ANALYSIS OF THE STRUCTURE OF CARBOHYDRATES WITH USE OF THE REGULARIZED DECONVOLUTION METHOD OF VIBRATIONAL SPECTRA

N.A. Nikonenko, D.K. Buslov, N.I. Sushko, R.G. Zhbankov

B.I. Stepanov Institute of Physics, Academy of Sciences of Belarus, Skaryna Ave. 68, 220072, Minsk, Belarus; e-mail: nataly@dragon.bas-net.by

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In analyzing the composition and structure of complex chemical compounds vibrational (IR and Raman) spectroscopy have been successfully used. However, analytical potentialities of the method of vibrational spectroscopy are restrained, to a great extent, by the low resolution of bands in the spectra of complex substances. The effective way for solving this problem is the use of corresponding methods for mathematical treatment of vibrational spectra.

The infrared spectra of polysaccharides (amylose and cellulose) as well as of their constituent monosaccharides (α- and β- D-glucose) and the results of their deconvolution are presented. The number of bands separated upon deconvolution in the 1200-920 cm⁻¹ spectral ranges exceeds the number of visualized absorption maxima in the room temperature spectra by a factor of more than two. It is shown that the results of deconvolution of the IR spectra of monosaccharides are in good agreement with the data of normal coordinate analysis of these compounds in the crystalline state. The manifestation in IR spectra of the investigated monosaccharides of factor group splitting of a number of nondegenerate fundamental vibrational modes of molecules in the crystalline state has been found. It has been shown that the glycosidic linkage formation in polysaccharides with 1→4 glycosidic linkage is characterized by the appearance of new absorption bands in the 1175-1140 cm⁻¹ spectral range, as compared to the infrared spectra of their constituent monomers. In the 1000-970 cm⁻¹ range, in the deconvolved IR spectrum of cellulose, absorption bands, which are not observed in the monomer spectrum, are separated. The number of bands in the above region remains unchanged for amylose, as compared to the spectrum of monomer α-D-glucose.

The results obtained can be used in investigating of the vibrational spectra of carbohydrates with the aim of solving various practically important problems of molecular spectral analysis.

CHEMOMETRIC METHODS IN ANALYSIS WITH MULTICHANNEL DETECTOR SPECTROMETRY

Yu.P. Turov

Institute of Petroleum Chemistry, Russian Academy of Sciences, 3, Akademichesky Ave., 634021, Tomsk, Russia, e-mail: tur@ipc.tsc.ru

Key words: chemometrics, data sets treatment, hyphenated analytical techniques, GC/MS, petroleum and environmental analysis

Chemometrics is the discipline concerned with the application of statistical and mathematical methods, as well as those methods based on mathematical logic, to chemistry.
The basic mathematical model of analytical chemistry conforms the experimental data sets – the multichannel \{NxM\} detector output signals – to \{LxM\} analyte contents and \{NxL\} their characteristics (spectra).

The modern hyphenated analytical instruments with multichannel detector - such as GC/MS, GC/FT-IR, MS/MS, 2D NMR, LC/DAD - can generate a multivariate matrix of data per sample. These data sets can viewed equivalently, as multivariate analytical signals, modulated by analyte concentrations or (and) instrument environment.

Most real samples for chemical analysis contain a huge mixture of compounds, for example, petroleum, fuels, food, environmental and forensic samples. The spectroscopy and chromatography of mixtures is an excellent and very widespread application of chemometrics, requiring techniques that are specifically aimed at and developed for the chemist.

The basic chemometric algorithm and iterative transformation procedure for multivariate experimental data sets is proposed and discussed based on working concentration simplex hypothesis – the sets of the experimental data points are situated within the (L-1)-dimensional simplex, constructed in the N-dimensional (N\gg L,M) initial nominal coordinate basis. Namely, the data points are located inside and on the surface of the space region limited by (L-1)-measured simplex with a unit height. Simplex apexes correspond to pure components, edges – to binary mixtures, sides – to triple mixtures \textit{etc}. Reconstructing a concentration simplex by means of truncated and censored sampling of mixture mass spectra, one can obtain (calculate) mass spectra for pure components and their concentrations.

Several special algorithms and procedures have been treated to solve this task, the solvability was analysed and their efficiency was demonstrated while processing the sets of MS and GC/MS data.

The approach potentialities and efficiency are demonstrated for GC/MS and complex data sets treatment in organic, petroleum and environmental analytical chemistry.

**SOLID-LIQUID EXTRACTION OF CADMIUM WITH N$_2$N-DIBUTYL-N'-BENZOYLTHIOUREA**

Ayla GÜNGÖR$^a$, Melek MERDİVAN$^b$

$^a$Balıkesir University, Environment Pollution Research Center, Balıkesir, 10100 Turkey

$^b$Dicle University, Science and Arts Faculty, Chemistry Department, Balıkesir, 10100 Turkey

**Key words**: solid phase extraction, N,N-dibutyl-N'-bezoylthiourea (DBBT), paraffin, cadmium, AAS.

The extraction behaviour of cadmium in nitric acid and hydrochloric acid solutions has been studied with N,N-dibutyl-N'-bezoylthiourea (DBBT) in paraffin, naphthalene.

In the range of pH 0-7, the complex was quantitatively extracted into both of them. For quantitative analysis by flame atomic absorption spectrophotometry, the organic phase was back-extracted with 1,25 M HNO$_3$. Ligand volume, pH range, ratio of aqueous volume to organic phase, as optimum conditions for determination were examined. The interferences of various ions were studied. The method has been applied to the determination of cadmium in synthetic samples and real samples and real samples such as mushrooms and wastewater.

**References**: