ANALYZER
HIDEN ISOCHEMA NEWSLETTER
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Introducing fast and accurate binary gas sorption measurements

Hiden Isochema is delighted to announce the launch of the new **IGA-003-MC**, a gravimetric sorption analyzer for binary gas sorption analysis using the IMB method. The IGA-003-MC is a turnkey system featuring a gravimetric analyzer, coupled mass spectrometer, and an optimized gas delivery and sampling system.

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hidenisochema.com/BinarySorption

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The IMB method is unique to Hiden Isochema instruments. It enables single component and binary data to be determined in one experiment and requires only a few grams of sample.

The **IGA-003-MC** allows precise control of pressure, composition and temperature conditions and operation is fully automated, including integrated mass flow and MS calibration routines.

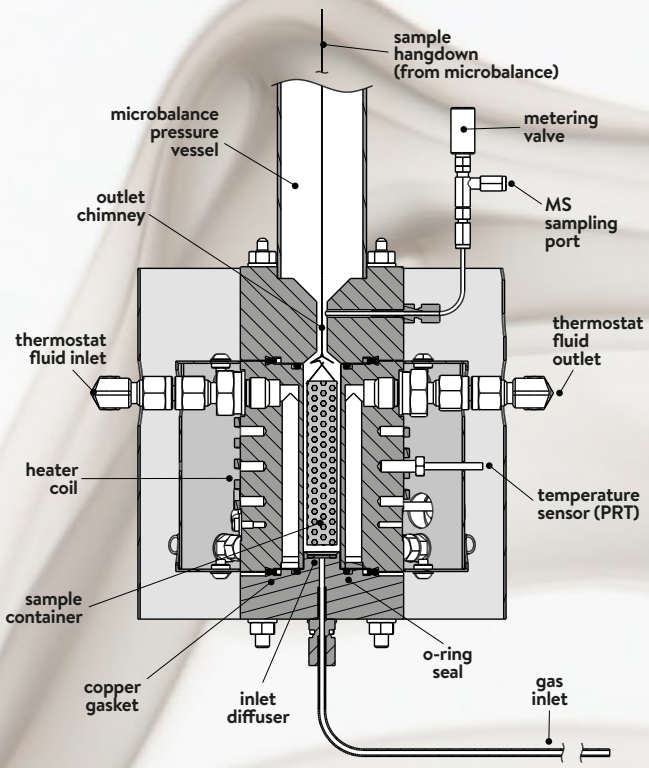


Figure 1: Specially designed IMB method reactor for the Hiden Isochema IGA-003-MC, with key components labeled (MS, mass spectrometer; PRT, platinum resistance thermometer).

IGA-003-MC

BINARY GAS SORPTION ANALYZER

Figure 2: Kinetic data measured for a binary N_2/CO_2 adsorption isotherm using the IMB method, showing the inlet and outlet mole fractions of each component.

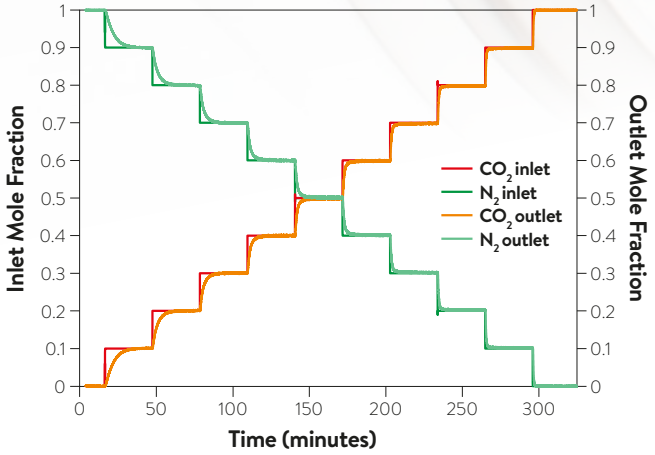
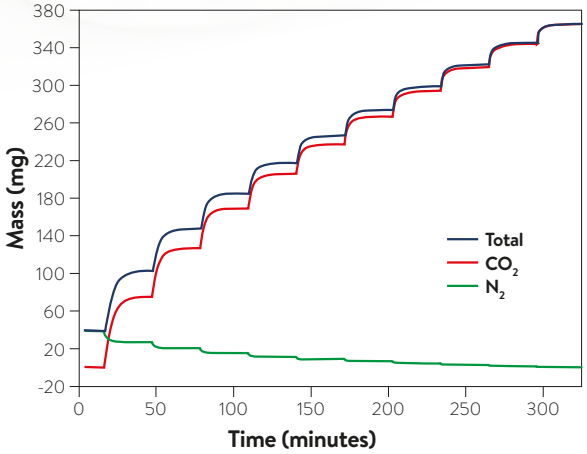


Figure 3: Kinetic data measured for a binary N_2/CO_2 adsorption isotherm using the IMB method, showing the total adsorbed mass and the mass of each component.



Binary Gas Sorption

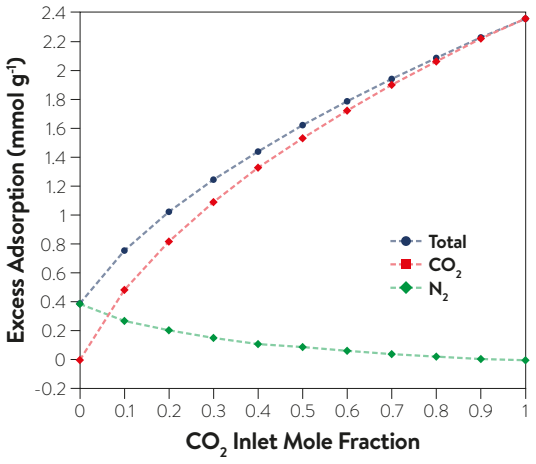
Measuring multicomponent gas sorption equilibria is essential to characterize and assess adsorbents for applications such as N_2 and O_2 production from air, natural gas upgrading, and H_2 purification. Both partial adsorbed quantities and selectivities, as a function of gas molar fraction, are required to accurately model such separation and purification processes. Traditional multicomponent methods are often time consuming and require large samples, but fast and accurate measurements, on only a few grams of material, are now possible using Hiden Isochema' s newly developed Integral Mass Balance (IMB) method.

The IMB Method

A controlled mixed gas flow is combined with in situ gravimetric measurement and outlet gas composition analysis using a mass spectrometer (MS). The gas mixture flows over a sample, suspended from the IGA microbalance, in a dedicated reactor with optimized flow path and MS sampling port (see Figure 1). Inlet flow rate and outlet gas composition analysis are combined with in-situ gravimetric data to calculate the partial adsorbed quantities, as a function of gas molar fraction. Total adsorbed quantities, meanwhile, are determined directly from the measured weight change. Full time-dependent data is recorded and analyzed to ensure steady state conditions are achieved at each step of the measurement. Both partial adsorbed quantities and selectivities can be rapidly and accurately measured at different temperatures, pressures and compositions. The IMB method is unique to Hiden Isochema's **IGA-003-MC** binary gas sorption analyzer.

For further information please contact our Sales and Application team at info@hidenisochema.com

Figure 4: Binary N_2/CO_2 adsorption isotherms for Norit RB1 activated carbon measured using the IMB method.



Example data from binary N_2 and O_2 adsorption on a porous carbon are shown in Figures 2-4. Kinetic data are presented in figures 2 and 3, and the final equilibrium binary gas adsorption isotherms are shown in Figure 4. The rapid measurement times –only a few hours for a full ten point binary gas adsorption isotherm – can be seen in Figure 4.

ACS Energy & Fuels award for excellence in publication awarded to Professor Mark Thomas

Congratulations to Professor Mark Thomas and his team from Newcastle University, UK, on being awarded the 2019 American Chemical Society (ACS) Energy & Fuels Joint Award for Excellence in Publication. Professor Thomas is a long term collaborator and customer of Hiden Isochema, and has published a large number of research articles reporting results of gas and vapor adsorption measurements on porous carbons, MOFs and natural materials.

The award is jointly made by the ACS Energy & Fuels Technical Division and the ACS journal Energy & Fuels, to recognize a notable article that has been published in Energy & Fuels during the previous 5 years. The 2019 award is for the article 'High-Pressure Methane Adsorption and Characterization of Pores in Posidonia Shales and Isolated Kerogens' [1] and follows Professor Thomas' earlier publication reporting methane adsorption in alum shale.[2]

This article reports a thorough study of the physical chemistry and petrology required to estimate the Gas-

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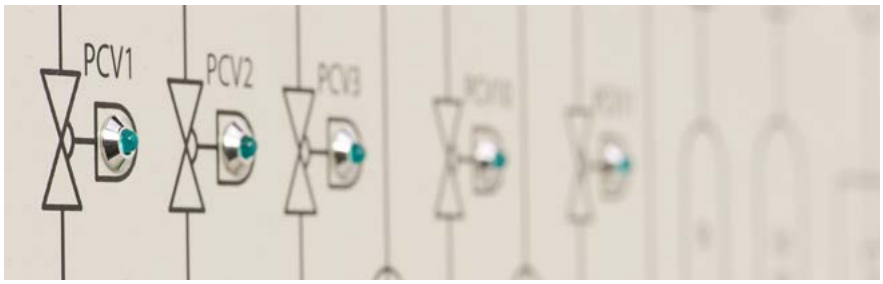

"It's fantastic to see Mark being recognised for his work in the area of shale characterization and we look forward to many more years of successful collaboration"

Dr. Mike Benham
Managing Director, Hiden Isochema

in-Place and the rate at which that gas can be extracted from a wide-spread, economically important shale formation that lies under Western Europe. An IGA-001 was used to measure low temperature N_2 and CO_2 adsorption isotherms to 1 bar, while CH_4 adsorption isotherms to 140 bar at several temperatures were measured using an IMI-HTP. Isotherms were measured on several shales and associated extracted kerogen samples, and analysed to obtain further thermodynamic values including isosteric enthalpies of adsorption.

In recognition of the award, Professor Thomas recently delivered a lecture at the Energy & Fuels Lectureship Symposium at the ACS Fall 2019 National Meeting & Exposition in San Diego.

Professor Thomas's current investigations include competitive adsorption in relation to the capture of carbon dioxide from flue gases on porous materials, and applications of porous materials and nanoparticles in electrodes.

References

[1] High-Pressure Methane Adsorption and Characterization of Pores in Posidonia Shales and Isolated Kerogens, T. F. T. Rexer, E. J. Mathia, A. C. Aplin and K. M. Thomas, Energy & Fuels, 2014, 28, 2886-2901.

[2] Methane Adsorption on Shale under Simulated Geological Temperature and Pressure Conditions, T. F. T. Rexer, M. J. Benham, A. C. Aplin and K. M. Thomas, Energy & Fuels, 2013, 27, 3099-3109.

Discovering more. seeing more.

DATE	CONFERENCE	LOCATION
25-29 Aug	ACS Fall Meeting	San Diego, USA
29-30 Aug	CFM2019	Brest, France
8-11 Sept	ILSEPT 2019	Sitges, Spain
9-10 Sept	39th Cement and Concrete Science Conference	Bath, UK
9-14 Sept	i-gass Gas Adsorption Summer School	Spetses, Greece
27-30 Oct	EuroMOF	Paris, France
10-15 Nov	AIChE Annual Meeting	Orlando, USA

PUBLICATION ROUND-UP

Gas Storage by Shales

Effects of energetic heterogeneity on gas adsorption and gas storage in geologic shale systems

J. Li, K. Wu, Z. Chen, W. Wang, B. Yang, K. Wang, J. Luo and R. Yu

Applied Energy 251 (2019) 113368. DOI: 10.1016/j.apenergy.2019.113368

High pressure methane adsorption isotherms, measured with an **IMI-HTP**, for two shale samples from the Sichuan Basin (China) are reported by scientists at China University of Petroleum and the University of Calgary. The isotherms, measured at three temperatures to 200 bar with the IMI-HTP's large capacity sample reactor, were fitted to a multi-site adsorption model and adsorbed quantities also extrapolated to higher pressures. The multi-site model was used to predict the adsorption energy distribution for each sample, which may be used as a description of the heterogeneity of shales, which is key to understanding and predicting their gas storage and production mechanism.

Sorption Kinetics/Zeolites

Nanoporous ZSM 5 Crystals Coated with Silicalite 1 for Enhanced *p*-xylene Separation

M. Miyamoto, S. Ono, Y. Oumi, S. Uemiya, S. Van der Perre, T. Virdis, G. V. Baron and J. F. M. Denayer

ACS Appl. Nano Mater. 2019, 2, 5, 2642-2650. DOI:10.1021/acsanm.9b00037

Improvement to ZSM-5's catalytic selectivity for *p*-xylene over other xylene isomers, due to silicalite-1 coating was studied using a range of techniques. An **IGA-002** was used to measure *p*-xylene isotherms with pressure points from $4x10^{-3}$ mbar, and separately, single step kinetic uptake curves for pure *p*-xylene and *m*-xylene vapor over pure ZSM-5 and silicalite-coated ZSM-5. For the kinetic study, vapor pressure was controlled at 10 mbar and temperature at 323 K throughout the analysis, which lasted up to 5 days. The coated sample exhibited significantly slower *m*-xylene sorption kinetics and faster *p*-xylene sorption kinetics, and also a stepped *p*-xylene isotherm not observed for the pure zeolite.

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