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Ion trap mass spectrometry

A survey of recent research activity in quadrupole ion trap mass spectrometry

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Abstract

A review of the quadrupole ion trap literature from 1995 to the present time showing the dynamic nature of ion trap mass spectrometry and the wide variety of applications. (Int J Mass Spectrom 212 (2001) 337–357) © 2001 Elsevier Science B.V.

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1. Introduction

"Every great adventure has a beginning, but it is the continuing of it until it is fully accomplished that yields the true glory" (Francis Drake). This survey of recent research activity in the field of quadrupole ion trap mass spectrometry covers the period from 1995, following immediately after publication of a series of three volumes titled Practical *Aspects of Ion Trap Mass Spectrometry* [1], to the present time. During this period, a tutorial titled "An introduction to quadrupole ion trap mass spectrometry" appeared [2] together with a review of mass spectrometry by Burlingame et al. [3]; this review includes many references to applications of the quadrupole ion trap. A view of quadrupole ion trap mass spectrometry at the turn of the century, that appeared in the 200th volume of *International Journal of Mass Spectrometry*, contained a selective review of the field since 1995 [4]. In striving for completeness in this survey, many of the references given in the selective review have been repeated here.

The vitality and diversity of the field of quadrupole ion trap mass spectrometry is clearly evident in the numerous references cited here. For those who may be contemplating involvement in the field of quadrupole ion trap mass spectrometry, Celia Henry has produced an interesting article titled, "Building a better trap" [5]. This article contains a comparative tabulation of the following quadrupole ion trap mass spectrometers: Esquire LC (www.bruker.com), LCQ and GCQ (www.Finnigan.com), LC/3DQMS (www.hii.hitachi.com), and Saturn 2000 (www.varian.com).

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A celebratory contribution to honor Professor R. Graham Cooks on the occasion of his 60th birthday.

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2. Theory

It has long been recognized that a real ion trap, having truncated electrodes and perforations in the electrodes for admission of electrons and ejection of ions, does not have a "pure" quadrupole field. The physical requirements of a real ion trap introduce deviations from ideality such that the field within the ion trap is no longer linear (a requirement of a pure quadrupole field); these deviations have been called field faults or superpositions of higher multipole fields. All rotationally symmetric fields without space charge can be described by a weighted sum of such multipoles. It was recognized by the early workers in this field, that is, by von Busch and Paul and by Dawson and Whetten, that field faults had a deleterious effect on the performance of instruments with two-dimensional quadrupole fields since such field faults caused ion losses by so-called nonlinear resonances. Once the problem encountered by Finnigan of mass shifts in the ion trap had been overcome by the serendipitous stretching of the ion trap geometry, the problem was ascribed to the presence of nonlinear resonances in the ion trap. The following question arises: if such resonances (or field faults or superimpositions of higher multipole fields) cause undesirable ion losses, can this mechanism of ion loss be employed for the deliberate ejection of ions in a mass-selective manner? This question of the influence of nonlinear resonances has been considered at length by Franzen et al. ([1], Vol. 1, Chap. 3) and it has been shown that the influence on modern mass scanning methods of weak higher multipole fields is surprisingly strong.

Further investigations have been carried out of higher order nonlinear resonances of an ion cloud in a Paul trap [6,7]. Sheretov et al. have investigated the action of modulated parametric resonances and the influence of these resonances on stability diagram structure [8]. Radial and axial secular frequencies in a quadrupole ion trap have been probed [9], as have the axial secular frequency shifts induced by field imperfections in nonlinear ion traps [10]. The correlated oscillations of two oppositely charged thermal ion clouds in a Paul trap and the facility, with which the clouds may be detected, have been examined [11].

With respect to mass resolution, an account of extended theoretical considerations for mass resolution in the resonance ejection mode has been reported [12]. The effect of phase-locking ac and rf voltages for high mass analysis has been investigated further [13]; this work is a continuation of earlier work by the same authors [14]. Titov has reported in a series of papers the separation of ions in imperfect fields in a quadrupole mass analyzer; the most recent paper on experimental verification is germane to ion traps also [15]. Sheretov has formulated a general theory for the optimization of the rf potential applied to electrodes of quadrupole ion traps [16]. For those of us who like to see images, Guan and Marshall have presented an interesting paper on stereoscopic views of three-dimensional ion trajectories in ion cyclotron resonance and quadrupole ion traps [17]. Finally, in this section, there is an account of further work on the characteristics of a broadband Fourier transform ion trap mass spectrometer [18].

3. Ion trap behavior

3.1. Ion motion and space charge

Vedel and coworkers have discussed an experimental study of the efficiency of ion storage in an rf ion trap [19], and Guan and Marshall have examined the equilibrium space charge distribution in a quadrupole ion trap [20]. A phenomenological description of space-charge effects in a squeezed ion cloud in an ion trap [21], and the results of an experimental investigation into the performance of ion traps using air versus helium as the buffer gas have been presented [22]. The examination by a time-of-flight (TOF) method of ions ejected from an ion trap revealed unduly large energy distributions in the ion cloud due to the effects of the rf drive potential amplitude. Vedel and coworkers have described an attempt to overcome this difficulty in their examination of well-defined chopping of the rf potential up to a frequency of 1.5 MHz combined with a TOF method [23]. Cooks' group has elucidated axial ion motion within the ion trap using dc pulse tomography [24]. Todd and coworkers have investigated “ghost” peaks caused by nonlinear fields in the ion trap mass spectrometer [25].

3.2. Mass shifts

Mass shifts continue to attract the attention of workers in the ion trap field. Mo and Todd have carried out an investigation of the mass shifts caused by coupling of ion motions in the ion trap mass spectrometer [26], whereas the group of Cox has made a determined assault on this problem; they have investigated mass shifts and local space charge effects observed in the quadrupole ion trap at higher resolution [27], the in situ optimization of the electrode geometry of the quadrupole ion trap for reducing mass shifts [28], the control of chemical mass shifts in the quadrupole ion trap through selection of the resonance ejection working point and the direction of the rf scan [29], resolution of isobaric and isomeric ions using chemical shifts in an ion trap mass spectrometer [30], and combined experiments and simulations on chemical mass shifts in ion trap mass spectrometry [31].

3.3. Simulations

There has not been a great deal of activity in the simulation area. The use of commercial packages for the calculation of ion trajectories has, perhaps, precluded research in this area. The Cooks group has carried out multiparticle simulations [32], a simulation study of nondestructive detection of ions in a quadrupole ion trap using a dc pulse to force coherent ion motion [33], and a multiparticle simulation of ion injection under the influence of helium buffer gas using short injection times and dc pulse potentials [34]. Yoshinari [35] has carried out a numerical analysis of the behavior of ions injected into an ion trap, and the March group has reported a comparison of three simulation programs for the calculation of ion trajectories in an ion trap [36]. André and coworkers have carried out simulation studies of a new operating mode for a quadrupole ion trap [37]. Sevugarajan and Menon have reported on a simulation study of the effect of geometric aberration, space charge, dipolar excitation, and damping on ion axial secular frequencies in a nonlinear Paul trap [38].

4. Gaseous ion studies

4.1. Ion reactivity

Trimethylsilyl derivatization of nucleic acid anions in the gas phase has been investigated [39] and the derivatization of alkylphenols has been employed for molecular ion stabilization [40]. A comparison of cation-binding affinities of quinones [41] and an evaluation of binding selectivities of caged crown ligands towards heavy metals has been carried out by Brodbelt and coworkers [42], and the noncovalent inclusion complexes of protonated amines with crown ethers [43] has been reported. Other studies of ion reactivity that have been reported are of Diels-Alder reactions of mass-selected ions [44], the gas-phase reactivity of positive and negative even-electron ions prepared from diethylmethylphosphonate ester in the external chemical ionization source of an orthogonal tandem quadrupole/ion trap instrument [45], experimental and theoretical studies of the gas-phase reactivity of the $(HO)_2P{=}O^+$ phosphonium ions towards methanol [46], and the reactions of polyatomic anions with cations [47]. An unexpected methyl loss has been observed in the electron impact ionization of chalcones [48].

4.2. Ion excitation

Continued research on the optimization of the ion excitation process is of great importance in collision-induced dissociation for tandem mass spectrometry. The group of McLuckey has reported on the thermal dissociation of ions derived from leucine enkephalin [49] and on the internal temperature of collisionally activated protonated leucine enkaphalin [50]. They have reported further of relative dissociation energy measurements using collisional activation [51], competition between resonance ejection and ion dissociation during resonant excitation [52], collisional activation of generations 1–5 of protonated poly (propylene imine) dendrimers [53], the relaxation of internally excited high-mass ions simulated under typical quadrupole ion trap conditions [54], and they have proposed a first-order model for the kinetics of collision-induced dissociation [55]. Collision-induced dissociation (CID) studies have been carried out with some carbamate and phenylurea pesticides [56], protonated and alkali metal cationized aminoglycoside antibiotics [57], iron ethoxide cations (by both CID and infrared-induced fragmentation) [58], and of all 64 protonated trimer oligodeoxynucleotides and 16 mixed base tetramer oligodeoxynucleotides [59]. Basic and Yost have reported CID breakdown surfaces for *n*-alkylbenzene molecular ions [60], whereas Tajima et al. have investigated the loss of CO from ionized *o*-, *m*-, and *p*-anisoyl fluoride, $CH_3OC_6H_4COF^{+\cdot}$ as a function of collision energy [61].

Multistep CID has been used for determination of the amino-acid sequence and gas-phase ion chemistry of valinomycin coordinated with lithium [62], and for isotope-specific MS^3 experiments with a protonated glutathione conjugate [63]. Wu has examined temperature and pressure effects in an ion source external to an ion trap using methane and oxygen as reagent gases [64].

4.3. Ion rearrangements

The group of O'Hair has found that the $[M–H–CO_2]^-$ anion from glycyl glycine undergoes rearrangement in the gas phase [65], and has attempted to determine whether N-protonated dehydroamino-2-byturic acid is formed upon loss of side chain water from protonated threonine [66].

4.4. Ion/molecule reactions

Much of the ion-chemistry research reported has been concerned with the reactions of ions, the kinetics of ion/molecule reactions, ion dissociation, and ion structure determination. The ions that have been studied are the phenylium cation with small oxygen- and nitrogen-containing molecules [67], isomeric parent ions from the dissociation of dimethylpyrroles [68], protonated polyamines [69], gas-phase H/D exchange reactions of polyamine complexes [70], perfluorotri-*n*-butylamine fragment ions [71], cluster ions of the form $H^+(H_2SO_4)_m(H_2O)_n$ [72], silane/hydrocarbon mixtures [73], and sodium ion attachment reactions [74,75]. Kinetic studies of gas phase reactions of the ion $C_5H_5Fe^+$ [76] and of the dissociation of a transient intermediate [77] have been carried out. The ion chemistry of carbon suboxide has been studied [78]. A chemometric approach to the evaluation of the parameters affecting the determination of reaction rate constants [79] has been made, and the effective ion temperatures in an ion trap have been estimated [80]. An effect of buffer gas temperature on the distribution of product ions in high mass tandem mass spectrometry has been found [81]. A TOF method has been used to estimate the kinetic energy of N^+ ions throughout the stability diagram [82].

The mass spectrometric investigation of antibiotics, for example, the post-column metal complexation of quinolone antibiotics [83], is commanding some attention as was seen in the above discussion [57] on the use of electrospray ionization (ESI) with the ion trap. Chemical ionization (CI) reactions studied were of oxygenated CI reagents with Vincamine [84], determination of the double bond position in functionalized monoenes using acetonitrile as CI gas [85], and ammonia CI for the structure determination of alkaloids [86]. The effects of functional group interactions on the gas-phase methylation and dissociation of acids and esters have been reported also [87]. Studies on the dissociation of ions have included alkali cationized polysaccharides [88], rearrangement of *n*-butylbenzene [89], the estimation of critical energies for ion dissociation via threshold collisional activation measurements [90], the dissociation of polyether-transition metal ion dimer complexes [91], the effects of heavy gases on CID of peptides [92], and the origin of the product ions observed [93].

Structure elucidation studies include those of radical cations [94], polyketide tetrasubstituted δ-lactones [95], and distinguishing the structural isomers of glutathione conjugates of estrone and estradiol [96], and of conjugated diene epoxides [97]. The sites of reaction of pilocarpine have been reported [98]. Elimination reactions studies are the deamination of protonated amines to yield protonated imines [99], denitration of nitroaromatic compounds by arylnitrile radical cations [100], and the elimination of CF_2 from trifluorocresols [101]. It is encouraging to observe that a library has been established of atmospheric pressure ionization (API) daughter mass spectra based on wideband excitation in an ion trap [102].

Ion chemistry [103,104], ion/ion reactions [105], and charge state effects [106] have been investigated. Ion/molecule reactions in phosphine/germane mixtures [107] have been reported, as has the use of ion/molecule reactions and mass spectrometry $(MS)^n$ for the characterization of conjugated ethylenic epoxides [108]. Reaction studies have been reported for dimethyl ether cations with polycyclic aromatic hydrocarbons (PAHs) [109], reactions in organophosphorus esters [110], and ionic species from acetonitrile with long-chain saturated and unsaturated alcohols [111]. Gas phase ion/molecule reactions have been carried out in a modified ion trap [112], and a Finnigan LCQ ion trap has been modified to permit ion/molecule reactions in two ways: (1) gas phase H/D exchange reactions followed by CID, and (2) CID prior to performing ion/molecule reactions. Ion/molecule complexes have been studied [113].

4.5. Chemical ionization

The temperature and pressure effects of negative CI [64] have been investigated, along with capillary liquid chromatography/atmospheric pressure chemical ionization mass spectrometry (LC/APCI/MS) [114], and comparative electron ionization (EI), CI, and CID product ion mass spectra of iminopyridine derivatives [115]. Negative-ion CI reactions of the hydroxy anion, $[OH]^-$, and the benzoyl anion, $[C_6H_5CO]^-$ [116] have been reported. Mass-selected reagent ion CI and MS^n techniques have been employed for structural analysis [117]. Acetonitrile has been found to be an effective reactant species for positive-ion CI of hydrocarbons [118]. Differentiation of microorganisms based on pyrolysis-ion trap mass spectrometry using CI has been achieved [119], and the effects of applied voltages and reaction times on negative-ion CI have been studied [120]. Analytical pyrolysis of methylated straw has been used to demonstrate in situ oxidative degradation of lignin side chains by two white-rot fungi [121].

5. Applications of electrospray ionization

The most active areas of ion trap research in the past seven years have been applications of ESI ([1], Vol. 2, Chap. 3; Vol. 3, Chaps. 5, 6, and 14), environmental applications ([1], Vol. 3, Chaps. 12 and 13), ion chemistry ([1], Vol. 1, Chaps. 5 and 8; Vol. 3, Chaps. 8–19), and instrument development ([1], Vol. 2, Chaps. 2, 3, and 7; Vol. 3, Chaps. 1, 6, and 11).

The relatively facile combination of ESI with a quadrupole ion trap has permitted many investigations of polar substances over a wide range of interests. Indeed, the investigations are so disparate and the range of investigations is so wide that particular emphases are difficult to discern. Recent researches are concerned chiefly with surveying the field and this activity may continue for the next five years. Nevertheless, the present researches are most informative with respect to both the specific compounds studied and the performance limits of the instruments. We have arranged this survey roughly in terms of method development, kinetic studies, MS^n, natural products, structural studies, gaseous dications, ion dissociation, metal complexation, and oligonucleotides.

The efficacy of the combination of ESI with the quadrupole ion trap is highly dependent on the injection efficiency and trapping of externally generated ions, and Doroshenko and Cotter [14,122–124] have contributed greatly to our understanding of this process. Let us consider first the manipulation of a sample prior to the admission of ions to the ion trap. Barroso and de Jong have developed a method of sheathless preconcentration applied to peptide analysis using capillary electrophoresis/ESI/ion trap [125]. Nanospray ionization [126,127] and microscale ESI for the investigation of peptides, protein digests [128], and bacterial investigations [129] have been reported.

Davis and Lee [130] have demonstrated a method known as “peak parking” which permits increased sample analysis times for components eluting from a liquid chromatograph. This method is particularly useful in that it permits high mass resolution over a narrow mass range, in addition to MS^2 and MS^3 within the ion trap, in the examination of small quantities of material. An optimized hydrophilic interaction chromatography-ESI/MS method has been applied successfully for the analysis of complex natural product mixtures [131]. A solution-based microscale approach for the determination of high-affinity noncovalent complexes (high-affinity ligands to the Src SH2 domain) from mixtures of compounds has been presented. The approach is based on capillary isoelectric focusing-coupled on-line with ESI ion trap mass spectrometry [132]. A microcapillary column switching high-performance liquid chromatography (HPLC)/ESI/MS system has been developed for the direct identification of peptides presented by major histocompatibility complex class 1 molecules [133], whereas high sensitivity peptide sequencing has been achieved with a magnetic sector/ion trap combination [134]. O’Hair and Reid have reported a combined experimental search for, and ab initio study of, stable gas-phase b_1 ions derived from aliphatic amino acids [135]. Derivatization of alkenes and alkynes for ESI/MS has been described [136].

Kinetic studies have been concerned with hydroiodic acid attachment as a chemical probe of gaseous protein ion structure [137] and gas phase H/D exchange kinetics, DI vs. D_2O [138–141]. The kinetic method has been employed in studies of competitive pyridine loss from platinum (II) ethylenediamine complexes [142]. McLuckey and coworkers have investigated ion–ion interactions of protonated pyridine with multiply charged oligonucleotides [143–145].

The propensity of the ion trap for tandem mass spectrometry MS^n has been demonstrated with reference to structural elucidation of erythromycins using MS^5 [146], determination of the anomericity of the glycosidic bond in Zn(II)-diethylenetriamine-disaccharide complexes using MS^n [147], and examination of the ESI/MS^n of poly(propylene oxide) [148]. Saponins have attracted some attention by way of an MS^n study of triterpenoid saponins [149], a method for the rapid identification of saponins in plant extracts by ESI/MS^n and LC/tandem mass spectrometry (MS/MS) [150,151], and their structural characterization by MS^n [152].

Other structural studies include a comparative study of four mass spectrometric methods for the elucidation of structure and fragmentation mechanisms of isomeric T-rich oligodeoxynucleotides [153], isomeric photomodified oligodeoxynucleotides [154], a collisional activation study of the gas-phase stability of double-stranded oligonucleotides and their noncovalent complexes [155], and the characterization of product ions of protonated oligonucleotides [156]. Structural studies have been carried out on ceramides that function as structural lipids in membranes, in the epidermis, hair, and nails of humans and animals [157]; ceramides function also as signal transducers.

Van Berkel and Zhou have reported on the observation of gas-phase molecular dications formed via the controlled-current electrolytic process inherent to ESI [158] and from neutral organics in solution via chemical electron-transfer reactions [159]. The dissociation, structure, and incidence of magic numbers in doubly charged cluster ions of the form $[(NaCl)_m(Na)_2]^{2+}$ [160] and of multiply charged cluster ions of alkali chlorides [161] have been reported. Purves and Li [162] and Lubman and coworkers [163–167] have reported on ion analysis for which an ion trap was used in combination with TOF.

The dissociation of a wide range of ion species has been reported. These reports include the collisional activation of deprotonated deoxymononucleoside and deoxydinucleoside monophosphates [168], decompositions of odd- and even-electron anions derived from deoxy-polyadenylates [169], the fragmentation of selected coumarin derivatives [170], MS/MS of model peptides modified with *trans*-2-hexenal [171], fragmentation of phosphopeptides [172], the role of the sulfhydryl group on the gas-phase fragmentations reactions of protonated cysteine and cysteine-containing peptides [173], characterization of cysteine residues and disulfide bonds in proteins [174], dissociation of doubly charged transition metal/polyoether/pyridyl ligand complexes [175], fragmentation pathways of selectively labeled uropranolol [176], and of propranolol-elated β-fluorinated amines [177], fragmentation mechanism of poly(3-hydroxybutanoic acid) end groups [178], and fragmentations of cholesterol acetate oxidation products [179].

Several contributions on metal complexation [180,181] have appeared including an evaluation of metal complexation as an alternative to protonation for ESI of pharmaceutical compounds [182], the synthesis and analysis of penta-coordinated diastereomeric Ni-N-glycoside complexes [183], stereochemical differentiation of mannose, glucose, galactose, and talose using zinc(III) diethylenetriamine [184], and a study of a tetrahedral supramolecular Ti_4L_4 cluster [185].

The structural characterization of penicillins [186], the determination and characterization of fumonisin mycotoxins [187], and the characterization of phenolic antioxidants from Maté (Ilex Paraguayensis)[188] have been reported.

Oligonucleotides have received some attention; for example, the identification and positional mapping of aflatoxin B_1-guanine adducts in oligonucleotides [189] has been reported along with small multiply charged oligonucleotides with a modified base [190], charge reduction of oligonucleotide anions [191], and charge state effects in the decompositions of single-nucleobase oligonucleotide polyanions [192].

The antitumour Catharanthus alkaloids have been examined [193], a comparison of gas chromatography (GC)/MS and HPLC/MS for the determination of polyhydroxyalkaloids in bluebells has been carried out [194], the anthocyanins of Catharanthus roseus in vivo and in vitro have been characterized [195], and mixtures of phosphatidyldiacylglycerols from Lactobacillus have been analyzed [196]. Other studies that have been reported are the characterization of supramolecular complexes by high mass resolution [197], determination of calcium binding sites in gas-phase small peptides [198], peptide sequencing [199], linkage position determination in cobalt coordinated pentasaccharides [200], analysis of *N*-acetylated hexosamine monosaccharides by ferrocenyl boronation [201], cyclodextrin-catalyzed oxidation of glutathione [202] and of cyclodextrin complexes with amino acids in eluates of gel permeation chromatography [203], charge derivatization of peptides to simplify their sequencing [204], determination of trans-4-hydroxy-2-nonenal at cellular levels[205], and collisional activation of both multiply protonated polypeptides where the disulfide linkage has been reduced [206] and of singly charged peptide ions [207].

Strategies for locating disulfide bonds in a monoclonal antibody [208] and the sequencing of anti-thyroxine monoclonal antibody Fab fragment [209] have been reported. Methods have been given for the determination of 5-methyl-2′-deoxycytidine in urine [210], urinary nucleosides [211], and of phenothiazines in whole blood [212]. Mass spectra of bile acids and other surfactants have been interpreted [213]. Investigations of poly [(R,S)-3-hydroxybutanoic acid] telechelics [214,215], the identification of self-assembly products from *N*-phosphoamino acids [216], the characterization of (methoxymethyl)melamine resins [217], application of directly coupled HPLC/NMR/MS to the identification and confirmation of quercitin glycosides and phloretin glycosides in apple peel [218], and methods for distinguishing N-Oxides from hydroxylated compounds [219] have been reported together with mass spectra of some new 3,4-dihydro-2[H]-pyridones [220].

6. Proteins and peptides

It should be noted that some publications concerning proteins have been discussed in the sections above. Wabnitz and coworkers have carried out a sequence determination of citropin peptides from the skin glands of the Australian Blue Mountains tree frog, litoria citropa [221–223]. The complete and rapid peptide and glycopeptide mapping of mouse monoclonal antibody has been reported [224], along with a study of brain distribution and exploratory pharmokinetics of a neuropeptide FF antagonist [225]. A microscale ESI interface incorporating a monolithic poly(styrene-divinylbenzene) support for on-line analysis of peptides and proteins has been described [226]; the performance of the monolith-filled ESI needle compared favorably or superior to needles filled with commercial particulate supports.

Yates et al. [227] have reported a method for comparing CID mass spectra of peptides. The method employs a cross-correlation analysis of a CID spectrum to a reference spectrum and normalizes the cross-correlation score to the autocorrelation of the CID spectra. The query spectrum is compared using both mass information and fragmentation patterns. Fragmentation patterns are compared to each other using a correlation function. From comparisons within a set of 2180 product ion mass spectra obtained from both TSQ and ion trap (LCQ) instruments, the scores obtained in the cross comparison of TSQ and LCQ product ion mass spectra were found to be only slightly lower than those obtained from cross comparisons of data from the same instrument.

Capillary isoelectric focusing MS has been used for stepwise mobilization of focused proteins [228], a method for structure determination of cyclodepsipeptides using sodium-directed selective cleavage of lactones has been proposed [229], de novo peptide sequencing with ^{18}O labeling has been carried out [230], and on-line derivatization of peptides for improved sequence analysis has been demonstrated [231].

Glish and coworkers have reported on an investigation of boundary-activated dissociation of peptide ions [232] and on C-terminal peptide sequencing using MS^n [233]. It is noteworthy that ≤ 8 stages of MS/MS were performed with both the quadrupole ion trap and ion cyclotron resonance mass spectrometers; whereas the same dissociation pathways were observed with each instrument, the branching ratios of competing pathways differed on occasion. Gatlin et al. have reported on the automated identification of amino acid sequence variations in proteins [234], and Bahr et al. have proposed a method for the differentiation of lysine/glutamine in peptide sequence analysis [235]. The analysis of noncovalent protein complexes up to 290 kDa [236], the electrospray-assisted modification of proteins as a radical probe of protein structure [237], and the identification of cysteine residues by isotopic labeling [238] have been reported.

The structural characterization of oligosaccharides has attracted the attention of several groups. Visuex et al. [239] reported that sequential tandem MS permitted the straightforward structural characterization of unknown carbohydrate moieties by comparison of their CID spectra with those of a set of reference compounds. Because the CID patterns are not dependent on the number of prior tandem mass spectrometric steps, structures can be assigned unambiguously by matching the spectra. Other structural studies are the characterization of oligosaccharide composition and structure [240], a probe for the versatile analysis and characterization of N-linked oligosaccharides [241], combined partial acid hydrolysis and ESI-MS [242], structural analysis of complex N-linked glycoprotein oligosaccharides [243], the facile detection of mono-, di-, and oligosaccharides by ESI-orthogonal TOF using 3-aminophenylboronic acid derivatization [244], and the direct structural analysis of 2-aminoacridone derivatized oligosaccharides [245]. Sheeley and Reinhold reported on the structural characterization of carbohydrate sequence, linkage, and branching in neutral oligosaccharides and N-linked glycans [246].

7. Drugs and drug metabolites

The determination of drugs and drug metabolites [247–250] is becoming increasingly important and the ion trap finds application in this area. The identification of synaptic metabolites of dynorphin A (1–8) by ESI/MS/MS [251], and the analysis of nitro musk compounds and their amino metabolites in liquid sewage sludges using both NMR and mass spectrometry [252] have been reported. A rapid and efficient approach to metabolite identification using NMR spectroscopy and LC/MS and LC/NMR/MS/MS has been proposed [253], as has the identification of drug metabolites in biological matrices by intelligent automated LC/MS/MS [254]. Monensin is a polyether monocarboxylic acid ionophore and refers to a group of factors comprising an antibiotic complex first isolated from Streptomyces cinnamonensis, and the in-source CID of monensin factors and related metabolites using ESI/MS has been investigated [255]. Taylor and coworkers have identified in vitro metabolites of Bupropion using ion trap mass spectrometry [256]. The high sensitivity of mass spectrometry permits detection and identification of minor impurities in drugs [257] and of unknown but related substances in commercial erythromycin samples [258]. The analysis of a commercial preparation of erythromycin estolates [259], the identification of tetracycline antibiotics [260], and ion trap determinations of anabolic steroids by GC/MS [261] and by negative ion CI/MS/MS [262] have been reported. Ion trap mass spectrometry has been used for the characterization of N-Methyl-1-(3,4-methylene-dioxyphenyl)-2-butanamine and N-Ethyl-3,4-methylenedioxyamphetamine, two widely distributed street drugs [263].

The ion trap has found application also in veterinary analyses. Avermectins represent a family of natural products with antiparasitic effectiveness and are used currently in the livestock industry. The structures of two new derivatives of ivermectin B_{1a} were determined by multistage tandem mass spectrometry [264]. Heller has reported that LC/MS is a timely response in regulatory analyses with specific reference to the identification of pentobarbitol in dog food [265] and the confirmation of seven β-lactam antibiotics in bovine milk [266]. GC/CI/MS has been used for the analysis of β-agonists [267], and HPLC combined with MS^n has been used in the determination of β-agonists in bovine urines [268].

8. GC/MS and GC/MS/MS

There is no doubt that the ion trap as a mass detector and as a tandem mass spectrometer, when combined with a capillary gas chromatograph, has brought about significant advances in the field of analytical chemistry. The announced advent by Varian Associates of a GC/MS instrument with enhanced mass resolution (full-width at half height of $\approx$0.1Th) will do much to revitalize a technique that has found wide application.

Bridleman and coworkers have employed enantioselective gas chromatography for the determination of methylsulfonyl polychlorinated biphenyls (PCBs) in arctic marine mammals [269]. Enantioselective analysis of adipose tissue of arctic ringed seal (Phoca hispida) and of polar bear (Ursus maritimus) revealed a strong dominance of certain enantiomers. For example, the enantiomer ratio of 3-CH_3SO_2-chlorinated biphenyl 149 was 0.32 and $<$0.1 in ringed seal blubber and polar bear fat, respectively. These low enantiomeric ratios are indicative of enantiomeric formation, metabolism, transport across cell membranes, or a combination of all three on both species. Quantitative determination of endocrine-disrupting PCBs and organochlorinated pesticides in human serum using GC/MS/MS with electron-capture detection has been reported also [270]. The determination of derivatized clenbuterol by use of GC/MS/MS using negative ion chemical ionization [271], the analysis of thermally labile pesticides by GC/MS and GC/MS/MS with a temperature-programmed injector [272], and the determination by GC/MS of the chloropropanols, 3-chloro-1,2-propandiol and 2-chloro-1,3-propandiol, in hydrolyzed vegetable proteins and seasonings [273] have been reported.

Pyrolysis/GC/MS has been employed for the determination of the chemical composition of wood casks for wine aging [274], the composition of sound and degraded wine bottle cork [275], essential oil and phenolic compounds in the Monks' pepper Vitex agnus-castus [276], for the characterization of the alkalilignin obtained from wheat straw soda-cooking effluents after treatment with Strepomyces strains [277], arsenic inorganic compounds [278], and for the monitoring of fugal-biotreated distillery wastewater using tramates sp. I-62 (CECT 20197) [279].

The determination of gestrinone in human urine by HPLC and GC/MS [280] and the quantitative analysis of α-tocopherol and deuterated isotopomers at subfemtomole levels in plasma using GC/MS/MS [281] have been reported. The positions of double bonds in di-unsaturated esters and a di-substituted γ-lactone have been determined by GC/MS of dimethyl disulfide derivatives [21], the behavior of catechin and ellagic acid has been investigated [282], and quantitative GC/MS/MS using resonant and nonresonant collisions in ion traps has been described [283]. The application of isotope dilution to ion trap GC/MS has been discussed [284], and isotope dilution was employed for quantification of 11-nor-delta-9-tetrahydrocannabinol-9-carboxylic acid [285].

9. Environmental applications

Numerous publications pertaining to the environment have appeared in the past six years or so. Clearly, great use is being made of the ion trap for the determination of many compounds of environmental interest. The publications are concerned with air analysis using a microwave plasma ionization source [286–288], structure elucidation of 2,4-dinitrophenylhydrazone derivatives of carbonyl compounds in ambient air by HPLC/MS and multiple MS/MS using negative ion APCI [289] and the real-time determination of aromatics in automobile exhaust by single-photon ionization ion trap mass spectrometry [290]. Aerosol particles have been investigated by real-time chemical analysis [291], laser desorption/ionization coupled to MS/MS for real-time monitoring of paraquat [292] and tributyl phosphate [293] on the surface of environmental particles, and by API/CI/MS for on-line characterization of organic aerosols formed from biogenic precursors [294].

The determination of semi-volatile organic compounds [295] and the evaluation of ion trap mass spectrometry for Environmental Protection Agency Method 8270 (semi-volatiles in solid wastes) [296] have been carried out. Volatile organic compounds in flue gas have been monitored using direct sampling ion trap mass spectrometry [297]. Determinations of polycyclic aromatic hydrocarbons [298,299] have been made and a new ion trap method that involves Rydberg electron capture mass spectrometry [300] has been described. PAHs in bitumen fumes during road paving have been determined by solid-phase extraction/GC/MS [301]. A GC/MS/MS method for the determination of 2,3,7,8, dioxins and furans at the sub-parts per trillion levels in high-fat foods has been described [302] and a comparison has been made [303] of three mass spectrometric methods (including the quadrupole ion trap) for the determination of dioxins/furans.

The examination of soils and explosives has attracted some attention. Ion-trap SIMS analyses of pinacolyl methylphosphonic acid [304] and of the nitrogen-based blister agents bis(2-chloroethyl)methylamine (HN-2) and tris(2-chloroethyl)amine (HN-3) and their hydrolysis products [305] on soil have been reported, along with elemental and organometallic analyses of soil using glow discharge MS and GC/MS [306]. High explosives vapor has been detected by glow discharge/ion trap mass spectrometry [307], and trace analysis of explosives in seawater has been affected using solid-phase microextraction and GC/MS [308]. An ESI/MS/MS CID study of explosives in an ion trap mass spectrometer [309] has been carried out.

Presently, there are concerns with pesticides in food, for example, chlormequat in pear extracts [310], carbamate and phenylurea pesticides studied by ion trap [56], herbicides, and farm run off. A comparison of MS/MS techniques using ion trap and hybrid mass spectrometers for studies of the degradation of herbicide by biofilm has been made [311].

The application of ion trap mass spectrometry to drinking water and river water has increased. In the opinion of the authors, there will be a significant growth in the application of mass spectrometry to natural waters. There is growing concern with the levels of pharmaceutical and neutraceutical products (and their metabolites), surfactants and their decomposition products, etc. in bodies of water from which drinking water supplies are drawn. For example, the determination of Bisphenol A and nonyl phenol in river water [312], the determination of persistent acid metabolites of nonylphenol ethoxylate surfactants [313], the identification and characterization of Fenton oxidation products of surfactants by ESI/MS [314], and the characterization (by LC/NMR and LC/MS) of dyes and other pollutants in the effluent of a textile company [315] have been reported recently. Solid-phase microextraction combined with quadrupole ion trap GC/MS has been employed for the determination of cannabonoids in water and in human saliva [316]. The chlorination of drinking water brings other problems necessitating the determination of organohalogen compounds [317,318] and determination of the main chlortoluron by-products during water disinfection by chlorine [319]. The determination of oxygenated polar organic compounds poses a particular problem due to lack of suitable methodologies; derivatization has been explored as a means to obviate this problem [320]. Ion trap mass spectrometry has been used for kinetic studies of stable isotope labeled vitamin A at low enrichment levels [321].

The characterization of a nerve agent O-ethyl S-2-diisopropylaminoethyl methyl phosphonothiolate (VX) on the surfaces of concrete samples by ion trap secondary ion mass spectrometry [322] together with a GC/MS study of organic films on stone monuments [323] have been reported. A sensitivity enhancement for the detection of chlorophylls has been found using atmospheric pressure ionization [324]. Two publications have appeared dealing with mass spectrometric methods for the identification of chemical markers of bacteria [325,326].

10. Lasers

Lasers have been used for a variety of purposes in ion trap experiments. For example, a laser was used as a probe for the determination of ion frequencies in a quadrupole ion trap where ions were first excited by a fast dc pulse [327], to excite N_2^+ for experimental studies of ion rotational relaxation processes in a 22-pole ion trap [328], and to dissociate selectively nitro groups in nitrocellulose [329]. Matrix-assisted laser desorption/ionization has been used both for the direct analysis of color couplers adsorbed on solid substrates [330] and in combination with an advanced stored waveform inverse Fourier transform technique [331].

In a study of laser ablation ion trap mass spectrometry, Gill and coworkers investigated storage field suppression and its effect upon analytical performance [332], and laser desorption has been used with membrane introduction mass spectrometry (MIMS) [333]. An arrangement of two lasers has been employed for organic surface analysis [334,335]. The Yost group has reported two studies of infrared multiphoton dissociation (IRMPD) of ions in an ion trap: first, they examined the effect of pulsed helium introduction on collisional quenching during IRMPD of ions obtained by ESI [336] and, second, they effected IRMPD in a quadrupole ion trap using a multipass optical arrangement [337].

11. Instrument development

Many interesting publications in the area of ion trap development have appeared in the past six years. These developments encompass ion traps of various shapes and sizes, ion traps modified for specific experiments both in the laboratory and in space, new methods of ion isolation, ion trapping, and ion detection.

Cooks' group has focused on the development and characterization of cylindrical ion traps [338]; such ion traps offer many advantages. A miniature cylindrical ion trap was constructed having $r_o=2.5$ mm and $z_o=2.88$ mm such that the volume was $\approx$1/64 of that of a commercial ion trap. Ion motion within the ion trap was simulated and the trap was used with a MIMS system for on-line reaction monitoring. Based on a 50% valley definition, a mass resolution, $m/\Delta m$, of $\approx$100 was observed [339]. Mass-selective operation [340] of and Fourier transform detection [341] in a cylindrical ion trap have been demonstrated. Recently, Cooks' group reported on a parallel miniature cylindrical ion trap array [342]. The same group had reported earlier on broadband nondestructive ion detection in a hyperbolic quadrupole ion trap [33,343]. Micro ion traps have been reported also [344,345]. Henry has commented on “The incredible shrinking mass spectrometers” [346].

Creaser et al. have described their application of a combined tandem ion trap/ion mobility spectrometer [347], and an ion trap/ion mobility/TOF mass spectrometer has been used to measure collision cross sections for 660 peptide ions [348]. Ion dynamics in a novel linear combined trap have been reported [349]. Novel ion trap devices continue to attract interest [350–352], and a dual quadrupole ion trap mass spectrometer has been evaluated [353].

Voyksner and Lee [354] have investigated the use of an octupole ion guide for ion storage and high-pass mass filtering in order to improve the quantitative performance of ESI with an ion trap, and Wu and Lin have determined the sensitivity of an external source to an ion trap [355]. Traldi and coworkers have employed wide band isolation in an ion trap for the analysis of polychlorobiphenyls [356]. Hyperboloid mass spectrometers have been prepared for space exploration [357], and a Paul trap has been adapted for study of the reaction of multiply charged cations with singly charged anions [105]. The confinement of ions in a quadrupole ion trap under the influence of a periodic pulsed potential [358] has been reported. Barinaga's group has observed enhanced abundance sensitivity for the resonant ion excitation of atomic ions generated in an inductively coupled plasma combined with an ion trap [359], whereas Duckworth and Barshick wondered in print as to what ion traps held for elemental analysis [360].

An ion trap has been employed as a secondary ion mass spectrometer [322,361], and a hybrid ion trap has been characterized [362]. Laser desorption has been optimized [363]. The uses of overtone and fundamental resonances for mass range extension have been compared [364]. The application of field-modulated selective ion storage that reduces ion loss [365], external customized waveforms [366], vector summed sine wave tailored waveforms for dipolar excitation of ions [367], automated strategies for obtaining standardized collisionally induced dissociation mass spectra [368], and a new ion ejection method [369] have been reported. Pulsed buffer gas introduction has been explored further [370,371], and a comparison of equilibrium ion density and trapping force in Penning, Paul, and combined ion traps [372] has been reported. Features of unstable ion trajectories have been explored [373,374]. Two further publications concern the rf and the effect of change of frequency on ion storage: the first [375] is an investigation of the effects of the rf potential on the trapping of externally generated ions in an ion trap whereas the second [376] concerns a frequency scan for the analysis of high mass ions (60–160 kDa) generated by matrix-assisted laser desorption/ionization in an ion trap.

12. Time of flight

The combination of a quadrupole ion trap with either a linear or a reflectron TOF mass spectrometer has proved to be a popular and productive research tool. Wilhelm et al. [377] carried out ion trajectory calculations for such a combination and reported on ion cloud motions as a result of collisions with jet-cooled molecules [378]. Qian's group reported on the development of a suitable data system [379] and procedures for MS/MS [380] with an ion trap/TOF combination. This group reported also on capillary LC/ion trap/TOF for structural confirmation of three recombinant protein isoforms [381], CID of multiply charged peptides [382], rapid mutation-site analysis of hemoglobin variants [383], detection of oligonucleotides [384], separation of tryptic digests [385], simulation of external ion injection [386], and the use of stored waveform inverse Fourier transform (SWIFT) technology [387].

Li and coworkers investigated the quantitative capabilities of an ESI/ion trap/TOF mass spectrometer [388] and characterized such an instrument for phenylthiohydantoin-amino acid analysis [389]. A cylindrical ion trap/TOF instrument has been used for the determination of dissociation rates of energy-selected benzene cations [390]. Quadrupole ion trap/TOF instruments have been employed with matrix-assisted laser desorption [391], whereas an ion trap/ion mobility/TOF instrument has been used for rapid and sensitive analysis of biomolecular mixtures [392].

13. Microfabricated devices

Microfabricated devices are being developed rapidly at this time so as to permit the analysis of minute quantities of material and to reduce analysis time. Figeys et al. have developed an integrated microfluidics MS/MS system for automated protein analysis [393]. This system has been used for the sequential automated analysis of protein digest; the system is compatible with the automated analysis of proteins separated by two-dimensional gel electrophoresis and detection at the low femtomole/μL level was demonstrated. The group of R.D. Smith has developed a microfabricated dialysis device for sample cleanup in ESI/MS [394] and an integrated microfabricated device for dual microdialysis of complex biological samples [395]. A micromachined chip-based ESI source [396], a microdevice with integrated liquid junction for facile peptide and protein analysis [397], and a nanoflow solvent gradient delivery from a microfabricated device for protein identification [393] have been developed.

14. Membrane introduction mass spectrometry

MIMS has been used in several applications; for example, Colorado et al. have used a fast response MIMS method for the determination of atmospheric isoprene using ion trap mass spectrometry; MS/MS of an adduct ion of isoprene with vinyl methyl ether and loss of methanol yielded isoprene concentrations of between 0.5 and 10 ppb [398]. MIMS has been used also for on-line monitoring of fermentation broths [399], organometallic analysis [400], direct sampling and analysis of volatile organic compounds in air [401], and for trace analysis of PAHs in water [402]. Thin pervaporation membranes for improved performance in on-line flow injection analysis have been investigated [403].

15. Capillary electrochromatography

An ion trap has been combined with capillary zone electrophoresis (CZE or CE) separation for the CZE/MS/MS (QIT) structural analysis of chromophore labeled disaccharides [404] and for characterization of β-agonists [405]. A comparison of the sensitivity of nanospray and microspray ionization techniques in CE/MS has been carried out [406]. The use of a mixed mode packing and voltage tuning for peptide mixture separation in pressurized CE with an ion trap/TOF mass spectrometer detector has been reported [407], along with an on-line preconcentrator for the CE/MS of peptides [408]. CE/MS has been applied to the analysis of bile acids and their conjugates [409], and to the characterization of peptides at the attomole level [410]. A CE has been interfaced in a poly(N-vinylpyrrolidone) (PVP) matrix to an ion trap for the analysis of oligonucleotides [411].

16. Method development

Todd and coworkers have reported on the automation of data acquisition and processing involving dynamically programmed scanning for an ion trap mass spectrometer [412], Drexler and Tiller have developed an “intelligent” fraction collection procedure to improve structural characterization by MS [413], Leary and coworkers have developed a saccharide topology analysis tool to be used in combination with MS/MS [414]. Tiller and Cunniff have discussed the advantages of increased sensitivity and mixture analysis with full-scan MS/MS for quantitative analysis with an ion trap mass spectrometer [415]. Rydberg electron-capture mass spectrometry has been applied to 1,2,3,4 tetrachlorodibenzo-p-dioxin [416]. The last publication is from the Cooks group and it concerns the characteristics of a broadband Fourier transform ion trap mass spectrometer [18].

17. Miscellaneous contributions

In categorizing a contribution as “miscellaneous” the authors of this review do not intend offense; rather, these contributions represent minor failures on the part of the authors in that we have failed in each case to discern the connectivity of these contributions to the categories chosen for this review.

There have been two ion trap publications concerning biomarkers. Karr and Walters have carried out sterane analysis [417] and the group of Smith has described the characterization of microorganisms and biomarker development from global ESI-MS/MS analyses of cell lysates [418]. Roussis et al. have used an ion trap for the investigation of low-energy ionization of hydrocarbons [419].

Brodbelt and coworkers have reported on solvent displacement in transition metal complexes [420] and ligand displacement reactions of dimer and trimer pyridyl ligand/transition metal complexes [421]. Leary and coworkers have investigated the influence of coordination number and ligand size on the dissociation mechanisms of transition metal-monosaccharide complexes [422]. Traldi and coworkers have used the ion trap as an ancillary tool in metallo-organic chemical vapor deposition studies [423] where as Tabet and coworkers have found stereochemical effects can be enhanced by using selective "self-ionization" under EI conditions in a quadrupole ion trap [424]. Groenewold et al. have characterized copper chloride cluster ions formed in secondary ion mass spectrometry [425], and Lifshitz and coworkers have presented new evidence in favor of a high (10 eV) C_2 binding energy in C_{60} [426]. Dahl and Appelhans have proposed a method for sample charge compensation using self-stabilizing ion optics [427].

18. Conclusions

The demonstrated sensitivity of the quadrupole ion trap and variations thereof, its ability to accumulate ions, its compatibility with a variety of ionization methods, its mass range, and its resolution have been used extensively in the period covered by this survey. The number of citations and the variety of research presented therein confirms our confidence in this extraordinary instrument for the pursuit of mass spectrometry for the benefit of mankind in the twenty-first century.

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