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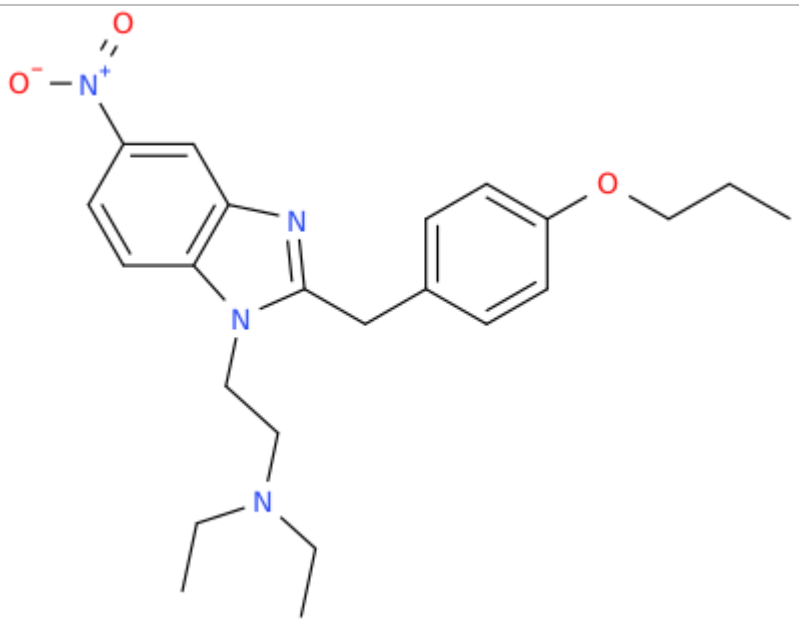
ANALYTICAL REPORT[1]

Protonitazene (C23H30N4O3)

diethyl(2-{5-nitro-2-[(4-propoxyphenyl)methyl]-1H-1,3-benzodiazol-1-yl}ethyl)amine

Remark – other active cpd. detected

Sample ID:	3024-21
Sample description:	powder - white
Sample type:	RM-reference material
Comments:	CAY Lot#0575807-34,
Date of entry (DD/MM/YYYY):	

Substance identified-structure[2] (base form)	
Systematic name:	diethyl(2-{5-nitro-2-[(4-propoxyphenyl)methyl]-1H-1,3-benzodiazol-1-yl}ethyl)amine
Other names:	N,N-diethyl-5-nitro-2-[(4-propoxyphenyl)methyl]-1H-benzimidazole-1-ethanamine; Pronitazene
Formula (per base form)	C23H30N4O3
M_w (g/mol)	410,52
Salt form:	HCl
StdInChIKey (per base form)	SJHUJFHOXYDSJY-UHFFFAOYSA-N
Other active cpd. detected	

[1] Approved by: Dr. Sonja Klemenc
[2] Created by OPSIN free tool: http://opsin.ch.cam.ac.uk/ **DOI:** 10.1021/ci100384d

Add.info (purity..)	≥98%

Report updates

date	comments (explanation)

Supporting information

Analytical technique:	applied	remarks
GC-MS (EI ionization)	+	NFL GC-RT (min): 17,68 BP(1): 86; BP(2): 107,BP(3) :87,
FTIR-ATR	+	direct measurement
GC-IR (condensed phase)	+	always as base form
HPLC-TOF	+	exact mass theoretical: 410,2318 / measured Δppm: -0,6

1. GC-MS (Agilent): GC-method is RT locked to tetracosane (9.258 min). Injection volume 1 µl and split mode (1:50). Injector temperature: 280 0C. Chromatographic separation: on column HP1-MS (100% dimethylpolysiloxane), length 30 m, internal diameter 0.25 mm, film thickens 0.25 µm. Carrier gas He: flow-rate 1.2 ml/min. GC oven program: 170 ^{0}C for 1 min, followed by heating up to 190 ^{0}C at rate 8 ^{0}C/min, then heating up to 293 0C at a rate of 18 ^{0}C/min, hold for 6.1 min, then heating at 50 ^{0}C/min up to 325 ^{0}C and finally 9.1 min isothermal. MSD source EI = 70 eV. GC-MS transfer line T= 235^{0}C, source and quadropole temperatures 280^{0}C and 180^{0}C, respectively. Scan range m/z scan range: from 50 (30 until 6 min.) to 550 (300 until 6 min) amu.

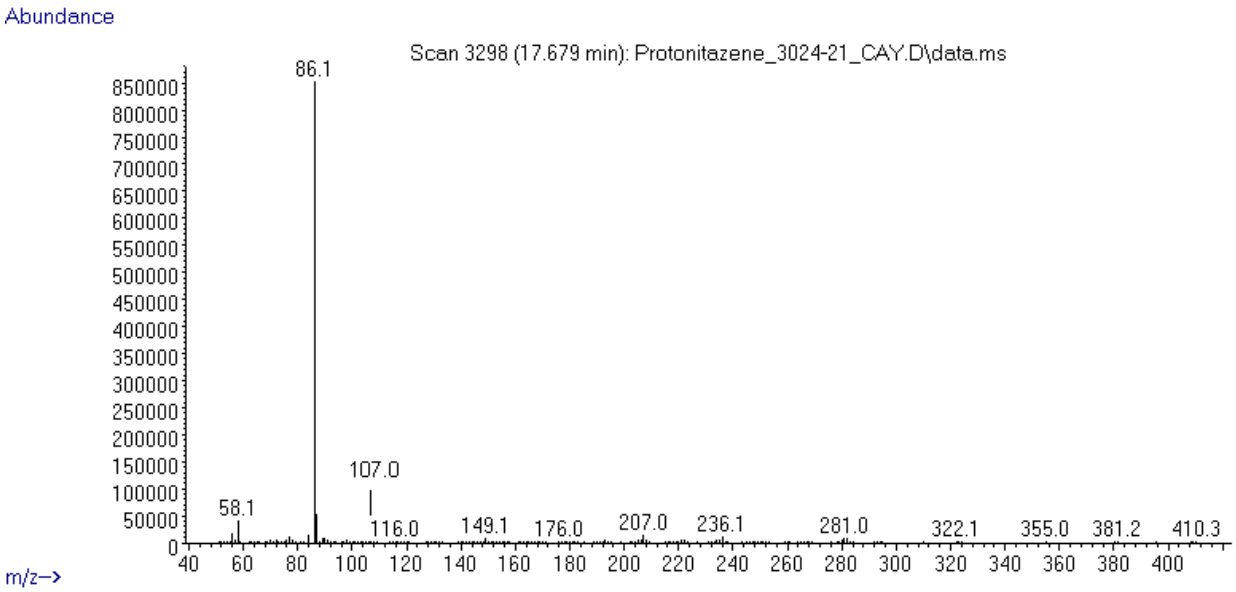
2. FTIR-ATR (Perkin Elmer): scan range 4000-400 cm^{-1}; resolution 4cm^{-1}

3. GC- (MS)-IR condensed phase (GC-MS (Agilent) & IR (Spectra analyses-Danny)
GC-method: Injection volume 1 µl and split mode (1:5). Injector temperature 280 ^{0}C. Chromatographic separation as above **(1).**
Split MS: IR = 1 : 9.
MSD source EI = 70 eV. GC-MS transfer line T= 235^{0}C, source and quadropole temperatures 280^{0}C and 180^{0}C, respectively. Scan range m/z scan range: from 50 (30 until 6 min.) to 550 (300) amu.
IR (condesed (solid) phase): IR scan range 4000 to 650, resolution 4 cm^{-1}.

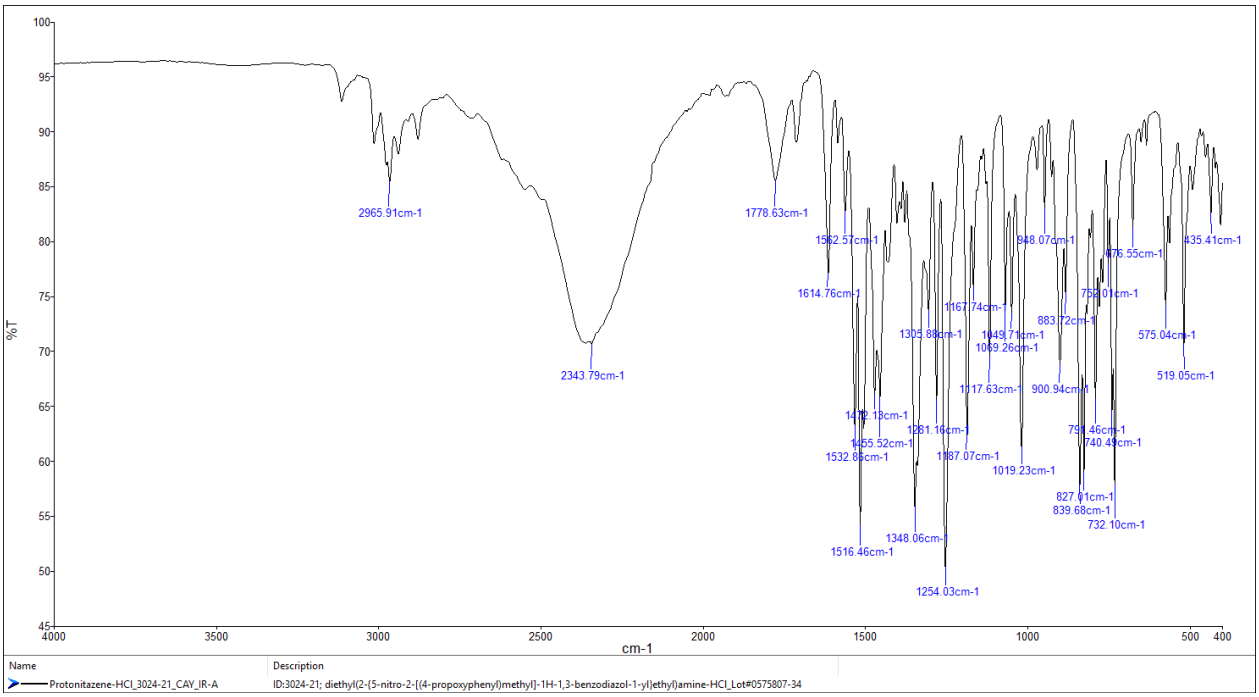
4. **HPLC-TOF** (Agilent): 6230B TOF with Agilent 1260 Infinity HPLC with binary pump, column: Zorbax Eclipse XDB-C18, 50 x 4.6 mm, 1.8 micron. Mobile phases (A) 0.1% formic acid and 1mM ammonium formate in water; (B) 0.1% formic acid in methanol (B). Gradient: starting at 5% B, changing to 40% B over 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 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.

ANALYTICAL RESULTS

MS (EI)



FTIR-ATR (direct measurement – sample as received)



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