TATA STEEL

TATA

Regulated Industry Permitting Team
Natural Resources Wales

13th May 2021

Our reference: EPR BL7108IM
Your reference: Application reference PAN-013746

Subject: Justification for Deviation from Testing Standards for Port Talbot Effluent Discharge

Tata steel recently informed NRW that there were some differences between the testing standards listed in the permit and those that were actually being conducted in the laboratory. Listed in Table S3.8 of EPR BL7108IM there is a column headed Monitoring Standard or method and that is the focus of this memo. Tata Steel committed to investigating all methodologies used to analysis effluent samples and reporting those which deviated from the prescribed method and those which matched.

When Tata Steel raised this issue with local NRW officers it was understood that this was an issue on a significant number of older permits. This is because testing methodologies improve over time, either for detection limits, consistency or time involved in getting an accurate result. The critical requirement of any test method is that it must be suitable for the sample matrix and the limit of quantification must be well below the consent limit for every analyte. We confirm that this is the case across all tests and analytes (where limits are defined). For this reason Tata Steel for first like to request that the column in table S3.8 be removed completely as there is a danger that it can quickly become out of date as methods improve. Meaning that Tata Steel could go out of compliance even though it was using a technically equivalent methodology that maybe be better than that specified in the permit. This would be preferable to replacing the standards listed in the permit with new ones. Secondly, many of the standards

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used in laboratories, both Tata Steel operated and external, tend to use in-house methodologies that are based on a standard rather than mirroring the standard in its entirety. For that reason, the “monitoring standard or method” would always be inaccurate regardless of the standard, unless we used in-house methodologies, which would be difficult to keep up-to-date.

However, if removal from the permit is not possible we have created a table below that shows the current parameters in the permit, the specified standard and how that analysis is currently being undertaken. Where required there is an explanation of the equivalence of the standard compared to that listed in the permit.

Sample location	Analyte	Standard Specified in Permit	Actual Method used
W1 (LSO)	BOD	BS EN 1899-1	Not tested.
W1 (LSO)	COD	BS 6068-2.34	In-house GRDS-9VCDUV based on BS ISO 15705:2002
W1 (LSO)	Cyanide	BS 6068-2.18	In-house RLES-6N6LKR based on BS 6068-2.18:1986 (steam distillation / colorimetry)
W1 (LSO)	Fe	BS EN ISO 15586:2003 (Atomic Absorption)	In-house RLOE-B63C9Z based on ISO 11885:1998 (ICP-OES, a multi-determinand method)
W1 (LSO)	Ni	BS 6068-2.29 (Atomic Absorption)	In-house RLOE-B63C9Z based on ISO 11885:1998 (ICP-OES, a multi-determinand method)
W5 (Cooling)	oil & grease	BS EN ISO 15680 (GC)	In house PMAE-9YDAUG based on ASTM D7066 US solvent extraction / infrared absoption (Horiba OCMA-500)
W1 (LSO)	Pb	BS 6068-2.29 (Atomic Absorption)	In-house RLOE-B63C9Z based on ISO 11885:1998 (ICP-OES, a multi-determinand method)
W1 (LSO)	pH	BS ISO 10523 (electrometric)	In house RLES-6NCEYU based on BS ISO 10523 (electrometric)
W4 (Afon)	pH	BS ISO 10523 (electrometric)	In house RLES-6NCEYU based on BS ISO 10523 (electrometric)
W1 (LSO)	Phenols	BS 6068-2 (colorimetry)	Monohydric: in-house MALN-8WYBUP based on: EN ISO 5667-3 (distillation) then ISO 6068-2-2.12: (colorimetry). Total Phenol: In-house RLES-6NBKVP based on: BS 6068-2-2.12:1984 (colorimetry).
W1 (LSO)	PAH	BS EN ISO 17993	Externally analysed by in-house method GEO81 (DLLME/GCMS)
W4 (Afon)	Soluble Iron	BS EN ISO 15586:2003 (Atomic Absorption)	In-house RLOE-B63C9Z based on ISO 11885:1998 (ICP-OES, a multi-determinand method)

W1 (LSO)	Sulphides	Ref: 228 (Blue Book - methylene blue)	In-house based on Ref 228 (test kits)
W1 (LSO)	TON	BS EN ISO 11732 (segmented flow analysis / colorimetry)	In-house ARJS-A3BANN based on Blue Book ISBN 0117515930
W1 (LSO)	Suspended Solids	BS EN 872 (Gravimetric)	In-house GRDS-0SSEZ7 based on BS EN872:2005
W3 (ABO)	Suspended Solids	BS EN 872 (Gravimetric)	In-house GRDS-0SSEZ7 based on BS EN872:2005
W4 (Afon)	Suspended Solids	BS EN 872 (Gravimetric)	In-house GRDS-0SSEZ7 based on BS EN872:2005
W1 (LSO)	Thiocyanate	BS EN ISO 10304-3 (HPLC/UV)	In-house RLES-6N7EFQ based on Blue Book method ISBN 0117519340 (colorimetry)
W1 (LSO)	Total Chromium	BS EN 1233 (Atomic Absorption)	In-house RLOE-B63C9Z based on ISO 11885:1998 (ICP-OES, a multi-determinand method)
W1 (LSO)	Total Hydrocarbons	BS EN ISO 15680 (GC)	In house PMAE-9YDAUG based on ASTM D7066 US solvent extraction / infrared absorption (Horiba OCMA-500)
W3 (ABO)	Total Hydrocarbons	SCA Blue Book 77	In house PMAE-9YDAUG based on ASTM D7066 US solvent extraction / infrared absorption (Horiba OCMA-500)
W4 (Afon)	Total Hydrocarbons	visible check	In house PMAE-9YDAUG based on ASTM D7066 US solvent extraction / infrared absorption (Horiba OCMA-500)
W4 (Afon)	Total Iron	BS EN ISO 15586:2003 (Atomic Absorption)	In-house RLOE-B63C9Z based on ISO 11885:1998 (ICP-OES, a multi-determinand method)
W1 (LSO)	Zn	BS 6068-2.29 (Atomic Absorption)	In-house RLOE-B63C9Z based on ISO 11885:1998 (ICP-OES, a multi-determinand method)

Analytes with the Correct Method

The following analytes have an in-house method that is based directly on the standard mentioned in the permit:

Cyanide, pH, Phenol, Sulphides, Suspended Solids

All other analytes deviate from the stated method.

Atomic Adsorption Spectroscopy

The standard BS EN ISO 15596 and BS 6068-2.29 relate to the use of Atomic Adsorption Spectroscopy (AAS) to analyse for metallic elements in the trade effluent. The following analytes should be tested using this method:
Iron, Nickel, Lead, Chromium and Zinc

This has been replaced with an in-house method based on ISO 11885:1998, which utilises Inductively Coupled Plasma – Optical Emission Spectroscopy (ICP-OES). Both techniques are suitable for detection of metallic elements, however ICP-OES provides lower limits of detection and allows multiple elements to be analysed at once meaning that the results are received many times faster than if AAS was used instead. The technique is considered to be a superior method for achieving rapid and accurate results.

Sealed Tube Method for Chemical Oxygen Demand

The standard listed in the permit for COD is BS 6068-2 (we have assumed this should actually read 6068-2.34), which details a number of different ways to determine COD including physical, chemical and biochemical. This has been replaced by a test kit which is a sealed tube pre-filled with reagents to give an accurate determination of the COD. These test kits are based on BS ISO 15705:2002 and have easily replicable results. The standard it replaces includes multiple techniques and a more standardised method.

Horiba method for Oil & Grease / Total hydrocarbons

Currently, the permit specifies the use of Gas Chromatography as per BS EN ISO 15680 in W1 and solvent extraction followed by infrared absorption as per SCA blue book 77 at W3. Tata Steel currently use a Horiba automated sampler for the analysis of oil in water. This sampler uses a solvent extraction followed by infrared absorption analysis. This technique is very similar to the SCA blue book 77 method and is an automated version of that standard and can detect any substance that is extracted by the solvent across a narrow infrared absorption band that best represents oils. This automated technique provides rapid easily repeatable results that have less potentially errors in sample preparation and result interpretation.

Gas Chromatography is a more accurate way of assessing the hydrocarbons in a sample, it gives more detail on the compounds present by the analysis of the volatilisation of the whole spectrum of hydrocarbons. However it is a slower technique and given that Tata Steel are performing multiple daily samples on the discharge, GC-MS would potentially cause delays in the delivery of the results. The permit only states that there is a require to report a total hydrocarbon value and therefore speciation of the hydrocarbon is unnecessary and would not be reported to NRW.

High Pressure Liquid Chromatography (HPLC) for PAH analysis

The techniques used for PAH analysis are very similar to that listed in the permit. BS 17993 refers to the use of HPLC after a Liquid-Liquid Extraction (LLE) for determination of 15 PAH compounds. Tata Steel currently use an external laboratory for testing of these samples that use a technique called Dispersive Liquid-Liquid Microextraction (DLLME) to extract the PAH compounds dissolved in a effluent sample. This is an automated extraction that disperses fine droplets of the extraction solvent throughout the sample to very rapidly partition the PAH compounds into the solvent phase and then extract the solvent for analysis. LLE is a similar

but manual technique that causes the partitioning of the PAHs into the solvent but in a slower, more labour intensive method.

Analysis is then conducted use Gas Chromatography – Mass Spectrometer which analysis the PAH in gas phase rather than in a liquid phase as in HPLC but the results are very similar and would be considered entirely equivalent. There is a potential for Tata Steel to bring this analysis in-house in the future, and if this was the case it would be LLE followed by GCMS.

High Pressure Liquid Chromatography (HPLC) for Thiocyanate

The permit states that Thiocyanate should be determined by HPLC as stated in BS EN ISO 10304-3. Tata Steel currently use an analytical technique based on colorimetry based on SCA Blue Book 100 that reacts the Thiocyanate with Iron nitrate to give a brown colouration and the strength of this colouration can be used to determine the concentration of Thiocyanate in the sample. This methodology gives an accurate determination of the thiocyanate in a sample and has a low limit of detection. It is a quicker test to perform and benefits the rapid turnaround of these samples as required. We therefore believe that this is an equivalent standard for determination of Thiocyanate.

BOD Testing

Tata Steel has been attempting to have BOD testing removed from permit requirements for a number of years due to the difficulty of performing the analysis as it requires several days of incubation to gain a valid result. In 2017 NRW responding saying they were developing an internal position on BOD testing and would not take enforcement action against Tata Steel for not analysing BOD. However, this has yet to be resolved.

Tata Steel have put in a permit variation during March 2021, once again asking for the removal of BOD testing from the testing schedule. The decision is with the permitting team and is unlikely to be resolved in the short term. Tata Steel is currently unable to complete BOD testing using its in-house laboratories and would need to approach external labs to have this done. We currently undertake COD analysis, which is analogous to BOD testing and given the difficulties of testing for BOD Tata Steel believe that the current testing regime is sufficient to ensure no impact from our effluent discharges.

Once this has been resolved we will have a better understanding of what standard is best to list in the permit.

Robert Naylor