ICS 71.040.40
CCS G 04

NATIONAL STANDARD OF THE PEOPLE'S REPUBLIC OF CHINA

GB/T 28633-2012

Surface chemical analysis -- X-ray photoelectron spectroscopy -- Repeatability and constancy of intensity scale

Issued on: July 31, 2012 **Implemented on: February 1, 2013**

Issued by: General Administration of Quality Supervision, Inspection and Quarantine of the People's Republic of China, Standardization Administration of the People's Republic of China

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Foreword

This standard was drafted in accordance with GB/T 1.1-2009 given rules.

This standard uses the translation method identical with ISO 24237.2005 (E) "Surface chemical analysis - X-ray photoelectron spectroscopy of intensity scale

Repeatability and consistency. "

The standard micro-beam analysis by the National Standardization Technical Committee (SAC/TC38) and focal points.

This standard was drafted. Analysis and Testing Center, Beijing Normal University.

Introduction

Analysis of surface X-ray photoelectron spectroscopy (XPS) is widely used material. From the measured photoelectron spectra give each element of the sample surface

Within the level of binding energy, the binding energy of the control elements in the sample identification table different elements (except hydrogen and helium). From the measured photoelectron peak intensity

Quantitative information can be obtained in these elements. Relative sensitivity factors apply formulas and equipment provided by the manufacturer, it can be quantified. spirit

Sensitivity factor is applicable to instruments is essential, usually after the instrument is installed, or in response to the peak intensity of the instrument was calibrated by a competent body/energy

After the function, sensitivity factor can be applied directly. In this standard, on the impact of peak intensity XPS measurement uncertainty of two important

The instrument factors. Repeatability (1) of the peak intensity measured; (2) peak intensity drifts over time.

For the analysis of trends and differences between similar samples, reproducibility is important. Factors affecting the measurement instrumentation repetitive

Are. stability, detector X-ray source is provided, the sensitivity of the instrument sample location data acquisition parameters and data processing methods. instrument

Intensity scale drift will affect the overall accuracy of the results of any quantitative analysis. This is due to instrument drift component, the electronic control unit

And detector aging and other factors. In the XPS instrument, it has been found that the equipment in use, the strength of the instrument/energy response function

It varies with the life of the instrument.

This standard describes a measuring instrument repeatability and consistency of the underlying strength of a simple method for instrument calibration, such as improved operating step

Step, re-set parameters or re-calibrate the instrument intensity/energy response function. Therefore, this method should be repeated periodically testing instruments, if

Instrument manufacturer or a qualified agency has checked the equipment to work properly, it is during this period of the present method is very useful. The method uses pure

Copper (Cu) sample for non-monochromatic aluminum (Al) or magnesium (of Mg) of the X-ray source X-ray photoelectron spectrometer, monochromator or aluminum (Al) X-ray source

X-ray photoelectron spectroscopy.

This method is not set forth all possible instrument errors exist, because it is very time-consuming test, and requires specialized knowledge and equipment. But

Describes the XPS instrument intensity scale common basic problem that the repeatability and drift. Can be used simultaneously when using this method

GB/T 22571-2008 [1] instrument calibration energy.

Surface chemical analysis - X-ray photoelectron spectroscopy

Repeatability and consistency of intensity scale

1 Scope

This standard specifies the routine analysis method for assessing the strength of the underlying repeatability and consistency of the method of X-ray photoelectron spectroscopy, the use of non

Monochromatic Al/Mg or monochrome AlX ray source. This standard applies only to instruments fitted sputter ion gun. This standard is not strength/energy ring

It should calibration function. This instrument calibration by the manufacturer or other qualified agencies to complete. This standard provides the data used to assess and confirm the instrument

Accuracy response function to maintain a constant strength during use/energy. This standard provides some may affect the consistency of instrument settings

instructions.

2 Symbols and Abbreviations

The average peak area A2 after deducting Shirley background Cu2p3/2 peak

A component value A2j A2 for j-th measured in a group of measurement values

A3 deduction Shirley background, the average peak area Cu3p peak

A component value A3j A3 for j-th measured in a group of measurement values

Pi i symbol for one of the three parameters of which

Subscript j independent measurements of one parameter Pij

Pi represents the mean of parameter A2, A3 and the amount of any one of A3/A2

The average of Pi parameters Pij j-th measurement

When U95 (Pi) 95% confidence limits mean uncertainty Pi

XPS X-ray photoelectron spectroscopy

delta 95% confidence limits (set by the analyst) if consistency tolerance value A3/A2 of

Energy delta instrument binding energy scale drift amount, equal to the measured Cu2p3/2 binding energy value of the peak maximum intensity minus 932.7eV

sigma (Pi) parameter Pi repeatability standard deviation

3 Methods Summary

Here is an overview of the method, in order to understand in detail in Chapter 4 steps. Measurement Cu2p3/2 and Cu3pX shot at suitable parameters

Ray photoelectron peak intensity, and get ready to foil reference material is necessary in order to evaluate X-ray photoelectron spectrometer by the method reproducibility

And consistency. Choose these peaks because they are close to the actual analysis of the binding energy of the high limit and low limit. These peaks are explicitly selected,

And a corresponding reference data.

The initial step to prepare reference material and instrument parameter settings see 4.1 to 4.5, the flow chart shown in Figure 1, the figure includes the relevant sub-headings.

If you have not had the intensity of repetitive measurement, the user needs to enter from 4.6 to 4.7. In 4.7, the measurement sequence is repeated 7 times

Strength Cu2p3/2 and Cu3p peaks. These data are given the repeatability standard deviation of the peak intensity. Repetitive elements from the following. X-ray source,

Spectrometer stability detector and the electronic power supply unit; peak intensity measurement of the sample position sensitivity and statistical noise spectrum at the peak.

In this method, it should be limited to the working conditions in order to ensure the strength was measured noise is relatively small. This is discussed in Appendix A. weight

The value of the complex will depend on the standard deviation of the sample positioning method. In 4.7.1, we need to use the same method of locating the sample, only the final calibration

The method of using the targeted sample to be effective.