Application Report XRD 29

D8 Family - SAXS Solutions

- SAXS² on Gold Nanoparticles

The D8 ADVANCE and D8 DISCOVER are true multipurpose solutions offering unmatched analytical performance with unrivaled ease-of-use for all XRD applications. The seamless integration of the outstanding EIGER2 R 500K detector perfectly fits to the DaVinci design and extends once more the analytical capabilities of the instruments.

This application note describes a Small Angle X-ray Scattering measurement of Au nanoparticles in solution (NIST SRM 8011) to extract structural information including size distribution using a D8 multipurpose diffractometer equipped with MONTEL mirror and EIGER2 detector.

For this application, the EIGER2's large field of view and exceptional dynamic range are true assets, enabling data collection in a single shot with extreme sensitivity for weak scatter at high q and high intensity linearity for low q signal near the direct beam. The panoramic Evacuated Flight Tube (EFT) and matching beamstop significantly improve the signal-to-background and signal-to-noise by virtually eliminating the parasitic air scatter while maintaining the detector’s full field of view.
The MONTEL mirror primary beam optic conditions the X-ray beam divergence in both equatorial and axial directions, avoiding smearing effects that would otherwise compromise data quality.

Further refinement of the X-ray beam size is achieved with a double pinhole collimator.

The gold nanoparticles in solution are a well characterized Standard Reference Material (SRM 8011) from the National Institute of Standards and Technology (NIST), and have a spherical character with SAXS size distribution of 9.1 nm +/- 1.8 nm. The sample was put in a glass capillary with 2 mm diameter.

A sample-to-detector distance of 330 mm was used to optimize q-coverage and q-resolution. Data was collected as a symmetric frame set of 3 frames centered about the direct beam, with a measurement time of 2500 s per frame. A second data set was collected with a capillary filled with the buffer solution, water in this case. The 2D data set (figure 1) shows the typical isotropic scattering from nanoparticles in solution.

DIFFRAC.SAXS was used to analyze the data, including integration into 1D, transmission calculation, background and buffer subtraction, and fitting.

DIFFRAC.SAXS offers a multitude of analysis options, from model free Guinier, Porod and Pair Distance Distribution Function (PDDF) methods to form factor model based analysis.

Figure 2 shows the result of a push button, model free PDDF analysis of the nanoparticle data. For spherical particles Dmax corresponds to the real space diameter while the real space radius R for a spherical particle is related to the radius of gyration $Rg=\sqrt{(3/5)}\,R$.
The PDDF result is thus in good agreement with the specified value.

To take the analysis a step further, the data was fitted to a model of monodisperse spheres. Figure 3 shows the excellent fit result corresponding to a radius of 4.35 nm (diameter of 8.7 nm), which is also in good agreement with the specified value.

The example clearly demonstrates that a multipurpose D8 instrument equipped with EIGER2 detector and DIFFRAC.SAXS software represents a complete solution for the investigation of nanomaterials.

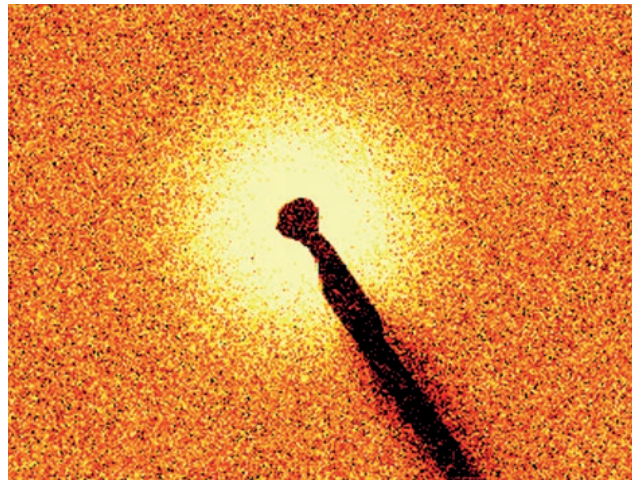

Figure 1: 2D SAXS data set of the gold nanoparticles.

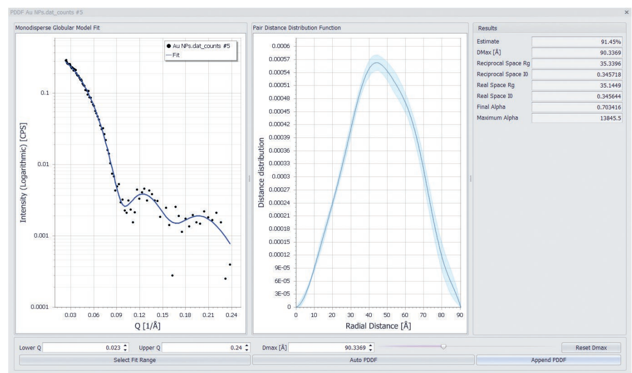

Figure 2: PDDF as determined with DIFFRAC.SAXS.
Dmax = 9.0 nm and real space Rg = 3.5 nm, which corresponds to a real space radius R of 4.5 nm.

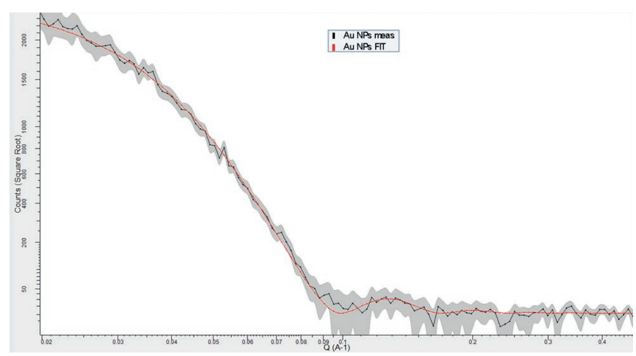

Figure 3: Integrated and background subtracted 1D data (black) fitted with the model of monodisperse spheres (red) in DIFFRAC.SAXS. In gray the error bars computed based on a Poisson distribution of the counts.

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