

Selected Abstracts

Papers published in 1991-94 listed in journal reference order.

1. Self-assembly of the fullerenes

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Acc. Chem. Res. 25(3) 1992 98-105

The methods of producing and observing fullerenes are discussed, especially for mols. $>C_{60}$. Large clusters can be prepd. and obsd. using a laser vaporization supersonic cluster beam source with a Fourier transform ion cyclotron resonance mass spectrometer. Mass spectra of clusters up to C_{350}^+ have been found. Published mass spectra are reproduced for carbon clusters of various sizes and of yttrium metallofullerenes. The only mechanism to fit the known facts for C_{60} formation is that of pentagon open graphitic sheet growth.

37 Refs. 8 Figs. 0 Tables

2. Characterization of fullerenes by mass spectrometry

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Acc. Chem. Res. 25(3) 1992 162-168

The applications of mass spectrometry to the discovery and more recent study of fullerenes are discussed. Early studies on the laser vaporization of graphite used time-of-flight Fourier transform techniques which revealed the existence of large C_n clusters. MS/MS studies were used to obtain the ionization energies and proton affinities. Fragmentations of fullerenes were followed in photodissociation-tandem time-of-flight expts. and high-energy CAD-MIKES studies. Fullerene endohedral complexes with He and Yt have been obsd. in the gas phase by MIKES and direct laser vaporization. The techniques of matrix-assisted laser desorption and electrospray ionization should be applied to the study of fullerenes.

48 Refs. 5 Figs. 0 Tables

3. Laser desorption ion trap mass spectrometry of the macromolecular component of coal

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Am. Chem. Soc. Div. Fuel Chem. Prepr. 38(1) 1994 318-323

Efforts are described to apply laser desorption ion trap MS to measure high mol. wt. mass spectra representative of the macromol. network of covalently bound heteroatoms and ultimately, to determine the chem. structure of org. sulphur contd. therein. Solvent exts. are also prepd. with pyridine. The matrix is 2,5-dihydroxybenzoic acid.

MS: ion trap, electrodes from a Finnigan MAT ITD 700, 6 kV conversion dynode, Nd:YAG laser 1064 nm.

A test compd. (bovine insulin) spectrum is shown but no spectra are obtd. from coal exts. The failure is attributed to sample prepn.

23 Refs. 2 Figs. 0 Tables

4. Role of on-line mass spectrometry for studying the structure/reactivity relationships and conversion processes of coal

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Am. Chem. Soc. Div. Fuel Chem. Prepr. 39(1) 1994 36-41

The role of MS in studies of structure/reactivity relationships and conversion processes of coal is reviewed. The basic MS configurations used in this field are described and include LAMMA (Laser Microprobe Mass Analyzer), FAB, direct probe pyrolysis (Py)-FIMS, Curie-point Py-MS, low eV EIMS, thermogravimetry-MS, mol. beam interfaces, GC/MS and Py-GC/MS.

42 Refs. 7 Figs. 0 Tables

5. Comparison of several contemporary ionization/mass analyzer techniques for large components of complex fossil-derived materials

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Am. Chem. Soc. Div. Fuel Chem. Prepr. 39(3) 1994 831-834

Ionization techniques (laser desorption (LD), DCI and desorption EI-high resolution MS) are compared for anal. of large components of complex fossil-derived materials. Argonne Premium Coal samples are employed.

MS1: Kratos MS50, EI, DCI: isobutane, samples on wire coil 200°-100°/min-700°, source 200°.

MS2: home-built linear TOF, or Kratos MALDI III reflectron TOF, Nd:YAG laser, 118 nm.

The DCI spectra show diff. mol. wt. distributions, but similar ion series. In general LD, DCI and EI give progressively lower distributions. The LD and DCI ions are directly comparable, while LD and EI produce diff. ion distributions. Information on neutrals is available from LD-photoionization. An ion series in the m/z 700-1000 range demonstrates an advantage of 10.5-eV ionization and TOF anal.

10 Refs. 3 Figs. 1 Table

6. Studies on advanced glycation end products by recent mass spectrometric techniques

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Amino Acids 6(1) 1994 65-96

Results obtd. by diff. MS approaches in the field of advanced glycation of proteins are reported and discussed in detail and compared with those obtd. by other anal. methodologies (fluorescence and absorbance spectroscopies, RIA, ELISA). They are subdivided in three main groups: anal. on degraded glycated proteins, direct anal. of glycated proteins and studies on the reaction between protected lysine and glucose. MS is a particularly valid anal. method in this field of research.

58 Refs. 17 Figs. 7 Tables

7. Protein molecular weight determination by MALD mass spectrometry: a superior alternative to gel filtration

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Anal. Biochem. 209(2) 1993 379-380

MALD MS is compared with gel filtration (Superdex 75 HR 10/30) for protein mol. wt. determinations. Sinapinic acid matrix is dissolved in MeOH and mixed with ferredoxin (FER, [CAS 9040-09-9]) or horse skeletal myoglobin [CAS 100684-32-0] to a molar ratio of 1000:1. Sample (500 pmol protein) is deposited on the stainless steel probe tip and allowed to dry.

MS: MALD TOF, 28 kV.

The FER mol. wt. is found to be 60% greater than the value calcd. using gel methods.

4 Refs. 1 Fig. 1 Table

8. Matrix-assisted ultraviolet laser desorption/ionization mass spectrometry applied to multiple forms of lipases

Hedrich, H.C.(1), Isobe, K.(2,3), Stahl, B.(4), Nokihara, K.(2), Kordel, M.(2), Schmid, R.D.(2), Karas, M.(4), Hiltenkamp, F.(4), Spener, F.(1)

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Anal. Biochem. 211(2) 1993 288-292

Matrix-assisted UV laser desorption/ionization MS is used to investigate heterogeneous patterns and mol. masses of microbial lipases from *Penicillium camembertii* (PC), *Geotrichum candidum* (GT) and *Pseudomonas sp.* (PS). Purified native and deglycosylated proteins are dialyzed or diafiltered against Na acetate buffer and lyophilized. MALD matrices are 2,5-dihydroxybenzoic acid and nicotinic acid.

MS: reflector-type TOF as previously described (Hiltenkamp and Karas, Methods Enzymol. 193, 1990 280).

Mass spectral peaks of the native glycosylated lipases from PC and GT are broader than those of the corresp. deglycosylated enzymes, indicative of heterogeneous glycosylations. Mol. masses determined for the deglycosylated species are in excellent agreement with those deduced from amino acid composition and sequence data. Conventional methods give only rough estimations of mol. masses.

25 Refs. 3 Figs. 1 Table

9. Peptide mass maps: a highly informative approach to protein identification

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Anal. Biochem. 214(2) 1993 397-408

A computer searching algorithm is used to identify protein sequences in the Protein Information Resource (PIR) database with peptide mass information (mass map) obtd. from proteolytic digests by microcapillary electrospray (ESP) LC/MS. Theor. anal. of the cytochrome c family demonstrates the ability to identify protein sequences in the PIR database with a high degree of accuracy using a set of six predicted tryptic peptide masses. The method is also applied to experimentally determined peptide masses for a small GTP-binding protein, protein from pig uterus, the human sex steroid binding protein and a thermostable DNA polymerase.

LC: Applied Biosystems 140A, 15 cm x 0.32 mm i.d. (5 μ m C₁₈), 0-100% acetonitrile (0.085% trifluoroacetic acid, TFA) in 0.1% aq. TFA, 2-4 μ l/min.

MS: Finnigan MAT TSQ 700, 20 keV conversion dynode, m/z 400-1800, 400 amu/s, CAD: Ar collision gas, 5 mtorr, 20-50 eV.

A set of obsd. masses < 50% of the total number of predicted masses can be used to identify a protein sequence in the database. A mass matching tolerance of 1 amu is used. Under these conditions, mass maps created by FAB/MS and MALDI TOF would also be applicable.

29 Refs. 2 Figs. 7 Tables

10. Site-specific characterization of glycoprotein carbohydrates by exoglycosidase digestion and laser desorption mass spectrometry

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Anal. Biochem. 218(1) 1994 34-46

A rapid and sensitive method for sequencing oligosaccharides (OG), using MALDI MS to monitor the digestion of glycopeptides by specific exoglycosidases, is described. The method is illustrated by characterization of recombinant human tissue inhibitor of metalloproteinases (TIMP), which has two glycosylation sites. Glycopeptides which span residues Asn30 and Asn78 are generated by tryptic digestion of 1 nmol of TIMP and sepd. by RP-HPLC. The OG composition of the glycoforms is inferred from the obsd. mass shifts following digestion by peptide-N-glycosidase F. Composition and sequence are then elucidated by digestion with specific exoglycosidases, using a total of 200 pmol of each glycopeptide. Glycopeptides from well-characterized proteins, fetuin, α_1 -acid glycoprotein and tissue plasminogen activator are also analyzed to confirm exoglycosidase specificity for glycopeptides and establish the quant. significance of the relative intensities of peaks in the mass spectra.

MS: Finnigan MAT Lasermat, pulsed N₂ laser, 337 nm.

Both TIMP glycosylation sites exhibit extensive heterogeneity comprising mainly fucosylated complex OG, but in diff. proportions. The Asn78 site also contains 4.4% nonfucosylated mannose (Man₄) OG. The merits and limitations of this approach as a universal method for OG anal. are discussed.

46 Refs. 7 Figs. 3 Tables

11. An approach to locate phosphorylation sites in a phosphoprotein: mass mapping by combining specific enzymatic degradation with matrix-assisted laser desorption/ionization mass spectrometry

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Anal. Biochem. 219(1) 1994 9-20

A rapid, pmol-scale method is described to locate phosphorylation sites in phosphoproteins by MALDI-TOF MS combined with enzymatic modification of the analyte. It involves (i) degradation of the phosphoprotein into small peptides by specific enzymatic or chem. reactions, (ii) identification of the phosphopeptides by -80 (or multiples of -80)-Da mass shifts in the mass spectra after dephosphorylation with alk. phosphatase, (iii) location of the phosphorylation sites by mass mapping. As the size of the protein increases, it is advantageous to fractionate the mixt. by HPLC. Bovine β -casein is analyzed by this method.

MS: Vestec VT2000 linear TOF, 30 kV, 337 nm, 3 ns pulse, matrices: α -cyano-4-hydroxycinnamic acid, sinapinic acid, 2,5-dihydroxybenzoic acid.

Conclusions about the specific phosphorylation sites of bovine β -casein coincide with previously reported results. From calcns., a mass spectrometer with 0.1% mass accuracy is sufficient, for mass mapping, to identify completely or partially digested tryptic peptides in the mass range of 100-8000 Da from bovine β -casein (mol. wt. 23,983).

48 Refs. 9 Figs. 4 Tables

12. From electrophoretically separated protein to identification: strategies for sequence mass analysis

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Anal. Biochem. 221(1) 1994 1-15

A review is given of electrophoretic techniques for protein sepn. and addresses the interface with amino acid sequence anal. and MS (MALDI-TOF, electrospray).

132 Refs. 5 Figs. 1 Table

13. Oligosaccharides from human milk as revealed by matrix-assisted laser desorption/ionization mass spectrometry

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Anal. Biochem. 223(2) 1994 218-226

Neutral and acidic oligosaccharide (OGs) fractions prep'd. from human milk are investigated by MALDI MS. The fraction of neutral OGs is sep'd. by GPC and the resulting subfractions are analyzed by pos. MALDI MS.

MS: reflectron TOFs, LAMMA 1000, 3 keV, Finnigan MAT TOF VISION 2000 (high mol. wt. OGs), 5 keV, post-accel. 20 keV, N₂ laser 337 nm.

Several low-mol. wt. glycans (degree of polymerization up to 13) are obs'd. with known structures. In addition, a variety of unknown large-sized carbohydrates is detected with mol. wts. 2242-8000. The large-sized glycans of low abundance are composed of both lactosamine and fucose residues attached to the lactose unit at the reducing end of the sugar chains with a highly variable stoichiometry. Following subfractionation by GPC, acidic (i.e., contg. sialic acid) glycans are analyzed using both pos. and neg. ion modes. Because of the inferior stability of acidic glycans, various matrices are applied and compared with respect to signal intensity, resolution and analyte stability.

53 Refs. 6 Figs. 3 Tables

14. Matrix-assisted laser desorption/ionization mass spectrometry of biopolymers

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Anal. Chem. 63(24) 1991 1193A-1203A

The history, principles, instrumentation and application to biopolymer analysis of matrix-assisted laser desorption/ionization (MALDI) are discussed. Ion sources of various designs exist but they all use a pulsed laser source which transfers its energy in 1-100 ns to avoid thermal decomposition of the sample. Time-of-flight mass spectrometers of linear or reflectron design are used, the latter giving improved mass resolution. To be detected, the ions are converted into electrons or low-mass ions at a conversion electrode, which begin the cascade in an electron multiplier. The sample matrices absorb the laser energy and separate the analyte mols. from each other. Typical matrix-analyte ratios are 100:1 to 50,000:1 and typical matrices are nicotinic or sinapinic acid. MALDI spectra for lipoproteins, a membrane protein and a monoclonal antibody are given. The future of this technique is briefly discussed.

44 Refs. 8 Figs. 1 Table

15. Integration of mass spectrometry in analytical biotechnology

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Anal. Chem. 63(24) 1991 2802-2824

The application of mass spectrometry to peptide and protein structure analysis is exam'd., with special reference to tandem high-performance MS and online LC/MS using FAB or electrospray ionization. The role of matrix-assisted laser desorption MS in protein anal. is also discussed. Many examples are drawn from the authors' lab. to illustrate the use of MS in sequencing blocked proteins, defining N- and C-terminal sequence heterogeneity, locating and correcting errors in DNA- and cDNA-deduced protein sequences, identifying sites of chem. modification and defining struc-

tural classes of carbohydrates at specific attachment sites in glycoproteins.

182 Refs. 24 Figs. 3 Tables

16. Development of resonance ionization spectroscopy for DNA sequencing and genome mapping

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Anal. Chem. 64(5) 1992 315A-328A

The methodology of resonance ionization spectroscopy (RIS) and its application to DNA sequencing is discussed. Sputter-initiated RIS (SIRIS) uses an Ar beam to produce neutral particles which are probed by the RIS laser beams to ionize all atoms of a selected element label (Sn, Fe). Laser atomization RIS (LARIS) replaces the ion beam with a laser beam and releases 3 orders of magnitude more material than SIRIS. The effects of charge compensation and the choice of the label are discussed. The potential of both procedures for rapid DNA sequence anal. after gel electrophoresis and for genome mapping is examd.

28 Refs. 8 Figs. 0 Tables

17. Microscopic organic analysis using two-step laser mass spectrometry: application to meteoritic acid residues

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Anal. Chem. 64(6) 1992 682-690

A laser desorption-laser ionization time-of-flight mass spectrometer is developed for the anal. of the org. constituents of particulates and inhomogeneous samples with a spatial resolution of *ca.* 40 μm . A pulsed, low-power IR laser desorbs intact mols. which are ionized by a pulsed UV laser (266 nm) and analyzed in a reflectron-type instrument. The detection limit for coronene is 8 fmol. Acid residues from 6 particulate meteorite samples (200 μm in diameter) are analyzed and the PAH and alkyl-PAH compositions are measured and compared.

58 Refs. 10 Figs. 2 Tables

18. Mass discrimination in laser desorption/Fourier transform ion cyclotron resonance mass spectrometry cation-attachment spectra of polymers

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Anal. Chem. 64(7) 1992 763-769

The laser desorption-ionization (LDI) Fourier transform ICR mass spectra of low-mol.-wt. poly(ethylene glycol) (PEG, [CAS 25322-68-3]) are recorded to assess the extent of mass discrimination associated with the gas-phase ionization process.

MS: K halide soln. in acetone-H₂O (1:1, v/v) aspirated onto stainless steel probe tip, followed by PEG dissolved in methylene chloride, 3.0 T superconducting magnet, 2 cubic trapped cells with common trap plate, electronics from FTMS-2000, LDI from Nd:YAG laser at 1064 nm, product ions trapped and detected without transfer in source-trapped cell at 2 V.

The mass discrimination is attributed to factors that influence the relative overlap of gas-phase neutral and ion populations that react to form the detected cation-attached products. Specifically, these are the laser power density, reactant masses, trap potential and distance between the trapped ion cell and the desorption site. Thus, the no. averaged mol. wts. for PEG-600, -1000 and -1500 vary by 7, 10 and 12%, resp., as the desorption site is displaced up to 10 cm from the cell. An accuracy of 5-10% is the best to be expected for ave. mol. wt. determinations.

28 Refs. 6 Figs. 1 Table

19. Substrate-assisted laser desorption of neutral peptide molecules

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Anal. Chem. 64(9) 1992 1041-1045

The laser desorption of neutral peptide mols. is greatly enhanced by applying a thin layer (500 monolayers) of sample over a layer of sinapinic acid, rather than mixing the two together. Samples are analyzed by laser desorption-PICI-Fourier transform mass spectrometry.

Laser desorption: Questek 2110 excimer laser (248 nm), 1-10 MW, fired 2-5 times before sample spot is exhausted.

MS: Fourier transform, 2-stage sample introduction system, electromagnet at 1 T, reagent ions prepd. by EI of volatile gas are trapped in cubic analyzer cell to react with neutrals, spectra recorded from a single laser shot, corresp. to 100 pmol.

Peptides with up to 10 amino acids are desorbed as intact mols., with int. energies 1 eV less than those desorbed directly from a metal surface. Typical CI spectra are given (e.g. for gramicidin S and Val-Pro-Leu). The substrate appears to absorb the laser energy then transfer it to the analyte which desorbs, while remaining itself on the probe surface. The system is not so effective when the peptide strongly absorbs the laser wavelength (e.g. 193 nm).

31 Refs. 4 Figs. 0 Tables

20. Mass-to-charge ratio upper limits for matrix-assisted laser desorption Fourier transform ion cyclotron resonance mass spectrometry

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Anal. Chem. 64(13) 1992 1461-1469

Radial and axial *m/z* upper limits, defined by 50% trapping efficiency in the absence of ICR excitation, are derived for FT/ICR/MS. They are calcd. as a function of magnetic field strength and trapping potential for cubic, tetragonal, screened tetragonal, cylindrical and hyperbolic traps. Since

an increase in trapping voltage lowers the radial limit but raises the axial limit, an optimum trapping potential is derived and evaluated. Elongated and screened traps have an upper limit of 14,000 u/e at 7.0 T. The cubic trap should reach m/z 12,000 u/e.

67 Refs. 6 Figs. 2 Tables

21. Matrix-assisted laser desorption mass spectrometry of proteins isolated by capillary zone electrophoresis

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Anal. Chem. 64(14) 1992 1594-1600

Proteins in soln. are sepd. by CZE and analyzed off-line by matrix-assisted laser desorption mass spectrometry (MALD). The detection limit for α -lactalbumin is 100 fmol injected into the CZE column. Horse heart myoglobin is stable in the CZE buffer, (cyclohexylamino)ethanesulphonic acid-KCl, (pH 9.0) for up to 1 month, so that isolates may be stored before anal. With porcine pepsinogen, > 50% degradation is obsd. within 5 min in trifluoroacetic acid. Mass measurement accuracies of $\pm 0.02\%$ are obsd. for small model proteins. The identification of protein isolates by N-terminal sequencing, mol. mass measurements and selective peptide mapping is discussed.

46 Refs. 8 Figs. 3 Tables

22. Axial introduction of laser-desorbed ions into a quadrupole ion mass spectrometer

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Anal. Chem. 64(18) 1992 2079-2083

Pulsed IR laser desorption is linked with a quadrupole ion trap by a fibre optic laser probe interface, which replaces optical windows in the mass spectrometer. The ions are formed externally and pass through the holes in one end-cap electrode without the need for auxiliary injection optics. Trapping is most effective with a high He buffer gas pressure (> 1 mtorr) and a long storage delay (> 50 ms) prior to detection and a low rf trapping potential (350-650 V_{0-p}) during the desorption pulse. The method allows desorption of biol. relevant mols. such as gramicidin S (mol. wt. 1141). Ion/mol. reactions are also demonstrated for desorbed alkali metal cations or halide anions with volatile organics such as polyethers.

34 Refs. 10 Figs. 0 Tables

23. Contribution to the isolation and characterization of buckminsterfullerenes

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Anal. Chem. 64(18) 1992 2143-2148

Buckminsterfullerenes generated by a high-energy electric arc discharge between 2 graphite electrodes are extd.

ultrasonically with tetrahydrofuran or toluene (20 min), dried and redissolved in dichloromethane for sepn. by HPLC (C₁₈, *n*-hexane) followed by laser desorption FT/MS.

MS: Extrel FTMS-2000, neg. mode, Nd:YAG laser, 266 nm, 10⁶ W/cm².

Purified C₆₀, C₇₀ and C₈₄ are obtd. and their mass spectra are measured. The HPLC fractions contain 80% (C₆₀), 20% (C₇₀) and < 2% higher fullerenes.

33 Refs. 7 Figs. 0 Tables

24. Comparative study of photodissociation and surface-induced dissociation by laser desorption Fourier transform mass spectrometry

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Anal. Chem. 64(19) 1992 2238-2243

Photodissociation (PD) and surface-induced dissociation (SID) are compared for the structural anal. of a porphyrin and 2 metalloporphyrins by laser desorption Fourier transform mass spectrometry. MS/MS/MS expts. are also carried out.

MS: Nicolet FTMS-2000, 7-T superconducting magnet, Tachisto 215 pulsed CO₂ laser for desorption, Lambda Physik EMG 201-MSD excimer laser for PD or to pump LP FL-2001 dye laser for UV and vis. PD.

Laser desorption-PD and -SID spectra and efficiency curves are obtd. PD efficiencies are the higher. Optimum structural information is obtd. from PD when parent ions are irradiated for long times (10-30 s) at 575 nm and short times (0.5-1.0 s) at 308 or 388 nm. SID gives max. conversion efficiency at 62 and 115 eV for 2 porphyrins. The MS/MS/MS ion spectra are used to give general guidelines for studies of nonvolatile compds. by the 2 techniques.

21 Refs. 9 Figs. 0 Tables

25. Time-of-flight mass spectrometry for the structural analysis of biological molecules

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Anal. Chem. 64(21) 1992 1027A-1039A

The basic operation of time-of-flight mass spectrometers and the factors affecting mass resolution and accuracy are described. For the anal. of large mols., the initial conditions of the ionization method and the fate of metastable ions are optimized to improve performance. Plasma desorption (PD) and matrix-assisted laser desorption instruments and reflectron designs are described. Strategies and applications to protein anal. are given with several examples.

36 Refs. 11 Figs. 1 Table