Electronic Supplementary Material (ESI) for Chemical Communications

Supplementary Information

Ionic liquids are compatible with on-water catalysis.

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General Experimental Details:

All solvents and reagents were used as received from commercial sources. Water was purified using a Millipore Milli-Q System (<18.2 MΩ.cm @25°C) or Millipore Elix system (< 15 MΩ.cm @25°C). Melting points were determined using a Stanford Research Systems Optimelt automated melting point system and are uncorrected. Infrared spectra were acquired neat on a Bruker Alpha-E ATR spectrometer. ^{1}H and ^{13}C NMR spectra were recorded on a Bruker ASCEND 500 (^{1}H frequencies 500 MHz; ^{13}C frequencies 125 MHz), a Bruker AVANCE DPX300 (^{1}H frequencies 300 MHz; ^{13}C frequencies 75 MHz) or a Bruker AVANCE DPX200 (^{1}H frequencies 200 MHz; ^{13}C frequencies 50 MHz). ^{1}H chemical shifts are expressed as parts per million (ppm) with residual chloroform (δ 7.26) as reference and are reported as chemical shift (δ_H); relative integral; multiplicity (s singlet, br broad, d doublet, t triplet, dd doublet of doublets, dt doublet of triplets, q quartet, m multiplet); and coupling constants (*J*) reported in Hz. ^{13}C NMR chemical shifts are expressed as parts per million (ppm) with residual chloroform (δ 77.1) as internal reference and are reported as chemical shift (δ_C); multiplicity (assigned from DEPT experiments). High resolution mass spectra were recorded on a Bruker ApexII Fourier Transform Ion Cyclotron Resonance mass spectrometer with a 7.0 T magnet, fitted with an off-axis Analytical electrospray source.

General Procedures for Cycloaddition Reactions:

All reactions were conducted in duplicate (or triplicate) and % conversion was calculated as [integral product]/([integral product] + [integral starting material]) determined by ^{1}H NMR analysis of the crude reaction mixture.

On-Water: A suspension of the diene/dipole (0.5/0.3 mmol) and the dienophile/dipolarophile (0.5/0.3 mmol) in water (4 mL) was stirred vigorously at the prescribed temperature (80 ^{o}C, 40 ^{o}C or room temperature) for the stated time. The reaction was extracted with ethyl acetate (4 mL) and the organic phase concentrated under reduced pressure.

At-Water: A suspension of the diene/dipole (0.5/0.3 mmol) and the dienophile/dipolarophile (0.5/0.3 mmol) in water (4 mL) was stirred gently (*ca* 250 r.p.m) at the prescribed temperature (80 ^{o}C, 40 ^{o}C or room temperature) for the stated time. The reaction was extracted with ethyl acetate (4 mL) and the organic phase concentrated under reduced pressure.

Neat: The diene/dipole (0.5/0.3 mmol) and the dienophile/dipolarophile (0.5/0.3 mmol) were stirred neat at the prescribed temperature (80 ^{o}C, 40 ^{o}C or room temperature) for the stated time. The reaction was concentrated *in vacuo* where appropriate to remove the unreacted volatile components.

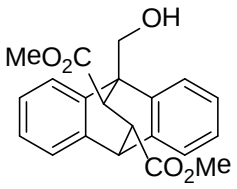
Solvent: A mixture of the diene/dipole (0.5/0.3 mmol) and the dienophile/dipolarophile (0.5/0.3 mmol) in solvent (0.5 mL, EtOH, BuOH, toluene, hexadecane or [BMIM][NTf_2])

was stirred gently at prescribed temperature (80 ^{o}C, 40 ^{o}C or room temperature) for the stated time. The volatile components were removed under reduced pressure.

On-Water/IL: A suspension of the diene/dipole (0.5/0.3 mmol) and the dienophile/dipolarophile (0.5/0.3 mmol) in water (4 mL) and [BMIM][NTf_2] (0.5 mL) was stirred vigorously at the prescribed temperature (80 ^{o}C, 40 ^{o}C or room temperature) for the stated time. The reaction was extracted with ethyl acetate (4 mL) and the organic phase concentrated under reduced pressure.

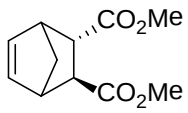
On-Water/Solvent: A suspension of the diene/dipole (0.5/0.3 mmol) and the dienophile/dipolarophile (0.5/0.3 mmol) in water water (4 mL) and organic solvent (0.5 mL, toluene or hexadecane) was stirred vigorously at the prescribed temperature (80 ^{o}C, 40 ^{o}C or room temperature) for the stated time. The reaction was extracted with ethyl acetate (4 mL) and the organic phase concentrated under reduced pressure.

Dimethyl 9-(hydroxymethyl)-9,10-dihydro-9,10-ethanoanthracene-11,12-dicarboxylate (8)[1]



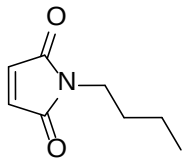
Following the general procedure (solvent), a mixture of 9-anthracenemethanol (104 mg, 0.50 mmol) and dimethyl fumarate (72 mg, 0.50 mmol) in *n*-butanol (0.5 mL) was stirred at 80 ^{o}C for 16 h. The solvent was evaporated and the crude residue purified by flash chromatography (8:2 hexane:ethyl acetate) to give compound **8** as a colourless oil (123 mg, 70%). ν_{max} (neat)/cm^{-1}: 2896, 1731, 1434, 1363, 1170, 1010; δ_H (500 MHz, $CDCl_3$): 7.65 (1 H, d, *J* 7.5, Ar-H), 7.38 (1 H, d, *J* 6.0, Ar-H), 7.29 (1 H, d, *J* 7.0, Ar-H), 7.19–7.12 (5 H, m, Ar-H), 4.83 (1 H, app. t, *J* 11.0, one of CH_2OH), 4.73 (1 H, s, 10-CH), 4.68 (1 H, d, *J* 12.0, one of CH_2OH), 3.63 (6 H, s, 2 × $COOCH_3$), 3.45 (1 H, s, 11-CH), 3.38 (1 H, s, 12-CH), 2.69 (1 H , bs, OH); δ_C (125 MHz, $CDCl_3$): 172.8 (C), 172.5 (C), 143.1 (C), 142.3 (C), 141.0 (C), 139.6 (C), 126.5 (CH), 126.4 (CH), 126.3 (CH), 126.3 (CH), 124.8 (CH), 122.8 (CH), 122.1 (CH), 61.5 (CH_2), 52.4 (CH_3), 52.3 (CH_3) 50.1 (CH, 50.0 (CH), 48.0 (CH), 46.3 (CH); m/z (ESI): Calculated for $C_{21}H_{20}O_5Na^+$ 375.1203, found 375.1205.

Dimethyl bicyclo[2.2.1]hept-5-ene-2,3-dicarboxylate (10)[2]



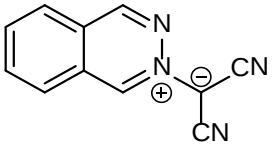
Freshly distilled cyclopentadiene (33 μL, 0.40 mmol) was added to a suspension of dimethyl fumarate (50 mg, 0.35 mmol) in water (4 mL) and the suspension was stirred vigorously for 150 minutes. Ethyl acetate (10 mL) was added and the phases were separated, the organic phase was dried over Na_2SO_4 and the solvent was evaporated to give the title compound (68.3 mg, 93%) as colourless oil. ν_{max} (neat)/cm^{-1}: 2953, 1726 (CO), 1435, 1265, 1163, 1017; δ_H (200 MHz, $CDCl_3$): 6.22 (1 H, dd, *J* 4, 4), 6.02 (1 H, dd, *J* 4, 4), 3.66 (3 H, s), 3.59 (3 H, s), 3.32 (1 H, t, *J* 4), 3.21 (1 H, br), 3.07 (1 H, br), 2.63 (1 H, dd, *J* 2, 6), 1.57 (1 H, dt, *J* 2, 10), 1.40 (1 H, ddd, *J* 2, 2, 10); δ_C (75 MHz, $CDCl_3$): 175.0 (C), 173.8 (C), 137.7 (CH), 135.3 (CH), 52.2 (CH3), 51.9 (CH3), 48.0 (CH), 47.7 (CH), 47.4 (CH2), 47.2 (CH), 45.7 (CH); m/z (ESI): 443 (100%, M2Na$^+$), 233 (45, MNa$^+$), 211 (69, MH$^+$).

***N*-Butyl maleimide (11)**



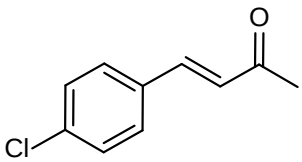
N-Butylamine (30 mL, 0.30 mol) was added dropwise over 30 min to a suspension of maleic anhydride (30 g, 0.30 mol) in $CHCl_3$ (150 mL) at 0 ^oC. The mixture was stirred for a further 1.5 h and then the solvent was removed under reduced pressure. The crude material (57 g) was taken up in MeOH/conc. HCl (5:1, 240 mL) and heated at reflux for 16 h. The MeOH was removed under reduced pressure and the aqueous residue extracted with ethyl acetate (3 × 100 mL). The organic extracts were dried over Na_2SO_4 and concentrated under reduced pressure. The crude residue was purified by flash chromatography (95:5 hexanes:ethyl acetate) to give compound **11** as a colourless oil (27.2 g, 60%). ν_{max} (neat)/cm^{-1}: 2960, 1697, 1442, 1205, 1179, 1113; δ_H (300 MHz, $CDCl_3$): 6.67 (2 H, s, 2 × C<u>H</u>), 3.51 (2 H, t, *J* 7.2, NC<u>H</u>$_2$), 1.59–1.51 (2 H, m, NCH$_2$C<u>H</u>$_2$), 1.33–1.26 (2 H, m, C<u>H</u>$_2$CH$_3$), 0.92 (3 H, t, *J* 7.3, CH$_2$C<u>H</u>$_3$); δ_C (125 MHz, $CDCl_3$), 170.9 (C), 134.0 (CH), 37.6 (CH_2). 30.6 (CH_2), 19.9 (CH_3) 13.5 (CH_3).

Dicyano(phthalazin-2-ium-2-yl)methanide (1)[3, 4]



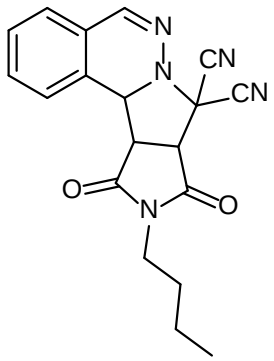
A solution of tetracyanoethylene-oxide (2.02 g, 14 mmol) in THF (100 mL) at 0 °C was treated with phthalazine (3.60 g, 28 mmol) and the resulting mixture stirred for 2 h. The resulting solid was collected by filtration, washed with cold THF (3 × 75 mL) and dried under vacuum to give the title compound as a yellow solid (2.60 g, 95%). mp: 253–255 °C (*lit.*[3] 252–254 °C); ν_{max} (neat)/cm^{-1}: 2190, 2157, 1614, 1566, 1447, 1346, 1250, 1218, 1155; δ_H (500 MHz, DMSO): 9.66 (1 H, s, Ar-H), 9.49 (1 H, s, Ar-H), 8.27 (1 H, d, *J* 8, Ar-U), 8.21 (1 H, d, *J* 8, Ar-H), 8.07 (1 H, t, *J* 8, Ar-H), 7.97 (1 H, t, *J* 8, Ar-H); δ_C (125 MHz, DMSO): 154.4 (CH), 136.0 (CH), 133.5 (CH), 130.2 (C), 129.0 (C), 128.6 (CH), 127.0 (CH), 123.4 (C), 118.0 (CH), 63.9 (C); m/z (ESI): Calculated for $C_{11}H_6N_4Na^+$ 217.0485, found 217.0485.

(*E*)-4-(4-Chlorophenyl)but-3-en-2-one (3)[5]



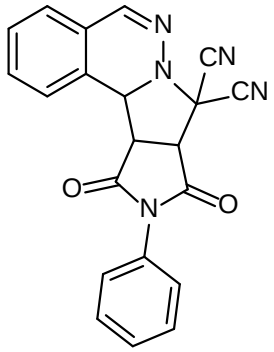
A solution of 4-chlorobenzaldehyde (2.81 g, 20 mmol) in acetone (5 mL) and water (20 mL) was heated to 40 °C and treated with NaOH (100 mg). The reaction was stirred for 16 h then cooled to room temperature. The acetone was removed under reduced pressure and the resulting mixture extracted with ethyl acetate (3 × 20 mL). The organic phase was dried over Na_2SO_4 and concentrated *in vacuo*. The crude residue was purified by flash chromatography (95:5 hexanes:ethyl acetate) to give the title compound as a pale yellow solid (2.90 g, 80%). mp: 55–56 °C (*lit.*[5] 50–54 °C); ν_{max} (neat)/cm^{-1}: 2966, 1607, 1585, 1487, 1404, 1358, 1247, 1198, 1087; δ_H (500 MHz, $CDCl_3$): 7.47 (1 H, d, *J* 8.5, Ar-H), 7.46 (1 H, d, *J* 16.2, CH=CHCO), 7.37 (1 H, d, *J* 8.5, Ar-H), 6.68 (1 H, d, *J* 16.5, CH=CHCO), 2.38 (3 H, s, COCH$_3$); δ_C (125 MHz, $CDCl_3$): 198.0 (C), 141.8 (CH), 136.5 (C), 133.0 (C), 129.4 (CH), 129.3 (CH), 127.5 (CH), 27.7 (CH_3).

10-Butyl-9,11-dioxo-9,10,11,11a-tetrahydro-8*H*-pyrrolo[3',4':3,4]pyrrolo[2,1-a]phthalazine-8,8(8a*H*,11b*H*)-dicarbonitrile (12)[4]



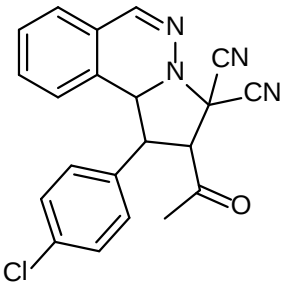
Following the general procedure (on-water/IL) a suspension of dicyano(phthalazin-2-ium-2-yl)methanide (60 mg, 0.3 mmol) and *N*-butylmaleimide (51 µL, 0.3 mmol) in water (4 mL) and [BMIM][NTf_2] (0.3 mL) was stirred vigorously at room temperature for 6 h. The reaction mixture was extracted with ethyl acetate (4 mL), filtered to remove the unreacted dipole and concentrated under reduced pressure. The crude residue was purified by flash chromatography (1:1 hexanes:ethyl acetate) to give the cycloadduct as a colourless powder (99 mg, 95%). Further recrystallization from ethanol gave **12** as a colourless crystalline solid (72 mg, 70%). mp: 169–170 ^{o}C (ethanol); ν_{max} (neat)/cm^{-1}: 2928, 1703, 1444, 1402, 1335, 1261, 1194, 1139, 1103; ; δ_H (500 MHz, $CDCl_3$): 7.74 (1 H, d, *J* 7.7, Ar-H), 7.69 (1 H, s, Ar-H), 7.55 (1 H, t, *J* 7.6, Ar-H), 7.45 (1 H, t, *J* 7.6, Ar-H), 7.34 (1 H, d, *J* 7.4, Ar-H), 4.91 (1 H, d, *J* 7.1, NCHCHCO) 3.90 (1 H, d, *J* 7.3, $CHC(CN)_2$), 3.84 (1 H, t, *J* 7.6, NCHCHCO), 3.48 (2 H, t, *J* 5.5, NCH_2CH_2), 1.40–1.44 ((NCH_2CH_2), 1.24–1.19 (2 H, m CH_2CH_3), 0.84 (3 H, t, *J* 7.4, CH_2CH_3); δ_C (125 MHz, $CDCl_3$): 171.8 (C), 169.5 (C), 147.2 (CH), 131.9 (CH), 129.5 (CH), 129.3 (C), 127.3 (CH), 127.1 (CH), 124.3 (C), 111.6 (C), 109.6 (C), 59.1 (CH), 58.6 (C), 50.7 (CH), 43.7 (CH), 39.9 (CH_2), 29.1 (CH_2), 19.8 (CH_2), 13.4 (CH_2), 13.4 (CH_3); m/z (ESI): Calculated for $C_{19}H_{18}N_5O_2^+$ 348.1455, found 348.1456.

9,11-Dioxo-10-phenyl-9,10,11,11a-tetrahydro-8*H*-pyrrolo[3',4':3,4]pyrrolo[2,1-a]phthalazine-8,8(8a*H*,11b*H*)-dicarbonitrile (14)[4, 6]



Following the general procedure (on-water/IL) a suspension of dicyano(phthalazin-2-ium-2-yl)methanide (60 mg, 0.3 mmol) and *N*-phenylmaleimide (53 mg, 0.3 mmol) in water (4 mL) and [BMIM][NTf_2] (0.3 mL) was stirred vigorously at room temperature for 1 h. The reaction mixture was extracted with ethyl acetate (4 mL), filtered to remove the unreacted dipole and concentrated under reduced pressure. The crude residue was purified by flash chromatography (1:1 hexanes:ethyl acetate) to give the cycloadduct as a colourless powder (101 mg, 90%). mp: 226–228 ^{o}C (*lit.*[6] 252–254 ^{o}C); ν_{max} (neat)/cm^{-1}: 1713, 1495, 1392, 1185, 1064; δ_H (500 MHz, $CDCl_3$): 7.72 (1 H, s, Ar-H), 7.71 (1 H, d, *J* 8.7, Ar-H), 7.54 (1 H, t, *J* 7.1, Ar-H), 7.45–7.41 (3 H, m, Ar-H), 7.39 (1 H, d, *J* 7.3, Ar-H), 7.34 (1 H, d, *J* 7.6, Ar-H), 7.19 (2 H, d, *J* 7.4, Ar-H), 5.04 (1 H, d, *J* 7.2, NCHCHCO) 4.10 (1 H, d, *J* 8.1, CHC$(CN)_2$), 4.00 (1 H, t, *J* 7.7, NCHCHCO); δ_C (125 MHz, $CDCl_3$): 170.9 (C), 168.6 (C), 147.0 (CH), 132.1 (CH), 130.8 (C), 129.7 (CH), 129.3 (CH), 128.9 (C), 127.4 (CH), 127.2 (CH), 125.9 (CH), 124.1 (C), 111.7 (C), 109.4 (C), 59.7 (CH), 59.3 (C), 51.32 (CH), 44.1 (CH). m/z (ESI): Calculated for $C_{22}H_{18}N_5O_3^+$ 400.1404, found 400.1404.

2-Acetyl-1-(4-chlorophenyl)-1,10b-dihydropyrrolo[2,1-a]phthalazine-3,3(2*H*)-dicarbonitrile (5)[3, 4]



Following the general procedure (on-water/IL) a suspension of dicyano(phthalazin-2-ium-2-yl)methanide (60 mg, 0.3 mmol) and (*E*)-4-(4-chlorophenyl)but-3-en-2-one (54 mg, 0.3 mmol) in water (4 mL) and [BMIM][NTf_2] (0.3 mL) was stirred vigorously at 40 ^oC for 24 h. The reaction was extracted with ethyl acetate (4 mL), filtered to remove the unreacted dipole and concentrated under reduced pressure. The crude material contained a mixture of two diastereomers (10:1). Flash chromatography (1:1 hexanes:ethyl acetate) gave the major isomer as a pale yellow solid (29 mg, 25%). mp: 161–163 ^oC (*lit.*[3] 162–163 ^oC); 1708, 1494, 1358, 1159, 1093, 1014; δ_H (500 MHz, $CDCl_3$): 7.64 (1 H, s, Ar-H), 7.50–7.42 (6 H, m, Ar-H), 7.35 (1 H, d, 7.1, Ar-H), 7.22 (1 H, d, *J* 7.4, Ar-H), 5.26 (1 H, d, *J* , 9.1, NCHCHCO), 4.27 (1 H, d, *J* 7.9, CHC$(CN)_2$), 3.87 (1 H, t, *J* 8.5, NCHCHCO), 1.89 (3 H, s, $COCH_3$); δ_C (125 MHz, $CDCl_3$): 204.3 (C), 144.4 (CH), 136.1 (C), 132.0 (CH), 130.2 (C), 130.0 (CH), 129.8 (CH), 129.8 (CH), 129.3 (C), 127.4 (CH), 126.1 (CH), 124.3 (C), 111.8 (C), 111.1 (C), 65.7 (C), 59.3 (CH), 58.8 (CH), 57.1 (CH), 31.4 (CH_3); m/z (ESI): Calculated for $C_{21}H_{15}Cl_{35}ON_4Na^+$ 397.0827, found 397.0826.

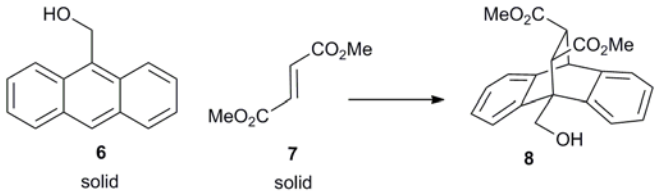


Table ESI-1. Effect of varying the amount of ionic liquid in the emulsion.

[BMIM][NTf$_2$]	*Experiment 1*	*Experiment 2*	*Average*	*IL:anthracene (mol:mol)*
1 mL	49%	55%	52%	6.8:1
0.5 mL	57%	64%	61%	3.4:1
0.25 mL	44%	50%	47%	1.7:1
0.1 mL	50%	39%	45%	0.7:1
0.05 mL	9%	16%	13%	0.3:1

Conditions: 9-methanol anthracene (0.5 mmol), dimethyl fumarate (0.5 mmol) water (4 mL), varied amounts of [BMIM][NTf$_2$] as co-solvent, 16 h, 80 ^{o}C. Results given as %conversion based on ^{1}H NMR integration.

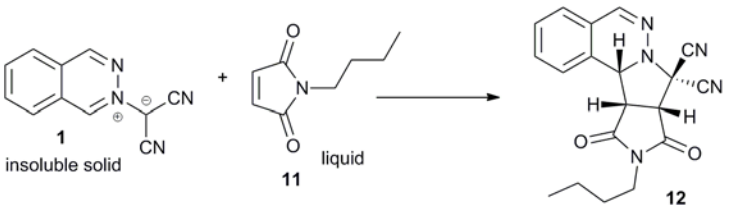


Table ESI-2. Reaction time required for completion.

Solvent	*Conversion after 6 h*	*Conversion after 16 h*
On-water	88%	100%
At-Water	53%	77%
Neat	84%	87%
EtOH	85%	-
[BMIM][NTf_2]	79%	-
Water/[BMIM][NTf_2]	100%	-
Water/toluene	74%	-
Toluene	78%	-

Conditions: Phthalazinium (0.5 mmol), *N*-butylmaleimide (0.5 mmol), water (4 mL), co-solvent (0.5 mL), at room temperature.

The dipole is very insoluble in most NMR solvents, so the reaction mixtures were diluted with ethyl acetate (4 mL), filtered and concentrated *in vacuo*. The percentage conversion was calculated relative to the remaining maleimide.

Additionally, the remaining insoluble dipole was collected after 6 hours, and the mass recovery was compared with the conversion by ^{1}H NMR analysis.

Table ESI-3. Comparison of conversion and mass recovery.

Solvent	*% conversion (by mass)*	*% conversion (by NMR)*
On-water	75-80%	76%
Neat	80-85%	82%

For both the on-water and neat reactions, the percentage conversion calculated by mass balance and by NMR was consistent; indicating that calculating conversion by NMR was a valid method.

The reactions for comparison of *endo*:*exo* ratios was conducted at 40 °C to ensure that minor fluctuations in ambient temperature (e.g. overnight) did not affect the reaction outcome.

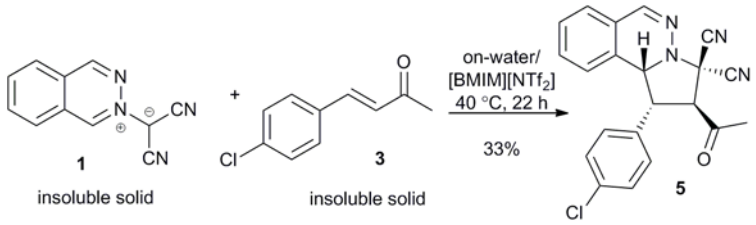


Table ESI-4. Comparison of *endo*:*exo* selectivity as a function of solvent. [4]

Experiment	*Conversion after 22 h*	*Conversion after 22 h*	*endo*:*exo*
On-water	0	0	-
Neat	0	0	-
[BMIM][NTf_2]	23%	15%	10:1
Water/[BMIM][NTf_2]	31%	35%	10:1
Water/toluene	0	0	-

Conditions: Phthalazinium (0.5 mmol), α,β -unsaturated ketone (0.5 mmol), water (4 mL), co-solvent (0.5 mL), at 40°C.

Ionic Liquid Re-use. The recyclability of ionic liquids is one of the attractive features of these reaction media and several recent examples have been published that re specific to the recovery and re-use of [BMIM][NTf_2], covering a range of reaction types.[7-12] In this work, the reuse of [BMIM][NTf_2] as a liquefier was demonstrated.

Following the general procedure (On-Water/IL), a mixture of 9-anthracenemethanol (104 mg, 0.50 mmol) and dimethyl fumarate (72 mg, 0.50 mmol) in water (4.0 mL) and [BMIM][NTf_2] (0.5 mL) was stirred vigorously at 80 ^{o}C for 20 h. The resulting mixture was extracted with EtOAc (5 mL) and the organic phase concentrated under reduced pressure. The residue was extracted repeatedly with toluene (5 × 1 mL) to give the IL, which was dried under vacuum and used in subsequent cycles without further purification. The organic extracts were concentrated under reduced pressure to give a mixture of the Diels-Alder adduct and starting materials and analysed by ^{1}H NMR.

Table ESI-5. Recycling [BMIM][NTf_2]

Cycle	*Recovery of IL (based on initial 0.5 mL)*	*Mass Recovery (SM and DA adduct)*	*% Conversion (by NMR)*
1	100%	72%	81%
2	96%	110%	76%
3	95%	101%	74%

Cycle	*Recovery of IL*	*Mass Recovery (SM and DA adduct)*	*% Conversion (by NMR)*
1	94%	82%	82%
2	89%	97%	81%
3	87%	95%	77%

References

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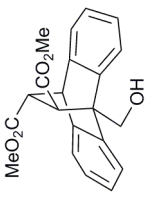
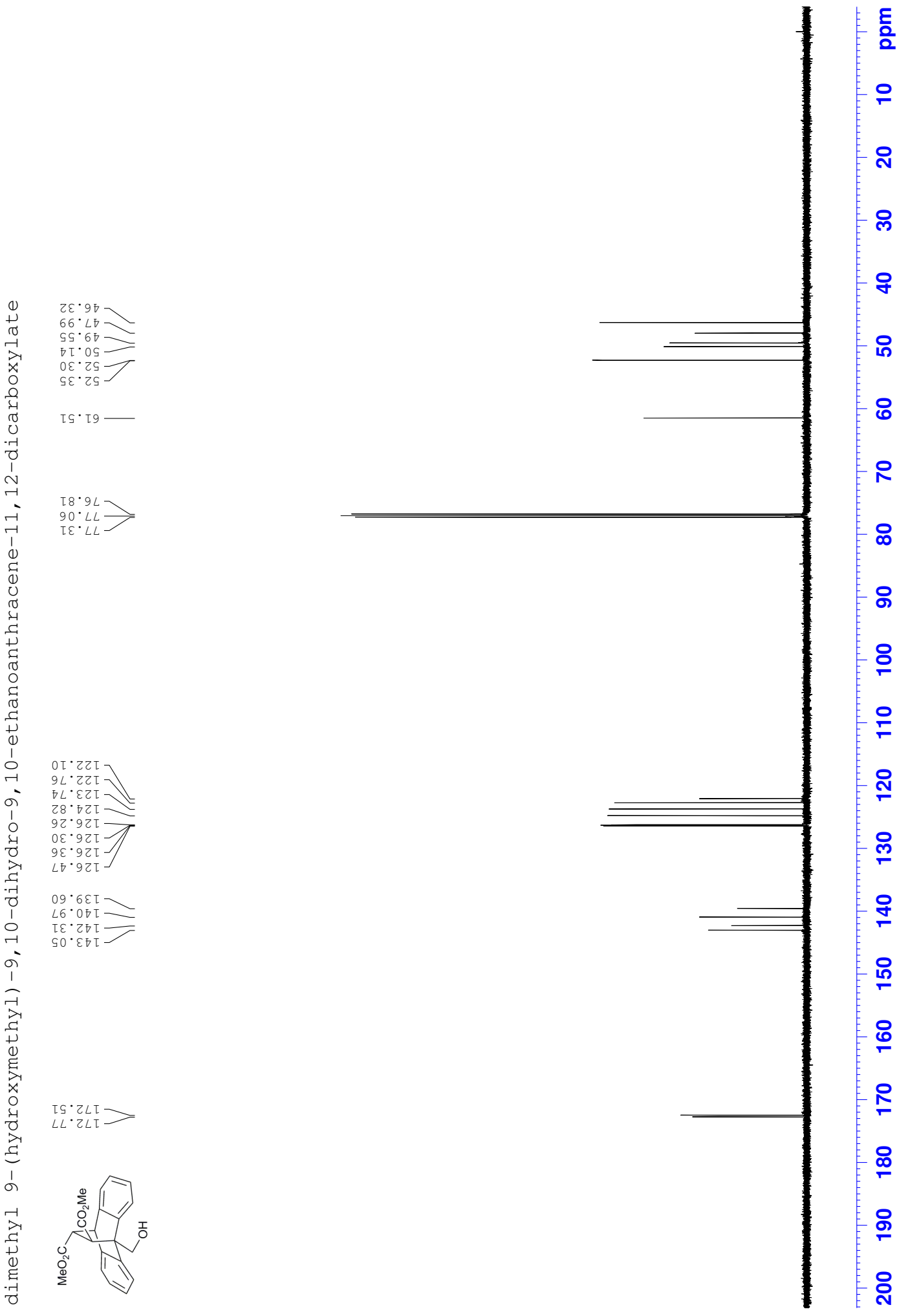
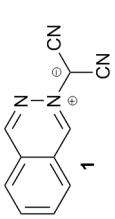
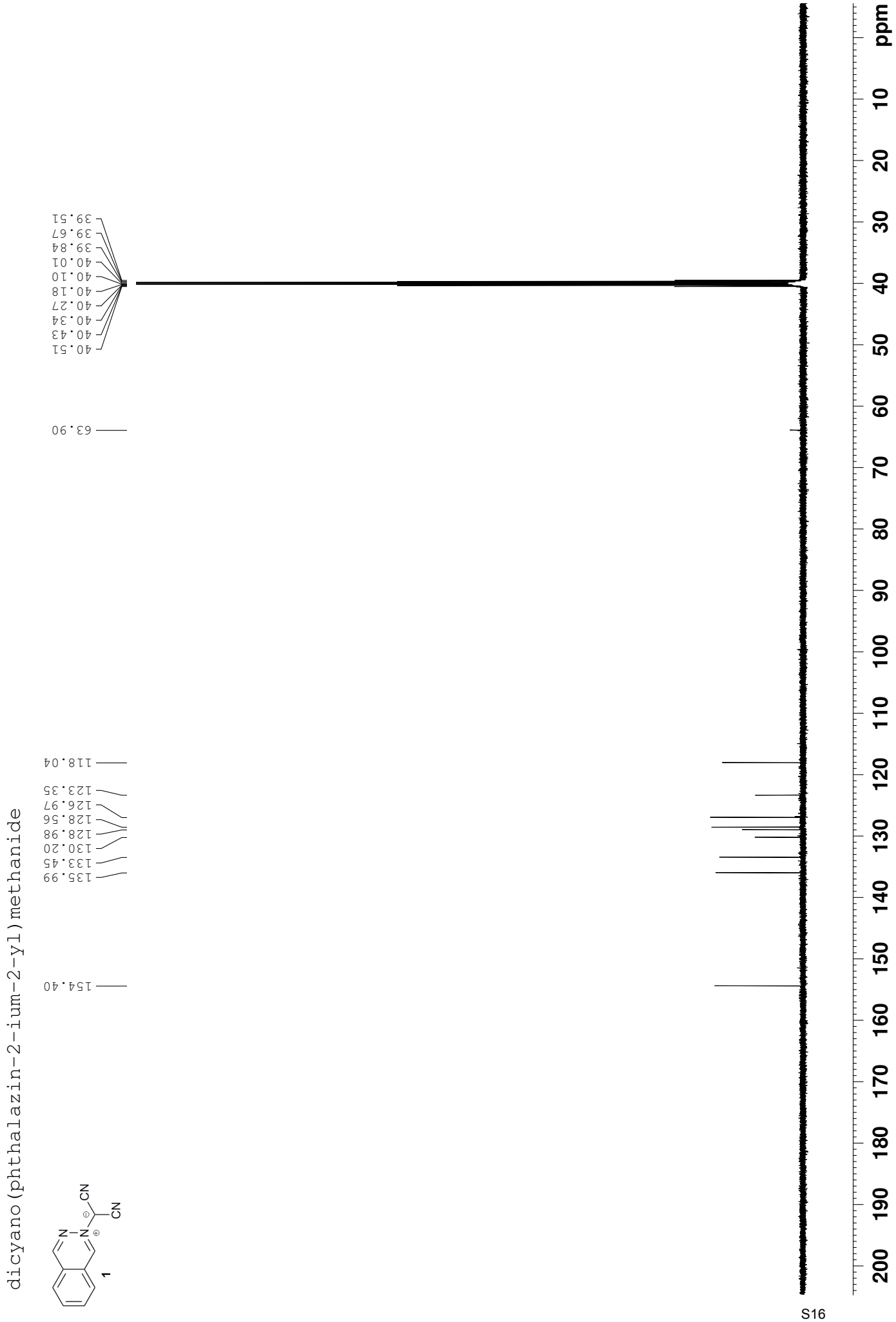
dimethyl 9-(hydroxymethyl)-9,10-dihydro-9,10-ethanoanthracene-11,12-dicarboxylate

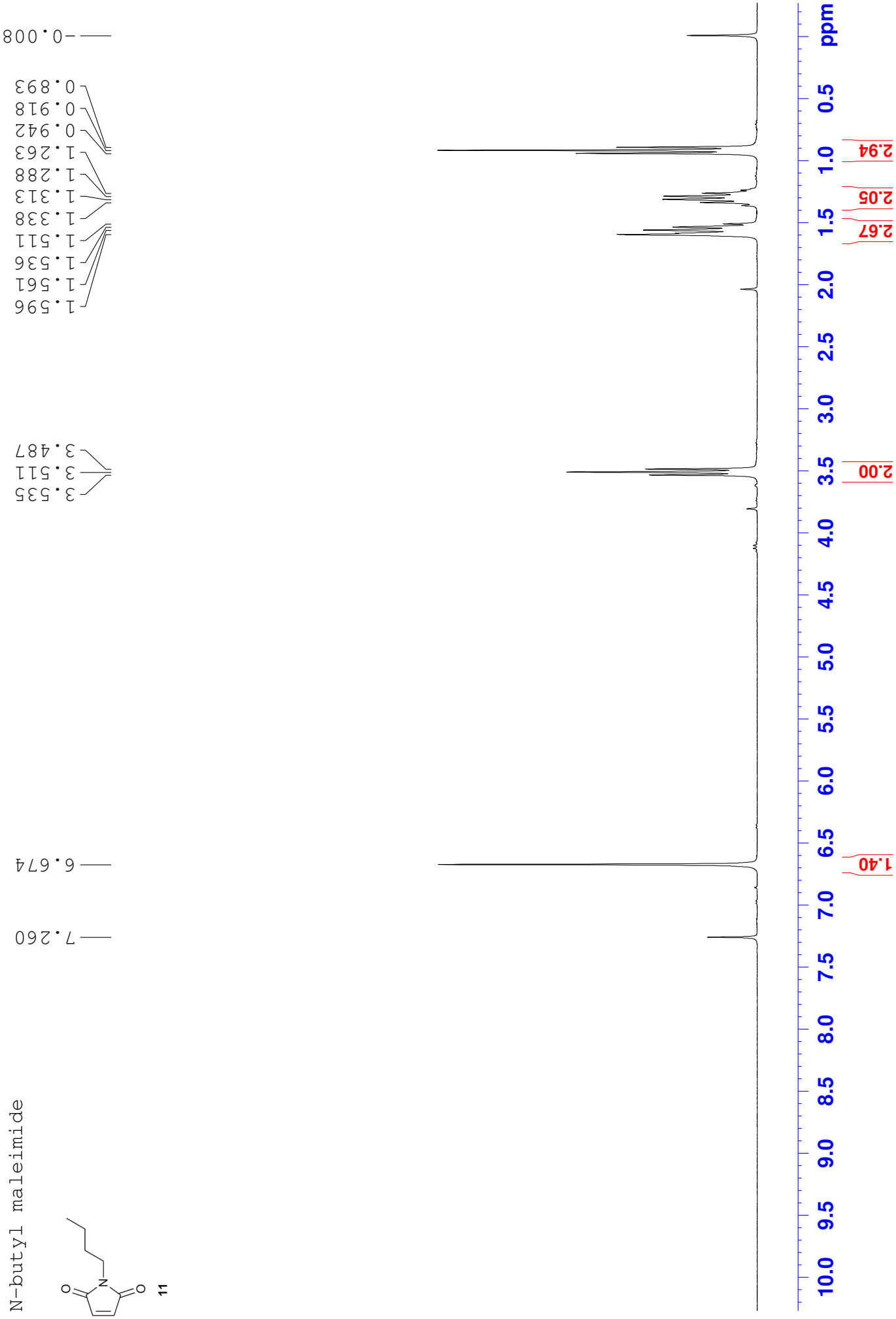


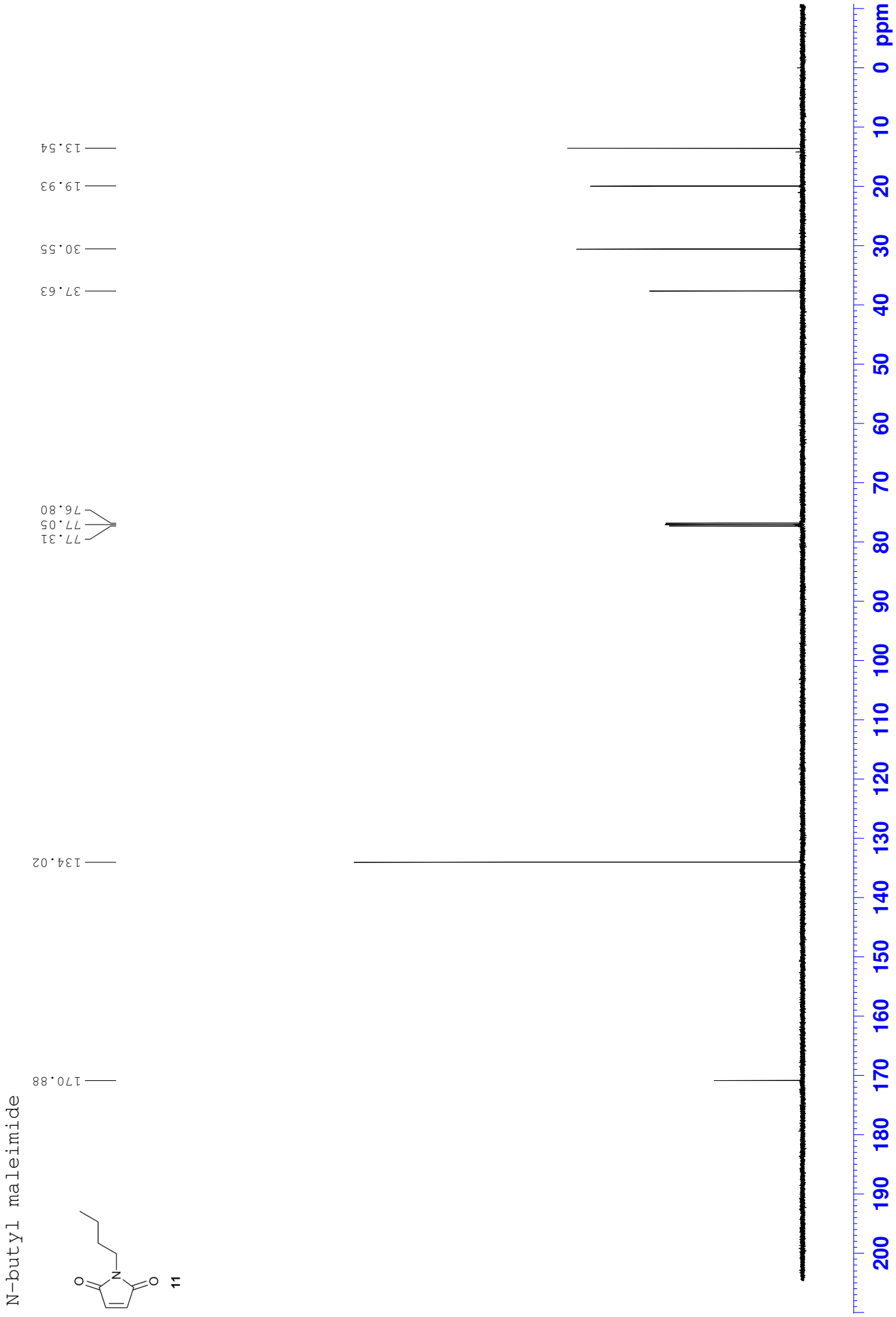


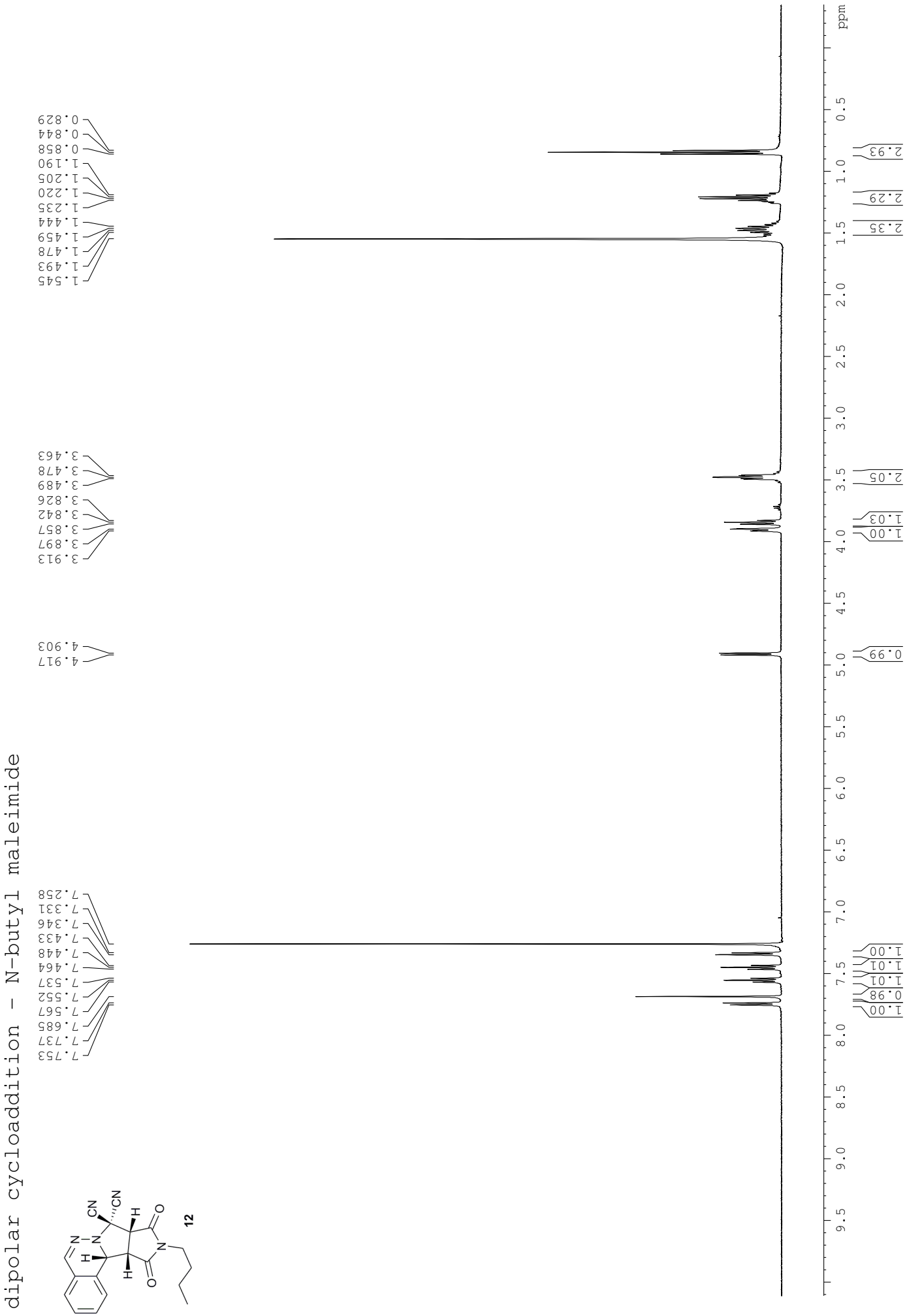
dicyano(phthalazin-2-ium-2-yl)methanide

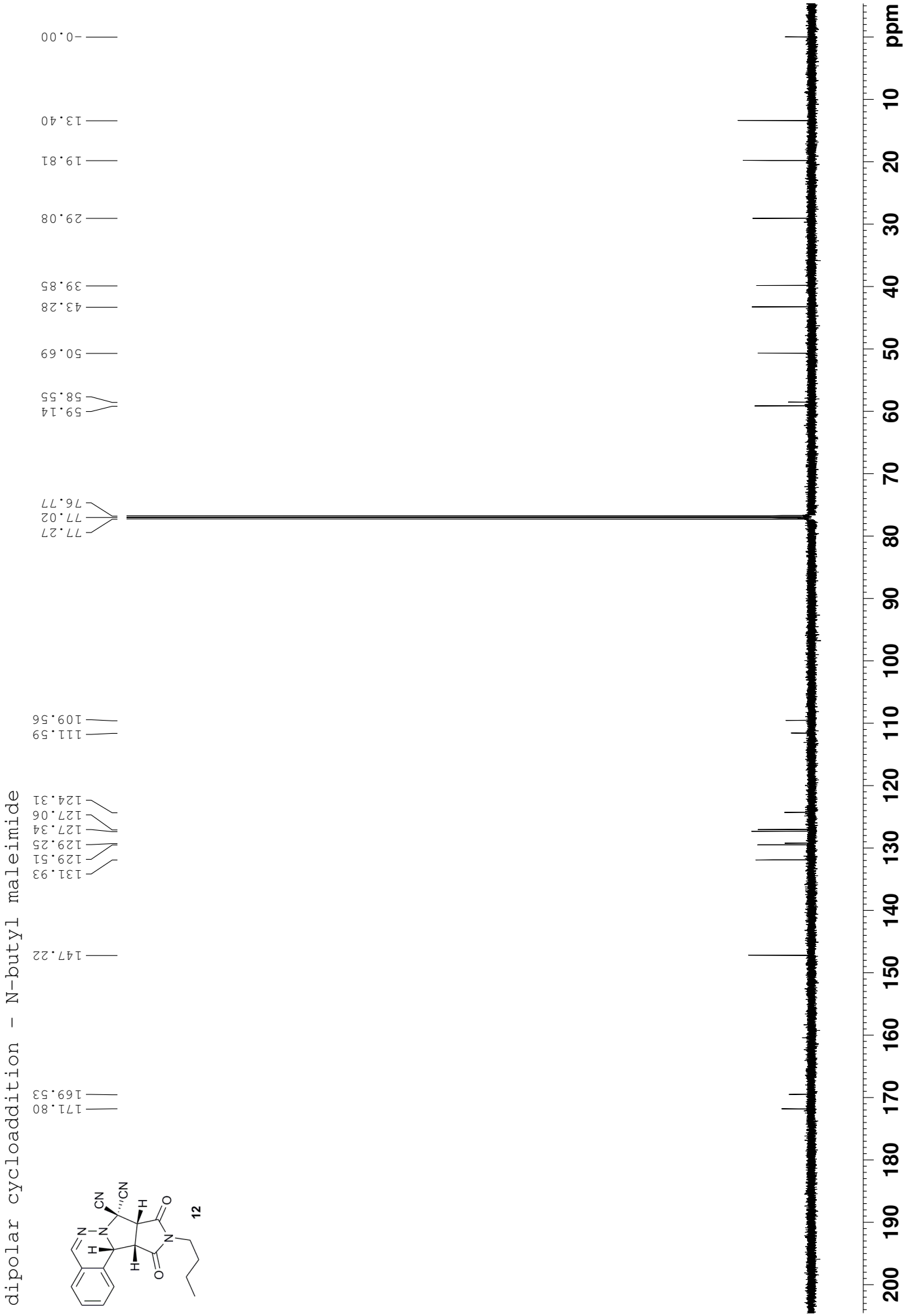



dicyano(phthalazin-2-ium-2-yl)methanide
1
CN
CN
N
N
154.40
135.99
133.45
130.20
128.98
128.56
126.97
123.35
118.04
63.90
40.51
40.43
40.34
40.27
40.18
40.10
40.01
39.84
39.67
39.51
ppm
10
20
30
40
50
60
70
80
90
100
110
120
130
140
150
160
170
180
190
200





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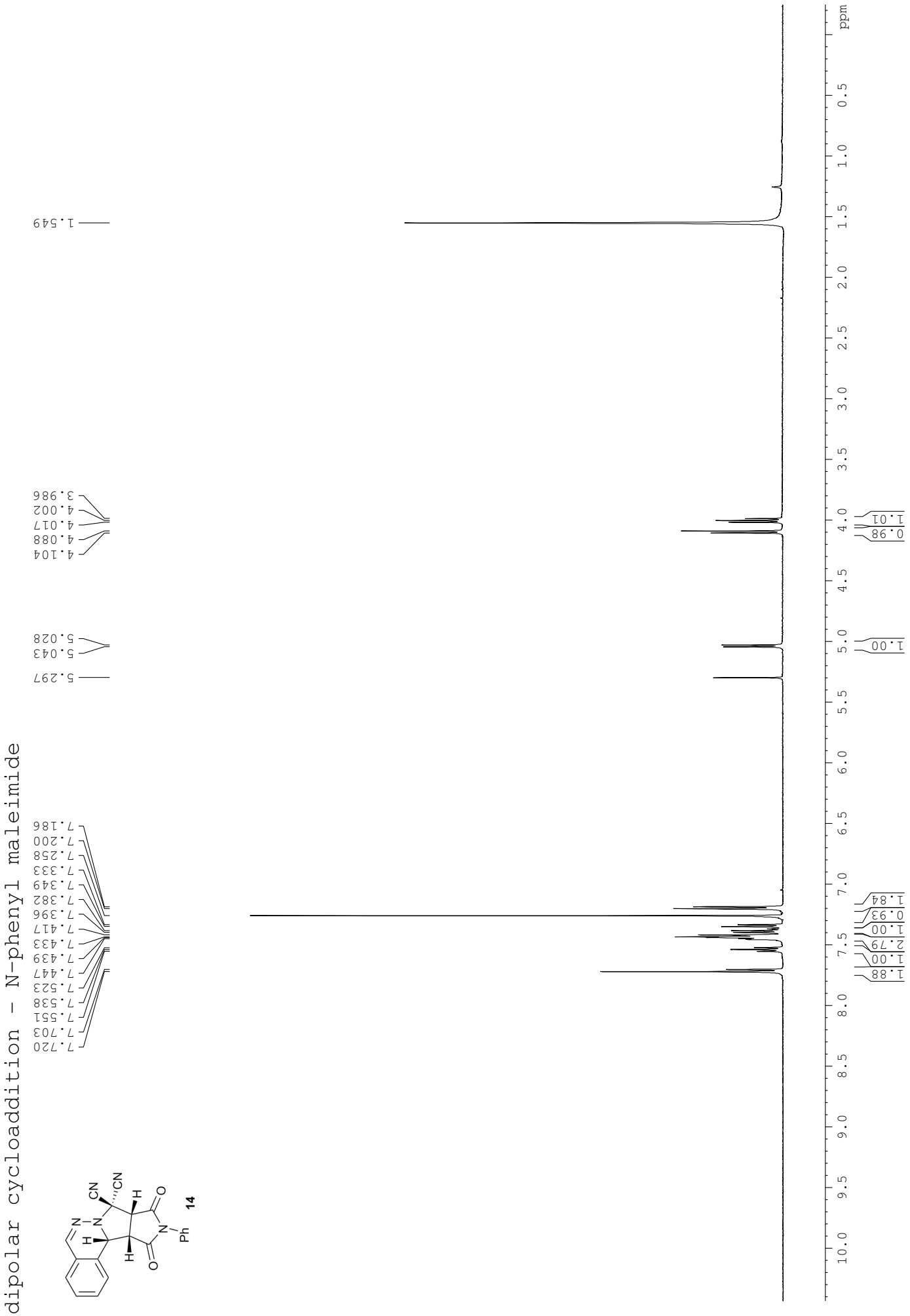
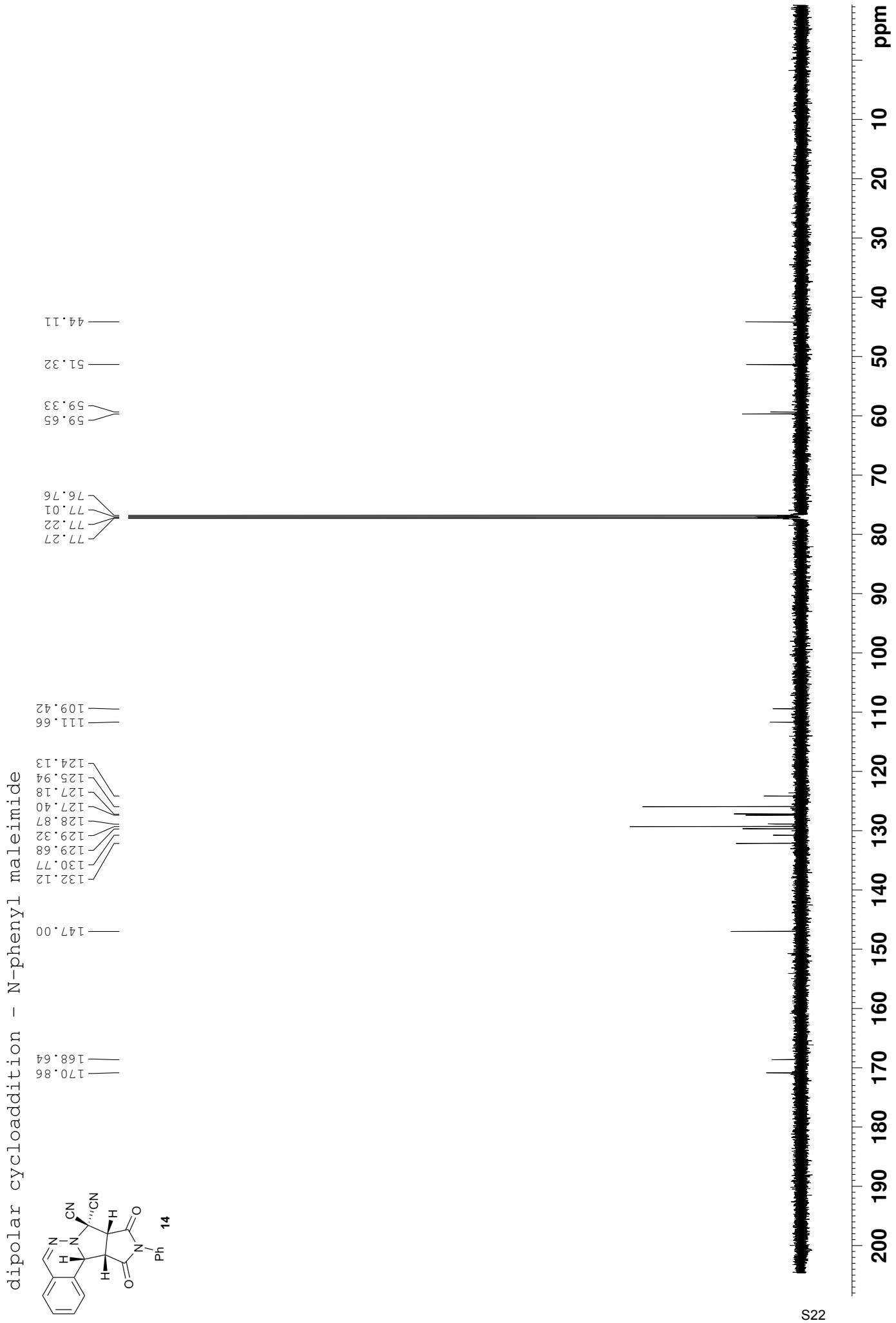
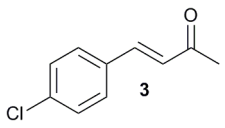
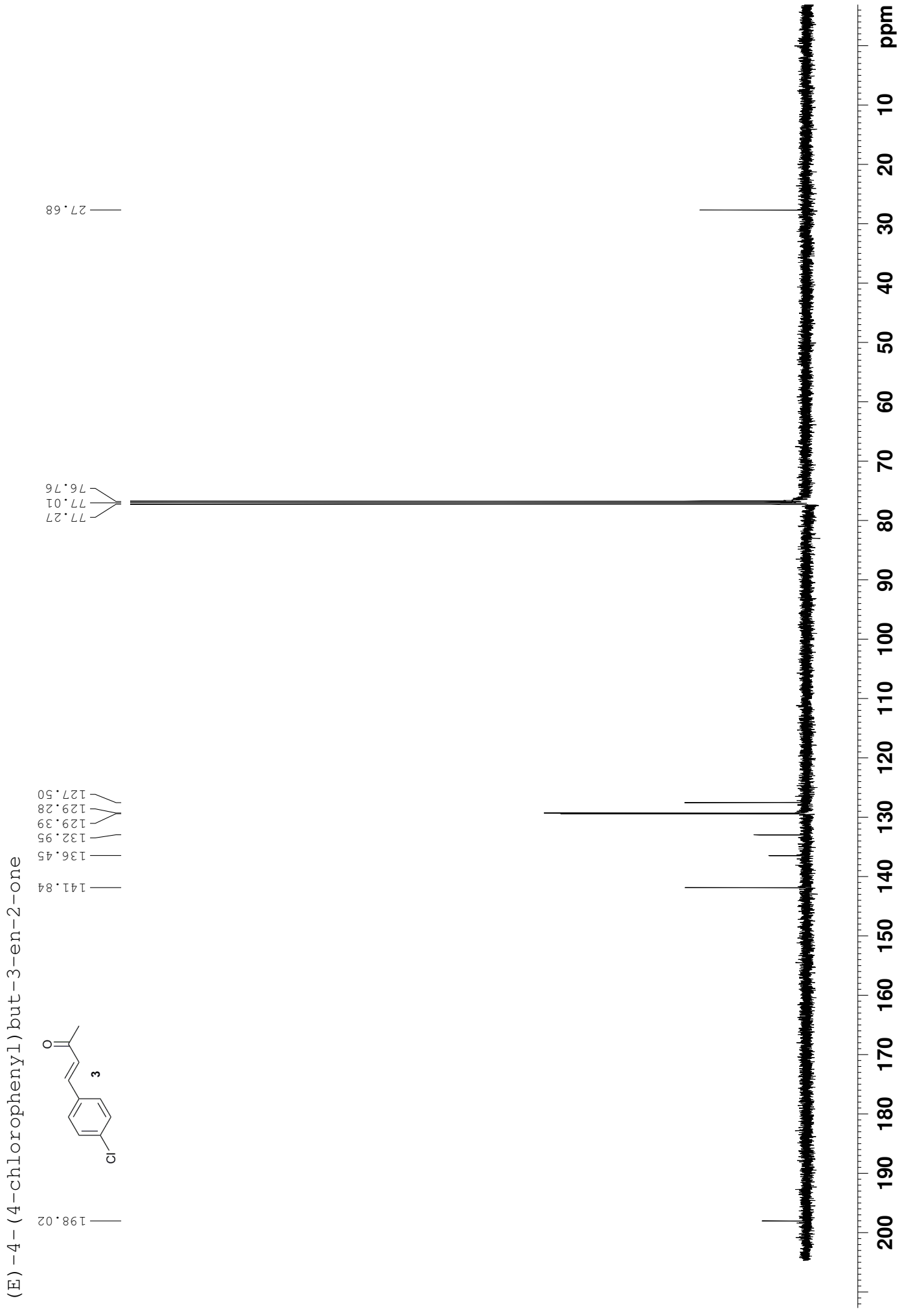
-0.00
13.40
19.81
29.08
39.85
43.28
50.69
58.55
59.14
76.77
77.02
77.27
109.56
111.59
124.31
127.06
127.34
129.25
129.51
131.93
147.22
169.53
171.80
ppm
10
20
30
40
50
60
70
80
90
100
110
120
130
140
150
160
170
180
190
200
CN
CN
H
O
N
N
H
H
O
N
12





(E)-4-(4-chlorophenyl)but-3-en-2-one




(E)-4-(4-chlorophenyl)but-3-en-2-one
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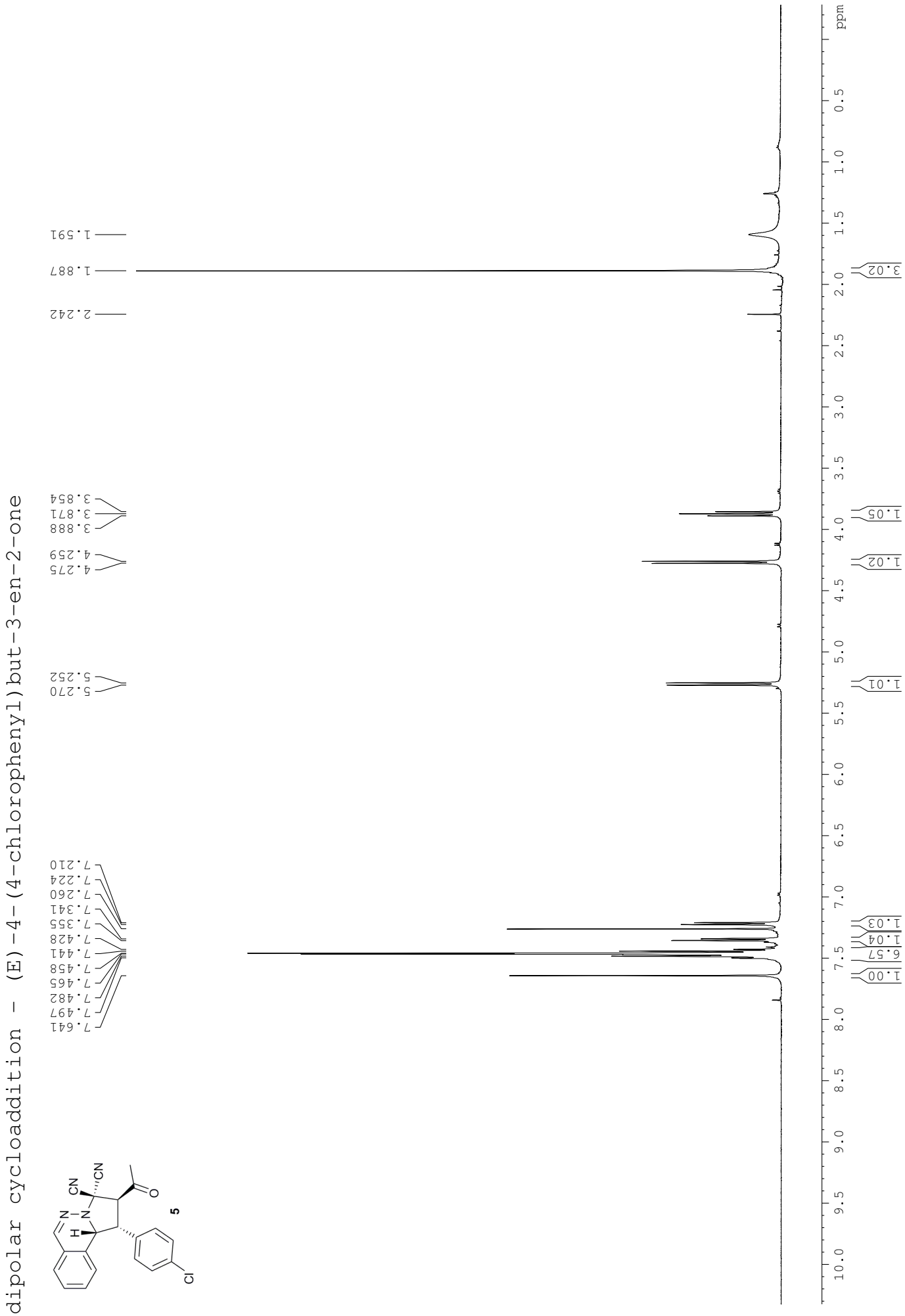
141.84
136.45
132.95
129.39
129.28
127.50
77.27
77.01
76.76
27.68
O
3
Cl
ppm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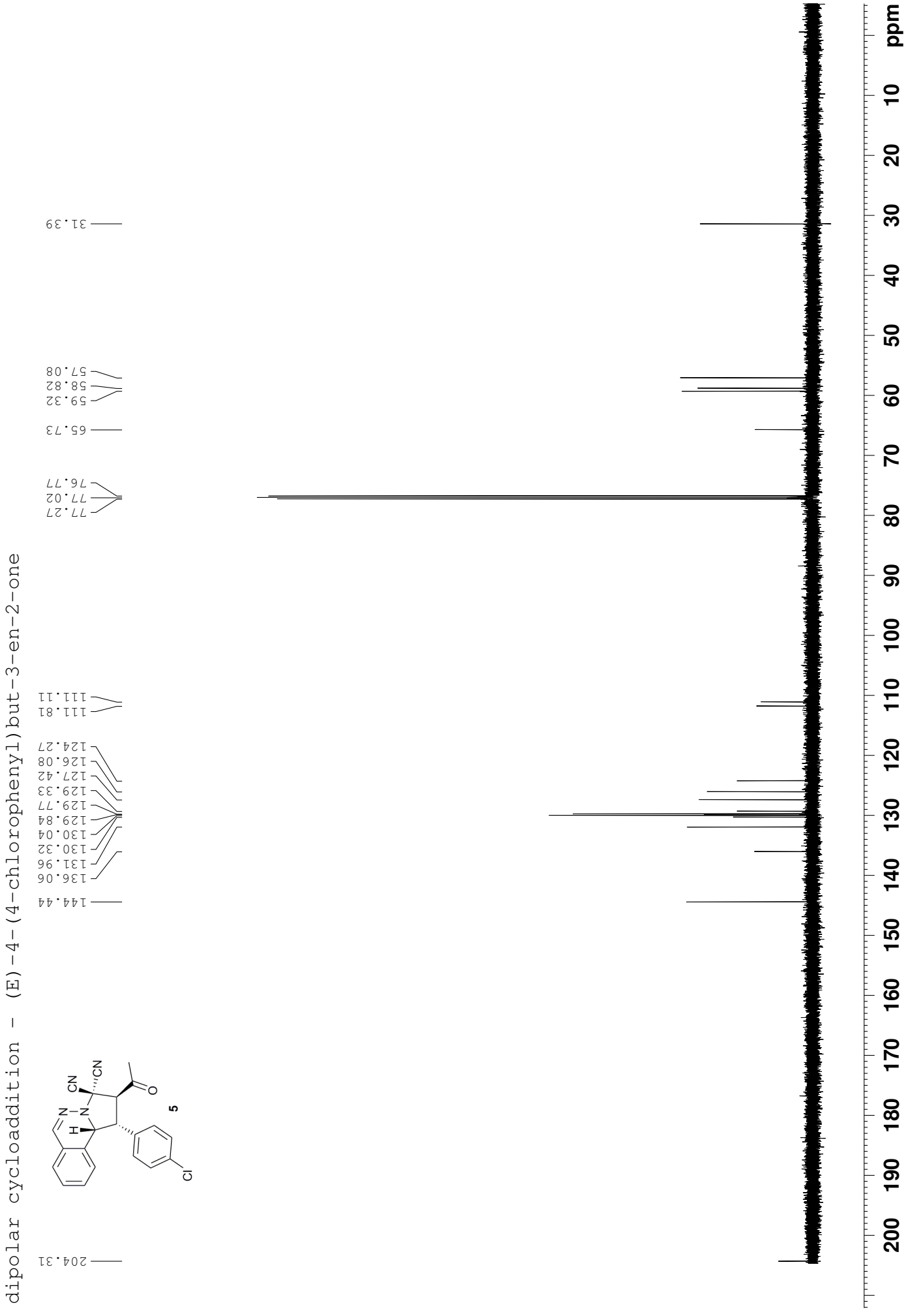
1.591
1.887
2.242
3.854
3.871
3.888
4.259
4.275
5.252
5.270
7.210
7.224
7.260
7.341
7.355
7.428
7.441
7.458
7.465
7.482
7.497
7.641
ppm
3.02
1.05
1.02
1.01
1.03
1.04
6.57
1.00
CN
CN
O
N
N
H
Cl
5


dipolar cycloaddition - (E)-4-(4-chlorophenyl)but-3-en-2-one
31.39
57.08
58.82
59.32
65.73
76.77
77.02
77.27
111.11
111.81
124.27
126.08
127.42
129.33
129.77
129.84
130.04
130.32
131.96
136.06
144.44
204.31
CN
CN
O
N
N
H
5
Cl
ppm
10
20
30
40
50
60
70
80
90
100
110
120
130
140
150
160
170
180
190
200