

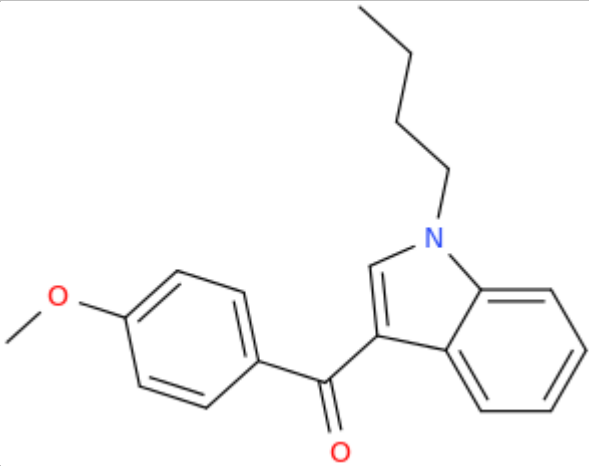
ANALYTICAL REPORT

RCS-4-C4-homolog (C₂₀H₂₁NO₂)

1-butyl-3-(4-methoxybenzoyl)-1H-indole

Remark – other NPS detected: none

| | |
|---|---|
| Sample ID: | 1957-18 |
| Sample description: | powder |
| Sample type: | RM-reference material /NFL- purchasing |
| Date of sample receipt (DD/MM/YYYY): | 08/08/2018 |
| Date of entry (DD/MM/YYYY) into NFL database: | 14/08/2018 |
| Report updates (if any) will be published here: | http://www.policija.si/apps/nfl_response_web/seznam.php |

| | |
|---|---|
| Substance identified - structure ¹ (base form) |  |
| Systematic name | 1-butyl-3-(4-methoxybenzoyl)-1H-indole |
| Other names | (4-methoxyphenyl)(1-butyl-1H-indol-3-yl)-methanone; BTM-4; E-4; OBT-199; SR-19; |
| Formula (per base form) | C ₂₀ H ₂₁ NO ₂ |
| M _w (g/mol) | 307,39 |
| Salt form/anions detected | base |
| StdInChIKey (per base form) | INTUAOVLJFCJIP-UHFFFAOYSA-N |
| Other NPS detected | none |
| Additional info (purity..) | 100 % |

¹ Created by OPSIN free tool: <http://opsin.ch.cam.ac.uk/> DOI: 10.1021/ci100384d

Report updates

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

Instrumental methods (if applied) in NFL

1. GC-MS (Agilent): GC-method is RT locked to tetracosane (9.258 min). Injection volume 1 ml 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 µm. 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 7.1 min, then heating at 50 °C/min up to 325 °C and finally 6.1 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. 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 µl. 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 (N₂) 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.

3. FTIR-ATR (Perkin Elmer): scan range 4000-400 cm⁻¹; resolution 4cm⁻¹

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

GC-method: Injection volume 1 ml 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 cm⁻¹.

5. IC (anions) (Thermo Scientific, Dionex ICS 2100), Column: IonPac AS19, 2 x 250mm; Eluent: 10mM KOH from 0 to 10 min, 10-58 mM from 10 to 40min; Flow rate: 0.25 ml/min; Temperature: 30°C; Suppressor: AERS 500 2mm, suppressor current 13mA; Inj. Volume: 25 µl

Supporting information

| Solubility in | result/remark |
|---------------------------------|---------------|
| CH ₂ Cl ₂ | soluble |
| MeOH | soluble |
| H ₂ O | |

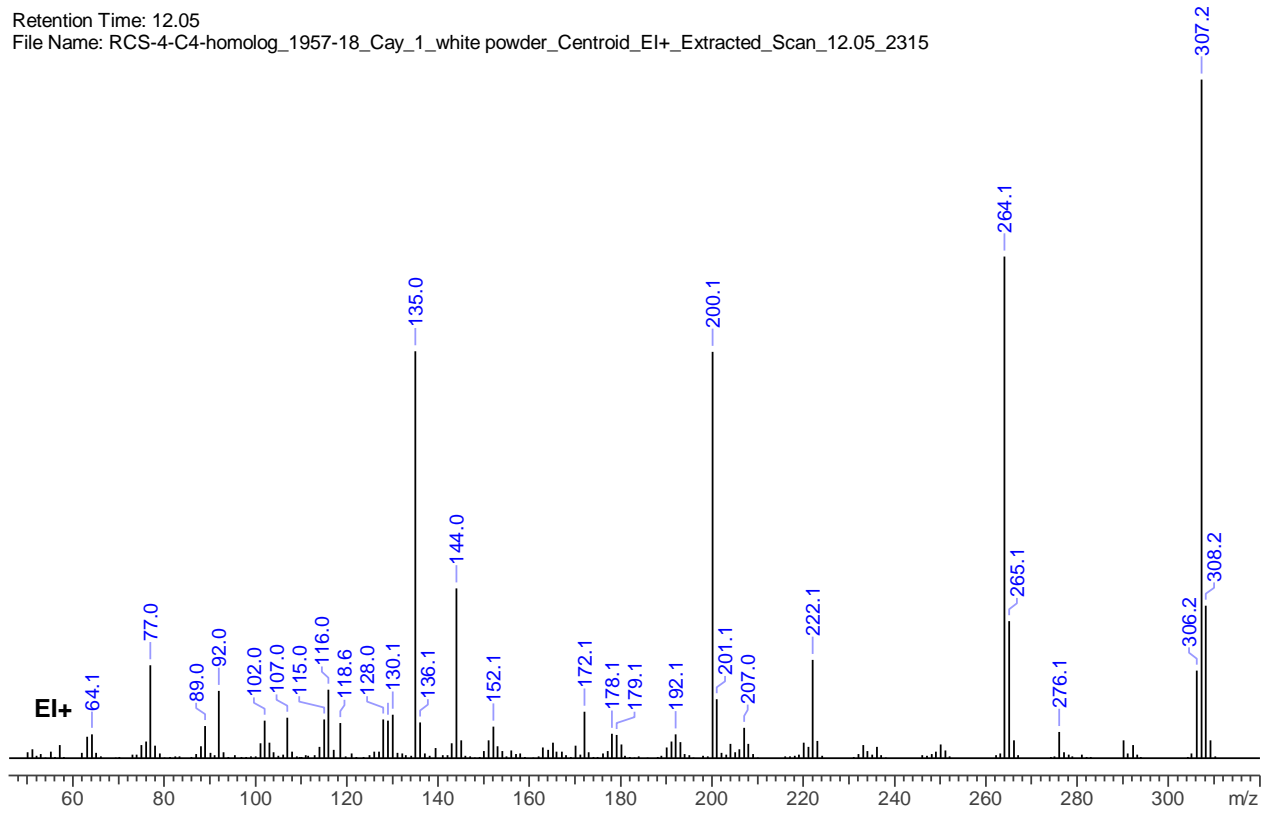
| Analytical technique: | applied | remarks |
|--|---------|---|
| GC-MS (EI ionization) | + | NFL GC-RT (min): 12,05 BP(1): 307; BP(2): 264,BP(3) :135, |
| HPLC-TOF | + | Exact mass (theoretical): 307,1572; measured value Δppm:0,01; formula:C ₂₀ H ₂₁ NO ₂ |
| FTIR-ATR | + | direct measurement (sample as received) |
| FTIR (solid phase) always as base form | + | |
| IC (anions) | | |
| NMR (in FKKT) | - | |
| validation | | |
| other | | |

ANALYTICAL RESULTS

MS (EI)

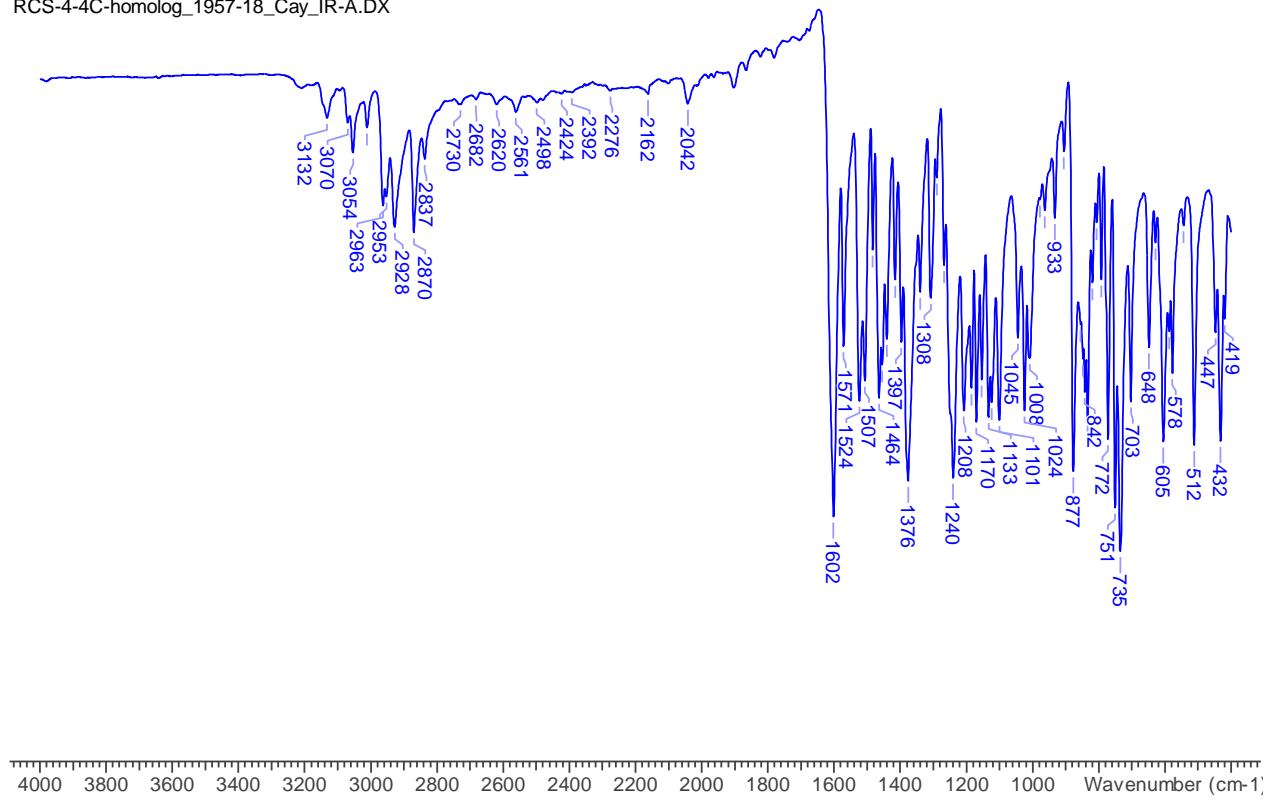
Retention Time: 12.05

File Name: RCS-4-C4-homolog_1957-18_Cay_1_white powder_Centroid_EI+_Extracted_Scan_12.05_2315

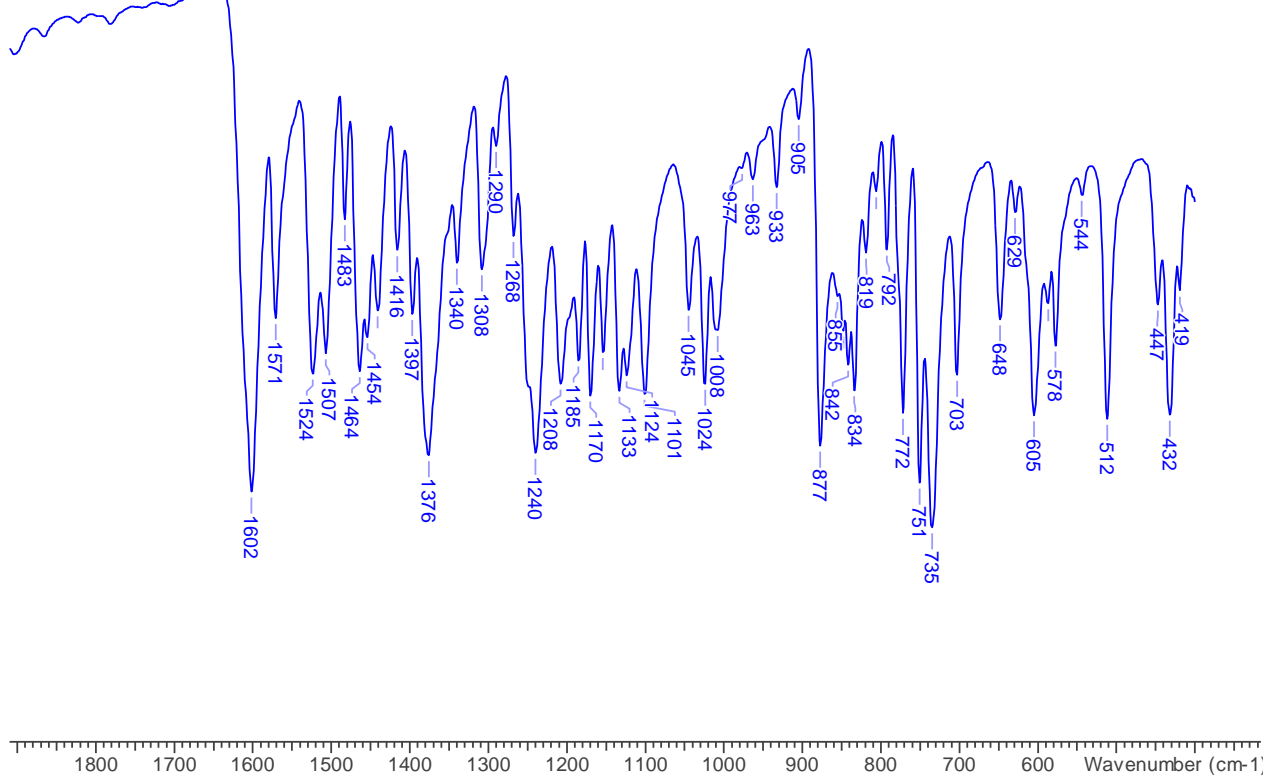


FTIR-ATR - direct measurement (sample as received)

RCS-4-4C-homolog_1957-18_Cay_IR-A.DX

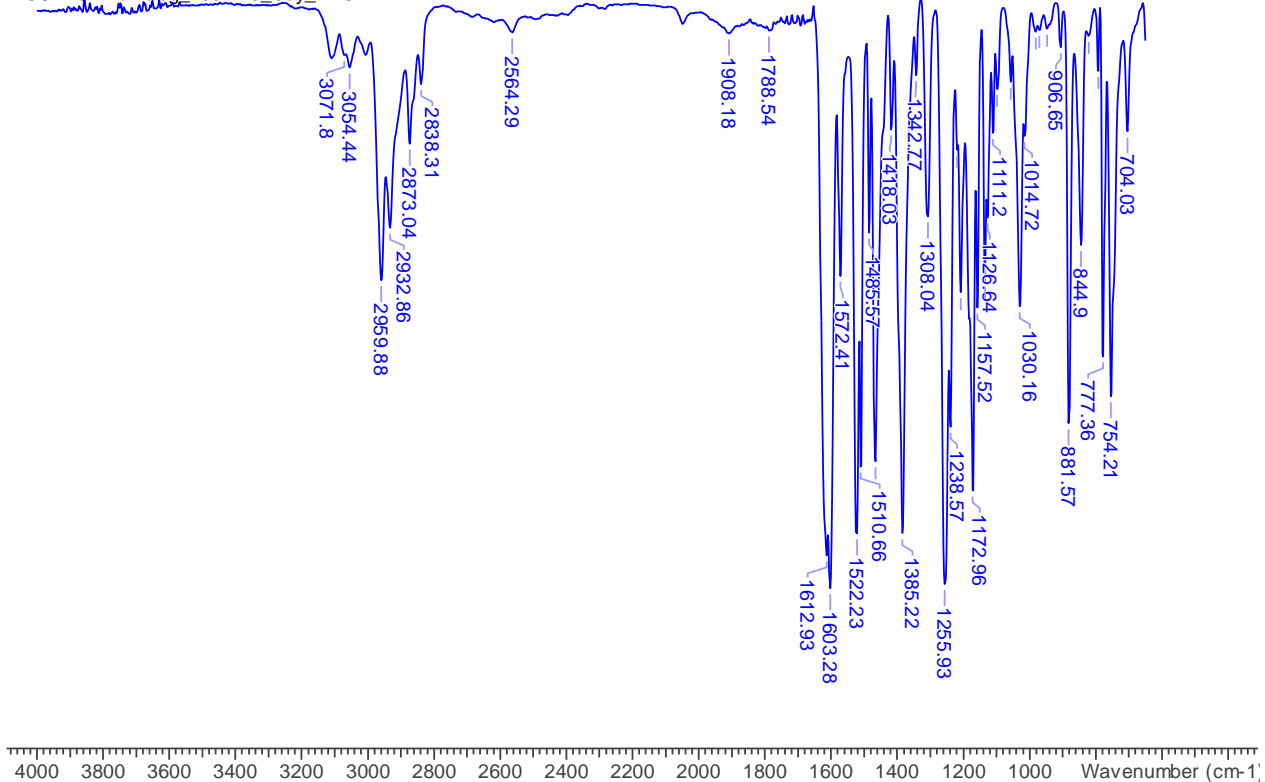


RCS-4-4C-homolog_1957-18_Cay_IR-A.DX



IR (solid phase – after chromatographic separation)

RCS-4-C4-homolog_1957-18_Cay_IR-C.DX



RCS-4-C4-homolog_1957-18_Cay_IR-C.DX

