ICP Guide

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Sample Preparation

The following considerations must be made:

- What is the *exact* nature of the problem?
- *Why* is ICP needed?
- *How* will the results be used following completion of this portion of the project?
- What is it that you want to accomplish?
- What is the purpose?
- What is the current situation or state of affairs?
- What is taking place that you need to understand, prevent, or improve?
- What decisions will be made based upon the data?

Preliminary sample issues:

The sample - the quantity, as well as the chemical and physical composition and properties must be considered. Any handling, safety, or stability issues must be noted. All safety data pertaining to the handling and disposal of the sample should be gathered. The sample matrix may be a suspected carcinogen, teratogen, mutagen, poison, or corrosive. Inhalation of fumes or dust may result in short as well as long term health effects. It may be flammable, explosive or irritating to the senses. Special storage, preservation or handling conditions may have to be observed. It may contain one or more functional groups that would require certain precautions when treated with chemicals during the preparation.

The analytes - the identity, chemical form(s) and approximate concentration level(s) of the analytes. Often times, only the identity of the analytes is known in which case a hypothesis of potential or likely forms should be postulated. What are the expected concentration levels?

- The selection of a preparation method is dependent upon:
  - the analyte(s)
  - the analyte concentration level(s)
  - the sample matrix
  - the required sample size

<u>Acid Digestion of Inorganic Samples:</u>

A liquid that is atomized using a nebulizer is the most reliable and convenient form of sample introduction to the inductively coupled plasma. If your sample is a liquid or soluble in a liquid then the sample preparation is relatively simple. If the sample is not soluble in a solvent then sample preparation techniques such as acid digestion, ashing, or fusion are required. The solvent used exclusively for the preparation techniques to be discussed is water.

Acid digestions have the advantage of retaining 'volatile' analytes (reflux condenser is needed for some trace elements) and the disadvantage of being tedious when large sample sizes are required. They are ideal if the sample size is < 1 gram. The acid that this guide will focus upon is nitric acid (HNO_3). Nitric acid is popular because of its chemical compatibility, oxidizing ability, availability, purity, and low cost.

Nitric acid* is used primarily in the preparation of inorganic sample types.[++] It is a very useful component in the destruction of organics but cannot by itself completely decompose organic matrices.

*All reference to HNO_3 will mean 69% 'concentrated' nitric acid unless specified otherwise.

[++]The conventional meaning of inorganic is intended along with the presence of low molecular weight water soluble organic cmpds. and organometallic cmpds. containing relatively small molecular weight organic components.
The following is a summary of some common inorganic dissolutions using nitric acid:

- **Dilute 10 - 15 % aqueous dilution** - Alkaline earth oxides, lanthanide oxides, actinide oxides, Sc_2O_3, Y_2O_3, La_2O_3.
- **1:1 HNO3 + H2O** - V_2O_5, Mn oxides, CuO, CdO, Hg oxides Tl oxides, Pb oxides, Bi oxides, Cu^0, Zn^0, Cd^0, Hg^0, Pb^0.
- **Concentrated (69%) HNO_3** - Mn^0, Fe^0 (hot), Co^0, Ag^0, Pd^0 (hot), Se^0, As^0, Bi^0, Re^0.
- **1:3 HNO_3 + HCl** - Pt^0, Au^0, steel, Fe/Ni alloys, Cu alloys, Cr/Ni steel.
- **1:1:1 HNO_3 + HF + H_2O** - The metal and oxides of Ti, Zr, Hf, Nb, W, Sn, Al, Si, Ge, Sb, Te, As, Se, Mo and numerous alloys and oxide mixtures containing one or more of these elements.

The only major group of elements not listed above are the alkaline earths, of which are all water soluble.

Visit http://www.inorganicventures.com/sample-preparation for a guide to show how to prepare samples with specific elements.

Do a search for papers that include detailed methods/protocols on the preparation of your sample or a very similar sample for ICP-OES analysis.

User Responsibilities of Samples:

- You must prepare your own sample in your lab. We do not have sample preparation facilities.
- You must provide your own standards.
    - You want a true blank (nanopure H_2O is best.)
    - You want a blank of the digestion solution without having run your sample through it.
    - You want calibration standards appropriate to your sample and project. (Going to Sigma Aldrich and searching for 'ICP standards' will bring up 150 choices.)
- You must use your own Falcon tubes –
    - for your sample, use 15 mL Falcon tubes
    - for your standards, calibrations, and blanks, use 50 mL Falcon tubes
        - Label Lids! Standards, calibrations, and blanks can be reused.

Resources you may find helpful as you consider your experiment and preparation of your samples:

http://physics.nist.gov/PhysRefData/ASD/lines_form.html for a searchable database of atomic emission spectra by element

http://www.aip.org/avsguide/refguide/opticalwav.html for a database that can tell you what elements share a particular emission wavelength

http://www.inorganicventures.com/periodic-table for element specific commonly used emission lines and possible interferences with each. This section of their website seems to contain information on all elements which their element specific preparation discussions lack

http://www.inorganicventures.com/spectral-interference-types-avoidance-and-correction for a brief overview of the types of interferences to be anticipated and some ways of addressing them

Thank you to Spencer Williams for pointing these out to me.

The first meeting for ICP training will be discussing the above issues.

We will have two more trainings; first, to familiarize you with the instrument, and second, to do the first set of your samples.

Please subscribe to the ICP mailing list to receive status updates of the ICP in terms of downtime for maintenance and other items:

https://mailman1.u.washington.edu/mailman/listinfo/icp-oes

ICP-OES Detection Limits

From:

www.perkinelmer.com/atomicspectroscopy

ICP-OES Detection Limits in µg/L			
Element	**Limit**	**Element**	**Limit**
Ag	0.6	**Mo**	0.5
Al	1	**Na**	0.5
As	1	**Nb**	1
Au	1	**Nd**	2
B	1	**Ni**	0.5
Ba	0.03	**Os**	6
Be	0.09	**P**	4
Bi	1	**Pb**	1
Br		**Pd**	2
C		**Pr**	2
Ca	0.05	**Pt**	1
Cd	0.1	**Rb**	5
Ce	1.5	**Re**	0.5
Cl		**Rh**	5
Co	0.2	**Ru**	1
Cr	0.2	**S**	10
Cs		**Sb**	2
Cu	0.4	**Sc**	0.1
Dy	0.5	**Se**	2
Er	0.5	**Si**	10
Eu	0.2	**Sm**	2
F		**Sn**	2
Fe	0.1	**Sr**	0.05
Ga	1.5	**Ta**	1
Gd	0.9	**Tb**	2
Ge	1	**Te**	2
Hf	0.5	**Th**	2
Hg	1	**Ti**	0.4
Ho	0.4	**Tl**	2
I		**Tm**	0.6
In	1	**U**	10
Ir	1	**V**	0.5
K	1	**W**	1
La	0.4	**Y**	0.2
Li	0.3	**Yb**	0.1
Lu	0.1	**Zn**	0.2
Mg	0.04	**Zr**	0.5
Mn	0.1		

Preferred Wavelengths of Perkin-Elmer 8300

Element	State	Wavelenth (nm)	Preference Order	BEC (mg/L)	DL mg/L (W, P, &F)	Relative Sensitivity (B)	Signal/Noise (S & T)	Intensity (W)
Ag	I	328.068	1	0.26	0.007	170000	2040.2	4200
Ag	I	338.289	2	0.43	0.013	82000	1261.2	2200
Ag	II	243.778	3	4	0.12	4200	28.2	23
Ag	II	224.874	4		0.5	880		3
Al	I	396.153	1	0.95	0.028	64000	268.4	2050
Al	I	308.215	2	1.52	0.045	23000	79.2	780
Al	I	394.401	3	1.59	0.047	32000	145.8	1050
Al	I	237.313	4	1	0.03		28.8	130
Al	I	309.271	5	0.77	0.023		141	1400
Al	II	167.022	6	0.05				
Ar	I	420.069	1				297.2	750.1
Ar	I	363.268	2					23
As	I	188.979	1	4.55		2100		1.7
As	I	193.696	2	1.79	0.053	5800		6.5
As	I	197.197	3	2.56	0.076	4300		5.8
As	I	228.812	4	2.78	0.083	4000	16	36
Au	I	267.595	1	1.04	0.031	35000		620
Au	I	242.795	2	0.59	0.017	27000		500
Au	II	208.209	3	1.43	0.042	7600		30
B	I	249.677	1	0.19	0.0057	79000	233.9	1150
B	I	249.772	2	0.16	0.0048	160000	457.9	2200
B	I	208.889	3	0.4	0.012	33000	5.4	75
B	I	182.528	4					
B	I	208.957	5	0.33	0.01	39000	8.5	140
B	I	182.578	6					
Ba	II	233.527	1	0.13	0.004	150000	615	1150
Ba	II	455.403	2	0.04	0.0013	1400000	5295.1	43000
Ba	II	493.408	3	0.08	0.0023	430000	220.6	16000
Ba	II	230.425	4	0.14	0.0041	110000	370.6	800
Ba	II	413.065	5	1.1	0.032	32000	197.9	1200
Be	II	313.107	1	0.02	0.0007	1900000		41000
Be	II	313.042	2	0.01	0.0003	2900000		64000
Be	I	234.861	3	0.01	0.0003	1400000		11500

Element	State	Wavelenth (nm)	Preference Order	BEC (mg/L)	DL mg/L (W, P, &F)	Relative Sensitivity (B)	Signal/Noise (S & T)	Intensity (W)
Bi	I	223.061	1	1.15	0.034	13000	36.9	66
Bi	II	190.171	2	10		850		
Bi	I	306.766	3	2.5		14000	51.8	380
Bi	I	222.821	4	2.78	0.083	6500	12	21
Bi	I	206.17	5	2.86	0.085	4600		6.5
Br	I	700.57	1					
C	I	193.03	1	1.49		5900		1.8
Ca	II	317.933	1	0.33	0.01	99000	6.1	1600
Ca	II	315.887	2	0.73		40000		950
Ca	II	393.366	3	0.01	0.0002	8300000	2674.8	450000
Ca	II	396.847	4	0.02	0.0005	3100000	1179.3	230000
Ca	I	422.673	5	0.33	0.01	180000		2900
Ca	I	227.546	6					5
Cd	I	228.802	1	0.09	0.0027	150000	664.5	1400
Cd	II	214.44	2	0.08	0.0025	170000	128.5	720
Cd	II	226.502	3	0.11	0.0034	140000	293.1	1000
Cd	I	361.051	4	7.69	0.23	6100	50	135
Ce	II	413.764	1	1.61	0.048	20000	82.2	
Ce	II	418.66	2	1.75	0.052	35000	87.5	1400
Ce	II	413.38	3	1.67	0.05	27000	83.4	1400
Ce	II	393.108	4	2	0.06	29000	16.2	
Ce	II	456.236	5	2.44	0.073	21000	24.8	800
Ce	II	401.239	6	2.5	0.075	27000	55.4	850
Cl	I	725.67	1					
Cl	I	782.139	2					
Co	II	228.616	1	0.23	0.007	65000	92.3	570
Co	II	238.892	2	0.2	0.006	72000	169.9	900
Co	II	230.786	3	0.32	0.0097	64000	61.9	400
Co	II	236.38	4	0.37	0.011	42000	77.5	400
Co	II	231.16	5	0.43	0.013	40000	64.6	320
Cr	II	267.716	1	0.24	0.0071	130000	411.7	2200
Cr	II	205.56	2	0.2	0.0061	64000	14.2	220
Cr	II	283.563	3	0.24	0.0071	170000	294.6	3700
Cr	II	284.325	4	0.29	0.0086	120000	196.9	2600
Cr	I	357.869	5	0.77	0.023	79000	284.5	2000
Cr	II	206.158	6	0.24	0.0071	55000	11.5	170
Cr	I	257.717	7					

Element	State	Wavelenth (nm)	Preference Order	BEC (mg/L)	DL mg/L (W, P, &F)	Relative Sensitivity (B)	Signal/Noise (S & T)	Intensity (W)
Cs	I	455.531	1	3333.33	100			0.7
Cs	I	459.32	2					0.2
Cu	I	327.393	1	0.32	0.0097	91000	467.5	4000
Cu	I	324.752	2	0.18	0.0054	180000	868	8000
Cu	II	224.7	3	0.26	0.0077	71000	161.3	350
Cu	II	213.597	4	0.4	0.012	35000	6.5	120
Cu	I	222.778	5	0.53	0.015	36000	52.3	130
Cu	I	221.459	6	0.77	0.023	21000	21.1	75
Dy	II	353.17	1	0.33	0.01	160000		
Dy	II	394.468	2	1.05	0.031	73000		
Dy	II	396.839	3	1.05	0.031	53000		
Dy	II	407.796	4	1.33	0.04	54000		
Er	II	337.271	1	0.34	0.01	150000		
Er	II	349.91	2	0.59	0.017	130000		
Er	II	339.2	3	1.06	0.031	45000		
Eu	II	381.967	1	0.09	0.0027	680000		
Eu	II	412.97	2	0.14	0.0043	280000		
Eu	II	393.048	3	0.19	0.0057	270000		
Fe	II	238.204	1	0.15	0.0046	110000	171.7	3500
Fe	II	239.562	2	0.17	0.0051	89000	130.3	2400
Fe	II	259.939	3	0.21	0.0062	170000	419.8	7000
Fe	II	234.349	4	0.34	0.01	50000	52.1	1100
Fe	II	234.83	5	0.43	0.013		22.9	
Fe	II	238.863	6	0.5	0.015	26000	35.1	700
Fe	II	273.955	7	0.67	0.02	61000	66	1600
Ga	I	417.206	1	2.22	0.066	29000		900
Ga	I	294.364	2	1.56	0.046	25000		650
Ga	II	209.134	3	9.09	0.272	1200		3
Gd	II	342.247	1	0.48	0.014	86000		
Gd	II	336.223	2	0.67	0.02	67000		
Gd	II	335.047	3	0.71	0.021	67000		
Gd	II	308.199	4	1.11	0.033	26000		
Ge	I	209.426	1	1.33	0.04	8600		35
Ge	I	265.118	2	1.61	0.048	15000		500
Ge	I	206.866	3	2	0.06	7500		21
Ge	I	303.906	4	3.45	0.103	9400		350

Element	State	Wavelenth (nm)	Preference Order	BEC (mg/L)	DL mg/L (W, P, &F)	Relative Sensitivity (B)	Signal/Noise (S & T)	Intensity (W)
Hf	II	277.336	1	0.53	0.015	46000		950
Hf	II	232.247	2	0.63	0.018	27000		230
Hf	II	264.141	3	0.63	0.018	51000		750
Hg	I	253.652	1	2.04	0.061	11000		230
Hg	II	194.168	2	0.83				22
Hg	I	404.656	3			1300		5
Hg	I	435.835	4	90.9	2.727	650		11
Hg	I	302.15	5	166.66	5	140		3.5
Hg	I	184.886	6					
Hg	I	546.074	7					5.5
Ho	II	345.6	1	1	0.0057	220000		
Ho	II	339.898	2	0.43	0.013	110000		
Ho	II	347.426	3	0.63	0.018	73000		
I	I	178.215	1	0.77				
I	I	206.188	2	4.3				
I	I	182.976	3	4.3				
I	I	206.163	4					
In	II	230.606	1	2.13	0.063	8200		80
In	I	325.609	2	4	0.12	8700		370
In	I	303.936	3	5	0.15	5300		240
In	I	451.131	4	6.25	0.187	7300		300
In	II	207.926	5	23.81	0.714	550		1
Ir	I	205.222	1	2.04	0.061	6400		1.2
Ir	II	224.268	2	0.91	0.027	20000		150
Ir	I	208.882	3	3.57	0.107	3700		9
Ir	I	237.277	4	8.33	0.25	3000		18
K	I	766.49	1	7.1				22
K	I	404.721	2	1428.57	42.857			0.8
La	II	408.672	1	0.33	0.01	210000		
La	II	379.478	2	0.33	0.01	150000		
La	II	407.735	3	0.48	0.014	110000		
La	II	384.902	4	0.83	0.025	60000		
Li	I	670.784	1	0.1		380000		12300
Li	I	610.362	2	1.5		34000		420
Li	I	460.286	3	28.57	0.857	1400		52
Li	I	413.256	4	250	7.5			

Element	State	Wavelenth (nm)	Preference Order	BEC (mg/L)	DL mg/L (W, P, &F)	Relative Sensitivity (B)	Signal/Noise (S & T)	Intensity (W)
Lu	II	261.542	1	0.03	0.001	670000		
Lu	II	291.139	2	0.21	0.0062	170000		
Lu	II	219.554	3	0.28	0.0083	50000		
Mg	I	285.213	1	0.05	0.0016	750000	123.4	17500
Mg	II	279.077	2	1	0.03	29000	8.5	830
Mg	II	280.271	3	0.01	0.0003	3500000	803.3	83000
Mg	II	279.553	4	0.01	0.0001	5800000	1569.2	99000
Mn	II	257.61	1	0.05	0.0014	580000	831	18000
Mn	II	259.372	2	0.05	0.0016	480000	672.2	13000
Mn	II	260.568	3	0.07	0.0021	340000	493.4	9900
Mn	II	294.92	4	0.26	0.0077	150000	176.6	8600
Mn	II	293.305	5	0.45	0.013	85000	79.9	2700
Mn	I	279.482	6	0.42	0.012	72000	78.7	2700
Mn	I	403.075	7	1.47	0.044	44000	110.1	2100
Mo	II	202.031	1	0.26	0.0079	46000	10.8	155
Mo	II	203.845	2	0.42	0.012	30000	7.2	90
Mo	II	204.597	3	0.42	0.012	30000	7.2	100
Mo	II	281.616	4	0.48	0.014	46000	244.6	2400
Na	I	589.592	1	2.33	0.069	22000		300
Na	I	330.237	2	62.5	1.875	640		8
Na	I	588.995	3	0.99	0.029	43000		650
Nb	II	309.418	1	1.2	0.036	28000		2500
Nb	II	313.079	2	1.67	0.05	23000		2200
Nb	II	269.706	3	2.33	0.069	16000		960
Nb	II	292.781	4	2.5	0.075	17000		870
Nd	II	406.109	1	3.23	0.096	23000		
Nd	II	401.225	2	1.69	0.05	33000		
Nd	II	430.358	3	2.5	0.075	26000		
Nd	II	424.738	4	5.88	0.176	9600		
Nd	II	384.824	5	6.25	0.187			
Ni	II	231.604	1	0.53	0.015	27000	98.1	620
Ni	II	221.648	2	0.34	0.01	55000	61.8	520
Ni	I	232.003	3	0.5	0.015	35000	73.9	410
Ni	I	341.476	4	1.61	0.048	30000	277.5	1400
Ni	II	227.022	5	0.83	0.025	19000	35.7	240

Element	State	Wavelenth (nm)	Preference Order	BEC (mg/L)	DL mg/L (W, P, &F)	Relative Sensitivity (B)	Signal/Noise (S & T)	Intensity (W)
Os	II	228.226	1	0.02	0.0006	760000		310
Os	II	225.585	2	0.01	0.0004	1100000		520
Os	II	189.9	3	0.04	0.0012	250000		
Os	I	222.798	4	0.09	0.0027	200000		30
P	I	213.617	1	2.56	0.076	5400	5.5	29
P	I	214.914	2	2.56	0.076	5400	4.6	20
P	I	178.221	3					
P	I	177.434	4	0.36				
Pb	II	220.353	1	1.43	0.042	13000	24.3	150
Pb	I	217	2	3.03	0.09	4900	11.5	50
Pb	I	261.418	3	4.35	0.13	4900	71.1	180
Pb	I	283.306	4	4.76	0.142	6600	34.9	340
Pb	I	224.688	5	11.11	0.333	1600		8.5
Pb	I	405.781	6	9.09	0.272	6800	17.3	320
Pd	I	340.458	1	1.47	0.044	26000		1000
Pd	I	363.47	2	1.82	0.054	22000		600
Pd	I	324.27	3	2.56	0.076	12000		420
Pd	II	248.892	4	3.45	0.103	6400		95
Pr	II	390.844	1	1.23	0.037	37000		
Pr	II	414.311	2	1.25	0.037	33000		
Pr	II	422.293	3	1.59	0.047	46000		
Pt	I	265.945	1	2.7	0.081	8900		230
Pt	II	214.423	2	1	0.03	14000		95
Pt	I	299.797	3			5700		110
Pt	I	204.937	4	2.38	0.071	5100		7
Pt	I	193.7	5	4.55	0.136	2200		5
Pu	II	340.11	1		0.05			
Pu	II	363.221	2		0.025			
Pu	II	453.614	3		0.015			
Pu	II	476.717	4		0.118			
Rb	I	780.023	1					6
Rb	I	420.185	2	1250	37.5			1
Re	II	197.248	1	0.2		54000		85
Re	II	227.525	2	0.23	0.006	94000		650
Re	I	204.908	3	2.63	0.078	4700		4.5

Element	State	Wavelenth (nm)	Preference Order	BEC (mg/L)	DL mg/L (W, P, &F)	Relative Sensitivity (B)	Signal/Noise (S & T)	Intensity (W)
Rh	I	343.489	1	2	0.06	16000		710
Rh	II	233.477	2	1.49	0.044	14000		95
Ru	II	240.272	1	1	0.03	20000		320
Ru	I	349.894	2	3.7	0.111	13000		280
Ru	II	279.535	3	5.26	0.157	5300		
S	I	181.975	1					
S	I	180.669	2	0.32				
S		182.563	3					
S	I	189.965	4					
Sb	I	206.836	1	1.1	0.032	12000	5.2	33
Sb	I	217.582	2	1.47	0.044	10000	23.1	55
Sb	I	231.146	3	2.04	0.061	8200	30.9	70
Sb	I	252.851	4	3.57	0.107	4800	48.3	85
Sb	I	204.957	5	6.67	0.2	1800		3.7
Sb	I	203.977	6	15.15	0.454	800		1.3
Sc	II	361.383	1	0.05	0.0015	1200000	343.3	
Sc	II	357.253	2	0.07	0.002	940000	215.3	
Sc	II	424.683	3	0.09	0.0027	620000	423.5	
Sc	II	357.634	4	0.12	0.0037	410000	152.1	
Se	I	196.026	1	2.5	0.075	4300		10.5
Se	I	203.985	2	3.85	0.115	3200		8.5
Si	I	251.611	1	0.4	0.012	51000	287.9	850
Si	I	212.412	2	0.56	0.016	11000	12.5	90
Si	I	288.158	3	0.91	0.027	37000	56.8	720
Si	I	252.851	4	1.05	0.031	29000	102.3	280
Si	I	221.667	5	1.39	0.041	13000	18.4	75
Sm	II	359.26	1	1.45	0.043	36000		
Sm	II	442.434	2	1.82	0.054	25000		
Sm	II	388.529	3	2.78	0.083	31000		
Sm	II	428.079	4	2.33	0.069	18000		
Sm	II	363.429	5	2.22	0.066	29000		
Sn	II	189.927	1	0.83				5.5
Sn	I	235.485	2	3.23	0.096	6000	33.1	28
Sn	I	283.998	3	3.7	0.111	9000	46.9	90
Sn	I	242.17	4	5.26	0.157	3900	21.6	20

Element	State	Wavelenth (nm)	Preference Order	BEC (mg/L)	DL mg/L (W, P, &F)	Relative Sensitivity (B)	Signal/Noise (S & T)	Intensity (W)
Sr	II	407.771	1	0.01	0.0004	3300000	2846	120000
Sr	II	421.552	2	0.03	0.0008	2300000	1506.7	63000
Sr	I	460.733	3	2.27	0.068	17000	5.4	400
Sr	II	232.235	4	3.45	0.103	4900		14
Ta	II	226.23	1	0.83	0.025	20000		125
Ta	II	240.063	2	0.95	0.028	29000		380
Ta	II	233.198	3	1.04	0.031	14000		170
Ta	II	267.59	4	1.47	0.044	15000		380
Ta	II	248.87	5	1.85	0.055	12000		195
Ta	II	209.133	6	9.09				
Tb	II	350.917	1	0.77	0.023	79000		
Tb	II	384.873	2	1.85	0.055	29000		
Te	I	214.281	1	1.37	0.041	10000		25
Te	I	238.578	2	5.88	0.176	2900		14
Te	I	226.555	3	38.46	1.153	400		0.7
Th	II	283.73	1	2.17	0.065	20000		720
Th	II	401.913	2	2.78	0.083	21000		1070
Th	II	339.204	3	3.33	0.1	13000		550
Ti	II	334.94	1	0.13	0.0038	370000	313.7	11000
Ti	II	336.121	2	0.18	0.0053	220000	252.8	8800
Ti	II	337.279	3	0.22	0.0067	180000	198.1	6800
Ti	II	334.903	4	0.25	0.0075	130000	98.8	1800
Ti	II	368.519	5	0.38	0.011	190000	142.6	4200
Tl	II	190.801	1	1.35		7800		4
Tl	I	276.787	2	4	0.12	5400		120
Tl	I	351.924	3	6.67	0.2	7700		120
Tm	II	313.126	1	0.17	0.0052	180000		
Tm	II	346.22	2	0.27	0.0081	210000		
U	II	385.958	1	8.33	0.25	7900		300
U	II	367.007	2	10	0.3	7300		180
U	II	409.014	3	11.24	0.337	5700		170
U	II	393.203	4	12.2	0.365	5200		150
U	II	424.167	5	15.38	0.461	2600		105
V	II	290.88	1	0.29	0.0088	140000	43.3	3800
V	II	310.23	2	0.21	0.0064	170000	203.8	7000
V	II	309.31	3	0.17	0.005	220000	221.1	9400
V	II	292.402	4	0.25	0.0075	140000	110.5	
V	II	311.071	5	0.33	0.01	150000	205	5600
V	II	270.093	6	0.59	0.017	39000	129.4	780

Element	State	Wavelenth (nm)	Preference Order	BEC (mg/L)	DL mg/L (W, P, &F)	Relative Sensitivity (B)	Signal/Noise (S & T)	Intensity (W)
W	II	207.912	1	1	0.03	13000	8.4	30
W	II	224.876	2	1.49	0.044	9300	44.2	75
W	II	239.708	3	1.85	0.0055	15000	49.6	150
W	II	248.923	4	2.44	0.073	8000	91.5	150
Y	II	371.029	1	0.12	0.0035	380000		
Y	II	324.227	2	0.15	0.0045	180000	154	
Y	II	360.073	3	0.16	0.0048	290000	348	
Yb	II	328.937	1	0.06	0.0018	620000		
Yb	II	369.419	2	0.1	0.003	650000		
Yb	II	289.138	3	0.29	0.0086	130000		
Zn	II	206.2	1	0.2	0.0059	54000	16.6	185
Zn	I	213.857	2	0.06	0.0018	240000	257	1020
Zn	II	202.548	3	0.13	0.004	76000	17.3	215
Zn	I	334.501	4	4.55	0.136	9600	22	95
Zn	I	330.258	5	7.69	0.23	6200	10.6	50
Zr	II	343.823	1	0.24	0.0071	190000	267.4	6500
Zr	II	339.197	2	0.26	0.0077	230000	292.2	8000
Zr	II	257.139	3	0.32	0.0097	83000	182.2	1300
Zr	II	354.262	4				16.7	450
Zr	II	357.247	5	0.33	0.01	170000	179.7	3800

Key to Table:

Background Equivalent Concentration (BEC) Test: The BEC value is the concentration of an element which would produce the same emission intensity as the plasma background measured at the analyte wavelength. The BEC serves as an indication of instrument sensitivity.

DL mg/L (W, P & F) Radial Detection Limits (mg/L) Winge, Peterson, and Fassel, *Applied Spectroscopy* 33, p. 206, 1979

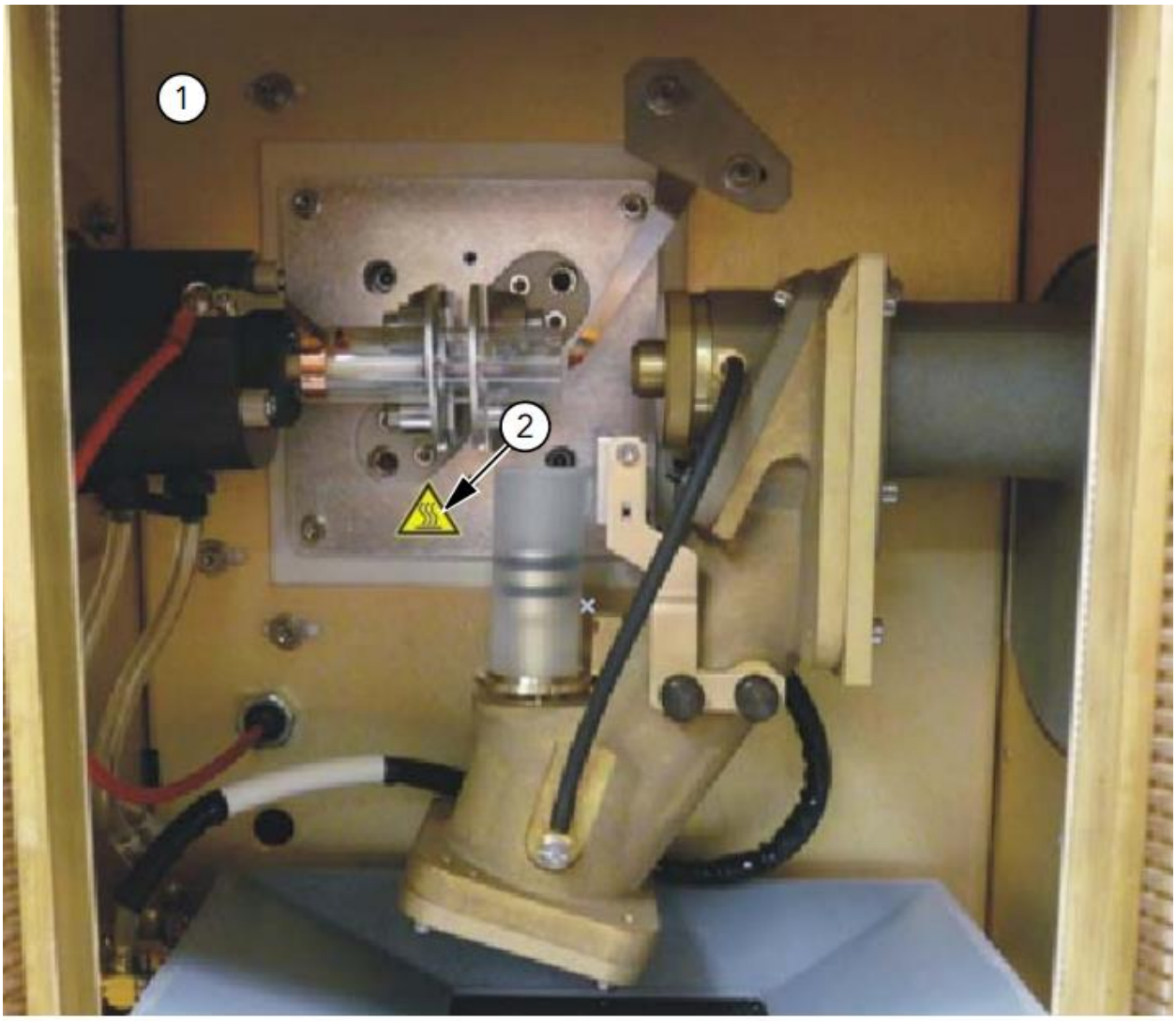
Rel Sens (B) Relative Sensitivity Boumans, *Line Coincidence Tables for ICP -AES*, Pergamon Press, 1980

Sig/Noise (S &T) Signal-to-Noise Ratio Schierle and Thorne, *Spectrochimica Acta* 50B, pp. 27-50, 1995

Intensity (W) Wohlers, *ICP Information Newsletter* 10, No.8, pp. 593-688, Jan 1985

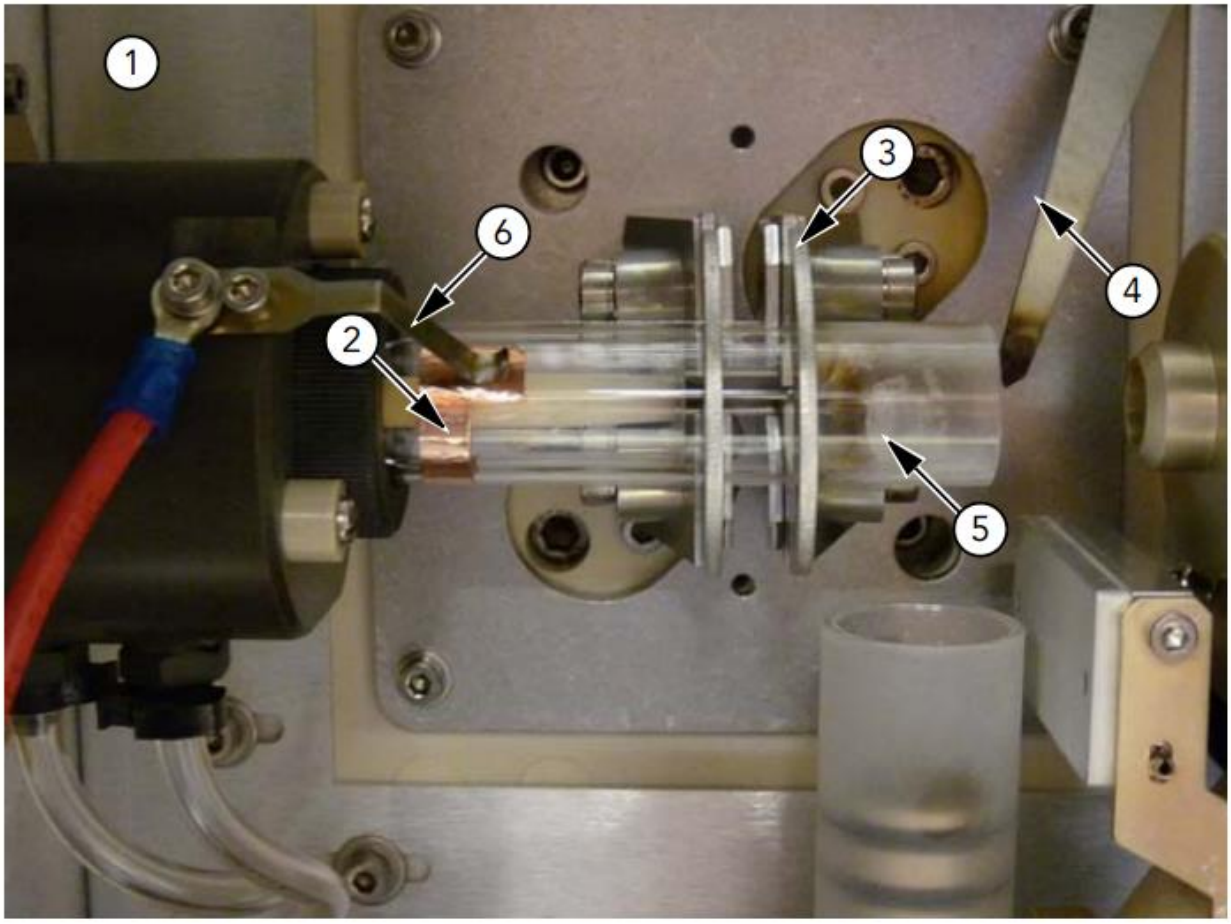
Spectrometer Information

Inside the Chamber



Location of warning labels in the sample compartment.

Item	Description
1	Inside Spray Chamber Compartment
2	Warning Label Hot Surface



Installing the torch into the sample compartment.

Item	Description
1	Front of Instrument Torch Compartment Door Open
2	Copper Foil Ignitor Contact
3	Plasma Induction Plates
4	Ground Pointer
5	Torch
6	Ignitor Contact Finger

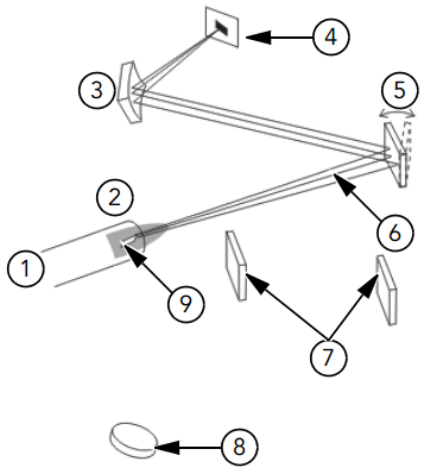
Plasma Viewing Configurations

The **Dual View (DV) instrument** can view the plasma either axially or radially. As shown in the next two diagrams of the transfer optics on the Dual View instrument, the torch is positioned horizontally in the sample compartment along the central axis of the spectrometer optics.

Changing from axial to radial viewing is a simple software command and is accomplished by computer control of a mirror located in the optical path.

Two toroidal mirrors image the plasma onto the entrance slit. The first mirror is computer-controlled and allows selection of radial viewing or axial viewing mode. For axial viewing, light emitted along the axis of the plasma is directed to the spectrometer optics. For radial viewing, the computer-controlled mirror is rotated slightly to capture light from the bottom of the plasma and direct it to the spectrometer optics.

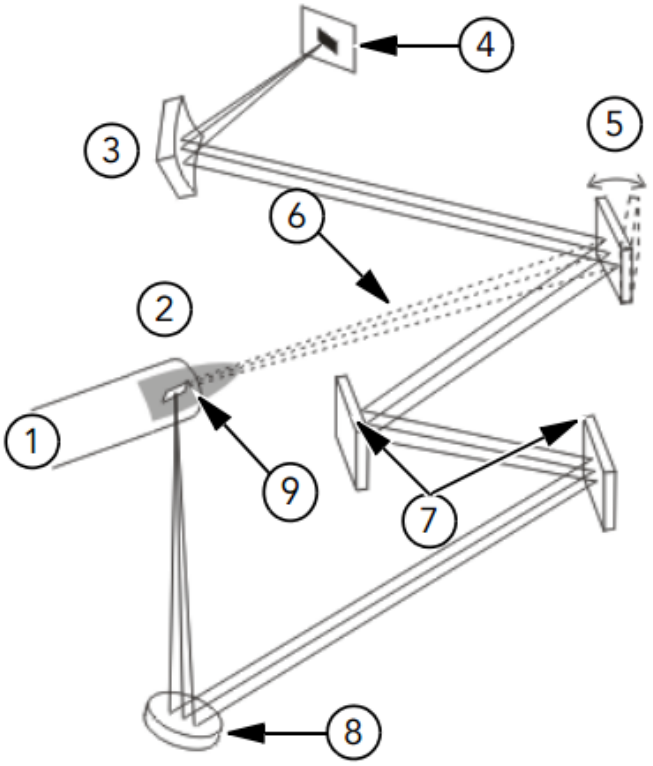
The computer-controlled mirror also adjusts plasma viewing in both the vertical and horizontal planes.



Transfer optics on the Dual View instrument shown in Axial viewing mode.

Item	Description
1	Torch
2	Plasma
3	Toroidal Mirror
4	Entrance Slit

Item	Description
5	Computer Controlled Toroidal Mirror in Radial Viewing Mode
6	Axial Viewing Mode (dotted Line)
7	Flat Mirrors
8	Spherical Mirror
9	Observation Zone

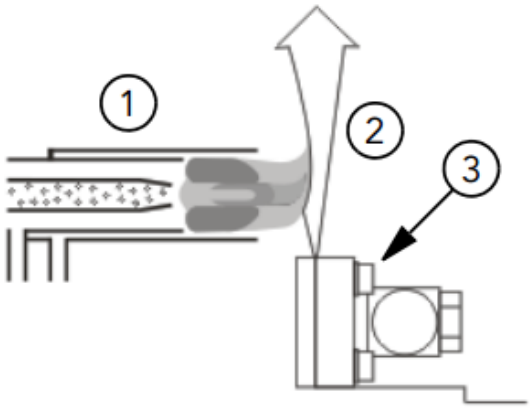


Transfer optics on the Dual View instrument shown in Radial viewing mode.

Item	Description
1	Torch
2	Plasma
3	Toroidal Mirror
4	Entrance Slit
5	Computer Controlled Toroidal Mirror in Radial Viewing Mode
6	Axial Viewing Mode (dotted Line)

Item	Description
7	Flat Mirrors
8	Spherical Mirror
9	Observation Zone

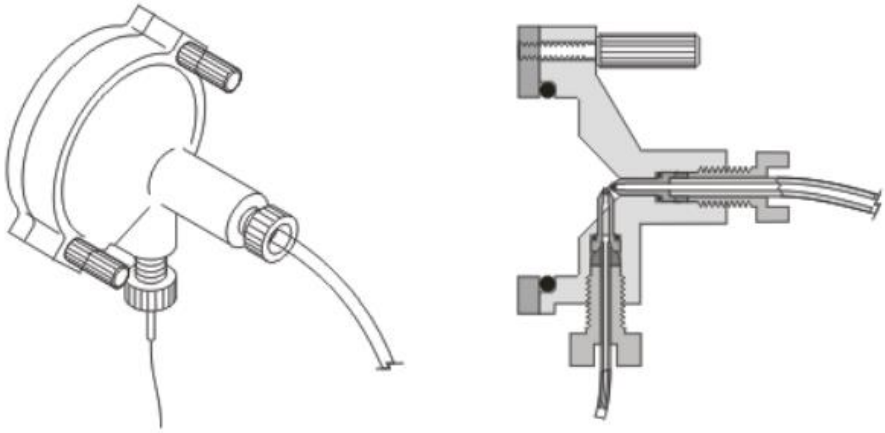
A flow of shear gas (compressed air or nitrogen) is directed at the plasma to "shear" off the tip of the plasma discharge. The shear gas minimizes the effects of selfabsorption by forcing cooler atoms from the tip of the plasma out of the optical path. The shear gas also keeps the purge window cool. The shear gas flow is turned on automatically when the plasma is ignited.



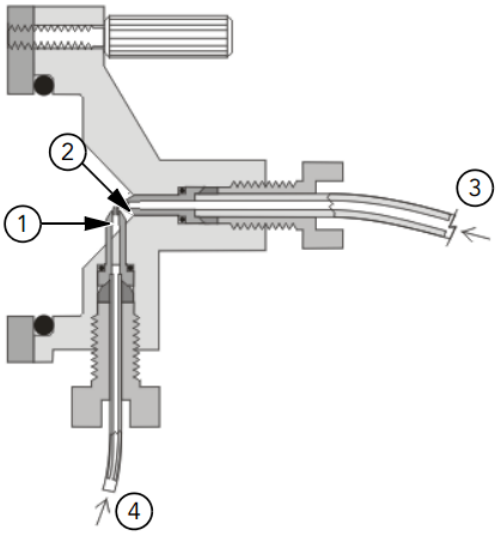
Use of shear gas.

Item	Description
1	ICP Torch
2	Shear Gas
3	Shear Gas Nozzle

Nebulizer Set Up



GemTip Cross-Flow nebulizer and end cap N0770546 (also shown in cross-sectional view).



Cross-section of GemTip Cross-Flow Nebulizer shown with Cross-Flow End Cap,

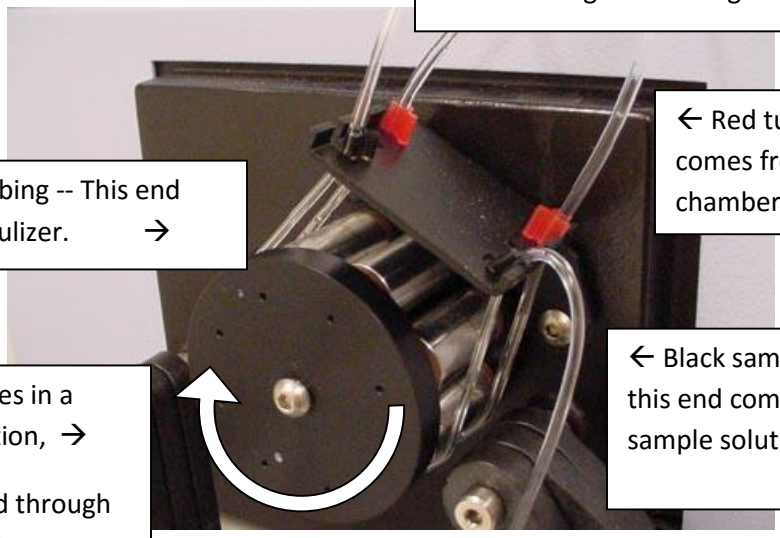
Item	Description
1	Clear GemTip
2	Red GemTip
3	Argon Inlet
4	Sample Inlet

RUNNING THE SPECTROMETER

Tubing and Pump Set Up

Reposition the tubing. The tubing color-coded black/black is used to bring the sample solution to the nebulizer. The tubing color-coded red/red goes from the mixing chamber to the waste bottle on the floor. The tubing is wrapped around the peristaltic pump in a clockwise direction. The black tubing is located in the front-most slot, and the red tubing is located in the rear-most slot.

Please note if any tubing appears damaged, do not attempt to repair or replace it yourself. Contact Adrienne through a phone call, email, or by finding her in the department.



Put on the tubing clamps. Be certain that the tubing goes into the groove in the clamp.

Flip up the tension lever on the rinse solution pump to the 4 o'clock position.

Instrument Login

There is no login on the computer. Simply click enter to go into Windows should you not be already in Windows.

After Windows comes up, double-click the WinLab 32 icon on the desktop. It takes approximately one minute for the instrument to initialize.

Creating a New Method

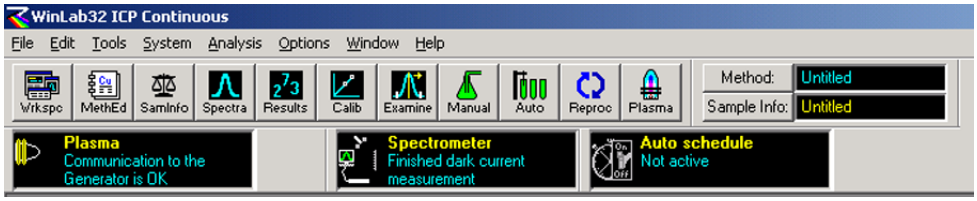
After Winlab 32 comes up and the instrument finishes initializing, you need to create a method if you choose not to use the Workspace template provided from training.

Perform the following steps to create a method:

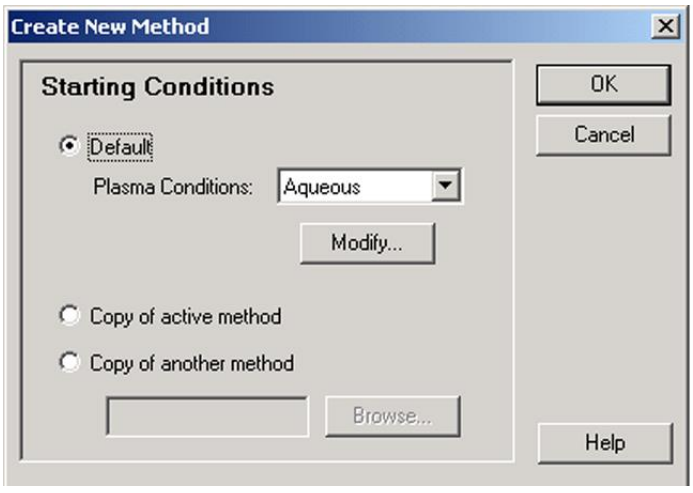
Click on the File menu, and then click on New, and Method.

Click on File →

and New, Method

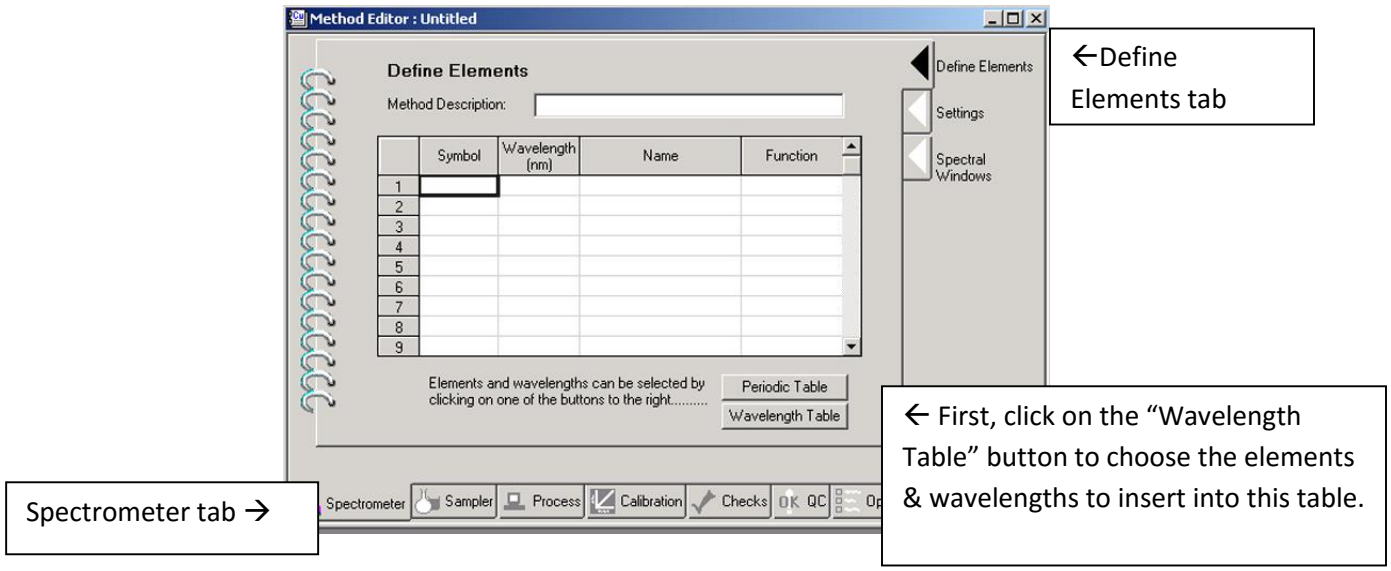


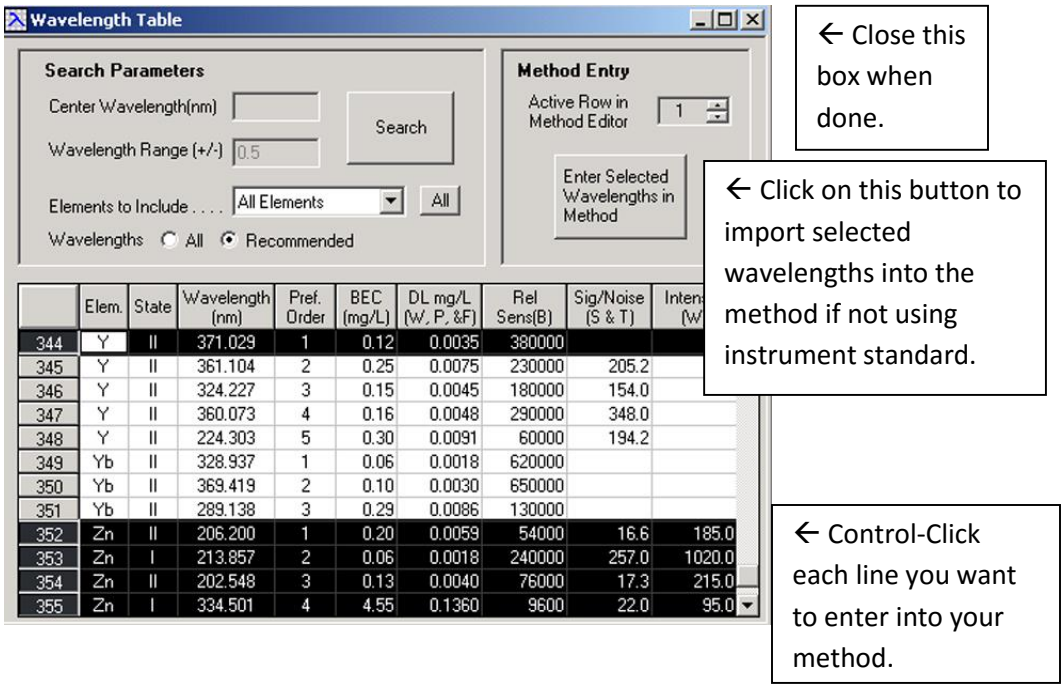
The following box comes up:

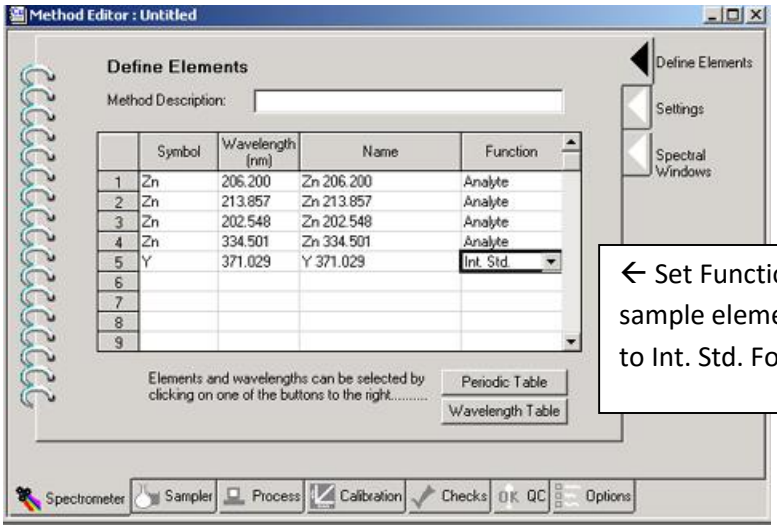


Use the “Robust” plasma conditions and click “OK”.

After you click OK, the following box appears, which lets you enter your elements and wavelengths. First, click on the "Wavelength Table" button to go to the list of elements and their wavelengths. Control-click each line of the table that you want to import into your method, and then click on "Enter Selected Wavelengths in Method" button. Once the selected wavelengths are entered into the method, Use the "Function" column to define the element as an "Analyte" for elements to be analyzed and "Int Std" for the element that is to be used as the internal standard.

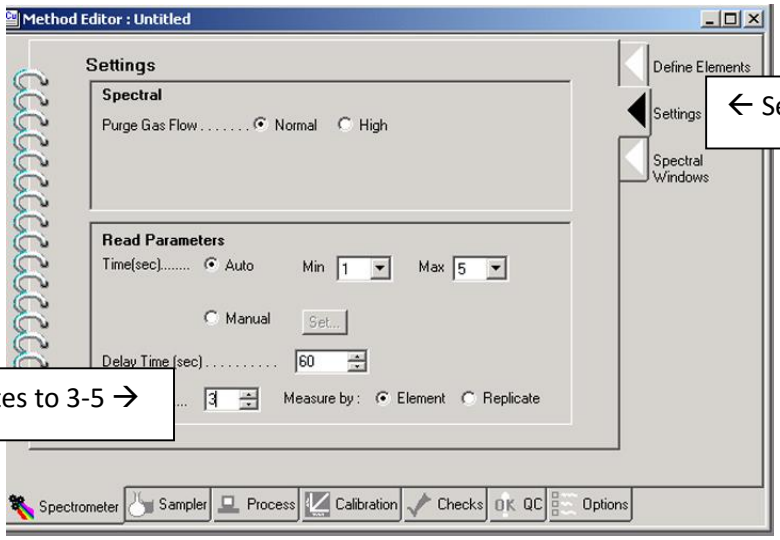






← Set Function to Analyte for sample elements, and set Function to Int. Std. For Internal Standards.

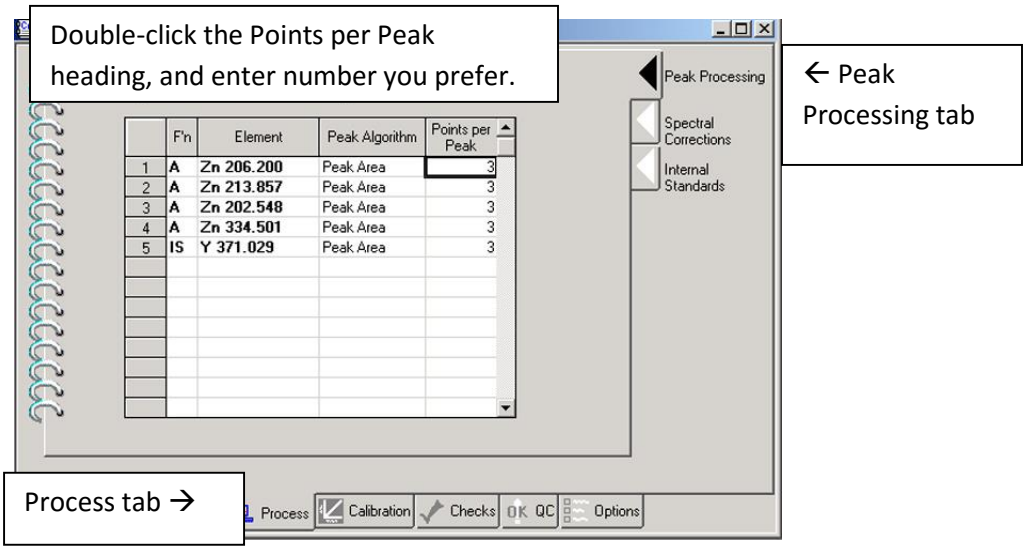
Click on the Settings tab on the right hand side. The following box will appear. Change the "Replicates" parameter to the number of replicates you wish to take, usually between 3 and 5.



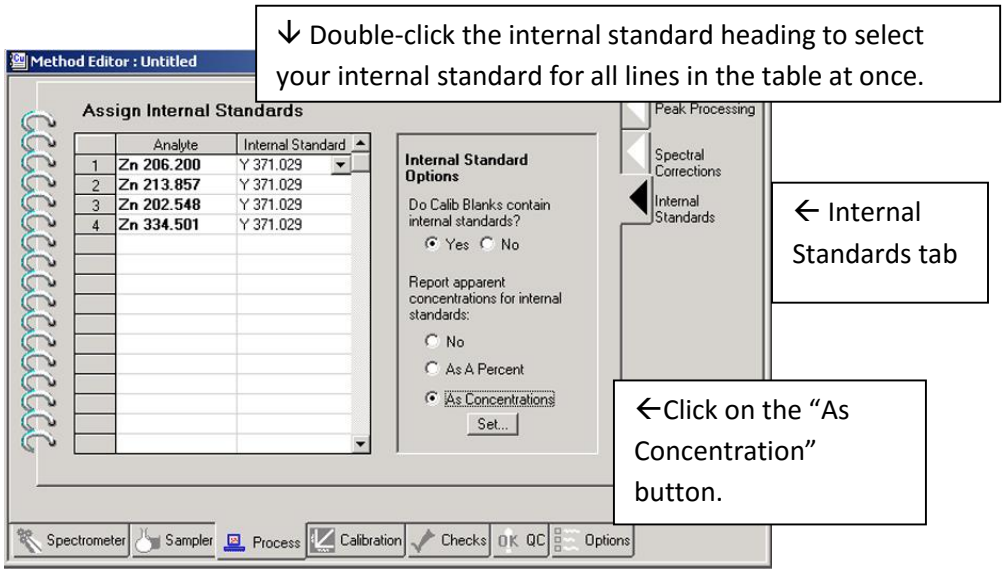
← Settings tab

Change Replicates to 3-5 →

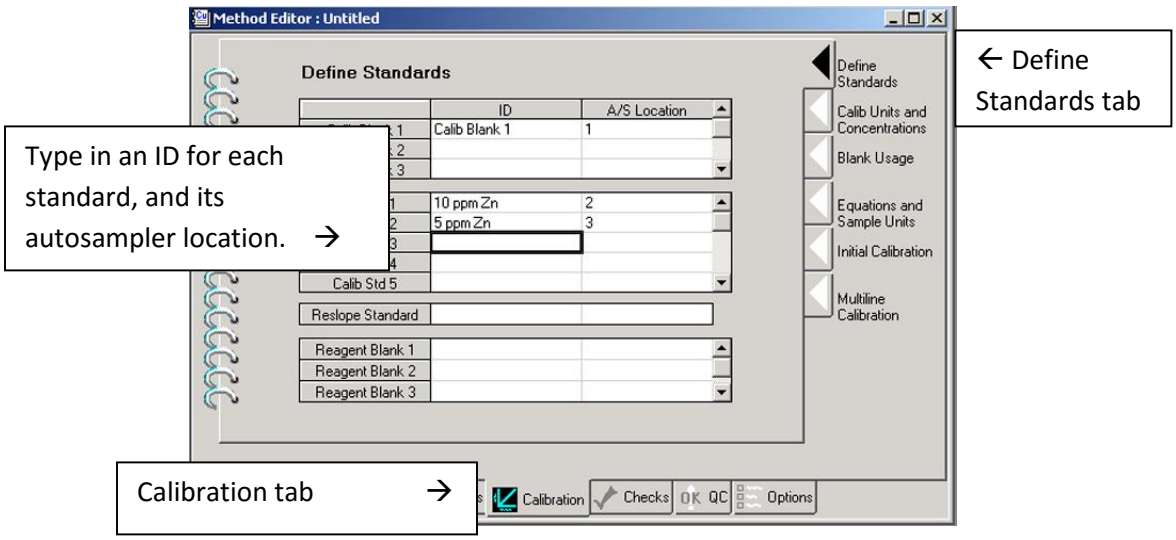
Next, click on the Process tab on the bottom. The following box appears. Change "Points per Peak" to a number you prefer for each line in the table by double-clicking the Points per Peak heading.



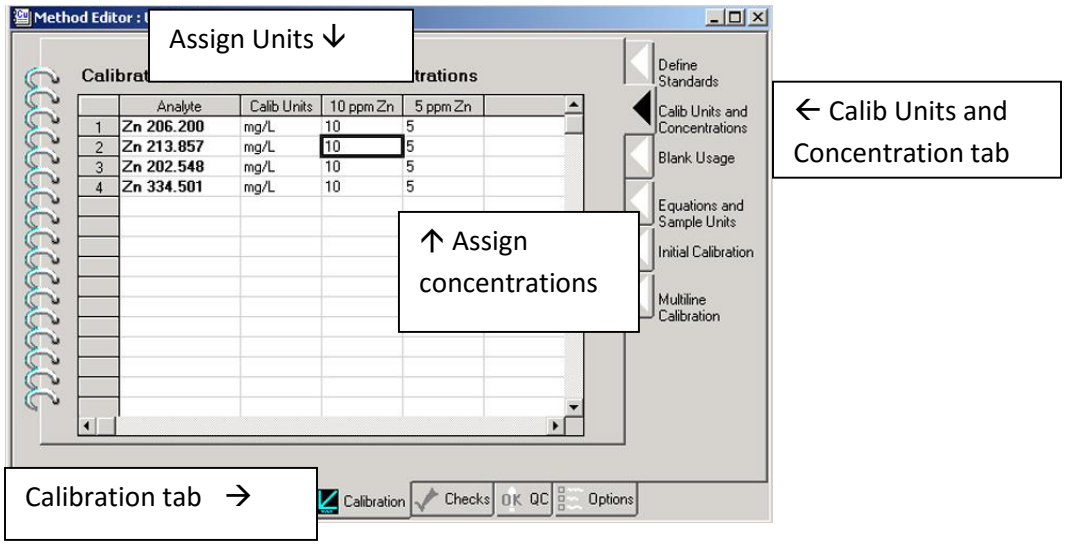
If using internal standards, click on the Internal Standards tab on the right. Double-click the internal standard heading to select the internal standard to use for each element in the table. Also, click on the "As Concentration" button.



Click on the Calibration tab on the bottom. Give an ID and a location for each standard you will be using. Load your calibration standards from lowest to highest concentration. The ID should say what the element is, and its concentration. The location is the numbered well in the autosampler tray.

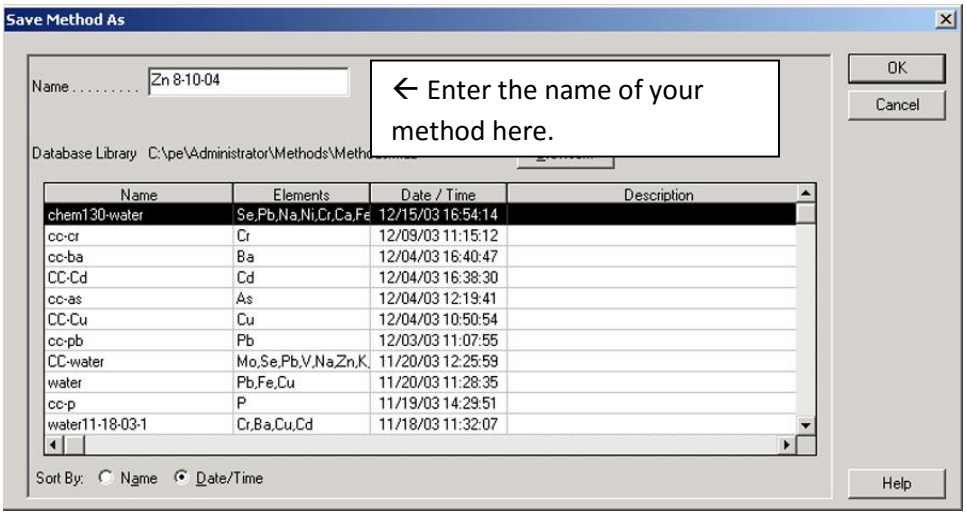


Click on the Calib Units and Concentrations tab. Enter the correct units to use for your standards, and enter the concentration of each standard.



Save your method. On the main page, click on the File menu, and click on Save As…, and Method. Enter a name for your method and click on OK.

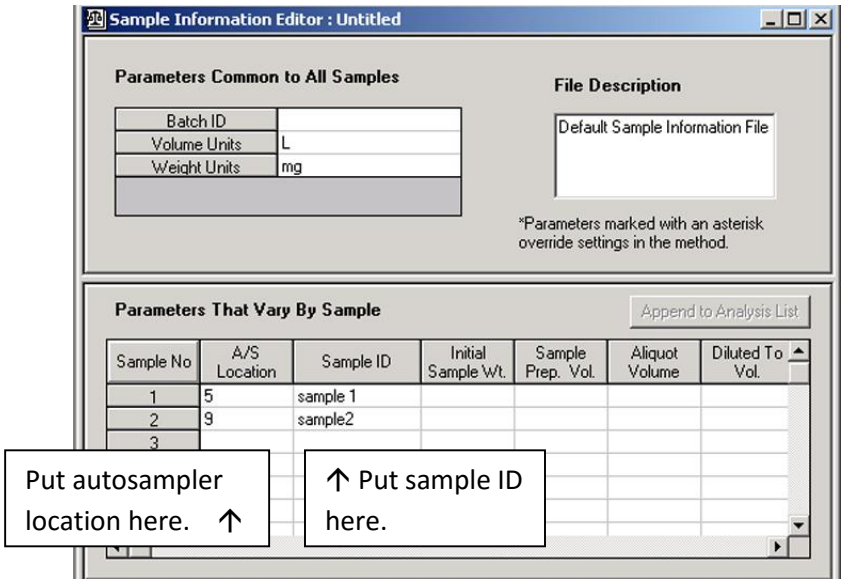
Under Edit menu, click 'Check Method' to be sure your method is free from errors. Fix any error that exist.



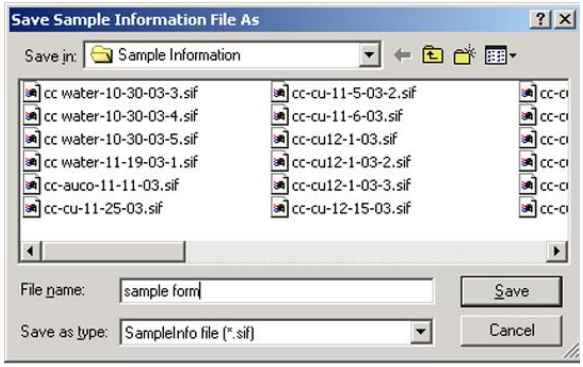
Creating a Sample Info File

Create a sample list. To do this, click on File, New, Sample Info File…

Fill in the table with the autosampler location and sample ID for each sample. Include one calibration standard from each element as a check.



Save your sample list. To do this, click on File, Save As, Sample Info File… Type in a name and click Save.



Close the Sample Information Editor.

Creating a Workspace File

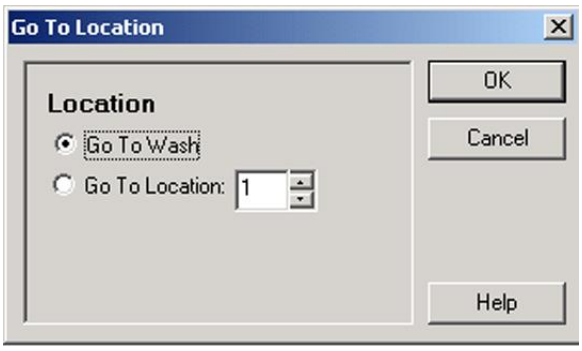
From the top icon toolbar open “Spectra”, “Calib”, and “Results”. Adjust the windows to fit on your page. Go to File, Save as, Workspace. Enter a name and save your workspace. Each time you use the instrument you can call up your workspace that you created to monitor your analyses.

Instrument Startup

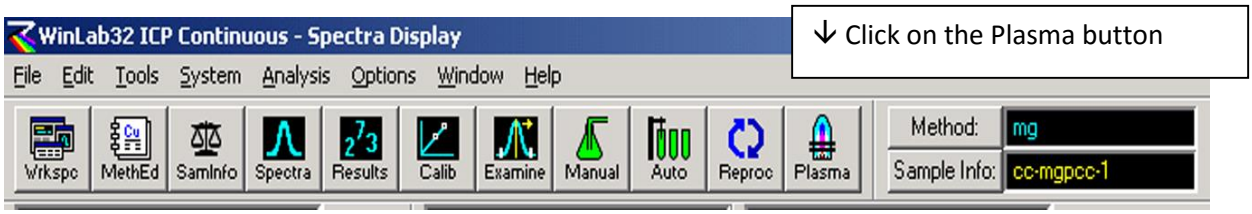
Put your samples, standards and blanks into the autosampler. Be sure to put them into the well positions that you specified in your methods.

Click on File, Open, Workspace…, and select your workspace file. This is a file that specifies what windows will be open on the desktop during data acquisition, and how they are arranged.

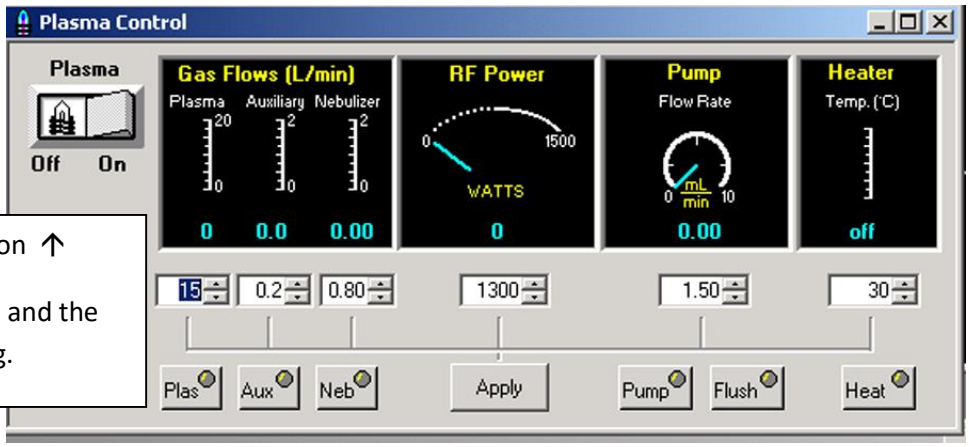
Push the F10 key on the keyboard. This will bring up a dialog box for the sample probe. Choose the option “Go To Wash”, and click OK. The rinse solution pump then starts, and the probe goes into the rinse solution.



Click on the Plasma button on the toolbar.

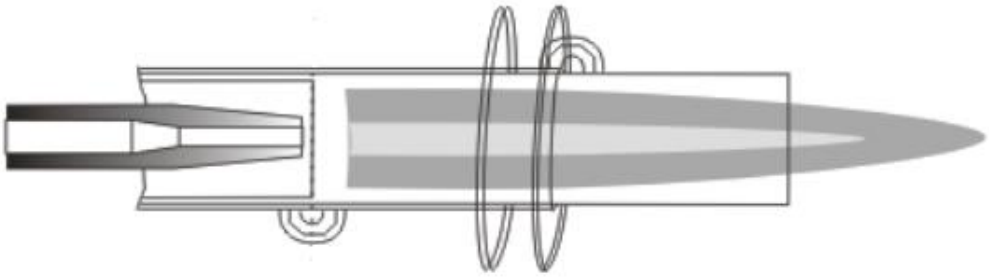


The Plasma Control box comes up. First, click on the "Pump" button. This turns on the sample pump. Verify that the solutions are flowing, and flowing in the right direction. Then click on the On button to light the plasma. Once you click the On button, a sequence of events takes place. First, it shuts off the sample pump, then it adjusts the gas flows, it lights the plasma, and then it turns the pump back on. Wait until after all variables come to equilibrium, then you can close the Plasma Control box or leave it open and move on.

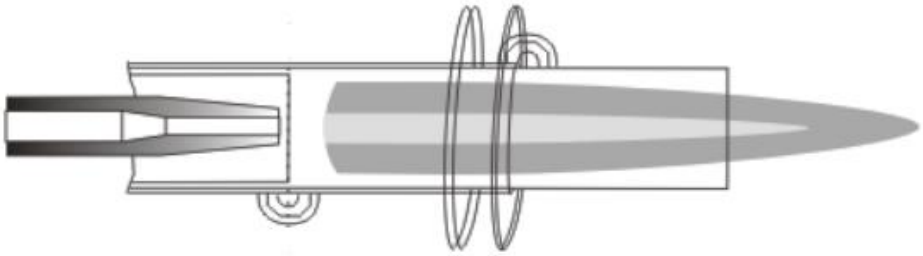


Flame Checks Before Moving to Analysis

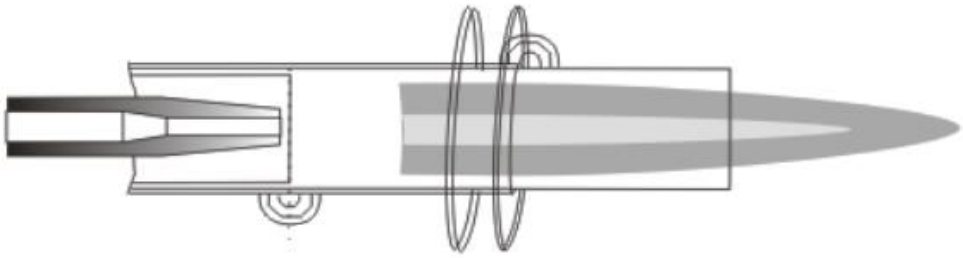
Check your flame shape. If any of the abnormal flame shapes are observed, please contact Adrienne through a phone call, email, or by finding her in the department.



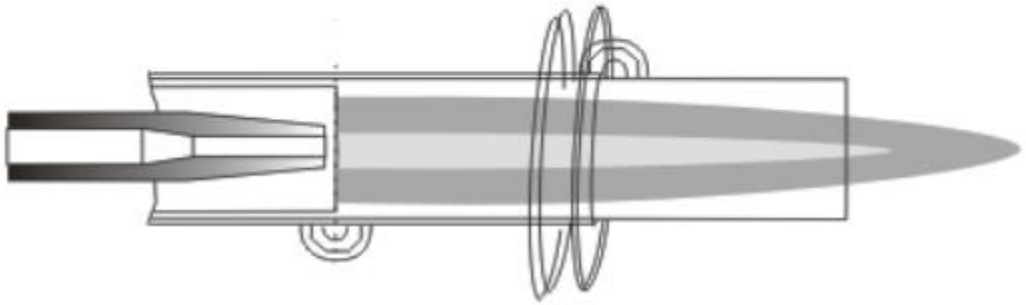
Normal plasma



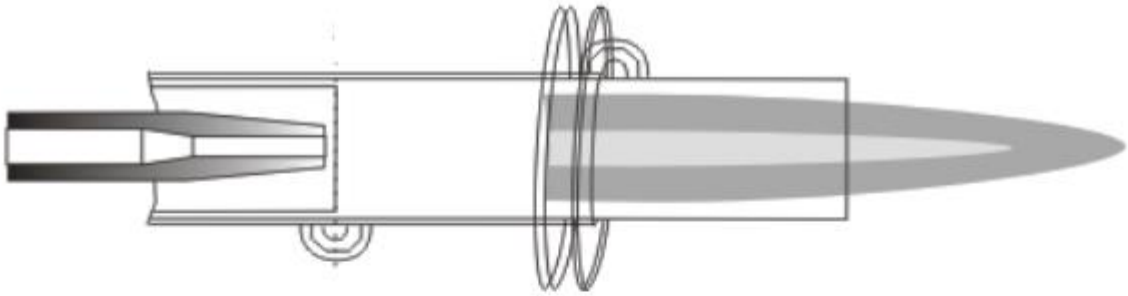
Rounded Plasma due to sample or air getting around the outside (typically subtle air leak).



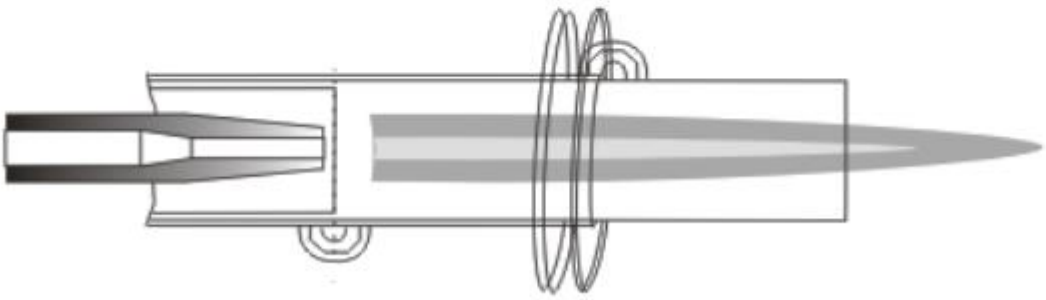
Aux Gas too high, air leak, or spray chamber temperature too high.



No Aux Gas (torch may be glowing).



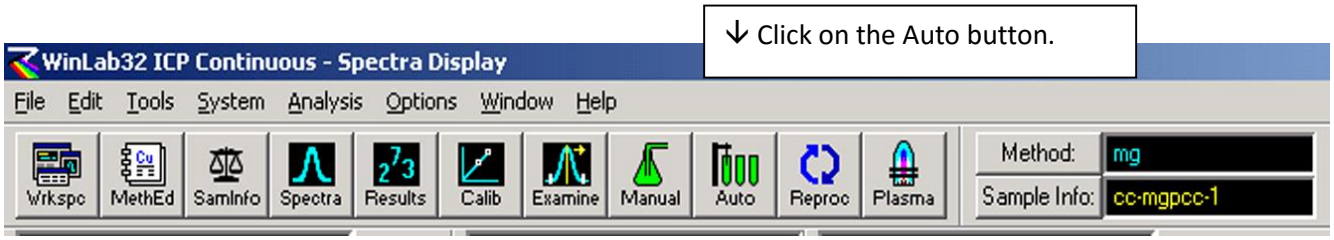
Air leak or spray chamber temperature too high.



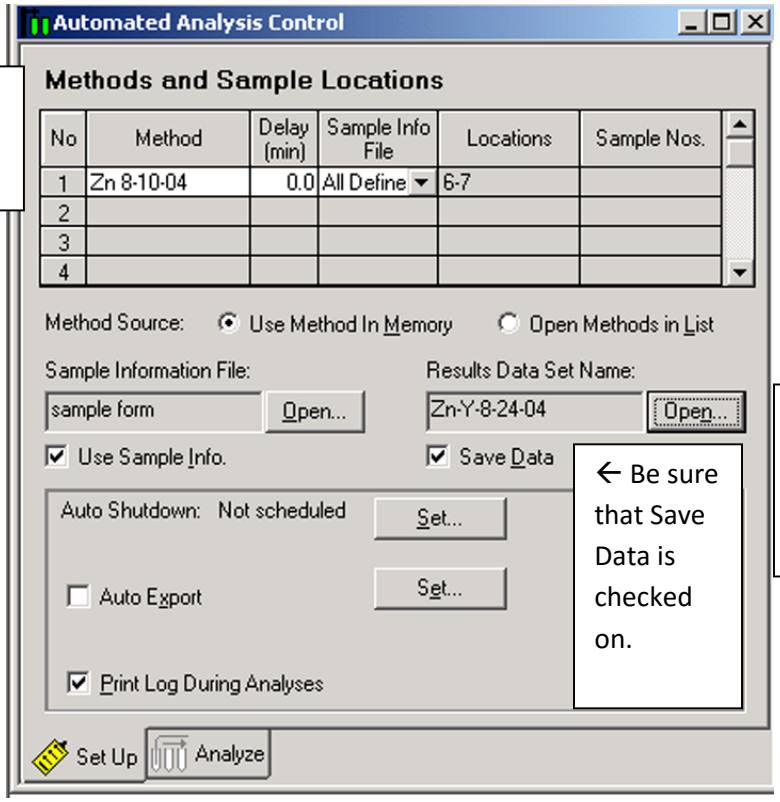
Thin plasma due to leak in plasma gas line.

Running Your Samples

Click on the Auto button on the toolbar. (This may already be open from your template workspace.)



The following dialog box will come up:



First, double-click the cell to put in your method. →

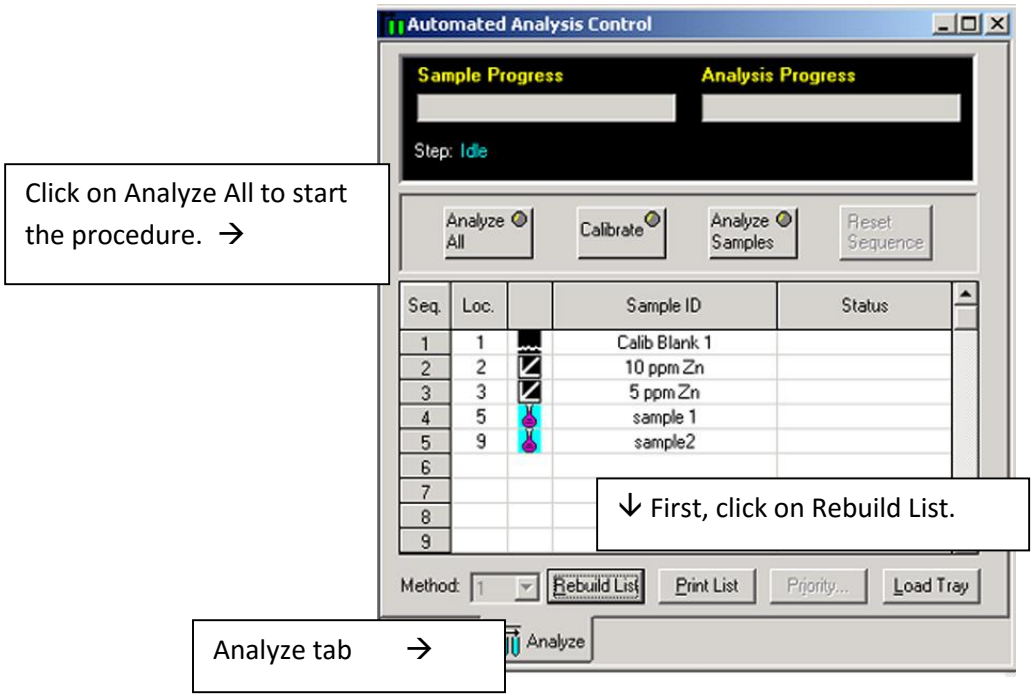
2nd, click on Open to put your sample information file in here. →

Be sure that Print Log During Analyses is checked on →

← Be sure that Save Data is checked on.

← 3rd, click on Open to give a name for your data results set.

Click on the Analyze tab. Click on the "Rebuild List" to update your list.



After you click on Analyze All, you can sit back and watch the analysis proceed. You can watch the spectra being collected, and can watch the calibration curves being generated, and will see the results being printed on the printer.

If you need to stop, you can click "Analyze All" again and select the appropriate stop.

How to Shut Down the Instrument When You Are Finished

Flush out the nebulizer after use. ***Flush*** with the stock <1% HNO_3 in water solution in the bottle by the autosampler (already set-up when autosampler finishes) for a minute or two, then hit F11 and let it flush with air for a minute or two before shutting the plasma down.

Click on the Plasma button to bring up the Plasma dialog box if not already open. Click on Off, to turn off the plasma.

Wait until the gases stop flowing before you exit from the program.

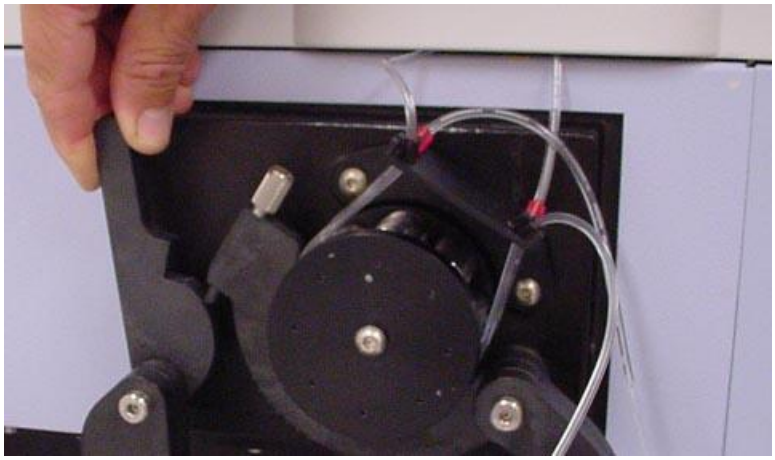
Exit from the program. If you wish to first export your data as a .csv or other format, see next section.

Do not log out of Windows.

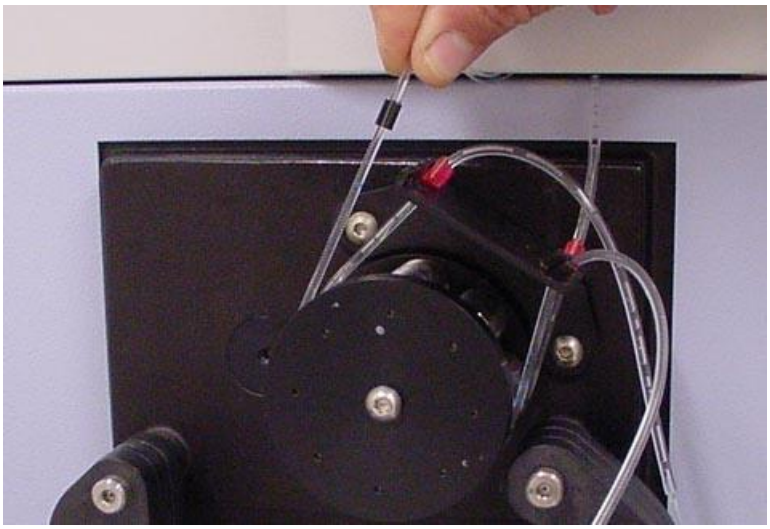
Leave the computer on.

Do not turn off any power switches on the instrument. They always remain on. Do not close or turn off gases.

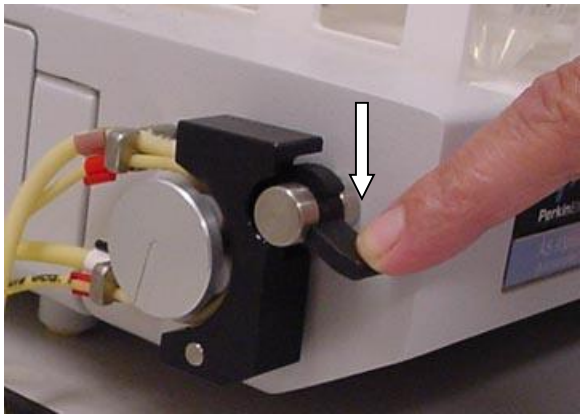
Release the clamps from the peristaltic sample pump.



Release the tubing.



Release the tension on the rinse solution pump.



Remove your standards and samples from the autosampler tray. You might want to keep the solutions in case you need to re-run anything at a later date.

Your data will print unless you have turned it off. Currently Results files are over-written on every run, so you will not be able to access your data after the next person uses the instrument.

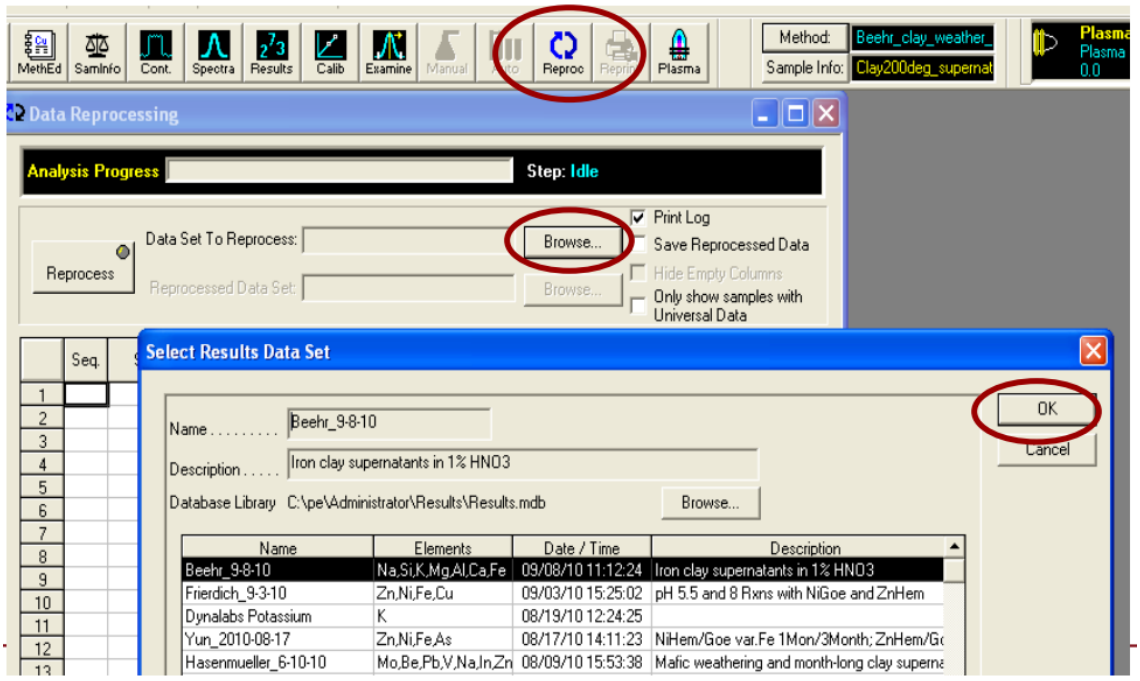
How to Get Your Data

Bring a USB to get your file from the results. Browse to C:\Users\Public\PerkinElmer\ICP\Data\Results\Results.mdb

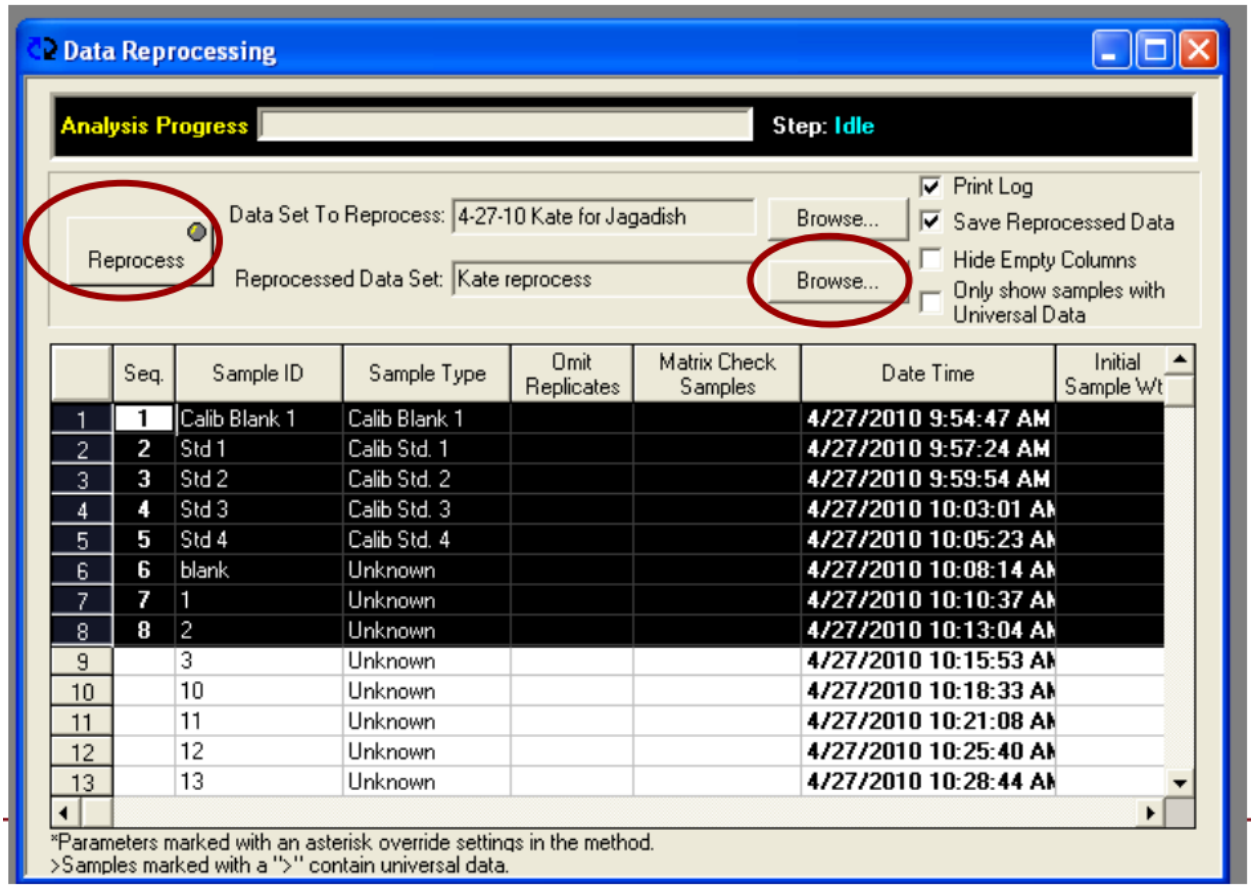
Overview of Windows for Handling Data: Data Manager Application.

Data Manager window. Use the Data Manager window toperform various data handling maintenance tasks. You can create, check, rename, copy, delete, and restore results data sets as desired. It is important to periodically delete and archive data sets to prevent libraries from becoming too large. Also, if you are running WinLab32 Enhanced Security use the Data Manager utility to view a master event log of all significant actions performed by a user as well as the revision history on all files and data objects.
Reprocess data after making adjustments to your method (you can change your calibration line type, remove standards, remove internal standard weighting, etc… but must save the method as the same name for reprocessing to work correctly)

Click on the “Reproc” icon, then click “Browse” by “Data Set To Reprocess” and highlight your data set, then click “Ok”



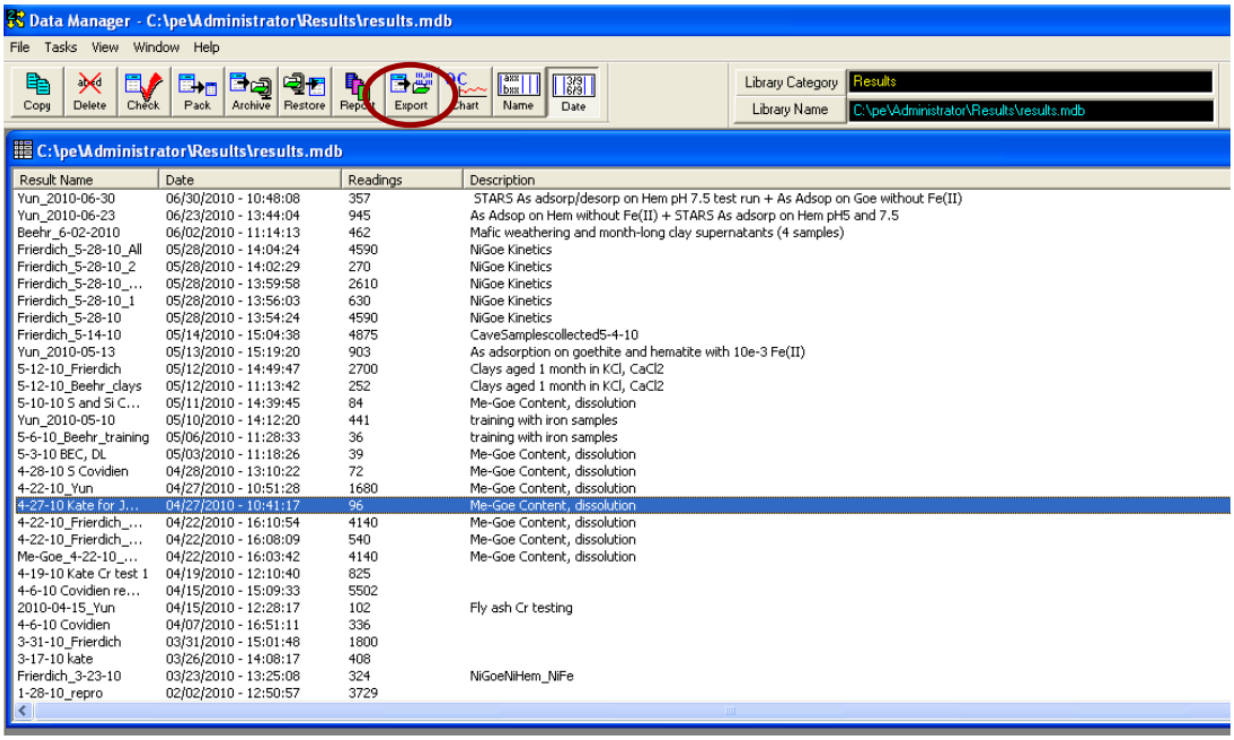
Click “Browse” by “Reprocessed Data Set” and name the reprocessed data file. Check “Print Log” and “Save Reprocessed Data”, uncheck all other boxes. Highlight your blank, standards, and samples, then click “Reprocess”.



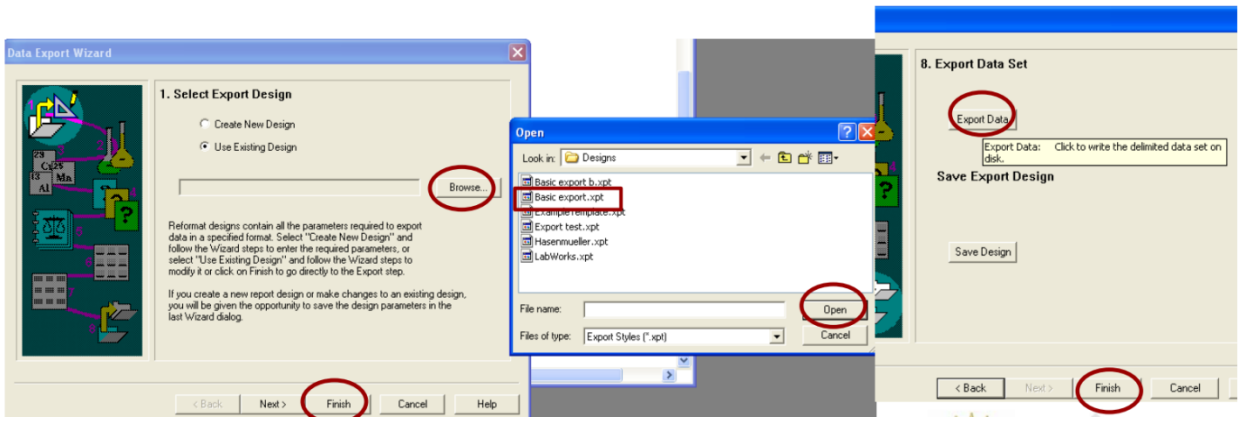
Reporting Wizard. Use Data Manager’s Reporting Wizard to create printed reports of selected data. This wizard is an interview-style series of dialogs to help you easily choose the data and the format in your report.

Export Wizard. Data Manager’s Export Wizard lets you select a subset of the data contained in a results data set and write it to a file that can be read by many other software applications, including spreadsheet and database management programs.

Go to the computer desktop and open “Data Manager” software. Highlight the dataset you want to export and click the “Export” icon



In the Data Export Wizard click “Use Existing Design”, then open “Basic export.xpt” or your own design. Click “Finish” to go directly to the export step or click “Next” to modify the design. If you clicked “Finish”, then click “Export Data”, then click “Finish”. Data will be saved in **C:\userdata\Data Manager Export\dataset name**



You can use your USB to collect your exported data.