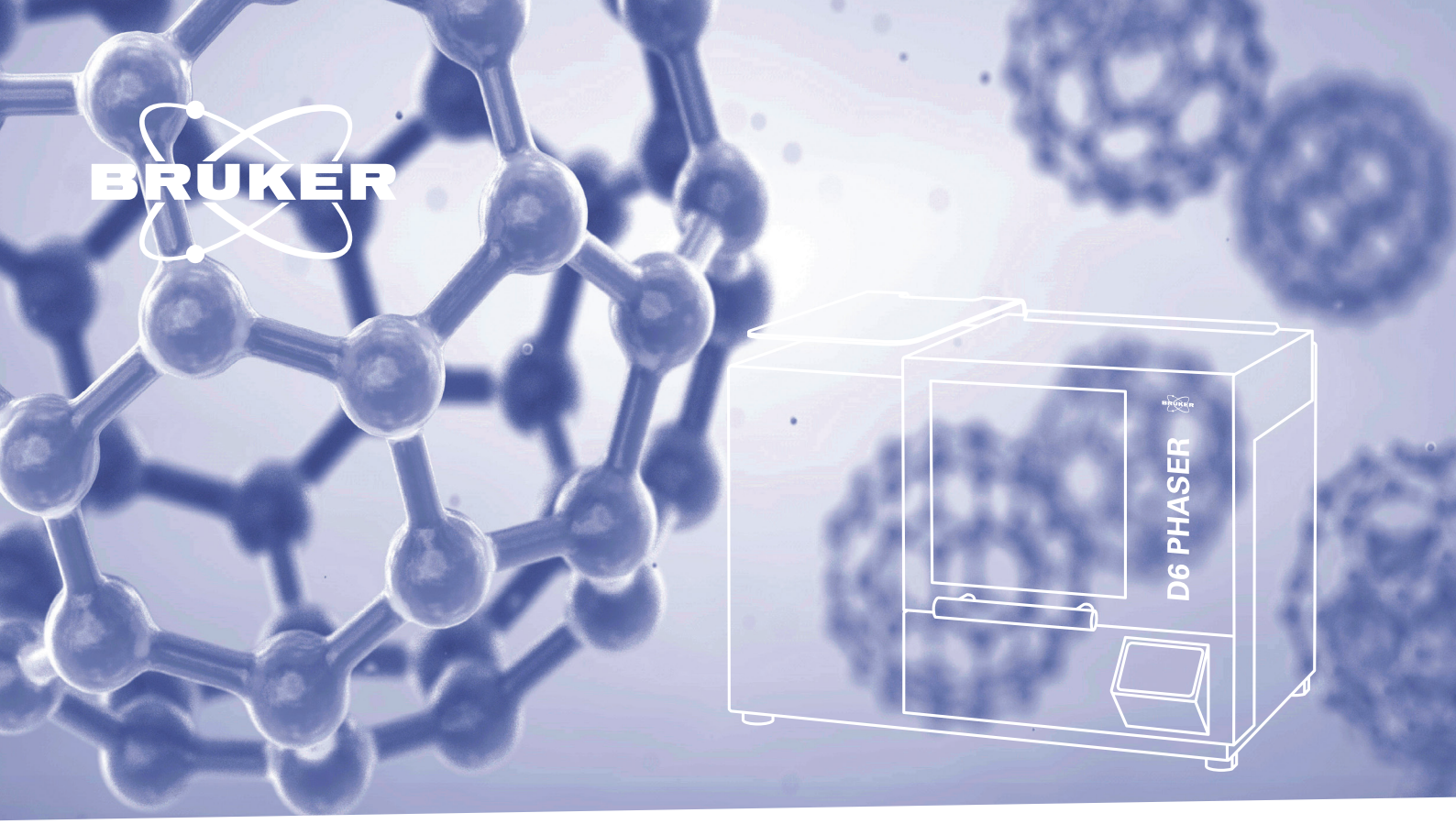

X-RAY DIFFRACTION

D6 PHASER – Benchtop XRD Total Scattering Analysis

Application Report 44

The D6 PHASER is a multipurpose benchtop diffractometer that is uniquely suited for modern materials research characterization. In this report, we present the capabilities of this system in capillary geometry for total scattering analysis.

Total scattering, also known as pair distribution function (PDF) analysis, demands high intensities and low background to observe weak diffuse scattering features in the diffraction pattern. The D6 PHASER diffractometer, equipped with a Mo source, 1200 W of power, and compact goniometer radius, combined with the excellent energy resolution of the LYNXEYE XE-T detector, offers a unique analytical solution for total scattering analysis in a benchtop instrument.

Low instrument backgrounds are not achieved through the detector electronics alone: careful removal of air scattering and other parasitic scattering features is also key to high quality PDF data.
The D6 PHASER uses the guard slit holder with beam stop and telescopic detector optics shaft together with the capillary stage to ensure the lowest possible instrument backgrounds.

Fixing and aligning the capillary to the goniometer center can also feel like a hassle. The D6 PHASER addresses this through the use of high precision magnetic mounting and alignment-free capillary holders, which take the guess work out of sample alignment. (For smaller diameter capillaries that require fine adjustment, a goniometer head is also available.)

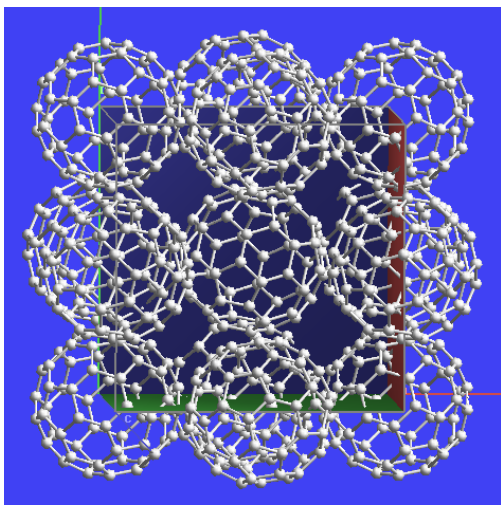

Figure 1
C_{60} molecules, decorating the lattice points of a face-centered cubic (fcc) lattice.

Figure 2
Capillary stage with alignment free base for large capillaries, primary side guard slit holder, beam stop, and secondary side telescopic detector optics shaft.

C_{60} is a perfect example to illustrate the importance of total scattering analysis. The C_{60} molecules are arranged in an fcc lattice (Figure 1), and the long-range order gives rise to sharp Bragg peaks. At room temperature, however, the molecules are spinning on their lattice points at a high rate, resulting in a large amount of diffuse scattering.

C_{60} powder was prepared in a 1mm Kapton capillary and measured on the D6 PHASER in capillary geometry equipped with a Mo source operated at 40 kV and 30 mA. The data was collected for 10 hrs. using a variable counting time (VCT) strategy, planned using the WIZARD plugin of DIFFRAC.MEASUREMENT. Even in a relatively short measurement time, the diffuse scattering features at high Q can be readily seen (figure 3, top). The bottom part of figure 3 nicely shows the well-defined intramolecular distances in C_{60} below 8 Å, characterized by sharp peaks in the PDF curve, while above 8 Å the peaks become much broader. This shows the atomic distances between different C_{60} molecules, which are less well-defined due to the rapid rotation of the molecules.

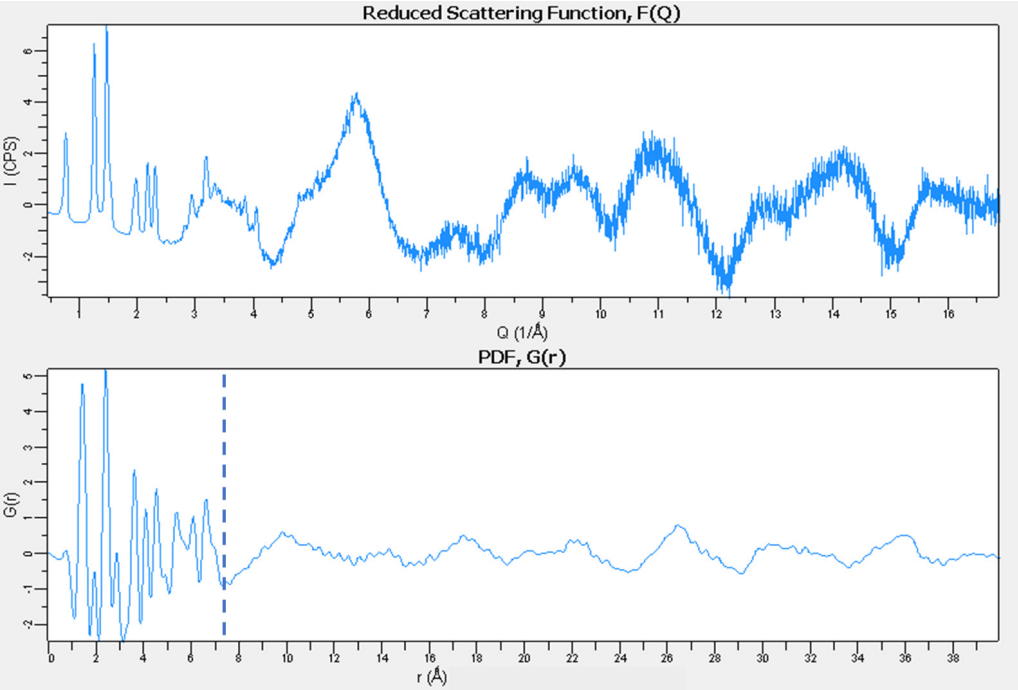

Figure 3
Reduced scattering function, F(Q) (top) and experimental PDF (bottom) collected on the D6 PHASER and converted using DIFFRAC.EVA v7.

C_{60} data were collected with a D6 PHASER 1200 W, Mo radiation, no filter, 4° primary side axial Soller collimator, primary variable divergence (constant aperture, 0.2 mm), 1 mm guard slit with beam stop, 2 mm telescopic slit, 4° secondary side axial Soller collimator. The measurement was performed as a continuous 1D scan from 3 to 145° 2Theta, 0.04° step width, exposure times between 0.6 and 18 sec per step (increasing with 2Theta), with a 4° opening of the LYNXEYE XE-T detector and in high resolution mode.

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