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Gas chromatographic and gas chromatographic-mass spectrometric analysis of high-purity monoisotopic hydrides of silicon and germanium

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At present time, there is a great interest to simple substances, including isotopically enriched ones, with the ultimately low content of impurities. A suitable method for production and ultra purification of the initial substances is their use in the form of volatile hydrides. Gas chromatography-mass spectrometry is the most promising method of analysis of high-purity substances which make it possible to reliably detect the impurities with high sensitivity. It is for the first time that the method of gas chromatography-mass spectrometry was used to determine the impurity composition monoisotopic silanes $^{28}SiH_4$, $^{29}SiH_4$, $^{30}SiH_4$ and germanes $^{72}GeH_4$, $^{74}GeH_4$, $^{76}GeH_4$. Introducing of gaseous samples into gas chromatograph was carried out by automatic two-position valve "Valco EH2C6WEZPH-CER5", connected with the developed sampling vacuum system. 56 impurity components have been determined including the permanent gases, arsine, phosphine, the homologs of silane, disiloxane, sulfur hexafluoride, carbon bisulfide, hydrocarbons, chloro- and fluoroorganic substances. The positive chemical ionization method was used to identify impurities missing in the individual state and thus, not included in the library of mass spectra. It is the first found that monoisotopic hydrides contain an increased concentration of substances that have displaced isotopic composition. The quantitative analysis has been conducted by the method of absolute calibration. The determination of substances missing in the individual state was based upon the dependence of analysis sensitivity on their ionization cross section. The limits of detection for impurities are $2x10^{-6}$ – $1x10^{-9}$ mol. %, which are by 8-20 times lower than those given in literature.

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Handling time misalignment and rank deficiency in liquid chromatography by multivariate curve resolution: Quantitation of five biogenic amines in fish

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Biogenic amines are used for identifying spoilage in food. The most common are tryptamine, 2-phenylethylamine, putrescine, cadaverine and histamine. Due to lack of chromophores, chemical derivatization with dansyl was employed to analyze these amines using HPLC-DAD. However, the derivatization reaction occurs with any primary or secondary amine, leading to co-elution of analytes and interferents with identical spectral profiles, and thus causing rank deficiency. When the spectral profile is the same and peak misalignment is present on the chromatographic runs, it is not possible to handle the data only with MCR-ALS, by augmenting on the time, or the spectral mode. To overcome both problems, this paper proposes a new analytical methodology for fast quantitation of these BAs in fish with HPLC-DAD by using the icoshift algorithm for temporal misalignment correction before MCR-ALS spectral mode augmented treatment. Limits of detection, REP and average recoveries, ranging from 0.14 to 0.50 µg mL-1, 3.5 o 8.8% and 88.08% to 99.68%, respectively. These results reaches quantification limits for the five BAs much lower than those established by FAO/WHO, and EFSA, all without any pre-concentration steps. The concentrations of BAs in fish samples ranged from 7.82 to 29.41 µg g^{-1}, 8.68 to 25.95 µg g^{-1}, 4.76 to 28.54 µg g^{-1}, 5.18 to 39.95 µg g^{-1} and 1.45 to 52.62 µg g-1 for TRY, PHE, PUT, CAD, and HIS, respectively. In addition, the proposed method spends less than 4 minutes in an isocratic run, consuming less solvent in accordance with the principles of green analytical chemistry.

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