

Color-Tec Method

Field-Based Analysis of Total Chlorinated Volatile Organic Halocarbons of Ex-Situ Soil and Water Samples



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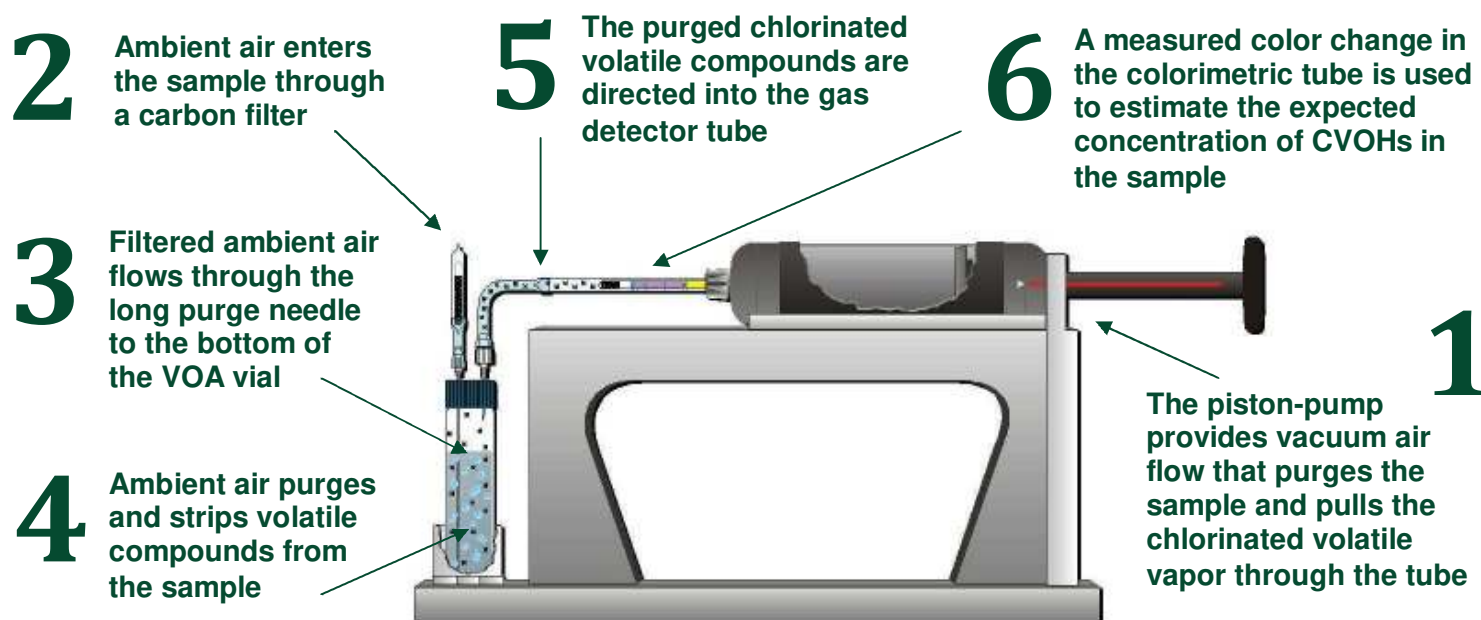
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Color-Tec is a field-based analytical method which combines sample purging with colorimetric gas detector tubes to detect total chlorinated volatile organic halocarbon compounds in any ex-situ liquid or solid sample at concentrations from ~3 µg/L or µg/Kg. Samples are analyzed in two minutes or less by purging the volatile compounds from the sample directly through the colorimetric tube, which is designed to produce a distinct color change when exposed to chlorinated compounds. Estimated sample concentrations are obtained by comparing the tube readings to a conversion table, which was developed based on comparison of the method values to GC/MS analysis of split samples.

*The Color-Tec Method was developed in 1997 by environmental professionals at **Ecology and Environment, Inc.** As the method is neither patented nor copyrighted, anyone may freely use it or manufacture/distribute commercial versions of it. There are currently two manufacturer/distributors of commercial kits containing the hardware and expendables items required to perform the method. These products are known as “AQR Color-Tec” and “QROS Color-Tec”.*

This SOP is a generic version of the method procedures manuals provided with these commercially available Color-Tec kits. In addition to presenting the detailed method procedures, this SOP provides a comprehensive list of items required to perform the method. The trademarked items/kit components mentioned in this SOP are the components used in the commercially-available kits. Users of the method who wish to assemble their own kits may use alternate brands/manufactures of the individual components with exception to the colorimetric tubes manufactured by Gastec[®], which are calibrated to the specific methodology provided in this SOP.



Standard Operating Procedures Manual

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1.0 Method History and Principles

The Color-Tec method was developed during 1997 by the environmental professionals at Ecology and Environment, Inc. while assessing/remediating the earliest sites addressed under the Florida Department of Environmental Protection's (FDEP) Drycleaning Solvent Cleanup Program. Since its development, the method has been used extensively at EPA, DOD, and various state regulatory agency sites to provide real-time, decision quality data at thousands of chlorinated solvent sites.

Color-Tec is a field-based analytical method which combines the use of colorimetric gas detector tubes (originally designed for occupational breathing-zone monitoring) with sample purging to detect very low (<3 µg/L or µg/Kg) concentrations of total chlorinated volatile organic halocarbons (CVOHs) in liquid and solid samples. Samples are analyzed by purging the volatile compounds from a groundwater or soil sample directly through the colorimetric tube, which is designed to produce a distinct color change when exposed to chlorinated compounds. Estimated sample concentrations are obtained by comparing the tube readings to a conversion table, which was developed based on comparison of Color-Tec readings to GC/MS analysis of split samples.

Each colorimetric tube contains an oxidizer (PbO₂) and a catalyst (H₂SO₄) which decomposes and converts the chlorinated compounds to hydrogen chloride, which discolors a reagent (4-phenylazodiphenylamine) in the tube from yellow to purple. The reaction formula provided by Gastec® for the PCE tube is as follows:



The colorimetric tubes react positively to all chlorinated volatile organic halocarbons, including saturated and unsaturated chlorinated alkenes and alkanes. The total response indicated by the detector tube (the distance that the color change travels through the tube) reflects the sum of the concentration of each individual chlorinated compound present in the sample. The method is primarily qualitative (detects the presence/absence of a compound or class of compounds).

The colorimetric gas detector tubes used in the method are designed to detect CVOHs in ambient air. **Color-Tec is an alternate use of these tubes**, which purges CVOHs from a water or soil sample and concentrates them into the colorimetric tube. When using colorimetric tubes for the Color-Tec method, the units (ppmV) printed on the tubes do not directly reflect the quantity of CVOHs present in the water or soil sample being analyzed. The Color-Tec tube reading (the distance that the color change travels through the tube) is a **relative response** to the amount of chlorinated-compound molecules that have been purged from the sample and directed into the tube. Therefore, the Color-Tec tube reading is a **unit-less** value used only to record the **relative response** for each analysis in order to facilitate comparison of that response to laboratory GC/MS analysis.

THE COLOR-TEC TUBE READING IS NOT THE SAMPLE CONCENTRATION!

The tube reading is a unit-less value which must be compared to laboratory results from split samples in order to yield an estimate of the actual concentration present in the sample.

This manual provides a conversion table, developed using comparison of Color-Tec tube responses to split-sample GC/MS analyses conducted on thousands of samples, which can be used to provide an estimate of the expected sample concentration based on the tube reading (See Table 2 on page 15).

2.0 Color-Tec Test Kit Description and Set-up

The Color-Tec Chlorinated VOH Soil/Water Test Kit System consists of two primary components:

1. A hardware kit which contains all **reusable equipment** needed to conduct the method, plus an optional carrying case; and
2. Expendables kits containing all **disposable components** needed for analysis of 10 water or soil samples using one of four available ranges of Gastec® 133-series tubes:
 - a. Ultra low range 133-LL tubes (expected detection range ~ 3 to 1200 µg/L or µg/Kg)
 - b. Low range 133-L tubes (expected detection range ~ 75 to 25,000 µg/L or µg/Kg)
 - c. Medium range 133-M tubes (expected detection range ~ 500 to 130,000 µg/L or µg/Kg)
 - d. High range 133-HA tubes (expected detection range ~ 5000 to 2,300,000 µg/L or µg/Kg)

2.1 Materials

2.1.1 Color-Tec Hardware (See Figure 1)

Item

Gastec® Piston pump
Color-Tec Pump Stand
Corning® Hot Plate
Stainless Steel Heating Pan
Nalgene® VOA Heating Rack
Thermometer
Decontamination Syringe
Pelican® hard case

2.1.2 Color-Tec Expendables (See Figure 2)

Item

Colorimetric Detector Tubes (133-LL, L, M, or HA)
Disposable Extraction Needle Assemblies (**single use only**)
Carbon Filter (may be re-used)
Carbon Filter Assembly (may be re-used)
Purge Needle (may be re-used)
40 Milliliter unpreserved VOA Vials (**single use only**)

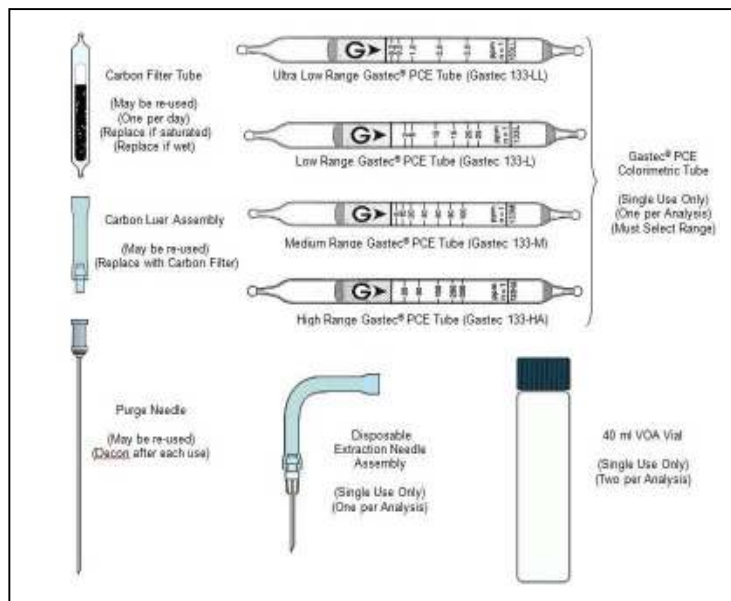
Quantity Per Analysis

1 per range, depending on concentration
1
1 for every 10 to 20 samples or more
1 for every 10 to 20 samples or more
1 (must be cleaned between each use)
2

Figure 1
Required Hardware



Figure 2
Expendables



2.2 Additional Supplies

The following items are suggested for use with the Color-Tec method to perform the listed functions.

Item	Purpose
Organic-free water	for soil sample extraction and equipment decontamination
Safety gloves	personal protection
Safety glasses	personal protection
120V AC power source	for hot plate
Permanent marker	labeling sample bottles
40 Milliliter VOA Vials	(Optional Item) for Color-Tec analysis and split samples

Performance of the Color-Tec method requires the use of two standard, unpreserved VOA vials per sample. These VOA vials are not included in the standard expendables kits, but may be added as an option. The user may wish to collect a quantity of split samples for laboratory analysis to provide comparison data which may be used to determine site-specific method detection limits and/or to tentatively quantify Color-Tec results. Split sampling will likely require three pre-preserved VOA vials per sample. Pre-preserved VOA vials for split samples are not available in the Color-Tec expendables kits.

2.3 Storage & Stability of Colorimetric Tubes

The Gastec® colorimetric tubes have a shelf-life of two years with refrigeration. Tubes should be stored at or below a temperature of 10°C/50°F when not in use. Colorimetric detector tubes are single-use (one tube per analysis) and should be used immediately after the tips are broken. Tube readings should be recorded immediately following analysis because the intensity of the color-change fades over time. Each box of tubes has an expiration date printed in red ink on the top of each box. When heating the tubes for use with the Color-Tec method, it is recommended that the tube temperature does not exceed 40°C/104° F. Other procedures and guidelines associated with the use of the tubes for their designed purpose (gas detection in ambient air) are included in the tube manufactures data sheets and tube instructions included in the tube packaging.

2.4 Heating Colorimetric Tubes and Samples

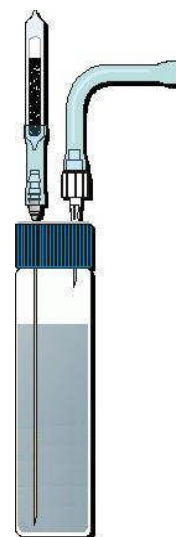
The colorimetric gas detector tubes used in the Color-Tec method were designed for the purpose of detecting volatile organic compounds (CVOHs) in ambient air. When using the tubes for analysis of ambient air, the calibrated operating temperature is 20°C/68°F. Using the tubes at temperatures above or below 20°C/68°F, for the purpose of testing ambient air, introduces error into the measurements requiring application of correction factors to correct that error. **Because Color-Tec is an alternate use of the colorimetric tubes which concentrates CVOHs from water or soil samples into the tubes, the units (ppmV) printed on the tubes have no direct relationship to the quantity of CVOHs dissolved in the water/soil sample being analyzed and the temperature correction factors used for analysis of ambient air are not required when using the colorimetric tubes as part of the Color-Tec method.** However, since the colorimetric tubes are more sensitive to the presence of chlorinated compounds at 40°C/104°F, and the purpose of the Color-Tec method is to detect the presence/absence of CVOHs in water at concentrations at the lowest concentrations possible, the tubes are heated to their optimum sensitivity (40°C/104°F) to maximize their detection capability.

The samples are also heated (in the VOA vials) to maximize contaminant volatilization and transfer of CVOHs from the water sample to the colorimetric tube. To heat the samples and colorimetric tubes, a hot plate is used to heat a water bath containing a test tube rack to hold the sample-filled VOA vials and unbroken colorimetric tubes. Special attention must be paid to the temperature of the water to avoid prolonged overheating the samples and tubes. The samples and colorimetric tubes should not be heated in excess of 40°C/104°F.

Given the size of the heating pan and VOA rack, generally only 3 sets of samples are heated at the same time. When a pair of VOAs is removed from the heating rack and placed on the pump stand, it can be replaced with a new pair for heating. After collection, samples should remain in a cool place until ready to be heated and analyzed. It is recommended to avoid heating the samples for more than about 2 minutes to avoid loss of CVOCs. Section 2.6 below, provides detailed water bath set-up and heating procedures.

2.5 Carbon Pre-Filter

Because ambient air is used to purge the samples, a carbon pre-filter is provided for attachment to the purge needle to prevent volatile airborne contaminants from passing through the sample and entering the detector tube during the purging process. To use the carbon pre-filter, break both tips of a carbon filter tube and insert the end of the tube onto the carbon lure assembly (make sure the air-flow arrows on the carbon tube point toward the carbon lure assembly), then tightly insert the male lure fitting on the carbon lure assembly into the female lure fitting on the purge needle (see Figure 2). At sites where little or no ambient air contamination is expected, a single pre-filter tube may be reused for several days. However, at sites where high concentrations of airborne chlorinated compounds are suspected or have been confirmed in the ambient air, the pre-filter tubes may need to be replaced more frequently. For most situations, one carbon filter per 10 samples is more than sufficient. Section 2.6 below, provides detailed carbon filter set-up and use procedures.



2.6 Color-Tec Work-Station Set-up

Pump Stand Set-up

1. Place the pump stand up-right on a flat stable surface.
2. Place the [®] piston-pump into the curved tray on the top of the pump stand as shown in the photo.



Corning[®] Hot Plate Set-up

1. Connect the AC power cord to the back of the hot plate.
2. Connect the other end of the AC power cord to a USA 120VAC electric outlet.
3. Place the hot plate on a flat stable surface.
4. Set the hot plate thermostat control to between dial setting 4 and 5.



Hot Water Bath Set-up

1. Fill the stainless-steel water bath pan with tap water to approximately 1.5-inches from the rim.
2. Insert the VOA rack into the water-filled, stainless-steel, water bath pan.
3. Remove the cap from a 40ml VOA vial, fill the VOA vial with tap water and place it into the VOA Rack as shown in the photo. Note: The bottom of the water-filled VOA vial should be slightly submersed in the water in the stainless-steel pan.
4. Place the stainless-steel water bath pan onto the heating surface of the hot-plate.
5. Open a box of low-level (133LL) Gastec® tubes and place several tubes into the water-filled VOA vial. Insert the yellow reagent end of the tubes into the bottom of the VOA vial. Note: Do not place tubes with broken tips in the water bath – heating must be accomplished before breaking the tube tips.
6. Turn on the Oakton® digital thermometer and insert the steel probe of the thermometer into the water-filled VOA vial with the colorimetric tubes. Maintain the water in the VOA vial containing the colorimetric tubes at approximately 100°F. The temperature of the water bath pan may be maintained slightly higher than the water in the VOA vial containing the colorimetric tubes to facilitate faster heating of the samples. Replace any water that evaporates from the water bath pan or the VOA vial containing the colorimetric tubes.



Heating Samples

1. Place both VOA vials containing the sample into the hot water bath for approximately 1 to 2 minutes.
2. Be sure that the VOA vials are tightly sealed before heating. Note: When properly heated, the VOA vials should feel warm in the hand – DO NOT OPEN VOA VIALS AFTER HEATING.



Carbon Filter/Purge Needle Set-up

1. Break both ends of a carbon filter tube using the tip breaker on the piston pump.
2. Connect a carbon filter luer assembly to the carbon filter tube by sliding the open end of the vinyl tubing over the broken end of the carbon filter tube. Note: The carbon filter is re-used for multiple purge cycles.
3. Attach the carbon filter assembly to a purge needle by inserting the carbon filter assembly luer fitting into the purge needle luer fitting.
4. The purge needle (with carbon filter) is now ready to use. Follow the steps in Section 4 to conduct sample analysis. **Important Note: Do not insert the purge needle into the sample VOA before completing steps 5 and 6 of Section 4.**

3.0 Sample Collection and Preparation

3.1 Liquid Sample Media

Collect the water or other liquid sample media directly from your sampling device into two 40 ml VOA vials by filling each vial to ~75% capacity (i.e. to about 1-inch below the shoulder of each vial). Tightly secure the caps onto the partially-filled VOA vials. The VOA vials containing the liquid sample to be tested must contain an air-filled headspace to accommodate purging. The caps must be tightened sufficiently to prevent loss of CVOHs during the time between sample collection and analysis (which includes the heating process) and to prevent air leakage during the purging process.

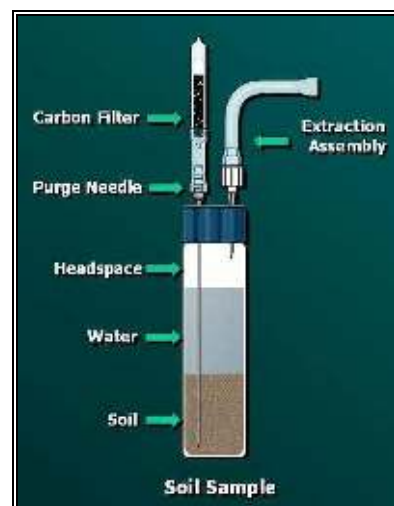


3.2 Solid Sample Media

Place about 1.5 inches of soil (or other solid sample media) into the bottom of each of two VOA vials (i.e. approximately 30 grams in each vial). Immediately after inserting the soil (or other solid sample media) into the two vials, add organic-free or other “clean” water to each VOA vial until they are both ~70 % full (i.e. to approximately 1-inch below the shoulder of each vial). Tightly secure the caps onto the partially-filled VOA vials. Once the caps are secure, shake the VOA vials vigorously for approximately for 5 to 10

seconds to thoroughly mix the soil and water. Additional mixing may be necessary for soil matrices comprised of clay-sized particles. The purpose of the mixing is to transfer any chlorinated compounds suspended in the soil matrix to the water to facilitate more effective purging.

IMPORTANT NOTE: The VOA vials containing the solid sample media and “clean” water must contain an air-filled headspace to accommodate purging. The caps must be tightened sufficiently to prevent loss of CVOHs during the time between sample collection and analysis (which includes the heating process) and to prevent air leakage during the purging process.



3.3 Purpose of the Second VOA Vial

The Color-Tec method is designed for use with two VOA vials (an original and a duplicate) for each sample collected. In certain situations, the duplicate sample may not be used in the performance of the method. However, the duplicate sample should always be collected in the event that it is needed to complete the analysis process. The duplicate sample may be used in either of the following situations:

1. When the initial test does not induce a color change in the colorimetric tube, the second VOA vial containing the duplicate sample, may be purged (using the same colorimetric tube) to increase the probability of detecting very low ($< 10 \mu\text{g/L}$) concentrations.
2. When the initial test induces a color change that exceeds the upper limit of the LL tube (a tube reading >3 for 100cc purge or >9 for 50cc purge), the extra VOA vial can be used to analyze the sample using higher range colorimetric tubes (133L or 133M) to tentatively quantify the higher concentration of chlorinated compounds in the sample.



4.0 Sample Analysis Procedure

1. Place both heated VOA vials (original & duplicate sample) into the two VOA holders on the pump-stand.
2. Remove a low-level tube from the hot water bath and wipe it dry.



3. Break both ends of the colorimetric tube using the tip breaker on the piston pump.
4. Insert the colorimetric tube into the pump inlet with the flow arrow (printed on the tube) toward the pump. **Note: Tube orientation is critical – the yellow reagent end of the tube is inserted in the pump.**



5. Connect a new extraction needle assembly to the colorimetric tube by sliding the open end of the vinyl tubing over the broken end of the colorimetric tube. This step must be completed before inserting the needle into the VOA (Prior to step 6).



6. Remove the protective cap from the extraction needle and insert the needle into the septa of the first VOA vial. **Note: Be sure that the tip of the extraction needle is positioned within the headspace of the VOA vial (above the water level). Do not insert the extraction needle as far as it will go into the headspace of the VOA vial, but rather only to a point slightly beneath the inside of the septa to reduce the possibility of sample water entering the extraction needle assembly and colorimetric tube during the purging process.**

7. Insert the purge needle (with carbon filter assembly) into the septa of the first VOA vial and push the tip of the needle to the bottom of the VOA vial.
IMPORTANT NOTE: Do not insert the purge needle before completing steps 5 and 6.

8. Align the 50ml label and red dot on the pump handle with the red dot on the pump shaft.
9. Pull the handle sharply until it locks in the 50ml (half pull) position.
10. Confirm that air is purging through the sample in the VOA vial.
11. Purge for approximately 30 seconds.



12. Check the yellow reagent in the tube for a color-change.
13. If no color-change reaction is visible or if the color reading is less than 1.5, rotate the pump handle ½ turn and pull the handle out to lock in the 100ml position.



14. Continue the 100ml purge until the flow cycle is complete. Note: Flow is complete when the end-of-flow indicator (located on the back of the pump handle) returns to its full brightness.

15. Check the yellow reagent phase in the tube for a color change.

16. If no color-change is visible, remove the extraction needle from the VOA with the vinyl tubing still attached to the low-level tube, rotate the pump handle ¼ turn and push the plunger back into the pump, remove the extraction needle from the first VOA vial and inject it into the septa of the second VOA (duplicate sample), then remove the purge needle from the first VOA vial and inject it into the septa of the second VOA (duplicate sample) - now re-pull the pump handle to lock into the 100ml position.

17. When the second 100ml purge cycle is complete, read and record the results.



For samples containing high concentrations ($>150 \mu\text{g/L}$) the resulting color-change may exceed the calibrated limit of the low-level tube, requiring the second VOA vial (duplicate sample) to be purged and analyzed by repeating steps 3 through 13 using a medium range (133L) or a high range (133M) tube.

For samples containing low ($<5 \mu\text{g/L}$) concentrations the color change does not usually begin until 100 CCs of air have purged through the sample. Furthermore, the color change induced at these low concentrations is very slight (below 0.5 on the tube scale) and appears as a slight darkening or light purple hue at the entrance of yellow reagent layer in the LL tube. When the sample contains higher concentrations ($>10 \mu\text{g/L}$) of chlorinated compounds, the resulting color change is an obvious light to dark purple, which propagates through the yellow reagent layer toward the pump end of the colorimetric tube. The tube reading (Color-Tec response) is obtained by matching the linear extent of the discolored reagent inside the tube to the calibration scale printed on the outside of the tube. Table 1 presents a troubleshooting matrix with causes and solutions potential problems.

Important Procedural Notes:

The disposable extraction needle assembly is intended for one use only. Decontamination and re-use of this part is highly discouraged because of the risk of contaminant carryover from the tubing and other plastic parts which can harbor contaminants from the previous analysis. Purge needles may be reused following decontamination using water and isopropanol.

Carbon filters should be discarded if they become wet from contact with sample water.

Never insert the purge needle into the VOA before the extraction needle assembly has first been connected to the colorimetric tube and inserted into the VOA headspace. If the purge needle is inserted first, the pressure inside the sealed VOA may force sample water up through the purge needle and into the carbon filter. Sample volatiles may be lost if the extraction needle assembly is inserted into the VOA headspace before connecting the tubing to the colorimetric tube.

To prevent clogging of the purge needle when inserting the purge needle into VOA vials containing soil samples, do not immediately push the bottom of the needle through the soil to the bottom of the vial; but rather temporarily position the base of the purge needle in the water above the soil until the pump handle has been pulled to begin air flow through the sample. Once air flow has been initiated, slowly extend the purge needle through the soil to the base of the vial. The air flow from the tip of the purge needle should reduce the potential for clogging as the needle moves through the soil. This procedure is especially helpful when working with clayey soils.

Troubleshooting Guide

Problem	Possible Cause	Solution
Sample does not appear to be purging (bubbling) after the pump handle has been pulled.	Clogged/blocked purge needle (long needle).	Use the decontamination syringe to check the purge needle for clogs. If clogged, clean the needle or use a new purge needle.
	Clogged/blocked extraction needle (short needle).	Use decontamination syringe to check the extraction needle for clogs. Use decontamination syringe to clean the needle or use a new extraction needle.
	Colorimetric tube is not securely connected to hand pump.	Remove and re-insert the colorimetric tube from the hand pump. If the fit seems loose, replace the hand pump inlet gasket.
	Colorimetric tube is not securely connected to extraction needle tubing.	Check the connection between the extraction needle tubing and the colorimetric tube. If loose, insert the colorimetric tube further into the extraction needle tubing.
	VOA cap is not tightly sealed.	Check the tightness of the VOA cap. Tighten if necessary.
	Colorimetric tube tips were not broken before connecting to hand pump and tubing.	Break both tips of the colorimetric tube before connecting to hand pump and tubing.
	Broken/bad plunger seal in hand pump.	Check the pump seal by holding your finger over the hand pump inlet while pulling the pump handle and lock into the 50cc position. If no vacuum is apparent, open the pump, remove the plunger, replace the plunger seal, and grease the new seal. Re-assemble the pump.
The colorimetric tube shows no reaction after purging a sample that contains chlorinated compounds. (False Negative)	Colorimetric tube is below the optimum operating temperature.	Heat the colorimetric tube to 40°C/104° F before using. It is also recommended to heat the sample. The recommended temperature for tubes and samples when using the Color-Tec Method is 40°C/104° F.
	Colorimetric tube was connected using reversed flow direction.	Use the flow direction arrows to properly align the tube. The purged air must pass through the black oxidizer phase and the white catalyst phase before entering the yellow reagent phase.
	The sample also contains a detectable concentration of xylenes or toluene.	Samples can be tested for the presence of xylenes and toluene using the Gastec® 122L colorimetric tube. The detection of chlorinated compounds may be diminished when xylenes or toluene are present in a sample.
The colorimetric tube indicates a reaction after purging a sample that contains no chlorinated compounds. (False Positive)	Chlorinated compounds are present at detectable concentrations the ambient air.	Test the ambient air using an LL tube to determine if chlorinated compounds are present at detectable concentrations. Attach the charcoal filter to the purge needle prior to purging samples.
	HCl vapor is present in the sample VOA or in the ambient air.	Avoid use of HCl in the area where Color-Tec is in use. Use only unpreserved VOAs for samples to be screened with Color-Tec.
	Water vapor has entered the yellow reagent phase of the tube indicating a positive reaction	Avoid purging more than 200 CCs through any sample. Stop purging before condensation inside the tube reaches the end of the black oxidizer phase. Avoid drawing any water from the sample VOA into the colorimetric tube.

5.0 Sample Purging and Detection Methodology

Samples may be purged using 50 cubic centimeters (cc), 100cc, or 200cc purge volumes. These various purge volumes are used in succession to maximize the low-level detection capability and detection range of each tube, thereby reducing the number of tubes needed to tentatively quantify the concentration of total chlorinated compounds in the sample. The pump stand is equipped with two VOA-vial holders to accommodate a second (duplicate) sample to be collected from each sampling location. This duplicate sample (collected and prepared in the same manner as the original sample) serves the following two potential purposes:

1. When purging the initial VOA vial does not induce a color change in the colorimetric tube, the second VOA vial containing the duplicate sample, may be purged (using the same colorimetric tube) to increase the probability of detecting very low ($< 10 \mu\text{g/L}$) concentrations.
2. When the initial test induces a color change that exceeds the upper limit of the LL tube (a tube reading > 3), the extra VOA vial can be used to analyze the sample using higher range colorimetric tubes (133L or 133M) to tentatively quantify the higher concentration of chlorinated compounds in the sample.

5.1 50cc Purge Volume

Initially, all samples are analyzed using a Gastec® 133-LL tube with a 50cc purge cycle. If the 50cc purge induces a color change reading of 1.5 to 3.0, read the calibration scale value aligned with the stained/unstained interface in the tube and use the pump stroke correction factors provided on Table 1 to determine the correct reading for a 50cc purge volume. If the concentration in the sample exceeds the upper detection limit of the tube (i.e. the color change moves beyond the upper limit of the calibration scale printed on the tube), repeat the analysis using duplicate samples and higher range tubes (133-L, 133-M, and 133-HA) until the color change reaction stops within the calibration scale on the tube. If the color change reaction exceeds the upper limit of the calibration scale of the HA tube, the sample contains a concentration of chlorinated compounds above the upper detection capability of the Color-Tec Method.

5.2 100cc Purge Volume

Following completion of the 50cc purge cycle, if the concentration in the sample has induced a color change in the tube which traveled less than half the distance of the calibrated portion of the reagent phase of the tube (less than a reading of approximately 1.5), pull the pump handle outward and lock it into the 100cc position to complete a full purge cycle. Record the value aligned with the stained/unstained interface on the tube. No correction factor is needed for a 100cc purge.

5.3 200cc Purge Volume

Following completion of the 100cc purge cycle, if the concentration in the sample has induced no color change reaction, remove the purge needle and extraction needle assembly from the VOA vial containing the original sample and insert them into the VOA vial containing the duplicate sample (which has also been pre-heating) and perform another 100cc purge cycle **using the same colorimetric tube**. To perform the transfer to the second vial, remove both needles from the original VOA vial and immediately insert both needles into the septa of the duplicate sample VOA vial. Before re-inserting the pump handle, temporarily remove the colorimetric tube from the tip of the hand pump and re-insert the pump handle completely into the pump while the tube is un-attached. Re-attach the colorimetric tube into the pump tip and pull the pump handle and lock it into the 100cc position. Following the complete second purge cycle, read the calibration scale value aligned with the stained/unstained interface in the tube and use the pump stroke correction factors provided on Table 1 to determine the correct reading for a 200cc purge volume.

Table 1
Purge Volume Correction Factors for 133-Series Tubes

Colorimetric Tube	Purge Volume	Quantity of Pump Pulls	Correction Factor
133-LL	50cc	Half Pull	Tube Reading x 3
133-LL	100cc	Full Pull	Tube Reading x 1
133-LL	200cc	Two Pulls	Tube Reading x 0.5
133-L	50cc	Half Pull	Tube Reading x 3
133-L	100cc	Full Pull	Tube Reading x 1
133-L	200cc	Two Pulls	Tube Reading x 0.5
133-M	50cc	Half Pull	Tube Reading x 2.5
133-M	100cc	Full Pull	Tube Reading x 1
133-M	200cc	Two Pulls	Tube Reading x 0.4
133-HA	50cc	Half Pull	Tube Reading x 3
133-HA	100cc	Full Pull	Tube Reading x 1
133-HA	200cc	Two Pulls	Tube Reading x 0.3 [∞]

6.0 Reading the Tubes

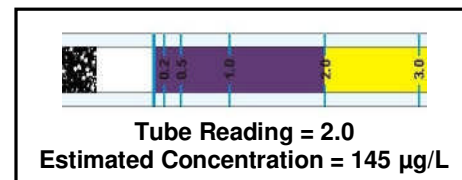
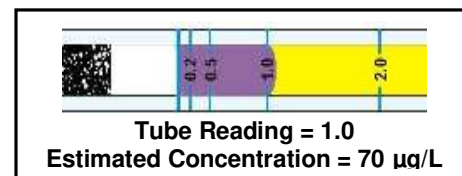
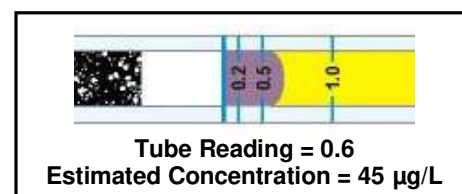
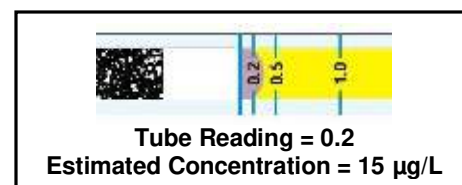
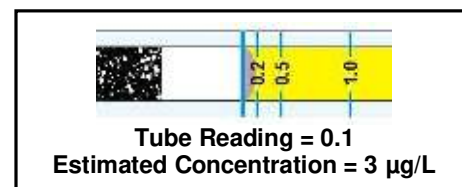
The basic Color-Tec method procedures are simple and intuitive; however, contaminant detection and semi-quantitative values are obtained through visual observation of the colorimetric reaction in the tubes, which is inherently subjective (especially in samples containing very low [$<5 \mu\text{g/L}$] total CVOHs). These low-level samples induce only a slight color change (i.e. slight darkening or light purple hue) prior to the 0.5ppm line on the tube scale at the entrance of yellow reagent layer in the LL tube. Samples containing concentrations of total chlorinated compounds above $5 \mu\text{g/L}$ usually induce a more apparent reaction within the LL tube.

6.1 Very Low Concentrations

When a sample contains very low concentrations ($<10 \mu\text{g/L}$) of chlorinated compounds, the resulting color change is not immediate or distinct. At these low concentrations the color change does not usually begin until between 100 and 200 CCs of air have purged through the sample into the tube. Furthermore, the color change induced at these low concentrations is very slight (below 0.5 on the tube scale) and appears as a slight darkening or light purple hue at the entrance of yellow reagent layer in the LL tube.

6.2 Low to Medium Concentrations

When the sample contains higher concentrations ($>10 \mu\text{g/L}$) of chlorinated compounds, the resulting color change is an obvious light to dark purple, which propagates through the yellow reagent layer toward the pump end of the colorimetric tube. The detected concentration level is obtained by matching the linear extent of the discolored reagent inside the tube to the calibration scale printed on the outside of the tube.



6.3 High Concentrations

When the sample contains high concentrations ($>100\text{ }\mu\text{g/L}$) of chlorinated compounds, the color change reaction occurs quickly and usually exceeds the upper detection level of the Gastec® 133LL tube. The higher the concentration of chlorinated compounds in the sample, the faster the color change reaction occurs and the further it propagates through colorimetric tube. Samples containing very high concentrations ($>1000\text{ }\mu\text{g/L}$) of chlorinated compounds, often discolor the entire yellow reagent layer in the LL tube before the pump handle has been fully extended. In these cases, the purging can be discontinued to allow for the current sample bottle to be re-tested using a higher range detector tube. There is no need to continue purging the sample when the detection level of the tube is exceeded. Each subsequently higher range tube (133L, 133M, or 133HA) is used to purge each new duplicate sample in succession until the color change reaction does not exceed the calibration range of the tube being used.

6.4 Recording Tube Readings

It is recommended to record the observed concentration value (tube reading), the range of the colorimetric tube (LL, L, M, or HA) and the final purge volume when logging Color-Tec results. For example, a reading of 2.5 observed on an LL tube using a 100 ml purge should be recorded as **2.5/LL/100**. Purge volume correction factors must be applied for Color-Tec values which were obtained using any purge volume other than 100cc. For example, a reading of 0.2 observed on an LL tube using a 200 ml purge should be recorded as **0.1/LL/200**. A reading of 60 observed on an M tube using a 50 ml purge should be recorded as **150/M/50**.

7.0 Estimating Sample Concentrations (Conversion Table)

The Color-Tec reading (the distance that the color change travels through the tube) is a **relative response** to the amount of chlorinated-compound molecules that have been purged from the sample and directed into the tube. Therefore, the units printed on the tubes are used only to record the **relative response** for each analysis in order to facilitate comparison to laboratory GC/MS methods.

To provide a field-ready estimate of the total chlorinated solvent concentration in liquid and solid samples based on the colorimetric tube reading, The developer of the Color-Tec method created a conversion table (see Table 2) based on statistical comparison of water samples collected from chlorinated solvent sites in which the Color-Tec and GC/MS methods were used to analyze split samples. An estimated concentration may be obtained by matching the Color-Tec tube response to either the median expected GC/MS concentration or the range of expected GC/MS concentrations provided on the comparison table. The potential range of corresponding analytical values associated with each positive tube reading increases significantly as the sample concentration increases. The estimated concentrations presented on Table 2 represent the central tendency of the comparison data. The actual analytical values obtained by laboratory analysis of split samples may differ substantially from this estimate and may fall outside of the corresponding ranges provided on Table 2.

The expected GC/MS concentrations presented in Table 2 are based on comparison of water sample data only. These conversion values may also be used for soil data; however, the potential range in expected GC/MS concentrations may be increased as a result of the difference in soil volumes used in the two methods and in the inherent heterogeneity of most soil matrices. However, the potential deviation factors included in the range of expected GC/MS concentrations column should be sufficient to account for the intrinsic analytical variability of most soil sample results.

THE COLOR-TEC TUBE READING IS NOT THE SAMPLE CONCENTRATION!

The tube reading is a unit-less value which must be compared to laboratory results from split samples in order to yield an estimate of the actual concentration present in the sample. This conversion table provides a reasonable estimate of the expected sample concentration based on the tube reading.

Table 2
Conversion of Color-Tec Readings (Relative Responses) to
Expected GC/MS Total Chlorinated Volatile Organic Halocarbon Concentrations

Gastec® Colorimetric Tube	Color-Tec Tube Reading (relative response) (unit-less)	Median Expected GC/MS (Laboratory) Concentration (µg/L or µg/kg)	Range of Expected GC/MS Concentrations (µg/L or µg/kg)	
			Low	High
133-LL	0	3	>0	5
	0.1	7	5	10
	0.2	15	10	20
	0.5	35	25	45
	0.8	55	40	75
	1	70	50	95
	1.5	110	75	140
	2	145	105	190
	2.5	190	130	245
	3	230	160	290
	5	380	260	490
	9	900	630	1,160
133-L	25	2,500	1,250	3,750
	35	4,400	2,200	6,600
	45	7,700	3,850	11,550
	55	15,000	7,500	22,500
	75	17,200	8,600	25,800
133-M	100	21,100	10,500	31,600
	200	46,000	23,000	69,000
	300	85,000	42,500	127,500
133-HA	500	225,500	112,800	338,300
	700	598,300	299,200	897,500
	900	1,587,500	793,800	2,381,300

Notes:

The **Color-Tec Tube Reading** (Color-Tec units) is the value printed on the colorimetric tube at the interface between the reacted and un-reacted reagent (the extent of the color change in the tube for a positive result).

The **Median Expected GC/MS Concentration** is the estimated concentration in micrograms per liter (µg/L) of total chlorinated volatile organic halocarbons (CVOHs) present in the sample for the corresponding Color-Tec tube response.

The **Range of Expected GC/MS Concentrations** is an estimated range of potential concentrations (µg/L or µg/kg) of total chlorinated volatile organic halocarbons (CVOHs) for the corresponding Color-Tec tube response.

The **Median Expected GC/MS Concentration** was obtained using statistical comparison of Color-Tec Method data and GC/MS (EPA Method 8260B) data. Comparison data were obtained from 5348 water samples collected from 152 chlorinated solvent (primarily PCE) sites in which the Color-Tec Method was used to analyze the samples in the field and either a laboratory-based or mobile GC/MS was used to analyze split samples.

The **Range of Expected GC/MS Concentrations** reflects the potential deviation in the **Median Expected GC/MS Concentration** based on Color-Tec Method/EPA Method 8260B comparison results. The potential error increases as the concentration increases. The initial deviation factor used for a Color-Tec Reading of zero is +/- 30% and increases to +/- 400% at a Color-Tec Reading of 900 units.

The **Median Expected GC/MS Concentrations** presented in this table are based on comparison of water sample data only. These conversion values may also be used for soil data; however, the potential error or range in expected GC/MS concentrations may be increased as a result in the difference in soil volumes used in the two methods and in the inherent heterogeneity of many soil matrices. The potential deviation factors included in the **Range of Expected GC/MS Concentrations** data should be sufficient to account for the intrinsic analytical variability of most soil sample results.

The expected GC/MS concentrations in this table are provided only to give Color-Tec Method users an approximate concentration for the Color-Tec Tube Response. Actual GC/MS results on split samples may be outside of the stated range for a given Color-Tec Tube Response.

Refer to the **Color-Tec Manual** for detailed information regarding general method principals and potential analytical variables.

8.0 Proposed QA/QC Procedures

As with any analytical method, standard sample preparation and quality assurance/quality control (QA/QC) procedures tailored to the specific project goals should be developed and followed precisely and consistently throughout the sampling and analysis program to insure consistent results and the lowest possible detection levels for all samples analyzed using the Color-Tec method. This section is intended to provide the Color-Tec user with a basic methodology for conducting QA/QC procedures which address various potential operational and procedural issues, such as analytical confidence, method performance, false positives/negatives, replicate accuracy, and contaminant carryover. Users of the Color-Tec method are encouraged to use the information provided in this section to develop project-specific QA/QC and sample handling procedures that insure the level of consistency and accuracy required for the user's sampling program.

8.1 Analytical Confidence and Method Performance

Using Color-Tec to analyze prepared sample spikes containing known concentrations of chlorinated compounds provides confidence that the method procedures are being performed properly performed and may provide a basis for estimating concentrations based on the low-range (133LL) colorimetric tube responses. Spiked sample concentrations should range between 10 µg/L and 200 µg/L to cover the detection range of the low-range (133LL) colorimetric tube. Most analytical laboratories will prepare spiked samples in VOA vials with specified compounds at specified concentrations. Conduct Color-Tec analyses on the spiked samples using the same procedures described in Sections 3 and 4 and record the results in your field log as described in Section 5.3. A 200cc purge using two VOA vials (as described in Section 4.3) may be required to produce a positive Color-Tec reading when testing spiked samples containing 10 µg/L or less of total CVOHs may require a 200cc purge to produce a positive Color-Tec reading.

Performance/confidence testing of the higher range tubes (133L, 133M, and 133HA) using high-concentration spiked samples is unnecessary because the high range tubes are usually not used unless the sample being tested has already exceeded the upper range of the low range tube, thus revealing that the sample being tested contains a sufficient quantity of chlorinated compounds to evoke a positive reaction from the next higher range tube. Given the inherent extreme variability of estimating high concentrations based on tube responses on the high range tubes (133L, 133M, and 133HA), comparison of high concentration (>500 µg/L) spiked samples generally

8.2 Chemical Inhibitors (False Negatives)

The presence of Toluene and Xylenes inhibits/diminishes the ability of the colorimetric tubes to detect CVOHs. At sites where the presence of these compounds is suspected to be present in the soil or water samples, QA procedures may include periodic testing of groundwater or soil samples and ambient air for the presence of toluene and xylenes using a Gastec® Toluene tube (the Toluene tube also detects xylenes). To conduct a test for the presence of compounds which could inhibit the detection of CVOHs use the Toluene (122L) tube to analyze a duplicate soil or water sample using the procedures described in Sections 2 through 4. Toluene (122L) tubes may be obtained from any distributor of Gastec® products.

8.3 Positive Interference (False Positives)

Chlorinated Volatile Organic Halocarbons. The Gastec® 133-series colorimetric tubes used to perform the Color-Tec method detect all chlorinated volatile organic halocarbons (CVOHs) present in each sample. Thus, individual CVOH compounds cannot be identified/isolated using this method. But rather, each positive tube reading represents the sum total of all CVOH compounds present in the sample as "total CVOHs". This detection of the entire class of compounds is an inherent effect of the colorimetric tube design and thus may not be avoided by any alteration of method procedures.

Water Vapor. A build-up of water vapor in the colorimetric tube in the oxidizer stage (black portion of the tube) and through the catalyst stage (white portion of the tube) can induce a subtle color change similar to that of a low-level positive result if the moisture reaches the reagent stage (yellow portion of the tube). This problem is easily avoided by observing the build-up of condensation inside the tube in the oxidizer stage during purging, and stopping the airflow before the condensation reaches the white catalyst stage. This condition rarely occurs before the maximum required purge volume of 200 CCs is achieved and contaminant presence or absence has been determined.

Hydrogen Chloride Vapor. Hydrogen chloride vapor is the reactant that causes the color change in the yellow reagent used in the PCE colorimetric tubes. The HCl vapor is formed when chlorinated halocarbons pass through the oxidizer and catalyst stages of the tube. Free HCl vapor can also be formed when strong hydrochloric acid comes into contact with air or calcium carbonate. Any source of free hydrogen chloride vapor which enters the colorimetric tube will cause a strong positive reaction. To minimize the risk of false positives from hydrogen chloride vapor, avoid the use of pre-preserved VOAs when using the Color-Tec method. Natural sources of hydrogen chloride vapor are rare.

Free Chlorine. Very high (>20,000 ppm) concentrations of free chlorine can cause a low-level positive reaction in the 133LL colorimetric tube. The conditions necessary for this positive interference rarely occur in groundwater or soil samples.

Contaminant Carryover. It is highly recommended that VOA vials and extraction needle assemblies be discarded following each test. Re-use of these expendable items may cause sufficient carryover of contaminants to cause a false positive result in subsequent samples.

8.4 Ambient Air Interference

Because the Color-Tec method uses ambient air as the purge gas, airborne chlorinated compounds at low concentrations can enter the sample and cause a positive reaction in the detector tube. Conversely, low concentrations of either toluene or xylenes present in the ambient air may enter the colorimetric tube and inhibit/diminish the tube's ability to detect CVOHs. To prevent airborne contaminants from entering the sample and detector tube during sample purging and analysis, the method is used with a carbon pre-filter attached to the purge needle. To determine whether airborne chlorinated contaminants are present, a PCE (133LL) colorimetric tube may be used periodically to test the ambient air at the location where the field testing is being performed. If airborne contaminants are present and the carbon filter is being used, the carbon filters can also be tested periodically using a colorimetric tube to determine if breakthrough is occurring. The ambient air may be similarly tested for the presence of xylenes or toluene using the PCE (133LL) colorimetric tube.

To conduct a test for the presence of chlorinated VOHs in the ambient air, break the tips of a PCE (133LL) or PCE (133LL) colorimetric tube and properly insert it into the hand pump. Pull and lock the pump handle into the 100cc position allowing ambient air to enter the colorimetric tube. *Note: Do not attach an extraction needle assembly to the colorimetric tube while performing this test.* Once the 100cc flow cycle is completed, carefully read the tube and record the results. A positive result indicates the presence of CVOCs in the ambient air at concentrations detectable by Color-Tec which would affect sample results unless the carbon filter assembly is attached to the purge needle (see Section 9). A negative result indicates that CVOCs are not present in the ambient air at concentrations detectable by Color-Tec and therefore will not affect sample results. It is recommended that the carbon filter assembly is used regardless of the ambient air testing results.

8.5 Duplicate Sample Testing Procedure

Duplicate or replicate samples are collected from the same sampling location, at the same time, using the same collection methods, and analyzed using the same procedures as the original samples for the purpose of determining both sampling and analytical method variability. Since a

second (duplicate) VOA vial is always collected for the Color-Tec method, a duplicate or replicate analysis may be performed on the second (duplicate) VOA vial any time that a positive result (color change) is evoked by the original sample (first VOA vial) without exceeding the upper limit of the low-level colorimetric tube. In those cases, the duplicate or replicate analysis is simply performed by using a new low-level colorimetric tube to analyze the duplicate sample in the second (unused) VOA vial. If sampling and method variability is low, the result of the duplicate test will be the same or similar to the results obtained from the original test. The relative percent difference (RPD) may be calculated to quantify any variability in the results.

8.6 Collection of Split Samples for Laboratory Analysis

It is recommended that sample splits be collected for laboratory comparison analysis from 5 to 20 percent of the total quantity of samples analyzed using the Color-Tec method. Given a sufficient quantity of split sample pairs and sufficient range of concentration values, the GC/MS-to-Color-Tec comparison data may be used to obtain estimated concentrations for samples in the data set which were analyzed only using the Color-Tec method. This can be achieved using linear regression analysis of the comparison data. Statistical analysis of the comparison data can also be performed to determine site-specific Color-Tec method performance data.

9.0 Safety Precautions

As with the use of any product, it is recommended that the user carefully review all product manuals and Material Safety Data Sheets (MSDS) provided with this product prior to use. Several components of the Color-Tec kit are products obtained from other manufacturers which have manuals including safety precautions. Users of the Color-Tec method should carefully review the manuals and safety precautions and should become familiar with the proper use of all components included in the Color-Tec kit. It is recommended that the procedures involved with the method be incorporated into the user's Site-specific Safety and Health Plan (SSHP). MSDSs for all chemicals provided as part of the Color-Tec kit are available upon request. The following precautions should be considered to reduce potential user safety risks associated with the performance of the Color-Tec method.

Activity	Potential Risk	Precaution
Breaking tube tips	eye injury, dermal puncture	safety glasses
Accidental tube breakage	dermal cuts, exposure to reagent	safety gloves
Use of purge/extraction needles	dermal puncture	use caution
Use of the hot plate	dermal burns, electric shock	limited setting
Use of PCE standards	dermal contact, dermal cuts	safety gloves

Additional Safety Notes:

- Use skin and eye protection while breaking colorimetric and carbon filter tubes;
- The thermostat dial setting of the Corning® Hot Plate should never be set above 5 for any heating purposes required by the Color-Tec method;
- Do not over-fill the water bath pan while heating the samples and tubes;
- Always conduct sample and tube heating activities on a flat, stable, surface.
- Keep all flammable or combustible materials away from the Corning® Hot Plate during sample and tube heating activities.
- Always use the stainless-steel water-bath pan properly filled with water for heating the samples and tubes – do not heat samples or tubes directly on the surface of the Corning® Hot Plate;
- Do not use any heat source to heat the water-bath, tubes, or samples other than the Corning® Hot Plate provided in the hardware kit.

Disposal of Expendable Materials:

- Re-cap all needles before disposal;
 - After re-capping each extraction needle, dispose of the extraction needle assembly while leaving the vinyl tubing attached to the colorimetric tube – Do not attempt to remove the extraction needle assembly from the tip of the colorimetric tube for disposal.
 - Place all used extraction needle assemblies and colorimetric tubes into a plastic container for disposal. A used wide-mouth drink bottle is usually sufficient.
 - Dispose of all sharps (needles and broken glassware) in accordance with any and all applicable local and/or federal rules or guidance.
 - Dispose of all colorimetric tubes as specified in the Gastec® MSDS and/or in accordance with any and all applicable local and/or federal rules or guidance.
 - Dispose of all VOA vials used to contain sample materials in accordance with any and all applicable local and/or federal rules or guidance.
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EPA Guidance Document References

Using Dynamic Field Activities for On-Site Decision Making
May 2003; OSWER No. 9200.1-40 EPA/540/R03/002; Chapter 5;
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<http://www.epa.gov/swertio1/download/char/procurement.pdf>

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EPA Triad Implementation References

Using Color-Tec for Source Identification and Delineation
Naval Construction Battalion Center Davisville North Kingstown, RI - 2008 Triad Conference;
http://www.umass.edu/tei/conferences/Triad_PDF/Anderson.pdf

Fast Track to Reducing Conceptual Site Model Uncertainty CH2MHill; Storage Tank Site ST-123 POL Fuel Yard;
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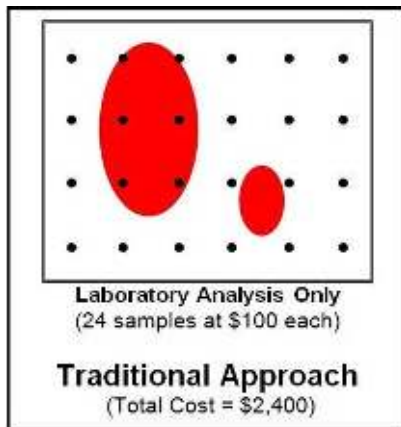
Successful Triad Implementations at Federal Sites Color-Tec Method locates source areas at Calloway Drum Recycling Site, Auburndale, Florida http://www.triadcentral.org/user/doc/TPP-Callaway-Field_Based_Decision_Approach.pdf



Color-Tec Method Applications

Source Area Identification at chlorinated solvent sites is highly complex given the low solubility of these compounds in water. Chlorinated solvent source zones often persist as suspended residual in unsaturated and saturated subsurface sediments for many decades. Surface water infiltration and groundwater flowing through the source zones slowly dissolves the suspended residual solvent leading to substantial aqueous phase contaminant plumes. Given the high volatility of most chlorinated compounds, residual solvents suspended in the unsaturated soil often leads to significant vapor phase contamination. The Color-Tec method is ideal for locating chlorinated solvent source areas by combining low level detection of all chlorinated compounds with low per sample cost to allow for significant expansion of sampling coverage compared to assessment approaches where only definitive analytical (laboratory) methods are employed to locate source areas. Definitive laboratory analysis provides high analytical accuracy, but sampling quantity is often limited to control costs, resulting in data gaps, sampling uncertainty, and low overall data quality. The low per-sample cost of Color-Tec method offers a 6:1 increase in analysis volume over laboratory methods, allowing for five times the sampling coverage for the same cost.

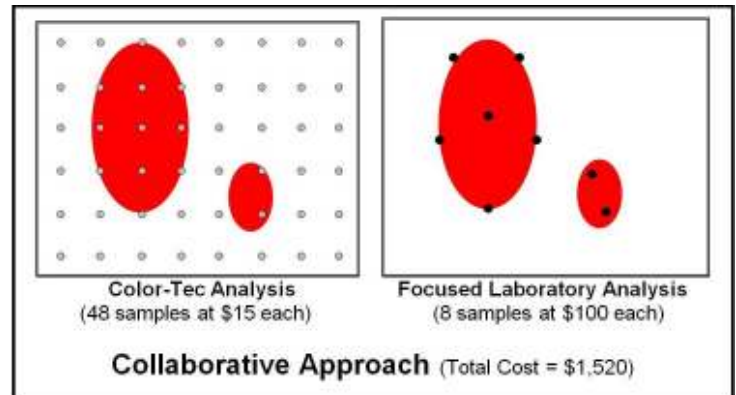
The illustrations below compare the traditional approach of source identification which uses only definitive laboratory analysis, to a collaborative approach which uses a high volume of Color-Tec data combined with a low quantity of definitive laboratory data. This collaborative approach combines high volume/low accuracy with low volume/high accuracy to achieve higher overall data quality than either method alone.



The diagram to the left shows the traditional site investigation scenario in which all samples collected are analyzed using only definitive analytical methods. The red areas represent previously unidentified source areas and black dots represent sampling locations intended to locate and delineate the contaminant plumes. Although this definitive-analysis only approach provides high analytical accuracy, the sampling quantity is often limited in order to control costs, resulting in data gaps, sampling uncertainty, and low overall data quality – and in this example the smaller source area remains undetected.

The two diagrams to the right show an investigation scenario in which a real-time measurement method, such as Color-Tec, is used to increase the overall sampling coverage,

resulting in reduced sampling uncertainty and increased overall data quality. In this example, the smaller source area is identified and the Color-Tec data is verified and confirmed by focusing a reduced quantity of definitive, laboratory-based, analysis of split-samples onto the most critical areas of the site. Combining Color-Tec with focused laboratory analysis in this manner provides increased overall data quality and analytical accuracy at significantly lower costs than conventional approaches which rely only on definitive laboratory-based analysis.



Groundwater Profiling is the collection of discrete samples at multiple depths and locations working outward from known source areas to define the lateral and vertical extent of a dissolved groundwater contaminant plume. The technique is used in conjunction with the Color-Tec method at chlorinated solvent sites to allow for immediate decisions regarding subsequent vertical and lateral sampling locations.

Soil Matrix Profiling is similar to groundwater profiling, but uses sampling of the unsaturated soil to define the lateral and vertical extent of the vapor phase contamination.

Groundwater Matrix Profiling (Residual Zone Mapping) is similar to groundwater or soil profiling, but uses sampling of saturated unconsolidated aquifer matrix to define the lateral and vertical extent of suspended residual DNAPL.

Surface Water/Sediment/Pore Water Impact Evaluation is the collection and analysis of sediment, sediment pore water, and surface water to locate and characterize groundwater impacts on surface water.