

GC-NCD-APCI-QTOF

Dual Detector in Quantitative Screening of 38 Stimulant-Type New Psychoactive Substances in Blood Without Using Reference Standards

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Materials and methods

GC with liquid autosampler



Flow splitter



Agilent 255 nitrogen chemiluminescence detector (NCD)



APCI source



Agilent 6540 QTOF mass spectrometer



Generic Sample prep

- Liquid-liquid extraction (LLE) with butyl chloride / ethyl acetate mixture (3:1)
- Primary and secondary amines were acylated with trifluoroacetic anhydride
- All evaporation steps were avoided
- 5 µl of the sample was injected to GC column (pulsed splitless)

Results and discussion

Primary amines

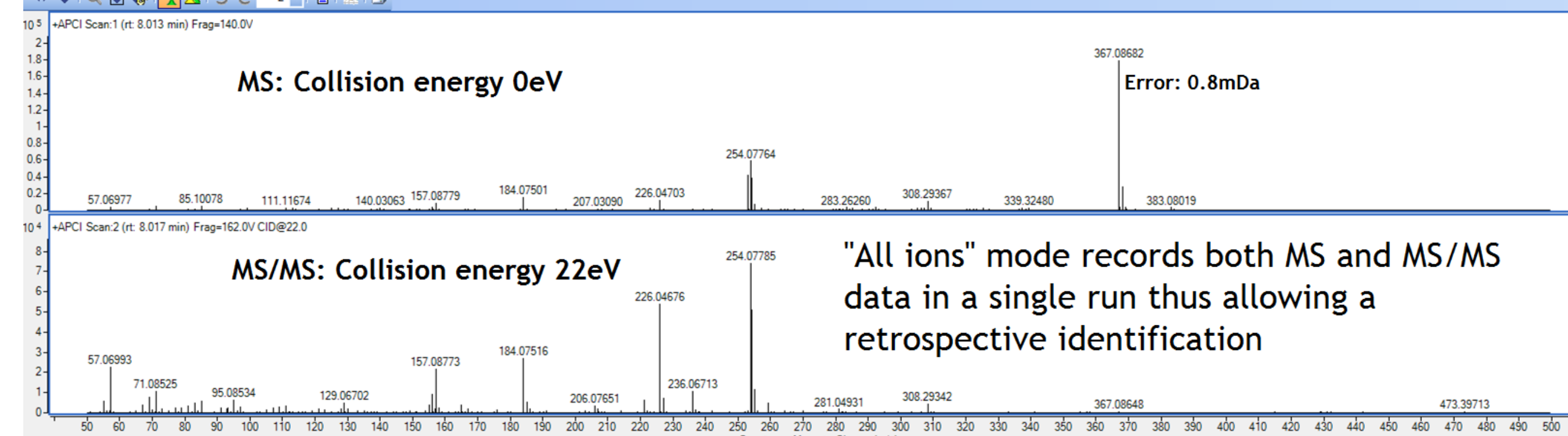
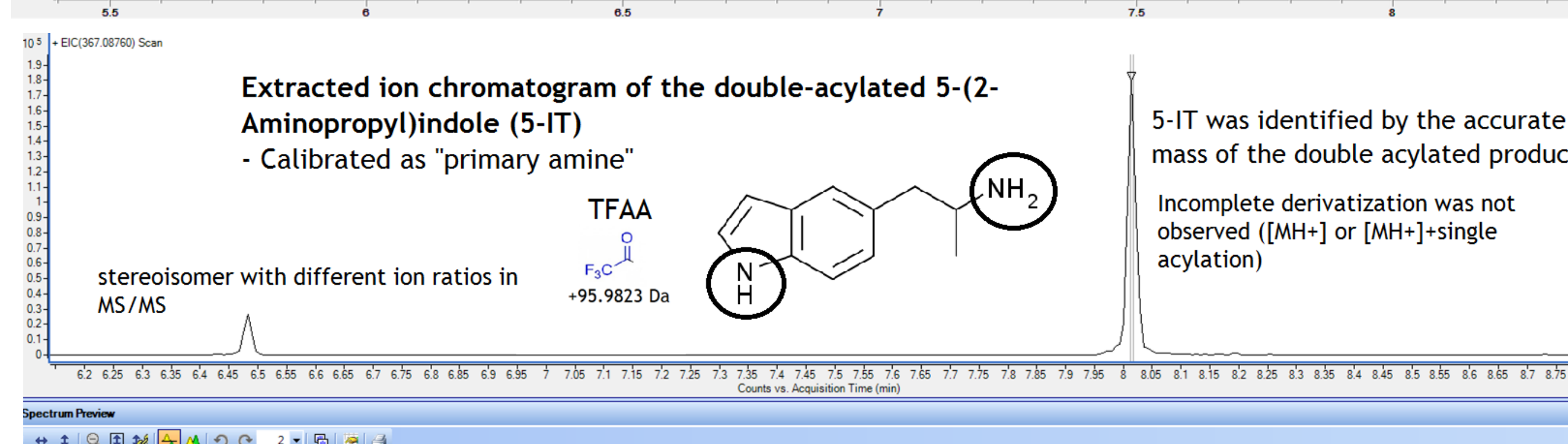
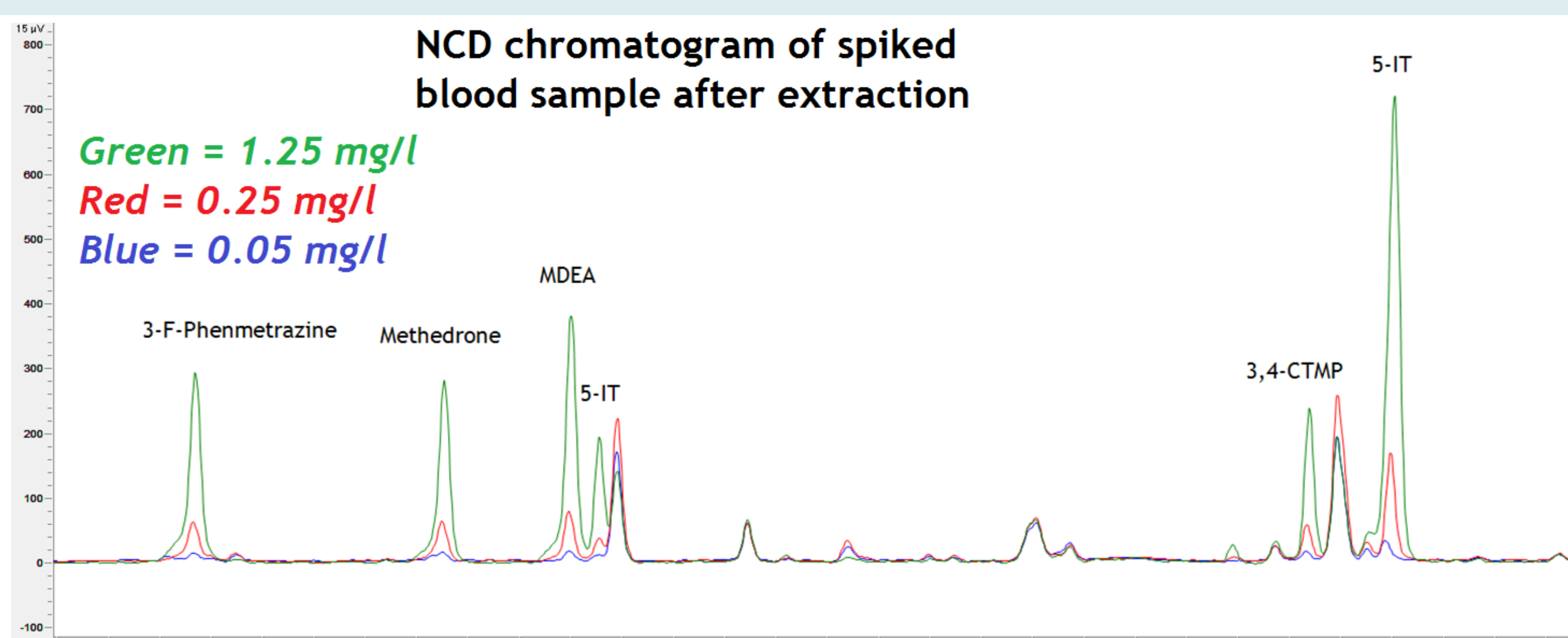
Compound	Spiked concentration					
	0.05 mg/l		0.25 mg/l		1.25 mg/l	
	Measured	Accuracy (%)	Measured	Accuracy (%)	Measured	Accuracy (%)
2C-B	0.034	68	0.174	70	1.075	86
2-PEA	0.035	70	0.183	73	0.996	80
Bromo-DragonFLY	0.038	76	0.170	68	1.065	85
MDAI	0.036	72	0.165	66	1.033	83
4-F-Amphetamine	0.046	92	0.243	97	1.211	97
4-MTA	0.053	106	0.262	105	1.170	94
4-Methylamphetamine	0.045	90	0.293	117	1.591	127
DOM	0.043	86	0.279	112	1.314	105
2C-T-2	0.034	68	0.182	73	0.927	74
DOB	0.045	90	0.240	96	1.007	81
MDA	0.032	64	0.235	94	1.119	90
2C-T-4	0.040	80	0.172	69	1.099	88
DOET	0.052	104	0.233	93	1.219	98
5-IT	0.034	68	0.285	114	1.588	127

Secondary amines

Compound	Spiked concentration					
	0.05 mg/l		0.25 mg/l		1.25 mg/l	
	Measured	Accuracy (%)	Measured	Accuracy (%)	Measured	Accuracy (%)
4-F-Methamphetamine	0.048	96	0.259	104	1.326	106
Propylamphetamine	0.055	110	0.271	108	1.417	113
2-DPMP	0.038	76	0.178	71	1.120	90
Camfetamine	0.038	76	0.255	102	1.445	116
Ethylcathinone	0.041	82	0.225	90	1.123	90
Ethylphenidate	0.044	88	0.232	93	1.281	102
m-CPP	0.032	64	0.179	72	0.961	77
Mephedrone	0.035	70	0.200	80	1.089	87
Methylphenidate	interferences		0.189	76	1.430	114
Pentdrone	0.059	118	0.261	104	1.421	114
Ethylamphetamine	0.040	80	0.248	99	1.277	102
MBDB	interferences		interferences		interferences	
Methamphetamine	0.051	102	0.291	116	1.405	112
4-MMA	0.048	96	0.263	105	1.414	113
Methiopropamine	0.061	122	0.302	121	1.562	125
3,4-CTMP	0.050	100	0.168	67	0.877	70
3-F-Phenmetrazine	0.034	68	0.210	84	1.063	85
MDEA	0.039	78	0.257	103	1.196	96
Methedrone	0.036	72	0.205	82	0.973	78

Tertiary amines

Compound	Spiked concentration					
	0.05 mg/l		0.25 mg/l		1.25 mg/l	
	Measured	Accuracy (%)	Measured	Accuracy (%)	Measured	Accuracy (%)
Dibutylone	interferences		0.239	96	1.117	89
α-PVP	0.039	78	0.234	94	1.572	126
4-F-α-PVP	0.035	70	0.325	130	1.432	115
Naphyrone	0.029	58	0.132	53	0.944	76
PCP	0.046	92	0.319	128	1.628	130



The presented instrumental combination was developed in response to lack of reference standards for NPS

NCD chromatogram may reveal new potentially harmful psychoactive compounds

Quantitative data from NPS or its metabolite can be used for toxicological assesment

Quantification and accurate mass based identification can be both done in retrospect

Quantification

- NCD's equimolar response to nitrogen allows quantification without reference standards
- Vast majority (>90%) of the new psychoactive substances (NPS) contain nitrogen
- Three external calibration standards were used to correct inaccuracies occurring in the sample preparation or GC injection
- Amphetamine for primary amines, MDMA for secondary amines and MDPV for tertiary amines
- LOQ was 0.05 mg/l