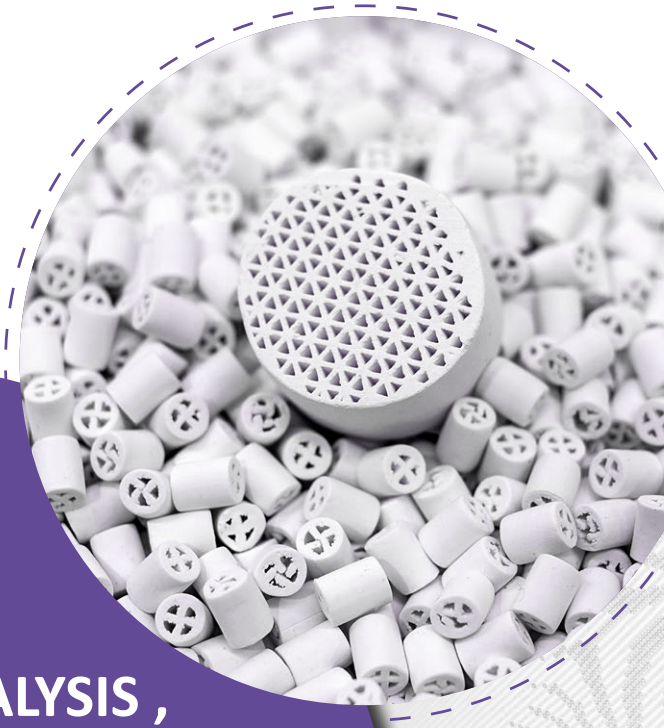
KEP Technologies

CATALYSTS
CHARACTERIZATION



THERMAL ANALYSIS , CALORIMETRY & GAS SORPTION SOLUTIONS

• SORPTION ISOTHERMS •
• SORPTION KINETICS •
• SELECTIVITY •
• HEAT OF SORPTION •
• COKE CONTENT •

setaram
KEP TECHNOLOGIES

YOUR CHALLENGES

The improvement of catalytic materials is a prime concern for developers and manufacturers. In a competitive and rapidly developing environment, you need technically reliable and economically profitable characterization solutions.

If you need to select the best catalyst for an application, or to define the applications of a new adsorbent, we have solutions to help! They can handle gas-solid, vapor-solid, or liquid-solid interactions to characterize materials' **sorption, desorption, selectivity data, coke content.**

COMMON CATALYSTS - STUDIES & SOLUTIONS

This brochure presents some of our solutions in this field and we encourage you to contact us for more information.

Manometry characterizes how much gas or vapor a material can capture. With these methods, you can measure and compare sorption isotherms.

Sorption Isotherms

If you're interested in separation processes, the best materials adsorb more of the desired gas, faster. You can compare sorption isotherms and kinetics to characterize selectivity against various gases.

Selectivity

If a solid substrate adsorbs a lot of gas but the sorption takes too long, it isn't applicable. With TGA and manometry, you can measure how fast the sorption takes place between the gas and the solid.

Sorption Kinetics

To select the best catalysts, you need to understand their surface chemistry. You can measure heat of sorption by coupling calorimetry with manometry. A large amount of heat means a strong interaction between the catalyst surface and the test molecule, and vice versa

Heat of Sorption

You can ensure your catalysts work for longer with TGA. A typical application of TGA is the quantification of the coke deposits on a catalyst's surface during its operation.

Coke Content

CUSTOMER TESTIMONIAL

"This instrument also allows us to inform our customers for catalytic applications where we will look at the acid-base of our products, their ability to absorb a more or less acid-base gas. These are things that we do with the CALVET PRO instrument from Setaram."

Livia Marra, R&D Project Manager
Baikowski

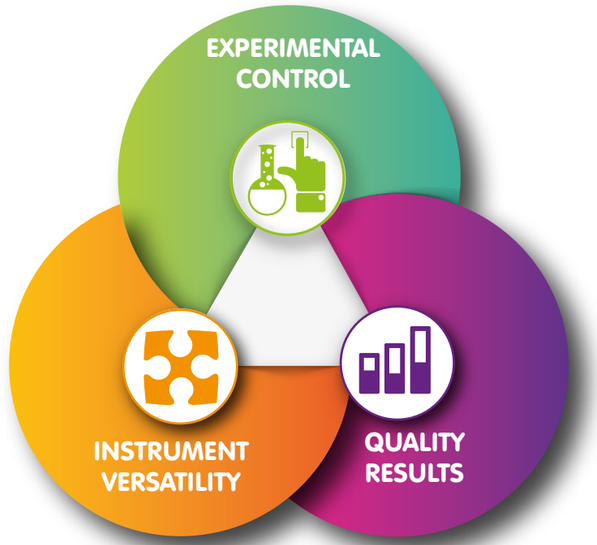
THE KEP TECHNOLOGIES ADVANTAGE

KEP Technologies is addressing it's offerings to the catalysts market by making available the widest and most versatile choice of solutions. Now you can consult with one company, KEP Technologies, to address your challenges across the broadest number of battery studies on the market.

Each solution embodies our "Reimagine Material Characterization" value proposition by delivering the three core customer benefits of **Experimental Control, Instrument Versatility and Quality Results.**

We believe solutions that provide these benefits will deliver the highest value to our customers.

In addition to our core customer benefits, we are able to provide **customized solutions** by harnessing the engineering and project management of our highly skilled organization.



CUSTOMIZED SOLUTIONS

Modular design allows for upgraded and tailored functionality
Access to all previous non-proprietary custom requests
Open access to our engineering development team

INSTRUMENT

GASPRO

VARIETY OF MODES OF OPERATION
ability to combine PCT, kinetics and cycle-life modes to 200 bar to determine the quantity and rate of sample/gas interaction and its ageing characteristics all in one instrument and operation

PRECISION MEASUREMENT OF SMALL SAMPLES
using the patented microdoser option

WIDE TEMPERATURE RANGE ENABLING A VARIETY OF APPLICATIONS
from sub-ambient operations up to 500+ °C with a customized solution

HIGH ACCURACY
to reduce cumulative error accross multiple measurement points

EXTERNAL CALORIMETER COUPLING CAPABILITY
to increase your research options

SPECIFICATIONS

Temperature range (°C)	-260 °C to 500 °C with different sample holder options Higher temperatures on request
Calibrated reservoirs	from ~12 ml to ~1.2 l
Sorption gas (Test gas)	Carbon Dioxide, Methane, Nitrogen, Argon, Hydrogen, Deuterium, Helium, Neon, Ammonia, n-alcanes from C2 to C6, more on request.
Operating pressure range	From vacuum to 200 bar
Sample pressure measurement	1 transducer for vacuum to 200 bar Accuracy < 0.025% full scale 1 transducer for vacuum to 15 bar Accuracy < 0.12% of the reading
Maximum sensitivity	3 µmole of gas (with the MicroDoser attachment)

For more information on specifications please consult the product information and brochures available on our website : www.setaramsolutions.com

APPLICATION

Methane adsorption into coal measured by the GASPRO

INTRODUCTION

Coalbed methane is an important source of energy in many countries. In contrast to a conventional gas reservoir, methane is stored by adsorption in to pores of the coal. In underground coal mining, it presents serious safety risks and is one of the main cause of coal mine accidents. Thus characterization of methane uptake in coal is essential to the development of new technologies to harness energy while mitigating environmental and underground mining risks. This application note shows the results of methane adsorption and desorption measurements on a coal sample at room temperature and up to 150 bar.

EXPERIMENT

CH_4 adsorption into a bituminous coal sample was measured at 25°C using a GASPRO Sievert's apparatus which was developed to study sorption of a variety of gases from vacuum up to 200 bar and from liquid He to 500°C.
Gas density temperature correction was done automatically by measuring the apparentfree gas volume at temperature using helium. The density of the entire sample was assumed to be 1.4kg/m3.

RESULTS AND CONCLUSION

The PCT isotherms of CH_4 adsorption and desorption for Illinois bituminous coal are shown in Figure1. The methane uptake is two times lower than that of CO2 reported for the same sample in AN654. Methane physisorbs into coal, thus its uptake depends on pore volume available in coal. The measured methane uptake of 20ml gas STP/ml sample is consistent with literature values for similar coal samples (4-25ml gas STP/ml). The GASPRO is well-suited for the detailed characterization of coal used in the study.

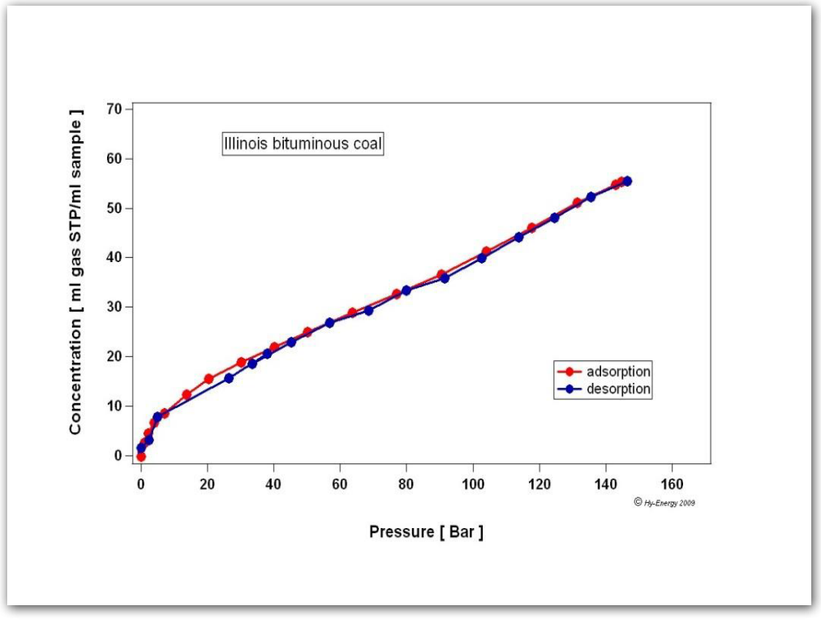


Figure 1: CH_4 sorption isotherm at 25°C for Illinois bituminous coal.

INSTRUMENT

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APPLICATION

CO_2 adsorption into coal measured by GASPRO

INTRODUCTION

In addition with the research for alternatives to reduce the use of hydrocarbon, every realistic future scenario confirms the continuous use of fossil fuel and thus the release of carbon dioxide (CO_2) in the atmosphere. Therefore high research effort is needed to find ways to efficiently store CO_2. Unminable coal bed are foreseen as potential solution for a sustainable storage. This application note shows the results of CO_2 adsorption on two coal samples using the manometric technique.

EXPERIMENT

Approx. half of a gram of the different coals in powder form (100 mesh) have been introduced in the standard sample holder (400°C/200 bar) of the GASPRO. After initial evacuation and subsequent volume calibration of the dead volume with helium, the pressure-composition temperature (PCT) isotherm of CO_2 on these coals were measured with the GASPRO.

RESULTS AND CONCLUSION

The samples show very different behaviours in term of CO_2 uptake as it can be noticed on the figure 1 at 40°C. The sub-bituminous coal shows a much higher uptake. It is also noticed that the saturation is reached for the bituminous coal. For the sub bituminous coal the CO_2 pressure was lower, but we can predict that saturation appears at the same pressure, as the beginning of an inflexion of the curve is detected. The comparison of the CO_2 uptake for the bituminous coal at different temperatures is shown on figure 2. The isotherm at lower temperature demonstrates that the saturation limit disappears in the studied pressure range.The GASPRO is well-suited for the detailed characterization of materials used in the study.

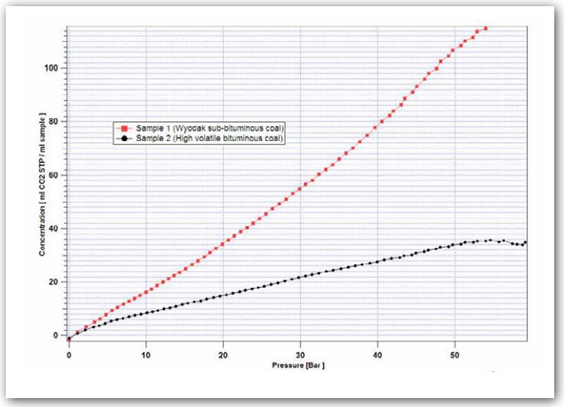


Figure 1: CO_2 sorption isotherms at 40°C for Illinois bituminous coal and Wyodak subbituminous coal.

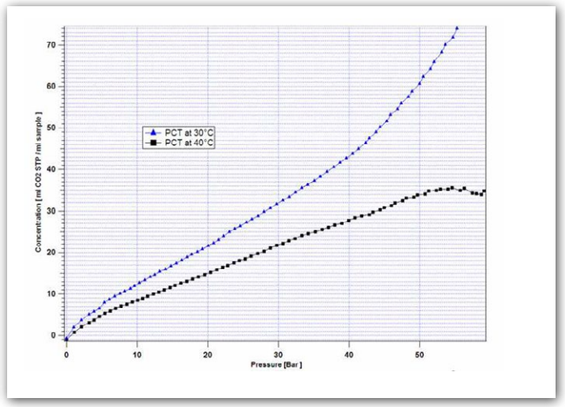


Figure 2: Comparison of the CO_2 isotherm of Illinois bituminous coal run at 30°C and 40°C

INSTRUMENT

THEMYS TGA

VARIETY OF ATMOSPHERE CONDITIONS
multiple carrier and reactive gas options

MODULAR ADAPTIONS ALLOWING
TGA only, DTA only, TG-DTA, and TMA up to 2400°C, DSC only and TG-DSC up to 1750°C all in one instrument

HIGH ACCURACY & VERSATILITY
hang-down symmetrical beam balance, specifically designed for TGA applications

EXTERNAL COUPLING CAPABILITY
designed for evolved gas analyzers (FTIR, MS, GCMS, MS-FTIR,or FTIR-GCMS)

SPECIFICATIONS

Temperature range (°C)	room temperature to 1750 or to 2400
Isothermal and temperature scanning (°C/min)	0.01 to 100
Sample volume (µl)	up to 2500 in TGA
Optional protected DTA rods for enhanced corrosion resistance, tricouple DTA rods for enhanced sensitivity, protected tricouple for combined advantages	

For more information on specifications please consult the product information and brochures available on our website : www.setaramsolutions.com

APPLICATION

CO_2 sorption on ZIF-8

INTRODUCTION

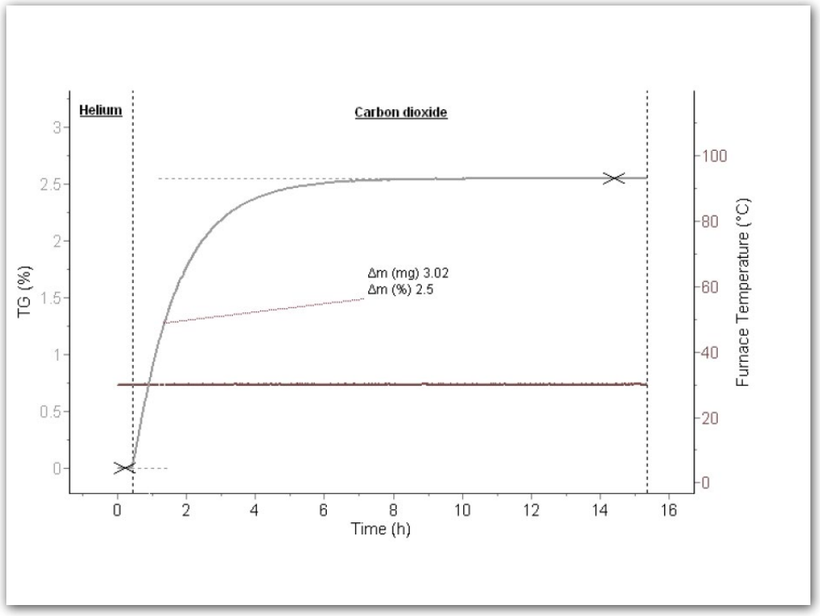
Zeolites Imidazole Frameworks (ZIF) are a class of metal organic frameworks. They can potentially be used to remove carbon dioxide from gas streams thanks to their highly porous structure. Thermogravimetric analysis can be used to determine the sorption capacity of porous materials against pure (like in the present example), or complex gas blends, together with the assessment of sorption kinetics at a given temperature.

EXPERIMENT

A 118.82 mg sample was pretreated at 100 °C under primary vacuum during 2 hours. It was then cooled down to 30 °C and its temperature stabilized under a flow of helium (20ml/min). At time zero, the signal is stabilized, but helium is flowed again during 20 more minutes, then the THEMYS gas panel switches from helium to carbon dioxide at the same flowrate.

RESULTS AND CONCLUSION

The mass variation (TG) signal was stabilized after 14 hours because of the saturation of the ZIF-8 sorption capacity. The mass uptake was determined thanks to Calisto data treatment software to be equal to 3.02 mg, i.e. 2.5%.
This example shows simple gas change possibilities. More complex gas blends can be handled / programmed with the PureGas, GasBlend, or MultiGasBlend options.



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Operating pressure range	From vacuum to 200 bar
Sample pressure measurement	1 transducer for vacuum to 200 bar Accuracy < 0.025% full scale 1 transducer for vacuum to 15 bar Accuracy < 0.12% of the reading
Maximum sensitivity	3 µmole of gas (with the MicroDoser attachment)

For more information on specifications please consult the product information and brochures available on our website : www.setaramsolutions.com

APPLICATION

Study of catalysts impact for improving the hydrogenation of an adsorbent by manometry

INTRODUCTION

The sorption capacity and kinetics of metal hydride are key parameters for their practical applications in hydrogen storage. Magnesium hydride (MgH_2) material has been receiving a great attention due to its high hydrogen storage capacity, cost effective and availability of Mg metal. Unfortunately, the slow hydrogenation kinetics is considered as the major barriers that limit this metal hydride to be utilized for fuel cell and automobile applications for example.
In this application note, the manometry technique has been used to study the impact of two catalysts on the hydrogen sorption kinetics of magnesium hydride material.

EXPERIMENT

GASPRO was used to test two samples of MgH_2 catalyzed with 5wt% of Ni and 5wt% of Nb_2O_5. Kinetics of hydrogen sorption have been studied by injecting hydrogen under 8bar at different temperatures from 200°C to 250°C.

RESULTS AND CONCLUSION

The results show that a small fraction of 5 wt.% Ni powder led to considerable improvement on the absorption kinetics of MgH_2. It is indicated by the short time required to absorb about 5.6 wt.% H_2 (within 43s). The saturation value of 5.8 wt.% H_2 was reached after 500s at 225°C and 250°C, as shown in Figure 1.
No significant difference of kinetic of sorption has been observed between the tests at 225°C and 250°C Adding fractions of 5 wt.% Nb_2O_5 to the MgH_2 powders also showed a clear acceleration of the hydrogenation process, as shown in figure 2. It can be noticed that the hydrogenation kinetics of MgH_2/5wt%Nb_2O_5 was positively affected by increasing the applied temperature from 200 °C to 250 °C.

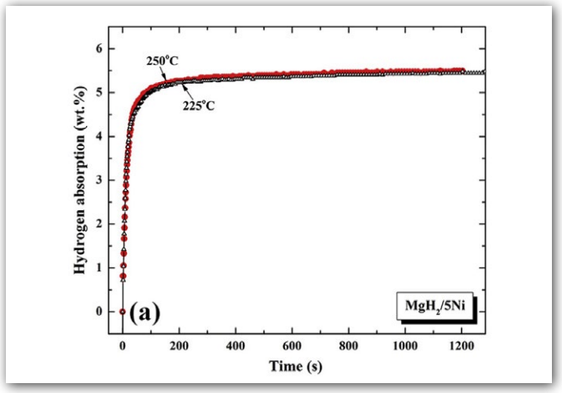


Figure 1: Effect of applied temperature on the hydrogen absorption kinetics of MgH_2 catalyzed with Ni with 5 wt.% Ni

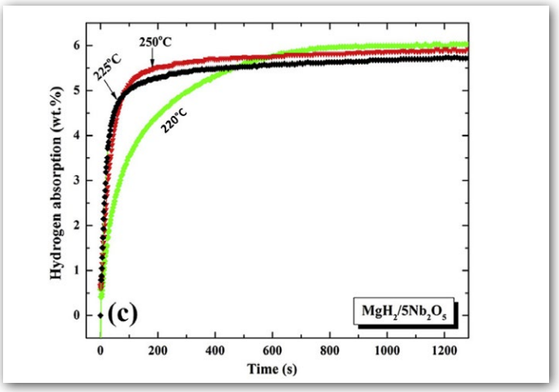


Figure 2: Effect of applied temperature and nano catalysts on the hydrogen absorption kinetics of MgH_2 catalyzed with 5wt.% Nb_2O_5

El-Eskandarany, S., Shaban, E., & Al-Shemmiri, A. (2014). Integrated Ni/Nb_2O_5 nanocatalytic agent dose for improving the hydrogenation/dehydrogenation kinetics of reacted ball milled MgH_2 powders. International Journal of Hydrogen Energy.

INSTRUMENT

THEMYS ONE

HIGH SENSITIVITY BALANCE FOR THE DETECTION OF SMALL MASS VARIATIONS
specifically designed for TGA analysis.

CONVENIENCE OF ONE FURNACE
to reach temperatures as high as 1600°C.

PLUG AND PLAY INTERCHANGEABLE RODS
to perform TGA only, TG-DSC, TG-DTA, and 3D high sensitivity/Cp measurements.

EXTERNAL COUPLING CAPABILITY
including evolved gas analyzers

SPECIFICATIONS

Temperature range (°C)	room temperature to 1600
Isothermal and temperature scanning (°C/min)	0.01 to 100
Sample volume (ml)	up to 1 in TGA
Evolved gas analyzers (FTIR, MS, GCMS, MS-FTIR, or FTIR-GCMS) for performing qualitative and quantitative gas characterization	

For more information on specifications please consult the product information and brochures available on our website : www.setaramsolutions.com

APPLICATION

Desorption of a zeolite studied by simultaneous thermal analysis techniques

INTRODUCTION

In recent years, hyphenated thermogravimetry and gas analysis techniques have experienced a wide development.
In such experimental set-ups, the gases evolved during decomposition can be transferred on-line or off-line to a gas analyzer. Usual gas analyzers are Fourrier transformed infra red spectrometers (FT-IR), gas chromatographs (GC), and mass spectrometers (MS) - probably the most employed.

EXPERIMENT

THEMYS ONE TG-DTA system was coupled to both Pfeiffer mass and Thermo FT-IR spectromers.
The analyzed sample is a proprietary formulated zeolite, whose carbon dioxide production during heat treatment needs to be carefully followed up.
The sample was heated between ambient and 1000°C at 5°C/min under a flow of nitrogen at 40mL/min. The MS was used in the multiple ion detection mode, with the following targeted molecules :

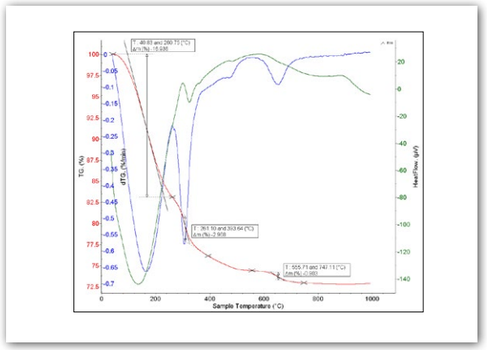


Fig 1 – Weight loss (TG), weight loss rate (DTG), and DTA signals for the zeolite desorption

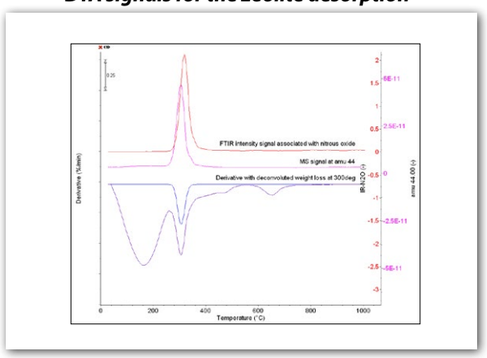


Fig 2 – Evolution of N_2O – The intensity variation of the absorption wavelength (2200cm-1) linked with nitrous oxide vibration confirms that the uma 44 detected by MS is not linked with CO_2 evolution.

RESULTS AND CONCLUSION

Figure 1 exhibits a three (TG) to four (DTG) steps mass loss of the zeolithe due to gas desorption.
As mentionned on figure 2 and figure 3, mass spectrometry signals, combined with both full FT-IR spectra and intensity variation follow-up of characteristic wavelengths, allowed to associate the second and third events respectively to nitrous oxide and ammonia evolution.
The presented work shows how multiple coupling techniques help understanding the mechanism of desorption of a zeolite, and more particularly how FTIR data could show that, what was firstly supposed to be CO_2 production, turned out to be nitrous oxide production.

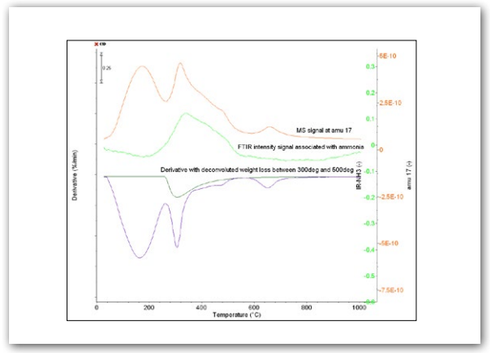


Fig 3 – Evolution of NH_3 – The intensity variation of the absorption wavelength linked with ammonia vibration indicates that the uma 17 detected by MS is firstly linked with water evolution, then to ammonia evolution (starts around 200°C).

INSTRUMENT

CALVET PRO DSC

HIGHEST HEAT MEASUREMENT ACCURACY
Calvet 3D sensor based on thermocouples with Joule effect calibration.

EXTERNAL COUPLING CAPABILITY
Designed to increase your research options, including manometry, BET, gas analyzers, humidity controllers and gas panels.

CONVENIENT INTERCHANGEABLE CRUCIBLES AND CELLS to perform even the most demanding experiments with one instrument : high pressure (500bar) and high vacuum (10-4 mbar) studies, pressure measurement and control, packed bed reactor experiments.

SPECIFICATIONS

Temperature range (°C)	Ambient to 830°C -120 to 200 °C (with cooling accessory)
Temperature accuracy (°C)	+/- 0.05*
Temperature precision (°C)	+/- 0.15*
Programmable temperature scaning rate (°C/min)	0.01 to 30
Enthalpy accuracy (%)	+/- 0.8*
Calorimetric precision (%)	+/- 0.4*
Crucible or cells volume (ml)	Up to 0.32 depending on the chosen design and material (aluminium, incoloy, graphite, alumina, platinum, etc)
Pressure (bar [psi])	400 [5,800] (measured and controlled); 500 [7,250] (resistant)

** Based on indium melting tests*

For more information on specifications please consult the product information and brochures available on our website : www.setaramsolutions.com

APPLICATION

Heat of adsorption of NH_3 on a zeolite by using a quartz tube reactor on a 3D sensor

INTRODUCTION

The investigation of gas adsorption on catalysts and more generally solid adsorbents requires a very good interaction between the reactive gas and the powder. The CALVET PRO DSC offers the main advantage to work with an open tube detection. This configuration allows the adaptation of different types of experimental crucibles, especially with the possibility of introduction of various types of gas under normal or high pressure. The quartz tube reactor is one option for the applications on catalysts, and more generally for all the adsorption investigations. It makes possible the simulation of the use of a plug-flow fixed bed reactor in heterogeneous catalysis.

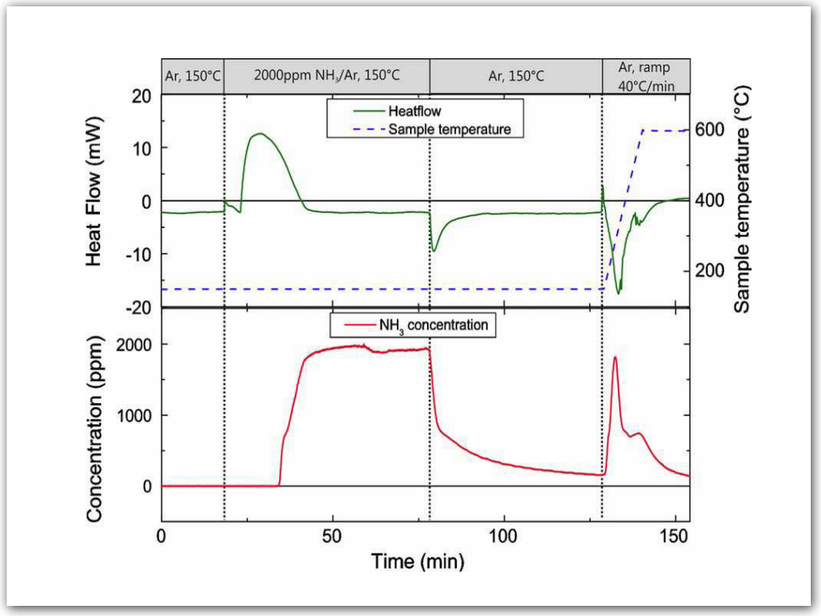
EXPERIMENT

The heat of adsorption of NH_3 on Cu-Beta zeolite is investigated at 150°C using the calorimeter connected with a FTIR analyzer. Prior to the test, the catalyst was first oxidized by using 8% O_2 at 500°C in order to ensure the removal of all ammonia from the surface.

RESULTS AND CONCLUSION

When ammonia was introduced at 150°C, an exotherm is observed corresponding to ammonia adsorption on the zeolite. The FTIR signal also gives information about the saturation of the catalyst. After the ammonia adsorption phase, the catalyst was exposed to Ar alone. An endotherm was observed due to desorption of loosely bound ammonia, with the corresponding decrease of the NH_3 concentration on the FTIR signal. Then a TDP test was run with a temperature ramp of 40°C/min resulting in a desorption endothermic peak and the corresponding variation of NH_3 concentration (FTIR signal).

(N. Wilken, K. Kamasamudram, N. W. Currier, J. Li, A.Yezerets, L. Olsson, Catalysis Today, 151 (2010) 237–243)



HEAT OF SORPTION

INSTRUMENT

CALVET

ISOTHERMAL OR TEMPERATURE SCANNING MODES
for increased flexibility

HEAT MEASUREMENT ACCURACY
with Calvet 3D sensor capturing 93-95% of all heat forms. The highest level on the market

CONVENIENT, INTERCHANGEABLE CELLS
to perform even the most demanding experiments using one instrument

WIDE TEMPERATURE RANGE
with low temperature version CALVET CRYO and high temperature version CALVET HT

SPECIFICATIONS

	CALVET	CALVET CRYO	CALVET HT
Temperature range (°C)	Ambient to 300	-196 to 200	Ambient to 600
Temperature accuracy (°C)	+/-0.3 *	+/-0.5 **	+/-1*
Temperature precision (°C)	+/-0.15*	+/-0.25**	+/-0.5*
Programmable temperature scanning rate	0.001 to 2°C/min	0.01 to 1°C/min	0.01 to 2°C/min
Enthalpy accuracy	+/-0.4 *	+/-0.2 **	+/-1*
Calorimetric precision (%)	+/-0.4*	+/-0.5**	+/-1.5*
Cells (ml)	Up to 12.5 (standard cell)	Up to 12.5 (standard cell)	Up to 7
Pressure measured and controlled (bar [psi])	350 [5,075]; 600 [8,700]; 1000 [14,600]	100 [1,450]; 600 [8,700]; 1000 [14,600]	100 [1,450]; 300 [4,350]; 400 [5,800]

** Based on indium melting tests ** Based on naphthalene melting tests*

For more information on specifications please consult the product information and brochures available on our website : www.setaramsolutions.com

APPLICATION

Characterizing CO_2 sorption properties of mesoporous silica based samples thanks to manometry & calorimetry

INTRODUCTION

Although manometry provides valuable information about the sorption properties of materials, its combination with other analytical techniques enables better characterization of the sorption phenomenon and of the surface properties of the material.
Calorimetry, that consists at measuring the heat flow from a material as a function of time and temperature, is very interesting from that point of view. GASPRO and the CALVET calorimeter can be coupled. The calorimeter records the heat released after the injection of gas doses by GASPRO. The manometric device records the subsequent pressure drop and its software calculates the corresponding adsorbed quantity per dose.

EXPERIMENT

Samples
2 mesoporous silica were tested : MCM-41-N2 and N3. They were modified to present amine functions at their surface.

Conditions
At 30°C, with 5 and 10 bar CO_2 doses, up to 30 bar.

RESULTS AND CONCLUSION

The heat of adsorption of one dose was divided by the corresponding amount of CO_2 adsorbed during the injection. The obtained “differential heat of sorption” is plotted against pressure (Figure 1).
Between 0 and 3 bar, the observed heat values are high.
They correspond to chemisorption between the amine functions and CO_2.
Above 3 bar, the heat values are of the order of magnitude of physisorption. This takes place in the available pores of the mesoporous silica base.
In the case of MCM-41-N3, the differential heat of sorption becomes higher at 15 bar. This is typically due to the formation of interactions between the adsorbed CO_2 molecules.

You can consult AN739 for more details about these measurements.

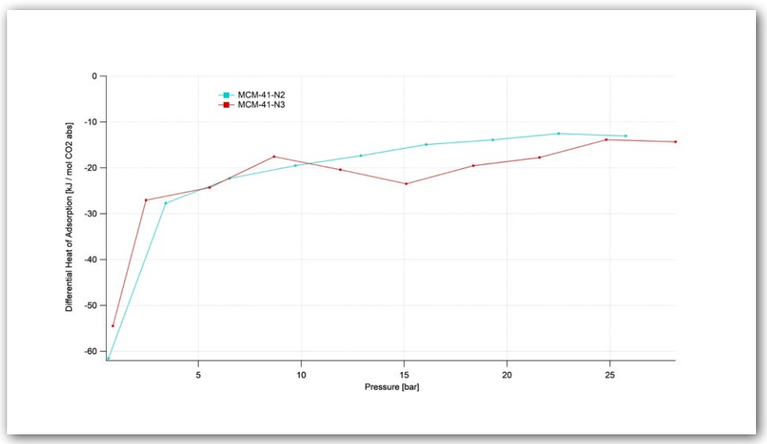


Figure 1 – Differential heat of sorption against pressure for MCM-41-N2 and MCM-41-N3

COKE CONTENT

INSTRUMENT

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Sample volume (ml)	up to 1 in TGA
Evolved gas analyzers (FTIR, MS, GCMS, MS-FTIR, or FTIR-GCMS) for performing qualitative and quantitative gas characterization	

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APPLICATION

Quantification of coke deposited on catalysts by TGA

INTRODUCTION

Solid catalysts can be used in the petrochemical industry to convert efficiently a given reactant into a valuable product with the highest yield possible. The conversion reaction takes place at the surface of the catalyst.
In such industries, the reactants are frequently organic substances containing carbon atoms.
During the reaction, carbon (or coke) can form deposits at the surface of the catalyst. Therefore, the access of fresh reactant to the surface of the catalyst becomes more and more difficult, leading to the catalyst deactivation. The present application note shows the use of TG-DTA technique for the quantification of coke deposited on catalysts during their operation.

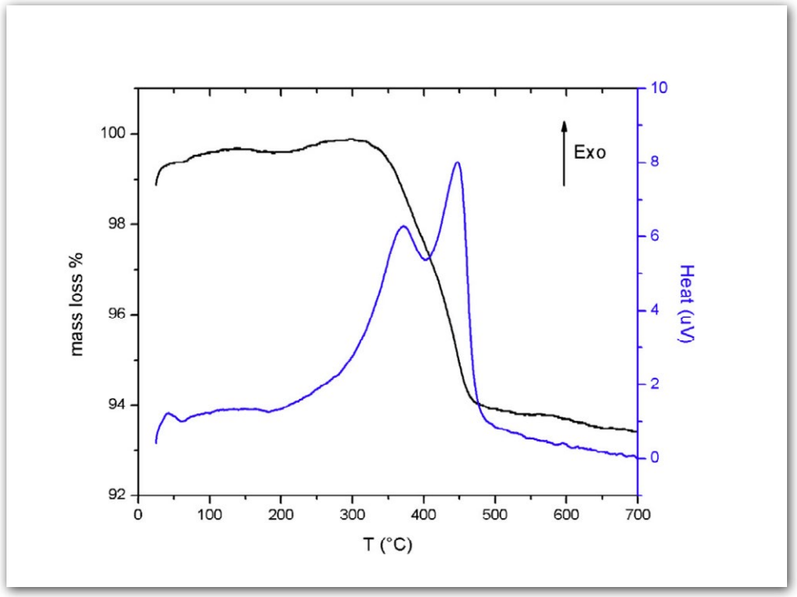
EXPERIMENT

THEMYS ONE TG-DTA thermal analyzer was used to determine the coke content on different PtSn based catalysts that were used for the production of hydrogen from kerosen.
The following thermal profile was applied :
-Heating from 20°C to 700°C at 5°C/min under a flow of synthetic air at 50ml/min.

RESULTS AND CONCLUSION

The figure below shows an example of TGA and DTA result obtained on a catalyst. A weight loss is observed in the range of 320°C to 480°C due to the combustion of the carbon. The values of mass loss in percentage are in the range of 3–7% for all the catalysts tested and from the author the carbon amount formed appears to correlate with the deactivation factor for each catalyst.
From the DTA analysis, two different peaks due to the combustion of different types of carbon maybe distinguished. The peak at 360°C is related to the carbon coke formed on the active metal phase of the catalyst and the second peak at 460–480◦C results from the combustion of the carbon coke formed on the catalyst support.

Reyes-Carmona, Á., Gianotti, E., Taillades-Jacquin, M., Taillades, G., Rozière, J., Rodríguez-Castellón, E., & J. Jones, D. (2013). High purity hydrogen from catalytic partial dehydrogenation. Catalysis Today, 26-32.237–243)



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