SAFETY is 'X' when the risk assessment is >7. ENVIRONMENT is 'X' if >=8. QUALITY is 'X' when inspection/record is required.	**Issue no. 1**	**SWP no. SWPQA0809**	**Date: 18/06/2014**	**By: A Jayne**
	PPE: Lab coat, safety glasses, safety shoes			
	EQUIPMENT: Zeenit 700 Analyser			
	Q=Quality, S=Safety, E=Environment			

STANDARDISED WORK PROCEDURE (CATEGORY 2)

Effluent Analysis by AAS

This method of analysis measures soluble and total nickel, copper and cobalt levels in Refinery Effluents by Atomic Absorption Spectroscopy.

The sample is atomised by an air-acetylene flame and passes into a beam of light produced by a hollow cathode lamp radiation source. The light emmitted passes through a monochromator to separate the element specific radiation and the absorbance of the radiation is measured by a detector.

Related Documents:

Occupational Risk Assessments:
SWPQA0809 RA

Environmental Risk Assessments:
SWPQA0809 RA

Chemicals:
Acetylene

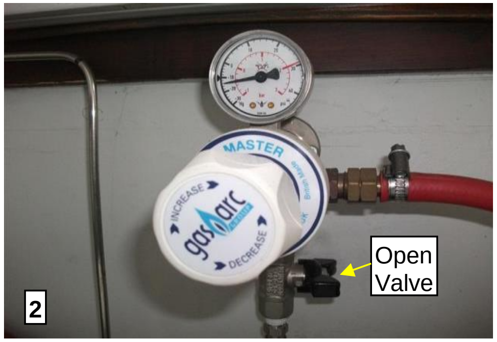
COSHH Risk Assessments:
Shortcut to COSHH Risk Assessments

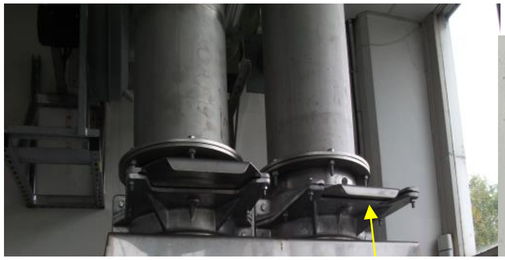
Material Safety Data Sheet:
Shortcut to Process MSDS
Shortcut to Supplies MSDS
Related Procedures:
WI0806

Standard Forms:

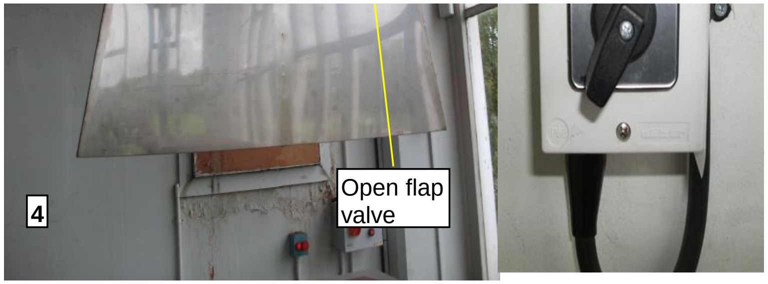
EH&S:	**Quality:**	**Production:**

No	Operating description	Q	S	E	Key points	Time
	Setting up the analyser					
1	Open the acetylene cylinder using the cyclinder key.				**Located in cylinder cage outside lab**	
2	Open the acetylene supply valve				**Located on wall next to instrument**	
3	Switch on acetylene alarm				**Alarm sounding indicates cylinder needs changing. Close cyclinder and replace.**	
4	Open the flap valve of the fume hood				**Fan switch on wall behind instrument should be in position 2**	
5	Turn on the Start button, (green button) the instrument comes on naturally and the computer is always on at the mains. Connect the computer and the instrument by opening the '**WinAAS**' icon and leaving for a few minutes to attain full function.				**This is the green button on the "window wall behind the instrument" and turns on the fans.**	
6	When the 'Start' menu appears, select '**Methods**' then 'Flame', if this option is not currently selected.					
7	Click the **'OK'** button and from the METHOD screen that appears, select the appropriate method and press the '**load**' button (in this instance, Ni is analysed first, so 'Ni_Eff' is selected).				**'Ni_Eff' - nickel in effluent, 'Cu_Eff' - copper in effluent, 'Co_Eff' - cobalt in effluent.**	
8	The 'Method Load File' appears- click the '**OK**' button.					
9	Wait until the instrument initialises					
10	Select the **'Flame'** icon at the top of the screen					
11	Click 'Ignite flame' button					
12	Once the flame has been lit, click 'Close'					
	Sample Preparation					
1	Refer to WI0806 'Determination of Total & Soluble Ni, Cu & Co, pH and Suspended Solids in Refinery Effluents'					

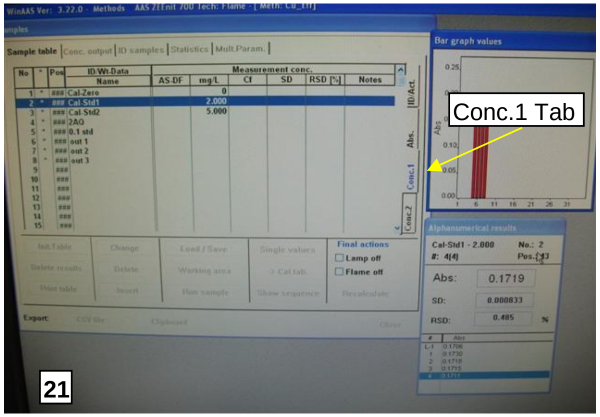




	Standard Preparation					
1	Shake the flask containing the standard before use.				Three standards are used for soluble Ni,Cu & Co: 2AQ, 5AQ and 0.1ppm. Distilled water is used as a blank. To analyse total Ni, Cu & Co, three different standards are used: 2TOT, 5TOT and 0.1ppm. TEZERO (TOT) is used as a blank.	
2	Dispense a small quantity of standard or blank into a secondary container as required.				**DO NOT dispense directly from the flask to avoid contamination. NEVER return standard back to original container - contamination may result**	
	Analysis of Soluble Nickel					
1	On the main menu along the top of the screen, click 'Samples'				**A list of each calibration standard and samples to be run will be listed**	
2	Click the 'Start/Conc' button on the bottom right hand side of the screen				**Follow the on-screen prompts**	
3	Dip the probe into the wash solution (a vessel containing distilled water), click 'OK'.					
4	Dip the probe into 'Cal-Zero' (the blank), click 'OK'.					
5	Dip the probe into the wash solution (a vessel containing distilled water), click 'OK'.					
6	Dip the probe into 'Cal-Std1' (2 ppm), click 'OK'.					
7	Dip the probe into the wash solution (a vessel containing distilled water), click 'OK'.					
8	Dip the probe into 'Cal-Std2' (5 ppm), click 'OK'.				**A calibration curve will be plotted using the absorbances obtained from Cal-Std1 and Cal-Std2.**	
9	Dip the probe into the wash solution (a vessel containing distilled water), click 'OK'.					
10	Dip the probe into '2AQ', click 'OK'.				**This step is used to check the calibration**	
11	Dip the probe into the wash solution (a vessel containing distilled water), click 'OK'.					



12	Dip the probe into '0.1ppm std', click 'OK'.				**This step is used to check the calibration**	
13	Dip the probe into 'outlet sol 1', click 'OK'.					
14	Dip the probe into the wash solution (a vessel containing distilled water), click 'OK'.					
15	Dip the probe into 'outlet sol 2', click 'OK'.					
16	Dip the probe into the wash solution (a vessel containing distilled water), click 'OK'.					
17	Dip the probe into 'outlet sol 3', click 'OK'.					
18	Dip the probe into the wash solution (a vessel containing distilled water), click 'OK'.					
19	Dip the probe into 'outlet total', click 'OK'.					
20	Dip the probe into the wash solution (a vessel containing distilled water), click 'OK'.					
21	To obtain the Ni concentrations, click on the 'Conc.1' tab and note the values displayed.					
	Analysis of Total Nickel					
1	Repeat the above steps using TEZERO (TOT) as the blank, 2TOT and 5TOT as the standards					
	Analysis of Soluble Copper					
1	Click 'Close' to close the current method					
2	On the main menu along the top of the screen, click 'Method'					
3	Select 'Cu_Eff'. Click 'Load', then 'OK'					
4	Repeat steps 1 - 21 under 'Analysis of Nickel in Effluent'					
	Analysis of Total Copper					
1	Repeat using TEZERO (TOT) as the blank, 2TOT and 5TOT as the standards				**Ensure method 'Cu_Eff' is loaded**	
	Analysis of Soluble Cobalt					



Conc.1 Tab

No	*	Pos	ID/Wt-Data Name	AS-DF	mg/L	Cf	SD	RSD [%]	Notes
1	*	###	Cal-Zero		0				
2	*	###	Cal-Std1		2.000				
3	*	###	Cal-Std2		5.000				
4	*	###	2AO						
5	*	###	0.1 std						
6	*	###	out 1						
7	*	###	out 2						
8	*	###	out 3						
9		###							
10		###							
11		###							
12		###							
13		###							
14		###							
15		###							

Cal-Std1 - 2.000 No.: 2
#: 4(4) Pos.: 13

Abs: 0.1719

SD: 0.000833

RSD: 0.485 %

1	Repeat steps 1 - 4 under 'Analysis of Copper in Effluent'				**Select method 'Co_Eff'**	
	Analysis of Total Cobalt					
1	Repeat using TEZERO (TOT) as the blank, 2TOT and 5TOT as the standards				**Ensure method 'Co_Eff' is loaded**	
	Closing Down the Instrument					
1	Leave the probe in the wash solution for 10 minutes to ensure the instrument is flushed free of any residual contaminants.					
2	Remove the probe from the wash solution.					
3	Click 'Flame' on the right hand side of the screen to extinguish the flame.					
4	Click 'X' to close the software.				**Message box "Save parameter set 0" appears - select No. Next message box, select 'Close'**	
5	Shut the flap on the fume hood.					
6	Press the red button on the control box					
7	Clean and tidy up the area.					
8	Close the acetylene supply valve.					
9	Switch off the acetylene alarm.					
10	Close the acetylene cylinder outside the laboratory building.					
