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Laser [213-nm] depth-profiling studies of V/Ni ratios in asphaltenes following liquid nitrogen pre-treatment

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Author's group has developed a rapid procedure of ablating asphaltene samples with a high-precision deep-UV laser (213 nm) following swift solidification by immersion in liquid nitrogen. Liquid nitrogen pre-treatment of such samples produces a solid mass whose material characteristics are suitable for depth-profiling studies. The petrified samples were brittle and fractured into discreet fragments under stress. Normally, laser ablation is limited to hard solids (that can resist deformation) and studies of such "jelly-like" samples are usually not possible because of "splashing" effects. Asphaltene samples were petrified in aliquots of liquid nitrogen and immediately subjected to laser irradiation. The study was conducted to investigate V/Ni ratios with depth and compare these ratios with the universal V/Ni ratio in the digested sample. Depth-profiling was limited to specific depths, and rapid spatial and sub-surface distributions of metal components in the petrified samples were achieved well before "thawing" set in. An Nd:YAG deep UV (213nm) laser ablation system was attached to a high-precision ICP-MS instrument. The petrified samples were placed in a special sample holder with dimensions 5 cmx5 cm. Samples were subjected to 213-nm laser irradiation; the level of the beam energy was 60%, with a beam diameter of 55 μm. The laser ablated successive depths of 5 μm at each point and penetrated the sample to a depth of about 25 μm. Additional beam characteristics were as follows: fluence at the sample surface: ~4.5 J/cm^2; dwell time: 5 s; repetition rate: 10 Hz. The experimental results showed promise and the distinct capability to record spectra in the absence of "splashing" effects. This pre-treatment approach is therefore, highly viable, and a useful contribution to metal characterization of asphaltenes

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