

result report template
base on per sample

Evaluation Study for new LC-MS/MS

1. Objective

The laboratory is acquiring a new LC-MS/MS system, to replace the existing LC-MS/MS system for the analysis of benzodiazepines in blood and urine and other non-routine drugs quantitation. This write up describes the details of sample evaluation and evaluation criteria used in this study. The current parameters setting for LC-MS/MS analysis in ESI mode is also available below.

2. Items and information provided for analysis (only one sample is provided per type)

2.1 Samples for evaluation (A)

S/N	Type (Methanol)	Standard	Internal Standard
		Mixture of ST1 & ST1	IS1
		Concentration (ng/ml)	
1	Neat (NA) *please dilute to lower concentration if required	500	
2	Neat (NISA) *please dilute to lower concentration if required		500
3	Unknown UA1	To determine concentration of ST1 & ST2 in ng/ml	10
4	Unknown UA2		10
5	Unknown UA3		10

2.2 Current laboratory's conditions:

Note: Tenderers are allowed to use any chromatographic and MS conditions for evaluation, except the analysis must be performed in ESI mode, using the mobile phase provided below and injection volume must be 5 µl.

Chromatographic Conditions	
Mobile phase A	2mM ammonium formate in water
Mobile phase B	Methanol
Injection volume	5 µl
Flow rate	0.4 ml/min
Column	100 x 2.1 mm Accucore C18 (2.6µm particle size)
Column temp	40°C
LC Gradient Elution Programme	
Time (min)	% B
0.01	30
1.0	30

9.0	90		
10.0	90		
10.1	30		
12.0	30		
MS conditions			
Ionisation mode	ESI		
Polarity	Positive		
Compound	Formula	M+H	MRM product ions
ST1	C ₁₉ H ₂₈ O ₂	289.2	109.1 97.1
ST2	C ₂₀ H ₂₈ O ₂	301.2	121.1 149.15
IS1	C ₂₁ H ₂₆ O ₅	359.2	147.05 267.05

3. Item and information provided for analysis (only one sample is provided per type)

3.1 Samples for evaluation (B)

S/N	Type	Standard	Internal Standard
		Mixture of L, 25C, 25B, BM & FN	IS2& IS3
		Concentration (ng/ml)	
1	Neat (NB) *please dilute to lower concentration if required	500	
2	Neat (NISB) *please dilute to lower concentration if required		500
3	Unknown UB1	To determine concentration of L, 25C, 25B, BM & FN in ng/ml	10
4	Unknown UB2		10
5	Unknown UB3		10

3.2 Current laboratory's conditions:

Note: Tenderers are allowed to use any chromatographic and MS conditions for evaluation, except the analysis must be performed in ESI mode, using the mobile phase provided below and injection volume must be 5 µl.

Chromatographic Conditions	
Mobile phase A	2mM ammonium formate and 0.1% formic acid in water
Mobile phase B	2mM ammonium formate and 0.1% formic acid in acetonitrile

Injection volume	5 µl		
Flow rate	0.5 ml/min		
Column	100 x 2.1 mm Accucore C18 (2.6µm particle size)		
Column temp	40°C		
LC Gradient Elution Programme			
Time (min)	% B		
0.0	20		
1.0	20		
8.0	70		
9.0	99		
9.01	20		
11.0	20		
MS conditions			
Ionisation mode	ESI		
Polarity	Positive		
Compound	Formula	M+H	MRM product ions
L	C ₂₀ H ₂₅ N ₃ O	323.8	223.15 207.10
25C	C ₁₈ H ₂₂ ClNO ₃	336.0	121.15 91.10
25B	C ₁₈ H ₂₂ BrNO ₃	382.9	121.10 91.10
IS2	C ₂₄ H ₁₇ D ₅ FNO ₂	381.2	155.05 127.0
BM	C ₁₄ H ₁₀ BrN ₃ O	316.0, 318.0	182.2
FN	C ₁₆ H ₁₂ FN ₃ O ₃	314.1	268.1 239.2 183.0
IS3	C ₁₇ H ₁₂ ClF ₃ N ₂ O	353.1	241.2 222.1

4. Evaluation criteria

- The result of the sample analysis must include the following:
 - Method parameters for the chromatographic system including mobile phase, column, injection volume, column temperature, pump flow rate, LC gradient elution programme, date of analysis and etc.;
 - Method parameters for the MS conditions including MRM transitions, dwell time, voltage setting and etc.; and
 - A report template as stated in this tender, refer to section C.3 in the tender specification for more detail.
 - All results submitted have to be presented in report format as specify in section C.3 in the tender specification.

- (e) All results have to be printed on a per sample basis including all the analytes.
- To determine the concentration of ST1, ST2, L, 25C, 25B,, BM & FN in the unknowns with following criteria:
 - (a) Linear model regression
 - (b) $r \geq 0.99$ or $r^2 \geq 0.98$
 - (c) $S/N \geq 10$
 - (d) Accuracy between $\pm 20\%$ of spiked concentration
 - (e) 2 MRM transitions per analyte (1 transition for quantitation and the 1 for qualifier)
 - (f) The MRM ion ratio shall satisfy the flowing criteria when compared with the calibrator:
 - Within $\pm 20\%$ if relative intensity $> 20\%$ of base peak; or
 - Within $\pm 30\%$ if relative intensity $\leq 20\%$ of base peak
 - (g) The retention time of unknown must be $\pm 2\%$ of the calibrator
 - (h) The peak shape must be in gaussian peak or symmetrical