

SAFETY is 'X' when the risk assessment is >7. ENVIRONMENT is 'X' if >=8. QUALITY is 'X' when inspection/record is required.	Issue no. 2	SWP no. SWPQA0806	Date: 18/06/2014	By: SB
	PPE: Safety glasses, lab coat, safety shoes and gloves.			
	EQUIPMENT: Sample tubes, beakers, flasks, measuring cylinders, centrifuge and ICP.			
	Q=Quality, S=Safety, E=Environment			

STANDARDISED WORK PROCEDURE (CATEGORY 3)

Analysis of Refinery Effluents. Determination of Total & Soluble Nickel, Cobalt & Copper , and pH.

The total and soluble nickel, copper and cobalt concentrations, the pH and the suspended solids are an important measure of the quality of the effluent. The analyses are specified by the Competent Authority in the consent limits within which Vale has to operate. This Work Instruction provides a standard method for the routine analysis of Ni, Co, Cu, pH & Suspended Solids in Refinery Effluents.

The soluble Ni, Cu and Co are determined directly on a portion of the effluent sample after centrifuging by Inductively Coupled Plasma (ICP). Another portion of the effluent is treated with 10% hydrochloric acid and also analysed by ICP after centrifuging. This determines the total Ni, Cu and Co content of the sample.

The pH is determined by the procedure set out in Document SWPQA0031. The suspended solids present, if not visually <10 mg/l, are measured by filtering a known volume of the sample and weighing the captured solids after drying in an oven. See SWPQA0808.

The Outlet samples from the Effluent Plant are analysed using the procedures detailed in this document. These consist of Sol 1 (AM) & Sol 2 (PM) samples covering a 24 hour period. These two effluent plant samples are analysed for soluble nickel and the average of these values is reported as the soluble nickel for the daily composite sample.

On a Monday there will be an additional four samples from the weekend. These are Sol 3, Sol 4, Sol 5 and Sol 6. Similarly, the average soluble nickel for each day is reported as the soluble nickel for that day.

Related Documents:

Occupational Risk Assessments:

[SWPQ0806 RA](#)
[SWPQA0030 RA](#),
[SWPQA0010 RA](#)

Environmental Risk Assessments:

Chemicals:

[Hydrochloric acid](#).

COSHH Risk Assessments:

[Shortcut to COSHH Risk Assessments](#)

Material Safety Data Sheet:

[Shortcut to Supplies MSDS](#)

Related Procedures:

[SWPQA0031](#)
[SWPQA0100](#)

Standard Forms:

[QA SF01](#)

Supervisor:		
J.R.Jones		

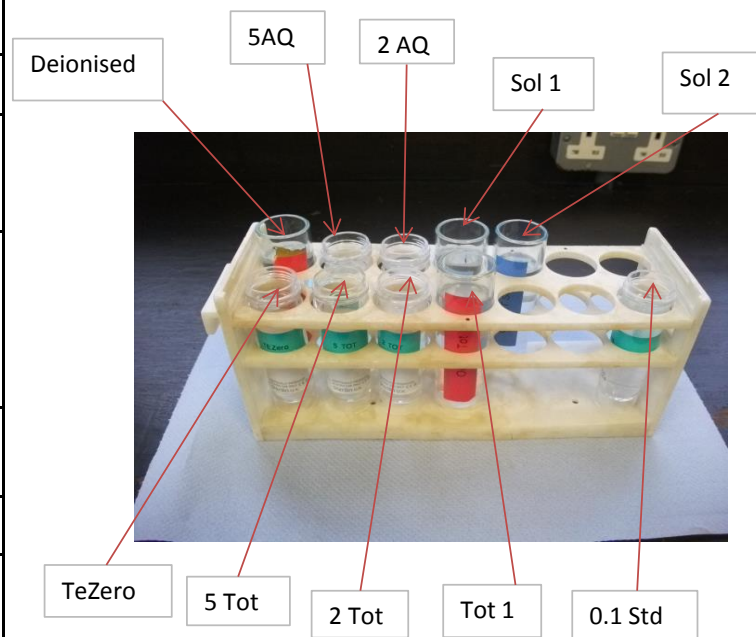
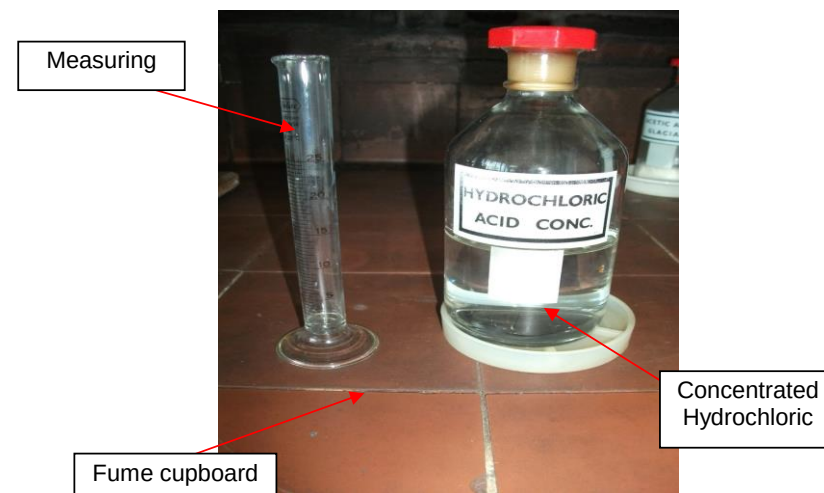
No	Operating description	Q	S	E	Key points	Time
ANALYSIS TUESDAY TO FRIDAY						
1	On Tuesday to Friday 1set of Effluent Outlet samples comes up each day: AM & PM samples.				The two samples cover a 24 hour period.	
2	Invert the AM sample to mix and pour into the OUTLET SOL 1 glass sample tube until 3/4 full. Measure 100ml of sample in a measuring cylinder and pour into a plastic conical flask labelled OUTLET 1 . Measure a further 75mls of sample into a measuring cylinder and place in a glass 1litre bottle labelled WEEKLY OUTLET COMPOSITE .				The collection of the Weekly Composite starts on a Tuesday and is completed on the following Monday. This is used for the Weekly analysis of Permanganate Value, Ammonia and Suspended Solids.	
3	Invert the PM sample to mix and pour into the OUTLET SOL 2 glass sample tube until 3/4 full. Measure 100ml of sample in a measuring cylinder and pour into a plastic conical flask labelled OUTLET 1. Measure a further 75mls of sample into a measuring cylinder and place in a glass 1litre bottle labelled WEEKLY OUTLET COMPOSITE.				The OUTLET 1 flask now contains 200mls of a composite daily sample that is used for pH and Total Ni, Co and Cu analysis. The composite is used to do a visual estimate for suspended solids. If the sample is clear, this is less than 10mg/l. If particles are present a further composite needs to be made and an appropriate volume filtered through a filter. See SWPQA0808 taking care to read the associated SWPQA0808 RA.	
4	Measure 100mls of sample from the OUTLET 1 flask and pour into the plastic OUTLET TOT1 beaker. Take this to the fume cupboard and add 10mls of concentrated Hydrochloric acid by measuring cylinder.		X		Care! Hydrochloric acid is corrosive and gives off strong acid fumes. It must be poured in a fume cupboard using the PPE listed above.	
5	Swirl gently to mix and pour into a glass tube labelled OUTLET TOT 1 until 3/4 full.					



Sol 1

Sol 2
Sample

6	Place the four tubes in a centrifuge and spin until the digital display indicates that it is safe to open. Replace tubes in the correct positions on tray.			It is important that all the sample tubes are centrifuged so that any small particles settle at the bottom and do not block tubes in the ICP. Tubes must be correctly placed in the centrifuge, ie, a tube in each holder, or two in opposite holders. Tubes should be filled to the same level. Turn on, close the lid and press the Start Stop button. The centrifuge spins for 2.5 minutes at 300RPMx10. When ready, and safe to open, the message 'OPEN' is displayed. Press the Lid Open button to release samples.	
7	Analyse for pH by pouring approximately 100mls of sample from the OUTLET 1 flask into the OUTLET pH 1 beaker. Carry out analysis as specified in SWPQA0030, taking note of the associated SWPQA0030 RA.				
8	Record the result in the work book and enter into LIMS.				
9	Set up the ICP as described in SWPQA0010 taking care to read the associated risk assessment SWPAQ0010 RA.	x			
10	Select the programme EFFDAILY.CMD . Place the Standards and deionised water in the correct positions in the sample tray as indicated in the programme.			Tray Layout: Deionised Water 2/8, 5AQ 2/9, 2AQ 2/10, 0.1 Std 2/7, TeZero 2/1, 5 TOT 2/2, 2 TOT 2/3, Sol 1 2/11, Sol 2 2/12, and Tot 1 2/4.	
11	Run the programme taking care to check that the AQC's are within limits.				
12	Record the results in the work book and enter into LIMS.				
13	Empty and rinse the sample bottles. Place in the lift so that they can be returned to the Effluent Plant.			If any bottles are dirty, discard and request more from the effluent plant.	



ANALYSIS ON MONDAYS					
1	On Mondays 3 sets of AM & PM samples are received from the previous Friday, Saturday and Sunday. This results in 6 sample bottles.				
2	The samples from Friday are poured into sample tubes Outlet SOL1 and Outlet SOL2 . 100mls of each sample is measured in a cylinder and poured into the OUTLET 1 flask which is used for pH and Total Ni, Cu and Co analysis in beakers OUTLET pH 1 and OUTLET TOT 1 respectively. The Total sample is acidified with 10mls of HCl. The tubes are then placed in the correct positions in the sample tray as specified in the programme EFFW.CMD. 75mls of each sample is also taken for the WEEKLY OUTLET COMPOSITE.	x		Care! Hydrochloric acid is corrosive and gives off strong acid fumes. It must be poured in a fume cupboard using the PPE listed above.	
3	The samples from Saturday are poured into sample tubes Outlet SOL3 and Outlet SOL4. 100mls of each sample is measured in a cylinder and poured into the OUTLET 2 flask which is used for pH and Total Ni, Cu and Co analysis in beakers OUTLET pH 2 and OUTLET TOT 2 respectively. The Total sample is acidified with 10mls of HCl. The tubes are then placed in the correct positions in the sample tray as specified in programme EFFW.CMD. 75mls of each sample is also taken for the WEEKLY OUTLET COMPOSITE.	x		Care! Hydrochloric acid is corrosive and gives off strong acid fumes. It must be poured in a fume cupboard using the PPE listed above.	

Six Effluent Samples

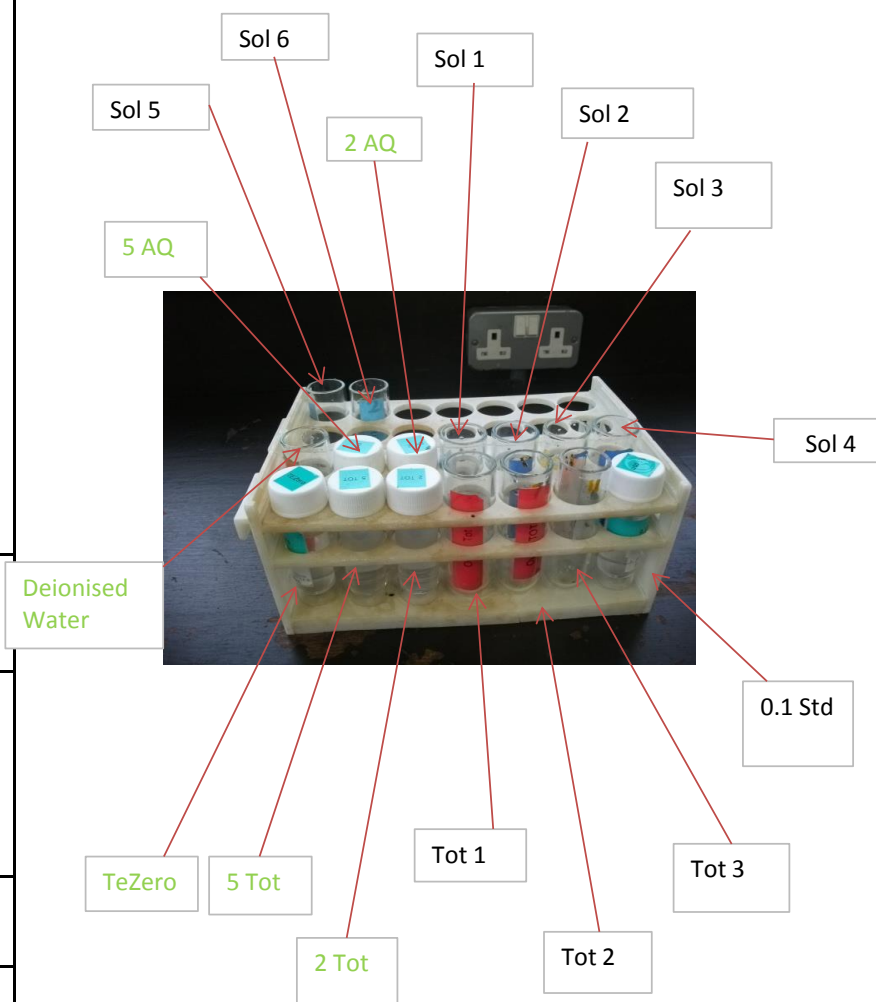


Plastic beakers and flasks



4	The samples from Sunday are poured into sample tubes Outlet SOL5 and Outlet SOL6. 100mls of each sample is measured in a cylinder and poured into the OUTLET 3 flask which is used for pH and Total Ni, Cu and Co analysis in beakers OUTLET pH 3 and OUTLET TOT 3 respectively. The Total sample is acidified with 10mls of HCl. The tubes are then placed in the correct positions in the sample tray as specified in programme EFFW.CMD. 75mls of each sample is also taken for the WEEKLY OUTLET COMPOSITE.	x	Care! Hydrochloric acid is corrosive and gives off strong fumes. It must be poured in a fume cupboard using the PPE listed above. It is important that all the sample tubes are centrifuged so that any small particles settle at the bottom and do not block tubes in the ICP. Tubes must be correctly placed in the centrifuge, ie, a tube in each holder, or two in opposite holders. Tubes should be filled to the same level. Turn on, close the lid and press the Start Stop button. The centrifuge spins for 2.5 minutes at 300RPMx10. When ready, and safe to open, the message 'OPEN' is displayed. Press the Lid Open button to release samples.	
5	Set up the ICP as described in SWPQA0010 taking care to read the associated risk assessment SWPQA0010 RA.	x		
6	Select the programme EFFW.CMD. Place the Standards and deionised water in the correct positions. See point 10 above.		Tray Layout: Deionised Water 2/8, 5AQ 2/9, 2AQ 2/10, 0.1 Std 2/7, TeZero 2/1, 5 TOT 2/2, 2 TOT 2/3, Sol 1 2/11, Sol 2 2/12, and Tot 1 2/4. Sol 3 2/13, Sol4 2/14, Sol 5 3/8, Sol 6 3/9, Tot 2 2/5, and Tot 3 2/6.	
7	Run the programme taking care to check that the AQC's are within limits.		Follow the Flowchart to troubleshoot any problems with the AQC's.	
8	Record the results in the work book and enter into LIMS.			
9	Empty and rinse the sample bottles. Place in the lift so that they can be returned to the Effluent Plant.		If any bottles are dirty, discard and request more from the effluent plant.	

ANALYSIS ON BANK Holiday
MONDAYS



Insert the AM and PM samples from the Bank Holiday Monday i.e. **SOL 7 and SOL 8** into positions **3/10 and 3/11**. Insert the Total sample from the Bank Holiday Monday i.e. **TOT 4** into position **3/01**.

These are inserted into the Weekend Effluent Tray Layout **EFFW.CMD** program. **IMPORTANT: DO NOT SAVE ANY CHANGES SO THAT THE PROGRAM REVERTS TO THE NORMAL WEEKEND PROGRAM.** This only needs to be done 5 times a year.