# Extending the Capabilities of Laboratory-Based PDF Measurements

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Pair Distribution Function (PDF) analysis based on total X-ray scattering enables direct access to interatomic distances in materials ranging from amorphous to crystalline states. It is particularly powerful for characterizing short-range order and structural disorder beyond the reach of average structure analysis methods such as Rietveld refinement.

Although total scattering experiments have traditionally relied on synchrotron radiation due to the requirement for high energy and high brilliance, recent advances in laboratory X-ray sources, detectors, and data processing have made wide-*Q*-range, high-quality PDF measurements feasible using laboratory diffractometers.

PDF measurements are most commonly performed in transmission geometry using capillary, and the methodology is well established. In contrast, applications of laboratory-based PDF measurements remain limited. In this study, we investigated three approaches to expand the applicability of laboratory PDF measurements: (1) *automated measurement of multiple capillaries*, (2) *in situ measurements*, and (3) *reflection-geometry measurements*.

(1) PDF measurements generally require long measurement times, improving measurement efficiency through automated sample exchange has been strongly required. However, implementation into the instrument has been challenging due to the requirements for wide-*Q*-range data collection and continuous capillary spinning during measurement. To address these technical demands, we developed a dedicated sample stage capable of them, enabling automated multi-sample measurements.

(2) The influence of parasitic scattering from chamber components on the quality of in situ measurements has not been sufficiently examined. We investigated strategies for suppressing parasitic scattering and evaluated their effectiveness. The methodology was applied to practical samples to verify whether reliable PDF analysis could be achieved under in situ conditions [1].

(3) Since PDF measurements are typically performed in transmission geometry, reflection geometry is required for bulk and thin film samples that are difficult to pulverize. We therefore examined the reliability of PDF measurements in reflection geometry, and we evaluated appropriate measurement and data analysis procedures.

In this session, we discuss the feasibility and expanded applicability of laboratory-based PDF analysis based on the results presented above.

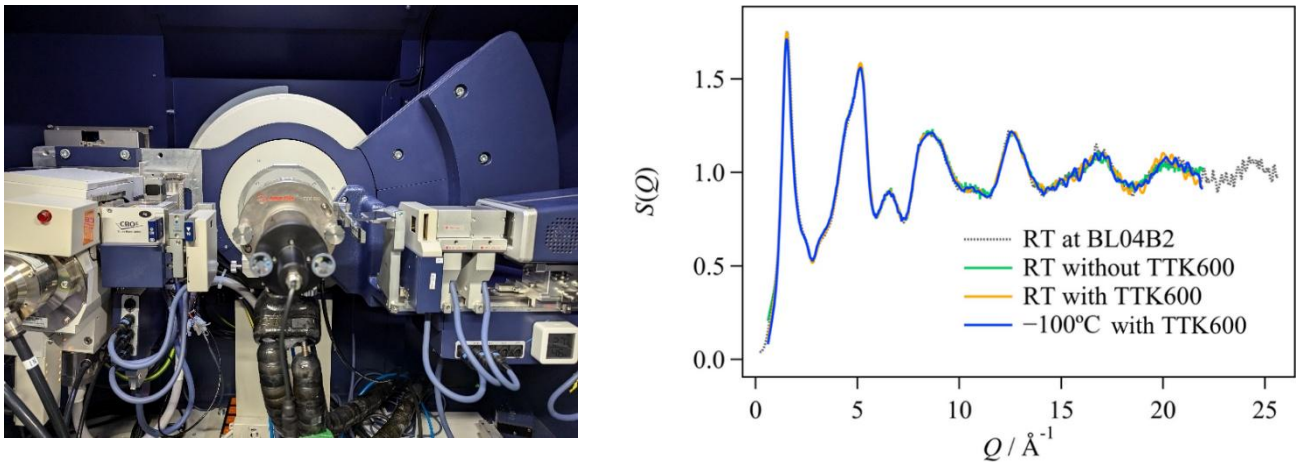



*Figure1. (Left) SmartLab with TTK600 chamber (Right) Comparison of the S(Q) values in the $SiO_2$ glass: black dotted line, room temperature (RT) at BL04B2 in SPring-8; green solid line, RT at SmartLab without the TTK600; orange solid line, RT at SmartLab with the TTK600; blue solid line, − 100 °C at SmartLab with the TTK600*

[1] Y.Shiramata, T.Konya, K.Kobayashi, Y.Ishii, S.Hiroi, H.Yamada&K.Ohara, ANAL.SCI(2025).41,777-782.