CONTAMINATED SOILS, SEDIMENTS AND WATER
Volume 10:
Successes and Challenges
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Foreword

Every spring, the University of Massachusetts – Amherst welcomes all “Soils Conference” Scientific Advisory Board members with open arms as we begin the planning process responsible for bringing you quality conferences year after year. With this “homecoming” of sorts comes the promise of reaching across the table and interacting with a wide spectrum of stakeholders, each of them bringing their unique perspective in support of a successful Conference in the fall.

This year marks the 20th anniversary of what started as a couple of thoughtful scientists interested in developing partnerships that together could fuel the environmental cleanup dialogue. Since the passage of the Superfund Law, regulators, academia and industry have come to realize that models that depend exclusively on “command and control” mandates as the operative underpinning limit our collective ability to bring hazardous waste sites to productive re-use. It is with this concern in mind that the Massachusetts Department of Environmental Protection privatized its cleanup program in 1993, spurring the close-out of over 20,000 sites and spills across the Commonwealth to date, in a manner that is both protective of human health and the environment while also flexible and responsive to varied site uses and redevelopment goals.

So we gather together again, this year, to hear our collective stories and share success and challenges just as we share stories at a family gathering. Take a read through the stories contained in these proceedings, Volume 10 of the Contaminated Soils, Sediments and Water. This jewel of a volume
contains a valuable collection of successes (and challenges) in the areas of environmental fate, heavy metals, modeling, MtBE and oxygenates, regulatory, remediation, risk assessment, site assessment and sampling methodology. As you can see, there is something for everybody. Most importantly, in our minds at least, is the embodiment of how, as a community, we have worked together toward the optimization of established approaches as well as embracing departures from traditional regulatory models in order to address the challenges posed by emerging unregulated constituents that threaten our natural resources.

It is with great joy and pride that we write this Foreword, an affirmation of our commitment to this international, one-of-a-kind conference. A conference that over the last 20 years has taken into account where we all have been – public and private sector alike – so we can responsibly chart where we need to go as stewards of the environment.

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An appreciated acknowledgement to **Denise Leonard**, Conference Coordinator, for her time and energy contributed throughout the year to the organization of The Annual International Conference on Soils, Sediment & Water at the University of Massachusetts, Amherst. Also acknowledgement to her assistant, **Holly Dodge**, for her support over the last year in her contribution to the compilation of this book and other aspects pertaining to the conference.
Edward J. Calabrese is a board certified toxicologist and professor of toxicology at the University of Massachusetts School of Public Health at Amherst. Dr. Calabrese has researched extensively in the area of host factors affecting susceptibility to pollutants and has authored more than three hundred papers in scholarly journals, as well as twenty-four books, including: Principles of Animal Extrapolation; Nutrition and Environmental Health, Vols. 1 and 2; Ecogenetic: Safe Drinking Water Act: Amendments. Regulations, and Standards; Soils Contaminated by Petroleum: Environmental and Public Health Effects; Petroleum Contaminated Soils, Vols. 1, 2 and 3; Ozone Risk Communication and Management; Hydrocarbon Contaminated Soils, Vols. 1, 2, 3, 4 and 5; Hydrocarbon Contaminated Soils and Groundwater, Vols. 1, 2, 3, and 4; Multiple Chemical Interactions; Air Toxics and Risk Assessment; Alcohol Interactions with Drugs and Chemicals; Regulating Drinking Water Quality; Biological Effects of Low Level Exposures to Chemicals and Radiation; Contaminated Soils; Diesel Fuel Contamination; Risk Assessment and Environmental Fate Methodologies; Principles and Practices for Petroleum Contaminated Soils, Vols. 1, 2, 3, 4, and 5; Contaminated Soils, Vol. 1; and Performing Ecological Risk Assessments. He has been a member of the U.S. National Academy of Sciences and NATO Countries Safe Drinking Water Committees, and the Board of Scientific Counselors for the Agency for Toxic Substances and Disease Registry (ATSDR). Dr. Calabrese also serves as Director of the Northeast Regional Environmental Public Health Center at the University of Massachusetts, Chairman of the BELLE Advisory Committee and Director of the International Hormesis Society.
Paul T. Kostecki, Vice Provost for Research Affairs, University of Massachusetts at Amherst and Associate Director, Northeast Regional Environmental Public Health Center, School of Public Health, University of Massachusetts at Amherst, received his Ph.D. from the School of Natural Resources at the University of Michigan in 1980. He has been involved with human and ecological risk assessment and risk management research for the last 13 years. Dr. Kostecki has co-authored and co-edited over fifty articles and sixteen books on environmental assessment and cleanup including: remedial Technologies for Leaking Underground Storage Tanks; Soils Contaminated by Petroleum Products; Petroleum Contaminated Soils, Vols. 1, 2, and 3; Hydrocarbon Contaminated Soils and Groundwater, Vols. 1, 2, 3 and 4; Hydrocarbon Contaminated Soils, Vols. 1, 2, 3, 4 and 5; Principles ad Practices for Petroleum Contaminated Soils; Principles and Practices for Diesel Contaminated Soils, Vols. 1, 2, 3, 4 and 5; SESOIL in Environmental Fate and Risk modeling; Contaminated Soils, Vol. 1; and Risk Assessment and Environmental Fate Methodologies. Dr. Kostecki also serves as Associate Editor for the Journal of Soil Contamination, Chairman of the Scientific Advisory Board for Soil and Groundwater Cleanup Magazine, as well as an editorial board member for the journal Human and Ecological Risk Assessment.

In addition, Dr. Kostecki serves as Executive Director for the Association for the Environmental Health of Soils (AEHS). He is a member of the Navy's National Hydrocarbon Test Site Advisory Board and a member of the Steering Committee for the Total Petroleum Hydrocarbon Criteria Working Group and the Association of American Railroads Environmental Engineering and Operations Subcommittee.

James Dragun, as a soil chemist (Ph.D. Penn State University in Agronomy), has a rich background in the fate of hazardous materials in soil systems and groundwater. He has assessed the migration and degradation of chemicals and waste of national concern in soil-groundwater systems such as dioxin, PBB, Radionuclides at Three Mile Island Nuclear Power Plant, PCB and petroleum spills, organics and inorganics at the Stringfellow Acid Pits, pesticides in San Joaquin Valley groundwater, and solvents in Silicon Valley groundwater. In addition, he has analyzed engineering controls designed to prevent the leakage of chemicals and wastes from landfills, surface impoundments, deepwell injection systems, underground storage tanks, land treatment systems, manufacturing and processing facilities, and hazardous waste sites. He has analyzed the chemical integrity and reactivity of materials used to treat and store hazardous and nonhazardous wastes. He has served as an expert reviewer of over 40 projects and programs involving
the siting, design, construction, performance, and failure mechanisms of landfills, land treatment systems, surface impoundments, and waste piles. In addition, he has authored exposure assessments for over 100 chemicals and wastes.

Widely recognized for his expertise, Dr. Dragun was appointed the primary technical advisor on exposure assessment to the Interagency Testing Committee, a consortium of 14 federal agencies that selects chemicals for potential regulatory control. He directs the Association of Official Analytical Chemist's development of standard methods to measure the migration and degradation of chemicals and wastes, and has authored test methods that are used today by environmental laboratories in North America, Europe, and Asia. His counsel and scientific findings have been disseminated and utilized by 24 nations including Japan, Canada, the United Kingdom, Australia, West Germany, Switzerland, Italy, France, Spain, Scandinavia, and the Netherlands.

Dr. Dragun is a member of Phi Kappa Phi and Sigma Xi, both international honorary scientific societies, and was awarded the U.S. EPA Bronze Medal for distinguished service in 1980.
PART 1: ENVIRONMENTAL FATE

CHAPTER 1

SLOW DESORPTION OF PHENANTHRENE FROM SILICA PARTICLES: INFLUENCE OF PORE SIZE, PORE WATER, AND AGING TIME

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Abstract: When micro-porous and meso-porous silica particles were exposed to aqueous phenanthrene solutions for various durations it was observed that sorbed-phase phenanthrene concentrations increased with aging time only for meso-porous but not micro-porous silicas. Desorption equilibrium was reached almost instantaneously for the micro-porous particles while both the rate and extent of desorption decreased with increasing aging time for the meso-porous silicas. These findings indicate that phenanthrene can be sequestered within the internal pore-space of meso-porous silicas while the internal surfaces of micro-porous silicas are not accessible to phenanthrene sorption, possibly due to the presence of physi-sorbed water that may sterically hinder the diffusion of phenanthrene inside water-filled micro-pores. By contrast, the internal surfaces of these micro-porous silicas are accessible to phenanthrene when incorporation methods are employed which assure that pores are devoid of physi-sorbed water. Consequently, when phenanthrene was incorporated into these particles using either supercritical CO₂ or via solvent soaking, the aqueous desorption kinetics were extremely slow indicating effective sequestration of phenanthrene inside micro-porous particles. Finally, a two-
compartment conceptual model is used to interpret the experimental findings and the implications for contaminant fate and transport are discussed.

**Key words:** Contaminant Fate and Transport, Contaminant Sequestration, Desorption Kinetics, Aging Methodology, Porous Silica, Phenanthrene, Two-Compartment Model.

1. **INTRODUCTION**

The remediation of aged hydrophobic contaminants in soils and sediments has in many cases been complicated by the extremely slow or incomplete release of these compounds from the mineral particles. It has been postulated that the slow desorption and related sequestration of these hydrophobic contaminants is most likely caused by several mechanisms such as the slow diffusion within either hard or soft organic matter domains or by sorption-retarded and sterically hindered diffusion in small mineral pores (Alexander, 1995; Huesemann, 1997; Hatzinger and Alexander, 1997; Luthy et al., 1997; Pignatello, 1990; Pignatello and Xing, 1996; Steinberg et al., 1987; Weber and Huang, 1996; Weber et al., 1998; Xing and Pignatello, 1997).

Considering that most naturally occurring soils and sediments contain significant amounts of organic matter, it is not surprising that most research has focused on elucidating the nature of contaminant sequestration in the various organic matter (OM) phases. In fact, it has been suggested by Cornelissen et al. (1998) that the presence of OM is more important for slow desorption than mineral micropores in soils and sediments with more than 0.1-0.5% OM. As a result, comparatively little contaminant sequestration research has been carried out to evaluate the role of mineral micropores in the absence of OM (Nam and Alexander, 1998; Huang et al., 1996; Farrell and Reinhard, 1994a, 1994b; Alvarez-Cohen et al., 1993; Werth and Reinhard, 1997a, 1997b; McMillan and Werth, 1999).

Huang et al. (1996) studied the aqueous sorption and desorption of phenanthrene in meso-porous silica gels (40Å, 100Å, and 150Å) and found that little or no phenanthrene sorption occurred on internal pore-surfaces. These investigators hypothesized that the presence of physi-sorbed water in silica pores results in the size-exclusion of phenanthrene from the interior pore space. They therefore concluded that the use of models that invoke solute diffusion in meso- and micro-porous mineral structures as a significant rate-limiting factor for sorption by soils and sediments is highly questionable. Nam and Alexander (1998) measured the biodegradation kinetics of phenanthrene that had been incorporated onto non-porous and
meso-porous (25Å, 60Å, and 150Å) silica particles via aqueous sorption. Since no significant difference in biodegradation rates between non-porous and porous silicas was observed, these investigators also concluded that the internal surfaces of these porous beads sorb little or no phenanthrene.

It is the objective of this research to further elucidate the various factors that affect slow desorption and sequestration of hydrophobic contaminants in mineral micro- and meso-pores in the absence of organic matter. Specifically, we are interested in how the pore-diameter, the presence (or absence) of water during the phenanthrene incorporation process, and the aging time influence the aqueous desorption kinetics of phenanthrene from silica particles.

2. MATERIAL AND METHODS

2.1 Silica Particles

The types of silica particles used in this study are listed in Table 1. Four batches of meso-porous silica particles ranging in size from 1 -10μ and median pore diameter (based on pore-volume) from 18Å to 76Å were synthesized using techniques similar to those described by Bruinsma et al. (1998) and Beck et al. (1992). Cetyltrimethylammonium chloride was used in combination with tetraethoxysilane to prepare the particles in batches 1 and 2. A cetyltrimethylammonium hydroxide/cetyltrimethylammonium chloride mixture in combination with sodium aluminate and mesitylene was employed to prepare the silicas in batches 3 and 4. The synthesized particles were calcined by heating using a temperature ramp from 20°C to 540°C under a nitrogen purge. Prior to use in the experiments, the cooled particles were ground lightly with a mortar and pestle to break up large aggregates.

<table>
<thead>
<tr>
<th>Particle Type</th>
<th>Particle Diameter (μ)</th>
<th>Median Pore Diameter (Å)</th>
<th>Surface Area (m²/g)</th>
<th>TOC (%, w/w)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Batch #1</td>
<td>1 - 10</td>
<td>18</td>
<td>825</td>
<td>0.0031</td>
</tr>
<tr>
<td>Batch #2</td>
<td>1 - 10</td>
<td>21</td>
<td>755</td>
<td>0.0096</td>
</tr>
<tr>
<td>Batch #3</td>
<td>1 - 10</td>
<td>76</td>
<td>858</td>
<td>0.0047</td>
</tr>
<tr>
<td>Batch #4</td>
<td>1 - 10</td>
<td>66</td>
<td>845</td>
<td>0.0068</td>
</tr>
<tr>
<td>Davisol</td>
<td>250 - 500</td>
<td>202</td>
<td>314</td>
<td>0.0078</td>
</tr>
<tr>
<td>Spheriglass</td>
<td>2</td>
<td>NA</td>
<td>2</td>
<td>ND</td>
</tr>
</tbody>
</table>

NA = Not Applicable, ND = Not Determined

Davisil silica gel with a median pore diameter of 202Å and a particle size range of 250-500μ was purchased from Supelco, Bellefonte, PA. Finally,
non-porous silica beads (i.e., spheriglass solid spheres) with a mean particle size of 2μ were purchased from Potters Industries, Inc., Carlstadt, NJ.

The total organic carbon (TOC) content of the silica particles was determined by placing an aliquot into a platinum crucible and heating it at 550°C for 16.5 hours. The carbon dioxide that was released as a result of this oxidation process was catalytically converted to methane which was subsequently analyzed by gas chromatography. The BET surface area and the pore-diameter distribution (based on pore-volume) of the silica particles was determined using a Micrometrics Surface Area Analyzer (Model 2010 Micrometrics Instrument Corp., Norcross, GA) according to procedures given in the operating manual (Micrometrics, 1995).

2.2 Hydration of Silica Particles

Preliminary aqueous sorption experiments involving dry silica particles from batches 1 and 2 indicated that phenanthrene sorption processes are significantly affected by changes in the silica surface chemistry that occur slowly when dry silica is exposed to water. In order to eliminate this confounding factor, all silica particles used in aqueous sorption experiments were pre-wetted in de-ionized water for one week. After the wetting period, the supernatant was carefully removed, and the sorption experiments were initiated by adding aqueous phenanthrene solution as described in more detail below.

In addition, all silica particles that were loaded with phenanthrene using non-aqueous methods (see details below) were also hydrated prior to phenanthrene loading to avoid the unusual aqueous desorption kinetics that are due to changes in silica surface chemistry. Five to ten grams of silica particles were equilibrated with 150 mL of de-ionized water over a period of 3 to 4 days at room temperature. The equilibrated particles were then filtered and dried under house vacuum in a dessicator containing Drierite for 5 to 6 days. The loss of water was monitored during the drying process. Drying was terminated when the weight of silica closely approximated its original starting weight. Additional water was then removed by subjecting the silica particles to high vacuum (4 to 5 X10⁻⁶ torr) for a period of 5 to 6 days. This procedure is known to remove all physi-sorbed water while chemi-sorbed water remains on the silica surfaces (Young, 1958).
2.3 Phenanthrene Sorption

2.3.1 Preparation of Phenanthrene Stock Solution

The phenanthrene stock solution was prepared as follows. 100 mg of ultrapure (99.5%+) phenanthrene (Aldrich Chemical Company) was placed into a small polyethylene bag (1.5" X 1.5", 4 mil). After the addition of 10 mL hexane (95% pure, Burdick and Jackson Chemical Company), the polyethylene bag was heat-sealed. The bag was slowly inverted until all phenanthrene crystals were dissolved. The hexane was evaporated by placing the bag into a hood for 2-3 days.

The bag was then transferred to an amber glass bottle (ca. 3.8 L) filled with a buffered (pH 7) solution containing 5 mg/L sodium bicarbonate (NaHCO₃) and 100 mg/L sodium azide (NaN₃) dissolved in de-ionized water (Huang et al., 1996). The bottle was sparged with nitrogen, capped, and then placed on a magnetic stirrer. The submerged polyethylene bag was mixed within the bottle until the aqueous phenanthrene concentration reached after 9 days equilibrium levels at 874 ug/L, which is close to the maximum reported solubility of this compound (Mackay et al., 1992). Aliquots of this phenanthrene stock solution were used in all sorption experiments.

This particular procedure was developed to assure that the aqueous solution is truly free of phenanthrene crystals that have been known to negatively affect the reproducibility of sorption and desorption experiments. In addition, in this method the use of solvents (e.g., methanol) that are commonly used to dissolve phenanthrene prior to the spiking of water was also avoided, thereby eliminating any potential negative influences that a co-solvent may have on sorption kinetics or equilibria.

2.3.2 Sorption Experiments

All sorption experiments were carried out in 30 mL amber centrifuge glass tubes with screw caps and Teflon-lined silicone septa. Prior to use, the glass tubes were ashed at 450°C for 4 hours to remove any potential organic materials that may interfere with phenanthrene sorption to silica particles. To initiate sorption experiments, 20 mL of the phenanthrene stock solution (874 ug/L) was added to 0.2 grams of silica particles (18Å, 76Å, and 202Å) that had been placed inside the glass tube. Thus, the water-to-solids ratio was equal to 100 in all sorption experiments.

During the sorption studies, the centrifuge tubes were tightly capped, covered with paper towels to protect against potential photo-oxidation of phenanthrene by fluorescent laboratory lights, and placed on a modified rock roller (Model NF-1, Lortone Inc.) @100 to 250 rpm for mixing. At specified
sampling times, the tubes were taken from the rollers and centrifuged at 4000 rpm (2960g) for 5 minutes. A supernatant sample (0.1mL) was taken from each tube and analyzed for phenanthrene as outlined in the Phenanthrene Analysis section. The glass tubes were again tightly capped and placed back on the roller until the next sampling event. For the “time zero” measurements, the glass tubes were briefly mixed manually (i.e., they were not put on the roller) and placed immediately in the centrifuge. In this case, the total time for mixing, centrifugation, and sampling took ca. 15 minutes.

A detailed mass balance calculation was carried out for each tube to determine the sorbed-phase phenanthrene concentration at termination of the sorption experiments. Thus, the mass of sorbed phenanthrene was computed as the initial mass of phenanthrene added to each tube minus any phenanthrene that was either removed via sampling or remained dissolved in the supernatant. An acetonitrile extraction of tubes and septa used in sorption experiments indicated that the mass of phenanthrene sorbed to glass walls or septa is negligible (< 0.1 ug per tube, or equivalently <5% (wt) of the initial mass of phenanthrene). In addition, control experiments carried out with tubes containing no silica particles confirmed that the observed decreases in aqueous phenanthrene concentrations are due to sorption and are not caused by biodegradation, photo-oxidation, or volatilization.

2.4 Incorporation of Phenanthrene into Silica Particles Using Non-Aqueous Methods

In the aqueous sorption methods outlined above, the pores of all silica particles were filled with water. In order to determine whether the presence of water has any significant effect on phenanthrene sequestration, we used the following three different “non-aqueous” methods to incorporate phenanthrene into the internal pore space of the silica particles in the absence of pore water. (Note: As outlined above, all silica particles were subjected to a specific hydration procedure that assured the elimination of all physi-sorbed water from the pores.)

2.4.1 Incorporation of Phenanthrene into Silica Particles Using Supercritical Carbon Dioxide

Phenanthrene (Aldrich, zone-refined) was used for all supercritical fluid (SCF) loading experiments. The SCF system consisted of a Dionex model SFE-703 supercritical extraction instrument that was modified to circulate supercritical carbon dioxide in a closed loop (Riley et al., 2001). Included in the closed loop system was a high-pressure stainless steel vessel (10 mL,
Keystone Scientific) used to dissolve the phenanthrene in supercritical carbon dioxide. A second vessel (10 mL) in the system contained the silica particles. An Eldex model B-100-S HPLC pump was used to circulate the supercritical solution through the closed loop system and a Shimadzu UV-2401PC spectrophotometer equipped with a custom-mounted high-pressure flow cell (Shimadzu SPD-M6A) was employed to monitor real-time changes in phenanthrene concentrations during loading.

The general procedure for the SCF incorporation of phenanthrene into the silica particles was as follows. Before starting the experiment, the two high-pressure vessels were removed from the system and loaded with appropriate amounts of silica (substrate vessel) and phenanthrene (sorbate vessel). After re-installing the vessels, the SFE-703 oven chamber was maintained at 30°C and the system was pressurized at 300 atm (4409 psi) with SFE-grade carbon dioxide. The supercritical carbon dioxide was then pumped through the sorbate vessel until all phenanthrene had been dissolved in CO2 as indicated by a stabilized UV absorbance reading. Following baseline stabilization, valves were switched to allow the phenanthrene containing supercritical CO2 to contact the silica particles in the substrate vessel. The solution was pumped through the substrate vessel for four hours. This contact time was long enough to ensure that phenanthrene in the circulating supercritical CO2 reached a steady-state concentration as indicated by a stabilized UV absorbance measurement. The circulating pump was then turned off, a valve was switched to depressurize the system, and the loaded silica particles were removed for use in aqueous desorption experiments. Using these procedures, rehydrated 21Å, 66Å and 202Å silica particles were loaded with phenanthrene resulting in final solid-phase concentrations of 2.9 ug/g, 5.7 ug/g, and 2.0 ug/g, respectively.

2.4.2 Incorporation of Phenanthrene into Silica Particles Using Solvent Soaking

Approximately 0.8 grams of rehydrated 21Å, 66Å and 202Å particles were each fully submerged in 10 mL methylene chloride containing 20 ug, 40 ug and 14 ug dissolved phenanthrene, respectively. The resulting slurry was mixed on a shaker table (@ 100 rpm) for four hours. A gentle stream of nitrogen was then used to evaporate the solvent while stirring the slurry periodically with a spatula until a constant weight was reached (ca. 3 hours). A subsample of the phenanthrene loaded particles was taken and analyzed for phenanthrene as outlined below. The solid-phase phenanthrene concentrations for the 21Å, 66Å and 202Å particles were 8.5 ug/g, 11.0 ug/g, and 1.1 ug/g, respectively (Note: g dry weight). All silica particles
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were placed into a freezer (-20 °C for 5 days) to immobilize the phenanthrene until the initiation of desorption experiments.

2.4.3 Incorporation of Phenanthrene into Silica Particles Using Solvent Spiking with Aging

This method involves the spiking of a small volume of solvent containing phenanthrene onto silica particles and the subsequent addition of water for moisture adjustment. It should be recognized that this phenanthrene incorporation procedure is a hybrid between a non-aqueous spiking procedure and an aqueous sorption experiment. It is most likely that phenanthrene is deposited on the outside surfaces of the silica particles during the spiking procedure while the subsequent addition of moisture will not only fill the pores with water but cause the dissolution of phenanthrene which in turn enables the diffusion along pores and aqueous sorption on silica surfaces. Despite the mechanistic complexity of this phenanthrene incorporation procedure, it was decided to evaluate this method because it is the "aging" technique that is most commonly reported in the literature (Chung and Alexander, 1998; Nam and Alexander, 1998; Hatzinger and Alexander, 1995; Kelsey and Alexander; 1997).

Approximately 1 gram of rehydrated 21Å, 66Å and 202Å particles were each spiked with 10µL methylene chloride containing 3 ug, 6 ug, and 2 ug dissolved phenanthrene, respectively. In addition, ca. 1 gram of dry 18Å particles were spiked with 10µL methylene chloride containing 110ug dissolved phenanthrene. All spiked silica particles were mixed every 30 minutes with a spatula for a total duration of four hours. A gentle stream of nitrogen was then used to evaporate the solvent while stirring the slurry periodically until a constant weight was reached (ca. 30 minutes). At this point, an aqueous solution containing 2% (wt) sodium azide was added to the spiked silica particles in order to adjust the moisture content to ca. 80% of the field capacity. The silica particles were subsequently mixed with a spatula for ca. 15 minutes and then transferred to an amber glass jar. The jar was tightly capped and stored in the dark at room temperature until the initiation of desorption experiments. The 21Å, 66Å and 202Å particles were aged in this manner for 61 days whereas the 18Å particles were aged for 100 days.

At the end of the aging period, the solid-phase phenanthrene concentrations were determined as outlined below. For the 18Å, 21Å, 66Å and 202Å particles, the phenanthrene concentrations were found to be 89 ug/g, 1.0 ug/g, 2.8 ug/g, and 0.9 ug/g, respectively. The corresponding moisture contents (g water/g moist silica) for these particles were 0.65, 0.66, 0.62, and 0.55, respectively. Finally, non-porous silica beads were spiked