



## Surface and core hole effects in X-ray photoelectron spectroscopy

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### ABSTRACT

In XPS analysis, two effects, which significantly reduce the measured peak intensity, are usually neglected: the core hole left behind in an XPS process which causes “intrinsic” excitations and excitations as the photoelectron pass through the surface region. We have calculated these effects quantitatively for various energies, geometries, and materials. Instead of considering the two effects separately, we introduce a new parameter, namely the correction parameter for XPS or  $CP_{XPS}$ , which takes into account both effects. We define this  $CP_{XPS}$  as the change in probability for emission of a photoelectron caused by the presence of the surface and the core hole in comparison with the situation where the core hole is neglected and the electron travels the same distance in an infinite medium. The calculations are performed within the dielectric response theory by means of the QUEELS–XPS software determining the energy-differential inelastic electron scattering cross-sections for X-ray photoelectron spectroscopy (XPS) including surface and core hole effects. This study has been carried out for electron energies between 300 eV and 3400 eV, for angles to the surface normal between 0° and 60° and for various materials. We find that the absolute effect is a reduction by 35–45% in peak intensities but that the variation in  $CP_{XPS}$  with material, angle and energy are  $< \pm 10\%$  for emission angle  $\leq 60^\circ$  and photoelectron energy  $\leq 1500$  eV. This implies that when XPS analysis is done using relative intensities, the combined effect of the surface and of the core hole is typically less than  $\approx \pm 10\%$  for geometries and energies normally used in XPS. In practice, it is however difficult to determine the bare peak intensity without the intrinsic electrons because the two overlap in energy.

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### 1. Introduction

Accurate quantitative chemical analysis of solid surfaces by electron spectroscopies as X-ray photoelectron spectroscopy (XPS) requires a correct understanding of several mechanisms. Particularly, a precise knowledge of inelastic scattering events experienced by the electrons in the solid is essential. Usually it is assumed that all the information on inelastic electron–solid interactions is contained in the inelastic mean free path (IMFP). Nevertheless, generally IMFPs used in XPS are derived from models considering an electron traveling in an infinite medium [1]. This does not take into account surface excitations [2,3] (i.e. excitations occurring while the electron is moving in the vacuum and in a shallow region in the medium) as well as energy losses originating from the static core hole created during the photoexcitation of the core electron [3–5] (excitations generated by the core hole are called “intrinsic” while excitations that take place during the photoelectron transport process are called “extrinsic”). It was previously found that the intrinsic excitations account for 20–50% of the measured intensity in an energy range  $\sim 15$ –25 eV below the peak energy [6,7]. In later papers [8,9], an extensive set of

experimental peak intensity ratios from elemental solids and alloys was compared to theory. The peak intensities were determined by different background correction methods. In these papers, it was pointed out that the Shirley and straight line backgrounds are not well defined because the operator must decide at what energy the peak starts and ends and the resulting peak intensities of course depend on this choice. This dependency was studied and it was found that it is important to select consistent values (either close to the peak or further away) to get the smallest deviations from theoretical peak intensity ratios. When this is done the RMS deviation of peak intensity ratios from theory was 35% for the Shirley and 25% for the straight line method. For the Tougaard background the energy range of the peak is determined by the algorithm and is thus not an input in the analysis. With this background correction, the determined peak intensity ratios deviated by 11% (RMS) from theory. The better agreement obtained with the Tougaard background is due to the intrinsic excitations which extend  $\sim 30$ –50 eV below the peak energy and which is accounted for by this background correction. In contrast, the Shirley and straight line backgrounds include a smaller or larger fraction of intrinsic electrons depending on the energy range applied to define the extension of the peak. To apply the Tougaard background, it is necessary to record the spectra in a wider energy range, and it is therefore still common to use the Shirley and the straight line backgrounds for a practical XPS peak intensity analysis. To be able to

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apply these methods for accurate peak intensity determination it is of interest to calculate the fraction of peak intensities that are in the peak and in the off peak regions (i.e. intrinsic electrons). If this can be calculated, the peak intensities determined with the Shirley and straight line backgrounds can be corrected to give the full peak intensity which would then be expected to give better agreement with theory.

It is the aim of the present paper to calculate the effects of both surface excitations and the core hole by a semi-classical dielectric response theory [3]. This model [2] was previously found to give a good quantitative description of experimental reflected electron energy loss spectra [10]. We describe the effects by a new parameter which is defined such that it can easily be understood and applied in practice by surface analysts.

In recent papers [11,12] about surface effects in reflection-electron-energy loss spectroscopy (REELS) and in elastic peak electron spectroscopy (EPES), we have introduced a new definition for the surface excitation parameter  $P_s(E, \theta)$  or SEP depending on the energy  $E$  of the incident electron and on its angle  $\theta$  with respect to the surface normal as follows: the SEP is the change in excitation probability for an electron caused by the presence of the surface in comparison to the situation where the electron travels the same distance in an infinite medium. According to this definition, a particular aspect of surface effects is taken into account, namely the Begrenzungs effect i.e. the decrease of the bulk inelastic cross-section close to the surface due to the coupling between the volume and surface modes that are orthogonal [13]. Consequently, with this definition, we removed all ambiguities for the practical use of SEP in EPES and, with this definition, the additional attenuation of the elastic peak intensity caused by the presence of the surface is simply  $\exp[-P_s]$ .

Similarly, we introduce here a correction parameter for XPS,  $CP_{XPS}(E, \theta)$ , taking both surface and core hole effects into account and defined as: the  $CP_{XPS}$  is the change in probability for emission of a photoexcited electron caused by the presence of the surface and the core hole in comparison with the situation where the core hole is neglected and the electron travels the same distance in an infinite medium. This  $CP_{XPS}$  parameter thus incorporates peak attenuations due to both the surface and the core hole in XPS. The practical application of this  $CP_{XPS}$  is straight forward. Thus the peak intensity should first be determined according to the usual procedure where these effects are neglected and using the IMFPs for the infinite medium and this peak intensity should then be multiplied by  $\exp[-CP_{XPS}]$ . Note that this factor should not be used with the Tougaard method since this already includes the effect of the core hole while the surface excitations are not included. In the present work, we determine  $CP_{XPS}$  for different material types (metal, semiconductor, insulator), for photoelectrons energies varying between 300 eV and 3400 eV and for exit angles of the photoelectron between  $0^\circ$  and  $60^\circ$ .

## 2. Theoretical model

The XPS model considered here [3] is based on the use of the surface reflection model [14] describing the interactions of electrons with semi-infinite media in terms of the dielectric properties of the bulk material and incorporates the effects of the surface and of the static core hole created during the photoionization process. We analyze here the case of an electron-hole pair created at depth  $x_0$  below the surface of a semi-infinite medium characterized by a dielectric function  $\epsilon(k, \omega)$ . The electron travels along a straight line with velocity  $v$ , energy  $E$  and angle  $\theta$  with respect to the surface normal, while the core hole is stationary with infinite lifetime. Within this model, the effective inelastic electron scattering cross-section  $K_{\text{eff}}^{\text{XPS}}(E, \hbar\omega, x_0, \theta)$  is defined as the average probability that the electron, excited at depth  $x_0$ , loses an energy  $\hbar\omega$  per unit energy loss and per unit path length traveled in the solid (the XPS in the

expression of  $K_{\text{eff}}$  stresses the presence of the core hole effects in the calculations).

As shown in Ref. [3], it is possible to express  $K_{\text{eff}}^{\text{XPS}}(E, \hbar\omega, x_0, \theta)$  in terms of the induced potential  $\Phi_{\text{ind}}(\mathbf{k}, \omega)$  created by the hole and the electron itself:

$$K_{\text{eff}}^{\text{XPS}}(E, \hbar\omega, x_0, \theta) = \frac{2}{(2\pi)^4 x_0 \hbar^2 \omega} \int_{-\infty}^{+\infty} dt \int d\mathbf{r} \rho_e(\mathbf{r}, t) \times \text{Re} \left[ i \int d\mathbf{k} \mathbf{k} v \Phi_{\text{ind}}(\mathbf{k}, \omega) e^{i(\mathbf{k}\mathbf{r} - \omega t)} \right], \quad (1)$$

where  $\rho_e(\mathbf{r}, t)$  is the charge density of the electron.  $\Phi_{\text{ind}}(\mathbf{k}, \omega)$  is obtained within the surface reflection model in which the potential of a system of moving charges in a semi-infinite medium is obtained by considering two infinite pseudomedia, the medium ( $M$ ) and the vacuum ( $V$ ). In the pseudomedia  $M$  and  $V$ , we have to consider all charges and their images. For XPS, the relevant charges are, for  $t > 0$ , the electron  $\rho_e = -e\delta(\mathbf{r} - \mathbf{x}_0 - \mathbf{v}t)$ , the core hole  $\rho_h = e\delta(\mathbf{r} - \mathbf{x}_0)$ , their images  $\rho_e^i = -e\delta(\mathbf{r} + \mathbf{x}_0 - \mathbf{v}^i t)$ ,  $\rho_h^i = e\delta(\mathbf{r} + \mathbf{x}_0)$  and fictitious surface charges  $\sigma_M$  and  $\sigma_V$  introduced to satisfy the boundary conditions (in previous expressions,  $\mathbf{x}_0 = (-x_0, 0, 0)$ ). We note that fictitious surface charges are determined by the requirement that the potentials, and the normal components of the displacement vectors in each pseudomedium must be continuous at the surface. Then, the resolution of Poisson's equation in Fourier space for each of the two infinite pseudomedia allows to obtain the induced potentials  $\Phi_{\text{ind}}^M$  and  $\Phi_{\text{ind}}^V$ .

This calculation implies the knowledge of the dielectric function of the medium  $\epsilon(\mathbf{k}, \omega)$  or equivalently the energy loss function (ELF)  $\text{Im}\{-1/\epsilon(k, \omega)\}$ . To evaluate this latter, we consider as a model the expansion in Drude–Lindhard type oscillators [15]

$$\text{Im}\left\{-\frac{1}{\epsilon(k, \omega)}\right\} = \sum_{i=1}^n \frac{A_i \hbar \gamma_i \hbar \omega}{(\hbar^2 \omega_{0ik}^2 - \hbar^2 \omega^2)^2 + \hbar^2 \gamma_i^2 \hbar^2 \omega^2} \theta(\hbar\omega - E_C) \quad (2)$$

with the dispersion relation:

$$\hbar\omega_{0ik} = \hbar\omega_{0i} + \alpha_i \frac{\hbar^2 k^2}{2m}. \quad (3)$$

$A_i$ ,  $\hbar\gamma_i$ ,  $\hbar\omega_{0ik}$  and  $\alpha_i$  are the strength, width, energy and dispersion of the  $i$ th oscillator, respectively and the step function  $\theta(\hbar\omega - E_C)$  is included to describe the effect of the energy band gap  $E_C$  in semiconductors and insulators. These parameters in the expansion are taken from Ref. [12,16] for the different materials studied here.

In XPS experiments, electrons from a wide range of depths are sampled. This implies to perform a weighted average of  $K_{\text{eff}}^{\text{XPS}}$  over the total of all path lengths  $x$  [3] with the weight function  $Q(E, x, \theta)$  that is the path length distribution function for those electrons that have undergone a single inelastic collision. The result is the inelastic scattering cross-section

$$K_{\text{sc}}^{\text{XPS}}(E, \hbar\omega, \theta) = \frac{\int_0^\infty dx Q(E, x, \theta) K_{\text{eff}}^{\text{XPS}}(E, \hbar\omega, x_0, \theta)}{\int_0^\infty dx Q(E, x, \theta)}. \quad (4)$$

$K_{\text{sc}}^{\text{XPS}}$  can be seen as the sum of 4 contributions:  $K_{\text{bulk}}$  (or  $K_{\text{intr}}$ ), i.e. the inelastic electron scattering cross-section (per unit energy loss and per unit path length traveled in the solid) for electrons moving in an infinite medium obtained from the theory of Lindhard [17];  $K_{\text{surf}}$ , the surface inelastic scattering cross-section in the absence of the hole;  $K_{\text{intr}}^{\text{XPS}}$ , the intrinsic contribution (that can also be divided into a bulk and a surface part,  $K_{\text{intr, bulk}}^{\text{XPS}}$  and  $K_{\text{intr, surf}}^{\text{XPS}}$ ) and  $K_{\text{interf}}^{\text{XPS}}$ , the interference term. The calculations are performed with the QUEELS–XPS software [18]. We again emphasize that the only input in the model to determine  $K_{\text{eff}}^{\text{XPS}}$  and thus  $K_{\text{sc}}^{\text{XPS}}$  is the dielectric function ( $k, \omega$ ) of the medium.

We note that phonon effects are neglected in the model. However, it has been shown [5,19] that energy loss due to electron–phonon interactions are insignificant in comparison to the processes considered here except for materials with very large energy band gap. Crystalline effects are also neglected in the present calculations. Crystalline effects can be important [20,21] and in particular will cause a substantial peak intensity variations with the emission angle [22] but this effect is small for polycrystalline materials.

Due to the analogy between SEP and  $CP_{XPS}$  calculated respectively for REELS and XPS, the procedure to obtain  $CP_{XPS}$  is very similar to the one used to get SEP that is extensively explained in Ref. [11] and will not be repeated here. Thus, we calculate the surface and hole contribution  $K_{SH}$  as  $K_{SH} = \int (K_{sc}^{XPS} - K_{inf}) d\hbar\omega$ . According to the definition of  $CP_{XPS}$  given above, we have

$$CP_{XPS}(E, \theta) = \frac{K_{SH}(E, \theta)}{\int K_{sc}^{XPS}(E, \theta) d\hbar\omega} \quad (5)$$

### 3. Results and discussion

Figs. 1 and 2 show  $CP_{XPS}$  as a function of emission angle  $\theta$  calculated for Si and Cu for electrons of energy  $E = 300, 1000, 3400$  eV. First, we observe that the angular distributions of  $CP_{XPS}$  are decreasing with the energy. But now, unlike the SEP angular distribution [11,12], the  $CP_{XPS}$  is not a monotonic increasing function of the angle but has a maximum at an angle that decreases with the energy. Moreover, the difference between the smallest and the largest  $CP_{XPS}$  values increases with the energy.

This behaviour can be understood by considering the following effects:

1. For increasing angles  $\theta$ , it is well known that extrinsic surface losses,  $K_{surf}$ , that account for  $\sim 95\%$  of the surface losses ( $K_{intr,surf}^{XPS}$  can thus be neglected) [3,23], are enhanced with respect to the bulk losses,  $K_{bulk}$  [2]. The reason is that, for glancing angles, electrons lose more energy to extrinsic excitations in the vacuum because they interact for a longer time with the solid compared to electrons emitted perpendicular to the surface.
2. Bulk intrinsic losses,  $K_{intr,bulk}^{XPS}$ , are an important part of bulk losses and their contribution decreases with increasing emission angle [3,23,24].

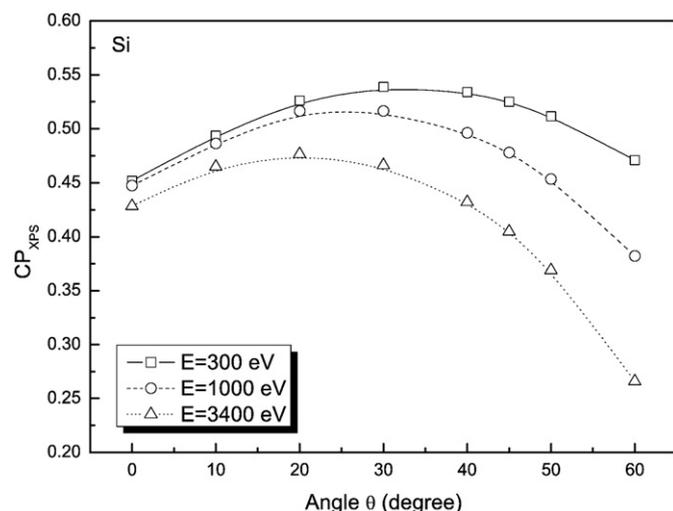


Fig. 1. Angular distribution of the  $CP_{XPS}(E, \theta)$  for Si and for energies of 300 eV, 1000 eV and 3400 eV.

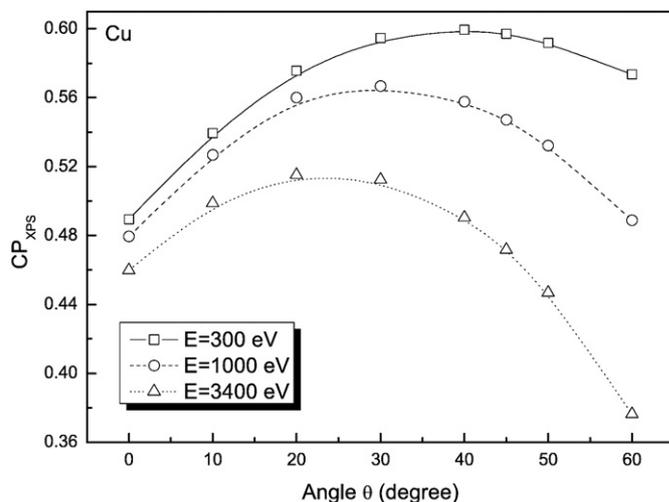


Fig. 2. Angular distribution of the  $CP_{XPS}(E, \theta)$  for Cu and for energies of 300 eV, 1000 eV and 3400 eV.

3. When energy  $E$  increases, the surface losses,  $K_{surf}$ , decreases relatively to the bulk losses,  $K_{bulk}$ . This has been known for a long time [25].
4. For increasing energy, the extrinsic contribution to the spectrum,  $K_{surf} + K_{bulk}$ , decreases [3]. This is similar to the case of REELS. However, for increasing energy, the intrinsic contribution,  $K_{intr,bulk}^{XPS}$  ( $K_{intr,surf}^{XPS}$  being negligible), increases, but saturates for very large energies [3]. This is because, for large energies, the electron moves, within the relaxation time of the plasmon, a large distance compared with the wavelength of the plasmon; therefore the potential around the core hole is large within the relaxation time of the plasmon and the probability for plasmon excitation is large. For small energies, we approach the adiabatic limit where the potential around the hole changes more slowly and the excitation probability is therefore smaller.

Thus, when the angle increases,  $CP_{XPS}$  is subject to two opposite effects: extrinsic surface excitation increases (point 1) and the hole contribution to bulk excitation decreases (point 2). This implies that, for increasing angle, the increase of surface losses due to surface excitations is (to some extent) compensated by the decrease of bulk losses due to the hole effect. As seen in Figs. 1 and 2, these effects give rise to an increase with angle in  $CP_{XPS}$  for small angles and a decrease for large angles. For increasing energy, the less relative importance of

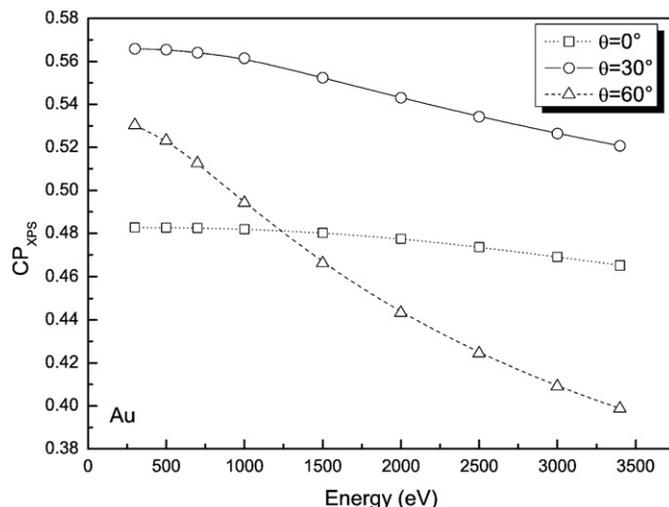


Fig. 3. Energy distribution of the  $CP_{XPS}(E, \theta)$  for Au and for angle of 0, 30 and 60°.

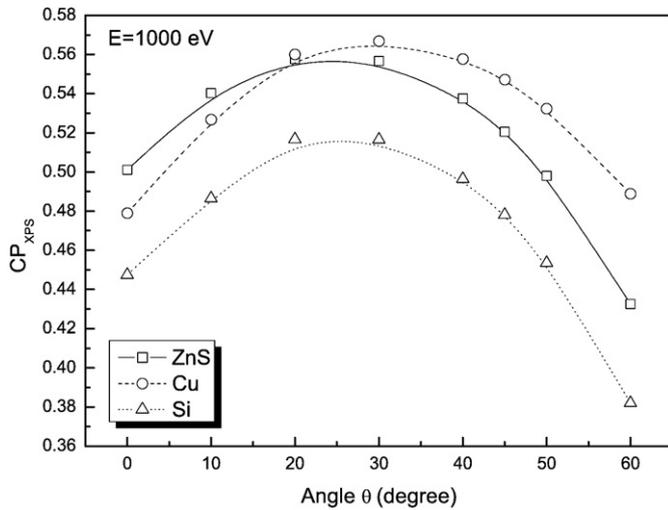


Fig. 4. Angular distribution of the  $CP_{XPS}(E, \theta)$  for Si, Cu and ZnS and for an energy of 1000 eV.

extrinsic surface excitation (point 3) and the increased relative contribution of the intrinsic excitation (point 4) imply a more pronounced decrease of  $CP_{XPS}$  for large angles in agreement with Figs. 1 and 2.

Fig. 3 shows  $CP_{XPS}$  as a function of the energy for three angles ( $\theta = 0^\circ, 30^\circ, 60^\circ$ ). For angles smaller than  $\sim 30^\circ$ ,  $CP_{XPS}$  is to a good approximation independent of the photoelectron energy. This is due to the compensation between a decrease in  $K_{surf}$  (point 3) and an increase in  $K_{intr,bulk}^{XPS}$  (point 4) with increasing energy. For  $\theta = 60^\circ$ , the variation with energy is large. This is because for  $\theta = 60^\circ$ ,  $K_{surf}$  is larger compared to  $K_{intr,bulk}^{XPS}$  and its decrease with energy will dominate the total behaviour.

Fig. 4 shows  $CP_{XPS}$  for  $E = 1000$  eV as a function of  $\theta$  for materials with very different dielectric properties: Cu (metal), Si (semiconductor), ZnS (insulator). The behaviour is quite similar for all materials and even the absolute values vary only by about  $\pm 10\%$ . The largest variations with energy is seen for  $\theta = 60^\circ$ . If we restrict the energy range to  $< 1500$  eV (which is the range applied in  $AlK\alpha$  and  $MgK\alpha$  excited XPS), the variation in  $CP_{XPS}$  is less than about  $\pm 10\%$  for all considered materials and geometries.

The results show that the correction in measured XPS peak intensities caused by the surface and the core hole is significant since the correction parameter is in the range  $0.45 \leq CP_{XPS} \leq 0.6$  which corresponds to a reduction in peak intensities of  $\sim 35\text{--}45\%$  (with  $\exp[-CP_{XPS}]$  as reduction factor). This is in good agreement with experimental observations [6,7]. However when XPS quantification is done by measuring relative intensities (which is the usual practice), the effects of the surface and of the core hole amounts to less than  $\pm 10\%$ . The present conclusions are also confirmed by various experiments. In Ref. [24], Biswas et al. actually find that, for Al 2s and Al 2p, the relative contribution of surface to bulk plasmon peaks is increasing with emission angle while the sum of the contributions is about constant. In Ref. [26], Suzuki et al. have determined the IMFPs for a Langmuir–Blodgett structure at a widely different emission angle from XPS peak shape analysis [27] and have obtained constant values, independent of the angle (for angles up to  $73^\circ$ ). In Ref. [28], Chen found that results from models neglecting the core hole effect deviate from experimental data. In Refs [8,9] the ratio of experimental peak intensities from a large set of peaks and materials was compared to theory and it was found that when the background correction was done with the Shirley and straight line methods, the deviation was considerably larger ( $\sim 35\%$  and  $25\%$  RMS

respectively) than when it was done with the Tougaard method ( $\sim 11\%$  RMS). In contrast to the first two, the latter includes the intrinsic core hole effects. For those peak intensity ratios where the uncertainty in theory is smallest, the difference is even more pronounced [8]. All three methods neglect the effect of the surface which is likely to be part of the reason for the remaining  $\sim 11\%$  deviation.

The present results show that the  $CP_{XPS}$  varies by less than  $\pm 10\%$ . If the peak intensities determined by the Shirley and straight line methods completely exclude the intrinsic electrons one would therefore expect the peak intensity ratios to deviate from theory by the same amount.

In practice the observed deviations for the Shirley and straight line methods can be considerably larger. The reason for this is probably that peak intensities determined with the Shirley and straight line methods include a smaller or larger part of the intrinsic electrons. The fraction of the intrinsic excitations which is included will then vary from peak to peak and from element to element when using the Shirley and straight line methods.

#### 4. Conclusion

The combined effect of the surface and core hole gives rise to a reduction in the XPS peak intensity in the range  $35\text{--}45\%$ . This total attenuation of the elastic peak intensity is calculated from the expression  $\exp[-CP_{XPS}]$  that must be used when the XPS intensities are determined by methods that exclude the effects of the core hole and the surface. However the variation is only  $\pm 10\%$  as a function of the angle, the energy or the medium (except for grazing angles and for large energies). This is due to a compensation between the surface effect and the core hole effect which vary in opposite directions. This implies that if a relative XPS analysis is done using peak intensities that exclude the intrinsic electrons, the error caused by neglecting the surface and of the core hole effects amounts to  $\pm 10\%$ . In practice, it is however difficult to determine the peak intensity without contributions from intrinsic effects because their energy range overlaps.

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