

## **TLC-FID (IATROSCAN) ANALYSIS OF HEAVY OIL AND TAR SAND SAMPLES**

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TLC-FID (thin layer chromatography-flame ionization detector, Iatroscan) has long been used as a rapid and inexpensive way to determine SARA fractions (saturate + aromatic = hydrocarbon, resin + asphaltene = polar) in crude oils and bitumen extracts. However, the analysis of heavy oils and tar sands (HOTS) poses a number of additional problems. HOTS have relatively high amounts of polar compounds that are retained near the origin on TLC rods, potentially causing both separation and quantitation problems. Additionally, the polar compounds have variable quantities of heteroatoms resulting in different FID response factors and thus further complicating the quantitation of the SARA fractions.

Two operational variables have been optimized with respect to improving the reproducibility of the Iatroscan data generated for HOTS samples. In addition to internal reproducibility, these operational parameters were also investigated with respect to obtaining SARA results that were more directly comparable to determination by asphaltene precipitation followed by open column chromatography using a silica gel alumina/column.

The first variable was to simply consider the size of the spot in which the sample was initially applied to the rod. Counterintuitively, it was observed that a very small spot (~2 mm) applied using an automated spotting device did not provide for better separation of the SARA fractions. More reproducible results were obtained by using a relatively rapid injection resulting in an initial spot size of about 5-6 mm. This result stems from the fact that SARA fractionation is not a normal chromatographic separation but rather a fractionation based on moving different polarity groups different distances up the rod by developing the rods with a series of different solvents to different distances. The apparent loss of resolution with the very small spot size may result from overloading of the silica at the origin and commensurate channeling of the low polarity solvents through the spot.

The second variable tested was to alter the properties of the silica rods by doping them with a small amount of transition metal. The effect of this was to increase the absolute response of the hydrocarbon fractions (Fig.1), especially the saturate compounds and to reduce the relative concentration of the polar compounds. Doping the rods with transition metal may also help to minimize the variability of response factor of the polar fractions.

Iatroscan SARA analysis of individual fractions obtained from asphaltene

precipitation and open column chromatography demonstrates that the two analytical techniques do not provide precisely the same separation. This is not surprising because the open column technique generally is carried out with both silica gel and alumina absorbents whereas the Iatroscan was carried out on silica rods or metal doped silica rods. It may not be possible to make a direct comparison of data generated by the two techniques because the polar fractions are fundamentally different and the Iatroscan technique also incorporates a response factor that is not an issue in the gravimetric detection of fractions from column chromatography. However, Iatroscan may well provide a higher degree of reproducibility of the amounts of polar fractions than column chromatography in the case of HOTS samples. In part, this reduced scatter may result from reduced oxidation during the TLC fractionation, reduction of physical occlusion, and also because evaporation of the solvent from a partially volatile sample is more consistent.

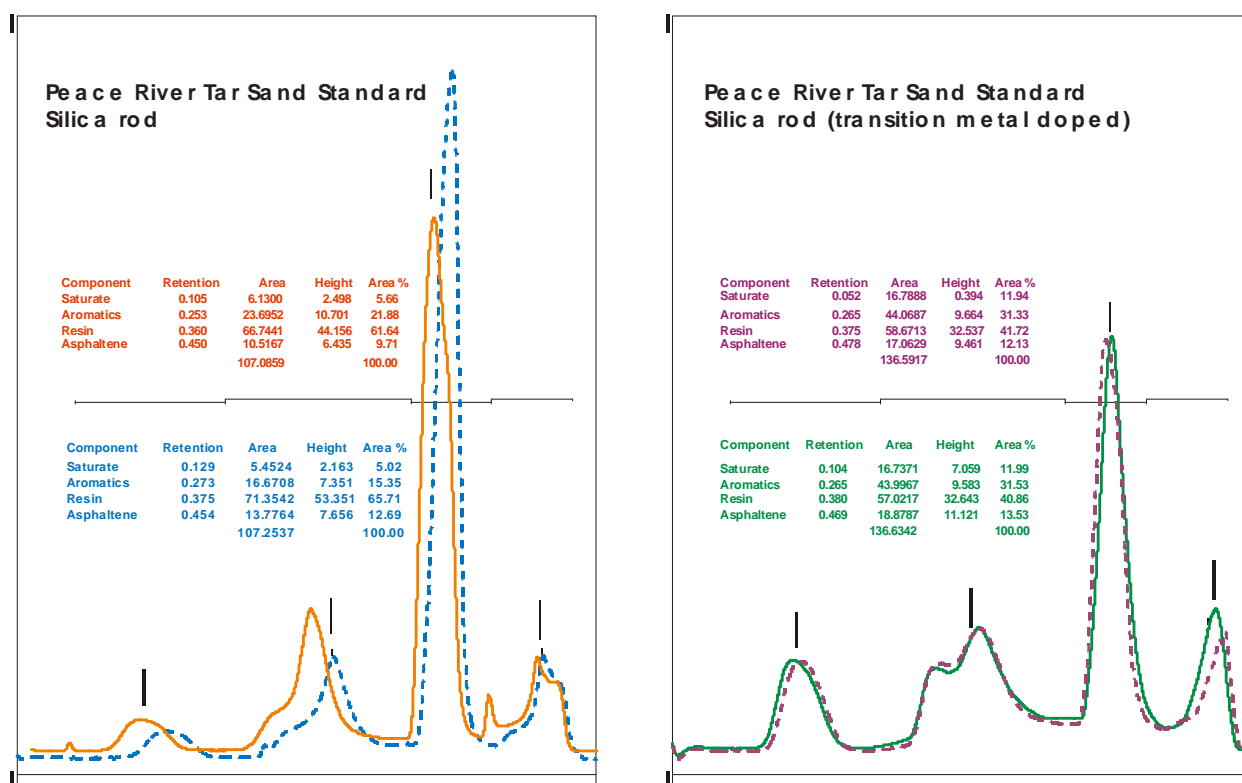


Figure 1. Comparison of absolute response and reproducibility of Iatroscan SARA analysis of a sample of Peace River Tar Sand bitumen.