**Record of Technical Anomaly**

**Technical Anomaly No: 419**

**PART 1: Issue, impact assessment and signing open**

**Raised by: Hannah Ford** **Date: 13/01/22**

**OP / Method: 224**

**Analytical sequence(s) affected** *(where appropriate)***: EC0789**

**Details** *(please tick relevant box(es) and provide supplementary information where required)***:**

□ QC point(s) above/below ±2SD □ Bracketing standard(s) outside limits

□ QC point(s) above/below ±3SD □ QC Recovery outside limits

□ QC point(s) outside expanded uncertainty R Other *(add details below)*

Interfering peak with area >400 in cal 0 (area 813). Attempted corrective action by tightening column nut to eliminate potential leak and re-ran cal 0 – interfering peak still present, area 968. Peaks also present in conditioning blank 70% injections (freshly prepared 70%) areas ranging from 765-1545. Also present in blank 70% injections throughout run, although area did drop off slightly.

Chart, line chart

Description automatically generated

Chart, line chart

Description automatically generated

Interference does not appear to have had significant impact on calibration – residuals for lowest 2 cal stds 2.5 and 5ppm were +1.4% and +1.6% respectively. Interfering peak area is (813/2466=) 33.0% of 2.5ppb cal area and (813/3999=) 20.3% of 5ppb cal area.



As 20.3% is a more tolerable proportion of interference than 33.0%, the reporting limit for this run was adjusted to 5ppb. All calibration levels were included in the curve as all residuals had passed.

It must be noted that MS response on this run was observed to be high in comparison to recent previous runs – abundance on tune was >31,000,000 compared to ~17,000,000 for run “EC0787 rerun” and internal standard peak area was between 27,000 – 33,000 compared to around 15,000 for “EC0787 rerun”. This high response, while beneficial to analyte sensitivity, would also have the undesirable effect of inflating interfering peaks as well as analyte peaks.

Some more data is to be gathered to determine if the acceptable limit for interference should be revised to a proportion of either 2.5ppb cal std area or internal standard area in the cal 0 injection, rather than an exact/fixed area limit which would not account for fluctuations in MS response.

□ No apparent reason for this anomaly

**Recommended Action:**

□ No action required out with the usual close monitoring of the Quality Control data in subsequent runs.

A thorough leak investigation to be performed before next run, with particular focus on seal between column and push-fit liner (a common source of leaks).

**Explanation why the issue does not impact data quality & why it isn’t a departure:**

Interfering peak has not impacted calibration or caused any quality control failures, and all results on run were much higher than the level at which interference may be suspected to impact results. The lowest reportable result on this run was 21.53ppb (sample S21-0931 @11.09ppb was rerun to check a discrepancy in training data).

**Management Review:**

***I agree to open this Technical Anomaly and confirm that this would not prevent results from being reported.***

**Authorised by:**

**(Technical/Services/Quality Manager)**

**Date:**

**PART 2: Follow-up actions and close-out**

**Follow-up actions conducted:**

□ No follow up required

**Signed off & closed by Quality Manager:**

**Date: \_\_\_\_\_\_\_\_\_\_\_\_**

