10.1071/CH15002_AC

Australian Journal of Chemistry 2015, 68(10), 1472-1478

Supplementary Material

Crystal structure studies towards the synthesis and applications of NHC-metal complexes derived from [2.2]paracyclophane

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Experimental Section

Materials and methods.

All chemicals were commercially purchased and used as received.

Solvents were reagent grade and purified by standard techniques. ^{1}H and ^{13}C spectra were recorded on Bruker AVANCE 300 at 298K. Chemical shifts were reported as δ values in ppm and referenced to using tetramethylsilane as an internal. Data are reported as (s = singlet, d = doublet, dd = doublet of doublet, ddd = doublet of doublet of doublet, t = triplet, dt = doublet of triplet, td = triplet of doublet, q = quartet, qd = quartet of doublet, m = multiplet, brs = broad singlet). Coupling constants were reported in Hertz (Hz). All high resolution mass spectra were obtained on an Agilent Technologies 6510 Q-Tof LC/MS. All reactions were monitored by TLC with silica gel-coated plates and visualized with a UV light at 254 nm. Melting points were recorded on a melting point apparatus and were uncorrected. Optical rotations were measured on a automatic polarimeter WZZ-2B equipped with a sodium lamp (λ = 589 nm) and reported as follows: $[\alpha]_D^T$ (c = g/100 mL, solvent). Enantiomeric excess was determined using HPLC on Chiralpak IA chiral column.

X-ray Crystallography.

The crystallographic data of **1-7** were collected on a Bruker Smart APEX CCD area-detector diffractometer with Mo-Kα radiation (λ = 0.71073 Å). The crystal data were solved by direct methods and refined by a full-matrix least-square method on F^2 using the *SHELXL-97* crystallographic software package.[S1] Full crystallographic data for **1-7** have been deposited with the **CCDC 1023337**, **CCDC 753625, CCDC**

1015372, CCDC 821397, CCDC 1017723, CCDC 1017741 and **CCDC 903887**.

These data can be obtained free of charge from The Cambridge Crystallographic Data Centre *via* www.ccdc.cam.ac.uk/data_request/cif.[S2]

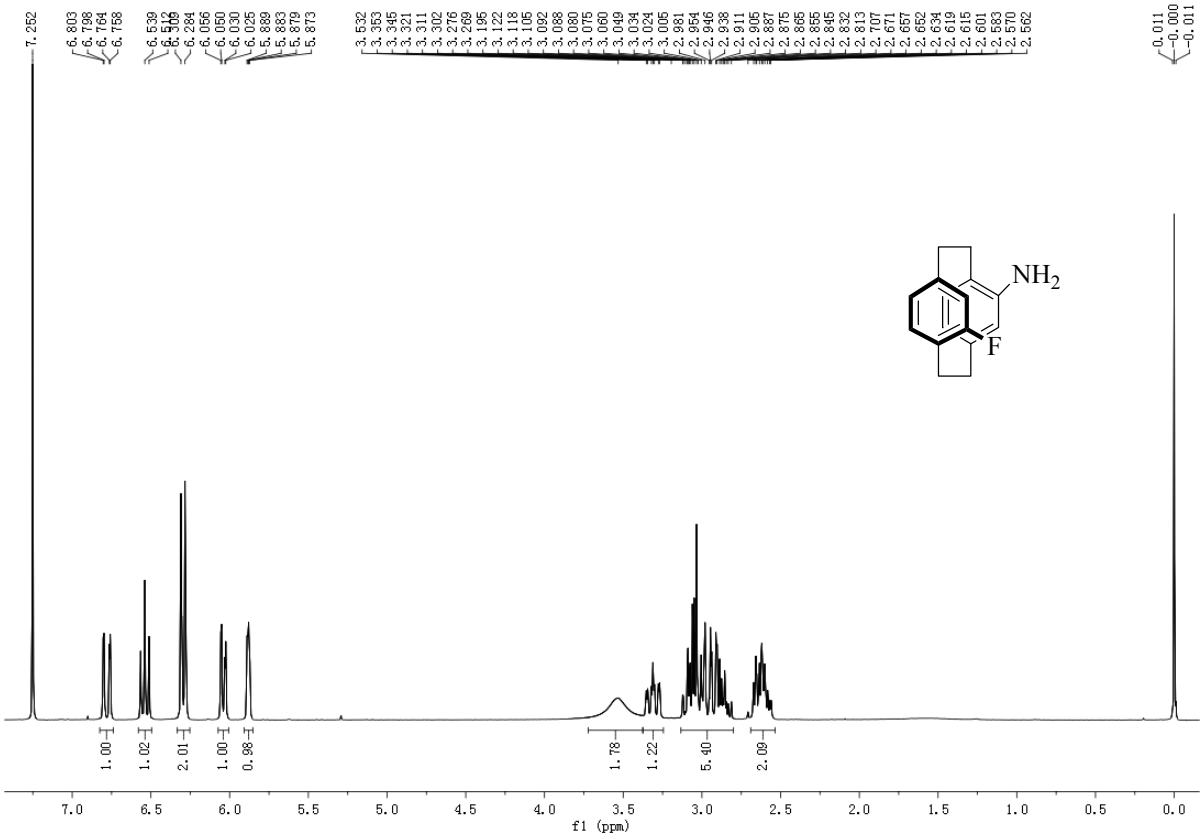
References

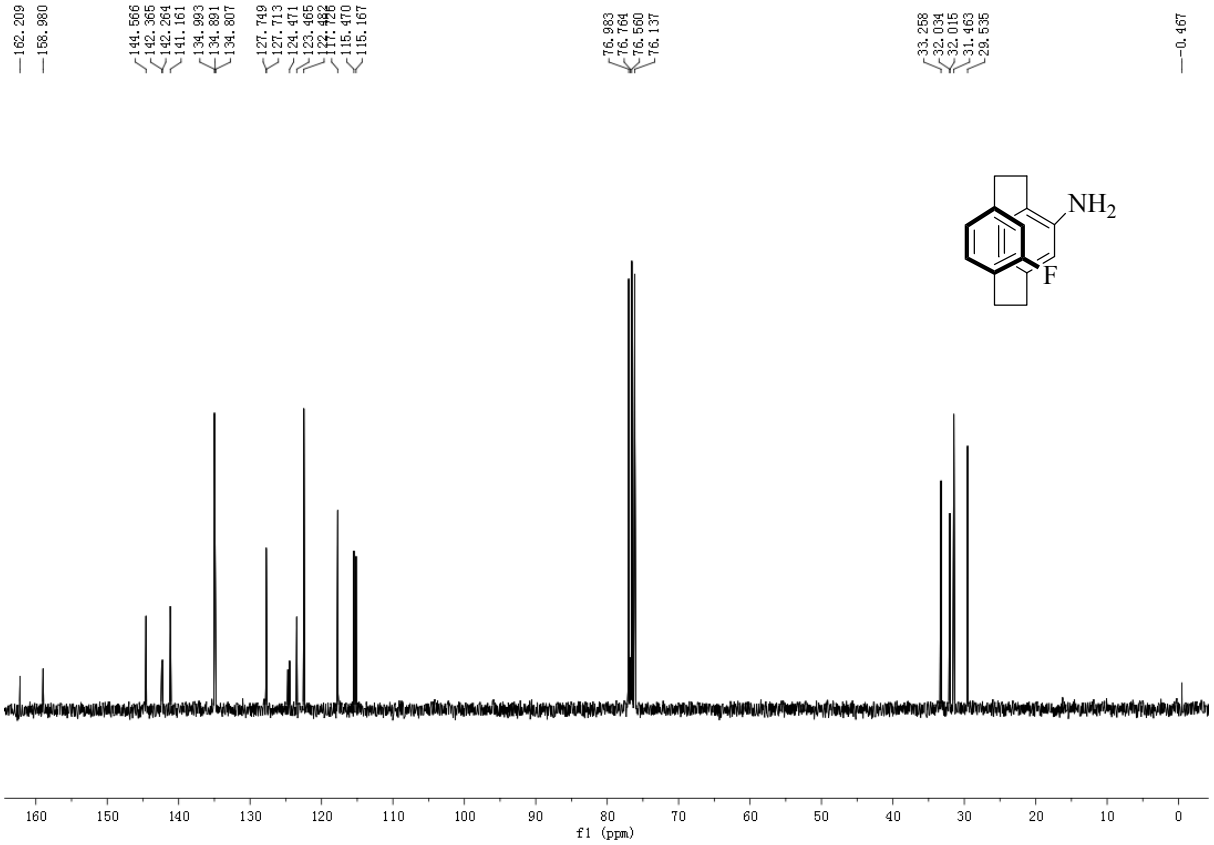
S1 (a) G. M. Sheldrick, *SHELXL97, Program for Crystal Structure Refinement*; University of Göttingen: Göttingen, Germany, 1997; (b) G. M. Sheldrick, *SHELXS97, Program for Crystal Structure Solution*; University of Göttingen: Göttingen, Germany, 1997.

S2 The checkcif program available at:

http://journals.iucr.org/services/cif/checkcif.html.

Spectra copies

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142.264
141.161
134.993
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127.749
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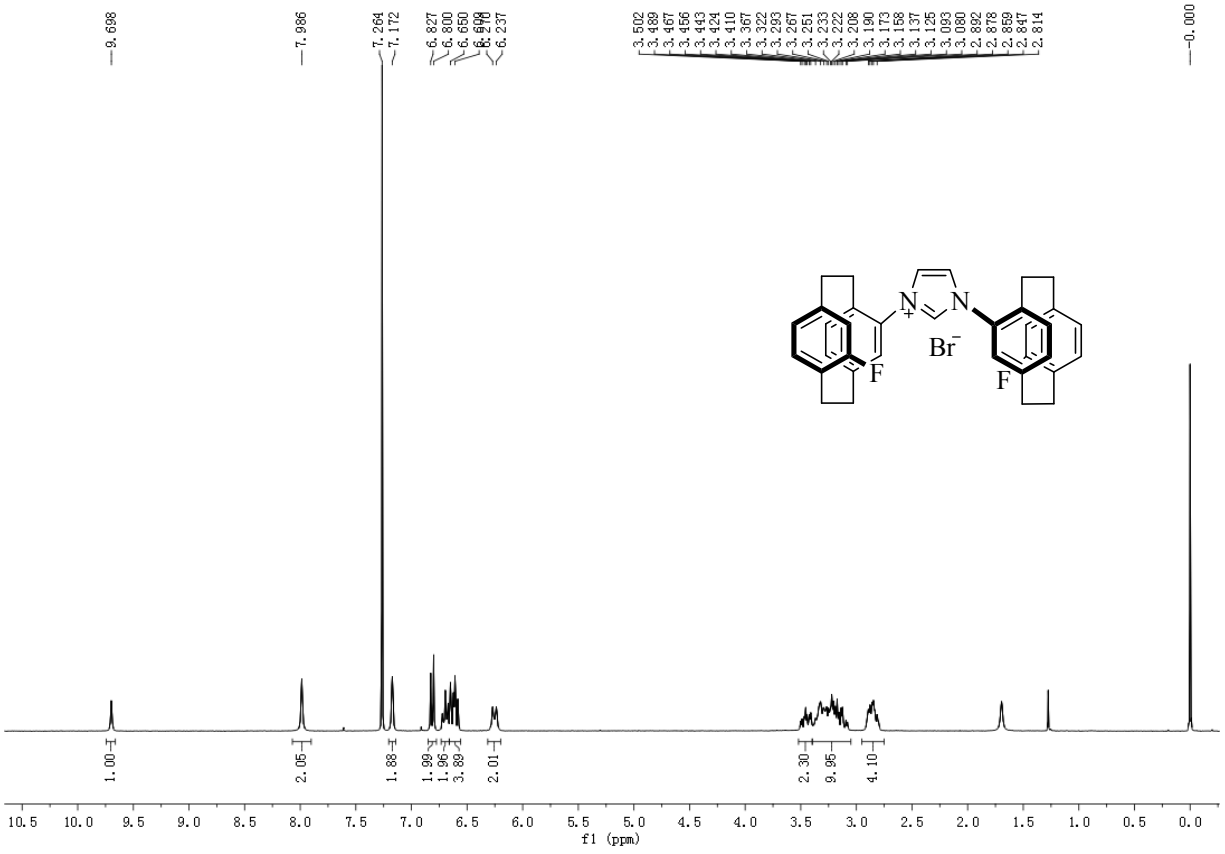
123.465
121.482
115.470
115.167
76.983
76.764
76.560
76.137
33.258
32.034
32.015
31.463
29.535
0.467
NH2
F
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)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6.237
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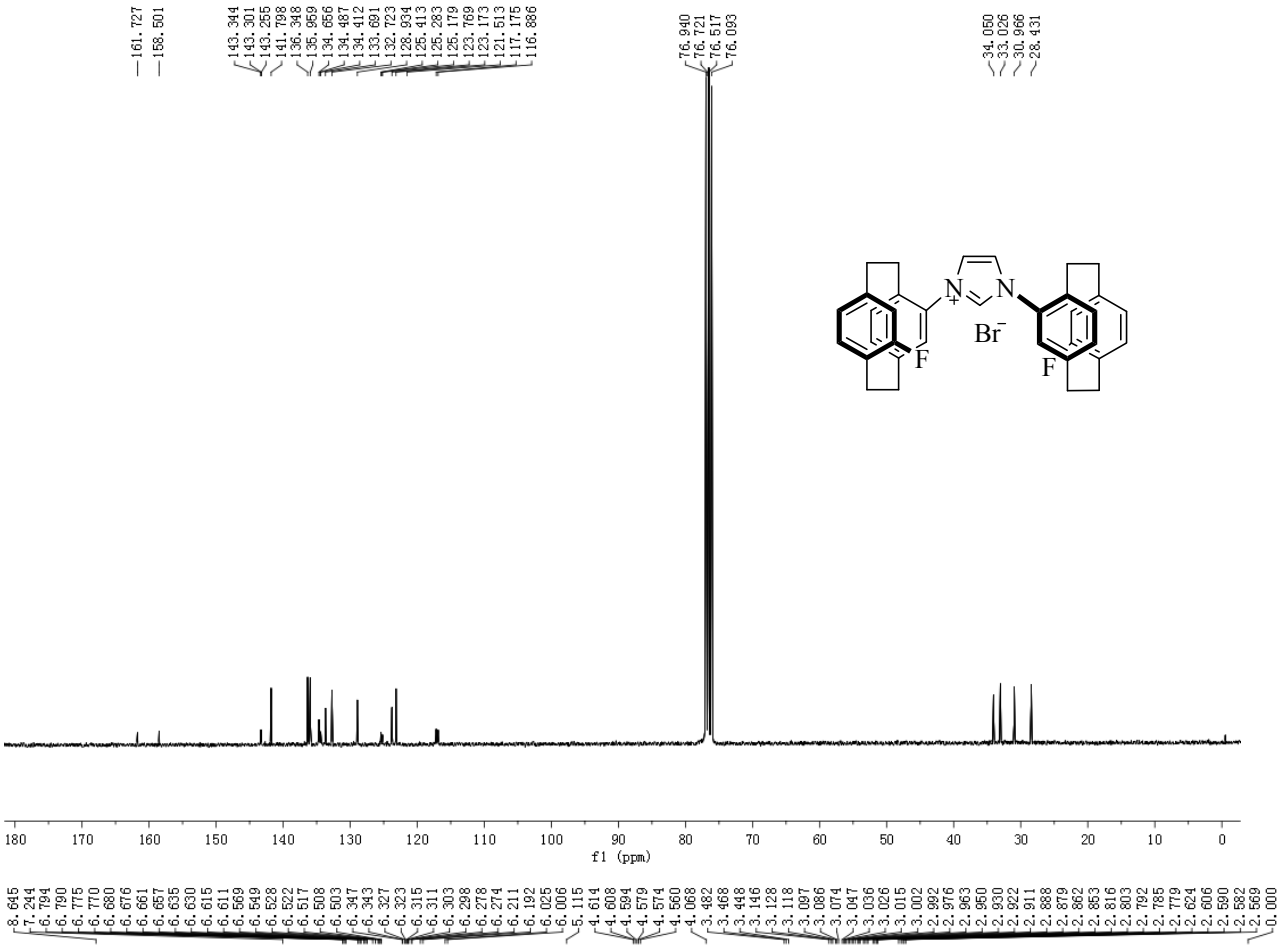
3.443
3.424
3.410
3.367
3.322
3.293
3.267
3.251
3.233
3.222
3.208
3.190
3.173
3.158
3.137
3.125
3.093
3.080
2.892
2.878
2.859
2.847
2.814
0.000
N+
N
Br−
F
F
1.00
2.05
1.88
1.99
1.96
3.89
2.01
2.30
9.95
4.10
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)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128.934
126.413
126.283
126.179
123.769
123.173
121.513
117.175
116.886
76.940
76.721
76.517
76.093
34.050
33.026
30.966
28.431
N
N
Br
F
F
f1 (ppm)


8.645
7.244
6.794
6.790
6.775
6.770
6.680
6.676
6.661
6.657
6.635
6.630
6.615
6.611
6.569
6.549
6.528
6.522
6.517
6.508
6.503
6.347
6.343
6.327
6.323
6.315
6.311
6.303
6.298
6.278
6.274
6.211
6.192
6.025
6.006
5.115
4.614
4.608
4.594
4.579
4.574
4.560
4.068
3.482
3.468
3.448
3.146
3.128
3.118
3.097
3.086
3.074
3.047
3.036
3.026
3.015
3.002
2.992
2.976
2.963
2.950
2.930
2.922
2.911
2.888
2.879
2.862
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2.779
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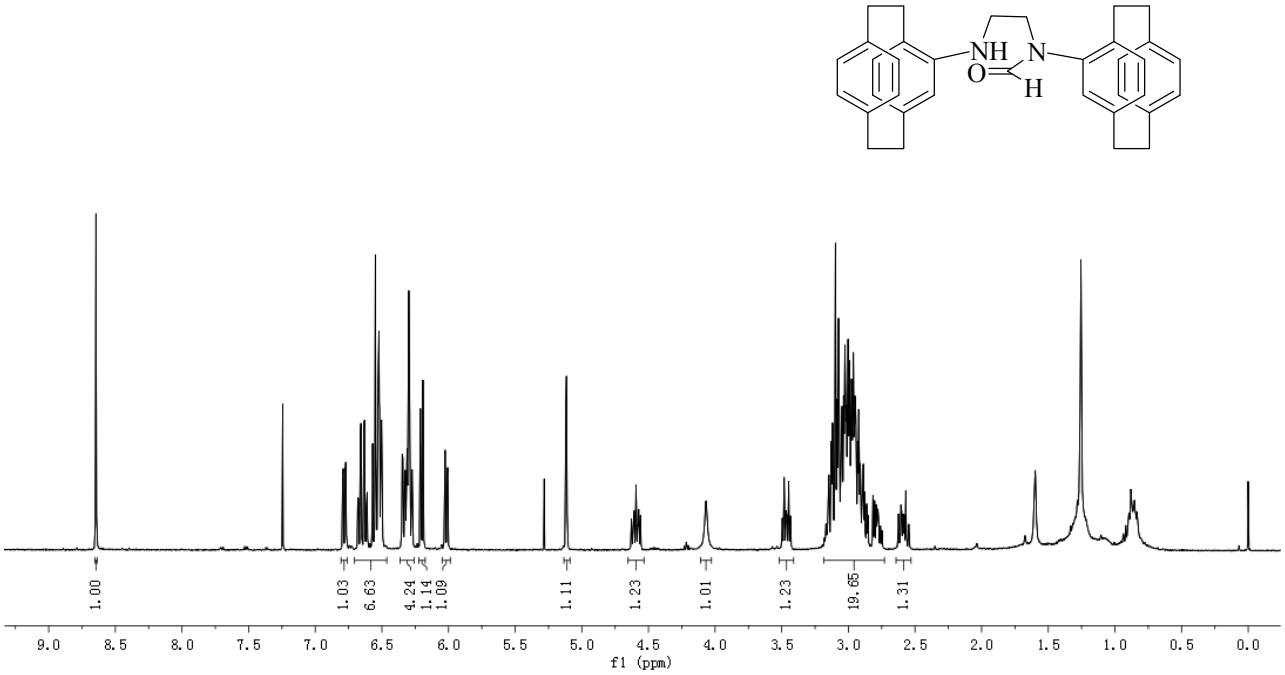
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O
N
H
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4.24
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1.09
1.11
1.23
1.01
1.23
19.65
1.31
f1 (ppm)