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McLaughlin, G, Morris, N, Kavanagh, PV, Power, JD, Twamley, B, O'Brien, J, Talbot, B, Dowling, G and Brandt, SD

The synthesis and characterization of the 'research chemical' N-(1-amino-3-methyl-1-oxobutan-2-yl)-1-(cyclohexylmethyl)-3-(4-fluorophenyl)-1H-pyrazole-5-carboxamide (3,5-AB-CHMFUPPYCA) and differentiation from its 5,3-regioisomer.

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### Article

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**The synthesis and characterization of the ‘research chemical’ *N*-(1-amino-3-methyl-1-oxobutan-2-yl)-1-(cyclohexylmethyl)-3-(4-fluorophenyl)-1*H*-pyrazole-5-carboxamide (3,5-AB-CHMFUPPYCA) and differentiation from its 5,3-regioisomer.**

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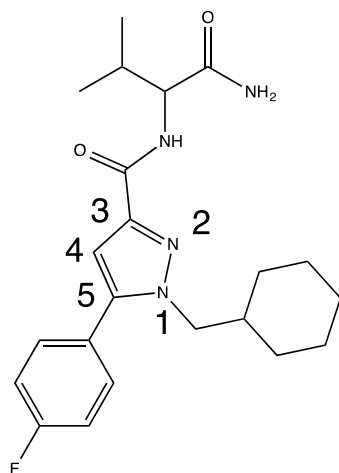
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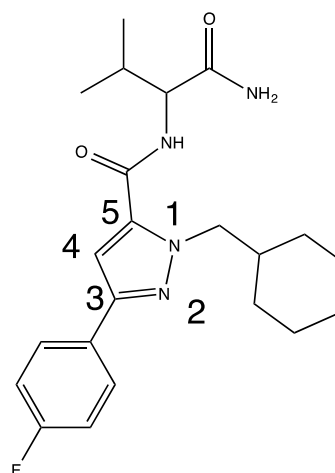
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5,3-AB-CHMFUPPYCA



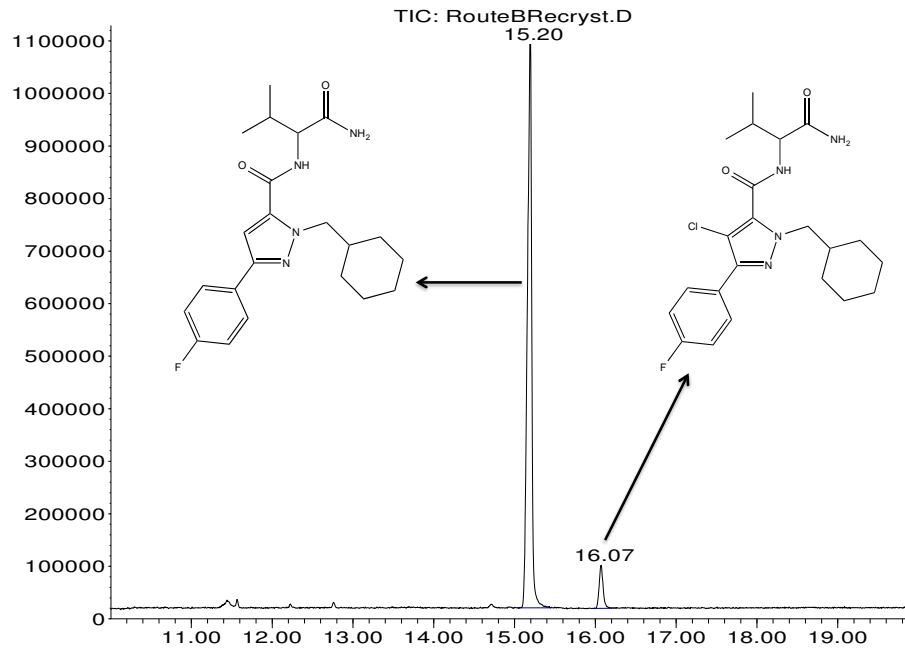
3,5-AB-CHMFUPPYCA

**Contents:**

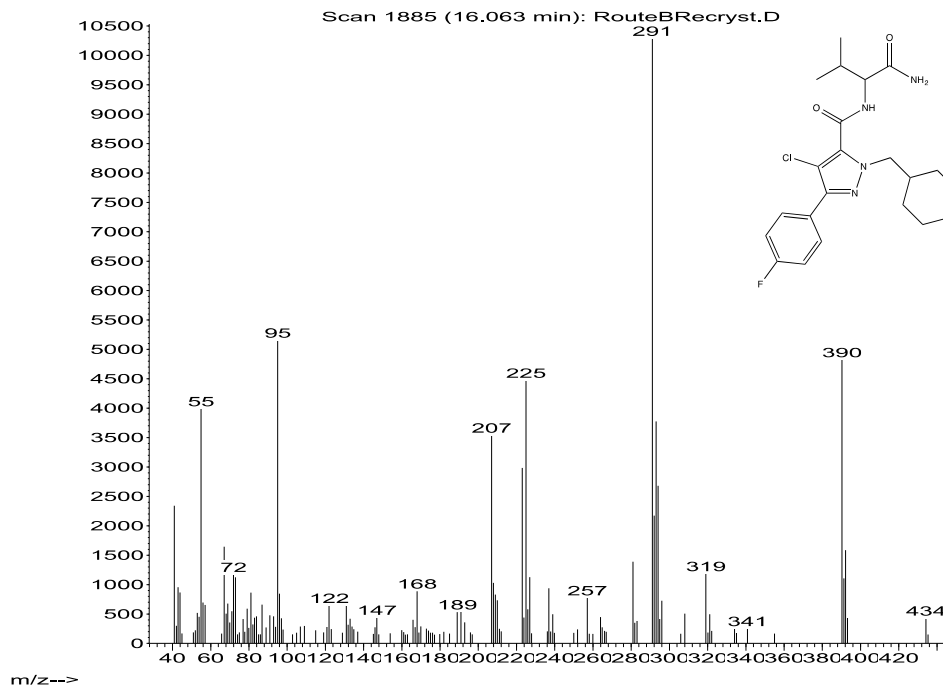
1. GC-MS data for the chlorinated by-product formed during the synthesis of 3,5-AB-CHMFUPPYCA
2. LC-MS data for the chlorinated by-product formed during the synthesis of 3,5-AB-CHMFUPPYCA
3. HR-MS data for the AB-CHMFUPPYCA isomers and vendor sample
4. Proton NMR spectra for the AB-CHMFUPPYCA isomers and vendor sample
5. Carbon<sup>13</sup> NMR spectra for the AB-CHMFUPPYCA isomers and vendor sample
6. Fluorine<sup>19</sup> NMR spectra for the AB-CHMFUPPYCA isomers and vendor sample
7. NMR Nuclear Overhauser effect experiment assignments
8. Additional x-ray crystallography data

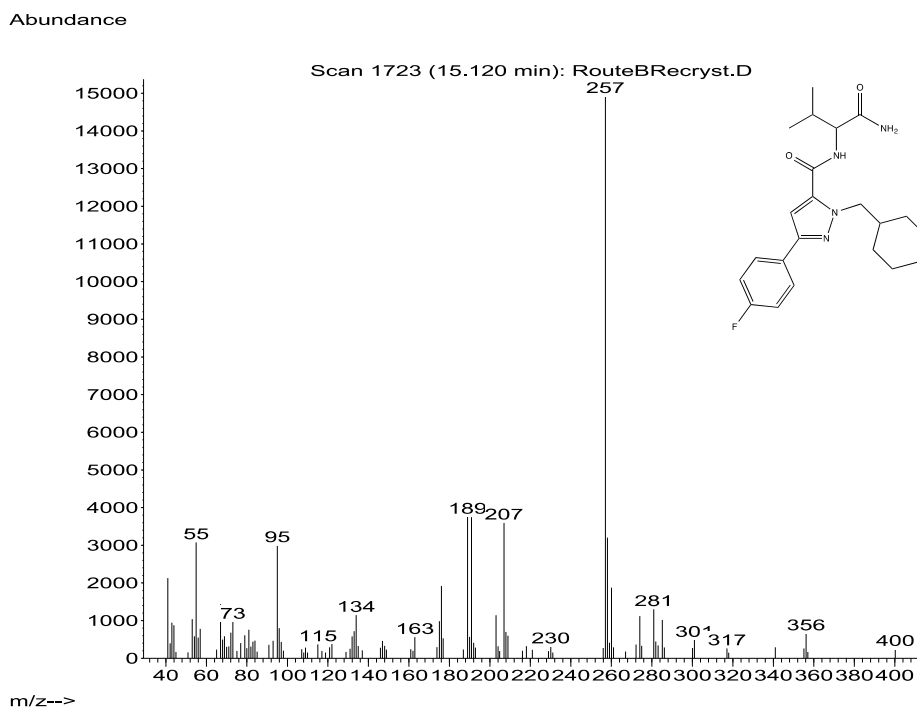
1. GC-MS data for chlorinated by-product formed during the synthesis of 3,5-AB-CHMFUPPYCA

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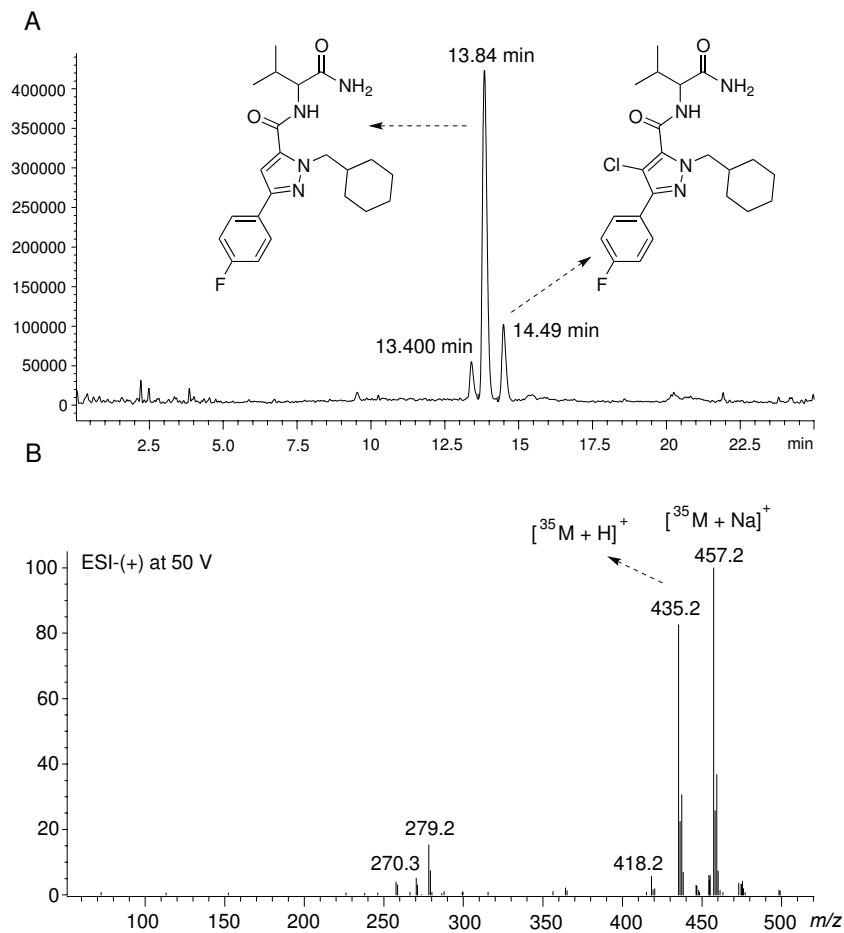


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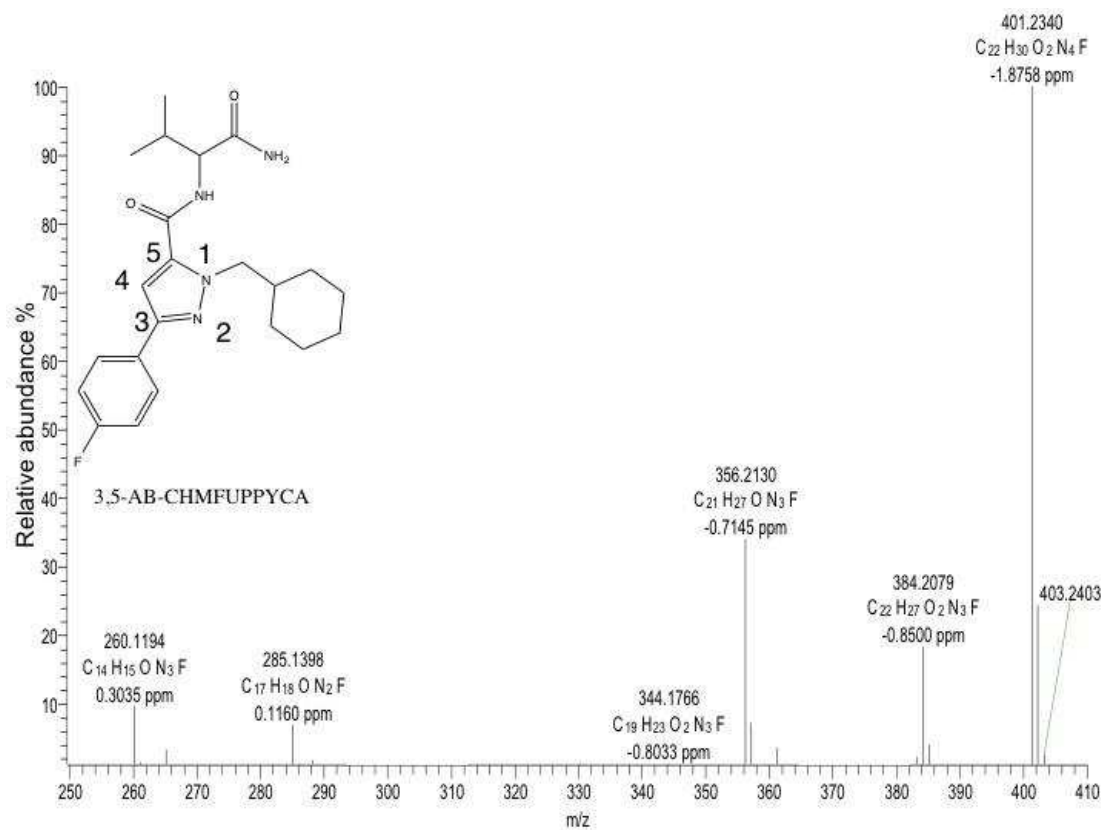
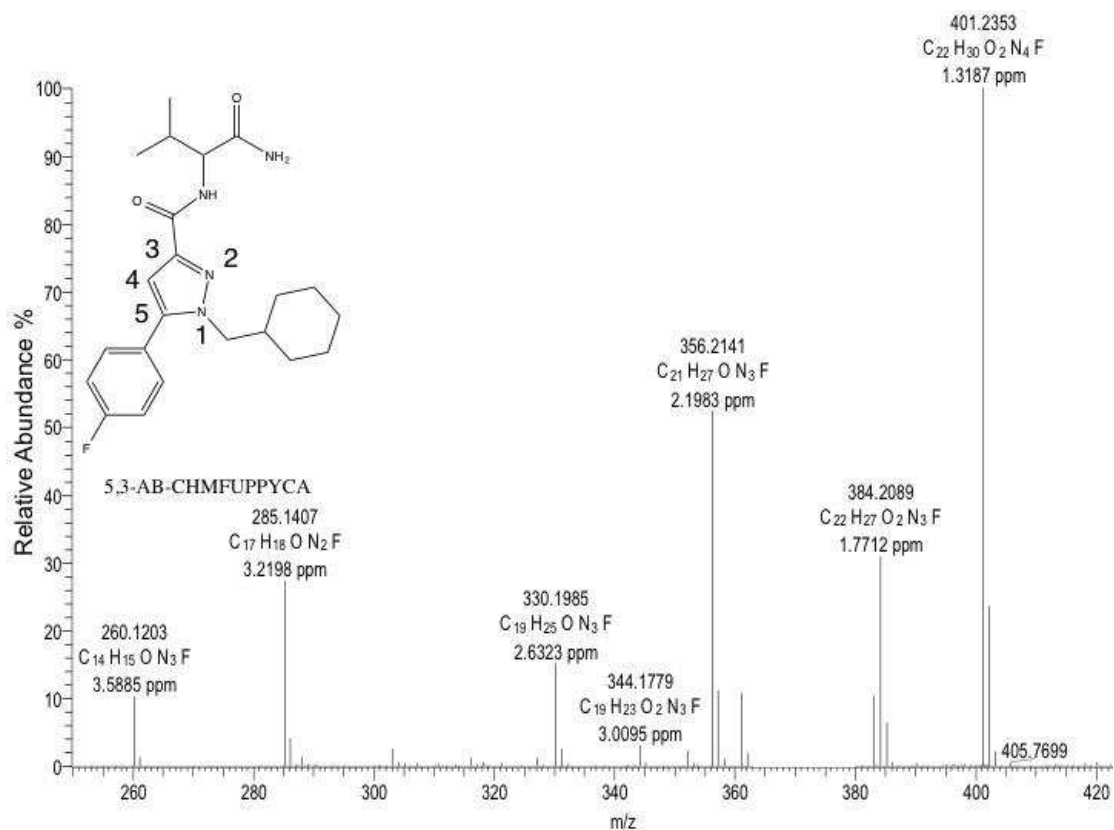


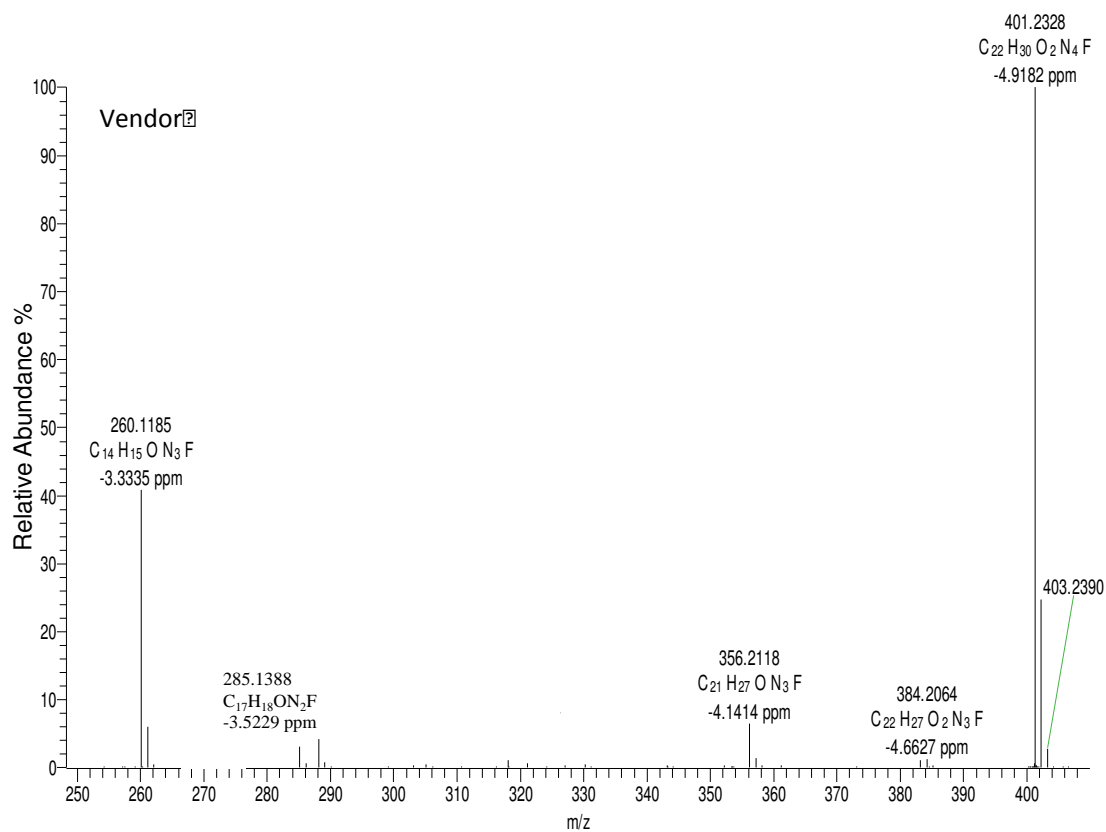


2. LC-MS data for the chlorinated by-product formed during the synthesis of 3,5-AB-CHMFUPPYCA



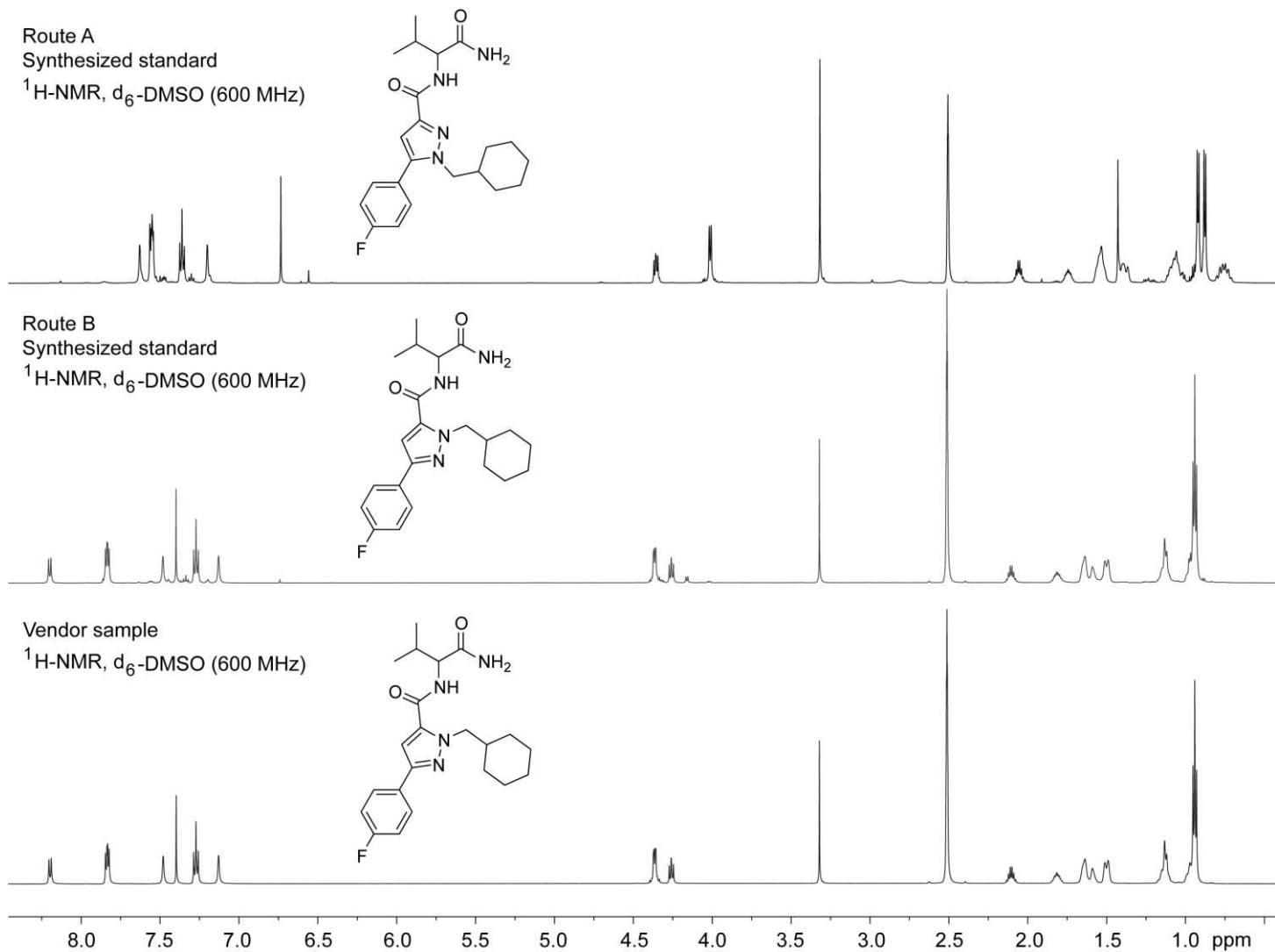
3. HR-MS data for the AB-CHMFUPPYCA isomers and vendor sample



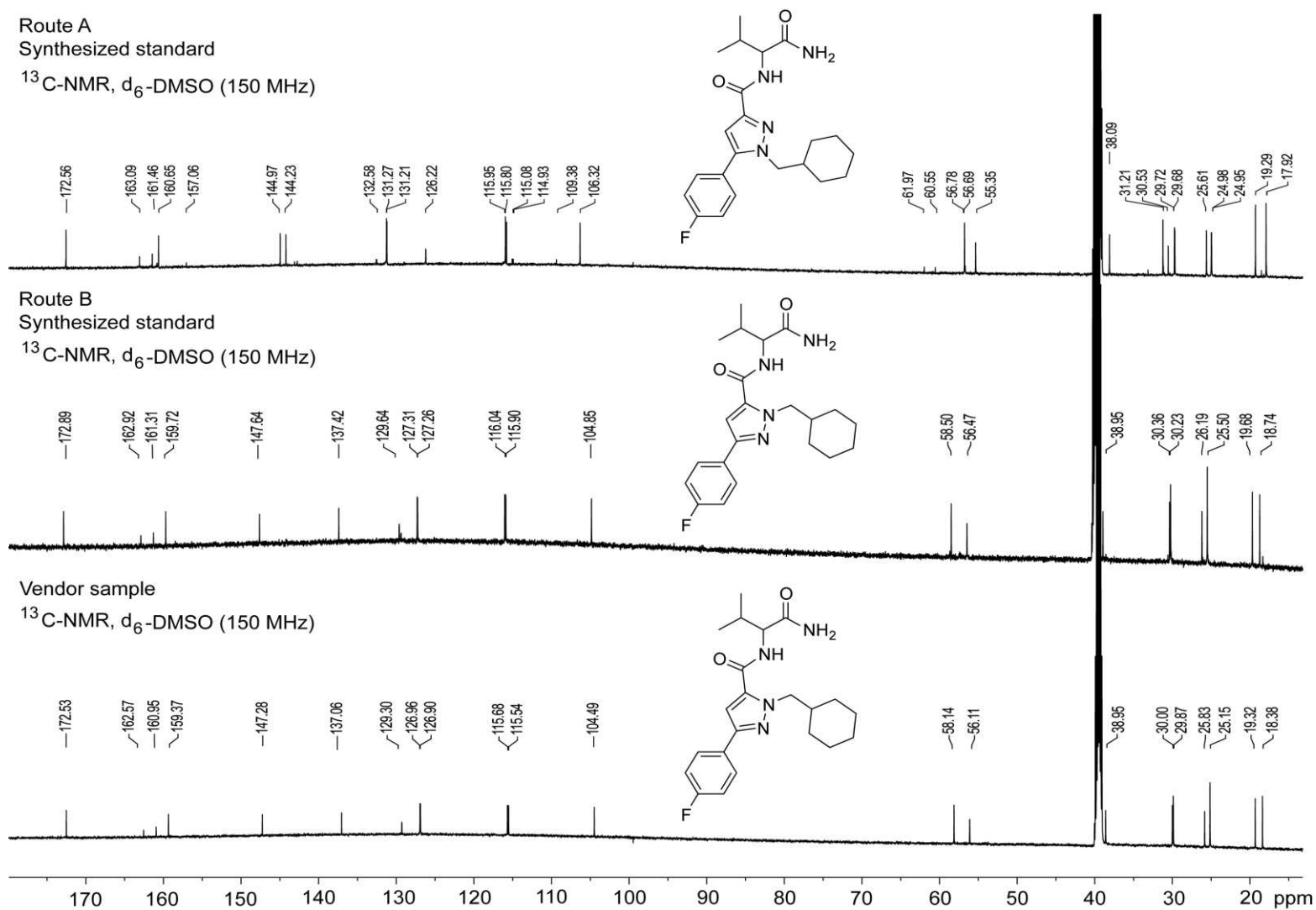




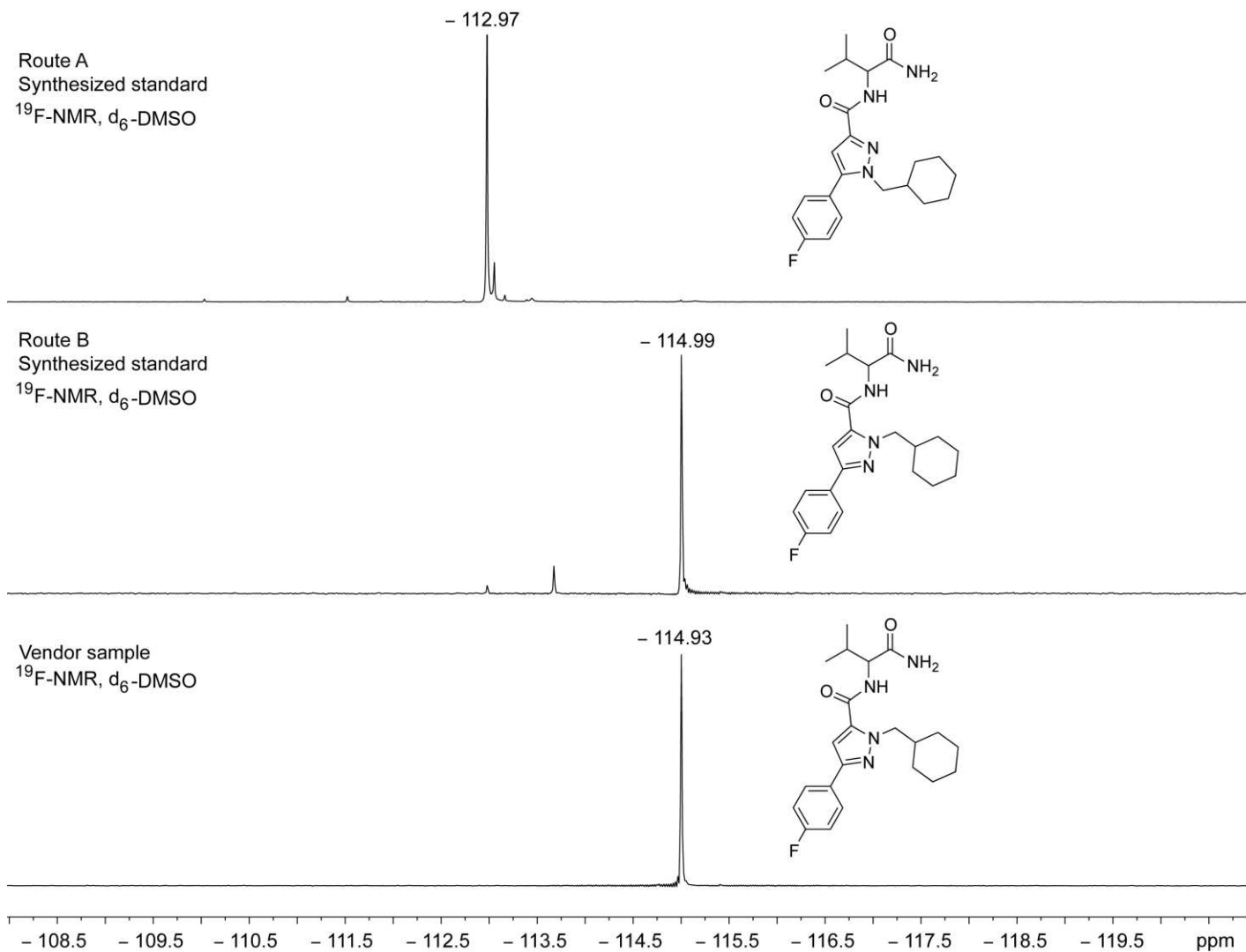
4. Proton NMR spectra for the AB-CHMFUPPYCA isomers and vendor sample



5. Carbon<sup>13</sup> NMR spectra for the AB-CHMFUPPYCA isomers and vendor sample

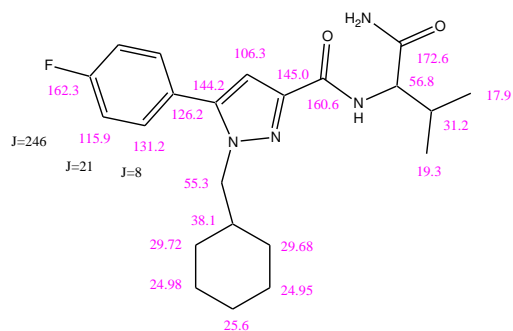


6. Fluorine<sup>19</sup> NMR spectra for the AB-CHMFUPPYCA isomers and vendor sample

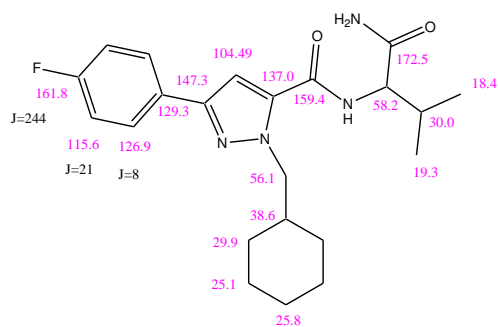


7. NMR nuclear Overhauser effect experiment assignments

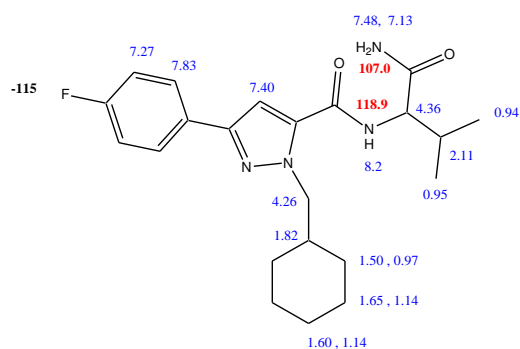
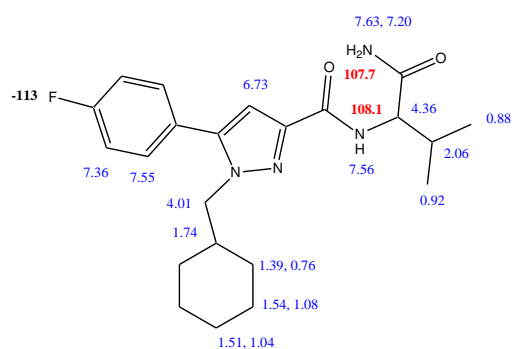
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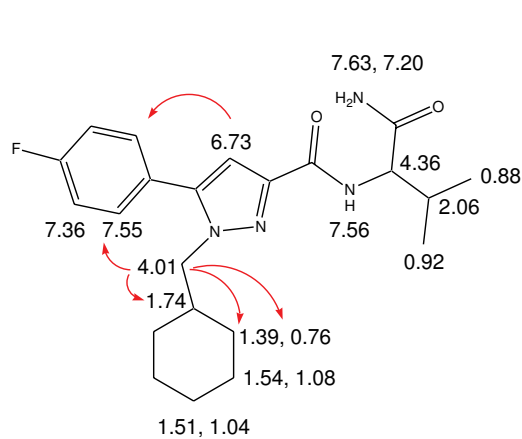
3,5-AB-CHMFUPPYCA



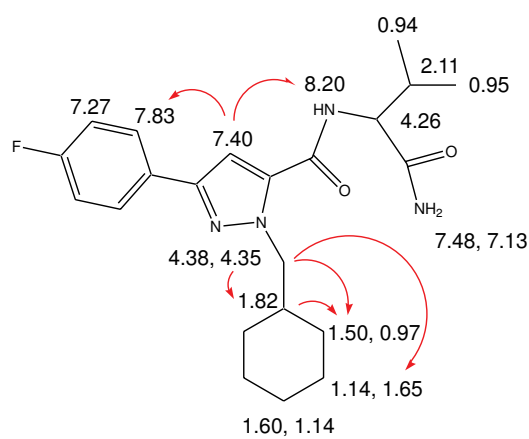
Carbon-13 NMR 150 MHz  $J_{19F-13C}$  coupling in Hz (black)



Proton (blue), Nitrogen-15 (red), Fluorine-19 (black) NMR



Spin coupling (Hz) : 7.27ppm (d,  $J_{HH}$  8.8, d,  $J_{19FH}$  8.8) , 7.83ppm (d,  $J_{HH}$  8.8, d,  $J_{19FH}$  5.0)



Spin coupling (Hz) : 7.27ppm (d,  $J_{HH}$  9.0, d,  $J_{19FH}$  9.0) , 7.83ppm (d,  $J_{HH}$  9.0, d,  $J_{19FH}$  5.5)

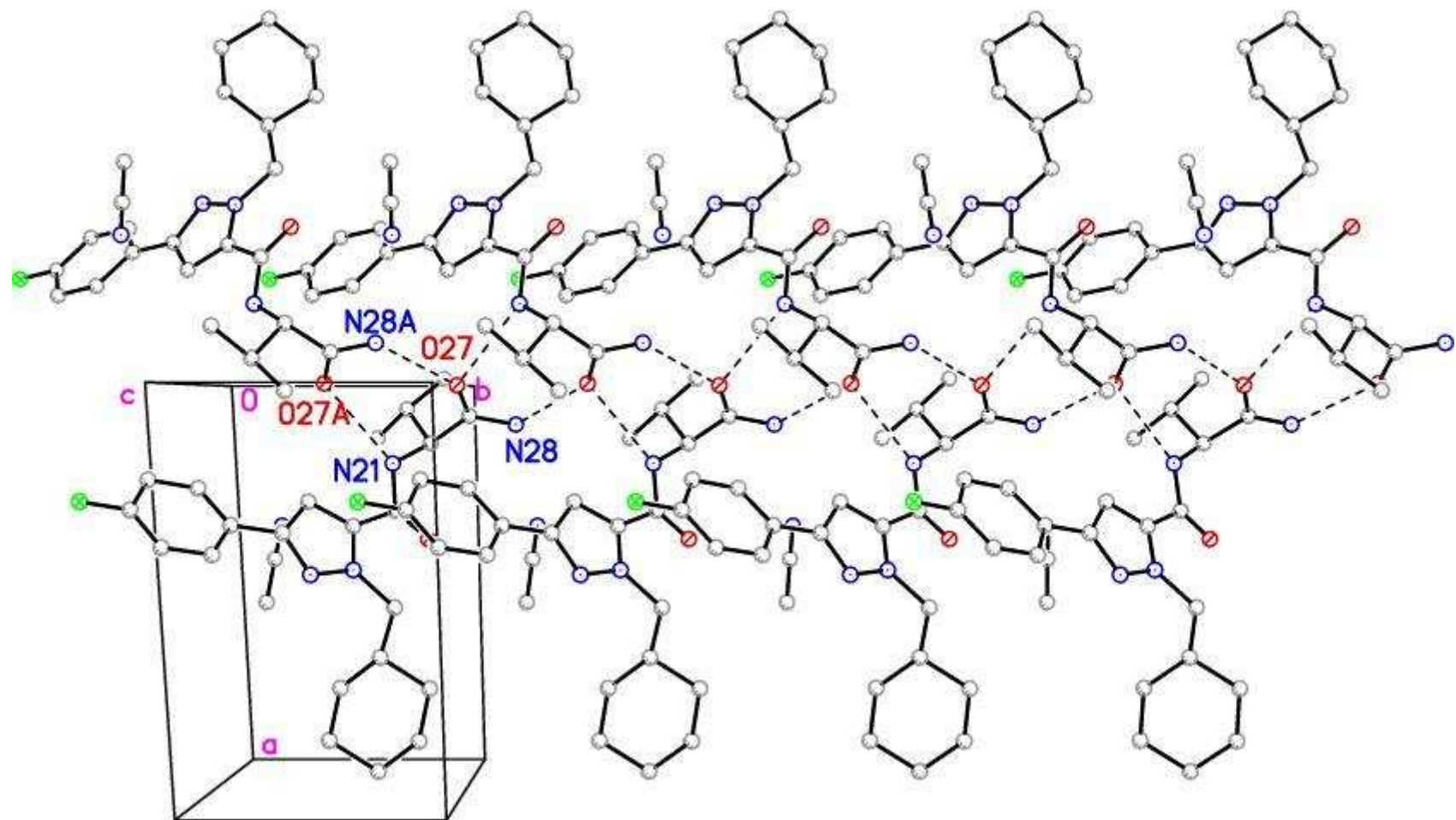
## 8. Additional x-ray crystallography data

Table 1. Hydrogen bonds for 3,5-AB-CHMFUPPYCA [ $\text{\AA}$  and  $^\circ$ ].

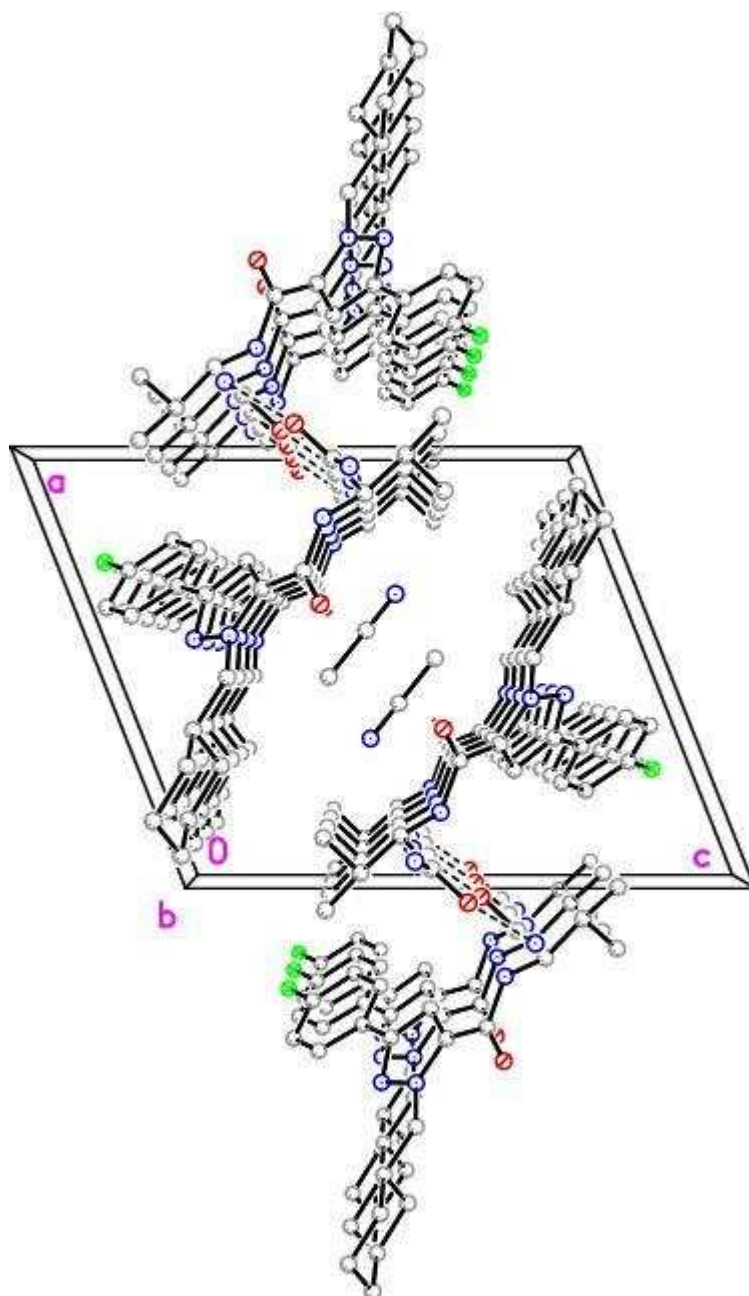
D-H...A	d(D-H)	d(H...A)	d(D...A)	$\angle$ (DHA)
C(11)-H(11)...O(27)#1	0.95	2.47	3.266(2)	141
N(28)-H(28A)...O(27)#2	0.88(3)	2.00(3)	2.856(2)	166(2)
N(28)-H(28B)...N(29)#3	0.90(3)	2.20(2)	3.039(2)	155(2)
N(21)-H(21)...O(27)#1	0.87(2)	2.11(2)	2.963(2)	167(2)

Symmetry transformations used to generate equivalent atoms:

#1  $-x, y-1/2, -z+1$  #2  $-x, y+1/2, -z+1$  #3  $x, y+1, z$



Hydrogen bonding network shown in dashed lines in 3,5-AB-CHMFUPPYCA showing the connectivity of the chain that extends parallel to the b axis. Unit cell shown and hydrogen atoms omitted for clarity.



Packing diagram of 3,5-AB-CHMFUPPYCA viewed down the b-axis showing the hydrogen bonded chain network and the channel occupied with acetonitrile. Dashed lines indicate hydrogen bonding. Hydrogen atoms omitted for clarity.