

A Novel Downwind Odor Sampling Strategy for Transient Events; Combined Metalized-FEP Gas Sampling Bag, Sorbent Tube Transfer and Thermal Reconstitution

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**Written for presentation at the 2009 ASABE Annual International
Meeting Sponsored by the American Society of Agricultural and
Biological Engineers
Reno, Nevada
June 21 to June 24, 2009**

ABSTRACT

Downwind odor impact characteristics can be very different depending upon the size of the upwind point-source, interim topography and wind patterns. At one extreme, the downwind odor plume from a relatively large confined animal feeding operations (CAFO), located on a flat open plain and under stable, near-straight-line wind conditions can be rather broad, sustained and predictable relative to a fixed downwind receptor site. Conversely, the plume from a small point-source (e.g. such as a vent stack) located on irregular topography and under rapidly shifting wind patterns can be intermittent and fleeting. These transient odor events can be surprisingly intense and offensive, in spite of their fleeting occurrence and perception. This work reports on efforts to develop a downwind odor sampling strategy which is optimized for sampling of such transient odor ‘spikes’. This approach is based on combined air sampling with improved-material bags and preconcentration onto sorbent tubes. Initial results have been very promising. For example, approximate 10 fold increases in target odorant yields were realized for 900 mL sorbent tube transfers from 1-2 second ‘burst’ odor event bag-captures; when compared to equivalent direct collections at the same downwind receptor location but during perceived (stable) odor ‘lull’ periods. Results-to-date targeting refinement and validation of this integrated strategy for transient odor events are presented. Application to general odor sampling and point-source differentiation utilizing tracer gases is also presented.

Keywords: malodor analysis, agricultural odor, turbulent dispersion, GC-Olfactometry, GC-O, solid phase microextraction, SPME, multidimensional gas chromatography, MDGC, process odor, dispersion modeling

INTRODUCTION

The authors previously reported on downwind odor sampling challenges encountered relative to a study focused on the Carthage Bottoms Industrial Area (i.e. CBIA) located on the northern edge of Carthage, Missouri (Wright et al, 2008). As summarized in the previous report, much of the challenge associated with these downwind assessments resulted from the surprisingly transient, odor ‘wave’ characteristic encountered. These odor bursts were typically, momentary ‘hits’ or odor sensations of only a few seconds duration interspersed with long periods where the odor was undetectable or only faintly detectable. This characteristic appeared to be the result of: (1) the at-distance downwind assessment format of a dispersed plume from the primary odor source; (2) relatively small point-source(s) carrying primary responsibility for the priority downwind impact and (3) frequent and rapid shifts in wind direction. It should be noted that this situation differs from that associated with many CAFO sources (Wright et al, 2005) in that CAFOs as odor sources can be rather broad (e.g. large manure piles, compost heaps, lagoons or field manure applications). In contrast, high-impact odor sources relative to the CBIA are believed to be traceable to comparatively small (i.e. approximately 1 to 6 ft diameter) roof or elevated stack vents.

These transient odor events are believed to be manifestations of the non-gaussian plume dynamic which some dispersion modeling researchers have previously attempted to address (Roberts, P.J.W. et al, 2001, Yu, Z. et al, 2008). In fact, these efforts suggest that current mathematical models which describe downwind plume dynamics as uniform gaussian distributions (Pasquill, F.; 1976) may be limiting; especially with respect to the issue of downwind odor impact. Regarding air toxics, for example, time-weighted average exposures can carry real significance with respect to predicting the cumulative impact on the health of downwind citizens. In contrast, from the standpoint of these same citizens, odor impact is best characterized as pass / fail or on / off events. Priority odorant concentrations are either below or above the recognition (i.e. or annoyance) threshold at any given moment-in-time. In the case of the former, the citizen-receptor is not impacted in the least; with respect to the latter the frequency and intensity of these above-threshold excursions will determine the perceived quality-of-life impact.

As a result of the transient nature of the CBIA odor events, it was shown to be very difficult to achieve reasonable odorant / VOC loadings in air samples for subsequent analytical efforts. This was especially true utilizing the initial solid phase microextraction (i.e. SPME) approach. The integrated strategy reported below was developed as a potential alternative for greater accuracy in sampling of such transients odor ‘spikes’. These results integrate some of the findings and strategies emerging from the USDA funded SBIR project, including: (1) metalized-FEP gas sampling bags for maximum recovery of highest impact odorants; (2) the necessity for minimizing sample storage time in the vapor state and (3) the potential for integrating sorbent tube storage with metalized-FEP bag vapor ‘grab’ collection to achieve constraint (2).

MATERIALS and METHODS

Multidimensional Gas Chromatography-Mass Spectrometry-Olfactometry:

MDGC-MS-Olfactometry (MDGC-MS-O) is an integrated approach combining multidimensional GC separation techniques, parallel olfactory detection by a human sensory investigator and conventional electronic detection by mass spectrometry (Wright et al, 1997). A commercial, integrated AromaTrax™ system from Microanalytics (a MOCON Company) of Round Rock, Texas was used for the MDGC-MS-O operations. With respect to MS operation; the Agilent 5975 B was operated in selected ion monitoring mode (SIM) and targeted selected challenge odorants as well as their associated tracer compounds during the dual point-source prioritization experiments.

Sorbent Tube Sampling:

A series coupled sorbent tube pair was utilized for this testing. The fore tube was packed with @ 2 cm (0.022g) of Tenax TA; an adsorbent of moderate strength. The aft tube was packed sequentially, with @ 1 cm (0.009g) Carbopack B and @ 1 cm (0.009g) Carboxen; adsorbents representing sequentially increasing adsorbent strength. The integrated tracer gas injection / sample bag to sorbent tube transfers were carried out utilizing a prototype Peltier cryotrap device. This device was set to control at @ 2 °C for increasing the trapping efficiency of the Tenax TA fore trap.

SPME Sampling:

Solid phase microextraction (Pawliszyn, 1997; Chai and Pawliszyn, 1995 Chai and Tang, 1997; Koziel and Pawliszyn, 2001; Koziel et al, 2006) utilizing a 1 cm Carboxen modified PDMS - 85 µm fiber was the headspace sampling technique which was utilized for the initial, Phase I efforts. SPME collections were carried out by direct fiber exposure at-distance and downwind of the scale model transient event generators. Variations in exposure times was used for cross-comparison purposes. All SPME downwind collections were carried out under ambient conditions present at the time of target odor detection by the principal investigator.

Weather Monitoring:

A Kestrel 4500 Pocket Weather Tracker was used during transient event and source differentiation experiments with the prototype scale model transient event generators described below. This unit is tripod mounted, configured for wind direction monitoring and incorporates comprehensive data logging capabilities.

Scale Model for Generation of Transient Odor Events:

A prototype was designed to permit up to 4 target odorants to be combined at selected ratios prior to being ejected from a small vent stack under controlled flow conditions. The target odorants are placed into one of three generator cartridges in an appropriate form depending upon the targeted odorant and the goal of the experiment. These forms include measured amounts of high purity solids such as naphthalene, permeation tubes for odorants of high volatility and odorant saturated film or fiber carrier materials for odorant 'surge release' simulation. Each cartridge is affixed with a blower under independent rheostat control. In this case the blowers used are relatively inexpensive hair dryers. The vent stack and odorant cartridge assemblies were fabricated from 3 inch schedule 40 PVC

and associated fittings. The stack vent terminates @ 7 ft above ground level and is shown in **Figure 2** below.

Tracer Gas Injection Strategy for Point-Source Prioritization:

Each of two independently positionable transient event generators was configured to permit steady-state emission of one characteristic odorant and one associated tracer gas. Generator #1 was configured for controlled release of the odorant / tracer pair, naphthalene and chloroform. Generator #2 was configured for controlled release of the contrasting odorant / tracer pair, dimethoxybenzene / methylene chloride. The tracer compounds were injected under controlled conditions utilizing a variation of the automated vaporizing injection technique as previously described by the principal investigator for the vinyl chloride purity assay analysis (ASTM D-5507).

RESULTS and DISCUSSION

As described above, significant challenges were encountered in sampling the transient at-distance, downwind odor events characteristic of the CBIA. In an effort to expedite development of an improved sampling strategy, a small scale transient odor event simulator was designed, constructed and carried through initial experimental evaluation. The prototype, as shown in **Figure 2** below, was developed in an attempt, to compress the distance and time factors responsible for drawing out sampling strategy optimization. Initial experimental efforts were carried out utilizing a binary odorant system and results with the model were very encouraging. Utilizing contrasting odorants, high purity naphthalene with its 'mothball' odor and dimethoxybenzene with its character-defining 'bluebonnet field' aroma, it was possible to immediately achieve replication of the transient event effect. In addition, a steady-state condition of several hours duration was achieved with the following characteristics: (1) odor recognition threshold @ 70 feet; (2) the at-distance (i.e. 50 to 70 feet) odor character was clearly dominated by dimethoxybenzene and (3) near-source (i.e. 5 to 10 feet) odor character was clearly dominated by naphthalene. After initial evaluation of the prototype transient event generator the device was applied to the development of a sampling strategy alternative to direct SPME exposure for the sampling of downwind transient odor events.

A Two-Step Strategy for Sampling of Transient Downwind Odor Events:

A number of alternatives for transient event odorant sampling were considered in light of the limitations shown for the direct SPME approach. One alternative approach for concentrating high-impact odorants is by sorbent tube directly and, like SPME, this will work well for odor events which are somewhat sustained (i.e. CAFO). However, as a result of the normal flow restrictions of packed adsorbent tubes there is still a limit to the volume of air which can be processed during brief transient events. For example, assuming a 30 cc/min peak flow rate through a sorbent tube under full vacuum (i.e. ~14.7 psi pressure differential), approximately 30 min is still required for concentrating odorants from a 1000 cc air sample. Unfortunately, this is a relatively long period in relation to transient odor events such as those encountered relative to the CBIA. Another alternative, pictured in **Figure 1** below, is a two-step process which has been shown to achieve a reasonable compromise between sample volume requirements and time

constraints. This two-step alternative integrates gas-bag and sorbent tube techniques in the following manner: (1) rapid, 1 liter grab samples of 1 to 2 seconds duration are vacuum drawn into metalized-FEP gas sampling bags during perceived momentary peak odor events and (2) these whole-air collections are followed by either immediate sampling of the captured bag contents through extended SPME fiber exposure or immediate transfer of the bag contents to packed sorbent tubes for transport, storage and eventual lab analysis. As shown in **Table 1** below, a 4X increase in target odorant response was achieved for 30 min SPME fiber exposures to 1-2 sec ‘burst’ bag-captured odor events; as compared to 3 min direct exposures to the same downwind receptor-site. It is noteworthy that this collection series was made during an extended period of relatively stable wind conditions.



Figure 1: Transient odor event peak sampler process. To the right is the left-most generator configured for controlled emission of the naphthalene / chloroform odorant / tracer pair. The first author is shown awaiting the characteristic sensory cue for the targeted transient event. Utilizing the 1 L manual gas tight syringe, the 1 L m-FEP gas sampling bags could be completely filled in 1-2 sec during the sensory cued events.

m-FEP Gas Bag Grab Sample / Extended SPME Exposure		
Indirect Series	Naphthalene Response	Response Differential
Run #1	10,524	
Run #2	10,318	
Average	10,421	4 X Direct

Direct Series

Fiber #1	2,624	
Fiber #2	2,451	
Average	2,538	0.25 X Indirect

Likewise, as shown in **Table II**, an average 11 fold increase in target odorant yield was realized for 900 mL sorbent tube transfers from similar 1-2 sec odor event bag-captures. This increased response is referenced to equivalent 900 mL direct sorbent tube collections at the same receptor-site but during perceived interim odor ‘lull’ periods. It is believed to be noteworthy that the sample points were selected to be at the approximate geometric center between the plume lateral downwind boundaries.

Table II**m-FEP Gas Bag Grab Sample / Sorbent Tube Transfer**

Indirect ‘Peak’ Series	Naphthalene Response	Relative Response
Run #1	83,915	13.5 X
Run #2	54,851	8.8 X
 Direct ‘Lull’ Reference		
Run #1	6,216	1X

Tracer Gas Integration to Controlled Model Stack Emissions:

The optimized transient event sampling strategy described above is potentially applicable to a number of downwind assessment challenges. One of these challenges is in the downwind impact prioritization of multiple, closely co-located upwind point-sources. Simply stated; if an investigator is successful in prioritizing the specific odorants most responsible for negative impact at an at-distance receptor-site he should be able to use this information to shift focus backward to prioritize from among multiple, upwind point-sources. This current effort explores the use of optimized transient event grab sampling in conjunction with signature odorant or tracer spiking of discrete upwind point-sources. Sulfur hexafluoride (SF6) and perfluorocarbon tracer (PFT) compounds have been widely referenced (Dietz et al, 1982; Dietz, 1986) for such VOC dispersion and air movement profiling studies. However, with respect to this application, others may also be appropriate and could be selected based upon: (1) relatively low odor impact; (2) high chemical stability; (3) relative absence from the normal environmental background of the target area and (4) safety and environmental impact considerations. For the exploratory purposes of this current effort, chloroform and methylene chloride were selected as the tracer compounds. These selections were made solely on the basis of availability and appropriateness of physical and analytical properties rather than any perception of applicability to expanded full-scale studies.

To insure a high degree of precision in the rate of tracer compound introduction, the liquid form compounds were injected and vaporized under controlled conditions utilizing a variation of the automated vaporizing injection technique (ASTM D-5507). This technique was previously described for the industry standard vinyl chloride purity assay analysis and has been used extensively to achieve a high degree of precision in that

analysis. The process in summary is: (1) the liquid tracer feed reservoir is pressurized with nitrogen, well beyond the compound's natural vapor pressure; (2) the over-pressured liquid is fed through a fixed restrictor which terminates in the heated vaporization chamber and (3) control and limitation of the tracer feed-rate is achieved by matching the liquid feed head pressure with the restriction (i.e. length and ID) of a tubular fixed restrictor. The naphthalene / chloroform ratio data summarized in **Table III** below was carried out to determine the precision which could be achieved utilizing this approach for tracer introduction into the model stack emission. The data reflects the results from 5 sequential sorbent tube collections from model Stack #1 which was configured for odorant / tracer pair emission. The 8.6% RSD value was felt to be excellent considering the fact that it reflects the complete range of experimental variability; (1) the odorant / tracer vaporization process; (2) stack emission process; (3) meteorological variability; (4) the transient event downwind sampling process and (5) the analytical process. It is possible, although unproven at this point, that the consistent upward trending of the ratio values (i.e. increasing naphthalene response relative to that of the chloroform tracer) stems from rushing the collection process before achieving naphthalene emission equilibrium.

Table III
Odorant / Tracer Pair Response Ratio Precision

Run Number	Naphth / CCl3 Response Ratio
Run #1	0.84
Run #2	0.98
Run #3	0.96
Run #4	0.95
Run #5	1.07
Mean	0.96
sd	0.082
%RSD	8.56
n	5

Integrated Sampling Strategy for Upwind Odor Point-Source Prioritization:

Based upon the precision reflected in the above odorant / tracer ratio data, it was felt that point-source prioritization should be relatively straightforward. These results suggest that by coordinating; (1) transient event peak grab sampling; (2) priority odorant identification / detection; (3) tracer compound relative abundance (i.e. or relative absence) and (4) coincident meteorological conditions, a definitive source prioritization from among multiple 'potential' up-wind point-sources should be achieved. To explore and refine the process, an expanded field test was carried out utilizing two independently controlled and positionable transient odor event generator stacks. Stack #1, as described above, was configured for controlled emission of the naphthalene / chloroform odorant / tracer pair. In contrast, Stack #2 was configured for controlled emission of the dimethoxybenzene / methylene chloride odorant / tracer pair. **Figure 2** below shows the integrated generator system which was utilized for this point-source prioritization study.

Shown in the right foreground are the two point-source generators. To the left background is the data logging weather station.



Figure 2: Integrated multiple point-source transient odor event generator system. To the right are the two ‘discrete’ point-sources. to the left background is the tripod mounted weather data logger.

For this initial field trial, two contrasting conditions were targeted: (1) transient ‘mothball’ odor events, indicating a naphthalene concentration spike and (2) transient ‘bluebonnet field’ odor events, indicating a dimethoxybenzene concentration spike. Six, series coupled sorbent tubes were distributed to reflect, triplicate naphthalene ‘peak’ events and triplicate dimethoxybenzene ‘peak’ events. Unfortunately, weather conditions turned un-favorable for initiating the test but once set-up was begun, we were forced to continue. Specifically, an approaching cold front and degrading wind conditions forced accelerated initiation of the transient event sampling process. Under the rapidly deteriorating wind conditions (i.e. both wind speed and direction variability) the transient odor events were found to be particularly brief; 1 to 3 sec in duration, at best. Under the rushed conditions it was not possible to insure that the generators were allowed to reach emission steady-state before starting the downwind collections. As surmised previously, the resulting non-steady-state situation appears to be reflected in the generally ‘ascending’ naphthalene / chloroform ratio values for the ‘mothball’ odor peak series (i.e. 1.10, 1.00 and 1.52). Adding to the weather related challenge, another problem arose due to the fact that the second generator (i.e. dimethoxybenzene / methylene chloride) had not been carried through a post-fabrication check-out prior to initiating this field trial. As a result, a number of unexpected mechanical problems were encountered during set-up which adversely affected control of both methylene chloride feed and stack total flow.

Table IV
Transient Event Sampling with Tracer Gas Injection for Point-Source Prioritization

'Mothball' Event	Naphthalene	CCl3	DMB
Run #1	220,450	200,750	6,592
Run #2	653,343	650,217	390
Run #3	584,887	386,036	480
Mean	486,227	412,334	2,487
'Bluebonnet Field' Event			
Run #1	26,522	<dl	4,203
Run #2	35,053	5,580	84,373
Run #3	17,428	2,988	3,771
Mean	26,334	2,889	30,782

As a result of the above complications, this experimental series is only considered significant from a system tuning perspective in advance of subsequent, fully integrated, field trials. Further, it should be viewed in the context of a 3 component strategy for point-source prioritization. In this context, naphthalene represents the 'mothball' transient odor event target, chloroform serves as the tracer gas for point-source #1 and dimethoxybenzene serves as the sensory que for timing the sampling event for comparative point-source #2. This strategy is believed to be appropriate for those situations where the goal is to differentiate the relative downwind impact of a primary suspect point-source relative to that of a 'potential' alternate.

Within the constraints imposed by the above stated context, the data shown does provide a number of observations which are believed to be significant. In particular, the first three collections, reflecting 'mothball' queued transient events, reflect consistent correlations between the sensory and analytical data. The chloroform average response during the 'mothball' peak events was 74 fold higher than for the highest individual chloroform response and 188 fold higher than the average chloroform response values during the contrasting 'bluebonnet field' queued transient events. Likewise, the naphthalene average response during the 'mothball' peak events was 14 fold higher than for the highest individual naphthalene response and 22 fold higher than the average naphthalene response values during the 'bluebonnet field' queued events. In contrast, the naphthalene / chloroform response ratio values were 1.10; 1.00 and 1.52 for an average of 1.21 and sd of 0.28 for a %RSD of 23.1%. This level of variation is higher than expected (i.e. previous field trial series results were avg = 0.96, sd = 0.082, %RSD = 8.56% for n=5) or would normally be acceptable, but given the above challenges should probably be expected. Follow-up field trials are planned after correcting the mechanical challenges believed responsible for the conflicting data shown for the contrasting 'bluebonnet field' sampling events. Planned efforts will be directed at expanding to a 4 component point-source differentiation strategy with the integration of the methylene chloride tracer gas into generator #2. This strategy is believed to be significant in that it adds a second layer

of downwind odor impact priority verification; a cross-check between two 'potential' upwind point-sources. Results from these follow-up, fully integrated, experiments will be presented in a later conference.

Transient sampling strategy implications for field odor assessments by Dynamic Dilution Olfactometry:

Efforts-to-date relative to the assessment of transient odor events have primarily been directed at the analytical approach to priority odorant monitoring. However, the transient event characteristic also magnifies the challenge associated with follow-up investigation of citizen odor complaints utilizing human 'sensors' and Dynamic Dilution Olfactometry (i.e. DDO), (ASTM E-679, 2004; ASTM E-1432; 2004 and CEN/TC264, 1999).

Typically, agency officials receive a complaint from downwind citizen and put an investigator on the road to drive the miles to the complaint site. As often happens, in the minutes or hours that it takes for this official response, the odor impact will have shifted to a new location. Likewise, even if the event it is still perceptible, it is likely to be difficult to get an accurate fix on the 'odor dilution number' when dealing with such a rapidly shifting target. Work is currently underway to apply the above transient event sampling strategies to DDO-based downwind odor assessment. These range from: (1) immediate assessment of transient event 'peak' bag-capture samples utilizing field DDO devices to (2) field odorant collection on adsorbent tubes followed by thermal de-sorption based whole-air reconstitution in the laboratory; just prior to DDO assessment. The key consideration relative to all of these related strategies is the constraint that samples must only be held in the whole-air form for very short periods of time; just long enough for analysis or transfer to the sorbed form for shipment / storage. Semi-volatile odorant recovery data to-date suggests that this time limit should likely be in the range of 15 to 30 min; in marked contrast to the 24 to 36 hr constraint reflected in many current protocols. Results from on-going efforts directed at application of the transient event sampling strategy to DDO will be presented in a later conference.

CONCLUSIONS

The challenge encountered and addressed in this work was the special case of transient downwind odor events; brief events of, typically, only a few seconds duration in spite of relatively significant peak odor intensities. Attempts to address this unique challenge have led to the development of a prototype transient event sampling strategy. Simply stated, the concept is: (1) rapid fill of a metalized-FEP bag at the instant of a perceived odor event followed by (2) immediate transfer of the bag contents onto an adsorbent tube for transport and storage prior to in-laboratory analysis. Encouraging initial evaluation of the concept and device have been carried out utilizing scale model transient odor event generator devices. A 4 fold increase in target odorant response for a 30 min SPME fiber exposures to 1-2 sec bag-capture odor events was shown; as compared to 3 min direct exposures to the same downwind location. Likewise, a 10 fold increase in target odorant yield was shown for a 900 mL adsorbent tube transfers from 1-2 sec odor event bag-captures; when compared to equivalent 900 mL direct collections at the same downwind location during perceived interim odor 'lull' periods. Efforts, reported herein, have subsequently been directed at applying this transient event sampling strategy to the

challenge of point-source prioritization. Despite a number of unexpected challenges during the initial field-trial attempt, preliminary data indicates that odor impact prioritization from among multiple ‘potential’ upwind point-sources is possible. The critical elements of this strategic application are: (1) correct downwind odorant impact prioritization and identification; (2) contrasting tracer gas injection; (3) tracer odorant injections, as required for contrasting sensory cue purposes; (4) rapid, sensory queued transient event grab sample capture and (5) adsorbent tube transfer for analyte stability during extended shipment and storage periods.

ACKNOWLEDGEMENTS

This work has been partially funded by the US Department of Agriculture under SBIR Phase II Grant – CSREES Award number 2007-33610-18619 to Microanalytics (a MOCON Company). However, any opinions, findings, conclusions, or recommendations expressed in this presentation are those of the authors and do not necessarily reflect the view of the US Department of Agriculture.

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