

Appendix E

Performance Specifications

Note: For the purpose of the following specifications, signal-to-noise is calculated as the ratio of the chromatographic peak height to twice the standard deviation of the noise.

E.1 Electrospray Positive Ion

The measured signal-to-noise ratio obtained from the chromatogram monitoring the transition m/z 609 to m/z 195 on injection of 5 pg of reserpine is $\geq 500:1$. This is based on a 5- μL injection of a 1 pg/ μL reserpine solution in 70:30 acetonitrile/water (no additives) at a flow rate of 200 $\mu\text{L}/\text{min}$ in MRM mode, 0.5 second dwell, span 0 Da.

The resolution of the precursor and product ions is <1 Da peak width at half-height.

E.2 Electrospray Negative Ion

The measured signal-to-noise ratio obtained from the chromatogram monitoring the transition m/z 503 to m/z 179 on injection of 25 pg of raffinose is $\geq 50:1$. This is based on a 5- μL injection of a 5 pg/ μL raffinose solution in 70:30 acetonitrile/water (no additives) at a flow rate of 200 $\mu\text{L}/\text{min}$ in MRM mode, 0.5 second dwell, span 0 Da.

The resolution of the precursor and product ions is <1 Da peak width at half-height.

E.3 MS Resolution

The resolution is demonstrated using a 1 $\mu\text{g}/\mu\text{L}$ solution of PPG 2000 in 50:50 acetonitrile/water containing 1-mM ammonium acetate. The peaks at m/z 2009.5 and 2010.5 should be resolved with a valley between them of no more than 15% of the height of the 2010.5 peak. It is recommended that fifteen 1-second scans are summed and the resulting spectrum smoothed (two passes, 0.5 Da SG).

E.4 Mass Measurement Accuracy

The mass measurement accuracy is measured from the mean of five repeat analyses of the $[M+NH_4]^+$ peak at m/z 1004.622 from 1 $\mu\text{g}/\mu\text{L}$ PEG 1000 in 50:50 acetonitrile/water containing 2 mM ammonium acetate. The mean measured mass will be 1004.622 \pm 0.05 Da. The standard deviation of the mean will be \leq 0.05 Da.

A mass calibration will be performed using the $[M+H]^+$ peaks from a separate analysis over the mass range m/z 700 to 1300 and the resolution on the $[M+H]^+$ peak at m/z 1031.62 must be between 0.3 and 0.4 Da wide at half height after smoothing.

E.5 APCI Positive Ion

Measured signal-to-noise ratio obtained from the chromatogram monitoring the transition m/z 331.2 to m/z 109.1 on injection of 50 pg of 17- α -hydroxyprogesterone will be \geq 70:1, using a 5 μL injection of a 10 pg/ μL 17- α -hydroxyprogesterone solution in 70:30 acetonitrile/water (no additives) at a flow rate of 1 mL/min in MRM mode, 0.2 second dwell, span 0 Da.

The resolution of the precursor and product ions will be <1 Da peak width at half height.