Invited Paper

Common Data Processing System for ISO Standards

Kazuhiro Yoshihara
ULVAC-PHI, Inc., 370 Enzo, Chigasaki 253-8522, Japan
kazuhiro_yoshihsra@ulvac.com

COMPRO originally was designed to treat spectral data taken on different machines in a computer.   COMPRO has been upgraded many times, and the next version will be 9.0.   ISO/TC201 publishes calibration procedures for energy and intensity scales.   However, users may feel difficulty to use these standards correctly without any guides.   Therefore, the main feature of COMPRO 9.0 is to support users when they calibrate and check their spectrometers.

1. Introduction

COMPRO (Common Data Processing System) can assess the data processing procedures proposed by scientists, calibrate a spectrum, and construct spectral databases[1].   COMPRO has been upgraded many times. From version 4, COMPRO can be downloaded from the home page of the Surface Analysis Society of Japan (http://www.sasj.gr.jp).   By using COMPRO, one can convert the format of spectral and depth profile data to ISO 14976 format[2], and also can attach sample information and calibration information of energy and intensity scales according to ISO 14975 format[3].   At present, COMPRO has data processing algorithms such as zooming, differentiation, smoothing, background subtraction, peak fitting, quantification, qualification, thin film analysis and so on.   COMPRO also has databases for physical properties of elements, standard spectra, reference spectra and AES absolute spectra measured by Prof. Goto.   The reference spectra database has been constructed by the volunteer work of members of the Surface Analysis Society of Japan[4].

ISO/TC201 publishes calibration procedures for energy and intensity scales.   They are ISO 15472[5], 17973[6] and 17974[7] for energy scale calibrations, and 21270[8], 24236[9] and 24237[10] for intensity scale calibrations.   However, users may feel difficulty to use these standards correctly without any guides.   Therefore, COMPRO 9.0 has a routine to obtain the calibration results for energy and intensity scales according to these ISO standards.   By following the instructions given by COMPRO 9.0, users can obtain the calibration properties of their spectrometers.
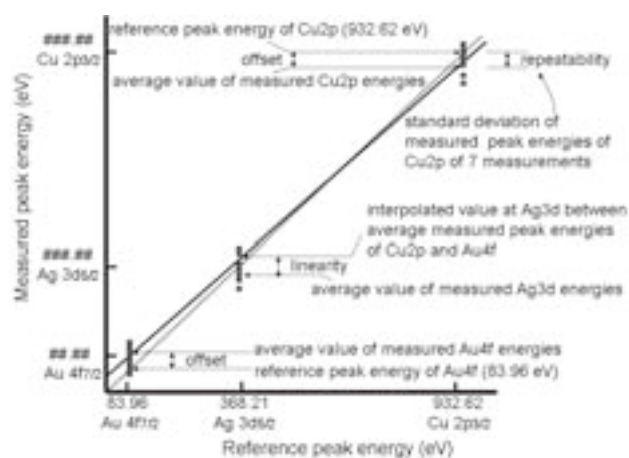

Fig.1 Schematic illustration of the energy scale calibration

2. Energy scale calibration

ISO/TC201 has published 3 standards for energy scale calibration.   ISO 15472 is for XPS.   ISO 17973 is for medium resolution AES, and ISO 17974 is for high resolution AES.   In this paper, ISO 15472 (energy scale calibration for XPS) will be introduced.   Figure 1 shows a schematic illustration of the energy scale calibration process for monochromatic Al XPS.   The ordinate shows measured peak energies and the abscissa shows reference peak energies given by ISO 15472.   ISO

15472 requests a user to measure three peaks. They are low (Au $4f_{7/2}$), medium (Ag $3d_{5/2}$), and high binding energy peaks (Cu $2p_{3/2}$). The energy scale calibration is defined by three parameters. They are the repeatability, linearity and offset. The repeatability is defined as the standard deviation of 7 measurements of the Cu $2p_{3/2}$ peaks. The offset is defined as the difference between average value of measured energies and reference energies for the Au $4f_{7/2}$ and Cu $2p_{3/2}$ peaks. A straight line can be drawn between the average measured peak energies of the Cu $2p_{3/2}$ and Au $4f_{7/2}$ peaks. Then an interpolated value of Ag $3d_{5/2}$ peak energy can be obtained. The linearity is defined as the difference between the interpolated value and the average value of measured Ag $3d_{5/2}$ energies.

In COMPRO, when one clicks [Analyzer] – [Energy] – [XPS] menu, one can start the energy scale calibration. The first thing to do is to select source energy and to set the tolerance limit for the error of one's measurements. The tolerance limit is based on the accuracy required in the laboratory's work and the stability of the instruments. If this is the first calibration, it is necessary to make 7 separate measurements of energies of the Au $4f_{7/2}$, Ag $3d_{5/2}$ and Cu $2p_{3/2}$. If one has already measured these peaks, COMPRO automatically reads the peak energies, and presents the energy calibration results. For regular calibration checks, it is sufficient to measure the peak energies of only two peaks (Au $4f_{7/2}$ and Cu $2p_{3/2}$). If one has already measured these data, COMPRO automatically reads the peak energies and calculates the Au $4f_{7/2}$ and Cu $2p_{3/2}$ offset values. The offset values are plotted in the control chart, as shown in Fig.2.
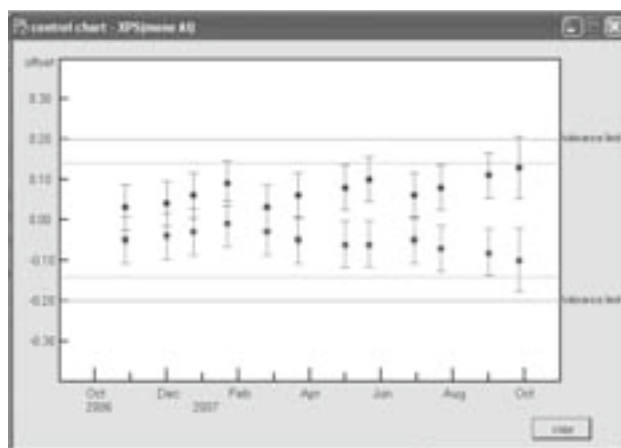

Fig.2 Control chart showing offset values from the energy scale calibration as a function of time from the first calibration

3. Intensity scale calibration

To obtain correct quantitative analysis, spectra intensities must be measured correctly. To that end, the following three items are important. The first one is the linearity of the intensity scale which is described in ISO 21270. Linearity means that the recorded spectral intensity must be proportional to the number of electrons entering into spectrometer. The second is repeatability, which means that intensities obtained from the same sample must be statistically identical. This is described in ISO 24236 (for AES) and ISO 24237 (for XPS). And the last one is constancy, which means that the measured intensities do not change with time. This is also described in ISO 24236 and ISO 24237.

3.1 Linearity of the intensity scale

ISO 21270 specifies two methods for determining the maximum count rate for an acceptable limit of divergence from linearity for AES and XPS. The first is to vary the source flux using a Cu sample. The second is a spectrum ratio method using Cu, stainless steel or the sample holder. In the first method, it is necessary to measure the count rate of Cu LVV for the more than 30 values of the beam current or anode emission current. In the second method, it is necessary to measure two spectra of Cu, stainless steel, or the sample holder at two X-ray source emission currents (100 % and about 25% of the maximum values).
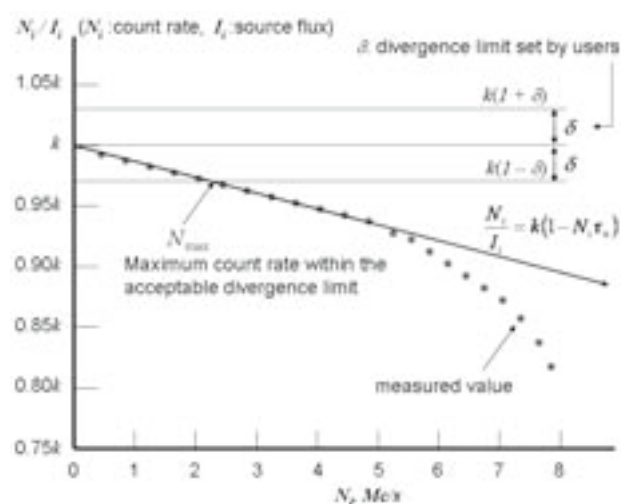

Fig.3 The relation between the ratio of count rate and source flux and the count rate

From the first method, one can obtain the relation between the ratio of count rate and source flux versus count rate as shown in Fig.3. Here, N_i is count rate, I_i is source flux, and the circles are the measured ratio. If one extrapolates the data points to zero count rate, one can get the ratio k, which is the ideal ratio without any counting losses due to the dead time at the electronics. The horizontal line at k is the ideal relation without any counting losses. If one selects an acceptable divergence limit, α, from linearity, the intersection of the extrapolated line and the divergence limit line gives the maximum count rate within the acceptable tolerance. The extrapolated line can be expressed as $N_i/I_i = k(1 - N_i\tau_n)$ where τ_n is the dead time of the counting electronics. If τ_n is known, the maximum count rate can be increased (but still remaining in the linear region of Fig.3) and a correction made for the counting losses.

If one has already measured the 30 spectra for Cu, COMPRO will give the intensity scale linearity result. When one selects <Analyzer> - <Intensity> - <varying source flux> menu, COMPRO automatically calculates the peak intensity and gives the result shown in Fig.4. In this case, maximum measured count rate is 1.2×10^6 counts/sec for $\delta = 2.5$ % and the dead time is 20ns.

For the second method, one shall measure two Cu spectra. When <Analyzer> - <Intensity> - <spectrum ratio> is selected, COMPRO automatically calculates the spectrum ratio and gives a simpler plot.

3.2 Repeatability and constancy of the intensity scale

There are two standards for repeatability and constancy, ISO 24236 for AES and ISO 24237 for XPS. The repeatability is defined as the standard deviation of ratios of Cu $2p_{3/2}$ and Cu 3p peak intensities for 7 measurements. (For AES, peak heights of the Cu MVV and Cu LVV peaks are used.) The constancy is defined as the stability of the ratio of the Cu $2p_{3/2}$ and Cu 3p intensities measured every three months. (For AES, the constancy is checked every two months)

COMPRO can give the calibration results by clicking <Analyzer> - < Intensity> - <Repeatability and constancy>. The first thing to do is to set the tolerance limit for constancy. It is then necessary to enter the
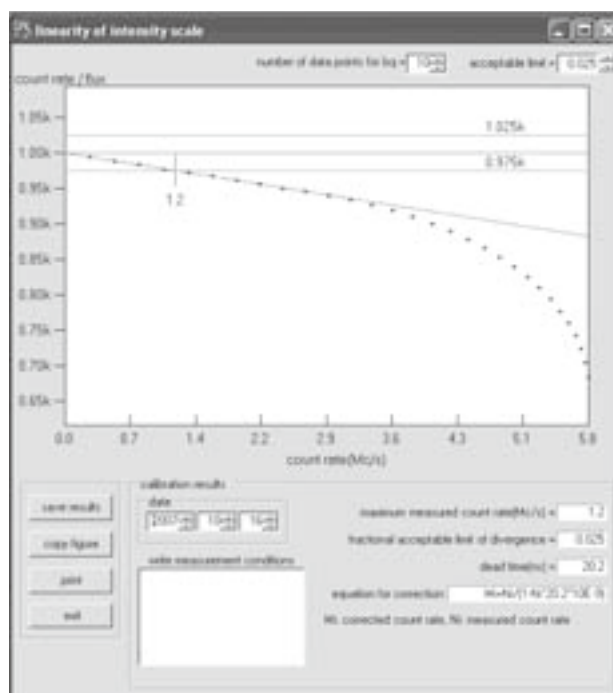

Fig.4 COMPRO screen for linearity check of the intensity scale.

intensities of Cu $2p_{3/2}$ and Cu 3p peaks for XPS. If one has already measured these spectra, peak intensities are automatically calculated after subtracting a Shirley background. Ratios of Cu 3p and Cu $2p_{3/2}$ intensities are calculated and the standard deviation is obtained. For constancy, it is necessary to enter one or two sets of Cu 3p and Cu $2p_{3/2}$ peak intensities, and COMPRO will calculate the ratio of the Cu 3p and Cu $2p_{3/2}$ intensities, and the results will be recorded as indicated in Fig.5.
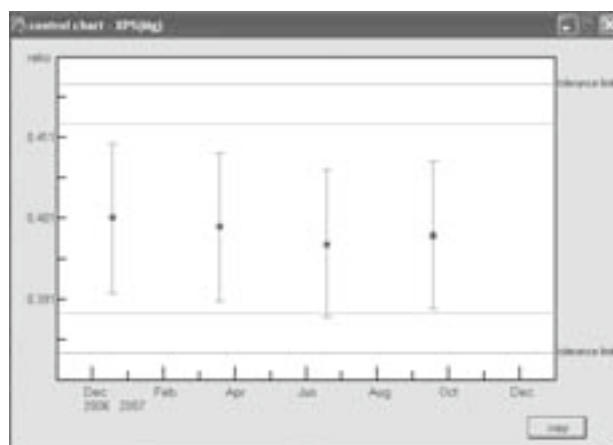

Fig.5 Control chart showing the ratios of the Cu 3p and Cu $2p_{3/2}$ intensities as a function of time

4. Conclusions

ISO standards are not so easy to use without guidance. The main feature of COMPRO 9 is to support users when they calibrate and check spectrometers using ISO standards. COMPRO 9 can be downloaded from http://www.sasj.gr.jp. ISO standards can be purchased from http://www.iso.org/iso/standrads_development.

5. References

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