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d'informations mutuelles

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## Contents

Editorial	1
Obituary Nicolaas Bloembergen	2
Portrait: Prof. Stefan Jurga	6
Calls for nominations	8
Report on Ampere NMR School 2017	10
Ampere NMR School 2017 Posterprizes: Johanna Kowalczyk, Raquel Saborano, Anne Selent	14
Report on 14 <sup>th</sup> ICMRM in Halifax 2017	22
Paul Callaghan Young Investigator Award: Jeffrey Simkins	24
ICMRM Poster Prize: Sarah Vashaee	28
ICMRM Image Beauty Competition: Krzysztof Klodowski and Mick Mantle	30
First announcement MRFood 2018	32
First announcement EUROMAR & ISMAR 2019 joint conferences	35
Executive Officers and Honorary Members of the AMPERE Bureau	36
Future conferences and AMPERE events	40

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## Editorial

Dear members of Groupement AMPERE,

the year 2017 is coming to its close and it is time to think about 2018. In the opening session of EUROMAR 2018 in Nantes (France) the AMPERE Prize for Young Investigators and the Raymond Andrew Prize for an outstanding Ph.D. thesis will be awarded. You find the calls for nomination in this issue. In fact, it may already be time to plan ahead for 2019, when EUROMAR and ISMAR will join forces for a meeting in Berlin. This conference will take place in an unusual week for EUROMAR (August 25<sup>th</sup>-30<sup>th</sup> 2019).

There is also sad news. Nicolaas Bloembergen passed away in September. Even if he received his 1981 Nobel Prize for Physics for his contribution to the development of laser spectroscopy, he is also one of the great heroes of early NMR where he contributed strongly to our modern understanding of relaxation. If you want to learn more about him, please read the obituary contributed by Rob Kaptein, Claudio Luchinat, and Rolf Boelens.

On a more upbeat tone we continue our series of portraits with the one of Stefan Jurga who likes 25 MHz as a frequency (find out why on p.6). Fitting to this portrait this Bulletin issue also contains the report on the AMPERE NMR School 2017, an event initiated by Stefan Jurga, and the scientific abstracts of poster award winners at this school. The reports on ICMRM 2017 in Halifax come not only with the Poster Prize abstract and a report on the work that won Jeffrey Simkins the Paul Callaghan Young Investigator Award, but also with the two winning pictures of an Image Beauty Competition. Make your own decision whether you like the abstract one or the spooky one more.

I wish you all the best for the holiday season and a good fresh start into the next year.



Gunnar Jeschke

Secretary General of Groupement AMPERE

## Obituary

### **Nicolaas Bloembergen: a pioneer in magnetic resonance and maser and laser physics.**

Rob Kaptein and Rolf Boelens,  
Department of Chemistry, Utrecht University,  
Padualaan 8, 3584 CH Utrecht, The Netherlands  
Claudio Luchinat,  
Magnetic Resonance Center (CERM) and  
Department of Chemistry, University of Florence,  
Via Luigi Sacconi 6, 50019 Sesto Fiorentino, Italy



Foto: Vetter (Spaarnestad Photo), Dutch National Archives, The Hague

**Nicolaas Bloembergen, Nobel laureate, died on September 5<sup>th</sup> at the age of 97 in Tucson, Arizona.** He was a true pioneer in two research areas: NMR spectroscopy, where he was the first to describe the fundamentals of nuclear spin relaxation, and in maser and laser physics.<sup>1</sup> In particular his work on nonlinear optics earned him the Nobel Prize in Physics in 1981.<sup>2</sup>

Nico Bloembergen was born in 1920 in The Netherlands. He started his study of physics in 1938 at Utrecht University mainly because Utrecht was conveniently close to Bilthoven, the town where he lived. He passed his master's exam in 1943 just a few weeks before the German army, that occupied Holland during World War II, closed the university. As conditions for his PhD research would be very poor in Holland during and just after the war, he moved in 1946 to Harvard University, where he was accepted as the first PhD student of Edward Purcell. Of course, NMR in condensed matter had just been discovered by Purcell and his co-workers Pound and Torrey (and independently by Felix Bloch at Stanford). For their discoveries Purcell and Bloch would share the Nobel Prize in Physics in 1952.

At Harvard, Bloembergen started to work at both experimental and theoretical aspects of NMR (he considered himself an experimentalist). However, his main accomplishment during the years 1946-1948 was his

ground-breaking work on nuclear spin relaxation, that became known as the BPP theory (after the authors Bloembergen, Purcell, and Pound). The Phys Rev paper of 1948 is a citation classic and was for many years the most cited paper in physics.<sup>3</sup> This theory explained the "motional narrowing" effect and formed the basis for many of the subsequent important experimental and theoretical advancements in the understanding of nuclear and electron relaxation. In 1947 Bloembergen met Prof. C.J. Gorter during a visit at Harvard. Gorter was an expert on paramagnetic relaxation at Leiden University and had made two unsuccessful attempts to discover NMR.<sup>4,5</sup> Gorter offered him to come to Leiden and finish his PhD thesis there. This was attractive to Bloembergen also because he had already passed the Dutch qualifying exams for a doctoral thesis. So he defended his doctoral thesis, which was largely based on the research done at Harvard, in 1948 at Leiden University.

Shortly after, he accepted an offer to return to Harvard. He started as a Junior Fellow of the prestigious Society of Fellows, which served as a pool of very talented scientists from which Harvard recruited its staff. In 1951 he was appointed Professor of Applied Physics and he stayed on the faculty until his retirement in 1990. Initially he continued with magnetic resonance. To quote a few of the most significant achievements by Bloembergen in the mid-50's in this area: the treatment of the so-called Fermi contact - or scalar - relaxation between nuclei, published together with I. Solomon,<sup>6</sup> who had published on nuclear dipolar relaxation the year before; his work on nuclear relaxation in paramagnetic solutions;<sup>7</sup> and, at the beginning of the 60's, together with L. O. Morgan, the theory of field-dependent electron relaxation.<sup>8</sup> This body of work has been highly cited, not only by physicists, but also by many experimental chemists and biochemists, and is universally known as the Solomon-Bloembergen-Morgan (SBM) model. The SBM model is at the basis of the important inter-atomic distance measurements by NOE's in biological macromolecules, and of the use of paramagnetic relaxation (PRE) as a further source of structural information when the biomolecule contains a paramagnetic metal ion or a radical center. Understanding relaxation is also very important in MRI, since the contrast in MRI scans is based on differences in relaxation times; furthermore, paramagnetic relaxation is at the basis of the development of contrast agents, paramagnetic metal complexes that are universally administered to enhance MRI contrast.

In the 50's Bloembergen became also interested in masers, which stemmed from the search for low-noise microwave amplifiers in the upcoming fields of data communication and radio astronomy. A crucial contribution was

his idea to use 3-level systems, using separate frequencies for excitation and stimulated emission. His familiarity with population inversion in NMR, either by  $180^\circ$  RF pulses or rapid passage methods led to this idea. The principle of 3 (or multiple) level systems led to the first practical continuous-wave masers. Bloembergen constructed a maser operating at a 21 cm wavelength, to detect the interstellar hydrogen line.<sup>9</sup> The same type of maser was later used by Penzias and Wilson to detect the cosmic background radiation as a remnant of the Big Bang, for which they earned the Nobel Prize.

The concept of the 3-level system was later also used to develop lasers. However, Bloembergen decided not to work on the development of lasers since he felt that this could better be done in large industrial laboratories such as IBM, GE, and Bell labs, which had embarked on this. Instead, he started to use powerful lasers to study the nonlinear optical behavior of materials. That work was awarded by the Nobel Prize. He realized that common properties of light such as reflection or diffraction, which were well understood at low intensity, would change in high-intensity laser beams. He worked out the fundamentals of nonlinear optics and is often considered the father of this field. This work found important applications in fast data communication using fiber optics.

Nico Bloembergen retired from Harvard in 1990. But far from resting on his laurels he took a new position as Professor at the University of Arizona in Tucson, where he studied fast dynamical processes of materials by femto-second laser spectroscopy.

Apart from his passion for science he was also a great teacher, very much loved by his students. In honor of his pioneering work in NMR the building of the Utrecht NMR group was named after Nicolaas Bloembergen in 2001.

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## Portrait: Prof. Stefan Jurga

- why magnetic resonance and why NMR and MRI?

Wonderful tools to better understand molecular and crystal physics, especially in finding time and space dependent interactions of atoms, molecules and nanosystems.

- what is your favorite frequency?

Generally, any frequency that allows to solve a physical problem. Since I started an adventure with NMR by constructing a solid state pulse spectrometer operating at 25 MHz, I like this frequency.

- what do you still not understand?

A lot of things in science; some of them are striking, specifically: 1) why superconductivity is not yet achieved at room temperature, 2) why neuroscience research models do not describe adequately the behaviour of human beings and more generally from the point of view of philosophy of science and 3) why there is a lack of a scientific models which would be able to couple science and mysteries in science.

- luckiest experiment you have ever done.

A measurement of spin-lattice relaxation times in the rotating field using a homemade solid-state NMR spectrometer operating at 25 MHz in the beginning of 70s. First such experiment in Poland.

- what was the worst mistake you have made during your lab time?

An explosion in a lab while preparing perchlorates samples for NMR measurements.

- most memorable conference story

It was a gala dinner at an NMR meeting in Crete: Ulrich Haebleren, dressed in an elegant suit, is jumping into a swimming pool to win a conference cake! Huge ovation of the audience!

- with whom (historical person) would you like to meet?

Marian Smoluchowski

- when do you get your best ideas?

While hiking in the woods.

- if you had just one month time for travelling - where would you go to?  
To see the biggest and wildest waterfalls in the world.

- your idea of happiness.

- 1) successful activities of my students and coworkers in science
- 2) development of some of the Polish research institutions where I left my "trace" as a result of my work in the Polish Government in the capacity of Secretary of State in the Ministry of Science and Higher Education and
- 3) observing my beloved grandchildren.



Position: Director of the NanoBioMedical Centre of the Adam Mickiewicz University, Poznań

Awards: Numerous Prizes of the Polish Minister of Science and Higher Education  
Numerous medals of recognition from Polish universities  
Twice election by the university community for rector of the Adam Mickiewicz University,  
Alexander von Humboldt Fellowship in Mainz  
Election for president by Santander Group Universities rectors  
Commander's Cross of the Order of Merit of the Republic of Poland  
Knight's Commander cross of the Order of St. Sylvester

Education:

1974: PhD in Physics at Adam Mickiewicz University Poznań (AMU),  
1975-1976: Research Associate at University of Liege, University of Illinois  
1976-1982: Teaching position in the Department of Physics, AMU  
1982: Visiting research position, Josef Stefan Institute, Lubljana  
1983-1986: Alexander von Humboldt and Max Planck Fellow, the Max Planck Institute for Polymer Research in Mainz with Professor H.W. Spiess  
1986-1991: Research and teaching position, Faculty of Physics and Mathematics, AMU  
1991: University Professor, Faculty of Physics, AMU  
1994-2016: Head of the Macromolecular Physics Department, AMU  
2010: Director of the newly funded NanoBioMedical Centre of AMU

Interests: Choral and classical music

Homepage: [www.staff.amu.edu.pl/~zfm](http://www.staff.amu.edu.pl/~zfm)

## Call for nominations AMPERE Prize for Young Investigators 2018

The AMPERE prize has recently been redefined and is now given to a young principle researcher (a "rising star") for her/his first achievements in her/his independent career. There is no strict age limit but typically researchers below the age of forty are envisioned. The prize is given biannually.

The committee now calls for Nominations for the AMPERE Prize 2018 for a young principal investigator in the field of magnetic resonance. The prize will be presented during the EUROMAR in Nantes (France) July 1<sup>st</sup> to July 5<sup>th</sup> 2018. The prize carries a value of € 2000.

You are kindly invited to submit nominations by e-mail to the president of the prize committee

beat.meier@nmr.phys.chem.ethz.ch

Suggestions must be received by **15<sup>th</sup> February 2018** and should include the following documents:

- Nomination letter
- Curriculum vitae
- List of publications and presentations at conferences

## Call for nominations Raymond Andrew Prize 2018

In memory of Professor Dr. Raymond Andrew and to honor his pioneering work in the field of magnetic resonance, the AMPERE Group has founded the Raymond Andrew Prize. The prize is awarded to young scientists for an outstanding PhD thesis in magnetic resonance.

For the Raymond Andrew Prize 2018 the AMPERE Prize Committee is seeking your help in searching for qualified candidates who completed their dissertation during the period of 2016/2017. The prize will be presented during EUROMAR in Nantes (France) July 1<sup>st</sup> to July 5<sup>th</sup> 2018.

You are kindly invited to submit nominations by e-mail to [andrewprize@ampere-society.org](mailto:andrewprize@ampere-society.org)

Suggestions must be received by **15<sup>th</sup> February 2018** and should include the following documents:

- Nomination letter
- Curriculum vitae
- List of publications and presentations at conferences
- PhD thesis in PDF

The thesis should be written in English. In exceptional cases, the thesis may also be submitted in triplicate as a hardcopy to the AMPERE Secretariat. Submissions that arrive too late will automatically be transferred to the next year. The prize committee will reconsider excellent contributions for two years in a row.

For a list of past Andrew Prize winners see:  
[www.ampere-society.org](http://www.ampere-society.org)

## Report on Ampere NMR School 2017 Zakopane, Poland

### Scientific Committee

B. Blümich (Aachen), Germany, J. Dolinšek (Ljubljana), Slovenia, F. Fujara (Darmstadt), Germany, S. Jurga (Poznan), Poland, W. Koźmiński (Warszawa), Poland, D. Lurie (Aberdeen), UK, MacKay (Vancouver), Canada, B. Meier (Zurich), Switzerland, S. Vega (Rehovot), Israel

### Organizing Committee

Stefan Jurga - Director

L. Szutkowska – Executive secretary, R. Markiewicz, K. Szutkowski, J. Jencyk, Z. Pietralik, M. Taube, K. Golba, A. Klimaszuk

The AMPERE NMR School was held from 25<sup>th</sup> of June to 1<sup>st</sup> of July 2017 in Zakopane (Poland) with over than 85 attendees, including 50 from abroad. The conference was organized by the NanoBioMedical Centre and the Department of Macromolecular Physics, Faculty of Physics of Adam Mickiewicz University in Poznań under the auspices of the Groupement Ampere.



The meeting attracted participants from 23 countries (25 nationalities) across the world with an interest in the basic and advanced NMR techniques, recent research and cooperation between scientists. Participants represented various countries such as Canada, Denmark, Colombia, Czech Republic, China, Denmark, Estonia, Finland, France, Poland, Russian Federation, Slovenia, Spain, Slovakia, Sweden, Somalia, Ukraine, UK.

The programme of the School covered the following topics: NMR relaxometry, NMR diffusometry, Solid State NMR, NMR of quadrupolar nuclei, MRI and Field Cycling MRI, Novel NMR techniques and NMR in biology, medicine and material science. The Programme comprised 21 lectures, 5 trainings including online transmissions from the NanoBioMedical Centre in Poznan:

- HIGH RESOLUTION SOLID STATE NMR  
Zbigniew Fojud and Jacek Jencyk
- DIFFUSION STUDIED BY NMR  
Kosma Szutkowski
- MRI: BASIC PRINCIPLES AND APPLICATION  
Tomasz Zalewski
- TWO-DIMENSIONAL NMR SPECTROSCOPY  
Igor Żukow, Łukasz Popenda
- NMR RELAXOMETRY  
Zbigniew Fojud and Jacek Jencyk

The lectures were given by:

Prof. Bernhard Blümich, Prof. Vladimir Chizhik, Prof. Matthias Ernst, Prof. Isabelle Felli, Dr. Fabien Ferrage, Prof. Franz Fujara, Prof. Jadwiga Tritt-Goc, Prof Katalin Kover, Prof. Wiktor Koźmiński, Prof. Danuta Kruk, Prof. Ilya Kuprov, Prof. David Lurie, Prof. Alex MacKay, Prof. Beat Meier, Prof. Michel Dieter, Dr. Jurgen Schmidt, Dr. Jiří Spěvaček, Prof. Siegfried Stapf, Prof. Janez Stepišnik. Dr. Ville Telkki and Dr. David Topgaard.



The participants had the opportunity to present their results during poster sessions which comprised 42 presentations. The posters were evaluated by the members of "the poster committee" composed of: Prof. Franz Fujara, Prof. David Lurie, Prof. Ernst Matthias, Prof. Janez Stepišnik.

The prizes were "Ex aequo" awarded to:

**Joanna Kowalczuk**

The structural parameters of rigid matrix of saccharide-based gel described by short and long diffusion regime analysis.  
Institute of Molecular Physics, Poznań, Poland

**Raquel Saborano**

Metabolism in liver disease.  
University of Birmingham, United Kingdom

**Anne Selent**

Quantifying the adsorption of flowing gas mixtures in porous materials by remote detection NMR.  
University of Oulu, Oulu, Finland

The winners were given prizes and diplomas.  
All abstracts of oral presentations and posters were published as printed proceedings (book of abstracts, see: ampere-society.org).



The social programme included an "All together party", Dinner in Regional restaurant with the folk music of the Tatra's region, a Dunajec Rafting Excursion, an Excursion to Bielska Cave (Slovakia) and hiking in the Tatra Mountains. Participants also had the opportunity to listen to organ recital performed by Prof. Dieter Michel.

The School was a successful event, as reflected in correspondence received from the participants after the event.

**The next edition of the School will be held in Zakopane (Poland) from 10<sup>th</sup> to 16<sup>th</sup> June 2018.**

## The structural parameters of rigid matrix of saccharide-based gel described by short and long diffusion regime analysis.

Institute of Molecular Physics, Polish Academy of Sciences, ul. M. Smoluchowskiego 17, 60-179 Poznań, Poland.

### Introduction

The application of the theoretical analysis of porous materials to the experimental results of the diffusion coefficient of toluene in gel was the main idea of this work. In the experiment, the time dependent diffusion coefficient of toluene in methyl-4,6-O-(p-nitrobenzylidene)- $\alpha$ -D-glucopyranoside (GlucO-NO<sub>2</sub>) matrix was investigated. The results can be used to estimate the tortuosity ( $\tau$ ) and the pore surface-to-volume ( $S/V_{pore}$ ) ratio of a porous material [1-4].

### Methods

The time dependent effective diffusion coefficient  $D_{eff}(\Delta)$  measurements of toluene in the GlucO-NO<sub>2</sub> organogels network were carried out with a Bruker AVANCE pulse spectrometer operating at 300 MHz and equipped with magnetic field gradients. The diffusion coefficients were measured using the pulse gradient spin echo (PGSE) pulse sequence introduced by Stejskal and Tanner [5].

### Results

Fig.1a presents the time-dependent diffusion coefficients  $D_{eff}(\Delta)/D_0$  of toluene for 2.0, 3.0, 4.0, and 5.0 wt. % gel as a function of the gradient pulse interval  $\Delta$ . The change in behavior of  $D_{eff}(\Delta)/D_0$  for 3 wt.% gel at the time limit of  $\sim 0.1$  s is clearly visible in Fig. 1b. In the short-time regime 0 - 0.1 s, the  $D_{eff}(\Delta)/D_0$  vs.  $\Delta$  dependence is a function of surface-to-volume ratio of the pores  $S/V_p$  in the system according to an equation proposed by Mitra et al. [3], in the long-time regime 0.1 - 0.3 s. The knowledge of the  $D_{eff}(\Delta)/D_0$  vs.  $\Delta$  dependence enables one to find with a very good approximation the tortuosity of the porous system according to an equation reported in literature as  $D_{eff}(\Delta)/D_0 = 1/\tau$  [1]. A horizontal dashed line in the Fig. 1b defines as inverse of tortuosity and so-called tortuosity limit. The tortuosity limit  $1/\tau$  calculated as a fitting parameter of long time diffusion regime gives 0.67, 0.61, 0.56 and 0.41 for 2.0, 3.0, 4.0 and 5.0 wt.% gel, respectively. Based on the analysis of the behavior of the time-dependent diffusion coefficient in all samples, the threshold

times were designated as 89, 65, 58 and 40 ms for 2.0, 3.0, 4.0 and 5.0 wt.% gel, respectively.

Fig. 2a and 2b present the values of the  $S/V_p$  ratio and calculated pore size as a function of GlucO-NO<sub>2</sub> concentration, respectively. On the basis of the  $S/V_p$  parameter the spherical pore diameter  $d$  can be calculated by relations  $S/V_p = 6/d$ . The size of pores can be also calculated using the time threshold by the mean squared displacement  $\langle r^2 \rangle$  relationship  $\langle r^2 \rangle = 2\Delta_{th}D_0$  [6-8].

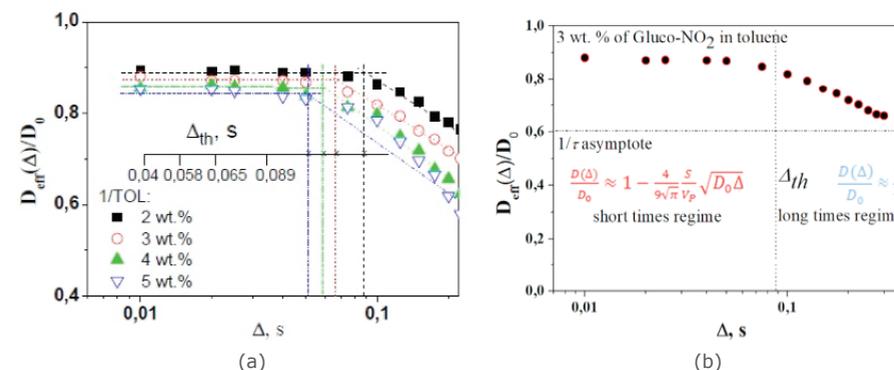


Fig.1. The time-dependent diffusion coefficient  $D_{eff}(\Delta)/D_0$  of toluene in the gel matrix of GlucO-NO<sub>2</sub> studied as a function of gelator concentration. The long and short diffusion regimes are separated by a threshold time  $\Delta_{th}$  which sets the time to change the slope of the measured diffusion values (a); The time-dependent diffusion coefficient  $D_{eff}(\Delta)/D_0$  ratio of toluene in the 3 wt.% GlucO-NO<sub>2</sub> concentration in gel (b)

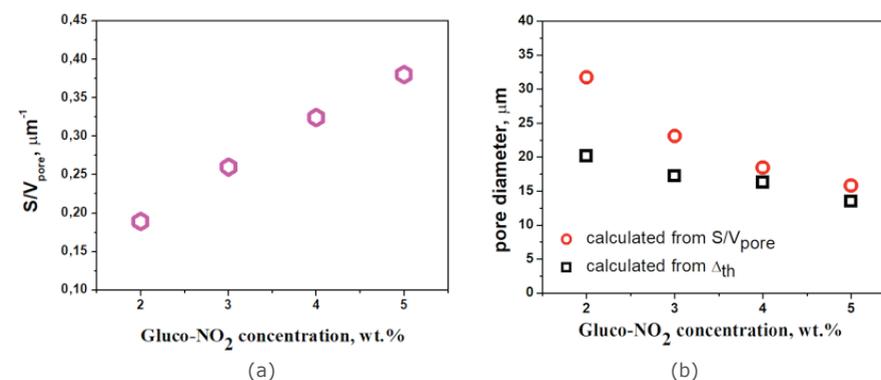


Fig.2. The  $S/V_p$  ratio (a) and pore diameter (b) as a function of GlucO-NO<sub>2</sub> concentration in gel.

On the basis of the obtained results, we can conclude that in the investigated gels the average pore sizes range from several to tens of micrometers. With a good approximation to a circular cross section shape the diameter of the barrier for diffused molecules in the gel can be calculated as  $b=6(\Phi^{-1}-1)V_p/S$  [9]. The organogel studied consists of a very large amount of toluene (the range from 95 to 98 wt.%) confined within a gel network composed from the gelator aggregates. In the range from 2.0 to 5.0 wt.% of gelator concentration in the samples the pore diameters  $d$  and porosity  $\Phi$  are changing about 5%. The increasing of diameter of the barrier  $b$  in the same range of gelator concentration in the samples are about 10 % (from 1.1  $\mu\text{m}$  to  $\sim 1.3 \mu\text{m}$  for 2.0% to 5.0 wt.% gel, respectively). The porosity of the samples decreases slowly as a function of gelator concentration with increasing of diameter of the element of rigid structure  $b$ . Such a situation is possible in systems in which the size of the aggregates forming the rigid matrix is at least several times smaller than the pore size. The tortuosity parameter suggests that the large aggregates formed from bundles of thin fibers in cross section have the irregular flattened shape similar to a multiple-star whose arms have different lengths.

#### Conclusion

In this work by theoretical analysis of diffusometry results the internal structure of the rigid matrix of a gel was described. The rigid matrix fibers thickness of 1.1-1.3  $\mu\text{m}$  and 30-15  $\mu\text{m}$  of the pore size (places filled with toluene) were calculated for gels in the range of 2.0 % to 5.0 % of Gluco-NO<sub>2</sub> concentration, respectively. The results corresponds very well with the optical microscopy image of 2.0 wt.% gel of Gluco-NO<sub>2</sub> in toluene published by M. Bielejewski et al. where the 1  $\mu\text{m}$  in thickness and 5 to 10  $\mu\text{m}$  in length fibers are clearly visible [10]. The details of this work were published in 2017 in the Journal of Materials Science [11].

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Poster Prizes Ampere NMR School 2017

**Raquel Saborano**

### **Metabolism in liver disease**

Raquel Saborano, Patricia Lalor, Michelle AC. Reed, Ulrich L. Günther  
University of Birmingham, B15 2TT Birmingham, United Kingdom

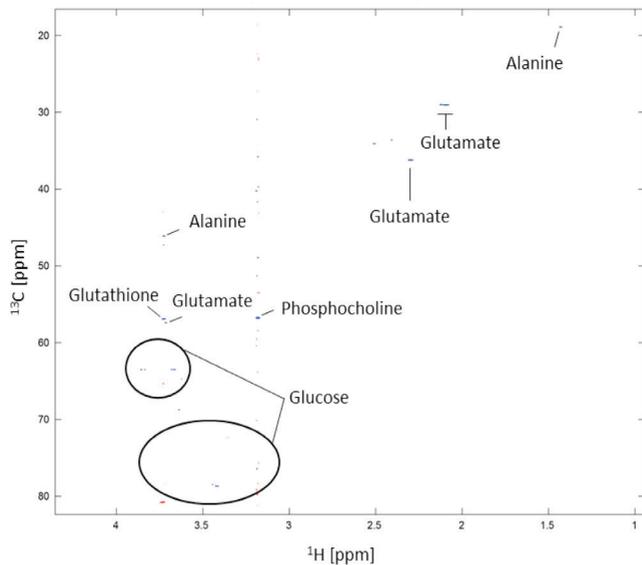
Non-alcoholic fatty liver disease affects a large portion of the world population and its incidence and prevalence are increasing to epidemic proportions. Metabolism has become an important new area in cell biology, giving information about biochemical processes in different pathologies. Metabolic regulation depends on the type of cell and its cellular environment, often controlled by cross-talk with stromal cells. In the case of the liver there is a number of cells interacting, which include hepatocytes (80% of the cell population), endothelial cells, Kupffer cells, stellate cells and others [1].

Analytical methods to study metabolism include mainly mass spectrometry and NMR. We have developed NMR methods for tracer-based metabolism where we feed cells <sup>13</sup>C-labelled metabolic precursors and use HSQC spectra to study site-specific label insertion in a series of metabolites [2].

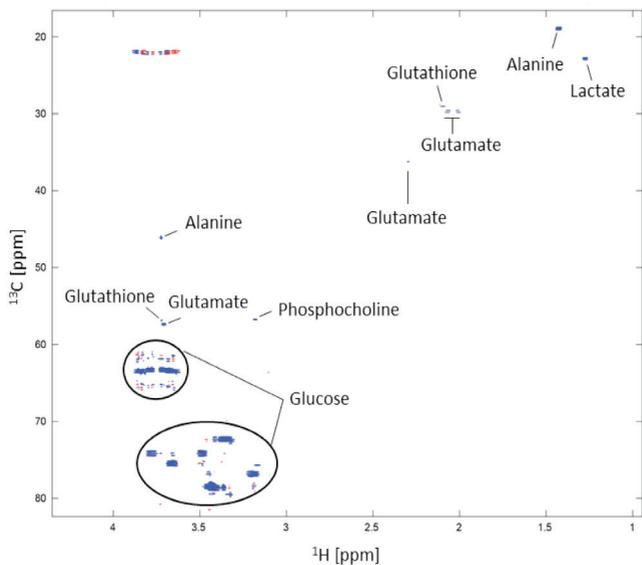
NMR has many advantages in this context and modern cryoprobes provide the sensitivity to work with mammalian cells. NMR spectra offer several parameters to study tracer-based metabolism, including intensities in <sup>13</sup>C-spectra or <sup>1</sup>H-<sup>13</sup>C HSQC spectra, but also line shapes and couplings. Coupling constants vary between different <sup>13</sup>C atoms and provide valuable structural information, coupling patterns can also be simulated to derive isotopomer ratios [3].

We have identified a number of <sup>13</sup>C-labelling schemes that can be used to study different metabolic mechanisms and describe NMR methods that can shed new light on metabolism in mammalian cell lines and primary cells.

Donor Liver



Donor Liver + 4.5H [U- $^{13}\text{C}$ ] Glucose



**Figure 1** –  $^1\text{H}$ - $^{13}\text{C}$  HSQC spectra of primary hepatocytes. Isolated hepatocytes from a human donor liver continue to be active and viable after being extracted from the organ. They were able to uptake the [U- $^{13}\text{C}$ ]Glucose and use it to produce alanine, lactate, glutamate, glutathione and phosphocholine, where  $^{13}\text{C}$  enrichment was observed.

References:

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## Quantifying the adsorption of flowing gas mixtures in porous materials by remote detection NMR

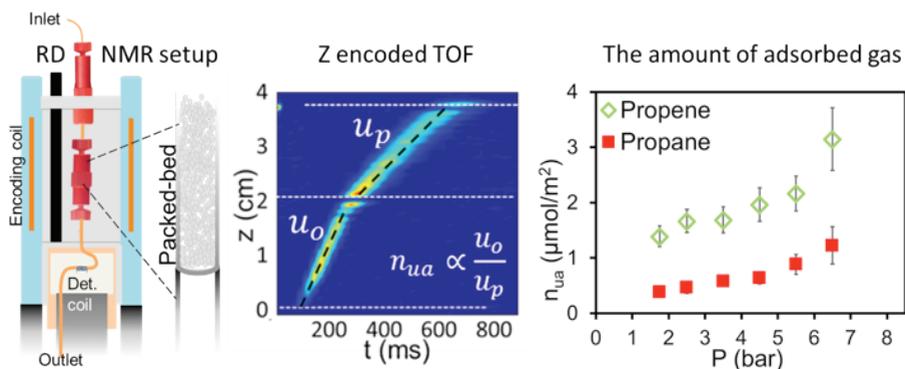
A. Selent,<sup>a</sup> V.V. Zhivonitko,<sup>b</sup> I.V. Koptug,<sup>b</sup> and V.V. Telkki<sup>a</sup>

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The development of efficient methods for gas adsorption studies is extremely important for a broad range of key technologies of modern society relying, e.g., on the large storage capacity of porous materials, heterogeneous catalysis and separation as well as purification of low-relative-volatility mixtures. Standard adsorption measurement techniques are slow and require an additional compositional analysis of non-adsorbed components of the gas mixture. We present a new, fast method for the characterization of competing adsorption of gas mixtures in porous media, based on spatially resolved remote detection time-of-flight nuclear magnetic resonance (RD TOF NMR).

In the study, gas flows through a powder-like porous material packed inside a capillary (called inlet capillary) close to the connection between the inlet and the outlet capillaries (see the figure below). Adsorption makes the average concentration of gas significantly higher in the sample area than in the outlet tubing, and therefore the average flow velocity in the sample region ( $u_p$ ) becomes lower than in the outlet tubing ( $u_o$ ). This can be clearly seen in the z-encoded TOF image as the slope in the sample region is much smaller than in the outlet capillary region (see the figure).



The novel adsorption measurement technique introduced uses the flow velocities, obtained from the slopes, to calculate the amount of adsorbed gas ( $n_{ua}$ ) based on mass balance [1]. The model states that the amount of adsorbed gas per unit of the surface area is directly proportional to the ratio of the observed velocities. An optimal experimental framework and theoretical model required for the investigations of single and multicomponent gases was developed and the obtained data was compared with standard measurements of gas adsorption isotherms. [2]

### References

[1] V. V. Zhivonitko, V.-V. Telkki, I. V. Koptug, *Angew. Chem. Int. Ed.* 51, 8054-8058 (2012)

[2] A. Selent, V. V. Zhivonitko, I. V. Koptug, V.-V. Telkki, Quantifying the adsorption of flowing gas mixtures in porous materials by remote detection NMR, (submitted for publication in *Micropor. Mesopor. Mat.*)

## Report on 14<sup>th</sup> ICMRM in Halifax 2017

The 14<sup>th</sup> ICMRM was organized under the auspices of the Spatially Resolved Magnetic Resonance (SRMR) Division of the Groupement AMPERE.

This year's conference was held on the campus of Dalhousie University in Halifax, Nova Scotia, Canada from August 13<sup>th</sup>-17<sup>th</sup>, 2017. The meeting attracted 180 delegates from 20 countries with an interest in MR spanning the engineering, physical and biological sciences.

### Conference Program:

The conference program comprised 2 plenary lectures, 4 educational lectures, 20 invited oral lectures, and 47 oral presentations selected by a review committee from submitted abstracts. In addition, 75 posters were presented during the two poster sessions. All posters were available for viewing throughout the conference. There were no parallel sessions.



The four educational lectures were presented by Sarah Codd (NMR Basics, Imaging, Flow and Diffusion), Jeff Dunn (Small Animal MRI/MRS), Andrew Webb (MR/MRI Hardware), and Dimitrios Sakellariou (Compact Magnetic Resonance Frontiers and Challenges). Guided by the SRMR Executive committee and the SRMR Division committee, this year's conference had a renewed emphasis on biomedical imaging. Invited lectures in many cases compared MRI to complementary microscopy methods. The 14<sup>th</sup> ICMRM included the "Colloquium on Mobile NMR", as with previous conferences. There were be two scientific sessions on "Mobile and Low field NMR", on Monday and Thursday.

### Erwin Hahn Lecture:

The ICMRM began with a plenary lecture given by R. Scott Hinks from GE Healthcare on Sunday afternoon. New this year was the Erwin Hahn lecture, named in honour of the late Erwin Hahn, one of the founders of magnetic resonance. An international selection panel chose Eiichi Fukushima to give the inaugural Erwin Hahn lecture. Unfortunately, an untimely bicycle accident prevented Eiichi from attending the conference. Bernhard Bluemich graciously stepped in at short notice to deliver a replacement plenary lecture.

### Competitions:

As with previous ICMRM conferences, a small group of young investigators were selected, based on reviewed abstracts, to present in a special session of the conference. The Paul Callaghan Young Investigator award, and a monetary prize, was given based on the best presentation, as judged by an expert panel. The 2017 Paul Callaghan Young Investigator winner is Jeffrey Simkins from Montana State University. The winner of the poster competition was Sarah Vashae from the University of New Brunswick, while the Image Beauty Competition was jointly won by Krzysztof Klodowski and Mick Mantle (pl. see following pages).

### Venue:

The conference was held in adjacent buildings on the Dalhousie University Campus in Halifax. The opening reception was sponsored by Bruker and held at one of Canada's most popular National Historic Sites, Citadel Hill. The conference dinner was sponsored by MR Solutions. The dinner was held in the Canadian Museum of Immigration at Pier 21 on the waterfront.

### Sponsorships:

The organizing committee is extremely grateful for the significant funding in the form of sponsorship from a number of sources including Bruker, MR Solutions, University of New Brunswick, Green Imaging Technologies/Oxford Instruments, Niumag, Cubresa, GE Healthcare, Royal Society of Chemistry, and Magritek. Without these sponsors the conference would not have been able to offer such an attractive academic and social programme.

### Local Organizing Committee:

Steven Beyea, James Rioux, Kimberly Brewer from Dalhousie University. Bruce Balcom, Igor Mastikhin, Ben Newling from the University of New Brunswick.

The Paul Callaghan Young Investigator award 2017

**Jeffrey Simkins**

## Oxygen Profile Characterization in Biofilm Systems Using $^{19}\text{F}$ Nuclear Magnetic Resonance Oximetry

JW Simkins, JD Seymour, KE Keepseagle and PJ Stewart, Chemical & Biological Engineering Department, Montana State University, Bozeman, MT, USA 59718

$^{19}\text{F}$  magnetic resonance has become a popular method in the medical field for quantifying oxygenation in blood, tissues, and tumors<sup>1-3</sup>. The technique, called  $^{19}\text{F}$  NMR oximetry, exploits the strong affinity of molecular oxygen for liquid fluorocarbon phases<sup>4</sup>, and the resulting strong linear dependence of  $^{19}\text{F}$  spin-lattice relaxation rate  $R_1$  on local oxygen concentration. The success of  $^{19}\text{F}$  NMR oximetry in clinical contexts naturally introduces the possibility of repurposing this method to measure oxygen in different systems.

Bacterial biofilms, aggregates of bacteria encased in a self-secreted matrix of metabolic products, are ubiquitous in industrial and clinical settings and, in both cases, oxygen gradients are a critical parameter in biofilm behavior. In the clinic, biofilms manipulate oxygen gradients to not only undermine host defenses but also dramatically reduce bacterial susceptibility to antibiotics, making them extremely difficult or impossible to treat<sup>5,6</sup>. For this reason, chronic wound oxygenation is known to be a significant predictor of clinical outcome<sup>7</sup>. In industry, regardless of whether biofilms are considered useful (e.g. wastewater treatment, bioremediation, or biofuel production) or deleterious (e.g. drinking water distribution systems, desalination, and food processing), oxygen represents one of the most important factors governing biofilm behavior. Optimization of such biofilm systems thus requires an in-depth understanding of how the biofilm establishes and responds to oxygen gradients.

However, measurement of oxygen distributions in biofilms is often cumbersome and in some cases intractable. In systems that permit it, such as a planar biofilm, the gold standard for oxygen quantification is the microelectrode, which is inserted to various depths within the biofilm and a 1D oxygen profile is constructed. The microelectrode has two hallmark drawbacks: first, the technique is limited to acquisition of one point at a time<sup>8</sup>, making it extremely time consuming, and second, insertion of the electrode is fundamentally invasive, and can cast doubt on obtained values<sup>9</sup>.

In systems such as a biofilm growing in a packed bed column, spatially-resolving oxygen concentration becomes unmanageable. NMR, entirely non-invasive and permitting spatial resolution, provides the potential to overcome these boundaries.

In the present work we develop methodology for quantifying oxygen distributions in different biofilm systems, focusing primarily on planar *Staphylococcus aureus* biofilms grown on agarose gel, a model of a chronic wound infection, and biofilms grown on packed bed columns, of which the porous geometry models, for instance, wastewater and soil biofilms. We explore the applicability of different fluorocarbons for different biofilm systems, based on properties such as signal density, fractional sensitivity to oxygen, emulsion stability, and volatility.

Construction of a calibration curve detailing the response of  $^{19}\text{F}$   $R_1$  to oxygen concentration is accomplished by bubbling gases of variable oxygen concentration through pure-phase fluorocarbon and allowing the system to reach equilibrium before measuring  $R_1$ <sup>3</sup>.  $R_1$  mapping is accomplished using inversion recovery in combination with a spin-echo imaging sequence. When a fluorocarbon with multiple peaks is used as the oxygen sensor, the excitation  $90^\circ$  pulse is used to chemically select the dominant spectral peak and the refocusing  $180^\circ$  pulse is used for the slice selection.

In the planar biofilm case, we demonstrate that injection of a stream of pure liquid hexafluorobenzene (HFB), a fluorocarbon with high signal density, into the growth medium immediately before measurement allows for observation of the oxygen-depth profile. Similarly, injection of a bolus of HFB into the medium records the exact oxygen concentration at that point. Following further development, this technique could be used in the clinic to evaluate oxygen penetration in chronic wounds, a metric which has been shown to be a strong predictor of clinical outcome.

For porous media biofilms, we incorporate the oxygen-sensing fluorocarbon into the matrix material itself. We use perfluorooctylbromide (PFOB), which readily forms stable emulsions, and incorporate the PFOB emulsion into 3 mm alginate beads by dripping an emulsion of PFOB-in-sodium alginate into a calcium chloride solution. In a subset of beads, biofilm growth is stimulated by inoculating with *Escherichia coli* before dripping. The effect of biofilm growth is investigated by packing a mixture of sterile and inoculated beads into a 10 mm inner diameter column and recording  $R_1$  profile (bulk, 1D longitudinal profile, 2D longitudinal, and 2D radial) over the course of microbial development. We correlate these measurements

with  $^1\text{H}$  velocity maps to yield, for the first time to our knowledge, a comprehensive comparison of fluid flow distribution and resulting oxygen distribution in a packed bed system. In addition to bacterial experiments, we also probe the timescale of oxygen depletion, both axially down the column and from within individual beads, when the transition is made from oxygen-saturated feed water to oxygen-depleted via nitrogen sparging. The PFOB bead system is modular, and can be modified as desired to alter conditions in the packed bed and observe subsequent changes on the system.

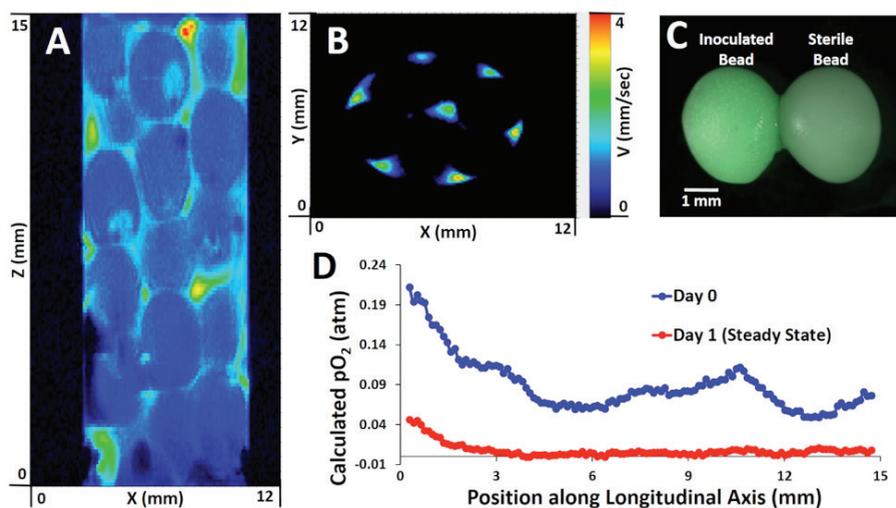


Fig 1. (A)  $^1\text{H}$  sagittal image of water flowing at 50mL/hr through a column containing PFOB-laden alginate beads. (B)  $^1\text{H}$  axial velocity map showing flow streamlines. (C) Fluorescent image of individual beads, with the inoculated bead exhibiting green fluorescence due to growth of *S. aureus*. (D) Longitudinal oxygen profile over course of bacterial growth calculated via  $^{19}\text{F}$   $R_1$  of PFOB.

Finally, we report progress toward encapsulation of hydrofluorether (HFE) in 300  $\mu\text{m}$  diameter alginate beads using a microfluidic device. HFE affords much higher signal-to-noise ratio than PFOB and is a frequently used fluorocarbon phase in microfluidic technologies. The small size and high signal afforded by these make them easy to incorporate into systems where oxygen levels are of concern and facilitate the measurement.

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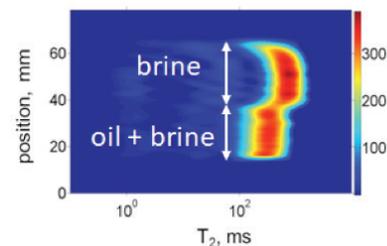
## Local diffusion and diffusion- $T_2$ distribution measurements in porous media

S Vashaee, B Newling, B MacMillan, F Marica, M Li and BJ Balcom, UNB MRI Centre, Department of Physics, University of New Brunswick, Fredericton, New Brunswick, E3B 5A3, Canada.

Slice-selective pulsed field gradient (PFG) and PFG- $T_2$  measurements are developed to measure spatially-resolved molecular diffusion and diffusion- $T_2$  distributions. A spatially selective adiabatic inversion pulse was employed for slice-selection. The slice-selective pulse is able to select a coarse slice, on the order of 1 cm, at an arbitrary position in the sample.

The new method can be employed to characterize oil-water mixtures in porous media. The new technique has an inherent sensitivity advantage over phase encoding imaging based methods due to signal being localized from a thick slice. The method will be advantageous for magnetic resonance of porous media at low field where sensitivity is problematic.

Experimental CPMG data, following PFG diffusion measurement, were compromised by a transient  $\Delta B_0(t)$  field offset. The off resonance effects of  $\Delta B_0(t)$  were examined by simulation. The  $\Delta B_0(t)$  offset artifact in D- $T_2$  distribution measurements may be avoided by employing real data, instead of magnitude data.



**Figure a**  
A  $T_2$  spatially-resolved map of a fully brine-saturated Bentheimer core plug injected with S6 oil, generated with SE-SPI method. The bottom of the sample (bottom of the image) includes a mixture of S6 oil and brine, but the top of the sample is still fully brine-saturated.

**Figure b**  
Adiabatic inversion SE-PFG- $T_2$  for slice-selective D- $T_2$  measurements of regions of interest.

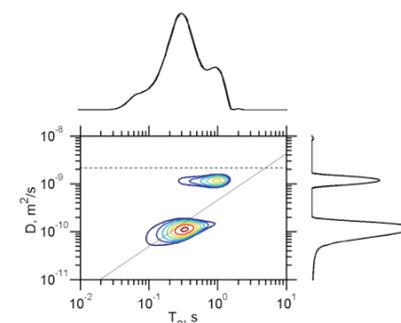
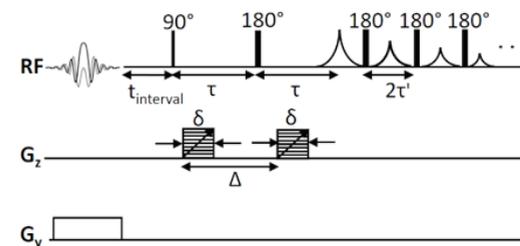


Fig. c

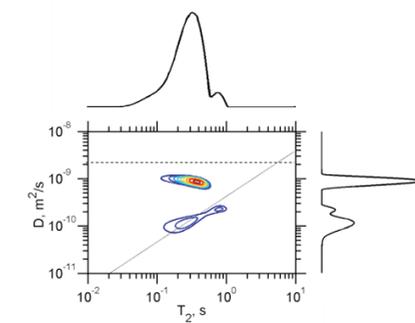


Fig. e

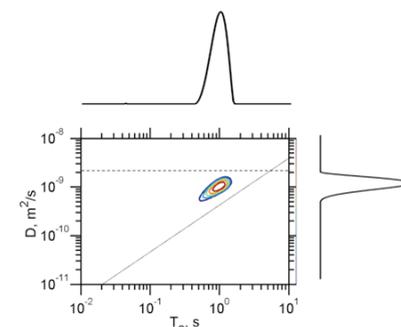
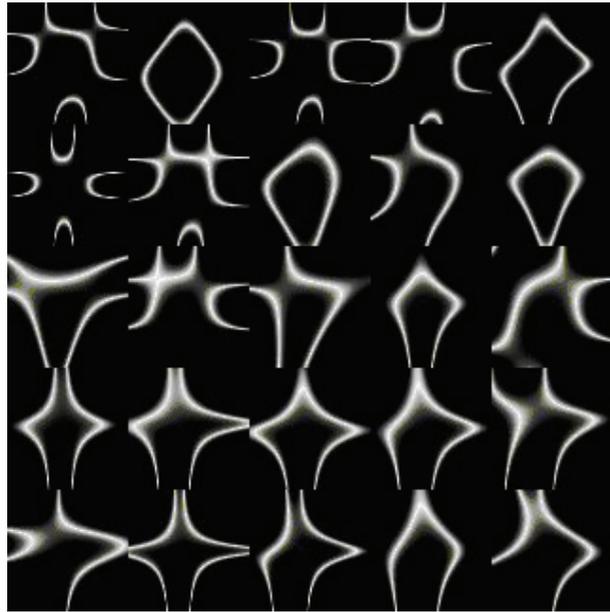


Fig. d

**Figure c**  
Bulk D- $T_2$  contour plot for S6 oil/brine-Bentheimer system, measured with bulk SE-PFG- $T_2$ . The brine and S6 oil are clearly separated.

**Figure d**  
The D- $T_2$  plot for a slice chosen from the bottom of the sample (bottom of the image) which includes both brine and S6 oil.

**Figure e**  
The D- $T_2$  plot for a slice chosen from the top of the sample which is fully brine-saturated.



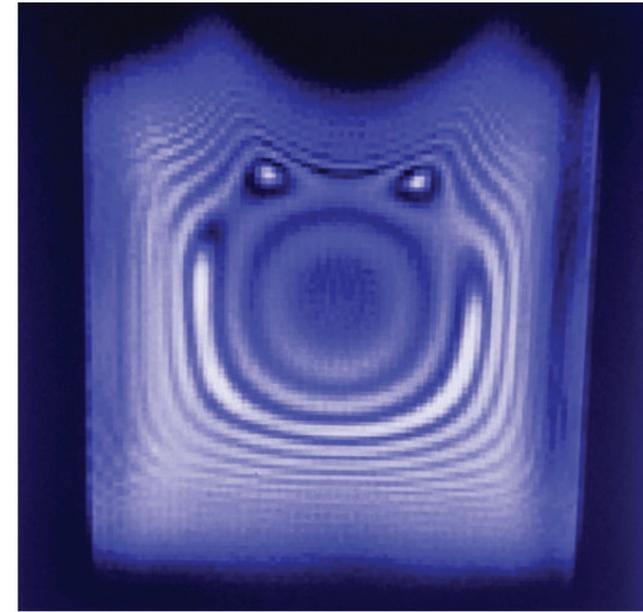
The image presents high contrast maps of the spatial inhomogeneities of the b-matrix elements derived through the BSD-DTI calibration, after bivariate polynomial fitting approximation. The origin of the image stems from the search for the best way of presentation of the data. In this case grey scale used together with very narrow window/level display pre-set resulted in a visually appealing, though not very informative picture. For more scientifically sound and useful visualization of the b-matrix inhomogeneities please refer to:

Approximation of the actual spatial distribution of the b-matrix in diffusion tensor imaging with bivariate polynomials, Kodowski K., Łukasik P., Krzyżak A. T., Proceedings of the 2016 Federated Conference in Computer Science and Information Systems, pp. 943-94. <https://annals-csis.org/proceedings/2016/pliks/fedcsis.pdf>

A general description of BSD-DTI can be found in:

Improving the accuracy of PGSE DTI experiments using the spatial distribution of b matrix, Krzyżak A. T., Olejniczak Z., Magnetic Resonance Imaging, 2015, 33(3), pp. 286-295.

DOI: <http://dx.doi.org/10.1016/j.mri.2014.10.007>



“An example when RARE imaging goes spookily wrong- pulse angle mayhem”.

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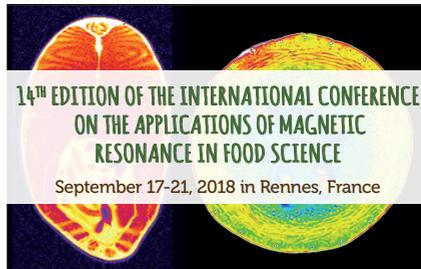
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The conference covers multiple aspects of the application of magnetic resonance to food



For the fourth time, the conference will be organized under the auspices of the groupement Ampere

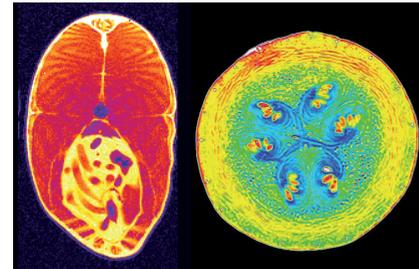


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## 14<sup>TH</sup> EDITION OF THE INTERNATIONAL CONFERENCE ON THE APPLICATIONS OF MAGNETIC RESONANCE IN FOOD SCIENCE

September 17-21, 2018 in Rennes, France



The conference covers multiple aspects of the application of magnetic resonance to food (including plants as food resources) and food products by presenting the latest innovations to understanding the functionality of foods, their processing, their stability and their impact on health and sensorial perception. Oral and poster presentations will deal with new techniques in low and high field NMR, quantitative NMR (qNMR), signal processing, food physics, postharvest and food technologies, foodomics, food authenticity, quality and safety as well as imaging and diffusometry.

## TENTATIVE PROGRAM

Details on the website [www.foodmr.org](http://www.foodmr.org)

MRIFood2018 is pleased to announce the presence of 7 invited speakers coming from all over the world (USA, Japon, Netherlands, Italy, France).

### Monday, sept. 17

12:00 ..... | Registration  
14:00 ..... | Welcome  
14:15 ..... | Tutorials  
17:30 ..... | Welcome Mixer

### Tuesday, sept. 18

9:00 ..... | Conference opening  
9:10-12:00 ..... | NMR/MRI development and signal processing  
12:15 ..... | Lunch, Posters  
13:30-18:00 ..... | Food Physics

### Wednesday, sept. 19

9:10-12:00 ..... | Postharvest Technologies  
12:15 ..... | Lunch, Posters  
13:30-18:00 ..... | Food Technologies

### Thursday, sept. 20

9:10-12:00 ..... | Foodomics and Food Chemistry (1<sup>st</sup> session)  
12:15-18:00 ..... | Excursion  
19:30 ..... | Conference dinner

### Friday, sept. 21

9:00-12:00 ..... | Foodomics and Food Chemistry (2<sup>nd</sup> session)  
12:00 ..... | Closing remarks

## ENJOY MORE...



Thursday, Sept. 20 : Visit of the Mont Saint Michel  
✓ [web.ot-montsaintmichel.com](http://web.ot-montsaintmichel.com)

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The entire city on foot from the congress center  
Easy to navigate on foot, with its many vehicle-phobic cobbled streets, Rennes old town offers its guests everything they could want once their day is over, where they can relax or enjoy or night out. A city centre where you can wander through the area surrounding the conference centre in a vibrant atmosphere of culture and leisure, with cinemas, theatres, concerts, medieval heritage, original boutiques, parks, luxury hotels, café terraces, restaurants...  
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Conference will be held at the conference centre "Le Couvent des Jacobins" located to the town city center. The "Convent des Jacobins" offers a large range of services that will ensure the success of the Conference.



First announcement

## EUROMAR & ISMAR joint conferences

August 25-30, 2019, Berlin



"Gathering the magnetic resonance community,  
displaying new scientific results,  
foreseeing future MR technologies"

## 2019 ISMAR & EUROMAR JOINT CONFERENCE

21<sup>ST</sup> ISMAR, 15<sup>TH</sup> EUROMAR,  
GDCh FGMR Discussion Meeting

August 25-30, 2019

Henry Ford Building, Freie Universität Berlin



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## Future conferences

### Ampere Events 2018

MRPM14 Magnetic Resonance in Porous Media	Gainesville (USA)	February 18-22 2018
15 <sup>th</sup> International Youth School-Conference ,Magnetic resonance and its applications, Spinus 2018	Saint Petersburg (Russia)	1-6 April 2018
Ampere NMR School 2018	Zakopane (Poland)	June 10-16, 2018
Euromar 2018	Nantes (France)	July 1-5, 2018
,HYP18` Hyperpolarized Magnetic Resonance 2018	Southampton (UK)	September 2-6 2018
FoodMR 2018	Rennes (France)	September 17-21 2018
Ampere Biological Solid-State NMR School	Palma de Mallorca (Spain)	October 21-26 2018

### Other Events 2018

SciX2018	Atlanta (USA)	October 21-26 2018
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### Ampere Events 2019

15 <sup>th</sup> ICMRM	Paris (France)	August 18-22 2019
ISMAR / Euromar 2019	Berlin (Germany)	August 25-30 2019