Magnox

Bradwell Site

SYSTEM: FED/ADAP Laboratory 185

PLANT CLASS: 2	PLANT CODE: 1000454	QA Grade: 2

JOB TITLE: New Main metals ICP-MS Method and Sample Preparation

THIS INSTRUCTION IS INTENDED FOR: (Delete as appropriate)	~~IMPLEMENTATION~~	REFERENCE USE
	~~CONTINUOUS USE~~	~~INFORMATION USE~~

RESPONSIBILITIES:

Minimum Staffing: 2 lab technicians.

Special Training: ICP-MS training

Verification (Post Holders): NRE

Hold Points (Post Holders): None

DOCUMENT CONTROL
Reason for Revision: ~~REVIEW~~ / ~~RE-ISSUE~~ / NEW ISSUE

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Signature:				
Date:	2/6/15	6/6/15	3/6/15	08-JUN-15

IMPLEMENTATION:
This document outlines the sample preparation and running of samples using the general ICP method on the ICP-MS NexIon 300 found in laboratory 185.

This method supersedes the general ICP-MS method described in BRAD/22429/OI/0097.

DESCRIPTION AND PURPOSE OF INSTRUCTION:
All sample preparation and running of samples must be carried out uniformly by all laboratory analysts in order to provide confidence in the laboratory results. The procedure is outlined in a series of step by step instructions.
This ICP method is for the determination of the following metals: Al, Zn, Ni, Fe, Cu, Pb, Cr, B and Cd in

(a)high concentration Mg/Na matrix for Sentencing Tank/ FMDT samples
(b) 20 -30% nitric acid/ 0-5% hydrogen peroxide medium for NOx scrubber samples

DOCUMENTATION:

References: See Section 2

Environmental and Risk Assessed: NO

COSHH: BRAD/0311/2014, BRAD/22429/RPT/00133 Chemical Risk Assessment for Pregnant Female Chemist working at Bradwell

PRECAUTIONS:

Radiological: The lab is a C2 area. Minimum PPE required: tyvek overall, overshoes, rubber gloves, hard hat or cloth cap and an EPD worn in a red belt.

Fire: Fire extinguisher and push button fire alarm available.

Lab Safety: Lab safety glasses and disposable gloves.

SPECIAL TOOLS AND MATERIALS:
Plastic volumetric flasks dedicated for ICP-MS use.

Perkin Elmer NexION v1.5 software for ICP-MS.

SPECIAL ARRANGEMENTS:

STATE OF PLANT:
Pre-commissioning and steady state.

Amendment History

The following changes have been made to this issue of the document.

Issue 2	to	Issue 3

Date of Change	Section or Paragraph	Amendment
May 2015	2.01 & 2.04 2.01 3.01 3.02 4.02 6.02 8.07 8.12 & 8.13 9.03 Appendix F Contents P34 no 15 Appendix I	Changed how long blank can be used for. Changed method for making blank. Extension of QC expiry date to 2 months .Clarification on type of volumetric flask. Deleted mention of tube zero. Modification of run order to remove blank between QC and sample. "May" changed to "should" Clarification of when calibration line needs to be re-run. Modified how concentration %RSD is calculated. Added" fail QCs must not be used to update control charts." Added units to the table. Added an explanation on how to use the table. Quoted values are now < . Updated page numbers. R2 values must be > 0.995 and < 1.0. New Appendix

Abbreviations / Acronyms

ADAP	Aqueous Discharge Abatement Plant
BEC	Background Equivalent Concentration
Cps	Counts per second
FED	Fuel Element Debris
FED D	Fuel Element Debris Dissolution
ICP-MS	Inductively Coupled Plasma – Mass Spectrometry
IS	Internal Standard
QC	Quality Control
%RSD	Relative Standard Deviation
SD	Standard Deviation

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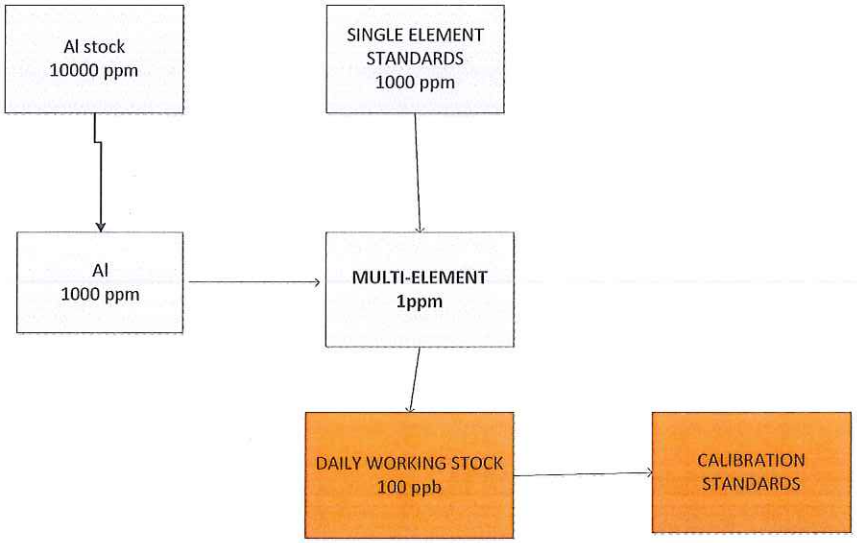
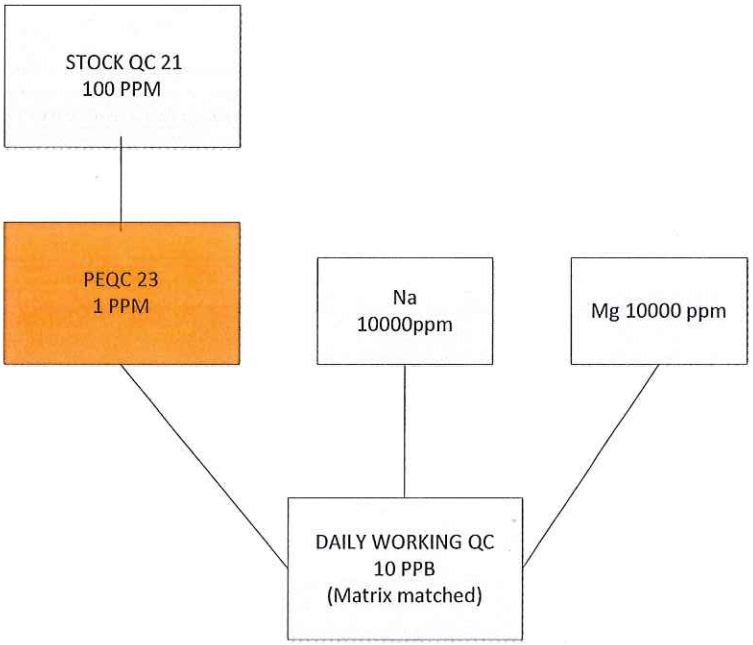
1 Process Flow Overview

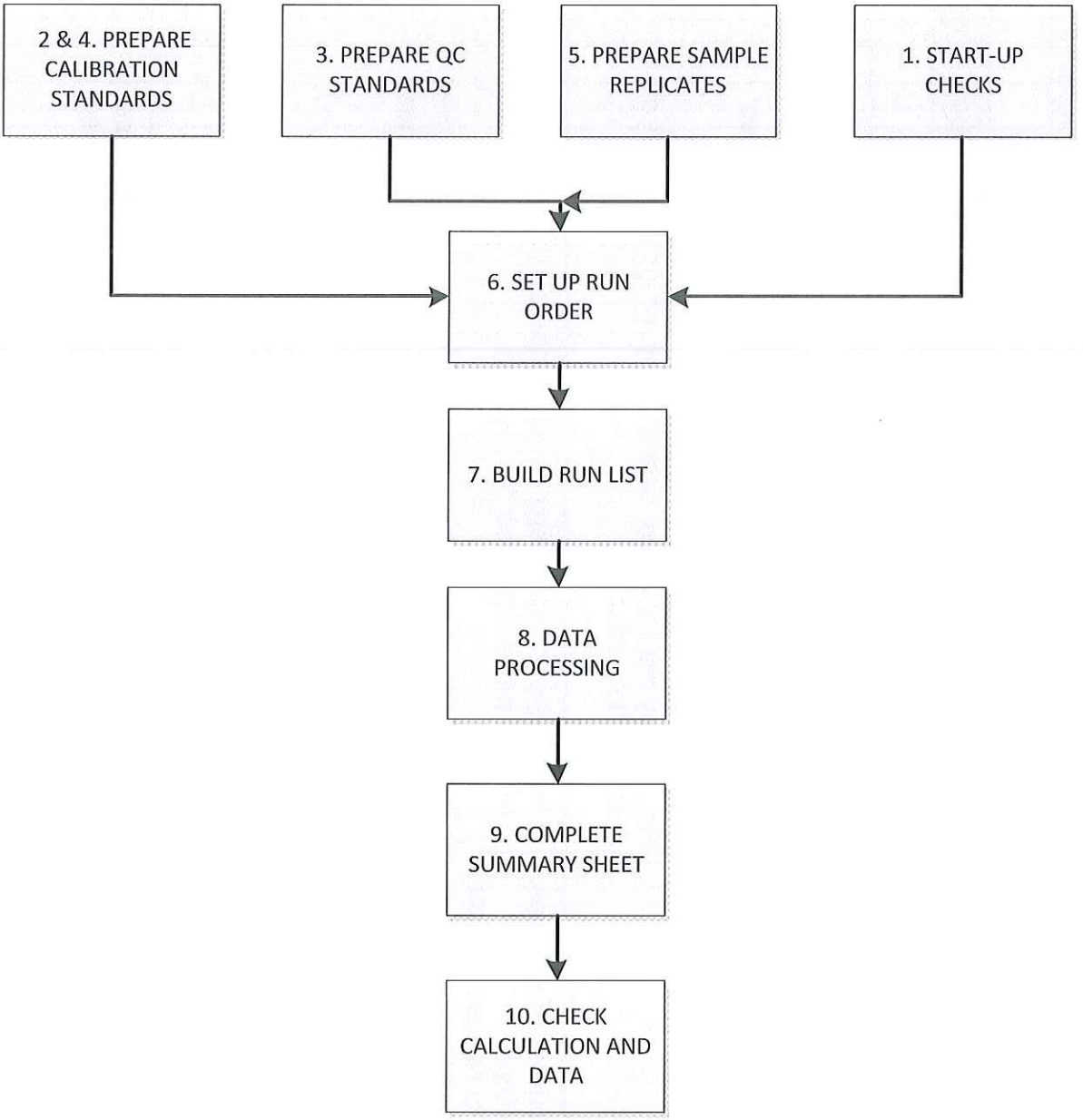
Preparation of calibration standards (Task 2 & 4)



Preparation of QC standards (Task 3)



WORK FLOW


2 & 4. PREPARE CALIBRATION STANDARDS
3. PREPARE QC STANDARDS
5. PREPARE SAMPLE REPLICATES
1. START-UP CHECKS
6. SET UP RUN ORDER
7. BUILD RUN LIST
8. DATA PROCESSING
9. COMPLETE SUMMARY SHEET
10. CHECK CALCULATION AND DATA

NOTE: The order of these sub-tasks may need not to be completed sequentially.

Step No:	Activity	Remarks/Notes	Team Members
Sub task 1: Start-up checks			
1.01	Perform the start-up checks as described in the procedure "ICP-MS Daily Performance Checks". The ICP-MS **must** pass these checks before use.	Reference: BRAD/22429/OI/0102 Also make a full visual check of all capillaries and pump tubes, nebuliser chamber etc. to ensure regular flow rates and correct instrument performance. Record any maintenance, observations, on-going issues etc in the ICP log book	Lab Technician
1.02	Before running any calibration curves or samples, the following criteria must be fulfilled: 1. For the analytes Be, In, U Net Intens. RSD must be < 3 % 2. Ce:Ce0 ratio must be less than 2.5% ie Net Intens. Mean < 0.025 3. Ce : Ce++ ratio must be less than 2.5% ie Net Intens. Mean < 0.025. Pass/fail decisions are highlighted in the daily performance check spreadsheet described in 1.02	In the case of failure of any of these criteria, a Smart-tune should be run to diagnose the problem. A failure in the double charged ratio (3) may be indicative of wear and tear or corrosion in the ICP cones. Typically these need changing every 4-6 months of continual use.	
1.03	Print out a copy of the daily performance check (if this has not been already done) . Record: 1. To which ICP this refers.	The monitoring is to ensure the instrument is being properly maintained and any deterioration in performance is picked up and remedied. Instructions on how to complete the daily performance	

Step No:	Activity	Remarks/Notes	Team Members
	2. Pass/fail of check 3. Initials of analyst. The data from the summary sheet should be recorded on the appropriate tab on: Y:\Decommissioning\FED Programme Working Area\FED & ADAP Commissioning Working Area\Labs\ICP External Calibration\Daily Calibration\Daily Performance Checks.xlsx The hard copy should be stored in a folder labelled ICP daily performance check.	checks summary sheet are detailed in Appendix E. Contact Lab Manager and/or Perkin Elmer engineer if unable Lab Technician to improve these metrics.	
Sub task 2: Preparation of Multi Element Stock			
2.01	• A 2% nitric acid solution should be prepared every 7 days, which can be used for blanks and dilution of standard stock solutions. • Open a 2L bottle of Type 1 water and pour out approximately 56 mL. • Add 56 ml of 70% Trace Select nitric acid. stopper and shake the bottle and record expiry date.	ALL STOCK SOLUTIONS AND QCS SHOULD BE TRANSFERRED TO CLEAN MEDICINE CUPS BEFORE ADDITION TO VOLUMETRIC FLASKS. DO NOT PLACE ANY PIPETTE TIPS INTO THE STOCK SOLUTIONS.	Lab Technician
2.02	• **NOTE: This multi-element stock solution should only be made up if the previous batch has run out, expired or become contaminated.** • Auto-pipette 0.1ml of the 1000ppm B, Al, Cr, Fe, Ni, Cu, Zn, Cd and Pb stock	Use the custom standard 1000ug/ml Al, B, Cd, Cr, Cu, Fe, Ni, Pb, Zn in 5% HNO3 to prepare.	

Step No:	Activity	Remarks/Notes	Team Members
	solutions into a clean 100ml volumetric flask • Bulk to 100ml line using 2% nitric acid solution, stopper and shake.		
2.03	• Pour into a new labelled 100ml polythene bottle.	The bottle should be labelled with: **1ppm Multi Element Stock and its expiry date** (2 months from date of preparation). Record the make-up of this solution in: Y:\Decommissioning\FED Programme Working Area\FED & ADAP Commissioning Working Area\Labs\ICP External Calibration\Calibration spreadsheets\ICP Standards Log.xlsx	Lab analyst
2.04	Daily Working Stock • Label a new 15ml ICP-MS tube with 'Daily Working Stock' and the day's date • Auto-pipette 9ml freshly made 2% HNO_3 • Add 1ml of the 1ppm Multi Element Stock • Securely lid and shake to make a 100ppb stock solution.	See 2.01 for make-up of 2% nitric acid.	Lab analyst
Sub task 3: Preparation of QCs			
3.01	**NOTE: This QC stock solution should only be made up if the previous batch has run out, expired or become contaminated.** PEQC23 dilution • Obtain a new 100ml polythene bottle and label **1ppm PEQC23 & expiry date** (2 month from date of making) • Auto-pipette 1ml 100ppm PEQC23 into a	Use dedicated 100ml plastic volumetric flask to avoid contamination. A custom standard which includes B,Al, Cr, Fe, Ni, Cu,Zn, Cd and Pb; this solution (PEQC23) should be used to prepare QCs. The numbering of QCs and expiry dates are listed in: Y:\Decommissioning\FED Programme Working Area\FED &	Lab analyst

Step No:	Activity	Remarks/Notes	Team Members
	clean 100ml volumetric flask • Bulk to 100ml line using 2% nitric acid solution.stopper and shake. • Pour into polythene bottle. • The bottle should be labelled **1ppm PEQC23 and its expiry date (2 months from date of preparation)**	ADAP Commissioning Working Area\Labs\ICP External Calibration\Calibration spreadsheets\ICP Standards Log.xlsx Record the make-up of this new solution in the above file.	
3.02	Daily working QC • Add approximately 50ml of 2% nitric acid into a clean plastic 100ml volumetric flask. • Auto-pipette 1ml of the 1ppm PEQC23 • Auto-pipette 0.3ml of the 10000ppm Na stock solution • Auto-pipette 0.6ml of the 10000ppm Mg stock solution . • Bulk to 100ml line using 2% nitric acid solution, stopper and shake.	The daily working QC is valid for 24 hours.	Lab analyst
Sub task 4: Preparation of Calibration Standards			
4.01	• Label five 15ml autosampler tubes with 1, 2, 5, 10, and 20ppb respectively		Lab analyst
4.02	• Using an auto-pipette transfer the volumes of 2% nitric acid and daily working stock (in this order) shown in TABLE 1		

TABLE 1	STD 1	STD 2	STD 3	STD 4	STD 5
2% HNO_3	9.9 ml	9.80ml	9.50ml	9.00ml	8.00ml

Step No:	Activity	Remarks/Notes	Team Members

Daily working stock	0.10ml	0.20ml	0.50ml	1.00ml	2.00ml
Concentration (ppb)	1	2	5	10	20
Total volume ml	10.00	10.00	10.00	10.00	10.00

	• Apply screw tops and shake. • These are the calibration standards which will be run through the instrument and are good for 24 hrs or until a QC standard fails, which ever is the sooner.		

Sub task 5: Sample Preparation

5.01	The solution must be diluted according to the following dilutions:	All samples are to be made up in clean sample vials.	Lab analyst
	Pre-neutralisation	X 2500	
	Supernatant	X 402	
	FMDT/sentencing tank (general, Hg)	X 402	
	NOx scrubber sample	X 1616	
5.02	Achieving x402 dilution: 1. Transfer 9.0 ml of 2% fresh nitric acid into the tube 2. Add 1.00ml of sample	This is a x10 dilution, followed by a x 40.2 dilution.	

Step No:	Activity	Remarks/Notes	Team Members
	3. In a second tube add 9.8ml of 2% fresh nitric acid, followed by 0.25ml of solution in point 2.		
5.03	Achieving x1616 dilution: 1. Transfer 9.8 ml of 2% fresh nitric acid into the tube. 2. Add 0.25ml of sample and mix. 3. In a second tube add 9.8ml of 2% fresh nitric acid, followed by 0.25ml of solution in point 2.	This is a x 40.2 dilution, followed by a x 40.2 dilution.	
5.04	Achieving x 2500 dilution 1. Transfer 9.8 ml of 2% fresh nitric acid into the tube 2. Add 0.20ml of sample. 3. In a second tube add 9.8ml of 2% fresh nitric acid, followed by 0.2 ml of solution in point 2.	This is a x 50 dilution, followed by a x 50 dilution.	
Sub-task 6: Setting up a Run Order			
6.01	Run files should be named according to the following format which allows identification Yymmdd icp no sample id sample point name analyst Eg150211 ICP1 A0613 FMDT2 AN	For sample point name reference BRAD/22405/OI/0166 and it will be listed under 'Sample Name'.	

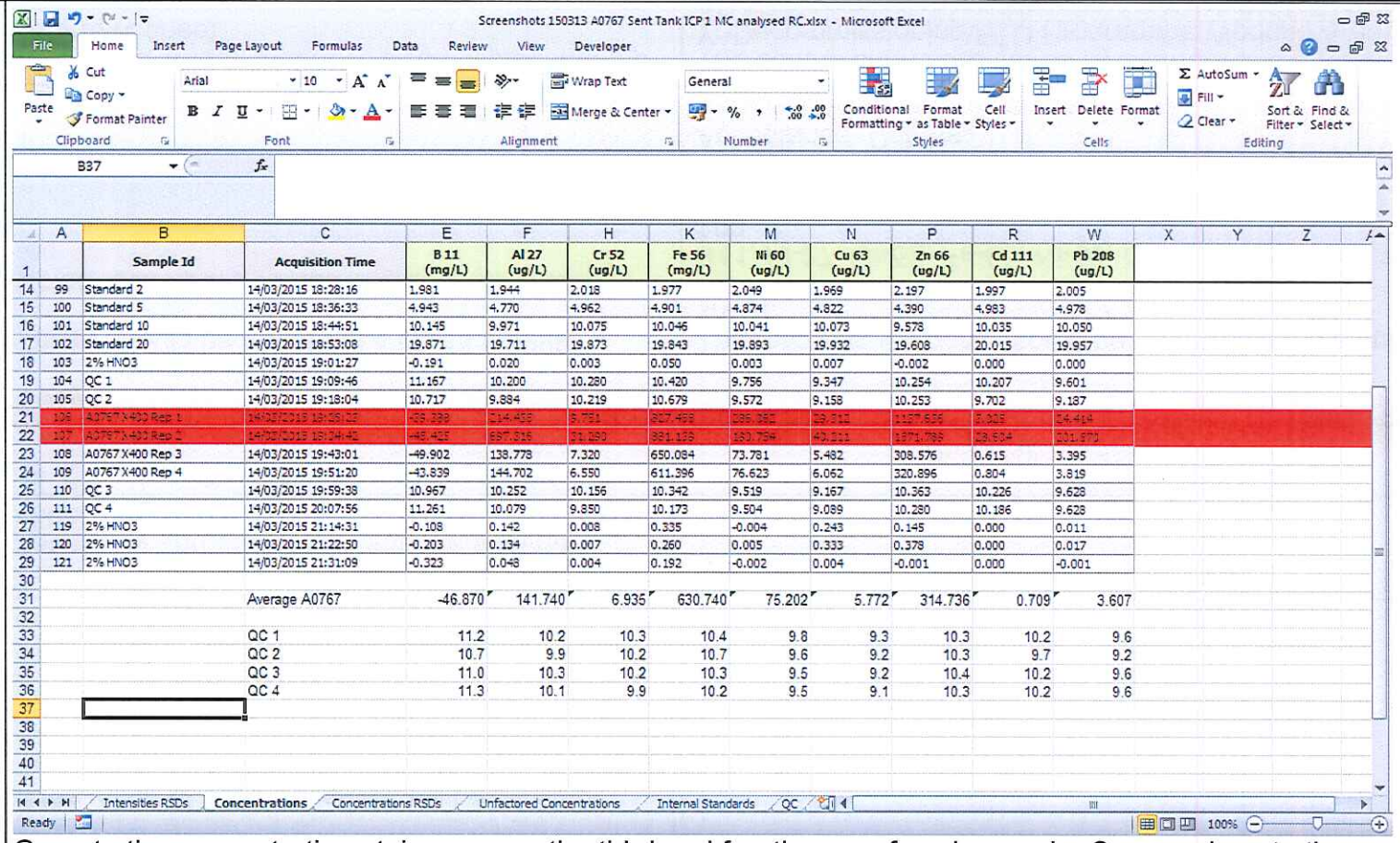
Step No:	Activity	Remarks/Notes	Team Members
6.02	• The run should be set up in the following order: Blank (x 10) Calibration Blank Standard 1 (1 ppb) Standard 2 (2 ppb) Standard 3 (5 ppb) Standard 4 (10 ppb) Standard 5 (20 ppb) Blank Blank QC QC Sample Sample Sample Sample QC QC Sample Sample Sample Sample QC QC Etc • Make a note of the dilution factor used eg	Blank here refers to 2% nitric acid. The method file to which this refers is Main Metals.mtd which is in the Methods/Commissioning ICP Methods file. The 10 blanks are required to determine the BEC. 10 separate tubes are not required, 3 x 50 ml blank tubes can be sampled 3- 4 times to give the 10 blanks. BEC determination is only required once a day. QC refers to daily working QC made up in 3.02. Each QC should be run from separate tubes. Correlation co-efficients (R^2) values should be checked either manually by plotting in Excel or using the Nexion software using the Quantitative Analysis Calibration report (See Appendix B). If $R^2 < 0.995$, the calibration run may need to be aborted and should be run, with new standards. This is in the case where removal of 1 calibration point does not improve $R^2 > 0.995$ Each new sample should be run 4 times and sandwiched between 2 QC standards. The first two replicates should be discarded from data analysis. Samples are deemed acceptable values if: 1. %RSD values of the duplicates are < 10 % (see section 8.04 for exceptions)	

Step No:	Activity	Remarks/Notes	Team Members
	A0747 FMDT x 402 in sample description. Write in columns for aliquot volume.	2. QC before and after the duplicates are within the instrument specific acceptance limits as defined by the Shewhart control charts (ie 3 standard deviations of the mean)	
6.03	• On the ICP-MS select the Method tab. • Click File→Open→Methods→Commissioning ICP Methods→Main Metals • Ensure parameters within your chosen method have not been altered. • Ensure there are sufficient levels of rinse solution, internal standard, QC's and standards for the desired method. The lowest level of blank solution, QC's and standards is 5ml. The minimum internal standard level is 100ml.	Each individual method shall contain a pre-defined set of parameters that have been specifically designed for samples that the lab shall receive. Should you wish to use this method but require to alter the parameter, then you MUST save the altered method separately and name it accordingly and save in methods/training. DO NOT SAVE OVER THE PREDEFINED MAIN METALS ICP METHOD.	Lab analyst
Sub task 7: Building a Run List			
7.01	• Click the Samples tab. • Ensure that the Batch tab is ticked for use with the autosampler. • **Only if run is long:** Tick the autostop button and select from the drop down menu "when batch complete". • Enter the desired information with the appropriate columns is correct. Check that the sample IDs correlate with the location of the samples within the autosampler. • Right click the method cell in order to open	The Samples tab is used to build the run list order. Columns that the analyst must complete are: • A/S Loc – autosampler location. Where the samples are located. • Batch ID – labeled on sample bottle e.g. NO_x Scrubber • Sample ID –labelled on samples delivered to the lab e.g. A0809 • Measurement Action – Run sample, run blank, stds and sample etc. Refer to page 158 of the Nexlon Software Guide for more detail.	Lab analyst

Step No:	Activity	Remarks/Notes	Team Members
	the desired method selected in sub task 6.02. Ensure that this method is filled down for all the samples requiring this type of analysis. • To select the entire run list to build, left click "Batch Index", this shall highlight all of the entries within the sample list. • Left click "Build Run List". Check the run list is in the order you desire. Ensure that the caps are off all the sample tubes you wish to run, as well as the internal standard tubing is within the correct solution. • Left click "Analyse Batch".	• Method – The desired method that was selected in sub task 6.02 • Description – e.g. quantitative analysis • Sample Type – determines what is in the tube at each autosampler location e.g. sample, QC spike etc. Refer to page 158 of NexIon Software Guide for more detail. • Fill in "aliquot volume" & "diluted to volume" An additional column that may require entering is the Wash Overide column. If a particular sample is suspected to be particularly concentrated even after dilution, then a longer wash override should be entered to ensure that there is no carry over into the next sample.	
7.02	SHUTTING DOWN • If the run is due to finish before the end of the shift, then remember to pump clean acid through both inlets, run dry. • Shut down plasma and disengage platens.	This applies to non-24 hr working pattern.	
Sub-task 8: Processing of data on Excel from NexION software.			
8.01	The results workbook from the run should be exported to Excel. This can then be processed manually.	Report view Tab-> bottom right-> Export all	
8.02	The tabs from the Main Metals Guide spreadsheet should be imported into the existing results worksheet.	Y:\Decommissioning\FED Programme Working Area\FED & ADAP Commissioning Working Area\Labs\ICP External Calibration\Calculation spreadsheets\Main metals Guide spreadsheet 20Jan15.xlsx	

Step No:	Activity	Remarks/Notes	Team Members
	Right click tab you want to copy, select move or copy and tick box " Create a copy". In the dropdown box select worksheet you are working on-> select OK. The instructions on the sheet should then be followed. For reference they are also reproduced below in 8:03 – 8:10.		
8.03	Copy the first 10 blanks and calibration standards from the intensities tab onto the calculations worksheet. QCs, samples and any subsequent blanks can be ignored.		
8.04	Insert IS correction factor columns between Zn 68 and Cd 111 and after Pb 208.		
8.05	To obtain IS correction factor, divide initial IS value by current IS value eg G$7/G7. Drag down to generate G$7/G8, G$/G9 etc..	B to Zn should use Sc45 as IS. Cd to Pb use Tb 159 as IS. Check Sc45 and Tb 159 column both should have the same numbers for that column.	
8.06	IS corrected values: Multiply each value by corresponding IS correction value.		
8.07	Average blank: Excluding the first four blanks, calculate the average.	The first four blanks should be discarded as these act as a rinse, the latter samples should be leveling off and reaching a baseline. These early blanks have contamination in them and will generate an artificially high baseline, leading to lower values. Use function =AVERAGE( ) to select region where blanks have reached stable values. Do not discard more than 5 of the blanks as some variation is required to give an indication of the noise level	

Step No:	Activity	Remarks/Notes	Team Members
8.08	Blank corrected: Subtract the average blank value from each value		
8.09	Calibration standards: Cut and paste standards 0 -20 ppb here and type in a column for the concentration values Calculate the correlation co-efficient R^2 using the formula =RSQ ($range x, range y). Calculate the slope of the calibration line using the formula =SLOPE($range x, range y).	In this case range x are the calibration standards, 0 -20. Range y are the cps that correspond to the calibration standards for each element. R2 >0.995, otherwise the calibration will need to be repeated. A single calibration point may be removed if it is clear that there is an outlier which may improve the R2 value eg in the case that 2 of the calibration points have been placed in the wrong order. The removal of a calibration point should be recorded in the comments section. Only 1 point can be removed. NB: Value of this line should be < 1, if x are the values from 0 -20 ppb	
8.10	Calculate the BEC: Using the value of an average blank (see 8.07) **BEC = average blank x slope x dilution factor**		

Step No:	Activity	Remarks/Notes	Team Members
8.11	 Go onto the concentrations tab, average the third and fourth reps of each sample. Copy and paste these values directly into the summary sheet tab as average value of sample. The QC values can also be directly copied into the summary sheet tab.		
8.12	On the concentrations tab, calculate the standard deviation between the third and fourth sample	Use function =STDEV()	

	A	B	C	E	F	H	K	M	N	P	R	W
1		Sample Id	Acquisition Time	B 11 (mg/L)	Al 27 (ug/L)	Cr 52 (ug/L)	Fe 56 (mg/L)	Ni 60 (ug/L)	Cu 63 (ug/L)	Zn 66 (ug/L)	Cd 111 (ug/L)	Pb 208 (ug/L)
14	99	Standard 2	14/03/2015 18:28:16	1.981	1.944	2.018	1.977	2.049	1.969	2.197	1.997	2.005
15	100	Standard 5	14/03/2015 18:36:33	4.943	4.770	4.962	4.901	4.874	4.822	4.390	4.983	4.978
16	101	Standard 10	14/03/2015 18:44:51	10.145	9.971	10.075	10.046	10.041	10.073	9.578	10.035	10.050
17	102	Standard 20	14/03/2015 18:53:08	19.871	19.711	19.873	19.843	19.893	19.932	19.608	20.015	19.957
18	103	2% HNO3	14/03/2015 19:01:27	-0.191	0.020	0.003	0.050	0.003	0.007	-0.002	0.000	0.000
19	104	QC 1	14/03/2015 19:09:46	11.167	10.200	10.280	10.420	9.756	9.347	10.254	10.207	9.601
20	105	QC 2	14/03/2015 19:18:04	10.717	9.884	10.219	10.679	9.572	9.158	10.253	9.702	9.187
21	106	A0767 X400 Rep 1	14/03/2015 19:26:23	459.389	214.459	8.791	827.468	286.382	39.511	1157.626	5.205	14.414
22	107	A0767 X400 Rep 2	14/03/2015 19:34:42	448.425	887.316	31.250	881.139	180.754	40.211	1871.788	28.684	201.870
23	108	A0767 X400 Rep 3	14/03/2015 19:43:01	-49.902	138.778	7.320	650.084	73.781	5.482	308.576	0.615	3.395
24	109	A0767 X400 Rep 4	14/03/2015 19:51:20	-43.839	144.702	6.550	611.396	76.623	6.062	320.896	0.804	3.819
25	110	QC 3	14/03/2015 19:59:38	10.967	10.252	10.156	10.342	9.519	9.167	10.363	10.226	9.628
26	111	QC 4	14/03/2015 20:07:56	11.261	10.079	9.850	10.173	9.504	9.089	10.280	10.186	9.628
27	119	2% HNO3	14/03/2015 21:14:31	-0.108	0.142	0.008	0.335	-0.004	0.243	0.145	0.000	0.011
28	120	2% HNO3	14/03/2015 21:22:50	-0.203	0.134	0.007	0.260	0.005	0.333	0.378	0.000	0.017
29	121	2% HNO3	14/03/2015 21:31:09	-0.323	0.048	0.004	0.192	-0.002	0.004	-0.001	0.000	-0.001
30												
31			Average A0767	-46.870	141.740	6.935	630.740	75.202	5.772	314.736	0.709	3.607
32												
33			QC 1	11.2	10.2	10.3	10.4	9.8	9.3	10.3	10.2	9.6
34			QC 2	10.7	9.9	10.2	10.7	9.6	9.2	10.3	9.7	9.2
35			QC 3	11.0	10.3	10.2	10.3	9.5	9.2	10.4	10.2	9.6
36			QC 4	11.3	10.1	9.9	10.2	9.5	9.1	10.3	10.2	9.6
37												
38												
39												
40												
41												

Step No:	Activity	Remarks/Notes	Team Members
	reps. If the value of both replicates are below BEC, then the concentration quoted is < BEC. There is no requirement to calculate %RSD; just write N/A. If one of the replicates is below BEC, then take BEC value in calculating the average, standard deviation and %RSD.		
8.13	Calculate the %RSD for each element where necessary and copy these into the summary sheet.	Use the equation %RSD=100*SD/average	
Sub-task 9 Completing Summary sheet			
9.01	Copy and paste R2 values from worksheet. These should be quoted to 4 dp eg 0.9999 Colour code pass values in green, fail values in red	See 8.08 The following isotopes should be reported in the summary sheet: B11, Al-27, Cr-52, Fe-56, Ni-60, Cu-63, Zn-66, Cd-111, Pb-208 Acceptance Criteria: If R2 > 0.9950 then pass otherwise fail	
9.02	Check QC values that have been copied into the summary sheet. Consult control charts for pass/fail criteria. If the values lie in between the upper and lower control limits ie mean ± 3 SD then QCs are	The control charts are stored by element and updated in Y:\Decommissioning\FED Programme Working Area\FED & ADAP Commissioning Working Area\Labs\ICP External Calibration\Control charts See Appendix D for instructions on how to complete the control charts..	