PROCEDIMENTO PARA LIGAR O LA-ICP-MS

1. Abrir todas as válvulas dos gases: Hélio 6.0, Nitrogênio 5.0 e Argônio líquido;









Nitrogênio 5.0



Argônio

2. Se precisar trocar o mount, é a primeira coisa que deve ser feita;

Monitor do Laser

3. Abrir o programa Chromium 2.3;



- 4. Clicar em gas controls;
- 5. Clicar em Evacuate;
- 6. Abrirá automaticamente uma janela: "Enable Automatic Flow Control", clicar em Yes;





7. Aguardar terminar todo o procedimento, até chegar em On line;

Gas Controls		
Laser Control Gas Cor	trols	
Sample Chamber V	alves	
Bypass		0
Online		
Purge		0 1
🗄 Purge Progress		
Evacuate		0
G Status:	Done	
Vacuum Motor	Off	
MFC1 Pressure	14.71	PSI
MFC2 Pressure	14.76	PSI
eQC		0
Mass Flow Controll	ers	
Auto Flow Control		
Shut Down All Flows	6	
MFC1: (He)	0.300	[0.300] LPM
MFC2: (He)	0.300	[0.300] LPM
MFC3: (N2)	0.0	[0.0] mLPM
Gas Control Setup		0

- 8. Desabilitar "Auto Flow Control";
- 9. Zerar o fluxo do gás manualmente:
- 0.0 LPM MFC1(He)
- 0.0 LPM MFC2(He);

das controis		
Laser Control Gas Con	trols	
Sample Chamber V	alves	
Bypass		۲
Online		0
🚽 Purge		0
🗄 Purge Progress		
Evacuate		0
Status:	Done	
Vacuum Motor	Off	
MFC1 Pressure	13.50	PSI
MFC2 Pressure	13.53	PSI
eQC	1	0
Mass Flow Controlle	ers o	1
Auto Flow Control	C 0	
Shut Down All Flows	6	
MFC1: (He)	0.000	0 [0.000] LPM
MFC2: (He)	0.000	9 [0.000] LPM
MFC3: (N2)	0.0	zerar o fluxo
Gas Control Setup		Zorur o huxo,

Monitor do ICP-MS

10. Em "Start and Stop Plasma", clicar em "On";



- 11. Abrirá a janela automaticamente: "Peristaltic Pump is switched off. Start Peristaltic Pump?";
- 12. Clicar em "No";



13. Aguardar todo o sistema ficar verde em "Start and Stop Plasma";

Start and S	itop Plasma • • • •
Off	
Stop	
<u>B</u> reak	
<u>E</u> merg. Off	
Se <u>t</u> tings	Plasma successfully started

Monitor do Laser

14. Habilitar o "Auto Flow Control";

Laser Control Gas Con	trols		
Sample Chamber Va	alves		
Bypass		۲	
Online		0	
I Purge		0	
🗄 Purge Progress			
= Evacuate		0	
Status:	Done		
Vacuum Motor	Off		
MFC1 Pressure	13.50	1	PSI
MFC2 Pressure	13.53 14		PSI
eQC		0	
Mass Flow Controlle	ers		
Auto Flow Control			
Shut Down All Flows	0		
MFC1: (He)	0.000		[0.000] LPM
MFC2: (He)	0.000		[0.000] LPM
MFC3: (N2)	0.0		[0.0] mLPM
Gas Control Setup			

Monitor do ICP-MS

- 15. Abrir a janela "Source Lenses";
- 16. Clicar em abrir pasta;
- 17. Clicar em "LR_Solução_Jet_Jetcone_Upb_Solange;
- 18. Clicar em "Open";

Scan Control	68 Zoom Optics	Cup Configuration	2 Detector Calibration	Center	Cup 🚺 Sourc	e Lenses 🕠	🛚 Inlet System
	File: Tune LR_U	Th_Pb_USP(RF NOVA)	_sandra.sle 🔶	LR_So	lução_Je	t_Jetc	one_UPb_Sola
.enses:	Extraction [V]:	85	2000.0		2000.0	- <u>17</u> [-	
6	Eocus [V]:	EE	-660.0	- + •	60.0		
	≚ • Defl. [V]:		0.02	•	0.02		
	⊻ - Defl. [V]:	EE	5.08	- • ·	5.08		
	<u>S</u> hape [V]:		230.	00++ 2	30.00		
	Bot Quad 1 [V]		1	9.90 + 1	9.90		
	Source Offset [V]:	ĒĒ	28.00		8.00		
	Guard Electrode	₂ On/ Off	Advanced >>	-			
			Analyzer	🛀 r Gate Open / (Closed		
			📄 🛛 🔮 HV On /	0#			
			🛅 🔮 Enable (CDDs			
			<u>R</u> esolution				

Monitor do Laser

19. Aguardar o fluxo de Hélio no "Auto Flow Control" chegar 0.300 LPM MFC1 e 0.300 LPM MFC2;

Laser Control Gas Con	trols		
Sample Chamber V	alves		
Bypass		0	
Online		۲	
🖃 Purge		0	
🗄 Purge Progress			
🖬 Evacuate		0	
∃ Status:	Done		
Vacuum Motor	Off		
MFC1 Pressure	14.71		PSI
MFC2 Pressure	14.76		PSI
eQC		0	
Hass Flow Controlle	ers		
Auto Flow Control			
Shut Down All Flows	6		
MFC1: (He)	0.300		[0.300] LPM
MFC2: (He)	0.300	19	[0.300] LPM
MFC3: (N2)	0.0		[0.0] mLPM
Gas Control Setup			0

Monitor do ICP-MS



20. Abrir a janela "Center Cup" e verificar se o "Faraday" está habilitado, se não estiver habilite-o;

- 21. Abrir a janela "Detector Calibration";
- 22. Clicar em "Baseline";
- 23. Clicar em "Ok", esperar o aparelho terminar o procedimento do "Baseline", a tecla ilumina novamente;

X Scan Control	S Zoom Optics	Cup Configuration	Detector Calibration	🥳 Center Cup	Source Lenses	• 11 Inlet System
Faraday Cups List Recalibrat 2	Break e aseline Gain	Ion Counters List Recalibrate List Recalibrate List List List List List List List List	d /otage e Level	1		

	Lintin	cups	
✓ Defocus		L5	
	IC1B	<u></u> ∠⊥4	☑ IC2
[IC 2 L5	L3	
first Mass of Center Cup:	176Hf	✓ L2 ✓ L1	VIL4
econd Mass of Center Cup:	176Hf	Center	☑ IC6 ☑ IC7
Jumber of Cycles (each 1.05s):	30	☑ H2 ☑ H3	<u> </u>
Pre baseline wait time [s] 23	10	₩ H4	

24. Clicar em "Gain";

X Scan Control 68 Zoom Optio	s 🖝 Cup Configuration 🔟 Detector Calibration 🥳 Center Cup 💼 Source Lenses 🔹 🗄 Inlet System
Faraday Cups Faraday Cups List Recalibrate Baseline aseline 24	Ion Counters List Recalibrate Yield Elateau Voltage
24	Dark Noise Level

- 25. Aguardar o aparelho terminar o procedimento do "Gain" e iluminar novamente, leva ~10 minutos;
- 26. Ir na janela "Watch Parameter" e clicar em "Analyzer Gate Open", o led ficará verde;

Watch Parameter	Tel		Watch Parameter	r
HV on Guard Electrode Coil Cooling OK Argon Pressure OK			HV on Guard Electrode Coil Cooling OK Argon Pressure OK	
Analogue			Analogue	
High Vacuum P <	2.77e-007		High Vacuum P <	2.85e-007
Ion Getter Press [mbar]	2.70e-009		Ion Getter Press [mbar]	3.66e-009
HV [V]	-9961.81	26	HV [V]	-9966.59
Field Probe [V]	0.569		Field Probe [V]	4.166
Fore Vacuum P <	1.60e-003		Fore Vacuum P <	1.57e-003
Switches			Switches	
Oon HV on			Oon HV on	
Skimmer Va	alve		Skimmer Va	alve
Hoff Analyzer Gate	: Open		Analyzer Gate	e Open

27. Ir na janela "Scan Control" - "Selected Mass" e selecionar a massa do Argônio: 79,.....



Scan Control	68 Zoom Optics	Cup Configurat	ion 🔽 Detect
Peak Control	79.91		27
Selected Cup:	C (Far.)	Set	28
		ts <u>B</u> reak	
	Peakso	an Peak <u>c</u> er	nter

- 29. Ir na janela "Watch Parameter" "Intensities", verificar a intensidade do Argônio;
- 30. Anotar a intensidade de Argônio no caderno, ela deve estar em aproximadamente 46V;



31. Voltar na janela "Scan Control" - "Selected Mass" e selecionar a massa 202 Hg;

22 Clicar om "Sot",				
Sz. ciicai eini set,	X Scan Control 66 ;	Zoom Optics	😎 Cup Configuratio	n 💆 Detector Ca
	Peak Control Selected 31	202Hg	•	
		Elements Peaksca	<u>B</u> reak	32

33. Abrir a janela: "Center Cup";

34. Desabilitar o "Faraday";



35. Voltar na janela "Scan Control" - "Selected Mass" e clicar em "Set";;

X Scan Control	68 Zoom Optics	😎 Cup Configura	stion 🛛 💆 Detector Ca
Peak Control			
Selected Mass	202Hg		
Selected Con	C (IC)	• Se	
	Element	ts Brea	k 35
	Peakso	an Peakge	anter

36. Voltar na janela "Watch Parameter" - "Intensities" e verificar a intensidade do 202 Hg;

37. Anotar no caderno a intensidade do 202 Hg; deve estar entre 5000 e 16000 cps aproximadamente;



- 38. Voltar na janela "Scan Control" e clicar em "Peakcenter";
- 39. Aguardar o término do peakcenter, isso ocorrerá quando a tecla estiver iluminada novamente;



- 40. Selecionar a massa para 204Pb em "Selected Mass", clicar em "Set";
- 41. Clicar em "Peakcenter", aguardar o término do peakcenter (tecla estiver iluminada novamente);

X Scan Control	68 Zoom Optics	Cup Configuration	💆 Dete
Peak C <u>o</u> ntrol			/
Selected <u>M</u> ass	с 204Рь		40
Selected C <u>u</u> p:	C (IC)	🔹 💽 🖌	
	<u>E</u> lements]
	Peaksca	n Peakcenter	
			41

- 42. Selecionar a massa para 207Pb em "Selected Mass", clicar em "Set";
 - Scan Control
 Scan Control
 Com Optics
 Cup Configuration
 Det

 Peak Control
 Peak Control
 42

 Selected Mass:
 207Pb
 42

 Selected Cup:
 C (IC)
 Set

 Elements...
 Break

 Peakscan
 Peakoenter

 43

43. Clicar em "Peakcenter", aguardar o término do peakcenter (tecla estiver iluminada novamente);

- 44. Ir na janela "Application Start";
- 45. Clicar no "X (Executive)";

{{{ Tune - [Neptune]	
III File Tune View Help	
	Accessories
Application Start	000
🗖 🖬 🖪 🗶 🕐 🞇	
Start and Stop Plasma	
45 Off Stop Break Emerg. Off Settings	✓ Plasma successfully started
Watch Parameter	
C regent resourcert	

- 46. Clicar na janela "Mass Calibration";
- 47. Verificar se o gráfico de calibração das massas está reto, ou se há alguma massa fora da reta;



- 48. Verificar qual é a massa virtual no Cup Configuration, a massa virtual fica no Faraday central "C";
- 49. Selecionar a massa virtual (279,.....) em "Selected Mass" e clicar em "Set";

C Scan Control	68 Zoom Op	optics Cup Configuration		Detector	Calibration
a					
A-1					
Actual Lup Pos	litions	Taxant famil	A	Circle Core	Chabin
Number	Species 💌	CDD Defl.[V]	Postion[mm]	Positioning	In position
L4-IC2					
L4-IC3	202Hg				
L4-IC4	204РЬ				
L4-IC5					
L4-F	206РЬ	84.450	84.466	Set	O Pos. OK
L3-IC6	207РЬ				
L3-F	208РЬ	68.430	68.413	Set	Pos. OK
L2-F		15.336	15.360	Set	O Pos. OK
L1-F		48.57	6.520	Set	O Pos. OK
IC1 C-F	219.76 🔶				
H1-F		6.861	54.862	Set	Undefined
H2-F	232Th	63.200	63.202	Set	O Pos. OK
H3-F		30.801	93.411	Set	Undefined
H4-F	238U	104.150	104.130	Set	O Pos. OK
H4-IC7					



Monitor do Laser

- 50. Calibrar as coordenadas do laser, clicar nas coordenadas, abrirá uma janela automaticamente "Move Stage
- 51. Clicar em "Home Stages";



Move Stages	5					
Go To/Step	Saved	Positions				
Move Mode	e	Position	I\$	Direction	Units	Step
0000	0510011	Υ 50			© µm	
() Step		⊠z 10	Reset		Omm	Stop Stop
Set XY	to (0,0)	9	Home Sta	ages	51]

52. Abrirá uma janela "Home Stages"; clicar nas coordenadas: "X", "Y" e "Z" e em "Start";



- 53. Quando termina a calibração a janela "Home Stages" se fecha automaticamente;
- 54. Fica aberta apenas a "Move Stages", fechá-la no canto direito da janela;

Move Stages				×
Go To/Step Saved	Positions			
Move Mode	Position	Direction	Units	
O Go to Position	⊠x 50	$\Rightarrow \triangleleft \triangleright$	⊚µm	Step
	Y 50	÷ 🗸 🛆		
Step	☑ z 10	÷ 🗸 🔺	Omm	Stop
	Reset			
Set XY to (0,0)	Cheme Mome	Stages		

55. Posicionar o laser no Padrao GJ, ir na coordenada "GJ-1" e clicar em "Go To";

No. 🗸 🔘 🥑 📥 Type	Description	Position	
	Default scan settings:	Χ, Υ, Ζ	
1 🗌 🗌 💭 💶 alinhamento braco		<u>Go To:</u> 82.615,84.895,20.124	4 <u>Edit</u>
2 🗌 🔲 💭 🔹 SENSOR		<u>Go To:</u> 74.130,11.380,20.277	7 <u>Edit</u>
3 🔲 🔲 🗌 📏 NIST-612		Go To: 61.910,57.050,20.247	7 <u>Edit</u>
4 🔲 🗖 🚺 🖕 GJ-1		Go To: 85.778,40.984,20.375	5 <u>Edit</u>
5 M TO M I GRAD SUP ESO		Gn Tn: 78.065.79.768.20.395	5 Edit

- 56. Posicionar o "spot" em uma área limpa do GJ;
- 57. Ativar o laser e furar o padrão;
- 58. Abrirá uma janela com a pergunta: "Do you want to open the laser shutter?", clicar em "Yes";



Contra Co	and Andrew PE. M.			1
Open Shutter	FI	RE Laser] 5	•
	Enable L	aser 🔘	×	
Laser Control			E	
Laser Control Gas.C	ontrols			
Open Shutter	FIF	RE Laser	1	
	Enable La	iser 🔾		
Warning				×
Do you war	nt to open th	e laser shutte	ar?	

Monitor do ICP-MS

59. Clicar em "Peakscan" na janela "Scan Control";

Peak Control		Deve esta	
Selected <u>M</u> ass:	219.76	•	
Selected C <u>u</u> p:	[cm] •	Set	
	Elements	<u>B</u> reak	
	Peakscan	Peakcenter	
Mass S <u>c</u> 59			
Start Mass [u]:	216	Start 🚺	
En <u>d</u> Mass (u):	223	Stop	
<u>N</u> umber of Steps:	100	Continuous <u>M</u> ode	
ntegration Time [s]:	0.131 -	📝 Integration Mode	
1997	C2 🔽 IC3	IC4	

60. Aguardar o gráfico terminar com os picos dos isótopos do Pb e U, os mesmos devem estar centrados em relação a massa virtual, se não estiverem mudar a massa virtual até os picos ficarem centrados;



61. Para alterar o valor da massa virtual, deve-se alterar o valor na janela "Cup Configuration", salvar

e na "Scan Control", fazer esse procedimento até os picos dos isótopos de Pb e U ficarem centrados em relação a massa virtual;

X Scan Cont	trol 🔗 Zoom (Optics 😎 Cup	Configuration	Detector	Calibration 😽 Ca						
Actual Cup I	Positions										
Cup Number	Species 🔻	Target [mm] CDD Defl.[V]	Actual Postion[mm]	Single Cup Positioning	Status In position						
L4-IC2							6.6				
L4-IC3	202Hg					-	X Scan Control	68 Z	oom Optics 🖤	Cup Configuration	ən 🔟 [
L4-IC4	204РЬ										
L4-IC5							Peak Control				
L4-F	206Pb	84.450	84.466	Set	Pos. OK		C. I		219.76		
L3-IC6	207РЬ				-	→	pelected Mass:		213.70	61	
L3-F	208РЬ	68.430	68.413	Set	Pos. OK		Selected C <u>u</u> p:		C (IC)	🔹 🚺 Set 🗸	
L2-F		15.336	15.360	Set	Pos. OK				Elements	Break	1
L1-F		61	6.520	Set	Pos. OK						
IC1 C-F	219.76 🔺							/ M	<u>P</u> eakscan	Peakcent	er 🚺
H1-F		6.861	54.862	Set	Undefined						
H2-F	232Th	63.200	63.202	Set	Pos. OK						
H3-F		30.801	93.411	Set	Undefined						
H4-F	238U	104.150	104.130	Set	Pos. OK						
H4-IC7											

Peak Control					
Selected Mass:	202Hg	÷			
Selected Cyp:	C (IC)	Set	t l		
	Elements	Break			
<u>M</u>	Peakscan	Peakcenter	1 🔼		
Mass Scan					
Start Mass [u]:	216	Start			
End Mass (u):	223	Stop			
Number of Steps:	100	Continuous	Mode		
Integration Time [s];	0.131 -	Integration I	Mode		
	C2 🔽 IC3	🔽 IC4			
EUCS 🚺	C6 🔤 IC7				

62. Para fazer ganho dos "MICs", colocar na massa 202Hg;

63. Deixar o sinal passando durante algum tempo pela multiplicadora de elétrons (SEM) - IC1;



64. Colocar ı	na massa	virtual:
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	Zoom Optics 🎔 Cu	p Configuration	W Detector Calibration	🛫 Center Cup	Source Lenses	• 1 Inlet Sys
Peak C <u>o</u> ntrol Selected <u>M</u> ass:	219.76	•				
Selected C <u>u</u> p:		Set	B			
	<u>E</u> lements <u>P</u> eakscan	Peak <u>c</u> enter				
Mass S <u>c</u> an						
S <u>t</u> art Mass [u]:	216	Start				
End Mass [u]:	223	Stop				
Number of Steps:	100	Continuous <u>N</u>	Mode			
Integration Time [s]:	0.131 💌	🔽 Integration M	tode			
	C2 🔽 IC3	VIC4				
IC5 🔽 I	C6 📃 IC7					

65. Deixar o sinal 202Hg passando pelo contador de íons (IC3), por um tempo; comparar se o sinal na SEM e ne correspondem, se sim, o sinal do 202Hg está calibrado;



66. Colocar na massa 204Pb;

Scan Control	S Zoom Optics 🔛	Cup Configuration	Detector Calibration	🥳 Center Cup	Source Lenses	• 11 Inlet System
Peak Control						
Colocted Mass:	204Pb					
Selected <u>M</u> ass.						
Selected C <u>u</u> p:	C (IC)	Set 🖌	J			
	Elements	Break	1			
	<u>Peakscan</u>	Peak <u>c</u> enter	J. AM			
Mass S <u>c</u> an						
	210	Chart				
S <u>t</u> art Mass [u]:	210	Sign				
Start Mass (u): End Mass (u):	223	Stop]			
Start Mass (u): En <u>d</u> Mass (u): <u>N</u> umber of Steps:	223 100	Stop]] 			

67. Deixar o sinal passando durante algum tempo pela multiplicadora de elétrons (SEM) - IC1;



68. Colocar na massa virtual:

Peak Cgntrol Selected Mass: 219.76 Selected Cup: CitCr Elements Break Elements Break Elements Break Elements Break Start Start Start Mass [u]: 216 Start Start Number of Steps: 100 Integration Time [s]: 0.131	Scan Control 68 .	Zoom Optics 🛛 😎 Cu	p Configuration 🛛 🔟	Detector Calibration	🥳 Center Cup	Source Lenses	• 11 Inlet System
Peak Cgntrol Selected Mass: 219.76 Selected Cup: CitCr Selements Break Peakscan Peakgenter Mass Sgan Start Mass [u]: 216 Start Start Mass [u]: 223 Stop Number of Steps: 100 Continuous Mode							
Selected Mass: 219.76 Selected Cup: CIEF Elements Break Peakscan Peakgenter Mass Sgan Start Mass [u]: 216 Start N Eng Mass [u]: 223 Stapp Number of Steps: 100 Continuous Mode	Peak Control						
Selected Mass: 213.76 Selected Cup: CftCr Set Elements Break Peakscan Peakcenter M Mass Sgan Start Mass [u]: 216 Start Mass [u]: 216 Start Mass [u]: 223 Stop Number of Steps: 100 Continuous Mode Integration Time [s]: 0.131	/ cak c <u>o</u> ntroi						
Selected Cup: CffCf Set Elements Break Break Break Break Break Break Break Break Break Break Break Break Break Break Deakscan Peakcenter Mass Scan Start Mass [u]: 216 Start Start Stop Number of Steps: 100 Continuous Mode Integration Time [s]: 0.131 V Integration Mode	Selected <u>Mass</u> :	219.76	•				
Elements Break Peakscan Peakcenter Mass Sgan Mass Sgan Start Mass [u]: 216 Start Start Start Start Dumber of Steps: 100 Continuous Mode Integration Time Is: 0131 Untegration Mode	Selected C <u>u</u> p:	Cher 🔹	Set				
Mass Sgan Start Mass [u]: 216 Start Mass [u]: 223 Stop Number of Steps: 100 Continuous Mode		Elemente	Break				
Peakscan Peakgenter Mass Sgan Start Mass [u]: 216 Start Mass [u]: 223 Stop Number of Steps: 100 Integration Time [s]: 0.131				-			
Mass Sgan Start Mass [u]: 216 End Mass [u]: 223 Stop Number of Steps: 100 Integration Time [s]: 0.131		<u>P</u> eakscan	Peak <u>c</u> enter	<u>N</u>			
Start Mass (u): 216 End Mass (u): 223 Number of Steps: 100 Integration Time [s]: 0.131	Mass Scan						
Start Mass [u]: 210 Start End Mass [u]: 223 Stop Number of Steps: 100 Continuous Mode Integration Time [s]: 0.131 Integration Mode		216					
End Mass [u]: 223 Stop Number of Steps: 100 Continuous Mode Integration Time [s]: 0.131 Integration Mode	Statt Mass (u):	210	Start	M			
Number of Steps: 100 Continuous Mode	En <u>d</u> Mass [u]:	223	Stop				
Integration Time [s]: 0.131 🗸 🔽 Integration Mode	<u>N</u> umber of Steps:	100	Continuous <u>M</u> oo	le			
	Integration Time [s]:	0131	Integration Mod	e			
		C2 🔽 IC3	V 1C4				
	🔲 IC5 🛛 💟 I	C6 [] 1C7					

69. Deixar o sinal 204Pb passando pelo contador de íons (IC4), por um tempo; comparar se o sinal na SEM e na IC4 correspondem, se sim, o sinal do 204Pb está calibrado;



70. Colocar na massa 207Pb;

Scan Control 68	Zoom Optics 🛛 😎 🕻	up Configuration 🛛 💆	Detector Calibration	🥳 Center Cup	Source Lenses	• 🗄 Inlet System
Peak Lontrol						
Selected <u>M</u> ass: (207РЬ					
Selected Cup:		Set 🚽				
	(
	Elements	<u>B</u> reak				
	Peakscan	Peak <u>c</u> enter	M			
N	35					
Mass S <u>c</u> an						
S <u>t</u> art Mass [u]:	216	St <u>a</u> rt 🖌	M			
End Mass [u]:	223	Stop				
Number of Steps:	100	Continuous Mod	_			
	1328	Continuous Mod	•			
Integration Time [s]	0.131 -	🛛 🔽 Integration Mode				

71. Deixar o sinal passando durante algum tempo pela multiplicadora de elétrons (SEM) - IC1;



72. Colocar	na	massa	virtual:
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Scan Control 🔗 .	Zoom Optics 🔛 Cu	ip Configuration 🛛 💆 D	etector Calibration	🥳 Center Cup	Source Lenses	• 🕅 Inlet Syste
Peak Control						
Selected <u>M</u> ass:	219.76	•				
Selected Cup:		Set				
	Elements	вгеак				
		Peak <u>c</u> enter				
Mass b <u>c</u> an						
Start Mass [u]:	216	Start				
En <u>d</u> Mass [u]:	223	Stop				
Number of Steps:	100	Continuous Mode				
integration (ime (s);	U.131 •					
	C2 📝 IC3	📝 IC4				
🗖 IC5 🛛 🔽 I	C6 📃 IC7					

73. Deixar o sinal 207Pb passando pelo contador de íons (IC6), por um tempo; comparar se o sinal na SEM e na IC6 correspondem, se sim, o sinal do 207Pb está calibrado;



73. Se o sinal do MIC não estiver coincidente com o sinal da SEM;

74. Verifique se é necessário aumentar ou diminuir o sinal no contador de íon (IC) para corresponder com o sinal na SEM;

75. Ir na janela "Application Start" e clicar em "X (Executive)";



76. Abrir a janela "Ion Counter";

77. Os MICs que devem ser alterados, são respectivamente: IC3 para 202Hg, IC4 para 204Pb e IC6 para o 207P

Ion	Counter Da	ta - Executive	-												
<u>F</u> ile	<u>V</u> iew <u>C</u> ust	omize <u>W</u> indow <u>H</u>	elp												
3	I 🕹 📅														
Mass	Calibration	Collector Ion Counter	Amp	olifier History	Log Files Auto	isampler									
Line	Ion counter	Installation Date	Туре	Dead Time [ns]	Dead Time Base Frequency [cps]	Dead Time Factor	Dark Current [V]	Max Current [V]	Plateau Voltage [V]	Operation Voltage [V]	Yield [%]	Mass [u]	Yield Date	Noise [cpm]	Noise Date
1	1	28/06/2000 08:40:37	IC	20.00000	1000.0	0.000000	2.11928e-010	0.000e+000	2400.076	2350.000	99.219	206.97600	30/11/2017 10:50:19	0.79	30/11/2017 10:26:53
2	2	10/01/2007 10:18:22	IC	70.00000	1000.0	0.000000	2.01331e-009	0.000e+000	2100.000	2850.000	80.000	206,97600	30/10/2017	7.55	30/11/2017 10:26:53
3	⇒ 3	10/01/2007 10:18:22	IC	70.00000	1000.0	0.000000	2.01331e-009	0.000e+000	2125.000	2890/000	88.500	206.97600	30/10/2017	7.55	30/11/2017 10:26:53
4	4	10/01/2007 10:18:22	IC	70.00000	1000.0	0.000000	1.55449e-008	0.000e+000	2150.000	2650.000	91.401	206,97600	30/10/2017	58.29	30/11/2017 15:33:38
5	5	10/01/2007 10:18:22	IC	70.00000	1000.0	0.000000	3.14713e-009	0.000e+000	2580.000	2950.000	92.287	206.97600	30/10/2017	11.80	30/11/2017 10:44:27
8	6	10/01/2007 10:18:22	IC	70.00000	1000.0	0.000000	4.86374e-009	0.000e+000	2280.000	2880.000	90.500	206.97600	30/10/2017	18.24	30/11/2017 10:44:27
7	7	10/01/2007 10:18:22	IC	70.00000	1000.0	0.000000	5.05447e-009	0.000e+000	2325.000	2700.000	93.123	206,97600	30/10/2017	18.95	30/11/2017 10:44:27
8	8	14/04/1998 12:07:00	IC	70.00000	1000.0	0.000000	0.00000e+000	0.000e+000	1200.000	0.000	100.000	0.00000	00:00:00	0.00	00:00:00
4							Ш								
													0	_	

78. Para calibrar o sinal nos MICs, deverá ser alterado o valor do "Operation Voltage" ou do "Yield (%) até o sinal no MIC ficar equiparado ao sinal da SEM (mutiplicadora de elétrons);

78.1 "Operation Voltage" funciona da seguinte forma: para aumentar o sinal, deve-se aumentar o valor da voltagem, que deve chegar no máximo à **3000**V e para diminuir o sinal, deve-se diminuir o valor da voltagem, faça essa mudança de 20 em 20V;

Mass	Calibration	Collector Ion Counter	Amp	blifier History	Log Files Auto	sampler						
Line	lon counter	Installation Date	Туре	Dead Time [ns]	Dead Time Base Frequency [cps]	Dead Time Factor	Dark Current [V]	Max Current [V]	Plateau Voltage [V]	Operation Voltage [V]	Yield [%]	Mass [u]
1	1	28/06/2000 08:40:37	IC	20.00000	1000.0	0.000000	2.11928e-010	0.000e+000	2400.078	2350.000	99.219	206.97600
2	2	10/01/2007 10:18:22	IC	70.00000	1000.0	0.000000	2.01331e-009	0.000e+000	2100.000	2850.000	80.000	206.97600
3	3	10/01/2007 10:18:22	IC	70.00000	1000.0	0.000000	2.01331e-009	0.000e+000	2125.000	2890.000	88.500	206.97600
4	4	10/01/2007 10:18:22	IC	70.00000	1000.0	0.000000	1.55449e-008	0.000e+000	2150.000	2650.000	91.401	206.97600
5	5	10/01/2007 10:18:22	IC	70.00000	1000.0	0.000000	3.14713e-009	0.000e+000	2580.000	2950.000	92.287	206.97600
6	6	10/01/2007 10:18:22	IC	70.00000	1000.0	0.000000	4.86374e-009	0.000e+000	2280.000	2880.000	90.500	206.97600
7	7	10/01/2007 10:18:22	IC	70.00000	1000.0	0.000000	5.05447e-009	0.000e+000	2325.000	2700.000	93.123	206.97600
8	8	14/04/1998 12:07:00	IC	70.00000	1000.0	0.000000	0.00000e+000	0.000e+000	1200.000	0.000	100.000	0.00000
•							III					

78.2 "Yield (%)": para aumentar o sinal no MIC, deve-se diminuir a porcentagem do "Yield" e para diminuir o sinal no MIC, deve-se aumentar a porcetagem do "Yield";

Mass	Calibration	Collector Ion Counter	Amp	olifier History	Log Files Auto	osampler						/
Line	Ion counter	Installation Date	Туре	Dead Time [ns]	Dead Time Base Frequency [cps]	Dead Time Factor	Dark Current [V]	Max Current [V]	Plateau Voltage [V]	Operation Voltage [V]	Yield [%]	Mass [u]
1	1	28/06/2000 08:40:37	IC	20.00000	1000.0	0.000000	2.11928e-010	0.000e+000	2400.076	2350.000	99.219	206.97600
2	2	10/01/2007 10:18:22	IC	70.00000	1000.0	0.000000	2.01331e-009	0.000e+000	2100.000	2850.000	80.000	206.97600
3	3	10/01/2007 10:18:22	IC	70.00000	1000.0	0.000000	2.01331e-009	0.000e+000	2125.000	2890.000	88.500	206.97600
4	4	10/01/2007 10:18:22	IC	70.00000	1000.0	0.000000	1.55449e-008	0.000e+000	2150.000	2650.000	91.401	206.97600
5	5	10/01/2007 10:18:22	IC	70.00000	1000.0	0.000000	3.14713e-009	0.000e+000	2580.000	2950.000	92.287	206.97600
6	6	10/01/2007 10:18:22	IC	70.00000	1000.0	0.000000	4.86374e-009	0.000e+000	2280.000	2880.000	90.500	206.97600
7	7	10/01/2007 10:18:22	IC	70.00000	1000.0	0.000000	5.05447e-009	0.000e+000	2325.000	2700.000	93.123	206.97600
8	8	14/04/1998 12:07:00	IC	70.00000	1000.0	0.000000	0.00000e+000	0.000e+000	1200.000	0.000	100.000	0.00000
•												

Monitor do Laser

79. Depois que os sinais nos MIC e na SEM estiverem equiparados, posicionar o "spot" em uma área limpa do



80. Ativar o laser e furar o padrão;

Laser Control		Laser Control	80
Laser Control Gas (Controls	Laser Control Gas C	ontrols
Open Shutter	FIRE Laser	Open Shutter	FIRE Laser
	Enable Laser 🔘		Enable Laser 🚫

81. Clicar em "Peakscan" na janela "Scan Control";

219.76			
C-(IE) -	Set		
Elements	Break		
Peakscan	Peak <u>c</u> enter		
216	Start ///		
223	Stop		
100	Continuous <u>M</u> ode		
0.131 👻	V Integration Mode		
	213.76 CT(E) Elements Peakscan 216 223 100 0.131	213.76 Set Elements Break Peakscan Peakgenter 216 Start 223 Stop 100 Continuous Mode 0.131 V Integration Mode	213.76 Set Elements Break Peakscan Peakgenter 216 Start 223 Start 100 Continuous Mode 0.131 V Integration Mode

82. Aguardar o gráfico terminar, os picos dos isótopos de Pb e U devem continuar centrados em relação a mas virtual;, se não estiverem centrados, altere o valor da massa virtual e faça o "Peakscan" no padrão GJ até ficarem centrados;



83. Se os isótopos estiverem centrados com a massa virtual o aparelho estará prontos para iniciar as análises.

PROCEDIMENTO PARA TROCA DE MOUNT

1. Desrosquear os parafusos do porta-amostra, desencaixar e retirar o porta-amostra;



2. Posição do porta-amostra retirado da câmara do laser com as amostras

localizadas no lado de cima do mount;



3. Colocar o porta-amostra na posição em que as amostras fiquem para baixo, retirar a placa, a mola e o mount que deverá ser trocado;





3 - Guardar o mount que foi retirado no seu respectivo pote;



- 4 Colocar o mount com a amostra na posição para baixo, seguindo a posição
- "Norte" do porta-amostra;

PROCEDIMENTO PARA TROCA DE FLUORETO DE ARGÔNIO - COMPUTADOR DO LASER

- 1. O fluoreto de argônio só deve ser trocado quando a "Voltage =1.2 V";
- 2. Abrir a pasta "ATLEX Laser Control";



3. Clicar em "Gas Control";

Communication Permisso	ns				
ser Control Gas Control	Settings Info			ATL Laser Advanced T	technik GmbH AT
Pressure [mbar]	Descenses over		Manual Valve Control		
5991 6044 7900	Laserhea	d Lase	Pre Pu r Vac	rge uum	
0	Auto Gas Exchange	Mirror Exchange	Transport and Storage	Gas Cylinder Exchange Start	Gas System Check
Laser Head Manifold	Gas action status: Idle				
rors: No errors					
atus: Laser Off					

4. Em "Auto Gas Exchange" clicar em "Start";

Communication Permisso	ons				
				ATL Laser	technik GmbH AT
				Advanced T	echnology Lasers
aser Control Gas Control	Settings Info				
Pressure [mbar]			Manual Valve Control		
5991 6044	nergen.		Pre	mix	
7900	41 2				-
					-
	Stop		Pu	rge	
				<u> </u>	
		The second s		-	
		Lase	er Vac	uum	
				di di i i i	
	Laserhea			< Pi	ump 🔶
	Laserhea			P	ump 🔶
	Laserhea Auto Gas Exchange	Mirror Exchange	Transport and	Gas Cylinder	Gas System Check
	Laserhea Auto Gas Exchange	Mirror Exchange	Transport and Storage	Gas Cylinder Exchange	Gas System Check
	Laserhea Auto Gas Exchange	Mirror Exchange	Transport and Storage	Gas Cylinder Exchange	Gas System Check
0	Laserhea Auto Gas Exchange	Mirror Exchange	Transport and Storage	Gas Cylinder Exchange Start	Gas System Check
0	Laserhea Auto Gas Exchange	Mirror Exchange	Transport and Storage	Gas Cylinder Exchange	Gas System Check
0 Laser Head Manifold	Laserhea Auto Gas Exchange Stat Gas action state: Idle	Mirror Exchange	Transport and Storage	Gas Cylinder Exchange	Gas System Check
0 Laser Head Manifold	Laserhea Auto Gas Exchange Stat Gas action state: Idle	Mirror Exchange	Transport and Storage	Gas Cylinder Exchange	Gas System Check
0 Laser Head Manifold irrors: No errors Xatus: Laser Off	Auto Gas Exchange Stat Gas action state: Idle	d Mirror Exchange	Transport and Storage	Gas Cylinder Exchange Start	Gas System Check



5. Abre automaticamente a janela: "Are both gas cylinders closed?" Clica em "Ok";

6. Abre automaticamente a janela: "Open Premix cylinder, set secondary pressure 6 bar";



7.Na câmera onde fica o cilindro de Fluoreto de Argônio, abrir a válvula do cilindro e a válvula do sistema;



8. Depois que abrir as válvulas, voltar no monitor e clica em "Ok";

ATLEX Laser Cor	atrol Software						-
 Communication 	on Permissor	IS			ATL Laser Advanced T	lechnik GmbH echnology Lasers	ATL
Laser Control	Gas Control	Settings Info					
Pressure	[mbar]			Manual Valve Contro	bl		
5989 7900	301	Stop Auto	gas exchange Ipen Premix cylinder, set sec bar	ondary pressure to 6	× ge um		
0		Auto Gas Exch	OK Cancel	Start	Gas Cylinder Exchange	Gas System Ch	eck
Laser Head	Manifold	Gas action status: Auto	gas exchange in progress				
Errors: No error	rs						
Errors: No error Status: Laser C	rs Xff						

9. Depois que o aparelho fizer toda a troca de gás, abrirá uma janela automaticamente, pedindo para fechar as válvulas do gás e do sistema;



10. Fechar as duas válvulas, voltar no monitor e clicar em "Ok";





11. Fechar o programa, clicar em "File", clicar em "Exit", abrirá uma janela"Exit laser control?", clicar "Yes".

ATL Lasertechnik GmbH Advanced Technology Lasers Atraced Technology Lasers Total Tot	Communication Perm	issons			
Advanced Technology Lasers				ATL Lasert	technik GmbH AT
Laser Control Ges Control 6025 6004 6025 6004 6025 6004 6025 6004 6025 6004 6025 6004 6025 6004 6025 6004 6025 6004 6025 6004 6025 6004 900 900	Lange Castral Cas Can	test Continue Info		Advanced Te	echnology Lasers
Pressure (mbar) 6025 0004 7900 Laser head Manifold Errors: No errors Status: Laser Off	Caser Control Gas Con	Settings into			
6025 6004 7900 Premix Purge Purge Vacuum Pump Auto Gas Exchange Sit laser control? Start Start Start Start Errors: No errors Status: Laser Off	Pressure [mbar]		Manual Valve Control	1.0	
7900 Purge Purge Vacuum Purge P	6025 6004	omergency	Pre	emix	
Frors: No errors Status: Laser Off	7900			_	
Stop Full ye Vacuum Pump Auto Gas Exchange Exit laser control Insport and Gas Cylinder Start Start Start Start			D	irao	
Laser Head Manifold Exchange Start Start Start Start Start Starts: Laser Off		Stop	FU FU	iige	
Laser Head Manifold Errors: No errors Status: Laser Off					
Laserhead Manifold Cas Exchange Cas System Check Cas Exchange Cas Excha			Laser Vac	cuum	
Errors: No errors Status: Laser Off				Construction of the second	
Auto Gas Exchange Start Star		Laserhead		PL	ump —
Errors: No errors Status: Laser Off	- 11	Laserhead	Exit laser control?	Pi	ump 🔶
0 Start Start Start Start Start Start Laser Head Manifold Gas action status. Idle Errors: No errors	- 11	Laserhead	Exit laser control?	Gas Cylinder	Gas System Check
O Start Start Start Start Laser Head Manifold Gas action status. Idle	11	Laserhead Auto Gas Exchange	Exit laser control?	Gas Cylinder Exchange	Gas System Check
Laser Head Manifold Gas action status. Idle Errors: No errors Status: Laser Off	11	Laserhead Auto Gas Exchange	Exit laser control?	Gas Cylinder Exchange	Gas System Check
Errors: No errors Status: Laser Off	0	Laserhead Auto Gas Exchange	Exit laser control? Exit laser control? Xee No Storage Stat Stat	Gas Cylinder Exchange Start	Gas System Check
Errors: No errors Status: Laser Off	0	Laserhead Auto Gas Exchange	Exit laser control? Exit laser control? Xee No Storage Storage Storage	Gas Cylinder Exchange Start	Gas System Check
Status: Laser Off	0 Laser Head Manifol	Auto Gas Exchange Start	Exit laser control? Exit laser control? Xee No Storage Start Start	Gas Cylinder Exchange Start	Gas System Check
Status: Laser Off	0 Laser Head Manifoli	Auto Gas Exchange Start Gas action status: Idle	Exit laser control?	Gas Cylinder Exchange Start	Gas System Check
	0 Laser Head Manifol Errors: No errors	Auto Gas Exchange Start	Exit laser control?	Gas Cylinder Exchange	Gas System Check

PROCEDIMENTO PARA DESLIGAR O ICP-MS

1. Center Cup - clicar em Center Cup

🐼 Zoom Optics	Oup Configuration	2 Detector Calibration	🕳 Center Cup	🚺 Source Lenses	• 1). Inlet System
File: Sandra.cen			1		
Switching					
dar.	Mode	Faraday			
NIF	mode	× Allerter			
E E	82497	+ 8249.7			
		00 9369			
	68 Zoom Optics File: Sandra.cen Switching lay	65 Zoom Optics De Oup Configuration File: Sendra.cen Switching lay Mode	65 Zoom Optics Cup Configuration 2 Detector Calibration File: Sendra.cen Switching lay Mode Faraday 1 - 82497 + 8249.7 2 Detector Calibration	65 Zoom Optics Cup Configuration 2 Detector Calibration Configuration 2 Detector Calibration Configuration 2 Center Cup File: Sendra.cen Switching lay Mode 9 Faraday	65 Zoom Optics Cup Configuration 2 Detector Calibration Center Cup 2 Source Lenses File: Sendra.cen Switching lay Mode Faraday 1 - 83697 + 8249.7 Particular (2007) - 2007 -

1.1. Clicar em Faraday - o quadrado deve ficar ticado (v)

🔨 Scan Control 🛛 🔗 Zoom Optic	s 💿 Cup Configuration	2 Detector Calibration	of Center Cup	🔕 Source Lenses	• 1) Inlet System
😹 🖬 🌒 File: Sandra.ce	en				
Faraday SEM Switching					
Faraday	Mode	Faraday			
		and and a second			
RPQ Tuning		and the second second			
Decelerator [V]:	82497	+ 8249.7			
A CONTRACTOR OF	and the second s	A 88 6 4 6			

- 2. Scan Control Clicar em Scan Control
- 2.1. Selected Mass: Clica na massa 7Li
- 2.2. Clica em Set



3.Tune Neptune

• When the state of the second state of the se	T start-start	
Not and		
	E	The Control of the Control of the C

3.1. Na tela do Tune ir no Watch Parameter - Hv on - clica em on - fica off e vermelho



3.2. Analyzer Gate Open - clica em on fica off e vermelho



3.3. Start and Stop Plasma

3.3.1. Clica em off



3.3.2. Todo o Sistema fica vermelho



PROCEDIMENTO PARA DESLIGAR O LASER

1. Chromium 2.3



1.1. Clicar em Gas Controls



1.1 Clicar em Bypass

G	as Controls	П.				
	¥ Laser Control 🛛 🖧	Gas Controls		1		
Ξ	Sample Chamber V	alves		/		
	Bypass		0			
	Online		0			
-	Purge		Ø			
	🗄 Purge Progress	<u> </u>				
E	Evacuate		0			
	🖃 Status:	Done				
	Vacuum Motor	Off				
	MFC1 Pressure	13.61		PSI		
	MFC2 Pressure	13.47		PSI		
Ξ	Mass Flow Controllers					
	Auto Flow Control					
	Shut Down All Flows	0				
E	MFC1: double-click to .	0.000		[LPM]		
	Setpoint (He)	0.000		[LPM]		
-	MFC2: double-click to .	0.000		[LPM]		
	Setpoint (He)	0.000		[LPM]		
۲	MFC3: double-dick to .	0.000		[mLPM]		
	Setpoint (N2)	0.000		[mLPM]		
	Gas Control Setup			0		

- 2. Clicar em File
- 2.1. Clicar em Exit



FECHAR OS GASES

1. Hélio - Fechar a válvula do Cilindro de He, girar sentido horário





2. Nitrogênio - Fechar o N2 - válvula na parede, virar no sentido horário





3. Argônio - Só pode fechar o Argônio quando no "Start and Stop Plasma o sistema estiver todo vermelho;

Image: Start and Stop Plasma

Image: Drift Settings

Settings

Image: Drift Setting Settin

3.1. Fechar a válvula verde na parede do Argônio, virar sentido horário.

