The determination of Titanium coating thickness on Steel with X-Supreme8000

Application Note
AN36XRF.v1

Galvanised steel is widely used in the construction of automotive bodies and the galvanising process applies a protective coating to the steel thereby ensuring long term corrosion resistance. Galvanising is the application of Zinc to the steel, however after the zinc is applied, an additional passivation layer i.e. a hard non-reactive surface film that further inhibits corrosion particularly when the sample is stored in air, is applied. This has been a chromate layer but recently due to environmental concerns, the Chromate layer has been replaced with alternative materials having similar properties. For one process for automotive bodies a Titanium passivation layer was applied to galvanised steel strips.

In the production process this Titanium passivation layer thickness needs to be monitored to ensure correct thickness for conformance to specification, and cost-effective production.

X-ray Fluorescence analysis (XRF) is a well respected analytical technique for the analysis of Titanium coatings and the **X-Supreme**8000 offers simple, rapid, accurate, elemental analysis with the instrument operated by either laboratory or production staff, operating on a 24/7 basis leading to high quality analysis with potential cost savings.

Sample Preparation and Presentation

To demonstrate the performance of the **X-Supreme**8000 for the analysis of Titanium coating on steel, six samples of known Titanium content (expressed in mg/m2) were used to derive a calibration. Using this calibration an "unknown" sample was measured to demonstrate instrument repeatability, together with a calculation for limit of detection, and this data is shown in table 1 on page 3.

The standards and samples were metal discs, which were cut into squares of approx 2.5 cm2 and placed into standard Oxford Instruments sample cells (part number Q59) which consists of a metal circular holder into which the sample is placed, and a spring loaded top holds the sample firmly in place. The complete assembly is then placed onto one of the 10 sample positions on the in-built sample carousel. Note: Up to 10 samples can be loaded at a time allowing unattended operation leading to maximised efficiency.

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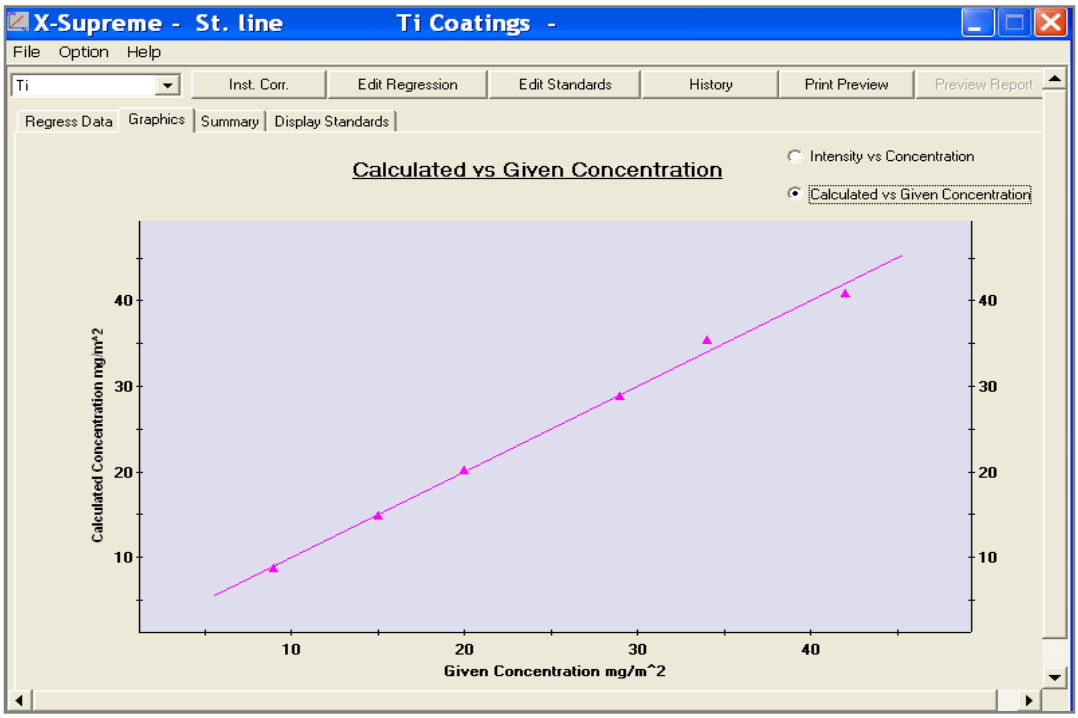
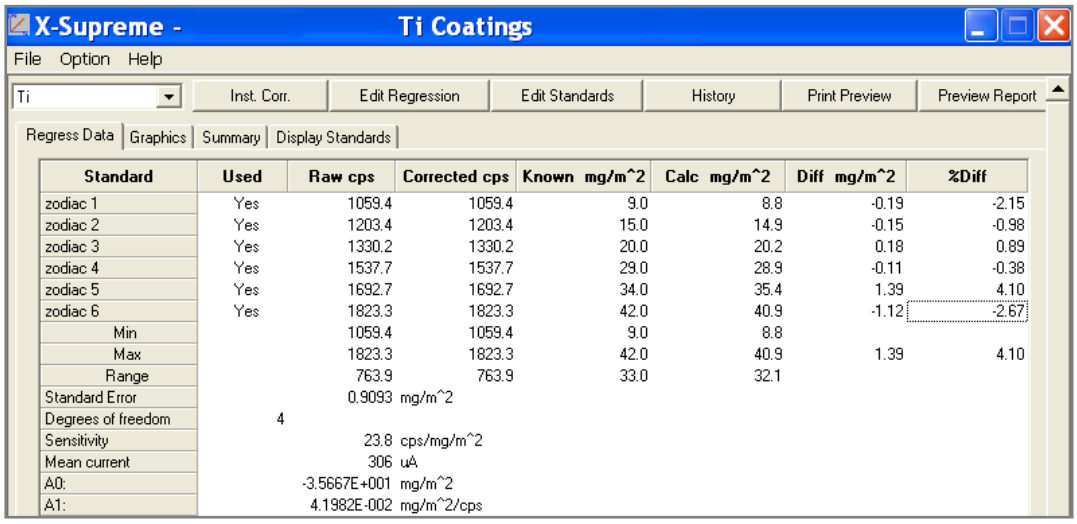
Performance and Results

For the determination of Titanium, the **X-Supreme** uses the *combined* power of the Tungsten target X-ray tube, and high resolution of the Silicon Drift Detector. The sample spinner was used to minimise any effect of sample homogeneity. Note: If additional elements such as Phosphorous also need to be determined then the helium purge option will be required to ensure high elemental sensitivity for phosphorous. Titanium determination was measured in "air path" mode.

A series of standards covering the Titanium concentration range 9 to 42 mg/m2 were measured and the resulting regression is shown below.





Standard	Used	Raw cps	Corrected cps	Known mg/m^2	Calc mg/m^2	Diff mg/m^2	%Diff
zodiac 1	Yes	1059.4	1059.4	9.0	8.8	-0.19	-2.15
zodiac 2	Yes	1203.4	1203.4	15.0	14.9	-0.15	-0.98
zodiac 3	Yes	1330.2	1330.2	20.0	20.2	0.18	0.89
zodiac 4	Yes	1537.7	1537.7	29.0	28.9	-0.11	-0.38
zodiac 5	Yes	1692.7	1692.7	34.0	35.4	1.39	4.10
zodiac 6	Yes	1823.3	1823.3	42.0	40.9	-1.12	-2.67
Min		1059.4	1059.4	9.0	8.8		
Max		1823.3	1823.3	42.0	40.9	1.39	4.10
Range		763.9	763.9	33.0	32.1		
Standard Error		0.9093	mg/m^2				
Degrees of freedom	4						
Sensitivity		23.8	cps/mg/m^2				
Mean current		306	uA				
A0:		-3.5667E+001	mg/m^2				
A1:		4.1982E-002	mg/m^2/cps				

Using the calibration curve on page 2 a Titanium coated sample was measured five times, i.e. leaving the sample in the instrument and taking five repeat readings, to demonstrate instrument repeatability (precision): Results are shown below.

	Ti (mg/m²)
#1	15.2
#2	15.3
#3	15.2
#4	15.0
#5	15.1
STD Dev	0.32

From all of the above data, Table 1 below shows the performance and results for the analysis of Titanium

Analyte	Concentration Range (mg/m²)	Standard error (mg/m²)	Theoretical limit of detection (mg/m²)	Guaranteed limit of detection (mg/m²)	Precision at mid range 95% confidence (mg/m²)	Measurement time (seconds)
Ti	9 - 42	0.91	0.5	0.75	0.50	60

Table 1

X-Supreme8000 instrument configuration

a) **X-Supreme** fitted with W tube and high performance SDD
b) Sample spinner (to reduce effects of sample inhomogeneity
c) One to ten sample cells P/No Q59 for loading of samples onto the sample carousel for unattended multi-sample operation
d) Instrument parameters: 8kV, 306uA, 60s, No primary beam filter, air path, spinner
e) Helium purge option (for P determination)

Conclusion

The above data shows the **X-Supreme**8000 offers simple, rapid, routine quality control analysis of Ti on galvanised steel. If required the **X-Supreme** can be operated by production staff offering 24/7 operation giving consistent product quality and optimisation of cost.

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