qNMR references (general and applied to plant extracts) for the webpage

1. **Dynamic residual complexity of natural products by qHNMR: solution stability of desmethylxanthohumol**

   ByChen, Shao-Nong; Lankin, David C.; Chadwick, Lucas R.; Jaki, Birgit U.; Pauli, Guido F.

   From Planta Medica (2009), 75(7), 757-762.Language:English, Database: CAPLUS, DOI: 10.1055/s-0028-1112209

   The use of chromatog.assays to assess the residual complexity of materials that are purified from natural sources by chromatog. means is, in a sense, a case of the fox watching the henhouse. Beside their static residual complexity, which is intrinsic to their metabolic origin, biol. active natural materials can also be involved in chem. reactions that lead to dynamic residual complexity. The present study examines the dynamics of the hop prenylphenol, desmethylxanthohumol (DMX), by quant. 1H-NMR (**qHNMR**) in a setting that mimics in vitro and physiol. conditions. The expts.provide a comprehensive, time-resolved, and mechanistic picture of the spontaneous isomerization of DMX into congeneric flavanones, including their 1H/2D isotopomers. Formation of the potent phytoestrogen, 8-prenylnaringenin (8PN), suggests that measurable estrogenic activity even of high-purity DMX is an artifact. Together with previously established **qHNMR** assays including purity activity relationships (PARs), dynamic **qHNMR** assays complement important steps of the post-isolation evaluation of natural products. Thus, **qHNMR** allows assessment of several unexpected effects that potentially break the assumed linkage between a single chem. entity (SCE) and biol. endpoints.

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2. **Purity-Activity Relationships of Natural Products: The Case of Anti-TB Active Ursolic Acid**

   ByJaki, Birgit U.; Franzblau, Scott G.; Chadwick, Lucas R.; Lankin, David C.; Zhang, Fangqiu; Wang, Yuehong; Pauli, Guido F.

   From Journal of Natural Products (2008), 71(10), 1742-1748.Language:English, Database: CAPLUS, DOI: 10.1021/np800329j

   The present study explores the variability of biol. responses from the perspective of sample purity and introduces the concept of purity-activity relationships (PARs) in natural product research. The abundant plant triterpene ursolic acid (1) was selected as an exemplary natural product due to the overwhelming no. yet inconsistent nature of its approx. 120 reported biol. activities, which include anti-TB potential. Nine different samples of ursolic acid with purity certifications were obtained, and their purity was independently assessed by means of quant. 1H NMR (**qHNMR**). Biol. evaluation consisted of detg. MICs against two strains of virulent *Mycobacterium tuberculosis* and IC50 values in Vero cells. Ab initio structure elucidation provided unequivocal structural confirmation and included an extensive 1H NMR spin system anal., detn. of nearly all J couplings and the complete NOE pattern, and led to the revision of earlier reports. As a net result, a sigmoid PAR profile of 1 was obtained, demonstrating the inverse correlation of purity and anti-TB bioactivity. The results imply that synergistic effects of 1 and its varying impurities are the likely cause of previously reported antimycobacterial potential. Generating PARs is a powerful extension of the routinely performed quant. correlation of structure and activity ([Q]SAR). Advanced by the use of primary anal.methods such as **qHNMR**, PARs enable the elucidation of cases like 1 when increasing purity voids biol. activity. This underlines the potential of PARs as a tool in drug discovery and synergy research and accentuates the need to routinely combine biol. testing with purity assessment.

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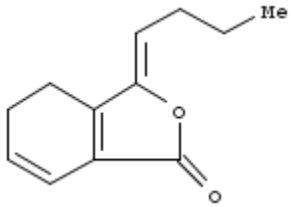
3. **Dynamic nature of the ligustilide complex**

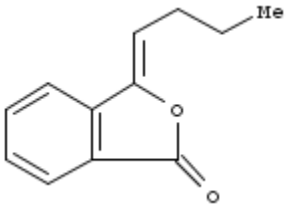
   BySchinkovitz, Andreas; Pro, Samuel M.; Main, Matthew; Chen, Shao-Nong; Jaki, Birgit U.; Lankin, David C.; Pauli, Guido F.

   From Journal of Natural Products (2008), 71(9), 1604-1611.Language:English, Database: CAPLUS, DOI: 10.1021/np800137n

   Monomeric phthalides such as Z-ligustilide (I) and Z-butylidenephthalide (II) are major constituents of medicinal plants of the Apiaceae family. While I has been assocd.with a variety of obsd. biol. effects, it is also known for its instability and rapid chem. degrdn. For the purpose of isolating pure I and II, a gentle and

rapid two-step countercurrent isolation procedure was developed. From a supercrit. CO2 fluid ext. of Angelica sinensis roots, the phthalides were isolated with high GC-MS purities of 99.4% for I and 98.9% for II and consistently lower **qHNMR** purities of 98.1% and 96.4%, resp. Taking advantage of molarity-based **qHNMR** methodol., a time-resolved study of the dynamic changes and residual complexity of pure I was conducted. GC-MS and (qH)NMR anal. of artificially degraded I provided evidence for the phthalide degrdn. pathways and optimized storing conditions. Parallel **qHNMR** anal.led to the recognition of variations in time- and process-dependent sample purity and has impact on the overall assessment of time-dependent changes in complex natural products systems. The study underscores the importance of independent quant. monitoring as a prerequisite for the biol. evaluation of labile natural products such as monomeric phthalides.

79. 

I 

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4. **Nuclear magnetic resonance method for quantitative and qualitative measurement of natural products**

By Pauli, Guido F.; Jaki, Brigit; Lankin, David

From PCT Int. Appl. (2008), WO2008051857A220080502.Language:English, Database: CAPLUS

Provided are various methods and systems for analyzing natural products by quant. proton NMR (**qHNMR**). A method is provided for quant. and qual. detn. of a natural product by 1HNMR and decoupling 13C nuclei from the protons in the sample contg. the natural product. The resultant spectrum wherein the decoupling provides a cleaner spectrum is used to provide both structural and quant. information about species within the sample. In an aspect, the decoupling is provided by globally optimized alternating-phase rectangular pulses (GARP). The methods presented herein are optionally used to detect impurities in a ref. material and verify the purity level of a ref. material. **QHNMR** is illustrated with taxol.

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5. **A routine experimental protocol for qHNMR illustrated with taxol**

By Pauli, Guido F.; Jaki, Birgit U.; Lankin, David C.

From Journal of Natural Products (2007), 70(4), 589-595.Language:English, Database: CAPLUS, DOI: 10.1021/np060535r

Quant. 1H NMR (**qHNMR**) provides a value-added dimension to the std. spectroscopic data set involved in structure anal., esp. when analyzing bioactive mols. and elucidating new natural products. The **qHNMR** method can be integrated into any routine qual. workflow without much addnl. effort by simply establishing quant. conditions for the std. soln. 1H NMR expts. Moreover, examn.of different chem. lots of taxol (paclitaxel) and a *Taxus brevifolia* ext. as working examples led to a blueprint for a generic approach to performing a routinely practiced 13C-decoupled **qHNMR** expt. and for recognizing its potential and main limitations. The proposed protocol is based on a newly assembled 13C GARP broadband decoupled proton acquisition sequence that reduces spectroscopic complexity by removal of carbon satellites. The method is capable of providing qual. and quant. NMR data simultaneously and covers various analytes from pure compds. to complex mixts. such as metabolomes. Due to a routinely achievable dynamic range of 300:1 (0.3%) or better, **qHNMR** qualifies for applications ranging from ref. stds. to biol. active compds. to metabolome anal. Providing a "cookbook" approach to **qHNMR**, acquisition conditions are described that can be adapted for contemporary NMR spectrometers of all major manufacturers.

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6. **Quantitative 1H NMR: development and potential of a method for natural products analysis**

By Pauli, Guido F.; Jaki, Birgit U.; Lankin, David C.

From Journal of Natural Products (2005), 68(1), 133-149.Language:English, Database: CAPLUS, DOI: 10.1021/np0497301

A review. Based on a brief revision of what constitutes state-of-the-art "quant. exptl. conditions" for 1H quant. NMR (**qHNMR**), this comprehensive review contains almost 200 refs. and covers the literature since 1982 with emphasis on natural products. It provides an overview of the background and applications of **qHNMR** in natural products research, new methods such as decoupling and hyphenation, and anal. potential and limitations, and compiles information on ref. materials used for and studied by **qHNMR**. The dual status of natural products, being single chem. entities and valuable biol. active agents that need to be purified from complex matrixes, results in an increased anal.demand when testing their deviation from the singleton compn. ideal. The outcome and versatility of reported applications lead to the conclusion that **qHNMR** is currently the principal anal. method to meet this demand. Considering both 1D and 2D 1H NMR expts.,**qHNMR** has proved to be highly suitable for the simultaneous selective recognition and quant. detn. of metabolites in complex biol. matrixes. This is manifested by the prior publication of over 80 reports on applications involving the quantitation of single natural products in plant exts., dietary materials, and materials representing different metabolic stages of microorganisms. In summary, **qHNMR** has great potential as an anal. tool in both the discovery of new bioactive natural products and the field of metabolome anal.

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7. **Quantitative NMR of bioactive natural products**

By Pauli, Guido F.; Jaki, Birgit U.; Lankin, David C.; Walter, John A.; Burton, Ian W.

Edited byColegate, Steven M.; Molyneux, Russell J

From Bioactive Natural Products (2nd Edition) (2008), 113-141.Language:English, Database: CAPLUS

A review discusses quant. NMR concepts, outlines **qNMR** technol., and provide examples of applications.

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8. **Evaluation of Glucoiberin Reference Material from *Iberis amara* by Spectroscopic Fingerprinting**

ByJaki, Birgit; Sticher, Otto; Veit, Markus; Froehlich, Roland; Pauli, Guido F.

From Journal of Natural Products (2002), 65(4), 517-522.Language:English, Database: CAPLUS, DOI: 10.1021/np0100800

Increasing worldwide regulations require increased efforts toward validation of anal. and pharmacol. ref. materials. A detailed survey of glucoiberin, a prototype lead constituent of therapeutic value, using 1D/2D NMR, MS, and X-ray spectroscopy provided precise phytochem. data for structure assignment. Quant. ref. validation was achieved by the recently proposed **qNMR** method.

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9. **qNMR - a versatile concept for the validation of natural product reference compounds**

By Pauli, Guido F.

From Phytochemical Analysis (2001), 12(1), 28-42.Language:English, Database: CAPLUS, DOI: 10.1002/1099-1565(200101/02)12:1<28::AID-PCA549>3.0.CO;2-D

A review with many refs.The validation of ref. compds.for natural products is a domain of the same physico-chem. methods that are used for their isolation, esp. those techniques involving coupled high-resoln. chromatog. Acknowledging the great problem of co-eluting impurities contained in the "biogenetic cocktail" of a plant ext., there is a strong demand for non-chromatog. alternatives in the quality assessment of ref. compds. Because of this, the concept of **qNMR** is introduced as a versatile tool based on qual. and quant.

1H-NMR allowing the precise and simultaneous detn. of both the compd. content as well as the amt. and nature of the impurities. As opposed to measuring carbons, 1H-NMR benefits from much higher sensitivity and is far more versatile for routine anal. with respect to time and cost. Since quantification of impurities is reliant upon their identification and, therefore, limited by knowledge about their structure, the concept emphasizes the high demand for qual. ref. dossiers including quality NMR data for profiling potential impurities which may be analogs, isomers, or degrdn. or oxidn. products of the ref. compds. The **qNMR** concept is developed with focus on its potential in the certification and quality control of ref. compds. Taking into account published work in the field of quant. NMR, selected natural products are analyzed in order to elaborate suitable exptl. parameters and to obtain preliminary validation data. The method is discussed with respect to sensitivity, precision and selectivity. Typical relative errors are found to be below 2% for the quantification of both the major analyte and the minor impurities even when the latter are contained at the 1% level only. Documentation of the conformity of signal integration and precision is based on measurements of a certified ref. std. Detn. of the natural 13C isotope is suggested as an elegant method of validation because the content values could be reproduced with errors below 1%. The **qNMR** concept offers a rapid and efficient way to assess the purity of natural products in a single anal. step without the need of performing multiple analyses, while still offering the option to retain the substance. Thus, **qNMR** pays tribute to the increasing demands in ref. compd. certification, but also holds out the prospect of easy access to the valid characterization of natural products tested in vitro or in vivo for their biol. and pharmacol. effects.

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10. **NMR based screening tool for quality control of botanical dietary supplements**

ByColson, Kimberly L.; Hicks, Joshua M.; Glinski, Jan A.; Gafner, Stefan; Ferrier, Jonathan; McIntyre, Kristina; Arnason, John T.; Cuerrier, Alain; Killday, Brian

From Abstracts, 41st Middle Atlantic Regional Meeting of the American Chemical Society, Wilmington, DE, United States, April 10-13 (2010), MARM-179.Language:English, Database: CAPLUS

Assessment of quality of **botanical** dietary supplements is challenging due to the complex nature of the mol. components that vary with growing location, seasonal conditions, harvesting conditions and processing conditions. The ability of **NMR** to analyze complex mixts.as a non-targeted fingerprinting method combined with rapid sample prepn. makes it an attractive anal. tool for the routine anal. of**botanical** exts. In this presentation we will show our work towards developing an **NMR** based quality control tool for crude **botanical** exts. including grape seed, pine bark, skullcap, ginseng, cranberry and blueberry. This presentation includes (1) evaluation of **NMR** reproducibility between different instruments and different operators to establish robust screening methods, (2) statistical methods used to characterize the **botanical** exts., (3) sample characterization to provide information such as the varietal, sample purity, and natural variation in samples, and (4) identification of the presence of single components within a crude ext. and **quantification** of these components.

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11. **An improved NMR method for the quantification of α-acids in hops and hop products**

ByHoek, Arie C.; Hermans-Lokkerbol, Ank C. J.; Verpoorte, Robert

From Phytochemical Analysis (2001), 12(1), 53-57.Language:English, Database: CAPLUS, DOI: 10.1002/1099-1565(200101/02)12:1<53::AID-PCA550>3.0.CO;2-E

A new and independent method for the **quantification** of α-acids in **hops** and **hop** products was developed. **NMR** was used as a qual. method for the complete assignment of all 1H- and 13C-**NMR** signals of the 3 main α-acids, cohumulone (1), **humulone** (2) and adhumulone. In a 13C 2-dimensional INADEQUATE expt., the 13C-**NMR** spectrum of 1 was unambiguously assigned via the carbon-carbon connectivities. Use of **NMR** as a **quant**. method allowed **quantification** of the pure and individual α-acids 1 and 2, and the detn. of the abs. concn. of solns. of these compds. **Quantification** of α-acids by **NMR** is less complicated and more reliable than the methods used until now.

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12. **Determination of the humulone composition in hop standards by 1H-NMR spectroscopy**

ByPusecker, Klaus; Holtzel, Alexandra; Albert, Klaus; Bayer, Ernst; Wildenauer, Manfred; Rust, Ulrich

From Monatsschrift fuer Brauwissenschaft (1997), 50(3/4), 70-74.Language:German, Database: CAPLUS

A method was developed for the **quantification** of **humulones** by 1H-**NMR** spectroscopy. This technique confirmed results obtained by combined HPLC conductometric titrn. on the detn. of**n**, ad, and co **humulones**. The reproducibility was the lowest for **n**-**humulone** (<1%).

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13. **Qualitative and quantitative analysis of terpene trilactones and flavonols in ginkgo products by 1H NMR in deuterated solvents**

ByWu, Tian-Shung; Li, Chia-Ying; Wang, Yu-Pei

From Taiwan.(2006), TW266874B20061121.Language:Chinese, Database: CAPLUS

Disclosure is an efficient method for qual. and **quant**. anal. of the active compds. in**ginkgo** products using the **NMR** instrument, particularly for the qual. and **quant**. anal. of**Ginkgo** terpene trilactones and flavonols. Till to date, there is no well method for the anal. of terpene trilactones and flavonols at the same time. We developed an alternative anal. method using 1H **NMR** spectrometry after a simple hydrolysis step to analyze terpene trilactones and flavonol aglycons simultaneously. This procedure is reliable and reproducible for the quality control of **Ginkgo** products, esp. for the qual. and **quant**. anal. of**Ginkgo** terpene trilactones and flavonols.

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14. **Method for rapid qualitative and quantitative analysis of active ingredients in *Ginkgo biloba* products**

ByWu, Tien-Shang; Lee, Chia-Ying; Wang, Yu-Pei

From Faming Zhuanli Shenqing Gongkai Shuomingshu (2006), CN1763514A20060426.Language:Chinese, Database: CAPLUS

The title method comprises hydrolyzing a ***Ginkgo*** *biloba* product sample with strong acid, and analyzing it by **NMR** spectroscopy, using H-12 of **Ginkgo** terpene trilactones and H-2' of flavonols as the targeting signal, a mixt. of deuterated agent and high-polarity solvents as the solvent, and 1,3,5-trimethoxybeneze as the internal std.

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15. **Quantitative analysis of ginkgolic acids from Ginkgo leaves and products using 1H-NMR**

ByChoi, Young Hae; Choi, Hyung-Kyoon; Peltenburg-Looman, A. M. G.; Lefeber, Alfons W. M.; Verpoorte, Robert

From Phytochemical Analysis (2004), 15(5), 325-330.Language:English, Database: CAPLUS, DOI: 10.1002/pca.786

The detn.of ginkgolic acids in **Ginkgo** products is one of the principal components of quality control. However, a no. of ginkgolic acids with different side chains may be present and this makes their anal. by conventional chromatog. methods more complex. In this study, 1H-**NMR** spectrometry was applied to the anal. of the total content of ginkgolic acids in leaves of ***Ginkgo*** *biloba* and in six types of com. **Ginkgo** products in the absence of chromatog. purifn. For this anal., protons H-3, H-4, and H-5, which are well sepd. in the range δ (ppm) 6.5-7.5 in the 1H-**NMR** spectrum, were utilized. For further confirmation, the correlations of H-3, H-4 and H-5 were examd. by 1H-1H COSY spectra in all exts. The **quantity** of the compds.was calcd. from the relative ratio of the integral of each peak to the integral of the peaks of a known amt. (100 μg) of anthracene used as an internal std. The **quant**. results obtained by 1H-**NMR** anal.were compared with those obtained by GC, which showed that the 1H-**NMR** method allows a simple

quantification of total ginkgolic acids in **Ginkgo** exts. without any pre-purifn. steps.

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16. **Efficient 1H Nuclear Magnetic Resonance Method for Improved Quality Control Analyses of Ginkgo Constituents**

ByLi, Chia-Ying; Lin, Chun-Hua; Wu, Chia-Che; Lee, Kuo-Hsiung; Wu, Tian-Shung

From Journal of Agricultural and Food Chemistry (2004), 52(12), 3721-3725.Language:English, Database: CAPLUS, DOI: 10.1021/jf049920h

An anal.method based on 1H-**NMR** spectrometry was used to resolve anal. problems with **Ginkgo**. After a simple hydrolysis step, an **NMR** anal.of the terpene trilactone H-12 signals and the flavonol aglycon H-2' (or H-2'/6' for kaempferol) signals was performed. By comparing the solvent effects on the resoln.of these signals, methanol-d4-benzene-d6 (65:35) was selected as the optimal 1H **NMR** solvent. The amts. of terpene lactones and flavonol aglycons in various com. **Ginkgo** products and **Ginkgo** leaves were detd. This newly developed **NMR** method enables the simultaneous anal. of terpene trilactones and flavonols and allows simple, rapid **quantification** of these compds. in pharmaceutical **Ginkgo** prepns.

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17. **Quantitative analysis of bilobalide and ginkgolides from *Ginkgo biloba* leaves and ginkgo products using 1H-NMR**

ByChoi, Young Hae; Choi, Hyung-Kyoon; Hazekamp, Arno; Bermejo, Paloma; Schilder, Yvonne; Erkelens, Cornelis; Verpoorte, Robert

From Chemical & Pharmaceutical Bulletin (2003), 51(2), 158-161.Language:English, Database: CAPLUS, DOI: 10.1248/cpb.51.158

1H-**NMR** spectrometry was applied to the **quant**. anal. of the bilobalide, ginkgolides A, B, and C in ***Ginkgo biloba*** leaves and 6 kinds of com. **Ginkgo** products without any chromatog. purifn. The expt. was performed by the anal. of each singlet H-12, which were well sepd. in the range of δ 6.0-7.0 in the 1H-**NMR** spectrum. However, the H-12 protons of bilobalide and ginkgolides may have overlapped with H-6 or H-8 protons of the **Ginkgo** flavonoids. Therefore, the optimum 1H-**NMR** solvent for the anal.of the compd. was selected through the evaluation of solvent effects on the resoln. of these signals from the compds. Acetone-d6-benzene-d6 (50:50) was found to be the best 1 among the solvents evaluated. The **quantity** of the compds.was calcd. by the relative ratio of the intensity of each compd. to the known amt. of internal std. (25 μg/mL), phloroglucinol. This method allows rapid and simple **quantitation** of underivatized bilobalide and ginkgolides in 5 min without any pre-purifn. steps.

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18. **Quantitation of bilobalide and ginkgolides A, B, C and J by means of nuclear magnetic resonance spectroscopy**

Byvan Beek, Teris A.; van Veldhuizen, Albertus; Lelyveld, Gerrit P.; Piron, Isabelle

From Phytochemical Analysis (1993), 4(6), 261-8.Language:English, Database: CAPLUS, DOI: 10.1002/pca.2800040604

A **quant**. **NMR** procedure for the detn.of bilobalide and ginkgolides A, B, C and J in ***Ginkgo biloba*** leaves and in phyto-pharmaceuticals without prior chromatog. sepn. of the mixt. was developed. The method was based on the comparison of the integral of each H-12 proton of the 5 **ginkgo** terpene trilactones with that of the olefinic protons of the internal std. (maleic acid). These protons are all well sepd. at 200 MHz and occur in a less crowded region of the **NMR** spectrum (6.15-6.50 ppm). The selectivity, reproducibility and sensitivity are comparable with HPLC with refractive index detection. The min. amt. that can be **quantified** within 30 min at 200 MHz is approx. 0.1 mg for all 4 ginkgolides and bilobalide. Advantages, in comparison with HPLC-RI, are that no ref. substances are needed and that for the occasional anal.of a limited no. of samples a very significant time-gain can be achieved.

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19. **1H NMR fingerprinting of soybean extracts, with emphasis on identification and quantification of isoflavones**

ByCaligiani, Augusta; Palla, Gerardo; Maietti, Annalisa; Cirlini, Martina; Brandolini, Vincenzo

From Nutrients (2010), 2, 280-289.Language:English, Database: CAPLUS, DOI: 10.3390/nu2030280

1H **NMR** spectra were recorded of methanolic exts. of seven soybean varieties (*Glycine max*.), cultivated using traditional and org. farming techniques. It was possible to identify signals belonging to the groups of amino acids, carbohydrates, org. acids and arom. substances in the spectra. In the arom.zone, the isoflavone signals were of particular interest: genistein, daidzein, genistin, daidzin, malonylgenistin, acetylgenistin, malonyldaidzin signals were assigned and these compds. were**quantified**, resulting in accordance with published data, and further demonstrating the potential of the **NMR** technique in food science.

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20. **NMR fingerprint analysis of soy bean isoflavone glycoside**

ByWen, Rui-Zhi; Tan, Ying; Huang, Juan-Juan

From Guangpu Shiyanshi (2006), 23(3), 450-453.Language:Chinese, Database: CAPLUS

By the fingerprint anal.of SOB's 1H-**NMR**, the signal assignments of three glucosides and the mass percentages of three aglycons were obtained. It is a useful method for the fingerprint and **quant**. anal. of mixt.

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21. **Using proton nuclear magnetic resonance as a rapid response research tool for methyl ester characterization in biodiesel**

Byter Horst, Marc; Urbin, Stephanie; Burton, Rachel; McMillan, Christina

From Lipid Technology (2009), 21(2), 39-41.Language:English, Database: CAPLUS, DOI: 10.1002/lite.200900004

Reliable and rapid anal.remains a high priority for quality control in biodiesel prodn. **Quantifying** biodiesel with alternative anal.tools such as proton **NMR** (1H **NMR**) can provide total Me esters distributions without significant sample pretreatment. Using unique spectra of individual Me esters, we investigate the feasibility of using 1H **NMR** spectroscopy to identify and **quantify** relative and abs. concns. of Me esters in a biodiesel.

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22. **Toward *Arabidopsis thaliana* hydrophilic metabolome: assessment of extraction methods and quantitative (1) H NMR**

ByGromova Marina; Roby Claude

From Physiologia plantarum (2010), 140(2), 111-27.Language:English, Database: MEDLINE

Our goal was to establish the hydrophilic metabolome of heterotrophic *Arabidopsis thaliana* cells grown in suspension, a cellular model of **plant** sink tissues. Water-soluble metabolites were **extracted** using four protocols: perchloric acid, boiling ethanol, methanol and methanol/chloroform (**M**/Chl). They were detected and **quantified** using (1) H **nuclearmagneticresonance** (**NMR**) spectroscopy at 400 MHz. **Extraction** yields and reproducibility of the **extraction** methods were investigated. The effects of cell harvest protocol, cell grinding and lyophilization and storage conditions on the measured metabolic profiles were also studied. These **quantitative** studies demonstrated for the first time that the four **extraction** protocols commonly used do lead to quite similar molecular compositions as analyzed by (1) H **NMR**. The **M**/Chl method proved effective and reliable to prepare series of physiologically significant **extracts** from **plant** cells for (1) H **NMR** analysis. Reproducibility of the detected metabolome was assessed **over** long periods of time by analyzing

a large number of separate **extracts** prepared from independent cultures. Larger variations in the **NMR** metabolite profiles could be correlated to changes in physiological parameters of the culture medium. **Quantitative** resolved (1) H **NMR** of cell **extracts** proved to be robust and reliable for routine metabolite profiling of **plant** cell cultures.

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23. **Quantification of artemisinin in *Artemisia annua* extracts by 1H-MNR**

ByCastilho Paula C; Gouveia Sandra C; Rodrigues Ana I

From Phytochemical analysis : PCA (2008), 19(4), 329-34.Language:English, Database: MEDLINE

Artemisinin is a polycyclic sesquiterpene lactone that is highly effective against multidrug-resistant strains of *Plasmodium falciparum*, the etiological agent of the most severe form of malaria. Determination of artemisinin in the source **plant**, Artemisia annua, is a challenging problem since the compound is present in very low concentrations, is thermolabile and unstable, and lacks chromophoric or fluorophoric groups. The ain of this study was to develop a simple protocol for the **quantification** of artemisinin in a **plantextract** using an (1)H-**NMR** method. Samples were prepared by **extraction** of leaf material with acetone, treatment with activated charcoal to remove chlorophylls and removal of solvent. (1)H-**NMR** spectra were measured on samples dissolved in deuterochloroform with tert-butanol as internal standard. **Quantification** was carried out using the using the delta 5.864 signal of artemisinin and the delta 1.276 signal of tert-butanol. The method was optimised and fully validated against a reference standard of artemisinin. The results were compared with those obtained from the same samples **quantified** using an HPLC-refractive index (RI) method. The (1)H-**NMR** method gave a linear **response** for artemisinin within the range 9.85-97.99 mm (**r**(2) = 0.9968). Using the described method, yields of artemisinin in the range 0.77-1.06% were obtained from leaves of the *A. annua* hybrid CPQBA x POP, and these values were in agreement with those obtained using an HPLC-RI.

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24. **Metabolite fingerprinting and profiling in plants using NMR**

ByKrishnan P; Kruger N J; Ratcliffe R G

From Journal of experimental botany (2005), 56(410), 255-65.Language:English, Database: MEDLINEDOI: 10.1093/jxb/eri010

A review. Although less sensitive than mass spectrometry (MS), **nuclearmagneticresonance** (**NMR**) spectroscopy provides a powerful complementary technique for the identification and **quantitative** analysis of **plant** metabolites either in vivo or in tissue **extracts**. In one approach, metabolite fingerprinting, multivariate analysis of unassigned 1H **NMR** spectra is used to compare the overall metabolic composition of wild-type, mutant, and transgenic **plant** material, and to assess the impact of stress conditions on the **plant** metabolome. Metabolite fingerprinting by **NMR** is a fast, convenient, and effective tool for discriminating between groups of related samples and it identifies the most important regions of the spectrum for further analysis. In a second approach, metabolite profiling, the 1H **NMR** spectra of tissue **extracts** are assigned, a process that typically identifies 20-40 metabolites in an unfractionated **extract**. These profiles may also be used to compare groups of samples, and significant differences in metabolite concentrations provide the basis for hypotheses on the underlying causes for the observed segregation of the groups. Both approaches generate a metabolic phenotype for a **plant**, based on a system-wide but incomplete analysis of the **plant** metabolome. However, a review of the literature suggests that the emphasis so far has been on the accumulation of analytical data and sample classification, and that the potential of 1H **NMR** spectroscopy as a tool for probing the operation of metabolic networks, or as a functional genomics tool for identifying gene function, is largely untapped.

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25. **Guaianolides from two subspecies of *Amphoricarpos neumayeri* from Montenegro**

ByDjordjevic Iris; Vajs Vlatka; Bulatovic Vanja; Menkovic Nebojsa; Tesevic Vele; Macura Slobodan; Janackovic Pedja; Milosavljevic Slobodan

From Phytochemistry (2004), 65(16), 2337-45.Language:English, Database: MEDLINE

Quantitative (1)H**NMR** measurements revealed delta(11(13)) sesquiterpene gamma-lactones as the main constituents (>or= 1% per weight of dried **plant** material) in the crude **extracts** of the aerial parts of *Amphoricarpos neumayerissp. neumayeri and ssp. murbeckii* from mountains Orjen and Visitor (Montenegro), respectively. Preparative silica gel chromatography afforded thirteen guai-11(13)-en-12,6alpha-olides, named amphoricarpolides (1-13), with the same relative (1alphaH,4betaH,5alphaH,7betaH) configuration of the basic skeleton. The common structural feature of lactones 2-13 was 3beta,15-dioxygenation pattern. The only exception was 1 (3-deoxyamphoricarpolide), containing a single oxygen substituent (15-OH). Eight of them exhibited an additional oxygen substituent, 9beta-OH (5 and 6), 2alpha-OH (8-12), or 2alpha-OAc (13). Compound 7 was epoxydated at 10alpha(14)-position, whereas the remaining lactones contained a 10(14) double bond.

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26. **Classification and correlation of St. John's wort extracts by nuclear magnetic resonance spectroscopy, multivariate data analysis and pharmacological activity**

ByRoos Gudrun; Roseler Christoph; Buter Karin Berger; Simmen Urs

From Planta medica (2004), 70(8), 771-777.Language:English, Database: MEDLINE, DOI: 10.1055/s-2004-827210

The use of proton **NMR** spectroscopy allows the analysis of complex multi-component mixtures such as **plantextracts** by simultaneous **quantification** of all proton-bearing compounds and consequently all relevant substance classes. Since the spectra obtained are too complicated to be analysed visually, the classification of spectra was carried out using multivariate statistical methods. The spectroscopic data of various **extracts** of St. John's wort (*Hypericum perforatum*) samples derived from 4 different accessions **extracted** with 6 distinct solvents were chemometrically evaluated and calibrated using the partial least square (PLS) algorithm. In a first approach, we found a consistent correlation for the spectroscopic pattern of the **extracts** and the corresponding IC (50) values derived from non-selective binding to opioid receptors. Consequently, the multivariate data analysis was used to predict the pharmacological efficacy of further St. John's wort **extracts** on the basis of their proton **NMR** spectra. In a second approach a PLS 2 model was used to predict the biological activity for eight St. John's wort **extracts** based on two pharmacological data sets: (i) non-selective binding to opioid receptors and (ii) antagonist effect at corticotrophin-releasing factor type 1 (CRF (1)) receptors. The PLS 2 model confirmed the useful application of the presented approach to assess the quality of medicinal herbs and **extracts** by spectroscopic analysis derived from bioactivity-related quality parameters.

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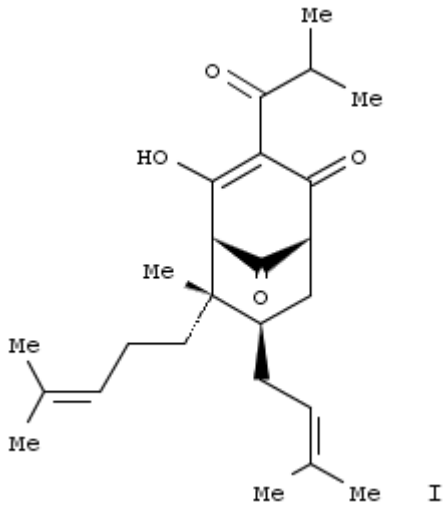
27. **Hyperatomarin, an antibacterial prenylated phloroglucinol from *Hypericumatomarium ssp. degenii***

BySavikin-Fodulovic, Katarina; Aljancic, Ivana; Vajs, Vlatka; Menkovic, Nebojsa; Macura, Slobodan; Gojgic, Gordana; Milosavljevic, Slobodan

From Journal of Natural Products (2003), 66(9), 1236-1238.Language:English, Database: CAPLUS, DOI: 10.1021/np030131o

As shown by **quant**. 1H-**NMR** measurements, a lipophilic ext. of the aerial parts of *Hypericum atomarium ssp. degenii* contained a high percentage (3.1% per wt. of dried **plant** material) of a prenylated phloroglucinol. The compd., named hyperatomarin, occurring in two tautomeric forms (I being the major one), was isolated by bioactivity-guided preparative TLC and was identified on the basis of spectral data interpretation. This isolated phloroglucinol exhibited activity against Gram-pos. (*Staphylococcus aureus* and *Micrococcus luteus*) and Gram-pos. spore-forming bacteria (*Bacillus subtilis* and B. IP 5832).



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28. **Comparison of two liquid-state NMR methods for the determination of saccharides in carrot (*Daucus carota* L.) roots**

ByWeberskirch, Linda; Luna, Alan; Skoglund, Sara; This, Herve

From Analytical and Bioanalytical Chemistry, No pp. yet given.Language:English, Database: CAPLUS, DOI: 10.1007/s00216-010-4311-6

To det. the saccharide content of **plant** tissues, the authors studied a new non-destructive and fast anal. method that the authors call "direct **quant**. proton **NMR** spectroscopy" (d q 1H **NMR**). The application of **quant**. proton **NMR** spectroscopy (q 1H **NMR**) to non modified **plant** tissues along with capillary tubes contg.a ref. compd. (for **quantification**) and deuterium oxide (for locking). Using this method, the saccharide content of samples of carrot (*Daucus carota* L.) roots was compared to the results given from similar samples by the formerly published q 1H **NMR** detn. of exts. obtained by the **O**'Donoghue/Davis method. The content in glucose and sucrose is significantly higher with the direct method than when an extn. step is included; the content in fructose is not significantly different. If a possible detection of saccharides included in glycosylated biol. compds. is to be excluded, a more complete extn. of saccharides is probably obtained using the direct method.

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29. **Effects of long-term cadmium exposure on growth and metabolomic profile of tomato plants**

ByHediji, Hedia; Djebali, Wahbi; Cabasson, Cecile; Maucourt, Michael; Baldet, Pierre; Bertrand, Anne; Boulila Zoghlami, Latifa; Deborde, Catherine; Moing, Annick; Brouquisse, Renaud; et al

From Ecotoxicology and Environmental Safety (2010), 73(8), 1965-1974.Language:English, Database: CAPLUS, DOI: 10.1016/j.ecoenv.2010.08.014

The **response** of tomato **plants** to long-term cadmium exposure was evaluated after a 90-days long culture in hydroponic conditions (0, 20, and 100 μ**M** CdCl2). Cadmium preferentially accumulated in roots, and to a lower extent in upper parts of **plants**. Abs. **quantification** of 28 metabolites was obtained through 1H **NMR**, HPLC-PDA, and colorimetric methods. The principal component anal.showed a clear sepn. between control and Cd treated samples. Proline and total ascorbate amts. were reduced in Cd-treated leaves, whereas α-tocopherol, asparagine, and tyrosine accumulation increased, principally in 100 μ**M** Cd treated leaves. Carotenoid and chlorophyll contents decreased only in 100 μ**M** Cd-mature-leaves, which correlate with a

reduced expression of genes essential for isoprenoid and carotenoid accumulations. Our results show that tomato **plants** acclimatize during long-term exposure to 20 µ**M** Cd. On the contrary, 100 µ**M** Cd treatment results in drastic physiol. and metabolic perturbations leading to **plant** growth limitation and fruit set abortion.

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30. **Comparative quantitative analysis of artemisinin by chromatography and qNMR**

ByLiu, Ning Qing; Choi, Young Hae; Verpoorte, Robert; van der Kooy, Frank

From Phytochemical Analysis (2010), 21(5), 451-456.Language:English, Database: CAPLUS, DOI: 10.1002/pca.1217

Since the discovery of artemisinin in the 1970s, many techniques based on diverse chromatog.techniques were developed to detect and **quantify** this important antiplasmodial compd. The accurate **quantification** of this compd. in the **plant** material is mainly needed for breeding purposes in order to cultivate higher yielding varieties. It is also important for the quality control of herbal prepns. contg. **plant** material. To evaluate the most common validated **quantification** techniques (LC-MS, HPLC-ELSD and TLC) and compare the results to **quant**. **NMR** spectroscopy (qNMR) in 8 different samples collected from around the world. The leaf material were extd. according to std. procedures and analyzed with the validated **quantification** techniques. For the qNMR anal.the authors did not employ a std. curve but instead used an internal std. (maleic acid) which is not chem. related to artemisinin. The authors found a significant difference between the results in this study. Compared with the qNMR results the HPLC-ELSD corresponded closely, followed by LC-MS. **Quantitation** with TLC led to an estn. range of -0.5 to +3.2 mg artemisinin/g of . These results imply that qNMR, with the addn. of an internal std., can be used to **quantify** artemisinin in samples in a rapid and reproducible manner. Copyright © 2010 John Wiley & Sons, Ltd.

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31. **qNMR: an applicable method for the determination of dimethyltryptamine in ayahuasca, a psychoactive plant preparation**

ByMoura, Sidnei; Carvalho, Felipe Garcia; Rodriguez de Oliveira, Carolina Dizioli; Pinto, Ernani; Yonamine, Mauricio

From Phytochemistry Letters (2010), 3(2), 79-83.Language:English, Database: CAPLUS, DOI: 10.1016/j.phytol.2009.12.004

Ayahuasca is an Amazonian **plant** beverage obtained by infusing the pounded stems of *Banisteriopsis caapi* in combination with the leaves of *Psychotria viridis. P. viridis* contains the psychedelic indole **N**,**N**-dimethyltryptamine (DMT). This assocn.has a wide range of use in religious rituals around the world. In the present work, an easy, fast and non-destructive method by **NMR** of proton (1H **NMR**) for **quantification** of DMT in ayahuasca samples was developed and validated. 2,5-Dimethoxybenzaldehyde (DMBO) was used as internal std. (IS). For this purpose, the area ratios produced by protons of DMT (**N**(CH3)2) at 2.70 ppm, singlet, (6H) and for DMBO (Ar(OCH3)2) at 3.80 and 3.89 ppm, doublet, (6H) were used for **quantification**. The lower limit of **quantification** (LLOQ) was 12.5 µg/mL and a good intra-assay precision was also obtained (relative std. deviation < 5.1%). The present 1H **NMR** method is not time consuming and can be readily applied to monitor this tryptamine in **plant** prepns. We believe that qNMR can be used for identification and **quantification** of many **plant**-based products and metabolites with important advantages, while comparing with other anal. techniques.

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32. **Isolation, characterization and quantification of artemisinin by NMR from Argentinean *Artemisia annua* L.**

ByRimada, Ruben S.; Gatti, Walter O.; Jeandupeux, Rene; Cafferata, Lazaro F. R.

From Boletin Latinoamericano y del Caribe de Plantas Medicinales y Aromaticas (2009), 8(4), 275-281.Language:English, Database:

CAPLUS

Artemisinin is a polycyclic sesquiterpene lactone present in leaves and inflorescences of wild *Artemisia annua* L. That substance is highly effective against multidrug-resistant strains of *Plasmodium falciparum*, which is the etiol. agent of the most severe form of malaria. The known procedures for the extn.and isolation of artemisinin were performed and optimized. The extns.were carried out with different solvents and/or their mixts. Chromatog.methods (TLC, HPLC and GC) were employed for the characterization and quality control of artemisinin and other metabolites presents in the solvents exts. QNMR method was also employed for detn. the artemisinin content in the solvents exts. The convenience of the above procedures was critically evaluated by comparison of the anal. results with those derived by applying the classic isolation methods by Soxhlet extn. The values of artemisinin content in Argentinean **plants** exts. were in the 0.2-0.3%. (p/p) range.

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33. **Metabolic acclimation to hypoxia revealed by metabolite gradients in melon fruit**

ByBiais, Benoit; Beauvoit, Bertrand; Allwood, J. William; Deborde, Catherine; Maucourt, Mickael; Goodacre, Royston; Rolin, Dominique; Moing, Annick

From Journal of Plant Physiology (2010), 167(3), 242-245.Language:English, Database: CAPLUS, DOI: 10.1016/j.jplph.2009.08.010

A metabolomics approach using 1H **NMR** and GC-MS profiling of primary metabolites and **quantification** of adenine nucleotides with luciferin bioluminescence was employed to investigate the spatial changes of metab. in melon fruit. Direct 1H **NMR** profiling of juice collected from different locations in the fruit flesh revealed several gradients of metabolites, **e**.g. sucrose, alanine, valine, GABA or ethanol, with increase in concns. from the periphery to the center of the fruit. GC-MS profiling of ground samples revealed gradients for metabolites not detected using 1H **NMR**, including pyruvic and fumaric acids. The **quantification** of adenine nucleotides highlighted a strong decrease in both ATP and ADP ratios and the adenylate energy charge from the periphery to the center of the fruit. These concn.patterns are consistent with an increase in ethanol fermn. due to oxygen limitation and were confirmed by obsd. changes in alanine and GABA concns., as well as other markers of hypoxia in **plants**. Ethanol content in melon fruit can affect organoleptic properties and consumer acceptance. Understanding how and when fermn. occurred can help to manage the culture and limit ethanol prodn.

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34. **Three Phenotypic Cultivars of *Salvia miltiorrhiza* Bunge**

ByDai, Hui; Xiao, Chaoni; Liu, Hongbing; Hao, Fuhua; Tang, Huiru

From Journal of Proteome Research (2010), 9(3), 1565-1578.Language:English, Database: CAPLUS, DOI: 10.1021/pr901045c

Metabonomic anal.is an important mol. phenotyping method for understanding **plant** ecotypic variations and gene functions. Here, the metabonomic variations assocd. with three *Salvia miltiorrhiza* Bunge (SMB) cultivars were systematically characterized using the combined **NMR** and LC-DAD-MS detections in conjunction with multivariate data anal. The results indicated that **NMR** methods were effective to **quant**. detect the abundant **plant** metabolites including both the primary and secondary metabolites whereas the LC-DAD-MS methods were excellent for selectively detecting the secondary metabolites. It was found that the SMB metabonome was dominated by 28 primary metabolites including sugars, amino acids, and carboxylic acids and 4 polyphenolic secondary metabolites, among which **N**-acetylglutamate, aspartate, fumarate, and yunnaneic acid D were reported for the first time in this **plant**. It was also found that three SMB cultivars growing at the same location had significant metabonomic differences in terms of metabs. of carbohydrates, amino acids, and choline, TCA cycle, and the shikimate-mediated secondary metabs. It was further found that the same SMB cultivar growing at different locations differed in their metabonome. These results provided important information on the ecotypic dependence of SMB metabonome on the growing environment and demonstrated that the combination of **NMR** and LC-MS methods was effective for

plantmetabonomic phenotype anal.
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35. **Analysis of metabolic variation and galanthamine content in Narcissus bulbs by 1H NMR**

ByLubbe, Andrea; Pomahacova, Barbora; Choi, Young Hae; Verpoorte, Robert

From Phytochemical Analysis (2010), 21(1), 66-72.Language:English, Database: CAPLUS, DOI: 10.1002/pca.1157

Galanthamine is a benzazepine alkaloid used as a drug to relieve symptoms of Alzheimer's disease. For pharmaceutical use this natural product has been extd. from the **plant** (Amaryllidaceae) or produced synthetically. Limited supply of the natural source and high cost of synthetic prodn.has led to a search for alternative sources of galanthamine. The bulbs of (Amaryllidaceae) have been identified as a potential source of raw material for galanthamine extn. Since inconsistent chem. compn.can be an issue with medicinal **plant** material, it is of interest to know whether large variations occur between bulbs grown in different geog. locations. Differences were evaluated in the overall metabolic profiles of bulbs grown in the 2 most important cultivation regions. 1H **NMR** and principal component anal.were used for an unbiased comparison of the bulb samples. Overall metabolite profiles were quite similar, but galanthamine levels could slightly discriminate samples by geog. region. 1H **NMR** was used for **quantitation** of galanthamine, and was found to be comparable to **quantitation** by HPLC. Compared with conventional chromatog.methods, sample prepn. for 1H **NMR** anal. is simple and rapid, and only a small amt. of **plant** material is required. Since useful qual. and **quant**. information about the metabolic state of bulbs can be obtained by 1H **NMR**, this method is useful for agricultural applications, and for quality control of raw material used in the pharmaceutical industry.

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36. **Metabolic Changes in Different Developmental Stages of *Vanilla planifolia* Pods**

ByPalama, Tony Lionel; Khatib, Alfi; Choi, Young Hae; Payet, Bertrand; Fock, Isabelle; Verpoorte, Robert; Kodja, Hippolyte

From Journal of Agricultural and Food Chemistry (2009), 57(17), 7651-7658.Language:English, Database: CAPLUS, DOI: 10.1021/jf901508f

The metabolomic anal.of developing *Vanilla planifolia* green pods (between 3 and 8 mo after pollination) was carried out by **NMR** spectroscopy and multivariate data anal. Multivariate data anal.of the 1H **NMR** spectra, such as principal component anal. (PCA) and partial least-squares-discriminant anal. (PLS-DA), showed a trend of sepn. of those samples based on the metabolites present in the methanol/water (1:1) ext. Older pods had a higher content of glucovanillin, vanillin, p-hydroxybenzaldehyde glucoside, p-hydroxybenzaldehyde, and sucrose, while younger pods had more bis[4-(β-D-glucopyranosyloxy)-benzyl]-2-isopropyltartrate (glucoside A), bis[4-(β-D-glucopyranosyloxy)-benzyl]-2-(2-butyl)tartrate (glucoside B), glucose, malic acid, and homocitric acid. A liq. chromatog.-mass spectrometry (LC-MS) anal.targeted at phenolic compd. content was also performed on the developing pods and confirmed the **NMR** results. Ratios of aglycons/glucosides were estd. and thus allowed for detection of more minor metabolites in the green vanilla pods. **Quantification** of compds.based on both LC-MS and **NMR** analyses showed that free vanillin can reach 24% of the total vanillin content after 8 mo of development in the vanilla green pods.

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37. **Composition of Indigo naturalis**

ByPlitzko, Inken; Mohn, Tobias; Sedlacek, Natalie; Hamburger, Matthias

From Planta Medica (2009), 75(8), 860-863.Language:English, Database: CAPLUS, DOI: 10.1055/s-0029-1185447

A proposal for a European Pharmacopoeia monograph concerning Indigo naturalis has recently been

published, whereby the indigo (1) and indirubin (2) content should be detd. by HPLC-UV. This method was tested, but problems were seen with the dosage of indigo due to poor soly. A **quant**. assay for indigo based on 1H-**NMR** was developed as an alternative. The HPLC and qNMR assays were compared with eight Indigo naturalis samples. The HPLC assay consistently gave much lower indigo concns. because soly. was the limiting factor in sample prepn. In one sample, sucrose was identified by 1H-**NMR** as an org. additive. Simple wet chem. assays for undeclared additives such as sugars and starch were tested with artificially spiked Indigo naturalis samples to establish their limits of detection, and sulfate ash detns. were carried out in view of a better assessment of Indigo naturalis in a future European monograph.

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38. **Detection of QTLs controlling major fruit quality components in peach within the European project ISAFRUIT**

ByDirlewanger, E.; Cardinet, G.; Boudehri, K.; Renaud, C.; Monllor, S.; Illa, E.; Howad, W.; Arus, P.; Croset, C.; Poessel, J. L.; et al

From Acta Horticulturae (2009), 814(Proceedings of the XIIth Eucarpia Symposium on Fruit Breeding and Genetics, 2007, Volume 2), 533538.Language:English, Database: CAPLUS

ISAFRUIT is a European integrated project aimed at increasing fruit consumption with the goal to improve health of the European citizens. One of the objectives of the work package GENFRUIT is to set the genetic basis of fruit quality by mapping the major genes and QTLs involved (Work package 6.1). This is performed on two peach (*Prunus persica* L. Batsch) and two apricot (*Prunus armeniaca* L.) progenies. Only results obtained on one of the two peach progenies, the F2 progeny of 207 hybrids issued from the 'Ferjalou' Jalousia x 'Fantasia' cross (JxF), will be described in this paper. This progeny is segregating for six Mendelian traits, peach or nectarine (G), flat or round fruit (S), non-acid or acid fruit (D), clingstone or freestone (F), male sterility (Ps), no fruit at maturity (af), as well as for **quant**. characters involved in fruit quality. This progeny was evaluated for various characters such as blooming and maturity date, fruit fresh, and dry wt., flesh firmness, juice sol. solid content; pH and acidity. The **quantification** of the flesh concn.in 15 to 20 major metabolites (glucose, fructose, sucrose, citrate, malate, major amino acids, and some major secondary metabolites) was performed in Bordeaux using proton **NMR** spectroscopy of polar exts. Phenolics profiling, using HPLC-DAD, was performed in Avignon. Candidate genes involved in fruit sugar or acid contents and fruit development were bin mapped in the ref. map 'Texas' x 'Earlygold' and in JxF. Preliminary results on QTL detection concerning fruit quality characters are presented. First colocalisations of QTLs with candidate genes were identified. In the future, the colocalisation of QTLs with candidate genes, and the colinearity of QTLs detected on the different populations analyzed in the ISAFRUIT project will be examd.

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39. **A rapid and simple determination of protoberberine alkaloids in Rhizoma Coptidis by 1H NMR and its application for quality control of commercial prescriptions**

ByLi, Chia-Ying; Tsai, Sung-I.; Damu, Amooru G.; Wu, Tian-Shung

From Journal of Pharmaceutical and Biomedical Analysis (2009), 49(5), 1272-1276.Language:English, Database: CAPLUS, DOI: 10.1016/j.jpba.2009.02.028

Simple, convenient, sensitive, and accurate anal.methods are needed for the anal. of alkaloid components in Rhizoma Coptidis in traditional Chinese herbal medicine, which has important bioactivity. In the present study, a highly specific and sensitive method using 1H **NMR** was developed for the **quant**. detn. of protoberberine alkaloids berberine, palmatine, coptisine, and jatrorrhizine in Coptis species and their com. traditional Chinese medicine prescriptions. A 1H **NMR** anal.of the H-13 signals of target protoberberine alkaloids was performed. By comparing the solvent effects on the resoln.of these signals, MeOH-d 4-benzene-d 6 (75:25) is selected as an optimal 1H **NMR** solvent. The **quantity** of the compds.is calcd. by the relative ratio of the integral values of the target peak for each compd. to the known amt. of the internal std.

anthracene. This method allows rapid and simple **quantitation** of protoberberine alkaloids from Coptis species and the more complex com. prescriptions in 5 min without any pre-purifn. steps. The recoveries of these alkaloids from *Coptis chinensis* are in the range of 93-105%. Limit of detection of berberine in the **plant** material or prescription is 0.03 mg/mL. The advantages of this method are that no ref. compds. are required for calibration curves, the **quantification** can be directly realized on a crude ext., and the better selectivity for protoberberine alkaloids and a very significant time-gain can be achieved, in comparison to conventional HPLC methods, for instance.

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40. **Proton NMR quantitative profiling for quality assessment of greenhouse-grown tomato fruit**

ByDeborde, Catherine; Maucourt, Mickael; Baldet, Pierre; Bernillon, Stephane; Biais, Benoit; Talon, Gilles; Ferrand, Carine; Jacob, Daniel; Ferry-Dumazet, Helene; Daruvar, Antoine; et al

From Metabolomics (2009), 5(2), 183-198.Language:English, Database: CAPLUS, DOI: 10.1007/s11306-008-0134-2

Tomato is an essential crop in terms of economic importance and nutritional quality. In France, the third most important region for tomato (*Solanum lycopersicum* L.) prodn.is Aquitaine where the major part of prodn. is now grown soilless under greenhouse conditions with harvest from March to Nov. Tomato fruit quality at harvest is a direct function of its metabolite content at that time. The aim of this work was to use a global approach to characterize changes in the fruit organoleptic quality at harvest under com. culture conditions during an entire season for two varieties and two different fertilization practices (with or without recycling of the nutrient soln.) for one variety. Abs. **quantification** data of 32 major compds.in fruit without seeds were obtained through untargeted (proton **NMR**, 1H-**NMR**) **quant**. profiling. These data were complemented by colorimetric anal. of ascorbate and total phenolics. They were analyzed with chemometric approaches. Principal component anal. (PCA) or partial least square analyses (PLS) revealed more discriminant metabolites for season than for variety and showed that nutrient soln. recycling had very little effect on fruit compn. These tendencies were confirmed with univariate analyses. 1H-**NMR** profiling complemented with colorimetric analyses therefore provided a diagnostic tool to follow the changes in organoleptic and nutritional quality of tomato. In addn. the **quant**. information generated will help to increase our knowledge on the mechanisms of **plantresponse** to environmental modifications.

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41. **1H NMR, GC-EI-TOFMS, and Data Set Correlation for Fruit Metabolomics: Application to Spatial Metabolite Analysis in Melon**

ByBiais, Benoit; Allwood, J. William; Deborde, Catherine; Xu, Yun; Maucourt, Mickael; Beauvoit, Bertrand; Dunn, Warwick B.; Jacob, Daniel; Goodacre, Royston; Rolin, Dominique; et al

From Analytical Chemistry (Washington, DC, United States) (2009), 81(8), 2884-2894.Language:English, Database: CAPLUS, DOI: 10.1021/ac9001996

A metabolomics approach combining 1H **NMR** and gas chromatog.-electrospray ionization time-of-flight mass spectrometry (GC-EI-TOFMS) profiling was employed to characterize melon (*Cucumis melo* L.) fruit. In a first step, **quant**. 1H **NMR** of polar exts.and principal component analyses (PCA) of the corresponding data highlighted the major metabolites in fruit flesh, including sugars, org. acids, and amino acids. In a second step, the spatial localization of metabolites was investigated using both anal. techniques. Direct 1H **NMR** profiling of juice or GC-EI-TOFMS profiling of tissue exts. collected from different locations in the fruit flesh provided information on advantages and drawbacks of each technique for the anal. of a sugar-rich matrix such as fruit. 1H **NMR** and GC-EI-TOFMS data sets were compared using independently performed PCA and multiblock hierarchical PCA (HPCA), resp. In addn. a correlation-based multiblock HPCA was used for direct comparison of both anal. data sets. These data analyses revealed several gradients of metabolites in fruit flesh which can be related with differences in metab. and indicated the suitability of multiblock HPCA for correlation of data from two (or potentially more) metabolomics platforms.

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42. **Prolonged root hypoxia induces ammonium accumulation and decreases the nutritional quality of tomato fruits**

ByHorchani, Faouzi; Gallusci, Philippe; Baldet, Pierre; Cabasson, Cecile; Maucourt, Mickael; Rolin, Dominique; Aschi-Smiti, Samira; Raymond, Philippe

From Journal of Plant Physiology (2008), 165(13), 1352-1359.Language:English, Database: CAPLUS, DOI: 10.1016/j.jplph.2007.10.016

Here we examd. the effects of root hypoxia (1-2% oxygen) on the physiol. of the **plant** and on the biochem. compn. of fruits in tomato (*Solanum lycopersicum* cv. Micro-Tom) **plants** submitted to gradual root hypoxia at first flower anthesis. Root hypoxia enhanced nitrate absorption with a concomitant release of nitrite and ammonium into the medium, a redn. of leaf photosynthetic activity and chlorophyll content, and an acceleration of fruit maturation, but did not affect final fruit size. **Quant**.metabolic profiling of mature pericarp exts. by 1H **NMR** showed that levels of major metabolites including sugars, org. acids and amino acids were not modified. However, ammonium concn.increased dramatically in fruit flesh, and ascorbate and lycopene concns. decreased. Our data indicate that the unfavorable effects of root hypoxia on fruit quality cannot be explained by two of the well-known effects of root hypoxia on the **plant**, namely a decrease in photosynthesis or an excess in ethylene prodn., but may instead result from disturbances in the supply of either growth regulators or ammonium, by the roots.

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43. **Quantitative 1H nuclear magnetic resonance metabolite profiling as a functional genomics platform to investigate alkaloid biosynthesis in opium poppy**

ByHagel, Jillian M.; Weljie, Aalim M.; Vogel, Hans J.; Facchini, Peter J.

From Plant Physiology (2008), 147(4), 1805-1821.Language:English, Database: CAPLUS, DOI: 10.1104/pp.108.120493

Opium poppy (*Papaver somniferum*) produces a diverse array of bioactive benzylisoquinoline alkaloids and has emerged as a versatile model system to study **plant** alkaloid metab. The **plant** is widely cultivated as the only com. source of the narcotic analgesics morphine and codeine. Variations in **plant** secondary metab.as a result of genetic diversity are often assocd. with perturbations in other metabolic pathways. As part of a functional genomics platform, we used 1H **NMR** (**NMR**) metabolite profiling for the anal. of primary and secondary metab. in opium poppy. Aq. and chloroform exts.of six different opium poppy cultivars were subjected to chemometric anal. Principle component anal.of the 1H **NMR** spectra for latex exts. clearly distinguished two varieties, including a low-alkaloid variety and a high-thebaine, low-morphine cultivar. Distinction was also made between pharmaceutical-grade opium poppy cultivars and a condiment variety. Such phenotypic differences were not obsd. in root exts. Loading plots confirmed that morphinan alkaloids contributed predominantly to the variance in latex exts. **Quantification** of 34 root and 21 latex metabolites, performed using Chenomx **NMR** Suite version 4.6, showed major differences in the accumulation of specific alkaloids in the latex of the low-alkaloid and high-thebaine, low-morphine varieties. Relatively few differences were found in the levels of other metabolites, indicating that the variation was specific for alkaloid metab. Exceptions in the low-alkaloid cultivar included an increased accumulation of the alkaloid precursor tyramine and reduced levels of sucrose, some amino acids, and malate. Real-time polymerase chain reaction anal.of 42 genes involved in primary and secondary metab. showed differential gene expression mainly assocd. with alkaloid biosynthesis. Reduced alkaloid levels in the condiment variety were assocd. with the reduced abundance of transcripts encoding several alkaloid biosynthetic enzymes.

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44. **Reduced oil accumulation in cottonseeds transformed with a Brassica nonfunctional allele of a delta-12 fatty acid desaturase (FAD2)**

ByChapman, Kent D.; Neogi, Purnima B.; Hake, Kater D.; Stawska, Agnes A.; Speed, Thomas R.; Cotter, Matthew Q.; Garrett, David C.; Kerby, Thomas; Richardson, Charlene D.; Ayre, Brian G.; et al

From Crop Science (2008), 48(4), 1470-1481.Language:English, Database: CAPLUS, DOI: 10.2135/cropsci2007.11.0618

In an effort to better understand the mechanisms that regulate oil accumulation and packaging in seeds, transgenic cotton lines were generated using a *Brassica napus* nonfunctional delta-12 fatty acid desaturase (FAD2) gene under control of the phaseolin promoter. Seeds of numerous transgenic **plant** lines had reduced oil content compared with null-segregating siblings or nontransformed seeds. Seed oil content was **quantified** by 1H-**NMR**, and was reduced to 12% or less of seed wt. in transgenics from 20% by wt. in nontransformed controls. Light- and electron-microscopic analyses of severely lowered lines, showed a redn. in overall cotyledon thickness and a disruption in cellular and subcellular organization. Lipid bodies and protein bodies were fewer in transgenics, and their size and distribution in cells was different than that in nontransformed seeds. Coincident with reduced storage reserves, sucrose levels were elevated in transgenic seeds. The overall effect of oil suppression was to selectively reduce the size of the embryo, but the seed coat and fiber properties remained unaffected in seeds. In fact there was a significant increase in lint percentage in all oil-suppressed lines examd. Overall we propose that expression of the nonfunctional Bnfad2 allele in cottonseeds disrupts normal oil biosynthesis and provides for redirection of carbon reserves.

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45. **Plant metabolomics: analytical platforms and integration with functional genomics**

ByHagel, Jillian M.; Facchini, Peter J.

From Phytochemistry Reviews (2008), 7(3), 479-497.Language:English, Database: CAPLUS, DOI: 10.1007/s11101-007-9086-9

A review. As the final downstream product of the genome, the **plant** metabolome is a highly complex, dynamic assortment of primary and secondary compds. Although technol. platforms to study genomes, transcriptomes and even proteomes are presently available, methods to pursue genuine metabolomics have not yet been developed due to the extensive chem. diversity of **plant** primary and secondary metabolites. No single anal. method can accurately survey the entire metabolome. However, recent tech., chemometric and bioinformatic advances promise to enhance our global understanding of **plant** metab. Sepn.-based mass spectrometry (MS) approaches, such as gas (GC) or liq. chromatog. (LC)-MS, are relatively inexpensive, highly sensitive and provide excellent identifying capacity. However, Fourier transform-ion cyclotron **resonance** (FT-ICR)-MS is better suited for rapid, high-throughput applications and is currently the most sensitive method available. Unlike MS-based analyses, **NMR** (**NMR**) spectroscopy provides a large amt. of information regarding mol. structure, and novel software innovations have facilitated the unequivocal identification and abs. **quantification** of compds. within composite samples. Due to the size and complexity of metabolomics datasets, numerous chemometric methods are used to ext. and display systematic variation. Coupled with pattern recognition techniques and **plant**-specific metabolite databases, broad-scope metabolite analyses have emerged as functional genomics tools for novel gene discovery and functional characterization. In this review, key metabolomics technologies are compared and the applications of FT-ICR-MS and **NMR** to the study of benzylisoquinoline alkaloid metab. in opium poppy are discussed.

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46. **Quantitative 1H NMR metabolomics reveals extensive metabolic reprogramming of primary and secondary metabolism in elicitor-treated opium poppy cell cultures**

ByZulak, Katherine G.; Weljie, Aalim M.; Vogel, Hans J.; Facchini, Peter J.

From BMC Plant Biology (2008), 8, No pp. given.Language:English, Database: CAPLUS, DOI: 10.1186/1471-2229-8-5

Opium poppy (*Papaver somniferum*) produces a diverse array of bioactive benzylisoquinoline alkaloids and has emerged as a model system to study **plant** alkaloid metab. The **plant** is cultivated as the only com.

source of the narcotic analgesics morphine and codeine, but also produces many other alkaloids including the antimicrobial agent sanguinarine. Modulations in **plant** secondary metab.as a result of environmental perturbations are often assocd. with the altered regulation of other metabolic pathways. As a key component of our functional genomics platform for opium poppy we have used proton **NMR** (1H **NMR**) metabolomics to investigate the interplay between primary and secondary metab. in cultured opium poppy cells treated with a fungal elicitor. Metabolite fingerprinting and compd.-specific profiling showed the extensive reprogramming of primary metabolic pathways in assocn. with the induction of alkaloid biosynthesis in **response** to elicitor treatment. Using Chenomx **NMR** Suite v. 4.6, a software package capable of identifying and **quantifying** individual compds.based on their resp. signature spectra, the levels of 42 diverse metabolites were monitored **over** a 100-h time course in control and elicitor-treated opium poppy cell cultures. Overall, detectable and dynamic changes in the metabolome of elicitor-treated cells, esp. in cellular pools of carbohydrates, org. acids and non-protein amino acids were detected within 5 h after elicitor treatment. The metabolome of control cultures also showed substantial modulations 80 h after the start of the time course, particularly in the levels of amino acids and phospholipid pathway intermediates. Specific flux modulations were detected throughout primary metab., including glycolysis, the tricarboxylic acid cycle, nitrogen assimilation, phospholipid/fatty acid synthesis and the shikimate pathway, all of which generate secondary metabolic precursors. The **response** of cell cultures to elicitor treatment involves the extensive reprogramming of primary and secondary metab., and assocd. cofactor biosynthetic pathways. A high-resoln.map of the extensive reprogramming of primary and secondary metab. in elicitor-treated opium poppy cell cultures is provided.

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47. **Bioactive agents from beach waste: Syringodium flotsam evaluation as a new source of -chiro-inositol**

ByNuissier, Gladys; Diaba, Faiza; Grignon-Dubois, Micheline

From Innovative Food Science & Emerging Technologies (2008), 9(3), 396-400.Language:English, Database: CAPLUS, DOI: 10.1016/j.ifset.2007.12.002

Flotsam from the seagrass *Syringodium filiforme* were assayed for inositols, a class of cyclitol well known for their biol. activities and applications. Free -chiro-inositol (LCI), a very rare natural occurring cyclitol, was isolated from aq. exts. of dried detrital leaves. The structure was unambiguously established by **NMR** and polarimetry. The LCI content of the crude aq. exts.prepd. from different batches of Syringodium flotsam was measured by **quant**. 1H-**NMR** spectroscopy. The high concns.found (2.3-2.5% dry wt.) offer promise for the exploitation of Syringodium flotsam as a new cheap source for nutraceutical or therapeutic applications, considering the demonstrated hypoglycemic action of LCI. Industrial relevance: In the West the demand for herbal drugs has reached a new high in recent years. As the demand for alternative medicine has grown, so have the harvesting and collection pressures for numerous ecologies that produce the medicinal **plants** of interest. There is evidence in literature that chiro-inositol can be used in managing diabetes. Flotsam of the seagrass *Syringodium filiforme*, which accumulate in huge **quantities** on the beaches of the Caribbean Sea, could become a new and interesting source to obtain exts. rich in chiro-inositol. Heretofore, there has been no market for Syringodium flotsam, so that the cost of the same is simply that of harvesting. Recovery of inositol from this waste material could offer very interesting economic possibilities to tropical coastal areas suffering from increased rates of unemployment.

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48. **An experimental implementation of chemical subtraction**

ByChen, Shao-Nong; Turner, Allison; Jaki, Birgit U.; Nikolic, Dejan; van Breemen, Richard B.; Friesen, J. Brent; Pauli, Guido F.

From Journal of Pharmaceutical and Biomedical Analysis (2008), 46(4), 692-698.Language:English, Database: CAPLUS, DOI: 10.1016/j.jpba.2007.12.014

A preparative anal.method was developed to selectively remove ("chem. subtract") a single compd. from a complex mixt., such as a natural ext. or fraction, in a single step. The proof of concept is demonstrated by the removal of pure benzoic acid (BA) from cranberry (*Vaccinium macrocarpon* Ait.) juice fractions that exhibit anti-adhesive effects vs. uropathogenic *Escherichia coli*. Chem. subtraction of BA, representing a major constituent of the fractions, eliminates the potential in vitro interference of the bacteriostatic effect of BA on the ***E****. coli* anti-adherence action measured in bioassays. Upon BA removal, the anti-adherent activity of the fraction was fully retained, 36% inhibition of adherence in the parent fraction at 100 μg/mL increased to 58% in the BA-free active fraction. The method employs countercurrent chromatog. (CCC) and operates loss-free for both the subtracted and the retained portions as only liq.-liq. partitioning is involved. While the high purity (97.47% by **quant**. 1H **NMR**) of the subtracted BA confirms the selectivity of the method, one minor impurity was detd. to be scopoletin by HR-ESI-MS and (q)HNMR and represents the first coumarin reported from cranberries. A general concept for the selective removal of phytoconstituents by CCC is presented, which has potential broad applicability in the biol. evaluation of medicinal **plant** exts. and complex pharmaceutical prepns.

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49. **Direct NMR analysis of cannabis water extracts and tinctures and semi-quantitative data on Δ9-THC and Δ9-THC-acid**

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

From Phytochemistry (Elsevier) (2007), Volume Date 2008, 69(2), 562-570.Language:English, Database: CAPLUS, DOI: 10.1016/j.phytochem.2007.07.018

Cannabis sativa L. is the source for a whole series of chem. diverse bioactive compds. that are currently under intensive pharmaceutical investigation. In this work, hot and cold water exts. as well as EtOH/water mixts. (tinctures) of cannabis were compared to better understand how these exts. differ in their overall compn. **NMR** anal.and in vitro cell assays of crude exts. and fractions were performed. Manufg.procedures to produce natural remedies can strongly affect the final compn. of the herbal medicines. Temp. and polarity of the solvents used for the extn. resulted to be 2 factors that affect the total amt. of Δ9-THC in the exts. and its relative **quantity** with respect to Δ9-THC-acid and other metabolites. Diffusion-edited 1H **NMR** (1D DOSY) and 1H **NMR** with suppression of the EtOH and water signals were used. With this method it was possible, without any evapn. or sepn. step, to distinguish between tinctures from different cannabis cultivars. This approach is proposed as a direct anal. of**plant** tinctures.

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50. **NMR-Based Metabolic Profiling and Metabonomic Approaches to Problems in Molecular Toxicology**

ByCoen, Muireann; Holmes, Elaine; Lindon, John C.; Nicholson, Jeremy K.

From Chemical Research in Toxicology (2008), 21(1), 9-27.Language:English, Database: CAPLUS, DOI: 10.1021/tx700335d

A review. We have reviewed the main contributions to the development of **NMR**-based metabonomic and metabolic profiling approaches for toxicol. assessment, biomarker discovery, and studies on toxic mechanisms. The metabonomic approach, (defined as the **quant**. measurement of the multiparametric metabolic **response** of living systems to pathophysiol. stimuli or genetic modification) was originally developed to assist interpretation in **NMR**-based toxicol. studies. However, in recent years there has been extensive fusion with metabolomic and other metabolic profiling approaches developed in **plant** biol., and there is much wider coverage of the biomedical and environmental fields. Specifically, metabonomics involves the use of spectroscopic techniques with statistical and math. tools to elucidate dominant patterns and trends directly correlated with time-related metabolic fluctuations within spectral data sets usually derived from biofluids or tissue samples. Temporal multivariate metabolic signatures can be used to discover biomarkers of toxic effect, as general toxicity screening aids, or to provide novel mechanistic information. This approach is complementary to proteomics and genomics and is applicable to a wide range

of problems, including disease diagnosis, evaluation of xenobiotic toxicity, functional genomics, and nutritional studies. The use of biol. fluids as a source of whole organism metabolic information enhances the use of this approach in minimally invasive longitudinal studies.

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51. **Quantitative analysis of sesquiterpene lactone cnicin in seven Centaurea species wild-growing in Serbia and Montenegro using 1H-NMR spectroscopy**

ByTesevic, Vele; Milosavljevic, Slobodan; Vajs, Vlatka; Janackovic, Pedja; Dordevic, Iris; Jadranin, Milka; Vuckovic, Ivan

From Journal of the Serbian Chemical Society (2007), 72(12), 1275-1280.Language:English, Database: CAPLUS, DOI: 10.2298/JSC0712275T

1H-**NMR** spectroscopy was applied for the **quant**. anal. of cnicin, a bioactive germacranolide type sesquiterpene lactone, in the aerial parts of seven wild-growing Centaurea species collected in Serbia and Montenegro. The anal. was performed by comparison of the integral of the one-proton signal of cnicin (H-13, δ5.75) with that of the two-proton singlet (δ6.98) of 2,6-bis(1,1-dimethylethyl)-4-methylphenol (BHT), used as the internal std. Cnicin, within the concn. range 1.06-6.12 mg/g, calcd. per wt. of the fresh **plant** material was detected in 6species, the exception being *C. salonitana*. This method allows the rapid and simple **quantification** of cnicin without any pre-purifn. step.

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52. **Antioxidative principals of *Jussiaea repens:* an edible medicinal plant**

ByHuang, Hai-Lan; Li, Dong-Li; Li, Xiao-Ming; Xu, Bo; Wang, Bin-Gui

From International Journal of Food Science and Technology (2007), 42(10), 1219-1227.Language:English, Database: CAPLUS, DOI: 10.1111/j.1365-2621.2006.01456.x

J. repens (JRL) is an edible medicinal **plant** and is also used as a vegetable by the local people in southwestern China. The crude ext. and its 4 fractions derived from JRL were evaluated for the 1,1-diphenyl-2-picrylhydrazyl radical-scavenging ability, hydroxyl radical-scavenging capacity and the potassium ferricyanide redn. property. The Et acetate-sol. fraction (EAF) and EAF6 (a subfraction derived from EAF) were the most valuable fraction and subfraction, resp. Furthermore, bioactivity-guided chromatog.fractionation revealed that 3 pure compds. greatly contributed to the antioxidant activities. Qual. and **quant**. analyses of the major antioxidant constituents in the ext. were systematically conducted by **NMR**, mass spectral analyses and RP-HPLC. The result demonstrated that rosmarinic acid (2.00 mg/g JRL dry wt.) quercetin 3-**O**-β-D-glucopyranoside (9.88 mg/g JRL dry wt.), and kaempferol 3-**O**-β-D-glucopyranoside (1.85 mg/g JRL dry wt.) were the major antioxidative constituents in JRL. These compds.are reported for the 1st time from this **plant**.

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53. **Quantitative metabolic profiles of tomato flesh and seeds during fruit development: complementary analysis with ANN and PCA**

ByMounet, Fabien; Lemaire-Chamley, Martine; Maucourt, Mickael; Cabasson, Cecile; Giraudel, Jean-Luc; Deborde, Catherine; Lessire, Rene; Gallusci, Philippe; Bertrand, Anne; Gaudillere, Monique; et al

From Metabolomics (2007), 3(3), 273-288.Language:English, Database: CAPLUS, DOI: 10.1007/s11306-007-0059-1

Tomato, an essential crop in terms of economic importance and nutritional quality, is also used as a model species for all fleshy fruits and for genomics of Solanaceae. Tomato fruit quality at harvest is a direct function of its metabolite content, which in turn is a result of many physiol. changes during fruit development. The aim of the work was to develop a global approach to characterize changes in metabolic profiles in two

interdependent tissues from the same tomato fruits. Abs. **quantification** data of compds.in flesh and seeds from 8 days to 45 days post anthesis (DPA) were obtained through untargeted (proton **NMR**, 1H-**NMR**) and targeted metabolic profiling (liq. chromatog. with diode array detection (LC-DAD) or gas chromatog. with flame ionization detection (GC-FID)). These data were analyzed with chemometric approaches. Kohonen self organizing maps (SOM) anal.of these data allowed us to combine multivariate (distribution of samples on Kohonen SOMs) and univariate information (component plane representation of metabolites) in a single anal. This strategy confirmed published data and brought new insights on tomato flesh and seed compn., thus demonstrating its potential in metabolomics. The compositional changes were related to physiol. processes occurring in each tissue. They pointed to (i) some parallel changes at early stages in relation to cell division and transitory storage of carbon, (ii) metabolites participating in the fleshy trait and (iii) metabolites involved in the specific developmental patterns of the seeds.

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54. **Probing the metabolic response of *Arabidopsis thaliana* to hypoxic stress using 1H-NMR**

ByBailey-Serres, Julia; Branco-Price, Cristina; Hamersky, Kayla A.; Larive, Cynthia K.

From Abstracts of Papers, 234th ACS National Meeting, Boston, MA, United States, August 19-23, 2007 (2007), ANYL-222.Language:English, Database: CAPLUS

The emerging field of systems biol., specifically metabolomics, has been used to study the **response** of *Arabidopsis thaliana* to hypoxic stress. Thirty small-mol. metabolites have been **quantified** after **plant** tissue was subjected to liq. extn. and proton **NMR** (1H-**NMR**) anal. Integral regions within **plant** ext. spectra were created to reflect **resonances** of fifty stds. involved in carbohydrate and amino acid metab. Peak areas were normalized to total integral intensity measured for each sample. Principal components anal. (PCA) was conducted to det. the most significant variances in the dataset. The PCA scores plots revealed good sepn. among the sample different treatments. Fold change between growth conditions and signal-log ratios were detd. and a t-test was used to establish confidence. Good correlations were obsd. between metabolic changes and those predicted from changes in gene expression levels.

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55. **Fast determination of the ripeness stage of strawberries using infrared spectroscopy combined with principal component analysis**

ByKwak, Chul Won; Choung, Dong-Ho; Min, Sung Ran; Kim, Suk Weon; Liu, Jang R.; Chung, Hoeil

From Analytical Sciences (2007), 23(7), 895-899.Language:English, Database: CAPLUS, DOI: 10.2116/analsci.23.895

The utility of IR spectroscopy for the detn.of strawberry ripeness has been successfully demonstrated. Transmission IR spectra were collected using dried liq. exts. from strawberry flesh. The overall IR feature provided fairly noticeable differences, and the ripeness stage was clearly identified using principal component anal. (PCA). Although all of the extd. components contributed to the resulting spectral features for discrimination, the variation of carbohydrate and amide residues played a major role for providing the selective spectral feature. **NMR** spectra were also collected to **quantify** the concns. of three small sugars (α-glucose, β-glucose and sucrose) as well as to evaluate the **NMR** spectral features at each ripeness step. The concns.of three sugars increased from early to late growth stages. Both IR and **NMR** spectroscopies were valuable to elucidate the metabolic signatures for the detg. of ripeness stage; however, IR spectroscopy could be more advantageous when fast and high throughput anal. is essential.

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56. **Nitrogen nutrition influences some biochemical responses to iron deficiency in tolerant and sensitive genotypes of Vitis**

ByJimenez, S.; Gogorcena, Y.; Hevin, C.; Rombola, A. D.; Ollat, N.

From Plant and Soil (2007), 290(1-2), 343-355.Language:English, Database: CAPLUS, DOI: 10.1007/s11104-006-9166-4

The effects of nitrogen source on iron deficiency **responses** were investigated in two Vitis genotypes, one tolerant to limestone chlorosis Cabernet Sauvignon (*Vitis vinifera* cv.) and the other susceptible Gloire de Montpellier (*Vitis riparia* cv.). **Plants** were grown with or without Fe(III)-EDTA, and with NO-3 alone or a mixt. of NO-3 and NH+4. Changes in pH of the nutrient soln. and root ferric chelate reductase (FC-**R**) activity were monitored **over** one week. **Quant**. metabolic profiling (1H-**NMR**) the activity of enzymes involved in org. acid metab. in root tips was detd. In iron free-solns., with NO3 as the sole nitrogen source, the typical Fe-deficiency **response** reactions as acidification of the growth medium and enhanced FC-**R** activity in the roots were obsd. only in the tolerant genotype. Under the same nutritional conditions, org. acid accumulation (mainly citrate and malate) was found for both genotypes. In the presence of NH4+, the sensitive genotype displayed some decrease in pH of the growth medium and an increase in FC-**R** activity. For both genotypes, the presence of NH+4 ions decreased significantly the org. acid content of roots. Both Vitis genotypes were able to take up NH+4 from the nutrient soln., regardless of their sensitivity to iron deficiency. The presence of **N**-NH+4 modified typical Fe stress **responses** in tolerant and sensitive Vitis genotypes.

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57. **Accumulation of glycinebetaine in rice plants that overexpress choline monooxygenase from spinach and evaluation of their tolerance to abiotic stress**

ByShirasawa, Kenta; Takabe, Tomoko; Takabe, Tetsuko; Kishitani, Sachie

From Annals of Botany (Oxford, United Kingdom) (2006), 98(3), 565-571.Language:English, Database: CAPLUS, DOI: 10.1093/aob/mcl126

Glycinebetaine (GB), a quaternary ammonium compd., is a very effective compatible solute. In higher **plants**, GB is synthesized from choline (Cho) via betaine aldehyde (BA). The first and second steps in the biosynthesis of GB are catalyzed by choline monooxygenase (CMO) and by betaine aldehyde dehydrogenase (BADH), resp. Rice (Oryza sativa), which has two genes for BADH, does not accumulate GB because it lacks a functional gene for CMO. Rice **plants** accumulate GB in the presence of exogenously applied BA, which leads to the development of a significant tolerance to salt, cold and heat stress. The goal in this study was to evaluate and to discuss the effects of endogenously accumulated GB in rice. Transgenic rice **plants** that overexpressed a gene for CMO from spinach (Spinacia oleracea) were produced by Agrobacterium-mediated transformation. After Southern and western blotting anal., GB in rice leaves was **quantified** by 1H-**NMR** spectroscopy and the tolerance of GB-accumulating **plants** to abiotic stress was investigated. Transgenic **plants** that had a single copy of the transgene and expressed spinach CMO accumulated GB at the level of 0.29-0.43 μmol g-1 d. wt and had enhanced tolerance to salt stress and temp. stress in the seedling stage. In the CMO-expressing rice **plants**, the localization of spinach CMO and of endogenous BADHs might be different and/or the catalytic activity of spinach CMO in rice **plants** might be lower than it is in spinach. These possibilities might explain the low levels of GB in the transgenic rice **plants**. It was concluded that CMO-expressing rice **plants** were not effective for accumulation of GB and improvement of productivity.

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58. **Covalent linkages between cellulose and lignin in cell walls of coniferous and nonconiferous woods**

ByJin, Zhenfu; Katsumata, Kyoko S.; Lam, Thi Bach Tuyet; Iiyama, Kenji

From Biopolymers (2006), 83(2), 103-110.Language:English, Database: CAPLUS, DOI: 10.1002/bip.20533

Covalent linkages between wall polysaccharides and lignin, esp. linkage between cellulose and lignin were discussed by carboxymethylation technique of whole cell walls of coniferous and nonconiferous woods.

Hydroxyl groups of **plant** cell walls polysaccharides were highly substituted, but not those of lignin by carboxymethyl groups under the used conditions, and sepd. into water-sol. and insol. fractions by water extn. Carboxymethylated wall polysaccharides linked covalently with lignin were distributed into the water-insol. fractions. Compn.of carboxymethylated sugar residues in the both fractions was analyzed **quant**. by 1H **NMR** spectroscopy after hydrolyzation with D2SO4 in D2O. More than half of cellulose linked covalently with lignin in coniferous wood, but only one-sixth of cellulose was involved in the linkage in nonconiferous wood. The major noncellulosic wall polysaccharides of coniferous wood also linked significantly with lignin. On the other hand, noncellulosic wall polysaccharides of nonconiferous wood were involved slightly in the covalent linkage with lignin. The situation of linkage between wall polysaccharides contg.cellulose and lignin was visualized by scanning electron micrographs.

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59. **Identification of Major and Minor Constituents of *Harpagophytum procumbens* (Devil's Claw) Using HPLC-SPE-NMR and HPLC-ESIMS/APCIMS**

ByClarkson, Cailean; Strk, Dan; Hansen, Steen Honore; Smith, Peter J.; Jaroszewski, Jerzy W.

From Journal of Natural Products (2006), 69(9), 1280-1288.Language:English, Database: CAPLUS, DOI: 10.1021/np0601612

The HPLC-SPE-**NMR** technique, supported by HPLC-MS measurements, was used to det. structures of major as well as some minor constituents of ethanol and petroleum ether exts. of*Harpagophytum procumbens* (Devil's claw) roots. This method was also shown to be applicable for rapid and precise online identification of secondary metabolites present in com. herbal products of *H. procumbens*. A total of 15 compds. (1-14 and 17) were identified from the ethanol and petroleum ether exts., including a novel Diels-Alder dimer 14. Optimization of the HPLC-SPE-**NMR** expts.included**quant**. 1H **NMR** measurements, detn. of trapping and elution efficiency, effect of multiple trapping of analytes, use of various deuterated solvents for SPE cartridge elution, and effect of post-column diln. ratio of eluent with water. Linear accumulation of apolar and relatively polar analytes was demonstrated for at least 8-10 repeated trappings, resulting in greatly improved signal-to-noise ratios in **NMR** spectra and reduced acquisition times. Thus, the HPLC-SPE-**NMR** technique provides an efficient means of identification of multiple components of crude exts. By allowing online generation of high-quality 2D **NMR** data without traditional purifn.of ext. components, the HPLC-SPE-**NMR** methodol. represents a paradigm shift in natural products research with respect to structure elucidation.

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60. **The structure of the O-specific polysaccharide of the lipopolysaccharide from Burkholderia gladioli pv. agaricicola**

ByKarapetyan, Gnuni; Kaczynski, Zbigniew; Iacobellis, Nicola S.; Evidente, Antonio; Holst, Otto

From Carbohydrate Research (2006), 341(7), 930-934.Language:English, Database: CAPLUS, DOI: 10.1016/j.carres.2006.02.010

A neutral **O**-specific polysaccharide contg. D-mannose, D-rhamnose and D-galactose was obtained by mild acid hydrolysis of the lipopolysaccharide of the **plant** pathogenic bacterium Burkholderia gladioli pv. agaricicola. By means of compositional analyses and **NMR** spectroscopy, the chem. repeating unit of the polymer was identified as a linear trisaccharide of the structure shown below, in which the mannose residue was **quant**. acetylated at C-2.

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61. **Validation of 1H NMR spectroscopy as an analytical tool for methylamine metabolites in urine**

ByLee, Martin B.; Storer, Malina K.; Blunt, John W.; Lever, Michael

From Clinica Chimica Acta (2006), 365(1-2), 264-269.Language:English, Database: CAPLUS, DOI: 10.1016/j.cca.2005.09.004

Methylamines have many metabolic roles and there is an increasing demand for their measurement. Glycine betaine is an important osmolyte, and a reservoir for Me groups. Proline betaine and trigonelline are important dietary betaines. Trimethylamine, derived from gut **flora**, is normally converted to trimethylamine oxide but in fish odor syndrome' is excreted as TMA. These compds.are all suitable for **quantification** by 1H **NMR** spectroscopy as they all have Me protons. Urine samples are acidified and 1H **NMR** spectra are obtained using presatn. for water suppression. Peak integrals or heights are compared to an internal std. of acetonitrile. Inter- and intra-assay CV's were < 5% for TMAO and creatinine, and < 10% for the other analytes. **Responses** were linear from 50 to 1000 μ**M** for all metabolites, and recoveries were ≥ 97%. Limits of detection using **NMR** are slightly higher than alternative HPLC assays (15-25 μ**M**). However, sensitivity is adequate for the detection of raised levels in urine, and sample anal. was complete in less than 5 min. 1H **NMR** spectroscopy is a convenient, rapid and economical option for the detn. of betaines and related compds. in urine in a single anal.

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62. **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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63. **Quantitative metabolic profiling by 1-dimensional 1H-NMR analyses: application to plant genetics and functional genomics**

ByMoing, Annick; Maucourt, Mickael; Renaud, Christel; Gaudillere, Monique; Brouquisse, Renaud; Lebouteiller, Benedicte; Gousset-Dupont, Aurelie; Vidal, Jean; Granot, David; Denoyes-Rothan, Beatrice; et al

From Functional Plant Biology (2004), 31(9), 889-902.Language:English, Database: CAPLUS, DOI: 10.1071/FP04066

Metabolic profiling by 1-dimensional (1-D) 1H-**NMR** was tested for abs. **quantification** of sol. sugars, org. acids, amino acids and some secondary metabolites in fruit, roots and leaves. The metabolite responsible for each peak of the 1H-**NMR** spectra was identified from spectra of pure compds. Peak identity was confirmed by the addn. of a small amt. of com.-available pure substance. 1H-**NMR** spectra acquisition was automated. 1H-**NMR** abs. **quantification** was performed with a synthesized electronic ref. signal and validated by comparison with enzymic or HPLC analyses; the correlation coeffs. between 1H-**NMR** data and enzymic or HPLC data were highly significant. Depending on the species and tissues, 14-17 metabolites could be **quantified** with 15-25 min acquisition time. The detection limit was approx. 1-9 μg in the **NMR** tube, depending on the compd. **Quant**. data were used for (1) a genetic study of strawberry fruit quality, (2) a functional study of tomato transformants overexpressing hexokinase and (3) a study of Arabidopsis phosphoenolpyruvate carboxylase transformants with several lines showing decreased activity of the

enzyme. Biochem.phenotyping of the fruits of a strawberry offspring allowed the detection of **quant**. trait loci (QTL) controlling fruit quality. Comparison of the roots of wild types and hexokinase tomato transformants using principal component anal.of metabolic profiles revealed that environmental factors, i**.e.** culture conditions, can significantly modify the metabolic status of **plants** and thus hide or emphasize the expression of a given genetic background. The decrease in phosphoenolpyruvate carboxylase activity (up to 75%) in Arabidopsis transformants impacted on the metabolic profiles without compromising **plant** growth, thus supporting the idea that the enzyme has a low influence on the carbon flux through the anaplerotic pathway.

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64. **Determination of isoperoxisomicine A1 content in peroxisomicine A1 batches by 1H NMR**

ByFernandez-Ramirez, Adrian; de la Luz Salazar-Cavazos, Ma; Rivas-Galindo, Veronica; Ceniceros-Almaguer, Lucia; de Torres, Noemi Waksman

From Analytical Letters (2004), 37(12), 2433-2444.Language:English, Database: CAPLUS, DOI: 10.1081/AL-200029366

Peroxisomicine A1 (PA1) is a natural product that shows biol. activity. After its extn.and isolation from the **plant**, there is still between 3 and 5% remaining of an isomer (isoperoxisomicine A1, isoPA1), which is also present in the original matrix. A simple and reliable **quantification** method was developed by proton **NMR** spectroscopy to det. the isoperoxisomicine amt. in these PA1 batches. Two methods were applied to the **quantification**: the relative area method and an abs. method by an internal std. No significant differences were found between them. The simplicity of the 1st one makes it desirable for routine anal. The validated method has adequate linearity and precision, because it allows measuring as low as 1% of isoPA1 content.

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65. **Quantitative analysis of cannabinoids from *Cannabis sativa* using 1H-NMR**

ByHazekamp, Arno; Choi, Young Hae; Verpoorte, Robert

From Chemical & Pharmaceutical Bulletin (2004), 52(6), 718-721.Language:English, Database: CAPLUS, DOI: 10.1248/cpb.52.718

A 1H-**NMR** method has been developed for the **quant**. anal. of pure cannabinoids and for cannabinoids present in *Cannabis sativa***plant** material without any chromatog. purifn. The expt. was performed by the anal. of singlets in the range of δ 4.0-7.0 in the 1H-**NMR** spectrum, in which distinguishable signals of each cannabinoid are shown. **Quantitation** was performed by calcg. the relative ratio of the peak area of selected proton signals of the target compds. to the known amt. of the internal std., anthracene. For this method no ref. compds.are needed. It allows rapid and simple **quantitation** of cannabinoids with a final anal. time of only 5 min without the need for a pre-purifn. step.

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66. **Quantitative analysis of ephedrine analogues from Ephedra species using 1H-NMR**

ByKim, Hye Kyong; Choi, Young Hae; Chang, Wen-te; Verpoorte, Robert

From Chemical & Pharmaceutical Bulletin (2003), 51(12), 1382-1385.Language:English, Database: CAPLUS, DOI: 10.1248/cpb.51.1382

Four ephedrine analogs such as ephedrine, pseudoephedrine, methylephedrine, and methylpseudoephedrine were detd. by 1H-**NMR** from Ephedra species. In the region of δ 5.0-4.0, the signals of H-1 attached to the same carbon with a hydroxyl, were well sepd. from each other in CDCl3. The amt. of each alkaloid was calcd. by the relative ratio of the intensity of H-1 signal to the known amt. of internal std., 200 μg of anthracene. This method allows rapid detn. of the **quantity** of four ephedrine alkaloids from Ephedra species. The amt. of these alkaloids was in the range of 1.0-2.0% of dry wt. depending on the **plant**materials.

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67. **L-5-hydroxytryptophan: Antioxidant and anti-apoptotic principle of the intertidal sponge *Hymeniacidon heliophila***

ByLysek, Nicola; Kinscherf, Ralf; Claus, Ralf; Lindel, Thomas

From Zeitschrift fuer Naturforschung, C: Journal of Biosciences (2003), 58(7/8), 568-572.Language:English, Database: CAPLUS

The intertidal sponge *Hymeniacidon heliophila* which survives under intense sunlight contains the antioxidant amino acid L-5-hydroxytryptophan (L-5-HTP) as major constituent. The content of L-5-HTP was detd. as (0.45 ± 0.23)% of the dry wt. by **quant**. **NMR**-spectroscopical anal. with an internal std. Other known antioxidants such as flavonoids, carotenoids or tocopherol derivs. were absent. Both the oxidn.potential and the concn. of L-5-HTP in H. heliophila correspond to the values obsd. for flavonoids being major antioxidants in **plants**. It was shown that L-5-HTP suppresses UV-induced apoptosis in human monocytes at the same concns. as it occurs in the sponge tissue.

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68. **The secoiridoid biophenols of *Olea europea L. drupes* and the role of their metabolites**

ByUccella, Nicola A.

From Acta Horticulturae (2002), 586(Vol. 2, Proceedings of the 4th International Symposium on Olive Growing, 2000, Vol. 2), 489-492.Language:English, Database: CAPLUS

A review. Secoiridoid biophenols (secoBP), contained in olive drupes, are enzymically activated to provide a complex mechanism for the biol. defense against pathogen attack. Their identification, **quantitation** and metabolic behavior are investigated, by HPLC, **NMR** and mol. dynamic expts. for the improvement of olive growing and of the prodn.of oil and table olives.

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69. **Determination of nitrobenzene oxidation products by GC and 1H-NMR spectroscopy using 5-iodovanillin as a new internal standard**

ByKatahira, Rui; Nakatsubo, Fumiaki

From Journal of Wood Science (2001), 47(5), 378-382.Language:English, Database: CAPLUS, DOI: 10.1007/BF00766789

The nitrobenzene oxidn.method was modified to obtain more reproducible data and more structural information about lignin, not only by gas chromatog. (GC) but also by proton **NMR** (1H-**NMR**) spectroscopy for **quant**. detn.of the oxidn. products and to simplify the procedures. The nitrobenzene oxidn.mixt. was directly extd. after acidification without preextn. of byproducts. The direct extn.made the extractive step easy and gave reproducible data. 5-Iodovanillin was selected as a new internal std. The reason for this selection was that 5-iodovanillin did not exist in the nitrobenzene oxidn. products from any **plant** species and had an aldehyde group whose peak did not overlap with the other aldehyde peaks on an 1H-**NMR** spectrum. Thus, the use of 5-iodovanillin enabled us to **quantify** p-hydroxybenzaldehyde, vanillin, and syringaldehyde in oxidn. products on the basis of 1H-**NMR** anal. as well as GC. Furthermore, more information about the condensed structure of lignin was derived by comparing the 1H-**NMR** and GC analyses.

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70. **Cambial coniferin content as an indicator of the health status of conifers**

BySteeves, V. J.; Savidge, R. A.

Edited bySavidge, Rodney A.; Barnett, J. A.; Napier, Richard

From Cell and Molecular Biology of Wood Formation (2000), 57-65.Language:English, Database: CAPLUS

The coniferin content in the cambium of conifers can be monitored by **quant**. **NMR** spectroscopy, concomitantly observing the extent of cambial cell division by microscopy. Coniferin was used to monitor the health status of Pinus resinosa whose vigor was manipulated by girdling, defoliation, debudding, and derooting.

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71. **Probing plant metabolism with NMR**

ByRatcliffe, R. George; Shachar-Hill, Yair

From Annual Review of Plant Physiology and Plant Molecular Biology (2001), 52, 499-526.Language:English, Database: CAPLUS, DOI: 10.1146/annurev.arplant.52.1.499

A review with 165 refs.Anal.methods for probing **plant** metab. are taking on new significance in the era of functional genomics and metabolic engineering. Among the available methods, **NMR** (**NMR**) spectroscopy is a technique that can provide insights into the integration and regulation of **plant** metab. through a combination of in vivo and in vitro measurements. Thus **NMR** can be used to identify, **quantify**, and localize metabolites, to define the intracellular environment, and to explore pathways and their operation. We review these applications and their significance from a metabolic perspective. Topics of current interest include applications of **NMR** to metabolic flux anal., metabolite profiling, and metabolite imaging. These and other areas are discussed in relation to **NMR** investigations of intermediary carbon and nitrogen metab. We conclude that metabolic **NMR** has a continuing role to play in the development of a **quant**. understanding of **plant** metab. and in the characterization of metabolic phenotypes.

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72. **Nuclear magnetic resonance spectroscopy for the detection of values of adsorbable organic halogens (AOX) caused by PVC**

ByDiehl, Bernd; Mertens, Melanie; Ockels, Werner

From WLB, Wasser, Luft und Boden (1999), 43(1-2), 28-29.Language:German, Database: CAPLUS

The application of a **NMR** method for the detn.of AOX caused by PVC in sewage sludge and sewer slime is described. Dried samples are subjected to a 2-fold Soxhlet extn. with THF (tetrahydrofuran) as solvent of the second step to selectively ext. PVC. From the THF ext. an 1H-**NMR** spectrum is taken, PVC is **quantifiedover** an internal std., and a theor. AOX value (caused by PVC) can be calcd. from the PVC amt. This method can be used to det. if high AOX levels in sewage sludge of communal treatment **plants** were produced by high PVC contents. PVC loads of 50-13,000 mg/kg dry residue were found in different sewage sludge samples corresponding to theor. AOX values of 29-7581 mg/kg dry residue. In addn., the anal.of sewer slime served to locate the point of PVC discharge into the sewer system.

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73. **Quantitative and qualitative study on natural rubber latex of Euphorbia larica Boiss and Euphorbia tirucalli L. and the whole plants**

ByEbrahimzadeh, H.; Aboulmaali, S.

From Iranian Journal of Polymer Science & Technology (Persian Edition) (1998), 11(2), 113-118.Language:Persian, Database: CAPLUS

The natural rubber is a polyisoprene of high mol. wt. Considering the wide range of applications in various industries, hospitals and the high cost for this raw material, many researchers have been involved to obtain

some new sources of natural rubber. In this work, the **quant**. measurement of rubber in the stem and the raw latex of the whole **plant** of two species in various seasons indicate that the highest amt. of rubber in spring time is obtained from the stem of **E**. Larica and in winter time it is obtained from the stem of **E**. Tirucalli L. The highest amt. of rubber from the raw latex of these two **plants** is obtained, however, in autumn and summer seasons. The degree of purity of the extd.rubber from the stem and the raw latex is detd. and studied by IR and 1H **NMR** spectroscopy. Further investigations on av. mol. wt. show that in winter the rubbers' wt. av. mol. wt. from **E**. Larica is 1.298x105 and that from **E**. Tirucalli is 1.29007x105.In addn., the extd.rubbers from these two species in spring and summer seasons have the highest and in autumn and winter seasons have the lowest no. av. mol. wts.

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74. **Determination of atropine and scopolamine by proton nuclear magnetic resonance spectroscopy**

ByNaqvi, A. A.; Mandal, S.; Verma, R. K.

From Phytochemical Analysis (1998), 9(4), 168-170.Language:English, Database: CAPLUS, DOI: 10.1002/(SICI)1099-1565(199807/08)9:4<168::AID-PCA408>3.0.CO;2-Q

A simple, accurate and specific proton **NMR** spectrometric method is described for the **quant**. detn. of atropine and scopolamine in **plants**. The method is based on a comparison of the integrated peak areas of the **N**-CH3 protons of both compds. (centered at 2.15 and 2.5 ppm) with that of the sharp singlet of 1, 4-dioxane (positioned at 3.5 ppm) which is used as the internal std.

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75. **Quantitative analysis of lipoxygenase metabolites in lipids by NMR-spectroscopy**

ByFeussner, Ivo; Porzel, Andrea; Wasternack, Claus; Kuhn, Hartmut

From BIOspektrum (1997), 3(5), 54,57-58.Language:German, Database: CAPLUS

A method is presented for the detn. of lipoxygenase metabolites in lipids by HPLC and online chemiluminescence detection, and the anal. of oxygenated lipids by 2-dimensional **NMR** and 1H-**NMR**. By this method, the **quant**. anal.of oxygenated lipids and their isomers is possible for the 1st time in lipid exts. from**plants** or animals.

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76. **The role of H-1 NMR Spectroscopy in K-Resin production**

ByStewart, Carol A.; Sardashti, Maziar; Wharry, Stephen

From Book of Abstracts, 214th ACS National Meeting, Las Vegas, NV, September 7-11 (1997), TECH-008.Language:English, Database: CAPLUS

K-Resin is a styrene/butadiene copolymer which is produced by Phillips Petroleum Company. H-1 **NMR** Spectroscopy plays a vital role in assuring product specifications of the polymer. K-Resin samples from the **plant** are sent directly to Phillips Research Center **NMR** Lab. for anal. Solns. H-1 **NMR** is used to produce high precision **quant**. data on the amts. and distribution of styrene and butadiene in the polymers. These data are used to calibrate the at-line near IR (NIR) instrument at the prodn. **plant**. The NIR measurement results are also validated periodically by H-1 **NMR**. An overview of the K-Resin prodn.at Phillips, and some exptl. details of the H-1 **NMR** measurement will be presented.

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77. **Characterization of complex substance mixtures by NMR**

ByMeusinger, R.; Muehl, O.

From Berichte - Deutsche Wissenschaftliche Gesellschaft fuer Erdoel, Erdgas und Kohle, Tagungsbericht (1996), 9603(Beitraege zur DGMK-Fachbereichstagung "Energetische und Stoffliche Nutzung von Abfaellen und Nachwachsenden Rohstoffen", 1996), 401-408.Language:German, Database: CAPLUS

Investigations of the mobile phases in solid natural products (**e**.g., wood resin, brown coal, oil **plants**) and the characterization of raw materials by applying the single-pulse (SP) **NMR** technique are presented. Direct qual. and **quant**. anal.of fatty acid esters and oils in sunflower and oil flax seeds by SP **NMR** was demonstrated. Applying the same method it could also be possible to study solid-to-fluid phase transitions of the materials.

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78. **Quality assessment of Radix Codonopsis by quantitative nuclear magnetic resonance**

ByLi Chia-Ying; Xu Hong-Xi; Han Quan-Bin; Wu Tian-Shung

From Journal of chromatography.A (2009), 1216(11), 2124-9.Language:English, Database: MEDLINE

Radix Codonopsis (Dangshen) is a famous traditional Chinese medicine and has long been used for replenishing energy deficiency, strengthening the immune system, lowering blood pressure and improving appetite in China, Japan and Korea. A highly specific **quantification** method using (1)H**NMR** has been developed for the simultaneous determination of novel quaternary ammonium alkaloids codotubulosine A and B, adenosine and 5-(hydroxymethyl)furfural in Radix Codonopsis materials Codonopsis pilosula, C. pilosula var. modesta, C. tangshen, C. tubulosa, C. subglobosa, C. clematidea, C. lanceolota and Campanumoea javanica collected from different regions of China and Taiwan. A solid-phase extraction with C-18 cartridge followed by elution with water can easily remove sugars the major components that may affect the determination of target constituents. In the (1)H**NMR** spectrum, the signals of **N**-CH(3) of codotubulosine A (delta 2.75) and B (delta 2.83), H-8 of adenosine (delta 8.15), and CHO signal of 5-(hydroxymethyl)furfural (delta 9.49) are well separated from other signals in [(2)H(4)]methanol. The **quantity** of the compounds was calculated by the relative ratio of the integral values of the target peaks of each compound to the known amount of internal standard pyrazine. The described **NMR** method is found to be relatively simple, specific, precise and accurate for the quality control of Radix Codonopsis herbs and no reference compounds are required for calibration curves, in comparison to conventional HPLC methods, for instance.

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79. **Analysis of organic compounds in aqueous samples of former ammunition plants**

ByLevsen, K.; Preiss, A.; Berger-Preiss, E.

From Proceedings of SPIE-The International Society for Optical Engineering (1995), 2504(Environmental Monitoring and Hazardous Waste Site Remediation, 1995), 350-61.Language:English, Database: CAPLUS

Approaches are presented for the extn. and anal. of explosives and related compds. in aq. samples from former ammunition prodn. sites. **Quant**. extn.of nitroaroms. and polar nitramines such as RDX and HMX is achieved by solid phase extn. with styrene-divinylbenzene polymers. Proton **NMR** (1H-**NMR**) was used to identify and **quantify** unknowns in ammunition wastewater. Automated multiple development high performance thin layer chromatog.was applied to the anal. of this class of compds.

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80. **Novel techniques for growth characterization and phytochemical analysis of plant cell suspension**

cultures

BySchripsema, Jan; Verpoorte, Robert

From Journal of Natural Products (1995), 58(9), 1305-14.Language:English, Database: CAPLUS, DOI: 10.1021/np50123a001

Major problems assocd.with**plant** cell suspension cultures are the variability and instability of the cultures. Therefore, characterization of a suspension culture in every expt. is necessary to allow a comparison between different expts. For this purpose, some new techniques were developed. A technique for growth characterization was developed based on the dissimilation of the cultures. The main advantages of this method are that it is non-destructive, it supplies a dissimilation curve for each individual flask, and it is accurate. Here the authors present an accurate calcn. for the curves and the most accurate way to measure them. Another technique was developed for the rapid **quantification** of the main org. intracellular components (esp. carbohydrates and amino acids). Aq. exts.are prepd. and the components are **quantified** by 1H-**NMR** spectroscopy. The main advantages of this method are non-selectivity and good sensitivity. The developed techniques were applied in an expt. in which cultures growing on media contg. different nitrogen levels were compared. The optimum **quantity** of nitrogen for growth and alkaloid prodn.was half the amt. normally present in Murashige-Skoog medium.

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81. ### High-performance liquid chromatographic and proton magnetic resonance spectroscopic methods for quality evaluation of Paeonia roots

ByCai, Y.; Phillipson, J. D.; Harper, J. I.; Corne, S. J.

From Phytochemical Analysis (1994), 5(4), 183-9.Language:English, Database: CAPLUS, DOI: 10.1002/pca.2800050403

The root of several Paeonia species is one of the most frequently used **plant** materials in traditional Chinese medicine, and has been shown to have a variety of pharmacol. properties. Owing to the differences in species, cultivation, harvesting and processing, the quality of the roots as com. medicinal herbs varies to a considerable extent. A **quant**. high-performance liq. chromatog.method has been developed for the quality evaluation of Paeonia root, and a no. of com. samples have been examd. A **quant**. 1H-**NMR** procedure has also been developed for this purpose, and this may find a wider application in the anal. of other **plant** materials.

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82. ### Identification and determination of 18α- and 18β glycyrrhetic acid

BySappe, Francoise; Archavlis, Albert; Latrides, Marie Christine; Artaud, Jacques

From Annales des Falsifications de l'Expertise Chimique et Toxicologique (1993), 86(919), 223-32.Language:French, Database: CAPLUS

Acid hydrolysis of glycyrrhizic acid of licorice (*Glycyrrhiza glabra*) roots yields 18β-glycyrrhetic acid, but the 18α isomer may also be present. Attempts to sep. the isomers by reverse-phase HPLC in the free form or as acetylated derivs.were unsuccessful. The free isomers were sepd. by TLC on silica gel (AcOH-MeOH-Et2NH, 7:4:1.5) when 3% of the 18α isomer was present. Proton **NMR** was the most sensitive method for **quantifying**<1% of the 18α isomer.

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83. ### Rapid and simple determination of O-acetyl groups bound to plant cell walls by acid hydrolysis and 1H NMR measurement

Byliyama, Kenji; Thi Bach Tuyet Lam; Kasuya, Natsuki; Stone, Bruce A.

From Phytochemistry (1994), 35(4), 959-61.Language:English, Database: CAPLUS

Acetyl groups on cell wall polysaccharides can be rapidly and conveniently **quantified** by 1H **NMR** spectroscopy following their liberation by acid hydrolysis in D2SO4-D2O. The method works equally well for **O**- and **N**-acetyl substituents. The method has been used to measure total acetyl groups in walls from grass and legume forage **plants**.

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84. **Examining the chemical composition of quinoid pigments from *Lithospermum erythrorhizon* Sieb. et Zucc. cell culture BK-39**

ByFedoreyev, S. A.; Denisenko, V. A.; Kulesh, N. I.; Krasovskaya, N. P.; Kozyrenko, M. M.; Bulgakov, V. P.; Zhuravlev, Yu. N.

From Khimiko-Farmatsevticheskii Zhurnal (1993), 27(6), 33-7.Language:Russian, Database: CAPLUS

The content and **quant**. ratio of quinoid pigments from *Lithospermum erythrorhizon* callus culture BK-39 were studied by HPLC and **NMR** techniques. The *Lithospermum erythrorhizon* cell culture produced 8 shikonin derivs. and 4 benzoquinonylfuran pigments. Three of them, i**.e.**, propionylshikonin, isobutyryl- and isovalerylbenzoquinonylfuran derivs., were found for the first time in *Lithospermum erythrorhizon* callus cells.

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85. **Investigation of extracts of plant cell cultures by proton nuclear magnetic resonance spectroscopy**

BySchripsema, Jan; Verpoorte, Robert

From Phytochemical Analysis (1991), 2(4), 155-62.Language:English, Database: CAPLUS, DOI: 10.1002/pca.2800020403

A method for the rapid phytochem.characterization of **plant** cell cultures, using 1H **NMR** spectroscopy, is described. Aq. exts.were prepd. from a large no. of **plant** cell suspension cultures. Following lyophilization, the exts.were redissolved in D2O contg. an internal std. to enable **quantification** and 1H **NMR** spectra were measured. In the spectra, signals from sugars and amino acids were obsd. The patterns obtained were characteristic of cell lines, with related lines typically showing similar characteristics, **e**.g., Tabernaemontana suspension cultures are characterized by their ability to accumulate large amts. of arginine, while Catharanthus cultures mainly accumulate glutamine. The amts. of amino acids accumulated were detd. mainly by the medium on which the cell cultures were growing. Murashige and Skoog medium supported nitrogen storage in amino acids, while Gamborg B5 medium did not. Ref. spectra of some of the most common constituents are presented, together with data enabling a direct **quantification** of these compds. in aq. exts.

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86. **Intra- and extracellular carbohydrates in plant cell cultures investigated by proton-NMR**

BySchripsema, J.; Erkelens, C.; Verpoorte, R.

From Plant Cell Reports (1991), 9(9), 527-30.Language:English, Database: CAPLUS

With the aim of **quantifying** intra- and extracellular carbohydrates, media and cell exts.from a *Tabernaemontana divaricata***plant** cell-suspension culture were investigated with 1H **NMR**. For suppression of the solvent peak the Meiboom-Gill modification of the Carr-Purcell (CPMG) spin-echo sequence was used after addn. of a paramagnetic relaxation agent (Mn2+) to the sample. Several aspects of this method were optimized (the Mn concn., the interpulse delay and the no. of spin-echo cycles) so as to obtain a rapid and easy method in which no pretreatment of media or cell-exts. was needed. Besides the speed and ease of the method, the direct identification of carbohydrates and other main components is also an advantage. The exhaustion of extracellular carbohydrates coincided with the max. amt. of intracellular carbohydrates. The intracellular carbohydrates, i**.e.** glucose and fructose, were consumed at a low rate, during several weeks.

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87. **Quantitative proton Fourier transform nuclear magnetic resonance spectroscopic analysis of mixtures of pyrrolizidine alkaloids from *Senecio vulgaris***

ByPieters, L. A. C.; Vlietinck, A. J.

From Fresenius' Zeitschrift fuer Analytische Chemie (1985), 321(4), 355-8.Language:English, Database: CAPLUS

An anal.method was developed to obtain qual. and **quant**. information on pyrrolizidine alkaloid mixts. from*S. vulgaris* (Compositae) by means of 1H Fourier-transform (FT) **NMR** spectroscopy. This **plant** contains not only seneciphylline, senecionine, and retrorsine, but also the corresponding geometrical **E**-isomers spartioidine, integerrimine, and usaramine. Some general instrumental conditions necessary for **quant**. FT **NMR** were established, which enabled the detn. of the total alkaloid level, the **N**-oxide level, the total Z/**E** ratio, and the amt. of each Z/**E** isomer pair with good precision. The possibilities of using this method to analyze pyrrolizidine mixts.from other Senecio species, and some advantages and limitations of **quant**. anal. by means of 1H FT **NMR** are discussed.

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88. **Proton NMR spectra of dioxane lignins of some plants of the mallow family**

BySmirnova, L. S.; Dalimova, G. N.; Abduazimov, Kh. A.

From Khimiya Prirodnykh Soedinenii (1980), (4), 560-3.Language:Russian, Database: CAPLUS

NMR spectral anal.of dioxane lignins of Althaea, kenaf, and cotton showed that the no. of arom. protons did not correlate with the no. of methoxy groups, demonstrating the differing extent of condensability among the studied compds. In all prepns.there was a small amt. of coumarin structure. The **NMR** spectra of the aliph.hydroxyl groups differentiated the primary (γ-OH) from the secondary (α-OH) ones, providing their **quant**. detn.

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89. **Quantitative determination of atractylon in Atractylodis Rhizoma and Atractylodis Lanceae Rhizoma by 1H-NMR spectroscopy**

ByHasada Keiko; Yoshida Takamitsu; Yamazaki Takeshi; Sugimoto Naoki; Nishimura Tetsuji; Nagatsu Akito; Mizukami Hajime

From Journal of natural medicines (2010), 64(2), 161-6.Language:English, Database: MEDLINE

(1)H-**NMR** spectroscopy was successfully applied to the **quantitative** determination of atractylon in Atractylodis Rhizoma (dried rhizomes of *Atractylodes ovata* and *A. japonica*) and Atractylodis Lanceae Rhizoma (dried rhizomes of *Atractylodes lancea* and *A. chinensis*). The analysis was carried out by comparing the integral of the H-12 singlet signal of atractylon, which was well separated in the range of delta 6.95-7.05 ppm in the **NMR** spectrum, with the integral of a hexamethyldisilane (HMD) signal at delta 0 ppm. The atractylon contents obtained by the (1)H-**NMR** spectroscopy were consistent with those obtained by the conventional HPLC analysis. The present method requires neither reference compounds for calibration curves nor sample pre-purification. It also allows simultaneous determination of multiple constituents in a crude extract. Thus, it is applicable to chemical evaluation of crude drugs as a powerful alternative to various chromatographic methods.

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90. **The transesterification of rapeseed and waste sunflower oils: Mass-transfer and kinetics in a**

laboratory batch reactor and in an industrial-scale reactor/separator setup

ByKlofutar B; Golob J; Likozar B; Klofutar C; Zagar E; Poljansek I

From Bioresource technology (2010), 101(10), 3333-44.Language:English, Database: MEDLINE

We have investigated the transesterification of rapeseed (RO) and waste sunflower (SO) oils with methanol in the presence of potassium hydroxide as a catalyst. The transesterification of tri-acylglycerols was first conducted in a batch reactor. The effect of the temperature on the reaction rates was studied at a constant molar ratio of the alcohol to tri-acylglycerols (6:1) and for a constant concentration of the catalyst (1.0wt%). Size-exclusion chromatography and (1)H**NMR** spectroscopy were used to **quantitatively** monitor the transesterification reaction. The mass-transfer coefficients of the tri-acylglycerols during the initial transesterification stage were found to be 0.2-1.2x10(-5)mmin(-1), depending on the type of oil and the temperature. Calculated activation energies implied that at higher temperatures the formation of mono-acylglycerols and glycerole was favored for the SO (93kJ/mol for the forward and 48kJ/mol for the backward reaction) and the RO (47kJ/mol for the forward and 36kJ/mol for the backward reaction), respectively. For the continuous industrial reactor/separator setup, the optimum methanol recycle ratio was established as 0.0550.

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91. **Detection of refined olive oil adulteration with refined hazelnut oil by employing NMR spectroscopy and multivariate statistical analysis**

ByAgiomyrgianaki Alexia; Petrakis Panos V; Dais Photis

From Talanta (2010), 80(5), 2165-71.Language:English, Database: MEDLINE

NMR spectroscopy was employed for the detection of adulteration of refined olive oil with refined hazelnut oil. Fatty acids and iodine number were determined by (1)H**NMR**, whereas (31)P **NMR** was used for the **quantification** of minor compounds including phenolic compounds, diacylglycerols, sterols, and free fatty acids (free acidity). Classification of the refined oils based on their fatty acids content and the concentration of their minor compounds was achieved by using the forward stepwise canonical discriminant analysis (CDA) and the classification binary trees (CBTs). Both methods provided good discrimination between the refined hazelnut and olive oils. Different admixtures of refined olive oils with refined hazelnut oils were prepared and analyzed by (1)H**NMR** and (31)P **NMR** spectroscopy. Subsequent application of CDA to the **NMR** data allowed the detection of the presence of refined hazelnut oils in refined olive oils at percentages higher than 5%. Application of the non-linear classification method of the binary trees offered better possibilities of measuring adulteration of the refined olive oils at a lower limit of detection than that obtained by the CDA method.

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92. **Comparison of mono- and di-saccharides release in aqueous solutions by raw or fried dice of onion (Allium Cepa L.) bulbs using quantitative nuclear magnetic resonance (qNMR)**

ByTardieu Audrey; Guerez Alice; Phana Sidarin; de Man Walter; This Herve

From Journal of food science (2009), 74(4), C319-25.Language:English, Database: MEDLINE

Although onion bulb tissues, either raw or thermally processed, are widely used as culinary ingredients in homes, in restaurants, and in the food industry, especially for sauces, little is known about the chemical constituents released from such systems. To get a straightforward and fast analysis of sugars released from onion dice soaked in model aqueous solutions, **quantitativenuclearmagneticresonance** (qNMR) spectroscopy was applied, and the effect of a preliminary thermal processing in oil was investigated. Soaking of raw or fried onion bulb dice at room temperature was followed for 11 d as a model of long-term storage. For the Armstrong cultivar, the extracted dry matter (in milligrams per gram of fresh weight) as well as the content in 3 sugars (glucose, fructose, and sucrose) increased up to a maximum after about 48 h of soaking. Frying induces no measurable new water-soluble compounds. However, extraction kinetics are

different (about 3 times faster with frying). Using additional microscopic studies, a possible extraction mechanism is proposed: compounds from sap-including sugars-would diffuse through conductive tissue channels.

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93. **Detection and quantification of phenolic compounds in olive oil by high resolution 1H nuclear magnetic resonance spectroscopy**

ByChristophoridou Stella; Dais Photis

From Analytica chimica acta (2009), 633(2), 283-92.Language:English, Database: MEDLINE

High resolution (1)H**NMR** spectroscopy has been employed as a versatile and rapid method to analyze the polar fraction of extra virgin olive oils containing various classes of phenolic compounds. The strategy for identification of phenolic compounds is based on the **NMR** chemical shifts of a large number of model compounds assigned by using two-dimensional (2D) **NMR** spectroscopy. Furthermore, 2D **NMR** was applied to phenolic extracts in an attempt to discover additional phenolic compounds. The (1)H **NMR** methodology was successful in detecting simple phenols, such as p-coumaric acid, vanillic acid, homovanillyl alcohol, vanillin, free tyrosol, and free hydroxytyrosol, the flavonols apigenin and luteolin, the lignans (+) pinoresinol, (+) 1-acetoxypinoresinol and syringaresinol, two isomers of the aldehydic form of oleuropein and ligstroside, the dialdehydic form of oleuropein and ligstroside lacking a carboxymethyl group, and finally total hydroxytyrosol and total tyrosol reflecting the total amounts of free and esterified hydroxytyrol and tyrosol, respectively. The absolute amount of each phenolic constituent was determined in the polar fraction by using anhydrous 1,3,5-triazine as an internal standard.

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94. **Determination of the content of fatty acid methyl esters (FAME) in biodiesel samples obtained by esterification using 1H-NMR spectroscopy**

ByMello Vinicius M; Oliveira Flavia C C; Fraga William G; do Nascimento Claudia J; Suarez Paulo A Z

From Magnetic resonance in chemistry : MRC (2008), 46(11), 1051-4.Language:English, Database: MEDLINE

Three different calibration curves based on (1)H-**NMR** spectroscopy (300 MHz) were used for **quantifying** the reaction yield during biodiesel synthesis by esterification of fatty acids mixtures and methanol. For this purpose, the integrated intensities of the hydrogens of the ester methoxy group (3.67 ppm) were correlated with the areas related to the various protons of the alkyl chain (olefinic hydrogens: 5.30-5.46 ppm; aliphatic: 2.67-2.78 ppm, 2.30 ppm, 1.96-2.12 ppm, 1.56-1.68 ppm, 1.22-1.42 ppm, 0.98 ppm, and 0.84-0.92 ppm). The first curve was obtained using the peaks relating the olefinic hydrogens, a second with the parafinic protons and the third curve using the integrated intensities of all the hydrogens. A total of 35 samples were examined: 25 samples to build the three different calibration curves and ten samples to serve as external validation samples. The results showed no statistical differences among the three methods, and all presented prediction errors less than 2.45% with a co-efficient of variation (CV) of 4.66%.

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95. **One-step rapid determination and purification of puerarin from Radix puerariae by n-octylamine-modified poly(methacrylate-co-ethylene dimethacrylate) monolith**

ByLv Yong-Qin; Tan Tian-Wei; Wang Man-Yi; Janson Jan-Christer

From Journal of chromatography.B, Analytical technologies in the biomedical and life sciences (2008), 871(1), 1-6.Language:English, Database: MEDLINE

n-Octylamine-modified poly(methacrylate-co-ethylene dimethacrylate) monoliths were prepared for rapid

screening, determination and one-step purification of puerarin from Radix puerariae(a crude extract of the root of *Pueraria lobata*). The modified monolith showed a specific surface area of 17.8 **m**(2) g(-1), an average pore size of 0.76 microm and a total porosity of 60.8%. Fast separation of **R**. puerariae crude extract was achieved within 5 min at a flow velocity of 722 cm h(-1) resulting in a puerarin purity of 97%, with a recovery of 85%. This demonstrates the potential of **n**-octylamine-modified poly(methacrylate-co-ethylene dimethacrylate) monolith for the rapid analysis and separation of isoflavonoids. Preparative scale sample loading (12 mg in 2 mL) resulted in a purity of 95%, and a recovery of about 69%. HPLC, FTIR, MS and (1)H**NMR** spectroscopy were used for the characterization and **quantification** of puerarin in isolated fraction.

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96. **Validation of a quantitative NMR method for suspected counterfeit products exemplified on determination of benzethonium chloride in grapefruit seed extracts**

ByBekiroglu Somer; Myrberg Olle; Ostman Kristina; Ek Marianne; Arvidsson Torbjorn; Rundlof Torgny; Hakkarainen Birgit

From Journal of pharmaceutical and biomedical analysis (2008), 47(4-5), 958-61.Language:English, Database: MEDLINE

A 1H-**nuclearmagneticresonance** (**NMR**) spectroscopy method for **quantitative** determination of benzethonium chloride (BTC) as a constituent of grapefruit seed extract was developed. The method was validated, assessing its specificity, linearity, range, and precision, as well as accuracy, limit of **quantification** and robustness. The method includes **quantification** using an internal reference standard, 1,3,5-trimethoxybenzene, and regarded as simple, rapid, and easy to implement. A commercial grapefruit seed extract was studied and the experiments were performed on spectrometers operating at two different fields, 300 and 600 MHz for proton frequencies, the former with a broad band (BB) probe and the latter equipped with both a BB probe and a CryoProbe. The concentration average for the product sample was 78.0, 77.8 and 78.4 mg/ml using the 300 BB probe, the 600MHz BB probe and CryoProbe, respectively. The standard deviation and relative standard deviation (**R**.S.D., in parenthesis) for the average concentrations was 0.2 (0.3%), 0.3 (0.4%) and 0.3mg/ml (0.4%), respectively.

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97. **Multivariate optimisation of microwave-assisted extraction of capsaicin from *Capsicum frutescens* L. and quantitative analysis by 1H-NMR**

ByNazari Fatemeh; Ebrahimi Samad Nejad; Talebi Mohammad; Rassouli Ali; Bijanzadeh Hamid Reza

From Phytochemical analysis : PCA (2007), 18(4), 333-40.Language:English, Database: MEDLINE

A simple and rapid microwave-assisted extraction (MAE) procedure combined with 1H-**NMR** spectrometry was developed and optimised for the extraction and **quantitative** determination of capsaicin in *Capsicum frutescens*. The influence of experimental variables, including irradiation power, extraction temperature and dynamic extraction time before reaching the selected extraction temperature, on the performance of the extraction procedure was systematically studied using a Box-Behnken experimental design followed by a conventional central composite design approach. Statistical treatment of the results together with results from some additional experiments suggested optimum extraction conditions as 120 degrees C and 150 W, for 15 min with acetone as extractant. The optimised MAE method provides extracts that can be analysed **quantitatively** using 1H-**NMR** without any preliminary clean-up or derivatisation steps. In the 1H-**NMR** spectrum of the crude extracts the doublet signal in the delta range 4.349-4.360 ppm was well separated from other **resonances** in deuterated chloroform. The **quantity** of the compound was calculated from the relative ratio of the integral value of the target peak to that of a known amount of dimethylformamide as internal standard. In comparison with traditional Soxhlet extraction, the proposed method is less labour-intensive and provides a drastic reduction of extraction time and solvent consumption. In addition, MAE showed higher extraction yield and selectivity, with comparable reproducibility and recovery, relative to both conventional Soxhlet and sonication methods.

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98. **High-performance liquid chromatography with nuclear magnetic resonance detection--A method for quantification of alpha- and gamma-linolenic acids in their mixtures with free fatty acids**

BySykora Jan; Bernasek Prokop; Zarevucka Marie; Kurfurst Milan; Sovova Helena; Schraml Jan

From Journal of chromatography.A (2007), 1139(1), 152-5.Language:English, Database: MEDLINE

While many naturally occurring mixtures of free fatty acids are conveniently analyzed by hyphenated technique of LC-**NMR**, a complete separation of alpha- and gamma-linolenic acids for their **quantitative** determination appears impossible at least by the methods of reversed phase HPLC. However, they can be differentiated and **quantified** from 1H **NMR** spectra measured in the course of isocratic acetonitrile-chloroform (90:10, with C8 and C18 columns in series) LC-**NMR** analysis without the need for any derivatization.

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99. **Sucrose, glucose, and fructose extraction in aqueous carrot root extracts prepared at different temperatures by means of direct NMR measurements**

ByCazor Anne; Deborde Catherine; Moing Annick; Rolin Dominique; This Herve

From Journal of agricultural and food chemistry (2006), 54(13), 4681-6.Language:English, Database: MEDLINE

Solutions obtained by heating carrot roots in water (stocks) are widely used in the food industry, but little information is available regarding the metabolites (intermediates and products of metabolism) found in the stock. The effect of treatment temperature and duration on the sugar composition of stocks was investigated directly by **quantitative** (1)H **NMR** spectroscopy, to understand the extraction mechanism when processing at 100 degrees C. Stocks prepared at three different temperatures (50, 75, and 100 degrees C) were investigated for up to 36 h. Three sugars (sucrose, glucose, and fructose) were detected and **quantified**. The concentrations of these three sugars reached a maximum after 9 h when the temperature of treatment was 50 or 75 degrees C. At 100 degrees C, the sucrose concentration reached a maximum after 3 h, whereas the concentration of glucose and fructose was still increasing at that time. Comparison of the kinetic composition of these carrot stocks with that of model sugar solutions leads to the proposal that the changes in stock composition result from sugar diffusion, sucrose hydrolysis, and hydroxymethylfurfural (HMF) formation.

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100. **Optimization of alkali-catalyzed transesterification of *Pongamia pinnata* oil for production of biodiesel**

ByMeher L C; Dharmagadda Vidya S S; Naik S N

From Bioresource technology (2006), 97(12), 1392-7.Language:English, Database: MEDLINE

Studies were carried out on transesterification of Karanja oil with methanol for the production of biodiesel. The reaction parameters such as catalyst concentration, alcohol/oil molar ratio, temperature, and rate of mixing were optimized for production of Karanja oil methyl ester (KOME). The fatty acid methyl esters content in the reaction mixture were **quantified** by HPLC and 1H **NMR** method. The yield of methyl esters from Karanja oil under the optimal condition was 97-98%.

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101. **Quantitative analysis of camptothecin derivatives in *Nothapodytes foetida* using 1H-NMR method**

ByLi Chia-Ying; Lin Chung-Hua; Wu Tian-Shung

From Chemical & pharmaceutical bulletin (2005), 53(3), 347-9.Language:English, Database: MEDLINE

A **quantitative** analysis using (1)H-**NMR** has been developed for the determination of camptothecin derivatives and trigonelline in *Nothapodytes foetida* root, stems and leaves. In the region of delta 9.5-5.5, the signals of H-7 of camptothecin (1), H-10 of 9-methoxycamptothecin (2), H-19 of pumiloside (3) and H-2 of trigonelline (4), were well separated from each other in DMSO-d(6). The **quantity** of the compounds was calculated by the ratio of the intensity of each compound to the known amount of internal standard 3,4,5-trimethoxybenzaldehyde. These results were compared with the conventional HPLC method. The advantages of the method are that no reference compounds are required for calibration curves, the **quantification** could be directly realized on a crude extract, an overall profile of the preparation could be directly obtained, and a very significant time-gain could be achieved, in comparison to conventional HPLC methods, for instance.

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102. **Evaluation of a methylation procedure to determine cyclopropenoids fatty acids from *Sterculia striata* St. Hil. Et Nauds seed oil**

ByAued-Pimentel Sabria; Lago Joao Henrique Ghilardi; Chaves Mariana Helena; Kumagai Edna Emy

From Journal of chromatography.A (2004), 1054(1-2), 235-9.Language:English, Database: MEDLINE

Cyclopropenoids fatty acids (CPFA) from *Sterculia striata* seed oil were characterized by gas chromatography/mass spectrometry (GC/MS) and **quantified** by gas chromatography-flame ionization detector (GC-FID) after derivation to fatty acid methyl esters using a cold base-catalyzed procedure. 1H **nuclearmagneticresonance** (**NMR**) analysis were done in oil and fatty acid methyl esters derivatives to **quantify** CPFA and verify artifacts formation during the base-catalyzed reaction. Similar **quantities** of CPFA were found in *S. striata* and *Sterculia foetida* seed oils before and after a base-catalyzed methylation by **NMR** analysis, with no artifact formation. These results were compatible with those obtained by GC-FID analysis. Transmethylation with KOH in methanol was an appropriated method to prepare cyclopropenoids fatty acids methyl esters and **quantify** them by GC and **NMR** analysis.

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103. **Quantitative analysis of strychnine and Brucine in Strychnos nux-vomica using 1H-NMR**

ByFrederich Michel; Choi Young Hae; Verpoorte Robert

From Planta medica (2003), 69(12), 1169-71.Language:English, Database: MEDLINE

A **quantitative** analysis using (1)H-**NMR** (Q-**NMR**) has been developed for the determination of strychnine and brucine in Strychnos nux-vomica seeds and stems. The advantages of the method are that no reference alkaloids are needed for calibration curves, the **quantification** could be directly realized on a crude extract, strychnine and brucine could easily be distinguished, an overall profile of the preparation (including non alkaloid compounds) could be directly obtained, and a very significant time-gain could be achieved, in comparison to conventional HPLC methods, for instance.

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104. **Extraction of chili, black pepper, and ginger with near-critical CO2, propane, and dimethyl ether: analysis of the extracts by quantitative nuclear magnetic resonance**

ByCatchpole Owen J; Grey John B; Perry Nigel B; Burgess Elaine J; Redmond Wayne A; Porter Noel G

From Journal of agricultural and food chemistry (2003), 51(17), 4853-60.Language:English, Database: MEDLINE

Ginger, black pepper, and chili powder were extracted using near-critical carbon dioxide, propane, and dimethyl ether on a laboratory scale to determine the overall yield and extraction efficiency for selected pungent components. The temperature dependency of extraction yield and efficiency was also determined for black pepper and chili using propane and dimethyl ether. The pungency of the extracts was determined by using an **NMR** technique developed for this work. The volatiles contents of ginger and black pepper extracts were also determined. Extraction of all spice types was carried out with acetone to compare overall yields. Subcritical dimethyl ether was as effective at extracting the pungent principles from the spices as supercritical carbon dioxide, although a substantial amount of water was also extracted. Subcritical propane was the least effective solvent. All solvents **quantitatively** extracted the gingerols from ginger. The yields of capsaicins obtained by supercritical CO(2) and dimethyl ether were similar and approximately double that extracted by propane. The yield of piperines obtained by propane extraction of black pepper was low at approximately 10% of that achieved with dimethyl ether and CO(2), but improved with increasing extraction temperature.

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105. **Determination of polydimethylsiloxanes by 1H-NMR in wine and edible oils**

ByMojsiewicz-Pienkowska K; Jamrogiewicz Z; Lukasiak J

From Food additives and contaminants (2003), 20(5), 438-44.Language:English, Database: MEDLINE

Fourier transform (1)H-**nuclearmagneticresonance** (**NMR**) spectroscopy was suitable for the **quantitative** determination of polydimethylsiloxanes (PDMS) in wine and edible oil samples. This approach offers highly specific qualitative and **quantitative** analysis due to silicone-specific location of proton signals linked to carbon atoms located directly next to silicon atoms (0-0.5 ppm), as well as a different location of signals in the range for different organosilicon structures. The method can be used for the control of PDMS at regulatory limits in foodstuffs (10 mg kg(-1)) using hexamethyldisiloxane (HDMS) as an internal standard. Samples were prepared by extraction under suitable conditions to separate the analyte, and with analyte enrichment before (1)H-**NMR** analysis. Analytical procedures were developed to permit the determination of PDMS at 0.06 mg kg(-1) in wine and at 6 mg kg(-1) in edible oils samples using readily available **NMR** instrumentation. It was, however, possible to lower the limit of detection to 6 microg kg(-1) for wine and to 60 microg kg(-1) for edible oils using a higher field instrument (500 MHz). Relative standard deviations (S(**r**)) were obtained for wine (0.028) and for oil samples (0.043), which when compared with values obtained for samples spiked with PDMS (0.021) indicated that the sample preparation was the main factor determining the precision of the method. The average recovery rates for PDMS were 97 and 95% for wine and edible oils, respectively. PDMS was detected in four brands of Italian wine, with Chianti-Rafaello containing the highest concentration (0.35 mg kg(-1)), and in four types of edible oils, highest concentration (11.9 mg kg(-1)) being found in Italian corn oil. None of the levels of PDMS found in the food samples exceeded the permissible standards laid down by the Codex Alimentarius Commission (10 mg kg(-1)), with the exception of the one corn oil sample.

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106. **Quantification of procyanidins in oral herbal medicinal products containing extracts of Crataegus species**

ByWittig Jorg; Leipolz Ingrid; Graefe Eva Ulrike; Jaki Birgit; Treutter Dieter; Veit Markus

From Arzneimittel-Forschung (2002), 52(2), 89-96.Language:English, Database: MEDLINE

According to the European Pharmacopeia a photometric assay is used for the estimation of procyanidins in Crataegi fructus. This assay is also most commonly used for procyanidin analysis in herbal medicinal products (HMPs) containing extracts of hawthorn (Crataegus species). In order to find an appropriate method for the determination of oligomeric and polymeric procyanidins by analysing various preparations containing extracts of Crataegus, the Ph. Eur.-method was compared to an HPLC-method with chemical

reaction detection (HPLC-CRD-method) and another conventional photometric assay using 4-dimethylamino-cinnamic-aldehyde (DMACA). Total procyanidins estimates obtained with the pharmacopeial method were, depending on the reference standard used, at least more than 50% higher than those obtained with the DMACA-assay. The determination of individual procyanidins could only be achieved by HPLC-CRD. Monomeric, dimeric, and trimeric procyanidins could be separated and detected individually, whereas no HPLC separation was possible for higher polymeric compounds. However, these compounds could be analysed as co-eluting groups. Using the DMACA method for the estimation of total oligomeric procyanidins and the HPLC-CRD method for **quantification** of the mono- up to trimeric procyanidins, some market leading herbal medicinal products from Germany containing extracts Crataegus species (C. monogyna Jacq., C. laevigata D.C., C. pentagyna Waldst. et Kit., C. nigra Waldst. et Kit, C. azarolus L.) were analysed. Procyanidin B2 (epicatechin-(4 beta-->8)-epicatechin) was isolated from Aesculus hippocastanum fruit shells as reference standard for calibration purposes. The structure elucidation was carried out by by means of MS and 1H-**NMR**. **Quantitative** 1H-**NMR** spectroscopy (qNMR) was applied for purity assessment.

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107. **A rapid and sensitive method for the analysis of cyanophycin**

ByErickson N A; Kolodny N H; Allen M M

From Biochimica et biophysica acta (2001), 1526(1), 5-9.Language:English, Database: MEDLINE

A method has been devised for the **quantitative** analysis of cyanophycin, based on (1)H**nuclearmagneticresonance** (**NMR**) spectroscopy, allowing determination of the nitrogen status of cyanobacteria. Cyanophycin is extracted with minimal washing from small volumes of cells and **quantified** by integration of the **NMR** peak attributed to the protons attached to the delta-carbon of arginine. Linear relationships were found between the amount of cyanophycin determined by this method and both known concentrations of cyanophycin solutions and the amount of cyanophycin determined using the standard chemical arginine assay.

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108. **In situ measurements of ribulose-1,5-bisphosphate carboxylase activity by nuclear magnetic resonance**

ByWang Z Y; Luo S; Sato K; Kobayashi M; Nozawa T

From Analytical biochemistry (1998), 257(1), 26-32.Language:English, Database: MEDLINE

High-resolution **NMR** spectroscopy is demonstrated to be capable of monitoring in situ the carboxylation reaction catalyzed by ribulose-1,5-bisphosphate carboxylase. Specific activities are determined for three enzymes from different sources containing higher **plant** and photosynthetic bacteria, and they are in agreement with those measured by other methods. Several important features of the reaction have been confirmed at the atomic level. A decrease in activity with time after the reaction started has also been observed for both enzymes with L8S8 and L2 structures from photosynthetic bacteria and higher **plants**, suggesting that the "fallover" of activity may be a more general phenomenon. 1H spectra obtained with H2O as solvent provide the most efficient **quantitative** measurement of the reaction product, 3-phosphoglycerate. 31P spectra give essentially the same result as 1H **NMR** but have the advantage of showing the degree of reaction at any time during the reaction. The incorporated carbon atom is unequivocally identified as the C-1 carbon of 3-phosphoglycerate from the 13C spectrum.

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109. **Comparison of capillary gas chromatography with 1H and 13C nuclear magnetic resonance spectroscopyfor the quantitation of pyrrolizidine alkaloids from *Senecio vernalis***

ByPieters L A; Hartmann T; Janssens J; Vlietinck A J

From Journal of chromatography (1989), 462, 387-91.Language:English, Database: MEDLINE

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110. **Vacuum liquid chromatography and quantitative 1H NMR spectroscopy of tumor-promoting diterpene esters**

ByPieters L A; Vlietinck A J

From Journal of natural products (1989), 52(1), 186-90.Language:English, Database: MEDLINE

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111. **Proton nuclear magnetic resonance studies of soybean lectin-monosaccharide interactions: computer analysis of complex binding behavior**

ByPreston C; Preston J M

From Archives of biochemistry and biophysics (1986), 247(1), 190-200.Language:English, Database: MEDLINE

1H **NMR** was used to **quantify** soybean lectin binding to monosaccharides, using presaturation of HOD plus a spin-echo sequence to observe sugar -NHCOCH3 and -OCH3 to below 0.01 mM. Binding is in the very-slow-exchange limit; there is no broadening or shifting and only unbound sugar is observed for pH 5 to 8 and 25 to 75 degrees C. Preliminary results were consistent with those previously reported for methyl 2-acetamido-2-deoxy-alpha-D-galactopyranoside (Me alpha-D-GalNAcp) (K = 3 X 10(4) liters mol-1 with four sites per tetramer). More detailed studies, however, gave concave Scatchard plots for methyl 2-acetamido-2-deoxy-alpha- and beta-D-galactopyranoside, (Me alpha- and Me beta-D-GalNAcp), best fitted using K1 values of (6-12) X 10(4) liters mol-1, K2 values less than or equal to 0.5 X 10(4) liters mol-1, and four sites of each type in D2O or 80% H2O at 25 degrees C and pH 7.2. Data for methyl alpha-D-galactopyranoside were fitted with K = 0.5 X 10(4) liters mol-1 and eight sites of the same K. Monosaccharides may be binding in the recently reported "hydrophobic sites" of soybean lectin. Both methyl 2-acetamido-2-deoxy-beta-D-galactofuranoside (Me beta-D-GalNAcf) and methyl 2-acetamido-2-deoxy-alpha-D-glucopyranoside (Me alpha-D-GlcNAcP) showed some binding by 1H **NMR**; K's were similar to those for the high-affinity sugars, but the occupancy was much lower. The soybean lectin in this study was saturated in Ca2+ (greater than or equal to 4 mol/tetramer), but low in Mn2+, with Mn2+ plus Mg2+ less than 4. We report new melting points for D-**N**-acetylgalactosamine, Me alpha-D-GlcNAcp, Me beta-D-GalNAcp, and Me beta-D-GalNAcf, and a fully listed program for fitting curved Scatchard plots using Apple IIc and IIe computers.

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