

Investigation of mesoporous MCM-41 material with ARL EQUINOX 3000 XRD

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Introduction

Mesoporous materials are a class of materials with pores ranging from 2 to 50 nm which is formed by a matrix usually made from amorphous silica, alumina or transition metal oxides. Such materials are used as catalysts or catalyst supports. However, the most commonly used type of material is mesoporous Carbon, which is widely used in energy storage devices. In contrast to Zeolites, the pores are larger, and their size and distribution can be easily adjusted.



MCM-41, together with MCM-48, is one of the most commonly used mesoporous material built from silica. The pores range from 2 to 6.5 nm and it is used as a catalyst for chemical reactions or as an adsorbent for wastewater treatment. More recent studies suggest the usage as a drug delivery system support material.

With X-ray diffraction (XRD) it is possible to directly measure the pore size and structural arrangement. Therefore, it is necessary to measure at very low ($<1^\circ 2\theta$) diffraction angles. The Thermo Scientific™ ARL™ EQUINOX 3000 is able to measure such low angles even in the standard setup without any special preparations (Figure 1).



Figure 1. ARL EQUINOX 3000 X-ray diffractometer

Instrument

The ARL EQUINOX 3000 is an entry-level full-scale laboratory instrument using a 3 kW generator powering a long fine focus X-ray tube connected to mirror optics (focal or parabolic) or a Ge (111) monochromator offering $K\alpha$ radiation with highest flux or brilliant $K\alpha_1$ radiation for highest resolution.

The ARL EQUINOX 3000 provides high flexibility and many accessories to cover applications ranging from QC/QA to In-Situ investigations. The unique curved position sensitive detector (CPS) that measures all diffraction peaks simultaneously and in real time, is especially designed for the asymmetric setup of the ARL EQUINOX instrument series. Therefore, both reflection and transmission measurements are easily obtainable without realigning the instrument.

Experimental

XRD measurements were carried out using a ARL EQUINOX 3000 XRD equipped with a Cu tube and a Ge (111) monochromator. The powdered sample was measured in reflection with an incidence angle of 0.5° for 10 minutes. Data was evaluated using MDI JADE 2010 and ICDD pdf4+ database.

Results

By choosing a low incidence angle of 0.5° it is possible to measure intensities starting from $0.74^\circ 2\theta$ (Figure 2). The measurement clearly reveals a diffraction pattern according to the structure of MCM-41 (Figure 2, green tick marks) with an excellent signal to noise ratio. Even reducing the measurement time would lead to evaluable data.

Conclusion

The ARL EQUINOX 3000 XRD in combination with the MDI JADE 2010 software suite and ICDD pdf4+ database is a suitable tool to analyze mesoporous materials down to very low diffraction angles of $0.74^\circ 2\theta$ and clearly identify the structure such as MCM-41 even after short measurement time. Such analyses require neither a specific setup nor special preparations. Therefore, the ARL EQUINOX 3000 is a tool especially suitable for QC/QA that can also satisfy more advanced analytical demands in academic institutions looking for a flexible and easy to use instrument.

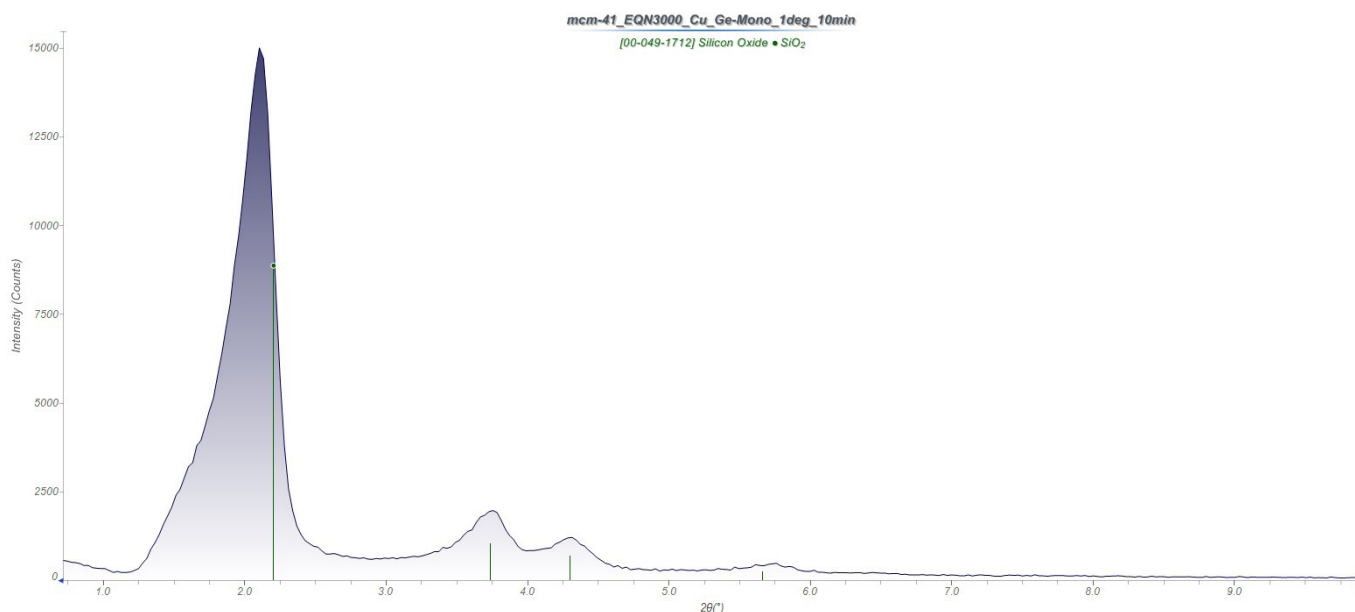


Figure 2: Diffraction pattern on MCM-41 from $0.74^\circ - 10^\circ 2\theta$ (reference positions for MCM-41 as green tick marks)

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