

you-try-it09.xlsx

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For use with:

Brian M. Tissue, *Basics of Analytical Chemistry and Chemical Equilibria*, (John Wiley: New York, 2013).

<http://www.achem.org>

Worksheets in this file

notes

This page with background information.

9.A-AAS-Fe

Unknown analysis with a non-linear calibration curve.

9.A-AAS-Mn

Additional set of data with a non-linear calibration curve.

9.B-AES

Multi-element analysis and calculating limit of detection (LOD).

9.C-ICP-MS

Visualizing 3D data and performing semi-quantitative analysis.

9.C-data

Raw data for making the ICP-MS data plot.

Background

Refer to Chapter 9 in the text for equations and explanations.

Each worksheet has instructions in the blue shaded box.

For step-by-step help see [you-try-it-09guide.pdf](#).

You-Try-It 9.A-Fe Atomic Absorption Spectrometry (AAS)

Table 9.A.1(Fe) lists AAS results for a set of Fe standard solutions and two unknown solutions. The unknown sample preparation and the instrument settings are listed in the box to the right.

1. Plot the standard data.
2. Determine the Fe concentration in the wine using a linear fit to the standards.
3. Determine the Fe concentration in the wine using a polynomial fit to the standards.
4. Assuming that the polynomial fit gave the correct answer, determine the error in the result using the linear fit.
5. Calculate the expected absorbance for the wine + spike.
Determine if a bias occurs for the Fe concentration due to a matrix effect.

Table 9.A.1(Fe)

run	[Fe] (ppm)	A	std dev
1	0.00	0.000	0.003
2	2.00	0.226	0.001
3	4.00	0.404	0.001
4	6.00	0.540	0.001
5	8.00	0.638	0.002
6	10.0	0.700	0.001
7	wine	0.148	0.001
8	wine + spike	0.395	0.001

A white wine sample was boiled to remove the ethanol.
It is reconstituted to its original volume with 0.2 M HCl in deionized water.
The "wine+spike" is a duplicate solution that has 3.33 ppm of Fe added.
The standards are diluted from a stock solution with 0.1 M HCl in deionized water.

Perkin-Elmer AAnalyst 200 Atomic Spectrophotometer with Cu-Fe-Mn-Zn lamp
air-acetylene flame
wavelength: 248.33 nm
integration time: 3 s
replicates: 4

The absorbance, A , values are the average of four replicates.
Replicate measurements allow us to identify outliers due to carryover or change in aspiration rate.
The **std dev** column lists the standard deviations for the replicates.
The std dev values are low in this data set and we do not need to look at individual measurements.

Data recorded during Fall 2017 in CHEM 4124 laboratory (M.Z.) at Virginia Tech.

You-Try-It 9.A-Mn Atomic Absorption Spectrometry (AAS)

Table 9.A.1(Mn) lists AAS results for a set of Mn standard solutions and two unknown solutions. The unknown sample preparation and the instrument settings are listed in the box to the right.

1. Plot the standard data.
2. Determine the Mn concentration in the wine using a linear fit to the standards.
3. Determine the Mn concentration in the wine using a polynomial fit to the standards.
4. Assuming that the polynomial fit gave the correct answer, determine the error in the result using the linear fit.
5. Calculate the expected absorbance for the wine + spike.
Determine if a bias occurs for the Mn concentration due to a matrix effect.

Table 9.A.1(Mn)

run	[Mn] (ppm)	A	std dev
1	0.00	-0.008	0.005
2	1.00	0.261	0.001
3	2.00	0.494	0.003
4	3.00	0.693	0.003
5	4.00	0.845	0.002
6	5.00	0.953	0.003
7	wine	0.315	0.002
8	wine + spike	0.677	0.002

Note that spike amount is different than in the Fe data set.

A white wine sample was boiled to remove the ethanol.

It is reconstituted to its original volume with 0.2 M HCl in deionized water.

The "wine+spike" is a duplicate solution that has 1.67 ppm of Mn added.

The standards are diluted from a stock solution with 0.1 M HCl in deionized water.

Perkin-Elmer AAnalyst 200 Atomic Spectrophotometer with Cu-Fe-Mn-Zn lamp

air-acetylene flame

wavelength: 279.5 nm

integration time: 3 s

replicates: 4

The absorbance, A , values are the average of four replicates.

Replicate measurements allow us to identify outliers due to carryover or change in aspiration rate.

The **std dev** column lists the standard deviation (M.Z. CHEM 4124 data 2017)

The std dev values are low in this data set and we do not need to look at individual measurements.

Data recorded during Fall 2017 in CHEM 4124 laboratory (M.Z.) at Virginia Tech.

You-Try-It 9.B Atomic Emission Spectrometry (AES)

Table 9.B.1 lists emission intensities for a series of standard solutions of metal analytes.

Table 9.B.2 lists repetitive measurements of a validation standard that is 0.100 ppm of each metal.

1. Plot the standard data in Table 9.B.1.
2. Plot the Ba standard data in Table 9.B.2.
3. Find the average and standard deviation of the replicate measurements in Table 9.B.3.
Use these results to determine the LOD for each metal.

Table 9.B.1. ICP Atomic Emission of Standard Solutions

Wavelength	Metal	Standard Concentration (ppm)				
		0.00	0.20	0.40	1.00	2.00
267.716	Cr	20.44	6854	14840	36670	73900
220.353	Pb	-8.4	348.7	1327	3283	6545
213.857	Zn	111.5	8221	15900	40370	81720
324.752	Cu	1665.64	41930	82550	202400	402400
259.939	Fe	180.88	9480	36580	90790	182700
257.610	Mn	-22.66	85370	173000	427400	854900

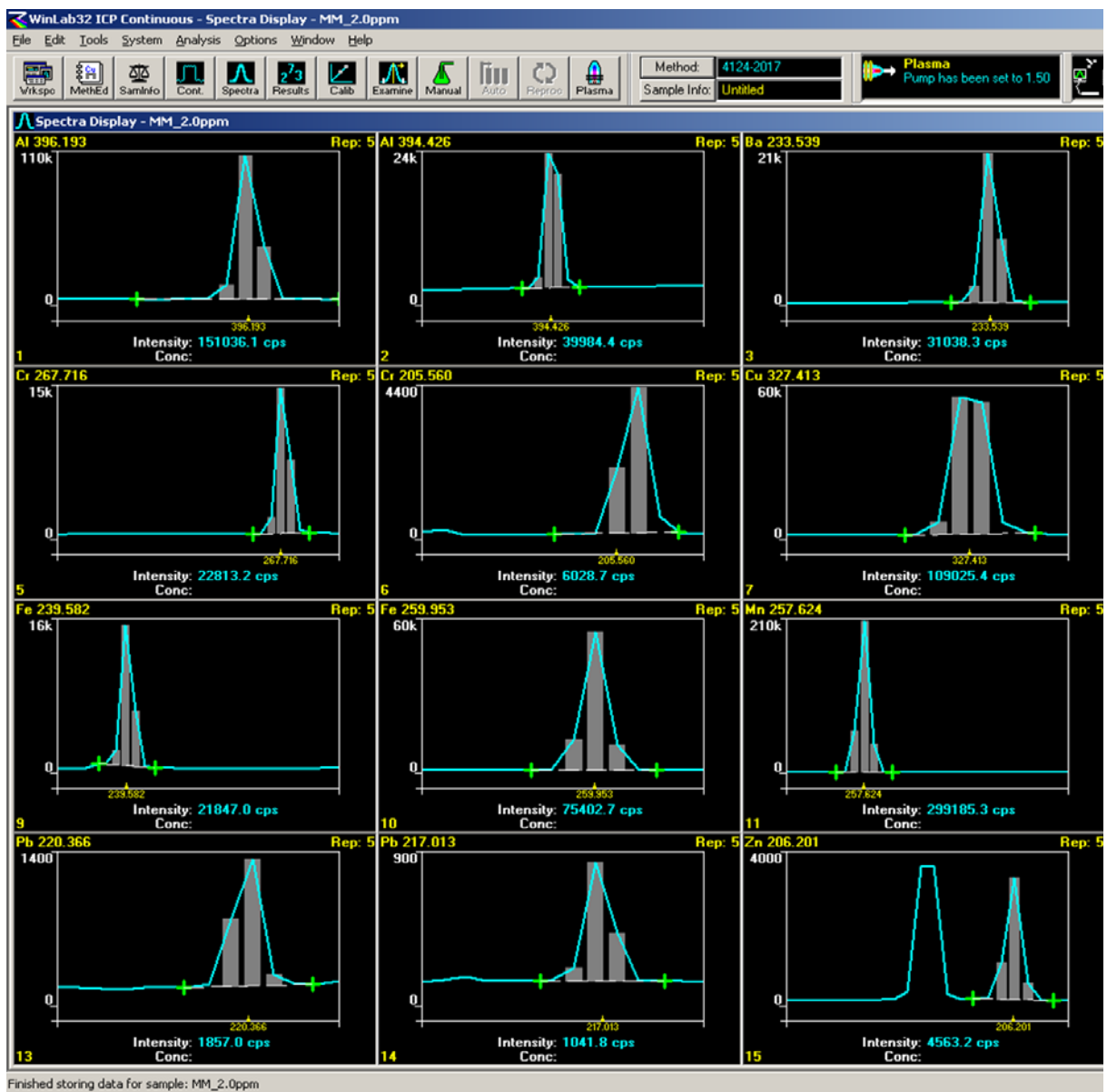
Table 9.B.2. ICP Atomic Emission of Standard Solutions

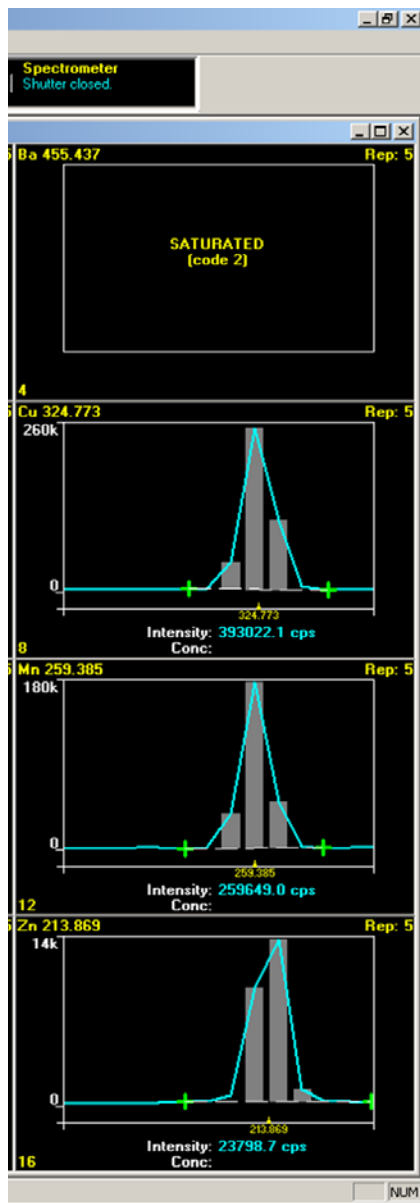
Wavelength	Metal	Standard Concentration (ppm)				
		0.00	0.20	0.40	1.00	2.00
283.560	Cr	1013.64	6469.9	18692.9	34306.7	68167.9
3274.393	Cu	516.8	23545.6	89451.7	207391.1	414704.7
233.520	Ba	21.1	3339	11781.7	31952.3	54655.2
455.403	Ba	-1522.38	546254.8	2158510.5	4652390.6	sat'd

Table 9.B.3. ICP Atomic Emission of 0.100 ppm Validation Standard

Run #	Pb (220.35)	Zn (213.86)	Ba (233.52)	Ba (455.40)
1	333	4064	3223	476122
2	333	4128	3311	463503
3	311	3999	3094	467987
4	377	4027	3293	475400
5	355	4188	3104	455123
6	366	3822	3299	476104

Sample spectra in a multielement analysis. Two spectral lines were measured for each element.





You-Try-It 9.C Inductively Coupled Plasma Mass Spectrometry (ICP-MS)

The 9.C-data worksheet contains LA-ICP-MS data for a melt inclusion in a geologic sample.

1. Create surface plots for the Al and Fe data that is in the 9.C-data worksheet.
2. Use the semi-quantitative sensitivities in the 9.C-data worksheet to calculate the fractional amounts of Al and Fe both within and outside of the melt inclusion.

The data set in the 9.C-data worksheet is taken from the sample data bundled with the "AMS (LA-ICPMS data reduction software)" package.

The readme.txt file states:

"This example was created by ablating a 8x8 array of
10 micron spots around a 40 micron melt inclusion."

Information available at: <https://www.geochem.geos.vt.edu/fluids/laicpms/ams.shtml>

The signal values for each spot are the average cps while the laser is on.

Row 5 in the 9.C-data worksheet gives the sensitivity in cps/ppm for selected elements.

These sensitivities are corrected for the isotopic abundance of each isotope.

LA-ICP-MS analysis of a melt inclusion.

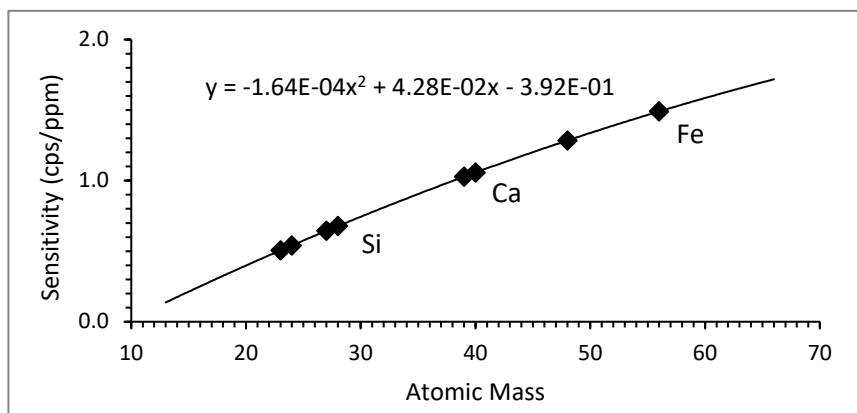
There are 64 measurements for an 8x8 array with 10 µm spatial resolution.

The signal values are average counts per second (cps) during the laser ablation.

major isotope	27	40	56						
Sensitivity (cps/ppm)	0.6440	1.0576	1.4905						
	Al	Ba	Ca	Dy	Eu	Fe	Gd	Hf	
	6764.8	0.0	14653.2		0.9	0.0	110861.3	0.0	0.0
	6871.7	0.2	14969.6		2.0	0.1	111137.6	0.0	0.0
	6544.6	0.2	14594.2		0.0	0.0	109937.0	0.0	0.0
	6894.1	0.0	14728.6		0.9	0.0	111371.1	0.3	0.0
	6631.8	0.0	14325.0		0.8	0.0	110956.2	1.0	1.1
	6695.2	0.2	14860.2		0.8	0.0	110247.3	0.0	0.0
	6732.3	0.2	15213.4		1.2	0.0	111249.4	0.2	1.0
	6699.1	0.2	16211.5		0.0	0.1	109866.2	0.0	0.0
	7024.2	0.0	15210.5		1.3	0.1	109576.9	0.0	0.0
	6944.3	0.0	15660.8		0.5	0.0	109763.2	0.3	0.4
	6792.7	0.3	14864.3		0.7	0.1	110429.1	0.0	0.8
	6893.8	0.0	16051.0		0.4	0.0	110113.5	1.0	0.0
	7010.3	0.2	15568.5		0.0	0.0	111878.1	0.2	0.8
	7085.6	0.2	15501.2		0.9	0.0	112312.8	1.7	0.7
	6846.6	0.0	14924.3		0.4	0.0	110887.9	0.0	0.4
	6876.1	0.0	15461.8		0.2	0.0	111634.4	0.0	0.4
	7442.0	0.0	15266.1		0.9	0.0	109634.1	0.0	0.8
	6958.8	0.0	16070.2		0.9	0.0	111312.2	0.3	0.0
	6953.7	0.0	15843.2		1.4	0.7	111441.8	0.0	0.0
	18549.7	123.6	18363.9		2.4	0.0	99371.6	2.1	0.8
	34327.3	286.2	22745.0		0.9	0.3	82995.4	0.0	3.8
	25954.3	205.9	19748.0		0.0	0.2	91396.9	3.2	0.6
	11139.3	46.2	16640.6		0.0	0.1	106218.8	2.3	0.5
	7593.3	9.0	16252.1		0.4	0.1	110904.9	0.1	0.0
	7134.7	0.0	15212.2		0.5	0.0	109417.4	0.0	0.4
	7584.3	1.7	16447.7		0.5	0.0	111315.9	0.0	0.4
	15033.2	77.6	18124.0		0.0	0.0	102384.0	0.0	1.2
	80780.2	900.0	36022.8		4.5	0.8	50827.5	0.3	5.4
	90069.7	934.4	38982.1		4.1	0.5	41728.1	0.5	4.8
	93386.6	1039.1	40756.0		7.7	0.5	40856.0	9.4	2.8
	65802.0	606.6	31412.4		2.9	0.8	66166.7	2.0	2.2
	11562.0	42.0	17354.9		0.9	0.1	110214.7	0.0	0.4
	7993.1	6.3	16339.6		0.0	0.0	111764.3	0.0	0.0
	7662.1	5.2	16287.7		0.0	0.1	111545.4	0.0	0.0
	34463.4	283.6	23158.2		0.9	0.3	84613.7	0.0	1.6
	84542.5	816.5	37848.2		6.4	0.7	47196.4	7.9	5.0
	93082.8	989.8	39637.7		4.3	0.7	42566.6	4.1	3.8
	88892.9	914.3	38609.9		3.9	1.2	40740.1	5.2	7.6
	90579.6	958.9	37452.1		6.8	1.2	44884.5	3.1	4.5
	14034.6	70.9	17716.2		2.0	0.3	106476.1	1.2	0.0
	8272.6	10.7	16176.3		0.9	0.0	112262.1	0.7	0.4
	7752.0	6.7	15736.3		0.9	0.0	111148.8	0.0	0.4
	19381.3	161.1	19207.1		0.0	0.0	101533.6	1.3	0.0
	79864.2	760.5	35532.7		3.8	0.7	53735.3	5.0	2.7
	91959.9	966.5	40471.5		6.5	0.2	42096.5	2.7	6.2
	92997.8	1032.2	39902.5		10.2	2.0	40766.4	3.2	3.8
	52704.7	528.1	27012.7		2.5	0.2	74218.7	2.8	2.8
	11233.2	41.4	18100.9		0.0	0.0	112600.9	1.2	0.0
	8220.7	11.7	16706.8		1.1	0.0	111486.9	0.0	0.4

7736.2	2.9	17152.4	0.9	0.0	112606.3	0.0	0.0
7985.7	11.0	16045.0	1.3	0.0	114014.7	0.0	0.8
22575.7	135.5	20906.7	3.1	0.1	100586.0	0.0	0.9
34783.4	312.5	23613.8	5.4	0.2	91174.5	0.6	0.6
28668.7	225.3	21905.6	2.8	0.0	96045.6	0.7	3.7
9579.5	21.2	17462.4	0.0	0.1	114094.7	0.0	0.0
8072.9	5.7	16966.2	0.9	0.1	113930.0	1.0	0.4
7911.5	2.1	17244.7	1.4	0.0	116855.7	1.9	0.0
7652.8	3.9	16825.0	0.0	0.0	115068.8	0.0	0.4
8085.4	5.1	16736.4	0.9	0.0	118054.3	0.0	0.0
7937.3	12.4	16213.9	1.4	0.1	114559.7	0.2	0.0
7999.8	5.9	17366.3	0.0	0.0	117219.3	0.3	0.0
7761.8	7.5	16697.4	2.1	0.0	114839.9	0.0	0.0
8042.6	0.3	17803.9	1.4	0.1	117990.6	1.1	0.0
7454.5	2.1	16961.8	1.4	0.0	115400.9	0.0	0.0

Plot of sensitivity versus atomic mass.



	39 1.0278		24 0.5407		23 0.5056					
Ho	K	La	Mg	Mn	Na	Nb	Nd	P	Pb	
	0.1	0.0	0.0	150925.4	2629.3	187.3	0.0	0.8	254.3	0.0
	0.0	3.3	0.2	155502.4	2715.2	309.5	0.0	0.1	272.3	0.0
	0.2	32.9	0.0	152792.5	2613.9	369.7	0.3	0.0	261.2	0.0
	0.0	0.0	0.3	153604.3	2686.0	240.2	0.9	0.2	238.1	0.0
	0.2	6.6	0.0	151948.6	2637.6	238.6	0.0	0.2	267.4	0.3
	0.1	70.1	0.0	152562.8	2626.9	199.5	0.0	0.1	286.1	1.6
	0.1	67.6	0.0	148444.3	2680.8	268.1	0.3	0.6	274.4	0.6
	0.3	142.4	0.0	153439.4	2640.9	174.1	0.0	0.0	263.0	1.3
	0.2	160.3	0.0	152016.5	2546.7	287.4	0.0	0.1	285.6	0.0
	0.1	90.9	0.0	152632.1	2585.9	321.1	0.6	0.0	212.0	0.0
	0.1	0.0	0.2	151946.5	2608.5	217.3	1.2	0.0	268.0	0.0
	0.0	49.7	0.0	156778.0	2573.7	291.6	0.3	0.1	254.4	0.0
	0.2	218.1	0.2	152419.0	2703.2	424.6	1.0	0.0	211.2	0.0
	0.1	83.2	0.2	156318.2	2729.6	290.5	0.3	0.1	225.3	1.4
	0.0	85.5	0.0	154865.7	2708.9	416.3	0.0	0.1	266.1	0.1
	0.2	77.1	0.0	153407.5	2634.1	214.7	0.0	0.0	176.0	0.6
	0.2	61.2	0.2	152171.2	2614.4	177.7	0.0	0.8	284.2	0.0
	0.1	114.4	0.0	153875.2	2632.9	321.9	0.0	0.0	253.9	0.0
	0.2	206.3	0.0	150867.5	2619.2	423.8	0.0	0.8	206.1	0.0
	0.2	3223.2	2.1	136583.3	2373.1	3521.7	0.7	0.9	244.5	1.4
	0.1	7320.1	7.6	105867.1	1952.2	7994.3	1.6	3.1	291.5	4.6
	0.1	5151.0	1.9	114257.9	2027.7	5452.4	1.5	3.4	159.8	3.3
	0.7	1363.8	1.6	148876.9	2533.8	1609.3	0.0	0.3	291.7	0.0
	0.3	293.7	0.2	156028.7	2615.6	568.4	0.0	0.0	245.9	0.4
	0.1	124.1	0.0	150951.0	2616.6	317.6	0.0	0.2	246.3	0.0
	0.1	225.5	0.2	152942.7	2555.6	468.4	0.0	0.2	220.3	0.0
	0.3	2438.7	1.5	139845.9	2401.8	2823.3	0.0	2.0	209.6	1.0
	1.5	16973.6	18.1	32254.0	909.9	18020.0	7.2	15.5	270.8	8.7
	0.7	18492.0	15.2	13444.1	608.4	20187.3	8.5	13.2	251.7	11.0
	1.1	19279.0	19.3	10785.1	630.5	20073.4	6.5	23.1	353.5	11.9
	1.0	13376.2	13.0	65859.9	1228.7	14303.9	2.6	8.9	323.0	7.6
	0.0	1235.3	0.9	148937.7	2623.4	1494.3	1.0	2.1	237.9	1.6
	0.0	282.9	0.0	154770.7	2649.1	457.2	0.0	0.0	238.7	0.0
	0.3	151.2	0.0	157768.8	2609.2	648.8	0.3	0.2	220.5	0.0
	0.6	6999.6	4.2	111367.4	1890.5	7717.6	2.9	0.8	278.0	2.3
	0.8	17082.3	15.5	26268.3	813.7	18487.6	5.6	11.9	353.1	9.6
	0.7	19279.9	21.0	12686.1	628.4	20911.3	6.6	20.2	384.1	12.2
	2.6	18480.0	14.3	11283.3	587.0	20429.3	4.8	21.8	307.3	11.2
	0.9	19099.0	17.1	21321.0	759.3	20376.2	6.5	13.7	264.7	10.2
	0.5	1661.7	1.3	146025.2	2476.5	2132.5	1.1	1.6	251.3	1.9
	0.0	333.2	0.2	154259.6	2612.0	594.5	0.0	0.2	180.6	1.0
	0.2	220.2	0.0	154791.4	2652.2	397.4	0.0	0.0	202.8	0.8
	0.0	3282.4	2.2	135261.8	2332.9	3658.4	2.6	2.8	290.4	0.7
	1.3	16196.7	13.8	37127.4	930.5	17151.7	4.9	15.9	267.4	10.1
	1.4	19450.9	15.2	12022.2	656.0	20509.0	6.2	9.0	273.5	10.4
	0.8	19183.0	16.8	10160.3	534.9	20808.3	6.2	22.0	266.5	11.6
	1.0	9947.8	7.5	87976.0	1603.6	10962.8	1.8	17.0	239.2	6.9
	0.3	1178.4	0.8	155846.3	2615.1	1363.7	0.4	2.3	239.1	0.6
	0.1	313.9	0.2	155943.3	2637.5	665.7	0.0	0.0	232.4	2.5

0.0	409.5	0.2	157993.8	2588.1	616.6	0.0	0.0	217.5	1.1
0.2	396.0	0.0	157892.6	2725.1	649.5	0.3	0.2	195.4	1.8
0.1	4060.9	2.7	132575.0	2297.5	4348.0	0.7	4.5	226.0	3.9
0.5	6473.3	3.9	114101.2	2024.1	7319.5	2.6	8.6	287.2	2.4
0.6	4962.8	3.1	124009.8	2229.0	5700.4	1.5	7.6	209.2	2.8
0.4	837.3	0.0	155467.5	2661.5	1219.7	0.0	0.0	189.4	1.4
0.0	374.6	0.2	159985.7	2701.0	707.7	0.3	0.0	169.7	0.9
0.2	179.5	0.4	159920.4	2683.1	573.6	0.3	0.0	211.6	2.1
0.3	139.2	0.2	157762.4	2687.7	508.2	0.6	0.0	210.9	1.6
0.0	104.1	0.4	162692.4	2745.6	631.2	0.3	1.5	170.1	1.8
0.1	144.4	0.4	158390.3	2686.4	594.7	0.0	0.2	151.2	0.9
0.2	202.8	0.5	161948.8	2740.7	561.5	1.0	0.8	210.6	0.5
0.0	141.5	0.0	158790.8	2692.9	468.3	0.3	0.2	204.3	0.5
0.1	240.0	0.4	163375.2	2722.4	642.7	0.0	0.8	165.6	1.6
0.3	117.6	0.0	162257.2	2656.3	551.0	0.3	0.0	160.6	0.0

	28 0.6778			48 1.2845		
Si	Sm	Sr	Ti	Y	Yb	
265037.1	0.0	0.1	1565.3	4.1	0.2	
260886.1	0.0	0.1	1547.0	3.8	0.4	
264287.4	0.0	0.2	1513.8	3.7	1.9	
262469.1	0.5	0.0	1526.5	4.3	1.8	
264511.0	0.0	0.0	1502.9	4.8	0.9	
264011.9	0.0	0.0	1527.2	5.1	1.0	
266300.3	0.4	0.2	1494.4	5.0	0.7	
262810.8	0.0	0.4	1340.4	6.7	1.0	
264243.5	1.1	0.8	1549.6	4.8	1.3	
263448.9	0.5	0.0	1619.8	5.3	1.4	
264326.7	0.5	0.1	1556.8	4.9	0.2	
259984.9	0.0	0.1	1406.4	5.8	0.5	
262249.6	0.0	1.2	1475.7	5.7	0.2	
259007.9	1.1	0.4	1555.8	5.5	2.1	
261568.1	0.4	0.0	1430.7	6.0	1.0	
262190.0	0.4	0.0	1373.1	6.0	1.0	
263624.8	0.0	0.3	1733.9	6.8	1.1	
261189.8	0.5	0.0	1615.2	7.5	0.0	
263705.8	0.0	0.1	1402.5	5.2	0.2	
265865.2	0.0	17.4	2243.0	8.9	1.9	
277287.0	1.2	50.9	2757.5	9.9	0.5	
277964.2	3.8	32.3	2879.9	9.8	1.3	
262421.4	0.6	7.0	1760.5	7.7	1.7	
258906.6	0.4	0.8	1534.0	6.9	1.8	
264957.7	0.0	0.5	1693.7	3.5	0.5	
261089.0	0.0	0.7	1550.8	4.9	1.2	
265881.4	1.2	15.6	2031.6	9.6	0.8	
290029.5	7.9	144.9	5875.3	28.5	4.7	
297597.7	3.0	158.3	6260.5	34.9	4.0	
295415.1	7.6	177.2	6398.0	31.0	2.2	
276413.0	2.1	103.5	4471.3	22.5	1.4	
259286.4	1.2	7.2	1751.3	9.5	1.1	
259121.5	0.5	1.7	1403.8	7.9	0.4	
257168.1	2.1	0.1	1513.0	5.9	1.5	
271820.5	0.0	48.0	3077.0	17.9	1.1	
291809.2	3.8	147.6	6110.7	31.6	2.9	
293476.3	4.6	170.4	6280.4	36.3	3.6	
300878.8	4.9	157.2	6370.7	26.8	3.4	
289558.0	4.9	161.7	6266.7	31.8	2.8	
260850.4	0.0	12.1	1668.1	8.1	2.2	
258979.7	0.5	2.0	1486.5	6.2	1.3	
260260.3	0.0	0.5	1318.2	5.8	0.2	
264044.8	0.0	16.1	2366.9	8.2	0.6	
286575.8	2.7	131.3	6027.7	30.3	3.3	
295009.5	4.9	169.2	6272.2	31.1	4.4	
296107.2	5.6	170.7	7040.0	29.5	4.8	
273220.5	3.3	76.3	3971.2	18.5	2.4	
252333.6	0.0	4.2	1868.8	3.2	0.2	
257748.6	0.4	1.7	1469.6	4.1	1.7	

255621.3	1.2	1.7	1505.7	6.9	1.1
255311.7	0.0	1.3	1463.6	8.3	1.1
262037.8	0.6	28.8	2300.0	6.4	0.6
265862.2	0.8	46.0	2755.8	17.7	1.7
263560.9	0.3	36.6	2878.7	12.6	2.0
254194.0	0.0	4.3	1525.1	5.4	0.9
253081.6	0.0	0.8	1464.4	7.5	1.1
251401.3	0.1	0.5	1585.8	5.4	0.2
254741.9	0.0	1.2	1547.8	7.0	1.1
248608.6	0.5	0.9	1730.8	5.7	1.6
254569.0	0.0	1.0	1730.8	7.5	2.4
249429.7	2.0	1.2	1563.0	5.4	0.2
254115.4	0.5	1.2	1521.4	7.4	1.5
247549.3	0.0	1.0	1532.4	7.3	0.2
251096.7	0.0	0.7	1686.2	5.9	0.8