

CESAMO'S EQUIPMENTS

1. NMR Equipments

Bruker AVANCE III 300 MHz

Avance III 300 (300-B) and NEO 300 (300-A) were equipped with a 5 mm BBFO probe (with z-gradients) which tunes to nuclei in the range between ^{109}Ag and ^{31}P . All NMR spectra were phased and baseline-corrected. Chemical shifts were referenced to the TMS signal.

Bruker AVANCE II 400 MHz for applications in liquid state

Avance II 400 is equipped with a 5 mm BBFO probe (with z-gradients) which tunes to nuclei in the range between ^{109}Ag and ^{31}P . All NMR spectra were phased and baseline-corrected. Chemical shifts were referenced to the TMS signal.

Bruker AVANCE II 400 MHz for applications in solid state

Avance II 400 is equipped with 4.0 mm double resonance (H/X) magic angle spinning probe for solid state NMR. The probe possesses an X channel that is tuneable from ^{31}P to ^{15}N . This probe has a maximum spin rate of 15 kHz. A double air-bearing probe and a zirconium oxide rotor were used. The MAS rate was in the 8 kHz. All NMR spectra were phased and baseline-corrected.

Bruker AVANCE III 600 MHz for applications in liquid state

Avance III 600 is equipped with a 5 mm BBI probe with z-gradients and tunes to nuclei in the range between ^{109}Ag to ^{31}P . The instrument is equipped with a BCU-05 unit for measurements between $-60\text{ }^{\circ}\text{C}$ to $+110\text{ }^{\circ}\text{C}$.

2. Mass spectrometry Equipments

HRMS@CESAMO

Mass spectrometry analyses (GCMS (EI and FI), field ionisation (FD) and electrospray (ESI)) were performed by the CESAMO (ISM, Bordeaux, France).

HRMS ESI spectra were obtained on a QExactive™ benchtop Orbitrap mass spectrometer coupled to a Vanquish UHPLC system (Thermo Scientific, San Jose, USA) using electrospray ionization mode. HRMS EI, FI and FD mass spectra spectra were obtained on an Accutof GCv mass spectrometer (JEOL).

Electrospray (ESI)

Mass spectra were performed by the CESAMO (ISM, Bordeaux, France) on a QExactive™ benchtop Orbitrap mass spectrometer coupled to a Vanquish UHPLC system (Thermo Scientific, San Jose, USA). The instrument is equipped with an ESI source and spectra were recorded in the **negative / positive** mode. Full MS scans were acquired over the range **50-1300** m/z with a mass resolution of **140,000**.

Analyses were processed by direct infusion (FIA). Samples were introduced by injection through a 20 µL sample loop into a **300** µL/min flow of **methanol** from the LC pump.

XCalibur software version 4.1 was used for data acquisition and FreeStyle software version 1.5 for processing (Themo Scientific, San Jose, USA).

Field Desorption

(FD) spectra were performed by the CESAMO (ISM, Bordeaux, France). The measurements were carried out on a TOF mass spectrometer AccuTOF JMS-T100GCv (JEOL, Tokyo, Japan) using a Field Desorption emitter with an emitter voltage of 10 kV. One to two microliters solution of the compound is deposited on the emitter.

GC-MS-FI

The GC-MS analysis was performed by the CESAMO (ISM, Bordeaux, France) on a 7890A Agilent Ultra Gas chromatograph (Agilent Technologies, Santa Clara, USA) coupled to a TOF mass spectrometer Accu TOF JMS-T100GCv (JEOL, Tokyo, Japan) using an Field Ionisation emitter with an emitter voltage of 10 kV.

Capillary GC analysis was performed on a **TG-5MS (30m×0.32mm i.d., ×0.25µm)** capillary column with helium as carrier gas. GC conditions were **50°C** at rate of **15 °C/min** to **310 °C**, carrier gas (He) flow rate of **1.2 mL/min**. Injection temperature was **200 °C** with **split/splitless** mode. The transfer line and source temperatures were maintained at **220 °C** and **180 °C**. A solvent delay of **2 min** was selected.

In the full-scan mode, field ionization (FI) mass spectra were recorded. The field ionization emitter was at 10 kV voltage, with 40 mA emitter current on a 10µm emitter.

GC-MS-EI

The GC-MS analysis was performed by the CESAMO (ISM, Bordeaux, France) on a 7890A Agilent Ultra Gas chromatograph (Agilent Technologies, Santa Clara, USA) coupled to a TOF mass spectrometer Accu TOF JMS-T100GCv (JEOL, Tokyo, Japan). Capillary GC analysis was performed on a **TG-5MS (30m×0.32mm i.d.×0.25µm)** capillary column with helium as carrier gas. GC conditions were **50 °C** at rate of **15 °C/min** to **310 °C**, carrier gas (He) flow rate of **1.2 mL/min**. Injection temperature was **200 °C** with **split/splitless** mode. The transfer line and source temperatures were maintained at **220 °C** and **180 °C**. A solvent delay of **2 min** was selected.

In the full-scan mode positive electron ionisation (EI), mass spectra in the range **60-800 (m/z)** were at 70eV electron energy.

MALDI@CESAMO

MALDI-MS spectra were performed by the CESAMO (Bordeaux, France) on an Autoflex maX TOF mass spectrometer (Bruker Daltonics, Bremen, Germany) equipped with a frequency tripled Nd:YAG laser emitting at 355 nm. Spectra were recorded in the positive-ion mode using the reflectron and with an accelerating voltage of 19 kV.

Samples were dissolved in **THF** at 10 mg/ml. The **IAA** matrix (**Indole acrylic acid**) solution was prepared by dissolving 10 mg in 1 ml of **THF**. A MeOH solution of cationisation agent (NaI, 10 mg/ml) was also prepared. The solutions were combined in a 10:1:1 volume ratio of matrix to sample to cationisation agent. One to two microliters of the obtained solution was deposited onto the sample target and vacuum-dried.

En vert, les conditions expérimentales à adapter suivant les échantillons.

Pour les analyses réalisées en GCMS basse résolution et LCMS, nous consulter pour avoir les spécificités de la méthode utilisée.

3. X-Ray diffraction Equipment

Crystallographic data were acquired at CESAMO (UMR 5255) on a Bruker APEX 2 DUO. A single crystal was mounted and immersed in a stream of nitrogen gas [$T = 150(2)$ K]. Data were collected, using a microfocus sealed tube of Mo K α radiation ($k = 0.71073$ Å) on a Kappa CD diffractometer. Data collection and cell refinement were performed using APEX2 2013.10-0 (Bruker AXS Inc.), and SAINT v8.34A (Bruker AXS Inc.). Data reduction was performed using SAINT v8.34A (Bruker AXS Inc.). Correction for absorption was performed using multi-scan integration as included in SADABS V2012/1 (Bruker AXS). Structure solutions were found by charge flipping methods (SUPERFLIP a computer program for solution of crystal structures by charge flipping in arbitrary dimensions (Palatinus & Chapuis, *J. Appl. Cryst.* (2007). 40, 786–790) EDMA: a computer program for topological analysis of discrete electron densities (Palatinus et al., *J. Appl. Cryst.* (2012). 45, 575-580)) and refined with (SHELXL) (G.M. Sheldrick, A short history of SHELX, *Acta Crystallographica Section A*, 64 (2008), pp. 112–122)

Si mesure à froid (généralement 150K) ...vérifier dans le cif ou demander

Si molybdène (généralement le cas) si non, mesure au cuivre : "Cu K α radiation ($k = 1.54184$ Å)"

Toute la partie grisée doit être validée avec Aline Lacoudre ou vérifiée dans le fichier cif car les logiciels de traitement des données peuvent changer...

Merci de citer le CESAMO comme lieu de réalisation des analyses,
Voici des exemples de propositions :

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or

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