2D-qNMR and different qHNMR experiments (relaxometry, imaging, etc)

1. **Characterization of mango juice by high-resolution NMR, hyphenated NMR, and diffusion-ordered spectroscopy**

   ByDuarte, Iola F.; Goodfellow, Brian J.; Gil, Ana M.; Delgadillo, Ivonne

   From Spectroscopy Letters (2005), 38(3), 319-342.Language:English, Database: CAPLUS, DOI: 10.1081/SL-200058713

   The application of **NMR** spectroscopy, hyphenated **NMR**, and diffusion-ordered spectroscopy (DOSY) to the characterization of mango juice, as an example of a complex food mixt., is described. The compositional changes taking place as a function of ripening were followed, and selected metabolites were **quantified** by integration of the corresponding **NMR** peaks. In this way, an overall view of the metabolite changes is obtained, enabling the study of the biochem. mechanisms involved in the ripening process. More than 50 compds.were identified by 1D- and 2D-**NMR**, but many ambiguous assignments remain due to spectral overlap or insufficient coupling information. The use of liq. chromatog. (LC-**NMR**) and LC-**NMR**/mass spectrometry (MS) enables a fuller characterization of the sol. pectin fraction to be made; its dependence on ripening stage is discussed. Finally, DOSY adds information on the Mr of many metabolites, including the pectin fractions of ripe and unripe mango juices, and enables further peak assignments to be made.

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2. **Quantification of compartmented metabolic fluxes in developing soybean embryos by employing biosynthetically directed fractional 13C labeling, two-dimensional [13C, 1H]nuclear magnetic resonance, and comprehensive isotopomer balancing. [Erratum to document cited in CA142:034686]**

   BySriram, Ganesh; Fulton, D. Bruce; Iyer, Vidya V.; Peterson, Joan Marie; Zhou, Ruilian; Westgate, Mark E.; Spalding, Martin H.; Shanks, Jacqueline V.

   From Plant Physiology (2006), 142(4), 1771.Language:English, Database: CAPLUS

   All abs. fluxes reported in μmol d-1 cotyledon-1 (in Figure 6 and text) should be multiplied by the integer 3. This error occurred because the combined dry wt. for three cotyledons (instead of that for a single cotyledon) was used while converting relative fluxes output by the program NMR2Flux to abs. fluxes. The relative fluxes (reported in carbon and mol per 100 carbon mol of Suc uptake) and reaction reversibilities (reported in %) remain unchanged. On page 3045, left column, first full paragraph, line 10, and though out the rest of the paper, the minor hexose hydrolysis product referred to as "5-hydroxymethyl furfural (HMF)" should read "hydroxyacetone (HyA)". These errors do not affect any of the results, conclusion, or interpretations of the data in the article.

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3. **Quantification of compartmented metabolic fluxes in developing soybean embryos by employing biosynthetically directed fractional 13C labeling, two-dimensional [13C, 1H] nuclear magnetic resonance, and comprehensive isotopomer balancing**

   BySriram, Ganesh; Fulton, D. Bruce; Iyer, Vidya V.; Peterson, Joan Marie; Zhou, Ruilian; Westgate, Mark E.; Spalding, Martin H.; Shanks, Jacqueline V.

   From Plant Physiology (2004), 136(2), 3043-3057.Language:English, Database: CAPLUS, DOI: 10.1104/pp.104.050625

   Metabolic flux **quantification** in **plants** is instrumental in the detailed understanding of metab. but is difficult to perform on a systemic level. Toward this aim, we report the development and application of a computer-aided metabolic flux anal. tool that enables the concurrent evaluation of fluxes in several primary metabolic pathways. Labeling expts.were performed by feeding a mixt. of U-13C Suc, naturally abundant Suc, and Gln

to developing soybean (Glycine max) embryos. Two-dimensional [13C, 1H]**NMR** spectra of seed storage protein and starch hydrolyzates were acquired and yielded a labeling data set consisting of 155 13C isotopomer abundances. We developed a computer program to automatically calc. fluxes from this data. This program accepts a user-defined metabolic network model and incorporates recent math. advances toward accurate and efficient flux evaluation. Fluxes were calcd. and statistical anal. was performed to obtain SDS. A high flux was found through the oxidative pentose phosphate pathway (19.99±4.39 μmol d-1 cotyledon-1, or 104.2 carbon mol. ± 23.0 carbon mol. per 100 carbon mol. of Suc uptake). Sep. transketolase and transaldolase fluxes could be distinguished in the plastid and the cytosol, and those in the plastid were found to be at least 6-fold higher. The backflux from triose to hexose phosphate was also found to be substantial in the plastid (21.72±5.00 μmol d-1 cotyledon-1, or 113.2 carbon mol. ±26.0 carbon mol. per 100 carbon mol. of Suc uptake). Forward and backward directions of anaplerotic fluxes could be distinguished. The glyoxylate shunt flux was found to be negligible. Such a generic flux anal.tool can serve as a **quant**. tool for metabolic studies and phenotype comparisons and can be extended to other **plant** systems.

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4. **Disintegration efficiency of pulsed electric field induced effects on onion (*Allium cepa* L.) tissues as a function of pulse protocol and determination of cell integrity by 1H-NMRrelaxometry**

ByErsus, Seda; Oztop, MecitHalil; McCarthy, Michael J.; Barrett, Diane M.

From Journal of Food Science (2010), 75(7), E444-E452.Language:English, Database: CAPLUS, DOI: 10.1111/j.1750-3841.2010.01769.x

The influence of elec. pulse protocol parameters on cell rupture of onion tissues was investigated in order to improve fundamental understanding and to enhance the processing of **plant** tissues with pulsed elec. fields (PEFs). The impact of PEF parameters on cell integrity of 20 mm dia, 4-mm thick disks of Don Victor onions (*Allium cepa L*.) was detd. by ion leakage measurements. Elec. field strength, pulse width, total pulse duration, and frequency effects were detd. in relation to their effects on cell damage as a function of pulse protocol. Elec. field strengths up to 500 V/cm increased the damage efficiency but there was no significant difference in efficiency beyond this field strength. Larger pulse widths increased the degree of tissue disintegration at a const. pulse no. Higher PEF efficiency was achieved with shorter pulse widths and a larger no. of pulses at a const. total treatment time. Lower frequencies caused a greater degree of disintegration at const. no. of pulses. 1H-**NMR**expts.were performed to det. the proton relaxation components of the PEF-treated onion samples and to obtain cell damage information nondestructively. Paramagnetic ion uptake by the onion sample was used to identify different proton relaxation components. Five different proton relaxation components were obsd. and changes in the 2 components representing different proton environments showed high correlations with ion leakage results (R2 = 0.99), indicating that T2 distributions can be used to obtain information about cell membrane integrity in PEF-treated samples. 1H-**NMR** proved to be an effective method for nondestructive **quantification** of cell membrane rupture in onions.

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5. **1H-NMR study of the impact of high pressure and thermal processing on cell membrane integrity of onions**

ByGonzalez, Maria E.; Barrett, Diane M.; McCarthy, Michael J.; Vergeldt, Frank J.; Gerkema, Edo; Matser, Ariette M.; Van As, Henk

From Journal of Food Science (2010), 75(7), E417-E425.Language:English, Database: CAPLUS, DOI: 10.1111/j.1750-3841.2010.01766.x

Proton **NMR** (1H-**NMR**) relaxometry was used to study the effects of high pressure and thermal processing on membrane permeability and cell compartmentalization, important components of **plant** tissue texture. High pressure treated onions were subjected to pressure levels from 20 to 200 MPa at 5 min hold time at initial temps. of 5 and 20 °C. Thermally treated onions were exposed for 30 min at temps. from 40 to 90 °C. Loss of membrane integrity was clearly shown by changes in transverse relaxation time (T2) of water at

temps. of 60 °C and above. Destabilization effects on membranes exposed to high pressure were obsd. at 200 MPa as indicated by T2 measurements and cryo-SEM (Cryo-SEM). T2 relaxation successfully discriminated different degrees of membrane damage based on the T2 shift of the vacuolar component. Analyses of the av. water self-diffusion coeff. indicated less restricted diffusion after membrane rupture occurred in cases of severe thermal treatments. Milder processing treatments yielded lower av. diffusion coeffs. than the controls. 1H-**NMR** proved to be an effective method for **quantification** of cell membrane damage in onions and allowed for the comparison of different food processes based on their impact on tissue integrity.

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6. **Chemical Structure and Heterogeneity Differences of Two Lignins from Loblolly Pine As Investigated by Advanced Solid-State NMR Spectroscopy**

ByHoltman, Kevin M.; Chen, Na; Chappell, Mark A.; Kadla, John F.; Xu, Ling; Mao, Jingdong

From Journal of Agricultural and Food Chemistry (2010), 58(18), 9882-9892.Language:English, Database: CAPLUS, DOI: 10.1021/jf101258x

Advanced solid-state **NMR** was employed to investigate differences in chem. structure and heterogeneity between milled wood lignin (MWL) and residual enzyme lignin (REL). Wiley and conventional milled woods were also studied. The advanced **NMR** techniques included 13C **quant**. direct polarization, various spectral-editing techniques, and two-dimensional 1H-13C heteronuclear correlation **NMR** with 1H spin diffusion. The 13C chem. shift regions between 110 and 160 ppm of two lignins were quite similar to those of two milled woods. REL contained much more residual carbohydrates than MWL, showing that MWL extn. more successfully sepd. lignin from cellulose and hemicelluloses than REL extn.; REL was also of higher COO, arom. C-C, and condensed aroms. but of lower arom. C-H. At a spin diffusion time of 0.55 ms, the **magnetization** was equilibrated through the whole structure of MWL lignin, but not through that of REL, indicating that REL is more heterogeneous than MWL.

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7. **Non-destructive quantification of water gradient in sludge composting with Magnetic Resonance Imaging**

ByDuval, F. P.; Quellec, S.; Tremier, A.; Druilhe, C.; Mariette, F.

From Waste Management (Oxford, United Kingdom) (2010), 30(4), 610-619.Language:English, Database: CAPLUS, DOI: 10.1016/j.wasman.2009.09.045

Sludge from a slaughter-house wastewater **plant**, and mixts.of bulking agent (crushed wood pallet) and sludge were studied by **NMR** (**NMR**). The **NMR** spin-spin relaxation (T2) and spin-lattice relaxation (T1) signals for sludge, wet crushed wood pallet and mixts. of sludge and bulking agent were decompd. into three relaxation time components. Each relaxation time component was explained by a non-homogeneous water distribution on a microscopic length scale and by the porosity of the material. For all samples, the T2 relaxation time value of each component was directly related to the dry matter content. The addn. of wet crushed wood to sludge induced a decrease in the relaxation time, explained by water transfer between the sludge and the wood. **MagneticResonance** Imaging (MRI) and respirometric measurements were performed on sludge and wood mixts. MR images of the mixts. were successfully obtained at different biodegrdn. states. Based on specific **NMR** measurements in an identified area located in the MRI cells, the results showed that gray levels of MR images reflected dry matter content. This preliminary study showed that MRI would be a powerful tool to measure water distribution in sludge and bulking agent mixts. and highlights the potential of this technique to increase the understanding of sludge composting.

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8. **White-rot fungus-mediated degradation of the analgesic ketoprofen and identification of intermediates by HPLC-DAD-MS and NMR**

ByMarco-Urrea, Ernest; Perez-Trujillo, Miriam; Cruz-Morato, Carles; Caminal, Gloria; Vicent, Teresa

From Chemosphere (2010), 78(4), 474-481.Language:English, Database: CAPLUS, DOI: 10.1016/j.chemosphere.2009.10.009

Ketoprofen is a nonsteroidal anti-inflammatory drug that has been detected in the environment in the range of ng L-1-μg L-1 due to its low degradability in some wastewater treatment **plants**. In this study, the use of the white-rot fungus *Trametesversicolor* to effectively degrade ketoprofen in a defined liq. medium was assessed. The fungus eliminated ketoprofen to nondetectable levels in 24 h when it was added at 10 mg L-1 whereas at low concn. of 40 μg L-1 it was almost completely removed (95%) after 5 h. Low extracellular laccase activity was detected in the *T. versicolor* cultures but the addn. of the laccase-mediator system did not lead to ketoprofenoxidn. The cytochrome P 450 inhibitor 1-aminobenzotriazole reduced ketoprofenoxidn. These data suggest that the first oxidn. step is cytochrome P 450 mediated. During time-course degrdn.expts., three intermediates were structurally elucidated and **quantified** by HPLC-DAD-MS and **NMR**: 2-[3-(4-hydroxybenzoyl)phenyl]-propanoic acid, 2-[(3-hydroxy(phenyl)methyl)phenyl]-propanoic acid, and 2-(3-benzoyl-4-hydroxyphenyl)-propanoic acid. The latter was reported for the first time in biol. systems. After 7 d of incubation, only small amts. of 2-[(3-hydroxy(phenyl)methyl)phenyl]-propanoic acid (0.08 mg) remained in the liq. medium in comparison with the initial ketoprofen dose (1.0 mg), suggesting possible mineralization of ketoprofen.

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9. **Two-dimensional J-resolved NRM spectroscopy: review of a key methodology in the metabolomics toolbox**

ByLudwig, Christian; Viant, Mark R.

From Phytochemical Analysis (2010), 21(1), 22-32.Language:English, Database: CAPLUS, DOI: 10.1002/pca.1186

A review. One-dimensional (1D) 1H **NMR** spectroscopy remains a leading anal. technol. in metabolomics. Advantages of this approach include relatively rapid spectral acquisition and **NMRresonances** that provide a direct measure of metabolite concn. based upon a single internal std. Severe spectral congestion can, however, significantly hinder both metabolite identification and **quantification**. Two-dimensional 1H -resolved (JRES) **NMR** spectroscopy retains many of the benefits of 1D **NMR**, but addnl. disperses the overlapping **resonances** into a second dimension, reducing congestion and increasing metabolite specificity. The usefulness of this approach to metabolomics was first realized 6 years ago, and since then it was used in biol., medical and environmental studies of **plants** and animals. Here the authors provide a basic introduction to the 2D JRES **NMR** expt. and then discuss strategies for spectral acquisition and processing in the context of metabolomics applications, concluding with some key recommendations: acquisition using a double spin-echo sequence with excitation sculpting; processing using the SEM window function, tilting and symmetricising, optionally followed by a skyline projection. Strategies for implementing JRES spectroscopy into the metabolomics toolbox are then considered, including its roles in metabolic fingerprinting, metabolite identification and metabolite **quantification**. Public resources and data stds. for JRES metabolomics are reviewed. The authors conclude by evaluating the advantages (**e**.g. increased spectral dispersion and confidence in metabolite identification; fully automated processing; reduced batch-to-batch variation) and disadvantages (**e**.g. longer acquisition times; higher tech. variability; phase-twisted lineshapes resulting in **quantification** errors) of 2D JRES **NMR**vs the established 1D approach for metabolomics. Copyright © 2009 John Wiley & Sons, Ltd.

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10. **Monitoring the postharvest ripening of tomato fruit using quantitative MRI and NMRrelaxometry**

ByMusse, Maja; Quellec, Stephane; Cambert, Mireille; Devaux, Marie-Francoise; Lahaye, Marc; Mariette, Francois

From Postharvest Biology and Technology (2009), 53(1-2), 22-35.Language:English, Database: CAPLUS, DOI: 10.1016/j.postharvbio.2009.02.004

MagneticResonance Imaging (MRI) was performed on tomato fruit during two 3-wk periods of postharvest ripening. Different image types were acquired to study macroscopic and microscopic structural changes. Air spaces were identified close to seeds and their shrinkage during the ripening period was estd. from the spin echo images. The development of the bubbles in the outer pericarp during ripening was estd. from the ratio of the long- and short-echo time gradient echo MRI images and supported by the macrovision imaging. Variations in the transverse (T2) and longitudinal (T1) relaxation times were detd. from**quant**. MRI images. They depended on the tissue type and matched fairly well between fruit. In the core, placenta, radial and outer pericarp, T2 decreased by about 25% from the initial values and T 1 by about 25-30% from the initial values during postharvest ripening. In the locular tissue the relaxation times had less marked trends than in other tissues: both T2 and T1 increased slightly until the eighth or ninth measurement day and after that it returned to its approx. initial value. Multi-component characteristics of T 2 and T1 decay were investigated by **NMR**relaxometry. They provided information about all major sub-cellular compartments and showed there was water redistribution among compartments during ripening. In addn. to the relaxometry measurements, water content, wt. loss and concn.of neutral sugars and acids were measured on some of the tomato fruit. Cell size and organization were investigated by macrovisionexpts. Although the overall dependence of the relaxation time on tissue type was to some extent explained by chem. compn.and cell dimension, no relationships between trends in MR data and tissue properties were established.

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11. **Effect of Severity on Hydrotreating of Demetallized Oil Monitored by 1H-NMR Spectroscopy**

ByGuzman, A.; Alvarez, M. C.; Nunez, M. L.

From Petroleum Science and Technology (2009), 27(8), 845-860.Language:English, Database: CAPLUS, DOI: 10.1080/10916460802455244

A study on arom.hydrogenation of Demetallized oil was carried out using a com. catalyst under pilot **plant** reaction conditions similar to those found in industrial processes. The feedstock was contacted with the catalysts in a trickled bed reactor unit at 330°, 350°, and 370°. A combination of physicochem.characterization of feed and products and 1H-**NMR** spectra was used to monitor changes in the arom. fractions caused by variation in reaction temp. Anal.of the 1H-**NMR** spectra, along with the **quant**. variation in the areas of the **resonance** lines, showed that the diaroms. with relatively long alkyl changes present in the lightest distn. cuts of the products were highly hydrogenated. In contrast, smaller changes in aromaticity in the heaviest fractions were obsd. under the same conditions. A limit of ∼2% of the integrals corresponding to the diarom.+ species suggests a thermodynamical limitation of hydrogenation under the studied reaction conditions.

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12. **Rapid and novel discrimination and quantification of oleanolic and ursolic acids in complex plant extracts using two-dimensional nuclear magnetic resonance spectroscopy-Comparison with HPLC methods**

ByKontogianni, Vassiliki G.; Exarchou, Vassiliki; Troganis, Anastassios; Gerothanassis, Ioannis P.

From AnalyticaChimicaActa (2009), 635(2), 188-195.Language:English, Database: CAPLUS, DOI: 10.1016/j.aca.2009.01.021

A novel strategy for **NMR** anal.ofmixts. ofoleanolic and ursolic acids that occur in natural products is described. These important phytochems.have similar structure and their discrimination and **quantification** is rather difficult. We report herein the combined use of proton-carbon heteronuclear single-quantum coherence (1H-13C HSQC) and proton-carbon heteronuclear multiple-bond correlation (1H-13C HMBC) **NMR** spectroscopy, in the identification and **quantitation** of oleanolic acid (OA) and ursolic acid (UA) in **plant**exts. of the Lamiaceae and Oleaceae family. The combination of 1H-13C HSQC and 1H-13C HMBC techniques allows the connection of the proton and carbon-13 spins across the mol. backbone resulting in the identification and, thus, discrimination of oleanolic and ursolic acid without resorting to physicochem. sepn. of the components. The **quant**. results provided by 2D 1H-13C HSQC **NMR** data were obtained within

a short period of time (~14 min) and are in excellent agreement with those obtained by HPLC, which support

the efficiency of the suggested methodol.

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13. **Quantitative NMRI studies of transient washwater addition to rising foam**

ByStevenson, Paul; Mantle, Mick D.; Tayler, Alexander B.; Sederman, Andrew J.

From Chemical Engineering Science (2009), 64(5), 1001-1008.Language:English, Database: CAPLUS, DOI: 10.1016/j.ces.2008.10.057

For the first time, **NMR** Imaging was used to provide non-invasive **quant**. data for transient washwater addn. to rising foam. Washwater is routinely added to flotation froths to aid rejection of unwanted gangue material from the conc. stream. The results show that washwater added to a mature foam (i**.e.**, one that has attained its equil. liq. fraction) travels down the column, whereas washwater added to an immature foam travels up the column. This observation has important implications for flotation **plant**practise; washwater added too early at start-up will not aid gangue rejection but will instead merely lead to a wetter conc. stream. This is explained theor. in the context of the hydrodynamic theory of rising foam.

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14. **Quantitative high-resolution online NMR spectroscopy in pharmaceutical reaction and process monitoring**

ByMaiwald, M.; Steinhof, O.; Sleigh, C.; Bernstein, M.; Hasse, H.

Edited byHolzgrabe, Ulrike; Wawer, Iwona; Diehl, Bernd

From NMR Spectroscopy in Pharmaceutical Analysis (2008), 471-491.Language:English, Database: CAPLUS, DOI: 10.1016/B978-0-444-53173-5.00020-2

A review.**Quant**. high-resoln.online**NMR** spectroscopy is the method of choice for investigating complex reacting mixts. We describe the use of **NMR** flow cells for pharmaceutical reaction and process monitoring where reactions and processes can be covered from several hours down to minutes.

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15. **Quantitative Analysis of Constituents in Heavy Fuel Oil by 1H Nuclear Magnetic Resonance (NMR) Spectroscopy and Multivariate Data Analysis**

ByNielsen, KatrineEllemann; Dittmer, Jens; Malmendal, Anders; Nielsen, Niels Chr.

From Energy & Fuels (2008), 22(6), 4070-4076.Language:English, Database: CAPLUS, DOI: 10.1021/ef800539g

Characterization of heavy fuel oil (HFO) is highly important to ensure tech., economically, and environmentally proper operation of the engines and power **plants** that use this source of energy. This applies in particular to the shipping industry. Here, we demonstrate that the combination of std. 1H **NMR**spectroscopy and multivariate data anal. can be employed for quick and accurate extn. of parameters pertaining to the phys. and chem. properties of complex suspensions, such as HFO. For 82 HFO samples of known origin, good prediction models were obtained for a large no. of characterization parameters, including the calcd. aromaticity index, the d., gross and net calorific values, and water and sulfur contents, as well as micro-carbon residue.

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16. **Quantitative imaging of oil storage in developing crop seeds**

ByNeuberger, Thomas; Sreenivasulu, Nese; Rokitta, Markus; Rolletschek, Hardly; Goebel, Cornelia; Rutten, Twan; Radchuk, Volodja; Feussner, Ivo; Wobus, Ulrich; Jakob, Peter; et al

From Plant Biotechnology Journal (2008), 6(1), 31-45.Language:English, Database: CAPLUS, DOI: 10.1111/j.1467-7652.2007.00294.x

In this article, we present a tool which allows the rapid and non-invasive detection and **quant**. visualization of lipid in living seeds at a variety of stages using frequency-selected **magneticresonance** imaging. The method provides **quant**. lipid maps with a resoln. close to the cellular level (in-plane 31 µ**m**× 31 µ**m**). The reliability of the method was demonstrated using two contrasting subjects: the barley grain (monocot, 2% oil, highly compartmentalized) and the soybean grain (dicot, 20% oil, economically important oilseed). Steep gradients in local oil storage were defined at the organ- and tissue-specific scales. These gradients were closely coordinated with tissue differentiation and seed maturation, as revealed by electron microscopy and biochem. and gene expression anal. The method can be used to elucidate similar oil accumulation processes in different tissues/organs, as well as to follow the fate of storage lipids during deposition and subsequent mobilization.

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17. **Advances of high-resolution NMR techniques in the structural and metabolic analysis of plant biochemistry**

ByEisenreich, Wolfgang; Bacher, Adelbert

From Phytochemistry (Elsevier) (2007), 68(22-24), 2799-2815.Language:English, Database: CAPLUS, DOI: 10.1016/j.phytochem.2007.09.028

A review. Rapid progress in instrumentation and software made **NMR** spectroscopy (**NMR**) one of the most powerful anal. methods in biol. sciences. Whereas the development of multidimensional **NMR** pulse sequences is an ongoing process, a small subset of two-dimensional **NMR**expts. is typically sufficient for the rapid structure detn. of small metabolites. The use of sophisticated three- and four-dimensional **NMR**expts.enables the detn. of the three-dimensional structures of proteins with a mol. wt. up to 100 kDa, and soln. structures of more than 100 **plant** proteins have been established by **NMR** spectroscopy. **NMR** has also been introduced to the emerging field of metabolomics where it can provide unbiased information about metabolite profiles of **plant**exts. In recent times, high-resoln.**NMR** has become a key technol. for the elucidation of biosynthetic pathways and metabolite flux via **quant**. assessment of multiple isotopologs. This review summarizes some of the recent advances of high-resoln. **NMR** spectroscopy in the field of **plant** sciences.

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18. **Method for Determining Molar Concentrations of Metabolites in Complex Solutions from Two-Dimensional 1H-13C NMR Spectra**

ByLewis, Ian A.; Schommer, Seth C.; Hodis, Brendan; Robb, Kate A.; Tonelli, Marco; Westler, William M.; Sussman, Michael R.; Markley, John L.

From Analytical Chemistry (Washington, DC, United States) (2007), 79(24), 9385-9390.Language:English, Database: CAPLUS, DOI: 10.1021/ac071583z

One-dimensional (1D) 1H **NMR** spectroscopy is used extensively for high-throughput anal. of metabolites in biol. fluids and tissue exts. Typically, such spectra are treated as multivariate statistical objects rather than as collections of **quantifiable** metabolites. The authors report here a two-dimensional (2D) 1H-13C **NMR**

strategy (fast metabolite **quantification**, FMQ, by **NMR**) for identifying and **quantifying** the ~40 most

abundant metabolites in biol. samples. To validate this technique, the authors prepd. mixts.of synthetic compds. andexts. from*Arabidopsis thaliana*, *Saccharomyces cerevisiae*, and *Medicago sativa*. The authors show that accurate (tech. error 2.7%) molar concns. can be detd. in 12 min using their **quant**. 2D 1H-13C **NMR** strategy. In contrast, traditional 1D 1H **NMR** anal.resulted in 16.2% tech. error under nearly ideal conditions. The authors propose FMQ by **NMR** as a practical alternative to 1D 1H **NMR** for metabolomics studies in which 50-mg (ext. dry wt.) samples can be obtained.

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19. **Flux quatification in central carbon metabolism of *Catharanthusroseus* hairy roots by 13C labeling and comprehensive bondomer balancing**

BySriram, Ganesh; Fulton, D. Bruce; Shanks, Jacqueline V.

From Phytochemistry (Elsevier) (2007), 68(16-18), 2243-2257.Language:English, Database: CAPLUS, DOI: 10.1016/j.phytochem.2007.04.009

Methods for accurate and efficient **quantification** of metabolic fluxes are desirable in **plant** metabolic engineering and systems biol. Toward this objective, the authors introduce the application of "bondomers", a computationally efficient and intuitively appealing alternative to the commonly used isotopomer concept, to flux evaluation in **plants**, by using *Catharanthusroseus* hairy roots as a model system. The authors cultured the hairy roots on (5% wt./wt. U-13C, 95% wt./wt. naturally abundant) sucrose, and acquired two-dimensional [13C, 1H] and [1H, 1H]**NMR** spectra of hydrolyzed aq. ext. from the hairy roots. Anal.of these spectra yielded a data set of 116 bondomers of β-glucans and proteinogenic amino acids from the hairy roots. Fluxes were evaluated from the bondomer data by using comprehensive bondomer balancing. The authors identified most fluxes in a three-compartmental model of central carbon metab. with good precision. The authorsobsd. parallel pentose phosphate pathways in the cytosol and the plastid with significantly different fluxes. The anaplerotic fluxes between phosphoenolpyruvate and oxaloacetate in the cytosol and between malate and pyruvate in the mitochondrion were relatively high (60.1±2.5 mol per 100 mol sucrose uptake, or 22.5±0.5 mol per 100 mol mitochondrial pyruvate dehydrogenase flux). The development of a comprehensive flux anal.tool for this **plant** hairy root system is expected to be valuable in assessing the metabolic impact of genetic or environmental changes, and this methodol. can be extended to other **plant** systems.

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20. **Substrate cycles in the central metabolism of maize root tips under hypoxia**

ByAlonso, Ana Paula; Raymond, Philippe; Rolin, Dominique; Dieuaide-Noubhani, Martine

From Phytochemistry (Elsevier) (2007), 68(16-18), 2222-2231.Language:English, Database: CAPLUS, DOI: 10.1016/j.phytochem.2007.04.022

Substrate cycles, also called "futile" cycles, are ubiquitous and lead to a net consumption of ATP which, in the normoxic maize root, have been estd. at about 50% of the total ATP produced. To evaluate their role, the authors studied the substrate cycles of maize root tips under an oxygen limitation of respiration (3% O2). Short-time labeling expts.with [U-14C]-Glc were performed to **quantify** the fluxes through sucrose and starch cycles of synthesis and degrdn. Steady-state labeling with [1-13C]-Glc followed by 1H **NMR** and 13C **NMR** anal. of sugars and free alanine was used to **quantify** fluxes in the central metabolic pathways, including the Glc-P/Glc cycle and the fructose-P/triose-P cycle of glycolysis. Comparison with results previously obtained in normoxia [Alonso et al., as mentioned above] showed that 3% O2 induced fermn. and reduced respiration, which led to a lesser amt. of ATP produced. The rates of Glc consumption, glycolytic flux and all substrate cycles were lower, but the proportion of ATP consumed in the substrate cycles remained unchanged. These findings suggest that substrate cycles are not a luxury but an integral part of the organization of the **plant** central metab.

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21. **Microchemical analysis of laser-microdissected stone cells of Norway spruce by cryogenic nuclear magnetic resonance spectroscopy**

ByLi, Sheng-Hong; Schneider, Bernd; Gershenzon, Jonathan

From Planta (2007), 225(3), 771-779.Language:English, Database: CAPLUS, DOI: 10.1007/s00425-006-0376-z

Stone cells (sclereids) in Norway spruce (*Piceaabies*) bark have been reported to be highly lignified tissues that are important in phys. defense against bark beetle invasion. Microchem.analyses of the low-mol. wt. compds. in the stone cells of Norway spruce were carried out using laser microdissection in combination with cryogenic **NMR** and mass spectrometry (LMD/**NMR**/MS). Two phenolic compds., the stilbeneastringin and the dihydroflavonoldihydroxyquercetin 3'-**O**-β-D-glucopyranoside, were identified indicating that stone cells are more than just repositories for lignin. Both of these compds. were also found to be present in other phloem tissue at a higher level than in the stone cells based on **quantification** by cryogenic 1H **NMR**. Our results suggest that stone cells may be involved in chem. as well as phys. defense against bark beetles and their assocd. microorganisms. This paper reports on the identification of secondary **plant** metabolites from a single laser-microdissected population of **plant** cells offering a sensitive new way to det. the chem. profile of specific **plant** cell types with a high degree of precision.

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22. **Development of a new distillation based process for trioxane production**

ByGruetzner, Thomas; Lang, Neven; Siegert, Markus; Stroefer, Eckard; Hasse, Hans

From Institution of Chemical Engineers Symposium Series (2006), 152, 336-343.Language:English, Database: CAPLUS

This paper reports on the development of a new process for the prodn. oftrioxane (C3H6O3), the cyclic trimer of formaldehyde (CH2O). Trioxane is synthesized from aq. formaldehyde solns. usingconcd. sulfuric acid as a catalyst and is mainly used for producing the high performance polymer poly(oxymethylene) (POM). As the POM market is continuously growing trioxane producers are expanding their facilities. For new **plants**, it would be highly desirable to replace the existing complicated trioxane process is by a simpler, more economic one. During the past two decades, powerful models were developed for describing vapor-liq. equil. of aq. formaldehyde solns., the educt for trioxane synthesis. These solns.are complex reacting multicomponent mixts. that are neither exptl. nor theor. easy to handle. The models give new opportunities for developing an improved trioxane process. In a first step, they were used in the present work for elucidating the phase behavior of the system formaldehyde/water/trioxane. Distn.line diagrams for that system were calcd. for the first time. They show a complex topol., including several pressure dependent azeotropes and distn. boundaries, α/α-anal. shows that pure trioxane can be obtained from by a pressure

swing distn. so that the undesired extn. step of the conventional process can be totally avoided. The resulting new process was also simulated rigorously. Distn.expts. were carried out to validate the results. They prove the feasibility of the sepns. in each column and, hence, of the entire process. For process design also reliable information on reaction kinetics is needed. Existing data on the trioxane synthesis is contradictory and unreliable. Therefore, expts.were carried out, in which the trioxane formation in highly concd. formaldehydesolns. contg. up to 0.1 g/g sulfuric acid was studied at temps. up to 115° with **quant**. 1H **NMR** spectroscopy. Using that method, for the first time reliable data on the kinetics of the trioxane formation were obtained. They were used for developing the reaction kinetic model for the process simulation.

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23. **Comparing metabolomes: The chemical consequences of hybridization in plants**

ByKirk, Heather; Choi, Young Hae; Kim, HyeKyong; Verpoorte, Robert; van der Meijden, Ed

From New Phytologist (2005), 167(2), 613-622.Language:English, Database: CAPLUS, DOI: 10.1111/j.1469-8137.2005.01448.x

Hybridization may lead to unique phytochem. expression in **plant** individuals. Hybrids may express novel combinations or extreme concns. of secondary metabolites or, in some cases, produce metabolites novel to both parental species. Here we test whether there is evidence for extreme metabolite expression or novelty in F1 hybrids between *Senecioaquaticus* and *Seneciojacobaea*. Hybridization is thought to occur frequently within Senecio, and hybridization might facilitate secondary metabolite diversification within this genus. Parental species express different **quantities** of several classes of compds. known to be involved in antiherbivore defense, including pyrrolizidine alkaloids, chlorogenic acid, flavonoids and benzoquinoids. Hybrids demonstrate differential expression of some metabolites, producing lower concns. of amino acids, and perhaps flavonoids, than either parental species. Despite evidence for **quant**. hybrid novelty in this system, **NMR** profiling did not detect any novel compds. among the **plant** groups studied. Metabolomic profiling is a useful technique for identifying qual. changes in major metabolites according to **plant** species and/or genotype, but is less useful for identifying small differences between **plant** groups, or differences in compds. expressed in low concns.

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64. **Characterization of mango juice by high-resolution NMR, hyphenated NMR, and diffusion-ordered spectroscopy**

ByDuarte, Iola F.; Goodfellow, Brian J.; Gil, Ana M.; Delgadillo, Ivonne

From Spectroscopy Letters (2005), 38(3), 319-342.Language:English, Database: CAPLUS, DOI: 10.1081/SL-200058713

The application of **NMR** spectroscopy, hyphenated **NMR**, and diffusion-ordered spectroscopy (DOSY) to the characterization of mango juice, as an example of a complex food mixt., is described. The compositional changes taking place as a function of ripening were followed, and selected metabolites were **quantified** by integration of the corresponding **NMR** peaks. In this way, an overall view of the metabolite changes is obtained, enabling the study of the biochem. mechanisms involved in the ripening process. More than 50 compds.were identified by 1D- and 2D-**NMR**, but many ambiguous assignments remain due to spectral overlap or insufficient coupling information. The use of liq. chromatog. (LC-**NMR**) and LC-**NMR**/mass spectrometry (MS) enables a fuller characterization of the sol. pectin fraction to be made; its dependence on ripening stage is discussed. Finally, DOSY adds information on the Mr of many metabolites, including the pectin fractions of ripe and unripe mango juices, and enables further peak assignments to be made.

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24. **Magnetic Imaging of Pyrolysis Feedstocks to model olefin product yields**

ByVirk, P. S.

From Preprints of Symposia - American Chemical Society, Division of Fuel Chemistry (2005), 50(1), 234-238.Language:English, Database: CAPLUS

A system for the **Magnetic** Imaging of Pyrolysis Feedstocks, acronym MIPF (pronounced with P silent) has been devised comprising three facets. First, sample prepn. incorporates internal stds. into the feedstock oils, to enable precise anal. of the **NMR**expts. Second, **NMR**expts.are performed to provide **quant**. C13 and H1 spectra, with spectral features elaborated by 1-D and 2-D procedures such as DEPT, COSY and HETCOR. Third, data anal.employs (1) an Integral Regions train, which provides coarse but complete information about all the carbon and hydrogen atoms in a feedstock, particularly the arom. C and H atoms, and (2) a

Canonical Groups train, which provides high-level information about chem. moieties, but detects only ∼1/3

of all the atoms in the feedstock, particularly those in **n**- and methylalkane chains. An example illustrates how the MIPF parameters of an AGO feedstock might presage its performance in an ethylene **plant**.

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25. **Quantification of compartmented metabolic fluxes in developing soybean embryos by employing biosynthetically directed fractional 13C labeling, two-dimensional [13C, 1H] nuclear magnetic resonance, and comprehensive isotopomer balancing**

BySriram, Ganesh; Fulton, D. Bruce; Iyer, Vidya V.; Peterson, Joan Marie; Zhou, Ruilian; Westgate, Mark E.; Spalding, Martin H.; Shanks, Jacqueline V.

From Plant Physiology (2004), 136(2), 3043-3057.Language:English, Database: CAPLUS, DOI: 10.1104/pp.104.050625

Metabolic flux **quantification** in **plants** is instrumental in the detailed understanding of metab. but is difficult to perform on a systemic level. Toward this aim, we report the development and application of a computer-aided metabolic flux anal. tool that enables the concurrent evaluation of fluxes in several primary metabolic pathways. Labeling expts.were performed by feeding a mixt. of U-13C Suc, naturally abundant Suc, and Gln to developing soybean (*Glycine max*) embryos. Two-dimensional [13C, 1H]**NMR** spectra of seed storage protein and starch hydrolyzates were acquired and yielded a labeling data set consisting of 155 13C isotopomer abundances. We developed a computer program to automatically calc. fluxes from this data. This program accepts a user-defined metabolic network model and incorporates recent math. advances toward accurate and efficient flux evaluation. Fluxes were calcd. and statistical anal. was performed to obtain SDS. A high flux was found through the oxidative pentose phosphate pathway (19.99±4.39 μmol d-1 cotyledon-1, or 104.2 carbon mol. ± 23.0 carbon mol. per 100 carbon mol. of Suc uptake). Sep. transketolase and transaldolase fluxes could be distinguished in the plastid and the cytosol, and those in the plastid were found to be at least 6-fold higher. The backflux from triose to hexose phosphate was also found to be substantial in the plastid (21.72±5.00 μmol d-1 cotyledon-1, or 113.2 carbon mol. ±26.0 carbon mol. per 100 carbon mol. of Suc uptake). Forward and backward directions of anaplerotic fluxes could be distinguished. The glyoxylate shunt flux was found to be negligible. Such a generic flux anal.tool can serve as a **quant**. tool for metabolic studies and phenotype comparisons and can be extended to other **plant** systems.

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26. **Quantitative high-resolution online NMR spectroscopy in reaction and process monitoring**

ByMaiwald, Michael; Fischer, Holger H.; Hasse, Hans

From ChemieIngenieurTechnik (2004), 76(7), 965-969.Language:German, Database: CAPLUS

A review.The application of high-resoln.**NMR** spectroscopy for the process control of **plants** is discussed. In contrast to anal. **NMR** applications, no deuterated solvents are used. The requirements on **NMR** spectroscopy for reaction and process monitoring are described, and the **quant**. evaluation of the **NMR** spectra is outlined. Two application examples are given. The 1st one deals with the reaction kinetics of a heterogeneously catalyzed ester formation, namely a mixt. of**n**-butanol, HOAc, **n**-butylacetate, and H2O. The 2nd example describes the reactive adsorption of acidic gases, like CO2, from aq. amine solns. This method can be used for the removal of CO2 from waste gases of power stations.

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27. **Improvements in metabolic flux analysis using carbon bond labeling experiments: bondomer balancing and Boolean function mapping**

BySriram, Ganesh; Shanks, Jacqueline V.

From Metabolic Engineering (2004), 6(2), 116-132.Language:English, Database: CAPLUS, DOI: 10.1016/j.ymben.2004.02.003

The biosynthetically directed fractional 13C labeling method for metabolic flux evaluation relies on performing a 2-D [13C, 1H]**NMR** expt. on exts. from organisms cultured on a uniformly labeled carbon substrate. This article focuses on improvements in the interpretation of data obtained from such an expt. by employing the concept of bondomers. Bondomers take into account the natural abundance of 13C; therefore many bondomers in a real network are zero, and can be precluded a priori-thus resulting in fewer balances. Using this method, the authors obtained a set of linear equations which can be solved to obtain anal. formulas for **NMR**-measurable **quantities** in terms of fluxes in glycolysis and the pentose phosphate pathways. For a specific case of this network with four degrees of freedom, a priori identifiability of the fluxes was shown possible for any set of fluxes. For a more general case with five degrees of freedom, the fluxes were shown identifiable for a representative set of fluxes. Minimal sets of measurements which best identify the fluxes are listed. Furthermore, the authors have delineated Boolean function mapping, a new method to iteratively simulate bondomer abundances or efficiently convert carbon skeleton rearrangement information to mapping matrixes. The efficiency of this method is expected to be valuable while analyzing metabolic networks which are not completely known (such as in **plant**metab.) or while implementing iterative bondomer balancing methods.

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28. **NMR-based flux map of cytosolic and plastidic metabolism in plants**

BySriram, Ganesh; Fulton, Bruce; Iyer, Vidya V.; Westgate, Mark E.; Spalding, Martin H.; Shanks, Jacqueline V.

From Abstracts of Papers, 227th ACS National Meeting, Anaheim, CA, United States, March 28-April 1, 2004 (2004), BIOT-182.Language:English, Database: CAPLUS

13C **NMR**-based metabolic flux anal.is a powerful diagnostic tool to study physiol. and identify metabolic engineering targets. Its implementation in **plants** is challenging since **plant**metab. exhibitscompartmentation - the same pathway(s) operate in parallel in two compartments. Often successful metabolic engineering requires identification of the target gene in the correct compartment. Consequently, evaluation of fluxes in individual compartments is needed. We developed a two-compartmental model for flux anal. of soybean (Glycine max) embryos, a model **plant** system. After feeding the embryos with U-13C sucrose, the isotopomer abundances of sink metabolites originating in the plastid (starch) and cytosol (hexose in glycosylated protein) were **quantified** using 2-D **NMR**. Fluxes through glycolysis and pentose phosphate pathways in these two compartments were evaluated using isotopomer balancing. Multiple-compartment flux maps are uncommon, and this is the first application of 13C flux anal. to**plants**, where fluxes of parallel pathways in distinct compartments were evaluated.

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29. **Flow encoded NMR spectroscopy for quantification of metabolite flow in intact plants**

BySzimtenings, Michael; Olt, Silvia; Haase, Axel

From Journal of Magnetic Resonance (2003), 161(1), 70-76.Language:English, Database: CAPLUS, DOI: 10.1016/S1090-7807(02)00183-0

An **NMR** flow **quantification** technique applicable to metabolite flow in **plants** is presented. It combines flow sensitive **magnetization** prepn. with slice selective spectroscopy. Flow encoded **NMR** spectroscopy is described to **quantify**, for the first time, flow velocities of metabolites in **plants** non-invasively. Flow sensitivity is introduced by **magnetization** prepn. based on a stimulated echo expt., prior to slice selective spectroscopy. For flow **quantification** eight different flow-weighted spectra are collected. With this flow prepn. very slow flow velocities down to 0.1 mm/s can be detected and small amts. of flowing metabolites can be obsd. despite the large background signal of stationary and flowing water. Important sequence optimization steps include appropriate choice of exptl. parameters used for flow encoding as well as complete balancing of eddy currents from the flow encoding gradients. The method was validated in phantom expts. and applied in vivo. Examples of **quant**. flow measurements of water and metabolites in phantoms and **plants** are provided to demonstrate the reliability and the performance of flow encoded spectroscopy.

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30. **Quantitative NMR microscopy of osmotic stress responses in maize and pearl millet**

ByVan der Weerd, Louise; Claessens, Mireille M. A. E.; Ruttink, Tom; Vergeldt, Frank J.; Schaafsma, Tjeerd J.; Van As, Henk

From Journal of Experimental Botany (2001), 52(365), 2333-2343.Language:English, Database: CAPLUS, DOI: 10.1093/jexbot/52.365.2333

The effect of osmotic stress (-0.35 MPa) on the cell water balance and apical growth was studied noninvasively for maize (*Zea mays* L., cv. LG 11) and pearl millet (*Pennisetumamericanum* L., cv. MH 179) by 1H **NMR** microscopy in combination with water uptake measurements. Single parameter images of the water content and the transverse relaxation time (T2) were used to discriminate between the different tissues and to follow the water status of the apical region during osmotic stress. The T2 values of nonstressed stem tissue turned out to be correlated to the cell dimensions as detd. by optical microscopy. Growth was found to be strongly inhibited by mild stress in both species, whereas the water uptake was far less affected. During the expt. hardly any changes in water content or T2 in the stem region of maize were obsd. In contrast, the apical tissue of pearl millet showed a decrease in T2 within 48 h of stress. This decrease in T2 is interpreted as an increase in the membrane permeability for water.

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31. **Simultaneous measurement of water flow velocity and solute transport in xylem and phloem of adult plants of *Ricinuscommunis*over a daily time course by nuclear magnetic spectrometry**

ByPeuke, A. D.; Rokitta, M.; Zimmermann, U.; Schreiber, L.; Haase, A.

From Plant, Cell and Environment (2001), 24(5), 491-503.Language:English, Database: CAPLUS, DOI: 10.1046/j.1365-3040.2001.00704.x

A new method for simultaneously **quantifying** rates of flow in xylem and phloem using the FLASH imaging capabilities of **NMR** (**NMR**) spectrometry was applied in this study. The method has a time resoln. of up to 4 min (for the xylem) and was used to measure the velocity of flows in phloem and xylem for periods of several hours to days. For the first time, diurnal time course measurements of flow velocities and apparent vol. flows in phloem and xylem in the hypocotyl of 40-d-old *Ricinuscommunis* L were obtained. Addnl. data on gas exchange and the chem. compn.of leaves, xylem and phloem sap were used to assess the role of

leaves as sinks for xylem sap and sources for phloem. The velocity in the phloem (0.250 ± 0.004 mm s-1) was const. **over** a full day and not notably affected by the light/dark cycle. Sucrose was loaded into the phloem and transported at night, owing to degrdn. of starch accumulated during the day. Concns.of solutes in the phloem were generally less during the night than during the day but varied little within either the day or night. In contrast to the phloem, flow velocities in the xylem were about 1.6-fold higher in the light (0.401 ± 0.004 mm s-1) than in the dark (0.255 ± 0.003 mm s-1) and vol. flow varied commensurately. Larger delays were obsd. in changes to xylem flow velocity with variation in light than in gas exchange. The relative rates of solute transport during day and night were estd. on the basis of relative flow and solute concns. in xylem and phloem. In general, changes in relative flow rates were compensated for by changes in solute concn. during the daily light/dark cycle. However, the major solutes (K+, NO3) varied appreciably in relative concns. Hence the regulation of loading into transport systems seems to be more important to the overall process of solute transport than do changes in mass flow. Due to transport behavior, the chem. compn.of leaves varied during the day only with regard to starch and sol. carbohydrates.

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32. **Rapid method for determining fat content in meat by using continuous wavenuclear magnetic resonance (CW-NMR) technique**

ByNagy, E.; Czegledi-Janko, J.; Elias, I.; Kormendy, L.

From ActaAlimentaria (2000), 29(4), 353-357.Language:English, Database: CAPLUS, DOI: 10.1556/AAlim.29.2000.4.5

Development of rapid methods is often needed for the in-line process control of the proximate compn. (**e**.g. fat or moisture content) of meat in the meat processing **plants**. This paper reports on the continuous wave **NMR** (CW-**NMR**) technique applied for detg. fat content in fresh meat. The interfering moisture content in meat was removed by microwave drying and the dried residue was transferred **quant**. into the **NMR**-tubes. The total anal.time was about 35 min. Expts. were performed with pork (with a fat content from 1.7% to 21%), beef (with a fat content from 1.0% to 16.1%), lard (rendered pork fat) and tallow (rendered beef fat) samples and with their combinations: lard-tallow, lard-lean pork, tallow-lean beef and lard-tallow-lean beef-lean pork. The regression (prediction) equations (**NMR**-signal vs. fat content detd. with the Soxhlet ref. method) of pork and beef did not differ significantly. However, there was a noticeable difference between the regression lines of pure lard and pure tallow. Moreover, the latter ones differed from the regression equations of pork, beef and of the various meat-fat combinations. The variability of the fatty acid compn.of the fat also seems to influence the stability of the calibration curves, because the sensitivity of the CW-**NMR** signal to the fatty acid compn. interferes with the **quant**. detn. of fat content in meat.

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33. **Identification and Quantification of Caffeic and Rosmarinic Acid in Complex Plant Extracts by the Use of Variable-Temperature Two-Dimensional Nuclear Magnetic Resonance Spectroscopy**ByExarchou, Vassiliki; Troganis, Anastasios; Gerothanassis, Ioannis P.; Tsimidou, Maria; Boskou, Dimitrios

From Journal of Agricultural and Food Chemistry (2001), 49(1), 2-8.Language:English, Database: CAPLUS, DOI: 10.1021/jf990928e

A combination of advanced **NMR** (**NMR**) methodologies for the anal.of complex phenolic mixts. that occur in natural products is described, with particular emphasis on caffeic acid and its ester deriv., rosmarinic acid. The combination of variable-temp. two-dimensional proton-proton double quantum filter correlation spectroscopy (1H-1H DQF COSY) and proton-carbon heteronuclear multiple quantum coherence (1H-13C HMQC) gradient **NMR** spectroscopy allows the identification and tentative **quantification** of caffeic and rosmarinic acids at 243 K in exts. from**plants** of the Lamiaceae family, without resorting to previous chromatog. sepn. of the components. The use of proton-carbon heteronuclear multiple bond correlation (1H-13C HMBC) gradient **NMR** spectroscopy leads to the complete assignment of the correlations of the spins of H2a and H3a with the ester and carboxyl carbons of rosmarinic and caffeic acid, even at room temp., and confirms the results of the above methodol. **Quant**. results are in reasonable agreement with reverse phase

HPLC measurements.
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34. **Quantification of water transport in plants with NMR imaging**

ByScheenen, T. W. J.; Van Dusschoten, D.; De Jager, P. A.; Van As, H.

From Journal of Experimental Botany (2000), 51(351), 1751-1759.Language:English, Database: CAPLUS, DOI: 10.1093/jexbot/51.351.1751

A new **NMR** imaging (NMRi) method is described to calc. the characteristics of water transport in **plant** stems. Here, dynamic NMRi is used as a non-invasive technique to record the distribution of displacements of protons for each pixel in the **NMR** image. Using the **NMR**-signal of the stationary water in a ref. tube for calibration, the following characteristics can be calcd. per pixel without advance knowledge of the flow-profile in that pixel: the amt. of stationary water, the amt. of flowing water, the cross-sectional area of flow, the av. linear flow velocity of the flowing water, and the vol. flow. The accuracy of the method is demonstrated with a stem segment of a chrysanthemum flower by comparing the vol. flow, measured with **NMR**, with the actual volumetric uptake, measured with a balance. **NMR** measurements corresponded to the balance uptake measurements with arms error of 0.11 mg s-1 in a range of 0 to 1.8 mg s-1. Local changes in flow characteristics of individual voxels of a sample (**e**.g. intact **plant**) can be studied as a function of time and of any conceivable changes the sample experiences on a time-scale, longer than the measurement time of a complete set of pixel-propagators (17 min).
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35. **The NMR Microscope: a Unique and Promising Tool for Plant Science**

ByIshida, Nobuaki; Koizumi, Mika; Kano, Hiromi

From Annals of Botany (London) (2000), 86(2), 259-278.Language:English, Database: CAPLUS, DOI: 10.1006/anbo.2000.1181

A review with many refs. An outline is given of **NMR** microscopy and its application to **plant** science. An **NMR** microscope non-destructively detects free water in tissues and creates anatomical images of the tissues. Since the **quantity** and mobility of cell-assocd.water is closely related to the condition of the cells,1H-**NMR** images represent physiol. maps of the tissue. In addn., the technique locates sol. org. compds. accumulated in the tissues, such as sugars in vacuoles or fatty acids stored as oil droplets in vesicles.23Na-**NMR** imaging is suitable for studying the physiol. of salt-tolerant **plants**. Diffusion measurements provide information about the transport of substances and ions accompanied by water movement. The recently developed techniques of three-dimensional imaging, flow-encoded imaging and spectroscopic imaging open up new opportunities for **plant** biologists. The **NMR** microscope is thus a unique and promising tool for the study of living **plant** systems in relation to morphol., the true features of which are often lost during prepn. for more conventional tissue anal. (c) 2000 Annals of Botany Company.
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36. **Application of nuclear magnetic resonance in agriculture**

ByGambhir, Prem N.; Nagarajan, Shantha

From PINSA-A: Proceedings of the Indian National Science Academy, Part A: Physical Sciences (1999), 65(6), 731-765.Language:English, Database: CAPLUS

A review, with 35 refs. In agricultural research, the major emphasis is given to anal. of large no. of samples for various chem. constituents and phys. properties. The method should be rapid and non-invasive esp. in germplasm evaluation and **plant** breeding programs. **NMR** technique which has the potential to meet both these demands has been extensively used in studies related to agricultural **plants** and their products. The

principle of free induction decay (FID) of low field **NMR** is used for rapid and non-destructive detn. of oil and moisture in oil seeds. Both spin-spin and spin-lattice relaxation times (T1 & T2) are exploited to obtain degree of satn. of oil in oil seeds and dry rubber content in natural rubber latex. They are extensively used to study water status and their cellular compartmentation in **plant** tissues and to develop a screening technique for drought tolerance in wheat. The application of high resoln.**NMR** esp. with 31P and 13C **nuclei**are also quite substantial. The fatty acid compn.of oil in a single intact seed is obtained using 13C **NMR**. Phosphorus **NMR** is extensively used to elucidate the mechanism of phosphate uptake and compartmentation in roots, anoxia in developing seeds and adaptation of roots to osmotic stress. The application of proton **NMR** imaging in studying the in vivo changes in water status in stems is also explored. Thus the review covers in detail the work carried out by our group using **NMR** techniques in characterizing and **quantifyingplant** traits of agriculturally important crops.

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37. **Fast NMR Flow Measurements in Plants Using FLASH Imaging**

ByRokitta, M.; Zimmermann, U.; Haase, A.

From Journal of Magnetic Resonance (1999), 137(1), 29-32.Language:English, Database: CAPLUS, DOI: 10.1006/jmre.1998.1611

A fast method for **quant**. **NMR** imaging of flow velocities in intact **plants** is described. The purpose of this method is to observe dynamic changes of flow velocity in the xylem of **plants** after fast changes of environmental conditions. The spatial image resoln.is 47 × 188 μm2 in-plane. The method applies a fast gradient echo sequence (FLASH). Compared to other flow **NMR** imaging sequences, the imaging time was reduced by a factor of 6 with comparable signal-to-noise ratio. A complete flow measurement consists of a set of 8 different flow weighted images with a total acquisition time of 3.5 min. (c) 1999 Academic Press.

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38. **Quantitative NMR imaging of kiwi fruit (*Actinidiadeliciosa*) during growth and ripening**

ByClark, Christopher J.; Drummond, Lynley N.; MacFall, Janet S.

From Journal of the Science of Food and Agriculture (1998), 78(3), 349-358.Language:English, Database: CAPLUS, DOI: 10.1002/(SICI)1097-0010(199811)78:3<349::AID-JSFA125>3.0.CO;2-X

Quant. 1H **magneticresonance** (MR) imaging was used to det. relaxation changes (T1, T2-CPMG) at regular intervals during growth and ripening of kiwi fruit (*A. deliciosa*vardeliciosa). Temporal trends and differences between flesh, locule and core tissue were found for both relaxation parameters. However, no consistent assocns.were found between non-destructive measurements and those for individual free sugars, sol. solids content (SSC) and macronutrients and micronutrients detd. on dissected companion samples. Increases of 200% in total free sugar concn.in flesh and 68% in SSC accompanied starch hydrolysis after harvest. Despite the magnitude of these changes, relaxation times remained unaltered. These observations were repeated in a 2nd investigation using A. arguta fruit and T1, T2, T2-CPMG and self-diffusion image contrasts. Here, SSC increased 125% during a compressed 15-day ripening period, while MR parameters like self-diffusion declined only 7-14% from harvest values. T2-CPMG relaxation was also investigated in aq. solns. contg. individual org. acids, sugars or pectate and juice from ripening fruit (4.7-15.5% SSC). Anal.ofsolns. and juices showed relaxation is indeed sensitive to increases in sugar compn. but relatively insensitive to changes in org. acids and sol. pectin at concns. normally found in fruit. Results imply that relaxation parameters detd. from MR images may not be appreciably influenced by processes that cause soln. compn. to vary dramatically, even though these changes are reflected in the relaxation properties of the juice itself. Possible reasons for this are discussed with regard to the impact of cell structure and **magnetic** field strength on relaxation processes.

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39. **Nuclear magnetic resonance microscopy of *Ancistrocladusheyneanus***

ByMeininger, M.; Stowasser, R.; Jakob, P. M.; Schneider, H.; Koppler, D.; Bringmann, G.; Zimmermann, U.; Haase, A.

From Protoplasma (1997), 198(3-4), 210-217.Language:English, Database: CAPLUS, DOI: 10.1007/BF01287570

The tropical liana *Ancistrocladusheyneanus*, which is known for its biol. active naphthylisoquinoline alkaloids, has been studied by **NMR** microscopy for the first time. The spatial resoln.of the cross-sectional **NMR** images was of the order of 20 **μm**. **Quant**. **NMR** relaxation time images of the root and the shoot show great contrast between different tissue regions. In addn., we obsd.the regional distribution of chem. compds. in*Ancistrocladusheyneanus*by chem.-shift **NMR** microscopy. The **NMR** imaging results were compared with light and fluorescence microscopic images and reveal the excellent tissue characterization using **NMR** technol.

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40. **Quantitative evaluation of NMR and MRI methods to measure sucrose concentrations in plants**

ByTse, T. Y.; Spanswick, R. M.; Jelinski, L. W.

From Protoplasma (1996), 194(1-2), 54-62.Language:English, Database: CAPLUS, DOI: 10.1007/BF01273167

Developing pea (*Pisumsativum* L.) seeds were chosen to evaluate the performance of various **NMR** and **magneticresonance** imaging (MRI) methods of detecting sucrose in **plants**. The methods included chem. shift selective imaging (CHESS), heteronuclear correlation via 13C-1H coupling (HMQC), and homonuclear correlation via 1H-1H coupling (DQF). The same expts.were also performed on sucrose phantom samples to evaluate the methods in the absence of the line broadening obsd. in**plant** systems. Using the spin echo technique for multi-slice imaging, we could discern the detailed internal structure of the intact seed with a resoln. of tens of microns. The proton spin-lattice relaxation time and linewidth as a function of the age of the seed were measured to optimize the efficiency of the **NMR** and MR expts. The age-dependent changes in these **NMR** parameters are consistent with the accumulation of insol. starch as age increases. Both the **NMR** and MRI results are in accord with the results of chem. anal., which reveal that the sucrose concn. is higher in the embryo than in the seed coat, and glucose is at low concn. throughout the seed. Of the three methods for proton observation, the enhanced version of the CHESS approach (CD-CHESS) provides the best combination of sucrose detection and water suppression. Direct observation of 13C is preferable to indirect detection using HMQC because of water signal bleed-through in samples with large (>200 Hz) linewidths.

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41. **2D and 3D DOSY 1H NMR, a useful tool for analysis of complex mixtures: application to herbal drugs or dietary supplements for erectile dysfunction**

ByBalayssacStephane; TrefiSaleh; Gilard Veronique; Malet-Martino Myriam; Martino Robert; Delsuc Marc-Andre

From Journal of pharmaceutical and biomedical analysis (2009), 50(4), 602-12.Language:English, Database: MEDLINE

Seventeen herbal dietary supplements, marketed as natural substances for the enhancement of sexual function, were analyzed by diffusion ordered spectroscopy (DOSY) (1)H**NMR**. The method allowed a global analysis of the samples with detection of both active and inactive ingredients present in these complex matrixes. Eight formulations contained compounds related to the synthetic phosphodiesterase-5 inhibitors. Sildenafil, tadalafil, vardenafil, hydroxyhomosildenafil, thiosildenafil, and the newly identified adulterant thiomethisosildenafil were detected. **Quantification** of these active ingredients was carried out by HPLC or **NMR**. In addition to these actives, about 30 compounds or excipients were characterized. This study ended up with a three-dimensional DOSY-COSY (1)H**NMR** experiment on a herbal formulation which provided both virtual separation and structural information.

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42. **Single-laboratory validation of an NMR method for the determination of aloe vera polysaccharide in pharmaceutical formulations**

ByDavis Bryce; Goux Warren J

From Journal of AOAC International (2009), 92(6), 1607-16.Language:English, Database: MEDLINE

This report presents a single-laboratory-validated **NMR** method for determining the **quantity** of aloe vera polysaccharide in product formulations. The ratio of signal intensities of the acetyl methyl protons to methyl protons of an internal reference varied linearly with concentration (r2 > 0.99) with a lower LOQ of 0.2 g/100 mL for two commercial aloe polysaccharide standards, Acemannan Hydrogel (AH) and Immuno-10 (I-10). The assay was used to **quantify** these standards in two nonacetylated polysaccharide matrices, dextrin and arabinogalactan, and in a pharmaceutical product. The concentrations of AH in samples containing the polysaccharide matrices agreed within 7% of values determined on the basis of weight and showed within- and between-run RSD values of < 3.5%. The assay of I-10 in the pharmaceutical product was within 7% of the expected values **over** a range from 50 to 125% of the targeted I-10 concentration, with a between-run RSD of < 7%. The assay showed no interference from other added polysaccharides or from other components of the pharmaceutical formulation and was independent of the molecular size distribution of the aloe polysaccharide present. The **NMR** assay can be used to validate aloe polysaccharide contained in a product and to follow any chemical degradation that may occur **over** time.

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43. **NMR quantification of trace components in complex matrices by band-selective excitation with adiabatic pulses**

ByRastrelli Federico; SchievanoElisabetta; Bagno Alessandro; Mammi Stefano

From Magnetic resonance in chemistry : MRC (2009), 47(10), 868-72.Language:English, Database: MEDLINE

The use of band-selective excitation with adiabatic pulses to rapidly obtain **NMR** spectra of trace components in the presence of strong signals is described, along with qualitative and **quantitative** examples from food matrices like olive oil and honey.

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44. **Direct NMR analysis of cannabis water extracts and tinctures and semi-quantitative data on delta9-THC and delta9-THC-acid**

ByPoliti M; Peschel W; Wilson N; Zloh M; Prieto J M; Heinrich M

From Phytochemistry (2008), 69(2), 562-70.Language:English, Database: MEDLINE

Cannabis sativa L. is the source for a whole series of chemically diverse bioactive compounds that are currently under intensive pharmaceutical investigation. In this work, hot and cold water extracts as well as ethanol/water mixtures (tinctures) of cannabis were compared in order to better understand how these extracts differ in their overall composition. **NMR** analysis and in vitro cell assays of crude extracts and fractions were performed. Manufacturing procedures to produce natural remedies can strongly affect the final composition of the herbal medicines. Temperature and polarity of the solvents used for the extraction resulted to be two factors that affect the total amount of Delta(9)-THC in the extracts and its relative **quantity** with respect to Delta(9)-THC-acid and other metabolites. Diffusion-edited (1)H**NMR** (1D DOSY) and (1)H **NMR** with suppression of the ethanol and water signals were used. With this method it was possible, without any evaporation or separation step, to distinguish between tinctures from different cannabis cultivars. This approach is proposed as a direct analysis of **plant** tinctures.

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45. **Inhibition of cellulase, xylanase and beta-glucosidase activities by softwood lignin preparations**

ByBerlin Alex; Balakshin Mikhail; Gilkes Neil; Kadla John; Maximenko Vera; Kubo Satoshi; Saddler Jack

From Journal of biotechnology (2006), 125(2), 198-209.Language:English, Database: MEDLINE

The conversion of lignocellulosic biomass to fuel ethanol typically involves a disruptive pretreatment process followed by enzyme-catalyzed hydrolysis of the cellulose and hemicellulose components to fermentable sugars. Attempts to improve process economics include protein engineering of cellulases, xylanases and related hydrolases to improve their specific activity or stability. However, it is recognized that enzyme performance is reduced during lignocellulose hydrolysis by interaction with lignin or lignin-carbohydrate complex (LCC), so the selection or engineering of enzymes with reduced lignin interaction offers an alternative means of enzyme improvement. This study examines the inhibition of seven cellulase preparations, three xylanase preparations and a beta-glucosidase preparation by two purified, particulate lignin preparations derived from softwood using an organosolv pretreatment process followed by enzymatic hydrolysis. The two lignin preparations had similar particle sizes and surface areas but differed significantly in other physical properties and in their chemical compositions determined by a 2D correlation HSQC **NMR** technique and **quantitative** 13C **NMR** spectroscopy. The various cellulases differed by up to 3.5-fold in their inhibition by lignin, while the xylanases showed less variability (< or = 1.7-fold). Of all the enzymes tested, beta-glucosidase was least affected by lignin.

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46. **Quantitative 1H-NMR imaging of water in white button mushrooms (*Agaricusbisporus*)**

ByDonker H C; Van As H; Snijder H J; Edzes H T

From Magnetic resonance imaging (1997), 15(1), 113-21.Language:English, Database: MEDLINE

MRI represents a valuable tool for studying the amount and physical status of water in **plants** and agricultural products, for example, mushrooms (*Agaricusbisporus*). Contrast in **NMR** images originates from the mixed influence of the fundamental **NMR** parameters, amongst others, spin-density, T2- and T1 relaxation processes. Maps of these parameters contain valuable anatomical and physiological information. They can, however, be severely distorted, depending on the combination of parameter settings and anatomy of the object under study. The influence of the tissue structure of mushrooms, for example, tissue density (susceptibility inhomogeneity) and cell shape on the amplitude, T2, and T1 images is analyzed. This is achieved by vacuum infiltration of the cavities in the mushroom's spongy structure with Gd-DTPA solutions and acquiring Saturation Recovery-Multispin Echo images. It is demonstrated that the intrinsic long T2 values in the cap and outer stipe tissue strongly relate to the size and geometry of the highly vacuolated cells in these spongy tissues. All observed T2 values are strongly affected by susceptibility effects. The T2 of gill tissue is shorter than T2 of the cap and outer stipe, probably because these cells are less vacuolized and smaller in size. The calculated amplitude images are not directly influenced by susceptibility inhomogeneities as long as the observed relaxation times remained sufficient long. They reflect the water distribution in mushrooms best if short echo times are applied in a multispin echo imaging sequence at low **magnetic** field strength.

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47. **Quantitative NMR microscopy on intact plants**

ByKuchenbrod E; Haase A; Benkert R; Schneider H; Zimmermann U

From Magnetic resonance imaging (1995), 13(3), 447-55.Language:English, Database: MEDLINE

Quantitative high resolution images on intact young maize **plants** were acquired by using **magnetization**-

prepared **NMR** microscopy. Although the spatial resolution is low compared with that of light microscopy, the calculated spin density and T1 maps exhibit contrasts that are in excellent agreement with photomicrographic images. The T2 map gives image contrasts that are not visible in a usual light microscopic image. The diffusion images show an anisotropic behavior of the water self-diffusion coefficient in the vascular bundles, which can be understood by the cell morphology in this **plant** section. This work demonstrates that **quantitative** imaging on intact **plant** systems is possible and that long total acquisition times are no obstacle. Furthermore, the different single parameter maps give a better insight into the morphology of **plants** under in vivo conditions.

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