Step No:	Activity	Remarks/Notes	Team Members
	passed. If the values lie outside the upper and lower control limits then QCs are failed. **If QCs have failed then the samples which the QCs bracket must be re-run.** Colour code pass values in green, fail values in red. If values lie outside the action limits ie mean ± 2SD then QCs, but within the control limits then the system must be investigated as to why the QCs are moving out of control and appropriate action taken. The control charts should be updated with the new QC data following completion of the summary sheet.	Inform chemistry manager and record on shift handover. Additional rules for these control charts.. 7 Consecutive points the same side of the mean represent a case where investigation should take place. Investigation should include but not be limited to checking expiry dates of solutions, evidence of deviation from existing procedure, inappropriate training of analyst, contamination. Any remedial action taken should be recorded by an e-mail to the whole chemistry team. This e-mail should also be stuck into the ICP logbook. Failed QCs should not be used to update the control charts.	
9.03	Copy and paste the following values: BEC (to nearest integer)		
9.04	Final sample value: If : Average sample value is > BEC and %RSD is < 10 %, then quote average. Average sample value is > BEC and %RSD is > 10.0 %, then consult Appendix F for wider %RSD	The sample may need to be re-run depending on what the data will be used for. If this is an FMDT tank "A" sample must be rerun. **NR is not an acceptable output.** **In the case of Al,** then NR will suffice as there is no	

Step No:	Activity	Remarks/Notes	Team Members
	limits in the case where the average is small compared to the discharge limits. which states state NR (No result) If Average value is < BEC, then quote < BEC. **For FMDT and sentencing tank samples,** **If BEC > discharge limit then the entire run needs to be repeated.** Highlight in green all pass values. Environmental discharge limits are pass/fail criteria for FMDT A samples. Environment discharge limits are found in BRAD/SI/ENV/002 Appendix K.	discharge limit for Al. there is no need to repeat a run just to obtain a value for Al. The value for concentration quoted in this case would be found in Appendix F.	
9.05	Report sheet should be completed (See Appendix C) to include analyst and QC sample references. Both the analyst and checker should sign and date the completed document. This hard copy will be attached to the rest of the sample standard request form (BRAD/22429/FORM/00122) and processed according to Sampling Procedure and Management (BRAD/22405/OI/0166 8.05) The checked electronic version should be saved in:	Note: If a series of QC has consistently failed, these should not be updated without consultation with the laboratory manager. Failure of the QCs must be investigated first.	**Lab analyst and checker**

Step No:	Activity	Remarks/Notes	Team Members
	Y:\readona\FED Project\FED Operations Project File Dv 22429\5-Environmental Waste Radiological Protection\5.1-Environmental\Lab Results\ICP RAW DATA In the appropriate ICP folder. The control charts must be updated with the new QC data following completion of the summary sheet.		
Sub-task 10: Check calculation and data.			
10.1	The data and calculations generated in Sections 8 & 9 **must** be checked by a second analyst to confirm the validity of the data. Detailed instructions are described in Appendix G.	Before embarking on an extensive checking exercise, the checker should just carry out some preliminary examination of the raw data eg intensities, concentrations tab to ensure that calibration lines, QCs etc look reasonable.	
10.2	If average value of sample or RSD values are unexpectedly high or low check for any transcription errors. If no errors can be found, then these values can be calculated manually to confirm the software results (see Appendix H).	Transcription errors include copying the wrong data from the worksheet, pasting values into wrong cells etc.	

Section 2: REFERENCE DOCUMENTS

Document	Revision No.	Title
Doc Ref.	Rev No.	Document Title.
BRAD/22429/OI/0101		Preparation of ICP-MS standards
BRAD/22429/OI/0102		ICP-MS Daily Performance Checks
BRAD/0311/2014		COSHH assessment
BRAD/22429/RPT/00133		Chemical Risk Assessment for Pregnant Female Chemist working at Bradwell

Appendix A:Cleaning of glassware.

Cleaning of General Plasticware for ICP-MS

Flasks should be left overnight with 10%HNO_3 solution, bulked to volume with deionized water. Label the flask stating that it contains 10% HNO_3. For use the next day, pour away this solution and bulk the flask with deionized water, shake with a stopper and then poured away. This rinsing process should be carried out three times before use.

Cleaning of Mercury Glassware for ICP-MS

Flasks should be left overnight with 10%HCl solution, bulked to volume with deionized water. Label the flask stating that it contains 10%HCl. For use the next day, pour away this solution and bulk the flask with deionized water, shake with a stopper and then poured away. This rinsing process should be carried out three times before use.

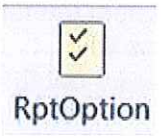
Cleaning of General Plasticware/Glassware e.g. beakers

General plasticware/glassware such as beakers and cylinders should be cleaned using 10%HNO_3 in distilled water. Once cleaned with 10% HNO_3 solution, the plasticware must be rinsed three times with distilled water thoroughly.

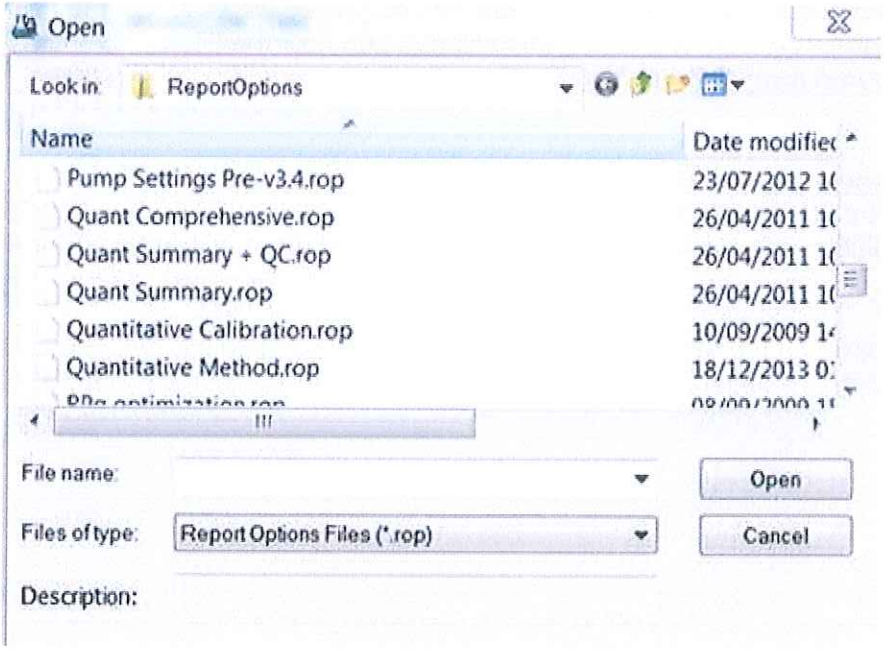
Any glassware/plasticware which is required for the sample preparation for mercury should be cleaned using 10%HCl solution. Once cleaned with 10%HCl solution, the glassware must be rinsed three times with distilled water thoroughly

Appendix B: Generating the calibration report

1. Open the report from the report options button



2. Select it from the list and open

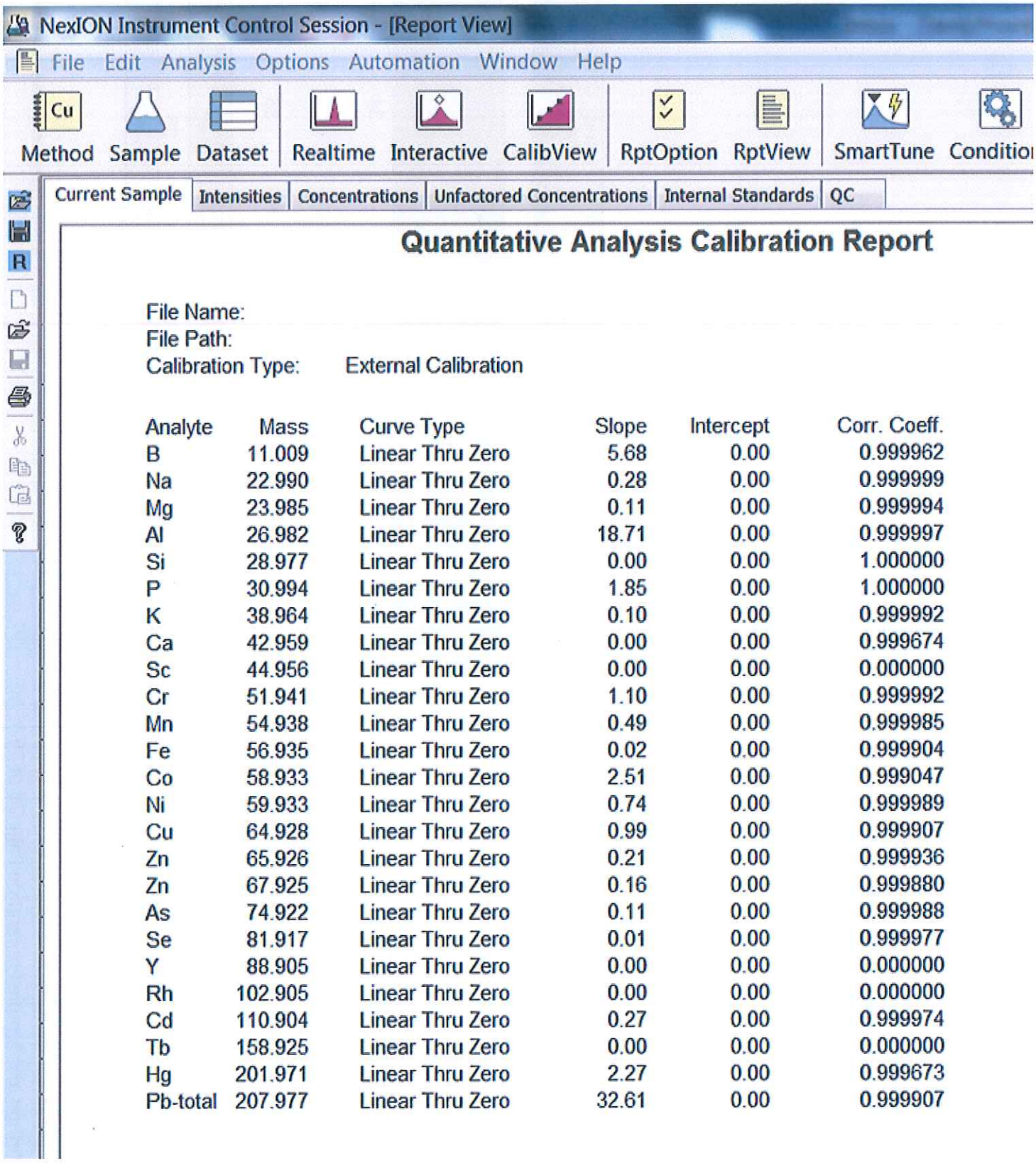


3. Open the dataset which contains the calibration you want to view

4. Highlight a line below the last calibrant

5. From the dropdown list in the dataset load the calibration

6. Click on report view and select the 'current sample' view tab



Quantitative Analysis Calibration Report

File Name:
File Path:
Calibration Type: External Calibration

Analyte	Mass	Curve Type	Slope	Intercept	Corr. Coeff.
B	11.009	Linear Thru Zero	5.68	0.00	0.999962
Na	22.990	Linear Thru Zero	0.28	0.00	0.999999
Mg	23.985	Linear Thru Zero	0.11	0.00	0.999994
Al	26.982	Linear Thru Zero	18.71	0.00	0.999997
Si	28.977	Linear Thru Zero	0.00	0.00	1.000000
P	30.994	Linear Thru Zero	1.85	0.00	1.000000
K	38.964	Linear Thru Zero	0.10	0.00	0.999992
Ca	42.959	Linear Thru Zero	0.00	0.00	0.999674
Sc	44.956	Linear Thru Zero	0.00	0.00	0.000000
Cr	51.941	Linear Thru Zero	1.10	0.00	0.999992
Mn	54.938	Linear Thru Zero	0.49	0.00	0.999985
Fe	56.935	Linear Thru Zero	0.02	0.00	0.999904
Co	58.933	Linear Thru Zero	2.51	0.00	0.999047
Ni	59.933	Linear Thru Zero	0.74	0.00	0.999989
Cu	64.928	Linear Thru Zero	0.99	0.00	0.999907
Zn	65.926	Linear Thru Zero	0.21	0.00	0.999936
Zn	67.925	Linear Thru Zero	0.16	0.00	0.999880
As	74.922	Linear Thru Zero	0.11	0.00	0.999988
Se	81.917	Linear Thru Zero	0.01	0.00	0.999977
Y	88.905	Linear Thru Zero	0.00	0.00	0.000000
Rh	102.905	Linear Thru Zero	0.00	0.00	0.000000
Cd	110.904	Linear Thru Zero	0.27	0.00	0.999974
Tb	158.925	Linear Thru Zero	0.00	0.00	0.000000
Hg	201.971	Linear Thru Zero	2.27	0.00	0.999673
Pb-total	207.977	Linear Thru Zero	32.61	0.00	0.999907

7. Print or save the report output

Appendix C: Example of completed report sheet

ICP-MS report sheet
A0734-sentencing
Tank
150224 ICP 1 A0734
De
MES 02
PEQC 04
DE

Pass
Fail

	Sample Id	Acquisition Time	B 11 (ppb)	Al 27 (ppb)	Cr 52 (ppb)	Fe 56 (ppb)	Ni 60 (ppb)	Cu 63 (ppb)	Zn 66 (ppb)	Cd 111 (ppb)	Pb 208 (ppb)
		R2 (4 dp)	0.9978	0.9978	0.9988	0.9984	0.9986	0.9976	0.9981	0.9987	0.9985
		QCs		10.8	10.3	10.5	10.4	9.6	10.9	10.8	10.1
				10.7	10.5	10.9	10.2	9.6	11.0	10.8	10.2
				11.0	10.3	10.7	10.2	9.4	10.9	10.8	10.1
				10.6	10.1	11.0	10.0	9.2	11.0	10.9	10.0
	Average value of sample		55	305	5	516	1	-1	34	0	0
		% RSD	30.0	22.9	1.3	19.5	99.4	-111.7	15.9	35.8	9.6
		BEC (main metals)	1136	320	13	479	8	40	86	2	7

		B 11 (ppb)	Al 27 (ppb)	Cr 52 (ppb)	Fe 56 (ppb)	Ni 60 (ppb)	Cu 63 (ppb)	Zn 66 (ppb)	Cd 111 (ppb)	Pb 208 (ppb)
	Final value (ppb)	< 1136	<320	<13	516	< 8	< 40	< 86	< 2	< 7
	Discharge limits(ppb)	**2,334,150**	n/a	**200**	**333,450**	**3,000**	**1,667**	**13,338**	**30**	**1,080**

Daily Performance PASS FAIL

R2	PASS	FAIL
QC	PASS	FAIL
RSD	PASS	FAIL
EA Limits	PASS	FAIL

Fe 56 has a large RSD at 20%. However the value of the replicates 444 and 587 as 4 orders of magnitude below the discharge limit.

Analyst

Checker

Appendix D: How to complete QC charts

How to complete QC charts

These should be completed on a regular basis, ideally after new validated QC values have been generated. Updating is important as it will help to identify any trends that are occurring in the system and allow preventative action to be taken in time.

The individual control charts for each element are stored in:

Y:\Decommissioning\FED Programme Working Area\FED & ADAP Commissioning Working Area\Labs\ICP External Calibration\Control charts

The control charts for each element are divided by ICP.

- From the verified QC data cut and paste the values for in the corresponding columns.

1. Date and time
2. QC standard
3. Machine
4. Analyst (who prepared standards and ran ICP)

- Under value column paste the mean value for each QC.
- The software should automatically calculate in the following columns:
  sample mean,
  standard deviation,
  sample standard deviation,
  lower control limit, (mean - 3 SD)
  upper control limit, (mean +3 SD)
  lower action limit (mean - 2 SD)
  upper action limit. (mean +2 SD)
- Check also that the graph has been updated with the values that have just been input.
- If the chart has not automatically updated it may be that the range to calculate the mean standard deviation, etc has not changed. Correct the values accordingly.
- Where a new QC standard is introduced, a vertical line should be put on the chart to indicate the transition between QCs. To help identify points, by hovering the cursor over a point this will bring up a label stating point number and value.

Appendix E: Daily Performance Checks

ICP-MS Daily Performance Checks

Performance checks are carried out to ensure the instrumentation is functioning correctly on the day it is used and to monitor day to day trends in performance. The latter can pick up developing problems and help prevent catastrophic failure. The data for each instrument must be recoded separately. Performance is assessed by looking at sensitivity across the mass range, levels of refractory oxides, levels of doubly charged ions, background and the reproducibility of the analyte signal.
To proceed with analysis ALL tests must PASS with the exception of 'Mg signal' which may occasionally fail due to Mg contamination in the system.

Procedure

1.1 Run the daily performance check routine using the 'Daily Set up solution'

1.2 Copy and paste the summary figures into the Daily Performance Spreadsheet.

1.3 Transcribe the poorest figure for analyte precision into column L (Poorest %RSD on analyte)

1.4 Do all tests PASS (with the possible exception of Mg)?

1.5 If YES – print out report and place in file. Proceed with analysis

1.6 If NO – follow the following sequence

1.6.1 **Re-run performance check solution**. If it passes this time proceed as above (1.5). If not go to 1.6.2

1.6.2 **Perform SmartTune**. If it passes this time proceed as above (1.5). If not go to 1.6.3

1.6.3 **Change pump tubing**. Re-run performance check solution. If it passes this time proceed as above (1.5). If not go to 1.6.4

1.6.4 **Prepare new solution**. If it passes this time proceed as above (1.5). If not go to 1.6.5

1.6.5 Consult with laboratory manager! There is a serious problem.

APPENDIX F: Reporting Criteria for %RSD greater than 10%

		Less than			Less than			Less than		
	Limit (ppb)	Average (ppb)	%RS	Report (ppb)	Average (ppb)	%RS	Report (ppb)	Average (ppb)	%RS	Report
B	53,235,000	5,000,000	70	<15,600,660	10,000,000	50	<24,900,00	15,000,000	30	<28,570,00
Cd	30	3	70	<9.3	6	50	<14.5	9	30	<18
Cr	4563	400	70	<1248	800	50	<1988	1200	30	<2260
Cu	38025	3800	70	<11436	8000	50	<19879	12000	30	<22600
Fe	7,605,000	760000	70	<2,372,203	1,500,000	50	<3,621,320	2,400,00	30	<4,521,320
Pb	1080	108	70	<337	200	50	<497	300	30	<554
Hg	7.5	1	70	<3.1	2	50	<5	3	30	<5.7
Ni	3000	300	70	<930	600	50	<1449	900	30	<1750
Zn	304200	30000	70	<93000	60000	50	<144900	90000	30	<175000
Al	None		>10	NR						

For sentencing tank and FMDT samples, if %RSD is greater than 10 % and average is above the values show in this table, the sample must be re-run.

This table details the values that should be reported for each element in the case that %RSD is greater than 10 %.

The reporting figures are based on the average + 3 standard deviations.

Example of how to use this Appendix:
For example if Ni average value = 18 ppb , % RSD = 30%, BEC = 9 ppb
Zn average value = 2390, % RSD = 65%. BEC = 118 ppb

Reading from left to right. For Ni the average 18 is less than 300, so you are allowed to have a tolerance of up to 70% RSD. In this case you quote the value from the table <930 ppb
For Zn the average 2390 is less than 30,000, so you are allowed to have a tolerance of up to 70% (you have 65%). So you can quote <93000 ppb directly from the table.

APPENDIX G:DATA CHECKING

Note: The role of the checker is to confirm that the data generated is correct, not repeat all the calculations from scratch. If at any point, a fundamental error is found, then the spreadsheet should be returned to the originating analyst to correct. If this is not possible, then the checker should perform the corrections. The checker now becomes the originating analyst and a different checker must be found. Inform the team leader of any delays that this may cause.

1. The password for the spreadsheets is " DavidPrice".
2. The name of QCs and calibration standard used should be recorded. For list of standards see: Y:\Decommissioning\FED Programme Working Area\FED & ADAP Commissioning Working Area\Labs\ICP External Calibration\Calibration spreadsheets\ICP Standards Log.xlsx
3. The analyst(s) who ran the ICP and the analyst who carried out the calculations should be recorded.
4. Check that the correct spreadsheet is being analysed ie date and the correct sample number.
5. Check that 2 IS correction factor columns have been inserted, one for Sc45 and one for Tb159. The correct formulas have been used for the 2 IS correction factors eg = K$7/K8, K$7/K9, etc down each column.
6. IS corrected counts =Initial value * IS correction factor
7. Sc 45 column has same value.
8. TB 159 column has same value.
9. Examine the choice of excluded blanks, which should be highlighted. Discard the first 4 blanks which are typically the highest values.. As long as the choice of data removed seems reasonable ie the remaining blanks are the lower values you can proceed
10. Check average blank correct formula EG " =average(D93:D99)"
11. Check SD. Correct formula. Eg "stdev(D93:D99)"
12. Blank corrected . Subtract average blank from each value. For each element subtract should always be from the same cell.
13. Copy the values of calibration standards. Check that the correct values are put in (ppb)
    For general metals: 0, 1, 2, 5, 10, 20.
    Quick Check values are increasing, eg 2 x 1 should be 2
    2x 5 should be 10
    2 x10 should be 20.

14. R2 should be calculated using the formula: "=RSQ($C114: $C119, D114: D119)." the concentration values should always be fixed hence $ sign.
15. R2 values must be > 0.995 and < 1.0.
16. No more than 1 point should be removed from calibration curve. **If R2 is still < 0.994 the calibration must be re-run.**
17. Slope should be calculated using the formula "=slope($C114: $C119, D114: D119)." Value must be < 1.00.
18. Dilution corrected concentrations.

When using the software:

Go onto the concentrations tab. Check that the correct sample reps (reps 3 & 4) have been averaged and copied over to the summary sheet.

Go onto the concentrations RSD tab. Check that the correct sample reps (reps 3 & 4) have been averaged and copied over to the summary sheet.

When calculating manually:

Blank corrected (from 9) should be multiplied by slope. Ensure that the correct dilution factor is being used:

Sample	Dilution factor
Pre-neut	X 2500
Post-GAC	X 402
Supernatant, Sentencing Tank, FMDT	X 402
NOx scrubber	X 1616

Discard 1st and 2nd replicate, only use 3rd and 4th replicate for average and SD.

" =average(D103:D104)"

" = stdev(D103:D104)"

To calculate % RSD =100 *SD/mean.

In general metals use BEC = slope x average blank

19. QC values:

When using the software:

Go onto the concentrations tab. The QC values should be copied directly from here onto the summary sheet. No further calculations are necessary.

When calculating manually:

QC = blank corrected counts x slope.

Values should fall in the region of 10.0. Check control charts of each element for up to date values. The specification limits are the lowest and highest control limit.

Y:\Decommissioning\FED Programme Working Area\FED & ADAP Commissioning Working Area\Labs\ICP External Calibration\Control charts

If QC has failed then sample needs to be re-run. Report NR. (No result)

20. Check that the correct values of R2, QC, sample average, BEC, %RSD have been pasted to the summary sheet.

21. Highlight green for pass values, red for fail.

    If Average value is < BEC, then quote < BEC.

    If %RSD > 10.0%, sample requires a re-run unless the element is Al which is run for information only, there is no discharge limit for Al.

    In the case where %RSD > 10%, consult Appendix F for reporting criteria for %RSD greater than 10%.

    In these qualifying cases you should quote the value in the table which is calculated as a value of average + 3 std. deviations. Otherwise NR (no result) must be quoted.

22. Ensure the columns are shaded accordingly:

Daily calibration check	PASS	FAIL
R2	PASS	FAIL
QC	PASS	FAIL
RSD	PASS	FAIL

EA limits	PASS	FAIL

Put name of analyst who carried out calibration check.

23. If the data check is OK, sign and date in the appropriate box.

APPENDIX H: MANUALLY CALCULATING DATA

Calculating dilution corrected concentrations:

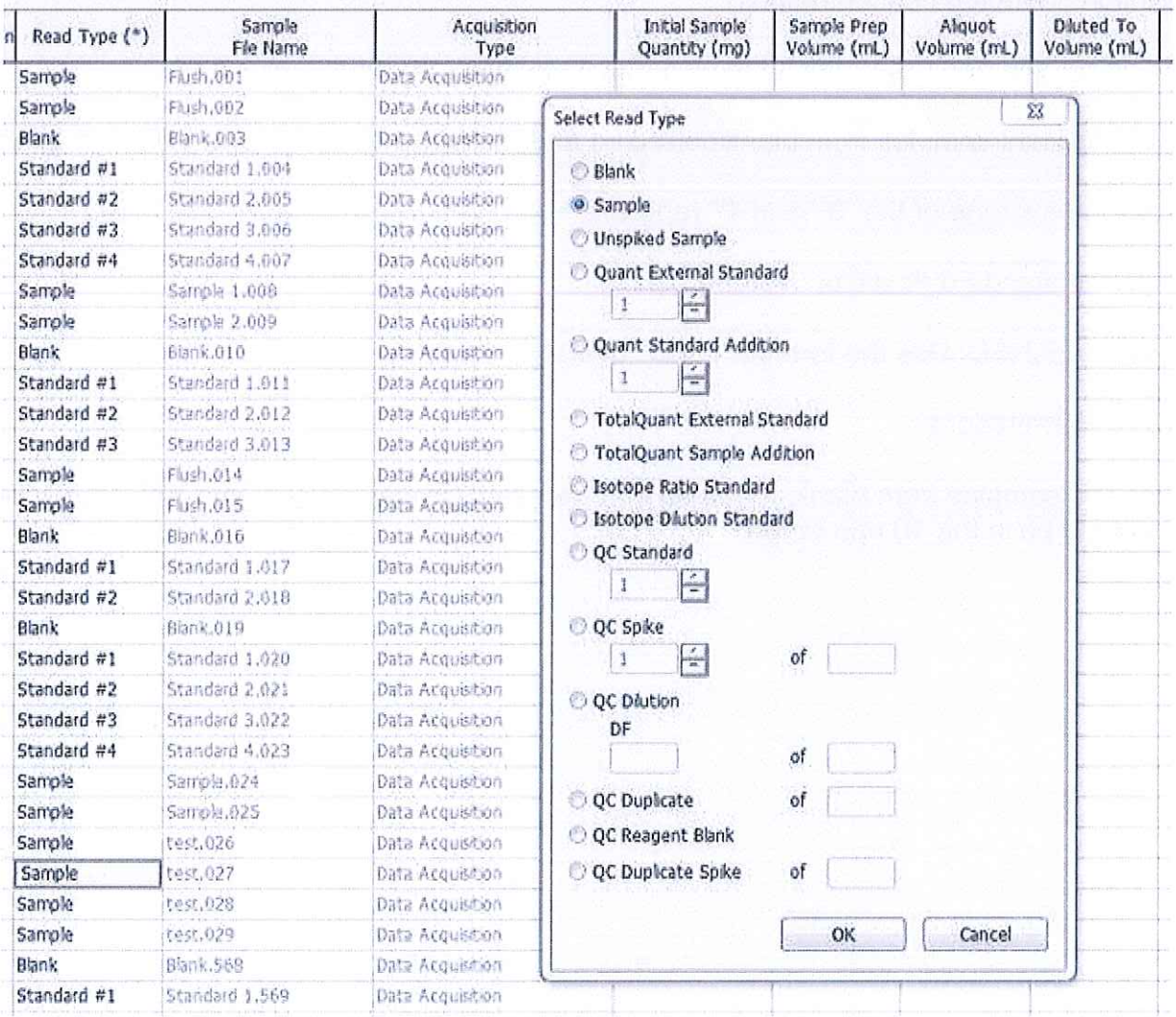
1) Include all sample and QC data on the worksheet and work through 8.04 – 8.10.
2) Copy the replicate samples from blank corrected and multiply the cps x slope x dilution factor
3) Calculate the average of the 3rd and 4th replicate
4) Calculate the standard deviation: =stdev( ).
5) Calculate the %RSD: Use the formula = 100* average( )/stdev( )

Calculating QC concentrations:

1) Copy the QC samples from blank corrected and calculate concentrations: cps x slope. These should be in the 10 ppb range.

APPENDIX I: Changing calibration blank

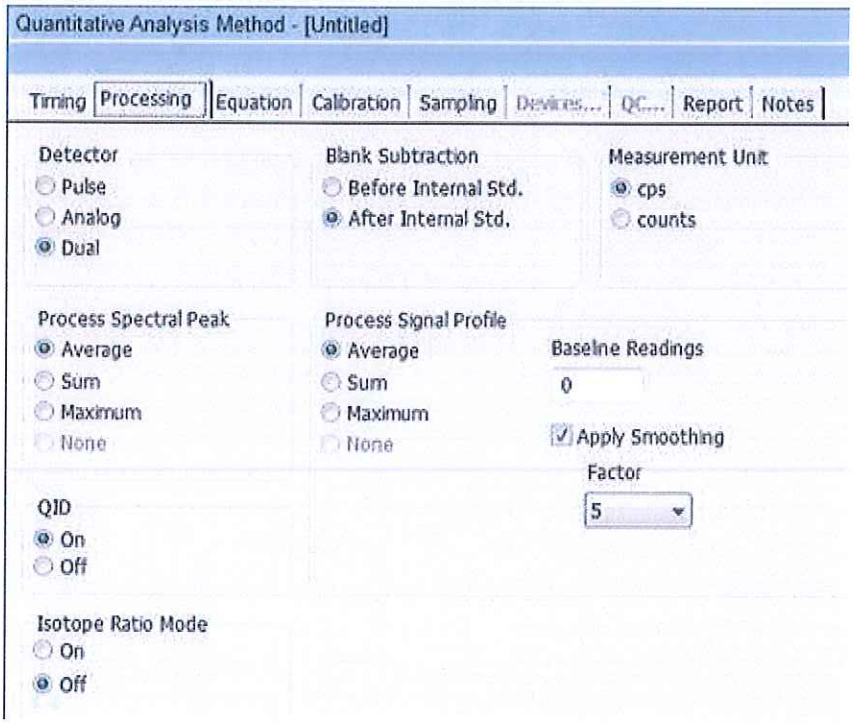
You can choose a different one in the dataset by changing 'sample' to 'blank'.



n	Read Type (*)	Sample File Name	Acquisition Type	Initial Sample Quantity (mg)	Sample Prep Volume (mL)	Aliquot Volume (mL)	Diluted To Volume (mL)
	Sample	Flush.001	Data Acquisition				
	Sample	Flush.002	Data Acquisition				
	Blank	Blank.003	Data Acquisition				
	Standard #1	Standard 1.004	Data Acquisition				
	Standard #2	Standard 2.005	Data Acquisition				
	Standard #3	Standard 3.006	Data Acquisition				
	Standard #4	Standard 4.007	Data Acquisition				
	Sample	Sample 1.008	Data Acquisition				
	Sample	Sample 2.009	Data Acquisition				
	Blank	Blank.010	Data Acquisition				
	Standard #1	Standard 1.011	Data Acquisition				
	Standard #2	Standard 2.012	Data Acquisition				
	Standard #3	Standard 3.013	Data Acquisition				
	Sample	Flush.014	Data Acquisition				
	Sample	Flush.015	Data Acquisition				
	Blank	Blank.016	Data Acquisition				
	Standard #1	Standard 1.017	Data Acquisition				
	Standard #2	Standard 2.018	Data Acquisition				
	Blank	Blank.019	Data Acquisition				
	Standard #1	Standard 1.020	Data Acquisition				
	Standard #2	Standard 2.021	Data Acquisition				
	Standard #3	Standard 3.022	Data Acquisition				
	Standard #4	Standard 4.023	Data Acquisition				
	Sample	Sample.024	Data Acquisition				
	Sample	Sample.025	Data Acquisition				
	Sample	test.026	Data Acquisition				
	Sample	test.027	Data Acquisition				
	Sample	test.028	Data Acquisition				
	Sample	test.029	Data Acquisition				
	Blank	Blank.568	Data Acquisition				
	Standard #1	Standard 1.569	Data Acquisition				

Clear the current blank and calibration before reprocessing. .

On the concentration data for the QCs the difference is likely to be due to the blank cps correction. It's an option in the method. You can subtract after internal standard correction.


Quantitative Analysis Method - [Untitled]
Timing
Processing
Equation
Calibration
Sampling
Devices...
QC...
Report
Notes
Detector
Pulse
Analog
Dual
Blank Subtraction
Before Internal Std.
After Internal Std.
Measurement Unit
cps
counts
Process Spectral Peak
Average
Sum
Maximum
None
Process Signal Profile
Average
Sum
Maximum
None
Baseline Readings
0
Apply Smoothing
Factor
5
QID
On
Off
Isotope Ratio Mode
On
Off