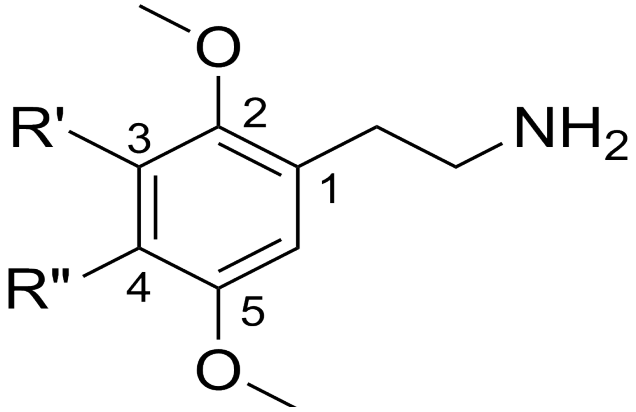
Identification and Quantification of Psychedelic Phenethylamine "2C" Series using SPE and LC-MS/MS



UCT Part Numbers

CSXCE106
Clean Screen XCEL®
I 130 mg / 6 mL

SLDA100ID21-3UM
Selectra® DA HPLC Column,
100 x 2.1 mm, 3 µm

SLDAGDC21-3UM
Selectra® DA guard column,
10 x 2.1 mm, 3 µm

SPHPHO6001-5
Select pH Buffer, Phosphate 6.0
pH / 100 mM

SLDGRDHLDR
Guard cartridge holder

Summary:

The frequent appearance of psychoactive phenethylamine designer drugs has rapidly grown within the past decade and has emerged as a matter of concern for all authorities involved. New phenethylamine drugs are being introduced because these compounds are not covered by existing legislation. Therefore, drugs cannot be considered as illicit drugs until their names are implemented. As a consequence, prompt legal action directed against their production, marketing, and consumption is not easily organized.

Among the most popular in the phenethylamine class are the "2C" compounds. Originally synthesized by Alexander Shulgin and named in such a manner as to denote the two carbon atoms between the phenyl and amine moieties. The prototype of the series, 2C-B, was introduced in 1974 and numerous analogues have since been created including 2C-I, 2C-D, 2C-P, 2C-E, 2CT2, etc. Potentially fatal intoxications have been reported involving 2C-E, 2C-T4 and 2C-T7.

Recently, a new group of 2C compounds has emerged on the market known as the N-methoxybenzyl ('NBOMe) derivatives of substituted phenethylamine. Since 2011 25B-NBOMe, 25C-NBOMe, and 25I-NBOMe have been among the compounds detected. These products are marketed under street names such as 'N-Bomb' and 'Smiles'. Like the 2C compounds, NBOMe drugs are expected to interact with alpha-adrenergic receptors, exhibiting hallucinogenic effects[1].

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Procedure:

1. Sample Preparation

a) To 1-2 mL urine add 3 mL 100mM phosphate buffer (pH 6.0)
b) Add appropriate volume and concentration of internal standard
c) Mix thoroughly

2. Apply Sample To Clean Screen® XCEL I Column

a) Load at 1 to 2 mL/minute.

3. Wash Column

a) 1 x 3 mL CH_3OH/Acetic Acid (98:2)
b) Dry column under full vacuum or pressure for 10-15 minutes

4. Elution

a) 1 x 3 mL CH_2Cl_2/IPA/NH_4OH (78:20:2)
b) Collect eluate at 1 to 2 mL/minute.
Note: Prepare elution solvent daily. Add IPA/ NH_4OH, mix, then add CH_2Cl_2 (pH 11-12).

5. Dry Eluate

a) Add 50 µL of 1% HCl in CH_3OH to each tube.
b) Evaporate fraction to complete dryness under stream of dry air or nitrogen at ~ 35 °C.
Note: A 1% HCl in CH_3OH solution has been used to prevent volatization by the formation of the hydrochloric salt of the drugs.

6. Reconstitute

a) Reconstitute sample in 100 µL of H_2O/ CH_3OH (50:50).

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Instrumental	
HPLC	Thermo Scientific™ Dionex™ Ultimate™ 3000 LC
Detector	Thermo Scientific™ TSQ Vantage™ MS/MS
Column	UCT Selectra DA, 100 x 2.1 mm, 3 µm
Guard Column	UCT Selectra DA, 10 x 2.0mm, 3 µm
Column Temperature	40 °C
Column Flow Rate	0.3 mL/min
Injection Volume	10 µL

Gradient Program		
Time (min)	**% Mobile Phase A (0.1% formic acid in H_2O)**	**% Mobile Phase B (0.1% formic acid in MeOH)**
0	60	40
1.5	60	40
3.5	35	65
5.0	35	65
5.5	5	95
7.0	5	95
7.1	60	40
11.0	60	40

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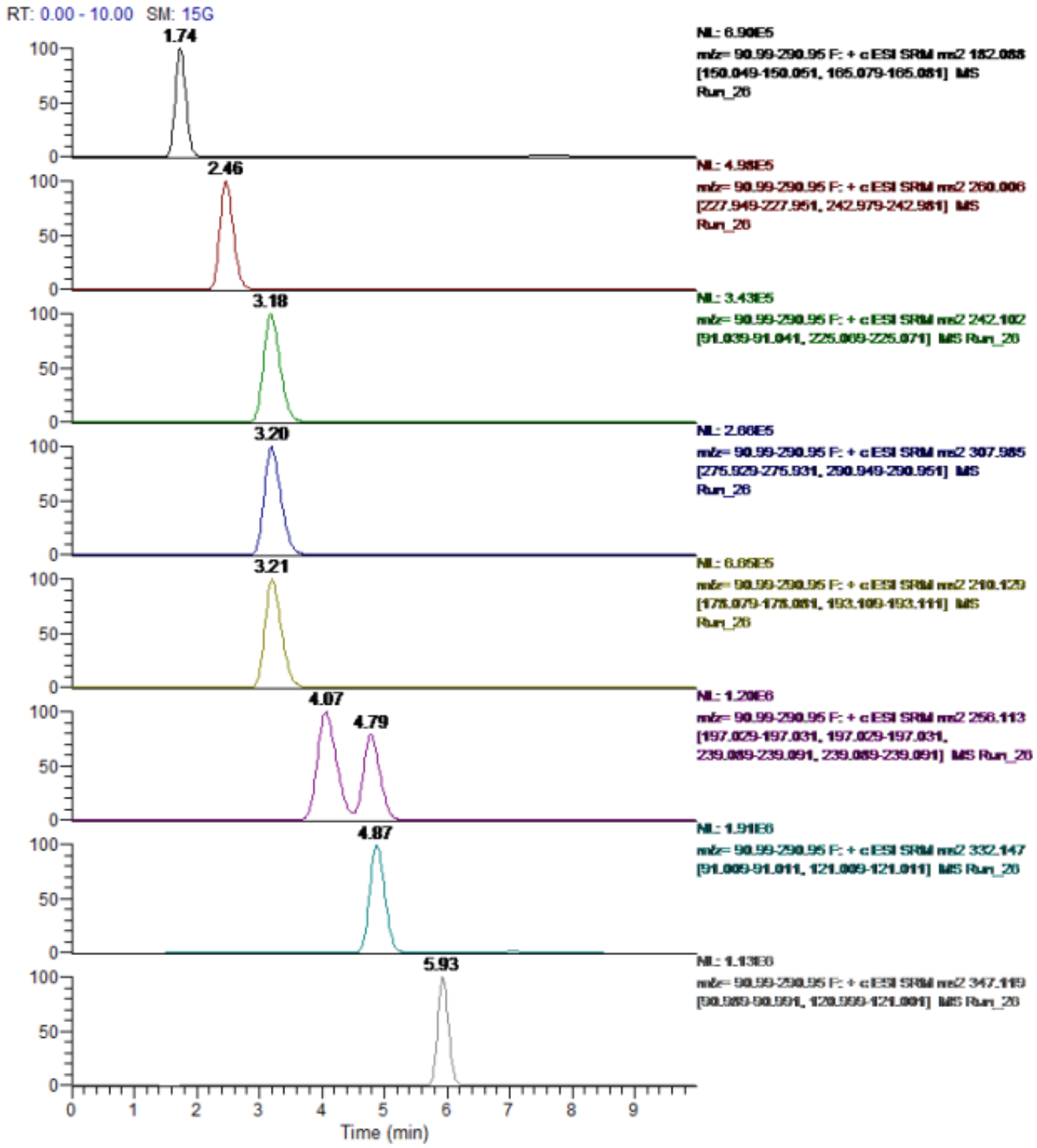

Figure 1: 16 Phenethylamine "2C" Series Compounds

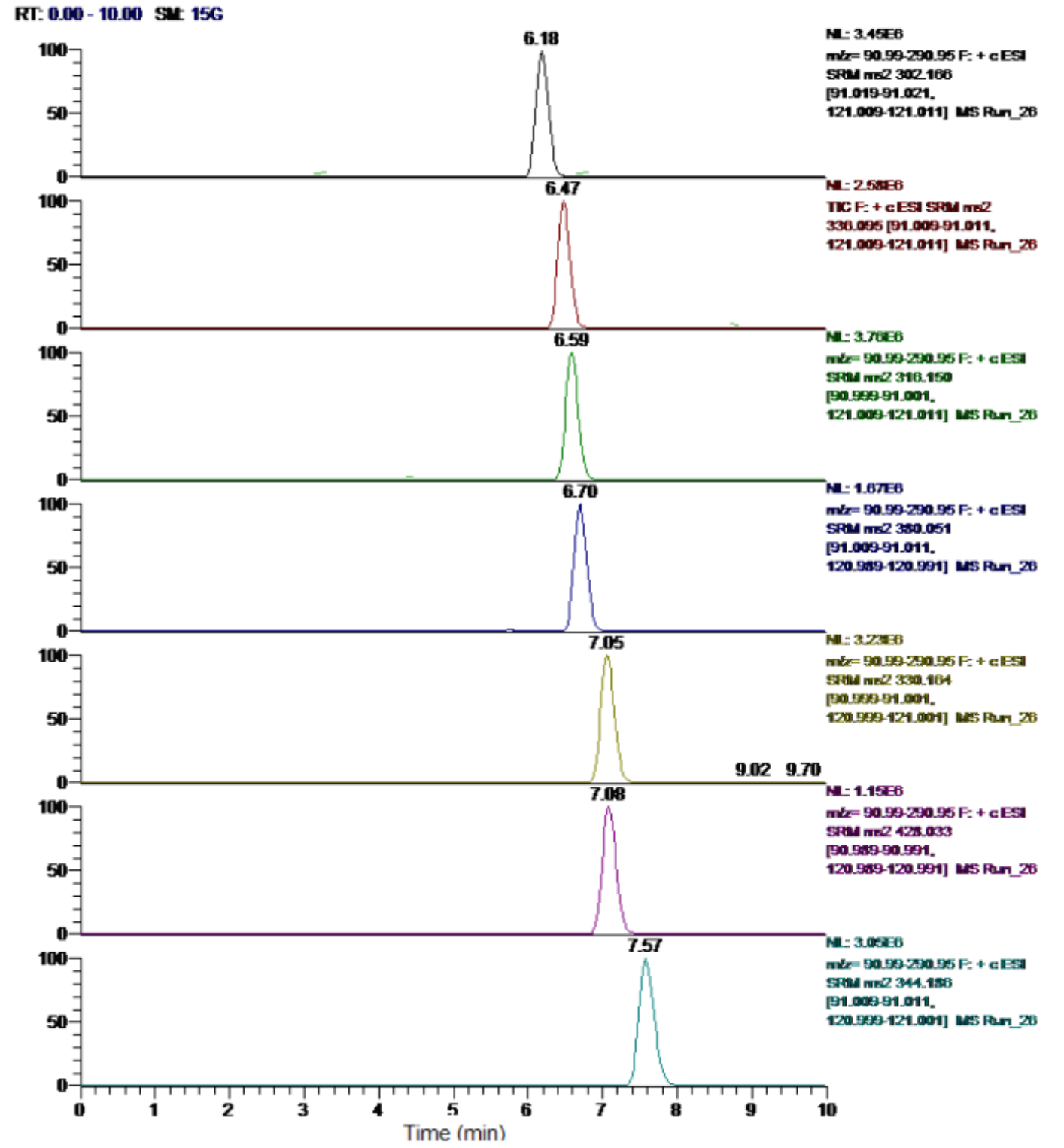

Figure 1 (cont.): 16 Phenethylamine "2C" Series Compounds

MRM transitions (ESI+, 50 ms dwell time)				
Analyte	Relative Retention Time (min)	Q1 ion	Q3 ion 1	Q3 ion 2
2C-H	1.74	182.088	165.080	150.050
2C-B	2.46	260.006	242.980	227.950
2C-T2	3.18	242.102	225.070	91.040
2C-I	3.20	307.985	290.950	275.930
2C-E	3.21	210.129	178.080	193.110
2C-T4	4.07	256.113	239.090	197.030
2C-T7	4.79	256.113	239.090	197.030
Mescaline-NBOMe	4.87	332.147	91.010	121.010
25N-NBOMe	5.93	347.119	90.990	121.000
25H-NBOMe	6.18	302.166	91.020	121.010
25C-NBOMe	6.47	336.095	91.010	121.010
25D-NBOMe	6.59	316.150	91.000	121.010
25B-NBOMe	6.70	380.051	91.010	120.990
25E-NBOMe	7.05	330.164	91.000	121.000
25I-NBOMe	7.08	428.033	90.990	120.990
25P-NBOMe	7.57	344.186	91.010	121.000

Results:

	10 ng/ml (n=5)			75 ng/ml (n=5)		
Analyte	Extraction Recovery	Matrix Effect	Extraction Efficiency	Extraction Recovery	Matrix Effect	Extraction Efficiency
2C-H	89%	23%	69%	93%	22%	73%
2C-B	105%	10%	94%	96%	7%	89%
2C-T2	98%	-6%	104%	93%	-1%	94%
2C-I	108%	10%	97%	96%	4%	92%
2C-E	107%	12%	94%	96%	4%	92%
2C-T4	104%	16%	87%	94%	3%	91%
2C-T7	103%	10%	93%	93%	3%	90%
Mescaline-NBOMe	104%	31%	72%	95%	8%	88%
25N-NBOMe	110%	24%	84%	95%	2%	93%
25H-NBOMe	103%	17%	85%	96%	3%	93%
25C-NBOMe	119%	24%	91%	96%	1%	95%
25D-NBOMe	105%	19%	86%	96%	1%	95%
25B-NBOMe	123%	29%	86%	96%	3%	93%
25E-NBOMe	115%	14%	87%	96%	4%	92%
25I-NBOMe	127%	33%	84%	95%	4%	92%
25P-NBOMe	123%	33%	83%	96%	6%	90%

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References:

[1] Poklis, J., Clay, D., & Poklis, A. (2014, February 16). 1. High Performance Liquid Chromatography with Tandem Mass Spectrometry for the Determination of Nine Hallucinogenic 25-NBOMe Designer Drugs in Urine Specimens. Journal of Analytical Toxicology, 113-121.

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