PRINCETON GEOSCIENCE

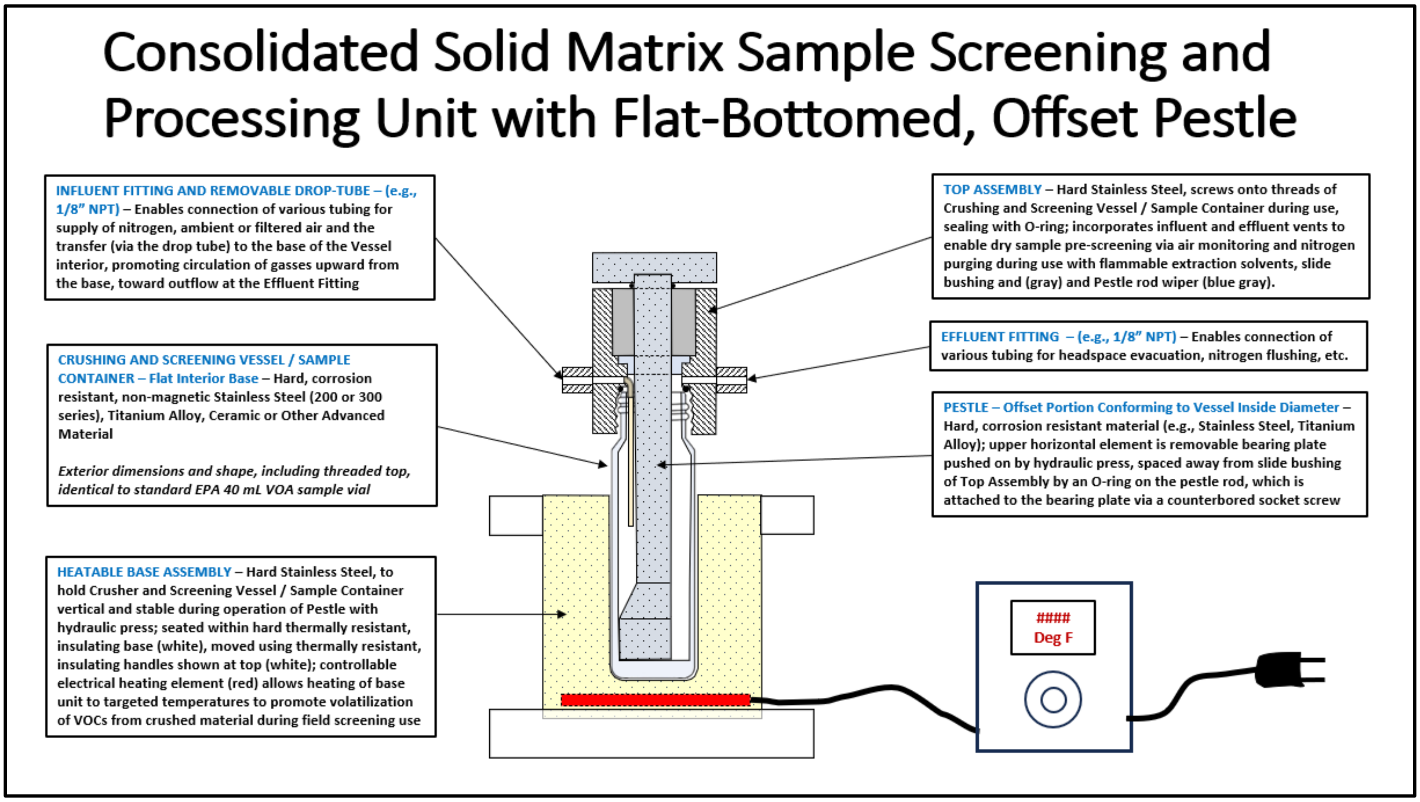
ROCK$^{\text{VOC®}}$ SYSTEM

DESCRIPTION & STATEMENT OF USEFULNESS

609-279-0008
princetongeoscience.com
209 Nassau St.
Princeton, NJ

ROCK$^{\text{VOC}}$™ System – Description and Statement of Usefulness
July 31, 2024

The ROCK$^{\text{VOC}}$™ System is a convenient new system for controlled screening and sampling to accurately measure volatile organic compound concentrations in rock, concrete and other consolidated, solid matrices; limiting or eliminating VOC loss inherent to other standard methods and equipment currently in use. It is further described under US patent number US 11,860,070 B1, issued January 2, 2024. A schematic illustration of the system is provided below and photos of the prototype are included on the following pages. A copy of the patent with its detailed description is attached.



Consolidated Solid Matrix Sample Screening and Processing Unit with Flat-Bottomed, Offset Pestle

INFLUENT FITTING AND REMOVABLE DROP-TUBE – (e.g., 1/8″ NPT) – Enables connection of various tubing for supply of nitrogen, ambient or filtered air and the transfer (via the drop tube) to the base of the Vessel interior, promoting circulation of gasses upward from the base, toward outflow at the Effluent Fitting

CRUSHING AND SCREENING VESSEL / SAMPLE CONTAINER – Flat Interior Base – Hard, corrosion resistant, non-magnetic Stainless Steel (200 or 300 series), Titanium Alloy, Ceramic or Other Advanced Material

Exterior dimensions and shape, including threaded top, identical to standard EPA 40 mL VOA sample vial

HEATABLE BASE ASSEMBLY – Hard Stainless Steel, to hold Crusher and Screening Vessel / Sample Container vertical and stable during operation of Pestle with hydraulic press; seated within hard thermally resistant, insulating base (white), moved using thermally resistant, insulating handles shown at top (white); controllable electrical heating element (red) allows heating of base unit to targeted temperatures to promote volatilization of VOCs from crushed material during field screening use

TOP ASSEMBLY – Hard Stainless Steel, screws onto threads of Crushing and Screening Vessel / Sample Container during use, sealing with O-ring; incorporates influent and effluent vents to enable dry sample pre-screening via air monitoring and nitrogen purging during use with flammable extraction solvents, slide bushing and (gray) and Pestle rod wiper (blue gray).

EFFLUENT FITTING – (e.g., 1/8″ NPT) – Enables connection of various tubing for headspace evacuation, nitrogen flushing, etc.

PESTLE – Offset Portion Conforming to Vessel Inside Diameter – Hard, corrosion resistant material (e.g., Stainless Steel, Titanium Alloy); upper horizontal element is removable bearing plate pushed on by hydraulic press, spaced away from slide bushing of Top Assembly by an O-ring on the pestle rod, which is attached to the bearing plate via a counterbored socket screw

Rock and concrete samples submitted to labs for VOC analysis are typically fragmented and loosely contained in jars. As such, they experience unquantified VOC loss into the headspace of the jars during transit and holding. Prior to analysis, the sample chunks must be crushed to increase surface area as an aid to extraction, prior to placement in extraction solvent. Additional unquantified VOC loss takes place with the crushing, which creates heat and increases sample surface area hundred- to thousand-fold.

This combined unquantified VOC loss is accepted under current analytical protocols, perhaps deemed negligible or, from a practical standpoint (in the absence of better methods), not worth further evaluation. For example, concrete that will be recycled may undergo crushing prior to re-use analogous to the crushing of the sample, releasing its VOC load at that time rather than later as a recycled product. And there are no current or proposed remediation standards for VOCs in rock; such analyses are usually undertaken in support of matrix-diffusion hydrogeological studies utilizing specialized equipment of limited availability which, while improved relative to standard approaches, still involves VOC loss inherent to crushing with exposure to air.

But it is possible to do better and there are historical and emerging case-specific reasons to try. Many recall when soil samples for VOCs were collected into standard VOA vials, inevitably with voids left into which VOC loss took place. And indoor air samples were commonly collected into plastic film bags. But monitoring needs have evolved as new and more protective remediation standards are set and with increasing awareness of contaminant fate and transport (e.g., vapor intrusion). So, we have better ways to obtain soil samples for VOCs and to sample indoor air today.

ROCK$^{\text{VOC}}$™ – Crusher and Sample Vial Assembly

U.S. Patent No. 11,860,070

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And improved approaches also are warranted for characterization of VOCs in consolidated, solid matrices such as rock and concrete. A few case examples may illustrate this.

- For concrete characterization, consider the scenario where the material will stay in place, such as the floor beneath a former drycleaning facility. There have been cases where subsurface vapor intrusion was thought to be the source of indoor air impacts, but a subsurface source was not found. If traditionally collected and analyzed concrete floor samples also fail to detect a VOC source, are those methods, which entail unquantified VOC loss, really reliable?
- Many areas have little or no soil above near-surface bedrock. In these settings, the solid matrix within which most of a contaminant discharge mass resides is typically not sampled during most remedial investigations, despite functioning as the reservoir for ongoing impacts to groundwater, soil vapor and indoor air. Whereas, if the extent of impacts within that rock matrix could be reliably determined, active remediation could in many cases be both feasible and cost-effective. An example is rippable shale, present at- or near-surface in much of industrialized Central NJ, precluding collection of soil samples. In one recent discussion, a developer who planned to construct a child-care facility on a former drycleaning site had a keen interest to identify and remove any vapor intrusion source material.

Digging out some additional rock, coincident with construction, was not going to be a problem for them. So, rock as a matrix in need of remediation is a reality at many sites, irrespective of the absence of any established "rock matrix remediation standards".

- Rock matrix sampling for hydrogeological studies is to date a highly specialized activity, technically out of reach for most remediation practitioners. But a readily deployed and accurate approach would enable characterization needed to estimate not only diffusion into rock matrices adjacent to fractures transporting VOCs in support of Technical Impracticability evaluations (a common current use) but also to support informed application of in-situ remediation efforts. As noted in NJDEP remediation guidance, such sampling can inform evaluation of both feasibility (beforehand) and effectiveness (post-remediation) of in-situ remediation.
- State promulgated groundwater remediation standards for some chlorinated VOCs have been lowered into the parts per trillion (ppt) range (e.g., New Jersey Ground Water Quality Standard of 35 ppt for vinyl chloride). With such compliance challenges, remedial investigations will necessarily become more complex, necessitating completion of rock matrix diffusion/back diffusion evaluations to discern to what extent the plume-area aquifer matrix itself may be a sink for, or contributing source of, ppt-level groundwater impact.

ROCK^{VOC}™ System – With Removable Heater and Temp Control

U.S. Patent No. 11,860,070 www.princetongeoscience.com

Sampling with the ROCK^{VOC} System begins the same as traditional rock or concrete sampling with coring and selection of representative sub-sample material to be analyzed from the core. System elements are used to crush the sub-samples under methanol, water or other extraction solvent, directly into specially manufactured 316 Stainless Steel 40-mL VOA vials that are also part of the System. The vials are compatible with standard laboratory auto-samplers, allow magnetic stir bars to be used and are recyclable and potentially re-usable. Standard, plastic VOA vial caps with silicone septa are used to seal the samples for shipment to the laboratory. The unquantified VOC loss inherent to traditional methods is thus eliminated with the ROCK^{VOC} System, through processing (crushing) and shipment within extraction solvent and wholly without exposure to air.

Crushing is accomplished using a capping unit (top assembly), specially designed crushing pestle, and base unit which are placed within a small benchtop hydraulic press or manually operated arbor press. The capping unit has threaded fittings to accommodate connection of influent and effluent tubing, enabling introduction and removal of gasses and fluids from the headspace of the stainless steel vial. The stainless steel base unit has a bored hole that accepts a thermal insertion probe connected to an electronic temperature controller. The thermal probe, in combination with the influent/effluent connections to the capping unit and field or lab screening equipment, enable highly representative, temperature-controlled VOC screening of rock and concrete samples. For example, an investigator could choose a temperature at or near the boiling point of a targeted VOC to expose all screening samples to, each for a similar time duration, prior to or after crushing. Within the enclosed, heated and controlled space of the ROCK^{VOC} System, screening can easily be conducted in a way that eliminates variables common to informal field screening approaches typically employed.

The ROCK^{VOC} System can be owned and operated by environmental consultants, laboratories or both. With the lab as owner, the specialized stainless steel VOA vials of the system are supplied to field crews, pre-filled with methanol, water or other extraction solvent (as done currently with soils), and the lab performs the crushing in the laboratory upon receipt of the vials returned by the field crew with rock sub-samples added. In this situation, field crews ship the vials within foam holders, kept upright, so that the rock sub-samples always remain below the level of the extraction solvent in the vials. This action ensures that the sub-sample always maintains contact with extraction solvent, limiting the potential for partitioning of VOCs into the headspace of the vial.

When environmental consultants own and operate the ROCK^{VOC} System, they have the option of conducting field screening, and sub-samples collected in the field are crushed into extraction solvent immediately after collection from the core. Although the environmental consultant field crew could potentially be responsible for adding reagent grade extraction solvent to the specialized stainless steel VOA vials, that function may likely be better provided by a laboratory or sampling container vendor. As such, entities other than just the environmental consulting firm will own at least portions of the ROCK^{VOC} System.

The Rock^{VOC} System addresses important shortcomings in traditional methods, offering improved reliability for VOC analysis, enabling more informed remediation decisions. It can also offer a highly specialized, accurate approach that may otherwise be out of reach for many remediation practitioners.

At present, we are seeking feedback from potential users and exploring options for making the system commercially available. If you have comments or potential interest as a research or commercial collaborator, please contact me. Thank you for taking time to review this information.

Jim Peterson, PG, LSRP - President

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Princeton Geoscience, Inc.
209 Nassau Street
Princeton, NJ 08542
Mobile 609-240-8650
jim@princetongeoscience.com
www.princetongeoscience.com