A.3 Silica Gel Cleanup (SGC)

What It Is

"Bulk" extractable TPH analyses such as SW-846 Method 8015B/C measure all organics present in a sample that (1) can be extracted using an organic solvent and (2) elutes on a gas chromatograph column within a selected boiling-point range. These extraction and quantitation methods are not specific to petroleum hydrocarbons, and the organics measured can include hydrocarbons (HCs) and nonhydrocarbons (non-HCs). SGC is a method used by the laboratory to "clean up" the sample extract before it is analyzed for TPH so that the extract contains primarily HCs. This cleanup is possible because of the different chemical properties between HCs and non-HCs. HCs contain only carbon and hydrogen atoms, and thus are relatively "nonpolar" molecules, and non-HCs contain an atom(s) in addition to carbon and hydrogen (such as oxygen, sulfur ,or nitrogen), and thus are relatively "polar" molecules. The most common non-HCs in groundwater or soil at petroleum release sites are:

- metabolites from biodegradation of the petroleum (e.g., compounds containing oxygen, such as organic acids and alcohols, etc.)
- natural organic matter (e.g., humic acids, etc.)
- sampling, laboratory, or ambient anthropogenic artifacts (e.g., phthalates or BPA)
- other nonpetroleum-related chemicals (e.g., chlorinated solvents)

What It Does

Silica gel (SG) is used for the cleanup because it is a polar substance and therefore adsorbs the polar non-HCs. The best SGCs are based on SW-846 Method 3630C, which is a column cleanup procedure (Figures A2-1 and A2-2). A glass column is packed with SG, the extract is poured onto the column, the SG preferentially adsorbs the polar non-HCs as the extract moves through the column, and the "cleaned up" extract is collected at the bottom and then analyzed for TPH. Lab surrogates are added to the extract before the procedure to monitor effectiveness of the procedure. Other SGC methods can be used and typically are based on adding a few grams of SG to the extract vial and shaking it for a specified time period. Research shows that the shake method is not as effective as the column cleanup for methylene chloride extracts.

How You Can Use It

SGC can be used for many reasons:

- To compare extractable TPH results to hydrocarbon (HC)-based regulatory criteria. SGC allows for an "apples-to-apples" comparison of HCs in the sample to HC-based criteria.
- To accurately measure the extractable-range HCs in the sample. SGC allows the HC signal not to be lost in the total organics mixture.
- To document that biodegradation is occurr ing at the site by comparing results for TPH without SGC to TPH with SGC for a single sample. Oxygen-containing compounds are removed from diesel during the refining process because they will have a detrimental impact on engine performance, so the difference between the without and with SGC is the non-HC content of the sample, which at many sites are the biodegradation metabolites.
- To provide better remediation design and monitoring. Non-HCs have much different physical and chemical properties than HCs, so understanding the site-specific content/proportions of the organics present is important.
- SGC CANNOT be performed on samples to be analyzed for volatile TPH (e.g., GRO/TPH-g) because (1) those samples are not extracted prior to being analyzed, and (2) the cleanup step could result in loss of volatiles.

SGC Schematic (USEPA 3630C)

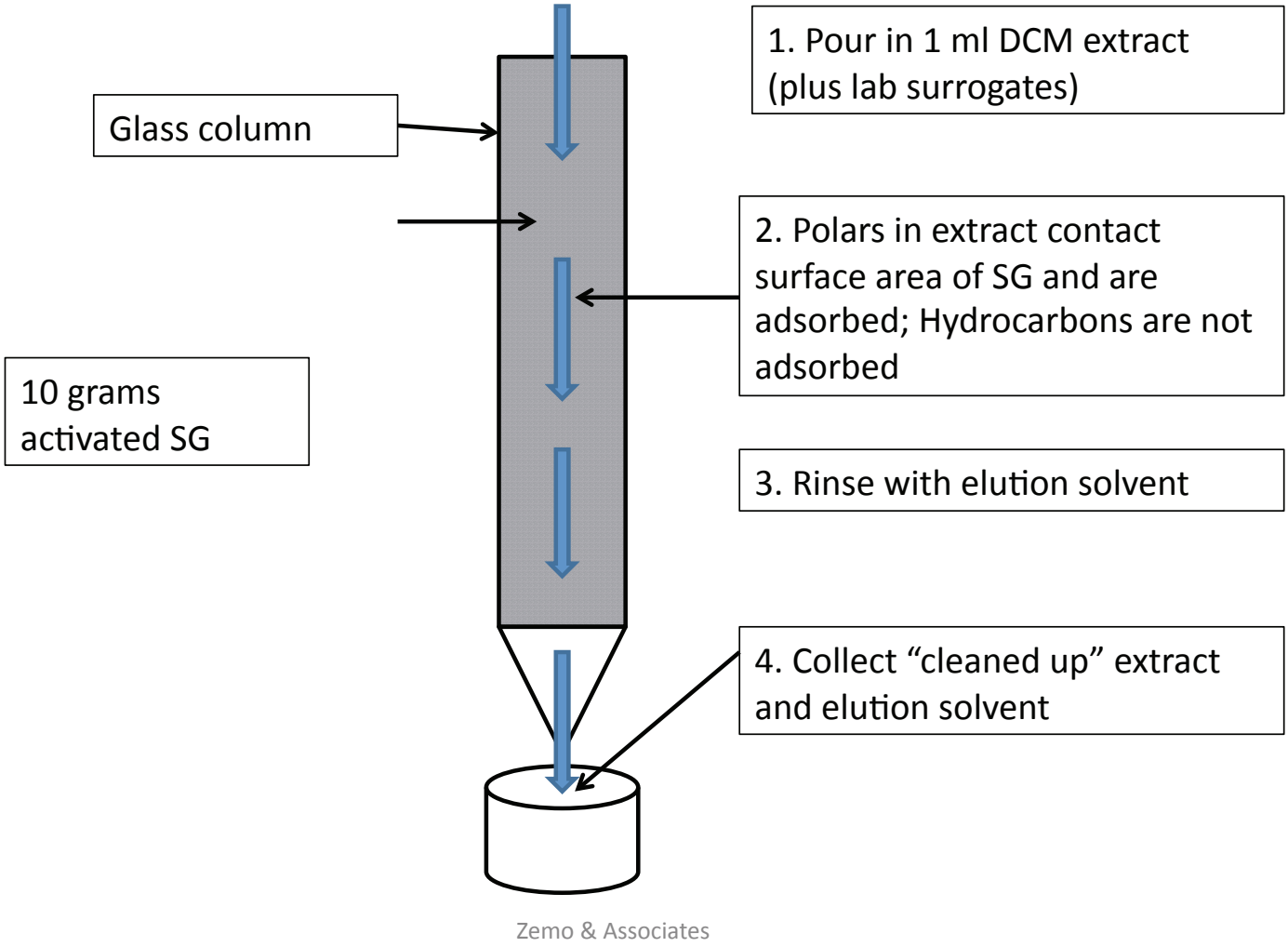

Figure A2-1. SGC schematic (USEPA 3630C)
(Source: Zemo and Associates, 2018.)

Figure A2-2. Laboratory column loaded with silica gel
(Source: Zemo and Associates, 2018.)

Note: Although not the focus of this fact sheet, a silica gel column is also used to separate aliphatic and aromatic HC classes for detailed "fractionated TPH" analyses; that method results in the separation of HCs and non-HCs as part of the procedure.

Silica Gel Use as Media to Separate Aliphatic and Aromatic Hydrocarbons

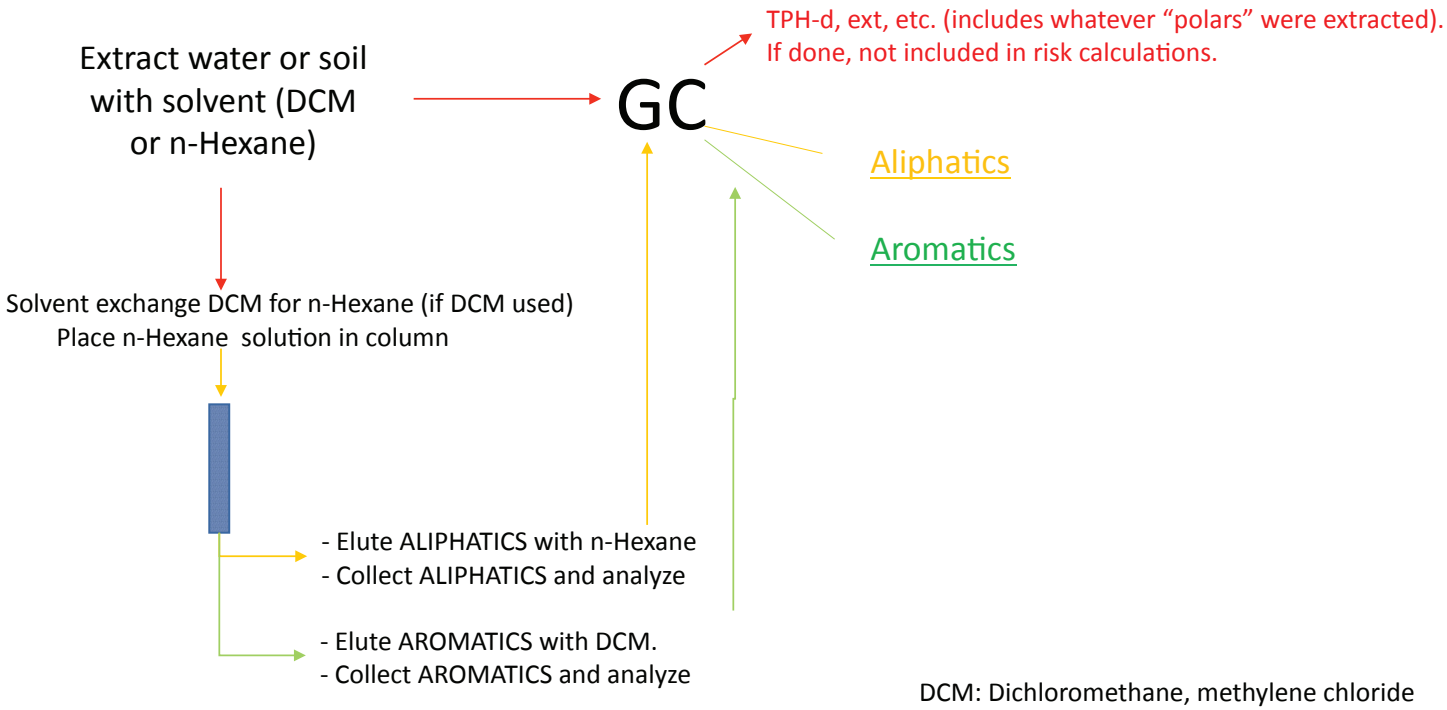

Figure A2-3. Silica gel use as media to separate aliphatic and aromatic hydrocarbons

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