



## LSD AND METABOLITES IN BLOOD, PLASMA/SERUM, URINE, TISSUE BY LC-MS/MS OR GC-MS CLEAN SCREEN<sup>®</sup> DAU EXTRACTION COLUMN

Part #

CSDAU020 – CLEAN SCREEN<sup>®</sup> DAU 200 mg, 10mL Tube

SBSTFA-1-1 – SELECTRA-SIL<sup>®</sup> BSTFA w/ 1% TMCS

SLDA100ID21-3UM – Selectra<sup>®</sup> DA HPLC Column 100 X 2.1 mm, 3  $\mu$ m

### 1. PREPARE SAMPLE:

To 1 mL of 100 mM phosphate buffer ( pH 6.0) add internal standards  
Add 1 -2 mL of blood, plasma/ serum, urine, or 1 g ( 1:4 ) tissue homogenate  
Mix/vortex and let stand for 5 minutes  
Add 2 mL of 100 mM phosphate buffer ( pH 6.0). Mix/vortex  
Sample pH should be  $6.0 \pm 0.5$ .  
Adjust pH accordingly with 100 mM monobasic or dibasic sodium phosphate.  
Centrifuge for 10 minutes at 2000 rpm and discard pellet

### 2. CONDITION CLEAN SCREEN<sup>®</sup> EXTRACTION COLUMN:

1 x 3 mL CH<sub>3</sub>OH  
1 x 3 mL D.I. H<sub>2</sub>O  
1 x 1 mL 100 mM phosphate buffer (pH 6.0)  
NOTE: Aspirate at full vacuum or pressure

### 3. APPLY SAMPLE:

Load at 1 to 2 mL/minute

### 4. WASH COLUMN:

1 x 3 mL D.I. H<sub>2</sub>O  
1 x 3 mL 100 mM acetic acid  
1 x 3 mL CH<sub>3</sub>OH  
Dry column (5 minutes at full vacuum or pressure)

### 5. ELUTE LSD AND METABOLITES:

1 x 3 mL Ethyl Acetate/ Acetonitrile: NH<sub>4</sub>OH (78: 20: 2 v/v)

or

1 x 3 mL CH<sub>2</sub>Cl<sub>2</sub>/ IPA/ NH<sub>4</sub>OH (78:20:2 v/v) Collect eluate at 1-2 mL /minute.

**NOTE:** Prepare elution solvent daily

Add IPA/ NH<sub>4</sub>OH, mix, then add CH<sub>2</sub>Cl<sub>2</sub> (pH 11-12)

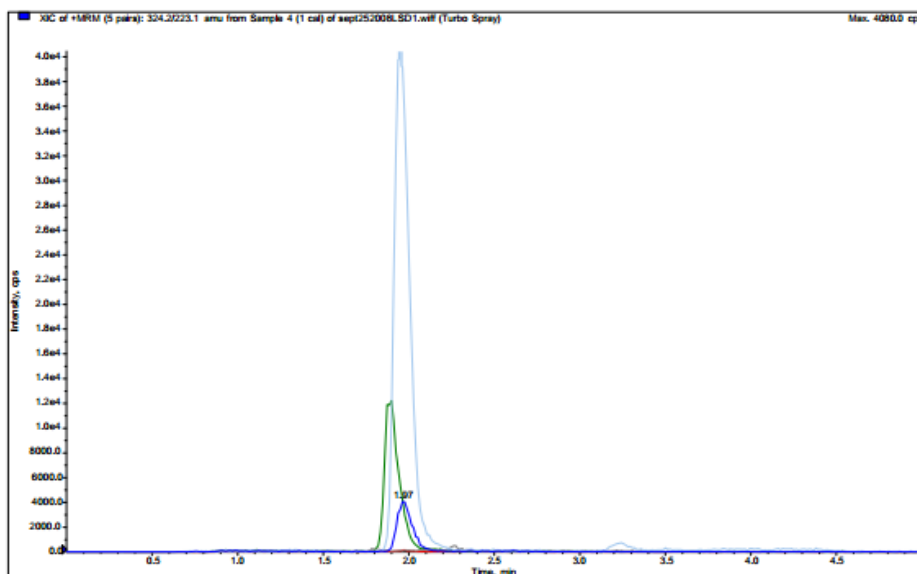
### 6. DRY ELUATE:

Evaporate to dryness at < 40 °C

### 7. RECONSTITUTE / DERIVATIZE:

- **LC-MS/MS:** Reconstitute sample in 100  $\mu$ L of mobile phase  
Inject 10  $\mu$ L.
- **GC-MS:** Dissolve residue in 50  $\mu$ L of Ethyl Acetate and 50  $\mu$ L of BSTFA w/ 1% TMCS and react at 70 °C for 30 minutes; Cool and inject 1-2  $\mu$ L

**INSTRUMENT CONDITIONS (LC-MS/MS):**  
**CHROMATOGRAM**



Analyte	MRM Transitions	
	Q1	Q3
1. LSD	324.2	223.1
2. Iso-LSD	324.2	281.1
3. Nor-LSD	310.2	209.1
4. OH-LSD	356.2	338.1
5. LSD-D <sub>3</sub>	327.2	226.1

**PARAMETERS**

**Mobile Phase A:** 0.1% Formic Acid in D.I. H<sub>2</sub>O

**Mobile Phase B:** 0.1% Formic Acid in Acetonitrile

**Flow Rate:** 0.5 mL/minute

**Polarity:** Positive

**Injection Volume:** 5 µL

**LC Column:** Selectra® DA HPLC Column 100 x 2.1 mm 3 µm

**Instrument:** API 3200 Qtrap MS/MS with Shimadzu Prominence UFLC

**Gradient:**

Time	%A	%B
0.00	30	70
3.00	90	10
3.01	30	70
5.00	30	70
5.01	STOP	

**INSTRUMENT CONDITIONS (GC-MS):**

Analyte	Quantify Ion	Qualifier Ion 1	Qualifier Ion
1. LSD-D <sub>3</sub> -TMS	298	296	271
2. LSD-TMS	395	293	268