

## Supplementary Information

### **Characterization of a recently detected halogenated aminorex derivative: *para*-fluoro-4-methylaminorex (4'F-4-MAR).**

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## **Methods**

### ***Reagents and chemicals***

For GC-MS analysis, GC-grade n-hexane and GC-grade acetone were purchased from Scharlau (Scharlab, Barcelona, Spain). For FTIR analysis, potassium bromide (KBr) was purchased from Scharlau.

### ***Sample treatment***

For FTIR analysis, sample was prepared with 5% of the unknown compound and 95% of potassium bromide, homogenized in an agate mortar and compressed under a pressure of 5000 kg/cm<sup>2</sup>.

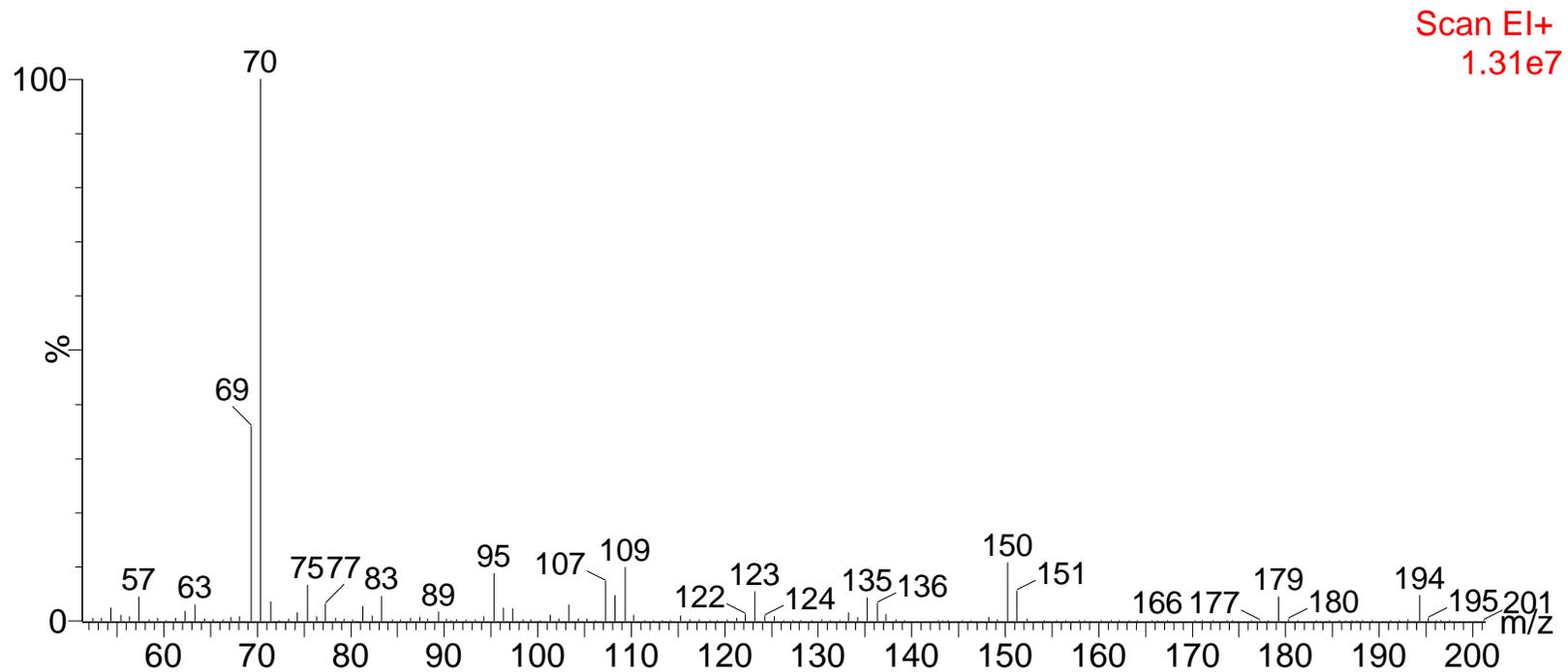
For GC-MS analysis, 10 mg of sample were extracted with 1 mL of acetone in an ultrasonic bath for 15 min. After centrifugation, the supernatant was five thousand-fold diluted with GC-grade n-hexane, and 1 µL of the extract were injected in the GC-MS system.

### ***Instrumentation***

For FTIR analysis, a Jasco FT/IR-6200 FTIR spectrometer (Jasco Inc., Easton, MD, USA) was used. Data acquisition was performed at 23 °C between 4000 and 400 cm<sup>-1</sup>, with a resolution of 4 cm<sup>-1</sup> and performing 32 acquisitions.

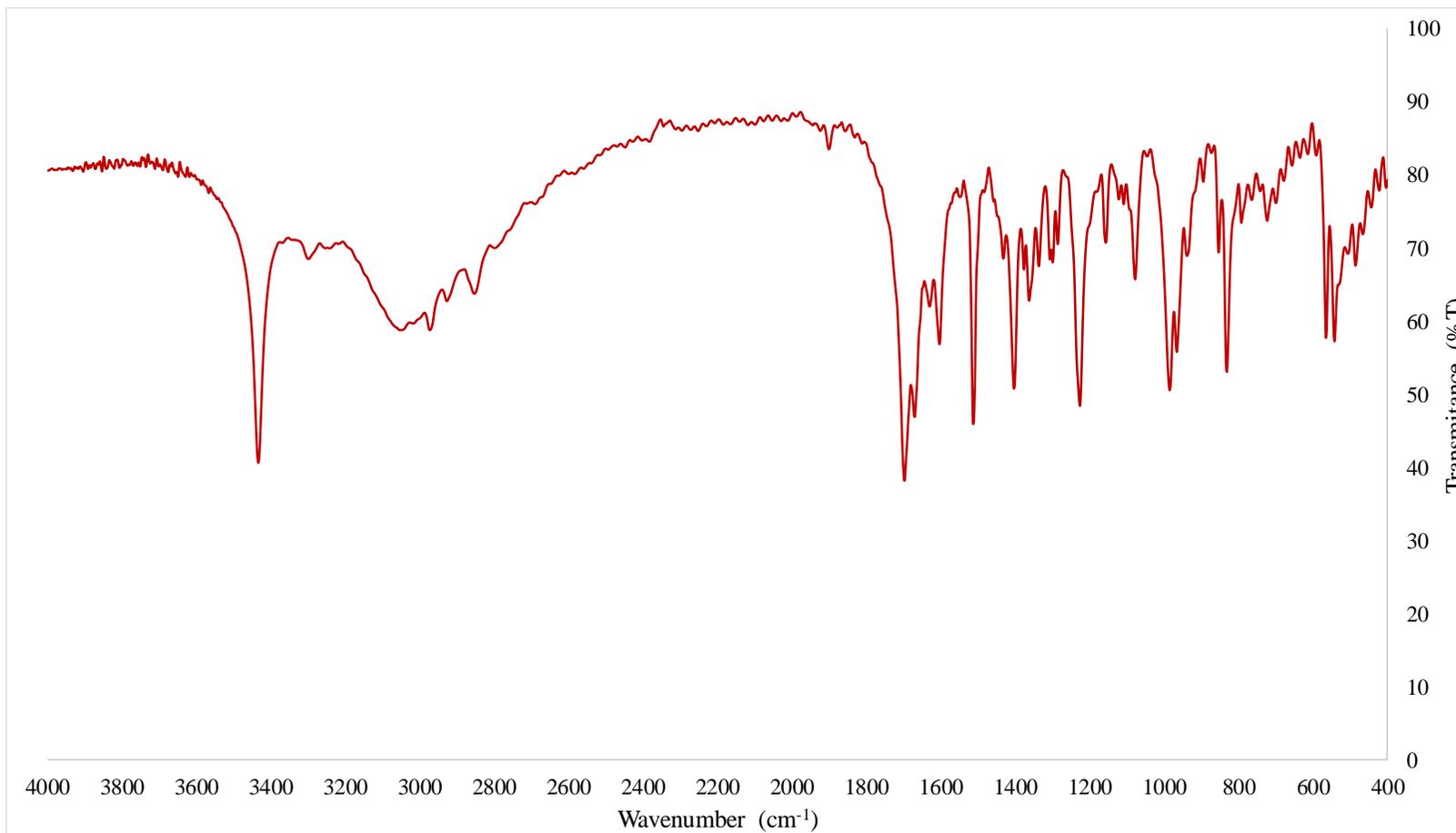
For GC-MS analysis, an Agilent 6890N gas chromatograph (Agilent Technologies, Santa Clara, CA, USA) equipped with an Agilent 7683 autosampler (Agilent Technologies) was coupled to a Quattro Micro GC triple quadrupole mass spectrometer (Micromass, Boston, USA) using an electron ionization (EI) interface. The injector and the interface were operated at 250 °C. 1 µL of sample was injected in splitless

mode using deactivated liners into a 30 m 0.25 mm i.d., 0.25  $\mu\text{m}$  film thickness DB-5MS column (Agilent). Helium (99.999%, Praxair, Valencia, Spain) was used as carrier gas at a flow rate of 1 mL/min. The oven temperature was initially maintained at 90  $^{\circ}\text{C}$  for 1 min and programmed to reach 300  $^{\circ}\text{C}$  at 20  $^{\circ}\text{C}$  per min. It was finally maintained at 300  $^{\circ}\text{C}$  for 1.5 min (total run time was 12 min). The mass spectrometer was operated in electronic ionization mode at 70 eV. MS system worked in SCAN acquisition mode, acquiring from  $m/z$  50 to 400 Da. Analytical data were acquired and processed using MassLynx data station operation software (version 4.0, Waters).



**Fig. S1.** GC-(EI)MS spectrum of the 4'-fluoro-4-MAR.

1  
2  
3



**Fig. S2.** FTIR spectrum of the 4'-fluoro-4-MAR.

4  
5  
6

EC-106

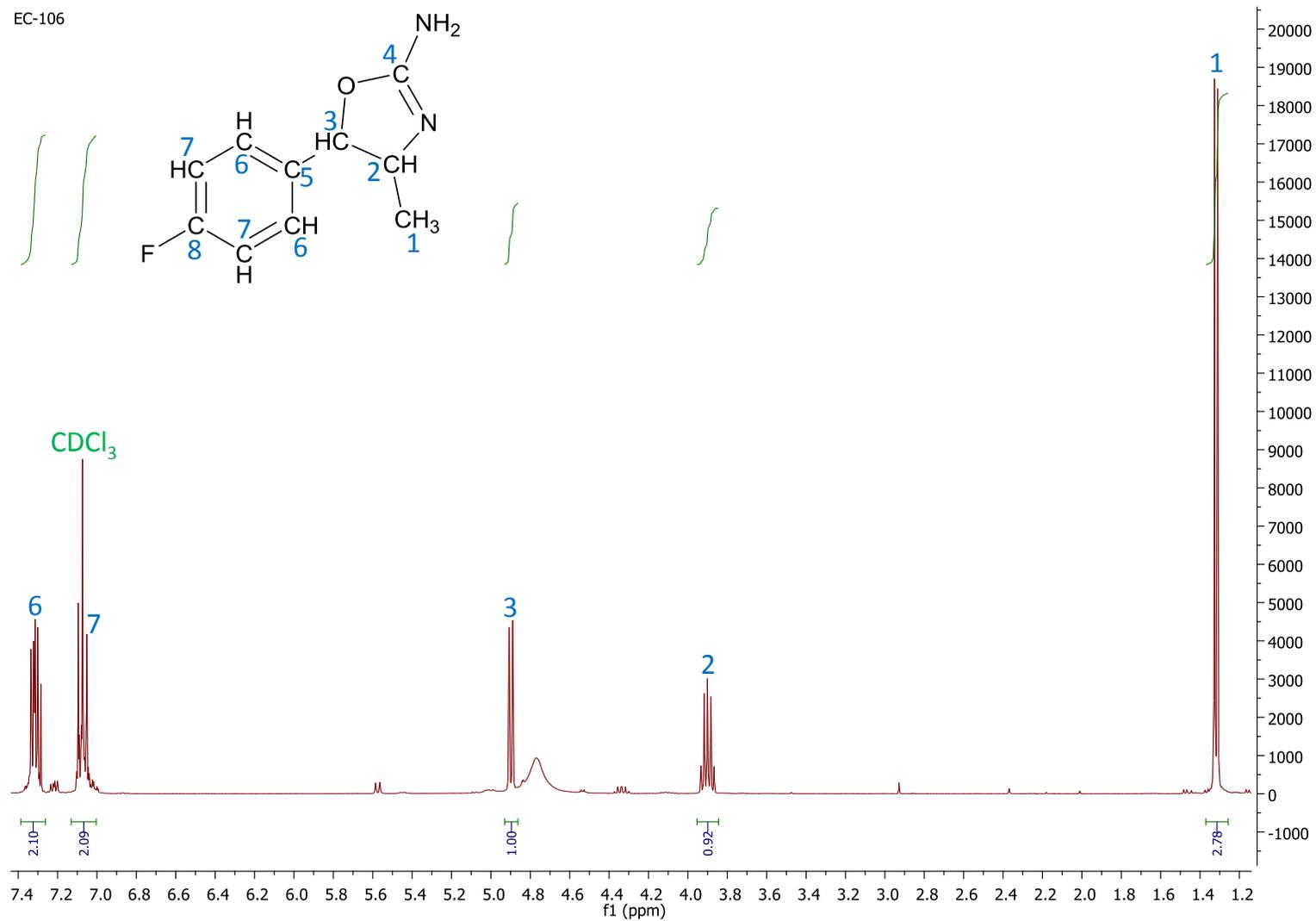


Fig. S3. <sup>1</sup>H NMR spectrum of the 4'-fluoro-4-MAR.

EC-106

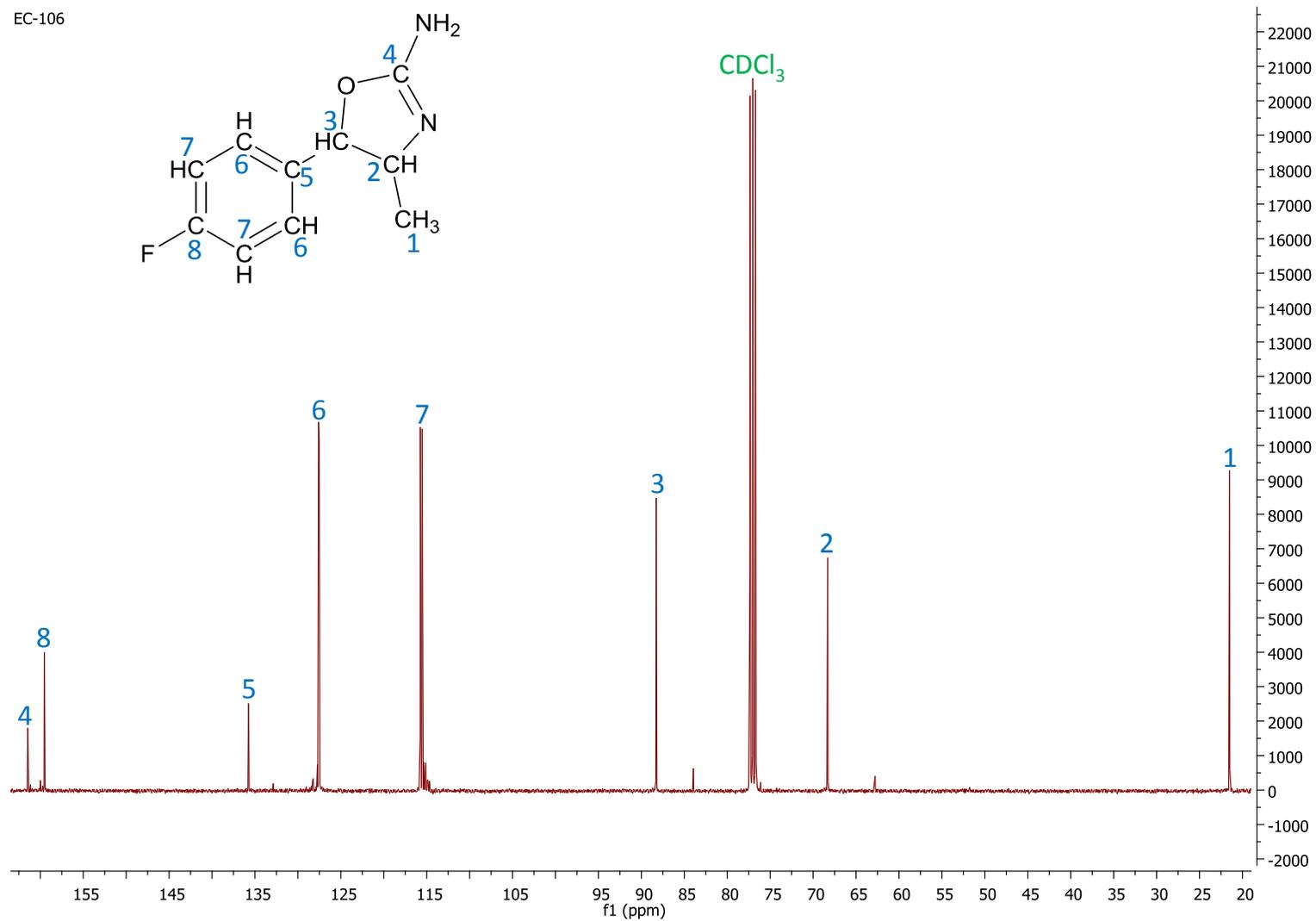


Fig. S 4. <sup>13</sup>C NMR spectrum of the 4'-fluoro-4-MAR.