

Title: Chemical Lab Sample Analysis

Rev. 6

Safe Job Procedure

LOTO Procedure: LTO-0005-MSM / LTO-0006-MSM/ LTO-0008-MSM (for Spectrometers)

LOTO procedure: LTO-0009-MSM (for Grinder / dust collector)

Responsible Position: QC Tech

Safety Apparel, Tools or Equipment Needed:

Side-Shield Safety Glasses/Goggles and/or Face Shield	Coverall and/or Greens	Steel Toe Boots w/ Metatarsals
Gloves	Ear Plugs or Ear Muffs	Coverall and/or Greens
Hard Hat (outside lab)	Long Sleeves	


Sequential Description of the Procedure

Hazards / Quality Impact

<i>Sample Preparation – All Samples</i>	
1.1 Retrieve sample from the vacuum tube chamber or drop tube. Wear gloves, assume all samples are hot and glass is also present.	SPECIAL HAZARDS: Eye injury, burns and cuts to fingers. Make sure no other sample is on the way before opening the door to the vacuum chamber, or another sample could be about to be dropped
1.2 Break stem from the sample. Wear the safety glasses and the tight fit rubber glove.	Pieces of glass from the sampler can fly away and get into the eyes

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<p>1.3 Use auto-grinder or manual disk grinder to obtain sufficient surface for testing.</p> <p>Auto-Grinder: Place sample on belt and push start button. <i>When the sample emerges at the end of the process, wait for the belt to come to a complete stop before picking up the sample.</i></p> <p>Disk Grinder: Use, coarse (24 or 36 grit) paper first, and then finish with fine (80 grit) paper. Grind sample at least 1/32" to ensure removal of the decarburized layer. Hold sample with the magnet*. Use tight-fit rubber gloves. Ensure grinding disk is securely fastened, and no one else is in the grinding room.</p> <p>*For grinding samples of billet or finished goods, it may be necessary to hold the sample directly with your glove. Also, billets are more likely to have slag around the sample edges, increasing the chance that debris will fly off the sample as you grind it.</p>	<p>Safety glasses required for all sample types</p> <p>If the belt is still turning, it could pull your fingertip into the tiny gap between the belt and the copper cooling plate.</p> <p>Grinding disk can tear and disintegrate into many flying pieces; sample can slip off from the holding magnet, finger tips can touch the grinding disc causing injury, grinding dust flying in the air.</p>
<p><i>Furnace Test Analysis</i></p>	
<p>2.1 Cool/Dry sample. Hold sample with glove or channel locks and place it on the copper plates to cool, if water is not used. If compressed air is used care must be taken not to blow scale or dirt into your face or others' faces. Use safety glasses.</p>	<p>The grinding process creates a very hot piece of metal, so treat it accordingly.</p>
<p>2.2 Take sample to Spectrometer and analyze it.</p> <ul style="list-style-type: none"> • Place the sample GENTLY on the spectrometer analyzer table to prevent cracking the insert • Make sure the sample is held in place securely by the sample holder to create good contact • Make sure sample door is fully closed before starting the analysis (if not – machine interlock will prevent analysis) 	<p>Sample can still be hot. Damage can occur to the insert in the analysis table. Electric shock if analysis table door is not closed.</p>
<p>2.3 Remove sample from the spectrometer. Be cautious when collecting all the samples in the brown bags before storing them.</p> <p>Inspect the burn to make sure it was a good burn – On primary ARL (4460) and secondary ARL (3460) good burn has black iris with white in the middle.</p>	<p>Sharp points from the broken stems.</p> <div data-bbox="959 1602 1127 1871">  </div> <p>Good burn has black iris and is white in the middle</p>

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<p>2.4 If the burn is good, make sure each element is below the maximum allowed for the intended product. Determine the Melters' aims for the C, Mn, Si, and V, if V is required.</p> <p>Communicate aims to the furnace crew. (If the furnace test is okay except for high S and P, see if the Melter can lower them enough to make the intended grade.)</p>	<ul style="list-style-type: none"> • Always look for the presents of Pb, and let the Melter and or Caster know if any is detected. • If Pb is above .01, respirators must be worn on the Caster. <p>(Q) Pay attention to the sound emitted from the spectrometer; a hollow sound is an indication that the sample is bad or the sample is not covering completely the hole in the analysis table</p>
<p>2.5 Place the FR sample in NUCSAFE LAB ANALYZER unit for additional Radiation Check (see SJP-0009-ENV)</p>	
<p>2.6 Place the samples inside the paper bag labeled with the correct heat number. Save samples in cabinet in the test room.(sample is typically retained for several weeks in case any question arises on chemistry)</p>	
<p><i>Ladle Top Analyses</i></p>	
<p>3.1 After burning the first ladle top and verifying that the burn is good, check the Carbon Equivalent (CE). If CE is at least a few points higher than the minimum required, and the individual elements are all within the minimum and maximum limits, instruct the Casting Foreman not to make any trim additions.</p> <ul style="list-style-type: none"> • If the silicon or manganese is in danger of falling below the allowed minimum, additions will have to be made. (This is true even if the carbon equivalent is okay.) 	
<p>3.2 When making merchant products, pay attention to the ratio of manganese to silicon. <i>As a rule of thumb, this ratio should not be lower than 3.0.</i> In extreme cases, manganese may have to be added to bring the ratio close to 3.0, even if the heat then has to be diverted.</p>	<p>(Q) Steel with too low of a manganese to silicon ratio will be difficult to cast due to entrapped air (not fully killed), and will result in a poor quality finished product.</p>
<p>3.3 If the chemistry is not withing the spec, give new aims to the casting crew.</p>	
<p>3.4 Repeat items 3.1 through 3.4 for each successive ladle top</p>	
<p>3.5 Place ladle samples into same paper bag as furnace test labeled with correct heat number.</p>	

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<i>Ladle Bottom & First Tundish Analyses</i>	
4.1 Analyze the ladle bottom or first tundish test as soon as it arrives, and determine whether the heat should be cast as the intended product.	
<p>General guideline for diverted heat assignment:</p> <ul style="list-style-type: none"> • Heats intended as grade 60, A706, or 400W should be diverted to a smaller bar size or to grade 40 if the carbon equivalent is below the minimum required for the intended product. • If the carbon is more than three points above the aim (or if it is above .30 when making A706 or 400W), divert the heat to 60 grade for a larger bar size. 	
4.2 When diverting a heat, it may be necessary to inform the torchman of the new cut length. If necessary, instruct him to add or subtract two or more billets per strand at the sequence, and request billet samples.	
<i>Tundish Finals</i>	
5.1 After receiving the second tundish samples grind all samples and run them on the spectrometer. Samples with a bad burn must be rerun.	
5.2 After obtaining good burns on each of the samples, check the results for evidence of segregation. This is indicated by a difference of at least 0.04 C, 0.10 Mn, or 0.07 Si from the highest to the lowest sample. If the heat is segregated it will normally be shown by the last tundish sample. Immediately notify casting crew of the finding & sample to isolate questionable material	(Q) If you see a low / high reading in one of the tundish samples, DO NOT just exclude the reading because the low/high reading may indicate that there is segregation in the heat. Re-grind the sample that is giving the abnormal reading and burn it again to verify the reading.
5.3 Review chemistry of the averaged tundish samples to determine whether the heat should be graded as originally intended.	
5.4 Analyze the standard at least every four hours, and drift correct the spectrometer if necessary.	
5.5 Place all tundish samples in the same paper bag for the heat.	
<i>Machine Maintenance – Changing Grinding Disc</i>	
1. Lock out grinder prior to changing grinding disk to prevent unexpected rotation of grinding wheel – see LOTO procedure LTO-0001-MSM	
2. Pull on the ON button to test to make sure the machine has been de-energized from the lockout	

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3. Remove protective ring from grinding head	
4. Peel off spent grinding disk from the grinding head, replace the proper grid sand paper on the grinding head – make sure the sand paper is well centered on the grinding head.	
5. Replace the protective ring onto the grinding head, make sure the ring sits properly on the table – look for uneven gap & adjust the ring position until it sits flat on the surface	
6. Unlock machine & ready for use	

Machine Maintenance – Troubleshooting ARL 3460

All major maintenance on the spectrometers are performed by specially trained professionals to prevent unintended modification to the high precision measuring equipment. However, there are several preventive maintenance items that should be performed lab technician/QC personnel on regular basis to ensure normal functioning of the equipment	
1. Gas filter cartridge <ul style="list-style-type: none">• Located in the front of the machine behind the small lower middle metal panel.• When the cartridge appears black – it shall be changed to prevent the black residual from moving to other parts of the machine.	(Q) Cartridge rating – 20 micron (resin – impregnated cellulose cartridge)
2. Vacuum Pump <ul style="list-style-type: none">• If it observed that the vacuum level of spectrometer goes below the optimal operating level (below 20 μm). The most common causes of such issue is due to vacuum pump malfunction• Contact lab supervisor if pump change is required	
3. Machine Temperature <ul style="list-style-type: none">• Spectrometers are highly sensitive measuring instrument, reading can be affected by the internal temperature of the machine and therefore the machine is internally regulated with thermocouples to ensure proper operation.	

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Title	Inserting/Exchanging Bruker Grinder Belt	Location	Quality Control
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Safety Apparel, Tools or Equipment Needed;

Hard Hat	Glasses w/ Side Shields	Steel Toed Boots w/ Metatarsals
Ear Plugs	Coverall and/or Greens	
Long Sleeves	Standard Hex Key Set	


Sequential Description of the Procedure

Hazards

<i>Belt Replacement</i>	
1. Lock out grinder prior to changing grinding belt to prevent unexpected rotation of grinding belt – see LOTO procedure LTO-0009-MSM	
2. Remove belt guard.	
3. Open the safety door on the right side by rotating the black knob located on the right side of the machine.	
4. Turn “Easy Lock” lever clockwise until it is in the unfasten position.	
5. Slide off old grinding belt.	
6. Put in new grinding belt so that it is lined up in the middle of the rollers.	

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<p>7. Turn the “Easy Lock” lever counterclockwise back to the tight position.</p>	
<p>8. Check the tension of the grinding belt. There should be minimal deflection when moderate pressure is applied. If not tight enough, turn the “Easy Lock” lever back to the unfastened position and rotate the adjacent black regulating knob clockwise slightly before retightening the lever and again checking the tension.</p>	
<p>9. Shut the safety door, replace belt guard, remove locks, and pull emergency stop button out to the “on” position.</p>	
<p>10. Follow “ADJUSTING THE BELT” procedure.</p>	
<p><i>Adjusting the Belt</i></p>	
<p>1. Set the program switch to position “0”. (The indicator light should now be flashing.)</p>	
<p>2. Hold the start button down to begin rotating the belt mechanism. Releasing the start button will stop the rotation.</p>	
<p>3. Inspect the position of the belt on the roller as it rotates and adjust accordingly.</p> <ul style="list-style-type: none"> a. Rotating the black regulating screw clockwise will cause the belt to migrate to the left side. b. Rotating the black regulating screw counterclockwise will cause the belt to migrate to the right side. 	
<p>4. Once the belt has a continuous straight motion in the position in the middle of the roller release the start button.</p>	
<p>ADJUSTING THE SAMPLE BRACKET</p>	

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1. Lock out grinder prior to changing grinding belt to prevent unexpected rotation of grinding belt – see LOTO procedure LTO-0009-MSM	
2. Remove belt guard.	
3. Open the safety door on the right side by rotating the black knob located on the right side.	
4. Place the sample in the V-slider as it would be “caught” during normal operation.	
5. Pull the piston rod down to apply pressure down on the sample.	
6. If the pressure point is not centered on the sample, loosen the two set screws counterclockwise and move the cylinder with its holder to the correct position.	
7. Tighten the set screws clockwise to lock the cylinder in place.	
8. Remove the sample from the machine.	
9. Shut the safety door, replace belt guard, remove locks, and pull emergency stop button out to the “on” position.	

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Title	Calibration Verification & Drift Correction of ARL 3460 and 4460 Spectrometers	Location	Chem. Lab
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Safety Apparel, Tools or Equipment Needed:

Coverall and/or Greens (optional)	Glasses w/ Side Shields	Steel Toed Boots w/ Metatarsals
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Note: The calibration of the primary spectrometer needs to be verified at the beginning of each shift and at approximately 4 and 8 hours into the shift. **The results of this verification shall be logged.** If the spectrometer is found to be out of calibration, use the following drift-correction procedure to bring it back within the acceptable range.

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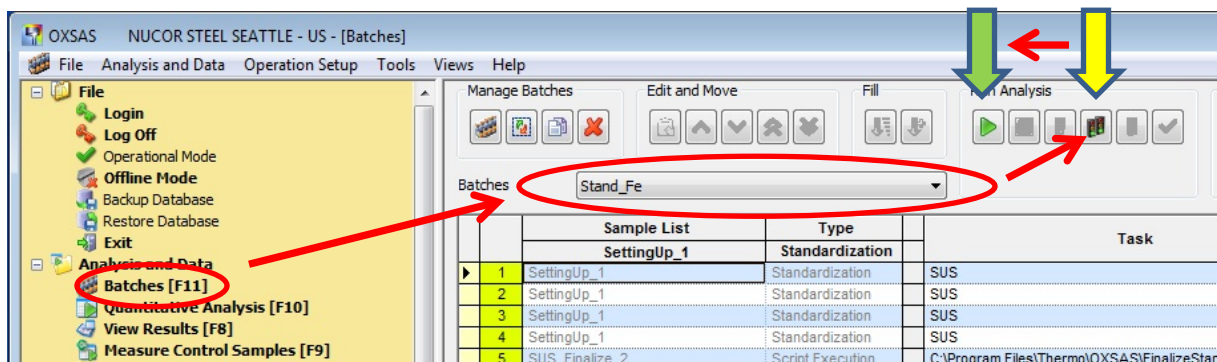
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Standardization Procedure for OXSAS Software on all Spectrometers

- 1) From the main screen, select “batches” (red circle below) or press F11.
- 2) In the drop down box select the batch that shows the 4 required SUS samples as well as the 5th “finalize” step.
- 3) Click on the button under the yellow arrow to run all SUS samples.
- 4) Click the “Run” button under the green arrow.



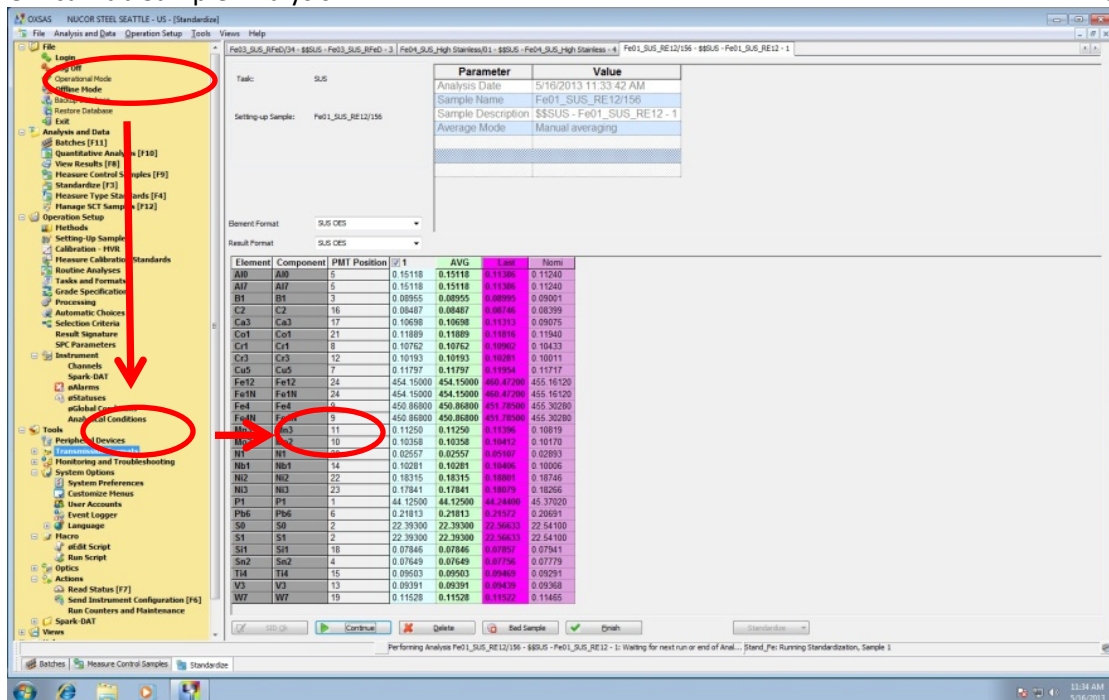
- 5) Check that the SUS sample ID matches the ID shown (circled) and place it on the spark stand.
- 6) Hit “continue” to analyze the sample (circled).
- 7) Adjust sample to test fresh area and hit “continue” to analyze the sample (circled) again.
- 8) Once you have two analyses that agree well, click “finish” (circled) to progress to the next sample and repeat steps 5 through 8. For most elements, agreeable results are under a standard deviation percentage of 3%. Some elements such as Calcium can typically be more variable so obtaining low SD% for these elements is not imperative. More attention should be paid to elements relevant to our process (C, Mn, S, P, Si, Cu, Cr, Mo, Sn, Ni, Nb, V, Pb, Al and N).

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This is what the screen will look like when you have burned multiple samples. Columns that are “checked” at the top will be used in the standardization. The SD% shows the variation, and this should be under 3% for the elements we are looking for.

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XSAS NUCOR STEEL SEATTLE - US - [Standardize]

File Analysis and Data Operation Setup Tools Views Help

Fe03_SUS_RFeD/34 - \$\$SUS - Fe03_SUS_RFeD - 3 Fe04_SUS_High Stainless/01 - \$\$SUS - Fe04_SUS_High Stainless - 4 Fe01_SUS_RE12/156 - \$\$SUS - Fe01_SUS_RE12 - 1

Task: SUS

Setting-up Sample: Fe01_SUS_RE12/156

Parameter	Value
Analysis Date	5/16/2013 11:33:42 AM
Sample Name	Fe01_SUS_RE12/156
Sample Description	\$\$SUS - Fe01_SUS_RE12 - 1
Average Mode	Manual averaging

Element Format: SUS OES

Result Format: SUS OES

Element	Component	PMT Position	1	2	3	4	AVG	Last	Nomi	SD	SD%
Al0	Al0	5	0.15118	0.15951	0.13731	0.14586	0.14478	0.11386	0.11240	0.006999	4.83
Al7	Al7	5	0.15118	0.15951	0.13731	0.14586	0.14478	0.11386	0.11240	0.006999	4.83
B1	B1	3	0.08955	0.09018	0.09171	0.09260	0.09128	0.08995	0.09001	0.001569	1.72
C2	C2	16	0.08487	0.08412	0.08507	0.08673	0.08556	0.08746	0.08399	0.001022	1.19
Ca3	Ca3	17	0.10698	0.10130	0.11480	0.15076	0.12418	0.11313	0.09075	0.023351	18.80
Co1	Co1	21	0.11889	0.11914	0.11853	0.11942	0.11895	0.11816	0.11940	0.000452	0.38
Cr1	Cr1	8	0.10762	0.17382	0.10772	0.11934	0.11156	0.10902	0.10433	0.006739	6.04
Cr3	Cr3	12	0.10193	0.10712	0.10226	0.10343	0.10254	0.10281	0.10011	0.000789	0.77
Cu5	Cu5	7	0.11797	0.12018	0.11899	0.11898	0.11865	0.11954	0.11717	0.000589	0.50
Fe12	Fe12	24	454.15000	458.04100	457.95400	455.53700	455.88033	460.47200	455.16120	1.925101	0.42
Fe1N	Fe1N	24	454.15000	458.04100	457.95400	455.53700	455.88033	460.47200	455.16120	1.925101	0.42
Fe4	Fe4	9	450.86800	453.75300	452.20800	450.67000	451.24867	451.78500	455.30280	0.836685	0.19
Fe4N	Fe4N	9	450.86800	453.75300	452.20800	450.67000	451.24867	451.78500	455.30280	0.836685	0.19
Mn3	Mn3	11	0.11250	0.12561	0.11063	0.11545	0.11286	0.11396	0.10819	0.002428	2.15
Mo2	Mo2	10	0.10358	0.11443	0.10353	0.10793	0.10501	0.10412	0.10170	0.002526	2.41
N1	N1	20	0.02557	0.02575	0.02575	0.02658	0.02597	0.05107	0.02893	0.000541	2.08
Nb1	Nb1	14	0.10281	0.10327	0.10416	0.10510	0.10402	0.10406	0.10006	0.001155	1.11
Ni2	Ni2	22	0.18315	0.29273	0.18416	0.19967	0.18899	0.18801	0.18746	0.009260	4.90
Ni3	Ni3	23	0.17841	0.18427	0.18098	0.18300	0.18080	0.18079	0.18266	0.002298	1.27
P1	P1	1	44.12500	44.78100	45.63700	45.74500	45.16900	44.24400	45.37020	0.905742	2.01
Pb6	Pb6	6	0.21813	0.21828	0.21715	0.21709	0.21746	0.21572	0.20691	0.000587	0.27
S0	S0	2	22.39300	22.61100	22.93400	23.00200	22.77633	22.56633	22.54100	0.333713	1.47
S1	S1	2	22.39300	22.61100	22.93400	23.00200	22.77633	22.56633	22.54100	0.333713	1.47
Si1	Si1	18	0.07846	0.07888	0.07805	0.07921	0.07857	0.07857	0.07941	0.000588	0.75
Sn2	Sn2	4	0.07649	0.07721	0.07900	0.07959	0.07836	0.07756	0.07779	0.001642	2.10
Ti4	Ti4	15	0.09503	0.09450	0.09400	0.09432	0.09445	0.09469	0.09291	0.000527	0.56
V3	V3	13	0.09391	0.09391	0.09329	0.09351	0.09357	0.09439	0.09368	0.000313	0.33
W7	W7	19	0.11528	0.11684	0.11474	0.11666	0.11556	0.11522	0.11465	0.000992	0.86

Performing Analysis Fe01_SUS_RE12/156 - \$\$SUS - Fe01_SUS_RE12 - 1: Waiting for next run or end of Anal... Stand_Fe: Running Stan

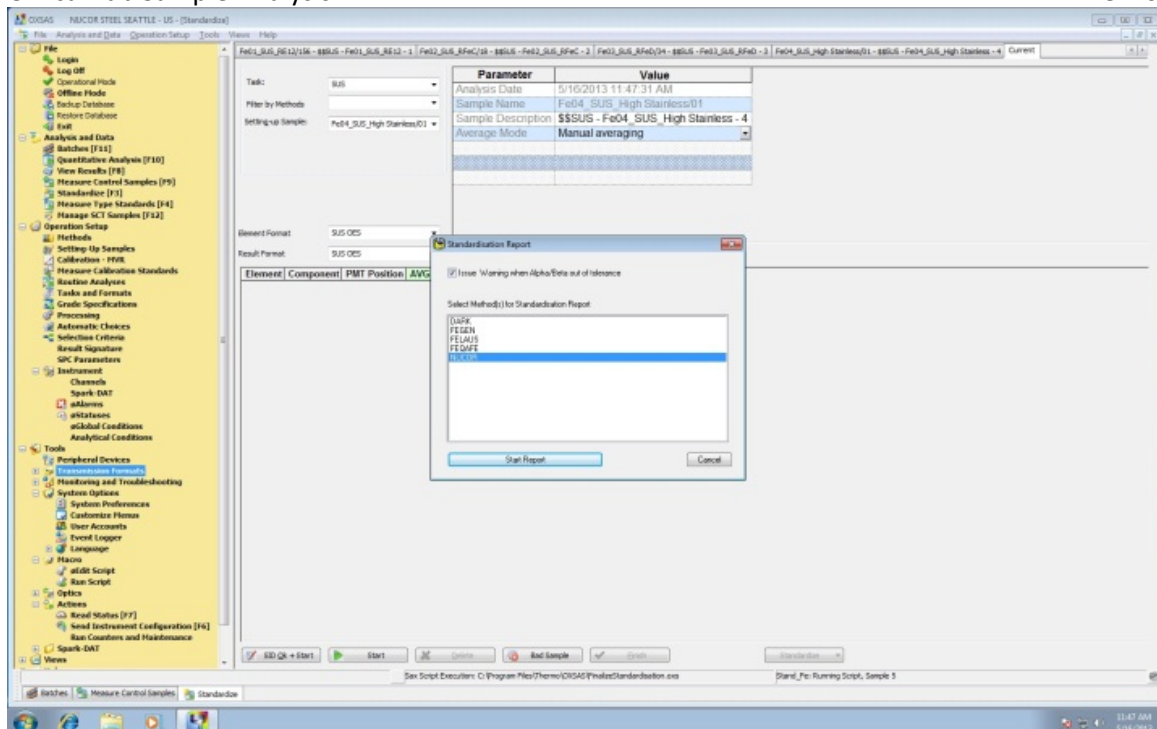
- 9) Once the 4th SUS standard had been analyzed, click “finish”, select “NUCOR” on the drop down and click “Start Report” ...and you are almost done!

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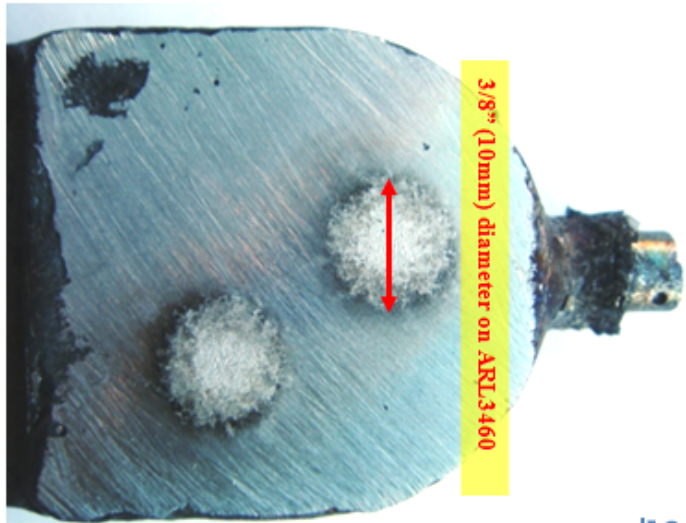
10) Analyze check standard and ensure the elements are within the specified ranges.

11) Document the verification

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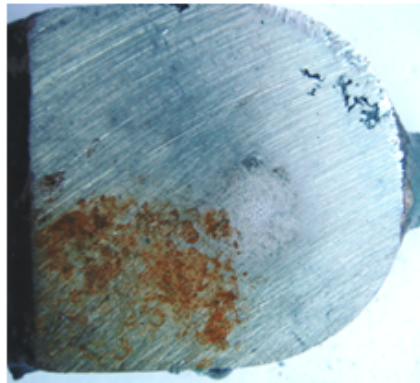
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Spectrometer Sample Burn Guideline

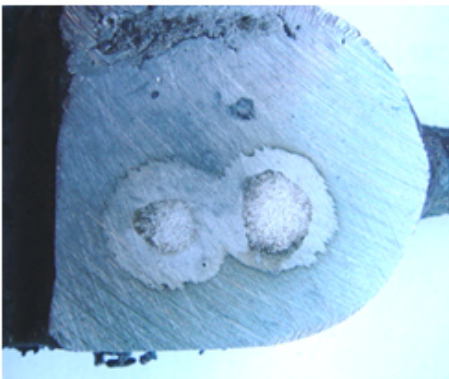


GOOD BURN

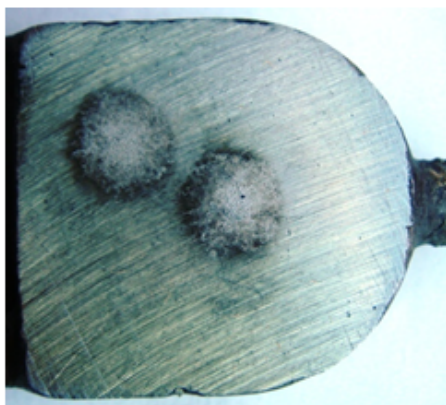
The burns have a distinct & even black ring around the edge, they're spaced far enough apart to avoid any interference from the other burn.
To achieve good burn, (1) machine must be warmed up & drift corrected for accuracy, (2) sample must be flat, dry & cool to touch, (3) contact with electrode holding down the sample is secure.



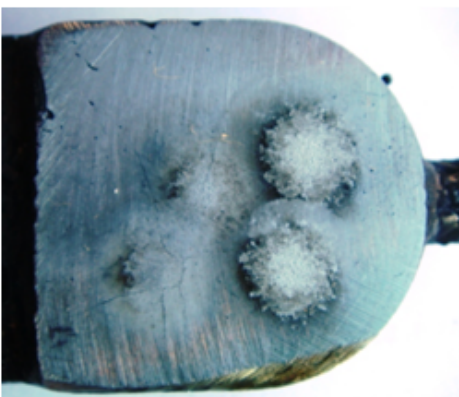
Too light This burn is too light and doesn't have a black ring – indicating the burn is not complete



Incomplete Burns are incomplete – burn size is small & no black ring and burn size is small



Too close Burns are too close together



Incomplete & overlapping Burns are incomplete (from surface crack) & overlapping. Regrind samples before making a additional burn if there is not sufficient space for good separate burns