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Laboratory testing of PAHs and alkylated PAHs in shellfish is typically performed using Gas Chromatography/Mass Spectrometry (GC/MS) in the Selective Ion Monitoring (SIM) mode. PAH (Polycyclic Aromatic Hydrocarbons) and alkylated PAH levels can be determined for a variety of shellfish, including shrimp, prawn, lobster, crayfish, crabs, clams, mussels, oysters and scallops. Analytical testing of seafood has received heightened attention recently due to the Gulf Oil Spill.

Analytical Procedure for Testing PAHs in Shellfish

The key to the analytical procedure is proper sample preparation: shucking, compositing (as appropriate to the project plan), and homogenization via mechanical mixing. Preliminary preparation under clean laboratory conditions prevents contamination by common PAHs. Decontamination of

sample preparation equipment is performed and monitored to assure clean conditions. The sample homogenate consists of homogenous slurry when prepared correctly. Columbia Analytical has been inspected and approved by various organizations (e.g. EPA, private industry, private consultants, etc.) during homogenization activities prior to receiving clearance to proceed with selected projects with high public, private, and government visibility.

After homogenization, samples are solvent extracted and subjected to cleanups via silica gel to remove interfering matrix components. Again, careful control of laboratory conditions and equipment is critical to assuring valid results. The cleanups are essential to remove interfering background components that mask the target compounds. Insufficient cleanup results in potential false positive results and/or elevated detection limits that render the data useless in many cases.

The SIM mode is used to further improve selectivity but also increases sensitivity. Typical reporting limits for shellfish range from 0.5 ng/g (ppb) to 5 ng/g (ppb), depending on the data quality objectives outlined in the project plan. The associated Method Detection Limits (MDL) are on the order of five to ten times lower than the reporting limit. When performing the low range determinations, the reporting limit of 0.5 ng/g (ppb) is derived from the lowest standard in the calibration curve.

The inclusion of alkylated homologs is critical to the forensic aspect of the determinations, which provides the connection to the source of the PAHs. The ratios of the various PAHs with substituted low molecular weight alkyl groups provide unique chemical characteristics that relate to the source of the contamination.

Columbia Analytical testing conforms with all requirements for production of a legally defensible data package. From receipt at the laboratory to final review of the report, every sample processing operation is recorded in the package, allowing the entire process to be re-created at a later time. Any anomalies associated with the analysis of the samples are discussed in narrative form. A standard deliverable includes full summaries of all quality assurance and quality control (QA/QC) operations associated with the analysis. Importantly, self-validating reporting software performs QA/QC checks that automatically document any anomaly in the data. This ensures that an issue can be addressed prior to releasing final reports.

Core List of PAH and Alkylated PAHs by EPA 8270/SIM*

1-Methylnaphthalene	C1-Benzothiophenes
2-Methylnaphthalene	C2-Benzothiophenes
Acenaphthene	C3-Benzothiophenes
Acenaphthylene	C2-Naphthalenes
Anthracene	C2-Fluoranthenes/Pyrenes
Benz(a)anthracene	C3-Fluoranthenes/Pyrenes
Benzo(a)pyrene	C1-Chrysenes
Benzo(b)fluoranthene	C2-Chrysenes
Benzo(b)thiophene	C3-Chrysenes
Benzo(e)pyrene	C4-Chrysenes
Benzo(g,h,i)perylene	C3-Naphthalenes
Benzo(k)fluoranthene	C4-Naphthalenes
Biphenyl	C1-Fluorenes
Chrysene	C2-Fluorenes
Dibenz(a,h)anthracene	C3-Fluorenes
Dibenzofuran	C1-Dibenzothiophenes
Dibenzothiophene	C2-Dibenzothiophenes
Fluoranthene	C3-Dibenzothiophenes
Fluorene	C4-Dibenzothiophenes
Indeno(1,2,3-cd)pyrene	C1-Phenanthrenes/Anthracenes
Naphthalene	C2-Phenanthrenes/Anthracenes
Naphthobenzothiophene	C3-Phenanthrenes/Anthracenes
Perylene	C4-Phenanthrenes/Anthracenes
Phenanthrene	C1-Naphthobenzothiophenes
Pyrene	C2-Naphthobenzothiophenes
	C3-Naphthobenzothiophenes
	C1-Fluoranthenes/Pyrenes

*More compounds are being added to this list. Please contact the laboratory for a list of the most current compounds.

For more information or Gulf spill support please contact the Columbia Analytical Team Lead, Greg Salata, at 360.501.3376.

 **Columbia Analytical Services**SM