

SOME APPLICATIONS OF MASS SPECTROMETRY IN BIOCHEMISTRY A THESIS SUBMITTED TO THE UNIVERSITY OF POONA FOR THE DEGREE OF DOCTOR OF PHILOSOPHY (IN BIOCHEMISTRY)



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in the first part of the thesis the major fragmentation modes operating in the permethylated fructose containing oligosaccherides have been descried with the aid of the available information in literature and metastable defocussing data it is possible to differentiste the various type of linking for the 1--->2 aldohexosyl fructose the intense peaks at m/e 205,m/e 219 and m/e 351 and the relatively less intense peaks at m/e 179 and m/e 409 appear to be characteristic. for the 1--->3,1--->4,1--->5 and 1--->6 linked aldohexosyl fructoses the reverse holds good the intesity ratios of m/e 279/m/e 249,m/e409 m/e 279 and m/e 279/m/e 219 are useful to differentiste between the various linkages in latter group of compounds it has been shown to be possible to locate the terminal ans nonterminal fructofuranose unit connected Via C<sub>2</sub> to another sugar moiety the relatively intense peaks at m/e 205 and m/e 219 observed in the spectra of raffinose and stachyose appear to be due to terminal fructofuranose unit connected Via C2.in melezitose where such a unit is in the middle of the chain the characteristic peaks are observed at m/e 391 and m/e359. it is not possible to differentiate between furanose and puranose forms in these compounds from the ratios of intensities of m/e 101 and m/e 88 praks the relative ease

of cleavage of the different bonds in turanose alditol permethyl ether has been determined with the aid of deuterium lablling studies

the important fragementation modes operating in some peracetylated aldosyl disaccharides have been established with the aid of metastable defocussing technique in part II. the peaks at m/e 317,m/e257,m/e243,m/e215,m/e197 and m/e 155 are helpful to differentiate between the various types of linkages these peaks are significant in the 1--->6 linked conpounds they are relatively insignificant in 1--->1 linked compound. for 1--->2,1--->3 and 1--->4 bonded compounds peaks at m/e317 and m/e243 are significant the ratios of intensities of the peaks at m/e331/m/e 317, m/e317/m/e245 and m/e317/m/e 229 are useful to differentiate between 1--->2,1--->3 and 1--->4 linkages. the ion kinetic energy spectra of these compounds have been examined characteristic differences are observed for 1--->1 and 1--->6 linked compounds . compounds with 1--->2,1--->3 and 1--->4 linkages gave more or less identical LKE spectra. it is also found to be possible to differentiate the 1--->2 linkage from the 1--->3 and 1--->4 linkage with the help of the ratios of some IKE peak intensities

part III deals with the application of mass

spectrometry as a quantitative analytical tool in pesticide residue analysis a method was standardised for the determination of malathion residues on wheat the method was used for determining the malathion residue in some wheat sample collected from the market the residues were found to very from 0.1 to 0.4 ppm which is far below the tolerance level recommended by FAO and WHO a general procedure was developed for the estimation of lindane and carbaryl residues in straw, rice and bran using spiked samples. sampleof rice bran ans straw which have received three applications of sevidol were analysed for lindane and carbaryl residues. the residue level of lindane and carbaryl in all the samples were well below the tolerence levels the last part of the thesis is on the syntesis assay and evaluation of a slow-releasign herbicide slowreleasing herbicides were prepared by chemcially binding 2,4-D with bagasse and saw-dust the saw dust product was prepared for comparison the conditions for the maximum incorporation of 2,4-D in the products were determined the 2,4-D content of the products was estimated by PH titration metod a mass spectrometric method was standardised for the estimation of 2,4-D in the soil it was used to determine the rate of release of 2,4-D form bagasse and saw dust prodcuts under field conditions appilcation of nitrogenous fertilizers to the soil increased

the rate of 2,4-D release satisfactory products were prepared for field trials containing sufficient quantities of free and combined 2,4-D the field trials were conducted on sugarcane crop the weed counts, weed weight and germination count of sugarcane were recorded the perliminary results are encouraging. more field trials are required for complete evaluation.