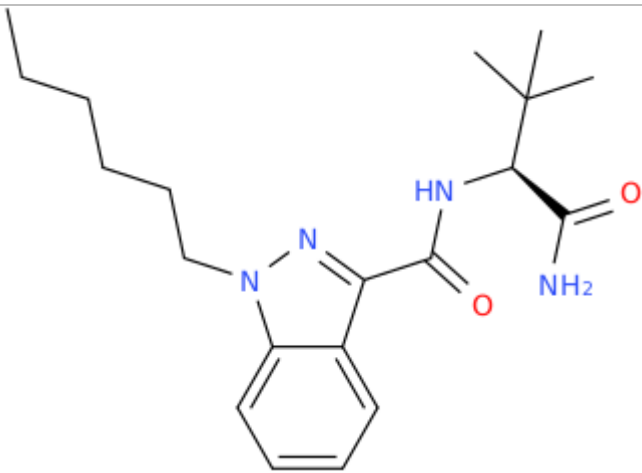


ANALYTICAL REPORT<sup>1</sup>ADB-HEXINACA (C<sub>20</sub>H<sub>30</sub>N<sub>4</sub>O<sub>2</sub>)

## (2S)-2-[(1-hexyl-1H-indazol-3-yl)formamido]-3,3-dimethylbutanamide

Remark – other active cpd. detected

|                             |                       |
|-----------------------------|-----------------------|
| Sample ID:                  | 2995-21               |
| Sample description:         | powder - white        |
| Sample type:                | RM-reference material |
| Comments:                   | CAY Lot#0611894-14,   |
| Date of entry (DD/MM/YYYY): | 25/10/2021            |

|                                                         |                                                                                            |
|---------------------------------------------------------|--------------------------------------------------------------------------------------------|
| Substance identified-structure <sup>2</sup> (base form) |         |
| Systematic name:                                        | (2S)-2-[(1-hexyl-1H-indazol-3-yl)formamido]-3,3-dimethylbutanamide                         |
| Other names:                                            | (S)-N-(1-amino-3,3-dimethyl-1-oxobutan-2-yl)-1-hexyl-1H-indazole-3-carboxamide; ADB-HINACA |
| Formula (per base form)                                 | C <sub>20</sub> H <sub>30</sub> N <sub>4</sub> O <sub>2</sub>                              |
| M <sub>w</sub> (g/mol)                                  | 358,49                                                                                     |
| Salt form:                                              | base                                                                                       |
| StdInChIKey (per base form)                             | PZMLDAGKYPJWHJ-QGZVFWFLSA-N                                                                |
| Other active cpd. detected                              |                                                                                            |
| Add.info (purity..)                                     | ≥98%                                                                                       |

<sup>1</sup> Approved by: Dr. Sonja Klemenc<sup>2</sup> Created by OPSIN free tool: <http://opsin.ch.cam.ac.uk/> DOI: 10.1021/ci100384d

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## Report updates

| date | comments (explanation) |
|------|------------------------|
|      |                        |
|      |                        |
|      |                        |
|      |                        |

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## Supporting information

| Analytical technique:   | applied | remarks                                                         |
|-------------------------|---------|-----------------------------------------------------------------|
| GC-MS (EI ionization)   | +       | NFL GC-RT (min): 11,76 BP(1): 229; BP(2): 314,BP(3) :145,       |
| FTIR-ATR                | +       | direct measurement                                              |
| GC-IR (condensed phase) | +       | always as base form                                             |
| HPLC-TOF                | +       | exact mass theoretical: 358,2369 / measured $\Delta$ ppm: -1,79 |

**1. GC-MS (Agilent):** GC-method is RT locked to tetracosane (9.258 min). Injection volume 1  $\mu$ l and split mode (1:50). Injector temperature: 280 °C. Chromatographic separation: on column HP1-MS (100% dimethylpolysiloxane), length 30 m, internal diameter 0.25 mm, film thickness 0.25  $\mu$ m. Carrier gas He: flow-rate 1.2 ml/min. GC oven program: 170 °C for 1 min, followed by heating up to 190 °C at rate 8 °C/min, then heating up to 293 °C at a rate of 18 °C/min, hold for 6.1 min, then heating at 50 °C/min up to 325 °C and finally 9.1 min isothermal. MSD source EI = 70 eV. GC-MS transfer line T= 235°C, source and quadropole temperatures 280°C and 180°C, respectively. Scan range m/z scan range: from 50 (30 until 6 min.) to 550 (300 until 6 min) amu.

**2. FTIR-ATR (Perkin Elmer):** scan range 4000-400  $\text{cm}^{-1}$ ; resolution 4 $\text{cm}^{-1}$

**3. GC- (MS)-IR condensed phase (GC-MS (Agilent) & IR (Spectra analyses-Danny)**

GC-method: Injection volume 1  $\mu$ l and split mode (1:5). Injector temperature 280 °C. Chromatographic separation as above (1). Split MS: IR = 1 : 9.

MSD source EI = 70 eV. GC-MS transfer line T= 235°C, source and quadropole temperatures 280°C and 180°C, respectively. Scan range m/z scan range: from 50 (30 until 6 min.) to 550 (300) amu.

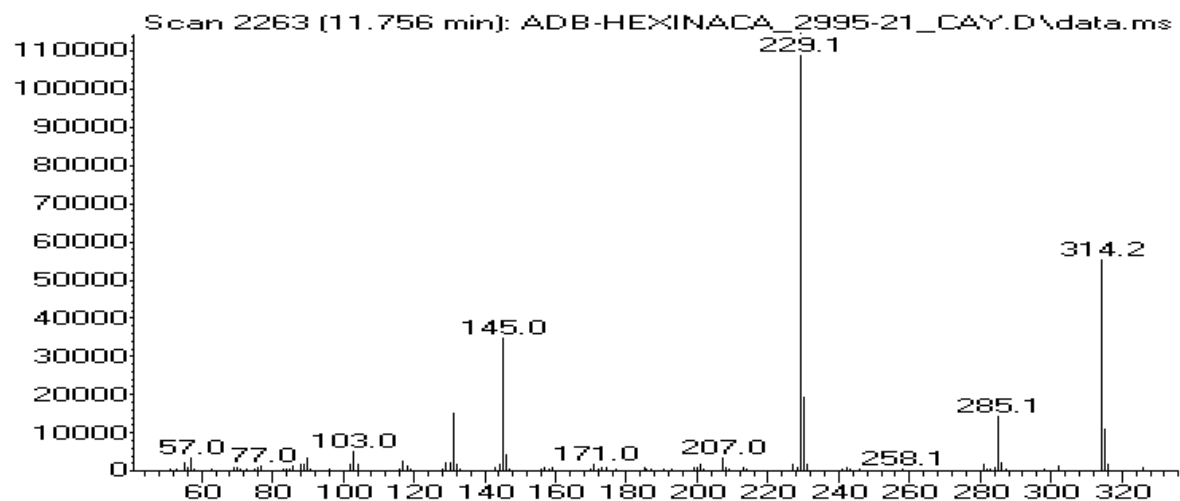
IR (condensed (solid) phase): IR scan range 4000 to 650, resolution 4  $\text{cm}^{-1}$ .

**4. HPLC-TOF (Agilent):** 6230B TOF with Agilent 1260 Infinity HPLC with binary pump, column: Zorbax Eclipse XDB-C18, 50 x 4.6 mm, 1.8 micron. Mobile phases (A) 0.1% formic acid and 1mM ammonium formate in water; (B) 0.1% formic acid in methanol (B). Gradient: starting at 5% B, changing to 40% B over 4 min, then to 70% over 2 min and in 5 min to 100%, hold 1 min and back to 5%, equilibration for 1.7 min. The flow rate: 1.0 ml/min; injection volume 1  $\mu$ l. MS parameters: 2GHz, Extended Dynamic range mode to a maximum of 1700 amu, acquisition rate 1.30 spectra/sec. Sample ionisation: by Agilent Jet Stream technology (Dual AJS ESI). Ion source: positive ion scan mode with mass scanning from 82 to 1000 amu. Other TOF parameters: drying gas (N<sub>2</sub>) and sheath temperature 325 °C; drying gas flow rate 6 l/min; sheath gas flow rate 8 l/min; nebulizer 25 psig; Vcap. 4000 V; nozzle 2000 V; skimmer 65 V; fragmentor 175 V and Octopole RF 750 V.

# ANALYTICAL RESULTS

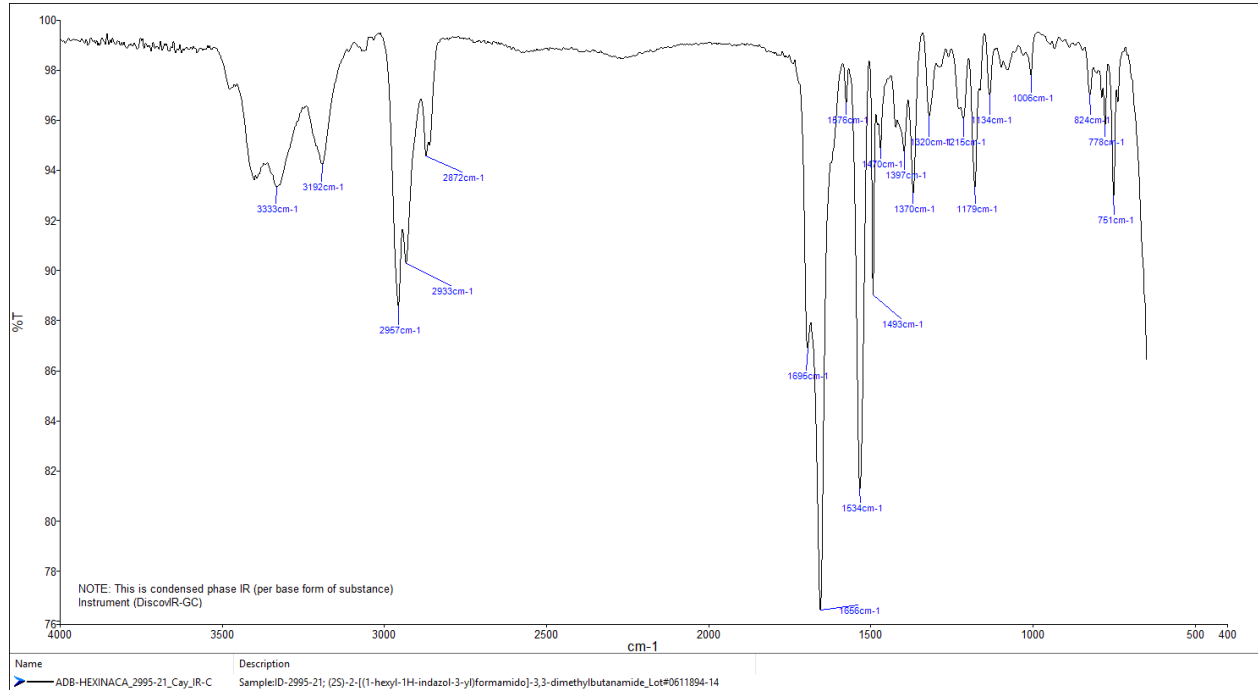
MS (EI)

Abundance



m/z-->

FTIR-ATR (direct measurement – sample as received)



IR- (condensed (solid) phase – after chromatographic separation) - spectrum per base form

