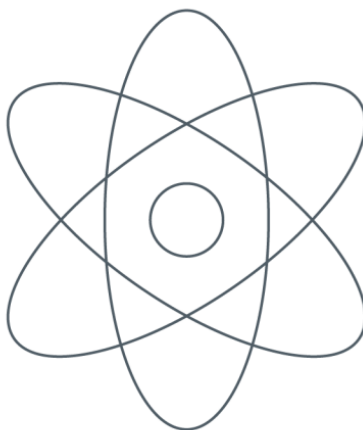


Note d'application

Ecstasy (MDMA) and Metabolites by LC-MS/MS

Waters Corporation

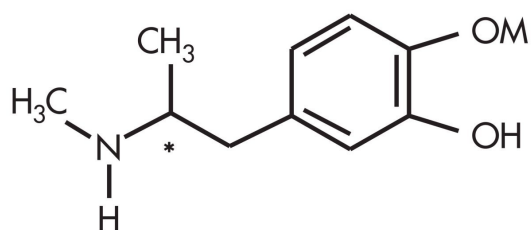


This is an Application Brief and does not contain a detailed Experimental section.

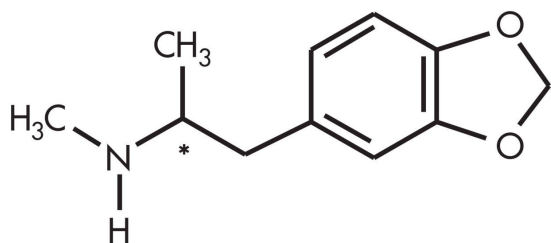
Abstract

This application brief highlights about the analysis of MDMA and its metabolites by LC-MS/MS method

Introduction



ECSTASYMETABOLITE
(HMMA)



ECSTASY (MDMA)

Experimental

HPLC Conditions

Column:	XTerra MS C ₁₈ 2.1 x 150 mm, 3.5 µm
Part number:	186000408
Mobile phase A:	20 mM NH ₄ HCO ₃ , pH 9.0
Mobile phase B:	MeOH
Flow rate:	0.2 mL/min
Isocratic mobile phase composition:	70% A; 30% B
Injection volume:	15 µL
Temperature:	30 °C
Detection:	MS ESI+
Instrument:	Alliance 2695, Micromass ZQ

MS Conditions

Instrument:	Waters/Micromass ZQ
Ion source:	Electrospray Positive
Source temperature:	150°C
Desolvation temperature:	350°C

OASIS® MCX EXTRACTION METHOD

Oasis® MCX Extraction Cartridge, 1 cc/60 mg
Part Number 186000252

CONDITION:

1 mL methanol/1 mL water

LOAD :

Prepare Sample glucuronidase, pH 5.2
8 hrs @ 37° 1 mL sample

WASH 1:

2 mL 0.1 N HCl
locks drugs by cation exchange

WASH 2:

1 mL methanol
removes acidic and neutral interferences

RE-EQUILIBRATE:

1 mL water

WASH 3:

1 mL 70:30 5% NH₄OH/methanol
removes most basic interferences

ELUTE:

2 x 1 mL methanol (10% NH₄OH)

Results and Discussion

Ions Monitored

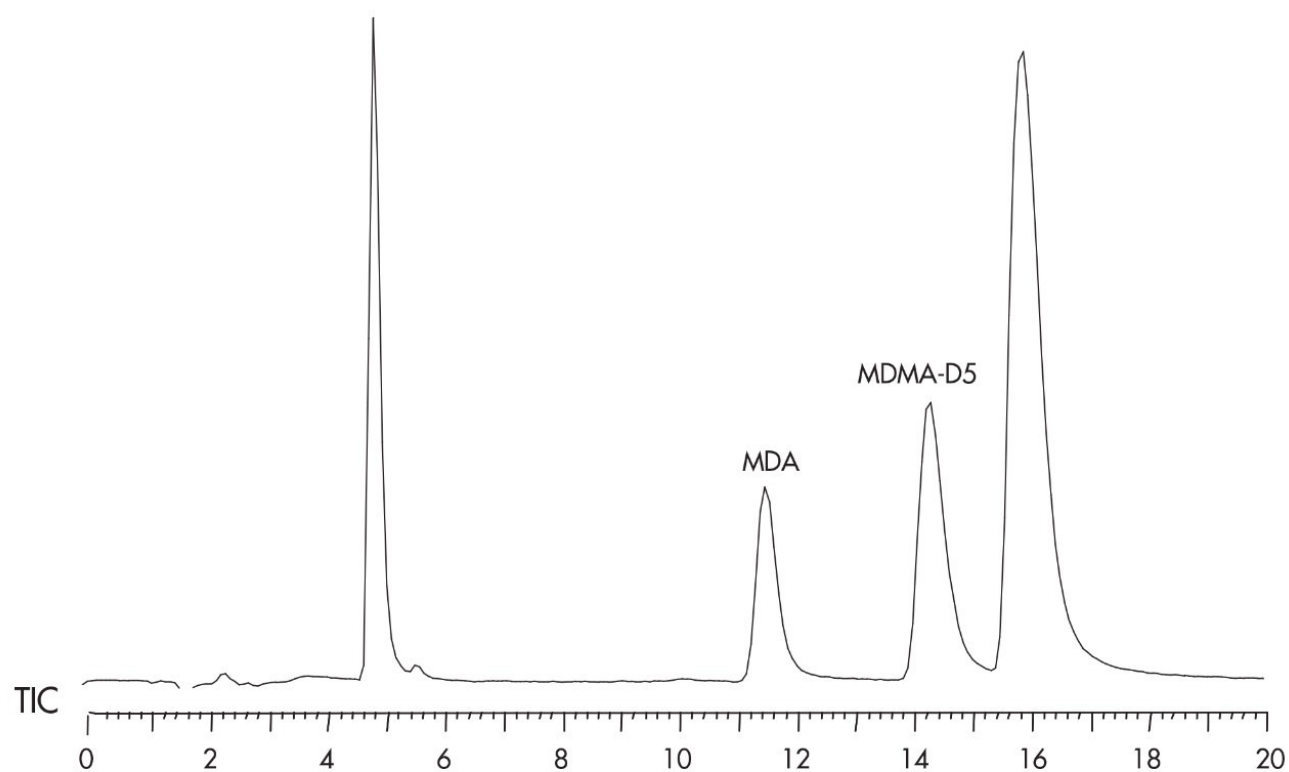
MDMA		HMMA	
Ion (<i>m/z</i>)	Cone (V)	Ion (<i>m/z</i>)	Cone (V)
194.11	25.0	196.16	20.0
163.08	37.5	165.08	37.5
135.00	55.0	137.00	55.0

MDA	
Ion (<i>m/z</i>)	Cone (V)
180.0	20.0
163.08	37.5
135.00	55.0

Analytes (LCMS)	Recovery (%)	Concentration (µg/mL)	RSD (%)
MDMA	108.0	0.10	9.8
	89.3	0.50	4.9
	88.1	1.25	4.6
	98.8	2.50	3.7
	99.9	5.00	5.7
MDA	103.0	0.10	8.8
	84.2	0.50	13.9
	83.8	1.25	9.8
	95.4	2.50	9.0
	104.5	5.00	13.4
	93.7	20.00	13.1
HMMA	90.5	0.04	8.2
	88.1	0.25	4.5
	84.8	0.50	5.4
	94.8	1.00	4.0
	100.0	2.00	5.3
	97.9	8.00	11.4

Compounds

1. 4-Hydroxy-3-methoxymetmamphetamine (HMMA).
2. 3,4-Methylenedioxyamphetamine (MDA)
3. MDMA-D5 (ISTD)
4. 3,4-Methylenedioxymethamphetamine (MDMA)



Analysis at pH 9 allows good peak shape and maximum retention for basic compounds with no modifiers that can interfere with LC-MS analysis. Do not try this with traditional silica based columns.

Featured Products

Alliance HPLC System <<https://www.waters.com/534293>>

WA31764.72, June 2003

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