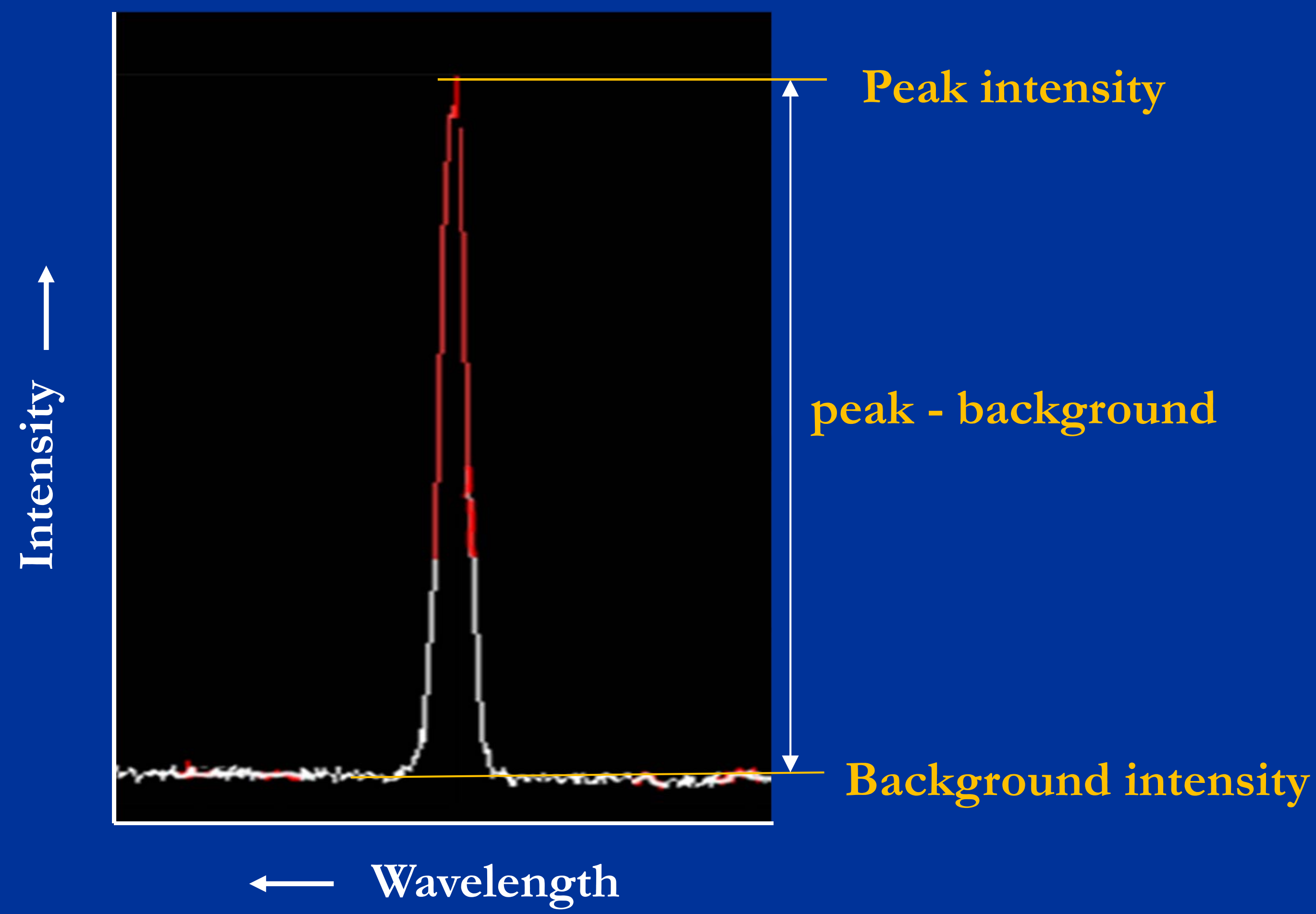
Quantitative Electron Microprobe Analysis

Wavelength Dispersive X-ray Spectrometry (WDS)

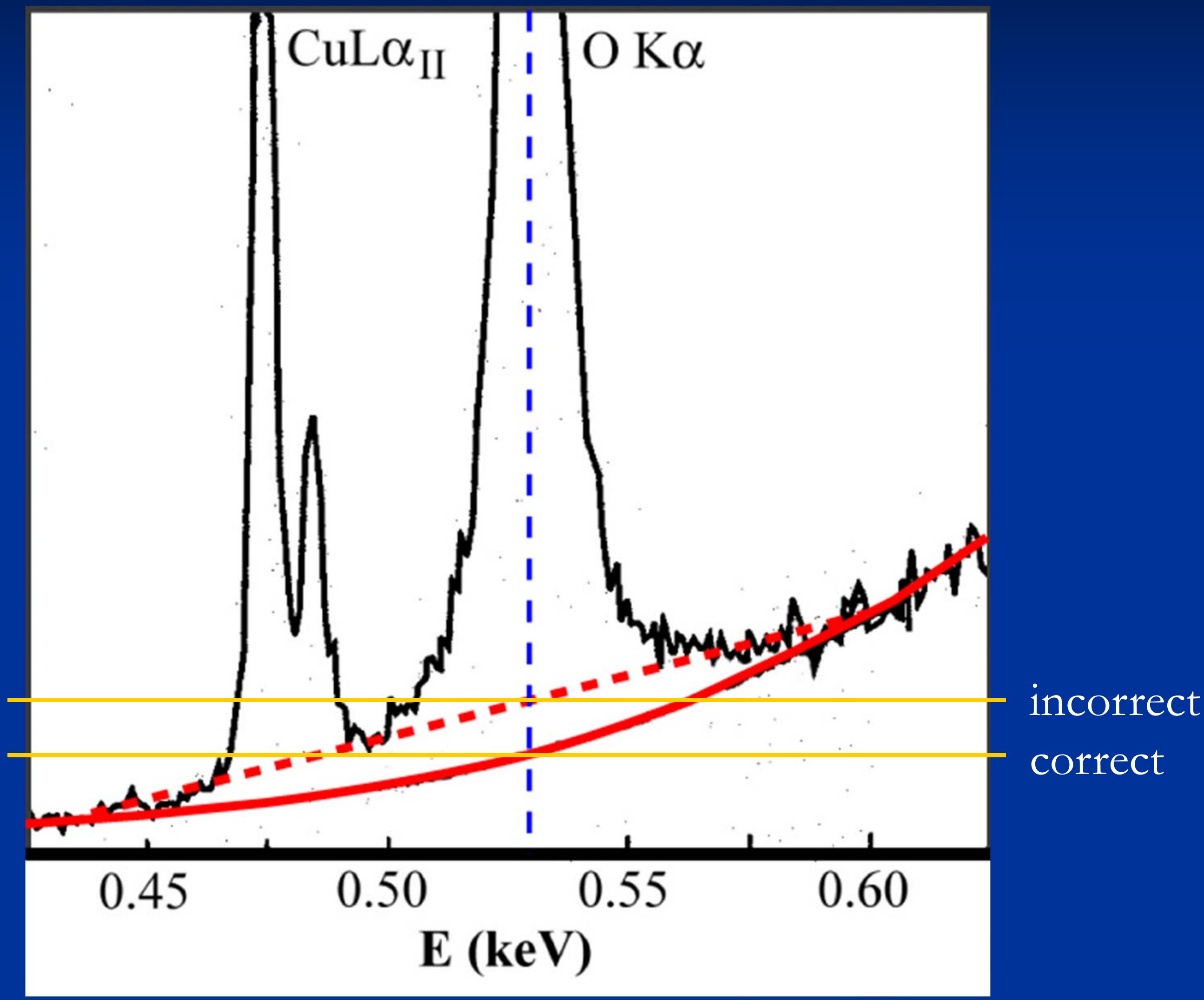
Goal: Measurement of concentration of elements in a microscopic volume

WDS: X-ray intensity measurement



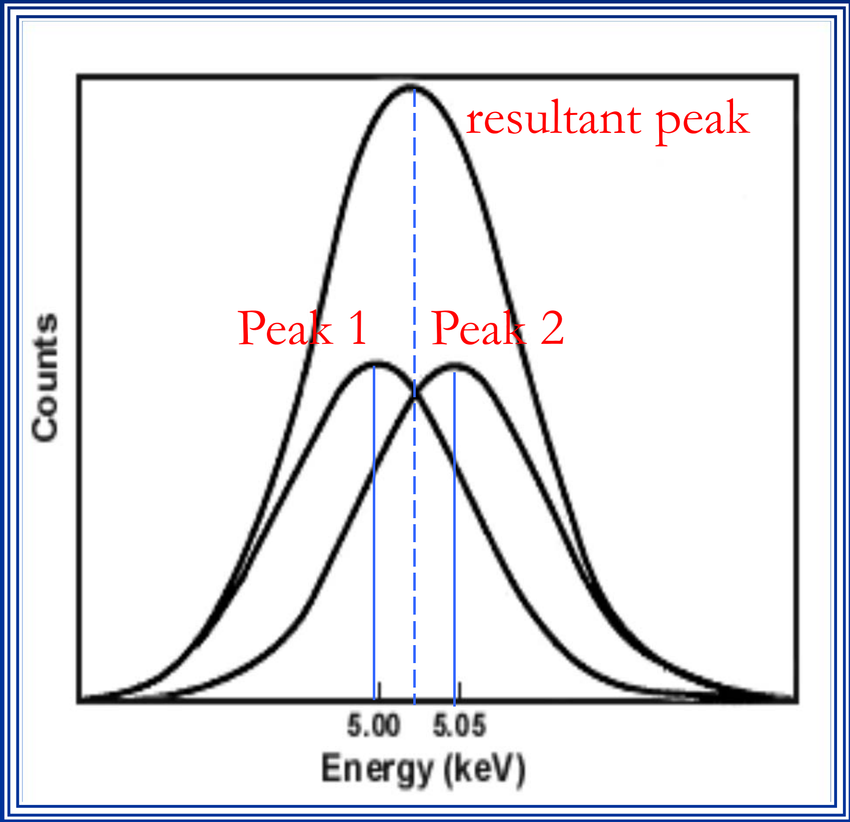
peak minus background intensity

Background modeling in X-ray spectra



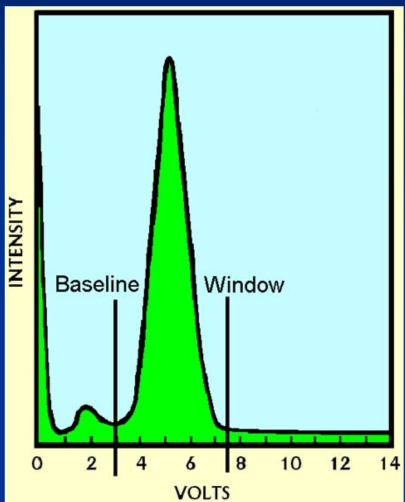
A polynomial fit to the background may be more accurate

Peak overlap in X-ray spectra

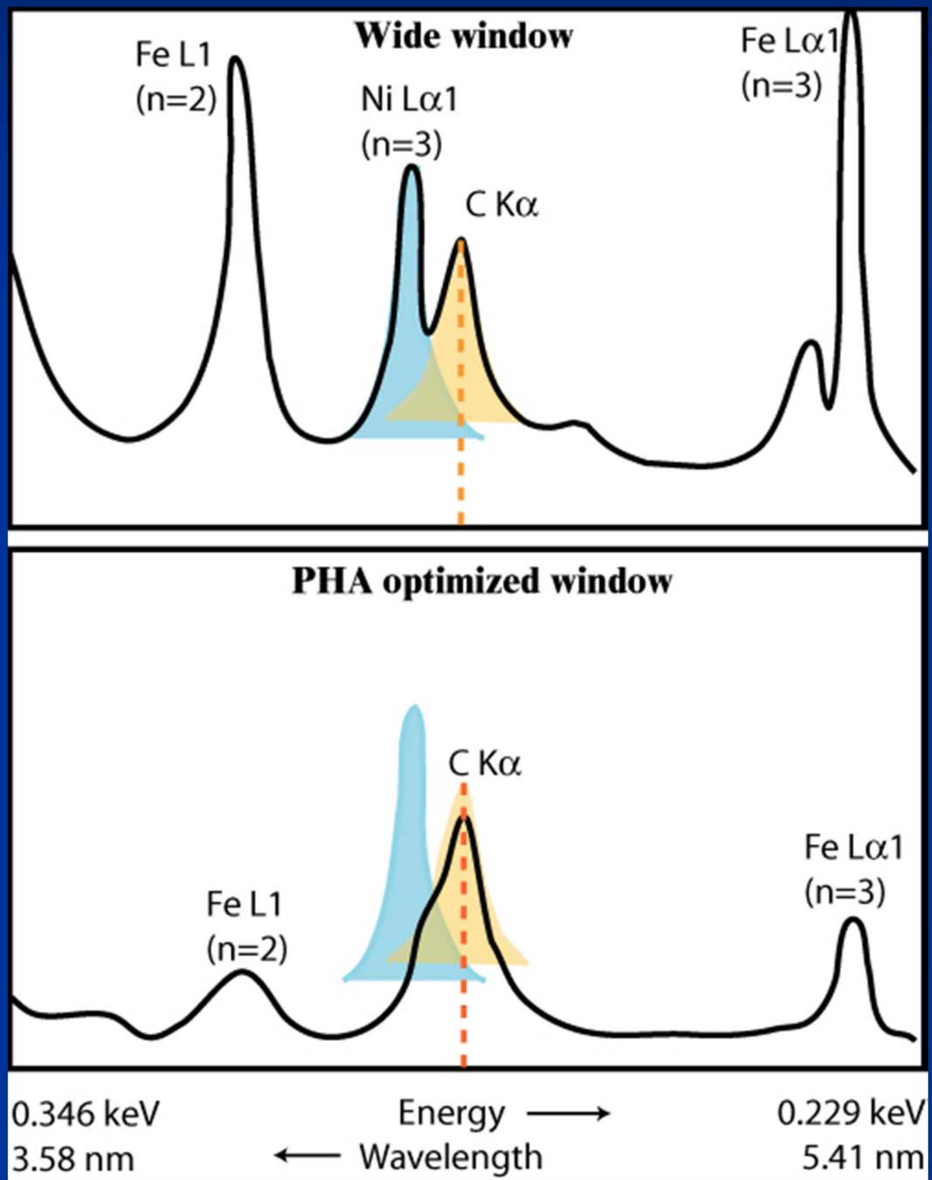


Overlap between Peak 1 and Peak 2 results in a broad single peak

WDS detector optimization with pulse height analysis (PHA)



SCA scan



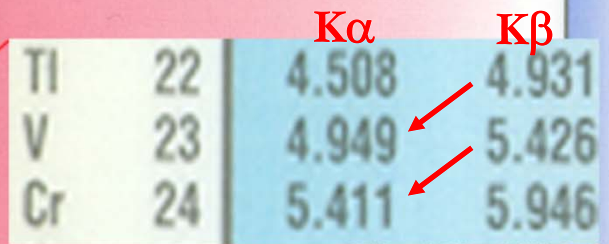
wide window

PHA optimized window

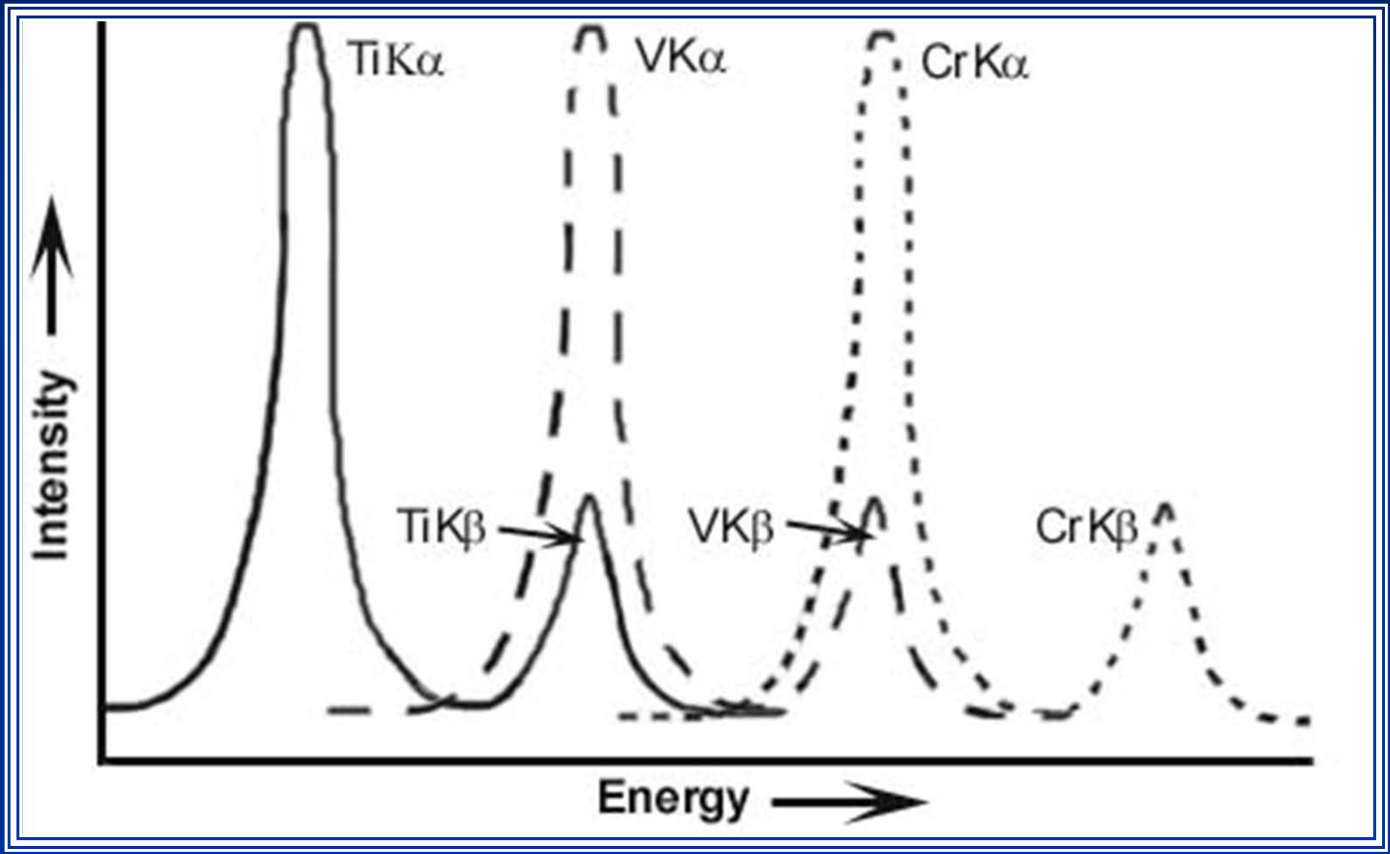
lower background and less peak overlap

Peak overlap: Kα and Kβ energies (keV) of Ti, V and Cr

Element	Atm. No.	Kα	Kβ( )	K_{ab}	Lα	$L\beta_1$(*)	$L\beta_2$(*)	$L\gamma_2$(*)	L_{IIIab}	L_{IIab}	L_{Iab}	M
Li	3	0.052		0.055								
Be	4	0.109		0.112								
B	5	0.183		0.192								
C	6	0.277		0.284								
N	7	0.392		0.400								
O	8	0.525		0.532								
F	9	0.677		0.687								
Ne	10	0.848		0.867								
Na	11	1.041		1.071								
Mg	12	1.253		1.303								
Al	13	1.486		1.560								
Si	14	1.739		1.840								
P	15	2.013	2.139(4)	2.143								
S	16	2.307	2.465(7)	2.470					0.136	0.164	0.193	
Cl	17	2.621	2.815(5)	2.819					0.202	0.203	0.238	
Ar	18	2.957	3.190(10)	3.202					0.245	0.247	0.287	
K	19	3.312	3.589(10)	3.607					0.294	0.297	0.341	
Ca	20	3.690	4.012(10)	4.037	0.341				0.349	0.353	0.399	
Sc	21	4.088	4.460(13)	4.488	0.395				0.406	0.411	0.462	
Ti	22	4.508	4.931(13)	4.964	0.452				0.454	0.460	0.530	
V	23	4.949	5.426(13)	5.463	0.511				0.512	0.519	0.604	
Cr	24	5.411	5.946(12)	5.988	0.573				0.599	0.593	0.742	
Mn	25	5.894	6.489(13)	6.536	0.637				0.639	0.650	0.762	
Fe	26	6.398	7.057(13)	7.110	0.705				0.707	0.721	0.849	
Co	27	6.924	7.648(13)	7.708	0.776				0.779	0.794	0.929	
Ni	28	7.471	8.263(13)	8.330	0.851				0.853	0.870	1.015	
Cu	29	8.040	8.904(13)	8.979	0.930				0.933	0.953	1.100	
Zn	30	8.630	9.570(13)	9.660	1.012				1.022	1.045	1.198	
Ga	31	9.241	10.262(14)	10.336	1.098				1.117	1.145	1.303	
Ge	32	9.874	10.978(14)	11.102	1.188				1.217	1.249	1.413	
As	33	10.530	11.722(15)	11.862	1.282				1.323	1.358	1.529	
Se	34	11.207	12.494(16)	12.652	1.419				1.434	1.474	1.652	
Br	35	11.907	13.286(16)	13.468	1.480				1.553	1.599	1.781	
Kr	36	12.631	14.107(16)	14.322	1.586				1.677	1.729	1.916	
Rb	37	13.373	14.956(16)	15.200	1.694				1.806	1.866	2.063	
Sr	38	14.140	15.830(16)	16.104	1.806				1.941	2.008	2.217	
Y	39	14.931	16.731(17)	17.035	1.922				2.079	2.154	2.376	
Zr	40	15.744	17.660(18)	17.996	2.042	2.124(45)			2.222	2.305	2.541	
Nb	41	16.581	18.729(8)	18.984	2.166	2.257(45)			2.370	2.464	2.710	0.355
Mo	42	17.441	19.599(17)	20.001	2.293	2.394(45)			2.523	2.627	2.880	0.331
Tc	43	18.325	20.608(16)	21.044	2.424	2.536(45)			2.677	2.794	3.055	
Ru	44	19.233	21.646(16)	22.116	2.558	2.683(45)			2.837	2.966	3.232	0.461
Rh	45	20.165	22.712(16)	23.216	2.696	2.834(40)	3.001(25)		3.002	3.144	3.416	0.496
Pd	46	21.121	23.806(17)	24.344	2.838	2.990(40)	3.171(25)		3.172	3.330	3.607	0.532
Ag	47	22.101	24.928(17)	25.512	2.984	3.150(40)	3.347(25)		3.350	3.525	3.807	0.568
Cd	48	23.106	26.081(18)	26.711	3.133	3.316(42)	3.528(25)		3.537	3.727	4.018	0.606
In	49	24.136	27.260(18)	27.937	3.286	3.487(75)	3.713(17)		3.730	3.939	4.237	
Sn	50	25.191	28.467(19)	29.190	3.443	3.662(75)	3.904(17)		3.928	4.157	4.464	0.691
Sb	51	26.271	29.396(19)	30.481	3.604	3.843(75)	4.100(17)		4.132	4.381	4.698	0.733
Te	52	27.468	30.974(19)	31.811	3.769	4.029(75)	4.301(17)		4.341	4.612	4.939	0.778
I	53	28.607	32.272(19)	33.167	3.937	4.220(75)	4.507(17)		4.558	4.853	5.191	
Xe	54	29.774	33.600(20)	34.590	4.109	4.420(50)	4.720(20)		4.781	5.103	5.452	
Cs	55	30.968	34.960(20)	35.987	4.286	4.619(50)	4.935(20)		5.011	5.357	5.720	
Ba	56	32.188	36.354(21)	37.452	4.465	4.827(50)	5.156(20)		5.246	5.622	5.995	0.972
La	57	33.436	37.771(21)	38.934	4.650	5.041(50)	5.383(20)		5.483	5.888	6.267	0.833
Ce	58	34.714	39.223(21)	40.453	4.839	5.261(50)	5.612(20)		5.723	6.160	6.547	0.883
Pr	59	36.020	40.771(21)	42.002	5.033	5.488(50)	5.849(20)		5.962	6.438	6.833	0.929
Nd	60	37.355		43.574	5.229	5.721(50)	6.088(20)		6.208	6.722	7.128	0.978
Pm	61	38.718		45.198	5.432	5.960(50)	6.338(20)		6.459	7.013	7.434	
Sm	62	40.111		46.849	5.635	6.204(50)	6.586(20)		6.716	7.312	7.747	1.081
Eu	63				5.845	6.455(50)	6.842(20)		6.979	7.618	8.059	1.131
Gd	64				6.056	6.712(50)	7.102(20)		7.242	7.930	8.385	1.185
Tb	65				6.272	6.977(50)	7.365(20)		7.514	8.251	8.715	1.240
Dy	66				6.494	7.246(50)	7.634(20)		7.880	8.582	9.050	1.293
Ho	67				6.719	7.524(50)	7.910(20)		8.066	8.915	9.398	1.347
Er	68				6.947	7.809(50)	8.188(20)		8.356	9.260	9.756	1.405
Tm	69				7.179	8.100(50)	8.467(20)	9.424(5)	8.648	9.615	10.119	1.462
Yb	70				7.414	8.400(50)	8.757(20)	9.778(5)	8.942	9.974	10.489	1.521
Lu	71				7.654	8.708(50)	9.038(20)	10.142(6)	9.247	10.343	10.872	1.581
Hf	72				7.898	9.021(50)	9.346(20)	10.514(10)	9.556	10.734	11.272	1.644
Ta	73				8.145	9.342(50)	9.650(20)	10.893(10)	9.875	11.130	11.680	1.709
W	74				8.396	9.671(50)	9.960(20)	11.284(10)	10.198	11.537	12.098	1.774
Re	75				8.651	10.008(50)	10.274(20)	11.683(10)	10.529	11.953	12.529	1.842
Os	76				8.910	10.354(50)	10.597(20)	12.093(10)	10.866	12.379	12.969	1.914
Ir	77				9.174	10.706(50)	10.919(20)	12.510(10)	11.210	12.818	13.421	1.977
Pt	78				9.441	11.069(50)	11.249(20)	12.940(10)	11.560	13.270	13.880	2.048
Au	79				9.712	11.440(50)	11.583(20)	13.379(10)	11.919	13.734	14.351	2.121
Hg	80				9.987	11.821(50)	11.922(20)	13.828(10)	12.284	14.212	14.840	2.195
Ti	81				10.267	12.211(50)	12.270(20)	14.289(10)	12.658	14.698	15.340	2.267
Pb	82				10.550	12.612(50)	12.621(20)	14.762(10)	13.038	15.203	15.852	2.342
Bi	83				10.837	13.021(50)	12.978(20)	15.245(10)	13.424	15.717	16.373	2.419
Po	84				11.129	13.445(50)	13.338(20)	15.741(10)	13.817	16.244	16.935	
At	85				11.425	13.874(50)	14.065(10)	16.249(10)	14.215	16.784	17.490	
Rn	86				11.725	14.313(50)	14.509(10)	16.768(10)	14.618	17.337	18.058	
Fr	87				12.029	14.768(50)	14.448(20)	17.300(10)	15.028	17.904	18.638	
Ra	88				12.338	15.233(50)	14.839(20)	17.845(10)	15.441	18.482	19.234	
Ac	89				12.650	15.710(50)	15.929(10)	18.405(10)	15.865	19.078	19.842	
Th	90				12.967	16.199(50)	15.621(20)	18.979(10)	16.296	19.679	20.459	2.991
Pa	91				13.288	16.699(50)	16.022(20)	19.565(10)	16.765	20.358	21.168	3.077
U	92				13.612	17.217(50)	16.425(20)	20.164(10)	17.162	20.943	21.766	3.165



Peak overlap corrections



$$I_{VK\alpha}^{corr} = I_{VK\alpha}^{meas} - \frac{I_{TiK\alpha}^{meas}}{I_{TiK\alpha}^{Ti-std}} I_{VK\alpha}^{Ti-std}$$

$$I_{CrK\alpha}^{corr} = I_{CrK\alpha}^{meas} - \frac{I_{VK\alpha}^{corr}}{I_{VK\alpha}^{V-std}} I_{CrK\alpha}^{V-std}$$

EPMA: Analytical procedure

- Sample preparation
- Qualitative analysis with EDS
- Standard intensity measurement (calibration)
- Measurement of X-ray intensities in the specimen
- Data reduction through matrix corrections

Sample preparation

- Sample cut and mounted in epoxy
- Polished first with coarse SiC paper, then with alumina grit slurry (final size: $\leq$0.25 μm) [1]
- Washed with water in ultrasonic cleaner [2]
- Dried with blow duster and air
- Carbon coated [3]

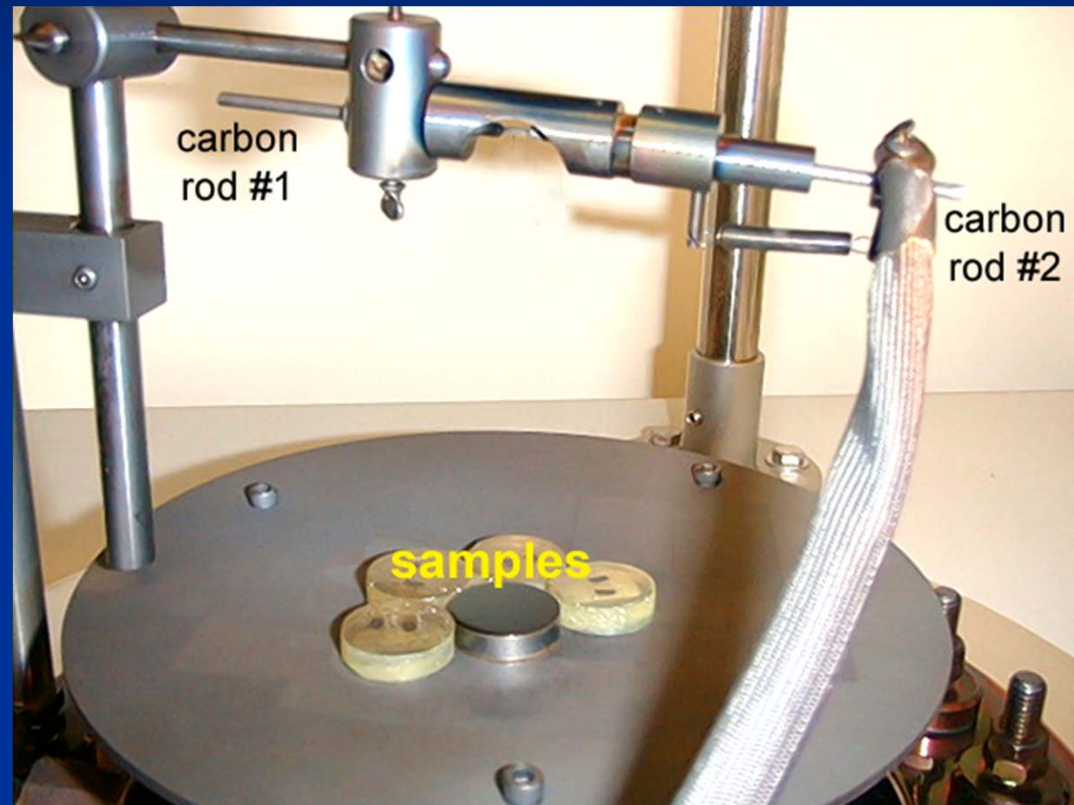
1: diamond paste or colloidal silica for some samples; dry polishing paper for water-soluble samples

2: ethanol may be used sparingly; cleaned with blow duster and cloth for samples that dissolve in water

3: for insulators; if standards are coated, however, all samples must be coated

Carbon coating

Vacuum vapor deposition



To monitor coat-thickness, a polished brass block is coated with the samples

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