

The package received on 10/18/17 and stored in the freezer, was opened.

- 2 small vials of vilagadone hydrochloride (HCl)

↳ 10 mg vial (cat # SML 1098-10mg) batch# 084M4714V

↳ 50 mg vial (cat # SML 1098-50mg) batch# 084M4714V

* 3 vials (1 draw, borosilicate glass, VWR # 66011-041) were charged with a small amount of sample and 3 different solvents (see details below); magnetic stir bars (VWR # 76001-874) were added, and the mixtures were stirred (100 rpm) overnight at room temperature (23-24 °C)

Vial ① (labeled IPA)

sample: 12 mg

iPA (isopropyl alcohol): 1 mL

Vial ② (labeled THF)

sample: 11 mg

THF anhyd (tetrahydrofuran): 1 mL anhydrous

Vial ③ (labeled H₂O^{GG})

sample: 11 mg

H₂O (distilled water): 1 mL

(*) PXRD : instrument : Bruker D2 Phaser

General protocol

- the sample was spread evenly on a low background sample holder

5 - for the θ , the sample was taken out of the freezer and measured immediately. The vial was then sealed with parafilm (VWR # 52858-000) and placed back in the freezer in a plastic bag that contains ~~indicator~~ ^{GG desiccant} packs

10 Method 1:

2θ : 3 - 40

step size : 0.02

time : 2 s

PSD opening: 2.5

15

file: VtCl.brawl

(*) The mixtures from the 3 vials (see page #1) were filtered (with suction) through Whatman Grade 1 filter paper (55 mm diameter, VWR # 28450-048)

20

- although some solid was observed on the filter paper, nothing could be recovered for further measurements. Careful scraping of the filter paper resulted in paper fibers collection.

- some solid observed in the filtrate after solvent evaporation

25

① * NMR

- approximately 2 mg of VHC1 (as received) were placed in an NMR tube (clean) and 0.7 mL deuterated DMSO (Cambridge Isotope Laboratories, Inc, cat# DLM-1070x0.75) was added and ^1H NMR spectrum recorded on a Bruker Avance 500 instrument

file: ggl-102617-1-1.bnmr

② * 2 vials (1 draw, microsilicate glass, VWR # 66011-041) were charged with a small amount of VHC1 and 2 different solvents (details below); magnetic stirbars were added (VWR # 76001-~~878~~) and the mixtures were stirred (100 rpm) overnight.

Vial 1 : taxa: 6.0251g w/lid \rightarrow labeled ^1PA 2
 sample: 11.6 mg
 ^1PA : 1 mL

Vial 2 : taxa: 6.0105g w/lid \rightarrow labeled TRF 2
 sample: 11.9 mg
 TRF aub: 1 mL

(*) The mixtures (page #3) were centrifuged (Sorvall Legend XF floor centrifuge, Thermo Scientific) at 3000 rpm for 15 min.

The supernatant was removed and added to another vial:

¹PA vial tara: 5.9625 g w/lid

5 TRF vial tara: 6.0257 g w/lid

- the solid vials were covered with Al foil (punched with a needle in few places) and dried at room temperature under vacuum

- the solvent was evaporated under vacuum (room temperature)

(*) A SC

- instrument: TA Instruments Q2000

- general protocol

- the aluminum pan (Tzero Hermetic Pan, cat # 901683.901) and the aluminum lid (Tzero Hermetic lid, cat # 901684.901) were

15 weighed, tared, and the sample (typically between 1-5 mg) was placed in the pan and the pan was sealed using the Tzero Press (cat # 901600.901) and the tie fet (cat # 901608.901)

- the measurements were done using pans with a pinhole (made with a 25G needle)

20 - all A SC measurements were done under nitrogen (10 mL/min flow rate)

- the instrument calibration was done on 08/19/17

Method

25 (#1) Temperature range: 25-350 °C

heating rate: 10 °C/min

small pieces of filter paper with solid were used for measurements (except for vials) which was sampled out from bottles

files: VHCI.001
 VHCI iPA.001
 VHCI TRF.001
 VHCI H2O.001

* TGA

- instrument: TA Instruments Q600

- general protocol

- a 90 μ l ceramic pan (alumina, cat # 960070.901) was loaded on the instrument and tared (using the instrument software controls)

- the sample (typically between 1-5 mg) was loaded into the pan, the furnace was closed, and the experiment started

- the instrument calibration was done on 08/31/2016

method

temperature range: room temperature ($\sim 20-25^\circ\text{C}$) - ⁶⁰⁰600 $^\circ\text{C}$

heating rate: $10^\circ\text{C}/\text{min}$

small pieces of filter paper with solid were used for measurements (except for VHCI which was sampled out from the bottle)

files: VHCI.001
 VHCI iPA.001

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