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Formation of cyclopropa[*c*]indole system in the course of Mn-mediated radical addition of cyclohexane-1,3-diones to N-substituted acrylamides

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Contents

1. Material and Methods

1.1. Chemistry. Melting points were determined on a Gallenkamp capillary melting point apparatus. IR spectra (ATR) were obtained with a Bruker Tensor27 spectrophotometer in the 400-4000 cm^{-1} range with 2 cm^{-1} resolutions. ^{1}H NMR and ^{13}C NMR spectra were recorded on a Varian Mercury-400 High performance Digital FT-NMR and Varian Oxford NMR300 spectrometers. High Resolution Mass Time-of-Flight spectra (TOF) were measured on an Agilent 1200/6210 LC/MS spectrophotometer. X-ray analysis was performed on an Bruker APEX II QUAZAR.. Thin layer chromatography (TLC) was performed on Merck aluminum-packed silica gel plates. Purification of products was performed by column chromatography on silica gel (Merck silica gel 60, 40-60 μm) or preparative TLC on silica gel of Merck ($PF_{254-366\ nm}$). All solvents (toluene, acetic acid, chloroform, ethyl acetate and hexane) were used with highest purity and anhydrous. *N*-Phenylmethacrylamide **2a** [S1], *N*-benzylmethacrylamide **2b** [S1], *N*-phenylacrylamide **2c** [S2] and 3,*N*-diphenylacrylamide **2d** [S3] were prepared by according to literature.

1.2. General Procedure for $Mn(OAc)_3$ Based Reaction. Salt $Mn(OAc)_3 \cdot 2H_2O$ (3 mmol, 0.85 g, 90 %) in acetic acid (10 ml) was heated in oil bath to 80 °C until it dissolved. At this temperature a solution of 1,3-dicarbonyl compound (2 mmol) and alkene (1 mmol) in acetic acid (5 ml) was added. The reaction was completed when the dark brown colour of the solution converted to yellow (30-60 min), and disappearance of the alkene was monitored by TLC. Water (20 ml) was added, and the products were extracted with $CHCl_3$ (3x20 ml). The combined organic phases were neutralized with saturated $NaHCO_3$ solution, dried over anhydrous Na_2SO_4 and evaporated. The crude products were purified by column chromatography on silica gel or preparative TLC using n-hexane/EtOAc as eluent.

1.2.1. 2,6,6-Trimethyl-4-oxo-*N*-phenyl-2,3,4,5,6,7-hexahydrobenzofuran-2-carboxamide (3a). Yield 57% (171 mg) as colorless oil. IR (ATR): 692, 750, 1028, 1232, 1398, 1527, 1597, 1625, 1679, 2931, 2958, 3309; δ_H (400 MHz, $CDCl_3$) 1.29 (3H, s), 1.30 (3H, s), 1.86 (3H, s), 2.41 (2H, s), 2.55 (2H, t, *J* = 2.0 Hz), 2.98 (1H, d, *J* = 15.6 Hz), 3.46 (1H, d, *J* = 15.6 Hz), 7.32 (1H, t, *J* = 7.6 Hz), 7.52 (2H, t, *J* = 7.6 Hz), 7.72 (1H, d, *J* = 7.6 Hz), 8.25 (1H, s, NH); δ_C (100 MHz, $CDCl_3$) 25.7, 28.4, 28.9, 34.2, 37.0, 37.9, 50.9, 77.2, 91.4, 112.1, 119.9, 124.9, 129.1, 136.8, 170.9, 172.7, 194.5; HRMS (m/z); $(M+H)^+$, $C_{18}H_{21}NO_3$: found 300.15941, calculated: 300.15822.

1.2.2. 1a,5,5-Trimethyl-3-phenyl-1,1a,5,6-tetrahydro-2*H*-cyclopropa[*c*]indole-2,7(3*H*)-dione (4a) Yield 20% (56 mg) as colorless solid mp: 120-123°C. IR (ATR): 692, 741, 1666, 1695, 1720, 2886, 2926, 2942; δ_H (400 MHz, $CDCl_3$) 1.15 (3H, s), 1.16 (3H, s), 1.47 (1H, d, *J* = 3.6 Hz), 1.60 (3H, s), 2.07 (1H, d, *J* = 3.6 Hz), 2.47 (1H, dd, *J* = 16.0, 0.8Hz), 2.40 (1H, d, *J* = 16.0 Hz), 5.05 (1H, s), 7.26 (2H, d, *J* = 6.0 Hz), 7.36 (1H, t, *J* = 7.2 Hz), 7.46 (2H, t, *J* = 7.6

Hz); δ_C (100 MHz, $CDCl_3$) 10.1, 28.5, 30.3, 30.9, 33.4, 35.7, 39.3, 54.6, 109.1, 126.9, 127.9, 129.3, 134.3, 139.8, 173.0, 206.0; HRMS (m/z); $(M+H)^+$, $C_{18}H_{19}NO_2$: found 282.15021, calculated: 282.14885.

1.2.3. 2-Methyl-4-oxo-*N*-phenyl-2,3,4,5,6,7-hexahydrobenzofuran-2-carboxamide (3b)Yield 35% (95 mg) colorless solid mp: 43-46°C. IR (ATR): 685, 760, 1025, 1236, 1410, 1520, 1575, 1642, 1678, 2922, 2961, 3321; δ_H (400 MHz, $CDCl_3$) 1.62 (3H, s), 1.98 (2H, m), 2.27 (2H, m), 2.45 (2H, m), 2.73 (1H, dt, *J* = 15.2, 2.0 Hz), 3.23 (1H, dt, *J* = 15.2, 2.0 Hz), 7.06 (1H, t, *J* = 7.6 Hz), 7.25 (2H, t, *J* = 8.8 Hz), 7.47 (2H, t, *J* = 7.6 Hz), 8.12 (1H, s, NH); δ_C (100 MHz, $CDCl_3$) 21.5, 23.9, 25.7, 36.4, 37.1, 91.1, 113.3, 120.0, 124.8, 129.0, 136.8, 170.9, 174.0, 195.2; HRMS (m/z); $(M+H)^+$, $C_{16}H_{17}NO_3$: found 272.12872, calculated: 272.128120.

1.2.4. 1a-Methyl-3-phenyl-1,1a,5,6-tetrahydro-2*H*-cyclopropa[*c*]indole-2,7(3*H*)-dione (4b) Yield 22% (58 mg) brown solid mp: 103-105°C. IR (ATR): 744, 794, 1598, 1669, 1699, 1710, 2944, 2996, 3080; δ_H (400 MHz, $CDCl_3$) 1.46 (1H, d, *J* = 3.6 Hz), 1.59 (3H, s), 2.04 (1H, d, *J* = 3.6 Hz), 2.38-2.67 (4H, m), 5.16 (1H, dd, *J* = 7.2, 2.0 Hz, H_{olef}), 7.25 (2H, dd, *J* = 7.6, 1.6 Hz), 7.33 (1H, tt, *J* = 7.6, 1.6 Hz), 7.43 (2H, td, *J* =7.6, 1.6 Hz); δ_C (100 MHz, $CDCl_3$) 10.0, 20.6, 30.9, 36.4, 39.2, 39.4, 98.1, 126.9, 127.9, 129.2, 134.3, 141.1, 173.0, 206.4; HRMS (m/z); $(M+H)^+$, $C_{16}H_{15}NO_2$: found 254.11852, calculated: 254.11755.

1.2.5. N-Benzyl-2,6,6-trimethyl-4-oxo-2,3,4,5,6,7-hexahydrobenzofuran-2-carboxamide (3c) Yield 26% (82 mg) colorless solid mp: 98-100°C. IR (ATR): 667, 768, 1034, 1267, 1454, 1547, 1580, 1640, 1667, 2934, 2967, 3323; δ_H (400 MHz, $CDCl_3$) 1.05 (3H, s), 1.08 (3H, s), 1.64 (3H, s), 2.19 (2H, s), 2.28 (2H, s), 2.75 (1H, dt, *J* = 15.2, 2.0 Hz), 3.23 (1H, dt, *J* = 15.2, 2.0 Hz), 4.40 (1H, dd, *J* = 14.4, 5.6 Hz), 4.48 (1H, dd, *J* = 14.4, 5.6 Hz), 6.94 (1H, t, *J* = 5.6 Hz, NH), 7.28 (5H, m); δ_C (100 MHz, $CDCl_3$) 25.5, 28.5, 28.6, 34.1, 36.9, 37.7, 43.1, 50.8, 77.3, 91.4, 111.6, 127.4, 127.5, 128.7, 137.8, 172.7, 173.4, 194.5; HRMS (m/z); $(M+H)^+$, $C_{19}H_{24}NO_3$: found 315.17888, calculated: 315.18289.

1.2.6. 3-Benzyl-1a,5,5-trimethyl-1,1a,5,6-tetrahydro-2*H*-cyclopropa[*c*]indole-2,7(3*H*)-dione (4c) Yield 4% (12 mg) yellow solid mp: 97-99°C. IR (ATR): 693, 729, 1603, 1668, 2868, 2955, 3032; δ_H (400 MHz, $CDCl_3$) 1.04 (3H, s), 1.15 (3H, s), 1.26 (1H, d, *J* = 3.2 Hz), 1.53 (3H, s), 1.93 (1H, d, *J* = 3.2 Hz), 2.40 (1H, dd, *J* = 16.0, 0.8 Hz), 2.30 (1H, d, *J* = 16.0 Hz), 4.38 (1H, d, *J* = 15.2 Hz), 4.76 (1H, d, *J* = 15.2 Hz), 5.05 (1H, s, H_{olef}), 7.20 (5H, m); δ_C (100 MHz, $CDCl_3$) 10.0, 18.4, 28.5, 30.4, 31.5, 33.5, 35.8, 39.1, 43.4, 54.6, 58.3, 108.8, 127.3, 127.5, 128.7, 136.2, 138.3, 173.9, 206.3; HRMS (m/z); $(M+H)^+$, $C_{19}H_{21}NO_2$: found 296.16568, calculated: 296.16450.

1.2.7. 6,6-Dimethyl-4-oxo-*N*-phenyl-2,3,4,5,6,7-hexahydrobenzofuran-2-carboxamide (3d) Yield 14% (40 mg) yellow solid mp: 144-146°C. IR (ATR): 689, 756, 1039, 1278, 1465, 1549, 1589, 1624, 1700, 2957, 2990, 3317; δ_H (400 MHz, $CDCl_3$) 1.13 (6H, s), 2.25 (2H, s), 2.41 (2H, s), 3.15 (1H, dd, *J* = 15.2, 7.2 Hz), 3.25 (1H, t, *J* = 15.2 Hz), 5.24 (1H, dd, *J* = 15.2,

7.2 Hz), 7.15 (1H, t, *J* = 7.6 Hz), 7.34 (2H, t, *J* = 8.4 Hz), 7.54 (2H, d, *J* = 8.0 Hz), 8.01 (1H, s, NH); δ_C (100 MHz, $CDCl_3$) 28.4, 28.9, 30.3, 34.2, 37.6, 50.9, 82.3, 112.1, 120.1, 125.0, 129.11, 129.15, 130.6, 168.2, 174.1, 194.4; HRMS (m/z); $M+H)^+$, $C_{17}H_{19}NO_3$: found 286.14426, calculated: 286.143770.

1.2.8. 6,6-Dimethyl-4,8-dioxo-*N*-phenylspiro[2.5]octane-1-carboxamide (5) Yield 20% (53 mg) yellow oil. IR (ATR): 692, 744, 1595, 1667, 1702, 2894, 3059, 3079; δ_H (400 MHz, $CDCl_3$) 1.05 (3H, s),1.16 (3H, s), 2.04 (1H, dd, *J* = 8.8, 3.6 Hz), 2.25 (1H, dd, *J* = 8.4, 3.6 Hz), 2.60 (4H, m), 2.85 (1H, t, *J* = 8.8 Hz), 7.09 (1H, t, *J* = 7.6 Hz), 7.29 (2H, t, *J* = 7.6 Hz), 7.45 (2H, d, *J* = 8.0 Hz), 7.79 (1H, s, NH); δ_C (100 MHz, $CDCl_3$) 24.2, 28.0, 28.6, 30.0, 43.9, 44.1, 52.5, 53.9, 120.3, 124.7, 128.90, 128.96, 137.2, 164.1, 204.4, 205.0; HRMS (m/z); $(M+H)^+$, $C_{17}H_{19}NO_3$: found 286.14463, calculated: 286.143770.

1.2.9. Ethyl 2,5-dimethyl-5-(phenylcarbamoyl)-4,5-dihydrofuran-3-carboxylate (6) Yield 51% (147 mg) pale yellow solid mp: 58-60°C. IR (ATR): 690, 750, 1062, 1242, 1523, 1597, 1662, 1701, 2985, 3315; δ_H (400 MHz, $CDCl_3$) 1.26 (3H, t, *J* = 6.8 Hz), 1.66 (3H, s), 2.30 (3H, t, *J* = 1.6 Hz), 2.88 (1H, dq, *J* = 15.6, 1.6 Hz), 3.36 (1H, dq, *J* = 15.6, 1.6 Hz), 4.15 (1H, dq, *J* = 7.6, 1.6 Hz, -OCH_2-), 4.17 (1H, dq, *J* = 7.6, 1.6 Hz, -OCH_2'-), 7.13 (1H, t, *J* = 7.2 Hz), 7.34 (2H, t, *J* = 8.8 Hz), 7.55 (2H, d, *J* = 7.6 Hz), 8.14 (1H, s, NH); δ_C (100 MHz, $CDCl_3$) 14.2, 14.3, 25.5, 40.6, 59.8, 87.6, 103.0, 119.8, 124.7, 129.0, 136.9, 164.6, 165.3, 171.7; HRMS (m/z); $(M+H)^+$, $C_{16}H_{19}NO_4$: found 290.13661, calculated: 290.13868.

1.2.10. 6,6-Dimethyl-4-oxo-*N*,2-diphenyl-2,3,4,5,6,7-hexahydrobenzofuran-3-carboxamide (7) Yield 46% (166 mg) colorless solid mp: 126-128°C). IR (ATR): 689, 786, 1017, 1278, 1502, 1565, 1589, 1638, 1679, 2941, 2994, 3315; δ_H (400 MHz, $CDCl_3$) 1.13 (3H, s), 1.55 (3H, s), 2.35 (2H, s), 2.45 (2H, d, *J* = 1.2 Hz), 4.12 (1H, d, *J* = 6.0 Hz), 6.57 (1H, d, *J* = 6.0 Hz), 7.06 (1H, t, *J* = 7.6 Hz), 7.33 (7H, m), 7.60 (2H, dd, *J* = 7.2, 1.2 Hz), 10.17 (1H, s, NH); δ_C (100 MHz, $CDCl_3$) 28.46, 28.48, 34.2, 38.0, 50.7, 54.4, 87.2, 109.4, 119.5, 123.8, 125.3, 128.5, 128.84, 128.88, 138.4, 139.9, 167.8, 178.9, 196.6; HRMS (m/z); $(M+H)^+$, $C_{23}H_{23}NO_3$: found 362.17654, calculated: 362.17507.

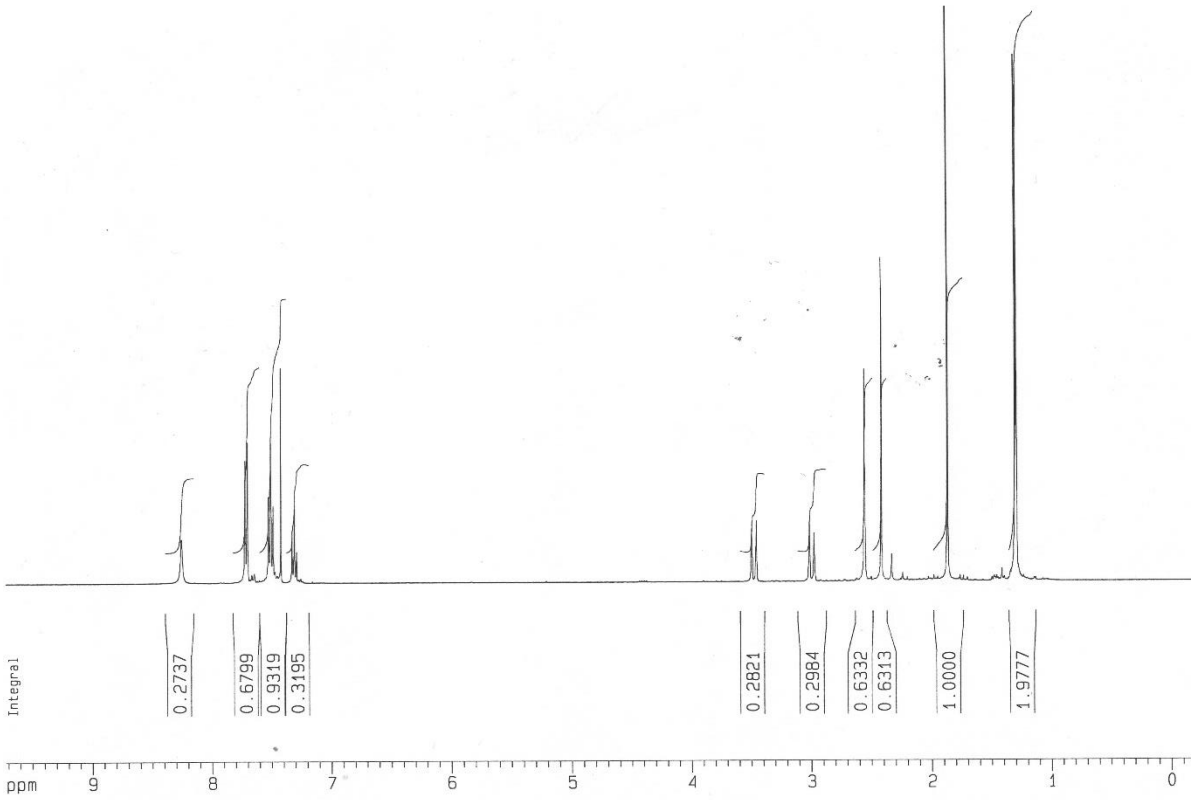
References

[S1] (a) R. A. Jacobson, *J. Am. Chem. Soc.*, 1945, **67**, 1998-1999; (b) B. Bentov, E. Reichmann, *J. Am. Chem. Soc.*, 1952, **74**, 845-847; (c) B. A. Abel, C. L. McCormick, *Macromolecules.*, 2016, **49**, 465-474.

[S2] I. L. Honigberg, W. H. Hartung, *J. Org. Chem.*, 1960, **25**, 1822-1824.

[S3] B. Narasimhan, D. Belsare, D. Pharande, V. Mourya, A. Dhake, *Eur. J. Med. Chem*., 2004, **39**, 827-834.

Compound 3a ^{1}H NMR



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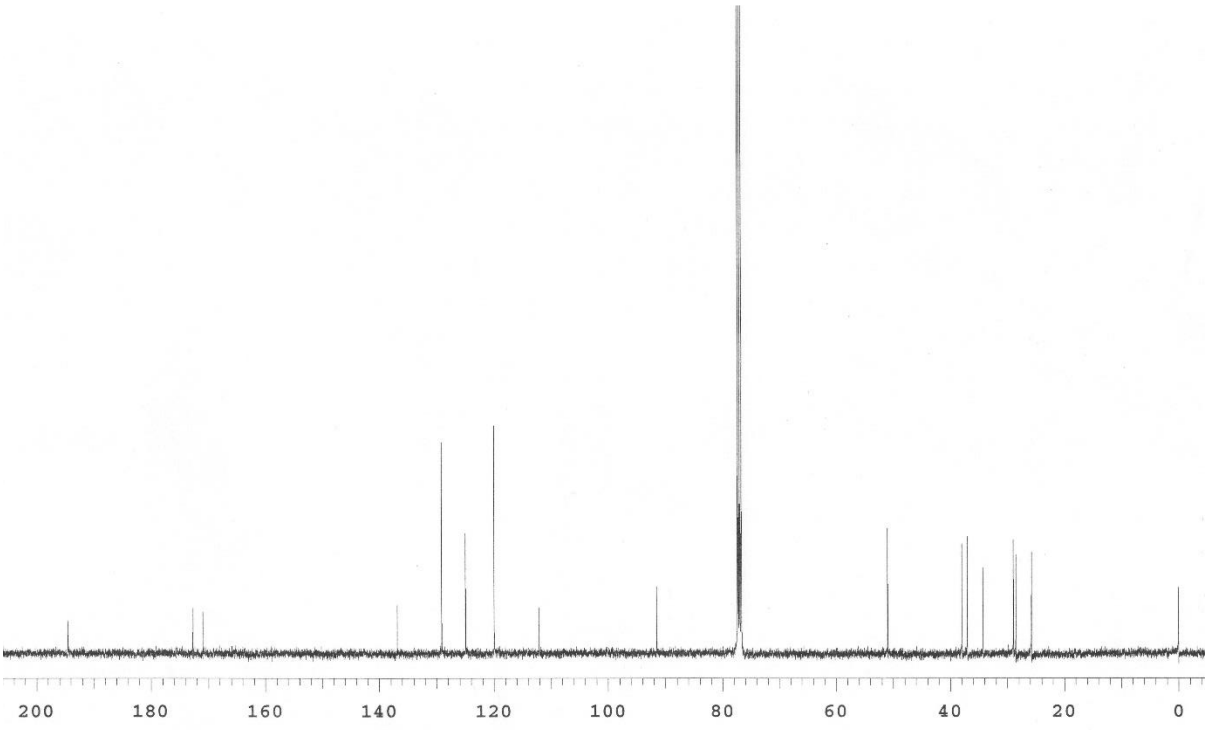

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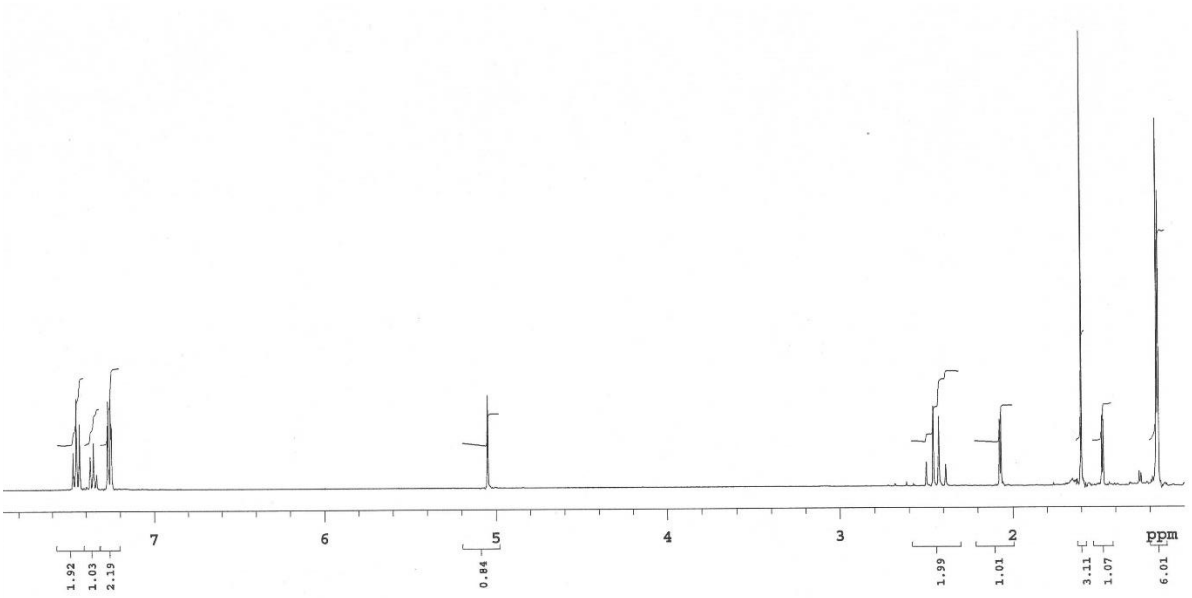

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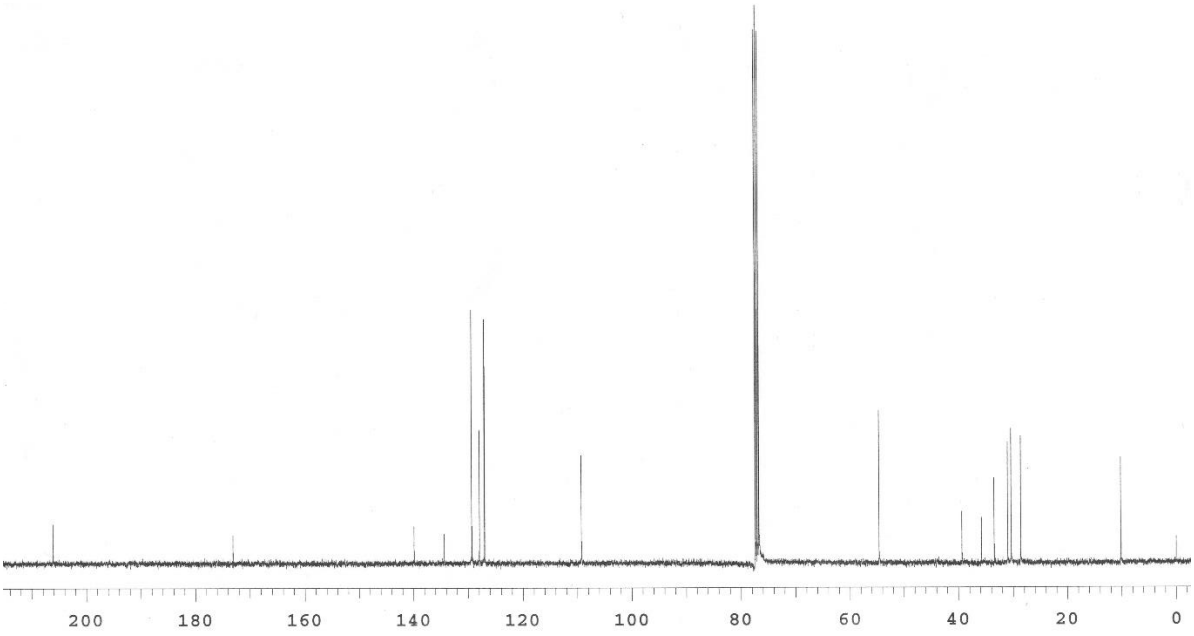

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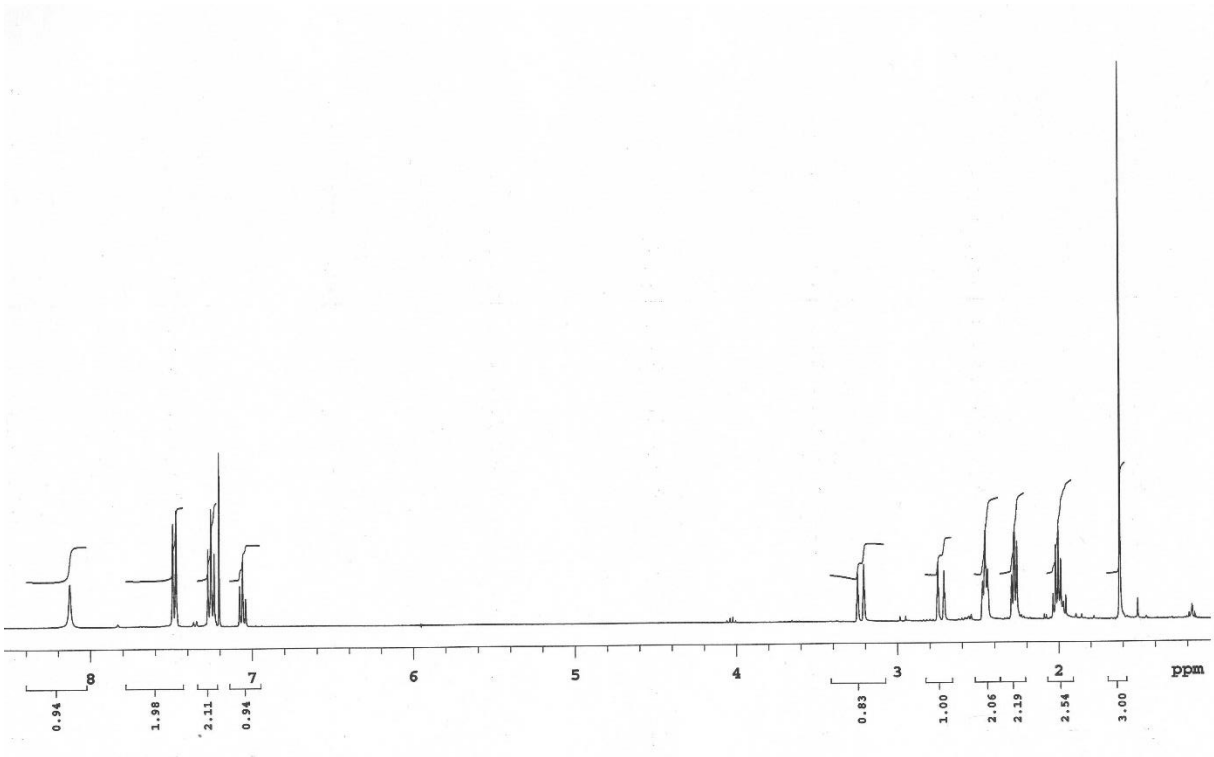

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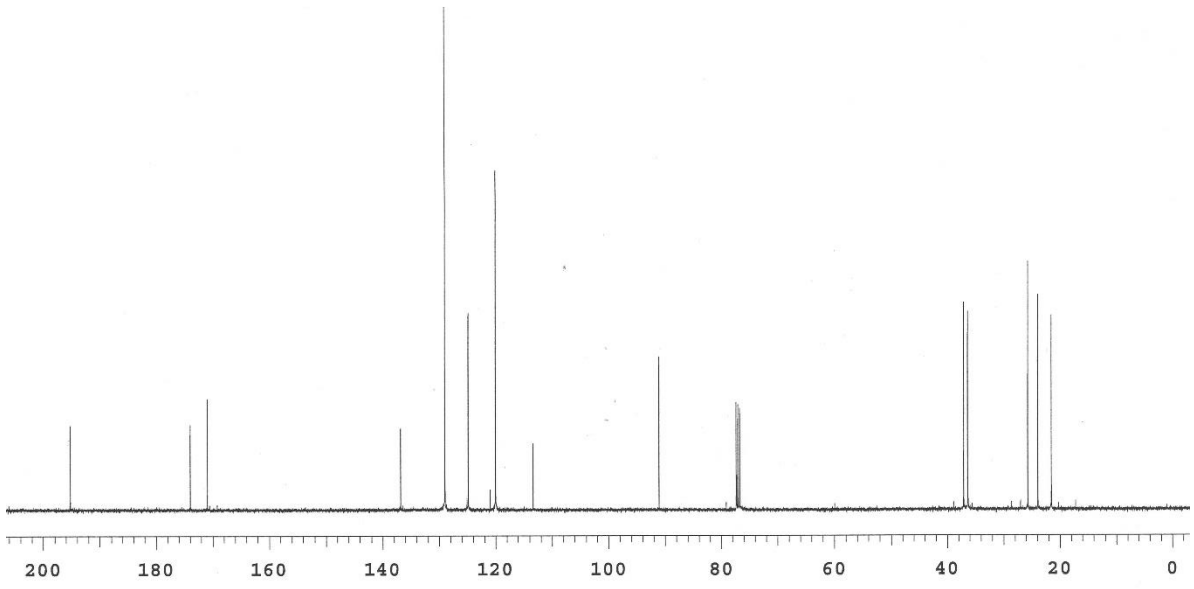

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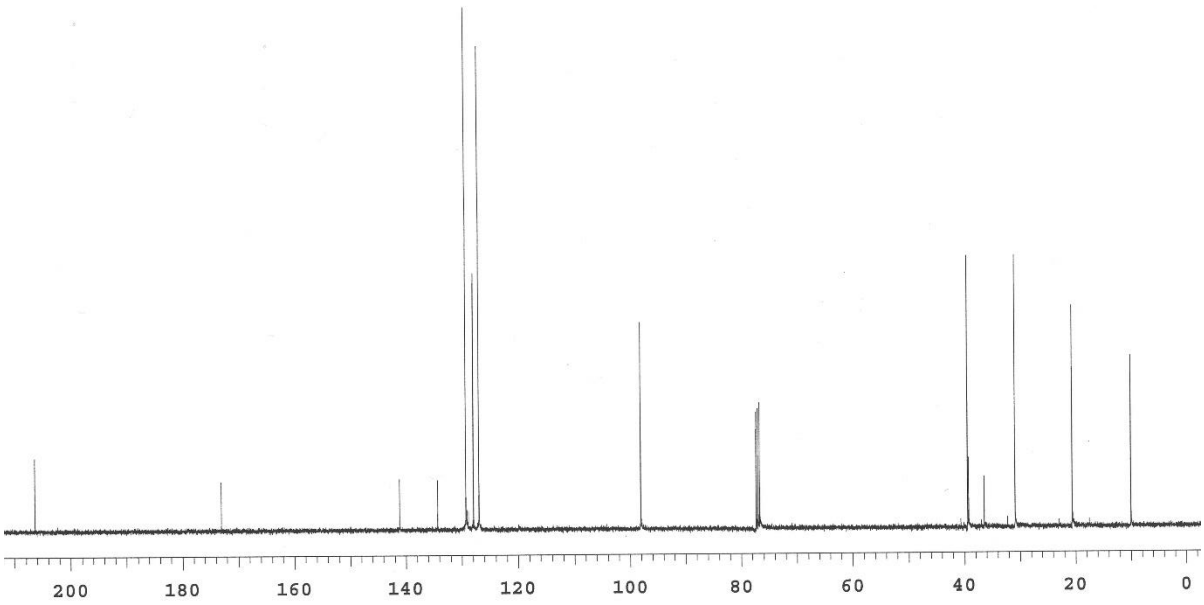
Compound 4b ^{13}C NMR



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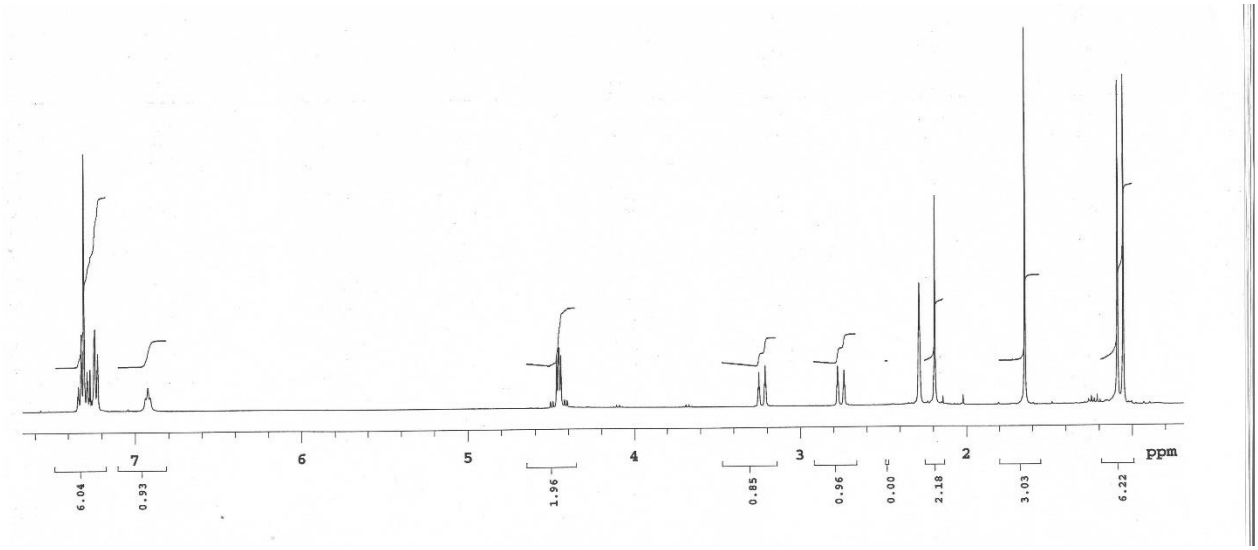

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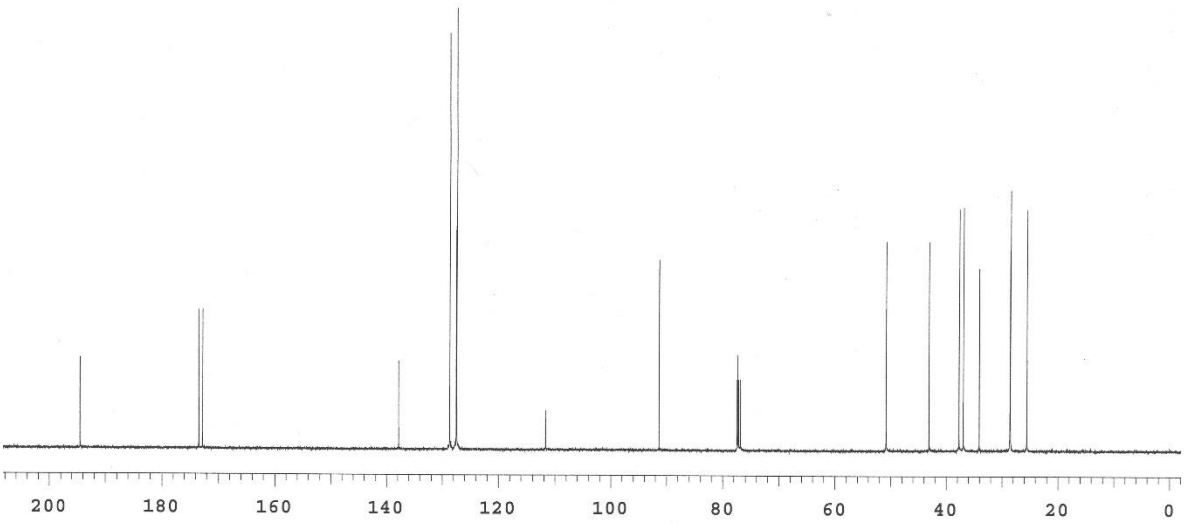

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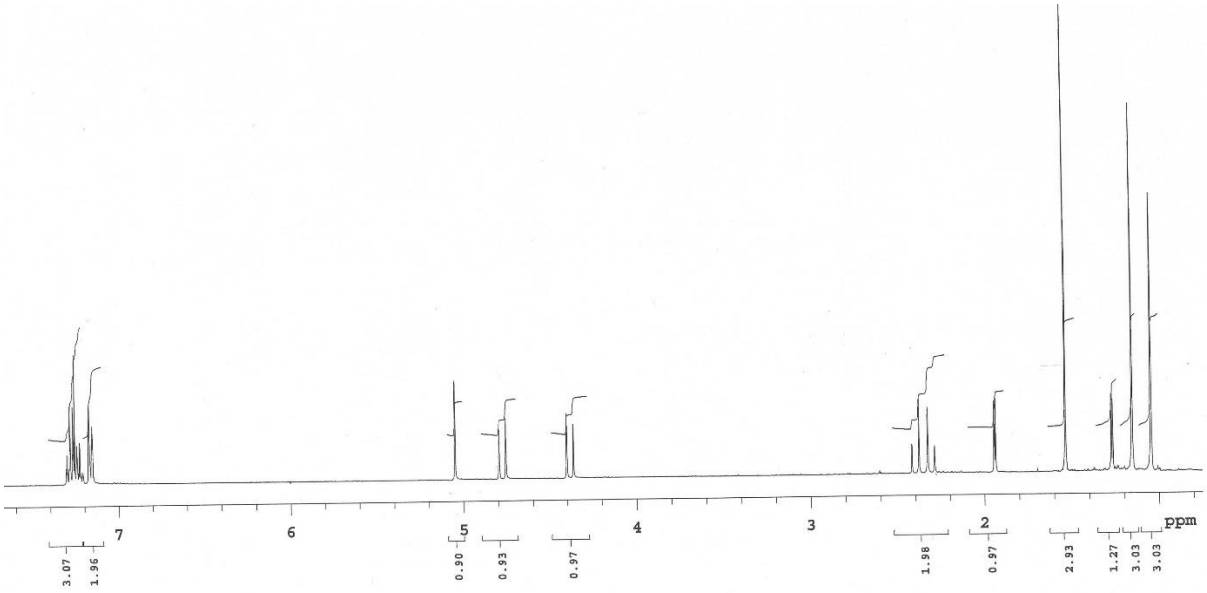

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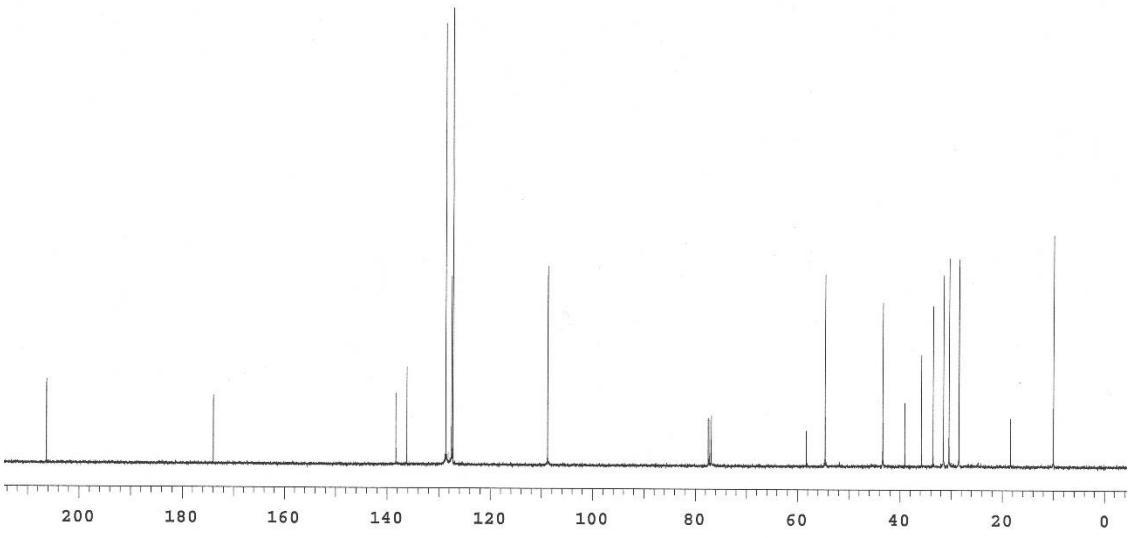

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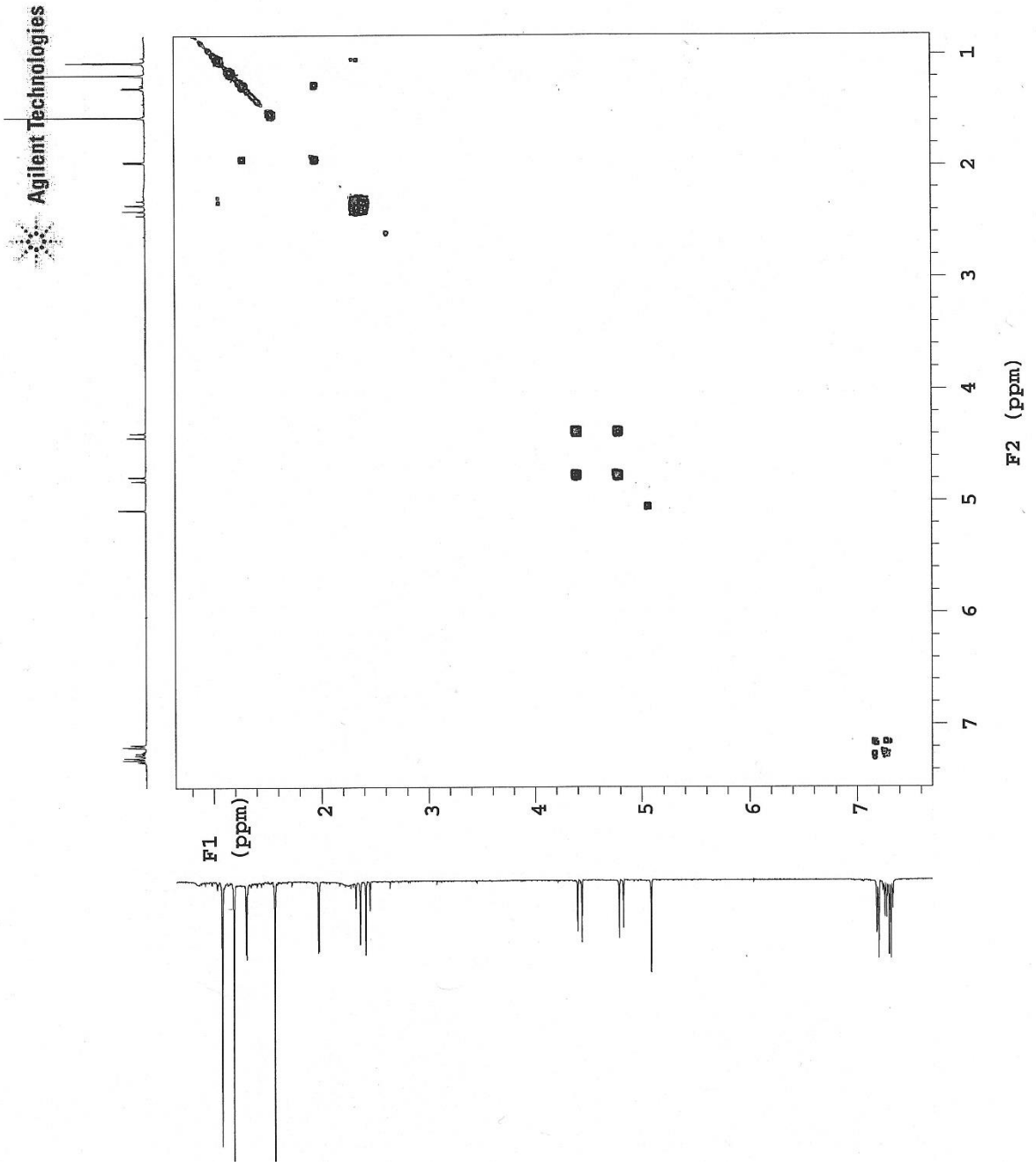

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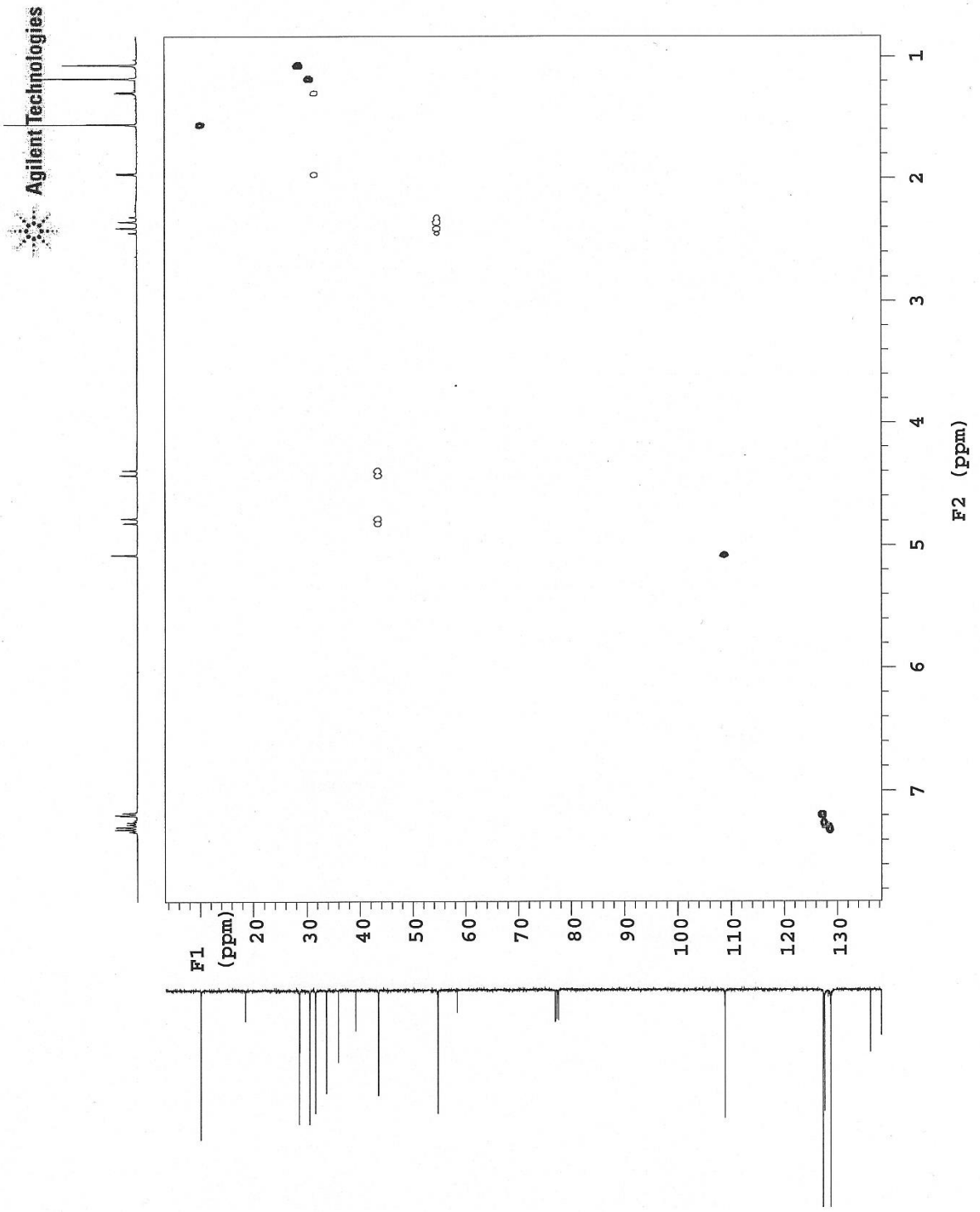

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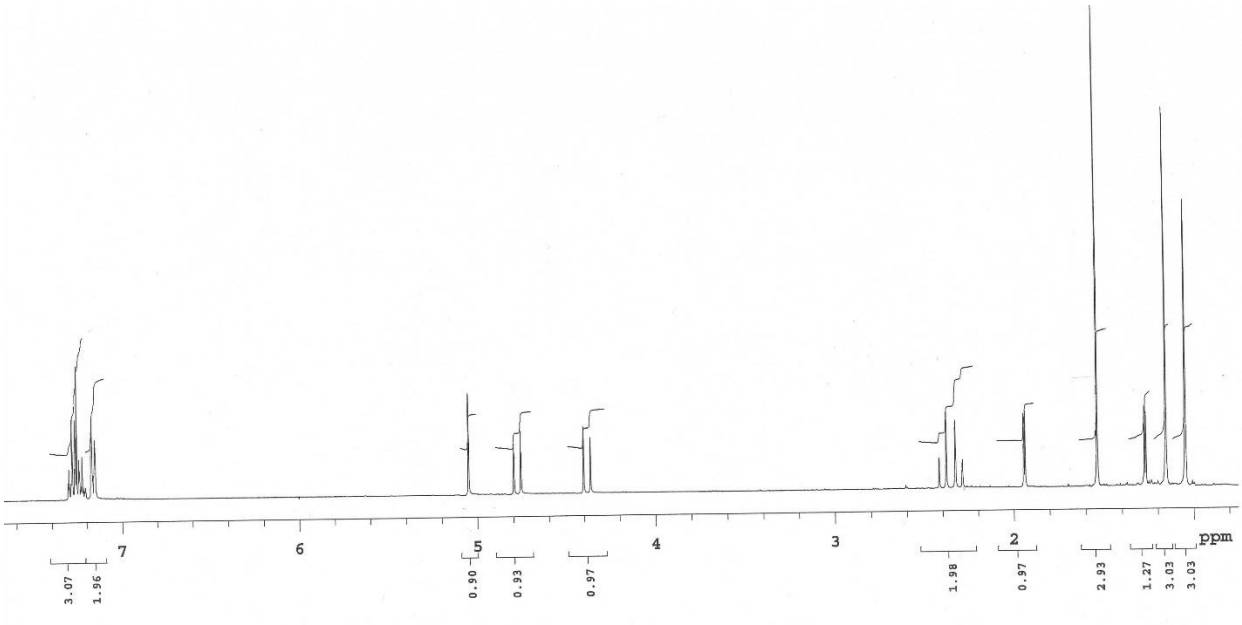

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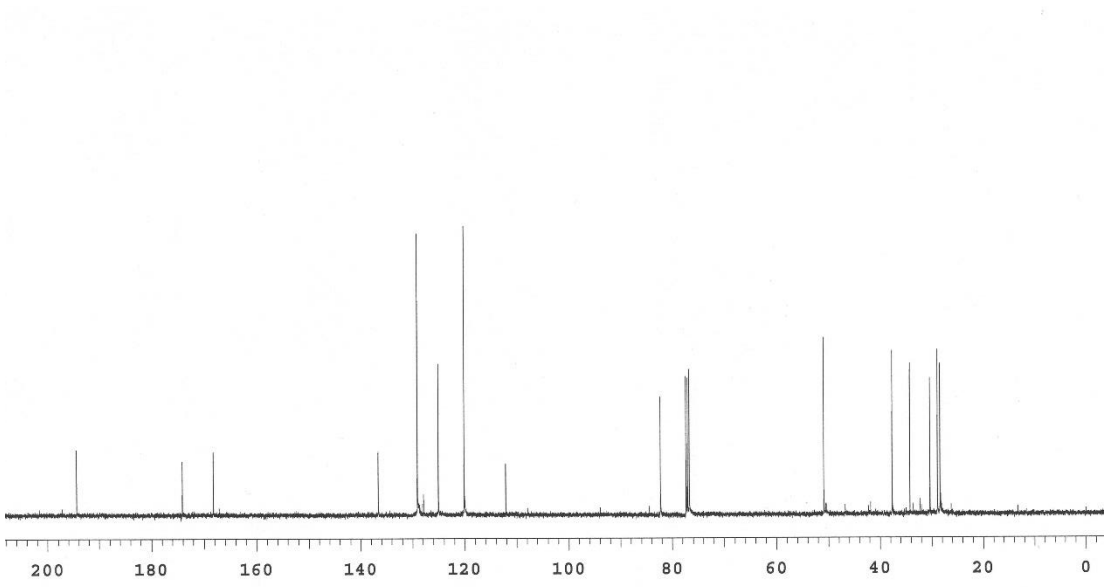

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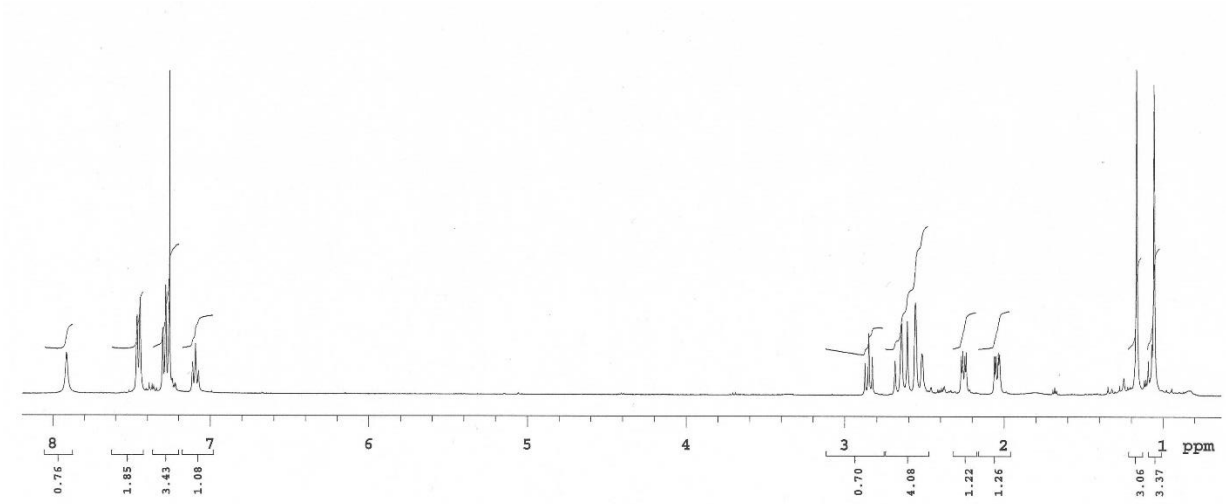

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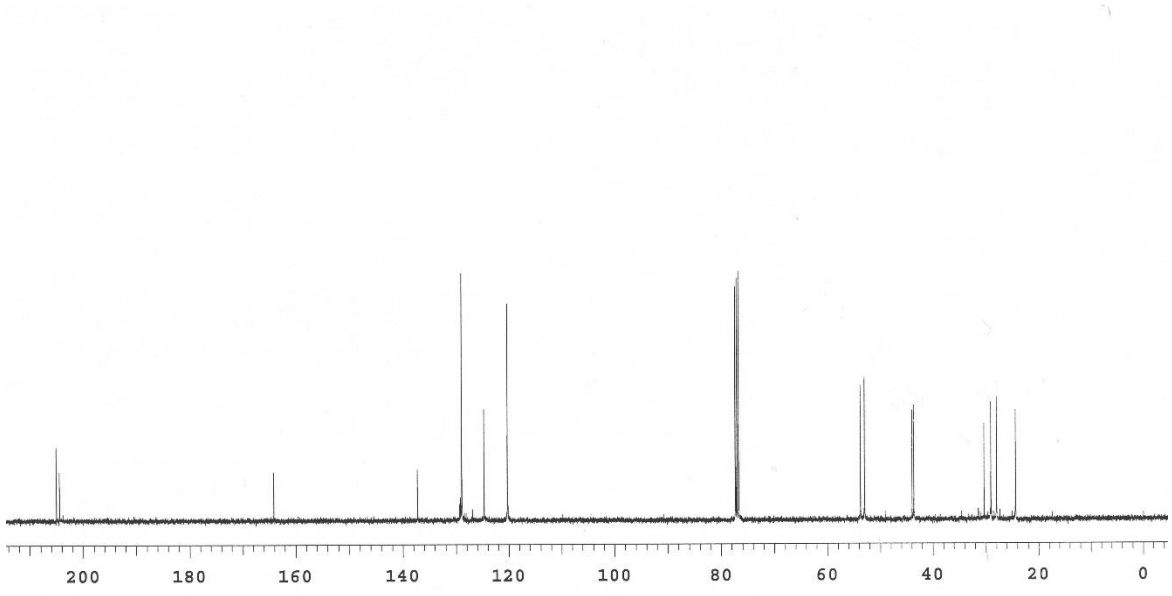

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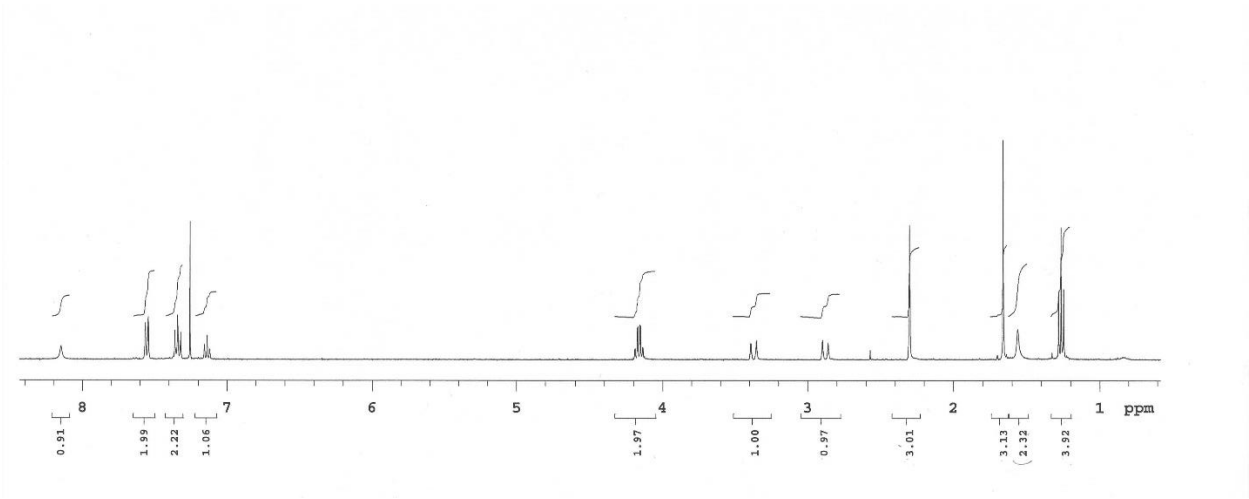

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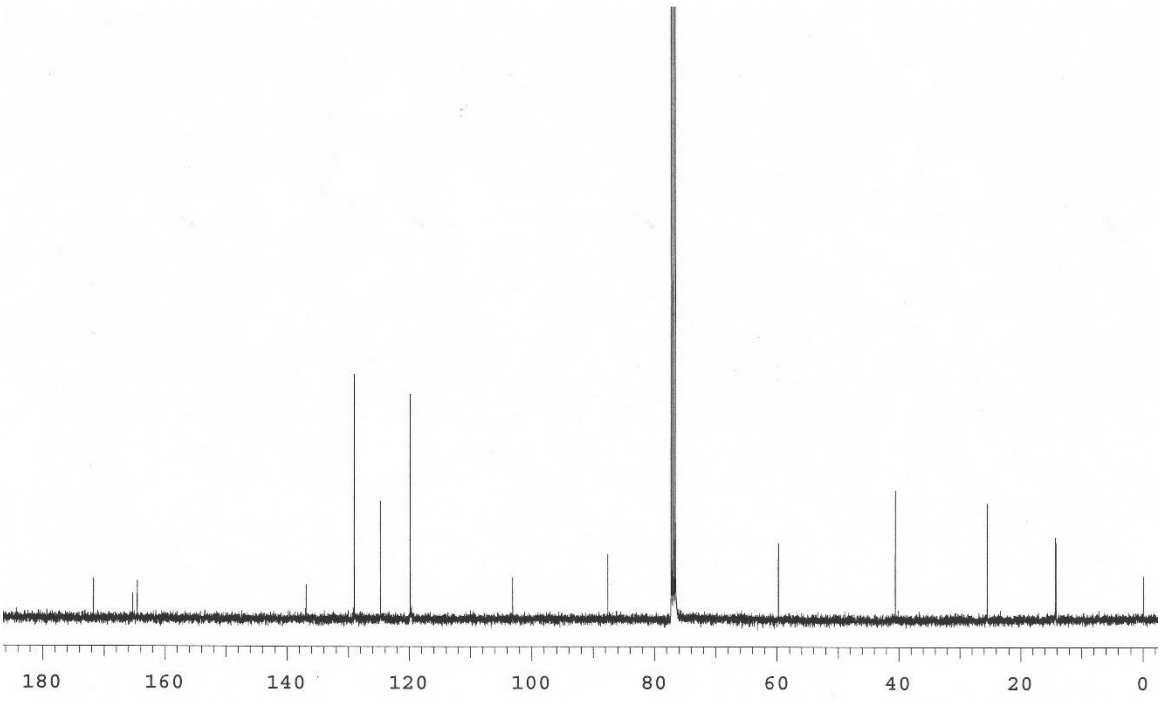

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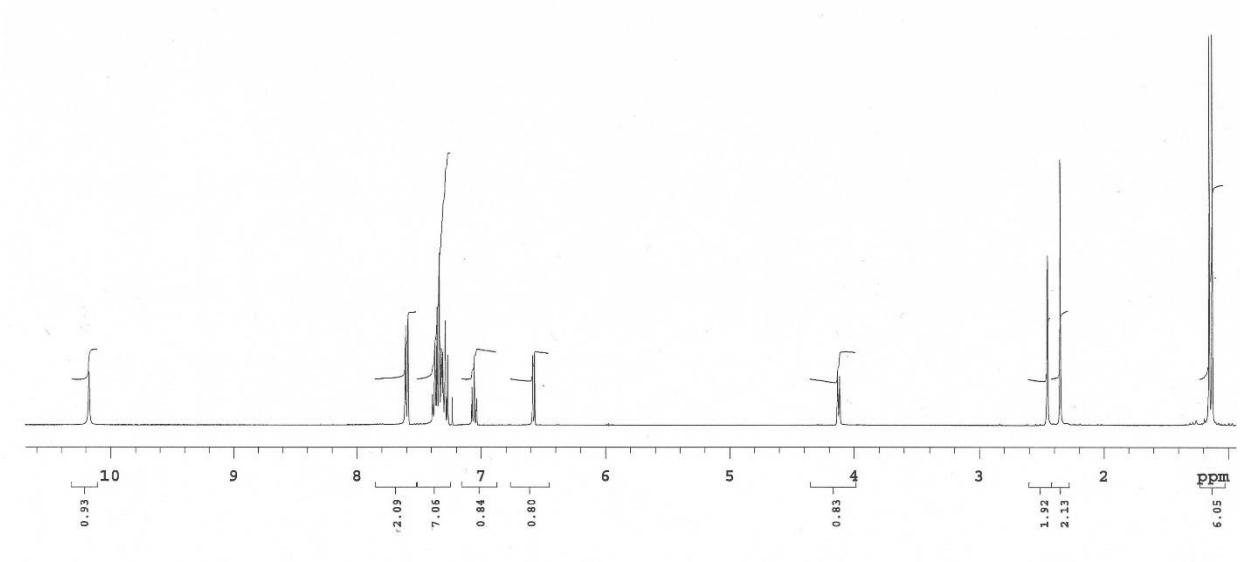

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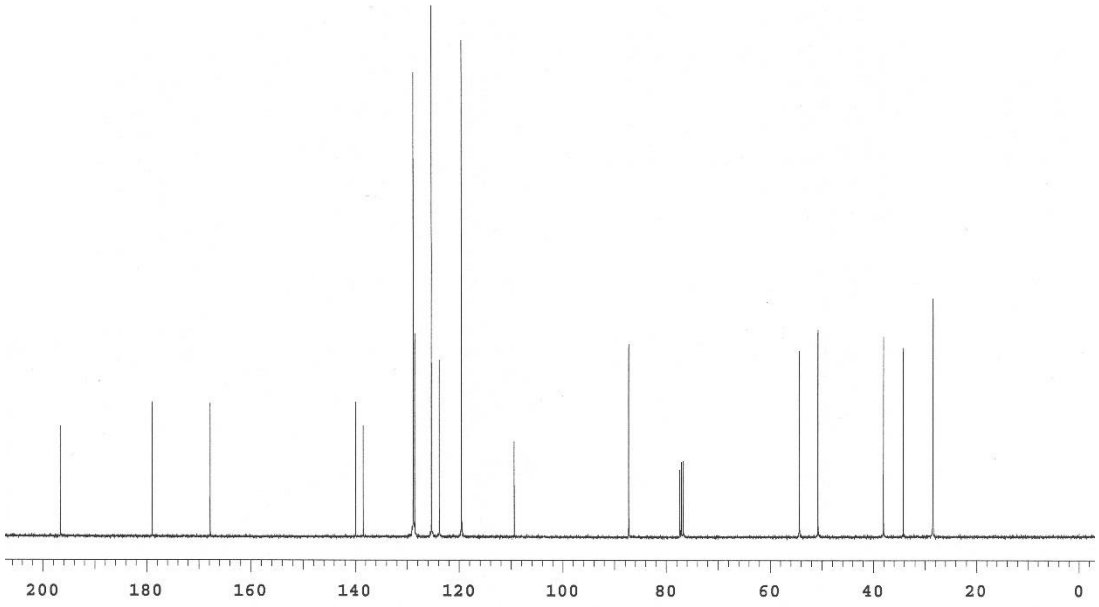

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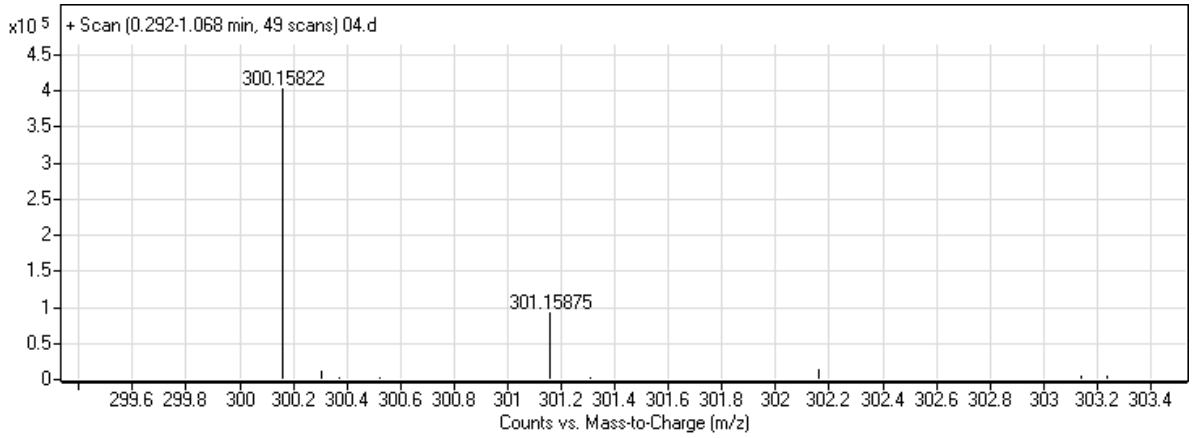

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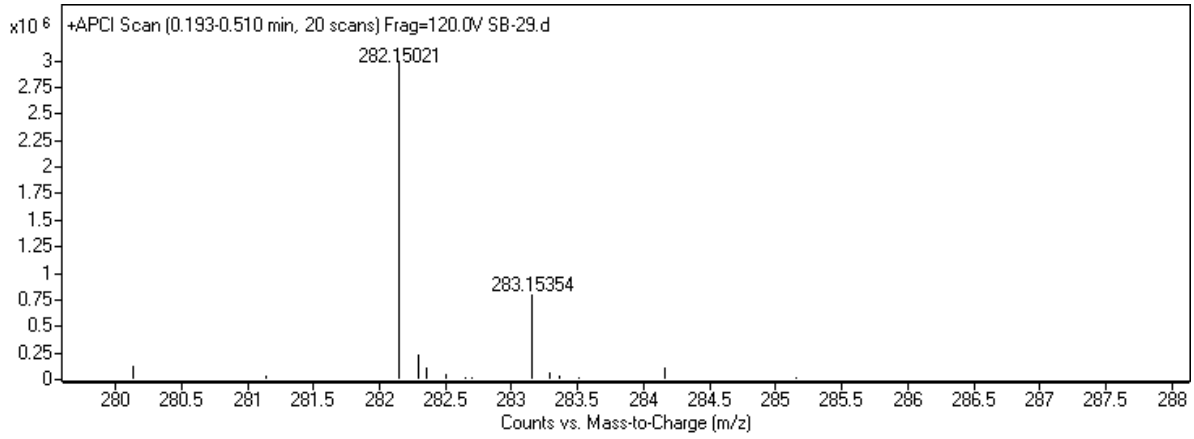
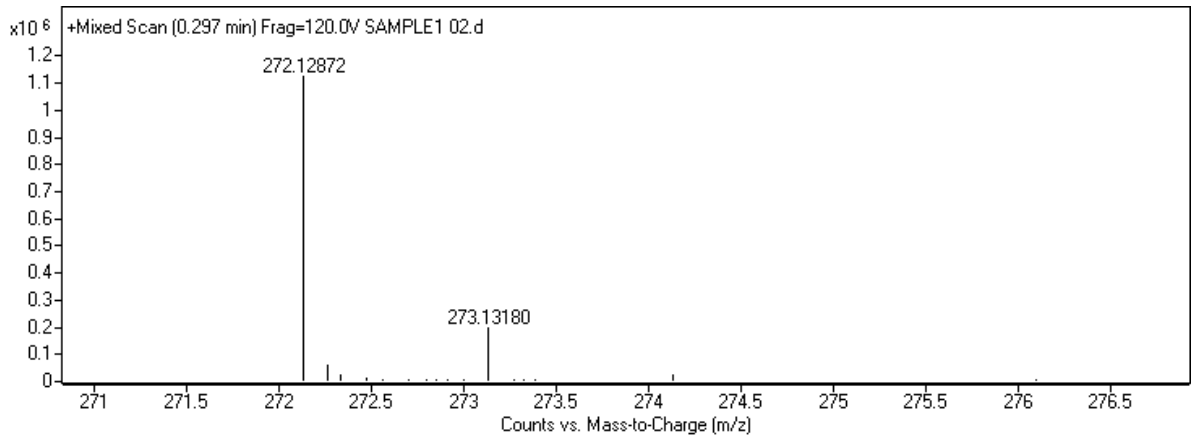
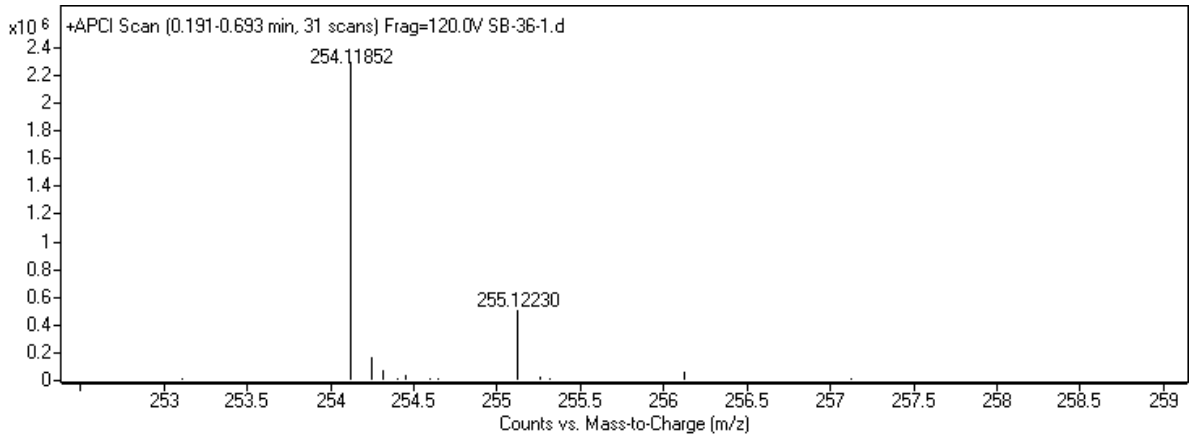


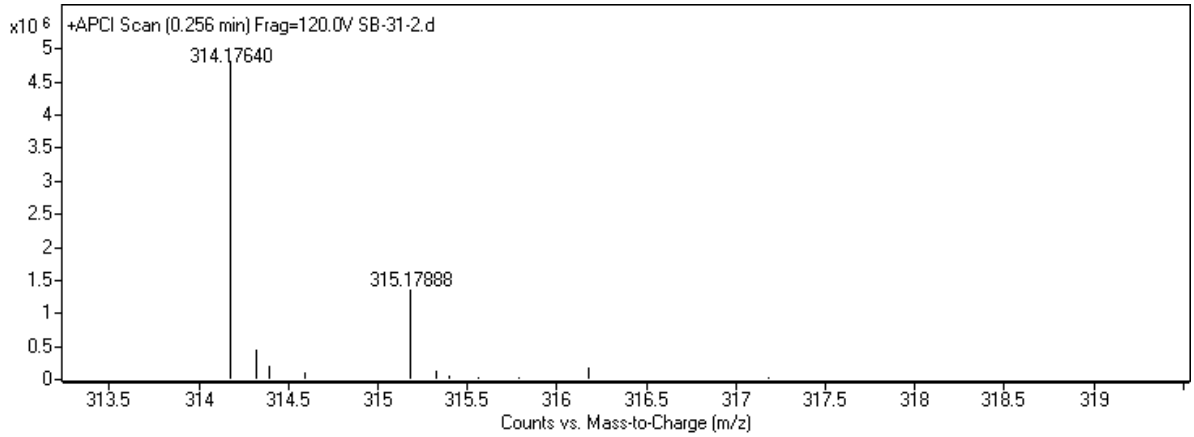
Compound 3b HRMS



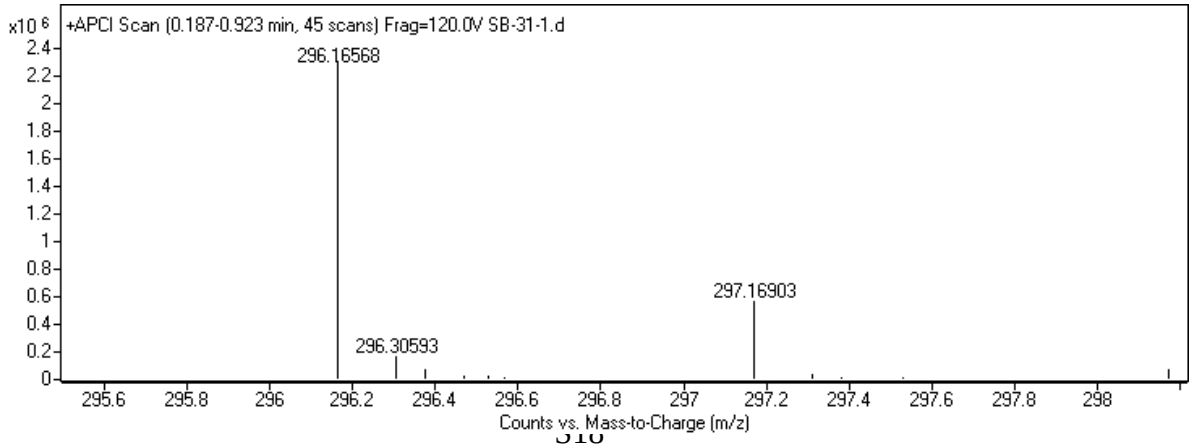
Compound 4b HRMS



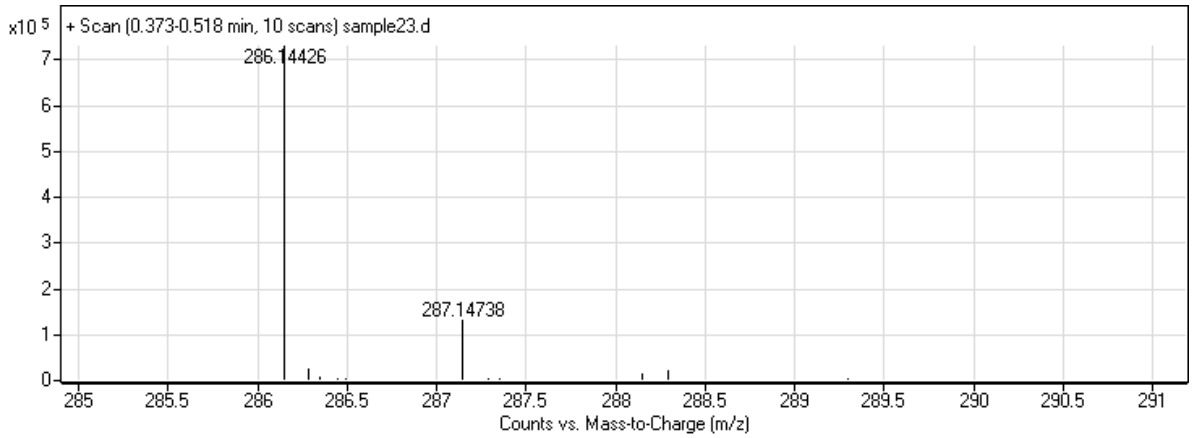
Compound 3c HRMS



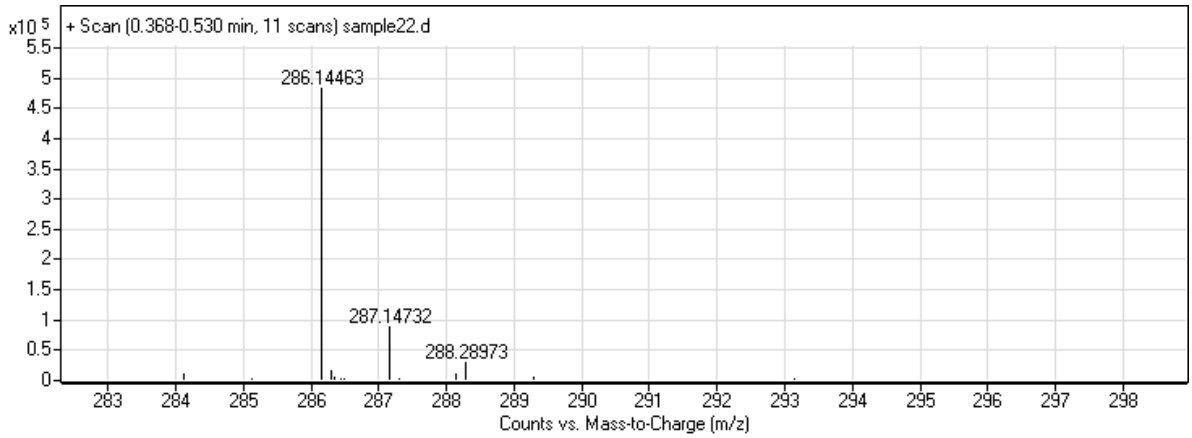
Compound 4c HRMS



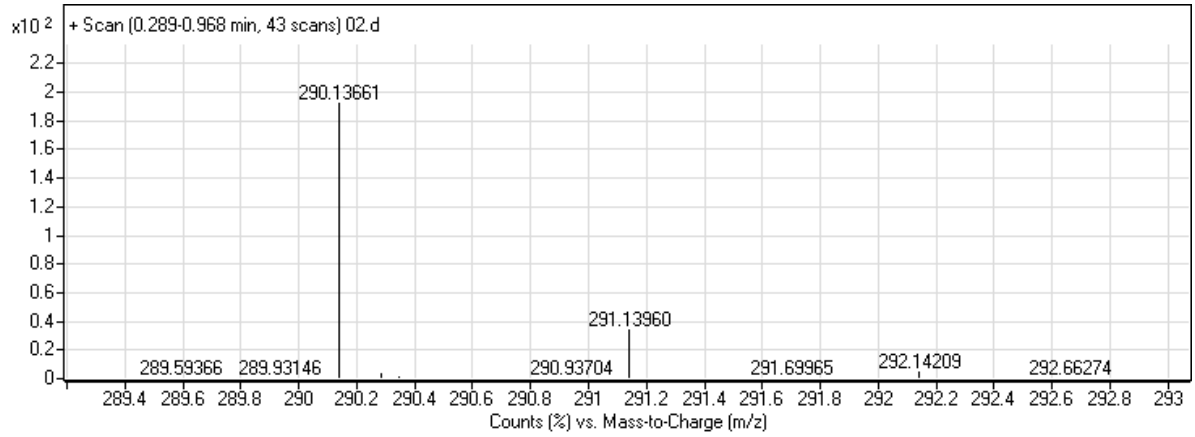
Compound 3d HRMS



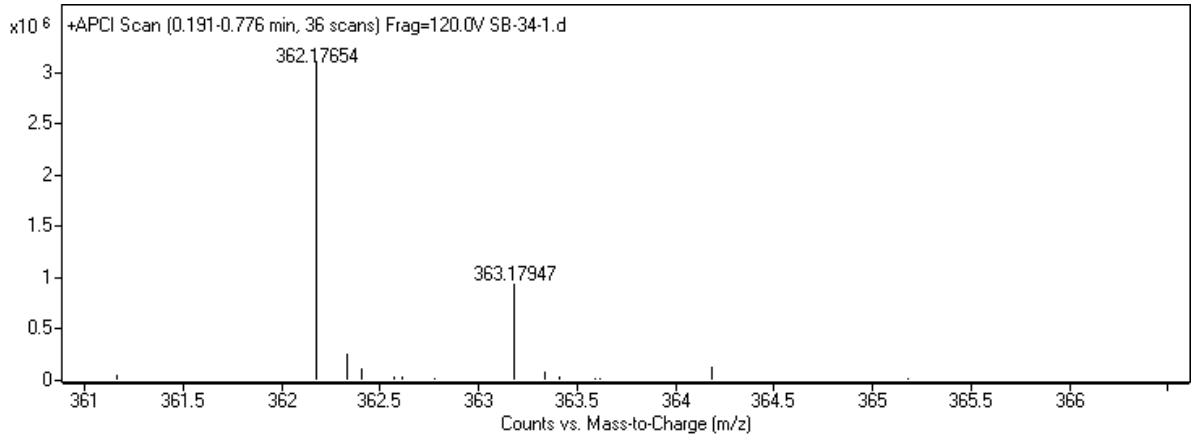
Compound 5 HRMS



Compound 6 HRMS



Compound 7 HRMS



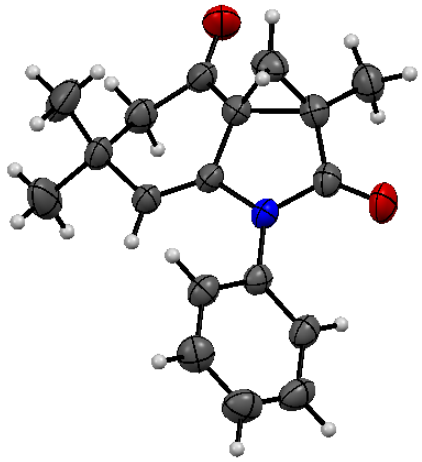
X-Ray Crystallography Information

X-ray data collection and structure refinement

Unit cell measurements and intensity data collection was performed on an Bruker APEX II QUAZAR three-circle diffractometer using monochromatized Mo Kα microfocus sealed-tube (λ = 0.71073 Å) using φ and ω technique at 120 K. Data integration and reduction were carried out with Bruker SAINT [S4] software package using a wide-frame algorithm. Absorption correction was performed using the multi-scan method (SADABS) [S4]. Space groups were determined using XPREP implemented in APEX2 [S4]. Structures were solved and refined using Bruker SHELXTL software package [S5]. All non-hydrogen atoms were refined anisotropically using all reflections with $I > 2\sigma(I)$. Aromatic and aliphatic C-bound H atoms were positioned geometrically and refined using a riding mode and Uiso(H) = 1.2Ueq(C). Crystallographic data and refinement details of **1-5** are summarized in Table S1. The molecular drawings were carried out with and Mercury program [S6].

[S4] Bruker APEX2 (Version 2014.1-1), SAINT (Version 8.34A) and SADABS (Version 2012/1). Bruker AXS Inc., Madison, Wisconsin, USA.
[S5] Bruker *SHELXTL* (Version 6.14). Bruker AXS Inc., Madison, Wisconsin, USA.
[S6] C. F. Macrae, P. R. Edgington, P. McCabe, E. Pidcock, G. P. Shields, R. Taylor, M. Towler, J. van de Streek, J. Appl. Cryst. 39 (2006) 453-457.

Crystallographic Data for 4a



Crystal Structure Report for Compound 4a

Crystal description
A colorless block-like specimen of $C_{18}H_{19}NO_2$, approximate dimensions 0.274 mm x 0.339 mm x 0.375 mm, was used for the X-ray crystallographic analysis. Instrument description The X-ray intensity data were measured.
Data collection The total exposure time was 2.99 hours. Integration The frames were integrated with the Bruker SAINT software package using a wide-frame algorithm. The integration of the data using a monoclinic unit cell yielded a total of 11513 reflections to a maximum θ angle of 25.00° (0.84 Å resolution), of which 2660 were independent (average redundancy 4.328, completeness = 99.8%, R_{int} = 3.20%, R_{sig} = 2.57%) and 2194 (82.48%) were greater than $2\sigma(F^2)$. Unit cell The final cell constants of a = 10.4868(11) Å, b = 8.0500(8) Å, c = 18.2365(16) Å, β = 101.892(6)°, volume = 1506.5(3) $Å^3$, are based upon the refinement of the XYZ-centroids of 4501 reflections above 20 σ(I) with 4.565° < 2θ < 53.64°. Scaling Data were corrected for absorption effects using the multi-scan method (SADABS). The ratio of minimum to maximum apparent transmission was 0.860. The calculated minimum and maximum transmission coefficients (based on crystal size) are 0.9700 and 0.9780.

Structure solution The structure was solved and refined using the Bruker SHELXTL Software Package, using the space group P 1 21/n 1, with Z = 4 for the formula unit, $C_{18}H_{19}NO_2$. Structure refinement The final anisotropic full-matrix least-squares refinement on F^2 with 194 variables converged at R1 = 4.08%, for the observed data and wR2 = 11.03% for all data. The goodness-of-fit was 1.048. The largest peak in the final difference electron density synthesis was 0.200 $e^-/Å^3$ and the largest hole was -0.151 $e^-/Å^3$ with an RMS deviation of 0.031 $e^-/Å^3$. On the basis of the final model, the calculated density was 1.240 g/cm^3 and F(000), 600 e^-. Structure Packing

Table S1 Sample and crystal data for **4a**

Identification code	17gtu071_B_4_1	
Chemical formula	$C_{18}H_{19}NO_2$	
Formula weight	281.34 g/mol	
Temperature	296(2) K	
Wavelength	0.71073 Å	
Crystal size	0.274 x 0.339 x 0.375 mm	
Crystal habit	colorless block	
Crystal system	monoclinic	
Space group	P 1 21/n 1	
Unit cell dimensions	a = 10.4868(11) Å	α = 90°
	b = 8.0500(8) Å	β = 101.892(6)°
	c = 18.2365(16) Å	γ = 90°
Volume	1506.5(3) $Å^3$	
Z	4	
Density (calculated)	1.240 g/cm^3	
Absorption coefficient	0.081 mm^{-1}	
F(000)	600	

Table S2. Data collection and structure refinement for 17gtu071_B_4_1.

Theta range for data collection	2.08 to 25.00°	
Index ranges	-12<=h<=12, -8<=k<=9, -21<=l<=20	
Reflections collected	11513	
Independent reflections	2660 [R(int) = 0.0320]	
Coverage of independent reflections	99.8%	
Absorption correction	multi-scan	
Max. and min. transmission	0.9780 and 0.9700	
Structure solution technique	direct methods	
Structure solution program	SHELXTL V6.14 (Bruker AXS)	
Refinement method	Full-matrix least-squares on F^2	
Refinement program	SHELXL-2014/7 (Sheldrick, 2014)	
Function minimized	$\Sigma\, w(F_o^2 - F_c^2)^2$	
Data / restraints / parameters	2660 / 0 / 194	
Goodness-of-fit on F^2	1.048	
Final R indices	2194 data; I>2σ(I)	R1 = 0.0408, wR2 = 0.1031
	all data	R1 = 0.0508, wR2 = 0.1103
Weighting scheme	$w=1/[\sigma^2(F_o^2)+(0.0503P)^2+0.4031P]$ where $P=(F_o^2+2F_c^2)/3$	
Extinction coefficient	0.0090(20)	
Largest diff. peak and hole	0.200 and -0.151 $eÅ^{-3}$	
R.M.S. deviation from mean	0.031 $eÅ^{-3}$	

Table S3. Atomic coordinates and equivalent isotropic atomic displacement parameters (Å^2) for 17gtu071_B_4_1.
U(eq) is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x/a	y/b	z/c	U(eq)
C1	0.40325(14)	0.27456(18)	0.65133(8)	0.0344(4)
C2	0.36055(15)	0.40219(19)	0.60762(8)	0.0396(4)
C3	0.26490(16)	0.3748(2)	0.53430(8)	0.0444(4)
C4	0.1871(2)	0.5338(2)	0.51256(11)	0.0720(6)
C5	0.3359(2)	0.3257(3)	0.47238(9)	0.0689(6)
C6	0.17135(17)	0.2348(2)	0.54564(9)	0.0472(4)
C7	0.23813(17)	0.0775(2)	0.57683(8)	0.0442(4)
C8	0.36271(15)	0.10196(18)	0.63167(8)	0.0379(4)
C9	0.47078(18)	0.9753(2)	0.63634(10)	0.0505(4)
C10	0.40890(15)	0.99693(19)	0.70220(9)	0.0414(4)
C11	0.33238(18)	0.8611(2)	0.72959(10)	0.0556(5)
C12	0.48016(15)	0.1183(2)	0.75831(8)	0.0413(4)
C13	0.54613(14)	0.41525(18)	0.76067(8)	0.0360(4)
C14	0.54073(15)	0.4511(2)	0.83429(8)	0.0429(4)
C15	0.60469(17)	0.5897(2)	0.86835(9)	0.0527(5)
C16	0.67141(17)	0.6936(2)	0.82963(10)	0.0560(5)
C17	0.67634(16)	0.6577(2)	0.75645(10)	0.0513(4)
C18	0.61503(15)	0.5185(2)	0.72206(9)	0.0417(4)
N1	0.48180(12)	0.27233(15)	0.72435(6)	0.0373(3)
O1	0.19470(14)	0.94030(16)	0.55914(7)	0.0646(4)
O2	0.52859(13)	0.08755(16)	0.82301(6)	0.0620(4)

Bond lengths

Table S4. Bond lengths (Å) for 17gtu071_B_4_1.

C1-C2	1.321(2)	C1-N1	1.4143(18)
C1-C8	1.475(2)	C2-C3	1.513(2)
C2-H2	0.93	C3-C4	1.526(3)
C3-C5	1.527(3)	C3-C6	1.536(2)
C4-H4A	0.96	C4-H4B	0.96
C4-H4C	0.96	C5-H5A	0.96
C5-H5B	0.96	C5-H5C	0.96
C6-C7	1.501(2)	C6-H6A	0.97
C6-H6B	0.97	C7-O1	1.2128(19)
C7-C8	1.486(2)	C8-C9	1.514(2)
C8-C10	1.532(2)	C9-C10	1.488(2)
C9-H9A	0.97	C9-H9B	0.97
C10-C12	1.498(2)	C10-C11	1.501(2)
C11-H11A	0.96	C11-H11B	0.96
C11-H11C	0.96	C12-O2	1.2100(18)
C12-N1	1.3880(19)	C13-C18	1.385(2)
C13-C14	1.386(2)	C13-N1	1.4259(19)
C14-C15	1.381(2)	C14-H14	0.93
C15-C16	1.376(3)	C15-H15	0.93
C16-C17	1.377(2)	C16-H16	0.93
C17-C18	1.377(2)	C17-H17	0.93
C18-H18	0.93		

Bond angles

Table S5. Bond angles (°) for 17gtu071_B_4_1.

C2-C1-N1	129.60(14)	C2-C1-C8	122.75(13)
N1-C1-C8	107.44(12)	C1-C2-C3	120.03(14)
C1-C2-H2	120.0	C3-C2-H2	120.0
C2-C3-C4	109.20(14)	C2-C3-C5	110.85(14)
C4-C3-C5	109.93(15)	C2-C3-C6	108.27(12)
C4-C3-C6	109.18(15)	C5-C3-C6	109.38(15)
C3-C4-H4A	109.5	C3-C4-H4B	109.5
H4A-C4-H4B	109.5	C3-C4-H4C	109.5
H4A-C4-H4C	109.5	H4B-C4-H4C	109.5
C3-C5-H5A	109.5	C3-C5-H5B	109.5
H5A-C5-H5B	109.5	C3-C5-H5C	109.5
H5A-C5-H5C	109.5	H5B-C5-H5C	109.5
C7-C6-C3	114.14(14)	C7-C6-H6A	108.7
C3-C6-H6A	108.7	C7-C6-H6B	108.7
C3-C6-H6B	108.7	H6A-C6-H6B	107.6
O1-C7-C8	121.98(15)	O1-C7-C6	123.16(15)
C8-C7-C6	114.85(13)	C1-C8-C7	117.24(13)
C1-C8-C9	116.48(13)	C7-C8-C9	119.53(13)
C1-C8-C10	106.87(12)	C7-C8-C10	125.04(13)
C9-C8-C10	58.50(10)	C10-C9-C8	61.36(10)
C10-C9-H9A	117.6	C8-C9-H9A	117.6
C10-C9-H9B	117.6	C8-C9-H9B	117.6
H9A-C9-H9B	114.7	C9-C10-C12	112.87(14)
C9-C10-C11	122.34(15)	C12-C10-C11	118.23(14)
C9-C10-C8	60.15(10)	C12-C10-C8	103.92(12)
C11-C10-C8	125.66(14)	C10-C11-H11A	109.5
C10-C11-H11B	109.5	H11A-C11-H11B	109.5
C10-C11-H11C	109.5	H11A-C11-H11C	109.5
H11B-C11-H11C	109.5	O2-C12-N1	125.19(15)
O2-C12-C10	125.66(15)	N1-C12-C10	109.15(12)
C18-C13-C14	119.82(15)	C18-C13-N1	119.65(13)
C14-C13-N1	120.53(14)	C15-C14-C13	119.39(16)
C15-C14-H14	120.3	C13-C14-H14	120.3
C16-C15-C14	120.78(16)	C16-C15-H15	119.6
C14-C15-H15	119.6	C15-C16-C17	119.63(17)
C15-C16-H16	120.2	C17-C16-H16	120.2
C16-C17-C18	120.34(17)	C16-C17-H17	119.8
C18-C17-H17	119.8	C17-C18-C13	120.03(15)
C17-C18-H18	120.0	C13-C18-H18	120.0
C12-N1-C1	111.91(12)	C12-N1-C13	124.19(12)
C1-N1-C13	123.72(12)		

Torsion angles

Table S6. Torsion angles (°) for 17gtu071_B_4_1.

N1-C1-C2-C3	-173.33(14)	C8-C1-C2-C3	0.7(2)
C1-C2-C3-C4	155.08(16)	C1-C2-C3-C5	-83.66(19)
C1-C2-C3-C6	36.3(2)	C2-C3-C6-C7	-53.72(18)
C4-C3-C6-C7	-172.51(14)	C5-C3-C6-C7	67.16(18)
C3-C6-C7-O1	-144.78(16)	C3-C6-C7-C8	35.29(19)
C2-C1-C8-C7	-22.1(2)	N1-C1-C8-C7	153.00(13)
C2-C1-C8-C9	128.99(16)	N1-C1-C8-C9	-55.87(17)
C2-C1-C8-C10	-168.34(14)	N1-C1-C8-C10	6.80(16)
O1-C7-C8-C1	-177.48(15)	C6-C7-C8-C1	2.4(2)
O1-C7-C8-C9	32.3(2)	C6-C7-C8-C9	-147.77(15)
O1-C7-C8-C10	-38.0(2)	C6-C7-C8-C10	141.88(15)
C1-C8-C9-C10	94.30(14)	C7-C8-C9-C10	-115.26(16)
C8-C9-C10-C12	-93.38(14)	C8-C9-C10-C11	115.60(17)
C1-C8-C10-C9	-111.13(14)	C7-C8-C10-C9	106.03(17)
C1-C8-C10-C12	-2.51(16)	C7-C8-C10-C12	-145.34(15)
C9-C8-C10-C12	108.62(14)	C1-C8-C10-C11	138.54(16)
C7-C8-C10-C11	-4.3(2)	C9-C8-C10-C11	-110.32(19)
C9-C10-C12-O2	-119.73(18)	C11-C10-C12-O2	32.6(2)
C8-C10-C12-O2	177.15(16)	C9-C10-C12-N1	60.37(17)
C11-C10-C12-N1	-147.33(15)	C8-C10-C12-N1	-2.76(16)
C18-C13-C14-C15	-0.2(2)	N1-C13-C14-C15	179.53(14)
C13-C14-C15-C16	1.2(2)	C14-C15-C16-C17	-1.1(3)
C15-C16-C17-C18	-0.1(3)	C16-C17-C18-C13	1.1(2)
C14-C13-C18-C17	-1.0(2)	N1-C13-C18-C17	179.34(14)
O2-C12-N1-C1	-172.48(15)	C10-C12-N1-C1	7.43(17)
O2-C12-N1-C13	2.7(2)	C10-C12-N1-C13	-177.37(13)
C2-C1-N1-C12	165.70(16)	C8-C1-N1-C12	-8.99(16)
C2-C1-N1-C13	-9.5(2)	C8-C1-N1-C13	175.77(13)
C18-C13-N1-C12	138.80(15)	C14-C13-N1-C12	-40.9(2)
C18-C13-N1-C1	-46.5(2)	C14-C13-N1-C1	133.75(15)

Anisotropic displacement parameters

Table S7. Anisotropic atomic displacement parameters (Å^2) for 17gtu071_B_4_1.

The anisotropic atomic 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}
C1	0.0369(8)	0.0394(9)	0.0265(7)	-0.0013(6)	0.0052(6)	-0.0018(6)
C2	0.0472(9)	0.0381(9)	0.0309(8)	0.0002(6)	0.0019(7)	-0.0049(7)
C3	0.0539(10)	0.0450(9)	0.0295(8)	0.0039(7)	-0.0026(7)	-0.0060(8)
C4	0.0841(15)	0.0534(12)	0.0611(12)	0.0108(9)	-0.0255(11)	0.0001(11)
C5	0.0821(14)	0.0913(15)	0.0327(9)	-0.0003(9)	0.0107(9)	-0.0226(12)
C6	0.0465(9)	0.0538(10)	0.0363(8)	0.0001(7)	-0.0031(7)	-0.0068(8)
C7	0.0562(10)	0.0448(10)	0.0310(8)	-0.0035(7)	0.0074(7)	-0.0101(8)
C8	0.0453(9)	0.0365(8)	0.0322(8)	0.0003(6)	0.0085(7)	0.0005(7)
C9	0.0548(10)	0.0435(10)	0.0557(10)	-0.0049(8)	0.0173(8)	0.0039(8)
C10	0.0447(9)	0.0382(9)	0.0404(9)	0.0055(7)	0.0066(7)	0.0033(7)
C11	0.0605(11)	0.0496(10)	0.0547(11)	0.0123(8)	0.0072(9)	-0.0045(9)
C12	0.0423(9)	0.0445(9)	0.0359(8)	0.0068(7)	0.0049(7)	0.0048(7)
C13	0.0322(8)	0.0410(9)	0.0311(8)	-0.0003(6)	-0.0021(6)	0.0052(7)
C14	0.0422(9)	0.0515(10)	0.0328(8)	0.0015(7)	0.0027(7)	0.0058(7)
C15	0.0543(10)	0.0616(12)	0.0371(9)	-0.0108(8)	-0.0028(8)	0.0090(9)
C16	0.0503(10)	0.0558(11)	0.0548(11)	-0.0114(9)	-0.0053(8)	-0.0041(9)
C17	0.0413(9)	0.0550(11)	0.0545(10)	-0.0005(8)	0.0026(8)	-0.0075(8)
C18	0.0377(8)	0.0500(10)	0.0361(8)	-0.0019(7)	0.0045(7)	-0.0004(7)
N1	0.0414(7)	0.0388(7)	0.0286(6)	0.0015(5)	0.0002(5)	0.0003(6)
O1	0.0842(10)	0.0499(8)	0.0524(8)	-0.0054(6)	-0.0030(7)	-0.0200(7)
O2	0.0780(9)	0.0584(8)	0.0402(7)	0.0141(5)	-0.0097(6)	0.0025(7)

Hydrogen atom coordinates and isotropic atomic displacement parameters

Table S8. Hydrogen atomic coordinates and isotropic atomic displacement parameters (Å^2) for 17gtu071_B_4_1.

	x/a	y/b	z/c	U(eq)
H2	0.3893	0.5089	0.6219	0.048
H4A	0.1431	0.5641	0.5518	0.108
H4B	0.1241	0.5157	0.4670	0.108
H4C	0.2451	0.6216	0.5052	0.108
H5A	0.3953	0.4124	0.4657	0.103
H5B	0.2735	0.3092	0.4264	0.103
H5C	0.3834	0.2246	0.4861	0.103
H6A	0.1173	0.2737	0.5793	0.057
H6B	0.1144	0.2101	0.4978	0.057
H9A	0.4514	-0.1257	0.6072	0.061
H9B	0.5589	0.0165	0.6405	0.061
H11A	0.3909	-0.2123	0.7618	0.083
H11B	0.2848	-0.2001	0.6875	0.083
H11C	0.2725	-0.0911	0.7570	0.083
H14	0.4945	0.3825	0.8605	0.051
H15	0.6026	0.6129	0.9180	0.063
H16	0.7130	0.7877	0.8527	0.067
H17	0.7213	0.7278	0.7301	0.062
H18	0.6199	0.4938	0.6729	0.05