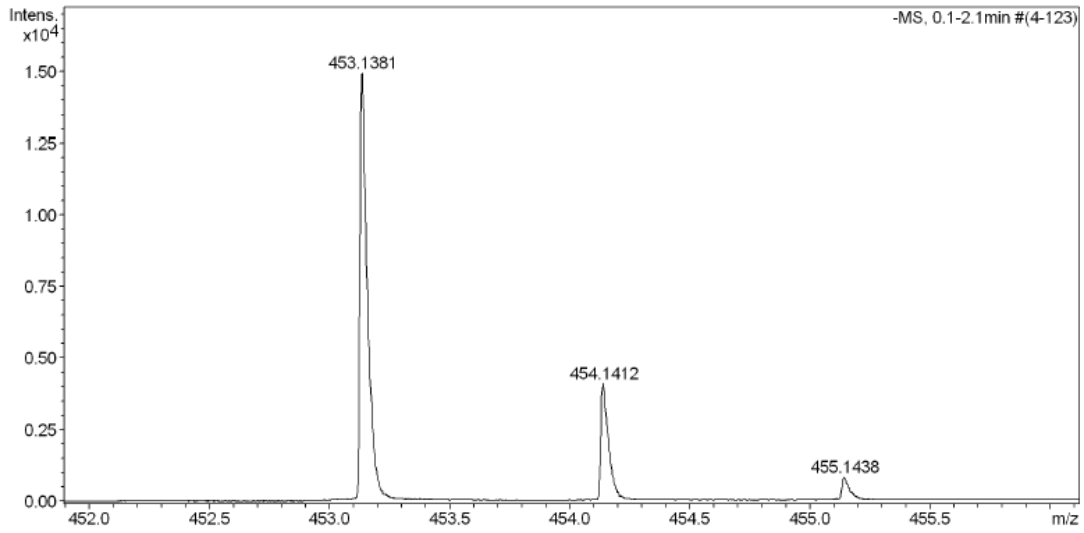
Analysis Info

Analysis Name	D:\Data\saba\saba454000001.d	Acquisition Date	8/30/2013 4:18:14 PM
Method	tune_mid.m	Operator	BDAL@DE
Sample Name	saba454	Instrument	maXis 21230
Comment			

Acquisition Parameter

Source Type	ESI	Ion Polarity	Negative	Set Nebulizer	0.4 Bar
Focus	Active	Set Capillary	2400 V	Set Dry Heater	180 °C
Scan Begin	200 m/z	Set End Plate Offset	-500 V	Set Dry Gas	4.0 l/min
Scan End	2900 m/z	Set Collision Cell RF	2000.0 Vpp	Set Divert Valve	Source





HRMS of (2*S*,5*R*,6*S*)-4-nitrobenzyl 6-((*R*)-1-hydroxyethyl)-3,7-dioxo-2-((phenylamino) methyl)-1-azabicyclo[3.2.0]heptane-2-carboxylate

Display Report

Analysis Info

Analysis Name	D:\Data\sachin\454000001.d	Acquisition Date	8/30/2013 4:27:33 PM
Method	tune_mid.m	Operator	BDAL@DE
Sample Name	454	Instrument	maXis 21230
Comment			

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.4 Bar
Focus	Active	Set Capillary	3500 V	Set Dry Heater	180 °C
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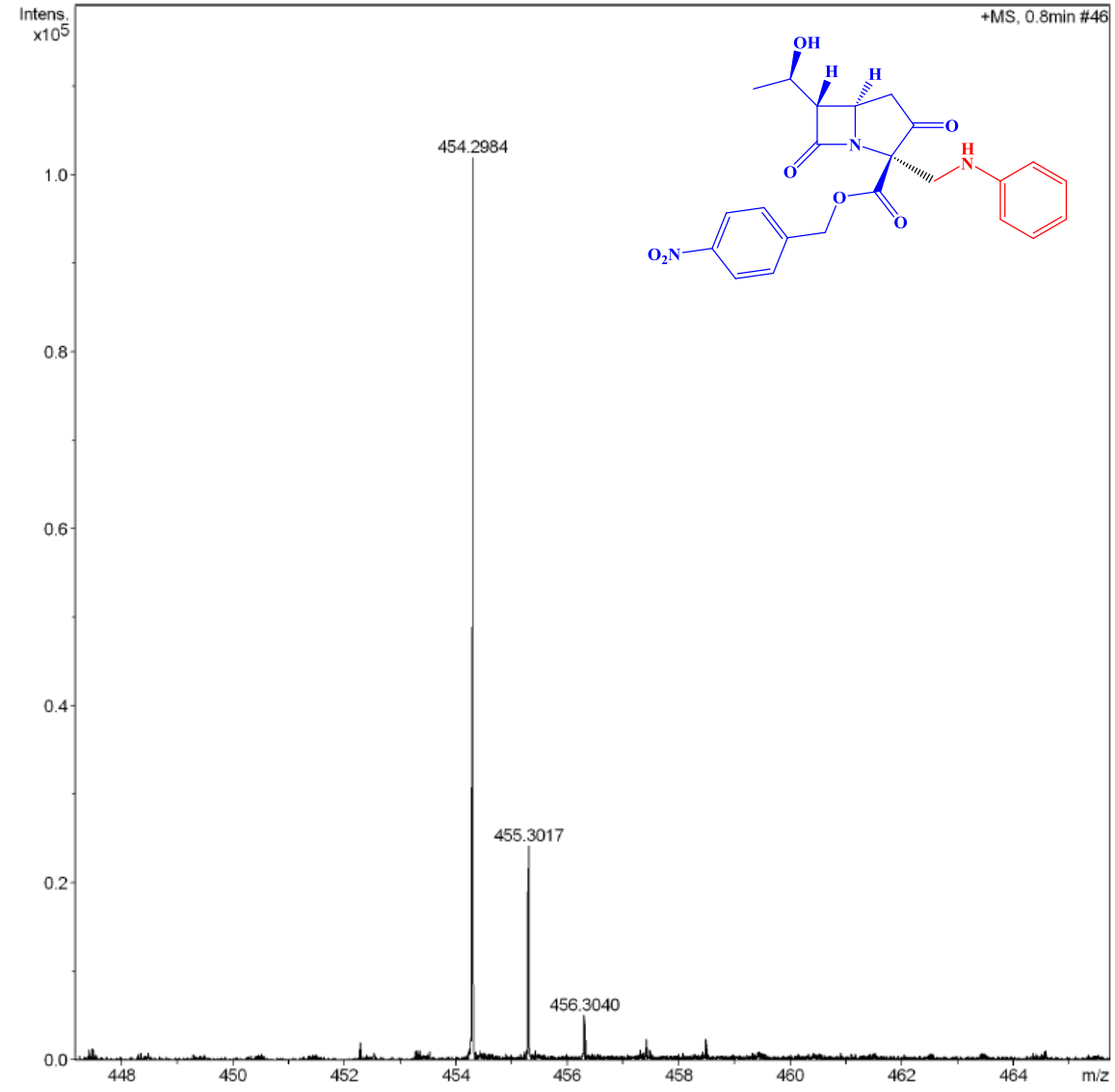
Scan End	2900 m/z	Set Collision Cell RF	2000.0 Vpp	Set Divert Valve	Source



HRMS of (*2S,5R,6S*)-4-nitrobenzyl 6-((*R*)-1-hydroxyethyl)-2-((4-methoxyphenylamino)methyl)-3,7-dioxo-1-azabicyclo[3.2.0]heptane-2-carboxylate

Display Report

Analysis Info

Analysis Name	D:\Data\thavi\methoxy.d	Acquisition Date	10/16/2013 12:55:41 PM
Method	fia.m	Operator	BDAL@DE
Sample Name	tunemix	Instrument	micrOTOF-Q 10139
Comment			

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.4 Bar
Focus	Not active	Set Capillary	4500 V	Set Dry Heater	200 °C
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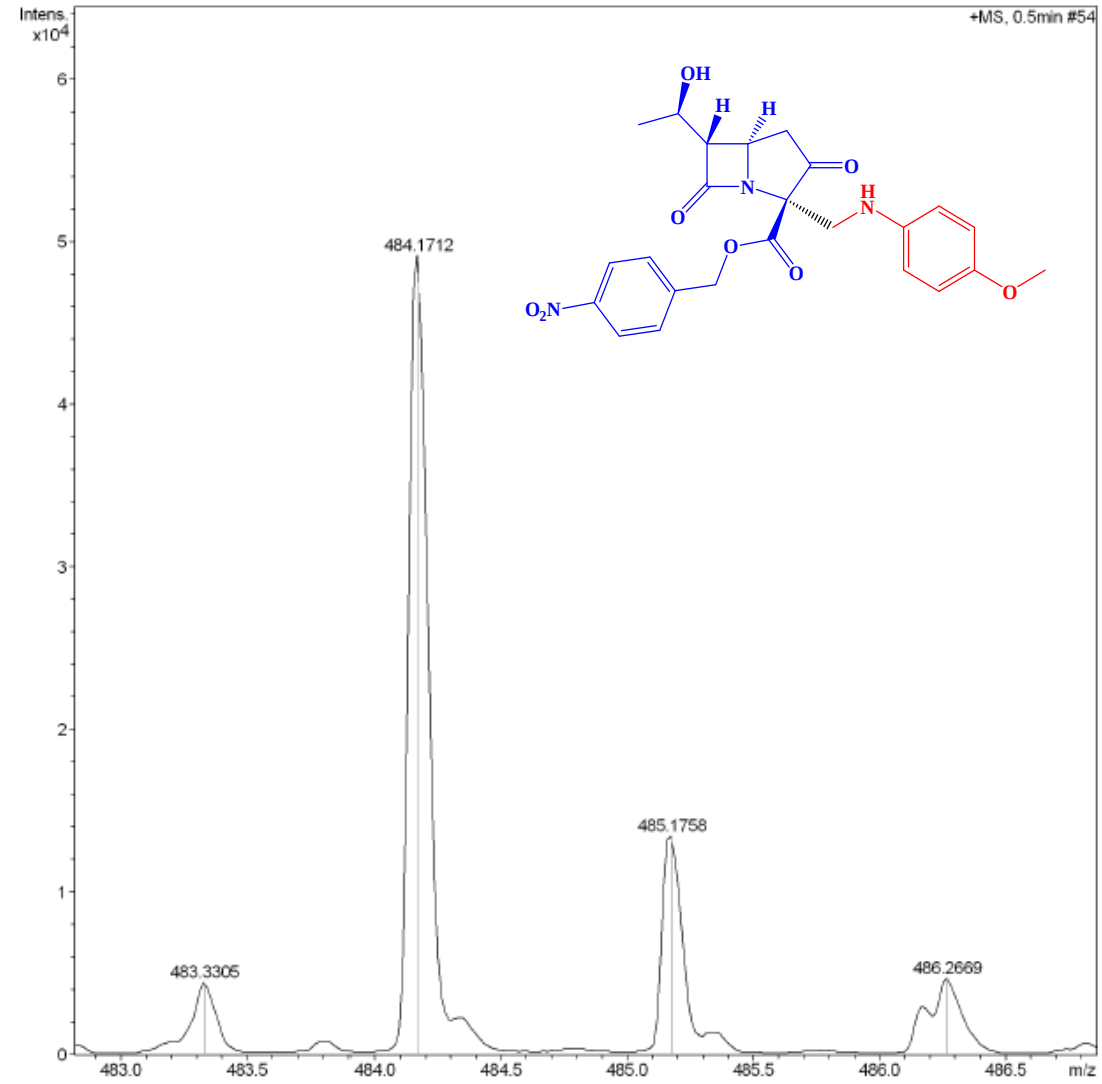
Scan End	3000 m/z	Set Collision Cell RF	500.0 Vpp	Set Divert Valve	Source



HRMS of (*2S,5R,6S*)-4-nitrobenzyl 2-((4-bromophenylamino)methyl)-6-((*R*)-1-hydroxyethyl)-3,7-dioxo-1-azabicyclo[3.2.0]heptane-2-carboxylate

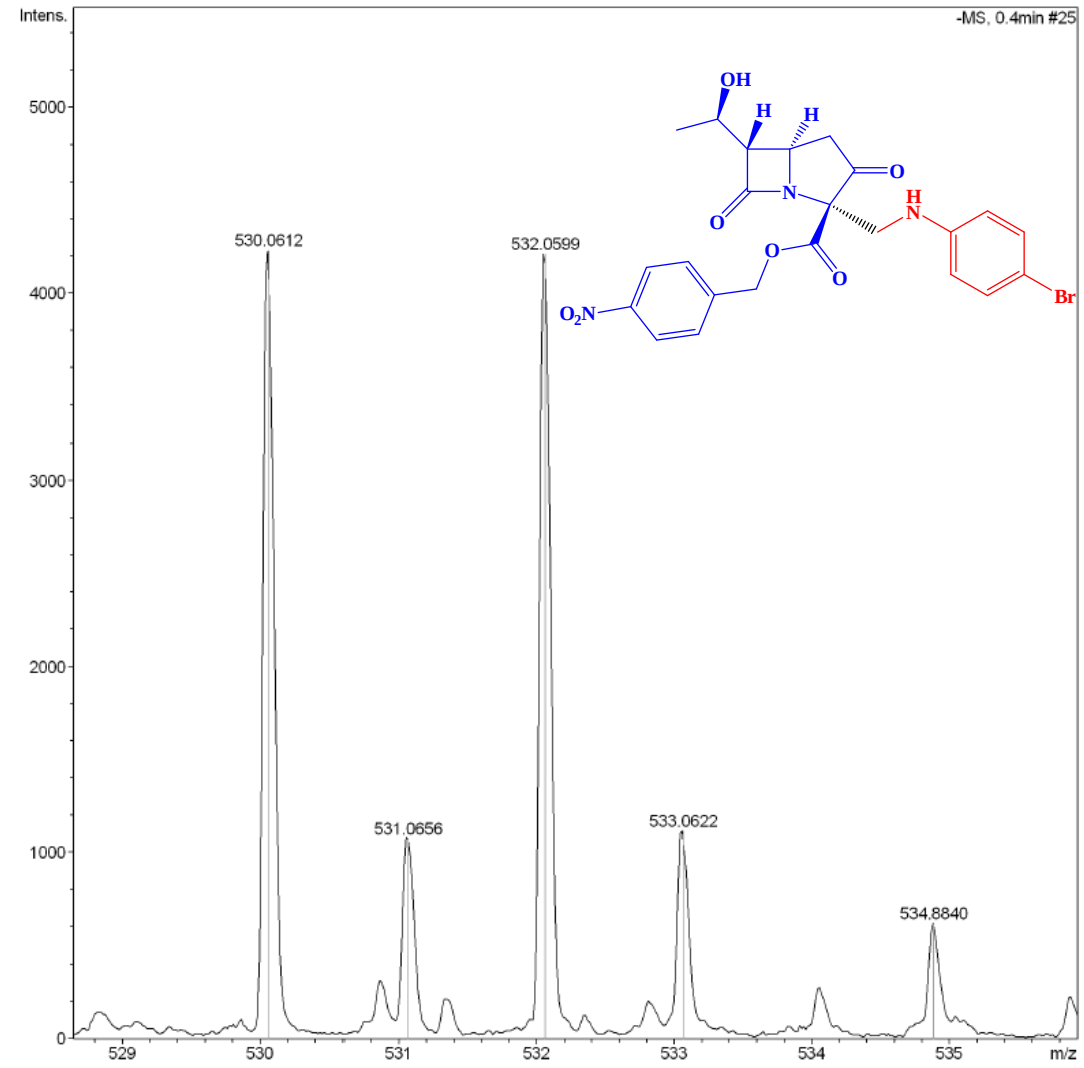
Display Report

Analysis Info

Analysis Name	D:\Data\thavi\bromo.d	Acquisition Date	10/16/2013 12:39:47 PM
Method	fia_neg.m	Operator	BDAL@DE
Sample Name	tunemix	Instrument	micrOTOF-Q 10139
Comment			

Acquisition Parameter

Source Type	ESI	Ion Polarity	Negative	Set Nebulizer	0.4 Bar
Focus	Not active	Set Capillary	3500 V	Set Dry Heater	200 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	4.0 l/min
Scan End	3000 m/z	Set Collision Cell RF	500.0 Vpp	Set Divert Valve	Source



Bruker Compass DataAnalysis 4.0 printed: 10/16/2013 1:07:26 PM Page 1 of 1

HRMS of (*2S,5R,6S*)-4-nitrobenzyl 6-((*R*)-1-hydroxyethyl)-2-((*S*)-1-(4-methoxy phenyl)-2-nitroethyl)-3,7-dioxo-1-azabicyclo [3.2.0] heptane-2-carboxylate

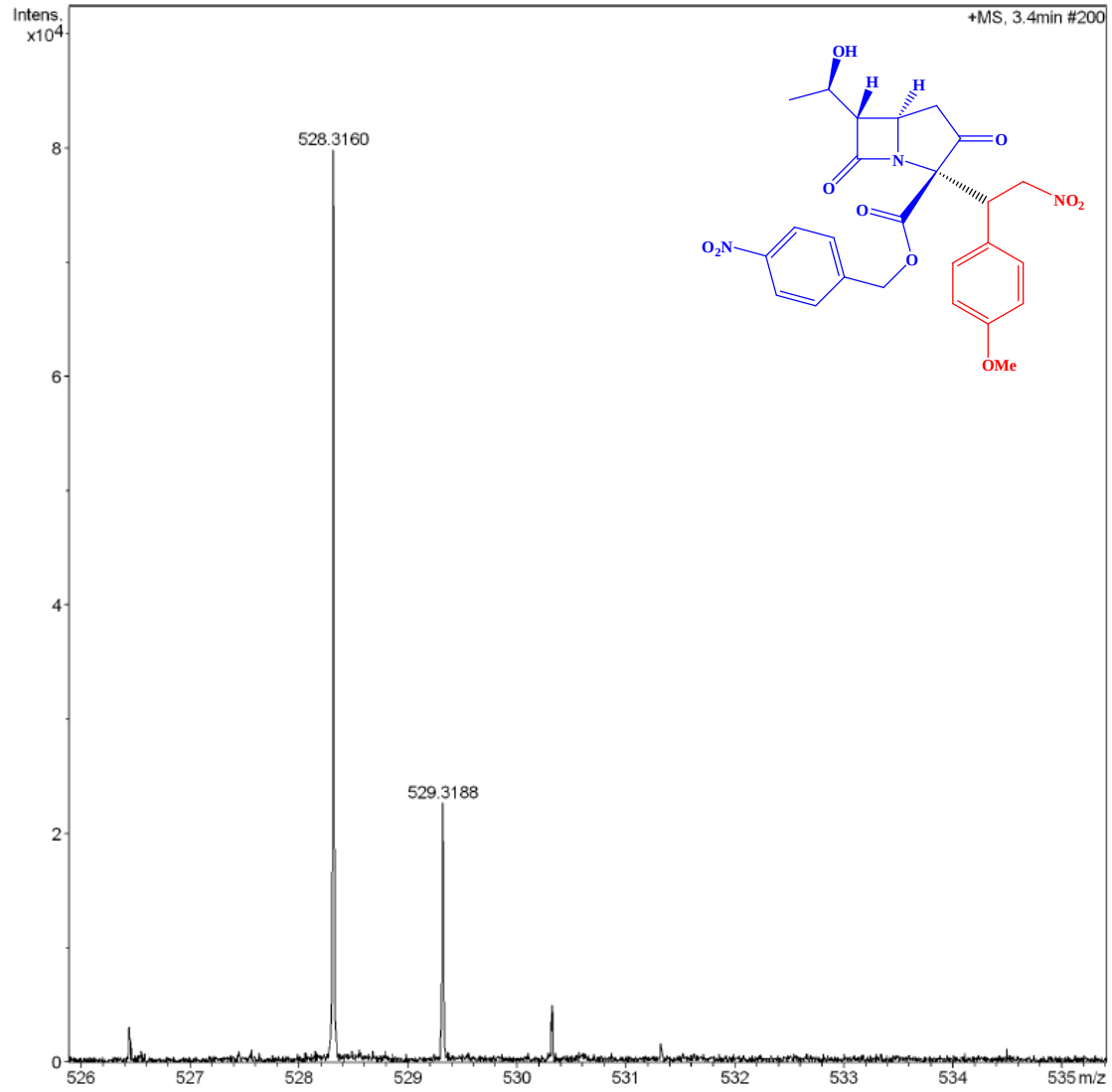
Display Report

Analysis Info

Analysis Name	D:\Data\sibusiso\st527000002.d	Acquisition Date	8/30/2013 3:17:00 PM
Method	tune_mid.m	Operator	BDAL@DE
Sample Name	st527	Instrument	maXis 21230
Comment			

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.4 Bar
Focus	Active	Set Capillary	3500 V	Set Dry Heater	180 °C
Scan Begin	200 m/z	Set End Plate Offset	-500 V	Set Dry Gas	4.0 l/min
Scan End	2900 m/z	Set Collision Cell RF	2000.0 Vpp	Set Divert Valve	Source



HRMS of (*2S,5R,6S*)-4-nitrobenzyl 6-((R)-1-hydroxyethyl)-3,7-dioxo-2-((*R*)-3-oxocy clopentyl)-1-azabicyclo[3.2.0]heptane-2-carboxylate

Display Report

Analysis Info

Analysis Name	D:\Data\sibusiso\430000001.d	Acquisition Date	8/30/2013 4:23:05 PM
Method	tune_mid.m	Operator	BDAL@DE
Sample Name	430	Instrument	maXis 21230
Comment			

Acquisition Parameter

Source Type	ESI	Ion Polarity	Negative	Set Nebulizer	0.4 Bar
Focus	Active	Set Capillary	2400 V	Set Dry Heater	180 °C
Scan Begin	200 m/z	Set End Plate Offset	-500 V	Set Dry Gas	4.0 l/min
Scan End	2900 m/z	Set Collision Cell RF	2000.0 Vpp	Set Divert Valve	Source