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Quantitation of N-terminal formaldehyde adducts to hemoglobin using UPLC-MS/MS

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Introduction: Formaldehyde (FA) is classified as carcinogenic by the International Agency for Research on Cancer. It is an important industrial and environmental chemical. Its occurrence in the environment created the need for data on human exposure to this chemical to assess the extent of exposure and potential health effects associated with the exposure. FA is highly reactive and forms covalent reaction products (adducts) with DNA and proteins. Hemoglobin adducts are successfully used for the assessment of human exposure to a wide range of chemicals. These adducts reflect the exposure over the past 3 month (lifetime of the erythrocyte). A reliable, accurate analytical method for measuring hemoglobin FA adducts was developed to address the need for data about human exposure to FA.

Methods: Sample preparation was performed using an automated liquid handler. Total hemoglobin content in the sample was determined using a commercial assay. We used TFE to denature proteins. After removal of TFE, hemoglobin was digested for 48 hours using trypsin at 48°C. We stopped the digestion reaction with formic acid and analyzed the resulting N-terminal peptide containing the adduct (FA-VHLTPPEEK) using UPLC-MS/MS (Thermo Accela UPLC with a Luna C18(2) 100 x 2.1 mm column and Thermo TSQ). Results were normalized to the amount of hemoglobin used for digestion. Quality control (QC) samples were prepared from erythrocytes in house for method validation.

Preliminary Data: The method is linear between 21.4 nmol/g - 177 nmol/g FA-VHLTPPEEK. The limit of detection is 3.4 nmol FA-VHLTPPEEK/g Hb and the limit of quantitation is 11.3 nmol FA-VHLTPPEEK/g Hb. The mean recovery is 96.4%±2.0%. The inter-assay and within-assay imprecision, expressed in %CV, determined with three QC samples containing different adduct levels range between 9.8% and 12.2%, and 7.1% and 13.8%, respectively. We measured FA-VHLTPPEEK in 192 RBCs specimens. FA-VHLTPPEEK was detected in all human samples, with FA-VHLTPPEEK concentrations ranging from 41.0 - 92.7 nmol/g Hb. This method is suitable for determining FA exposure in the general population.

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Synthesis and structural elucidation of Ni (II), Cu (II), Zn (II) and Cd (II) metal complexes of oxazoline based ligands

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Synthesis of 2-(2'-hydroxyphenyl)-2-oxazoline(2-Phox) and 2-(4-hydroxyphenyl)-2-oxazoline(4-Phox) and their complexes with Ni (II), Cu (II), Zn (II) and Cd (II) have been done. They have been characterized by elemental analysis, magnetic susceptibility, NMR (HH-COSY, HSQC, HMBC), UV-vis, IR spectroscopy and conductance measurement. The complexes were formed in 1:2 (M: L) ratio. Their molar conductance of one millimolar solution measured in DMF indicated them to be non-electrolyte. On the basis of these studies a tetrahedral geometry has been assigned for all the complexes.

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