A one-pot multicomponent coupling/cyclization for natural product herbicide (±)-thaxtomin A

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Electronic Supplementary Information (ESI)

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Experimental

General: All reagents and solvents were purchased from commercial sources and used without further purification. Compounds were purified using flash chromatography with Whatman Purasil 60 Å 230-400 mesh silica gel or by preparative TLC using Dynamic Absorbents Inc. silica gel TLC 250 μm w/h F-254, catalog no. 84111. Proton and carbon NMR spectra were recorded using a Bruker Avance III 600 spectrometer. The residual solvent signal in CD_3OD was referenced to 3.31 and 49.0 ppm in proton and carbon spectra respectively, while the residual signal in CD_3CN was referenced to 1.96 ppm in proton and 1.3 ppm in carbon. Mass spectral data were taken on a Waters ESI-MS, model LCT premier XE. All reactions involving microwave irradiation were conducted with a capped vial using a CEM Discover System. Methyl isocyanide was prepared as described in Schuster, R. E., Scott, J. E., Casanova, J. *Organic Syntheses* **1966**, *46,* 75. (DOI: 10.15227/orgsyn.046.0075) Ethyl 2-(4-nitro-1H-indol-3-yl)acetate was prepared as described in Bell, I.; Stump, C. A. "Preparation of spirolactam-containing tricyclic aryl and heteroaryl amides as CGRP receptor antagonists for the treatment of headaches, including migraine and cluster headaches". WO2008153852A1, 2008. (page 44 scheme 7, excluding the *N*-alkylation step).

2-(4-Nitro-1H-indol-3-yl)acetaldehyde (**3**). Procedure modified from Revelant, G.; Dunand, S.; Hesse, S. P.; Kirsch, G., *Synthesis* **2011**, 2935-2940. 9.40 mL of a 1M DIBAL-H (9.40 mmol) solution in THF was added slowly to 0.528 g (2.12 mmol) ethyl 2-(4-nitro-1H-indol-3-yl)acetate dissolved in 14 mL of anhydrous DCM at 4 ^{o}C. The reaction was allowed to warm to room temperature over 1 hour and then quenched with 4 mL methanol added dropwise. After gas evolution had stopped, 30 mL of 20% sodium potassium tartrate was added and the resulting slurry was filtered through a pad of celite. The celite was then washed several times with 10:1 DCM:MeOH, subsequently the filtrate was washed with saturated $NaHCO_3$ and brine solution to provide the crude alcohol, which was taken to the next step without further purification. To the crude alcohol was added 35 mL of acetonitrile and 1.79 g (6.40 mmol) of IBX and the suspension was heated at 100 ^{o}C for 1 hour. After the reaction had cooled to room temperature it was filtered through a pad of celite and then dried. The crude was then purified by flash column chromatography using DCM:MeOH to provide **3** (0.301 g, 70%) as a dark yellow solid, m.p. 116-118^{o}C. Data for **3**. ^{1}H NMR (600 MHz, CD_3CN): 9.92 (bs, 1H), 9.76 (s, 1H), 7.87 (d, *J* = 7.8 Hz, 1H), 7.81 (d, *J* = 7.8 Hz, 1H), 7.42 (s, 1H), 7.26 (t, *J* = 7.8 Hz, 1H), 4.02 (s, 2H); ^{13}C NMR (150 MHz, CD_3CN): δ 201.5, 143.4, 140.5, 131.1, 121.4, 120.3, 119.4, 118.5, 107.3, 43.1; ESI HRMS: $[M+Na]^+$ calcd for $C_{10}H_8N_2NaO_3$ 227.0433, found 227.0424.

3-Hydroxyphenylpyruvic acid (**5**). Procedure from Wong, H. N. C.; Xu, Z. L.; Chang, H. M.; Lee, C. M., *Synthesis* **1992**, 793-797 with modified purification as described below. 3.00 g (24.6 mmol) 3-hydroxybenzaldehyde, 3.45 g (29.5 mmol) of *N*-acetyl glycine, 2.62 g (31.8 mmol) sodium acetate, and 11.6 mL of acetic anhydride were combined and heated at 120 ^{o}C for 5 hours. After the reaction had cooled to room temperature 12 mL of ice water was added. The resulting precipitate was filtered and washed with 50% aqueous EtOH (4 x 5 mL) then recrystallized from acetone. The orange solid (4.18 g)

and 102 mL of 3M HCl were combined and heated at 100 ^{o}C for 3 hours and 40 minutes. The reaction was allowed to cool, the precipitate filtered off and the filtrate extracted (3 x 35 mL) ethyl acetate. The combined organic layers were then concentrated and subsequently dissolved in a minimal amount of diethyl ether. The remaining dark brown solid was filtered off and the solvent was removed under reduced pressure to provide **5** (36% over two steps, 1.60 g) as a yellow solid, m.p. 160-162 ^{o}C. ^{1}H NMR (600 MHz, CD_3OD): δ 7.34 (s, 1H), 7.15 (m, 2H), 6.67 (m, 1H), 6.41 (s, 1H); ^{13}C NMR (150 MHz, CD_3OD): δ 168.4, 158.3, 142.2, 137.6, 130.1, 122.5, 117.2, 115.6, 111.6; ESI LRMS: $[M-H]^-$ found 179.2.

(±)-Acyclic Ugi precursor (**2**). To 0.010 g (0.049 mmol) 2-(4-nitro-1H-indol-3-yl)acetaldehyde was added 0.10 mL of MeOH followed by 4.2 □L of methyl amine solution (40% in water). The imine formation was allowed to take place over 5 minutes at room temperature. Then 2.6 μL (0.049 mmol) of methyl isocyanide and 0.0088 g (0.049 mmol) of 3-hydroxyphenylpyruvic acid were added sequentially and the reaction was left to stir overnight. The reaction was then concentrated under reduced pressure to an oil and purified by preparative TLC with 20:1 DCM:MeOH (3 times) to provide **2** (0.0080 g, 37%) as a yellow-orange solid, m.p. 101-103 ^{o}C. Data for **2**, as a mixture of two rotamers. ^{1}H NMR (600 MHz, CD_3OD): δ 7.94 (d, J = 7.8 Hz, 1H), 7.88 (d, J = 7.9 Hz, 1H), 7.77 (d, J = 8.0 Hz, 1H), 7.73 (d, J = 8.0 Hz, 1H), 7.38 (s, 1H), 7.32 (s, 1H), 7.26 (t, J = 8.0 Hz, 1H), 7.22 (t, J = 8.0 Hz, 1H), 7.10 (t, J = 7.8 Hz, 1H), 6.95 (t, J = 7.8 Hz, 1H), 6.71 (m, 2H), 6.58 (m, 3H), 6.31 (m, 1H), 6.15 (m, 1H), 5.09 (dd, J_1 = 5.9 Hz, J_2 = 9.6 Hz, 1H), 4.73 (dd, , J_1 = 4.6 Hz, J_2 = 9.7 Hz, 1H), 3.79 (d, J = 17.4 Hz, 1H), 3.74 (d, J = 15.3 Hz, 1H), 3.62 (d, J = 15.3 Hz, 1H), 3.58 (dd, , J_1 = 4.6 Hz, J_2 = 14.9 Hz, 1H), 3.51 (dd, J_1 = 5.9 Hz, J_2 = 14.9 Hz, 1H), 3.23 (dd, J_1 = 9.6 Hz, J_2 = 14.9 Hz, 1H), 3.13 (dd, J_1 = 9.7 Hz, J_2 = 14.9 Hz, 1H), 3.01 (s, 3H), 2.75 (s, 3H), 2.70 (s, 6H), 2.57 (d, J = 17.3 Hz, 1H); ^{13}C NMR (150 MHz, CD_3OD): δ 199.0, 198.5, 171.8, 171.6, 169.31, 169.28, 158.9, 158.4, 144.0, 143.8, 141.18, 141.15, 134.9, 134.2, 131.4, 130.9, 130.7, 130.2, 122.1, 121.8, 121.6, 121.2, 120.2, 119.9, 119.6, 119.1, 119.0, 118.6, 117.8, 117.7, 115.5, 115.0, 110.5, 110.3, 63.4, 58.8, 47.4, 46.2, 31.9, 29.7, 27.5, 27.1, 26.6, 26.4; ESI HRMS: $[M+Na]^+$ calcd for $C_{22}H_{22}N_4NaO_6$ 461.1437, found 461.1440.

(±)-**Anti-1**. To 0.022 g (0.050 mmol) of acylic Ugi (**2**) was added 0.50 mL of MeOH, then 0.070 mL (0.50 mmol) triethylamine and the mixture was allowed to stir for 30 minutes at room temperature. The reaction was then concentrated to dryness and purified by preparative TLC 20:1 DCM:MeOH (3 times) to provide **anti-1** and **syn-1** (0.017 g, 77%, dr 10:1). Pure **anti-1** is a yellow solid with a m.p. of 207-208 ^{o}C. This purification procedure is sufficient for the separation of the diastereomers if desired, however this material was utilized for the preparation of (±)-thaxtomin A therefore **anti-1** and **syn-1** were combined. Data for **anti-1**. ^{1}H NMR (600 MHz, CD_3OD): δ 7.75 (d, J = 7.8 Hz, 1H), 7.67 (d, J = 8.0 Hz, 1H), 7.16 (m, 2H), 7.04 (t, J = 7.6 Hz, 1H), 6.67 (m, 1H), 6.43 (m, 2H), 3.56 (dd, J_1 = 3.7 Hz, J_2 = 15.3 Hz, 1H), 3.31 (m, 2H, overlapping with solvent residual peak), 3.13 (d, J = 13.4 Hz, 1H), 3.00 (d, J = 13.4 Hz, 1H), 2.80 (s, 3H), 2,79 (s, 3H); ^{13}C NMR (150 MHz, CD_3OD): δ 168.4, 167.7, 158.6, 144.3, 140.8, 136.7, 130.4, 130.1, 121.7, 121.0, 120.2, 118.7, 118.3, 117.4, 115.5, 108.8, 87.50, 63.0,

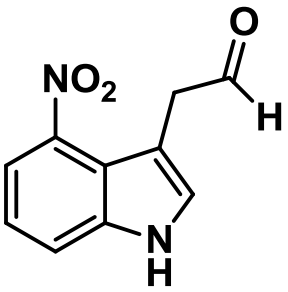
44.9, 32.9, 30.4, 28.1; ESI HRMS: $[M+Na]^+$ calcd for $C_{22}H_{22}N_4NaO_6$ 461.1437, found 461.1455.

(±)-Thaxtomin A (**syn-1**), from **anti-1**. To 0.017 g (0.039 mmol) of the diastereomeric mixture was added 1.6 mL MeOH and 0.19 mL of 1M KOH (0.20 mmol) in MeOH. The reaction mixture was subsequently heated to 70 ^oC for 30 minutes (5 minute ramp) with a CEM Discover microwave. After the reaction had cooled, 12 μL (0.21 mmol) of acetic acid was added, then the reaction was concentrated and the crude material purified by preparative TLC using 20:1 DCM:MeOH (3 times) to provide **syn-1** and **anti-1** (0.014 g, 85%, dr 4:1). Pure **syn-1** is an orange solid, m.p. 205-206 ^oC. Data for thaxtomin A, **syn-1**. ^1H NMR (600 MHz, CD_3OD): δ 7.84 (dd, *J* = 7.9 Hz, 1H), 7.69 (d,*J* = 8.0 Hz, 1H), 7.23 (t, *J* = 8.1 Hz, 1H), 7.18 (t, *J* = 8.0 Hz, 1H), 6.95 (s, 1H), 6.75 (m, 1H), 6.70 (m, 2H), 3.86 (dd, J_1 = 6.4 Hz, J_2 = 8.8 Hz, 1H), 3.31 (overlapping with solvent residual peak, 1H), 3.13 (d, *J* = 13.5 Hz, 1H), 3.03 (s, 3H), 2.82 (s, 3H), 2.60 (dd, J_1 = 6.3 Hz, J_2 = 14.1 Hz, 1H), 1.61 (dd, J_1 = 8.9 Hz, J_2 = 14.1 Hz, 1H); ^{13}C NMR (150 MHz, CD_3OD): δ 168.3, 166.8, 159.1, 143.6, 141.1, 137.4, 132.5, 131.2, 122.7, 121.0, 119.8, 119.3, 118.6, 118.4, 115.9, 110.5, 88.0, 64.6, 43.5, 34.2, 33.5, 28.5; ESI HRMS: $[M+Na]^+$ calcd for $C_{22}H_{22}N_4NaO_6$ 461.1437, found 461.1460.

(±)-Thaxtomin A (**syn-1**), from acyclic starting material. To 0.016 g (0.036 mmol) of the Ugi precursor was added 1.6 mL MeOH and 0.18 mL of 1M KOH (0.18 mmol) in MeOH. The reaction mixture was subsequently heated to 70 ^oC for 30 minutes (5 minute ramp) with a CEM Discover microwave. After the reaction had cooled, 11 μL (0.19 mmol) of acetic acid was added then the reaction was concentrated and the crude material purified by preparative TLC using 20:1 DCM:MeOH (3 times) to provide **syn-1** and **anti-1** (0.009 g, 56%, dr 4:1), alternatively the crude material could be recrystallized using 20:1 DCM:MeOH and hexanes. Data matched that of thaxtomin A prepared from the diastereomer as described above.

(±)-Thaxtomin A (**syn-1**), one pot procedure. To 0.010 g (0.049 mmol) 2-(4-nitro-1H-indol-3-yl)acetaldehyde was added 0.10 mL of MeOH followed by 4.2 μL (0.049 mmol) of methyl amine solution (40% in water). The imine formation was then allowed to take place over 5 minutes at room temperature. Then 2.6 μL (0.049 mmol) of methylisocyanide and 0.0088 g (0.049 mmol) of 3-hydroxyphenylpyruvic acid were added sequentially and the reaction was left to stir for 20 hours. The reaction was then diluted with 0.80 mL of MeOH and 0.13 mL of 1M KOH (0.13 mmol) in MeOH. Following this addition, the reaction mixture was heated to 70 ^oC in a microwave reactor for 30 minutes (5 minute ramp time). After the reaction had cooled to ambient temperature, the base was neutralized with 0.008 mL (0.14 mmol) of AcOH. The solvent was then removed under vacuum and the crude material purified by preparative TLC with 20:1 DCM:MeOH (4 times) to provide **syn-1** (0.0068 g, 32%) and **anti-1** (0.0017, 8%). The yield for **anti-1** was calculated by ^1H NMR because of the presence of an inseparable impurity. Data for **syn-1** and **anti-1** matched that as reported above.

^{1}H and ^{13}C NMR Spectra



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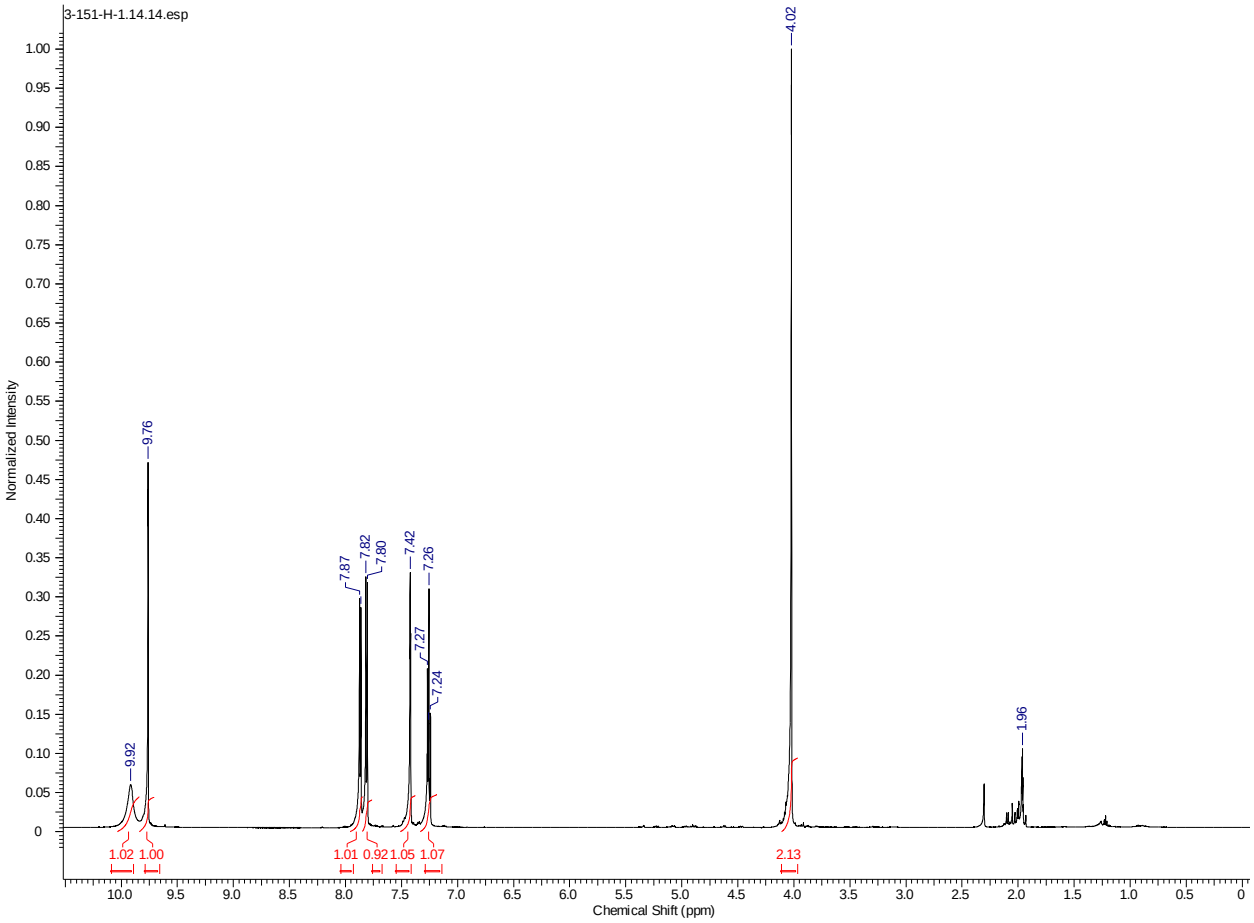

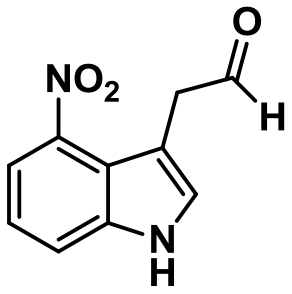
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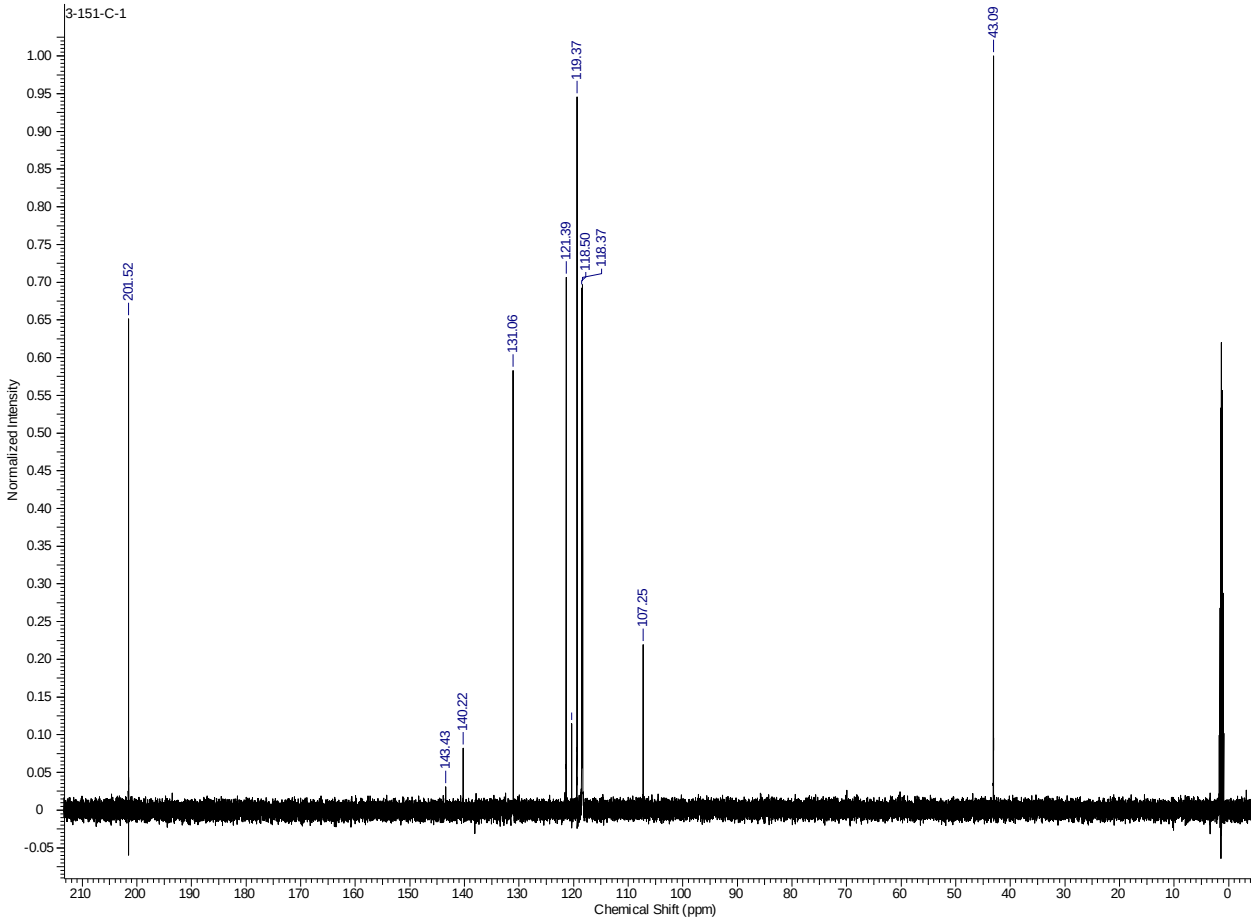
H

3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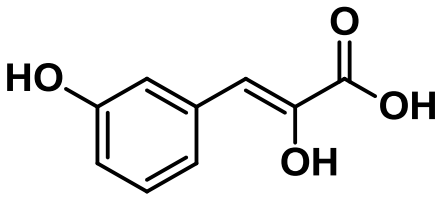
118.37
107.25
43.09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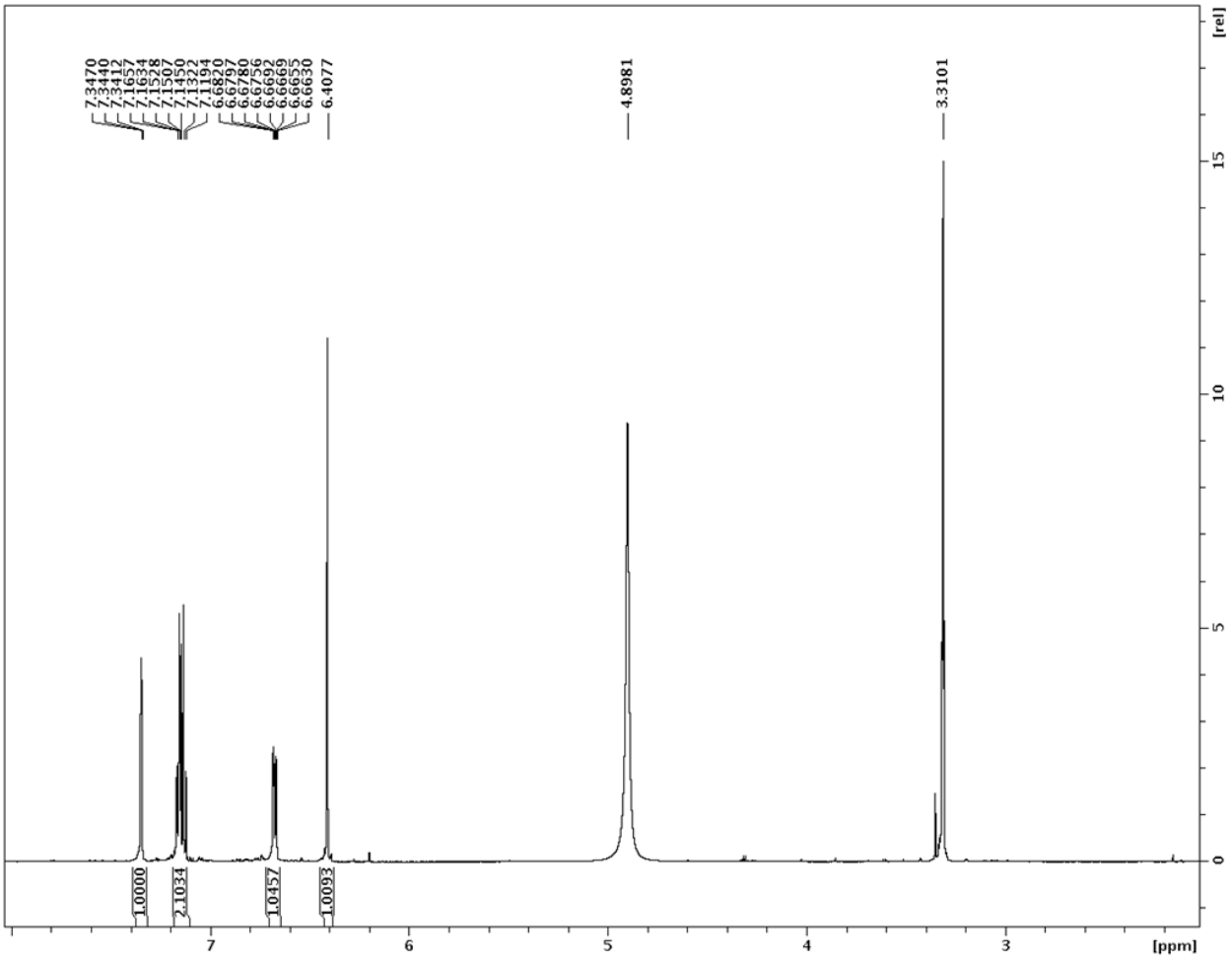
O
OH
OH

5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6.6780
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6.6692
6.6669
6.6655
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6.4077
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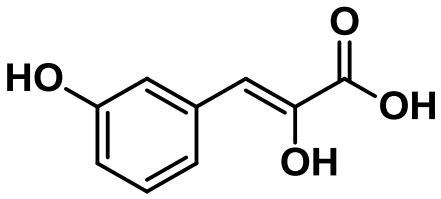
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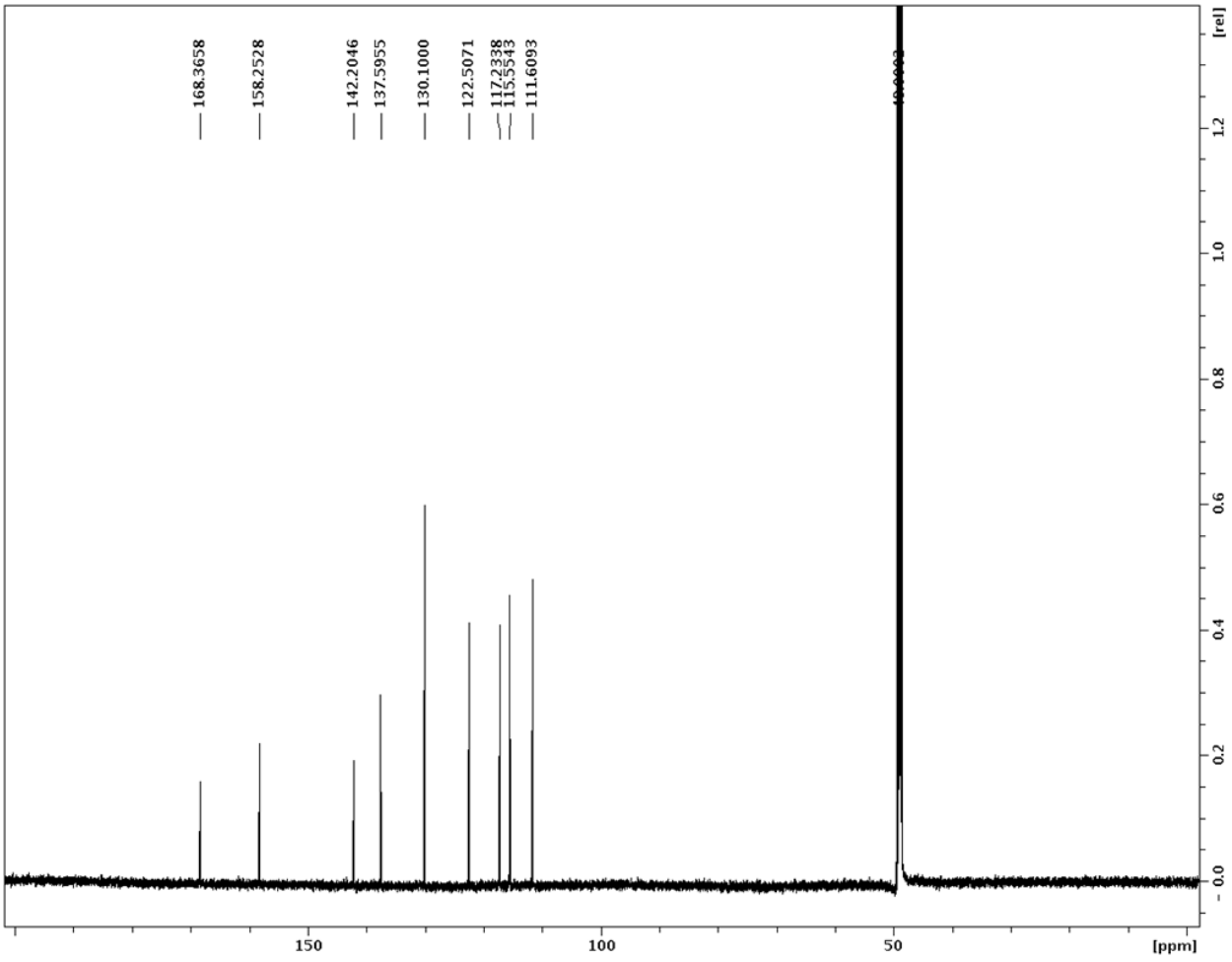
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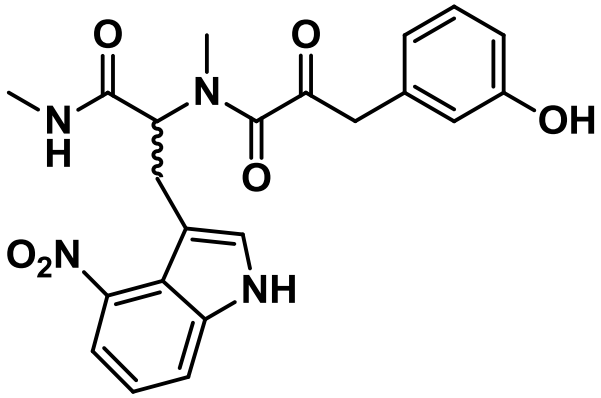
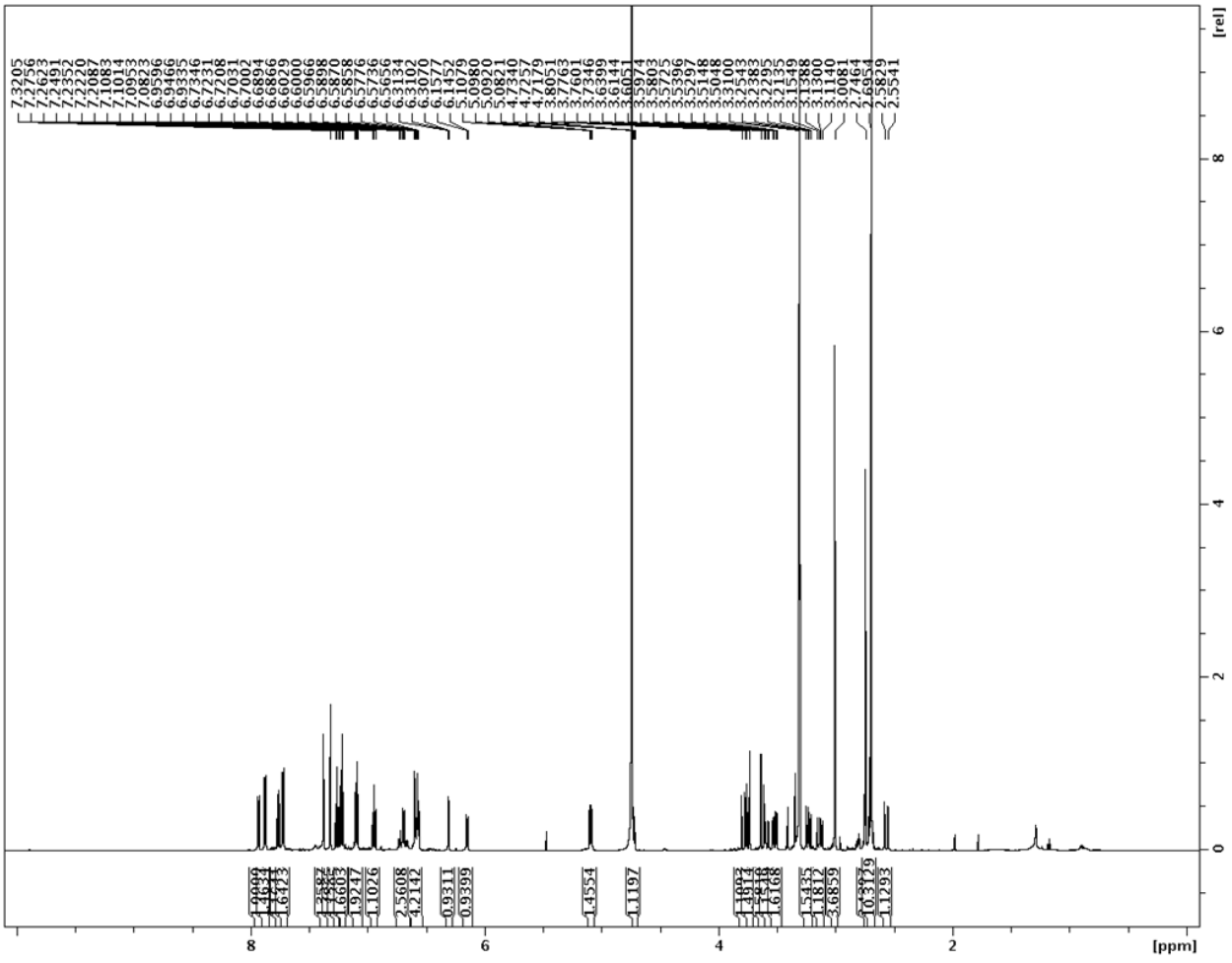
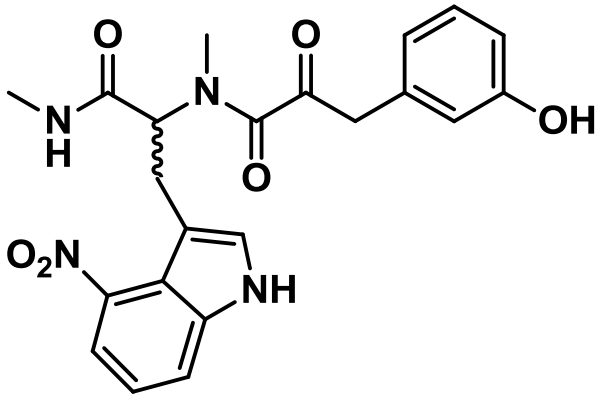
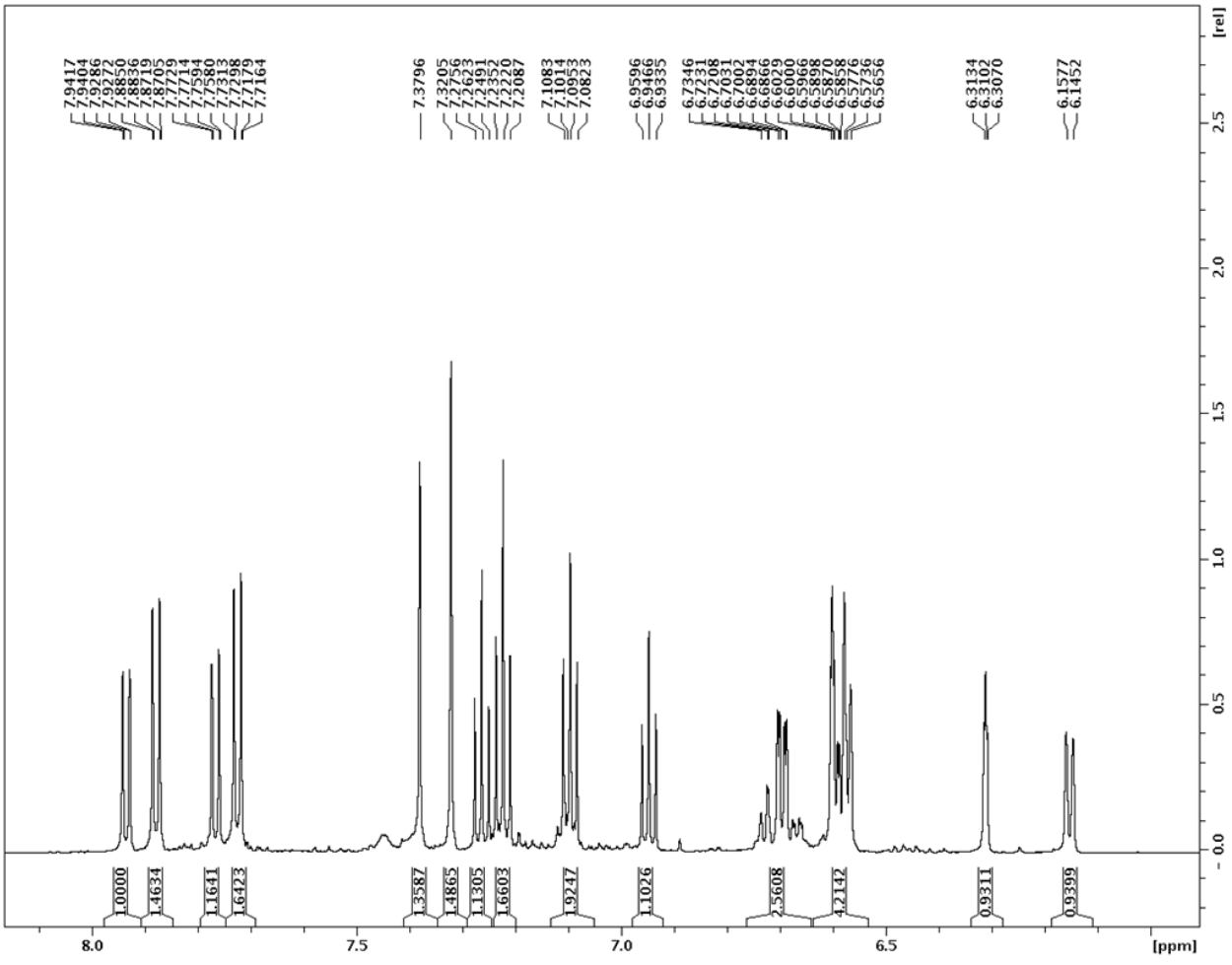


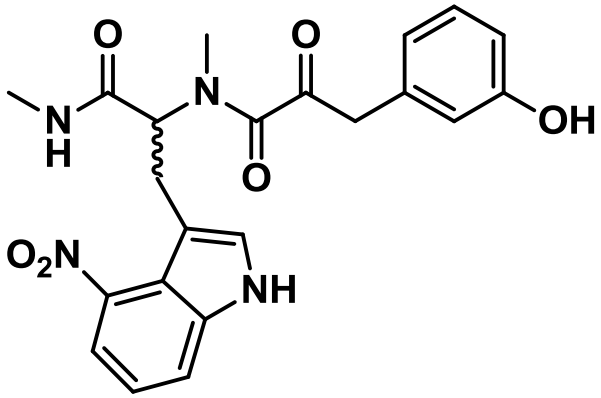
(±)-**2** as a mixture of rotamers



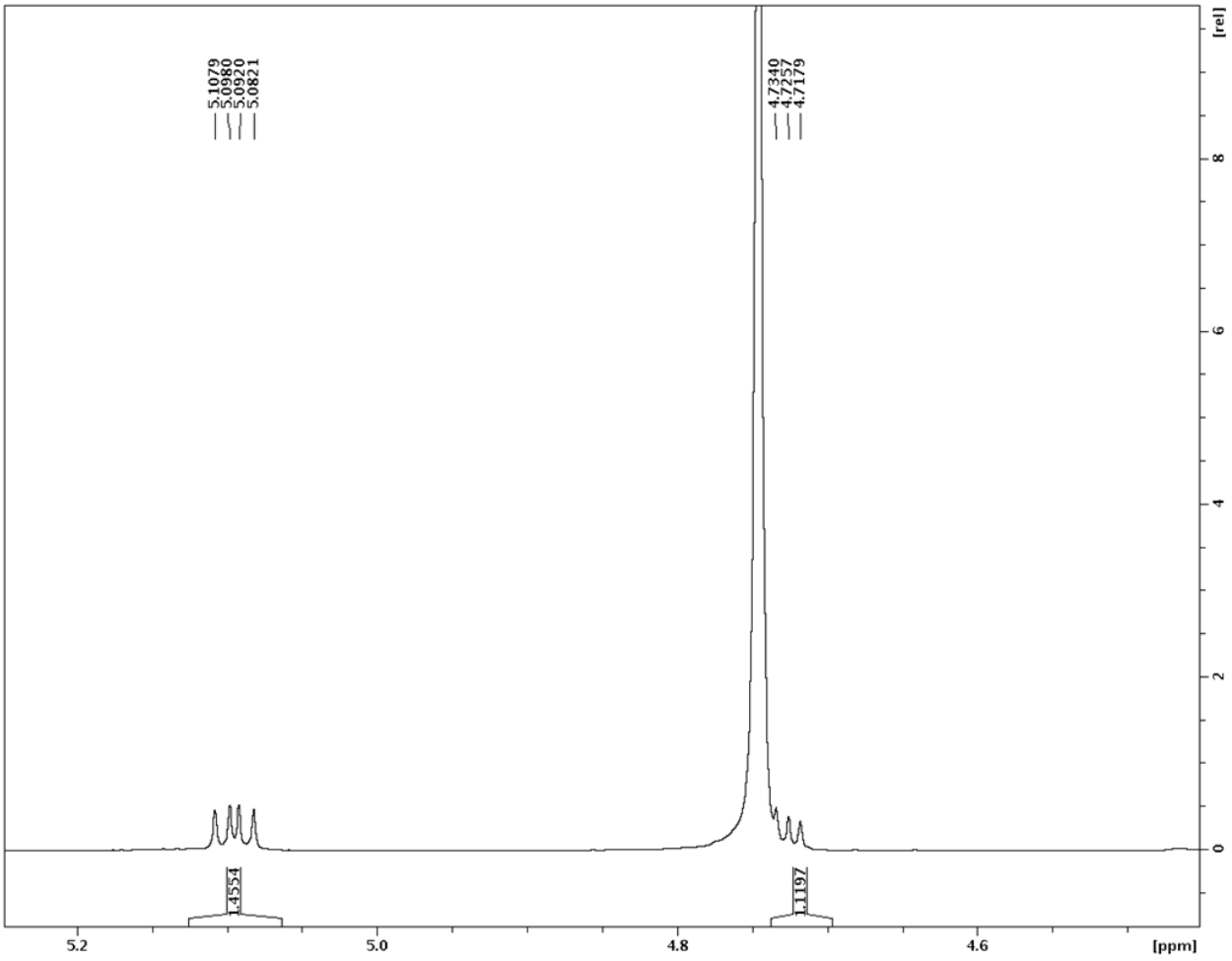


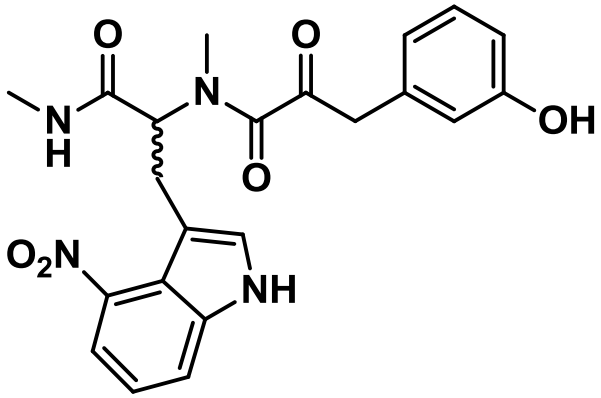
(±)-**2** expansion 8 - 6 ppm



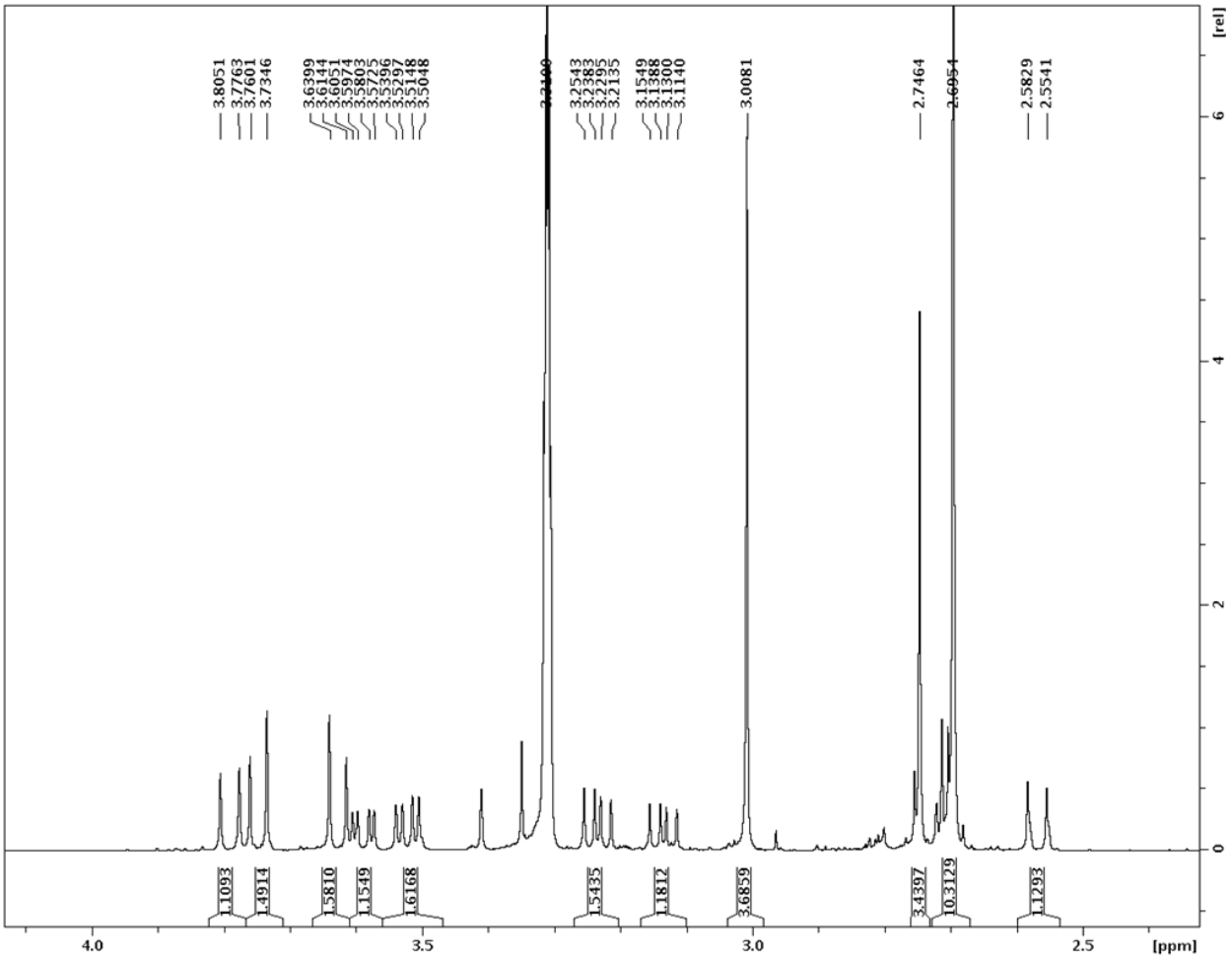


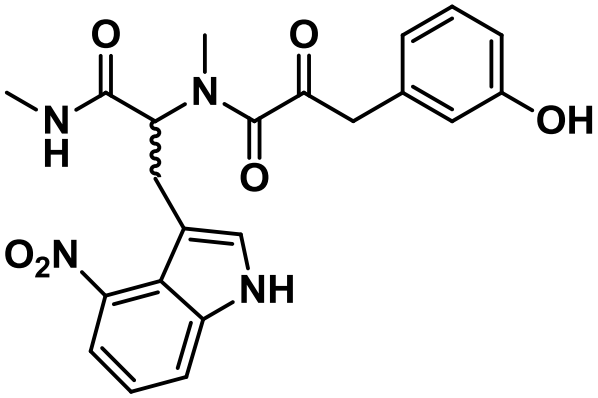
(±)-**2** expansion at 5 ppm



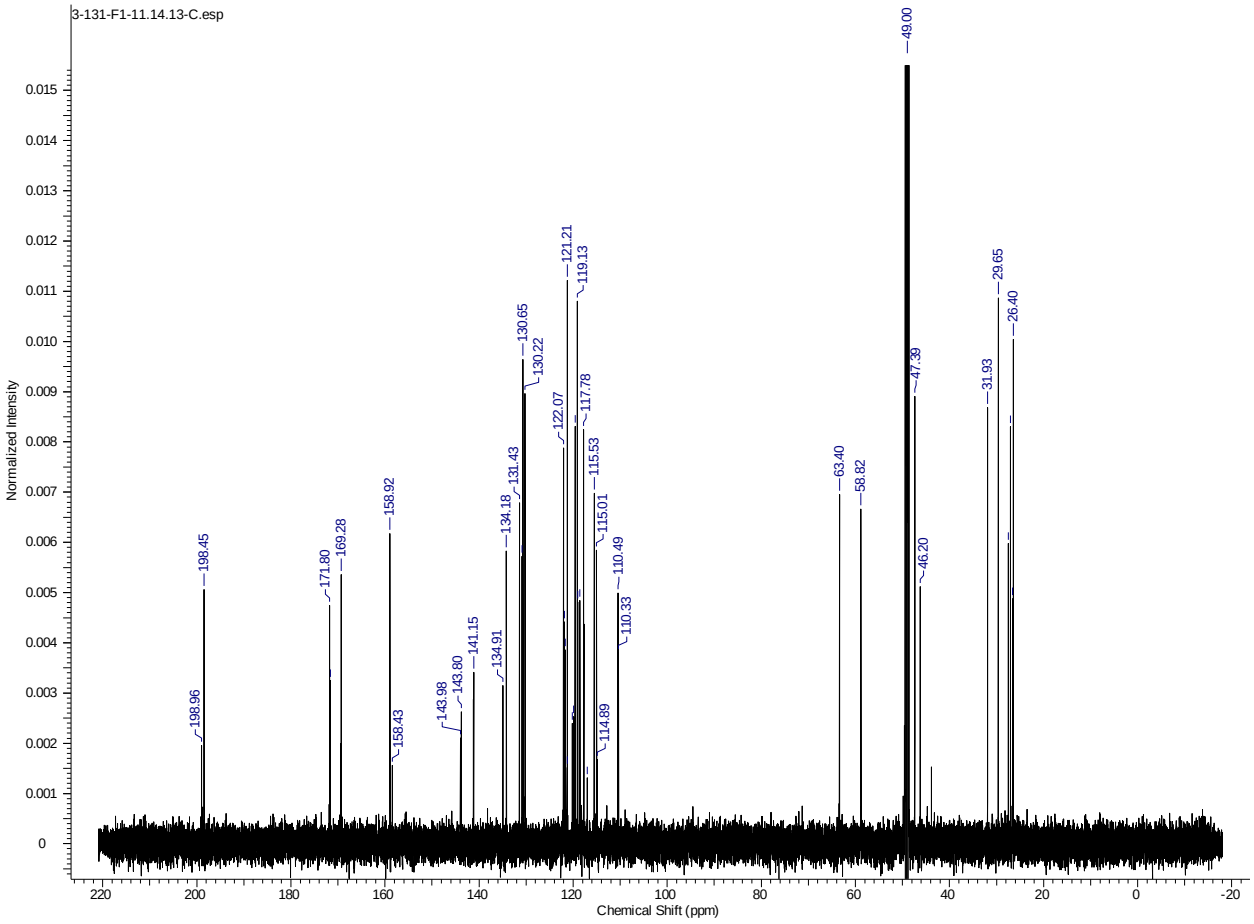


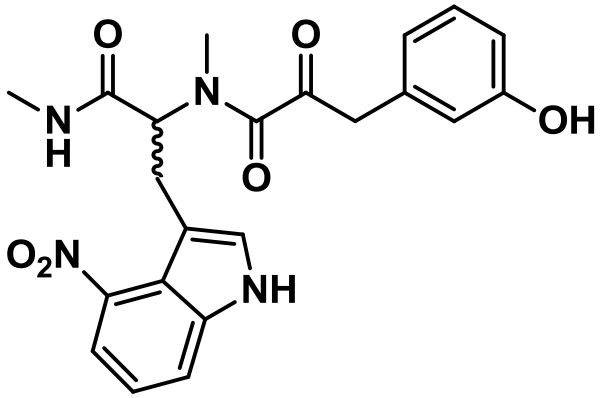
(±)-**2** expansion 4 – 2.5 ppm



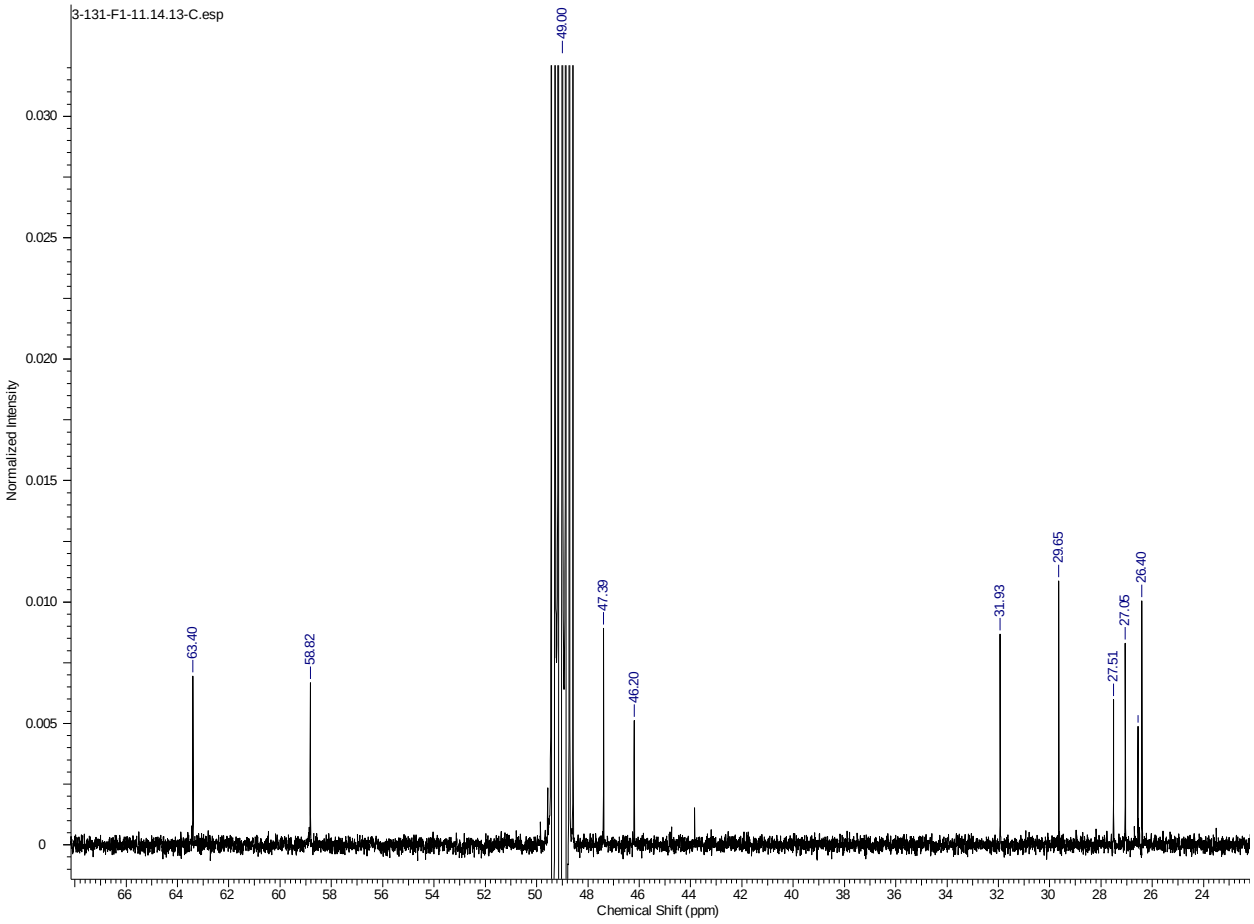


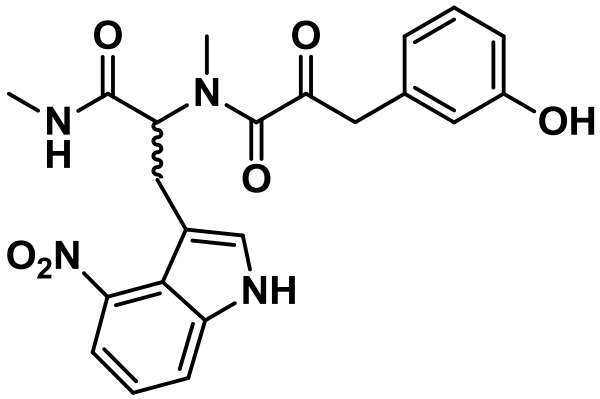
(±)-**2** as a mixture of rotamers



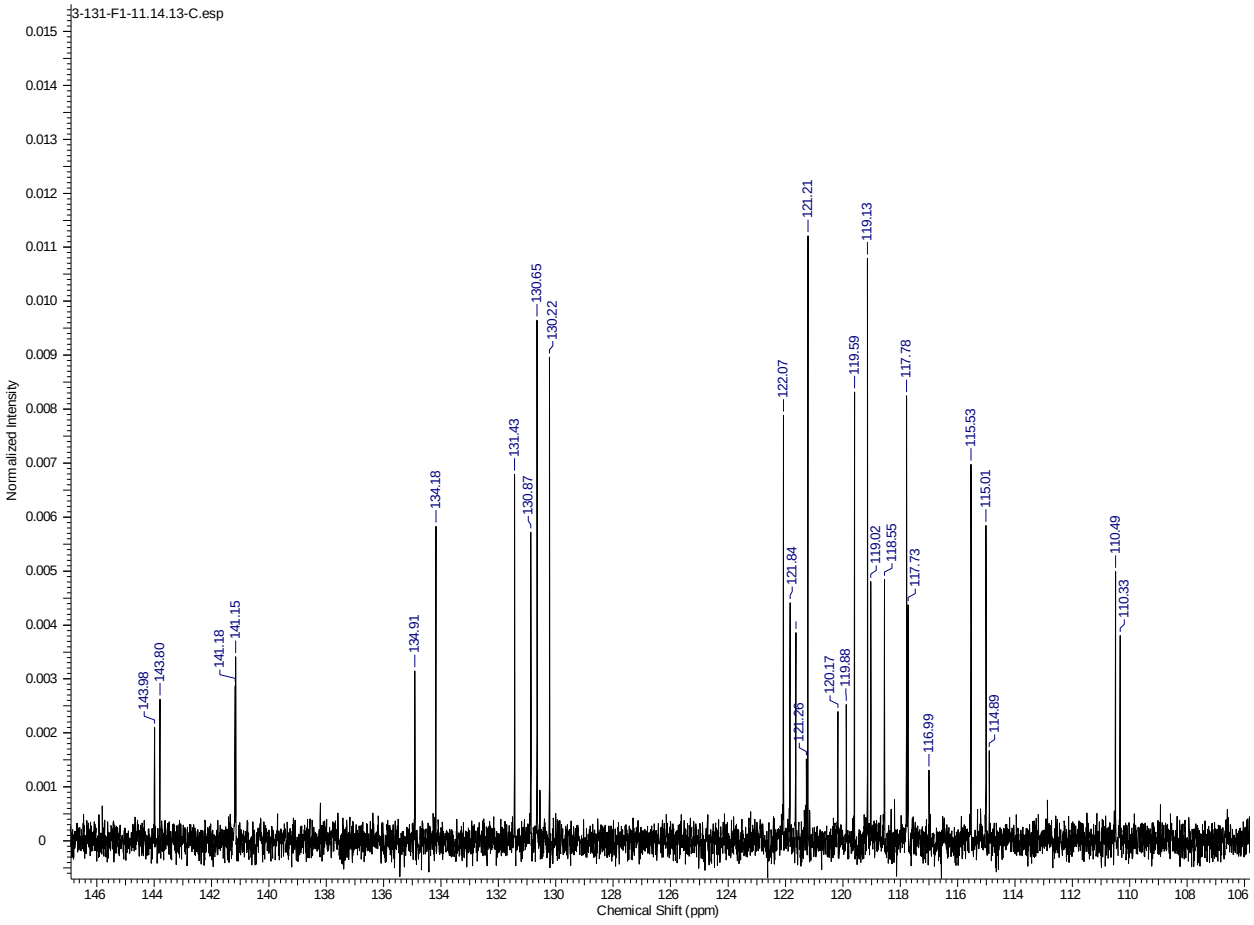


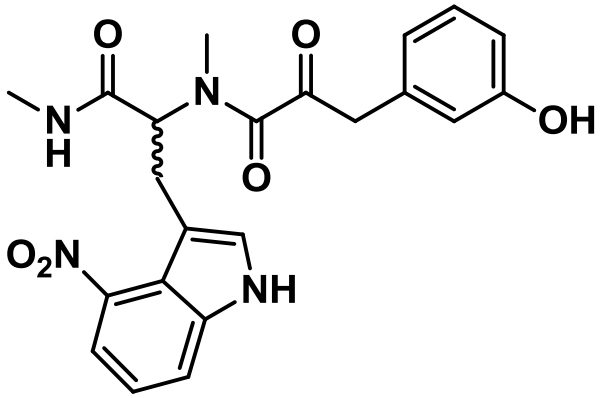
(±)-**2** expansion 66 - 24 ppm



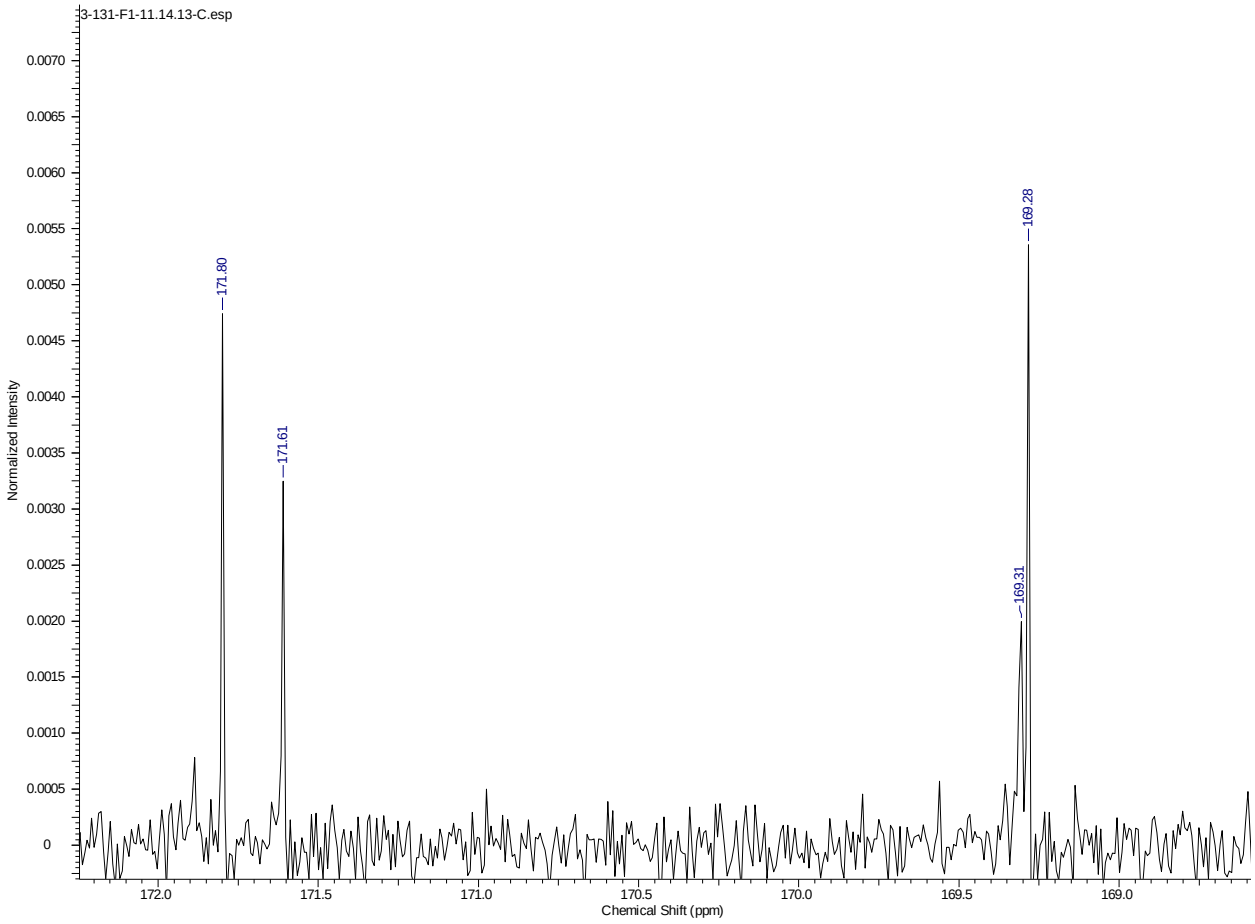


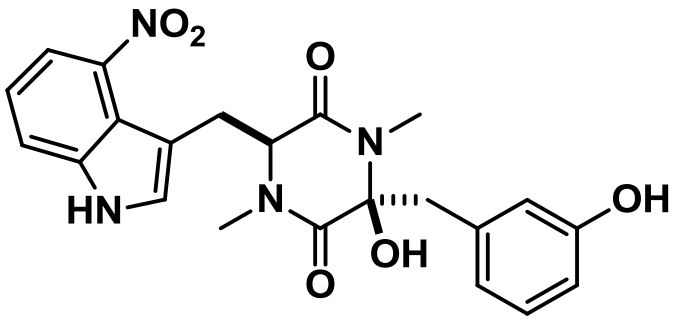
(±)-**2** expansion 146 - 106 ppm





(±)-**2** expansion 172 – 169 ppm

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HN
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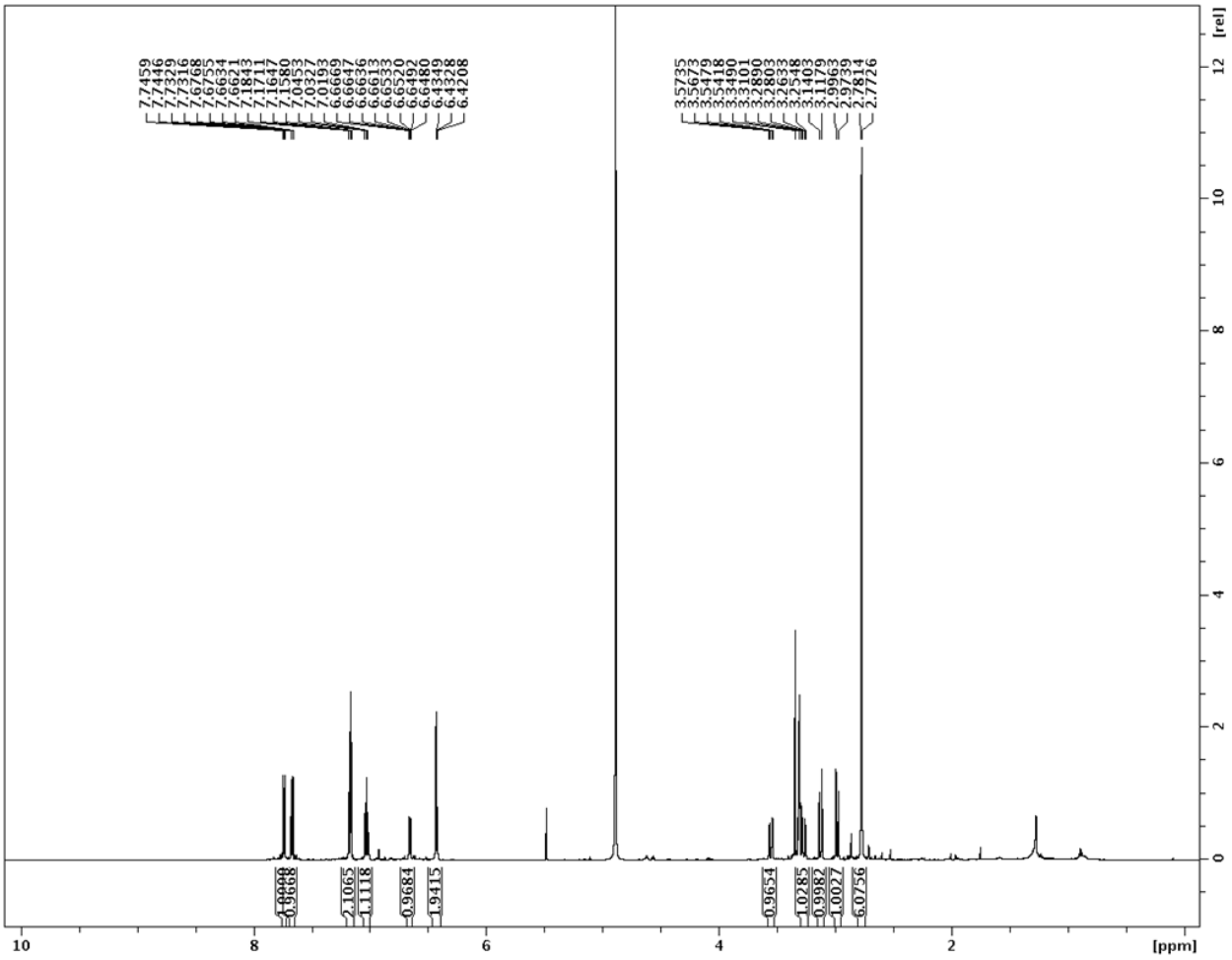
O

(±)-**anti-1**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0.9668
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1.9415
0.9654
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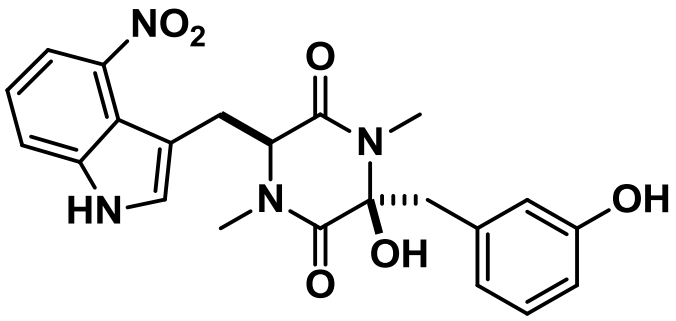
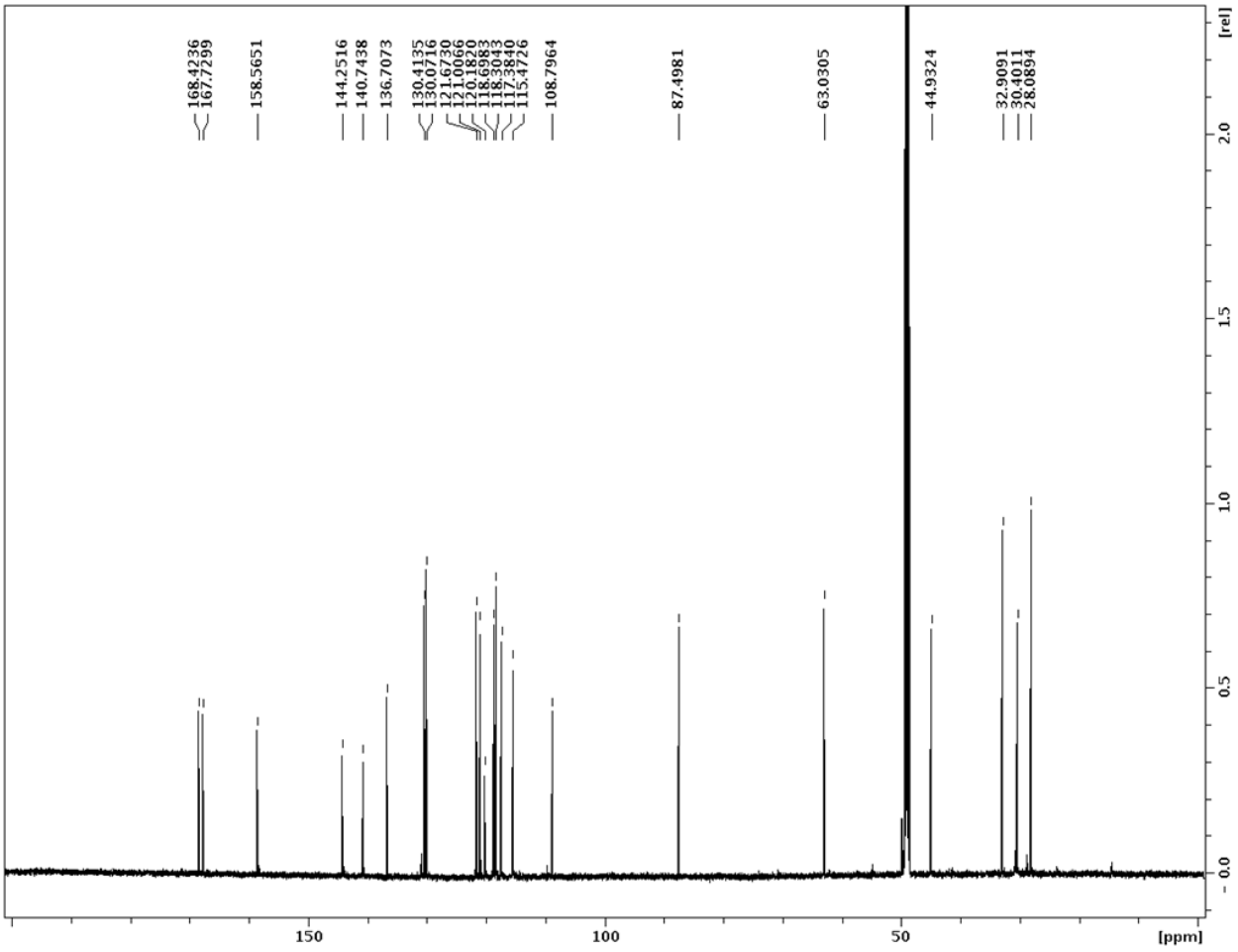

NO2
O
N
HN
N
OH
OH
O

(±)-**anti-1**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120.1820
118.6983
118.3043
117.3840
115.4726
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87.4981
63.0305
44.9324
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28.0894
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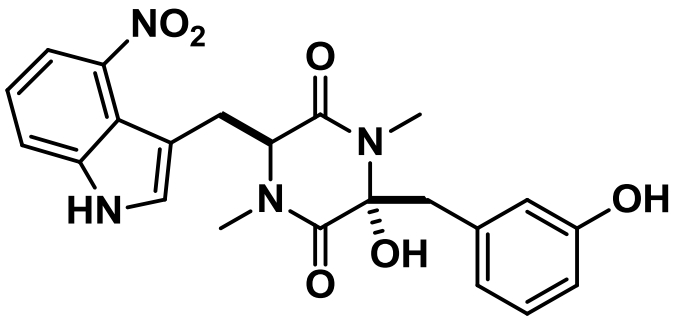
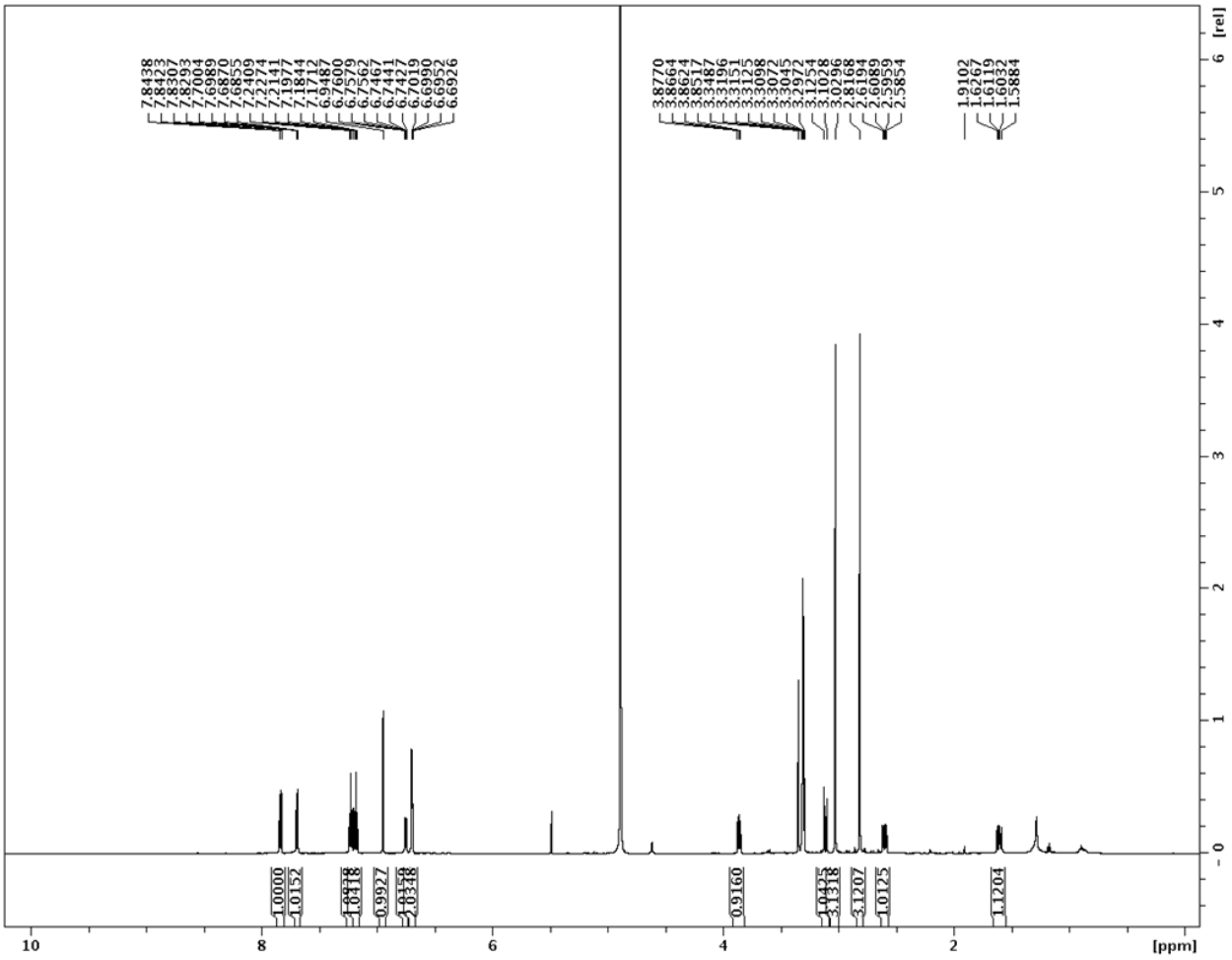

NO2
O
N
HN
N
OH
OH
O

(±)-**syn-1**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7.2274
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7.1977
7.1844
7.1712
6.9487
6.7600
6.7579
6.7562
6.7467
6.7441
6.7317
6.7019
6.6990
6.6952
6.6926
3.8770
3.8664
3.8624
3.8517
3.3487
3.3196
3.3151
3.3115
3.3068
3.3027
3.3045
3.2972
3.1254
3.1028
3.0296
2.8168
2.6194
2.6089
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2.5834
1.9102
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1.6119
1.6032
1.5884
1.0000
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1.0418
1.0418
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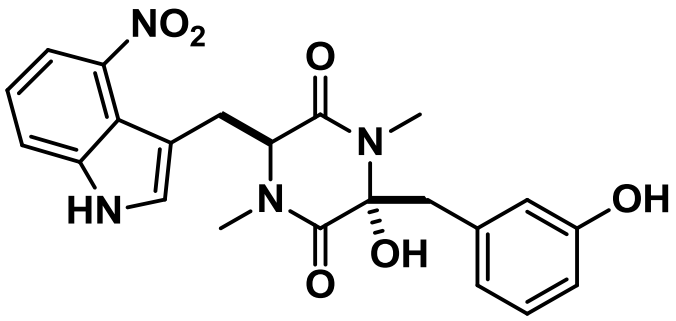
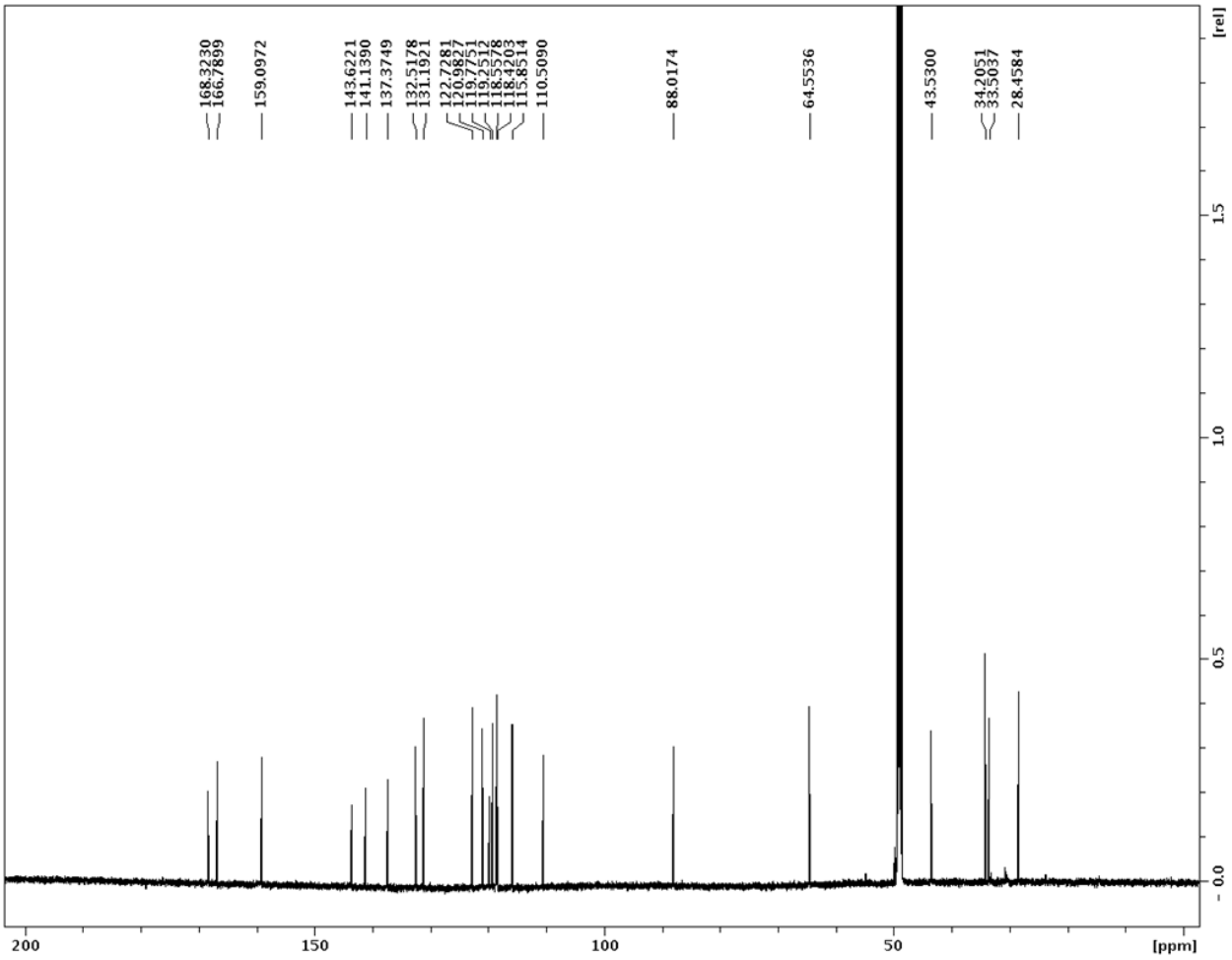
8
6
4
2
[ppm]
[rel]


NO2
O
N
HN
N
OH
OH
O

(±)-**syn-1**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118.5576
118.4203
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88.0174
64.5536
43.5300
34.2051
33.5037
28.4584
[rel]
1.5
1.0
0.5
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200
150
100
50
[ppm]

Crystallography Information for (±)-thaxtomin A (syn-1)

Crystals of x-ray crystallographic quality were obtained by slow evaporation from MeOH at 3 ^{o}C yielding orange crystals.

A yellow needle-like specimen of $C_{22}H_{22}N_4O_6$, approximate dimensions 0.015 mm x 0.018 mm x 0.172 mm, was used for the X-ray crystallographic analysis. The X-ray intensity data were measured.

The total exposure time was 92.23 hours. The frames were integrated with the Bruker SAINT software package using a narrow-frame algorithm. The integration of the data using a triclinic unit cell yielded a total of 9047reflections to a maximum θ angle of 68.67° (0.83 Å resolution), of which 3726 were independent (average redundancy 2.428, completeness = 90.9%, R_{int} = 2.25%, R_{sig} = 2.60%) and 3185 (85.48%) were greater than 2σ(F^2). The final cell constants of a = 8.1838(6) Å, b = 8.4093(6) Å, c = 18.4956(15) Å, α = 83.381(7)°, β = 77.526(4)°, γ = 63.273(5)°, volume = 1109.80(14) Å^3, are based upon the refinement of the XYZ-centroids of 3662 reflections above 20 σ(I) with 11.78° < 2θ < 136.2°. Data were corrected for absorption effects using the multi-scan method (SADABS). The ratio of minimum to maximum apparent transmission was 0.897. The calculated minimum and maximum transmission coefficients (based on crystal size) are 0.6750 and 0.7530.

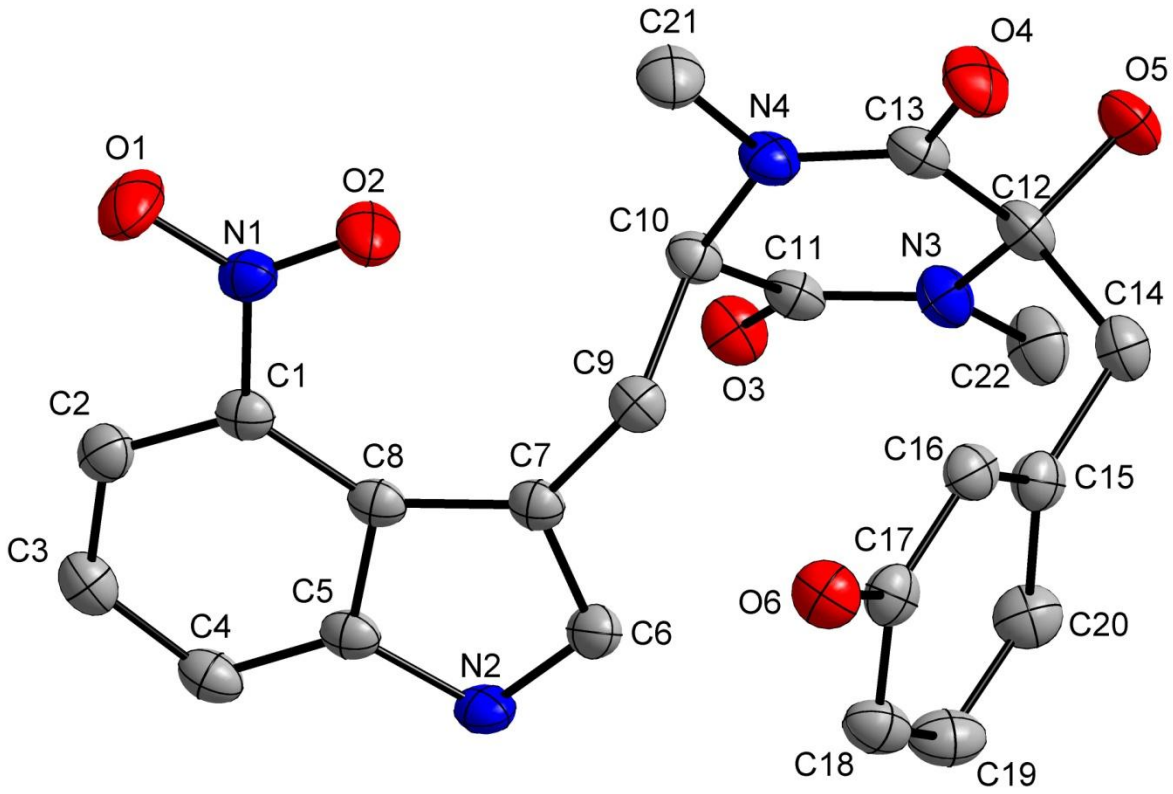
The structure was solved and refined using the Bruker SHELXTL Software Package, using the space group P -1, with Z = 2 for the formula unit, $C_{22}H_{22}N_4O_6$. The final anisotropic full-matrix least-squares refinement on F^2 with 398 variables converged at R1 = 4.06%, for the observed data and wR2 = 10.97% for all data. The goodness-of-fit was 1.041. The largest peak in the final difference electron density synthesis was 0.566 e^-/Å^{3}and the largest hole was -0.476 e^-/Å^3 with an RMS deviation of 0.042 e^-/Å^3. On the basis of the final model, the calculated density was 1.408 g/cm^3 and F(000), 496 e^-.

CCDC #	1000681
Chemical formula	$C_{22}H_{22}N_4O_6$
Formula weight	438.43
Temperature	160(2) K
Wavelength	1.54178 Å
Crystal size	0.015 x 0.018 x 0.172 mm
Crystal habit	yellow needle
Crystal system	triclinic
Space group	P -1
Unit cell dimensions	a = 8.1838(6) Å α = 83.381(7)°

	b = 8.4093(6) Å	β = 77.526(4)°
	c = 18.4956(15) Å	γ = 63.273(5)°
Volume	1109.80(14) $Å^3$	
Z	2	
Density (calculated)	1.408 Mg/cm^3	
Absorption coefficient	0.884 mm^{-1}	
F(000)	496	

Theta range for data collection	2.45 to 68.67°	
Index ranges	-9<=h<=9, -9<=k<=9, -22<=l<=21	
Reflections collected	9047	
Independent reflections	3726 [R(int) = 0.0225]	
Coverage of independent reflections	90.9%	
Absorption correction	multi-scan	
Max. and min. transmission	0.7530 and 0.6750	
Structure solution technique	direct methods	
Structure solution program	SHELXS-97 (Sheldrick, 2008)	
Refinement method	Full-matrix least-squares on F^2	
Refinement program	SHELXL-97 (Sheldrick, 2008)	
Function minimized	$\Sigma w(F_o^2 - F_c^2)^2$	
Data / restraints / parameters	3726 / 0 / 398	
Goodness-of-fit on F^2	1.041	
Δ/σ_{max}	0.001	
Final R indices	3185 data; I>2σ(I)	R1 = 0.0406, wR2 = 0.1042
	all data	R1 = 0.0482, wR2 = 0.1097
Weighting scheme	$w=1/[\sigma^2(F_o^2)+(0.0513P)^2+0.6481P]$ where $P=(F_o^2+2F_c^2)/3$	
Largest diff. peak and hole	0.566 and -0.476 $eÅ^{-3}$	
R.M.S. deviation from	**mean** 0.042 $eÅ^{-3}$	

ORTEP diagram of (±)-thaxtomin A (syn-1) with hydrogen atoms omitted



Crystallography Information for (±)-anti-1

A specimen of $C_{22.20}H_{22.80}N_4O_{6.20}$, approximate dimensions 0.050 mm x 0.136 mm x 0.137 mm, was used for the X-ray crystallographic analysis. The X-ray intensity data were measured.

The total exposure time was 28.67 hours. The frames were integrated with the Bruker SAINT software package using a narrow-frame algorithm. The integration of the data using a monoclinic unit cell yielded a total of 21585 reflections to a maximum θ angle of 59.84° (0.89 Å resolution), of which 3100 were independent (average redundancy 6.963, completeness = 100.0%, R_{int} = 2.45%, R_{sig} = 1.29%) and 2963 (95.58%) were greater than $2\sigma(F^2)$. The final cell constants of a = 13.4151(3) Å, b = 11.3424(3) Å, c = 14.8403(3) Å, β = 111.2467(10)°, volume = 2104.61(9) $Å^3$, are based upon the refinement of the XYZ-centroids of 9986 reflections above 20 σ(I) with 7.621° < 2θ < 119.5°. Data were corrected for absorption effects using the multi-scan method (SADABS). The ratio of minimum to maximum apparent transmission was 0.907. The calculated minimum and maximum transmission coefficients (based on crystal size) are 0.6820 and 0.7520.

The structure was solved with direct methods and subsequent Difference Fourier Syntheses and refinements. Non-hydrogen atoms were refined with anisotropic atomic displacement parameters, disordered non-hydrogen atoms were refined with isotropic atomic displacement parameters. H-atoms with occupancy of 0.8 or 1 were located from Difference Fourier Syntheses and refined with isotropic atomic displacement parameters. Hydrogen atoms with occupancy of 0.5 were calculated and Hydrogen atoms with occupancies less than 0.5 were ignored.

The hydroxyphenyl-group is disordered over several positions. Three different conformations were deconvoluted. Conformation "B" can be found in the crystal with a 50% probablility having the OH-group located on the same side of the molecule as the NO2 group. The aromatic rings of two conformers "A1 and A2" overlap and are refined with an occupancy of 0.5, while the OH-groups of these conformers are disordered over two positions with occupancies of 0.3 (A1) and 0.2 (A2). Stabilization of these conformers results from the different possibilities of H-bonding.
The OH-groups of conformer B and A1 form a H-bond to the keto - O5, while conformer A2 is in close contact to the free solvent molecule methanol.

The methanol molecules are disordered over an inversion center and were refined with a
scattering factor simulating 50% C and 50% O occupancy.
H-atoms with occupancy of less than 0.5 were not included in the refinement.

The final anisotropic full-matrix least-squares refinement on F^2 with 366 variables converged at R1 = 4.17%, for the observed data and wR2 = 11.06% for all data. The goodness-of-fit was 1.075. The largest peak in the final difference electron density

synthesis was 0.531 $e^-/Å^3$ and the largest hole was -0.373 $e^-/Å^3$ with an RMS deviation of 0.050 $e^-/Å^3$. On the basis of the final model, the calculated density was 1.404 g/cm^3 and F(000), 934 e^-.

Chemical formula	$C_{22.20}H_{22.80}N_4O_{6.20}$
Formula weight	444.84 g/mol
Temperature	100(2) K
Wavelength	1.54178 Å
Crystal size	0.050 x 0.136 x 0.137 mm
Crystal system	monoclinic
Space group	P 1 21/n 1
Unit cell dimensions	a = 13.4151(3) Å α = 90°
	b = 11.3424(3) Å β = 111.2467(10)°
	c = 14.8403(3) Å γ = 90°
Volume	2104.61(9) $Å^3$
Z	4
Density (calculated)	1.404 g/cm^3
Absorption coefficient	0.872 mm^{-1}
F(000)	934

Theta range for data collection	3.81 to 59.84°
Index ranges	-15<=h<=15, -11<=k<=12, -16<=l<=16
Reflections collected	21585
Independent reflections	3100 [R(int) = 0.0245]
Coverage of independent reflections	100.0%
Absorption correction	multi-scan
Max. and min. transmission	0.7520 and 0.6820
Refinement method	Full-matrix least-squares on F^2
Refinement program	SHELXL-2014 (Sheldrick, 2014)
Function minimized	$\Sigma w(F_o^2 - F_c^2)^2$
Data / restraints / parameters	3100 / 0 / 366
Goodness-of-fit on F^2	1.075
Final R indices	2963 data; I>2σ(I) R1 = 0.0417, wR2 = 0.1095
	all data R1 = 0.0431, wR2 = 0.1106
Weighting scheme	$w=1/[\sigma^2(F_o^2)+(0.0523P)^2+2.1110P]$ where $P=(F_o^2+2F_c^2)/3$

Largest diff. peak and hole	0.531 and -0.373 eÅ^{-3}
R.M.S. deviation from mean	0.050 eÅ^{-3}

ORTEP diagram of (±)-anti-1 with hydrogen atoms omitted