



## Ebatco Nano

A Bimonthly Newsletter

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### **Nano Brief**

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Ebatco Academy is returning with a new course titled Contamination, Impurity, and Unknown Material Identification.

This course will be held on September 19<sup>th</sup>-20<sup>th</sup> and it will cover how to identify unknown materials, contaminations, and impurities using FTIR, Raman, SEM/EDS, and XRD techniques. Seats are filling up, so call or email soon to secure your seat and also take advantage of the 10% discount by registering by August 20<sup>th</sup>. See the attached brochure for more information.

Ebatco will be exhibiting at the following upcoming events:

- September 29<sup>th</sup> – October 2<sup>nd</sup>, Printing for Fabrication 2019, Parc 55, San Francisco, CA, representing Kyowa Interface Science Co., Ltd.
- September 29<sup>th</sup> – October 3<sup>rd</sup>, MS&T19, Booth # 530, Oregon Convention Center, Portland, OR
- December 1<sup>st</sup> – 6<sup>th</sup>, Materials Research Society Fall Meeting & Exhibit, Hynes Convention Center, Boston, MA

Please stop by our booth to discuss the incredible world of surface sciences, nanotechnologies, nanomaterials, and nano/micro scale material and device characterization with our staff scientists. We hope to see you there!

### **Ebatco**

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As we continue to grow our business we have hired on new talents to expand our expertise and testing lab service offerings. Please join us in welcoming the newest addition to the Ebatco team: Ms. Yesenia Vega.

Ms. Yesenia Vega graduated in the Fall of 2018 from the University of Minnesota-Twin Cities with a Bachelor of Science degree in Chemistry. During her undergraduate education, she was a Research Assistant working in the area of computational chemistry. Her

project focused on using DFT to study the catalytic cycle of the oxidative dehydrogenation of propane using Zr-based metal organic frameworks. She also participated in an REU program funded by the NSF in which she synthesized and characterized polynuclear Fe complexes for sensing applications. She is enthusiastic about applying her research skills and chemistry background onto lab services of chemical analysis and materials characterization.

## Case Study

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### **Quantitative Composition Determination of Powder Mixtures Using XRD**

Material composition is an essential piece of information for engineering, quality assurance, trace element analysis, process controls, and research and development. In ceramics, alloys, steels, geology, and many other fields knowing the composition and crystallographic phase of a material is vital to accurately predicting its performance. With x-ray diffraction (XRD), it is possible to determine the type of material and weight fraction of multiple components in the same sample. XRD has the advantage of being non-destructive and does not require large sample volumes. This method works best for any polycrystalline sample with randomly oriented grains that are less than 10  $\mu\text{m}$  in size, which includes many metals, alloys, geological samples, powders, ceramics, and cements.

There are three stages to measuring composition with XRD: acquiring the diffraction pattern, identifying the crystallographic phases present in the sample, and refining a model to determine the amount of each phase. Materials are identified by comparing the sample's diffraction pattern to those in a pattern library containing hundreds of thousands of standards. Each crystallographic phase has a unique diffraction fingerprint, allowing for the identification of the materials present in the sample. The final step is fitting the whole powder pattern to a theoretical model. The total intensity of each phase's pattern is proportional to the amount present in the sample. A model of the phases is used to calculate a theoretical diffraction pattern which includes strain, changes in stoichiometry, texture, or sample and instrument misalignments. These variables are refined using the Rietveld method and produce composition measurements good to 5 wt % for many applications.

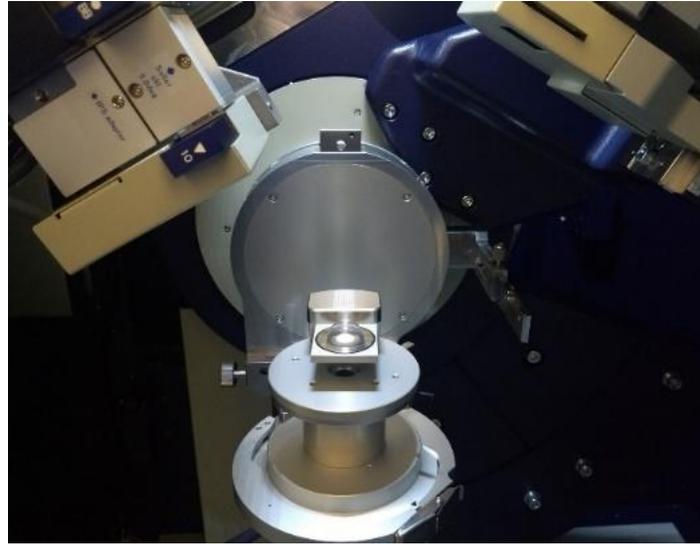


Figure 1. Powder mixture in low-background sample holder during XRD experiment.

In this demonstrative experiment, a mixture of two known, polycrystalline powders was tested for phase identification and composition measurement using a Rigaku SmartLab X-ray Diffractometer. The powder mixture was placed in a low-background sample holder and pressed to produce a smooth, flat surface, as shown in Figure 1. The Cu x-ray source, detector, and sample were all aligned. A nickel foil was used to selectively absorb the Cu  $K_{\beta}$ . The specular powder diffraction pattern was measured with the D/TeX 1D detector at  $2^{\circ}/\text{min}$  over a  $2\theta$  range of 15 to  $110^{\circ}$ .

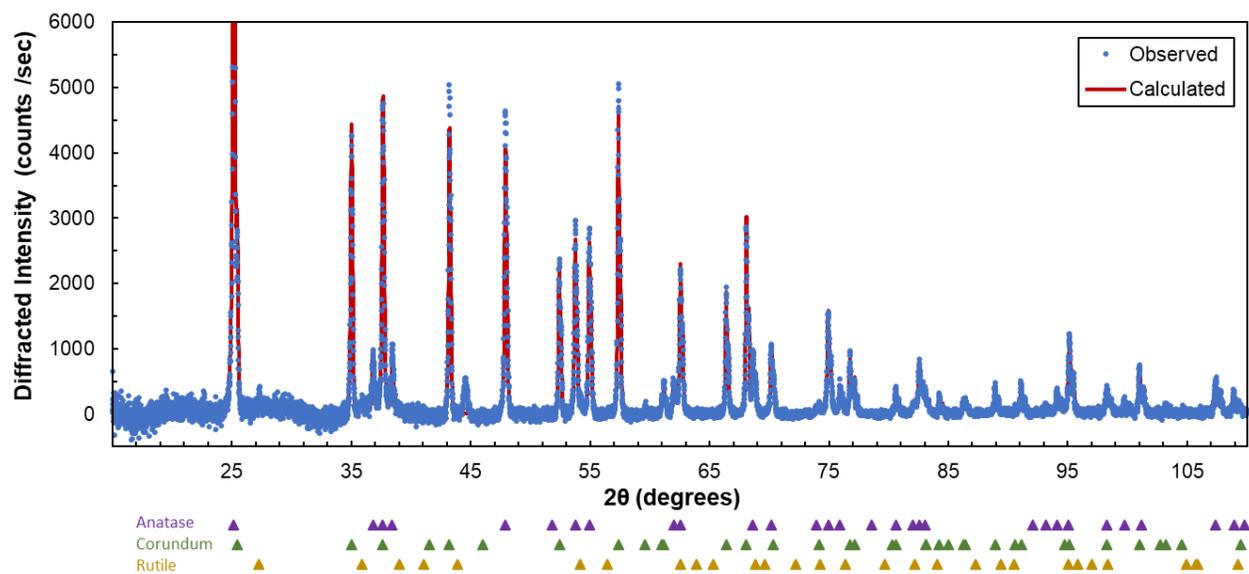


Figure 2. Experimental and calculated x-ray diffraction pattern from the powder mixture. The ideal peak positions for anatase, corundum, and rutile are marked with purple, green, and gold triangles, respectively.

The experimental diffraction pattern from the powder mixture is shown above in Figure 2. The phases were identified as corundum ( $\text{Al}_2\text{O}_3$ ) and anatase ( $\text{TiO}_2$ ) with the PDXL 2 analysis program using the Crystallography Open Database powder diffraction library. Rutile (another form of  $\text{TiO}_2$ ) may be present at or below 1.1 wt %. Reference markers below the data indicate the ideal peak positions for each phase. The whole pattern was fit to a model in PDXL 2 in which the Lorentz polarization, the  $K_{\alpha 1}$  and  $K_{\alpha 2}$  splitting, and sample self-absorption were accounted for. The weight fraction, lattice constants, thermal vibration parameters variables were fit to the experimental pattern for each phase. The resulting calculation from the best-fit model closely matches the experimental pattern ( $R_{wp} = 2.55\%$ ) and is shown as the red line in Figure 2.

As an independent check for the XRD results, the elemental composition of the sample was determined using Energy Dispersive X-ray Spectroscopy (EDS). The weight percent of each phase was calculated assuming ideal stoichiometry. As EDS does not distinguish between crystallographic phase, the weight percent of  $\text{TiO}_2$  is a sum of anatase and rutile. As seen below in Table 1, the composition measurements from XRD and EDS are in agreement.

Table 1 Powder Mixture Composition Results from XRD and EDS

Chemical Formula	Phase	XRD	EDS
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		(wt. %)	(wt %)
Al <sub>2</sub> O <sub>3</sub>	Corundum	59.8 ± 2.2	62.48 ± 3.55
TiO <sub>2</sub>	Anatase	39.1 ± 2.2	37.52 ± 4.09
	Rutile	1.1 ± 2.1	

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