10.1071/CH14351_AC

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Supplementary Material

Design and synthesis of piperazine–based ionic liquids for liquid–liquid extraction of Cu(II), Ni(II) and Co(II) from water

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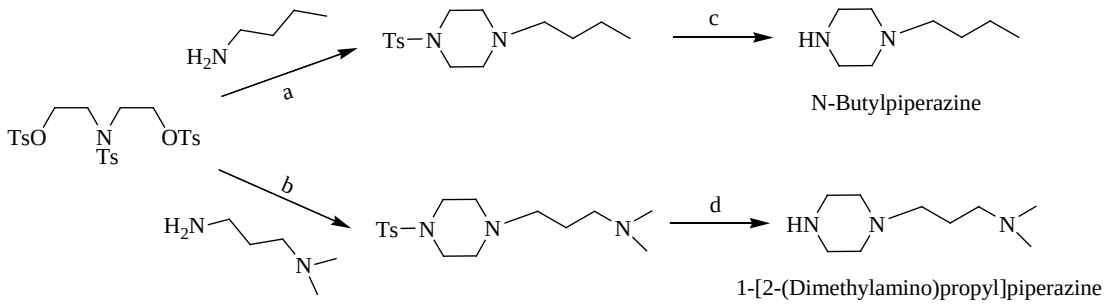
Synthesis of piperazine functional groups

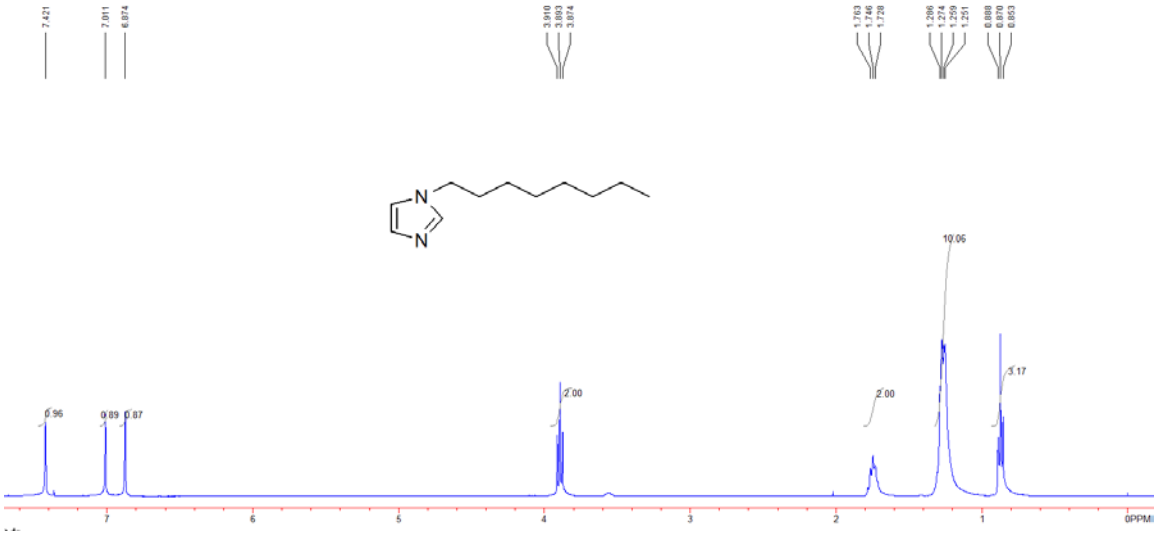
Two N–heteroalkyl–N′–tosylpiperazines were synthesized according to protocols published in the literature (Huang JY, Xu WY, Xie HJ and Li SJ, One-Step Cyclization: Synthesis of N–Heteroalkyl–N′–tosylpiperazines. *J Org Chem* **77**: 7506–7511 (2012)). Subsequently, tosyl group was removed by sulfuric acid (98 v/v%, 10 mL) with stirring at 120 °C for 24 h under N_2 atmosphere. The mixture was cooled and adjusted pH to 10 with aq. NaOH. The resulting solution was extracted with $CHCl_3$ (10 mL ×3). The combined organic phases were concentrated under reduced pressure and the residue was dried under vacuum to give mono-substituted piperazines.



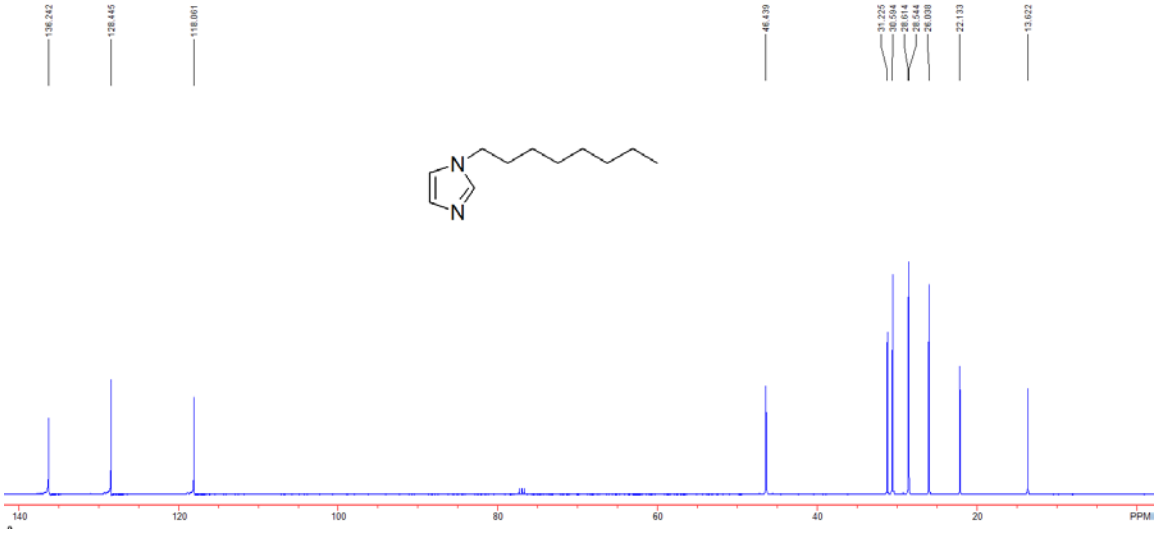
Scheme S1. Synthetic route of mono-substituted piperazines. Reaction Conditions: (a, b) K_2CO_3, MeCN, reflux under N_2 atmosphere, 12 h; (c, d) H_2SO_4, N_2 atmosphere, 120°C, 24 h.

Compound 1

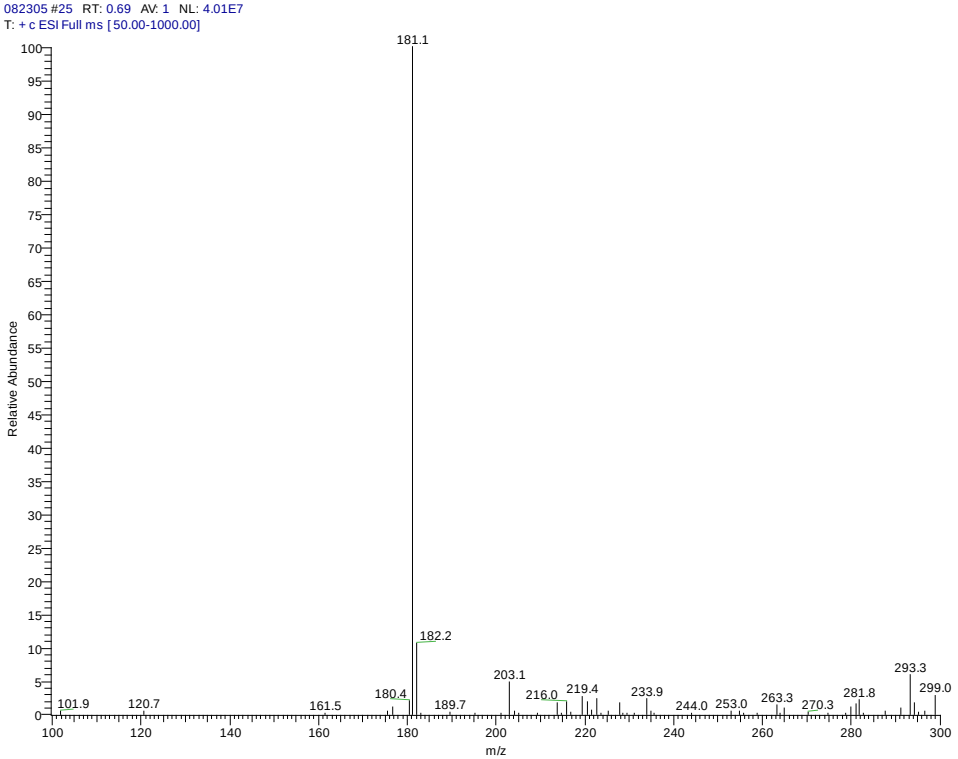
^{1}H NMR



^{13}C NMR



ESI–MS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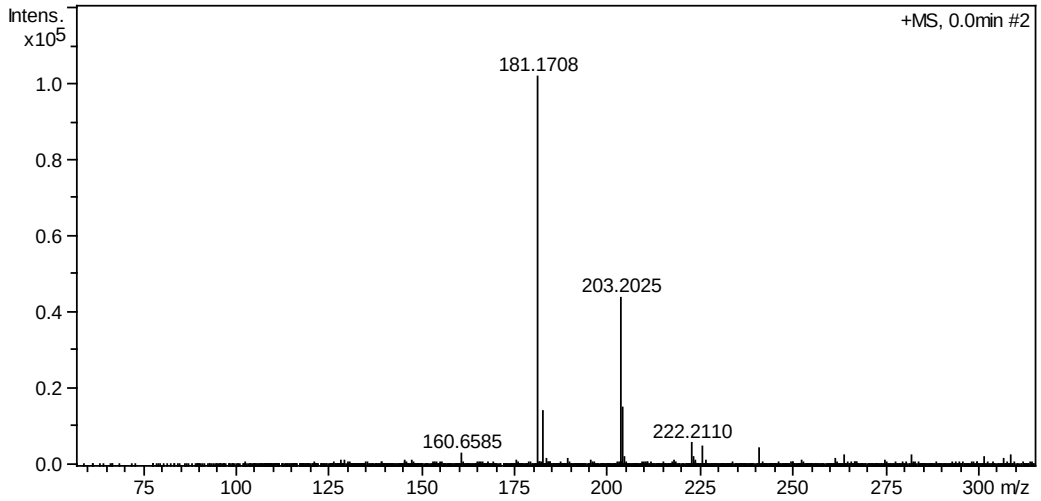
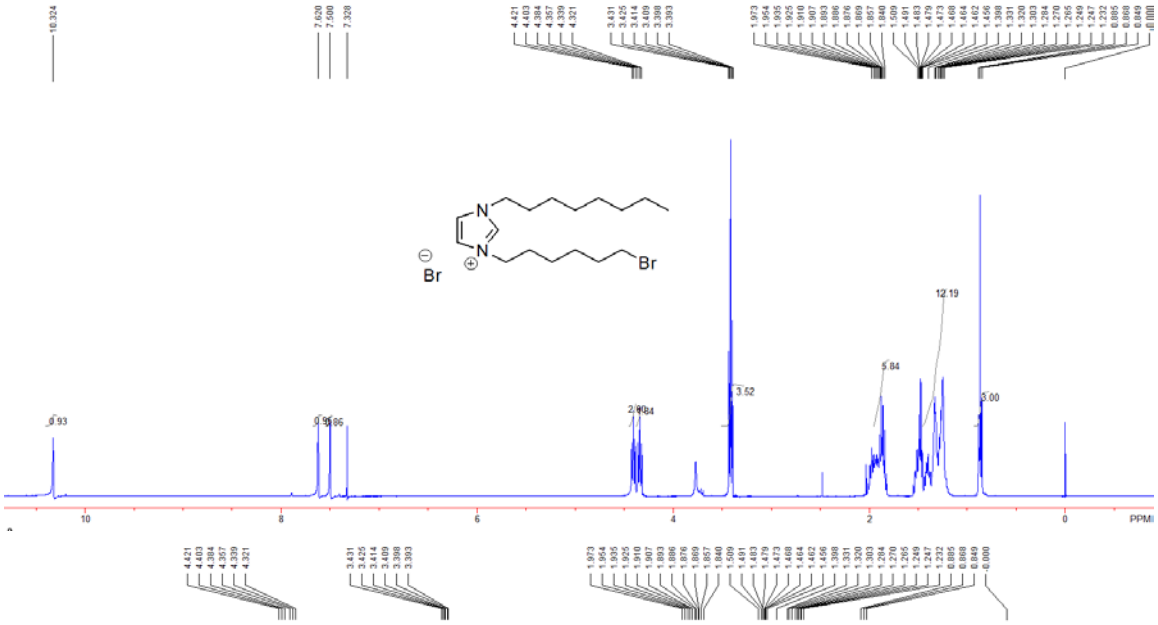
Relative Abundance
m/z
181.1
182.2
203.1
293.3
299.0
101.9
120.7
161.5
180.4
189.7
216.0
219.4
233.9
244.0
253.0
263.3
270.3
281.8

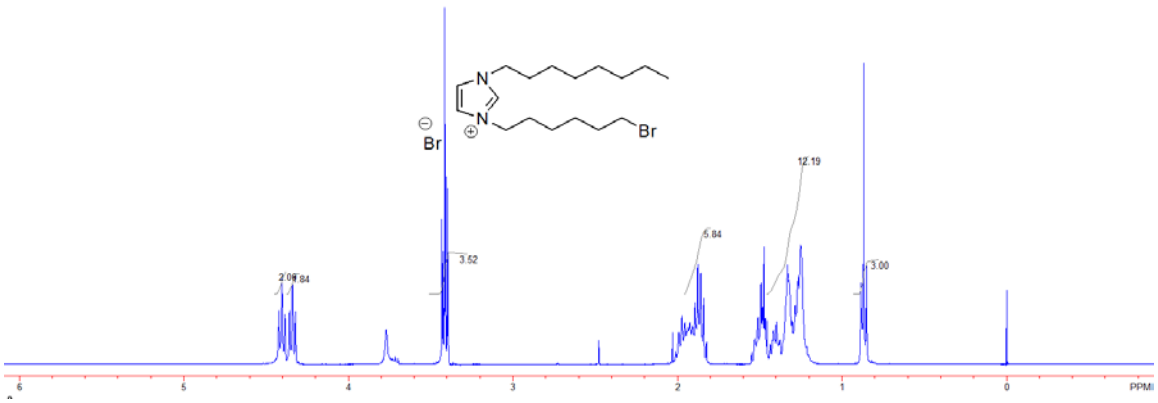
HRMS


Intens.
x10^5
+MS, 0.0min #2
181.1708
203.2025
160.6585
222.2110
m/z

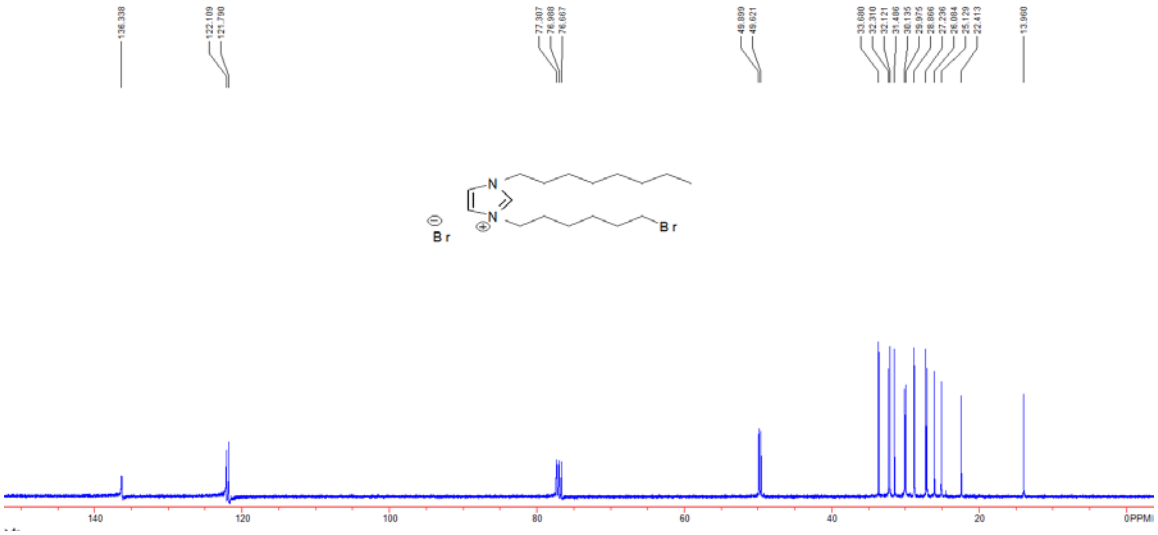
Compound 2

^{1}H NMR

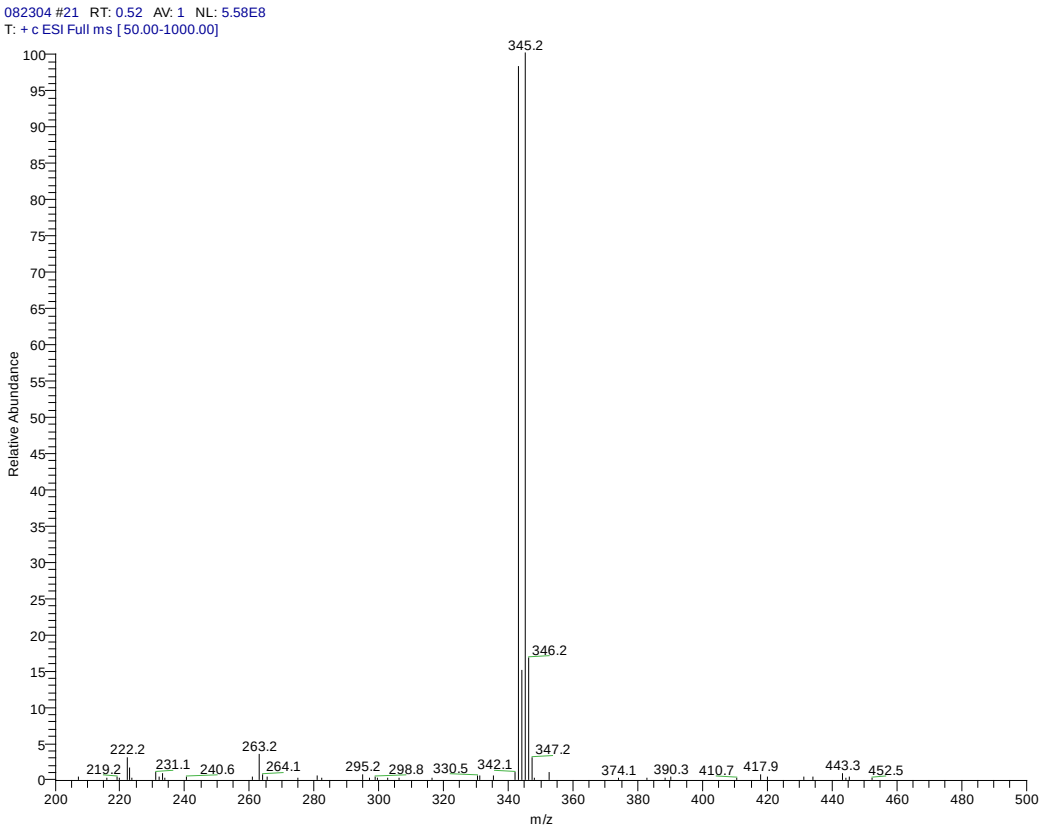




^{13}C NMR



ESI–MS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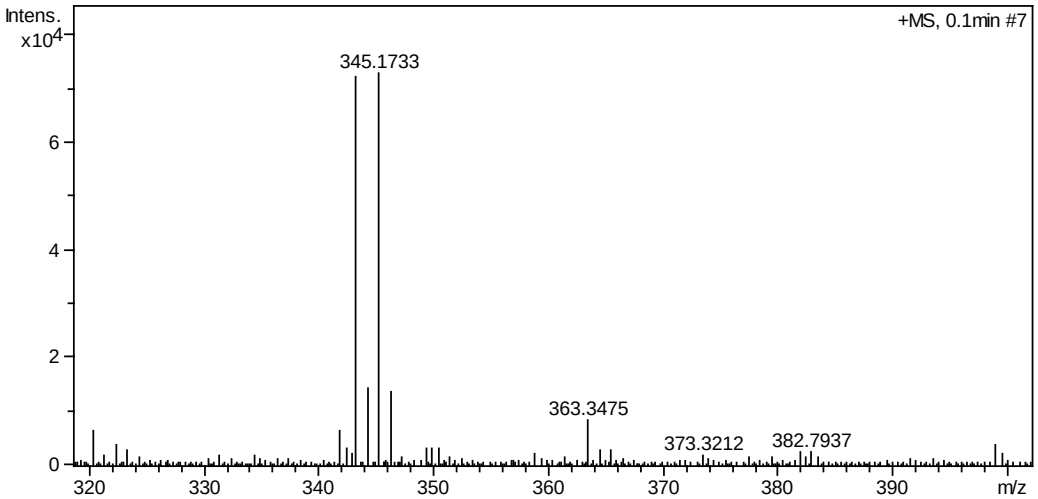
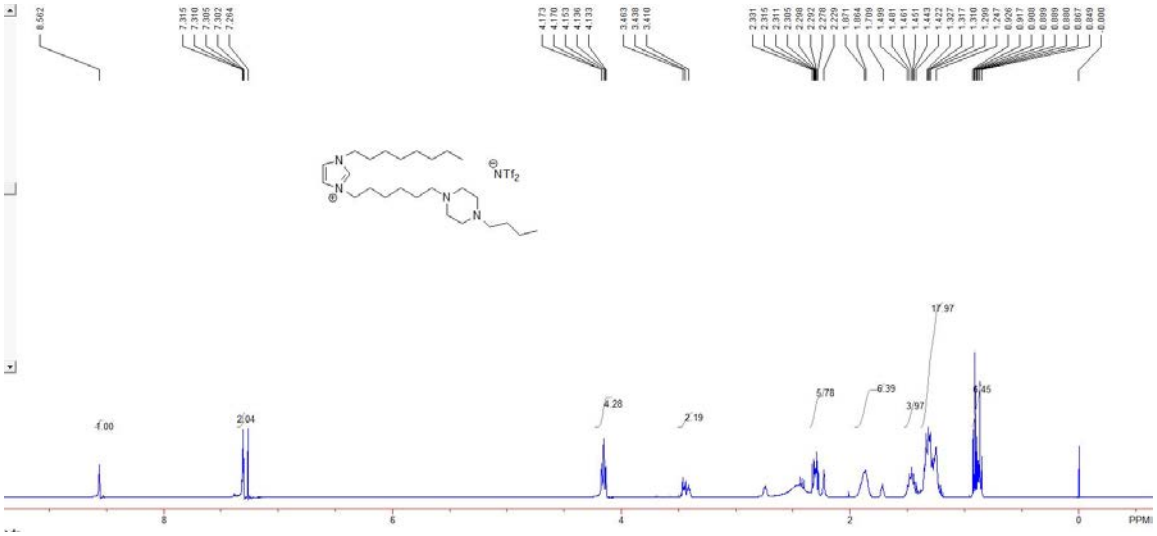
T: + c ESI Full ms [50.00-1000.00]
Relative Abundance
m/z
345.2
346.2
347.2
222.2
219.2
231.1
240.6
263.2
264.1
295.2
298.8
330.5
342.1
374.1
390.3
410.7
417.9
443.3
452.5

HRMS

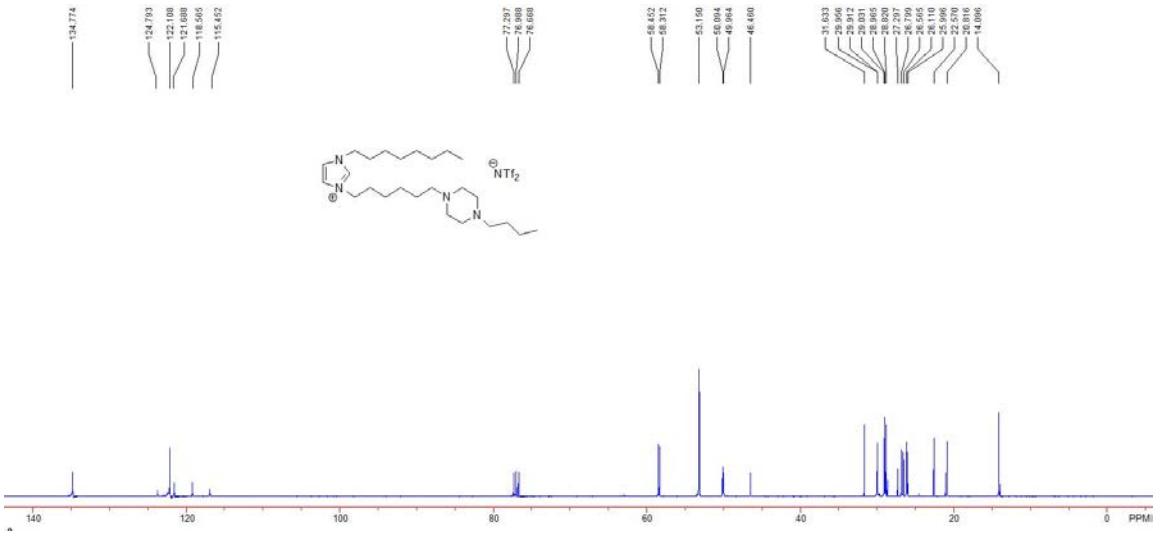

Intens.
x10^4
+MS, 0.1min #7
345.1733
363.3475
373.3212
382.7937
m/z

Compound 3a

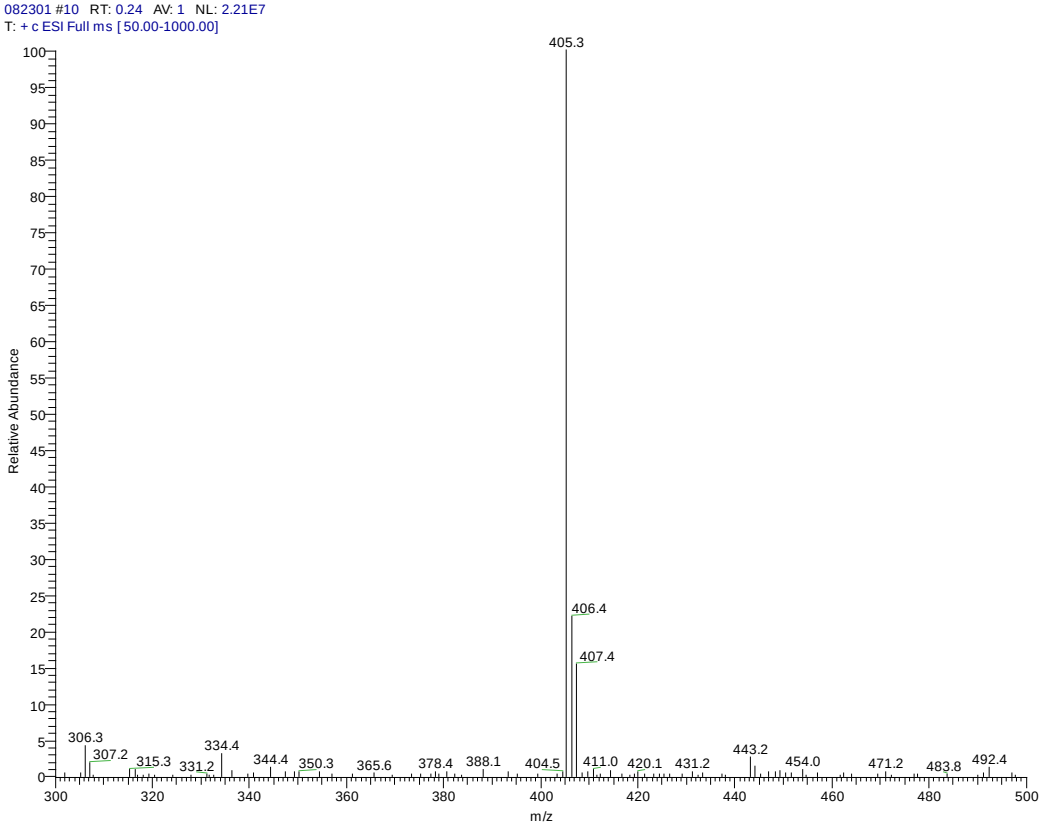
^{1}H NMR



^{13}C NMR



ESI–MS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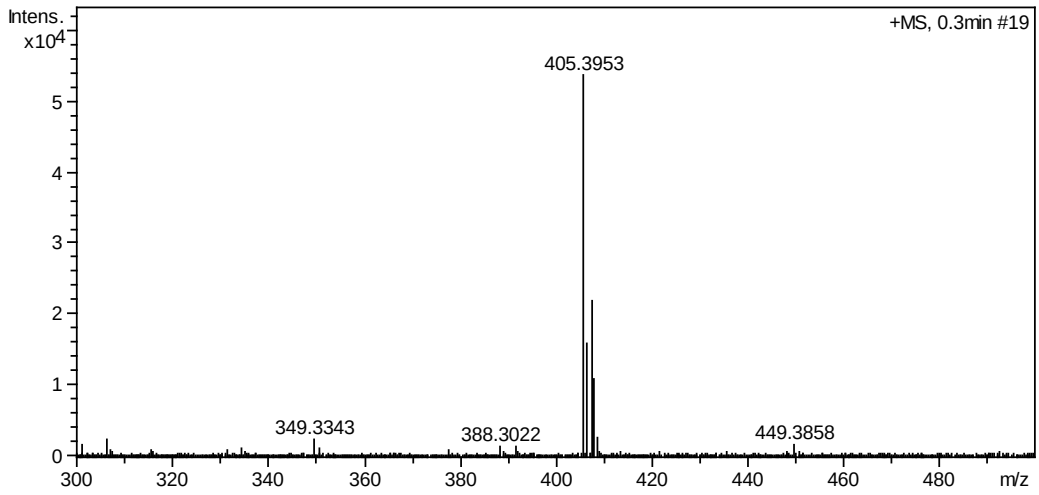
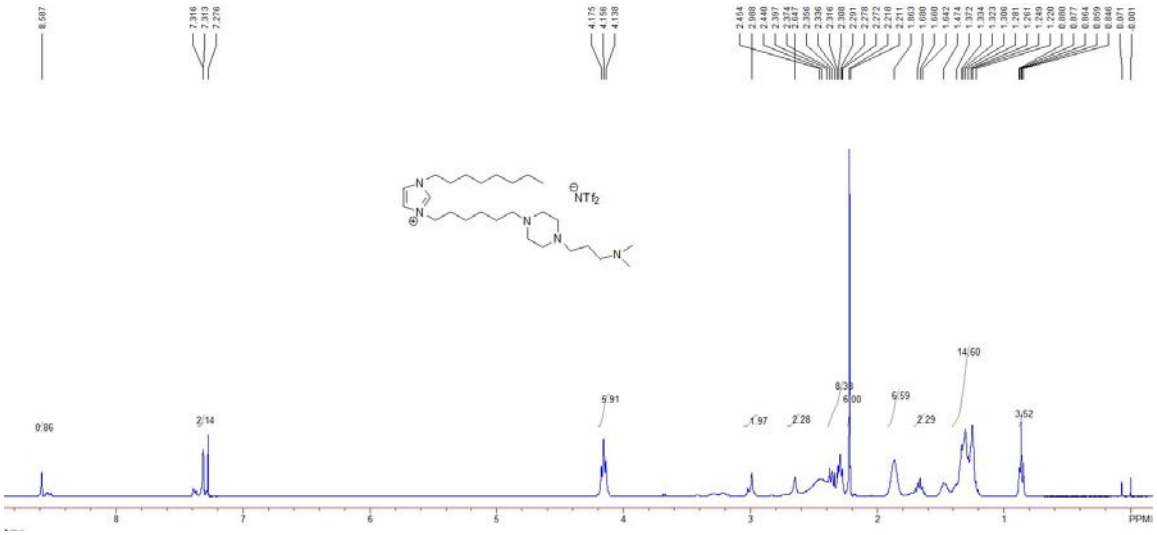
T: + c ESI Full ms [50.00-1000.00]
Relative Abundance
m/z
405.3
406.4
407.4
306.3
307.2
315.3
331.2
334.4
344.4
350.3
365.6
378.4
388.1
404.5
411.0
420.1
431.2
443.2
454.0
471.2
483.8
492.4

HRMS

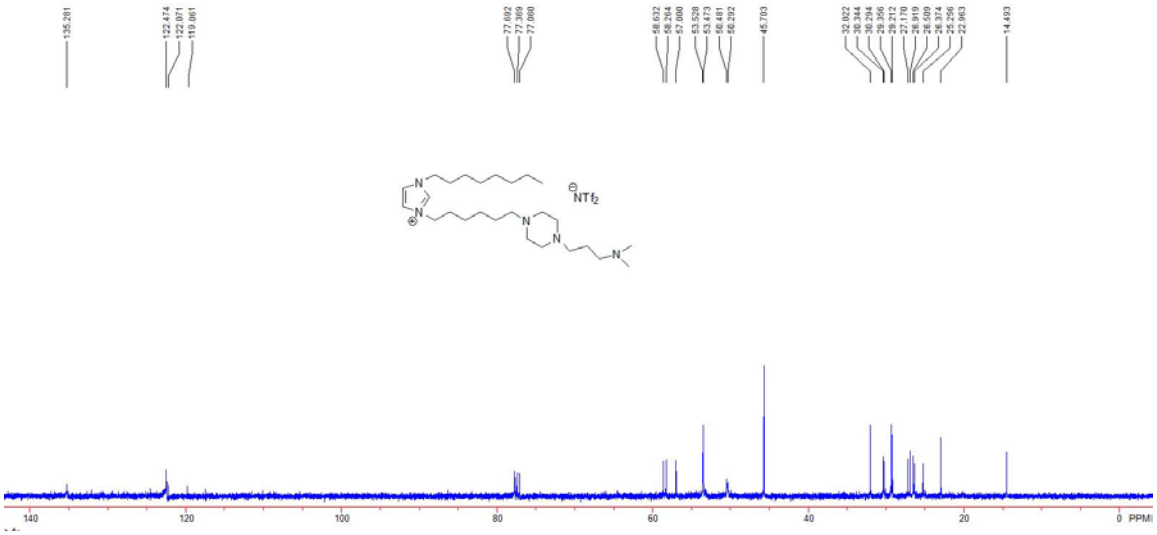

Intens.
x10^4
+MS, 0.3min #19
405.3953
349.3343
388.3022
449.3858
m/z

Compound 3b

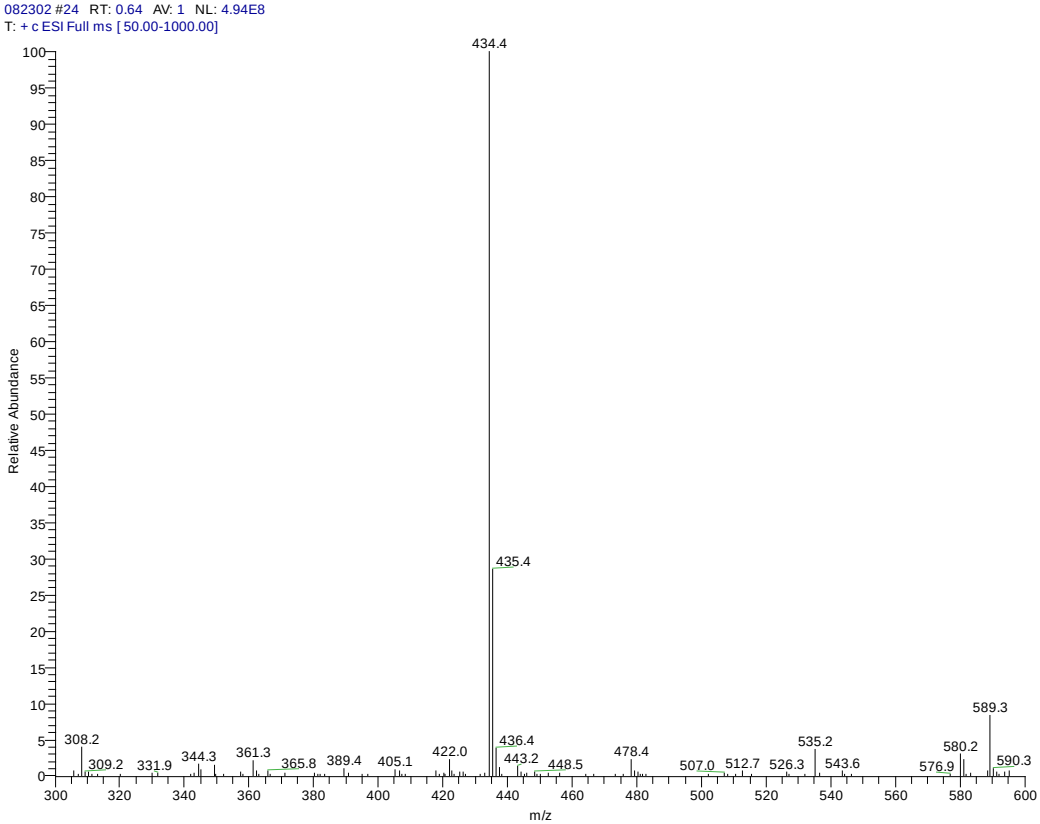
^{1}H NMR



^{13}C NMR



ESI–MS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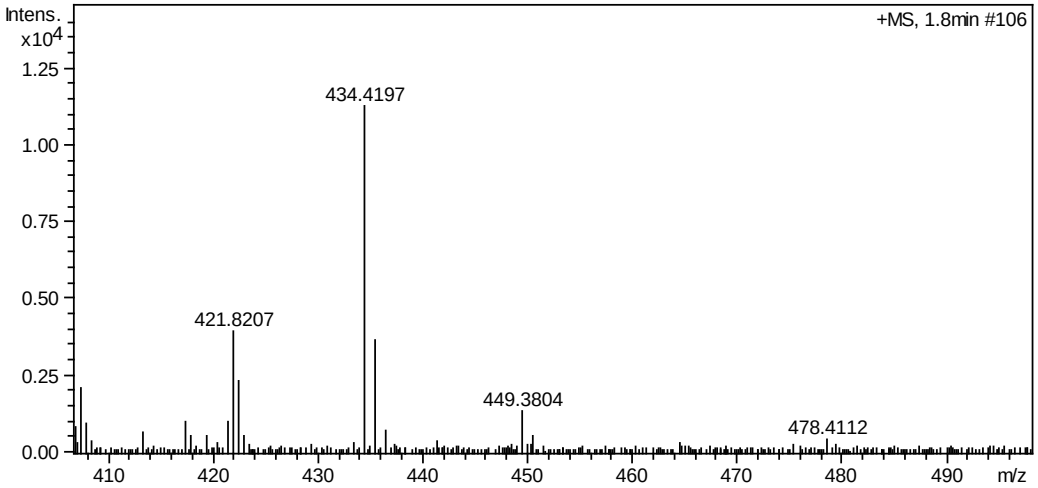
T: + c ESI Full ms [50.00-1000.00]
Relative Abundance
m/z
434.4
435.4
436.4
308.2
309.2
331.9
344.3
361.3
365.8
389.4
405.1
422.0
443.2
448.5
478.4
507.0
512.7
526.3
535.2
543.6
576.9
580.2
589.3
590.3

HRMS


Intens.
x10^4
+MS, 1.8min #106
434.4197
421.8207
449.3804
478.4112
m/z