Preliminary Testing and Acceptance of Instrument SX100 Prior to Shipment

Documentation of performance in accordance with following preliminary specifications shall be provided by the Contractor prior to shipping the Instrument.

The Instrument will be preliminarily accepted once University personnel receive, review, and approve of the following preliminary specifications:

- 1. Electron Gun specifications in Section 13.1.3;
- 2. Electron Column specifications in 13.2.3 and 13.2.10;
- 3. Wavelength Spectrometer specifications in 13.3.13;
- 4. Secondary and backscattered specifications in 13.4.1 and 13.4.3;
- 5. Light Optical specifications in 13.5.4 and 13.5.5;
- 6. Stage specifications in 13.8.1 and 13.8.2;
- 7. Interface specifications in 13.9.1 and 13.9.2;
- 8. Software specifications in 13.10.2.i.

Once University receives and approves the preliminary specifications, it shall notify Contractor that Preliminary Acceptance has occurred, and that the Instrument should be shipped.

Comply, but according to any modifications of these specifications detailed in the corresponding sections above.

Results of factory specification tests will be communicated to the University for Review prior to shipment.

Electron Gun specifications in Section 13.1.3

<u>13.1.3</u> Gun will produce a regulated current range from at least 1 nA to 200 nA (1 to 500nA desired) with a linearity of better than 0.2% as measured using a NIST traceable electrometer.

Comply, but CAMECA cannot supply the electrometer

DESIRED: 1 to 500 nA regulated beam current range

Comply, 0.5 to 1000nA range (normally supplied only as part of the GEOCHRON option but this capability will be supplied independently in this case).

Tuning	Tuning
Beam Align	Beam Align
Aperture (μm) 150 💌	Aperture (μm) 150 💌
I (nA)	I (nA)
Regulation Off On	Regulation Off On
Beam Focus Hysteresis	Beam Focus Hysteresis
Wobble Off On	Wobble Off On
Size (μm) 🛛 💌	Size (μm) 🛛 💌
Stigmatism Aid Off On	Stigmatism Aid Off On
Tuning	Tuning
Tuning Beam Align	Tuning Beam Align
Tuning Beam Align Aperture (μm) 150	Tuning Beam Align Aperture (μm) 150 💌
Tuning Beam Align Aperture (μm) 150 ▼ I (nA) 500.0 ▼	Tuning Beam Align Aperture (μm) 150 Γ I (nA) 1000. Γ
Tuning Beam Align Aperture (μm) 150<	Tuning Beam Align Aperture (µm) 150 ▼ I (nA) 1000. ▼ Regulation Off On
Tuning Beam Align Aperture (μm) 150< ▼	Tuning Beam Align Aperture (μm) 150 ▼ I (nA) 1000. ▼ Regulation 0ff On Beam Focus Hysteresis
Tuning Beam Align Aperture (μm) 150 I (nA) 500.0 Regulation Off Beam Focus Hysteresis Wobble Off Off	Tuning Beam Align Aperture (μm) 150 ▼ I (nA) 1000.▼ Regulation 0ff On Beam Focus Hysteresis Wobble 0ff On
Tuning Beam Align Aperture (μm) 150 I (nA) 500.0 Regulation Off Beam Focus Hysteresis Wobble Off O Size (μm) 0	Tuning Beam Align Aperture (µm) 150 ▼ I (nA) 1000.▼ Regulation Off On Beam Focus Hysteresis Wobble Off On Size (µm) 0 ▼

Electron Column specifications in 13.2.2, 13.2.3 and 13.2.10

<u>13.2.3</u> High voltage instability must be no more than +/- 0.005% per hour (+/- 50 ppm) as determined by repeated Duane-Hunt limit test on EDS (with Cu K α /L α calibration) or equipment supplied by University (see 13.2.2),

We will supply the test results from the electronic department. Our specification is +/- 50ppm relative per hour over the complete available range.

<u>15 kV – 10 nA</u>



<u>20 kV – 20 nA</u>



<u>13.2.10</u> The beam monitoring aperture (faraday) current variation shall be less than 0.3% (+/- 0.15%) when measured on both a pure carbon sample and on a pure Fe sample at both 10KeV and 25KeV @ 50nA beam current and enough replicate measurements to achieve sufficient precision. The stage position (stepper motor winding circuits) shall have an effect on the beam current of less than 0.1% (+/- 0.05%),

Comply

<u>10 KeV</u>

Faraday current on C = Faraday current on Fe =	50.63 nA 50.61 nA
Faraday current variation =	0.039 %

<u>25 KeV</u>

Faraday current variation =	0.042 %
Faraday current on Fe =	47.93 nA
Faraday current on C =	47.91 nA

Wavelength Spectrometer specifications in 13.3.13

13.3.13 Each supplied analyzing crystal count rate and P/B specification (as measured on pure metals unless otherwise noted) must meet or exceed 80% of the Contractor's specifications and 100% of the specifications in this document:

Please refer to the table of standard crystal count rate and P/B specifications in the Standard Performance Specifications document. Please note that some CAMECA specifications refer to different X-Ray lines than are present in this table, and that PC1.5 and PC2.5 are delivered without performance specifications.

PET

Spec	Xtal Number	Element	Sin Theo	Sin Real	Sin delta	Cs Peak	Cs Back-	Cs Back+	Peak/Back	BaseLine	Wind	Bias	Gain
1	940	Cr	26173	26156	17	21646.	84.8	74.5	272.	1520	4040	1325	727
1	940	Ti	31417	31402	15	23018.	56.1	50.7	431.	1130	4108	1324	712
1	940	Ag	47488	47477	11	3567.	19.4	15.6	204.	560	3221	1319	702
4	939	Cr	26173	26174	-1	21642.	86.3	75.8	267.	1517	4043	1323	608
4	939	Ti	31417	31408	9	21687.	54.4	47.5	426.	1129	4108	1309	688
4	939	Ag	47488	47464	24	2946.	16.0	13.8	198.	560	3221	1304	693
5	941	Cr	26173	26236	-63	29443.	129.1	104.2	252.	1509	4051	1826	677
5	941	Ti	31417	31460	-43	28148.	76.8	65.0	397.	1124	4108	1823	670
5	941	Ag	47488	47506	-18	6086.	36.1	28.5	188.	560	3219	1817	636

TAP

Spec	Xtal Number	Element	Sin Theo	Sin Real	Sin delta	Cs Peak	Cs Back-	Cs Back+	Peak/Back	BaseLine	Wind	Bias	Gain
1	937	Si	27738	27708	30	44111.	23.0	59.8	1066.	560	5000	1322	2131
1	937	Al	32464	32441	23	41911.	18.3	49.6	1235.	560	5000	1322	2133
1	937	Mg	38500	38476	24	34177.	27.8	36.2	1068.	560	5000	1321	2054
4	916	Si	27738	27735	3	48057.	22.8	60.0	1161.	560	5000	1319	1879
4	916	Al	32464	32453	11	46360.	19.1	53.2	1283.	560	5000	1309	2011
4	916	Mg	38500	38480	20	36219.	27.9	36.9	1117.	560	5000	1307	1970

LIF

Spec	Xtal Number	Element	Sin Theo	Sin Real	Sin delta	Cs Peak	Cs Back-	Cs Back+	Peak/Back	BaseLine	Wind	Bias	Gain
5	874	Ge	31146	31135	11	13116.	83.2	76.8	164.	1768	2786	1823	295
5	874	Fe	48085	48073	12	16994.	34.6	23.9	582.	954	2252	1815	296
5	874	Ti	68264	68251	13	5617.	7.7	5.5	854.	560	1850	1809	269

LPET

Spec	Xtal Number	Element	Sin Theo	Sin Real	Sin delta	Cs Peak	Cs Back-	Cs Back+	Peak/Back	BaseLine	Wind	Bias	Gain
2	198	Cr	26173	26167	6	63955.	248.4	195.3	288.	1518	4042	1303	804
2	198	Ti	31417	31419	-2	60056.	138.7	120.0	464.	1128	4108	1302	796
2	198	Ag	47488	47491	-3	8361.	40.9	33.4	225.	560	3220	1296	768
3	197	Cr	26173	26162	11	80144.	349.6	291.1	250.	1523	4037	1900	1185
3	197	Ti	31417	31416	1	66793.	191.8	155.4	385.	1129	4108	1899	1147
3	197	Ag	47488	47482	6	14878.	78.5	63.3	210.	560	3221	1887	1104

LLIF

Spec	Xtal Number	Element	Sin Theo	Sin Real	Sin delta	Cs Peak	Cs Back-	Cs Back+	Peak/Back	BaseLine	Wind	Bias	Gain
3	153	Ge	31146	31162	-16	54789.	346.1	312.7	166.	1765	2786	1868	409
3	153	Fe	48085	48117	-32	57253.	110.1	79.0	605.	952	2252	1859	384
3	153	Ti	68264	68266	-2	19654.	20.4	15.4	1099.	560	1849	1850	356

LTAP

Spec	Xtal Number	Element	Sin Theo	Sin Real	Sin delta	Cs Peak	Cs Back-	Cs Back+	Peak/Back	BaseLine	Wind	Bias	Gain
2	49	Si	27738	27740	-2	134554.	178.6	204.0	703.	560	5000	1319	1964
2	49	Al	32464	32466	-2	121765.	120.2	167.9	845.	560	5000	1311	2081
2	49	Mg	38500	38497	3	91665.	108.6	115.5	818.	560	5000	1312	1996

MPC 1

Spec	Xtal Number	Element	Sin Theo	Sin Real	Sin delta	Cs Peak	Cs Back-	Cs Back+	Peak/Back	BaseLine	Wind	Bias	Gain
1	215	F	30992	30439	553	21917.	197.6	63.9	168.	560		1504	555
1	215	0	39991	39402	589	3888.	60.5	17.0	100.	560	5000	1502	552
1	215	С	74778	73947	831	1013.	5.8	0.0	173.	560		1500	532
4	216	F	30992	30370	622	23377.	158.6	61.7	212.	560		1487	537
4	216	0	39991	39250	741	4066.	48.3	15.6	127.	560	5000	1485	554
4	216	С	74778	73677	1101	1009.	4.1	0.0	246.	560		1500	489

MPC 2

Spec	Xtal Number	Element	Sin Theo	Sin Real	Sin delta	Cs Peak	Cs Back-	Cs Back+	Peak/Back	BaseLine	Wind	Bias	Gain
1	280	0	25432	25442	-10	5700.	0.0	76.8	74.	560		1508	828
1	280	С	47555	47172	383	22398.	326.7	202.8	85.	560		1504	981
1	280	В	72772	72286	486	2621.	36.3	0.0	72.	560		1504	981

Secondary and backscattered specifications in 13.4.1 and 13.4.3

<u>13.4.1</u> A secondary image resolution of 70 angstroms (0.007 um) at 20 KeV or better using a tungsten filament on a sample of Au particles on carbon and a beam current of at least 100 pA. It is desired to meet this specification also at 300 pA;

The 7nm resolution is met at 25keV with a beam current of 3pA.



DESIRED: meet above spec at 300 pA

<u>13.4.3</u> A backscattered electron atomic number resolution must be at least 0.1 Z at Z=29 or better (must easily contrast α/β brass sample) at both 10 KeV and 5 KeV using a 5 nA beam;

This specification is realized at 20kV. It has never been tested at other voltages.

BSE ATOMIC RESOLUTION

Point	Comment	Cu	Zn	Total
1	phase 1	55.46	44.54	100
2	phase 1	55.27	44.73	100
3	phase 1	55.43	44.57	100
4	phase 1	55.37	44.63	100
5	phase 1	55.11	44.89	100
6	phase 2	61.83	38.17	100
7	phase 2	62.91	37.09	100
8	phase 2	62.91	37.09	100
9	phase 2	63.49	36.51	100
10	phase 2	63.79	36.21	100

Average phase 1 on Cu	55.328	0.55328
Average phase 1 on Zn	44.672	0.44672
Average phase 2 on Cu	62.986	0.62986
Average phase 2 on Zn	37.014	0.37014

Average Atomic % phase 1	29.44672
Average Atomic % phase 2	29.37014

Result 0.07658



Light Optical specifications in 13.5.4 and 13.5.5

<u>13.5.4</u> Optical depth of focus less than +/- 1 um at 300-400x verified using hi-res video capture images on a suitable test specimen;



Z = 344 (5µm unfocused)

<u>13.5.5</u> The image shift in transmitted light must be less than 1 um as the transmitted optical light polarizer are rotated. The transmitted optical system must be compatible with all sample mounting systems;

Comply



Camera

Stage specifications in 13.8.1 and 13.8.2

<u>13.8.1</u> High speed and 100% reliable servo or stepper stage motors (linear optical encoder position verification desired but not required) with 0.5 um movement on all three axes. The stage must remain within 0.1 degrees level (+/- 0.05 degrees) relative to all Spectrometer take off angles at all times. This shall be verified by measuring k-ratios for each Spectrometer tuned to the same element (see Section 13.3.9). For this reason, there shall be NO stage tilt option in the sample stage assembly;

'100% reliable' would imply 0% chance of any failure at any time in the motor's life, and we cannot guarantee this. Local movement controlled to $0.1\mu m$. There is no stage tilt option. Please see note at section 13.3.9.

DESIRED: linear optical encoder position verification for stage

Comply

Linear optical encoders on X, Y and Z Axis: No tilt on the sample stage. 0.1 µm minimum increment in image or quanti line scans acquisition. **13.8.2** Minimum of 5 mm/sec speed (10 mm/sec desired) and less than or equal to $\pm/-1$ micron reproducibility ($\pm/-$ 0.5 micron desired) for X and Y axis positioning, and 1 mm/sec speed (2 mm/sec desired) and less than or equal to $\pm/-1$ micron reproducibility ($\pm/-$ 0.5 micron desired) for Z axis positioning as determined by driving from a point of interest to stage limits and back to the point of interest at 10,000X in SE image mode for X and Y and reflected light for Z over 200 times without discernable failure of reproducibility;

Speed for X and Y motions: 15mm/sec Reproducibility for X and Y motions: within 1µm with backlash. Speed and reproducibility for Z are not specified.



Interface specifications in 13.9.1, 13.9.2, 13.9.3 and 13.9.4

<u>13.9.1</u> All automation, acquisition and control software interface functions of the Contractor's instrument (stage and Spectrometer motor automation, PHA/counting, column/beam control, etc.) shall be specified and documented in English;

Comply.





13.9.2 Complete documentation of interfaces and communication protocols to main instrument microprocessor(s) and/or hardware systems and subsystems shall be provided in English to allow University the option to interface directly to the probe hardware in the future;

Such documentation is not completely in English, assistance is available.



Software specifications in 13.10.2.i.

<u>13.10.2</u> Microprobe analytical, acquisition and automation software should have the following attributes:

I) Qualitative x-ray microanalysis software shall provide wavelength scan data acquisition, sample step-scan data acquisition (count times from 0.01 to 3600 seconds minimum), automatic peak identification for a compete K, L, M, dataset similar to the latest NIST x-ray line database (including higher order reflections), and graphical display/manipulation of data, zoom, pan, data cursor, etc.;

Clarification required: 0.01 to 3600 seconds is total acquisition time or channel dwell time?

WDS Spectra on Andradite

g ¹ Sx - Settings (Andradite.wdsSet) File Beam/SEM Options Acquire Now M 때 한 딸 D ☞ 대 매 매]			
Comments :				WDS Spectra
Beam		SEM		
HV (KV) 20 ▼ I (nA) 10.	00 🔻	Qu	iality	Low Med. High User
Size (µm)	•	5	12*384	▼ 0.320 s ▼
Scanning Off	On	SEM1		SE 💌
Raster Length (µm) 604.2	60 🔽	SE	EM2	SE
-X-Rays				
Wds Crystals	F	Range		
1 OPET TAP PC1 PC2	Full	Rel.	Abs. O	Ref 39332 0 Ka 🕅 🕰
2 LPET DLTAP	۲	0	۰	Start: 22418 End: 84160
3 LLIF OLPET	۰	0	0	Start: 22378 End: 83947
4 PET o TAP PC1 PC25	۲	0	0	Start: 21810 End: 83742
5 DIF PET	0	۲	0	Ref 48085 Fe Ka 👫 📣 - 1000 + 1000

