Applications: Geo- and Cosmosciences

DETERMINATION OF TRACE ELEMENTS IN CRUDE OILS AND ORGANIC MATERIALS EXTRACTED FROM OIL-FORMING SOURCE ROCKS IN CHINA BY INAA

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A combination of Soxhlet extraction, column chromatography and INAA with GC and GC-MS was used in this work for the study of the abundance patterns of 36 trace elements and their correlation in nearly 100 samples of crude oil and organic materials extracted from oil-forming source rocks from 10 main Chinese oilfields. The low V/Ni ratios of 0.01 to 0.3 state the oil-forming continent environment. The geochemical significance hinted from other trace element abundances is also briefly discussed.

Introduction

The abundances, distributions and chemical species of trace elements in crude oils and organic materials extracted from oil-forming source rocks can provide valuable geochemical information about origin, migration and evolution of petroleum.\(^1\),\(^2\) In addition, they are essential to refining processing and environmental pollution control as well.

However, the concentrations of trace elements in crude oil and organic extracted material are quite low, in most cases at some \(\mu g/g\) or lower. Conventional chemical analysis often has the danger of contamination caused by reagent blank or of volatile loss during ashing and preparation process of organic samples. Thus, some authors applied NAA to determine trace elements in crude oils in the world.\(^3\),\(^4\) In this work we systematically studied trace elements in crude oil and organic materials extracted from oil-forming source rocks in China with INAA, mainly because the related data are scarce in the literature.
Experimental

Crude oil samples

Collaborated with institutes of petroleum in China, we collected various crude oil samples from 10 main Chinese oilfields: Jianghan, Zhongyuan, Shengli, Liaohe, Kalamayi, Biyang, Dongfeng, Renqiu, Dagang and Shonghuajiang. About 100 mg samples of each crude oil were taken and rinsed with 9:1 (volume) benzene-methanol solution for 24 hours, then filtered in a funnel containing anhydrous sodium sulfate. The treated oil samples were concentrated to dryness. Aliquots of 20 to 40 mg samples were used for column chromatography separation, gas-chromatography (GC) and GC-mass-spectroscopy (MS). The rest was for INAA.

Organic materials extracted from rocks

Each 50–100 g of the 80–100 mesh oil-forming source rock samples was extracted in a Soxhlet extractor with 200–250 ml of 9:1 benzene-methanol or chloroform at 80 or 65 °C, respectively. The extracted organic solution was filtered to remove the fine rock powder in it, then concentrated to dryness for column chromatography separation and analysis.

Group fraction separation

The above oil and organic material samples were fractionated as follows:

1) Precipitation and filtration of asphaltene. 10 to 30 mg of the oil or organic material samples were dissolved in 30–40 ml n-hexane, then left to stand overnight to make asphaltene precipitated. The asphaltene fraction was filtered and repeatedly washed by n-hexane until the solvent became colorless. Again, the asphaltene in the container and filter was washed off by dichloromethane or chloroform. After concentration to dryness, it is subjected to NAA and column chromatography.

2) Column chromatography separation. After filtering and separation from asphaltene, the organic samples were subjected to column chromatography fractionation, and divided into 3 group fractions: saturated hydrocarbons, aromatic hydrocarbons and nonhydrocarbons. The first fraction was eluted with n-hexane, followed by aromatic hydrocarbons and non-hydrocarbons with dichloromethane and anhydrous alcohol, respectively. The 7:3 (volume) silica gel (60–80 mesh) and neutral Al₂O₃ (100–120 mesh) were used as absorbent, beforehand activated for 4 hours at 180 and 400 °C, respectively.

The fraction of saturated hydrocarbons is only for GC and GC-MS, not subject to INAA, since the concentrations of trace elements in it are too low. The aromatic