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### G2 X-Ray Photoelectron Spectroscopy (XPS) Analysis of Etched Dental Crown Metal Surfaces Demonstrates Prior Immersion in Hydrochloric Acid

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After attending this presentation, attendees will be aware of a technique that will allow for quantitative elemental analysis of material surfaces with the ability to determine oxidation states and infer molecular structure. This technique has been used in this study to determine that the specific corrosive in which a dental crown was immersed was hydrochloric acid. It may have uses in other forensic situations in which quantitative surface analysis becomes important.

This presentation will impact the forensic science community by illustrating the use of a technique that potentially extends the analytical repertoire available to forensic odontologists and other practitioners.

Four distinctive dental Porcelain Fused to Metal (PFM) crown artifacts were received in connection with a forensic matter. It was suspected that these crowns had been immersed in hydrochloric acid for seven days. Previous analysis using scanning electron microscopy and Electron Dispersive Spectroscopy (EDS) had demonstrated that the items had been immersed in a corrosive substance but could not determine specifically what the corrosive had been.<sup>1</sup>

XPS is a surface analysis technique that permits both quantitative estimation of the elements present in a sample and determination of their oxidation state. Depth of information is only from the outer five to ten nanometres of the surface being analyzed. It operates by using a high-energy X-ray beam to excite electrons and etch a thin layer (approximately ten nanometres) of the sample surface. The number and kinetic energy of these electrons is measured and the machine produces a graph specific for the chemical and electronic state of each of the elements present. This study hypothesized that it would be capable of determining the specific chemical identity of the corrosive in this matter; specifically, finding evidence of Cl<sup>-</sup> ions would be sufficient to permit this conclusion. No prior preparation of the surface was required.

One of the crowns was selected and an area of the crown was exposed to the X-ray beam in a hard vacuum. The kinetic energies of the resulting electrons were analyzed and charted by the machine.

This study was able to determine the elemental composition of the crown at the scanned location. It confirmed the makeup of the crown as first determined by EDS. Crucially, it also showed the presence of chlorine.

To test the hypothesis that chlorine would be found only in the surface (indicating immersion in HCl), an *in situ* argon ion gun was used to etch 20 nanometres of the surface from the sample and then the surface was re-analyzed. The comparison between the two scans confirmed the presence of the two peaks characteristic of chlorine, demonstrating its presence in the surface layer, probably as the chloride of nickel, molybdenum, or both. The post-etch analysis showed a greatly reduced level of chlorine, confirming the hypothesis that the chlorine was confined to the surface level only.

The presence of chlorine in the surface layer of the metal component of the dental crown, and the reduction in its level as the test moved more deeply into the metal, confirmed the hypothesis that the corrosive substance in which this crown had been immersed was Hydrochloric Acid (HCl).

This presentation provides information on the use of XPS to determine the surface composition of a dental crown believed to have been immersed in hydrochloric acid, and demonstrates that the technique can be used to demonstrate this fact. This analytical technique may be of relevance to forensic analysts who are required to make similar determinations.

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#### Forensic Odontology, Analytical Technique, XPS