



PMI Just In Case

Positive Material Identification
Ferritscope Testing

QUALITY DOCUMENT

CALIBRATION OF NITON XRF ALLOY ANALYSER

DOCUMENT No

QD-CAL-PMI-01

REVISION

01

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1.0 Purpose

To define the scope and method of carrying out calibration of a Niton XRF Alloy Analyser.

2.0 Scope

This procedure is a standard procedure that describes the methods for carrying out calibration using the in-built self-calibrating function of the Niton XRF Alloy Analyser and also an annual comparison check against a Certified Reference Material (CRM) of known chemical analysis.

3.0 References

Niton XL3T 800 Alloy Analyser Operating Manual.
API RP578 – Material Verification Program for New and Existing Alloy Piping Systems
Positive Material Identification Procedure QD-PMI-01

4.0 Definitions

PMI (Positive Material Identification)

Physical testing of materials to determine the chemical composition and positively verify material composition by determining the alloy content of a component or a weld without the need to remove samples for analysis.

XRF (X-ray Fluorescence Spectroscopy)

A method of PMI using a portable X-ray fluorescence analyser to verify the chemical elements that establish the positive identification for a particular material.

When activated on a test item, an XRF analyser produces short wavelength electromagnetic rays. These emitted rays interact with (excite) certain elements. As a result, the excited elements then re-emit a new wavelength within a discrete energy band that is identifiable by the analyser.

When used along with material standards, the portable analyser can determine approximate chemical quantities for the target elements.

CRM (Certified Reference Material)

A reference material with one or more properties whose values are certified by a technically valid procedure accompanied by or traceable to a certificate or other documentation, which is issued by a Certifying Body.

5.0 Responsibilities

Personnel applying this procedure shall be familiar with this procedure and be competent in the use of a Niton XL3T 800 Alloy Analyser

6.0 Safety

- All equipment shall be inspected for obvious defects prior to calibration.

7.0 Equipment

PMI test equipment and its calibration shall meet the following criteria:

- Be suitable for the range of material compositions to be verified
- Be suitable for the range of surface curvatures and orientations to be encountered such as welds and small bore fittings.
- Be sufficiently portable to gain access to each checkpoint under evaluation.
- Be capable of providing 'direct read-out' quantitative results (% composition) of the required chemical elements.

8.0 Calibration

8.1 For standard inspection the Niton XL3T 800 Alloy Analyser detector shall be used as a comparator check analysis during set-up.

Activation of the analyser self calibration mechanism must only be done by authorised PMI Just In Case personnel.

- i. Once the machine is switched on select '**Utilities**' on the screen menu
- ii. Select '**Calibrate**'
- iii. Select '**Calibrate Detector**' – the analyser will automatically begin the calibration cycle which takes approximately 1 minute.
- iv. **RES** will appear once calibration is complete – ensure **RES** is under 220eV.
- v. If **RES** is greater than 220eV quarantine analyser and consult with Niton UK.

These set-up checks shall be carried out as a minimum:

- Each time the analyser is switched on

and re-confirmed at least at the following times:

- At intervals not exceeding each 50 determinations carried out
- Each time a different Standard or material range is required
- If there is a significant change in equipment or material temperature
- Whenever the equipment has been accidentally knocked or adjusted
- Whenever any error is suspected
- Immediately at the end of a work session

8.2 A comparison check against a Certified Reference Material (CRM) must be done annually as a minimum. The CRM grade 1¼ Chromium and ½ Molybdenum is certified by Analytical Reference Materials International and bears the Part Number (Q.A.No): IARM 35HN.

- i. Obtain 5 readings (minimum of 20 seconds per reading) of the CRM
- ii. Record all readings on Calibration Certificate – see appendix II
- iii. Calculate mean readings for Molybdenum (Mo), Iron (Fe), Manganese (Mn) and Chromium (Cr) and compare against CRM Certificate No 35HN-01112007-IARM-P.
- iv. In the event of sporadic, inconsistent readings quarantine the analyser and consult with Niton UK.

9.0 Reporting

On completion of self calibration, in accordance with section 8.1 above, the operator must date and sign the Certificate of Calibration – refer to Appendix I.

Reporting of the comparison check against the CRM must be done annually as a minimum – refer to Appendix II.

Certified Reference Material (CRM) – refer to Appendix III.

All records to be kept for a minimum of 3 years.

APPENDIX II

NITON XL3T 800 ALLOY ANALYSER - CALIBRATION CERTIFICATE

SERIAL No: 47632

Certified Reference Material (CRM): Grade 1¼Cr ½Mo / UNS K11572 Part No (Q.A.No): IARM 35HN

Reading	Time	Alloy1	Sb	Sb Error	Sn	Sn Error	Pd	Pd Error	Ag	Ag Error	Al	Al Error	Mo	Mo Error	Nb	Nb Error
1	08/06/2010 19:44	1 1/4 Cr : 0.37	< LOD	0.024	< LOD	0.035	< LOD	0.025	< LOD	0.194	< LOD	80	0.507	0.015	< LOD	0.004
2	08/06/2010 19:51	1 1/4 Cr : 0.00	< LOD	0.031	< LOD	0.044	< LOD	0.033	< LOD	0.228	< LOD	80	0.483	0.019	< LOD	0.006
3	08/06/2010 19:51	1 1/4 Cr : 0.00	< LOD	0.028	< LOD	0.042	< LOD	0.031	< LOD	0.178	< LOD	80	0.482	0.018	< LOD	0.005
4	08/06/2010 19:52	1 1/4 Cr : 0.13	< LOD	0.024	< LOD	0.035	< LOD	0.025	< LOD	0.178	< LOD	80	0.506	0.015	< LOD	0.004
5	08/06/2010 19:52	1 1/4 Cr : 0.00	< LOD	0.024	< LOD	0.035	< LOD	0.025	< LOD	0.179	< LOD	80	0.503	0.015	< LOD	0.004
Mean			< LOD		< LOD		< LOD		< LOD		< LOD		0.496		< LOD	

Reading	Time	Alloy1	Zr	Zr Error	Bi	Bi Error	Pb	Pb Error	Se	Se Error	W	W Error	Zn	Zn Error	Cu	Cu Error
1	08/06/2010 19:44	1 1/4 Cr : 0.37	< LOD	0.003	< LOD	0.002	< LOD	0.002	< LOD	0.006	< LOD	0.057	< LOD	0.023	< LOD	0.046
2	08/06/2010 19:51	1 1/4 Cr : 0.00	< LOD	0.004	< LOD	0.002	< LOD	0.013	< LOD	0.009	< LOD	0.086	< LOD	0.032	< LOD	0.067
3	08/06/2010 19:51	1 1/4 Cr : 0.00	< LOD	0.004	< LOD	0.009	< LOD	0.03	< LOD	0.008	< LOD	0.088	< LOD	0.027	< LOD	0.056
4	08/06/2010 19:52	1 1/4 Cr : 0.13	< LOD	0.003	< LOD	0.005	< LOD	0.009	< LOD	0.006	< LOD	0.065	< LOD	0.018	< LOD	0.049
5	08/06/2010 19:52	1 1/4 Cr : 0.00	< LOD	0.003	< LOD	0.009	< LOD	0.008	< LOD	0.007	< LOD	0.063	< LOD	0.017	< LOD	0.047
Mean			< LOD		< LOD		< LOD		< LOD		< LOD		< LOD		< LOD	

Reading	Time	Alloy1	Ni	Ni Error	Co	Co Error	Fe	Fe Error	Mn	Mn Error	Cr	Cr Error	V	V Error	Ti	Ti Error
1	08/06/2010 19:44	1 1/4 Cr : 0.37	< LOD	0.095	< LOD	0.287	97.49	0.182	0.626	0.068	1.116	0.05	< LOD	0.055	< LOD	0.076
2	08/06/2010 19:51	1 1/4 Cr : 0.00	< LOD	0.101	< LOD	0.372	97.51	0.237	0.513	0.084	1.158	0.065	< LOD	0.068	< LOD	0.104
3	08/06/2010 19:51	1 1/4 Cr : 0.00	< LOD	0.102	< LOD	0.349	97.54	0.223	0.567	0.08	1.071	0.059	< LOD	0.066	< LOD	0.095
4	08/06/2010 19:52	1 1/4 Cr : 0.13	< LOD	0.087	< LOD	0.288	97.55	0.184	0.609	0.067	1.075	0.049	< LOD	0.052	< LOD	0.081
5	08/06/2010 19:52	1 1/4 Cr : 0.00	< LOD	0.079	< LOD	0.28	97.58	0.179	0.543	0.064	1.107	0.049	< LOD	0.054	< LOD	0.081
Mean			< LOD		< LOD		97.53		0.572		1.105		< LOD		< LOD	

LOD = Lack of Detection

DATE OF NEXT CALIBRATION – 8th JUNE 2011

