NACIONALNI FORENZIČNI LABORATORIJ
NATIONAL FORENSIC LABORATORY

Vodovodna 95
1000 Ljubljana
SLOVENIJA

T: +386 (0)1 428 44 93
E: nfl@policija.si
www.policija.si

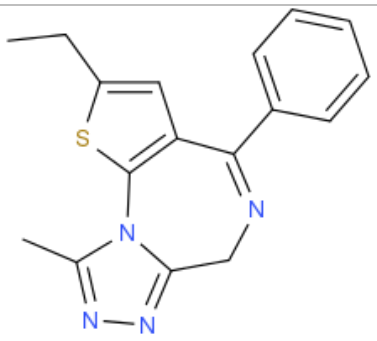
ANALYTICAL REPORT

Deschloroetizolam (C17H16N4S)

2-ethyl-9-methyl-4-phenyl-6H-thieno[3,2-f][1,2,4]triazolo[4,3-a][1,4]diazepine

Remark – other NPS detected: none

Sample ID:	1174-15
Sample description:	tablet - pink
Sample type:	test purchase
Comments:	RESPONSE -purchasing
Date of entry:	4/17/2015

Substance identified-structure[i] (base form)	
Systematic name	2-ethyl-9-methyl-4-phenyl-6H-thieno[3,2-f][1,2,4]triazolo[4,3-a][1,4]diazepine
Other names	
Formula (per base form)	C17H16N4S
M_w (g/mol)	308,4
Salt form	base
Smiles	C(C)C1=CC=2C(=NCC=3N(C2S1)C(=NN3)C)C3=CC=CC=C3
Compound Class	Benzodiazepines
Other NPS detected	none
Add.info (purity..)	

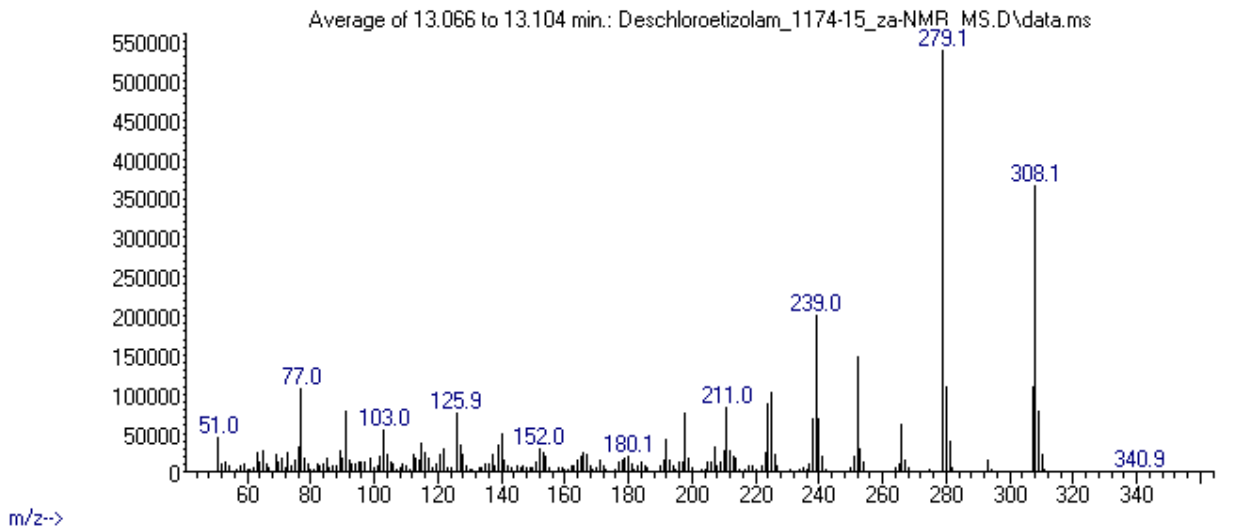
This report has been produced with the financial support of the Prevention of and fight against crime Programme of the European Union (grant agreement number JUST/2013/ISEC/DRUGS/AG/6413). The contents of this report are the sole responsibility of the National Forensic Laboratory and can in no way be taken to reflect the views of the European Commission.

Supporting information

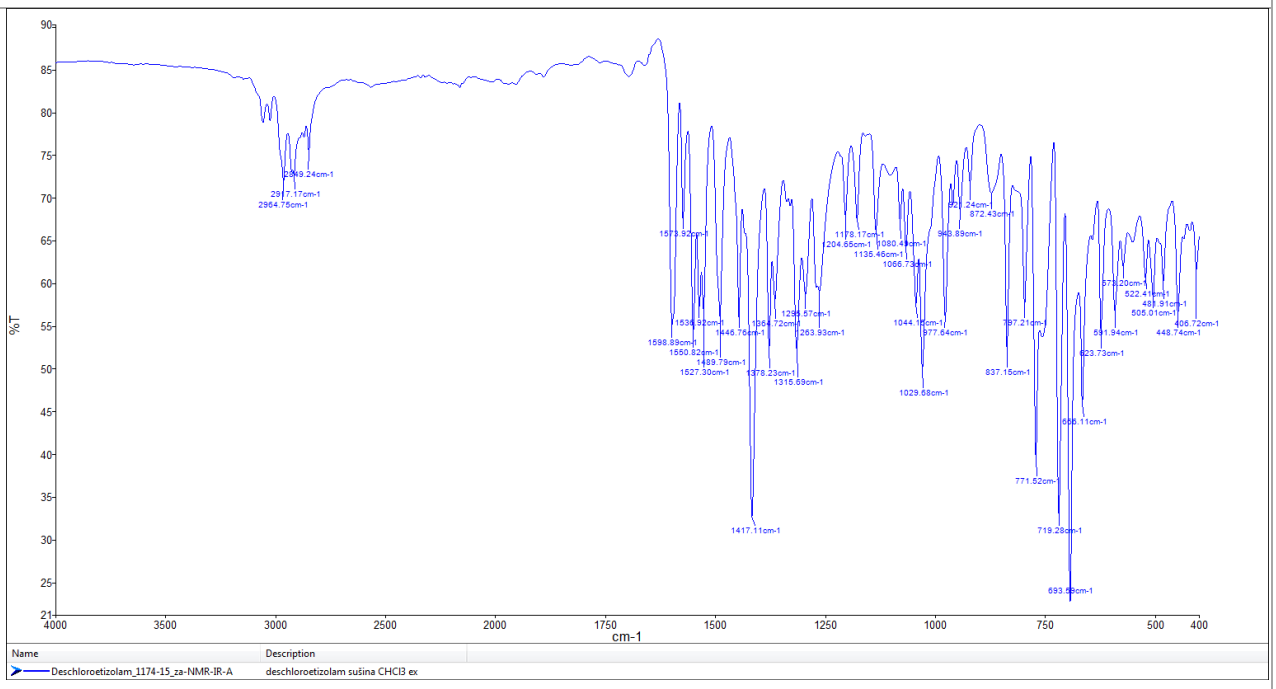
Analytical technique:	applied	remarks
GC-MS (EI ionization)	+	NFL GC-RT (min): 13,08 BP(1): 279; BP(2): 308,BP(3) :239,
FTIR-ATR	+	dried CHCl3 extract from tablet
FTIR (condensed phase) always as base form	-	
HPLC-TOF		Exact mass (theoretical): 308,1096; measured value Δppm:-2,43; formula:C17H16N4S
NMR-confirmed	+	partialy
validation		MS spectrum consistent by the one published in EMCDDA EDND database and in the literature: http://link.springer.com/article/10.1007/s11419-015-0277-6#page-1
other		

GC-MS (Agilent): GC-method is RT locked to tetracosane (RT=9.53 min). Injection volume 1 ml and split mode (1:50)for GC-MS instruments and 1:5 for GC-MS-FTIR(condensed phase). Injector temperature: 280 ^{0}C. Column: HP1-MS (100% dimethylpolysiloxane), length 30 m, internal diameter 0.25 mm, film thickens 0.25 mm. Carrier gas He: flow-rate 1.2 ml/min. GC oven program: 170 ^{0}C for 1 min, followed by heating up to 293 ^{0}C at a rate of 18 ^{0}C/min, hold for 6.1 min, than heating at 50 ^{0}C/min up to 325 ^{0}C and finally 2.8 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. m/z scan range: from 50 (40) to 550 amu.
FTIR-ATR (Perkin Elmer): scan range 4000-400 cm-1; resolution 4cm-1
FTIR (Spectra analyses-Danny): scan range 4000 to 600, resolution 4cm-1

MS spectrum (EI)



FTIR - ATR



[i] Created by OPSIN free tool: http://opsin.ch.cam.ac.uk/ **DOI:** 10.1021/ci100384d

Target Compound Screening Report

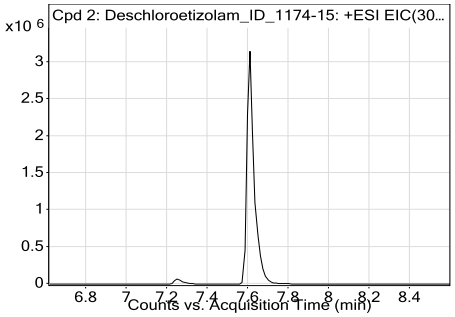
Data File	Deschloroetizolam_ID_1174-15_TOF.d	**Sample Name**	Deschloroetizolam
Sample Type	Sample	**Position**	P1-F7
Instrument Name	SG13170002	**User Name**	
Acq Method	droge general-2-4-2015-XDB-C18-ESI-poz.m	**Acquired Time**	4/10/2015 10:03:30 AM
IRM Calibration Status	Success	**DA Method**	Droge_Default.m
Comment	red tablet, extract in MeOH		

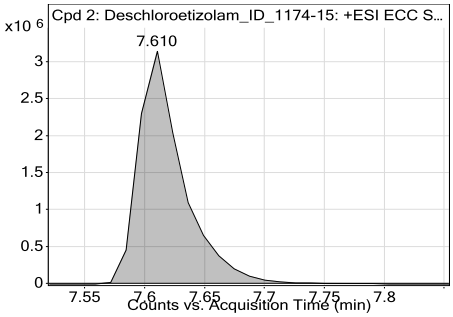
Compound Table

Label	Tgt Name	MFG Formula	Tgt Formula	Obs. RT	Obs. Mass
Cpd 2: Deschloroetizolam_ID_1174-15	Deschloroetizolam_ID_1174-15	C17 H16 N4 S	C17 H16 N4 S	7.61	308.1103

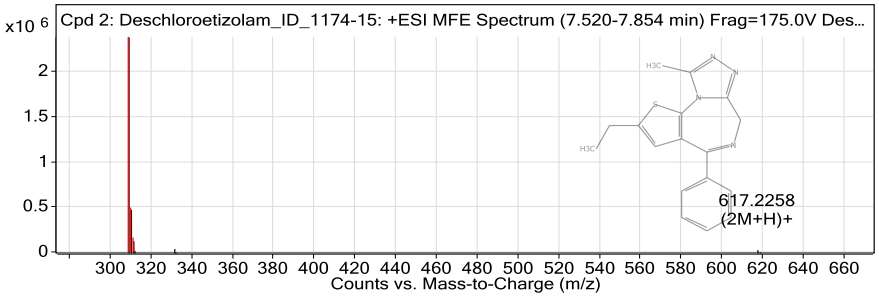
Name	*Obs. m/z*	Obs. RT	Obs. Mass	DB RT	DB Formula	DB Mass	DB Mass Error	Tgt Formula	Find Cpds Algorith
Deschloroetizolam_ID_1174-15	309.1177	7.61	308.1103	7.61	C17 H16 N4 S	308.1096	-2.43	C17 H16 N4 S	Find by Molecular Feature

Compound Chromatograms

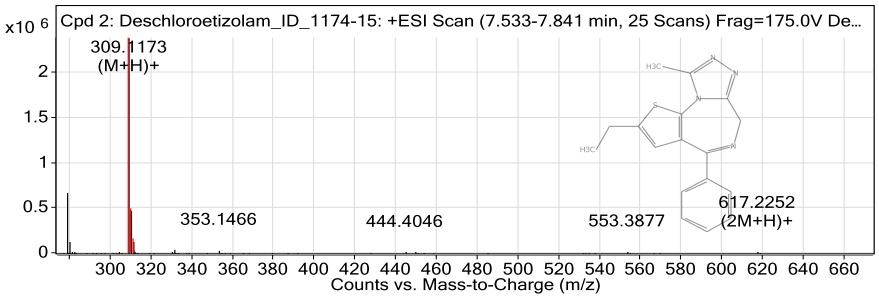




MFE MS Zoomed Spectrum



MS Zoomed Spectrum



MS Spectrum Peak List

Obs. m/z	Charge	Abund	Formula	Ion/Isotope
309.1177	1	2386072	C17 H16 N4 S	(M+H)+
310.1199	1	481698.52	C17 H16 N4 S	(M+H)+
311.116	1	141143	C17 H16 N4 S	(M+H)+
312.1171	1	22869.43	C17 H16 N4 S	(M+H)+
331.0991	1	45489.21		(M+Na)+
332.1025	1	10623.43		(M+Na)+
333.0947	1	3885.45		(M+Na)+
617.2258	1	34732.21		(2M+H)+
618.2278	1	13298.98		(2M+H)+
619.2266	1	5903.71		(2M+H)+

--- End Of Report ---

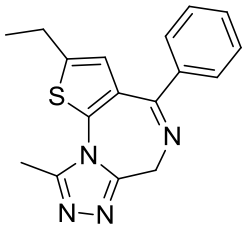
University
of Ljubljana

Faculty *of Chemistry and Chemical Technology*

Večna pot 113
P. O. Box 537
SI-1001 Ljubljana
Slovenia
Phone: +386 1 479 8558
janez.kosmrlj@fkkt.uni-lj.si

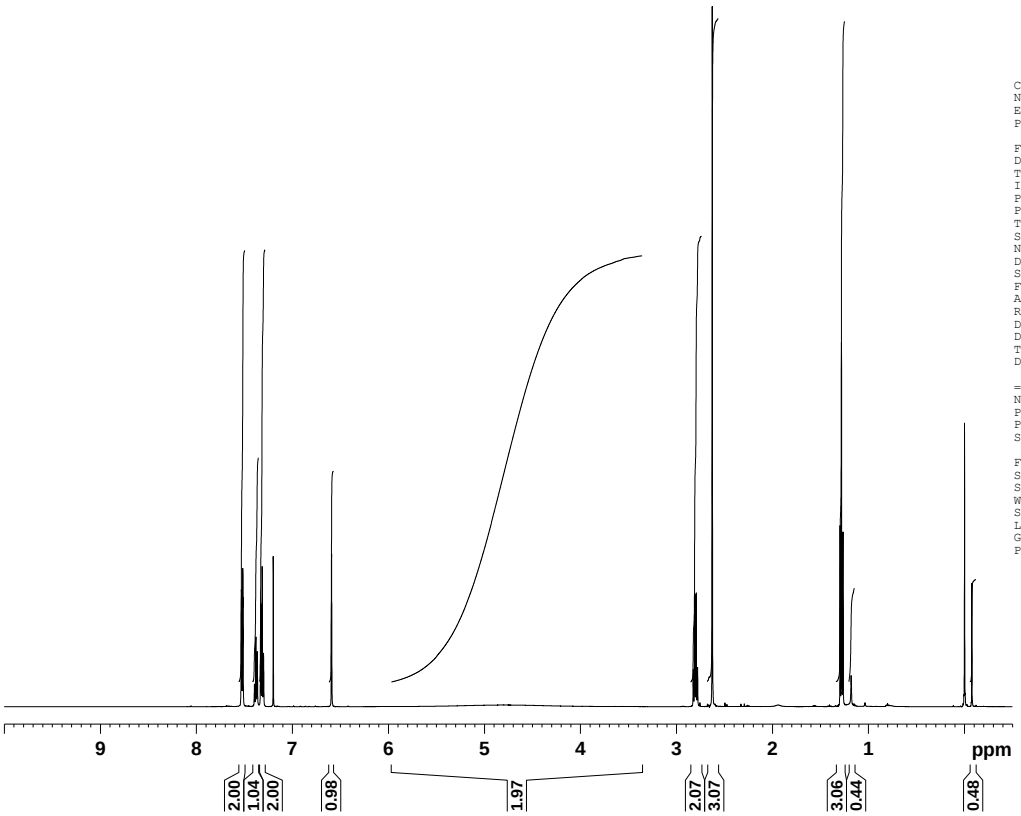
Co-funded by the Prevention of and Fight against Crime Programme of the European Union

REPORT

Sample ID:	**1174-15**
Our notebook code:	P-1174-15
NMR sample preparation:	15 mg dissolved in 0.7 mL $CDCl_3$
NMR experiments:	^1H, ^{13}C, ^1H–^1H *gs*-COSY, ^1H–^{13}C *gs*-HSQC, ^1H–^{13}C *gs*-HMBC, ^1H–^{15}N *gs*-HMBC.
Proposed structure with chemical name:	
Comments:	- Two resonances of impurities are detected in ^1H NMR (0.08 ppm and 1.18 ppm) and ^{13}C NMR (–1.02 ppm and 28.67 ppm) (see the supporting information). - 1D and 2D NMR spectra confirm some parts of the suggested structure (**A**):  **A** i.e.: a) the appearance of ^1H NMR with the corresponding integration, and b) the number of ^{13}C NMR resonances with the corresponding chemical shifts are both in agreement with structure **A**. The lack of protons in the fused ring system and thus the lack of connectivity pattern in 2D NMR spectra prevent unambiguous structure elucidation.
Supporting information:	Copies of ^1H and ^{13}C NMR spectra in $CDCl_3$
Author:	Prof. Dr. Janez Košmrlj
Date of report:	June 20, 2015

This report has been produced with the financial support of the Prevention of and fight against crime Programme of the European Union (grant agreement number JUST/2013/ISEC/DRUGS/AG/6413). The contents of this publication are the sole responsibility of the Author and can in no way be taken to reflect the views of the European Commission.

P-1174-15



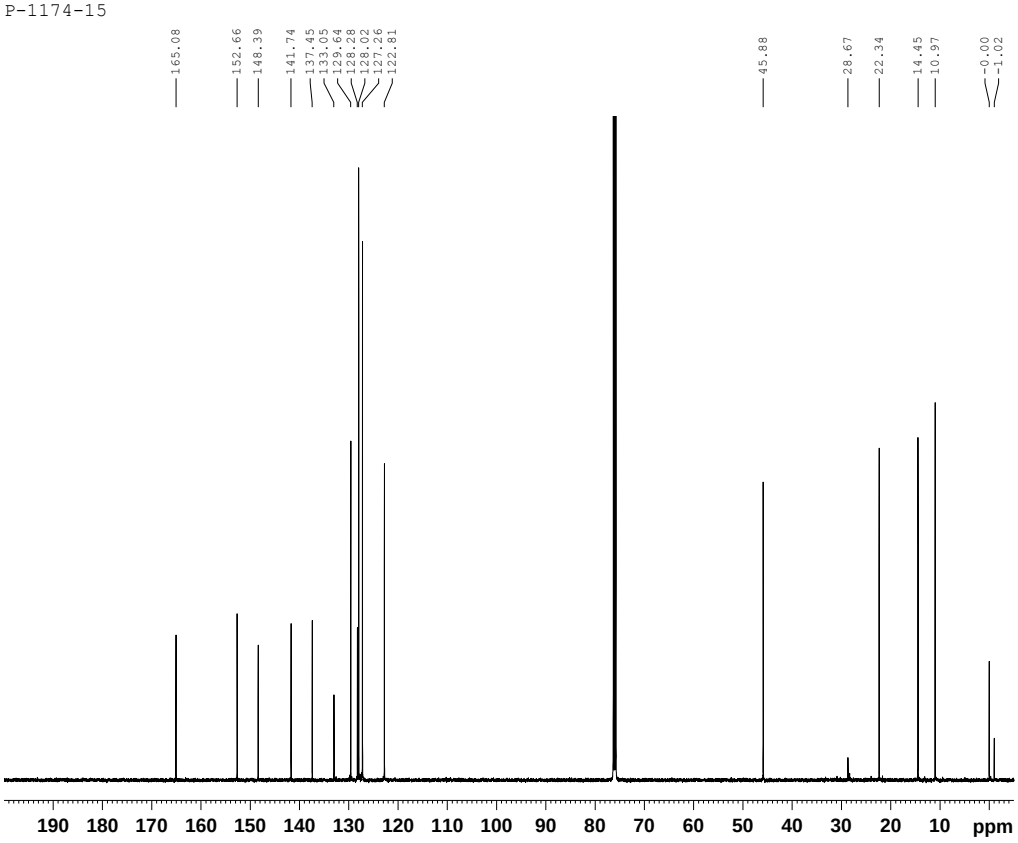
Current Data Parameters
NAME P-DESCHLOROETIZOLAM
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20150424
Time 6.55
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 10330.578 Hz
FIDRES 0.157632 Hz
AQ 3.1719923 sec
RG 71.8
DW 48.400 usec
DE 6.50 usec
TE 296.0 K
D1 1.00000000 sec

======== CHANNEL f1 ========
NUC1 1H
P1 8.90 usec
PLW1 26.00000000 W
SFO1 500.1330885 MHz

F2 - Processing parameters
SI 65536
SF 500.1300433 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

P-1174-15



Current Data Parameters
NAME P-DESCHLOROETIZOLAM
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20150424
Time 8.48
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 3072
DS 4
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010548 sec
RG 2050
DW 16.800 usec
DE 6.50 usec
TE 296.0 K
D1 1.00000000 sec
D11 0.03000000 sec

======== CHANNEL f1 ========
NUC1 13C
P1 9.00 usec
PLW1 122.00000000 W
SFO1 125.7703637 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PLW2 26.00000000 W
PLW12 0.32179001 W
PLW13 0.20595001 W
SFO2 500.1320005 MHz

F2 - Processing parameters
SI 32768
SF 125.7579188 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40