

EDXRF spectrometer recalibration for MXene synthesis products

Date: 2022-12-09

Tags: Kai Kai 2020 Synthesis Optimisation Purity EDXRF

Created by: James Bird

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Goal : Recalibrate PANalytical MiniPal 4 EDXRF spectrometer to retrofit data from powder Ti_3C_2 MXene synthesis products collected using Omnian Fundamental Parameters approach ([\[Experiment\] EDXRF of MXene synthesis products I](#)) to improve on the original calibration attempt described in [\[Experiment\] EDXRF spectrometer calibration for MXene synthesis products](#)

Procedure :

Sample and instrument preparation

- Produce a larger set of standards with composition closer to that of the MXene synthesis products (synthesised in [\[Experiment\] Optimisation of MXene Synthesis - Execution of Plackett-Burman Screening Design](#)) mixed with an SiO_2 sand internal standard (in proportions detailed in [\[Experiment\] XRD analysis of freeze-dried MXene synthesis product with an internal standard - PANalytical X'Pert Pro](#))

Compounds containing all of the EDXRF-detectable elements ($Z > Z_{\text{Na}}$) contained in a $\text{Ti}_3\text{C}_2\text{T}_x$ synthesis product spiked with silica (Ti, Al, Si, Cl) were mixed as powders in known and measured proportions, as detailed in the table below. Twelve standards, instead of eight, are prepared by combining NaCl, SiO_2 and Ti_3AlC_2 (Kai Kai 2020 batch, $\approx 98\%$, $< 75 \mu\text{m}$) KaiKai to as best as possible cover the target concentration range in equally-spaced increments. The maximum concentration of each compound is 86, 54 and 98 wt% for Ti_3AlC_2 , NaCl and SiO_2 , respectively. Sodium chloride increments were more closely spaced in the chlorine concentration range of 0-10wt% based on the initial EDXRF analyses of the [\[Experiment\] EDXRF of MXene synthesis products I](#) and QPA in XRD ([\[Experiment\] Quantitative Phase Analysis \(QPA\) of MXene synthesis product PXRD patterns](#)). Target total mass is ~ 250 mg to match those used in the Omnian approach.

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Standard N° / #	m(Ti3AlC2) / mg	m(NaCl) / mg	m (SiO2) / mg	Total / mg
1	0	5.8	237	242.8
2	16	4.8	228.6	249.4
3	39.7	9.9	200.8	250.4
4	75.7	14.2	156.2	246.1
5	74	16.9	167.1	258
6	94	18.5	137.7	250.2
7	111.3	21.5	117.4	250.2
8	124.5	20.7	98.4	243.6
9	149.1	96.2	3.4	248.7
10	186.7	10	64.3	261
11	201.2	10.4	57	268.6
12	218.5	12.2	20.2	250.9

- Homogeneity ensured by manual shaking of powder-containing vials until colour continuity visibly achieved
- Powder transferred to a sample holder to cover the entirety of a Mylar (C₁₀H₈O₄) film of 28 mm diameter (3.6 µm thick and density 1.37 g cm⁻³)
- Samples loaded into the EDXRF instrument (Panalytical MiniPal 4)
- Helium flow ensured

Software operation

In the MiniPal software program, a new application was defined, where the analytical program is first assembled, such that:

- Sample type defined as pressed powder sample (despite not being pressed) with no binder

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- Spinner used
- None of balance element/compound, normalisation to 100% or default oxides required
- Repeat factor = 1

Required measuring channels/elements are selected to detect Ti, Al, Si and Cl K_α emission lines and measuring conditions are defined as below:

Condition name	Anode voltage / kV	Current / μ A	Filter	Medium	Measurement time /s
	5.5	1000	None	Helium	1600
	12	395	Thin aluminium	Helium	1600
	30	300	Ag	Air	1600

The above conditions were selected to reproduce the Omnian approach used previously, in [\[Experiment\] EDXRF of MXene synthesis products I](#), to ensure retrofitting of the initial outputs to this recalibration is plausible. Some exceptions are present, namely the use of a current of 395 μ A in the condition, resulting from the software optimising the conditions of a representative sample. The representative chosen was standard 7 due to its midrange concentration values for each compound. The current was initially set at 750 μ A to achieve the maximum anode power of 9W. The optimisation procedure moderated the current to ensure the detector receives up to, but not in excess of 50,000 cps in order to optimise photon detection. The other exception is the use of air for the medium in the condition, which should have no impact on the detection of higher energy photons, for which this condition is intended. The condition was measured purely to quantify the intensity of elastically (Compton) scattered Rh K_α tube lines to be able to deploy the Compton correction model, as otherwise no elements of interest lie in this range.

Calibration of the system for this application then follows by defining the measured concentrations of each detectable element/compound (channel) in each

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standard calibrant as defined below.

Standard N° / #	[SiO ₂] / wt%	[Al] / wt%	[Cl] / wt%	[Ti ₃ C ₂] / wt%
1	97.611	0.000	1.449	0.000
2	91.660	0.889	1.167	5.526
3	80.192	2.198	2.398	13.656
4	63.470	4.265	3.500	26.494
5	64.767	3.977	3.973	24.705
6	55.036	5.209	4.485	32.360
7	46.922	6.168	5.213	38.315
8	40.394	7.086	5.155	44.021
9	1.367	8.312	23.464	51.638
10	24.636	9.918	2.324	61.613
11	21.221	10.386	2.349	64.519
12	8.051	12.074	2.950	75.010

Calculation and identification of the appropriate line groups is automatically carried out in a spectrum evaluation using non-linear least squares fitting based on the AXIL algorithm (developed at the University of Antwerp). All standard samples are measured under the conditions to extract net intensities for the line groups of interest. Deconvolution is performed, to ensure the background fit and identification of any other present line groups doesn't impede accurate calculation of the line group intensities of interest. And finally, regression analysis is performed, taking into account regression correction factors. The background ranges and fitting procedure parameters are detailed in the table below, set equal to those used in Omnian analysis.

Condition name	Start energy / keV	End energy / keV	Background model	Nr of iterations	Preprocessor
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	1.25	5.25	Axil filter	10	Square root
	1.25	9.3	Axil filter	16	Square root
	20.5	30	Axil filter	8	Square root
	9.3	20.5	Axil filter	30	Square root
	0.6	7.75	Axil filter	24	Square root

This application differs from the previous, in that μ values are calculated empirically in the software (presumably using the Lachance-Trail algorithm), rather than with Fundamental Parameters, to determine the extent to which the matrix attenuates or enhances X-ray transmission. These μ values are summarised in the matrix correction factors, M_i , defined in the equations below, where R_i is the net relative intensity of deconvoluted line group, C_j is the concentration of each analyte in a measurement, μ_j its associated μ value, C_i the concentration of the analyte of interest and D_i , E_i and F_i are the fit parameters of the regression.

$$C_i = D_i + E_i \cdot (R_i M_i) \text{ where } M_i = [1 + \sum_{j=i \dots n, j \neq i} \mu_j \cdot C_j]$$

Results :

The calculations carried out in the MiniPal software programme gave the following outputs given in the table below: the measured net intensity (after deconvolution) of each line group in each calibrant, $I(\text{cps})$, the matrix correction factor for each element in each standard, M , and the product of the two, R_{corr} . Deconvolution was performed for all emission lines of the elements Na, Al, Si, S, Cl, Ca, Ti, Cr, Fe, Ni, Cu and of all K_α lines of Rh (L and K_β lines are omitted). Ar and Au lines (emitted from air and the hardware, respectively) are also omitted to avoid negative fitting.

The silicon calibration curve had no corrections applied to it as there was no notable reduction in associated errors when doing so. All data are plotted in files named EDXRF_Caliv2_Ident#.png, where # is the sample identifier listed in the

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table below, using Python script EDXRF_Plot.py uploaded to Github. Plots were produced via importing of .mp2 spectra files exported from MiniPal, where file nomenclature is AppliStandard_ID#_.mp2 such that # is a number identifier as described previously, and is either Cl-V, Na-S or Ni-Ag dependent on the condition under which the measurement was made (see earlier table describing conditions).

Element	Chlorine			Aluminium			Silicon (SiO2)	Titanium (Ti3C2)			
Ident / #	I (cps)	Matrix (M)	R_Corr / cps	I (cps)	Matrix (M)	R_Corr / cps	I (cps)	I (cps)	Matrix (M)	R_Corr / cps	Conc. sum / wt%
1	54.76	-2.00E-05	-1.10E-03	27.799	2.60E-05	7.23E-04	9090.24	15.322	2.10E-05	3.22E-04	99.06
2	220.11	-1.00E-05	-2.20E-03	573.758	3.10E-05	1.78E-02	14720.7	8532	2.50E-05	2.13E-01	99.24
3	66.989	-1.00E-05	-6.70E-04	1240.89	4.00E-05	4.96E-02	5803.31	33987.4	2.50E-05	8.50E-01	98.44
4	501.325	0.00E+00	0.00E+00	1333.02	6.20E-05	8.26E-02	6949.84	30467.3	3.30E-05	1.01E+00	97.73
5	28.096	4.00E-06	1.12E-04	1209.48	6.30E-05	7.62E-02	1016.62	22520.8	3.50E-05	7.88E-01	97.42
6	117.468	9.00E-06	1.06E-03	1903.48	6.50E-05	1.24E-01	2493.44	65922.8	3.30E-05	2.18E+00	97.09
7	635.19	7.00E-06	4.45E-03	1539.58	7.60E-05	1.17E-01	2843.25	38250.1	3.60E-05	1.38E+00	96.62
8	129.434	8.00E-06	1.04E-03	2162.77	6.90E-05	1.49E-01	551.186	81772.1	2.80E-05	2.29E+00	96.66
9	149.212	1.53E-04	2.28E-02	2215.48	7.90E-05	1.75E-01	51.391	85860.5	3.20E-05	2.75E+00	84.78
10	60.622	-1.00E-05	-6.06E-04	2209.19	1.00E-04	2.21E-01	383.091	81366.6	4.30E-05	3.50E+00	98.49
11	73.863	-1.00E-05	-7.39E-04	2181.68	1.04E-04	2.27E-01	491.161	88063.8	4.30E-05	3.79E+00	98.47
12	67.146	1.00E-06	6.71E-05	2278.88	1.03E-04	2.35E-01	119.675	84986.8	4.00E-05	3.40E+00	98.08

Each of the calibration curves constructed for this dataset are plotted below, with their associated fit equations placed nearest each plot alongside their R^2 values.

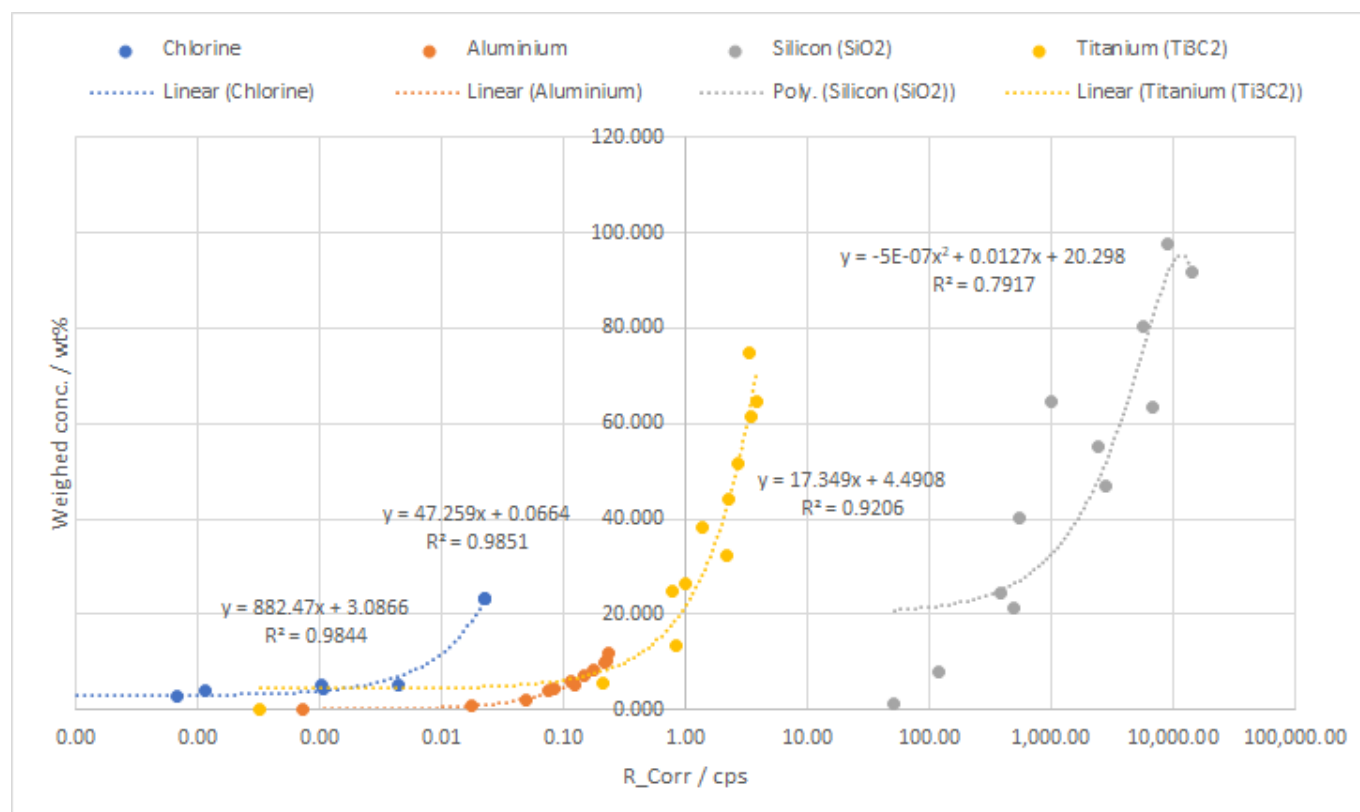
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Despite the calibration curves having high R^2 values for all elements bar silicon, it proved impossible to reproduce the alpha values and hence the matrix correction factors which were provided by the software when using any common variation of the Lachance-Trail algorithm. Without the correct form of the equation needed to correct R_i values, retrofitting of the data collected under Omnian conditions is impossible. As a result, a script (EDXRF_MatrixCoeffCalc3.py) was written to deduce alpha, D & E values via multiple linear regression of all calibrant concentrations and R_i for each emission line. The outputs of each regression, with comparison to the raw data, are given in plots named Calibration_.png, where is the calibrated emission line. The outputs are also summarised in the table below. As the SiO_2 concentrations are known, the Si line group is not subject to the regression analysis.

Channel	Al	Cl	Ti (Ti3C2)
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Standard deviation /wt%	0.29	0.31	3.04
D Value	0.335	3.897	9.042
E Value	0.021	6.322	0.018
Alpha for Al	10.723	-0.879	12.910
Alpha for Cl	6.508	65.636	-3.577
Alpha for SiO ₂	11.932	-1.860	12.785
Alpha for Ti ₃ C ₂	66.614	-5.463	80.200

With alpha values and the equation now known, another script (EDXRF_ApplicationRetrofitv2.py) was used to retrospectively extract concentrations of each element in the samples initially analysed in Omnian conditions ([\[Experiment\] EDXRF of MXene synthesis products I](#)), but only once the intensities of each line group analysed in the condition (Ti and Cl) have their intensity scaled proportional to the relative current used in Omnian analysis versus the application, such that:

$$R_{\text{curr}} = R_{\text{raw}} / (I_{\text{Omni}} / I_{\text{Appli}})$$

where I_{Appli} is the fixed anode current used for the condition in the application/calibration, I_{Omni} is the current used in Omnian analysis (where autocurrent selection is applied - see table below), R_{raw} is the net intensity any line group of samples measured in the Omnian 3 condition (see table in [\[Experiment\] EDXRF of MXene synthesis products I](#)) and R_{curr} is the current-corrected intensity value carried forward in the retrofitting calculations.

Ident	Omnian 3 anode current (I_{Omni}) / μA
1	248
2	206
3	159

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4	711
5	203
6	171
7	399
8	370
9	215
10	503
11	461
12	577
13	195
14	750
15	750
16	209

All calculated concentrations are listed in the table below for each sample, and the sum of the concentrations given in the final column named 'SUM'.

Sample Run / #	[Cl] / wt%	[Al] / wt%	[SiO2] / wt%	[Ti3C2] / wt%	SUM /wt%
1	3.75	0.62	77.74	19.26	101.37
2	3.64	1.51	73.86	24.10	103.11
3	7.90	1.91	15.89	27.48	53.18
4	3.91	0.57	97.65	11.03	113.17
5	3.44	1.67	59.36	25.25	89.72
6	13.80	1.34	14.59	22.09	51.82
7	-3.74	0.47	82.66	15.13	94.52
8	-0.82	0.46	79.20	15.43	94.27
9	4.43	0.52	76.58	24.47	106.01

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10	4.34	0.63	92.51	12.85	110.32
11	2.07	0.51	80.52	13.60	96.70
12	4.51	0.64	73.05	12.66	90.86
13	1.24	0.38	20.41	19.56	41.59
14	4.44	0.38	69.94	9.72	84.48
15	4.20	0.46	86.84	10.91	102.41
16	2.86	0.68	48.02	21.39	72.95

Conclusions

This recalibration was an attempt to remove some of the remaining discrepancies between the Omnian and initial calibration conditions, such as:

- Difference in sample matrix (undetected elements)
- Background fitting
- Condition definitions

These differences were either removed completely, accounted for or minimised as much as possible. Despite this, these calibration-calculated concentrations are difficult to trust, considering there are some negative values of concentration (in the case of chlorine) and sum concentrations are often not close to 100%.

Expected titanium concentration (assuming the non-quartz fraction is ~74 % titanium from MAX phase stoichiometry) plotted against EDXRF measured concentrations and scaled to 100% does give a reasonably good fit to a linear correlation in the plot below, although this would of course ideally be closer to an $x=y$ relationship.

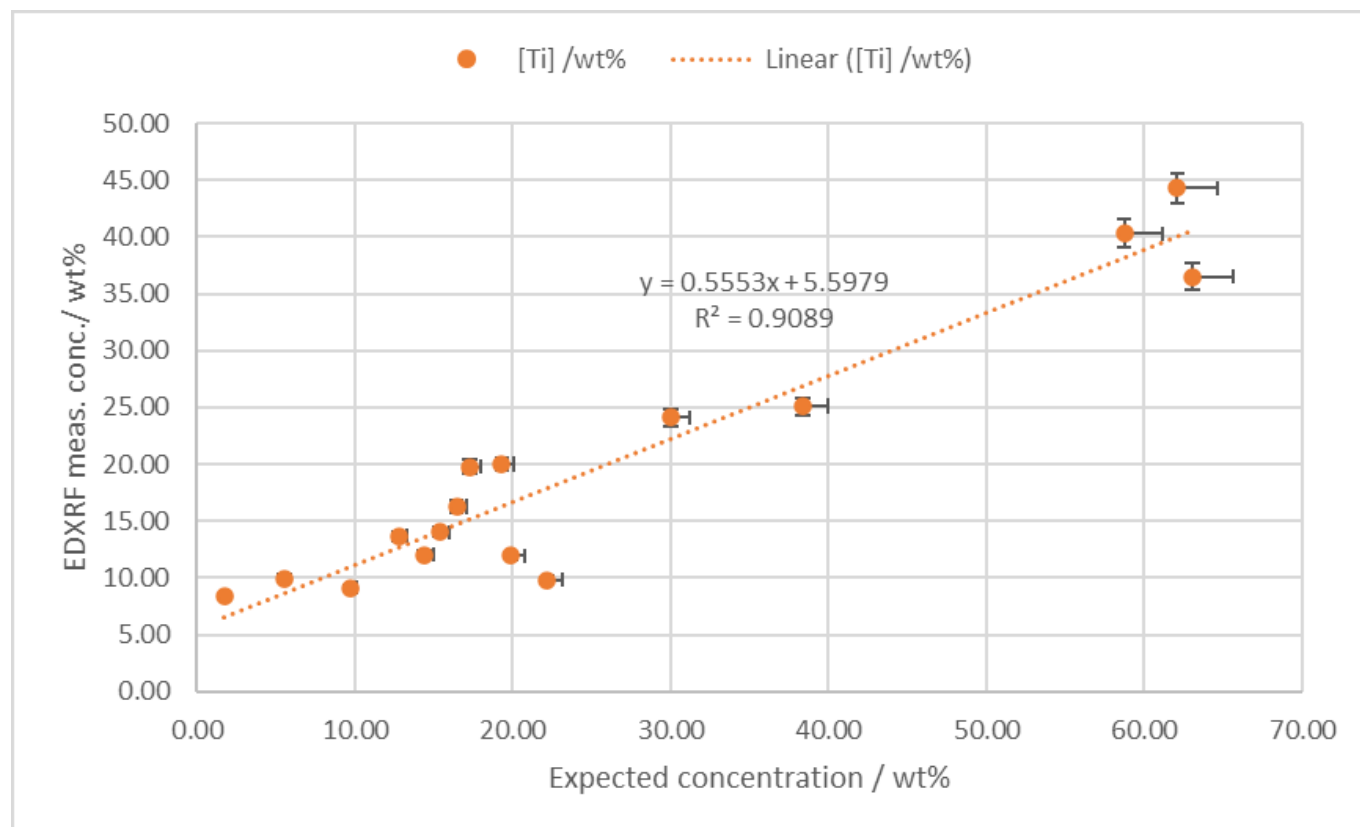
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In attempting to identify a source of these errors, kernel density estimates (KDEs) were produced to inspect the distribution of the intensity values registered in both the calibration and the Omnian analyses for each line group. Attached plots named KDE_Comparison.png, where is the name of the line group whose intensity distribution is depicted. These plots are produced using Python script KDE_EDXRF_Intensities.py uploaded to Github. It is expected that all of the Omnian measured intensities would lie within the range over which intensities are collected from the calibrations - this is true for all line groups with the exception of chlorine, which explains how negative concentrations have been output. In comparing the KDEs of the Omnian and calibration-measured intensities of the silicon line group, we remark that the distribution of Omnian-measured intensities is much narrower than is expected based on the known calibrant concentration range from 15-98% (see table above). There is no difference between these Omnian and calibration measurement conditions, Omnian 4 and , respectively. All

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other comparative KDE intensity distributions seem reasonable.

The final source of measurement variation to be scrutinised is the Omnian approach's ability to automatically adjust current to ensure the total count rate stays in an optimum range (as close to, but less than, 50,000 cps), and the calibration's inability to do so. The effect of this issue is demonstrated in attached plots KDE_Comparison_Condition_Cl-V.png and KDE_Comparison_Condition_Na-S.png, produced using Python script [KDE_EDXRF_Counts.py](#) (again uploaded to Github). For the /Omnian 4 condition, total count rate is always kept within the 0-50,000 cps range for both the calibration and the Omnian analyses, as expected seeing as the autocurrent function never kicked in during Omnian analyses. The same is not however true for the application collected data for the condition, where the maximum count rate was exceeded in half of the analyses. This has considerable impact on the validity of the calibration for both the Cl and Ti line groups which fall within that condition, as the detector accuracy is decreased.

Unfortunately, the inability to accurately measure the concentration of any single phase precisely impacts the ability to measure all concentrations precisely due to the definition of matrix-correction factors and by extension, R_{corr} . This limitation within the software hinders further progress with this technique from a measurement-optimisation perspective. If this issue could be overcome, a further improvement on the method used would see line-overlap corrections used for Al/Si $K_{\alpha 1}$ and Cl K_{α} /Rh L_{α} , for which some overlap is evident in plots EDXRF_Caliv2_Ident#.png.

Other sample-based issues could include:

- Remaining discrepancies between calibration standards and samples (i.e. matrix dissimilarity)
- Loose-powder sample type

Although Omnian analysis permits the use of loose powder samples if powder mass, density and thickness is known and input (in order to apply corrections), all

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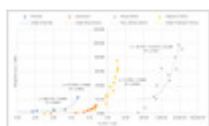
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calibration methods in the literature recommend samples are homogeneous, flat with a polished surface and have infinite thickness with respect to the incident radiation and XRF emissions. Although masses were kept fairly consistent across all analyses, consistent densities, thicknesses and percentage coverages could not be assured. Glaring sample dissimilarities are of course the morphological difference between two-dimensional Ti_3C_2 MXene and its three-dimensional Ti_3AlC_2 parent with particle size $< 75 \mu\text{m}$, and the absence of Na in Omnian-measured samples.

Attached files

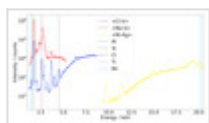
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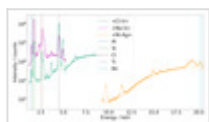
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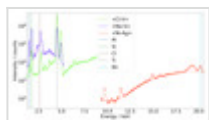
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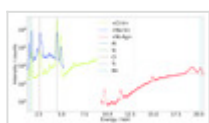
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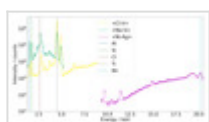
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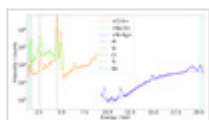
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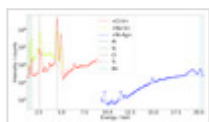
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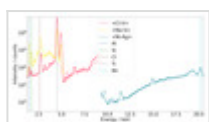
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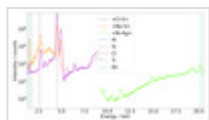
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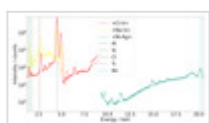
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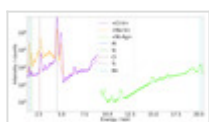
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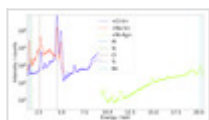
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AppliStandard_ID8_Ni-Ag.mp2

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AppliStandard_ID9_Cl-V.mp2

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AppliStandard_ID9_Na-S.mp2

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AppliStandard_ID10_Cl-V.mp2

sha256: 96ff7ebd9be148f1e0c31f0e04f21fc1cf2d771fa11fc29f0b2bdbaa0c31c082

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AppliStandard_ID9_Ni-Ag.mp2

sha256: 15675181bd8d6f5dd09868574f958ecdcae264affe6aea37f1d95a479300c76c

AppliStandard_ID10_Na-S.mp2

sha256: 12ea1957f652744c22f66e969088259418d153d74fd637e67df4c4fbe53fd304

AppliStandard_ID10_Ni-Ag.mp2

sha256: 687910edee6fe3da9420b93c7074410f0ca36352d0b1f471567fa037b92ebe80

AppliStandard_ID11_Cl-V.mp2

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AppliStandard_ID11_Na-S.mp2

sha256: a85a3a8e3b0c4d51b6846559af4ed65062bf0f88ba19adcc6b7225a6a7700e45

AppliStandard_ID11_Ni-Ag.mp2

sha256: cf6c05e2827f67d916187d65a874a849171f26984d60efc17583d81f9ba5eced

AppliStandard_ID12_Cl-V.mp2

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AppliStandard_ID12_Na-S.mp2

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AppliStandard_ID12_Ni-Ag.mp2

sha256: 4a89aa9f73ea236c8a54e4bfcde4fed447eaab71a3514881f9d538dd9ce689f7

AppliStandard_ID1_Cl-V.mp2

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AppliStandard_ID1_Na-S.mp2

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AppliStandard_ID1_Ni-Ag.mp2

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AppliStandard_ID2_Cl-V.mp2

sha256: 8842a6a103d77c0191879f899379a9d33d8dcfee2606fc1641cde3d8625b2342

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AppliStandard_ID2_Na-S.mp2

sha256: f262651011e13600dcce3c6905ff02b8a4771b4e41f8aa64d6663b9cbcf0f160

AppliStandard_ID2_Ni-Ag.mp2

sha256: 18e11d38e5745642e07d84ffc54f9045ee15d77a16d982bb9c5966915c62f4fb

AppliStandard_ID3_Cl-V.mp2

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AppliStandard_ID3_Na-S.mp2

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AppliStandard_ID3_Ni-Ag.mp2

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AppliStandard_ID4_Na-S.mp2

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AppliStandard_ID4_Ni-Ag.mp2

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AppliStandard_ID5_Cl-V.mp2

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AppliStandard_ID5_Na-S.mp2

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AppliStandard_ID5_Ni-Ag.mp2

sha256: c60efc85424e3dd136e7d6988c4e967546285d597d3483609e18e79276041872

AppliStandard_ID6_Cl-V.mp2

sha256: c23ea0e33b2ff65e407f375fceb66e7a17e1bf71bd4ab5f51d3734ab13f8f270

AppliStandard_ID6_Na-S.mp2

sha256: c9d32cebcbfababc247f76f026275dc276cc7fe292fe768ce82fa35b2df0eea36

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AppliStandard_ID7_Cl-V.mp2

sha256: af23da3e1fd35c1b4a784ac13f33c2ef33e847c6170dbb509b2e0ef9da42b5f1

AppliStandard_ID6_Ni-Ag.mp2

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AppliStandard_ID7_Na-S.mp2

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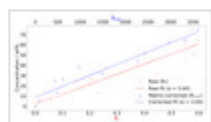
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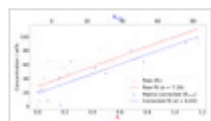
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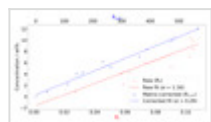
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Calibration_Al.png

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Calibration_Cl.png

sha256: f740072dd651125bd9998ba85b5bff82edfd34bf02c8c15b21e22cb776eaa17d

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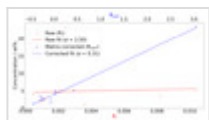
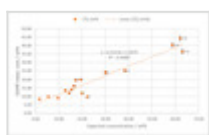


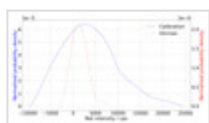
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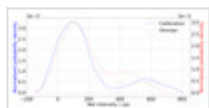
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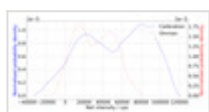
KDE_Comparison_Cl.png

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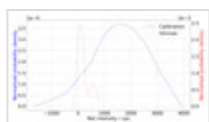
KDE_Comparison_Ti.png

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KDE_Comparison_Al.png

sha256: 9a8aeac5461248a7c76421bf5906542fc235df1f96ab27c8405db513781fd706



KDE_Comparison_Condition_Cl-V.png

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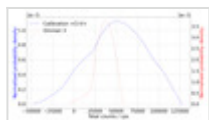
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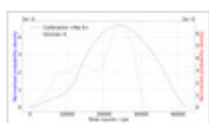
Created by: James Bird

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KDE_Comparison_Condition_Na-S.png

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Link: <https://frankel-elab.manchester.ac.uk/experiments.php?mode=view&id=75>