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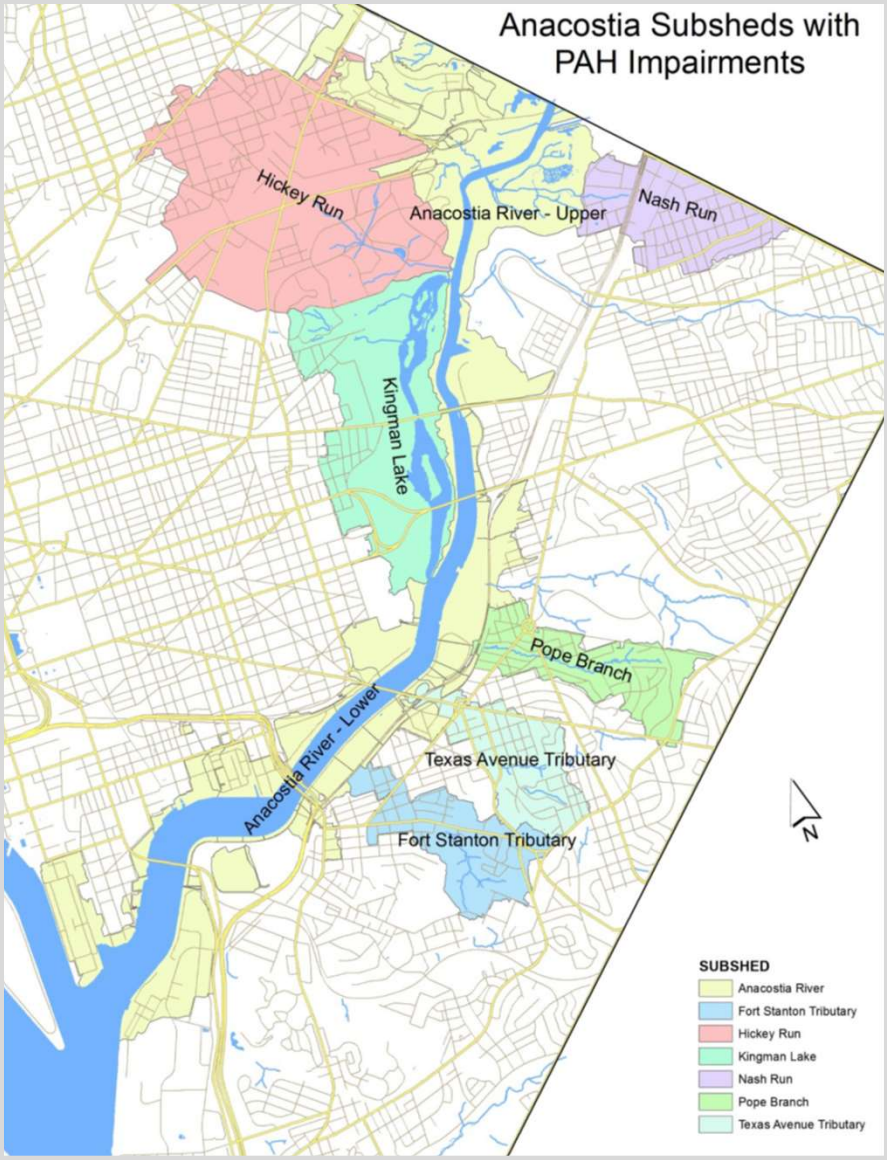
**Final Project Report: Protocol Development to Certify Low-PAH Sealants**

**Background:**

In 2008, Washington, D.C. (the District) joined a number of jurisdictions across the country in banning pavement sealants containing coal tar, a byproduct of the production of coke and coal gas, to protect District residents and waterways from a source of toxic contamination. Research has identified coal tar pavement sealant products as a major source of Polycyclic Aromatic Hydrocarbons (PAHs), a class of toxic contaminants found in coal, crude oil, and gasoline. PAHs are threats to both human health and aquatic wildlife; the EPA has classified 7 PAHs as probable human carcinogens and 16 PAHs as Priority Pollutants. PAHs are a common toxic pollutant in urban waterways, including several tributaries of the Chesapeake Bay. The Anacostia River and several of its tributaries are listed as PAH-impaired as well as Baltimore Harbor.

***FIGURE 1***

**The Anacostia River flows through northeast and southeast DC is PAH-impaired as well as several of its tributaries**



Pavement sealants are products applied to asphalt-based driveways and parking lots as a protective barrier to enhance the aesthetics of the space and to prolong the life of the asphalt. As the sealant becomes worn through exposure to the elements and vehicles, PAHs enter the environment through volatilization and as dust transported by tires, wind, and rain.

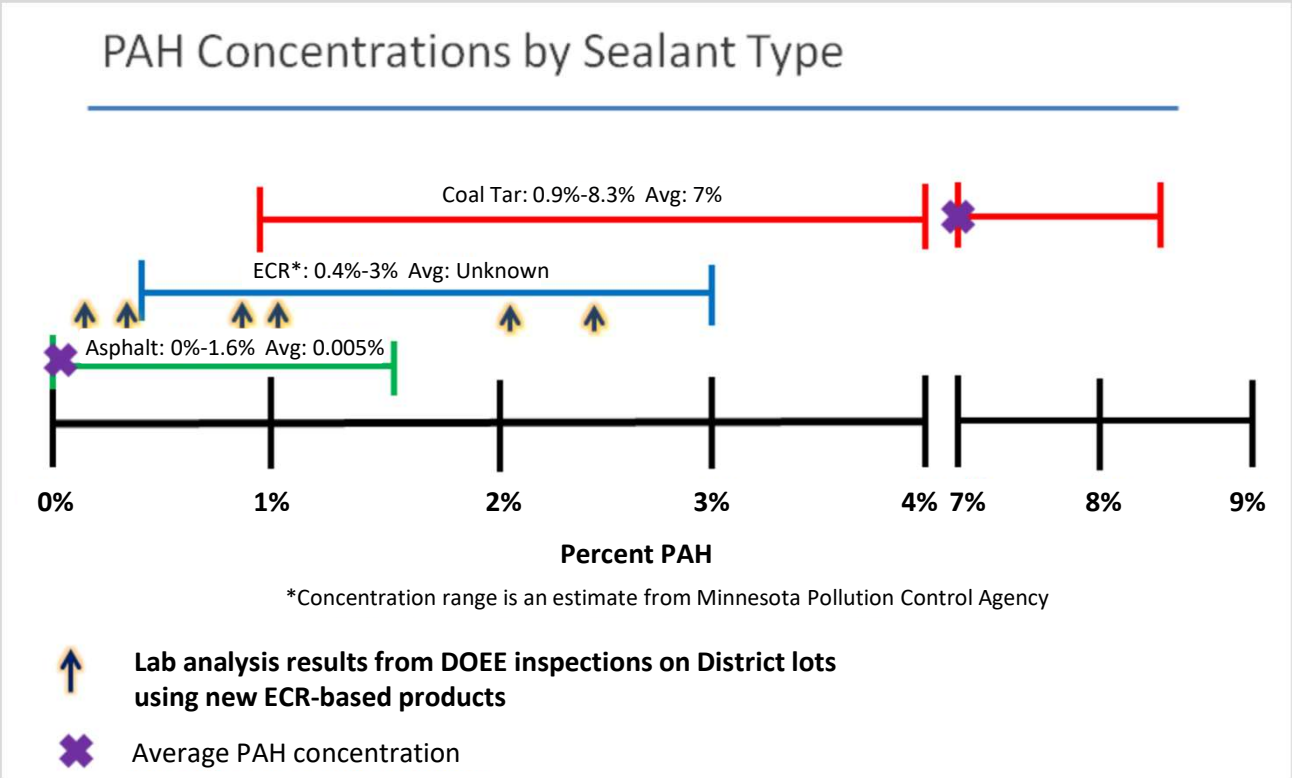
At the time of the coal tar ban's passing, the only available alternative sealant type to coal tar was asphalt-based, which has significantly lower concentrations of PAHs. Coal tar sealants average 7% PAHs, ranging from 0.9%-8.3%, while the average PAH concentration of asphalt-based sealant is closer to 0.005% (Figure 2). The District's Department of Energy & Environment (DOEE) is responsible for enforcing the District's pavement sealant ban and routinely inspects properties for compliance with the law. DOEE has not encountered a coal tar product during routine inspections since 2014.

In 2017, DOEE inspectors identified a new class of pavement sealant products being used on several District properties containing Ethylene Cracker Residue (ECR), also known as steam-cracked asphalt, a byproduct of petroleum distillation. Sealant products containing ECR are relatively new to the market and largely unstudied, but through laboratory analysis and industry research DOEE confirmed that these products contain PAH concentrations similar to coal tar.

Industry experts estimate the PAH concentration of ECR-based products range between 4,000 parts per million (ppm) to 30,000 ppm (0.4%-3%) PAHs, which is supported by analysis of parking lots inspected by DOEE in the District. DOEE completed lab analyses on five of the seven lots confirmed to have used these new products in the District and found PAH concentrations ranging from 0.13% to 3% (Figure 2).

***FIGURE 2***

**Average PAH concentrations and ranges for asphalt, coal tar, and ECR-based sealant products**




DOEE was a part of a growing group of stakeholders across the country noticing the emerging threats in ECR products. With assistance from the Huron River Watershed Council, 15 jurisdictions in southern Michigan became the first in the

country to pass a ban on any sealant product containing PAH concentrations higher than 0.1% by weight. When data on the use and threats of ECR products in the District was shared with DC Council, lawmakers followed suit in expanding the ban on coal tar pavement sealants to include any product containing ECR or with a PAH concentration greater than 0.1% by weight. The new law became effective in March, 2019. The limit of 0.1% is not so restrictive to prevent the continued use of asphalt-based sealants, which studies have shown have little to no measurable impact on aquatic wildlife (Mahler et al., 2015, Titaley et al., 2016), but prevent harmful products from being used on properties within the District. The District was the first jurisdiction of its size in the country to pass a PAH-limit law and has since been joined by other cities such as Austin, Texas and Charlotte, North Carolina.

As the regulatory agency responsible for implementing the District's pavement sealant ban, DOEE's first step once the amendments were passed was to research how to accurately and consistently identify PAH concentrations in pavement sealants, and quickly found that a protocol does not exist. Pavement sealant manufacturers do not make their product PAH concentrations public.

While a standard EPA method exists for testing for PAHs in substances like waste samples and liquids similar to sealants (EPA Method 8270D), the structure and chemistry of PAHs are known to be highly volatile/unstable, which means the methodology for sample preparation could significantly impact the results of the analysis. DOEE and other regulators responsible for enforcing PAH-limit laws need a standardized protocol for testing PAHs specific to pavement sealants in order to enforce the requirements and educate the regulated community about what can and cannot be used. Ideally, one standardized protocol would be used by all jurisdictions with PAH-limit laws to reduce confusion among the regulated community and to promote collaboration and support among regulators.

**Project Summary:**

DOEE sought funding from the GIT Toxic Contaminants Workgroup in 2018 to fund a contractor to create a standardized protocol for testing PAH concentrations in pavement sealants and to use the approved protocol to create a "Gold" (products with 1,000 ppm PAHs or below) and "Silver" (10,000 ppm PAHs or below) list of pavement sealants. The project would be co-chaired by DOEE and the EPA's Chesapeake Bay Program (CBP) and overseen by a steering committee including regulators, subject matter experts, and stakeholders from across the country, with all deliverables to be made publicly available by DOEE.

Administered by the Chesapeake Bay Trust, the 2018 Request for Proposals for Goal Implementation Team (GIT) Funded Projects included the "Protocol Development to Certify Low-PAH Sealants" project as Scope #7 for a maximum bid of $85,000. Sitelab Corporation was awarded the contract in June 2019. Since 1998, Sitelab manufacturers portable hydrocarbon instruments and provides consulting services to the environmental remediation and oil and gas industries with expertise in laboratory analytical methods and hydrocarbon forensic analysis.

This report summarizes work performed for the project's goals. This report follows the objectives outlined in CB Trust's GIT 2018 "Table 2: Defining Project Scope of Work" document.

**Task 1 Summary**

The goal for this task was to develop a robust, scientifically sound, Standard Operating Procedure (SOP) or "Quality Assurance Protocol" for testing and certifying the PAH content in pavement sealants. This protocol was designed to be a national standard for use around the country where communities want to ban and regulate the use of high PAH products.

**Task 1.1** The goal for this task was to form a Review Committee The purpose of the Review Committee was to help guide SOP design and guarantee quality control. Scientists, regulators and nonprofit stakeholders were invited to the committee in order to provide feedback on progress of the project and assist in making key decisions. The ultimate goal

was to ensure the protocol created under the project reflected the needs of regulators on a national scale. The committee was formed by Sitelab with assistance from DOEE. Members are listed below:

| Name: | Affiliation: |
|---|---|
| Steve Greason | Sitelab Corporation |
| Nancy Rothman | New Environmental Horizons, Inc. |
| Lillian Power | DOEE, Dept. of Energy & Environment (Project Lead) |
| John Materi | DOEE, Dept. of Energy & Environment |
| Greg Allen | U.S. Environmental Protection Agency |
| Rebecca Esselman | U.S. Environmental Protection Agency |
| Tom Ennis | City of Austin, TX, Watershed Protection Dept. |
| Hilary Swartwood | Huron River Watershed Council |
| Leonard Schugam | Maryland Dept. of the Environment |
| Stan Edwards | Montgomery County, MD |
| Barbara Mahler | USGS Austin, TX (volunteer, limited attendance) |

Review Committee meetings were conducted fairly regularly until delays caused by the Covid-19 pandemic. Meeting agendas and conference calls were scheduled by DOEE and meeting minutes were recorded by Sitelab Corporation. The last meeting with the Review Committee was in September 2020. Since then, meetings were conducted with DOEE only, with comments and feedback received by Review Committee members when emailing the protocol drafts and test data.

Attempts were made to add members from sealcoat manufacturers and industry trade organizations, but few trade organizations exist and it was decided to work independently with manufacturers instead to avoid any conflicts or show favoritism. Sitelab was successful establishing relationships with multiple manufacturers, who offered samples of their products to help with this project. Much of this effort contributed to the outreach plan conducted in Tasks 3 and Task 4.

**Task 1.2** The goal of this task was to develop testing protocols and procedures for a pavement sealant certification program. Sitelab developed a draft laboratory "QA Protocol" with Nancy Rothman from New Environmental Horizons, Inc., Sitelab's consultant, who's an experienced expert in lab analytics and protocol development. The protocol was designed to meet and qualify sealcoat products having concentrations below DOEE's 1,000 ppm (Gold) and 10,000 ppm (Silver) certification levels. These limits match the PAH limits regulated throughout the country. See Figure 3.

***FIGURE 3***

**17 PAH Compounds**

1. Naphthalene
2. 2-Methylnaphthalene
3. Acenaphthylene
4. Acenaphthene
5. Fluorene
6. Phenanthrene
7. Anthracene
8. Fluoranthene
9. Pyrene
10. Benzo(a)anthracene
11. Chrysene
12. Benzo(b)fluoranthene
13. Benzo(k)fluoranthene
14. Benzo(a)pyrene
15. Indeno(1,2,3-cd)pyrene
16. Dibenz(a,h)anthracene
17. Benzo(g,h,i)perylene

- Total PAH is calculated by adding the 17 compounds.
- Test Method 8270D includes the EPA's priority pollutant compounds, which are regulated and have specific toxicity limits to human health and the environment.
- The PAH content in sealants varies, depending on what type of hydrocarbons they're made with.
- The protocol was designed to qualify products having Total PAH concentrations below DOEE's 1,000 ppm (Gold) and 10,000 ppm (Silver) limits.

**Current Bans in U.S. With and Without PAH Limits**

**Coal Tar Bans with 1,000 ppm PAH Limit**

States: 0

Federal Districts
Washington, D.C.

Counties & Cities
Wisconsin: 15
Michigan: 12
North Carolina: 2
Maryland: 1
Texas: 1

Austin, TX, is the largest municipal ban with a PAH limit. See below:

https://coaltarfreeusa.com/bans-2/

**Coal Tar Bans with 10,000 ppm PAH Limit**

States: 2
New York
Maine

Counties & Cities
Wisconsin: 1

**Coal Tar Bans without a PAH Limit**

States: 2
Minnesota
Washington

Counties & Cities
Illinois: 14
Pennsylvania: 9
Michigan: 3
Texas: 3
Maryland: 3
Wisconsin: 2
Kansas: 1
Maine: 1
Massachusetts: 1
South Carolina: 1
New York: 1

Prior to developing the protocol, a year was spent acquiring different products made with asphalt, ethylene cracked residue (ECR) and coal tar analyzing both “wet” and “dry” samples. PAHs are highly volatile, which means the PAH concentrations detected via lab analysis in a sealant sample can vary significantly depending on whether the sample comes directly from the container (“wet” sample) or after it’s been applied to a surface and allowed to dry (“dry” sample). The dry sample could have lower PAH concentrations than the wet sample as the more volatile PAHs enter the air, with the PAH concentrations continuing to drop the longer you allow the sample to dry. With approval from the steering committee, Sitelab prepared both wet and dry samples for analysis to determine whether results varied significantly based on preparation method.

See Figure 4 for examples. Samples were donated by manufacturers or purchased from retail stores and analyzed for PAHs by Pace Contest and Alpha Analytical Laboratories to determine how much or how little PAHs exist and see how the two laboratories compared to each other. The water content in sealcoats vary, most products contain about 50% water plus coal tar, asphalt or other ingredients. Wet sample results are corrected for moisture content and reported on a dry weight basis in mg/Kg. Sitelab developed SOPs to dry sealcoats indoors to try and mimic field conditions, where ‘scrapings’ from parking lots are typically collected, and to see how much or how little PAHs are lost during the drying process.

Concentrations exhibited by both laboratories were similar in both dry and wet samples. Pretrial results confirmed that using Method 8270D, regardless of dry vs. wet testing, accurately reflected zero PAH concentrations in the asphalt-based products and very high PAH concentrations in the ECR and coal tar products.

***FIGURE 4***

**Samples Acquired from Retail Stores & Manufacturers**

Asphalt and coal tar sealcoats were purchased in 5-gallon buckets at retail stores or online. Buckets were mixed using a paint mixer and samples were collected into glass vials for the laboratory.

ECR and coal tar sealcoat samples were also provided by several manufacturers.

**Procedure for Preparing Dried Samples**

Wet samples were dried indoors using aluminum weigh dishes for 2 days at 70°F using a hygrometer to monitor humidity and temperature. Sample pieces were then crushed or cut into fragments and transferred into glass vials for the laboratory.

**Task 1.3** The goal of this task was to confirm the ability to test sealant samples according to EPA Method 8270C to determine PAH concentration. Sitelab Corporation demonstrated the ability to accurately measure the concentrations of PAHs using certified laboratories performing EPA Method 8270D or 8270E (updated versions to C). This method detects and reports 17 PAH compounds. See Figure 3 for details.

Samples in Task 1 were measured by the two laboratories for PAHs by EPA Method 8270D, plus EPA Method 8270D-SIM and Mass DEP’s EPH Method. These three methods were selected to determine the best test method for the program and what their limitations are, including accuracy with cost. The 8270D-SIM test detects up to 80 PAH compounds. This includes the 17 target PAHs plus other alkylated or ‘cousin’ compounds. This test was chosen to determine how many other PAHs exist in the sealcoats. This is an expensive test and not many laboratories can perform it. The EPH test detects the 17 compounds and detects the aromatic and aliphatic hydrocarbon fractions. This test was chosen to better understand the full hydrocarbon content in the sealcoats.

Samples used in Task 1 included two different brands each of asphalt, ECR and coal-tar based products, representing the 3 major types of sealcoats applied to driveways and commercial parking lots. The six products were tested to check the protocol and to gather more information on the products themselves to confirm our assumptions (ECR and coal tar are high-PAH products and asphalt are low). In addition, in order to see if these products vary during the manufacturing process or if manufacturers use different ingredients to make their products at different locations, samples were collected and tested in two batches. These were acquired by purchasing bucket brands (Latexite, Blackjack and Sakrete) stamped with dates made over 4 weeks apart and by samples collected by manufacturers made at two factories (Black Diamond and FedSpec) or at the same factory made 3 weeks later (Neyra Force). Batches are shown as "B1" and "B2" in Figure 5 and were tested in wet and dry form.

***FIGURE 5***

**Performance Data Reported in Sitelab Task 1 Summary Report. Total 17 PAHs by 8270D in ppm (mg/Kg)**

| | CONTEST LAB 8270D + EPA 3546 **WET B1** | CONTEST LAB 8270D + EPA 3546 **WET B2** | AVG. CONC. | STD DEV | ALPHA MANSFIELD 8270D-SIM + EPA 3540C **WET B1** | ALPHA WESTBORO 8270D + EPA 3580A **WET B2** | AVG. CONC. | STD DEV | COMBINED LABORATORY RESULTS **WET SAMPLE BATCHES** AVG. CONC. | STD DEV |
|---|---|---|---|---|---|---|---|---|---|---|
| LATEXITE | ND | ND | 0 | 0 | 22 | ND | 0 | 0 | 0 | 0 |
| BLACKJACK | ND | ND | 0 | 0 | 9 | ND | 0 | 0 | 0 | 0 |
| NEYRA FORCE | 18,825 | 21,917 | 20,371 | 1,546 | 35,742 | 19,711 | 27,727 | 8,016 | 24,049 | 7,903 |
| BLACK DIAMOND | 28,300 | 20,699 | 24,500 | 3,801 | 36,575 | 38,051 | 37,313 | 738 | 30,906 | 8,045 |
| FEDSPEC | 64,620 | 57,920 | 61,270 | 3,350 | 74,541 | 67,002 | 70,771 | 3,769 | 66,021 | 6,859 |
| SAKRETE | 102,640 | 127,190 | 114,915 | 12,275 | 178,447 | 162,658 | 170,552 | 7,895 | 142,734 | 34,261 |

| | CONTEST LAB 8270D + EPA 3546 **DRY B1** | CONTEST LAB 8270D + EPA 3546 **DRY B2** | AVG. CONC. | STD DEV | ALPHA MANSFIELD 8270D-SIM + EPA 3540C **DRY B1** | ALPHA WESTBORO 8270D + EPA 3580A **DRY B2** | AVG. CONC. | STD DEV | COMBINED LABORATORY RESULTS **DRY SAMPLE BATCHES** AVG. CONC. | STD DEV |
|---|---|---|---|---|---|---|---|---|---|---|
| LATEXITE | ND | ND | 0 | 0 | 4 | ND | 0 | 0 | 0 | 0 |
| BLACKJACK | ND | ND | 0 | 0 | 5 | ND | 0 | 0 | 0 | 0 |
| NEYRA FORCE | 17,989 | 15,280 | 16,635 | 1,355 | 37,848 | 31,223 | 34,535 | 3,312 | 25,585 | 10,740 |
| BLACK DIAMOND | 20,158 | 18,333 | 19,246 | 913 | 46,031 | 25,589 | 35,810 | 10,221 | 27,528 | 12,715 |
| FEDSPEC | 62,410 | 83,710 | 73,060 | 10,650 | 89,131 | 75,863 | 82,497 | 6,634 | 77,779 | 11,603 |
| SAKRETE | 108,139 | 107,280 | 107,709 | 429 | 155,890 | 109,043 | 132,467 | 23,424 | 120,088 | 23,879 |

As expected, ECR and coal tar products contained very high concentrations and are well above the 10,000 ppm limit, regardless of test method, batch, or sample preparation method. Asphalt-based products contained no or very low PAHs. In fact, the 80 compound test performed by 8270D-SIM, detected very few other PAH compounds in the asphalt sealants; PAH concentrations were well below the 1,000 PAH ppm limit.

The Task 1 round of testing was very successful. Using the two laboratories proved useful to account for any differences from one lab to the other. It was decided to only test wet samples for the QC Protocol and to use EPA Method 8270D or 8270E only, as originally specified, to analyze the PAH content in sealcoat products. This method testing wet sealcoats did not yield significantly different results from dry preparation and exhibited the most consistent results. The EPH method proved useful in determining the aromatic and aliphatic hydrocarbon content, but produces low PAH concentrations and is not suitable for this application. The 8270D-SIM test was helpful in knowing the full PAH content in the sealants and exhibited lower detection limits, but was costly to perform and therefore determined not to be an economically viable option for manufacturers who will be responsible for paying for testing. This round of testing verified that 8270D was sensitive and accurate enough to distinguish between high and low PAH products while also being considerably more economical than the more sensitive 8270E-SIM method.

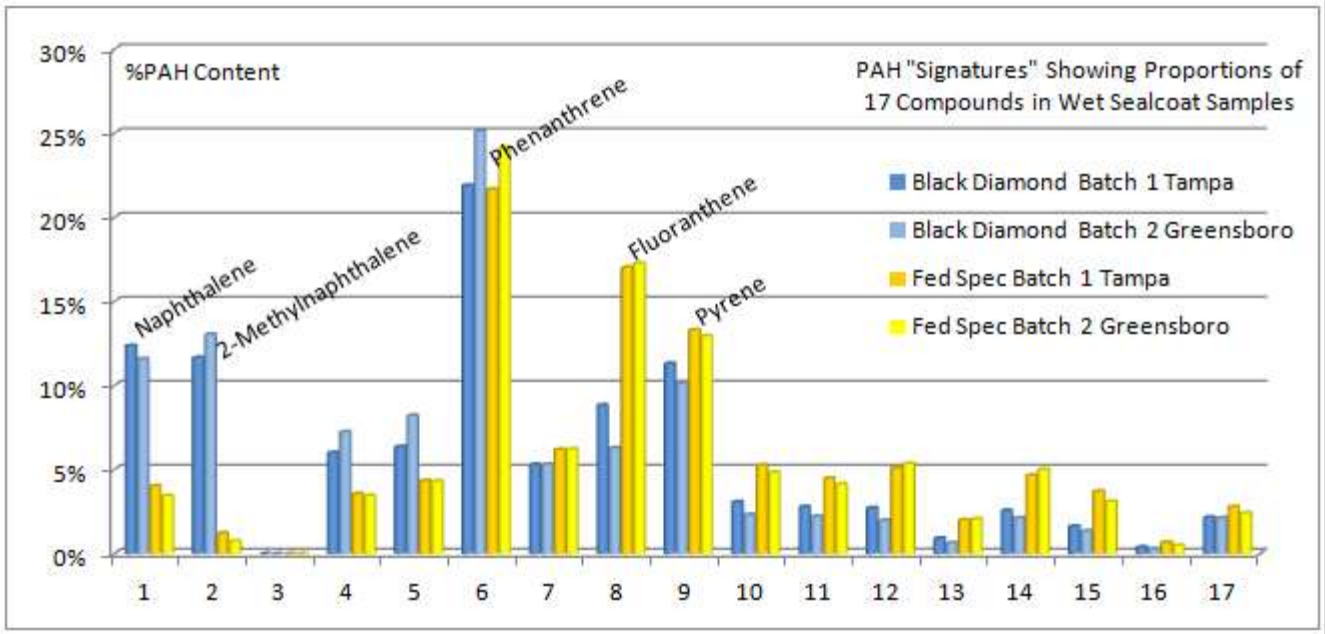
To ensure accuracy, the QA Protocol was revised to include a number of QA/QC measurements to qualify products for the certification program. Laboratories used must be "NELAP certified." NELAP stands for National Environmental Laboratory Accreditation Program and is a national accreditation program developed by The NELAC Institute. This program is similar to ISO standards and fosters the generation of data of known and documented quality.

Laboratories must perform a Method Blank (solvent), two Laboratory Control Samples (solvent plus PAH spike) and two Matrix Spikes (sealcoat plus PAH spike) with each sealcoat sample. Surrogate compounds are also added to these samples and the surrogate recoveries and spike recoveries in the Blank, LCS, LCSD, MS and MSD must also be reported and fall within the lab's acceptance limits for each. The laboratories must report any flags or outliers exhibited in the data. An Appendix was added to the QA Protocol. This included an Example Data Package and Data Review Process. These were developed to help manufacturers and regulators interpret the lab reports.

Sitelab provided reports summarizing the laboratory results for each product tested. These reports included photos of samples, PAH screening analysis using Sitelab's UVF-Trilogy analyzer and in some cases, PAH forensic graphs comparing the hydrocarbon ingredients found in different types of sealcoat products. See Figure 6 for example.

***FIGURE 6***

**PAH Forensic Graph Comparing ECR and Coal Tar Products with Most Abundant Compounds Highlighted**




Gemseal's Black Diamond (ECR) and Fed Spec (Coal Tar) were collected from two different plants. PAH concentrations in each compound were similar, including the percent PAH content graphed here. ECR-based sealcoats contain more lighter compounds, while coal tar-based sealcoats contain more heavier compounds. Both types are most abundant with Phenanthrene, which makes up 20-25% of the PAH content. These signatures were similar to other ECR and Coal Tar products tested.

**Task 1.4** The goal of this task was to develop a Quality Assurance Project Plan (QAPP). A draft QAPP was completed by Sitelab's consultant, Nancy Rothman from New Environmental Horizons, Inc.

**Task 2 Summary:**

The goal of this task was to validate the procedures and quality assurance/quality control (QC/QC) mechanisms in the protocol to ensure sealcoat products are measured accurately and the data is defensible.

**Task 2.1** The goal of this task was to create SOPs for labs responsible for testing products for certification. The improved QA Protocol developed by Sitelab and New Environmental Horizons, Inc., with input from DOEE and the Review Committee was used for the Task 2 round of testing.

**Task 2.2** The goal of this task was to acquire a range of available pavement sealant products for testing SOPs. Sealcoats having high, medium and low concentrations of PAHs were split and analyzed by four NELAP certified laboratories from around the country. This was to ensure the protocol yielded accurate, consistent results. In addition, the labs used four different EPA extraction methods to prepare the samples for analysis. Method 8270D is an analytical method and allows for different extraction methods be used. Sealcoats are extracted in methylene chloride solvent and then extracted using different technologies; soxhlet, ultrasonic and microwave oven are commonly used methods and were chosen to see how much variation occurs in the test results. EPA Method 3580A by waste dilution is also popular and was used by Alpha in Task 1, but this extraction method produces high detection limits and is not suitable for the certification program.

One of the samples chosen for Task 2 included an asphalt-based sealcoat cross-contaminated with coal tar. This was first discovered using Sitelab's PAH screening test. This was also the case with several other samples received from manufacturers who make all three product types at their facilities. Manufacturers were advised of this problem and new products were collected and submitted for analysis with no PAHs detected. It was fortunate, however, these samples were available since the PAH concentrations were between the Gold and Silver PAH limits. To date, no products have been found having PAHs between these two ranges. Sealcoats either have no PAHs or contain PAHs above the Silver limit.

Figure 7 highlights some of the data in Task 2. No considerable variations were found across the labs, confirming the SOP as written is robust and user-friendly by NELAP labs. The four extraction methods used produced similar results. The wet vs. dry results were also similar as before in Task 1. The laboratories reported sufficient detection limits and the QC data met the protocol's limits, performing best testing in the Driveshield asphalt-based product. QC performance testing samples with high PAHs exhibited lower surrogate and spike recoveries due to matrix interference in the sealcoat. These outliers, however, were identified in the laboratory reports.

***FIGURE 7***

| 4 Labs Used for EPA 8270D PAHs | 4 EPA Extraction Methods Used | Driveshield-Wet ppm (mg/Kg) | Neyra Force-Wet ppm (mg/Kg) | Asphalt*-Dry ppm (mg/Kg) | Asphalt*-Wet ppm (mg/Kg) | Wet Duplicate ppm (mg/Kg) → | % RPD |
|---|---|---|---|---|---|---|---|
| Pace Contest | 3546 Microwave | ND <42 | 27,872 | 2,049 | 2,837 | 2,560 | 10.3% |
| EMSL | 3546 Microwave | 9 ppm | 13,354 | 1,732 | 2,778 | 2,789 | 0.4% |
| Pace Contest | 3540C Soxhlet | ND <26 | 19,303 | 1,750 | 2,062 | 2,356 | 13.3% |
| GEL | 3541 Auto Soxhlet | ND <9.6 | 14,339 | 2,312 | 1,866 | 1,545 | 18.8% |
| Analab | 3550C Ultrasonic | ND <2.6 | 28,701 | 2,318 | 2,105 | 1,586 | 28.1% |
| EMSL | 3550C Ultrasonic | 10 ppm | 20,320 | 1,453 | 2,676 | 2,516 | 6.2% |
| **Combined Laboratory Results, Good Correlation Exhibited** | | | | | | | |
| Average Total PAH Concentration: | | ND <10 ppm | 20,648 | 1,936 | 2,387 | 2,225 | |
| Standard Deviation: | | 0 | 6,511 | 349 | 423 | 530 | |

*This brand of asphalt sealcoat contained residual coal tar due to sample collection error by Manufacturer. The same tank was used to prepare both products at plant.

**Task 2.3** The goal of this task was to run test samples through protocols for quality control, ensuring samples are handled consistently and correctly.

Sealcoat products were run through the QA Protocol. Each lab in Task 2 was given the protocol and reported the Blank, LCS, LCSD, MS, MSD and other information as required. In this case, the 4 laboratories detected similar concentrations in the samples, regardless of which extraction method was used and the quality control tests passed the appropriate limits.

This confirms different labs and the extraction methods they use to analyze samples for PAHs by 8270D is consistent and accurate.

The only change made to the protocol after Task 2 was by DOEE, replacing the CAM-IIB acceptance limits with tighter acceptance limits. Acceptance limits were tightened with guidance from the EPA's Chesapeake Bay Program and the Region III Quality Control (QC) Officer to ensure only high quality data is used to accept or reject products for the Low-PAH Product Lists. DOEE will be reviewing the protocol periodically and can update components like recovery as needed. Should labs consistently fail to meet the stricter limits, DOEE is able to loosen limits as more data reflects need. See Figure 8 for details.

***FIGURE 8***

**Quality Assurance Protocol: PAH Measurement Performance Criteria**

| Data Quality Indicators | Measurement Performance Criteria | CAM-IIB Limits | DOEE Limits in Protocol |
|---|---|---|---|
| Sensitivity | Method Blank: | <QL (29.4 ppm each compound) | |
| Accuracy/Precision | LCS/LCSD Recoveries:<br>LCS/LCSD RPD | 40%-140%<br>RPD ≤30% | 60%-140%<br>RPD ≤30% |
| Accuracy | Surrogate Recoveries: | 30%-130% | 70%-130% |
| Accuracy/Precision | MS/MSD Recoveries:<br>MS/MSD RPD | 40%-140%<br>RPD ≤30% | 60%-140%<br>RPD ≤30% |
| Precision/Representativeness | Lab Sample Duplicate: | RPD ≤50% | RPD ≤50% |
| Completeness | 100% sample collection<br>100% laboratory analysis | Data Completeness Check | |

- Labs must perform and report QA/QC analysis with each product tested. This includes a Blank, LCS, LCSD, MS, MSD and Duplicate to ensure results are accurate. Labs normally use the Mass DEP's CAM-IIB limits to qualify data.
- Surrogate and spike recoveries in sealcoat products can often be low due to matrix interference.
- DOEE uses tighter limits, but specifies: "Data packages with qualifiers falling outside but close to stated ranges (at least above 50% recovery) may still be submitted without reanalysis for consideration by DOEE if all other QC data is found within acceptance ranges."

**Task 2.4** The goal of this task was to finalize the QAPP and have it approved by EPA prior to the start of product certification. The final QAPP was completed and approved by Durga Ghosh, the U.S. EPA's QC Officer assigned to this project.

Changes made to the original draft QAPP included the addition of specific reporting limits for each compound and other improvements to the Method 8270D performance criteria tables and the removal of the Method 8270D-SIM and EPH Method tables and SOPs since it was decided to not use these methods in the Task 2 round of testing.

**Task 3 Summary:**

The goal of this task was to develop an outreach plan for recruiting manufacturers to test and certify the PAH content in their products. Much of this effort began early on in the project.

**Task 3.1** The goal of this task was to notify sealant manufacturers of the new certification program and recruit products for testing. By searching the internet, Sitelab created a large database of different manufacturers and brands of products available around the country. Manufacturers were contacted by phone or email encouraging them to participate. Sitelab created a chain-of-custody form for manufacturers with specific instructions to collect and submit samples for analysis.

Currently, there are over 100 different products available in this industry. In general, throughout most of the United States, sealcoat products used on commercial properties, like shopping centers and office parking lots, are coal tar-based and are applied by sealcoating service companies. Most products used on driveways by homeowners are asphalt-based and are sold in buckets at retail stores.

As a result of the coal tar bans around the country, manufacturers have introduced new "alternative coal tar" products made with ethylene cracked residues (ECR or steam cracked residues). The source of hydrocarbons used in each product is identified by the CAS number listed in the composition section in each product's Material Safety Data Sheet (MSDS), available on the internet. Sitelab has been updating this database on a frequent basis and has all the MSDS on file. Figure 9 summarizes all the products available.

***FIGURE 9***

**Types of Parking Lot Sealcoats, Total PAH Concentration Ranges and Products Available in U.S. Market**

| ❶ Asphalt-Based Sealcoats<br>Hydrocarbons CAS# 8052-42-4 | ❷ ECR-Based, "Cracked Residue" Sealcoats<br>Hydrocarbons CAS# 64742-90-1 | ❸ Coal Tar-Based Sealcoats<br>Hydrocarbons CAS# 65996-93-2 |
|---|---|---|
| ● Total PAHs = 0 ppm (ND <50) | ● Total PAHs = 20,000 to 30,000 ppm | ● Total PAHs = 50,000 to 180,000 ppm |
| ● Number of products in market = 86 | ● Number of products in market = 9 | ● Number of products in market = 29 |
| ● Qualify for DOEE's Gold <1,000 ppm or Silver <10,000 ppm certification | ● Do not qualify, PAHs > 10,000 ppm | ● Do not qualify, PAHs > 10,000 ppm |

❹ Other: Latex-Based Sealcoats, CAS# 25085-34-1 or CAS# 25067-01-0. Products are made with soybean oil or acrylic latex (like paint). Number of products in market = 6. These products have not yet been tested but are expected to have no or little PAHs.

Based on the test results performed in Tasks 1 and 2, it was decided to recruit asphalt-based products for the program since these products contain no/little PAHs and pass DOEE's 1,000 ppm Gold certification level. A total of 86 products have been identified. A small 4$^{th}$ group of latex-based sealcoats also exist. Sitelab has acquired several of these products and UVF screening analysis performed detected no/low PAHs. ECR and coal tar products contain PAH concentrations greater than 10,000 ppm and would not qualify for DOEE's Gold or Silver certification levels.

Interest and feedback received from manufacturers was both positive and negative. Some of the bigger companies who make coal tar, ECR and asphalt-based products, like Neyra Industries and Gemseal, were very willing to submit their asphalt-based sealcoat products for analysis. They had tested their products for PAHs already and understood the importance of this program. However, most others contacted have been reluctant to participate, despite the fact that testing was offered for free in the exploratory testing phase of this contract. This reluctance is mainly due to the new regulations and bans impacting the industry, which has been using coal tar since the mid 20th century.

Some manufacturers too are hesitant to submit samples for fear their formulas will be shared to the public. This is a very competitive industry and many manufacturers are not familiar with PAH testing and that laboratory data is strictly confidential. In response to concerns expressed by industry, DOEE has drafted a User's Guide as a resource, which explains that DOEE will not test for anything other than PAH concentrations using the approved testing protocol.

It's expected these concerns will alleviate over time as more companies participate in this program and as more bans go into effect in the future. Sealmaster and STAR, for example, recently submitted samples in order to be used in the City of Austin, Texas, starting in 2022. These manufacturers were contacted several years prior but were not willing to send samples. New York, the first State to ban coal tar and high PAH products, will also have a big impact when enforcement begins next year. Many manufacturers contacted are members of PCTC, a pro-coal tar industry trade organization, which to some extent, has hindered the recruiting efforts. Visit PCTC's website: https://www.pavementcouncil.org

The "Parking Lot Sealcoat List" in Figure 10 identifies all the products available and which brands have been tested.

***FIGURE 10***

## PARKING LOT SEALCOAT LIST: JUNE 27, 2022


EACH PRODUCT LISTED HAS MATERIAL SAFETY DATA SHEETS (MSDS) AVAILABLE.
CAS NUMBER IN MSDS SECTION "COMPOSITION" IDENTIFIES SOURCE OF HYDROCARBONS.

**ASPHALT-BASED SEALCOATS**
SOURCE OF HYDROCARBONS: CAS# 8052-42-4

| MANUFACTURER | BRAND NAME | TESTED | COMMENTS |
|---|---|---|---|
| ASPHALT COATINGS ENGINEERING | ACE SEAL | | |
| ASPHALT COATINGS ENGINEERING | ACE SEAL HD | | |
| ASPHALT SYSTEMS, INC. | GSB-88 GILSONITE | | CONTAINS UP TO 12% DIESEL FUEL |
| ASPHALT SEALCOATING DIRECT | BIG A | | |
| B&E SEALCOATS | SAFE SEAL | | NO MSDS AVAILABLE |
| CASCADE | ARMOSEAL A100 | | |
| CASCADE | ARMOSEAL A100-HD | | |
| DALTON ENTERPRISES | LATEXITE AIRPORT GRADE | YES | <1,000 PPM, MEETS DOEE "GOLD" LIMIT |
| DALTON ENTERPRISES | LATEXITE COLOR GRADE | | |
| DALTON ENTERPRISES | LATEXITE OPTIMUM | | |
| DALTON ENTERPRISES | LATEXITE PREMIUM | | |
| DALTON ENTERPRISES | LATEXITE SAND MIX SEALER | | |
| DALTON ENTERPRISES | LATEXITE SEALRIGHT | | |
| DALTON ENTERPRISES | LATEXITE THERMASEAL | | |
| DALTON ENTERPRISES | LATEXITE ULTRASHIELD | | |
| DALTON ENTERPRISES | LATEXITE ACRYLIC GRADE | | |
| DALTON ENTERPRISES | LATEXITE XL1000 | | |
| DALTON ENTERPRISES/PMI | SUPERSEAL DRIVE MATE 6 | | |
| DALTON ENTERPRISES/PMI | SUPERSEAL NEW DRIVE 3 | | |
| DALTON ENTERPRISES/PMI | SUPERSEAL XS10 | | |
| DEWITTS | 518-DMAX DRIVESHIELD | YES | <1,000 PPM, MEETS DOEE "GOLD" LIMIT |
| DEWITTS | DS2000 | | SAMPLE AVAILABLE AT SITELAB |
| DEWITTS | DS4000 | | SAMPLE AVAILABLE AT SITELAB |
| DEWITTS | DS5000 | | SAMPLE AVAILABLE AT SITELAB |
| DEWITTS | DS6000 | | SAMPLE AVAILABLE AT SITELAB |
| ENVIRONMENTAL SEALER SUPPLIES | ENVIROKOTE | | |
| GARDNER GIBSON | APOC 330 | | |
| GARDNER GIBSON | BLACKJACK DRIVEASEAL 200 | | |
| GARDNER GIBSON | BLACKJACK DRIVEKOTE 500 | | |
| GARDNER GIBSON | BLACKJACK DRIVEMAXX 500 | | |
| GARDNER GIBSON | BLACKJACK NEWBLACK 300 | YES | <1,000 PPM, MEETS DOEE "GOLD" LIMIT |
| GARDNER GIBSON | BLACKJACK DRIVEMAXX 700 | YES | <1,000 PPM, MEETS DOEE "GOLD" LIMIT |
| GARDNER GIBSON | BLACKJACK DRIVEMAXX 1000 | YES | <1,000 PPM, MEETS DOEE "GOLD" LIMIT |
| GARDNER GIBSON | BLACKJACK ULTRAMAXX 1000 | | |
| GARDNER GIBSON | CAS73 COMMERCIAL SEALER | | |
| GARDNER GIBSON | GAS6470 COMMERCIAL SEALER | | |
| GARDNER GIBSON | GAS6480 COMMERCIAL SEALER | | |
| GARDNER GIBSON | DRIVESEAL 2 | | |
| GARDNER GIBSON | DRIVESEAL 4 | | |
| GARDNER GIBSON | DRIVESEAL 8 | | |
| GARDNER GIBSON | DRIVESEAL 10 | | |
| GARDNER GIBSON | BJ ARMORMAX | | |
| GEMSEAL | GUARDIAN AE | | |
| GEMSEAL | GUARDIAN PM | YES | <1,000 PPM, MEETS DOEE "GOLD" LIMIT |
| GUARDTOP | NEYRA GUARDTOP | YES | <1,000 PPM, MEETS DOEE "GOLD" LIMIT |
| GUARDTOP | ULTRAHIGH | | |
| HENRY | HE130 | YES | <1,000 PPM, MEETS DOEE "GOLD" LIMIT |
| HENRY | HE175 | | |
| HENRY | HE200 | | |
| HENRY | HE532 | | |
| INTEGRATED PAVEMENT SOLUTIONS | HAS | | NO CAS#, ONLY "PETROLEUM ASPHALT" |

***FIGURE 10***

**ASPHALT-BASED SEALCOATS *CONTINUED***
SOURCE OF HYDROCARBONS: CAS# 8052-42-4

| MANUFACTURER | BRAND NAME | TESTED | COMMENTS |
|---|---|---|---|
| JETCOAT | DRIVEWAY SEALER 25705 | | |
| JETCOAT | EZ STIR 25735 | YES | <1,000 PPM, MEETS DOEE "GOLD" LIMIT |
| JETCOAT | PREMIUM 25745 | | |
| JETCOAT | SELECT 25775 | | |
| JETCOAT | STANDARD 25715 | | |
| JETCOAT | SUPREME 25765 | | |
| JETCOAT | ULTRA 25755 | | |
| KST | KOOLSEAL KS0073300 | | |
| KST | KOOLSEAL KS0073600 | | |
| KST | KOOLSEAL KS0073900 | | |
| NEYRA INDUSTRIES | JENNITE AE | | |
| NEYRA INDUSTRIES | NEYRA AE | YES | <1,000 PPM, MEETS DOEE "GOLD" LIMIT |
| NEYRA INDUSTRIES | PAVESHIELD | YES | <1,000 PPM, MEETS DOEE "GOLD" LIMIT |
| RAYNGUARD | OVERCOAT | | |
| RAYNGUARD | STEEL GUARD | | |
| RAYNGUARD | STEEL GUARD 65 | | |
| SEABOARD | AE-36 | | SAMPLE AVAILABLE AT SITELAB |
| SEABOARD | EM-50-TT | | |
| SEABOARD | EQUINOX LN-11 GILSONITE | | SAMPLE AVAILABLE AT SITELAB |
| SEALMASTER | ASPEN | | |
| SEALMASTER | EZ STIR | | |
| SEALMASTER | LIQUID ROAD | YES | <1,000 PPM, MEETS DOEE "GOLD" LIMIT |
| SEALMASTER | LIQUID ROAD ULTRA | | |
| SEALMASTER | MASTERSEAL | YES | <1,000 PPM, MEETS DOEE "GOLD" LIMIT |
| SEALMASTER | MASTERSEAL PMM | YES | <1,000 PPM, MEETS DOEE "GOLD" LIMIT |
| SEALMASTER | MASTERSEAL PMM ULTRA | | |
| SEALMASTER | MASTERSEAL ULTRA | | |
| SEALMASTER | OPTIPAVE | | |
| STAR | MICROPAVE | YES | <1,000 PPM, MEETS DOEE "GOLD" LIMIT |
| STAR | MICROPAVE AVIATOR | | |
| STAR | MICROPAVE PRO BLEND | | |
| STAR | MICROPAVE SUPREME | | |
| U.S. SEAL INTERNATIONAL | PITCH BLACK | YES | <1,000 PPM, MEETS DOEE "GOLD" LIMIT |
| VANCE BROTHERS | ULTRA SEAL | | ALSO CONTAINS COKE, CAS# 64743-05001 |
| VELVETOP | ASPHALT SEALER | | |

**LATEX-BASED SEALCOATS**
SOURCE OF HYDROCARBONS: ALKYD LATEX (SOYBEAN OIL) or ACRYLIC LATEX, CAS#25085-34-1 or CAS# 25067-01-0

| MANUFACTURER | BRAND NAME | TESTED | COMMENTS |
|---|---|---|---|
| AEXCEL | BIOSEALCOAT | | SAMPLE AVAILABLE AT SITELAB |
| REICHHOLD | BECKOSOL AQ-510 | | |
| DALTON ENTERPRISES | ACRYLIC GRADE SEALER | | |
| DALTON ENTERPRISES | LIFT DRIVEWAY OVERLAY | | |
| GARDNER GIBSON | ARMOR MAXX | | |
| REICHHOLD | BECKOSOL AQ-510 | | |

**ECR-BASED, "CRACKED RESIDUE" SEALCOATS**
SOURCE OF HYDROCARBONS: CAS# 64742-90-1

| MANUFACTURER | BRAND NAME | TESTED | COMMENTS |
|---|---|---|---|
| BREWER | ECLIPSE 167710 | | |
| CRAFCO | ACTIONPAVE LP | | |
| DALTON ENTERPRISES | XL 1000 | | |
| GEMSEAL | BLACK DIAMOND | YES | PAHs >10,000 PPM, DOES NOT QUALIFY |

***FIGURE 10***

**ECR-BASED, "CRACKED RESIDUE" SEALCOATS *CONTINUED***
SOURCE OF HYDROCARBONS: CAS# 64742-90-1

| MANUFACTURER | BRAND NAME | TESTED | COMMENTS |
|---|---|---|---|
| GEMSEAL | BLACK DIAMOND XL | | |
| GEMSEAL | BLACK DIAMOND XL | | |
| NEYRA INDUSTRIES | NEYRA FORCE | YES | PAHs >10,000 PPM, DOES NOT QUALIFY |
| SEALMASTER | LP SEALER | | |
| STAR | TRITON | YES | PAHs >10,000 PPM, DOES NOT QUALIFY |
| VELVETOP | SUPER PAVEMENT SEALER | | |

**COAL TAR-BASED SEALCOATS**
SOURCE OF HYDROCARBONS: CAS# 65996-93-2

| MANUFACTURER | BRAND NAME | TESTED | COMMENTS |
|---|---|---|---|
| BREWER | BREWER COTE | | |
| BREWER | POLY COTE | | |
| BREWER | SPEC COTE | | |
| COSMICOAT | SPEC SEAL | | |
| CRAFCO | ACTION PAVE RT | | |
| FACTORY DIRECT CHEMICALS | ASPHALT SEALER | | |
| GEMSEAL | FED SPEC | YES | PAHs >10,000 PPM, DOES NOT QUALIFY |
| GEMSEAL | TARPRIME POLYTAR | | |
| GEMSEAL | PRO BLEND | | COAL TAR AND ASPHALT MIX |
| MAINTENANCE, INC | ADVANCED FORMUA J16 | | |
| MAINTENANCE, INC | FASS DRI | | |
| NEYRA INDUSTRIES | JENNITE | | |
| NEYRA INDUSTRIES | TARCONITE | | |
| SAKRETE | BLACKTOP SEALER | YES | PAHs >10,000 PPM, DOES NOT QUALIFY |
| SAKRETE | FLO COAT | | |
| SAKRETE | PREMIUM SEALER | | |
| SEABOARD | TE1 | | |
| SEABOARD | TE2 | | |
| SEABOARD | TE36 | | SAMPLE AVAILABLE AT SITELAB |
| SEALMASTER | CONCENTRATE SMT 100 | | |
| SEALMASTER | EZ STIR | | |
| SEALMASTER | POLYMER MODIFIED | | |
| SEALMASTER | S1000 | | |
| SEALMASTER | ULTRA | | |
| STAR | STARSEAL | | |
| STAR | STARSEAL AVIATOR | | |
| STAR | STARSEAL PROBLEND | | |
| STAR | STARSEAL SUPREME | | |
| VANCE BROTHERS | PROTEC TAR | | |

Many brands exist that contain the same source of hydrocarbons used to make the product, but include special polymers and other additives to enhance the look and longevity. This is the case with most brand varieties sold at retail stores. The three brands of Blackjack tested, for example, have no PAHs and are made at the same plant with the same asphalt. Despite this, every brand of product must be tested to be certified and each product must be retested every two years to stay certified. These requirements are specified in the QA Protocol.

Tasks 4 and 5 Deliverables for this contract originally included using the approved protocol developed and finalized in Tasks 1 and 2 to create a "Gold" (under 1,000 ppm) and "Silver"(under 10,000 ppm) list of sealant products (Task 4), to be maintained and made publicly available by DOEE after the end of the contract period (Task 5). Several unforeseen delays and budget restrictions prevented Sitelab from completing these tasks.

Tasks 1 and 2 took longer than anticipated and exceeded the original budget as DOEE and CBP prioritized additional testing of products in various conditions by multiple labs to confirm the protocol was robust, consistent, and usable. With approval from the steering committee, DOEE and CBP decided that the most important deliverable of the contract was a well-thought out protocol manufacturers could trust and regulators could use, which was achieved as described in Tasks 1 and 2 above.

The Testing Protocol and Appendix is now publicly available for use by any regulator, manufacturer, or stakeholder at:
https://www.site-lab.com/DOEE-QA-PROTOCOL-APRIL1-2022.pdf

https://www.site-lab.com/CBTRUST-DOEE-PROTOCOL-APPENDIXES-JUNE23-2021.pdf

In addition, the final protocol needed to undergo regulatory approval in the District of Columbia before use could be permitted for DOEE's Gold and Silver lists, which took an unforeseen 9+ months to complete in 2021 and put the project on hold for close to a year. Rather than ask Sitelab complete Task 4 under a constrained, untenable timeline, it was decided efforts were best focused into a detailed, quality final report before the contract deadline of July 1, 2022.

While products were not tested for the Gold and Silver lists under this contract, partnering regulators in Austin, Texas and Mecklenburg County, North Carolina have begun using the testing protocol in partnership with Sitelab to begin building the lists.

DOEE plans to coordinate efforts in creating the Gold and Silver Lists of pavement sealant products with partnering regulators, and has actively begun enforcing the Low-PAH Limit Law in the District using the approved Testing Protocol developed under this contract. DOEE plans to periodically review the protocol, inviting feedback from manufacturers, regulators, and laboratories, in order to ensure the protocol is working as intended, making edits or updates as needed.

To date, a total of sixteen sealcoat products have been tested which meet DOEE's Gold PAH limit and qualify for the certification program. See Figure 11 for details. All of these products are asphalt-based. Nine of these products were tested for the City of Austin, TX, under a different contract. Sitelab began working with Austin testing sealcoat products used in their city. Austin adopted this protocol for their own certification program. This work began in the Spring of 2021, picking up where the work with DOEE left off. No products were tested for DOEE since December 2020.

Starting in February 2022, Sitelab began working with Mecklenburg County in Charlotte, NC, for a similar contract as Austin. One sealcoat product was tested. Laboratory reports and Sitelab summary results performed for Austin and Mecklenburg County have been shared with DOEE. Sealant products tested in the future will also be shared as soon as they become available.

The list of qualified products is posted on Sitelab's website:
https://www.site-lab.com/pahs-coal-tar-sealcoats-certification.htm

***FIGURE 11***

**List of 16 Products Tested-to-Date Which Qualify for DOEE Gold Certification Level**

| **Manufacturer** *In Alphabetical Order* | **Product Brand Name** | **Laboratory Used** | **Lab Report ID No.** | **Date Reported** | **Sitelab Project Performed For** |
|---|---|---|---|---|---|
| Dalton Enterprises | Latexite Airport Grade | Alpha Analytical<br>Pace Contest Labs | L2020460<br>20E0772 | June 12, 2020<br>May 28, 2020 | CBTRUST, DOEE |
| Dewitts | 518-Dmax Driveshield | Pace Contest Labs<br>EMSL<br>Analab<br>GEL Labs | 20J1559<br>012012058<br>943228<br>525695 | November 16, 2020<br>November 25, 2020<br>November 20, 2020<br>November 19, 2020 | CBTRUST, DOEE |
| Gardner Gibson | Blackjack DriveMaxx 300 | Alpha Analytical<br>Pace Contest Labs | L2020460<br>20E0772 | June 12, 2020<br>May 28, 2020 | CBTRUST, DOEE |
| Gardner Gibson | Blackjack DriveMaxx 700 | Alpha Analytical<br>Pace Contest Labs | L2020460<br>20E0772 | June 12, 2020<br>May 28, 2020 | CBTRUST, DOEE |
| Gardner Gibson | Blackjack DriveMaxx 1000 | Pace Contest Labs | 21H1136 | September 3, 2021 | CITY OF AUSTIN |
| Gemseal | Guardian PM | Pace Contest Labs | 21H1136 | September 3, 2021 | CITY OF AUSTIN |
| Guardtop | Neyra Guardtop | Pace Contest Labs | 21H1136 | September 3, 2021 | CITY OF AUSTIN |
| Henry | Henry HE130 | Pace Contest Labs | 22E0555 | May 19, 2022 | CITY OF AUSTIN |
| Jetcoat | Jetcoat E-Z Stir | Pace Contest Labs | 22E0560 | May 25, 2022 | MECKLENBURG CO |
| Neyra Industries | Neyra AE | Pace Contest Labs | 21B0465 | March 9, 2021 | CBTRUST, DOEE |
| Neyra Industries | Paveshield | Pace Contest Labs | 21B0465 | March 9, 2021 | CBTRUST, DOEE |
| Sealmaster | Liquid Road | Pace Contest Labs | 21H1136 | September 3, 2021 | CITY OF AUSTIN |
| Sealmaster | Masterseal | Pace Contest Labs | 21H1136 | September 3, 2021 | CITY OF AUSTIN |
| Sealmaster | Masterseal PMM | Pace Contest Labs | 21H1136 | September 3, 2021 | CITY OF AUSTIN |
| STAR | Micro-Pave | Pace Contest Labs | 22B1349 | March 7, 2022 | CITY OF AUSTIN |
| U.S. Seal International | Pitch Black | Pace Contest Labs | 22D2076 | May 11, 2022 | CITY OF AUSTIN |

Note 1: Laboratory reports performed for each manufacturer's product are strictly confidential and are not available to the public.

Note 2: Products tested by Sitelab Corporation for Department of Energy & Environment in Washington, DC, funded by a U.S. EPA Grant with Chesapeake Bay Trust and contracts with City of Austin, Texas, Watershed Protection Department and Mecklenburg County, North Carolina, Charlotte-Mecklenburg Storm Water Services.