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STANDARD OPERATING PROCEDURE

Conducting a Trapping Efficiency Study for Air Monitoring using Standard in Solvent

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KEY WORDS

Trapping media, bed height, sampling duration, air flow volume

APPROVALS

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Environmental Monitoring Branch organization and personnel, such as management, senior scientist, quality assurance officer, project leader, etc., are defined and discussed in SOP ADMN002.

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1.0 INTRODUCTION

When conducting air monitoring for pesticide residues the appropriate trapping medium, the duration of sampling, and the volume of air drawn through the medium need to be chosen. The selection of the trapping medium is generally made in consultation with the chemistry laboratory or by searching for developed methods for the pesticide. The laboratory will develop and test a method for extracting the pesticide (analyte) from the trapping medium. The trapping efficiency study follows method development at the lab. The kinds of trapping medium are discussed in SOP (Standard Operating Procedure) FSAI001.00.

Duration of sampling and volume of air are determined by many factors including toxicology, set tolerance levels, breathing rate and/or based on the ability of the trapping medium to trap the pesticide. The decisions made regarding medium, duration and flow must be tested to determine if the procedure is effective at trapping the pesticide. Additionally, environmental influences need to be examined such as the effect of temperature and/or humidity. Oxidation products should also be analyzed to determine the rate of conversion.

1.1 Purpose

This SOP describes how to conduct a trapping efficiency study to determine if the duration of sampling and air flow are appropriate for retaining the pesticide of interest in the trapping medium under similar environmental conditions as the proposed study. The type of trapping efficiency study described here is based on direct application of analytical standard dissolved in solvent as opposed to the analyte in a gaseous or air-borne particulate-adsorbed state. While results from this type of study provide information on the ability of the trapping medium to retain the analyte under controlled conditions, recoveries from solvent-based spikes may not necessarily reflect actual recoveries from gaseous field samples due to other effects (Biermann and Barry, 1999).

1.2 Definitions

1.2.1 **Bed height** refers to the height of the resin in a tube, cartridge or glass jar. Bed height refers to the distance the air is pulled through the trapping medium. For example, the same volume of resin can have an increased bed height by using a tube with a smaller diameter.

1.2.2 The **airflow rate** is the amount of air drawn through the trapping medium per unit time, usually expressed as milliliters per minute or liters per minute. The **total volume** of air drawn through trapping medium is the airflow rate multiplied by the duration of the monitoring.

1.2.3 **Sorbent sample tube** or **sorbent tube** refers to tubes, cartridges and glass jars that may be packed with a variety of trapping mediums such as charcoals and XAD resins.

1.2.4 **Break-through** occurs when the analyte (pesticide) is not completely retained on the first (primary) sampling tube in a series of sampling tubes. The primary sampling tube is located furthest from the air pump intake. Breakthrough is indicated by detection of the analyte in the second (or sometimes third) tube in a series. The second and third tubes are referred to as back-up tube(s), Numbering and naming of the tubes is discussed in SOP FSAI001.00.

2.0 MATERIALS

2.1 Sample pumps that will be used for field monitoring

2.2 Labeled sorbent sample tubes, in tandem or triplicate (described in FSAI001.00 in 3.4.2)

2.3 Equipment listed in SOP EQAI001.00 and EQAI002.00 to attach sorbent tubes to pumps

2.4 Air flow calibration equipment described in EQAI001.00 and EQAI002.00

2.5 Temperature monitoring device, preferably with a data logger (see EQWE002.00, EQWE003.00 and EQOT001.01)

2.6 Weather monitoring station if necessary (see EQWE002.00 and EQWE003.00)

2.7 Pesticide analyte standard prepared by the chemist, and supplies specified in the laboratory spiking SOP

2.8 Glass wool, or additional tube with glass wool to spike into

2.9 Latex gloves

2.10 Pen and tape for labeling pumps

2.11 COC (Chain of Custody) for each tube filled out as in ADMN006.00

3.0 PROCEDURES

After selecting the sorbent material, type of container, and flow volume needed, the next step is to conduct the trapping efficiency study to determine if the combination will be effective and also if it will work under the environmental conditions expected during the main study.

3.1 Determining spike level

3.1.1 There should be a minimum of at least 3 spike levels. At least 3 repetitions of each treatment combination should be used. More levels and repetitions should be used depending on factors such as how frequently the method is used, regulatory importance and the magnitude of estimated maximum concentration relative to the MDL. A treatment combination consists of each unique combination of sampling duration, flow rate, spike level, and any other factors studied.

3.1.2 The spike levels should be chosen to evenly span the range of expected concentrations in the field, with the lowest level close to the detection limit, the highest level comparable to the highest expected concentrations in the field, and the remaining level(s) distributed evenly between the low and high levels. While the range of detections in the proposed field monitoring is generally unknown, they can be determined based on experience or roughly estimated by data collected from similar analytes in the same trapping material, expected application rate for the planned field monitoring, and the physicochemical properties of the analyte. The project leader in conjunction with the laboratory and the Quality Assurance Officer should determine the spike levels.

3.1.3 Sampling duration is an additional variable that may be included in a trapping efficiency study by choosing different time intervals at the same spike level. Since a trapping efficiency study is a test of both duration of sampling and airflow rate with a particular trapping medium, the combination of these variables to be tested should encompass those to be used in the monitoring study.

3.1.4 Always include a matrix blank (un-spiked sorbent tube) as a control.

3.1.5 Inform the lab liaison and lab of the chosen spike levels so the lab can prepare the pesticide standard.

3.2 Environment selection and data recording

3.2.1 Site Selection

3.2.1.1 Choose the site for the trapping efficiency study based on expected environmental conditions during field monitoring. If hot weather is expected, conduct the trapping efficiency study outdoors in hot conditions. If the monitoring will be conducted indoors, the efficiency study should be conducted inside under similar conditions. If a wide range of weather conditions is expected (year round sampling), then trapping efficiency studies should be conducted in a variety of conditions. These may include rain, fog, and various temperatures.

3.2.1.2 Vehicle exhaust and volatile chemicals should be kept clear of the study site.

3.2.2 Weather data recording

3.2.2.1 Information about the weather should be recorded. Note the temperature range during the study, relative humidity, and other weather conditions (sunny, cloudy etc.).

3.2.2.2 A temperature data logger (EQOT001.01) is useful to collect temperature data over the duration of the trapping efficiency study.

3.2.2.3 A weather station with a data logger may be useful if the project leader decides detailed weather information is necessary for the trapping efficiency study (EQWE002.00, EQWE003.00).

3.3 Set-up

3.3.1 Select the same model air pumps as will be used for field monitoring. For the trapping efficiency study, one pump is needed for each sample and one extra pump for a blank matrix sample. A sample consists of the primary and back-up sorbent tube(s).

3.3.2 Calibrate sample pumps following EQAI001.00 and EQAI002.00 at the planned flow rate selected for field monitoring. The flow rate depends on the type of sorbent tube used, so calibrate with the same type and number of tubes to be used in the study.

3.3.3 Record airflow rate for each pump, generally on tape attached to the pump.

3.3.4 Set pumps at selected trapping efficiency study location. Set sorbent tube holders (usually some type of clip) at a height similar to the height that will be used in the field monitoring by using rods, camera tripods or stakes. A lab cart is useful for holding the pumps. Make sure all power needs (battery, or electrical) are met. Set-ups will vary based on the type of pumps used, location and type of sorbent tubes used. Figure 1 illustrates an example of a set up for a trapping efficiency study using Lo-vol XAD4 resin tubes and SKC AirChek HV30 sample pumps.

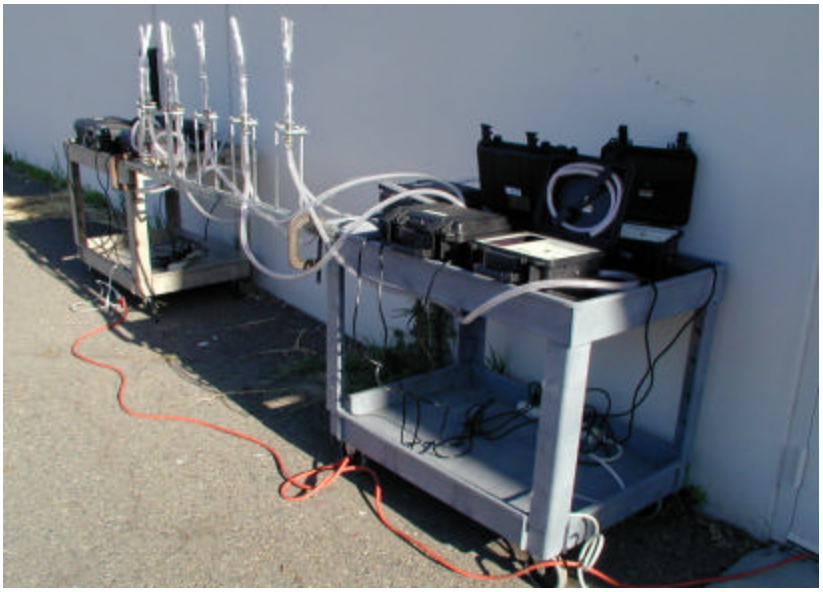

Figure 1. Equipment

3.3.5 Attach the primary and back-up tube(s) to the pump in series. Make sure the correct end of each tube is up (see SOP EQAI001 and EQAI002). The primary tube is the furthest from the pump air intake.

3.3.6 The spike is generally applied to glass wool or other material that is in the top of the primary tube. Alternately, an additional tube packed with glass wool may be attached to the front of primary tube and the spike applied to that additional tube. The Environmental Monitoring Branch has used both methods of spiking for trapping efficiency tests.

3.3.7 For relatively non-volatile analytes, the spiking procedure does not demonstrate the ability of the sampling system to collect the analyte from air, but does serve as a measure of potential breakthrough. The Branch has used the spiking method in 3.3.6 for analytes that are not expected to be in gaseous form, but may be adsorbed to particulates such as air-borne dust.

3.3.8 If the analyte is highly volatile despite being in solvent, the analyte may need to be spiked into an enclosed system. In this case the analyte may be spiked into an enclosed flask while an air pump draws air simultaneously from the flask with a sorbent tube in series. This method is similar to that utilized by Biermann and Barry (1999).

3.4 Spiking

3.4.1 Turn on all air pumps. Check the airflow rate with the calibrator, and record values on the COC. There should be a COC for every sample, and every tube must be labeled as described in FSAI001.00.

3.4.2 Generally a chemist should perform the spiking procedure. The chemist should wear disposable gloves and follow their laboratory SOPs for the spiking procedure. If necessary, the precision of the spiking technique can be determined by spiking five sample tubes with only glass wool and analyzing them.

3.4.3 The chemist then uses a syringe to spike appropriate amount of analyte into the glass wool of the primary tube, the extra glass wool tube or as described in 3.3.7 or 3.3.8.

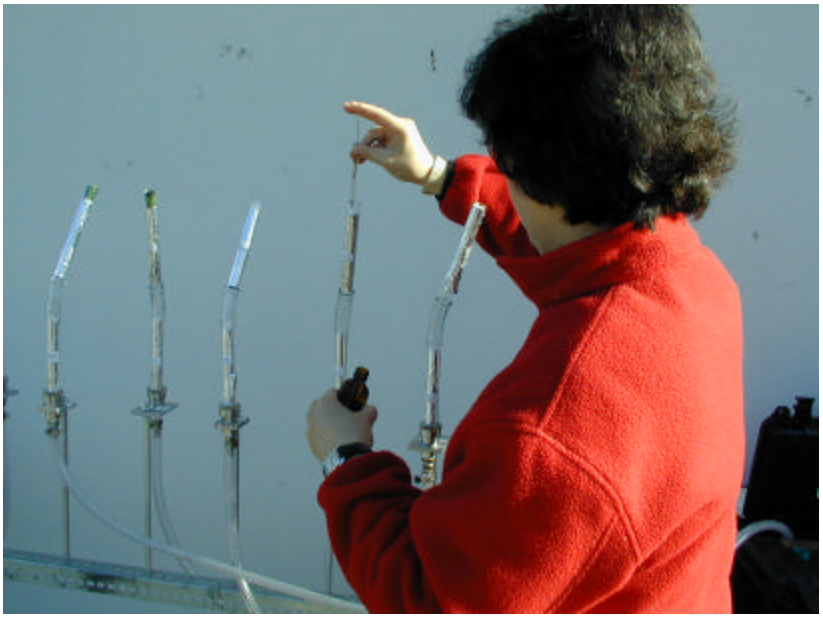

Figure 2. Chemist Spiking

3.4.4 Record the time of the spike and begin the air pumps immediately.

3.4.5 The pumps should run for the same time interval as selected for the field monitoring.

3.4.6 Periodically check pumps to make sure they are still operating. Check to make sure tubes are still attached. Record any problems or unusual conditions on the corresponding COC.

3.5 Ending trapping efficiency testing

3.5.1 Just prior to turning off the pumps and removing the tubes, take airflow rate readings with the calibrator. Then turn off one pump at a time and record ending flow, the ending time and runtime on the matching COC.

3.5.2 Remove the sorbent tubes from the pump one at a time. Cap each one prior to removing the next one.

3.5.3 Store the sorbent tubes as the method calls for, usually on dry ice or in a freezer until the laboratory can analyze them.

3.6 Analysis of Samples

3.6.1 Each sorbent tube must be analyzed separately. All primary tubes should be analyzed in the same extraction set to minimize variability. The back-up tubes can be analyzed in a second extraction set to determine if break-through occurred during the study.

3.6.2 All trapping efficiency samples should be analyzed as soon a possible to avoid storage dissipation.

4.0 REMEDIAL ACTION IN CASE OF MALFUNCTION

4.1.1 If a pump stops before the appropriate end time, record the run time listed on the pump, if available. Remove the sample tube(s) as per sections 3.5.2 - 3.5.3.

4.1.2 The project leader will need to determine if the sample results are usable or if some or all the trapping efficiency study needs to be repeated.

5.0 STUDY-SPECIFIC DECISIONS

5.1 Break-through

5.1.1 If there is no analyte detected in the back-up tubes, and there is satisfactory recovery appropriate for the analytical method, then the trapping medium, volume of air and time interval should be effective for field monitoring under the same environmental conditions.

5.1.2 If the analyte is detected in the back-up tubes, then the parameters need to be changed and a new trapping efficiency study needs to be conducted, or use a back-up tube in the field. If a back-up tube is to be used, a trapping efficiency study needs to be conducted with 3 tubes to ensure the first two tubes retain the analyte.

5.1.3 If the recoveries of the standard spike are low or not detectable, then the medium is unable to trap the analyte under the conditions of the study (assuming the sorbent material was tested in the lab and worked). Another sorbent material should be tested. If there is no sorbent material that works, at the discretion of the project leader and senior scientist, the method may possibly be used, but the poor recovery must be well documented and discussed in the monitoring report. All other alternatives should first be explored.

6.0 CALCULATIONS

6.1 Trapping efficiency

6.1.1 To calculate the total trapping efficiency use the following equation:

Primary tube recovery (µg/sample usually)=A
Back-up tube or tubes recovery=B
Amount spiked by chemist=C
Amount recovered in optional glass wool spiking tube (described in 3.3.6)=D

$$(A+B)/(C-D)*100=\%\ \text{trapping efficiency}$$

6.1.2 In general, the recovery in spiking tube D should be less than C. If this is not the case then the results are of limited applicability. Calculate % trapping efficiency for all replicates. Statistical analysis

of the data should be conducted to determine any systematic trends in trapping efficiency with spike level, run time, flow rate, or other factors studied. Assuming there is no effect due to these factors, calculate the mean and standard deviation of percent trapping efficiency based on all replicates.

6.2 Breakthrough

6.2.1 To calculate breakthrough use the following equation:

Back-up tube or tubes recovery (µg/sample usually) =B
Amount spiked by chemist=C
Amount recovered in optional glass wool spiking tube (described in 3.3.6)=D

B/(C-D)*100=% breakthrough

6.2.2 Calculate percent breakthrough for all replicates. Statistical analysis of the data should be conducted to determine any systematic trends in breakthrough with spike level, run time, flow rate, or other factors studied. Assuming there is no effect due to these factors, calculate the mean and standard deviation of percent breakthrough based on all replicates.

6.3 Percent conversion from parent analyte to metabolite.

6.3.1 Some analytes convert to metabolites during the process of air sampling. Percent conversion can be determined if only the parent analyte is spiked and the sample is analyzed for both the parent and the metabolite. If the spiking standard contains both the parent and metabolite, then percent conversion cannot be determined.

6.3.2 To calculate percent conversion use the following equation:

Primary tube recovery of metabolite (µg/sample usually)=E
Back-up tube or tubes recovery of metabolite=F
Amount spiked by chemist of parent analyte=C
Amount recovered in optional glass wool spiking tube of parent (described in 3.3.6)=D

Amount recovered in optional glass wool spiking tube of metabolite=G

E+F/C-D-G*100= % conversion

6.3.3 Calculate percent conversion for all replicates. Statistical analysis of the data should be conducted to determine any systematic trends in conversion with spike level, run time, flow rate, or other factors studied. Assuming there is no effect due to these factors, calculate the mean and standard deviation of percent conversion based on all replicates.

7.0 REFERENCES

Biermann, H. and T. Barry. 1999. Evaluation of Charcoal Tube and SUMMA Canister Recoveries for Methyl Bromide Air Sampling. Environmental Monitoring Branch Report EH 99-02. California Department of Pesticide Regulation.