# NMR of carbohydrates, lipids and membranes

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DOI: 10.1039/9781849732796–00344

## 1 Introduction

This is our first review for RSC Specialist Periodical Reports on the field of NMR of carbohydrates, lipids and membranes, which from 1995 until now has been written by Dr. Hounsell. Our contribution covers the literature published between June 2009 and May 2010. The number of the papers devoted to the structure and interactions of these compounds is quite large although we included in our review only those papers which were accessible, peer-reviewed and printed. The reviewed material has been arranged in sections devoted to the structure of the compounds being discussed and interactions between them. It contains two distinct main parts. In the first part the literature on carbohydrates and on their non-covalent interactions to peptides/proteins and to synthetic/natural products has been outlined. In this part also the data on glycopeptides has been included. In the second part the papers devoted to proteins/peptides – lipids interactions in the membranes, lipidated proteins, lipoproteins, lipids and membranes, and glycolipids have been collected. A special section has been devoted to metabonomic studies and finally, in the last one the new NMR methods designed to study sugars, peptides/proteins and lipids have been briefly discussed.

## 2 Carbohydrates

### 2.1 Sugar structure

Numerous authors have utilized NMR for elucidation of the structure of complex oligo- and polysaccharides derived from various organisms. Since these saccharides are of crucial importance, *e.g.* for cellular biology, pharmaceutical therapy and industrial applications, a survey of this literature is presented below. The eukaryotic and prokaryotic saccharides are discussed separately.

**2.1.1 Eukaryotic polysaccharide structure.** Abronina *et al.*[1] have utilized 2D NOESY to establish the stereochemistry at C-1 of the newly developed anomeric *O*-benzoylated α- and β-D-mannopyranosyl azides. The solid state $^{13}$C NMR method have been used by Mossine *et al.*[2] to evidence that D-fructose-N-allylaniline crystallizes exclusively in the β-pyranose form. Tafazzoli and Giasi[3] have presented empirical formula linking anomeric carbon chemical shift with the glycosidic bond dihedral angles useful in conformational analysis of cyclodextrins. 1D $^{1}$H or $^{13}$C NMR has been employed to evaluate the degree of deacetylation of chitosan (marine polysaccharide) by de Alvarenga *et al.*;[4] also to estimate an average degree

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of polymerization of highly ordered cellulose II by cellodextrin phosphorylase by Hiraishi *et al.*;[5] to compare different synthetic routes for starch acetates by Volkert *et al.*;[6] to elucidate the effect of the reaction parameters on the structure of octenyl succinic anhydride–modified hyaluronic acid polysaccharide by Eenschooten *et al.*[7]

A similar method has been used by Shin *et al.*[8] to determine the abundance of α-1,6-glucisidic linkages in starch-related α-glucans (determinant of glucose release by lytic enzymes) and by Kimmel *et al.*[9] to analyze the formation of glycosidic linkages of N-unsubstituted 4-hydroxyquinolin-2-(1H)-ones (potential pharmaceutics).

1D and 2D NMR homo- and/or heteronuclear spectra have been used by Guerrini *et al.*[10,11] to detect and quantify sulfated polysaccharides contaminating heparin[10] and to detect various components of sidestream heparin,[11] and by Deng *et al.*[12] to elaborate the procedure for structural characterization of rhamnogalacturonans, plant cell wall heteropolysaccharides.

The conformational features of hyaluronic acid have been investigated by Gargiulo *et al.*[13] in isotropic and anisotropic conditions using standard 2D homo- and heteronuclear experiments.

Homo- and/or heteronuclear NMR spectra, *e.g.* COSY, ROESY, HSQC, HMQC-TOCSY, HMBC, PANSY, NOESY-HSQC TILT, have been used for determination of the linkage and/or structure of the polysaccharides isolated from various tissues of eukaryotic organisms (see Table 1).

Guilherme *et al.*[85] have used 1D NMR to estimate the structure of a vinyl-functionalized pectin and solid-state $^{13}C$ CP-MAS NMR to elucidate the formation of hydrogels. The crystallinity of cellulose in plant organs has been analyzed by Takahashi *et al.*[86] using $^{13}C$ CP-MAS NMR. Manni *et al.*[87] have used $^{13}C$ NMR with CP-MAS technique to estimate the structure of chitin prepared by the enzymatic deproteinization of shrimp wastes.

Rudd *et al.*[88] have presented a review on application of high resolution NMR to the analysis of glycosaminoglycans (*e.g.* heparin, heparan sulfate).

**2.1.2 Prokaryotic polysaccharide structure.** 1D and 2D NMR homo- and/or heteronuclear spectra have been used for determination of the linkage and/or structure of the polysaccharides isolated from prokaryotic cells (see Table 2).

## 2.2 Peptide/protein-sugar binding (non-covalent interactions)

**2.2.1 Effect on peptide/protein.** Several NMR techniques including chemical shift mapping, saturation transfer difference (STD) NMR experiment, competition STD, laser photo CINDP and NMR titration have been employed for identification of the binding sites, for analysis of mode and orientation of binding, and for determination of the dissociation (or binding) constants of sugars to proteins in protein-sugar complexes. One or more of these techniques have been used to study binding of several mono- and disaccharides to the C-terminal domain of an R-type lecitn from the earthworm *Lumbricus terrestris* by Hemmi *et al.*;[141] of Neu5Acα2Me to the carbohydrate-binding cleft mutants of the rotavirus spike protein by Kraschnefski *et al.*;[142] of the Lewis$^x$ trisaccharide to the macrophage galactose-type C-type lectin 1 by Sakakura *et al.*;[143] of dimeric CD69NG70 with ManNAc and GlcNAc by Kavan *et al.*[144]

**Table 1** Eucaryotic saccharides which structures have been evaluated with the aid of NMR spectroscopy

| polysaccharide type | organism | reference |
|---|---|---|
| tumor associated antigen $Le^aLe^x$ central fragment | *Homo sapiens* | 14 |
| phenylethanoid glucosides containing jacaranone | *Jacaranda glabra* | 15 |
| wall mannan (effect of oxidative and osmotic stresses) | *Candida albicans* serotype A | 16 |
| tyramine-based hyaluronan hydrogels | | 17 |
| heparin-mimicking polymer | | 18 |
| $Glc_3Man$ | | 19 |
| xylomannan (thermal hysteresis-producing, containing a fatty acid component) | *Upis ceramboides* | 20 |
| (1,6)-$\beta$-glucan (cell wall) | *Saccharomyces cerevisiae* | 21 |
| dendric sulfated cellobiose cluster with polylysine | | 22 |
| acidic polysaccharide LBP-1 | *Lycium barbarum* fruit | 23 |
| $(1\rightarrow3)$-$\beta$-D-glucan carboxymethylated-sulfated derivative, antitumor agent | glucan of *Poria cocos* | 24 |
| oligasaccharides obtained through enzymatic synthesis | mannosultransferases from *Candida* sp. | 25 |
| polysaccharide | *Chroogomphis rutilus* fruiting bodies | 26 |
| heteroglycan | *Collema flaccidum* | 27 |
| polysaccharide | *Auricularia polytricha* fruiting bodies | 28 |
| rhamnoglucuronan obtained through oxidation of gellan exopolysaccharide | | 29 |
| fucosylated chitooligosaccharides | synthesized by human $\alpha$-1,6-fucosyltransferase | 30 |
| polysaccharide (chemically sulphated, dietary supplement) | *Ganoderma lucidum* | 31 |
| lignins | wood | 32 |
| xylooligosaccharides | hydrothermal processing of rice husk | 33 |
| lacquer polysaccharide chemically sulfated | sap of *Rus vernicifera* | 34 |
| acidic galactan | *Pomarea lineata* eggs | 35 |
| cyclic $\beta$-1,3-heptaglucan | *Phanerochaete chrysosporium* recombinant mutated glycosynthase | 36 |
| fructopyranose oligosaccharides | fermented beverage of plant extract | 37 |
| galactomannan, chemically sulfated | guar gum | 38 |
| $(1\rightarrow3)$-linked 2-*O*-$\beta$-D-xylopyranosyl-$\alpha$-L-arabinofuranosyl side chains | cereal arabinoxylans | 39 |
| glucan | *Rubus crataegifolius* roots | 40 |
| polysaccharide | *Amorphophalluscampanulatus* corm | 41 |
| glucan | *Pleurotus florida* | 42 |
| polysaccharide | *Launaea acanthodes* | 43 |
| starch esters | enzymatic esterification using *Staphylococcus aureus* lipase | 44 |
| heteropolysaccharide | *Solenum melongena* unripe fruits | 45 |
| heteropolysaccharide | *Amaranthus* tricolor stems | 46 |
| galactosyl derivatives | transglycosylation with $\beta$-galactosidase of *Aspergillus orizae* | 47 |
| exopolysaccharide | *Tolypocladium* sp (fungus, mycelial fermentation) | 48 |
| chondroitin sulfate | *Scyliorhinus canicula* (dogfish) | 49 |
| polysaccharides | *Grifola frondosa* (mushroom) | 50 |

**Table 1 (*Continued*)**

| polysaccharide type | organism | reference |
|---|---|---|
| polysaccharides | *Lycopersicon esculentum* (unripe tomato) | 51 |
| sulfated polysaccharide | defatted rice bran | 52 |
| cell wall polysaccharides | *Abelmoschus esculentus* (pod of okra) | 53 |
| lignin and polysaccharide | plant cell wall components | 54 |
| chemically phosphorylated (1→3)-$\beta$-D-glucan | *Poria cocos* | 55 |
| polysaccharide | *Astragalus membranaceus* roots | 56 |
| polygalacturonic acid | *Maytenus ilicifolia* leaves | 57 |
| heteropolysaccharide | *Psidium guajava* fruits | 58 |
| (1→3)-$\beta$-D-glucan | *Dictyophora indusiada* fruiting body | 59 |
| chitosan polysulfate | chemically modified | 60 |
| (1→6)-$\beta$-D-glucan | *Bulgaria inquinans* fruiting body | 61 |
| phenyl-adducted cyclodextrin | | 62 |
| glucuronoxylan | poplar wood | 63 |
| (1→3)-$\beta$-D-glucan (paramylon and curdian) derivatives | oxidized by 2,2,6,6-tetra-methylpiperidine-1-oxyl radical (TEMPO) | 64 |
| $\beta$-D-glucan | oxidized by 2,2,6,6-tetramethylpiperidine-1-oxyl radical (TEMPO) | 65 |
| deacetylated chitosan | chemically derived | 66 |
| light-curable chitosan | chemically modified | 67 |
| $\alpha$-1,5-L-arabinobiose | released by hydrolase of *Fusarium graminearum* from plant cell wall polysaccharide | 68 |
| heteropolysaccharide | *Tremella aurantialba* mushroom | 69 |
| dextran | produced by *Leuconostoc mesenteroides* dextranosucrase | 70 |
| deprotonated $\beta$-cyclodextrin | formed in alkaline solutions | 71 |
| fucogalactan | *Ganoderma lucidum* fruiting body | 72 |
| (1→6)-$\beta$-D-glucan | *Agaricus bitorquis* (mushroom) | 73 |
| 3-*O*-methylated mannogalactan | *Pleurotus pulmonarius* (mushroom) | 74 |
| lignin-type polymers | with different $\gamma$-carbon functionality | 75 |
| arabinoxylans | maize, rice and wheat brans | 76 |
| $\alpha$,$\alpha$-trehalose-based polyacetals and macrocyclic acetals | synthetic environmental-friendly polymers | 77 |
| hydrolytic and glycosylated products | *Aspergillus niger* $\alpha$-glycosidase | 78 |
| oligosaccharides from stachylose | hydrolysis by Pectinex Ultra | 79 |
| arabinoxylan | wheat grain | 80 |
| glucans | *Caripia montagnei* (mushroom) | 81 |
| (1→6)-$\beta$-D-glucan | *Malassezia sympodialis* (fungus) | 82 |
| mucin-type *O*-glycans | *Echinocccus granulosus* (cestode) | 83 |
| sodium alginate derivatives obtained after photolytic depolymerization | | 84 |

In addition to the usage of some of the above mentioned techniques the 3D solution structure of the protein-sugar complexes have been also calculated with NMR restraints by Shahzad-ul-Hussan *et al.*[145] for chititriose nad chitotetraose bound to the cyanobacterial lectin MVL; by Koharudin *et al.*[146] for sucrose bound to LKAMG, the designed chimera of TbCVNH; by Siebert *et al.*[147] for sialic acid containing oligosaccharides to lectin SHL-1; by Nesmelova *et al.*[148] for gal-1 in the lactose bound and unbound states.

Competition saturation transfer difference method combined with isotope editing and filtering schemes has been offered by Féher *et al.*[149]

**Table 2** Prokaryotic cells saccharides which structures have been established using NMR spectroscopy

| polysaccharide type | bacterial strain | reference |
|---|---|---|
| exopolysaccharides | nitrogen fixing bacteria *Burkholderia kukuriensis* | 89 |
| *O*-polysaccharide | haloalaliphilic bacteria *Halomans alkaliantarctica* | 90 |
| *O*-antigen containing ethanolamine phosphate | *Shigella flexneri* | 91 |
| complete core region from LPS | *B. cepacia* | 92 |
| α-glucosides produced by maltose phosphorylase | *Lactobacillus acidophilus* | 93 |
| O-tetrasaccharide from flagellin | *Pseudomonas syringae* | 94 |
| N-tetrasaccharide from flagellin | *Methanococcus maripaludis* | 95 |
| O-polysaccharide | *Salmonella O55 Escherichia coli O103* | 96 |
| 2-branched (1,3)-$\beta$-D-glucans | lactic bacteria | 97 |
| exopolysaccharide | *Streptococcus thermophilus* | 98 |
| *O*-polysaccharides | *E. coli O123* and *Salmonella enterica O58* | 99 |
| *O*-polysaccharides | *Aeromonas bestiarum* strain 207 | 100 |
| oligosaccharide | *Arcobacter halophilus* | 101 |
| *O*-specific polysaccharides | *Azospirilium brasilience* sp. 107 and S27 and *A.lipoferum* RG20a | 102 |
| exopolysaccharide | *Rhizobium sp.* | 103 |
| *O*-polysaccharides | *E. coli* | 104 |
| (1→3)-$\beta$-D-glucan (curdlan) rate of sulfation | *Alcaligenes faecalis* | 105 |
| exopolysaccharide (effect of carbohydrate source) | *L. fermentum* | 106 |
| *O*-specific polysaccharide | *Hafnia alvei* | 107 |
| heparosan K5 polysaccharide (quantification in growth medium) | *E. coli* | 108 |
| exopolysaccharide | *L. johnsonii* | 109 |
| core oligosaccharide segment of lipopolysaccharide | *Yokenella regensburgei* | 110 |
| *O*-polysaccharide | *Mesorhizobium loti* and *M. amorphae* | 111 |
| *O*-polysaccharide | *E. coli* | 112 |
| polysaccharide containing 3-*O*-acetylglycerol | *Streptococcus pneumoniae* | 113 |
| cell wall polysaccharides | *Bifidobacteriumbifidum* | 114 |
| *O*-polysaccharide | *Yersinia pseudotuberculosis* | 115 |
| lipopolysaccharide | *Neisseria meningitidis* | 116 |
| cell wall glycopolymers | *Kribbella* spp. | 117 |
| polysaccharide | *Vibrio vulnificus* | 118 |
| *O*-polysaccharides | *Providencia alcalifaclens* and *Proteus vulgaris* | 119 |
| exopolysaccharide (evan) | *Halomonas* sp. | 120 |
| *O*-polysaccharide containing abequose | *Citrobacter freudii* | 121 |
| *O*-polysaccharide | *E. coli* | 122 |
| exopolysaccharide (pullulan) | *Aureobasidium pullulans* | 123 |
| O-polysaccharide | *Vibrio anguillarum* | 124 |
| polysaccharides | *Rahnella aquatilis* | 125 |
| polysaccharide | *V. vulnificus* | 126 |
| polysaccharide | *V. vulnificus* | 127 |
| *O*-polysaccharide | *Salmonella Mara* | 128 |
| *O*-polysaccharide | *Plesiomonas shigelloides* | 129 |
| *O*-polysaccharide | *Azospirillum lipoferum* | 99, 130 |
| *O*-polysaccharide | *Cronobacter muytjensii* | 131 |

**Table 2** (*Continued*)

| polysaccharide type | bacterial strain | reference |
|---|---|---|
| *O*-polysaccharide | *Acinetobacter baumannii* | 132 |
| co-aggregation receptor polysaccharide | *Streptococcus oralis* | 133 |
| oligosaccharide | *Haemophilus parainfluenzae* | 134 |
| high molecular weight exopolygosaccharide | *L. pentosus* | 135 |
| *O*-polyaccharide | *Taylorella equigenitalis* | 136 |
| *O*-polyaccharide | *Shigella shigelloides* | 137 |
| lipopolyaccharide | *Loktanella rosea* | 138 |
| lipopolyaccharide and lipid A | *Bacteriovorax stolpii* | 139 |
| exopolysaccharides | *Paenibacillus polymyxa* | 140 |

It allows the separation of the STD signals of the labelled reference sugar ligand from that of the natural abundance hit compound in the case of strong signal overlap. Diehl *et al.*[150] have shown with $^{15}N$ spin relaxation experiments that gelectin-3 backbone exhibits an increase in conformational entropy upon binding lactose. The cross-polarization with polarization inversion (CPPI) solid-state experiments have been applied by Patching *et al.*[151] to study binding of D-glucose to the *E. coli* sugar transporter Ga1P and its mutants in membrane preparations. Interaction of lectin $B_4$ of *Vicia villosa* with α-D-GalNAc-$\beta^3$-peptide (a foldamer with antibacterial and antiproliterative properties) has been analyzed by saturation transfer difference (STD) NMR spectroscopy by Kaszowska *et al.*[152]

Miller *et al.*[153,154] have used $^{1}H$-$^{15}N$ HSQC and pulse field gradient (PFG) to show binding of galectin-1 (lectin) to α-galactomannan Davanat[153] and to prove that carbohydrate-binding domain on human galectin-1 is more extensive for complex glycan than for simple saccharides.[154] Sulfated heparin tetrasaccharide interactions with a complement factor H module 7 (a model of glucosaminoglican–protein complex formation) have been analyzed by Blaum *et al.*[155] using $^{15}N$,$^{13}C$ HISQC and $H_2CN$ experiments. Ribeiro *et al.*[156] have elucidated the principles of lectin-based drug design by developing combined strategy to identify lead compounds using STD NMR.

**2.2.2 Effect on sugar.** NMR based conformational studies of α-D-mannopyranosyl-(1→6)-α,β-D-mannose complexed with *Allium sativam* agglutinin I and concanavalin A have been reported by Mazumder and Mukhopadhyay.[157] High resolution real-time NMR has been applied by Guyett *et al.*[158] in monitoring of intermediates in the conversion of UDP-α-D-glucoronic acid to UDP-α-D-xylose and UDP-α-D-apiose by a UDP-apiose/UDP-xylose synthase.

## 2.3 Sugar-sugar binding (non-covalent interactions)

Weak $Ca^{2+}$-mediated interactions between two synthetic trisaccharides, 1-AII and 1-S@Au, have been proven by Santos *et al.*[159] using diffusion-ordered (DOSY) and (TR-NOESY) NMR experiments.

## 2.4 Sugar to RNA binding (non-covalent)

Zakhour *et al.*[160] have characterised the binding of Galα3GalαOMe to rN82 VLPs employing STD NMR experiments.

## 2.5 Sugar binding (non-covalent) to synthetic and natural compounds

$^1$H NMR titration has been used in quantitative binding studies of di- and mono-saccharides to dimesithylmethane-derived receptors by Mazik and Buthe.[161]

$^1$H NMR spectra have been employed to estimate interaction of dextran with poly(methyl vinyl ether-co-maleic anhydride) nanoparticles by Porfire *et al.*;[162] to analyze the formation of non-inclusion complex between meglumine antimoniate (antileishmanial drug) and β-cyclodextrin by Ribeiro *et al.*;[163] and the formation of colchicine:β-cyclodextrin complex, potential colchicine delivery system for treatment cutaneous diseases, by Singh *et al.*[164] The same approach has been used by Provencher *et al.*[165] to test carboxymethylated cyclodextrines (α-, *β*- and *γ*-) in the presence of lanthanide ions as chiral NMR solvating agents for aromatic substrates with phenyl, naphtyl, pyridyl, indoline and indole rings, and by Xin *et al.*[166] to test series of branched cationic β-cyclodextrin derived polymers as anionic drug carriers. The structures of copolymers have been characterized using $^1$H NMR by Zhang *et al.*[167] for β-cyclodextrin/poly(L-leucine) copolymers; by Gou *et al.*[168] for miktoarm star copolymer composed of 14 poly(ε-caprolactone) arms and 7 poly(ethylene glycol) arms with β-cyclodextrin as core moiety; by Maffeo *et al.*[169] to investigate the ability of EDTA-type cyclodextrins (bearing 6, 7 and 8 bis(carboxymethyl)amino(iminodiacetic acid groups) to coordinate with lanthanide ions (potentially useful as contrast agents in MRI). 1D NMR has also been employed by He *et al.*[170] for structural characterization of the hyaluronic acid-poly(butyl cyanoacrylate) nanoparticles and by Mercê *et al.*[171] for cholecalciferol:*β*-cyclodextrin complex.

1D together with 2D NMR experiments have been used to characterize the (*S*)-7,8-dihydrokavain and β-cyclodextrin inclusion complex by Pesticelli *et al.*;[172] to confirm the structures of monoacyl cyclodextrines (α-, β- and γ-) derivatives by Martina *et al.*[173] Various drug:cyclodextrin complexes have been characterized by the aid of 1D and 2D experiments: the conformation and stability of the *N*-1-decyl-ferrocenylmethylamine:carboxymethyl-β-cyclodextrin complex (redox-responsive vesicles) by Zhang *et al.*;[174] the structure of the *N*,*N*'-bis(ferrocenylmethylene)diaminohexane:β-cyclodextrin complex by Zhang *et al.*;[175] the structure of aspartame:cyclodextrins complex by Sohajda *et al.*;[176] the structure of the 5,10,15,20-tetrakis(*N*-methylpyridinium-4-yl)porphyrin:cyclodextrin complex by Mosinger *et al.*;[177] the structure of the antidepressant trazodone hydrochloride:hydroxypropyl-β-cyclodextrin complex by Misiuk *et al.*[178] A similar approach has been used to elucidate the structure of various cyclodextrins:risperidone (antipsychotics) complexes by Danel *et al.*[179] and the structure of biodegradable star polymer functionalized with β-cyclodextrin inclusion complex by Setijadi *et al.*[180]

2D ROESY spectra have been used to elucidate the structure of cyclodextrin:DNA (pUC18 plasmid) complex as a model delivery system for gene-therapy by Aachmann *et al.*;[181] the structure of cyclodextrin:formoterol (pulmonary drug) complex by Thi *et al.*;[182] the structure of a complex built of tripod molecule containing an aromatic core bearing three peracetylated cyclodextrins and pesticide by Mallard-Favier *et al.*[183] and the structure of heptakis-[6-deokxy-6-(2-aminoethylsulfanyl)]:β-cyclodextrin complex by Gómez-Biagi *et al.*[184] The structures of

α-tocopherol:β-cyclodextrin and quercetin:γ- cyclodextrin complexes have been investigated by Koontz *et al.*[185] using $^{13}$C CP-MAS NMR.

New types of ligands have been synthesised and their binding properties to the carbohydrate recognition domain have been studied with competitive binding NMR method: Stokmaier *et al.*[186] have studied binding of a series of triazole monovalent ligands to the asialoglycoprotein receptor (ASGP-R); Murthy *et al.*[187] have studied binding of aromatic mannose disulfide derivatives to concanavalin A.

### 2.6 Identification and quantification of sugars

1D and 2D NMR spectra have been used for characterization or identification of sugars. With the aid of NMR Toida *et al.*[188] have characterized oligosaccharides in depolymerised chondrotoin and dermatan sulphates; Huang *et al.*[189] have characterised the structure of β-(1,4)-linked mannopyranoses, the predominant components of the Lan3-2 oligosaccharide liberated from glycoproteins of *Hirudo medicinalis*; Volpi and Maccari[190] have characterized dermatan sulphate purified from marine clam *Scapharca inaequivalvis*.

$^{1}$H NMR has been used by Bose *et al.*[191] for quantification of sugar monomers in a modified hydrolysis procedure of hardwood carbohydrates. $^{13}$C NMR has been used for quantification of several biological processes: by Liddell *et al.*[192] to quantify the lactate production in glucose metabolism under hydrogen peroxide stress in cultured astrocytes.

*In vivo* $^{13}$C NMR has been used by Castro *et al.*[193] to monitor the consumption of α- and β-anomers of the specifically labelled glucose in different glucose uptake systems as well as to quantify intracellular metabolites in extracts. $^{13}$C CPMAS NMR has been applied by Metzger *et al.*[194] to quantify the concentration of polysaccharides and proteins in EPS of *Pseudomnas putida* nad *Aureobasidum pullulans*.

Sitkowski *et al.*[195] have applied DOSY experiment for screening heparin samples for oversulfated chondroitin sulphate contamination.

## 3 Glycosylated proteins

A subset of glycosylated proteins existing in cells plays specific glycosylation-dependent roles. On the other hand structural elucidation of glycosylated proteins is a challenging task because of their complexity and diversity. Various NMR techniques have been employed in this field of research.

### 3.1 Glycoprotein structure

For the first time a pure arabinogalactan-protein has been isolated by Göllner *et al.*[196] and the primary structure of its sugar part determined with the aid of $^{13}$C NMR. NMR methods have been used Yoshida-Moriguchi *et al.*[197] for identification of a phosphorylated O-mannosyl glycan on the mucin-like domain of recombinant α-DG. Interaction surfaces between domains of fibronectin have been identified by Vakonakis *et al.*[198] from chemical shift perturbations measured in $^{1}$H-$^{15}$N HSQC spectra. The structure of O-glycan of Q-mucin from jellyfish has been established by Urai

*et al.*[199] using combination of $^{1}H$, $^{13}C$ and $^{31}P$ NMR. The structure of 3 disialyl-Le$^{x}$ hexaccharide antigen of human colorectal cancer has been proposed by Robbe-Masselot *et al.*[200] with the aid of COSY spectra. The effect of glycosylation on the *cis*/*trans* isomerisation of prolines of the hinge peptide of human serum immonoglobulin A1 has been analysed by Narimatsu *et al.*[201] with $^{1}H$-$^{13}C$ HSQC NMR.

Segmental $^{13}C/^{15}N$ labelling of protein but not carbohydrate moiety has enabled Slynko *et al.*[202] determination of the structure of *N*-linked glycoprotein of *Campylobacter jejuni* using 2D $^{13}C$-filtered-filtered NOESY. Clément *et al.*[203] have characterized the structure of fucoidan from brown algae using 1D and 2D NMR while its interaction with the protein C4 (of human complement system) has been analyzed using STD-NMR and TRNOESY experiments. Human galectin-1 and galactose-containing ligands structures have been analysed using $^{1}H$-$^{15}N$ HSQC NMR experiments by Meynier *et al.*[204]

### 3.2 Glycopeptides

2D homo- and hetero-nuclear NMR spectra have been used by Matsushita *et al.*[205] to solve the 3D structure of MUC1-related *N,O*-glycopeptide and its two *O*-glycopetide fragments. Temperature coefficients of amide protons have been measured by Tam *et al.*[206] in their studies of solution conformation of C-linked antifreeze truncated glycoprotein AFGP-8 analogues. A series od monoglycolsylated antifreeze glycopeptides analogues have been synthesised by Heggemann *et al.*[207] and their structures confirmed with NMR. Lu *et al.*[208] have determined the structure of a new antifungal glycopeptide of *Burkholderia contaminans* with 2D NMR and Wu *et al.*[209] using $^{13}C$ and HMBC spectra have elucidated the structure of *Ganoderma lucidum* fruit glycopeptide with sugar dependent antioxidant activity.

## 4 Proteins/peptides – lipids interactions in the membranes

For many years biological membranes were considered to be a passive structural scaffold for interacting and even embeded proteins. Development of new NMR techniques made studies on the lipid-protein interaction feasible revealing the existence of dynamic structural equilibrium between both counterparts. Recent studies using simplified model lipid-protein interacting systems and also natural partners interactions are summarized below.

### 4.1 Effect on lipid

Russel-Schulz *et al.*[210] have used $^{2}H$ solid state spectroscopy to study perturbation of DPPC/POPG bilayers by SP-$B_{8-25}$, a synthetic peptide comprising the N-terminal helix of the essential lung surfactant protein. POPC/POPG membrane perturbation caused by the human cathelicidin antimicrobial peptide LL37 has been studied by Thennarasu *et al.*[211] with the aid of $^{31}P$ SSNMR. Changes in the solid state $^{13}C$ and $^{31}P$ chemical shifts and decrease of the corresponding $T_1$ relaxation time have been observed by Ausili *et al.*[212] for phosphatidylglycerol unreached membranes upon the interaction with the C-terminal domain of Bax protein. The depth of

membrane penetration of the NPY analogue, paramagnetic [Ala$^{31}$, TOAC$^{32}$]-NPY, have been studied by Thomas *et al.*[213] with $^{1}$H MAS NMR. In the case of zwitterionic phospholipids and charged lipids NPY was located in the upper chain region and in the head group region of membrane, respectively.

The effect of mycosubtilin on phase transition of DPMC has been checked by Nasir *et al.*[214] with HR-MAS NMR. The DMPC and DMPG supramolecular organization and phase transition upon interactions with penetratin and antimicrobial RL16 peptide have been monitored by Alves *et al.*[215] with $^{31}$P SSNMR. Membrane disordering effect has been studied for the series of histidine-rich amphipathic cationic peptides with $^{2}$H SSNMR by Mason *et al.*[216] It has been shown by Vostrikoy *et al.*[217] using $^{2}$H SSNMR that the introduction of a single arginine residue into a hydrophobic TM helix results either in a small reorientation of the helix or in a dynamic switching between TM and interfacial orientations of the helix. Yang *et al.*[218] have used the same techniques to study the effect of a C-terminal peptide of surfactant protein B on mechanically oriented POPC/POPG bilayers. The influence of the pore-forming cytolysin, equinatoxin II on the lipid order and bilayer morphology of multilamellar vesicles has been investigated by Dreschler *et al.*[219] with $^{31}$P and $^{2}$H solid-state NMR. Interactions of the gp144 lytic transglycosylase with DPMC and DPMG membranes have been studied using $^{2}$H and $^{31}$P SSNMR by Cloutier *et al.*[220] Using these techniques Antharam *et al.*[221] have shown that the C-terminus of lung surfactant protein B alters lipid organization in DPPC/POPG and POPC/POPG lipid systems. The structure and motions of several membrane-mimetic systems have been studied in the presence of the HIV fusion peptide (HFP) constructs by Gabrys *et al.*[222] with the aid of $^{2}$H and $^{31}$P SSNMR; the HFP-induced membrane curvature was observed. The interaction of pardaxin with zwitterionic and anionic vesicles have been studied by Vad *et al.*[223] using $^{13}$C PISEMA. Location of two different potassium channels, the KvAp and HsapBK in DMPC/DHPC micelles and POPC vesicles has been studied by Biverståhl *et al.*[224] using $^{13}$C and $^{2}$H SSNMR.

Attenuation of signals in $^{1}$H NMR spectra of POPC/POPG membranes embedded with voltage-sensing domains has been used by Krepkiy *et al.*[225] to study membranes structure and hydration. The equilibrium between oleic acid free and bound to equine lysozyme in ELOA complex has been studied by Nielsen *et al.*[226] using $^{1}$H NMR.

1D and 2D NMR have been used by Marty *et al.*[227] to search for a membrane-binding motif of the chloroplast signal recognition particle receptor and to analyze its interaction with lipids and by Gouttenoire *et al.*[228] to characterize the amphipathic segment ($\alpha$-helix) responsible for membrane association of hepatis C virus (HCV) protein 4B using deuterated micellar dodecylphospholine or deuterated trifluoroethanol as membrane mimetic medium. Tong *et al.*[229] have resolved the conformation of the GD2 ganglioside (targeted for cancer diagnosis, prognosis and therapy by antibody mAb 3F8), free and bound to mAb, by STD NMR and various 2D experiments. Lesovoy *et al.*[230] have analyzed binding of recombinant neurotoxin II from *Naja oxiana* with liposomes (DOPC/DOPS/CHO) using $^{31}$P NMR and 2D $^{1}$H-$^{15}$N HSQC spectra.

Solid-state MAS $^{31}$P NMR has been used by Sani *et al.*[231] to track *ex vivo* the behaviour of mitochondrial membrane lipids (PE, PC, Cl) during physiological processes; by Cheng *et al.*[232] to analyze the effect of antimirobial peptides (aurein) on perturbation of lipid headgroups of the bilayers (PMPC/DMPG and POPC/POPG); by Nakazawa *et al.*[233] to study the mode of interactions of amyloid β peptide (Aβ) with DMPC membranes. Similarly, Madine *et al.*[234] have elucidated the effect of α-synuclein on PC/PG vesicle integrity using $^{31}$P NMR. The effect of membrane-fusogenic 'Leu-Val' peptides (mimicking natural transmembrane peptide sequences) on the changes of phospolipid (POPC/DOPS/DOPE) phase order has been elucidated by Agrawal *et al.*[235] using solid-state 1D $^{31}$P NMR and 2D NOESY experiment.

Solid-state $^{2}$H NMR has been used by Jean-François *et al.*[236] to elucidate the effect of cateslytin, antimicrobial peptide, on model membrane (DMPC/DMPG/ERG) structure; by Pabst *et al.*[237] to observe the perturbation of PG bilayers (membrane thickness) caused by the antimicrobial peptide peptidyl-glycylleucinecarboxyamide. Solid-state $^{31}$P and $^{2}$H NMR have been used by Kim *et al.*[238] to observe the antimicrobial peptides (magainin-2 and aurein-3) induced formation of pores of thinned lipid bilayer. Interaction and dynamics of a 21-mer cytotoxic peptide (that acts as ion channel) with phospholipid (DMPC or DPPC)/DHPC bicelles have been elucidated by Ouellet *et al.*[239] with solid-state $^{2}$H, $^{13}$C, $^{15}$N and $^{31}$P NMR. De la Serna *et al.*[240] have investigated the lipid dynamics in the pulmonary surfactant membranes reconstituted from the lung surfactant hydrophobic components (lipids and proteins) using pulse-field gradient $^{31}$P NMR. Lind *et al.*[241] have analyzed the effect of two model peptides with different hydrophobic length on the phospholipid dynamics in different bicelles using natural abundance $^{13}$C relaxation measurements; diffusion constants were measured using a modified Stejskal-Tanner spin-echo experiment.

### 4.2 Effect on peptide/protein

The intact transmembrane domain from human amyloid protein precursor (APP), hAPP-TM has been produced and purified in the amount sufficient to measure its NMR spectra in DPC micelles by Park *et al.*[242] The 3D structure and orientation of rat IAPP in DPC micelles have been studied by Nanga *et al.*[243] by solution NMR. Smith *et al.*[244] with the solid-state NMR have confirmed that hIAPP binds to the regions of negative curvature in bicelles. Grimaldi *et al.*[245] have probed conformational dependence of the amyloid peptide, Aβ(16–35) in the response to negative charge modifications of the micelle surface. Sato *et al.*[246] have shown that the amyloid precursor protein transmembrane (APP TM) helix is disrupted at the intracellular membrane boundary near ε-cleavage site. Structures of rat and human $IAPP_{1–19}$ in DPC micelles have been determined by Nanga *et al.*[247] Park *et al.*[248] have charcterised bovine antimicrobial peptide lactophoricin in DPC micelles using $^{1}$H-$^{15}$N HSQC spectra.

The temperature dependence of the partition coefficient of an eleven amino acids peptide, substance P in isotropic bicells has been studied by Kim *et al.*[249] using pulsed filed gradient (PFG) NMR diffusion technique.

Vermeer *et al.*[250] using $^{13}C$ solid state NMR have shown that the TM7 segment of subunit *a* from $H^+$-V-ATPase from *Saccharomyces cerevisiae* is immobile in phospholipid bilayer.

Changes in the $^1H$ NMR spectra of the N-terminal region of CGI-58 upon DPC titration measured by Gruber *et al.*[251] revealed strong protein-micelle interaction. Chemical shift perturbations of $C_\alpha$, $C_\beta$ and $H_N$ of acyl carrier protein from *Plasmodium falciparum* substantiate with NOE effects have been used by Upadhyay *et al.*[252] to analyze the interactions of ACP molecule with the acyl intermediates in the fatty acid synthesis pathway. Evans *et al.*[253] using chemical shift perturbations and 2D homo- and hetero-nuclear spectra have calculated a series of 3D structures of the derivatised act ACPs from *S. coelicolor* A3(2) bound with different mimics of early polyketide intermediates.

3D NMR solution structures of *E. coli apo*-ACP, acyl-ACP and butyryl-ACP have been solved by Wu *et al.*[254] and by that means the conformational dependence of the protein on the ligand size has been revealed. The solution structure of an ACP domain from a fungal type I polyketide synthase has been solved by Wattana-amorn *et al.*[255]

Chemical shift perturbations have been used to investigate the interactions of ATP with cytochrome c and its E26N mutant by Patriarca *et al.*[256] (using $^1H$, $^{15}N$ of amide groups); of a small antimicrobial 'PFR' peptide with SDS and DPC micelles by Zorko *et al.*[257] (using $H_\alpha$).

The orientation of the heterodimeric antimicrobial peptide in the membrane has been determined by Resende *et al.*[258] using $^2H$ solid state NMR. Orientational distributions of monomeric membrane-bound peptides have been predicted theoretically by Esteban-Martin *et al.*[259] and validated by comparison of the predicted $^2H$ solid state NMR quadrupolar couplings with experimental ones. Strandberg *et al.*[260] have studied synergistic transmembrane insertion of the heterodimericPGLa/magainin 2 complex using $^2H$ solide-state NMR and peptides labelled with Ala-$d_3$ in different positions. Rui *et al.*[261] have simulated orientation of protegrin-1 monomer and dimer in DLPC and POPC bilayers. The tilt angle of hCB1(T377-E416) within the phospholipid bilayer has been determined using six single $^{15}N$-labelled peptides and $^{15}N$ solid phase NMR by Tiburu *et al.*[262] The results of Ellena *et al.*[263] studies of the structure and dynamics of Syb(1-116) in lipid micelles have indicated the presence of helical propensities in the TM domain and at the beginning and end of the SNARE motif.

Using different liquid or solid state NMR experiments the 2D and/or 3D structure, dynamics and/or interactions of peptides/proteins with membrane have been revealed: of kisspeptin decapeptide analogs bound to POPC by Lee *et al.*;[264] of bradykinin bound to DOPC/DOPE by Bonechi *et al.*;[265] of Ste11SAM bound to DPC by Bhunia *et al.*;[266] of RP-1 antimicrobial peptide bound to DPC by Bourbigot *et al.*;[267] of Gp41 peptide P1 bound to DPC by Coutant *et al.*;[268] of hedistin in DPC micelles by Xu *et al.*;[269] of the N-terminal region of equinatoxin II bound to several membranes by Drechsler *et al.*;[270] of the isolated voltage-sensing domain of the potassium channel KvAP bound to DPC/LDAO by Shenkarev *et al.*;[271] of the potassium channel KcsA-Kv1.3 embedded in asolectin liposomes by Ader *et al.*;[272] of the penicillin-binding protein 5 anchor

peptide embedded into DPC micelle by O'Daniel *et al.*;[273] of Ser-16 phosphorylated phospholamban in mechanically oriented DOPC/DOPE by Chu *et al.*;[274] of the N27A phospholamban in MLVs by the same group;[275] of oxidized and reduced y1fatc, the FATC domain of yeast TOR1, bound to membrane-mimetic bicelles by Dames;[276] of Pa4 pardaxin in LPS micelles by Bhunia *et al.*;[277] of EphA2 transmembrane domain embedded in DPMC/DHPC bicelles by Bocharov *et al.*;[278] of the hinge deleted melittins in DPC micelles by Saravan *et al.*;[279] of lung surfactant peptide $KL_4$ in POPC/POPG and DPPC/POPG lipid vesicles by Long *et al.*;[280] of the antifungal Psd1 pea defensin with PC, DPC and CMH vesicles by de Medeiros *et al.*;[281] of sticholysin I with DPC by Castrillo *et al.*;[282] of the intra-membrane histidine kinase Ybdk TM domain in DPC micelles by Kim *et al.*;[283] of several neuropeptides and their analogues in DPC micelles by Zdobinsky *et al.*;[284] of HIV-1 virus protein U cytoplasmic domain in DPC micelles by Wittlich *et al.*;[285] of the dengue virus fusion peptide in DPC micelles by Melo *et al.*;[286] of a double transmembrane fragment of a G-protein coupled receptor in LPPG micelles by Neumoin *et al.*;[287] of the PC1/3 DCSG-sorting domain in CHAPS micelles by Dikeakos *et al.*;[288] of the critical transmembrane segment XI in DPC micelles by Lee *et al.*;[289] of the 4F antiatherogenic peptide in DMPC discs by Mishra *et al.*;[290] of CD4mut comprising the human CD4 transmembrane and cytoplasmic domains in DPC micelles by Wittlich *et al.*;[291] of Nogo-66, the extracellular domain in DMPC vesicles and DPC micelles by Vasudevan *et al.*[292]

Haney *et al.*[293] have reviewed solution structures of amphibian antimicrobial peptides in the presence of membrane mimetic micelles or bicelles obtained with solution NMR. Thomas *et al.*[294] have systematically studied using $^2$H SSNMR the extent of helix kink caused by a single proline within the isolated TM helical domain of WALP19 in DOPC, DPMC or DPLC. The influence of membrane curvature (in the order: MLV, SUV, DPC, DM) on the structure of the phospholipase C-δ1 pleckstrin homology domain has been investigated with the aid of $^{13}$C SSNMR by Uekama *et al.*[295]

The secondary structure of the cav-1(94-102) juxta-membrane segment of caveolin-1 in interaction with various membrane models has been determined by Le Lan *et al.*[296] using 2D NOESY and proton/deuterium exchange experiments. In addition, the localization of the peptide in DPC micelles has been established with intermolecular NOEs. Water-accessible residues in the trasmembrane β-barell of OmpX in DHPC solution have been mapped by Catoire *et al.*[297] with hydrogen/deuterium exchange measurements. The secondary structure of TM2-$GABA_A$ peptide within the phospholipid bilayer has been determined by Kandasamy *et al.*[298] with the aid of REDOR experiments and its orientation in membrane has been measured with 2D PISEMA. Salnikov *et al.*[299] using 2D $^{15}$N-δ *vs.* $^1$H-$^{15}$N dipolar coupling solid–state NMR correlation spectroscopy have found that ampullosporin A and alamethicin adopt mixed $\alpha/3_{10}$-helical structures in their trans-membrane configuration. Location of HIV fusion peptide and its cross-linked oligomers in cholesterol-containing membranes have been studied by Qiang and Weliky[300] using $^{13}$C-$^{31}$P REDOR. The same group has compared the structures of FP34, N70 and FP-hairpin constructs of HIV gp41 in membranes with physiologically relevant cholesterol content

and in membranes without cholesterol using REDOR spectra.[301] The effects of anesthetics on the tilt and rotational angles of TM2 helices (the second transmembrane domains of the neuronal α4β2 nicotinic acetylcholine receptor) in magnetically aligned 14-O-PC/6-O-PC have been observed by Cui *et al.*[302] in PISEMA spectra. Cholesterol has been found to reduce pardaxin's dynamic and tilt in phospholipid membrane by Ramamoorthy *et al.*[303] using BB-PISEMA.

It has been shown by Williamson *et al.*[304] in studying amyloid polypeptide, IAPP binding to lipid using CPMG that the amount of helix formed affects the rate of amyloid assemble.

Temperature-dependent relaxation study ($^1$H $T_{1\rho}$) of membrane-bound influenza A M2 transmembrane peptide carried out by Cady and Hong[305] has revealed amantadine induced better puckering of peptide tetramers. The same group have used $^{13}$C and $^{15}$N SSNMR to show the role of Ser31 in amantadine binding and its influence on the bound peptide structure[306] and $^{13}\{C^2H\}$ REDOR to establish a 0.3 Å resolution structure of this binding site.[307]

Duchardt *et al.*[308] using 2D ROESY and NOESY techniques have demonstrated that the cyclic form of a cell-penetrating peptide from the N-terminal domain of human lactoferricin undergoes a conformational transition when interacting with heparan sulphate proteoglycans.

Solid-state $^{13}$C-$^{13}$C correlation spectroscopy of membrane (DTPC/DTPG) associated influenza virus fusion peptide, IFP, has been used by Sun and Weliky[309] to confirm the existence of helix-turn-helix motif and pH dependent two conformational states of the peptide.

Bodner *et al.*[310] using $^1$H-$^{15}$N HSQC, lipid-to-amide NOE transfer experiments, $^{15}$N TROSY experiments for $R_2^T$ measurements and PFG NMR experiments have found that α-synuclein binds to DOPE/DOPS/DOPC SUVs with stable multiple competitive modes depending on lipid/αS stoichiometry. The same group have viewed the phospholipid binding properties of the disease variants of α-synuclein using solution NMR methods.[311]

From the combined use of solid state NMR and MD simulations a large structural flexibility has been concluded by Vogel *et al.*[312] for the peptide that represents the terminal seven amino acids of human N-Ras protein in DPMC. SSNMR and MD studies performed by Lam *et al.*[313] for equinatoxin II N-terminus in DPMC and DPMC/SM bilayers have indicated significant environment dependent secondary structure differences. Selective labelling has been used by Lange *et al.*[314] to monitor the nucleotide binding site of the bacterial ABC transporter ArtMP in nucleotide-bound or –unbound states in native lipid environment.

$^{15}$N chemical shift tensors have been used by Chemenkev *et al.*[315] to investigate fast in-plane motions of piscidin along the membrane surface. Different binding modes to DPC micelles for different cyclotides have been found by Wang *et al.*[316] It has been postulated by Vos *et al.*[317] that NMR spectroscopy yielding high-resolution structure at atomic level should be complemented with other 'low resolution data' in order to properly describe membrane-bound proteins; 'I' and 'L' states of M13 protein served as an example. The results of the NMR investigations supported with EPR and

SAXS methods of an integral membrane protein interactions with different detergents carried out by Columbus *et al.*[318] have suggested that the completeness of the NMR observations depend on the matching of the micelle dimensions to the proteins hydrophobic surface.

SSNMR approaches used to study topological equilibria and dynamics of membrane peptides have been reviewed by Salnikov *et al.*[319]

The 3D structure of the SARS coronavirus envelope protein channel in DPC micelles has been established by Pervushin *et al.*[320] using several NMR techniques including paramagnetic probes and residual dipolar couplings. Activation energy of excited state (T- to R-state transition) of the integral membrane protein phospholamban DPC-bound has been measured using CPMG at different temperatures by Traaseth and Veglia.[321] The secondary structure and dynamics of green proteorhodopsin in DPMC/DPMA environment have been analyzed by Shi *et al.*[322] with the aid of 3D and 4D MAS SSNMR spectroscopy. Using 2D SSNMR difference spectroscopy for rhodopsin/transducer (SRII/HtrII) complex from *Natronomonas pharaonis* in a natural membrane environment Etzkorn *et al.*[323] have identified residues that may act as a functional module around the retinal binding site during the early events of protein activation. The use of $^{2}$H SSNMR methods used for study interactions of the retinal cofactor with rhodopsin in detergent micelles has been reviewed by Brown *et al.*[324] The binding of rhomboid protease from *Pseudomonas aeruginosa* to lipid membranes (DDM, DPC, LMPC, LPPG) has been studied with $^{1}$H-$^{15}$N HSQC by Sherratt *et al.*[325] The CRINEPT-TROSY experiment designed to provide $^{1}$H-$^{15}$N correlations has been used for the first time by Caillet-Saguy *et al.*[326] to characterise a large *holo*- and *apo*-HasA-HasR complex in DPC micelles. The 3D structure of Rv1761c (127 residues) from *Mycobacterium tuberculosis* in DPC micelles has been determined by Page *et al.*[327] The binding between human profilin I and inositol 1,4,5-triphosphate has been investigated by Richer *et al.*[328] with NMR titration experiments. Veldkamp *et al.*[329] have shown that DMPC/DHPC bicelles promote dimerization of SDF-1 protein. Barbar *et al.*[330] have analyzed the effect of cholesterol binding on the structure of steroidogenic acute regulatory (StAR) protein structure by using solution-state $^{1}$H-$^{15}$N HSQC spectra.

### 4.3 Simultaneous elucidation of the effects on lipid and protein

Solution NMR has been used by Bourbigot *et al.*[331] to determine the structure of an antimicrobial peptide IL-8α in SDS micelles, and solid-state $^{31}$P, $^{15}$N and $^{2}$H NMR to probe the interaction of the peptide with oriented POPC/POPG bilayers. No evidence of bilayer disruption was found in this case. The effect of the dermadistinctin K on the organisation of the lipid bilayer with $^{31}$P solid-state NMR has been studied by Verly *et al.*[332] and the alignment of the peptide within a lipid membrane has been investigated by $^{15}$N solid state NMR. Antharam *et al.*[333] have analyzed the secondary structure of $KL_4$ peptide interacting with lipid bilayers and studied the effect of $KL_4$ on lipid head-group interactions and lipid acyl chain dynamics with $^{31}$P NMR and $^{2}$H NMR, respectively. 3D structures of C-terminal analogues of human β-defensin-3 in the presence of DPC/POPG micelles have been determined with 2D NOESY spectra by Bai *et al.*[334] In addition,

lipid phase partition of the peptides has been characterized with DOSY experiments. The 2D heteronuclear NMR spectra in lipid nanodiscs have been reported for an IMP by Glück *et al.*;[335] for VDAC-1 by Raschle *et al.*[336]

To ensure that the functionally active state of an IMP protein is present in the conventional membrane mimetic, Shenkarev *et al.*[337] have proposed as a reference the 2D $^{1}H,^{15}N$-fingerprint of a protein measured in a lipid-protein nanodiscs. Morrissey *et al.*[338] have reviewed usage of solid-state NMR in combination with nanodiscs as a platform for blood clotting proteins-membrane interactions.

$^{13}CO$-$^{31}P$ and $^{13}CO$-(16-$^{19}F$) REDOR have been applied by Qiang *et al.*[339] to trace membrane locations for several different HIV fusion peptide constructs. Charge- and hydrogen bond stabilized interactions of side-chains of Arg and Lys residues of a cell-penetrating peptide with the lipid phosphate groups have been analysed by Su *et al.*[340] with the aid of $^{13}C$-$^{31}P$ REDOR. It has been shown by Tang and Hong[341] using a variety of solid-state NMR techniques that PG-1, the arginine-rich-β-hairpin anti-microbial peptide, causes toroidal pore defects in the anionic membrane and, in addition, that the peptide structure is membrane dependent. The Arg-depleted PG-1 mutant ($\Delta_{4,18}$ $G_{10}$) has been shown by the same group[342] to form incomplete insertion of the peptide at the membrane surface.

Bhattacharjya and Ramamoorthy[343] have reviewed the mechanisms of action of two highly potent helical antimicrobial peptides, MSI-78 and MSI-594, with membrane. They discussed peptide structure-function correlations at the atomic level on the basis of NMR data. Solid state $^{15}N$ and $^{31}P$ NMR have been applied by Chenal *et al.*[344] for studying pH dependence of the diphteria toxin T domain interaction with POPC/POPG bilayer.

Proteorhodopsin has been reconstituted in DPMC/DMPA liposomes by Shi *et al.*,[345] and $^{13}C$ and $^{15}N$ backbone and side-chain chemical shifts for 103 out of 238 residues of this protein were assigned. The stability of CCR5 functionally reconstituted in rHDL has been shown by Yoshiura *et al.*[346] to be sufficient for methyl directed transferred cross-saturation (TCS) experiments for studying CCR5 with MIP-1α ligand interactions. $^{2}H$ and $^{31}P$ NMR have been used by Meier and Seelig[347] for investigation of the effect of the detergents of the *n*-alkyl-β-D-glucopyranoside class on the ordering of lipid bilayers and dynamics of membrane-embedded peptides.

## 5 Lipidated proteins

Besides primary amino acid composition also posttranslational lipidation of proteins is substantial for their hydrophobicity. The resulting biological effects are thought to be crucial for the association of lipidated protein with the cellular membranes as well as for protein-protein interaction, protein folding and stability. Additionally, the reversibility of the lipidated protein – lipid membrane association still remains a point of debate. Several recent papers have been focused on these topics.

It has been shown by Mascioni *et al.*[348] using 3D $^{15}N$-edited NOESY spectra and paramagnetic 5-doxyl stearic acid that the folded portion of the $Pam_3Cys$ lipidated rLP2086-B01 anchored to the micelle does not interact with its surface. Katre *et al.*[349] have found that recombinant ApolPBP1

becomes a pH sensor upon lipidation. High resolution NMR spectra have been obtained by Valentine *et al.*[350] for myristolyated recoverin and HIV-1 matrix protein both encapsulated in riverse micelles.

Dynamic properties of lipidated outer membrane protein LP2086-B01 with those of the non-lipidated free protein in solution have been compared by Mascioni *et al.*[351] using T1, T2 and $^{15}N(^{1}H)$NOE experiments. Epand *et al.*[352] using $^{31}P$ SS NMR have identified for the first time phosphatidic acid as one of the lipids bound to the bovine mitochondrial ADP/ATP carrier. Scheidt *et al.*[353] have used $^{2}H$ (powder spectra) and $^{31}P$ NMR (static spectra) to investigate the structure and dynamics of the myristoyl chain of myr-Src (tyrosine kinase) in phospholipid bilayers. Interaction of N-terminally acetylated peptide (used as a proxy for the phospholamban PLB) with the surface of the DMPC/DOPG membranes has been analyzed by Hughes *et al.*[354] using wide line $^{2}H$ and $^{31}P$ MAS NMR. Liu *et al.*[355] have presented the structure of myristoylated ADP-rybosylation factor1 (ARF1) and an assessment of the influence of myristoylation on association of ARF1 with lipid bilayer by solution NMR methods ($^{13}C$ excited $CC_mH_m$-TOCSY, NOESY-$^{15}N$ HSQC, HNHA, $^{1}H^{1}H$ TOCSY-$^{15}N$ HSQC experiments). The structure of $C_{15}$-surfactin-O-methyl ester, surface active lipopeptide produced by *Bacillus subtilis*, has been established using $^{1}H$ and $^{13}C$ NMR by Liu *et al.*[356]

Brunsveld *et al.*[357] have summarized the literature data on membrane binding of lipidated Ras peptides and proteins, among other methods solid-state $^{2}H$ and $^{31}P$ MAS NMR have been discussed.

## 6 Lipoproteins (non-covalent complexes)

Lipoprotein complexes are important objects of structural studies because of their involvement in both intercellular lipid transport. Modulation of these processes might lead to the development of pharmacologically important protocols (*e.g.* hypercholesterolemia diagnosis and treatment). A number of publications have been devoted to elucidation of these problems.

A series of loihichelins A-F, siderophores produced by the marine bacterium *Halomonas* LOB-5 have been characterized by Homann *et al.*[358] with the aid of proton and proton-carbon spectra. The structure of the ApoE(130-149)-CR17 fusion construct has been solved with NMR data and the minimal interface between both parts has been characterized with changes in chemical shifts by Guttman *et al.*[359] Sinnaeve *et al.*[360] have applied $^{3}J_{HH}$ couplings to show that the conformation of the backbone of the cyclic lipodepsipeptide Pseudodesmin A does not change between two solvents chloroform and acetonitrile. The binding mode of the domain V of plasma protein β2GPI with four LA modules from the low-density lipoprotein receptors LDLR and ApoER2 has been studied by Lee *et al.*[361]

$^{1}H$ NMR has been used by Carr *et al.*[362] to measure lipoprotein subclass particle concentration (different class produce a distinct methyl signal whose amplitude is directly proportional to lipoprotein particle concentration); by Wang *et al.*[363] to estimate the amount of low- and very low-density lipoproteins in rat serum after exposure to chlorpyrifos and carbaryl; by Burdge *et al.*[364] to measure the plasma lipoprotein size and

concentration after acute fish oil consumption; by Mora *et al.*[365] to elucidate the lipoprotein particle profiles (size and number) as a potential cardiovascular disease clinical marker. The same group[366] has elucidated the lipoprotein particle profiles (size and number) in association with clinical type 2 diabetes.

Intercellular lipid transport has been addressed by the study on the structure of lipid binding protein. Zornetzer *et al.*[367] have analyzed the effect of fatty acyl chain length on the dynamics of the Acyl Carrier Protein by $^{15}$N-$T_1$, $^{15}$N-$T_2$, and $^{1}$H-$^{15}$N heteronuclear NOE.

## 7 Lipids and membranes

Biological membranes play dual role in living cells - they protect cell integrity and simultaneously constitute the permeable barrier responsible for nutrients supply and environmental signal transmission. These functions are secured by specific membrane composition. Many researchers exploited various NMR techniques to elucidate structural and functional aspects of natural and model lipid membranes and hydrophobic cellular components.

### 7.1 Lipid structure and dynamics

1D and 2D NMR have been applied for structural elucidations: to estimate the structure of uvaol (triterpene) and other lipids of *Carpobrotus edulis* (inhibitors of the P-glycoprotein - the efflux pump responsible for the multidrug resistance of malignant cells) by Martins *et al.*;[368] to describe the structure of dehydrocostus lactone, proapoptotic component of hexane extract of *Saussurea lappa* Kim *et al.*;[369] to identify the structure of myelin penta- and hexa-acetyl-galactosyl-ceramides from rat brain by Podbielska *et al.*;[370] to describe the structures of 16 dihydro-β-agarofuran sesquiterpenes, potential modulators of P-glycoprotein dependent multidrug resistance by Torres-Romero *et al.*;[371] to elucidate the structure of nosokomycins, new antibiotics produced by *Streptomyces* sp. by Uchida *et al.*;[372] to identify the structures of chemically synthesized fourteen ursolic acid and oleanolic acid saponins (with N-acetyl-β-D-glucosamine oligasaccharide residues) by Wang *et al.*[373]

Capyk *et al.*[374] have identified the hydroxyl group at carbon-26 of cholesterol derivative molecule by 1D NMR which confirmed the classification of mycobacterial cytochrome P450 125 as a steroid C26-hydroxylase. 1D and 2D spectra have been used by Nguyen *et al.*[375] to identify the structure of 17,20,24-trihydroxyvitamin D2 – product of human cytochrome P450scc. A similar approach has been used by Tuckey *et al.*[376] to elucidate the structure of 1α,20-dihydroxyvitamin D3 – product of human cytochrome P450scc. Analogous experiments have been performed by Makarieva *et al.*[377] to elucidate the structure of isorhizocchalin, a bipolar sphingolipid of *Rhizochalina incrustata*; by Ohnuki *et al.*[378] to establish the structures of haplofungins, inositol phosphorylceramide synthase inhibitors from *Lauriomyces bellus*; by Bao *et al.*[379] to establish the structures of bicyclic α,ω-dicarboxylic acid derivatives from a colonial tunicate; by Sandjo *et al.*[380] to determine the structures of terpenoid derivatives from the leaves of *Triumfetta cordifolia*; to estimate the structures of acylglycerols from the

glandular trichome exudate of *Paulownia tomentosa* by Asai *et al.*;[381] the structures of acetylenic fatty acids, triterpenes and trigliceride from the leaves of *Hymenodictyon excelsum* by Nareeboon *et al.*;[382] the structure of $C_{37}$ skeletal carotenoid from the clam, *Paphia amabilis* by Maoka *et al.*;[383] the structure of the etheroleic acid as a predominant product of linoleic acid metabolism by divinyl ether synthase of the Lily-of-the-Valley roots by Ogorodnikova *et al.*;[384] by Gaenko *et al.*[385] to elucidate the structure of lipid components of *Clostridium butyricum* spores (with antitumor activity); by Tayone *et al.*[386] to determine the structure of achaetolide, polyhydroxylated 10-membered macrolide, of *Ophiobolus* sp.; by Whitson *et al.*[387] to establish the structures of fibrosterol sulfates (sulfated sterol dimers with an inhibitory activity against protein kinase C) of the sponge *Lissodendoryx fibrosa;* by Morinaka *et al.*[388] to identify the structures of amaroxocanes, sulfated dimeric sterols of the sponge *Phorbas amaranthus;* and by Qin *et al.*[389] to determine the structures of globosterol, polyhydroxylated steroid of the fungus *Chaetomium globosum*.

The following structures have been elucidated with the aid of 1D and 2D NMR spectroscopy: of cholesterol carboxyaldehyde formed upon reaction of cholesterol with singlet molecular oxygen and ozone by Uemi *et al.*;[390] of two prenylated flavonoids, potential inhibitors of acyl-coenzyme A:cholesterol acyltransferase by Choi *et al.*;[391] of 13-*cis*-retinoyl ferrocene derivatives by Long *et al.*;[392] of four novel oxylipins of the corn of *Dracontium loretense* by Benavides *et al.*;[393] of chlorosulfonolipids of the alga *Ochromonas danica* by Kawahara *et al.*;[394] of diterpenoid, multidione, from the stems of *Jatropha multifida* by Das *et al.*;[395] of the ascorbate-polyethylene glycol-DSPE conjugate (potential pharmaceutical nanocarrier) by Salmaso *et al.*;[396] of the products formed *in vitro* by soybean lipoxygenase by Zheng *et al.*;[397] of tanacetamide D, a ceramide from the leaves of *Tanacetum artenisioides* by Hussain *et al.*[398] and the structures of six lipidyl pseudopteranes of coral *Pseudopterogorgia acerosa*, potential inhibitors of protein tyrosine phosphatase by Kate *et al.*[399]

The structures of chlorosulfonolipids of the alga *Ochromonas danica* have been determined by Bedke *et al.*[400] using $^{1}H$,$^{13}C$ NMR and 2D HETLOC and HSQMBC experiments.

Ohyama *et al.*[401] have elucidated the biosynthetic pathway of phytosterol in *Arabidopsis thaliana* by estimation of the structure of the metabolically labelled sterols using $^{1}H$,$^{13}C$ and $^{13}C$-{$^{1}H$}{$^{2}H$}NMR.

Phospholipid composition of human lenses has been re-evaluated by Estrada *et al.*[402] using $^{31}P$ NMR, and lipid composition of bull muscles has been analyzed by Dannenberger *et al.*[403] using $^{13}C$ NMR. Gylfason *et al.*[404] have analyzed the phospholipid composition of lipid rafts isolated from brush border membrane of Atlantic cod with $^{31}P$ NMR, and Frederick *et al.*[405] analyzed the morphology and organization of hydrated dispersions of dioleoyl-bis(monoacylglycero)phosphate with wide line $^{31}P$ NMR.

The structure of chemically synthesized lipid derivatives has been studied, *e.g.* Cui *et al.*[406] have estimated the structure of chemically synthesized methyl esters of eicosapentaenoic acid monoepoxides, Hojabri *et al.*[407] have characterized the fatty-acid derived diisocyanate and biobased polyurethane, Li *et al.*[408] have elucidated the structure of chemically synthesized

(using selenium dioxide) allylic hydroxylated derivatives of the C18 unsaturated fatty acids, Banday *et al.*[409] have determined the structures of various chemically synthesized cholesteryl esters. Rawling *et al.*[410] have described the stereoselective synthesis of monounsaturated ω-3 fatty acids and analyzed their structure using $^{1}H$-$^{13}C$ HSQC experiment. Lessig and Fuchs[411] have followed the chemical (HOCl-mediated) degradation of unsaturated plasmalogens in PC membranes by using $^{31}P$ NMR. Guillén *et al.*[412] have elucidated the degradation process of the sunflower oil upon frying by estimation of the structure of its component-linoleic acid using $^{1}H$ NMR. Baillif *et al.*[413] have investigated the fatty acid elongation and desaturation steps in *Fusarium lateritium* by determination of the structure of methyl linoleate by quantitative $^{2}H$-{$^{1}H$}NMR in isotopic and chiral oriented solvents. Kooijman *et al.*[414] have investigated the pH-dependent ionization of phosphatidylinositol bi- and triphosphates in mixed PI/PC membranes using $^{31}P$ MAS NMR.

### 7.2 Lipid – lipid interactions

$^{31}P$ NMR has been used by Ahyayauch *et al.*[415] to analyze the composition of the PC/SM/CHO membranes, by García-Pacios *et al.*[416] to analyze the interaction of sphingosine-1-phosphate with DEPE membranes, by McMullen *et al.*[417] to examine the effect of cholesterol on the structure and organization of the PG membranes. The size and morphology of small bicelles formed by mixtures of dimyristoylphospatidylcholine (DMPC) ordi-O- tetradecylPC with dihexanoylphospatidylcholine or di-O-hexylPC has been assessed by $^{1}H$ and $^{15}N$ NMR by Wu *et al.*[418] Thermodynamic and structural behavior of equimolar mixtures of POPC and nonionic detergents (tetraethylene glycol ethers $C_n$; $n = 8, 12, 16$) has been elucidated by Pfeiffer *et al.*[419] using $^{2}H$ and $^{31}P$ NMR.

Model membrane (POPC/POPA/cholesterol) remodeling (interleaflet diffusion of cholesterol) has been analyzed by Bruckner *et al.*[420] using $^{13}C$ and $^{31}P$ NMR. Hoeller *et al.*[421] have characterized the commercial soybean microemulsions by generating self-diffusion coefficients of the individual lipid components by $^{1}H$- and $^{31}P$ NMR and DOSY experiment.

1D and 2D approaches have been used by Sasaki *et al.*[422] to measure the effect of pH on sphingosine and sphingosine-1-phosphate aggregation. Sivanandam *et al.*[423] have elucidated the motion of PC in the membranes with a focus on the interfacial region of the membrane using $^{13}C$ field cycling NMR relaxation studies of sn-2-carbonyl-$^{13}C$-PC.

Aucoin *et al.*[424] have compared the $^{1}H$ MAS NOESY experiment with the $^{1}H$ MAS RFDR (radiofrequency driven dipolar decoupling) on DMPC membranes to measure lipid motion and membrane – amino acids interactions. NMR-based molecular ruler for determining the depth of intercalants within the lipid bilayer have been developed Cohen *et al.*[425,426] using $^{1}H$- and $^{13}C$ NMR. Cohen *et al.*[427] have also noticed the aggregate formation in the intercalation of the long-chain fatty acids esters into liposomes.

Solid-state $^{2}H$ NMR has been used to elucidate various questions: Brownholland *et al.*[428] have investigated lipid organization - phase separation in binary mixtures of bipolar ($C_{20}$BAS) and monopolar (POPC) lipids;

Davis *et al.*[429] have followed the coexistence of liquid-ordered and liquid- disordered phases in ternary mixtures DOPC/DPPC-$d_{62}$/CHO; Soni *et al.*[430] have analyzed the effect of *trans* configuration of unsaturated fatty acid on their molecular organization in a phospholipid membrane; Orädd *et al.*[431] have investigated the effect of sterol on the lipid order and bilayer rigidity (transverse relaxation study). The same method has been used by Hsueh *et al.*[432] to analyze the effect of ergosterol on the physical properties of the POPE bilayer; by Morrow *et al.*[433] to follow the structural behavior of binary systems composed of $C_{16}$-ceramide or $C_{16}$-ceramide-1-phosphate with DPPC and by Juhasz *et al.*[434] to quantitatively characterize the coexisting phases in DOPC/DPPC/CHO mixtures using.

A new spin-labelled phospholipid analogue (doxyl-POPC) has been proven by Bunge *et al.*,[435] using solid-state $^{2}$H NMR, to be more appropriate to assess the versatile dynamics of POPC membranes than saturated analogue used earlier.

*Reviews*

Wassall *et al.*[436] have presented a review paper on the polyunsaturated fatty acid – cholesterol interactions and domain formation in the membranes; application of several NMR techniques has been described (*e.g.* $^{17}$O NMR - the measurement of membrane permeability, $^{2}$H NMR - the measurement of membrane order).

Lindblom *et al.*[437] have reviewed the application of the pulse field gradient (pfg)-NMR method for measurements of translational diffusion of molecules in macroscopically aligned lipid bilayers.

### 7.3 Lipid – drug interactions

Lipid vesicles (liposomes) have been proven to be a system of choice for the delivery of pharmaceuticals to the cells. Design and optimalization of the lipid vehicle requires the deep knowledge of the drug-liposome structure – it has been studied by NMR in several papers mentioned below.

The effect of curcumin on the model membrane (DMPC/DHPC bicelles) structure has been studied by Barry *et al.*[438] using solid-state $^{31}$P- and $^{14}$N NMR. Changsan *et al.*[439] have characterized the liposome (CHO/PC) suspensions containing rifampicin by $^{2}$H and $^{31}$P NMR solid-state NMR. Matsumori *et al.*[440] have analyzed the interactions between amphotericin B with ergosterol in deuterated ERG/POPC bilayers using $^{2}$H NMR. The same group[441] has investigated the amphotericin B-amphotericin B bimolecular interactions in sterol-containing POPC membranes by $^{13}$C{$^{19}$F}REDOR experiment using $^{19}$F- and $^{13}$C-labelled amphotericin B.

Interactions of cisprofloxacin with DOPC/DPPC and DOPC/DPPG membranes have been elucidated by Bensikaddour *et al.*[442] using static $^{31}$P NMR experiment. Xue *et al.*[443] have used $^{1}$H NMR to elucidate the hydrogen bonding between the gelator molecules (cholesteryl derivatives containing phthaloyl, isophthaloyl and terephthaloyl moieties) and various solvents.

Castro *et al.*[444] have analyzed the interactions between anesthetics (lidocaine and short chain alcohols) and DMPC membranes using $^{2}$H NMR, 2D $^{1}$H-$^{13}$C separated local field MAS. The effect of aminoglycoside

antibiotics (gentamicin, tobramycin and amikacin) on the thermodynamic properties of the DPPC membranes has been studied by Jia *et al.*[445] using $^{31}$P NMR.

Al-Abdul-Wahid *et al.*[446] have used a solution NMR ($^{13}$C,$^{1}$H $T_1$-HSQC) to measure the amphiphile immersion depth and orientation in membrane model system (built of dodecylphosphocholine).

## 8 Glycolipids

The structure of various natural glycolipids has been analyzed using NMR. $^{1}$H NMR has been used by Xu *et al.*[447] to determine the structure of fucosyl glycosphingolipid of brine shrimp and by Tani *et al.*[448] to establish the structure of neogala-series glycosphingolipids of the fungus *Hirsutella rhossiliensis*. Chechetkin *et al.*[449] have determined the structure of pathogen-inducible oxylipins (monogalactosyldiacylglycerol derivatives with esterified divinyl ether residues) from flax using $^{1}$H NMR and COSY spectra.

1D $^{1}$H- and $^{13}$C and 2D spectra have been used to determine the structure of two cholestane glycosides of the rhizomes of *Dioscorea septemloba* by Liu *et al.*[450] and to evaluate the structure of the cell-surface glycolipid of the spirochete *Spirochaeta aurantia* by Paul *et al.*[451] The structure of undecaprenyl phosphate-β-D-galactosamine, a sugar donor for biosynthesis of Lipid A has been analyzed by Wang *et al.*[452] using $^{1}$H, $^{13}$C, $^{31}$P NMR and 2D experiments.

## 9 Metabonomic studies

Development of NMR methodology and simultaneous rapid progress in computer technology made the high-throughput studies on cellular metabolom feasible. Elaboration of metabolomic approaches rapidly speeds up the basic studies and provides the perspective for the development of the new diagnostic tools. Numerous papers described below utilized such approaches for specific clinical problems using either the model systems (*in vitro* or *in vivo*) or the magnetic resonance imaging the tissue samples or the entire body.

$^{1}$H CPMG NMR spectra have been used by Wu *et al.*[453] to elucidate plasma metabolic profiles of functional dyspepsia patients, DF patients treated with acupuncture and healthy control subjects using $^{1}$H CPMG NMR spectra, and by Huo *et al.*[454] to follow biochemical changes in the serum of type 2 diabes mellitus patients treated with metformin hydrochloride.

$^{1}$H NMR has been applied to investigate the biochemical profiles of livers of zebrafish *Danio rerio* by Ong *et al.*[455] to examine metabolism in the liver and other tissues in Peroxisome Proliferator-Activated Receptor (PPAR)-α–null mice and wild type controls during aging by Atherton *et al.*[456] (tissue extracts have been analyzed), to assess the effect of dietary cholesterol in the development of fatty liver disease (quantitative profiling of liver extracts of mouse model) by Vinaixa *et al.*[457]

Dai *et al.*[458] have analyzed the water depletion induced metabonomic changes in *Salvia miltiorrhiza* (four sequential leaf extracts) by $^{1}$H NMR and 2D COSY, TOCSY, $^{1}$H *J*-RES, HSQC and HMBC spectra.

Profiling of human gut bacterial metabolism has been performed by de Graaf *et al.*[459] using metabolic labelling with [U-$^{13}$C]glucose of the *in vitro* model of human intestinal fermentation followed by $^{13}$C NMR and 2D HSQC experiment.

Logan *et al.*[460] have reported the characterization of the carbohydrate modifications on *Campylobacter jejuni* flagellin using metabolomics-based approaches with application of 1D $^{1}$H and $^{13}$C NMR and 2D experiments.

Klawitter *et al.*[461] have elucidated the metabolic characteristics of leukaemia cells – cell extracts were analyzed by $^{1}$H and $^{13}$C NMR.

Metabolomic analysis of the response of growing pigs to dietary L-arginine supplementation has been performed on animal serum using $^{1}$H NMR, 1D NOESY, 2D COSY and TOCSY spectra were collected by He *et al.*[462]

Allen *et al.*[463] have analyzed the impact of light on soybean embryos growth and metabolism by multiple labelling experiments and direct flux measurements employing $^{13}$C and $^{1}$H NMR.

Guénin *et al.*[464] have analyzed the therapeutic effect of methionine deprivation on melanoma tumours by $^{1}$H HR MAS NMR metabolomic analysis of pieces of intact melanoma tissues.

Raina *et al.*[465] have followed the efficacy of silibinin on prostate cancer metabolism in mouse prostate model using quantitative $^{1}$H HR-NMR metabolomics.

The metabolic profile of human healthy and neoplastic colorectal tissues has been obtained using *ex vivo* $^{1}$H HR MAS NMR by Righi *et al.*[466]

Li *et al.*[467] have applied metabolic profiling to follow the *Schistosoma mansoni* infection in mice using $^{1}$H MAS NMR of the tissues; 2D COSY and TOCSY spectra were also acquired.

Duarte *et al.*[468] have monitored the effect of cell handling and storage on cell integrity using $^{1}$H HR MAS NMR; CPMG and 2D TOCSY and HSQC spectra.

Martin *et al.*[469] have evaluated the effect of different dairy-based food products on early atherogenesis in hiperlipidemic hamster using plasma-based $^{1}$H NMR metabolomics, 1D CPMG and 2D COSY and HSQC spectra.

$^{1}$H NMR metabolomics of human plasma revealed strong sex dependent profile in 17-year-old individuals; 1D CPMG spectra were collected by Bertram *et al.*[470]

Yao *et al.*[471] have examined the effect of phospholipids on the emulsion stability recovered after aqueous extraction of soybean flour and flakes by profiling and quantifying the phospholipids with $^{31}$P NMR.

Vauclare *et al.*[472] have followed the metabolic rearrangement during dark-induced carbohydrate starvation in soybean nodules using $^{13}$C- and $^{31}$P NMR.

Sébédio *et al.*[473] have reviewed the recent advances in metabolomics tools including application of NMR techniques. Lane *et al.*[474] have described the methodology and approaches, including NMR, to stable isotope tracing in cells, animal models and in human subjects in the context of clinical cancer metabolomics.

## 10 New NMR methods

### 10.1 For sugars

Meier *et al.*[475] have proposed 3D H2BC experiment, very useful for sequential assignment in the analysis of complex carbohydrates, which yields simultaneously heteronuclear one- and two-bond and COSY correlations.

Vermillion and Price[476] have shown that [*carbonyl*-$^{13}$C]acetate labelling of sugars allows to obtain their 2D and 3D diffusion ordered $^{13}$C spectra (SIE-DOSY $^{13}$C NMR) in several minutes and hours, respectively.

Xia and Margulis[477] have implemented *J* coupling calculations in their Fast Sugar Structure Prediction Software which allows to investigate the solution structure of sacharides.

α-Cyclodextrin has been shown by Rudzińska *et al.*[478] to be a convenient chemical shift reagent for determination of the enantiomeric composition of α-hydroxyalkenephosphonic acids by 1D $^{31}$P NMR and 2D ROESY spectra.

### 10.2 For peptides/proteins

Soong *et al.*[479] have implemented two-dimensional proton-evolved local-field (2D PELF) pulse sequences which can be used for the measurements of a broad range of heteronuclear dipolar couplings, allowing a complete mapping of protein dynamics in a lipid bilayer environment; magnetically aligned bicelles containing cytochrome $b_5$ served as an example.

New sensitivity enhanced HETCOR solid-state experiments (2D SE-HETCOR, 3D HETCOR-SLF and 3D SE-PISEMAI-HETCOR) have been presented by Gopinath *et al.*[480] for oriented systems – the integral membrane protein sarcolipin oriented in lipid bicelles served as an example.

Bertelsen *et al.*[481] have presented a new method to obtain the local dynamics of membrane proteins by measuring $^{1}$H-$^{15}$N dipole-dipole coupling, $^{15}$N anisotropic chemical shift and $^{2}$H quadrupole coupling for a single residue; helix-tilt angle, wobbling and oscillatory rotation around the helix were measured by them for peptaibol alamethicin oriented in DPMC. The $CF_3$-Ala labelling strategy combined with $^{19}$F SSNMR have been applied by Maich *et al.*[482] to study orientation and tilt of petaibols in membranes.

Shi *et al.*[483] have proposed a refinement protocol to determine the structure, topology and depth of insertion of membrane proteins utilizing both solution and solid-state NMR data. This hybrid approach has been applied by this group to study the structure and topology of monomeric phospholamban in lipid membrane.[484]

Fu *et al.*[485] have reported the improvement in characterization of the topology of $^{15}$N-labelled piscidin aligned in hydrated lipid bilayer by the use of $^{1}$H-$^{15}$N HETCOR NMR spectroscopy at 900 MHz field.

Kouzayha *et al.*[486] have presented a $^{31}$P and $^{15}$N MAOSS solid-stateNMR strategy to study the effects of inserting transmembrane peptides in lipid bilayers.

Wang[487] has applied $^{1}$Hα-$^{13}$Cα crosspeak intensity plots from HSQC spectra (called HSQC waves) of micelle-bound antimicrobial peptides for indentifying key membrane–anchoring residues.

Holt *et al.*[488] have introduced a new $^{2}$H SSNMR based strategy for the analysis of orientation of TM peptides that provides more realistic value for

tilt angle of peptide in the presence of hydrophobic mismatch; for WALP23 peptide in DPMC membrane the value of 21° was determined.

Shenkarev *et al.*[489] have tested lipid-protein nanodiscs in NMR structural studies of membrane proteins.

Abdine *et al.*[490] have used cell-free expression and solid-state NMR for structural study of MscL, the selectively labelled membrane protein.

Franzmann *et al.*[491] have described the background of the method based on paramagnetic relaxation enhancement for determining at atomic resolution the orientation and insertion depths of peptides in micelles.

McDermott[492] has reviewed application of MAS SSNMR technique to study the structure and dynamics of membrane proteins of moderate size in lipid bilayers.

Van Horn *et al.*[493] have discussed the impact of window functions on NMR based paramagnetic relaxation enhancement measurements in membrane proteins.

Water-edited solid-state NMR spectroscopy that allows probing protein conformation in a lipid bilayer has been described by Ader *et al.*[494]

Segmental isotope labelling of glycoproteins has been reviewed by Skrisovska *et al.*[495] in relation to NMR investigations.

The $^{14}$N-PISEMA experiment which correlates $^{14}$N quadrupolar coupling and $^{1}$H-$^{14}$N dipolar coupling has been proposed by Qian *et al.*[496] as a sensitive probe for peptide orientation in planar membranes.

### 10.3 For lipids

Espindola *et al.*[497] have used a selective homonuclear decoupling of multiple protons simultaneously that allows a fast and reliable determination of specific coupling values from complex spectra and permits determination of relative configuration of small molecules.

Petzold *et al.*[498] have established a semiconstant time 2D $^{1}$H-$^{31}$P COSY experiment that allows identification and quantification of phospholipids in complex mixtures; phospholipids of *Helicobacter pylori* have been analyzed as an example.

Lundbom *et al.*[499] have elucidated the detailed echo time behaviour of the selected fatty acid and explored the *in vivo* feasibility of long TE spectroscopy in characterizing human adipose tissue using $^{1}$H NMR with a PRESS sequence.

Roberts *et al.*[500] have used the high resolution $^{31}$P field cycling NMR to measure the spin-lattice relaxation rate to explore the energy barriers associated with phospholipid motion in unilamellar vesicles.

Ciesielski *et al.*[501] have proposed the use of Lee-Goldburg decoupling in high resolution natural abundance $^{13}$C CP-MAS NMR to investigate changes in molecular and segmental dynamics of membrane lipids.

## 11 Data bases for sugars

Based on NMR parameters SOACS index, a new type of query of glycan sequences in *GlycomeDB*, several available unified web databases has been formulated by Maes *et al.*[502]

## 12 Miscellaneous

Long and Yang[503] using $^{15}N$ relaxation measurements have reported on MES buffer interference with functionally relevant human liver fatty acid binding protein (hLFABP) dynamics (μs – ms).

Nine new cannabinoids have been isolated and their structures solved by Radwan *et al.*[504] Endocannabinoid binding to the cannabinoid receptors has been reviewed by Reggio;[505] this includes NMR studies. The analysis of the structure of the C-terminus of the human cannabinoid receptor in the presence of DPC micelles carried out by Ahn *et al.*[506] has revealed two amphiphathic helices. Tiburu *et al.*[507] have reported membrane dependent structure of a human cannabinoid receptor-1 helix domain.

Quantitative characterisation of insertion depth and relative orientation of ganglioside GM1 in DPMC/CHAPSO bilayer has been done by DeMarco *et al.*[508] utilizing paramagnetic relaxation data. The interactions between micelles of ganglioside lyso-GM1and amyloid β have been characterised with various NMR techniques by Yagi-Utsumi *et al.*[509]

The interactions of the C2B domain of Syt1, p40 synaptotagmin with SUV in the presence of $Cu^{2+}$ cations have been characterised by Kathir *et al.*[510] using $^{1}H$-$^{15}N$ HSQC spectra.

$^{15}N\{^{31}P\}$REDOR experiments have been used by Garimella *et al.*[511] to measure the distances between D-alanine amine groups of teichoic acid and phosphate groups within cell wall fragments of *Bacillus subtilis* in the presence and absence of $Mg^{2+}$ ions.

Ogino *et al.*[512] have introduced $^{15}N$ labelled Tβ4 into living mammalian cells by reversible membrane permeabilization and measured in-cell NMR spectra of this protein.

Kielec *et al.*[513] have employed reverse micelle encapsulation to solubilise the protein in a low-viscosity solvent. This approach allows using of standard triple-resonance NMR methods in structural studies. The approach has been applied by the authors to a 54 kDa construct of the heterodimeric potassium channel KcsA in CTAB/HDAB micelles.

A cross-polarization 31P NMR has been applied by Fotakis *et al.*[514] for studying conformational changes and dynamics in DPPC multilamellar bilayers under influence of antihypertensive $AT_1$ antagonist, Losartan.

Bolaamphiphile-class surfactants composed of two maltoside headgroups connected by long saturated alkyl chain have been tested using NMR methods by Li *et al.*[515] for their ability to stabilize a solubilised membrane protein, DAGK.

## 13 Abbreviations used

| | |
|---|---|
| CHAPS-3 | [(3-cholamidopropyl)-dimethylammonio]-1-propansulfonate; |
| CHAPSO | 3-[(3-cholamidopropyl)-dimethylammonio]-2-hydroxy-1-propanesulfonate; |
| CHO | cholesterol; |
| Cl | cardiolopin; |
| CMH | glucosylceramide; |
| CTAB | cetyltrimethylammonium bromide; |

| | |
|---|---|
| DDM | dodecyl maltoside; |
| DEPE | 1,2-dielaidoyl-sn-glycero-3-phoshoetanolamine; |
| DHAB | dihexadecyldimethylammonium bromide; |
| DHPC | 1,2-dihexanoyl-*sn*-glycero-3-phosphocholine; |
| DMPA | 1,2-dimyristoyl-sn-glycero-3-phosphate; |
| DMPC | 1,2-dimyristoyl-*sn*-glycero-3-phosphocholine; |
| DMPG | 1,2-dimyristoyl-*sn*-glycero-3-phosphoglycerol; |
| DOPC | 1,2-dioleoyl-*sn*-glycero-3-phosphocholine; |
| DOPE | 1,2-dioleoyl-*sn*-glycero-3-phosphoethanoloamine; |
| DOPG | 1,2-dioleoyl-sn-glycero-3-[phospho-ra*c*-(1-glycerol)]; |
| DOPS | 1,2-dioleoyl-*sn*-glycero-3-phospho-L-serine; |
| DPC | dodecylphospatidylcholine; |
| DPPC | 1,2-dipalmitoyl-sn-glycero-3-phosphocholine; |
| DPPS | 1,2-dipalmitoyl-*sn*-glycero-3-phosphoserine; |
| ERG | ergosterol; |
| GUV | giant unilamellar vesicles; |
| LMPC | 1-myristoyl-2-hydroxy-*sn*-glycero-3-phosphocholine; |
| LPPG | 1-palmitoyl-2-hydroxy-*sn*-glycero-3-[phospho-ra*c*-(1-glycerol)]; |
| LUV | large unilamellar vesicles; |
| PC | phospatidylcholine; |
| POPA | 1-palmitoyl-2-oleoyl-*sn*-glycero-3-phosphate |
| POPC | 1-palmitoyl-2-oleoyl-sn-glycero-3-phosphocholine |
| POPG | 1-palmitoyl-2-oleoyl-sn-glycero-3-phosphoglycerol |
| SM | sphingomyelin; |
| SUV | small unilamellar vesicles |

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