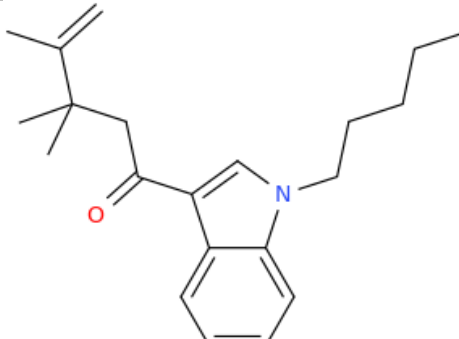


## ANALYTICAL REPORT

UR-144 Degradant (C<sub>21</sub>H<sub>29</sub>NO)**3,3,4-trimethyl-1-(1-pentyl-1H-indol-3-yl)pent-4-en-1-one**Remark – other active cpd. detected **none**

Sample ID:	2166-20
Sample description:	liquid - transparent
Sample type:	RM-reference material
Comments:	CAY Lot#0463169-14,
Date of entry (DD/MM/YYYY):	15/05/2020

Substance identified-structure <sup>1</sup> (base form)	
Systematic name:	3,3,4-trimethyl-1-(1-pentyl-1H-indol-3-yl)pent-4-en-1-one
Other names:	KM-X1 Degradant; UR-144 3,3,4-Trimethylpentenoyl isomer
Formula (per base form)	C <sub>21</sub> H <sub>29</sub> NO
M <sub>w</sub> (g/mol)	311,47
Salt form:	base
StdInChIKey (per base form)	NBJHWTCAQOYUND-UHFFFAOYSA-N
Other active cpd. detected	none
Add.info (purity..)	≥98%; 5mg/0.5ml methanol solution

<sup>1</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): 9,84 BP(1): 214; BP(2): 229,BP(3) :296,
FTIR-ATR	-	direct measurement
GC-IR (condensed phase)	+	always as base form
HPLC-TOF	+	exact mass theoretical: 311,2249 / measured $\Delta$ ppm: 0,02

**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 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. 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.

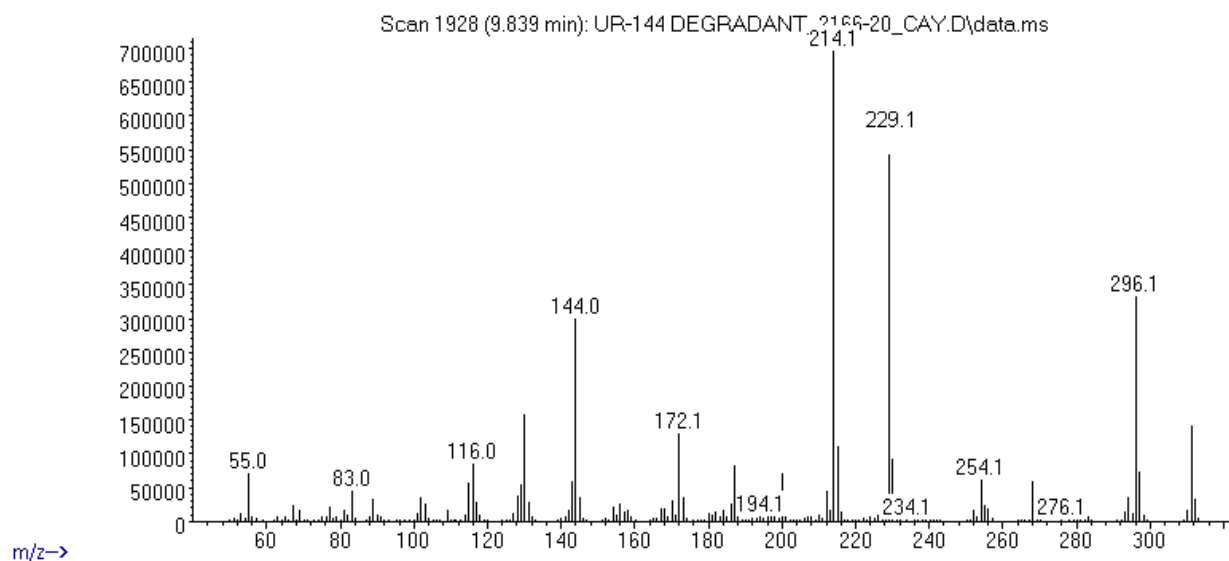
IR (condensed (solid) phase): IR scan range 4000 to 650, resolution 4  $\text{cm}^{-1}$ .

**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



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

