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THE PARTITIONING OF MAJOR, MINOR, AND TRACE ELEMENTS DURING SIMULATED IN-SITU OIL SHALE RETORTING

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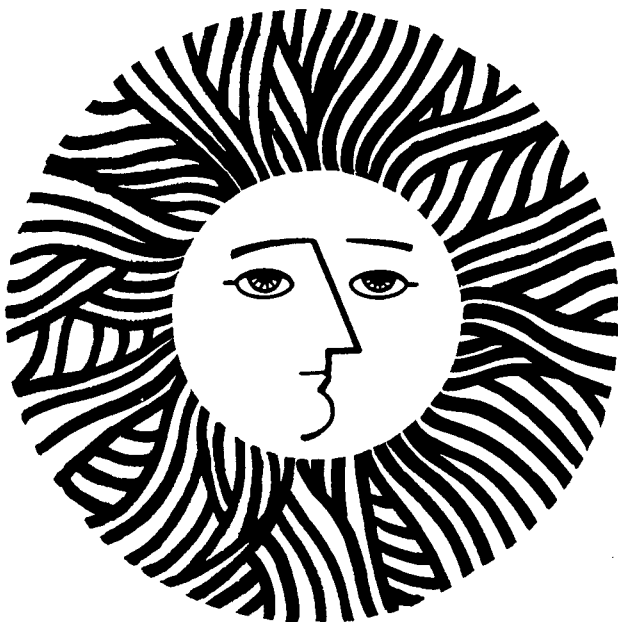
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April 10, 1980

TO: Art Hartstein
FROM: Richard Fish, Bob Giaouque, and Phyllis Fox
RE: March Monthly Progress Report
The Partitioning of Major, Minor, and Trace Elements
During Simulated In-Situ Oil Shale Retorting
LBID-196

NAVY CORE PROJECT

All analyses, data reduction, and quality control checks were completed for both core holes and a draft of the final report was written.

IDENTIFICATION OF POTENTIAL ORGANIC COMPOUNDS AS
LIGANDS OF METALS IN OIL SHALE PROCESS WATERS

We have continued our studies on the isolation and identification of organic ligands potentially associated with trace metals in oil shale process waters. The experimental approach, as before, includes extraction of the retort water (pH ~9) with methylene chloride, concentration of the methylene chloride extract, and adsorption of this residue on a dry Florisil column. The Florisil column is then eluted with hexane, methylene chloride, and isopropyl alcohol under controlled gradient conditions using both high performance liquid chromatographic pumps. Fractions of 4.0 ml are collected automatically with a fraction collector and those that have ultraviolet absorptions at 254 nm are grouped, concentrated, and analyzed using capillary column gas chromatography. The grouped fractions of interest are then analyzed by capillary column gas chromatography/electron impact mass spectrometry.

We have presently analyzed three retort water samples: Geo-kinetics', Laramie Energy Technology Center's 150-ton retort water, and Occidental's retort water. Using the techniques discussed, we have identified a series of ligands which include

methyl-substituted phenols, substituted pyridines, aliphatic nitrogen heterocycles, methyl-substituted quinoline compounds, and cyclic aliphatic ketones. It is interesting to note that all three retort water samples had a somewhat similar profile of bands of compounds absorbing at 254 nm in the ultraviolet (Figures 1, 2, and 3).

We are also measuring trace element concentrations in the various fractions eluted from the Florisil column. In this way, we hope to be able to correlate the organic ligands with the metal content and thereby infer the types of organometallic compounds and complexes that may be present. This month, we initiated these analyses for Zn using graphite furnace atomic absorption spectrometry. A methylene chloride extract (100 ml) of 150-ton retort water gave a Zn value of approximately 13 ppm. Analyses of several fractions containing the nitrogen aliphatic and aromatic heterocycles yielded Zn concentrations of about 1 ppm.

RHF:lmv

Figure 1. Plot of solvent composition versus fractions collected for a gradient elution experiment with Geokinetics' retort water. Bands represent ultraviolet absorption at 254 nm.

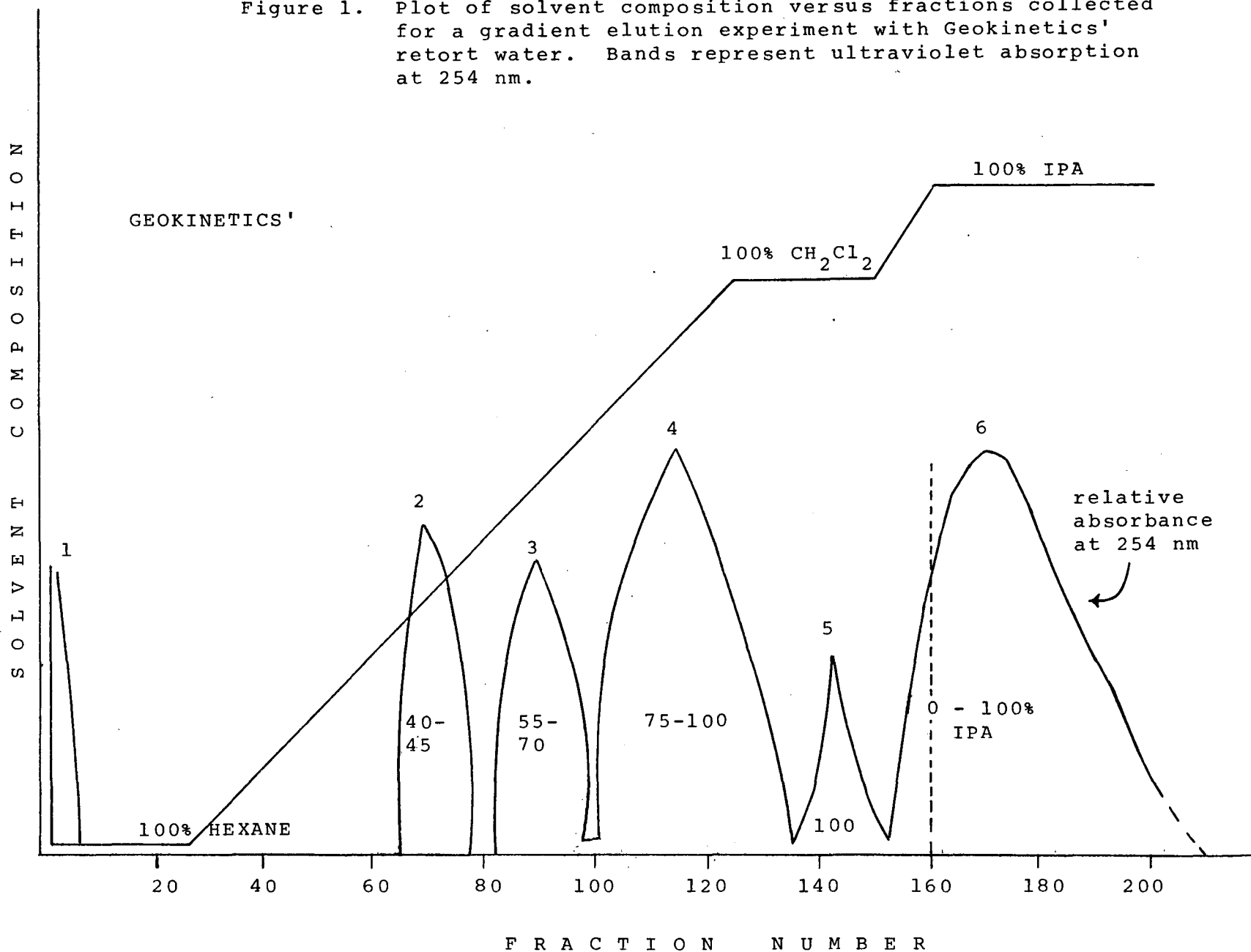


Figure 2. Plot of solvent composition versus fractions collected for a gradient elution experiment with 150-ton retort water. Bands represent ultraviolet absorption at 254 nm.

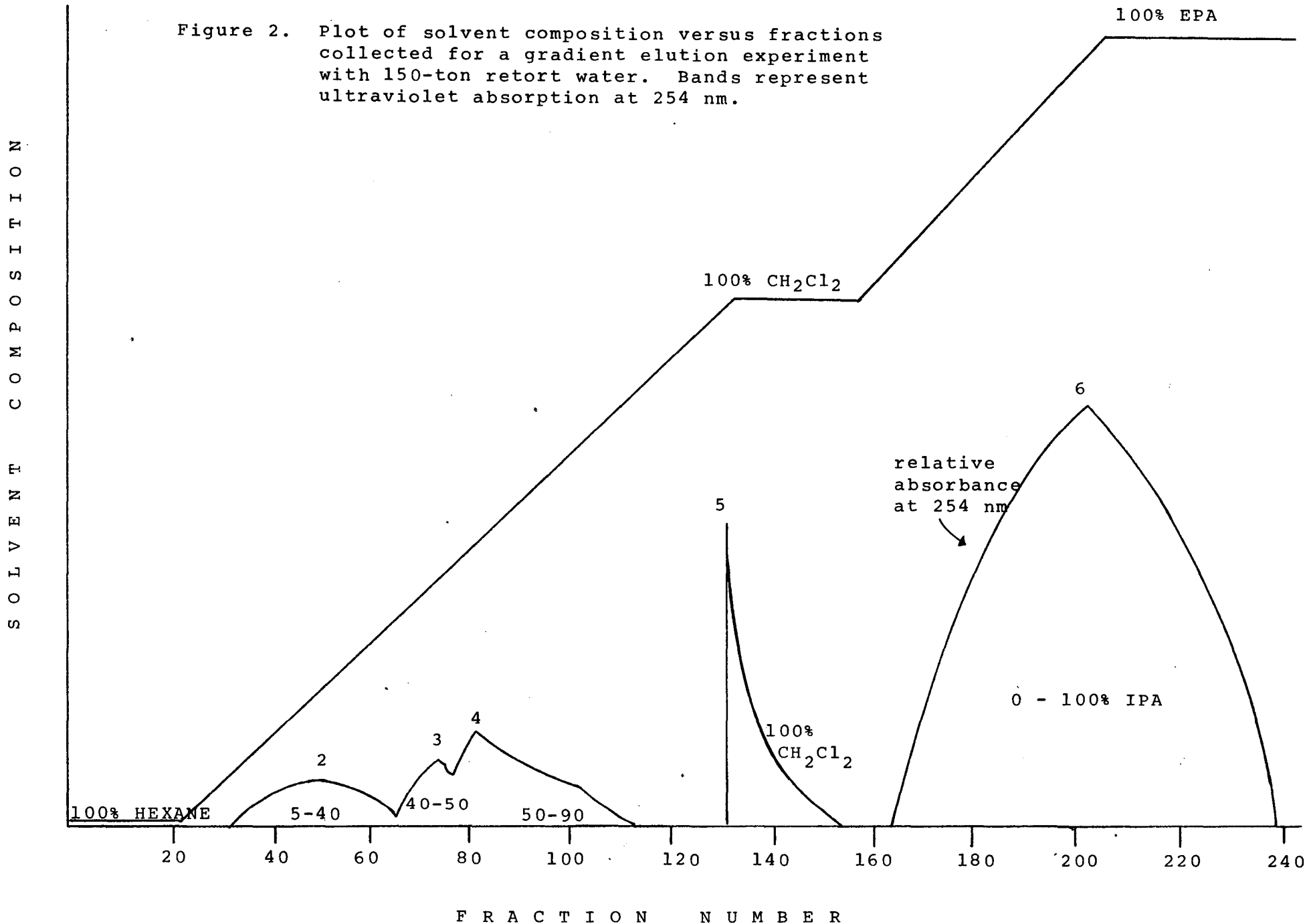
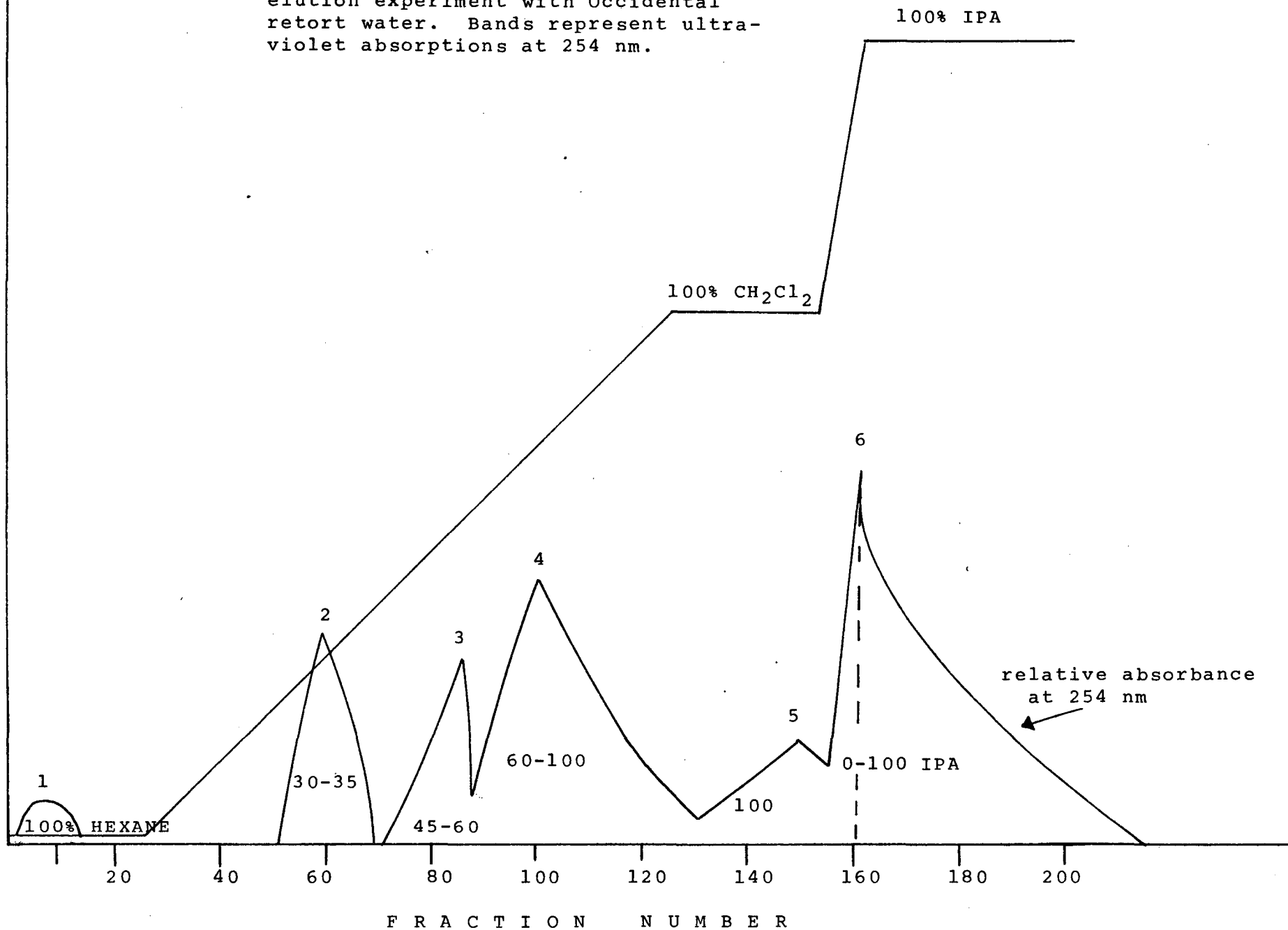


Figure 3. Plot of solvent composition versus fractions collected for a gradient elution experiment with Occidental retort water. Bands represent ultraviolet absorptions at 254 nm.

S O L V E N T C O M P O S I T I O N S



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