



Colloidal Activated Carbon (CAC) Groundwater Sampling Guidance Document

Best Practices for Collecting Samples Following CAC Applications





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Introduction



Figure 1 - Image of groundwater with suspended colloidal carbon poured into a bucket. The groundwater appears quite dark but once tested was safe to send to an analytical laboratory for accurate samples.

During injection, PlumeStop® or PetroFix® may flow into nearby monitoring wells, as evidenced by sampling the well and finding the watercolor to be black. This occurrence is not uncommon and is simply the result of the colloidal activated carbon (CAC) of the product transported through the natural flux zones of the aquifer. In most situations, the observance of PlumeStop or PetroFix in monitoring wells, or even in collected soils samples, is desired because this tells us that we are getting proper distribution at a site. At most sites, groundwater treated with PlumeStop or PetroFix will clarify to safe sampling concentrations by the first quarter after injection.

Most sites can be sampled by 3 months post application even if darkened water is present because CAC concentrations have fallen to safe sampling concentrations.

REGENESIS' rule-of-thumb is to sample your site no earlier than 3 months post application.

However, we recognize that some situations may require sampling sooner or at a minority of sites, PlumeStop or PetroFix may stay suspended longer than normal (> 3 months) at concentrations not considered safe for sampling. This document is intended to give our customers a comprehensive evaluation of techniques to help attain good groundwater samples at any phase of the project.

Table 1 and Table 2 (next pages) summarize best practices to prevent PlumeStop or PetroFix from interfering with commercial analytical methods and the specific techniques are described in order in this document.



Figure 2 - Image of groundwater samples in a VOA bottle and exactly at 100 mg/L with some visible light coming through. Anything at this concentration or lower is safe to send to an analytical laboratory.

PetroFix can be safely and accurately sampled at concentrations below 100 mg/L which is still dark. A person can just see through 100 mg/L of colloidal carbon in groundwater when in a 40mL VOA bottle. Very little colloidal carbon is needed to darken the water. **DO NOT** evaluate the ability to sample with only visual observations of groundwater in large plastic drink containers, buckets, etc. because larger volumes of PetroFix appear darker than in 40mL VOAs.

Always use 40mL VOA bottles to begin the evaluation of sampling appropriateness. Field concentration test kits are available from REGENESIS for PlumeStop projects or shipped with each PetroFix order.

Table 1: Guidance for Sampling <Q1

Category	Time Frame	Technique	Importance
Prevent PlumeStop® or PetroFix® in samples	Prior or during application	Standard well sampling practice ¹	Recommended
		Over purge wells or points during sampling	Recommended
		Rehabilitate and desilt wells if low-flow sampling cannot be used	Recommended
		<i>In situ</i> flocculation, CaCl ₂ parking around wells	Recommended
		Passive diffusion bags (collect baseline before injection) ²	Recommended
		Install and develop temporary sentinel piezometer	Optional
Treat PlumeStop® or PetroFix® in samples	After Application	Standard well sampling practice ¹	Recommended
		Over purge wells or points during sampling	Recommended
		Rehabilitate and desilt wells if low-flow sampling cannot be used	Recommended
		<i>In situ</i> flocculation, CaCl ₂ parking around wells	Recommended
		Wait and sample when colloidal carbon <100 ppm	Optional
		Passive diffusion bags (PDBs) ²	Optional
		Filter (<0.4 micron) ³	Optional
		VOA sample clarification with alum ⁴	Optional

1. Remove tubing and bailers between events, keep wells watertight, use standard development methods, allow post-purge recovery time, gently lower bailers/meters, and don't allow any sampling equipment to touch the bottom of the well.
2. Take baseline before CAC injection; Not all analytes can be measured with PDBs (see acceptable analytes on page 14)
3. State and analyte-specific – would need to be pre-approved by state and client.
4. Modified lab technique would need to be pre-approved by state and client.

Table 2: Guidance for Sampling >Q1

Category	Time Frame	Technique	Importance
Prevent PlumeStop® or PetroFix® in samples	Prior or during application	Standard well sampling practice ¹	Recommended
		Over purge wells or points during sampling	Recommended
		Rehabilitate and desilt wells if low-flow sampling cannot be used	Recommended
		<i>In situ</i> flocculation, CaCl ₂ parking around wells	Optional
		Passive diffusion bags (collect baseline before injection) ²	Optional
		Install and develop temporary sentinel piezometer	Optional
Treat PlumeStop® or PetroFix® in samples	After Application	Standard well sampling practice ¹	Recommended
		Over purge wells or points during sampling	Recommended
		Rehabilitate and desilt wells if low-flow sampling cannot be used	Recommended
		<i>In situ</i> flocculation, CaCl ₂ parking around wells	Recommended
		Wait and sample when colloidal carbon <100 ppm	Recommended
		Passive diffusion bags (PDBs) ²	Optional
		Filter (<0.4 micron) ³	Optional
		VOA sample clarification with alum ⁴	Optional

1. Remove tubing and bailers between events, keep wells watertight, use standard development methods, allow post-purge recovery time, gently lower bailers/meters, and don't allow any sampling equipment to touch the bottom of the well.
2. Take baseline before CAC injection; Not all analytes can be measured with PDBs (see acceptable analytes on page 14)
3. State and analyte-specific – would need to be pre-approved by state and client.
4. Modified lab technique would need to be pre-approved by state and client.

Well Housekeeping

Standard Practices



Figure 3 - Image of tubing left in well and coated with PetroFix

At various remediation sites, the age of the wells or poor maintenance practices can affect PlumeStop or PetroFix sampling. One example is the accumulation of settled fines in the bottom that may have colloidal carbon attachment and that churn up or re-suspend in a monitoring well during normal groundwater sampling activities. The resuspension of darkened fines is often mistaken for *in situ* colloidal carbon suspensions. This section is intended to provide remediation practitioners with solutions to overcome sampling issues related to monitoring wells during groundwater sampling.

Good well-keeping practices go a long way in ensuring good samples are collected from monitoring wells. Here is a list of minimum practices that should be observed for every site.

1. Keeping monitoring well plugs (aka, J-Plugs), monitoring well covers, bolts, and gaskets water-tight. Loosely fitting plugs and well covers permit stormwater and sediments to enter the monitoring well.
2. Remove all tubing, bailers, and rope from monitoring wells after each sampling event (i.e., dedicated sampling tubing should not be used since colloidal carbon can coat tubing).
3. Utilize industry-wide well development and purging methods before sampling.
4. After purging, allow wells proper recovery time before sampling. Where there are concerns with colloidal carbon affecting the sampling, we recommend waiting at least 4 hours, but up to 24 hours where necessary, between purging and sampling.
5. Sampling should be performed by gently lowering the bailer into the well.
6. Water level meter probes should be gently lowered into the well.
7. Bailers, tubing, or sampling equipment should not contact the bottom of the well at any time during the sampling process

Over Purging



Figure 4 - Dark water pulled from a well and indicating that over purging or other well rehabilitation may be necessary.

Where colloidal carbon is present near a monitoring well, over purging may help to restore the monitoring well to a representative sampling point. Typically, purging conducted before sampling will remove approximately 1-3 well pore volumes or about 2-5 gallons from a 2" internal diameter (ID) monitoring well. During over purging, 5-10 pore volumes or more are recommended to be removed. Over purging can be completed using a downhole well pump or a bailer. The colloidal carbon in the monitoring well should begin to decline during the purging process. If over purging is effective, purge water may still be relatively turbid; however, the dark carbon color should fade and become gray or translucent.

The purge water in a 5-gallon bucket can be misleading (see Figure 1) and is not recommended to determine effectiveness because turbidity can affect the color. To determine if the over purging is adequate, we recommend looking at occasional samples of the purge water in a clear 40 ml vial or similar clear containers. We recommend waiting at least 24 hours before performing groundwater sampling upon over purging. As a cautionary note, we recommend stopping if the purge water from the well during the over purging process does not appear to become clearer after ten pore volumes. We also recommend stopping if over purging needs to be conducted on more than two events. Alternatively, the well may require rehabilitation, or a Calcium Chloride flush, as discussed in later sections.

Well Rehabilitation

In some cases, standard housekeeping practices and over purging are ineffective because there are more significant problems with the monitoring well itself. Occasionally, fine silts can build up at the base of the well screen and surrounding well pack. These fine silts are often coated with colloidal carbon following a PlumeStop or PetroFix injection, and these particles can cause a persistent problem for groundwater sampling. Being mobilized by the typical well purging and sampling processes, these fine silts can carry contaminants into the monitoring well, otherwise not present in the dissolved phase groundwater remediation with PlumeStop. For the scenario just described, well rehabilitation may be the solution.

Surge blocks, well pumps, and vac-trucks are all equipment options for well rehabilitation. Whether a well pump or vac truck is used is up to the prescriber. The process to rehabilitate a well can take between 1-4 hours, depending upon the severity of the problem and the size of the well. Each of these methods and their effectiveness are discussed below.

A vac truck is ideal where a significant amount of silt and sediment is present on the bottom of a well. Vac trucks can place a stinger tube down the well and rapidly remove the silt and sediment, along with purge water. Vac trucks are limited in subsurface reach and, depending upon barometric conditions, generally struggle to draw water/sediment from deeper than 35 feet.

A downhole well development pump can be used for deeper wells or where a vac truck is not suitable. Not all downhole pumps effectively remove sediments at the base of a well, and the process can damage some. It's best to make sure to know how much sediment might be present at the bottom of the well and select the right pump for the job.

Along with extracting purge water and sediments, surging the well with a surge block is highly recommended because it will enhance the quality of the well rehabilitation. The surging process quickly moves water in and out of the well screen, reordering and recompacting the filter pack. Surge blocks for shallow 2" diameter wells can be easily operated by hand. A well development truck with a lift might be needed for deeper wells or larger diameter wells to work the surge block. It's ideal to alternate the surging process with purging using the development pump or vac truck. Adding clear potable water down the well will also help enhance the well rehabilitation process, especially at sites where the hydraulic conductivity may be low, and the wells don't naturally produce much water.

As described in Over Purging methods, using a clear glass 40 ml vial to view the water quality changes during well rehabilitation is highly recommended. Upon completing the well rehabilitation process, it is advisable to flush the well with a CaCl_2 solution (see next section for specific recommendations). The CaCl_2 flush will help the residual colloidal carbon flocculate and remain in the aquifer.

In Situ Flocculation for Aquifer Clarification

Some practitioners wish to prevent colloidal suspensions near key wells because they know they want to sample soon after an injection or increase the likelihood that groundwater will be in a safe sampling range at any point post-injection. With extra work, practitioners can “park” PlumeStop or PetroFix colloidal suspensions through chemical flocculation techniques. The primary infield flocculation technique that we recommend is the injection or flooding of Calcium Chloride (CaCl_2) **separate from the PetroFix application**. CaCl_2 can flocculate and destabilize PlumeStop or PetroFix which results in improved aquifer clarification within a few days to weeks.

Here are usage examples of parking:

- Flood CaCl_2 into monitoring wells and a very limited distance from those wells immediately after PetroFix applications to “park” colloidal carbon. This accelerates the clarification of the aquifer near those wells and aids in sampling.
- Inject CaCl_2 post-injection into the formation in critical sampling areas or areas where one wishes to minimize the initial flux of PetroFix.

PLEASE NOTE: the use of CaCl_2 should only be used post-injection and carefully.

DO NOT CO-MIX or CO-INJECT CaCl_2 with PlumeStop or PetroFix and only apply in a separate application.

Site-wide parking of PlumeStop or PetroFix will deleteriously affect the distribution of the product. One should decide if they want to use, or have the option, of using CaCl_2 so that it can be included in the Underground Injection Control (UIC) application.

Calcium Chloride (CaCl₂) Parking

Health and Safety



Figure 5 - Example of 83% CaCl₂ flakes.

CaCl₂ is non-toxic and, generally, is safe to use when handled properly. However, CaCl₂ can generate enough heat to cause burns and equipment damage when mixed in water at high concentrations. This section is intended to give technical and safety guidance on CaCl₂ use as a PlumeStop or PetroFix parking agent. To that end, the following mixing threshold is established, which will alleviate most safety concerns.

- CaCl₂ should never be mixed at a concentration above 1 lb CaCl₂/ gal water.
- CaCl₂ should always be added to the total volume of water to minimize heat generation and ensure higher concentrations are not created.
- Please read and follow all material handling and safety instructions on supplier packaging.

Calcium Chloride Mixing

Standard Calcium Chloride Solution:

- 0.5 lb CaCl₂/gallon of water equivalent to 150 lb per 300-gallon mix tank.
- The purity of the calcium chloride should be 85% or higher.
- Calcium chloride flakes are recommended over pellets because they will dissolve faster.
- The volume of calcium chloride solution to be injected should be equivalent to 4 saturated well volumes.

Recommended Procedure for Applying Calcium Chloride Well Flush



Figure 6 - Client performing a well flush.

1. Calculate the saturated well volume:

$$V = \pi r^2 \text{ saturated length where "r" is the radius of the borehole.}$$

2. Multiply this volume by 4. Four well volumes will provide a good flush of the surrounding aquifer material.
3. Use a 55-gallon drum or supplementary poly tank if available and fill it with the requisite volume.
4. Slowly add the CaCl_2 amount calculated above and mix with a drill mixer. Mix until all CaCl_2 is dissolved.
5. Secure expansion plug with bypass (see table and links below) to well and attach hosing and pump. Expansion plugs with bypass are the simplest way to pump with pressure into wells of various sizes.
6. Make sure hosing and fittings match and are rated for appropriate psi tolerance. Rating should be greater than the max PSI of the pump.
7. Pump recommendations: pool pump, trash pump, air diaphragm pump, Hydracell, Moyno.
8. See expansion plug information and links below.
9. Pump the required amount of CaCl_2 mixture into the well.

REGENESIS recommends flushing the well at 3-5 gallons per minute or higher without going above a pressure equivalent to 1 psi per foot of well depth.

Expansion Plugs with Bypass



Use the bypass tube on these plugs to add air, water, and other fluid to your pipeline or to relieve pressure while testing. Maximum air back pressure is the amount of pressure a plug can withstand without moving. Maximum water back pressure refers to the pressure resulting from the height of the water above the plug.

Size	For Pipe ID	Max. Back Pressure		O'all Ht.	Temp. Range, °F	Material		Bypass			Each	
		Air, psi	Water, ft. of head			Seal	Bypass Cap	Pipe Size	Thread Type	Gender		
Style C Iron Stem												
1/2	0.47"-0.50"	Not Rated	46	4 1/4"	30° to 150°	Natural Rubber	Metal	1/16	NPT	Male	2644K15	\$18.37
3/4	0.72"-0.75"	Not Rated	46	5 1/8"	30° to 150°	Natural Rubber	Metal	1/8	NPT	Male	2644K16	21.13
1	0.97"-1.00"	Not Rated	46	5"	30° to 150°	Natural Rubber	Metal	1/4	NPT	Male	2644K18	22.70
2	1.75"-2.00"	Not Rated	34	10 3/8"	30° to 150°	Natural Rubber	Metal	1/2	NPT	Male	2644K21	18.37
3	2.75"-3.00"	Not Rated	34	10 1/2"	30° to 150°	Natural Rubber	Metal	1/2	NPT	Male	2644K22	23.87
4	3.63"-4.00"	Not Rated	23	10 1/8"	30° to 150°	Natural Rubber	Metal	1/2	NPT	Male	2644K23	27.60
5	4.50"-5.00"	Not Rated	23	12 5/8"	30° to 150°	Natural Rubber	Metal	1/2	NPT	Male	2644K24	57.74
6	5.50"-6.00"	Not Rated	23	12 1/2"	30° to 150°	Natural Rubber	Metal	1/2	NPT	Male	2644K25	40.69
8	7.50"-8.00"	Not Rated	4	14 3/4"	30° to 150°	Natural Rubber	Metal	1/2	NPT	Male	2644K26	91.31
10	9.50"-10.00"	Not Rated	4	14 7/8"	30° to 150°	Natural Rubber	Metal	1/2	NPT	Male	2644K31	132.05
12	11.50"-12.00"	Not Rated	4	14 5/8"	30° to 150°	Natural Rubber	Metal	1	NPT	Male	2644K32	232.86

Expansion plug link: <https://www.mcmaster.com/expansion-plugs-with-bypass/expansion-plugs-with-bypass/>

Adapter link: <https://www.mcmaster.com/pipe-fittings/thick-wall-plastic-pipe-fittings-for-water/>

Passive Diffusion Bags



Figure 7 – Example passive diffusion sampler.

Passive diffusion bag (PDB) samplers can sample groundwater where PlumeStop or PetroFix is present because it cannot diffuse through these bags, so the groundwater collected will be free of PlumeStop or PetroFix.

The significant advantage of PDBs is the confidence that you can sample groundwater for VOCs at any time post-injection based on your desired sampling time frames and without interference from any suspended PlumeStop or PetroFix. Furthermore, according to the Interstate Technology Regulatory Council (ITRC), PDB sampling is cost-effective and a viable alternative to standard or low-flow purge and sample techniques for collecting volatile organic compound (VOC) data at monitoring wells. PDB samplers, typically low-density polyethylene bags filled with water, have been shown in multiple studies to provide accurate groundwater VOC measurements.

PDBs come in 1-to-4-foot lengths and are filled with clean water and hung into a well for a minimum of two weeks. PDB sampling is achieved as VOCs in groundwater diffuse through the wall of the bag and into the bag water, which eventually comes to equilibrium with the surrounding well water.



Figure 8 - Sample pulled from a PDB bag and free of suspended carbon.

PDB Implementation Tips

- **Take Baseline PDB samples before injection.** State regulatory agencies might ask you to compare PDB's to standard groundwater samples (i.e., low-flow sampling or bailing) to prove that they correlate. With this in mind, **we recommend you take baseline samples alongside PDB samples before any PlumeStop or PetroFix application, where time-critical monitoring will begin shortly afterward.**
- **Hang PDBs from the same vertical interval that you have historically sampled from.** To get accurate groundwater VOC values compared to past or present results, it is critical to hang a PDB from the same vertical sampling interval in a monitoring well used from other sampling efforts. This is because groundwater contamination can stratify in an aquifer, and a plume may have different vertical groundwater concentrations intersecting a monitoring well screen. Those differences can be detected by PDBs hanging in a well. This phenomenon also is true for standard low-flow sampling. For example, at a hydrocarbon site, it is common for higher groundwater concentrations to be present in the aquifer near the surface of the aquifer where the smear zone resides versus at the bottom of a well where it is not likely present. However, multiple studies show that when PDBs are hung in the same zone that you usually collect standard or low-flow purge samples, they will correlate and provide accurate VOC concentrations.

PDB Analyte Limitations

Please note that PDBs are unsuitable for monitoring certain organic compounds (MTBE, TBA) or ionic species (nitrate, sulfate). They are currently only effective for early VOC measurements and not the entire suite of parameters you may use need to sample. Non-VOC parameters may need to be measured using standard sampling techniques after the PlumeStop or PetroFix suspension has mostly been clarified from groundwater. We recommend you become familiar with available resources on PDBs (the first ITRC FAQ listed left) and guidelines from the suppliers of PDBs.

Here are some helpful links:

PDB References

- ITRC FAQ On PDBs And List Of VOC's Showing Good Sample Correlation:
<https://www.itrcweb.org/Documents/PDBFAQs2.pdf>
- Users Guide for Polyethylene-Based PDBs:
<https://www.itrcweb.org/GuidanceDocuments/DSP-1a.pdf>
- USEPA Clu-In.org Guidance On Diffusion Samplers:
[https://clu-in.org/characterization/technologies/default.focus/sec/Passive\(nopurge\)Samplers/cat/DiffusionSamplers/](https://clu-in.org/characterization/technologies/default.focus/sec/Passive(nopurge)Samplers/cat/DiffusionSamplers/)

PDB Suppliers

- ALS:
<https://www.alsglobal.com/en-us/services-and-products/environmental/sampling/passive-diffusion-bags-pdbs>
- EON Products Incorporated:
<https://store.eonpro.com/store/c/71-Water-Sampling-Pumping.aspx>

Compounds Showing Good Correlation in Laboratory Tests (Average differences in concentration of 11 percent or less between diffusion sampler water and test vessel water)			
Benzene	1,3-Dichlorobenzene	Naphthalene	
Bromodichloromethane	1,4-Dichlorobenzene	1,1,2,2-Tetrachloroethane	
Bromoform	Dichlorodifluoromethane	Tetrachloroethene	
Chlorobenzen	1,2-Dichloroethane	Toluene	
Carbon tetrachloride	1,1-Dichloroethene	1,1,1-Trichloroethane	
Chloroethane	cis-1,2-Dichloroethene	1,1,2-Trichloroethane	
Chloroform	trans-1,2-Dichloroethene	Trichloroethene	
Chloromethane	1,2-Dichloropropane	Trichlorofluoromethane	
2-Chlorovinyl ether	cis-Dichloropropene	1,2,3-Trichloropropane	
Dibromochloromethane	Dibromochloromethane	Vinyl chloride	
Dibromomethane	trans-1,3-dichloropropene	Total xylenes	
1,2-Dichlorobenzene	Ethyl benzene		

Compounds Showing Poor Correlation in Laboratory Tests (average differences in concentration greater than 20 percent between diffusion sampler water and test vessel water)			
Acetone*	Methyl-tert-butyl ether	MIBK*	Styrene

Source: Compounds tested under laboratory conditions for use with passive diffusion bag samplers (Vroblesky and Campbell, 2001).

*T.M. Sivavec and S. S. Baghel, 2000, General Electric Company, written communication.

Table 3 - ITRC FAQ document on passive diffusion bags analytes showing good correlation with PDBs

Install and Develop Sentinel Piezometers

Sentinel piezometers can be installed to detect the spread of PlumeStop or PetroFix before reaching a monitoring well or another sensitive receptor. By performing real-time observations of the sentinel well, the remediation practitioner should have sufficient time for a response to be implemented during the injection to maximize or minimize the appearance of PlumeStop or PetroFix at that well or receptor. The main reasons for using sentinel wells are to fine-tune PlumeStop or PetroFix dilutions to achieve proper ROI when there are not enough nearby monitoring wells for the same observations, to detect that lateral or vertical spread to unwanted zones or receptors, and finally, in some circumstances to aid in the minimization of PlumeStop or PetroFix in nearby wells to aid in early sampling.

The location of the piezometer is determined based on the existing grid or barrier injection layout, the groundwater flow direction and the groundwater flow velocity, and the need for the sentinel well. If sentinel piezometers are installed, we recommend that multiple units be installed to measure spread at different locations. For example, multiple piezometers can help document sufficient distribution at the site while also helping to minimize spread at other sensitive areas.

Sentinel Wells to Monitor Distribution

If sentinel wells are used within a grid to manage and monitor the spread of PlumeStop or PetroFix, we recommend placing them equidistant within the grid and where monitoring well coverage is insufficient. While injections are being performed, these piezometers should be monitored for PlumeStop or PetroFix. Ideally, CAC concentrations of several hundred to several thousand mg/L should be observed if distribution and injection overlap is achieved. If such concentrations are not observed during the injection, the remediation practitioners should re-evaluate injection volumes (by increasing injection dilutions), boost pressure, re-evaluate injection tooling used, adjust the spacing, or a combination of all. More on this subject is discussed in the direct push application instructions for PetroFix www.petrofix.com/resources

Sentinel Wells to Minimize Distribution to a Critical Receptor

The remediation practitioner may seek to minimize suspended carbon's impact at a critical well or receptor. In the case of the monitoring well, groundwater results may be needed relatively soon after injection and it is essential to minimize the concentration of CAC flowing through and around that monitoring well. As a cautionary note, minimizing the spread of PlumeStop or PetroFix by using sentinel wells may interfere with the performance by limiting the beneficial spread of CAC at densities that would offer better performance. Please use sentinel wells and concentration adjustments judiciously.

We recommend that sentinel wells be placed 1 to 2 feet directly between injection points and the monitoring well where you want to minimize impact, or at least 5 feet from a critical receptor (i.e., water body). By monitoring real-time, the pumping of CAC can be stopped or slowed once the sentinel piezometers show detections of materials. We feel that it is appropriate to allow the sentinel piezometer to reach concentrations of up to a few hundred mg/L, which would attenuate in concentration to the nearby well or receptor.

Sentinel wells can be effectively used with CaCl_2 parking efforts by verifying parking zones.

Wait and Sample When CAC <100 PPM



Figure 9 - Client periodically sampled wells and used field concentration test kit (next section) to evaluate when sampling was safe. In this case, groundwater sampling was delayed.

If a well is impacted, the best solution is to delay sampling and analysis until PlumeStop or PetroFix has had more time to deposit onto the soil, resulting in clarified groundwater samples. **Two to three months is enough at many sites, although it can take longer at some sites.** The time to equilibrate in the subsurface is correlated with soil clay and silt content. Generally, increased clay and silt content will decrease the time for PlumeStop or PetroFix to sorb and equilibrate. Divalent cations (ex. calcium or magnesium) in groundwater also speed up the clarification process.

If PlumeStop or PetroFix is observed in a groundwater well during the application, the well can be flushed with clear water (i.e., no reagent). The “PetroFix Well Flushing” Technical Bulletin provides more information on clear water well flushing. If sampling at least four weeks post-PlumeStop or PetroFix application, extended low flow purging of the monitoring well may improve the water clarity.

As a rule of thumb, if a sample is placed in a 40-mL VOA vial and you can see through the vial, it is probably safe to sample. The inability to effectively see through a vial is approximately 100 mg/L of PlumeStop or PetroFix (see the following figure of various PlumeStop or PetroFix concentrations for reference).

If you are interested in independent research on showing when suspended carbon from PlumeStop or PetroFix interferes with laboratory samples, please view the webinar entitled [“Remediation of Chlorinated Solvents in Groundwater with PlumeStop: Analytical Challenges and Solutions”](#) which was given by Heather Lord, Ph.D., who at the time was the Environmental Research and Development Manager for Maxxam Labs (now Bureau Veritas Labs). At roughly 15 minutes, Heather begins discussing the ranges where PlumeStop (or PetroFix) does not cause significant lab interference (around 100 mg/L) and positive results from passive diffusion bag samplers.

Unfortunately, no commercial laboratory prep procedures can easily remove PlumeStop or PetroFix from samples before analysis without deviating from standard methods. Filtering the 1 to 2-micrometer diameter particles from suspension is possible (see later section), although difficult and not always an accepted approach by every regulatory agency.

While centrifuging is possible, commercial labs typically do not have the necessary centrifuges to separate PlumeStop or PetroFix effectively.

If you need further technical assistance addressing the interference of PlumeStop or PetroFix when sampling, please get in touch with REGENESIS at info@REGENESIS.com or info@petrofix.com.

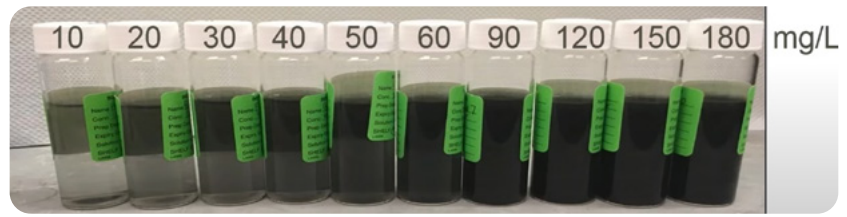


Figure 10 - PlumeStop or PetroFix concentrations in 40 mL VOA vials. If a vial can be seen through (~<100 mg/L), sending the sample to the lab is safe.

CAC Field Concentration Test Kit

Kit Contents:

- 50 mg C/L standard
- Tall test vial
- Small dosing syringe
- Large dilution syringe

NOTE: you will need a source of clean water. Tap or bottled drinking water is acceptable.

REGENESIS does provide simple in-field PlumeStop or PetroFix testing kits to semi-quantitatively determine the activated carbon concentration in groundwater samples following the injection of PlumeStop or PetroFix. The kit is meant to aid in deciding if a well requires flushing, judging the influence of an injection event, and following the change in suspended carbon well over time. Using the kit as described below will resolve activated carbon concentrations of 0-5000 mg C/L.

All PetroFix shipments come with one (1) field concentration test kit taped to the top of a drum or tote in the shipment. For PlumeStop projects which are injected turnkey by REGENESIS, the field crews will have available kits. The instructions in the kit explain how to dilute the sample and how to calculate CAC concentrations. Please contact REGENESIS at info@REGENESIS.com or 949-366-8000 if you need to replace a test kit.

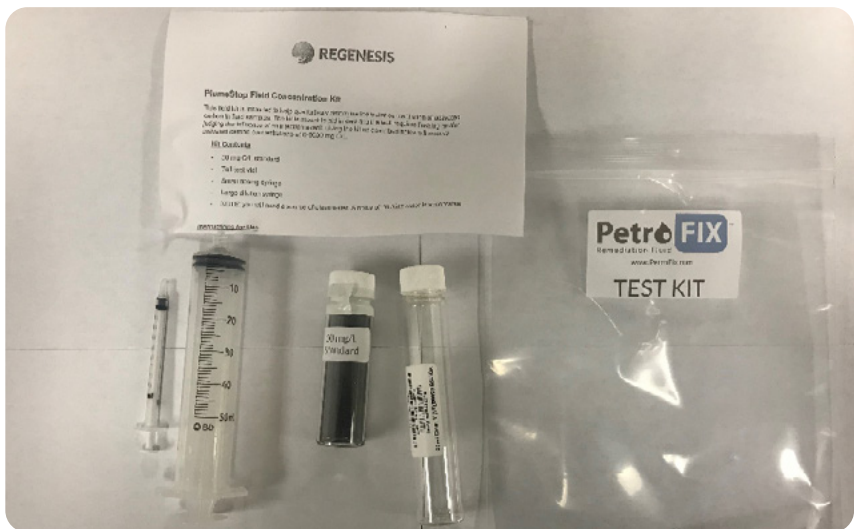


Figure 11 - Image of a field concentration test kit.

Filtering PlumeStop or PetroFix From Samples

In some instances, filtration as a step to remove low levels of CAC may be considered. Of the various methods available to remove CAC from water samples, filtration is the least recommended and most likely to receive pushback from regulators. Filtration is difficult because the filters rapidly become clogged by the CAC at higher concentrations, 300 mg/L or above. This filter cake build-up can also bias low concentration aqueous analytical results because of the high CAC layer that filtered water must travel through as it reaches the filter membrane. Additional issues with filtration include sorption of target analytes to the filter membrane and potential volatilization of lighter organic compounds such as BTEX.

Notes on best practices when filtering:

- Only attempt filtration to remove low levels of CAC (approximately 300 mg/L or lower)
- To avoid sorption of analytes to the filter, use glass fiber filter membranes (GFF) or other polar, low-affinity type materials.
- Minimize the headspace on both sides of a filtration setup. Volatilization of many VOCs is rapid and will bias the results

If filtration is being considered, it is strongly recommended to first consult with the regulating agency receiving the data to decide if the proposed sampling method will be acceptable.

In VOA Sample Clarification With Alum

A final option to obtain groundwater samples if additional fieldwork, waiting, or other methods are not desired or otherwise not successful, then groundwater samples can be safely sampled when treated with a powerful flocculant known as aluminum sulfate (alum). Field treatment of VOA samples with alum will remove CAC from the water matrix within hours while maintaining the integrity of any desired analysis. As stated throughout this document the presence of CAC above approximately 100 mg/L can have a negative impact on the methods and instruments used to quantify volatile organic compounds (VOCs) in water by standard methods like EPA 8260. This method is compatible with analytical methods used to measure VOCs, cVOCs, TPH-G and TPH-D.

Note, given this new approach clients or regulators may have questions about adding alum to samples and the potential to affect results. REGENESIS has confirmed with our own labs and through independent, outside lab testing that the use of alum does not bias results. Additional information on the subject can be found in a separate technical bulletin at www.REGENESIS.com or www.PetroFix.com website and doing a keyword search for **“CAC Alum Flocculation Method Validation”** which will identify the latest copy of this document which we anticipate may be updated in the future.

Alum Approach

For standard volatile organic carbon analysis by EPA 8260, this is achievable by adding a small amount (approx. 1 g/L) of alum (potassium aluminum sulfate, a food additive) to the sample at the time of collection. The addition of alum will promote the flocculation and settling of the suspended CAC, thus clarifying the sample, and allowing a clear aliquot to be taken for workup and analysis by the standard purge and trap method commonly performed as a part of EPA 8260. Because the carbon and water have reached equilibrium by the time of collection, there is slight to no bias between the before and after contaminant groundwater concentration following removal of the suspended CAC from the sample. Alum is commonly used in municipal water supplies to reduce turbidity before distribution in public water utilities.

Alum Settling Agent Kit

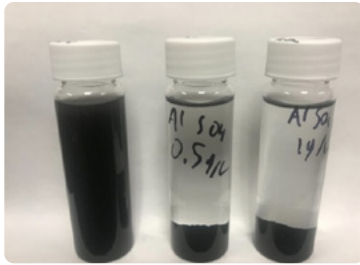


Figure 12 - Before and after alum treatment of suspended CAC in VOA vials.

Settling Agent Dosing Guide

Sample Volume	40 mL
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Settling agent	40 mg
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Settling agent	2 scoops
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Alum kits are provided by request and are used for settling suspended colloidal activated carbon (CAC) in 40mL VOA vials so that the remaining clear solution (supernatant) can be analyzed for contaminants by instrumental methods. Once the settling agent is added to the sample of black water, the carbon will begin to settle rapidly and be ready for analysis in roughly an hour. This method is appropriate for water samples containing around 5000 mg/L of CAC or less.

The lab receiving an alum-treated sample must allow the vials to stand undisturbed after receipt until the CAC has settled by an acceptable amount. If vials are set aside immediately after receipt by the lab, there will be adequate time for settling to occur within the method hold time. The lab requires no other special action.

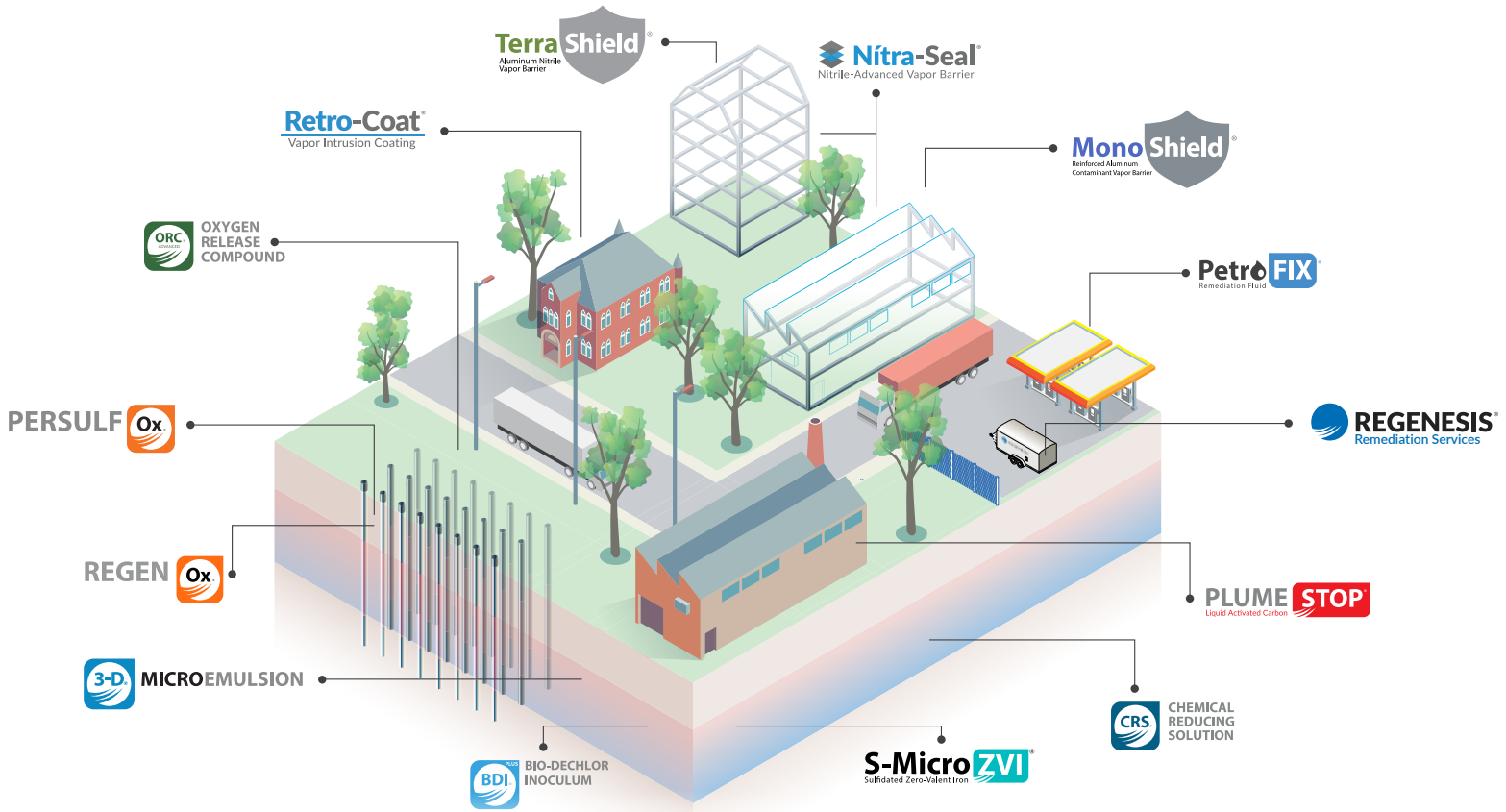
Kits can be obtained by emailing info@REGENESIS.com or info@petrofix.com and referencing your project. Alum can be sourced separately as well and applied per the kit directions below.

Contents:

- 1x Vial of 10 g settling agent (aluminum sulfate hydrate, alum)
- Dosing spoon to deliver 20-40 mg of alum

Procedure:

1. Obtain 40 mL of water to be tested in a 40 mL VOA vial.
2. Deposit 2 scoops of the settling agent into the vial.
3. Shake the vial for 30 seconds.
4. Allow at least 1 hour for carbon to completely settle before testing clear water



About REGENESIS

At REGENESIS we value innovation, technology, expertise and people which together form the unique framework we operate in as an organization. We see innovation and technology as inseparably linked with one being born out of the other.

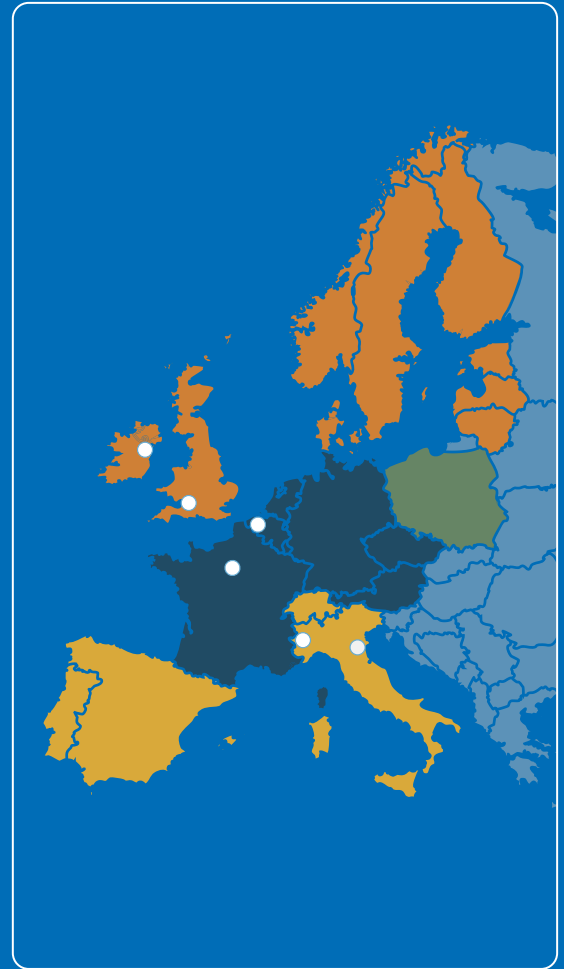
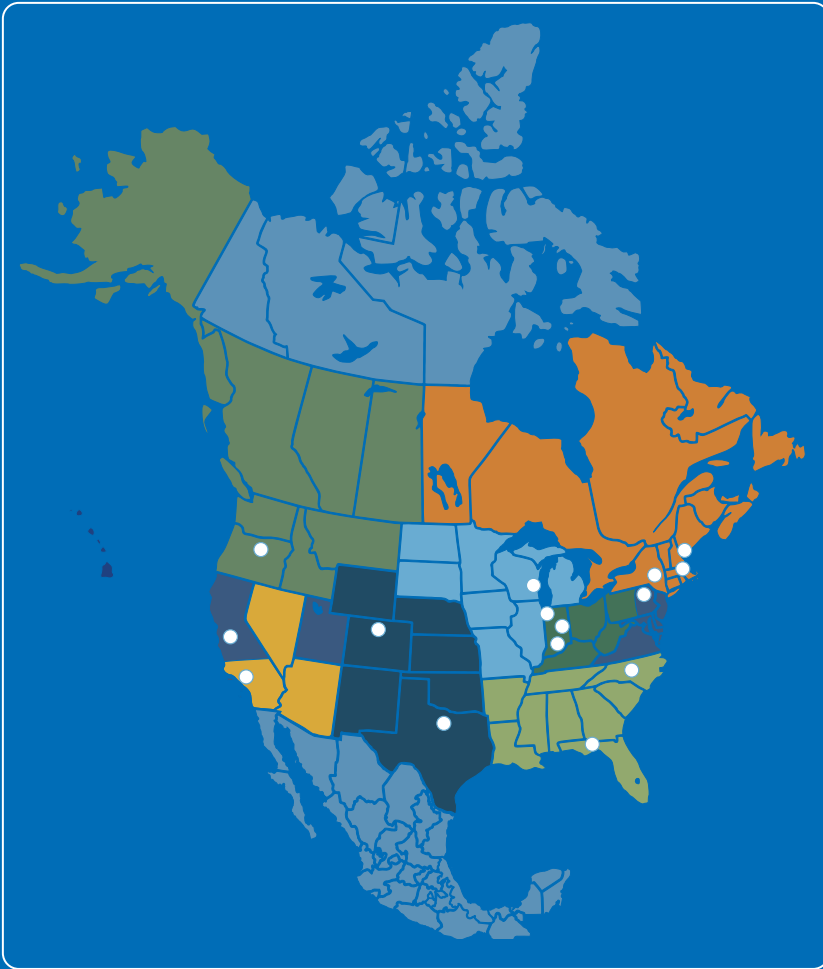
Inherently, innovation imparts new and better ways of thinking and doing. For us this means delivering expert environmental solutions in the form of the most advanced and effective technologies and services available today.

We value expertise, both our customers' and our own. We find that when our experienced staff collaborates directly with customers on complex problems there is a high potential for success including savings in time, resources and cost.

At REGENESIS we are driven by a strong sense of responsibility to the people charged with managing the complex environmental problems we encounter and to the people involved in developing and implementing our technology-based solutions. We are committed to investing in lasting relationships by taking time to understand the people we work with and their circumstances. We believe this is a key factor in achieving successful project outcomes.

We believe that by acting under this set of values, we can work with our customers to achieve a cleaner, healthier, and more prosperous world.

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Colloidal Suspension Sampling Guidance Document 4122022-v9



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