PHOTOVAC 10A10 PORTABLE GAS CHROMATOGRAPH OPERATION

SOP#: 2107
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1.0 SCOPE AND APPLICATION

This standard operating procedure (SOP) pertains to the use, calibration and maintenance of the Photovac 10A10 portable Gas Chromatograph. The Photovac 10A10 Gas Chromatograph is used for field and laboratory screening of air, soil gas, water, and soil headspace samples for chlorinated and nonchlorinated alkenes and aromatic hydrocarbons down to the 1 to 20 part per billion (ppb) range.

These are standard (i.e., typically applicable) operating procedures which may be varied or changed as required, dependent on site conditions, equipment limitations or limitations imposed by the procedure or other procedure limitations. In all instances, the ultimate procedures employed should be documented and associated with the final report.

Mention of trade names or commercial products does not constitute U.S. EPA endorsement or recommendation for use.

2.0 METHOD SUMMARY

The Photovac 10A10 is a battery/AC operated photoionization detector (PID) portable Gas Chromatograph. It is a field instrument capable of monitoring for many organic vapors using an ultraviolet light source and a photoionization detector. Gaseous contaminants are ionized as they emerge from the column. The ions are then attracted to an oppositely charged electrode which causes a current and electronic signal to a strip chart recorder.

The samples will be introduced into the 10A10 via gas tight syringes. As the compounds are detected by the PID, the resulting response will be recorded by an attached strip chart recorder or alternately, an integrator/plotter system.

3.0 SAMPLE PRESERVATION, CONTAINERS, HANDLING, AND STORAGE

This section is not applicable to this SOP.

4.0 INTERFERENCES AND POTENTIAL PROBLEMS

This instrument is not to be exposed to precipitation or high humidity. Liquids are not to be injected into this instrument. The instrument is best utilized in a stable, temperature controlled environment. It is advisable to avoid combustion fumes while using the instrument in the field because they can contaminate the columns. Readings can only be reported relative to the calibration standard used. High alkane concentrations may interfere with the resolution and detector response of early-eluting chlorinated alkenes, and aromatic compounds.

Since the Photovac is a GC, the target compounds are identified by their retention times (RT). If the RT of the sample peak(s) match the RT of the standard peak(s) they are assumed to be identical. If any non-target compounds has the same RT, it can be misidentified as a target compound.

5.0 EQUIPMENT/APPARATUS

The following equipment is required for Photovac operation:

- Photovac 10A10 Gas Chromatograph, with manual and power cord
- Extra source lamp
- Photovac lamp tuning screwdriver
- Extra columns/fittings
- Ultra Zero Air carrier gas
- Two stage regulator, with quick-connect fitting

- One flowmeter per Photovac (either bubble-meter, rotameter, or Gilibrator)
- Septa, 6 mm diameter
- Syringes, gas-tight, 10 µL to 1 mL
- VOA vials filled with activated charcoal, for syringe cleaning
- Integrator or strip-chart recorder, with appropriate connections
- Labels
- Tools - large adjustable wrench, wrenches (5/16" to 9/16"), screwdrivers (Flat head and Phillips head), nosepliers, jeweler's screwdrivers, Allen wrenches
- Duct tape
- Teflon tape
- Power strip
- Snoop
- Kimwipes

6.0 REAGENTS

- Carrier gas cylinder (compressed ultra zero air, 0.1 ppm total hydrocarbons)
- Headspace calibration standards - Supelco A and B or equivalent
- Certified gas calibration standards with a ±2% level of accuracy (Scott Speciality Gas, Matheson Gas, or other reliable sources)

7.0 PROCEDURES

7.1 Laboratory Operation

1. The carrier gas (air) cylinder is attached to the 10A10 and a maximum of 40 psi is delivered via the second stage of the dual stage regulator.

2. The flow rate will vary according to the target compounds in question and the column used. The carrier gas flow is adjusted using the curled knob to the left of the 10A10, labeled Column 1 or Column 2. The flow is measured by attaching a flow meter to the vent port at the top left of the unit (Figure 1, Appendix A). Once the flow is set, the PID will stabilize after approximately 1/2 hour warm-up time. The output is then set at 10 mV using the offset knob in the center of the unit.

3. An interface cable is attached from the output lead on the 10A10 to a strip chart recorder or more preferably a plotter-integrator such as the Hewlett Packard 3396A. The voltage input and/or attenuation is selected on the chart or integrator to keep peaks on scale. Check that the electrical controls are set as follows:

   a. Power Switch to "Off"
   b. Charge Switch to "Off"
   c. Attenuation Switch to "100" (lowest sensitivity)
   d. Offset dial to "zero"
   e. Connect chart recorder to the coaxial Output connector
   f. Set the chart recorder to 100 mV full scale and chart speed to 1 cm/min
   g. Plug the Power Cord into the panel socket and the red AC indicator light will come on.
   h. The instrument is now in its Power Down condition and is ready for starting.

4. With the chart recorder off, switch on the Power switch. The red source off indicator will light and stay on for up to five minutes. During this time, the lamp-start sequence is being automatically initiated.

5. As soon as the source off light is extinguished, the meter should show a high reading which should fall as conditions in the photoionizing chamber stabilize.

6. Establish an acceptable base line on the chart recorder.

The instrument is now ready for calibration.

7.2 Calibration Procedure

Photovac Incorporated conducts an instrument calibration and includes the chromatogram as a component of that instrument's instruction manual. A check of the instrument's performance can be accomplished by duplicating the factory calibration check and comparing the results. Refer to Appendix B for Calibration and Maintenance Schedule. The procedure is as follows:

1. Take a clean 1-L sample bottle, or a clean 1-L Tedlar bag fitted with a septum cap, and completely flush with good quality bottled air.

2. Go to the factory calibration data sheet and calculate the required amounts of each calibration compound required to generate an air standard (1-L total volume) which is identical to that run by Photovac in the factory calibration.

3. Using an appropriate volume gas-tight syringe, aspirate the required amounts of each compound from the headspace of the storage bottles at room temperature, and inject it into the purged 1-L sample bottle. Be careful to fully flush the syringe with clean air between each compound.

4. Allow 10 minutes for the standard to equilibrate.

5. Using a clean 100-µL gas-tight syringe, aspirate the required injection volume from the 1-L standard. With a crisp and snappy action, inject the standard into the proper "injection port" of the Photovac 10A10.

6. Start and mark the strip chart recorder. The resulting chromatogram should be similar to the factory calibration chromatogram, under similar conditions.

7. A simple calibration curve can be constructed by injecting the same volume of several standards with varying concentration levels of the target compounds. Alternatively, a calibration curve can also be constructed by injecting various volumes (10-1,000 µL) of the same standard. In this case the response of the standards and samples should be normalized to one injection volume. Both standards and samples present in Tedlar bags can be diluted in the field.

7.3 Field Operation

1. Prior to any field analyses, check to ensure that the instrument is operational and clean. Remove closure fittings on the "Detector Out" port. Closure fittings may have been engaged to prevent static contamination.

2. Check that the lecture bottle gas supply is adequate (charge supply is 1800 psi and should last approximately three days or less depending on carrier flow rates).

3. Set the pressure regulator to zero (fully counterclockwise) and turn on the main valve of the lecture bottle.

4. Slowly turn the regulator control clockwise until air begins to escape from the quick-disconnect connection. Allow the line to purge for 10 seconds.

5. Plug the quick-disconnect fitting into the free "carrier in" port. Shut off and disconnect the laboratory air supply. Adjust the lecture bottle regulator to 40 psig. Set the required flow rate as described previously using a bubble meter, calibrated rotameter, or Gilibrator.

6. With the instrument in the power down mode, disconnect the AC power supply. This automatically switches the instrument to battery power. The instrument is now completely self-contained, and together with a battery powered recorder, may be taken into the field. Check the battery charge on the Photovac.

7. The instrument is now ready to be run through the start up procedures described under Laboratory Operation, Parts 4-8 of the Manual.

8. If there are significant changes in ambient temperature when moving the instrument from place to place, the column will require time to stabilize thermally. At higher sensitivities, a non-thermally stabilized column will manifest itself as baseline drift.

9. For troubleshooting information, refer to Appendix C.

7.4 Shut Down

1. Turn the Power Switch to "Off".

2. Reduce the carrier gas flow to 2-5 cc/min.

3. Place instrument on low charge while on the bench, and maintain as described in Maintenance and Calibration Schedule Section.

4. Unplug the unit except when charging batteries.

8.0 CALCULATIONS

1. Calibration Curve

A calibration curve of at least three (3) concentrations must be constructed for each target compound. A straight line equation in the form of y = (m)(x) + b (where: x = concentration, y = area counts, m = slope, and b = the intercept) is fit to the standards raw data. The (x), or the unknown concentration for the sample, is determined from the above straight line equation. Non-linear data is indicative of detector response range limitations.

Alternatively, sample concentration can be calculated as below:

$$[Sample] = [Std]\frac{(A_1)(V_2)}{(A_2)(V_1)}$$

A_1 = peak area of sample
A_2 = peak area of standard
V_1 = injection volume of sample
V_2 = injection volume of standard

2. Standard Response Generation/Duplication of Factory Calibration Date

If appropriate gas standard mixtures are not available, gas standards can be made using the headspace from 40-mL VOA bottles, with Teflon-lined septa screw caps, partically filled with the desired neat volatile liquid. Factory instrument response is generally determined using the following three compounds:

Compound	P_{VAP} 20EC
methylene chloride	347 mm Hg
n-hexane	126 mm Hg
benzene	74 mm Hg

These compounds are toxic and should be stored and worked with under a hood. The general formula for preparing a standard from the headspace above a volatile liquid is:

$$V_{HS} = \frac{760}{P_{VAP}}(C)(V)$$

where:

V_{HS} = Volume of headspace (µL)
P_{VAP} = Vapor pressure of liquid (mm Hg) (Use appropriate tables to determine compound vapor pressure if working environment temperature is not 20EC.)
C = Desired concentration (ppm)
V = Volume of standard vessel (liters)

A pre-determined volume of neat liquid headspace may be introduced to the standard vessel through the septa if using a Tedlar bag with the appropriate fitting. Bags or vessels used should be labelled with content concentrations, date, and time of preparation.

9.0 QUALITY ASSURANCE/ QUALITY CONTROL

There are no specific quality assurance activities which apply to the operation of the Photovac. However, the following general QA procedure applies:

1. All instrumentation must be operated in accordance with operating instructions as supplied by the manufacturer, unless otherwise specified in the work plan. Equipment checkout and calibration activities must occur prior to sampling/operation and they must be documented.

10.0 DATA VALIDATION

This section is not applicable to this SOP.

11.0 HEALTH AND SAFETY

When working with potentially hazardous materials, follow USEPA, OSHA, and corporate health and safety practices.

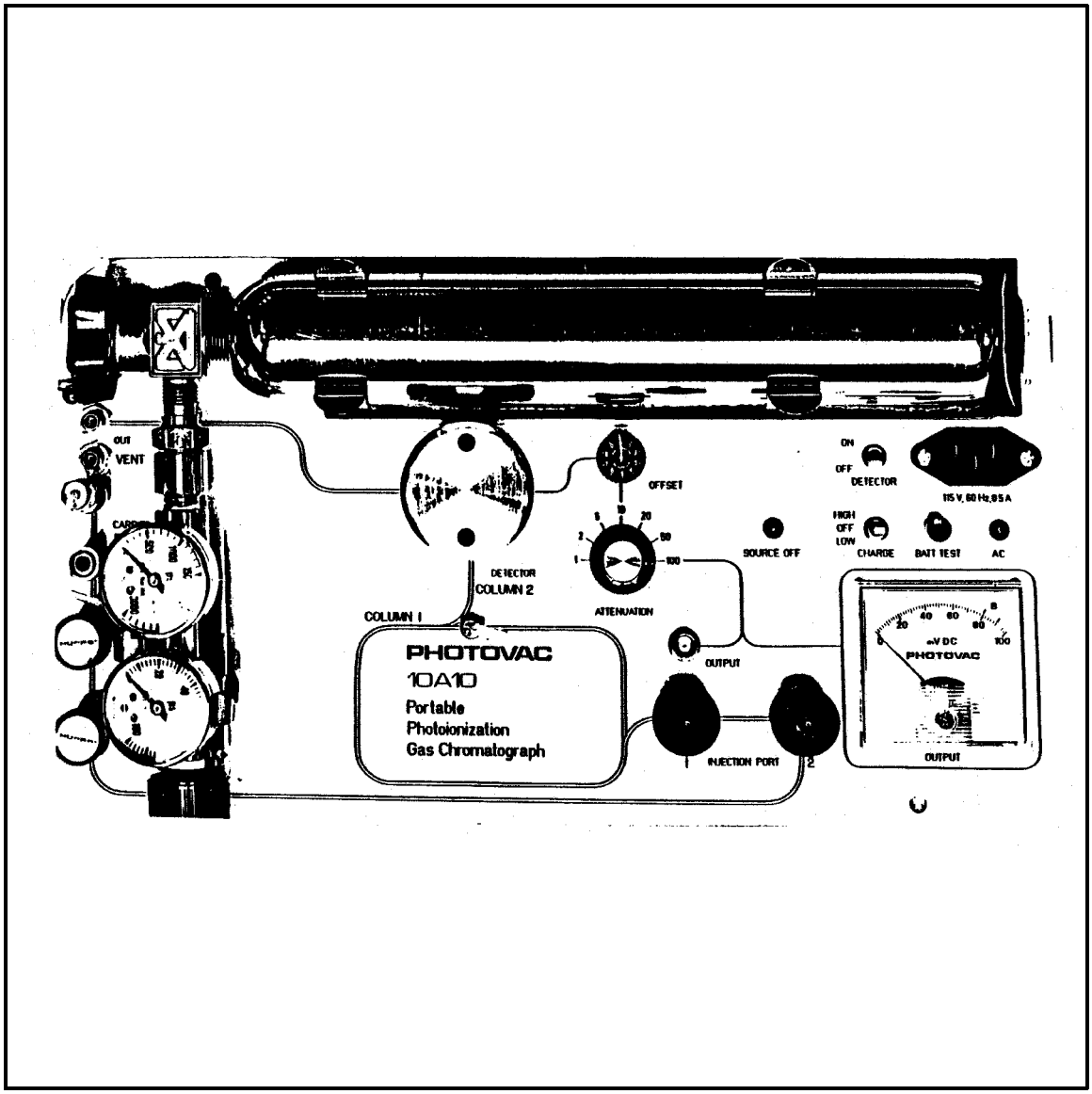
12.0 REFERENCES

Photovac 10A10 Operations Manual, Photovac International, Thornhill, Ontario, Canada L3T 2L3.

APPENDIX A

Figures

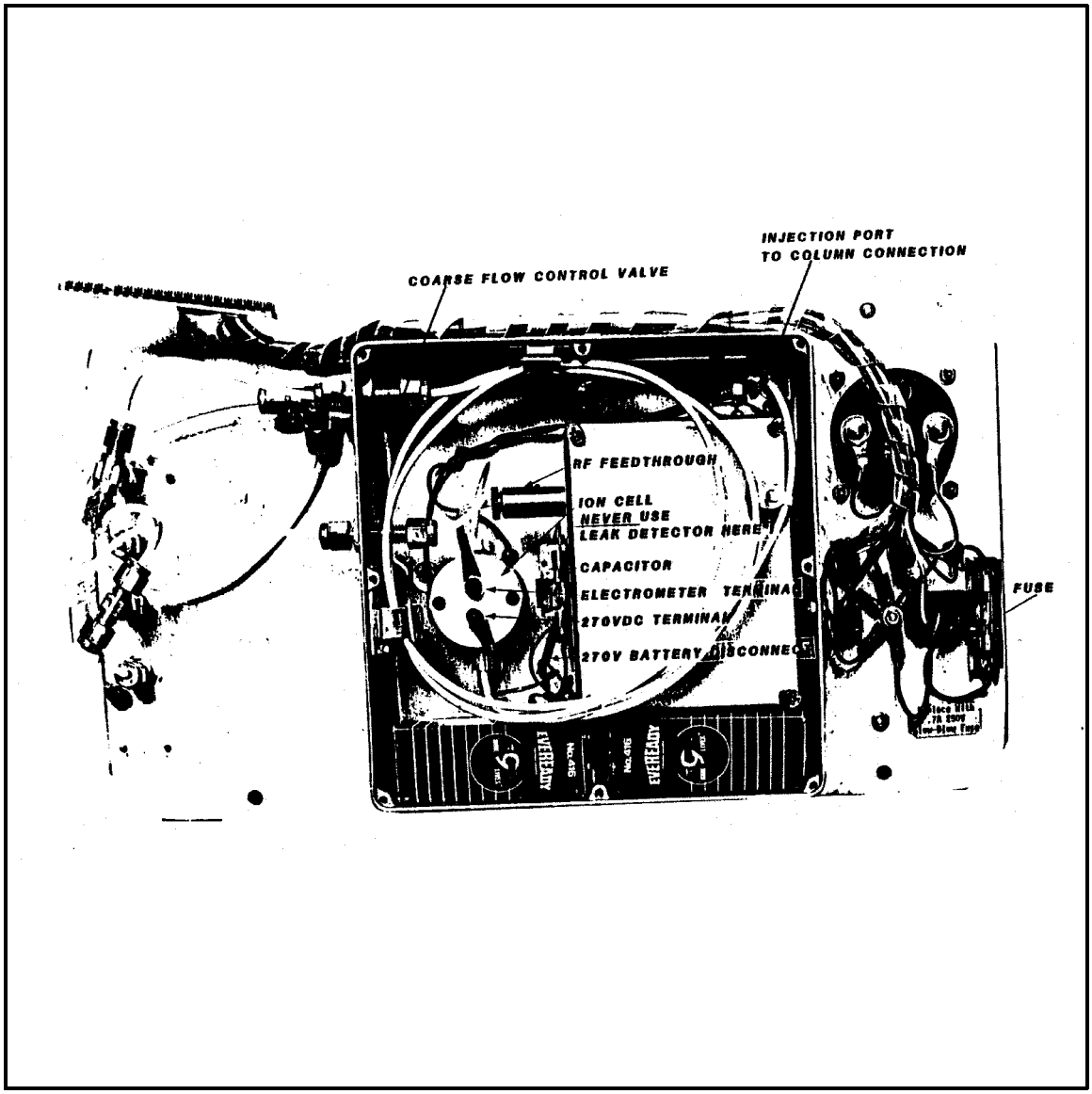
FIGURE 1. Instrument Panel



APPENDIX A (Cont'd)

Figures

FIGURE 2. Ion Cell Chambers



APPENDIX B

Maintenance and Calibration Schedule

Maintenance and Calibration Schedule

Function	Frequency
Battery charge when instrument has been operating exclusively on wall current with no use of battery.	Every three months for 10 hours on LOW
Battery charge when instrument has been operated off batteries.	After each use, 1 1/2 hours of high charge for every hour of use (Do not over-charge)
Calibration	With each 24 hour period of use
Septum Change	After approximately 40 injections
Column Reconditioning	Every three months or after heavy use, or when installing a new column

All maintenance and calibration functions should be documented

Septum Change

The 10A10 uses a Teflon faced silicon rubber 6 mm diameter septum. Hamilton "Micro Sep" F-138 is suitable. The septum can easily be replaced as follows:

1. Unscrew septum retainer.
2. Remove the old septum using the needle of one of the gas tight syringes available for sample injection.
3. Inset the new septum, teflon face down.
4. Carefully screw the retainer back into place firmly, but without overtightening.
5. A 10 to 20 minute stabilization period may be required because the carrier gas flow is temporarily interrupted when the septum is changed.

Column Maintenance

1. The standard Photovac 10A10 is equipped with a 4 ft. x 1/8" OD Teflon tube packed with 3% SE-30 on 80/100 mesh Chromosorb G suitable for running field surveys and analyses requiring detailed separations. Normally the column will be connected for manual operation.
2. New columns must be conditioned overnight with ultra-high purity helium (or nitrogen, at a temperature of 100EC, at a flow rate of 10 cc/min. Reconditioning of older columns is accomplished under the same conditions.
3. To access the column, utilize the following procedure:

    a. Never remove the panel while the instrument is connected to the main power supply.
    b. Disconnect the AC cord.
    c. Disconnect the chart recorder lead.
    d. Disconnect the lecture bottle carrier gas supply.

e. Remove the 4 Phillips screws securing the panel to the case and remove the screw attaching the lid.
f. Grasp the panel assembly by the cylinder clamp. Gently lift the rear of the panel clear of the case rim and ease the panel assembly backward from the front rim. Lift the panel assembly clear.
g. Gently remove the wire harness connection from the circuit board into which it is plugged. Remove the 9 Phillips screws from the gold box and lift clear the lid/circuit board subassembly. The interior of the column/ion cell chamber is now accessible (Figure 2, Appendix C).
h. To remove the column, locate the 2 compression fittings at either end of the column (ion cell body and injection part). Using a 5/6" open-ended wrench, loosen these fittings. Unscrew the fitting with the finger and remove column.
i. To replace the column reverse the previous steps and take special care not to damage the threads on the compression fittings. Fittings are made finger tight and then the 5/16" open-ended wrench is used to give an additional 1/8" turn to assure fitting seating.

APPENDIX C

Troubleshooting Guide

Troubleshooting Guide

PROBLEM	PROBABLE CAUSE	REMEDY
1. No Chromatographic Response	No carrier gas flow	Check at "OUT" port with flow gauge
	Batteries flat (if on battery operation)	Plug into AC and check again
	Electrometer staturated	Turn "attenuation" to, set meter to 0, if "Offset" reads 10 or more, the instrument is saturated
2. Unacceptable Baseline drift	Unit has been subjected to large temperature change	Allow to stabilize until clear
	A very concentrated sample has recently between introduced, resulting in excessive "tailing"	Allow to self-purge until clear
	Unacceptable contamination levels in carrier gas supply	Change carrier gas supply and allow instrument to stabilize
	The unit is charging and the resulting heat is affecting the column	Turn "Charge" switch to "OFF"
3. Deterioration of sensitivity	Syringe has leaky plunger	Try a new syringe
	Column needs conditioning	Condition column
	Septum leaking	Change septum
	Column fittings leak	Disassemble and check for leaks around fittings, while under pressure, with soap solution
	Deterioration due to ozone contamination after 1-3 years of operation	Decrease attenuation Replace detector
4. Unacceptable low Frequency noise	Column needs conditioning	Condition column

PROBLEM	PROBABLE CAUSE	REMEDY
5. Peaks elute very slowly	Carrier flow-rate is too slow	Increase flow-rate
6. Peaks eluting too fast	Carrier flow-rate is too high	Decrease flow-rate
7. Peak has flat top	Electrometer has saturated	Lower injection volume. Pre-dilute sample and repeat
8. Peak is asymetric with considerable tailing	Improper injection	Repeat
	Flow is too slow	Increase flow
	Improper injection technique	Repeat
	Peak is developing from earlier injection	Allow greater time between injections or install shorter column
	Compound is wrongly matched to column	Select appropriate column
	Power supply inadequate	
Source “OFF” light stays on after 5 min.	Batteries low (if battery operation)	Plug in AC connector
		Adjust potentiometer under aluminum cylindrical case by 45E clockwise increments
	Wire attachments to power supply not secure	Secure wire connections
	Tube driver mismatched	Contact Photovac for advice at (516) 351-5800
	Power supply inadequate	
9. Electrometer does not return to zero	Electometer saturated	Allow carrier flow for extended period without sample injection