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Jens Weitkamp – in Memoriam

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In Memory of Prof. Dr.-Ing. Jens Weitkamp

Prof. Jens Weitkamp was a pioneer in the field of zeolite catalysis, especially for applications such as hydrocracking, isomerization, alkylation, but also selective ring opening of polynuclear aromatics. This is an extended version of the closing remarks delivered at the Symposium in honor of Professor Jens Weitkamp on February 11th, 2020, at the DECHEMA house, Frankfurt, Germany.

Keywords: Catalysis, Diffusion, Jens Weitkamp, Obituary, Zeolites

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1 Introduction

It is in the very nature of man and distinguishes him from all other creatures to ask himself the question why he has become what he is – or, to put it in a larger context: Where do we come from? What are we? Where are we going?

We all are, to a large extent, products of our environment and, notably, a product of the people whom we have come into contact with. Over many stages of my career, I had the great fortune to enjoy the companionship of Jens Weitkamp, benefitting from his advice and support and having found in him a prudent advisor and a true, reliable friend. Many readers certainly share this experience. I decided to deliver my words in a very personal way with confidence that everybody would recognize own experience with a great scientist and, simultaneously, with a compassionate human being.

2 First Contacts

My first contacts with Jens Weitkamp date back to the time of the approaching and finally merging two parts of Germany. In the field of zeolite science and technology, this approach occurred with the two sides seeing each other as equals – pleasantly differing from the situation in many other fields of science. In this context, I am happy to refer to Jens Weitkamp's entry in the guest book of Harry Pfeifer's research group on the occasion of his 65th birthday as displayed in Fig. 1. Harry Pfeifer was my mentor at Leipzig University and worldwide recognized for his merits in marrying zeolite sciences with nuclear magnetic resonance [1–3].

Harry Pfeifer's advice to concentrate on the application of nuclear magnetic resonance to diffusion in zeolites set the course of my further career. It was Jens Weitkamp who drew my attention to the decisive role that diffusion plays in catalysis since performance can never be faster than the rate by which, after their conversion, product molecules get away from the active sites, leaving place for new reactant molecules [4, 5].

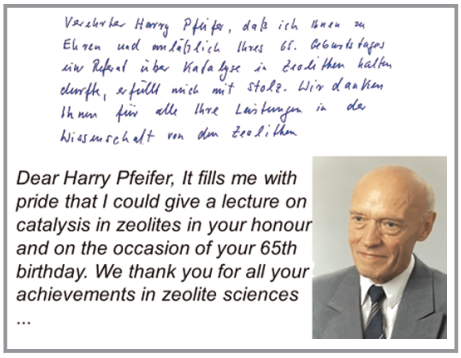

Figure 1. Jens Weitkamp's entry in the guestbook of the Department of Interface Physics at Leipzig University, headed by Prof. Harry Pfeifer on the occasion of his 65th birthday in February 1994.

3 Beginning of a Fruitful Cooperation

Our first cooperation concerned the special case that the molecules are confined to small channels prohibiting their mutual passage, not uncommon for zeolites. Transport

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under such conditions is referred to as single-file diffusion. Fig. 2 describes the peculiarities of mass transfer under such conditions. The probability distribution of displacements z as a function of time t (with the file length assumed to be infinitely large) is still a Gaussian, but the mean square displacement $<z^2(t)>$, as a measure of the speed of molecular propagation, increases in proportion with only the square root of time rather than with time itself. The replacement of product molecules by fresh reactants in porous catalysts occurs, therefore, under single-file conditions at a much smaller rate. Though this phenomenon had been referred to in the literature already quite in general [6, 7], its consequences on the performance of catalytic reactions have remained, quite surprisingly, out of the focus of investigation. Our joint efforts to provide a quantitative correlation between the effectiveness factor of catalytic reactions and the type of molecular propagation including mass transfer under single-file conditions [8] attained, accordingly, substantial interest.

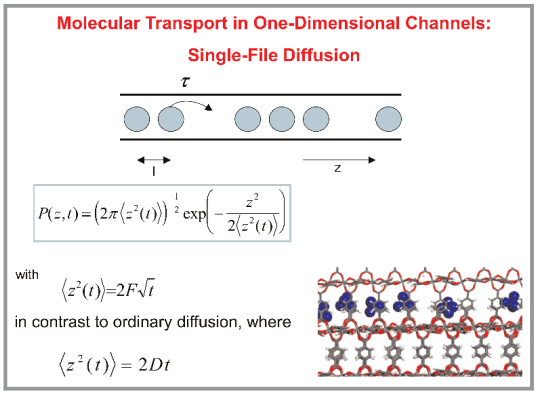

Figure 2. The effect of "single-file confinement": Molecules in sufficiently narrow channels (bottom right) cannot pass each other, leading to a dramatic retardation in molecular propagation. Reprinted with permission from Ref. [13], Copyright 2019 American Chemical Society.

Among the readership of our joint paper was Wolfgang Sachtler of North-Western University, Evanston, IL, USA, who based a series of papers on our findings [9–12]. I very happily remember how Jens Weitkamp introduced us to each other and how – catalyzed by this cordial introduction and by the fact that Wolfgang Sachtler was born and grew up in the little town Delitzsch in Leipzig's immediate vicinity – we eventually became good friends.

He and his wife Anne Lore are shown in Fig. 3, together with Randy Snurr, one of Wolfgang Sachtler's young colleagues at an event commemorating the 30th anniversary of the Center for Catalysis and Surface Science at Northwestern University on August 25, 2015.

Randy provides another bridge to Jens Weitkamp. During his time as a Humboldt fellow at Leipzig University, Randy was involved in both modeling [14] and experimental measurement [15], dealing among others with the conversion of isopropanol to propene in zeolite NaX.



Figure 3. Wolfgang M. H. Sachtler, Anne Lore Sachtler and Randall Q. Snurr (from right; courtesy: Northwestern University).

In these investigations (see Fig. 4), the potential of NMR spectroscopy was exploited for determining the relative amounts of the reactant and product molecules from the respective intensities of the NMR signal (the "free induction decay") and their diffusivities by studying the attenuation of the NMR signal (the "spin echo") under the influence of the pulsed field gradients. The diffusivity of the smaller and, hence, more mobile propene molecules is found to increase

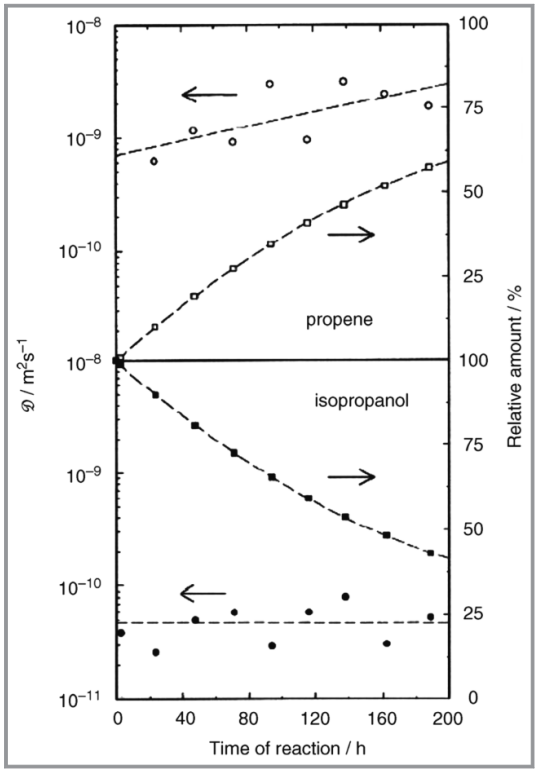

Figure 4. Time dependence of the relative amount (right ordinate scale) of isopropanol (bottom) as the reactant and of propene (top) as the product in zeolite NaX during conversion at 473 K and their diffusivities (left ordinate scale) as determined by PFG NMR, from [16].

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with increasing reaction time, which may be easily referred to a decrease in transport inhibition by the decreasing concentration of the bulkier isopropanol molecules.

It was with these investigations and thanks to Jens Weitkamp's intermediation that we physicists in Leipzig started to substantially benefit from the competent and generous support of DECHEMA, under whose auspices these investigations have been performed (Fig. 5).

4 Joint Networking

With Jens Weitkamp on board, we physicists did even dare applying for a network dedicated to transport optimization during fluid catalytic cracking within the Competitive and Sustainable Growth program of the European Commission. Our application was based on the ability of PFG NMR to record molecular mobilities over varying length scales as illustrated by Fig. 6.

This includes in particular diffusion measurement in the interior of the individual crystals and over larger distances. From these data one could conclude that the conversion rate of chemical reactions is controlled by intra*particle* diffusion rather than by intra*crystalline* diffusion. Fig. 7 illustrates that this conclusion could be nicely confirmed by direct experimental evidence. Considering a series of catalyst particles (catalysts 1 to 4) produced with identical Y-type zeolite crystals under different formulation conditions, catalysts with the largest intraparticle diffusivities were found to give rise to the largest conversion rates. The investigations have been performed within a network involving ten partner institutions, notably from industry. Without Jens Weitkamp's help and influence we would never have been able to assemble such a powerful community, notably pushed forward thanks to Michael Stöcker with SINTEF and Roger Gläser as Jens Weitkamp's habilitand in these days.

A second finding of these studies concerned the influence of the pore space formed during zeolite dealumination as an integral step in manufacturing the catalyst particles. This enhancement in mesoporosity turned out to leave the intra-



Figure 5. Jens Weitkamp with the DECHEMA managing directors, Gerhard Kreysa, and Kurt Wagemann, respectively, on the occasion of JK's "official" retirement in 2009 in Leipzig (left) and at the Wachenburg near Weinheim on the occasion of Klaus Unger's 80th birthday in 2016. (Source right photo: K. K. Unger).

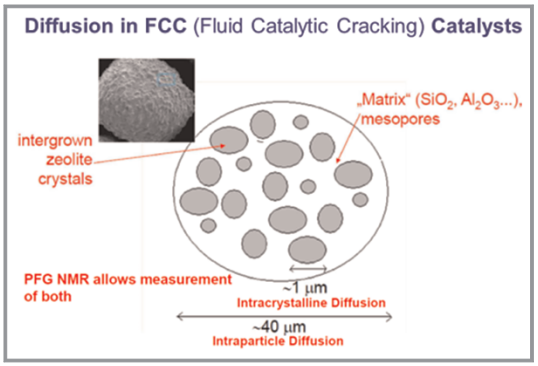

Figure 6. The potential of pulsed field gradient (PFG) NMR for recording mass transfer in an FCC catalyst particle (top left). Depending on the chosen observation time, diffusivities both in the interior of the individual zeolite crystals (the intracrystalline diffusivities) and through the whole catalyst particle (intercrystalline or intraparticle diffusivities) can be determined.

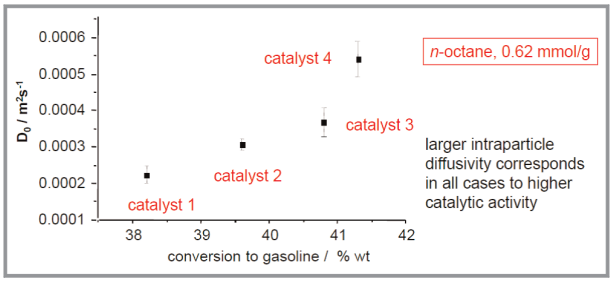

Figure 7. Correlating catalytic performance with intraparticle diffusivities: the conversion rate was observed to increase with an increase in the intraparticle diffusivity D_0 (with the zeolite crystals and, hence, the intracrystalline diffusivities kept constant in all catalyst particles). Adapted with permission from Ref. [17], Copyright 2005 American Chemical Society.

crystalline diffusivity, as recorded with *n*-octane, essentially unaffected [18]. This observation significantly differs from the message of PFG NMR diffusion measurement of, e.g., propane in mesoporous zeolite NaCaA [19], where the presence of mesopores gave rise to a diffusivity enhancement of more than two orders of magnitude [20]. This difference, however, may be easily explained by having in mind that the space of mesopores as generated in zeolite NaCaA penetrates the zeolite crystal as a continuous network, while the mesopores emerging during dealumination of Y-type zeolites are, obviously, separated from each other. Direct experimental evidence of such a behavior has been provided by three-dimensional transmission electron microscopic observation [21]. In Ref. [18], this finding was additionally confirmed by following the uptake of 1,3,5-triisopropylbenzol (1,3,5-TIPB). This molecule is too bulky for permeating the genuine Y-type pore space. The pore space corresponding to

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the actually adsorbed amount of 1,3,5-TIPB, was found to be notably exceeded by the total space of mesopores as resulting from analysis of the nitrogen isotherms. This, once again, excludes the option that zeolites of type USY are permeated by a coherent network allowing molecular exchange between the zeolite interior and its surroundings. This, however, is among the decisive criteria for ensuring that the existence of mesopores can in fact result in any substantial transport enhancement [22–25].

5 Monitoring Diffusion and Catalytic Conversion

In addition to the diffusivities and concentrations of all involved components, the in-depth investigation of a reaction process should ideally be based also on knowledge of the distribution of the involved components within the catalyst. In principle, NMR does as well provide this type of information. In fact, the detection of this possibility was honored in 2003 with the Nobel Prize in Medicine to Paul Lauterbur and Peter Mansfield for their foundation of magnetic resonance imaging (Fig. 8).

Thanks to Harry Pfeifer's personal contact to Paul Lauterbur (top left in Fig. 9), our group in Leipzig was probably the first one to apply this technique in chemical engineering [26]. In these early days we still used the name zeugmatography as Paul Lauterbur had originally called it. We have, primarily, introduced this technique as an alternative to X-ray tomography [27] with the option to operate with, essentially, any (proton-containing) guest molecule, and not only with those accommodating particularly heavy nuclei as a prerequisite for X-ray tomography. Fig. 9 introduces the type of information that has become accessible in these old studies by IR imaging. It displays the evolution of the distribution of (a finite amount of) guest molecules (*n*-butane) that has been introduced on top of a bed of previously activated (i.e., evacuated) crystals of NaCaA-type zeolites contained in the NMR sample tube. Such concentration profiles become directly accessible by analyzing the frequency dependence of the NMR signal if the constant magnetic field is superimposed by a field gradient in direction of the axis of the NMR tube. While molecular uptake in a bed of large crystals occurs essentially simultaneously over the whole bed of crystals, attaining final equilibrium during a couple of seconds (top), within a bed of small crystallites the offered amount of guest molecules is found to be adsorbed within only a small layer close to the bed surface, once again during the course of (tens of) seconds (bottom left). This process, however, is followed by a second, much slower process, leading during the course of tens of minutes till hours to an equilibration of the guest molecules over the whole bed of crystals.

With resolutions in the (sub-)millimeter range and seconds, this type of information may attain substantial relevance in the assessment of bulk phenomena in chemical engineering, including the knowledge-based optimization of adsorption columns [28–30]. It did not suffice, however, for providing a reliable resolution of concentration profiles within the individual crystals. For this purpose, another experimental approach was required.

Being impressed by the far-reaching potential of microimaging by IR and interference microscopy for exactly this type of application, Jens Weitkamp enthusiastically supported our efforts to establish these techniques as alternatives. Fig. 10 provides an introduction to these techniques in a nutshell. The crystal under study (center bottom) is placed under a microscope (left). Information about the guest concentration is attained in interference microscopy (IFM) by analyzing the interference of a light beam passing the crystal with a calibration beam (top center), yielding information about the refractive index of the crystal, which – in turn – is a function of the guest concentration. IR microscopy (IRM) is based on the message of an array of detectors. The attained signal (top right) provides information about the type of molecules and

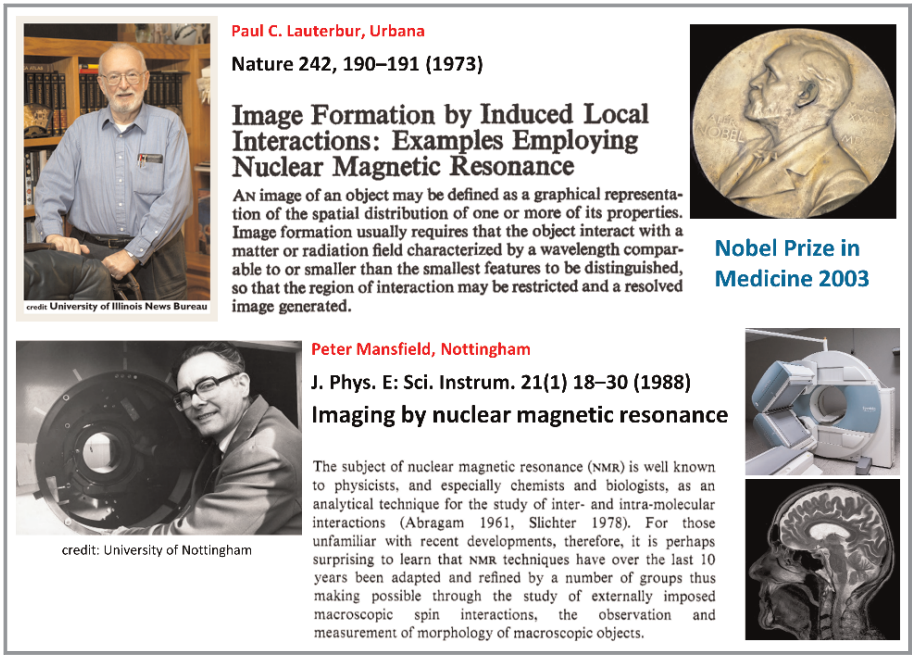

Figure 8. In recognition of their groundbreaking work in the application of NMR spectroscopy for medical imaging, Paul Lauterbur and Peter Mansfield were awarded the Nobel Prize in Medicine in 2003.

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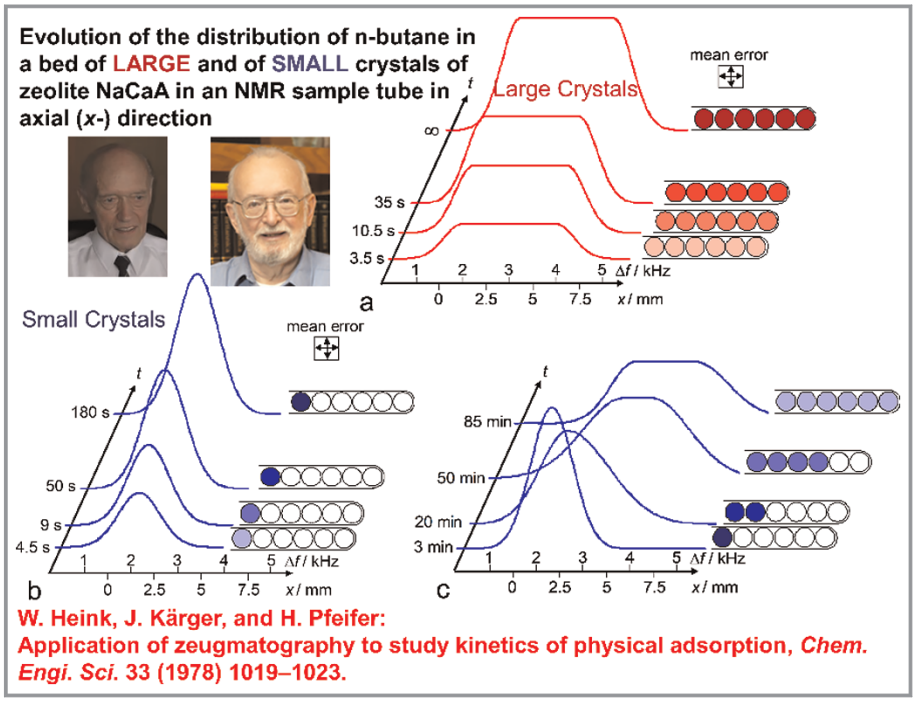

Figure 9. Early-days' magnetic resonance imaging (MRI – still under the name "zeugmatography") of the evolution of the distribution of *n*-butane molecules within a bed of zeolite crystals of type NaCaA (after [26]).

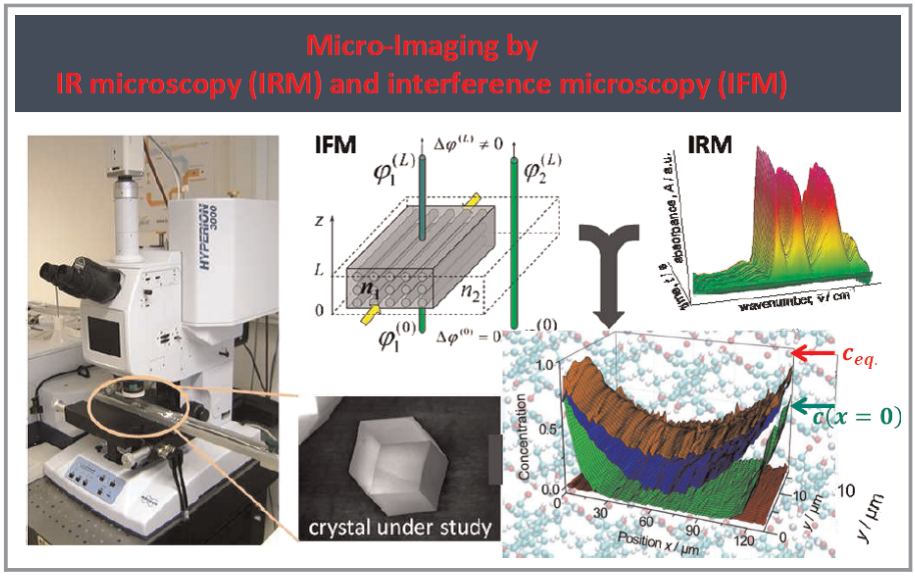

Figure 10. Microimaging in a nutshell: With the nanoporous crystal/particle (bottom center) in the focus of a microscope (left) it is possible to monitor the evolution of intracrystalline concentration (bottom right) by analyzing in interference microscopy (IFM, top center) the interference patterns of light beams passing the crystal with those passing their surroundings, and in IR FT microscopy (IRM, top right) the signals received by an array of IR detectors.

their relative amount within the space element under study (with a minimum size of 2.7 µm). In both IFM and IRM it is the integral in observation direction rather than the local concentration itself that one is able to record. This, however, does not mean any limitation in significance if mass transfer may be implied to occur in only the plane perpendicular to the observation direction. This may be easily ensured with 1d and 2d pore spaces and may quite generally be cared for by an appropriate (partial) sealing of the particle/crystal under study [31, 32]. Bottom right shows a typical example of the transient concentration profiles (in this case obtained during molecular uptake) as attainable by either technique. A more detailed introduction may be found in [33–36] and in Sect. 12.2 of [37].

The potential of microimaging was developed and explored in a number of case studies performed in close cooperation between Stuttgart and Leipzig with joint funding by the European Commission, DECHEMA and the German Science Foundation. In this context, zeolites of type ferrierite proved to serve as an ideal host system. Depending on the way of synthesizing and pretreatment [38, 39], uptake and release (of, e.g., short-chain alcohols) could be "switched" to occur through either, predominantly, the smaller (and shorter) 8-ring channels or the 10-ring channels, oriented along the longitudinal extension of the crystals [40–42]. With both channels open, molecular uptake was clearly observed to occur predominantly along the broader channels, though with a nice exception: Pre-adsorption of ethanol was observed to be followed by methanol uptake preferentially through the narrow channels [35]. Now, after the period of ethanol pre-adsorption, the wider 10-ring channels were obviously blocked by already so many ethanol molecules that the subsequent methanol adsorption was faster through the narrower, but not yet that much populated 8-ring channels.

Owing to its ability to simultaneously record the concentration profiles of different molecular species [43–46], IR

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imaging is ideally suited for recording the evolution of the concentration profiles of the reactant and product molecules during molecular conversion in nanoporous catalysts. In Fig. 11 this potential is exemplified with the transient concentration profiles of the reactant and product molecules recorded during the hydration of benzene to cyclohexane in a nanoporous glass plate (the porous catalyst) containing finely dispersed nickel operating as a catalyst in this reaction. Top and bottom faces were sealed gas-tight, allowing molecular uptake and release to take place through only the edge ($x = 0$) of the platelets. The information presented in the figure is taken from the area highlighted in yellow in the scheme shown top right. The full lines are best fits of the solution of the diffusion-reaction equation solved with the simplifying assumption of a first-order reaction with the diffusivity D coinciding for the reactant and product molecules and with both the diffusivity and the rate constant k of conversion being independent of loading.

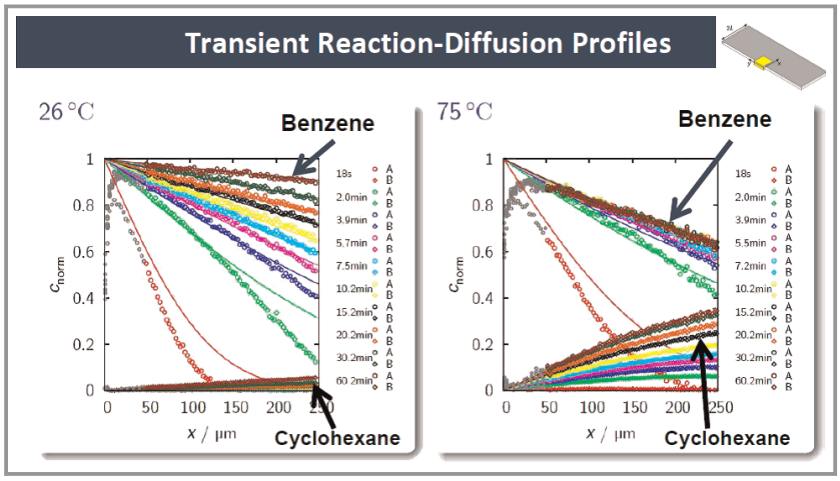

Figure 11. Monitoring the transient concentration profiles of benzene and cyclohexane, during benzene hydration to cyclohexane, in a nanoporous glass plate with finely dispersed nickel used as a catalyst (symbols). The full lines are best fits of the solution of the diffusion-reaction equation, after [32]. Copyright Wiley-VCH GmbH.

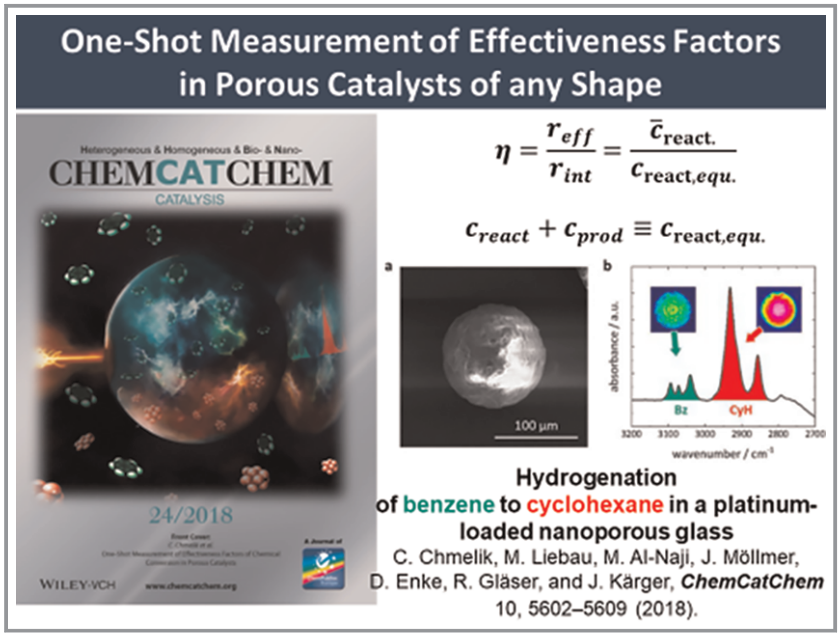

Figure 12. Analysis of the intensity of the absorption lines in the IR spectrum, fingerprinting the various components involved in a chemical reaction provides direct access to the mean reactant and product concentrations within a porous catalyst and, thus, to the effectiveness factor of the reaction [47].

A temperature increase from 26 °C (left) to 75 °C (right) is seen to enhance the propagation rate of the diffusion front into the material and, in particular, to substantially accelerate the conversion, giving rise to a much larger concentration of product molecules within the porous catalyst under dynamic equilibrium. Stationarity, i.e., dynamic equilibrium, is seen to be finally attained when the rate of benzene uptake (following the decay in benzene concentrations towards the particle interior), the conversion rate from benzene to cyclohexane within the particle and the rate of cyclohexane release (following the decay in cyclohexane concentrations towards the surroundings) coincide. With knowledge of the concentration profiles, also the mean concentrations of the reactant and product molecules are known and, hence, the effectiveness factor of the reaction.

Since information about the mean concentration is directly provided by the intensity of the characteristic IR bands, such type of measurement – allowing a one-shot determination of the effectiveness factor – is possible for any particle shape. This possibility became even the cover story in the Christmas issue of *ChemCatChem* in 2018 as displayed with in Fig. 12. We received warm congratulations on this from Jens Weitkamp, who was happy to see the continuation of our cooperation, now with, notably, Roger Gläser, as one of his "own" people.

6 Networking Continued

From the very beginning of our contact, Jens Weitkamp was aware of the significance of diffusion in catalysis and adsorption science and technology in general. Thus, he became one of the strong supporters of the idea to focus the

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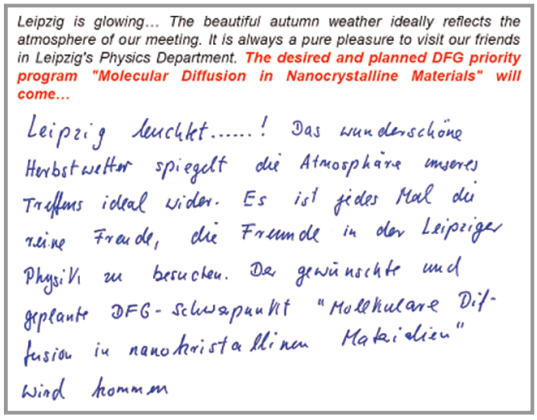
diverse activities in this field, starting with a priority program under the auspices of the German Science Foundation. A corresponding entry in the guest book of our group as shown in Fig. 13 demonstrates his engagement.


Leipzig is glowing... The beautiful autumn weather ideally reflects the atmosphere of our meeting. It is always a pure pleasure to visit our friends in Leipzig's Physics Department. The desired and planned DFG priority program "Molecular Diffusion in Nanocrystalline Materials" will come...

Leipzig leuchtet.....! Das wunderschöne Herbstwetter spiegelt die Atmosphäre unseres Treffens ideal wider. Es ist jedes Mal die reine Freude, die Freunde in der Leipziger Physik zu besuchen. Da gewünschte und geplante DFG-Schwerpunkt "Molekulare Diffusion in nanokristallinen Materialien" wird kommen

Figure 13. Jens Weitkamp's entry in the guestbook of the department of Interfacial Physics of Leipzig University on November 10, 2000.

It was with great pleasure that Jens Weikamp became aware of the continuation and worldwide accomplishment of this endeavor within an IUPAC group (Fig. 14).

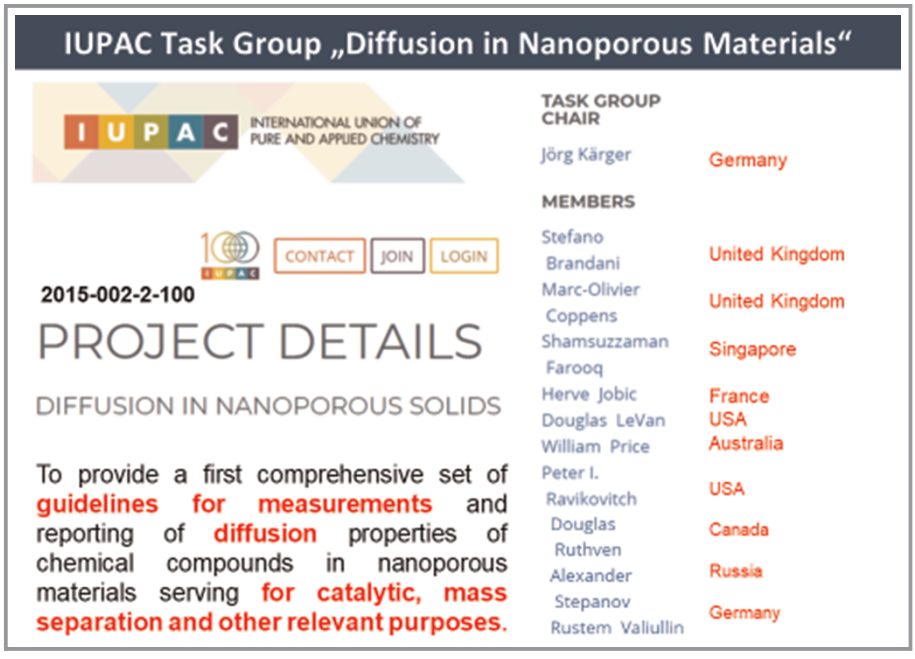


Figure 14. Diffusion in Nanoporous Solids: Formation of an IUPAC task group in continuation of the cooperation with Jens Weitkamp, and with many more colleagues and friends all over the world (https://iupac.org/projects/project-details/?project_nr=2015-002-2-100, see also [48] and further contributions to a thematic issue of the Adsorption Journal).

7 Commemorating a Great Scientist and a Cordial Friend

I had the privilege and pleasure to publish jointly with Jens Weitkamp a total of 30 papers as a result of a most productive and reliable cooperation and fruitful discussions. Equally important, these were occasions of intellectual enjoyment: enjoyment about the sharpness of his mind and the precision of his words, and all this combined with a wonderfully dry humor. I once told him about the receipt of an e-mail, which concluded with the remark: "For reasons of time efficiency, I have refrained from proofreading this e-mail". In this case, Jens Weitkamp said to me, he would have answered with a return mail: "For reasons of time efficiency, I have refrained from reading this e-mail".

The topics of common interest and, hence, of our discussions were by far not restricted to zeolites, catalysis, and diffusion. Once he surprised me with Sigmund Freud's reflections about "Jokes and their relation to the unconscious" as a book present – which immediately reminded me of his remark concerning e-mails and time efficiency.

In another book present (Fig. 15) Jens Weitkamp surprised me with novel views on one of our greatest German geniuses, Johann Wolfgang von Goethe. Born in Frankfurt, Goethe came to Leipzig as a student and learned – clearly among others – to handle the tools, which he later had to apply as a minister at the court in Weimar. His obligations notably included assurance of a high level in teaching and research at the University of Jena in Thuringia, obligations that also Jens Weitkamp has met in the best possible way, both as a vice Rector at Stuttgart University and as the Founding Rector of the German University in Cairo.

Jens Weitkamp might also have shared with Goethe the fascination of the country and might have totally agreed with him, when he was saying "I've always loved being here … I think it comes from the harmony in which everything is here, the area, the people, the climate, the do's and don'ts." [50].

The bottom left of Fig. 15 shows Jens Weitkamp in front of the "Rudelsburg", on a hiking tour jointly with Jürgen Caro and Christine Pfeifer, Harry Pfeifer's spouse. Jens Weitkamp always appreciated the persons and not the offices they held. His visits to Leipzig therefore included meetings with Harry Pfeifer and his spouse even long after Harry

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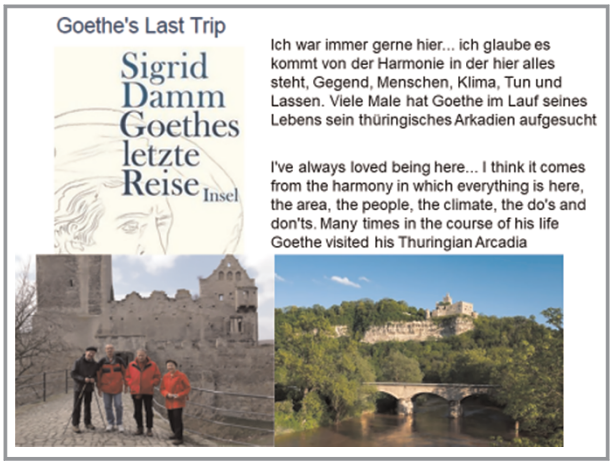

Figure 15. Left: Jens Weitkamp during a hiking tour in front of the Rudelsburg above the Saale River close to Jena, jointly with Christine Pfeifer, Jürgen Caro [49] and JK (with the sticks). Top: excerpt from Sigrid Damm's "Goethes letzte Reise" [50]. Right: Rudelsburg (Copyright: Saale-Unstrut-Tourismus e.V., Wolfgang Kubak (Merseburg)).

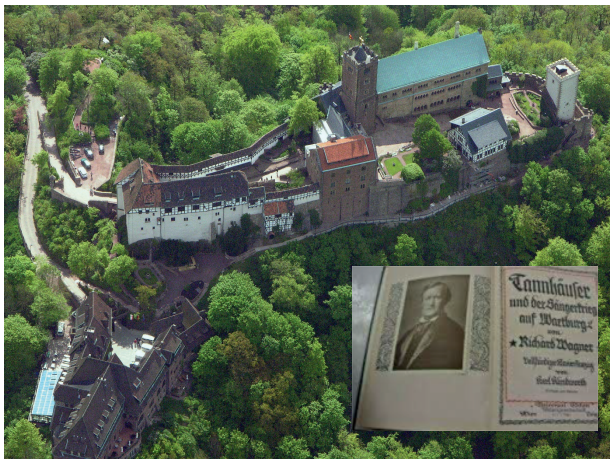

Figure 16. Commemorating a recording of Wagner's Tannhäuser, which I received as a present from Jens Weitkamp, with the narration on and near the probably most famous castle in Thuringia, the Wartburg.

Pfeifer's retirement – and Jens held on to this tradition even after Harry's death.

Jens Weitkamp's love for this landscape was, most likely, additionally nourished by his love for music, notably the music of Richard Wagner, also a child of the city of Leipzig. It was probably no coincidence that I once received a wonderful recording of the opera Tannhäuser from him, with the action of the opera taking place on and around the presumably most famous castle in Thuringia, the Wartburg (Fig. 16).

In 2003 the Saxon Academy of Sciences and Humanities in Leipzig elected Jens Weitkamp as a corresponding member of its Mathematical-Natural Sciences Class. With Saxony, Saxony-Anhalt and Thuringia, it represents the central German region in the Union of the German Academies of Sciences.

To this day, famous scientists like Wilhelm Ostwald and Werner Heisenberg among its members set the standards [51]. With very good reason, the Academy President during these days, Pirmin Stekeler-Weithofer, said therefore in his congratulatory letter to Jens Weitkamp on his 70th birthday in June 2012 (Fig. 17):

"The Saxon Academy of Sciences and Humanities considers itself lucky to have a scientist of your international reputation and national standing in its ranks."

and

"Your lecture on 'Zeolites and Catalysis' is still in my very best memory as an impressive example of the synergy of basic research and application."

8 Farewell

In the future, we will have to renounce the great joy and grace of listening to Jens Weitkamp. We will miss the keenness of his mind and his warm-heartedness. Taking up my initial questions: "Where do we come from?" and "What are we?", I guess, everybody will agree: He meant a lot to us. Without his beneficial foresight and influence, science and thus we ourselves would not have become what we are. And where are we going? Nobody knows. But Jens Weitkamp's keen spirit will certainly be with us. Thank you, Professor Weitkamp, thank you, dear Jens, for having been with us and farewell.

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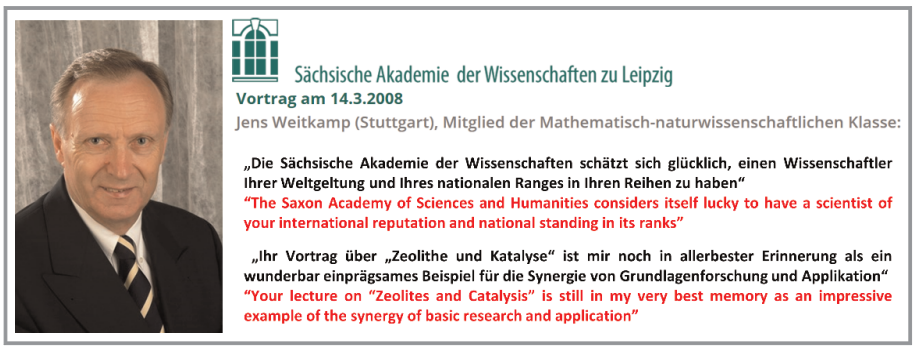

Figure 17. Jens Weitkamp as a corresponding member of the Mathematical-Natural Sciences Class of the Saxon Academy of Sciences and Humanities in Leipzig, elected on February 14, 2003. Quotations from the president's congratulation letter on the occasion of his 70th birthday in 2012.

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