CASE STUDY

Air Quality Monitoring

Customer Story

PerkinElmer Collaboration Helps Improve How Pace Analytical Services Collects and Analyzes Air Samples

Success of technology saves time, money, and raises the bar on partnerships

One of the largest testing laboratories in the U.S., Pace Analytical Services, headquartered in Minneapolis, MN, has provided over 35 years of scientific know-how, problem-solving expertise and state-of-the-art instrumentation to support a multitude of analytical testing requirements. The Environmental Services group has inorganic, organic and radiochemistry capabilities—specializing in the analysis of trace level contaminants in air, groundwater, drinking water, wastewater, biota and soil samples.

The Pace/PerkinElmer Partnership Providing Industry Solutions

The Pace air laboratory has been certified to run US EPA Compendium Method TO-15, for the Determination of Volatile Organic Compounds (VOCs) in Air Using Summa Canisters and Analyzed by GC/MS[1] for over ten years, and has a substantial customer base who requests this analysis. However, understanding the benefits of using Method TO-17, for the Determination of VOCs in Air Using Active Sampling on to Sorbent Tubes[2], they wanted to incorporate this technique into their portfolio. Pace had heard about PerkinElmer's Soil Vapor Intrusion tube (SVI) that extends the hydrocarbon range for the TO-15/TO-17 target compounds to pyrene, and made contact with them in July of 2010 to discuss instrumentation, tube technology and the necessary expertise required to run the analysis.

Both companies worked together to come up with experiments to prove that the new sorbent tubes, the thermal desorption procedure, and the analytical conditions were suitable for the analysis of the target list of compounds. Because of this successful partnership, the relationship grew into a collaborative research agreement expanding the use of TO-17 into additional areas of air monitoring.

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For the Better

The New Challenge

Pace laboratories are known for their innovative solutions to environmental challenges and are always looking for ways to improve testing procedures by forming partnerships with their clients and vendors to meet regulatory commitments, optimize methodologies and identifying process improvements wherever they can.

The air monitoring team at Pace approached PerkinElmer, because they wanted to respond to demands they were getting from their clients for an alternative to the traditional methods for the collection and analysis of BTEX (benzene, toluene, ethyl benzene and xylenes) and the 16 regulated PAHs (polynuclear aromatic hydrocarbons) in ambient air. One procedure in particular that required improvement was the testing of hazardous volatile organic compounds (VOCs) and semi-volatile compounds (SVOCs) in air at monitoring locations near manufactured gas plant (MGP) remediation sites around the country. When soil excavation of these sites occurs, fence line monitoring is required to quantify toxic components which could escape from the remediation site into the adjacent residential areas.

Currently, this analysis is performed using EPA Method TO-15 for the target VOC compounds with a focus on BTEX compounds and EPA Method TO-13A[3] for the target SVOCs, specifically the 16 regulated PAHs (polynuclear aromatic hydrocarbons) from naphthalene to Benzo(g,h,i)perylene.

Nathan Eklund, who leads the Pace Air Monitoring Sales and Marketing teams, says that while Pace has a vast amount of experience running traditional air quality tests using EPA Methods TO-13A and TO-15, these methods are relatively time consuming, and expensive. Pace was therefore looking for a single, cost effective, and safer method in order to respond to the demands of its clients and enhance laboratory efficiency.

To make this happen, Pace proposed a collaboration with PerkinElmer, as Eklund explained:

> *"We were familiar with PerkinElmer's thermal desorption equipment and we also felt that its recognized knowledge on sorbent selection would be beneficial to developing a robust alternative testing method built around EPA Method TO-17 for the collection of both BTEX and PAHs saving us time and money at each level in the process."*

Why the Change?

So why make this change, because EPA Methods TO-13A and TO-15 have been the industry-standard for over 15 years and will continue to be requested and used for many air-monitoring projects in the foreseeable future. However, it is important to emphasize that TO-13 is very labor intensive and expensive, which uses methylene chloride ($MeCl_2$) solvent to ensure cartridges are clean prior to sampling taking 16 hours, and glassware is clean prior to extraction. $MeCl_2$ is a toxic solvent so there is a risk for worker exposure. The extraction process uses 300 mL of solvent which takes 16 hours, and then the extract is concentrated to a 1 mL volume. During this concentration step, the environment is being contaminated with the solvent being evaporated. Also the holding times for analyzing the samples is only seven (7) days, compared to TO-17 analysis, which is approximately 28 days. In addition TO-13 requires surficial power supply to operate, creating a potential hazard to workers at the MGP sites where soil excavation is occurring.

EPA method TO-15 also has some limitations. The Summa canisters and necessary accessories are more expensive relative to sorbent tubes. Because of their larger size, it is more costly to ship and occupies a significant amount of laboratory space. The cleaning equipment for canisters also occupies a great deal of space and the process of cleaning canisters can often take many hours. Another limitation of TO-15 is that it cannot recover the PAH target analyte list which makes TO-15 impossible to achieve the goal of measuring both VOCs and SVOCs in a single method.

Benefits of Using a One Method Approach

There are many significant advantages to using a one method approach. Analyzing one sample instead of two will be more efficient for the analytical laboratory carrying out the work, and one sampling media, will be a more cost-effective solution, by saving Pace's clients time and money.

In addition, after the samples have arrived at the lab, they require no preparation. Once the sample tubes are placed on the thermal desorber autosampler, the instrument automates the process of desorbing the analytes from the tube, and injecting the sample into the analytical column for detection and analyzed by GC/MS.

In addition, it is well-recognized that the thermal desorption process is significantly more efficient than the Soxhlet extraction and also requires significantly less operator intervention.

From a performance standpoint, TO-17 can sample larger volumes; therefore the achievable detection limits are significantly lower with TO-17 than with TO-15. It can recover components as heavy as C44 while TO-15 cannot recover components heavier than C12. In addition, sorbent tubes are cleaned during the desorption cycle so the cleaning process time is insignificant.

From Collaboration to Innovative Solution

The partnership to work on Method TO-17 to replace Methods TO-13A and TO-15 began in October, 2012 when the Pace sales team initially contacted PerkinElmer's application scientist Lee Marotta about extending the list of analytes to include all 16 regulated PAHs and satisfy this market need for a one method solution. PerkinElmer's role in this collaborative agreement was to provide the supplies, the equipment, the design of a new sorbent tube with an analyte range from 1, 3-butadiene to Benzo(g,h,i) perylene, and expertise, while Pace's would provide the scientist and the location for the method development. Both companies worked together to come up with experiments to prove that the new sorbent tubes, the thermal desorption procedure, and the analytical conditions were suitable for the analysis of the target list of compounds.

The success of this collaboration summed up by Roberta Provost who led the Pace evaluation team:

> *"Vendor collaborations are a great way to streamline and reach the method development milestones at an accelerated rate, and Lee's support in particular has been integral to the success of our partnership with PerkinElmer. Just a few months ago, another vendor that provides us with passive sampling media was not able to deliver in time. Lee was able to save the day by pulling together a suitable sampling kit, which we could ship directly to our client."*

The scope of the collaboration was to design a tube and method to meet the real-world challenge of running a single analytical testing technique using Method TO-17 for both volatile and semi-volatile compounds. This was no small task, as there were unique problems in selecting a sorbent material to trap such a wide range of analytes. However, that challenge was overcome with the development of a new desorption tube designed to meet the demands of the greater boiling point component range found in both VOC and SVOC analytes. The tubes contain multiple layers of different charcoal-based sorbents arranged so that the sample is exposed to increasingly stronger sorbents as it penetrates the tube. This design prevents the absorption of the heavier components onto the stronger sorbents that would not release them. Instead, the heavier components are adsorbed onto the weaker sorbents in the front of the tube while the lighter components are adsorbed onto the stronger sorbents in the back end of the tube. The weaker sorbents protect the strong adsorbents from irreversible adsorption and leave the tube clean, typically after one desorption cycle. The new tube, shown in Figure 1, handles the broadest analyte range from 1, 3 butadiene to Benzo[g,h,i]perylene. The new tube's analyte range is from C4 to C40 which easily recovers this multi-ring PAH.

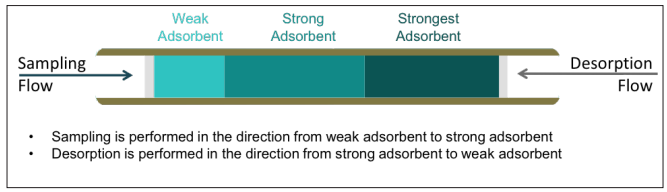

Figure 1. The new PerkinElmer desorption tube for trapping both VOC and SVOC analytes in air using multiple adsorbents (range from C4 to C40).

These new tubes are designed to fit onto the PerkinElmer TurboMatrix 650 ATD automatic thermal desorber. This instrument features a two-stage thermal desorption process that concentrates analytes before they are introduced into the PerkinElmer Clarus SQ8 GC-MS system[4]. TurboMatrix thermal desorbers provide high-temperature desorption capability that allow the determination of analytes up to C44 hydrocarbons. The automatic addition of internal standard calibration mixtures into the tube before sampling and analysis helps to ensure sample integrity and improves analytical quantification and accuracy. To minimize water vapor entering the system, which can quench target response in MS detection, the system offers optimized dry purging of both the tube and the trap, ensuring efficient water elimination even for high moisture air samples. The system is shown in Figure 2.

Figure 2. The automated thermal desorption system and Clarus GC/MS used in the collaboration.

Study data indicates that up to 50 liters of 95% relative humidity air can be sampled requiring only a two minute dry purge. However, further studies using 300 liters of air are planned. The full capabilities of the new methodology were not only evaluated using performance metrics, such as breakthrough volume, calibration linearity, reporting limits, precision, recovery, and carryover, but also by carrying out the analysis of real-world field samples[5]. The results are currently being developed for publication in a peer-reviewed journal in the Fall of 2014[6].

The Benefits of Collaboration

PerkinElmer's new desorption tube is now used on a routine basis and it is changing the way Pace and its customers conduct environmental testing. In fact, productivity and customer satisfaction at Pace Labs has significantly improved thanks to this single method approach. The benefits of the TO-17 thermal desorption tubes are that they use no solvent, rendering it a more environmentally friendly method. In addition the tubes are fitted with a small preset pump in the lab, so they can sample for up to several weeks with an extended battery pack. Their small size also allows for low-cost shipping, and there is no preparation required at the lab prior to analysis, which provides complete adsorption and recovery of all the target analytes.

"We would describe this collaboration as one of the most important relationships that the Air Lab at Pace Analytical Services has ever developed," says Amy Jacobson, Laboratory Manager of the Pace Air Monitoring Lab. *"It has been mutually beneficial and continues to open doors for both companies based on insight into each partner's side of the business. There is no question that the collaboration has not only produced a cost-effective, green solution to the problem, but has also allowed us to develop new markets, helped us significantly improve internal business practices, and increased our expectations of other vendors."*

References

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2. U.S. Environmental Protection Agency, Compendium Method TO-17, Determination of Volatile Organic Compounds (VOCs) in Ambient Air Using Active Sampling on to Sorbent Tubes. Office of Research and Development, Cincinnati, OH. January, 1999.
3. U.S. Environmental Protection Agency, Compendium Method TO-13A, Determination of Polycyclic Aromatic Hydrocarbons (PAHs) in Ambient Air Using Gas Chromatography/Mass Spectrometry (GC/MS). Office of Research and Development, Cincinnati, OH. March 18, 1999.
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