APPLICATION NOTE

MICROTRAC MRB

PARTICLE CHARACTERIZATION

part of VERDER scientific

The reproducibility of the pore size distribution measurement by AFSM

BELSORP instruments

The patented Advanced Free Space Measurement (AFSMTM; US Patent:6.595.036) is a highly accurate method for the determination of dead volume, which does not require to keep the level of liquid refrigerant constant. With AFSM™ it is possible to measure the free-space change continuously considering the change of liquid refrigerant level, the room temperature change during sorption measurement and the temperature change of the refrigerant due to oxygen dissolution. Therefore, the pore size distribution as well as the specific surface area can be evaluated with highest accuracy (see Application Note No. 4 AFSMTM: Improved reproducibility of the BET specific surface area by AFSM).

Figure 1 shows up to five nitrogen sorption isotherm of mesoporous alumina (BAM-PM-104), carbon black (LGC2102) and porous glass (BAM-PM-122) at 77 K (left) including an enlarged section of relevant relative pressure range (right).

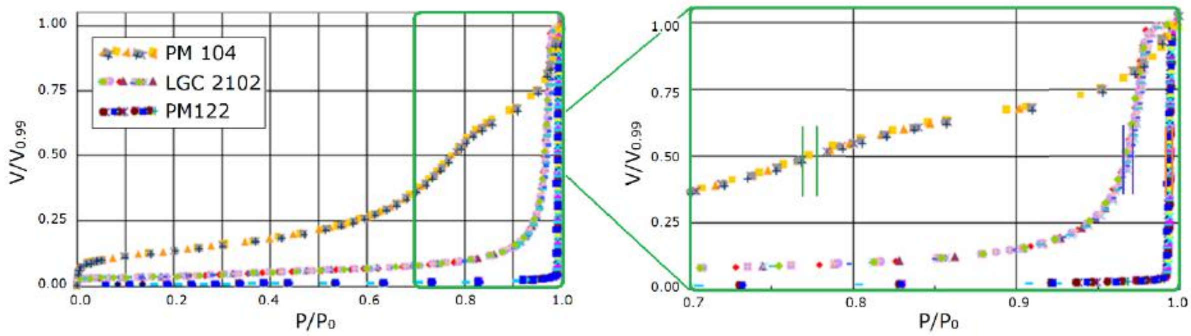

Fig. 1 Nitrogen sorption isotherm at 77 K and reproducibility of PM104, LGC2102 and PM122 (left) and enlarged section of relevant relative pressure range (right)

It is shown that the nitrogen sorption isotherms are highly reproducible with small deviations by using AFSM technique. As shown in Fig. 1 the gas uptake of each material occurs at different relative pressures, while

[1] AFSM™; US Patent:6.595.036

strongest increase of gas uptake is marked (marking in Fig. 1, right). Consequently, all three materials have different pore size distribution, respectively mean pore radius.

Fig. 2 shows the reproducibility of pore size distribution calculation of the upon mentioned materials with different mean pore radius. The calculation of pore size distribution is based on the compensation of the meniscus radius rc and the thickness t using Kelvin equation at 77 K (see Equation 1 and 2).

Kelvin equation at 77 K: $$r_c = \frac{-0.416}{\log(p/p_0)} \quad (1)$$

$$\rightarrow \quad r_p = r_c + t \quad (2)$$

Figure 2 points out that the calculation of pore size distribution is highly reproducible. The highest increase of uptake of PM 122 is at relative pressure of ~0.995, which corresponds to a pore radius of ~195 nm (diameter: 390 nm; cf. Fig. 2, orange). Although this pore diameter is near to the maximum of resolution (pore diameter of 500 nm), the measurement error is about 6%.

In case of PM 104, the smallest pores of our examples, the highest increase of nitrogen uptake is about a relative pressure of 0.76, which corresponds to a pore radius of ~4 nm. A measuring error of about 1% (see green marking, Fig. 2) is obtained. Consequently, the smaller the pores are, the smaller is the deviation/measurement error.

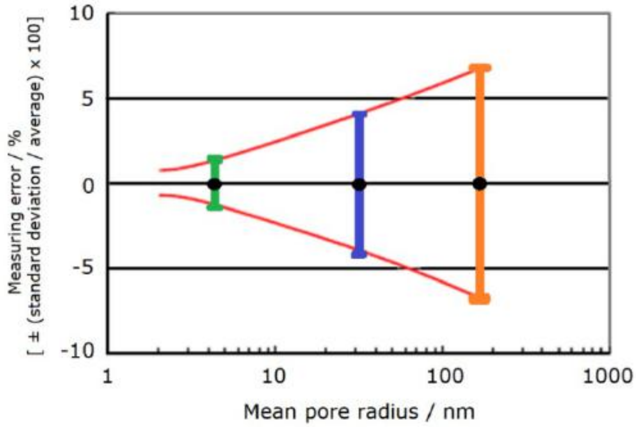

Fig. 2 Reproducibility of pore size distribution of PM 104 (green), LGC 2102 (blue) and BAM PM 122 (orange) (corresponding to Fig. 1, right)

Summarized, the AFSM technique of BELSORP series deliver isotherms with high accuracy. As a result, the calculation of pore size distribution has lower measuring errors compared to other common techniques.