APPLICATION REPORT

Rigaku

XRF 176

QUANTITATIVE ANALYSES OF TITANIUM ALLOYS

USING MULTI-ELEMENT SIMULTANEOUS X-RAY FLUORESCENCE SPECTROMETER "SIMULTIX 12"

INTRODUCTION

Since Ti alloys are light, strong and corrosion resistant, they are widely used for structural materials for airplanes, space development, heat exchangers for power generation, scientific plants and other materials.

Described below are examples of analyses using the Simultix 12, which can analyze Ti alloys quickly and with high precision:

1. INSTRUMENT AND MEASURING CONDITIONS

Instrument : Multi-element simultaneous X-ray fluorescence spectrometer Simultix 12
X-Ray Tube : Rh target end window type
Excitation Condition : 50kV - 70mA

Analysis Line	Mn	Cr	Cu	Mo	Fe	Al
Attenuator	Out	Out	Out	Out/In*	Out	Out
Analyzing Crystal	LiF(200)	LiF(200)	LiF(200)	LiF(200)	LiF(200)	PET
Detector	S-PC	S-PC	S-PC	SC	S-PC	S-PC

Analysis Line	V**	Sn	Si	Zr	Ni	Ti
Attenuator	Out	Out	Out	Out	Out	In
Analyzing Crystal	LiF(200)	LiF(200)	RX4	LiF(200)	LiF(200)	LiF(200)
Detector	S-PC	SC	S-PC	SC	S-PC	S-PC

* Samples with concentrations of 1 mass% or less were measured with "Attenuator: Out".

** Vanadium was measured using a goniometer for Ti alloy analyses and a standard general-purpose goniometer.

2. STANDARD SAMPLES

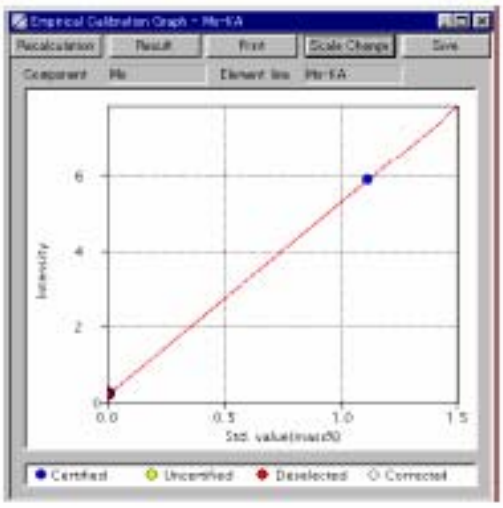
	Sample Name	Kind
NIST	641	8Mn (A)
	642	8Mn (B)
	643	8Mn (C)
	644	2Cr-2Fe-2Mo (A)
	645	2Cr-2Fe-2Mo (B)
	646	2Cr-2Fe-2Mo (C)
	654b	6Al-4V
	1128	15V-3Al-3Cr-3Sn
	1133	5Al-2Sn-2Zr-4Cr-4Mo
IARM'S™	174A	Tl-CP-4
	175A	6Al-4V
	176A	6Al-4VELI
	177A	6AL-2Sn-4Zr-2Mo
	178A	6Al-6V-2Sn

3. SAMPLE PREPARATION

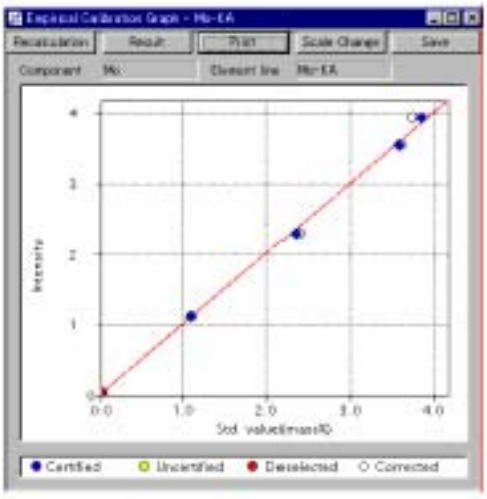
Each sample was ground using a belt surfacer with Al_2O_3 #240.

4. CALIBRATION CURVES

As for Mo, no problem arises for samples with concentrations of 1 mass% or less, but the counting loss of the detector occurs in a range exceeding 1 mass%. To suppress this counting loss, analyses were made using the “automatic calibration curve selection function”, which automatically selects a calibration curve (measuring condition) for an analysis from one that uses the attenuator and one that does not.



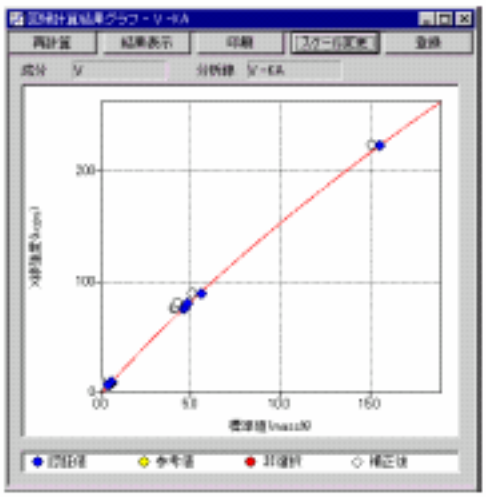
Calibration Curve for Mo
(Acc.: 0.022 mass%)
Samples with concentrations less than 1 mass% were measured without the attenuator.



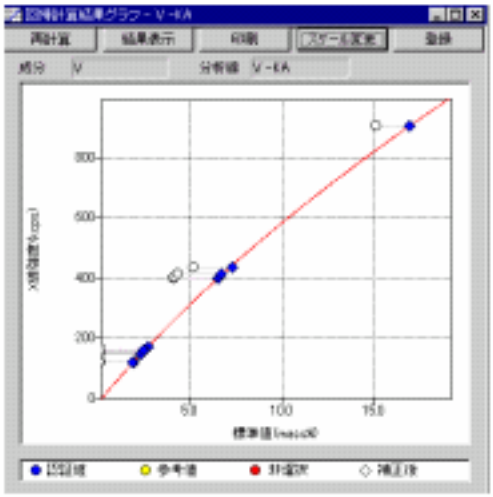
Calibration Curve for Mo
(Acc.: 0.027 mass%)
Samples with concentrations of 1 mass% or more were measured with the attenuator (1/32).

To analyze trace vanadium, by using a dedicated goniometer with higher angular resolution, an overlap correction coefficient for the Ti-Kβ1 spectrum line can be made smaller and analyses can be made more accurately than when a standard vanadium goniometer is used.

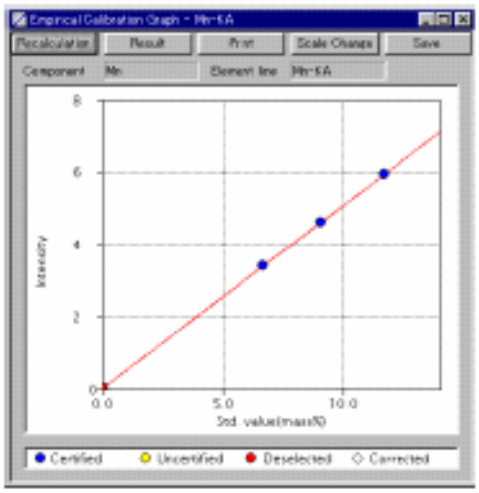
Prepared calibration curves are shown below:



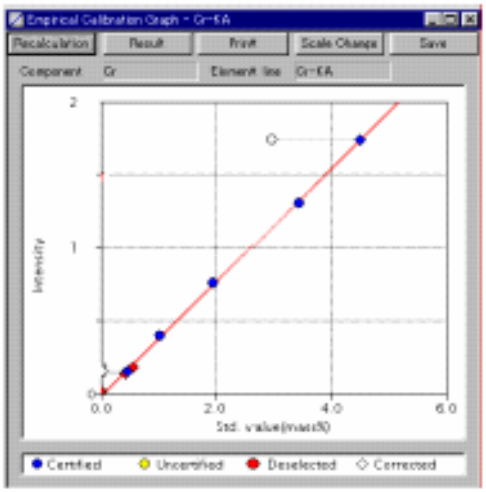
Calibration Curve for V
(Goniometer for Ti Alloys)
(Acc.: 0.045 mass%)



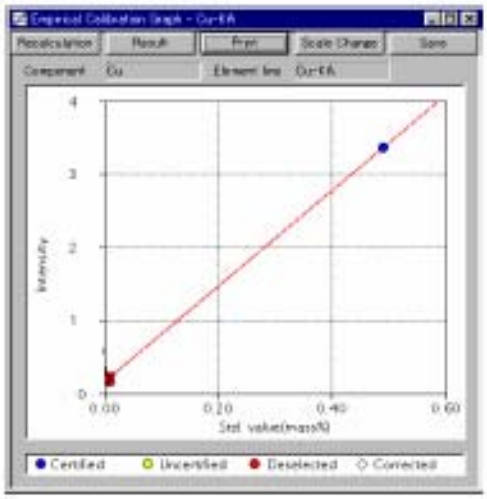
Calibration Curve for V
(Standard Goniometer)
(Acc.: 0.050 mass%)



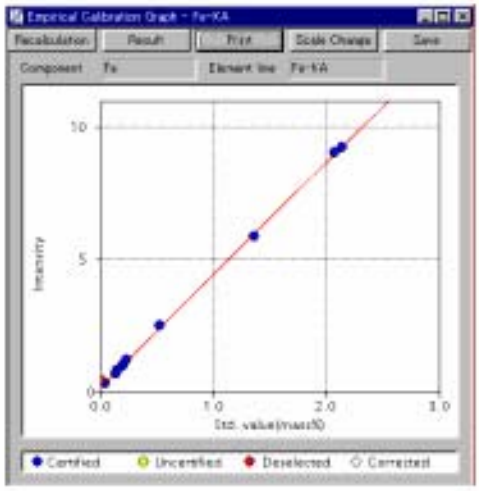
Calibration Curve for Mn
(Acc.: 0.011 mass%)



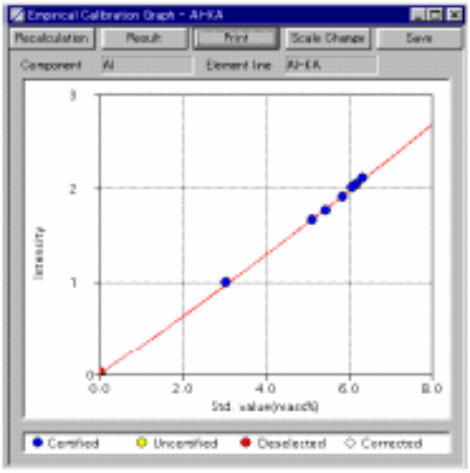
Calibration Curve for Cr
(Acc: 0.01mass%)
Overlap correction with V is required.



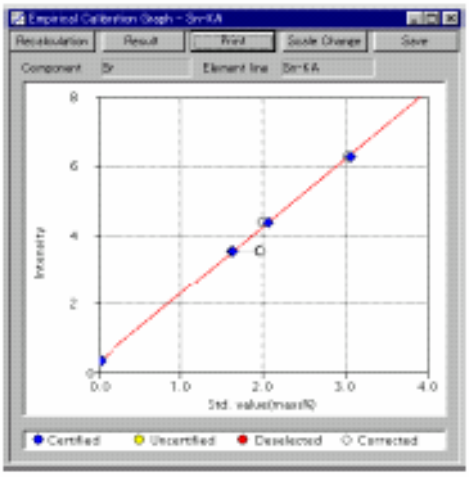
Calibration Curve for Cu
(Acc.: 0.062 mass%)



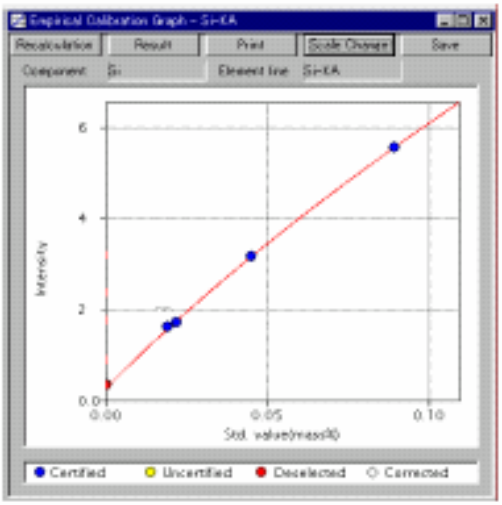
Calibration Curve for Fe
(Acc.: 0.013 mass%)



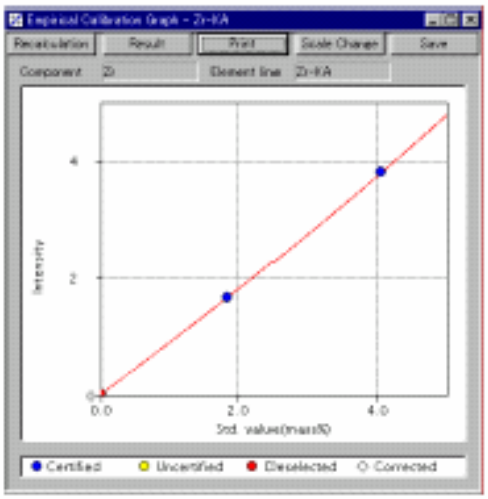
Calibration Curve for Al
(Acc.: 0.036 mass%)



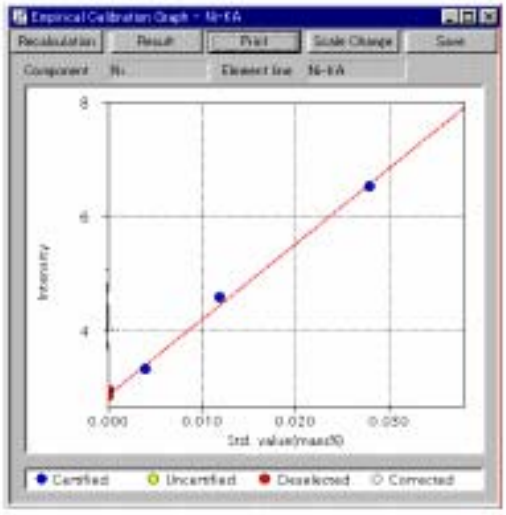
Calibration Curve for Sn
(Acc.: 0.078 mass%)
Matrix correction with Zr, Cu, Al, Mo and V is required.



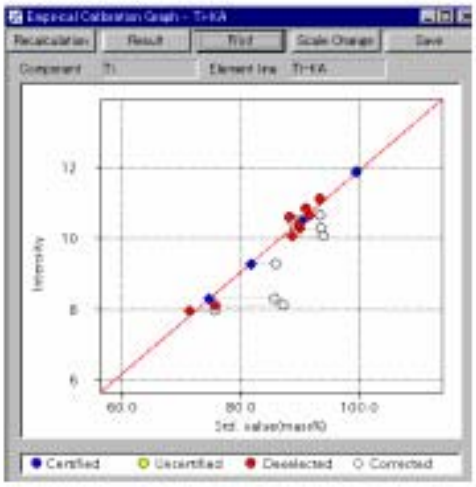
Calibration Curve for Si
(Acc.: 0.00056 mass%)



Calibration Curve for Zr
(Acc.: 0.056 mass%)



Calibration Curve for Ni
(Acc.: 0.00085 mass%)



Calibration Curve for Ti
(Acc.: 0.15 mass%)
Matrix correction with Mo, Sn, Zr and V is required.

5. CHECKING OF REPRODUCIBILITY

Sample: 177A (6Al-2Sn-4Zr-2Mo)

(Unit: mass%)

Element	Mo	Fe	Al	V (*1)	V	Sn	Si	Zr	Ni
Standard Value	2	0.045	5.87	0.006	0.006	1.97	0.089	4.05	0.007
Average	1.98	0.043	5.85	0.0073	0.00015	1.99	0.089	4.08	0.009
Standard Deviation	0.0009	0.0003	0.0032	0.0005	0.00018	0.0008	0.00035	0.0012	0.00011
Coefficient of Variation	0.045	0.72	0.053	7.37	120	0.041	0.39	0.030	1.22

*1: About Goniometer Exclusively for Ti Alloy Analyses

Since Ti-Kβ1 overlaps with V-Kα, an optical system (goniometer exclusively for Ti alloy analyses) with higher resolution is used. This improves the accuracy for trace V as can be seen at "V (*1)" in the above table. However, since resolution is inversely proportional to sensitivity, sensitivity lowers a little when this optical system is used.

Sample: NIST1128

(Unit: mass%)

Element	Cr	Fe	Al	V (*1)	V	Sn	Ti
Standard Value	2.96	0.134	3.05	15.13	15.13	3.04	75.96
Average	2.96	0.125	3.07	15.13	15.13	3.04	75.65
Standard Deviation	0.0019	0.00046	0.0023	0.0092	0.0049	0.0010	0.015
Coefficient of Variation	0.06	0.37	0.074	0.061	0.032	0.034	0.02

Sample: NIST1133

(Unit: mass%)

Element	Mo	Fe	Al	Sn	Si	Zr	Ti
Standard Value	3.72	0.144	5.13	1.98	0.027	1.84	87.16
Average	3.75	0.144	5.13	1.98	0.019	1.84	87.14
Standard Deviation	0.0013	0.00049	0.0029	0.00081	0.00014	0.00098	0.017
Coefficient of Variation	0.034	0.34	0.057	0.041	0.75	0.053	0.019

6. CONCLUSION

From the analysis results of NIST standard samples shown above, accuracy can be checked using differences between the standard values and the average values, and precision can be checked using the standard deviations and coefficients of variation.

By using a multi-element simultaneous X-ray fluorescence spectrometer, high precision analyses can be made more quickly and accurately.

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