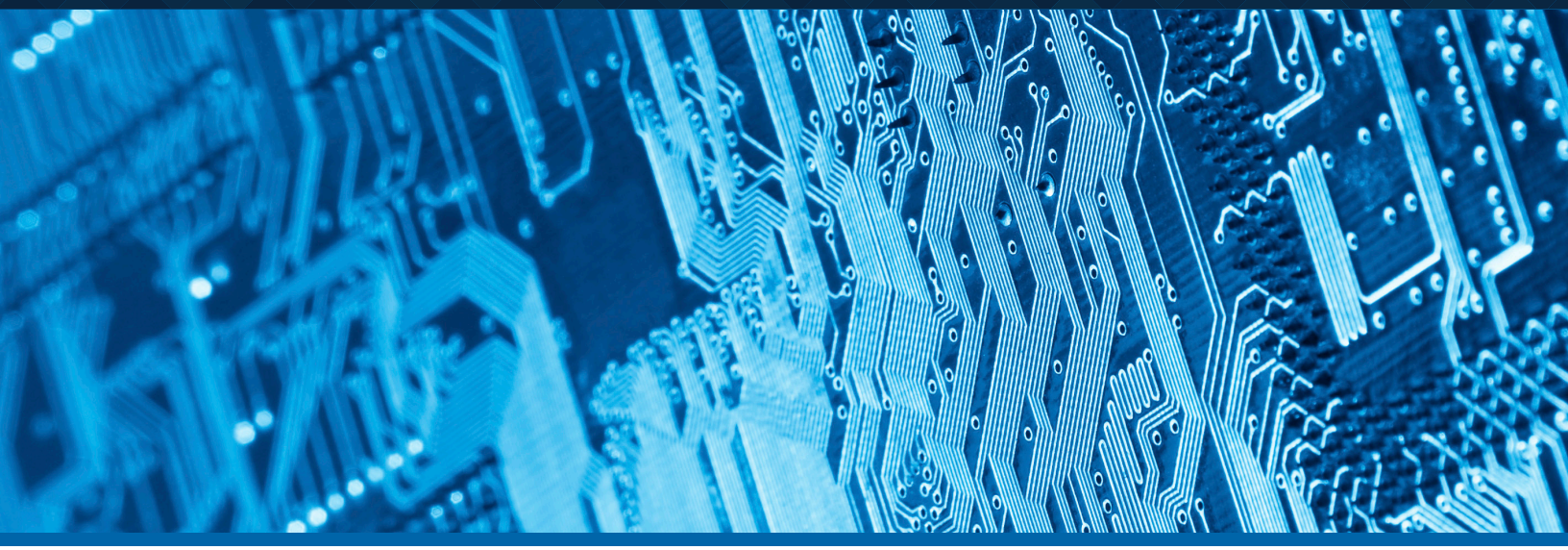

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prep*FAST* CARBON – An Automated Laboratory System for Low PPT Detection of High Molecular Weight Organic Contaminants in Semiconductor Grade Chemicals

Brief

prep*FAST* CARBON combines the high sensitivity of electrospray ionization (ESI) and accurate mass determination of time-of-flight mass spectrometry (TOF-MS) with advanced automation to enable the real time detection of trace organics in semiconductor solvents, acids, and bases with parts per trillion (ppt) detection limits. prep*FAST* CARBON provides superior speed of analysis and improved detection limits over traditional methods of chromatographic separation.

- Barcode reading for sample identification
- Uncapping and recapping of sample bottles
- Ultraclean sample enclosure
- Inline standard addition calibrations
- Automated quantification of targeted contaminants
- Automated near quantitative estimation of other contaminants
- Qualitative analysis between samples via spectral difference plots

Figure 1. A prep*FAST* CARBON bench attached to an Agilent QTOF-MS

Introduction

Purity requirements for materials used in semiconductor fabrication process are becoming more stringent as the industry moves towards 3 and 2 nm nodes. While SEMI specifications are mainly focused on trace metals, there is a growing concern about organic impurities as they can be precursors to nanoparticle and residue formation on wafer surfaces during the drying process. The 2023 IRDS roadmap recognized this, projecting that the requirement for high molecular weight organic contaminations (>10 carbon atoms) in IPA will decrease from 50 ppb in 2023 to 10 ppb in 2027. Contaminated chemicals have impacted operations at fabrication facilities in the recent past, resulting in huge financial impacts. prep*FAST* CARBON is the solution for identifying and quantifying organic contaminants in IPA and other semiconductor grade solvents, meeting the current and future requirements outlined by the IRDS roadmap.

System Details

prep*FAST* CARBON is a highly automated lab system for detection and quantification of low ppt level organics and anions in a large variety of solvents, through automated sample capping, recapping, barcode reading, and more.

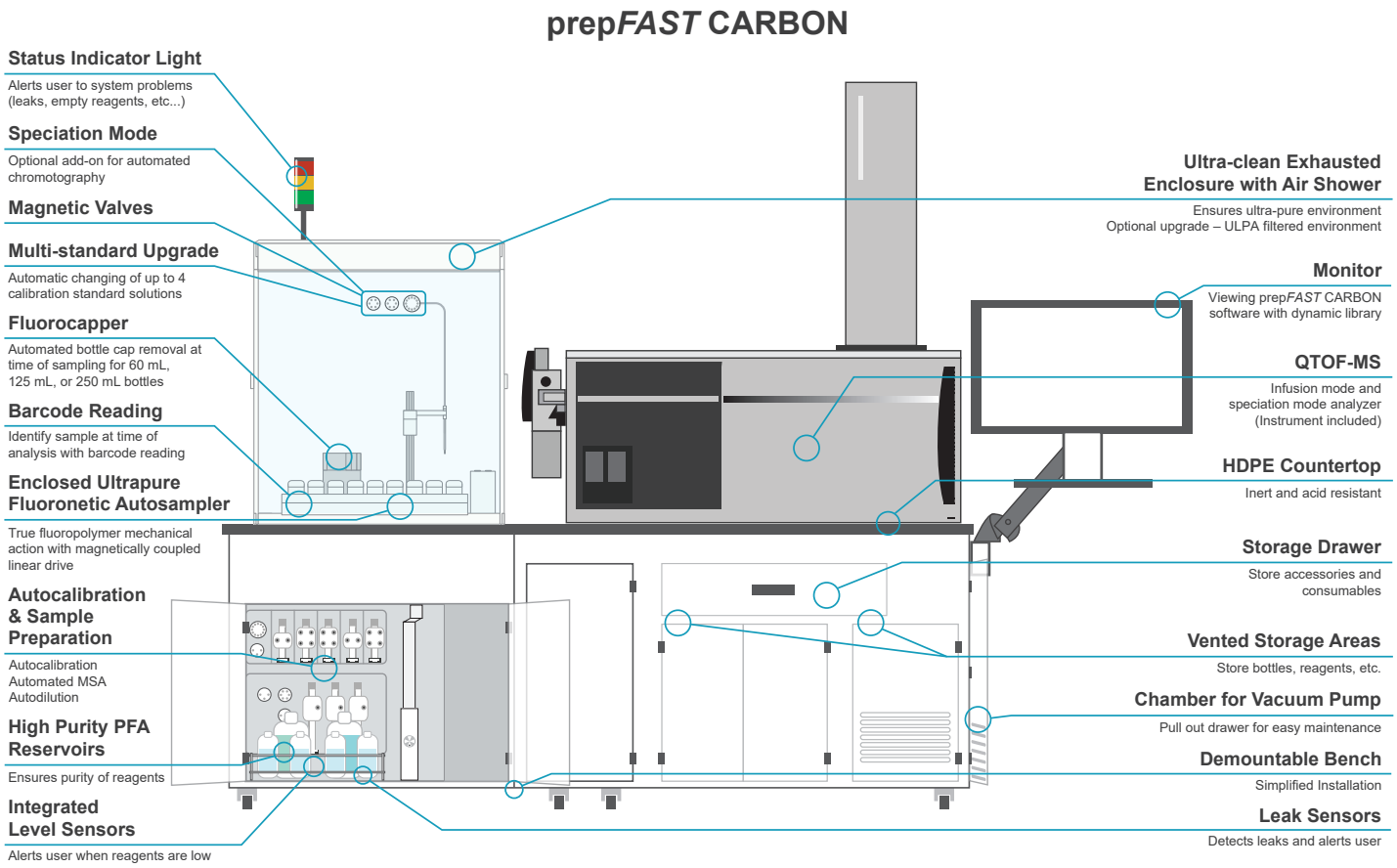


Figure 2. Features diagram of the prep*FAST* CARBON

Introduction *(Continued)*

Table 1. A list of semiconductor chemicals that can be analyzed by prep*FAST* CARBON. The list includes the most chemicals used in the semiconductor industry. Other chemicals can also be analyzed.

	Chemicals Analyzed by prep*FAST* CARBON										
Chemical	UPW	IPA	PGME	PGMEA	Cyclohexanone	Ethyl Lactate	nBa	NH_4OH	TMAH	H_2O_2	H_2SO_4
Type	Water	Solvent	Solvent	Solvent	Solvent	Solvent	Solvent	Base	Base	Oxidizer	Acid

Advanced Automation

prep*FAST* CARBON automatically scans PFA barcoded bottles before sampling. Then, it uncaps, aliquots sample, and recaps the sample bottle quickly and efficiently. The data obtained from this sample remains associated with the barcode throughout the entire facility. Standards can be automatically added to the sample to generate a calibration curve. Response factors from the calibration are used in quantification of contaminants.

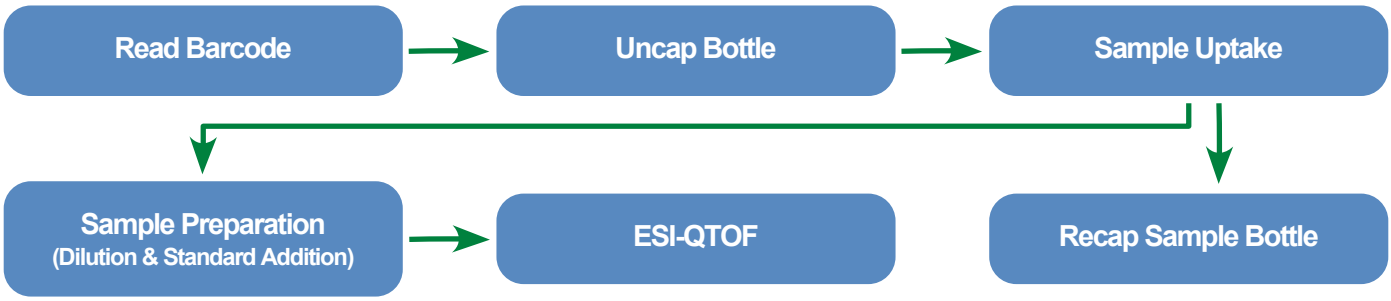


Figure 3. Workflow diagram for analysis using the prep*FAST* CARBON system

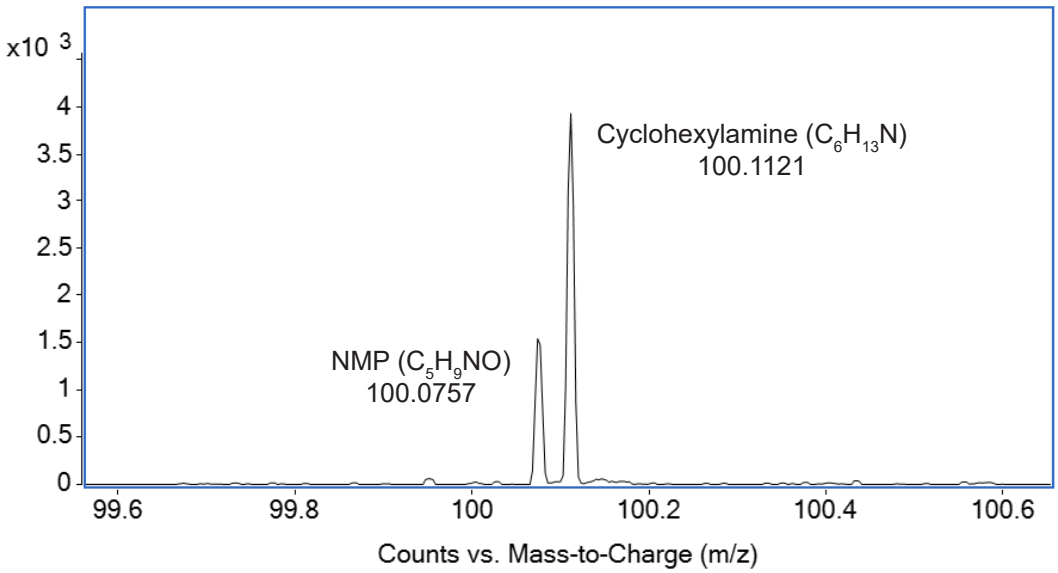


Figure 4. ESI-QTOF allows detection and peak differentiation up to 0.0001 amu.

Simplified Preparation and Analysis

In-line addition of both internal standard and calibration standards provides a means to limit human error and contamination, improve analysis speed, and minimize hands-on lab work.

In prep*FAST* CARBON's infusion mode, a single sample can be analyzed in both positive and negative ion modes. Extracted spectra reproducibly maintains high resolution and mass accuracy.

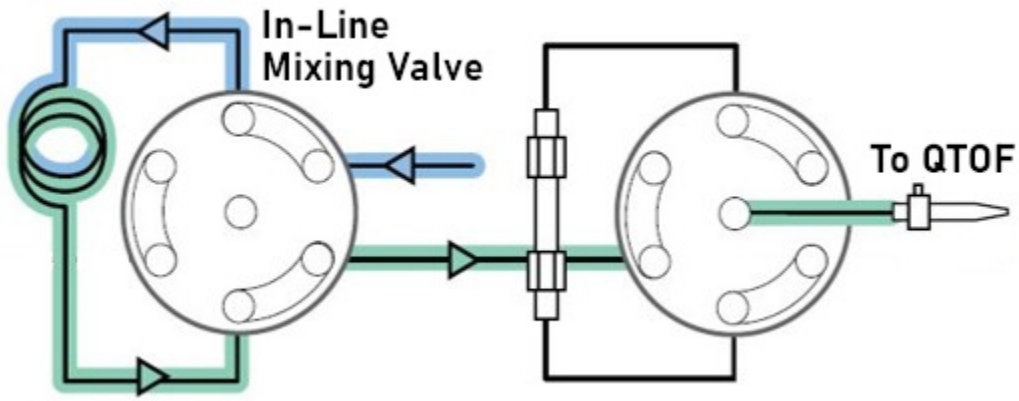


Figure 5. Diagram of the prep*FAST* CARBON during in-line standard spiking

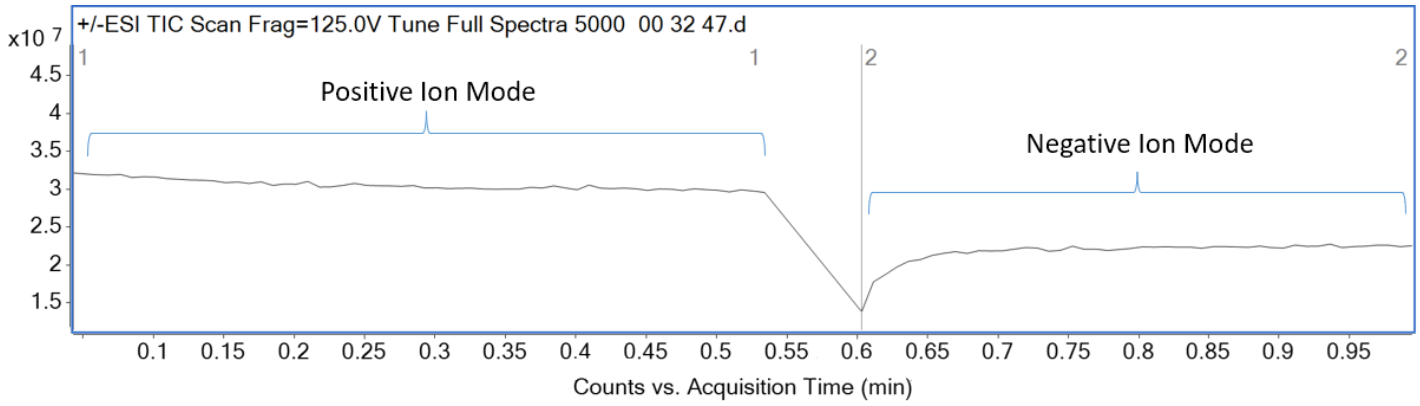


Figure 6. Total-Ion chromatogram of a sample as seen in the provided data analysis software. Each sample is run in both positive and negative ion mode, displayed in real time.

Autogenerated Calibration Curves

- prep*FAST* CARBON system automatically introduces calibration standards at user-defined levels
- prep*FAST* CARBON software automatically generates calibration curves for use in quantitation

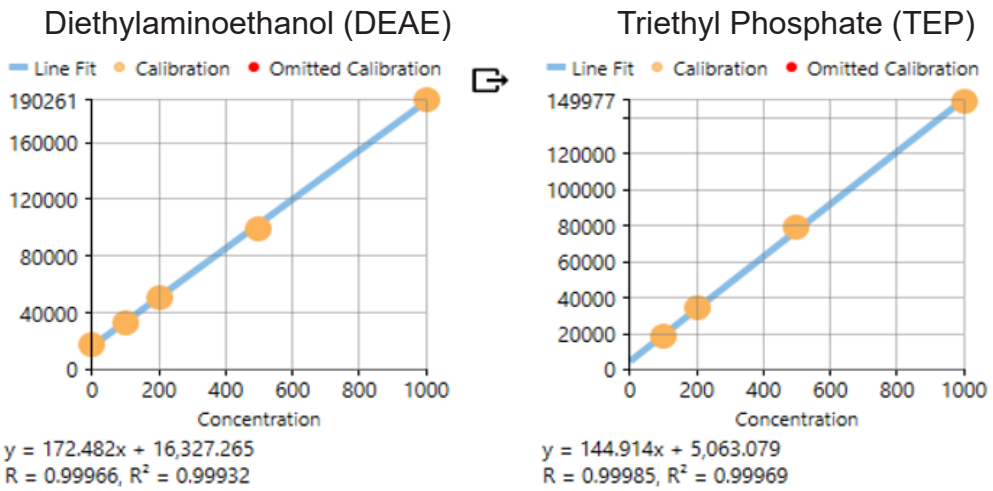


Figure 7a. Autogenerated calibration curves at the ppb level by prep*FAST* CARBON software.

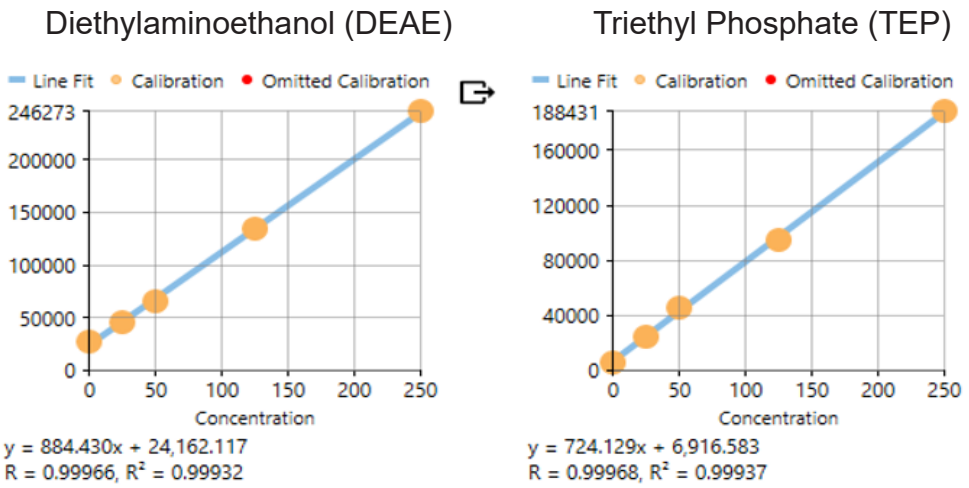


Figure 7b. Autogenerated calibration curves at the ppt level by prep*FAST* CARBON software.

Dynamic Calibration Libraries

prep*FAST* CARBON's processing software utilizes an expansive data library to identify compounds based on the measured mass. Users can add new contaminants to the dynamic library. Quantitative results of user selected library compounds are automatically generated by NearQuant.

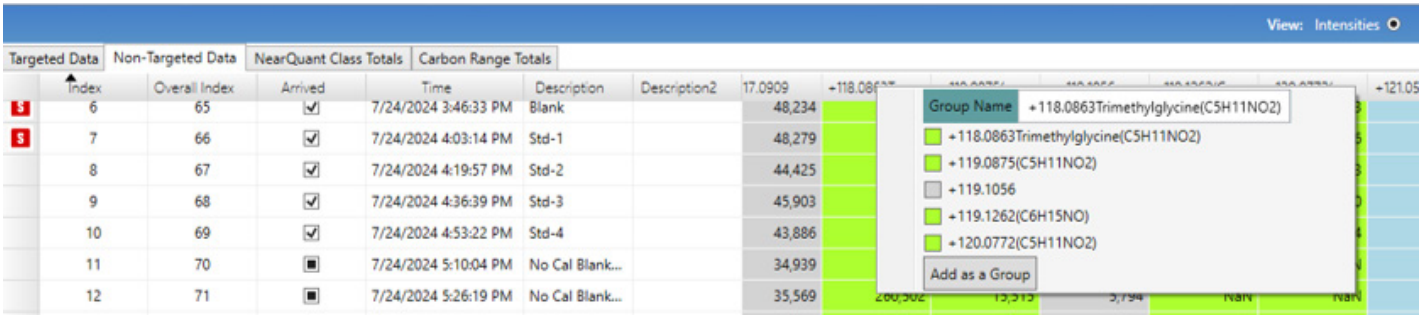


Figure 8. Example of a dynamic calibration library

NearQuant

NearQuant is an algorithm of classifying compounds based on the principle that compounds with similar properties (i.e. Amines, Sulfates) ionize similarly. NearQuant generates approximate quantitation of unknown compounds with mass accurate, autogenerated formulas utilizing response factors obtained from targeted compounds in a user's calibration standard.

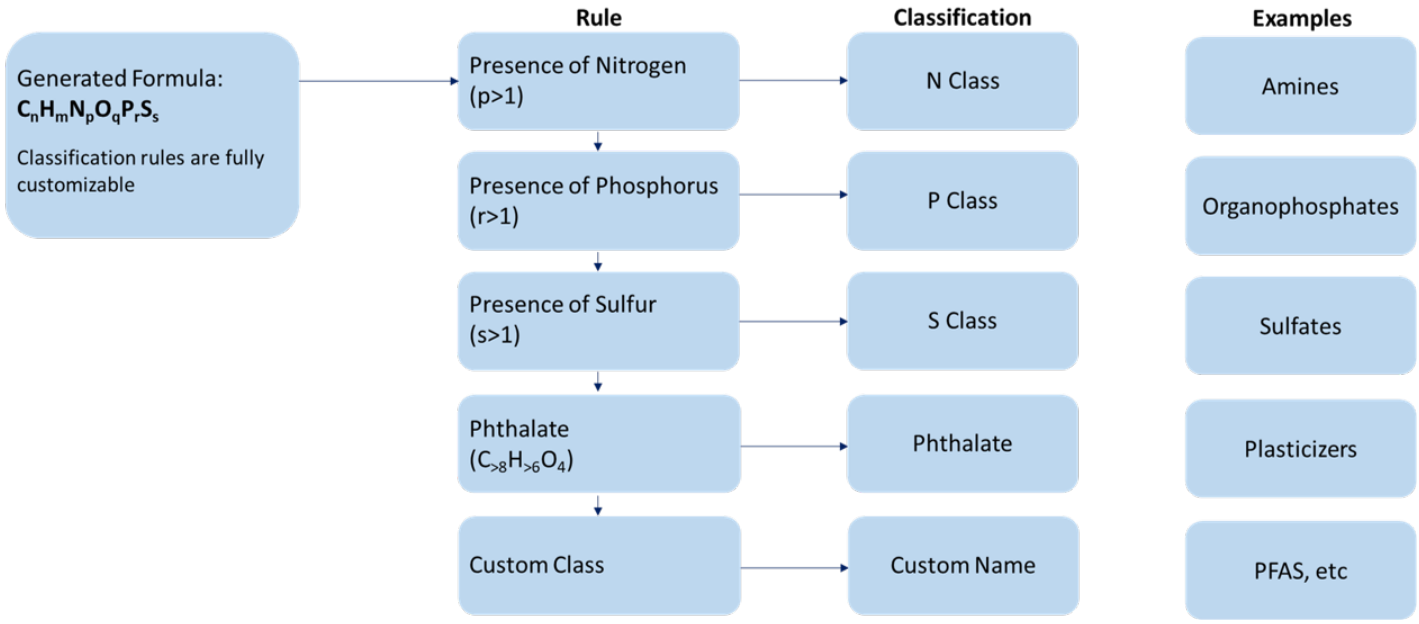
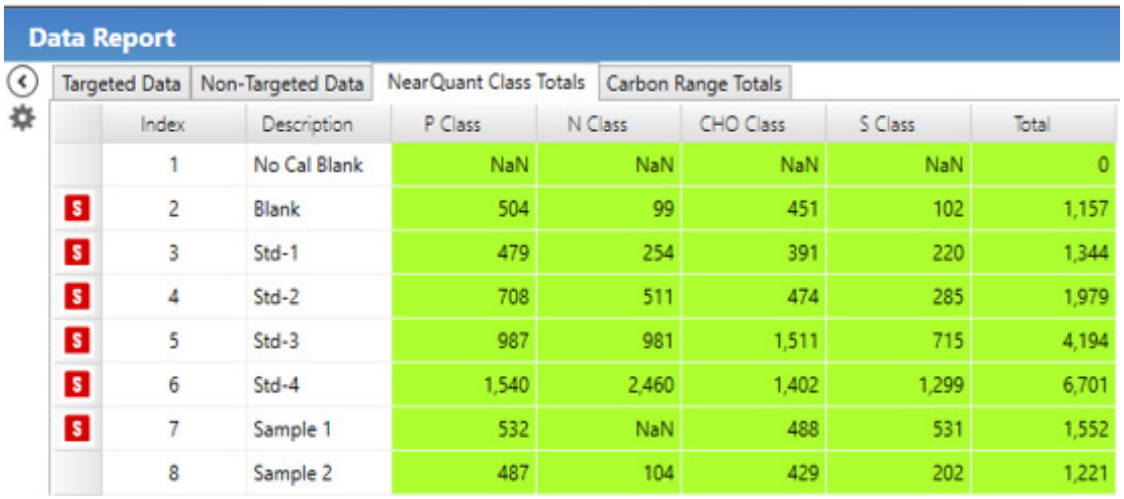


Figure 9. NearQuant algorithm for approximating unknown compound concentration

NearQuant *(Continued)*

NearQuant is configured to group by class. The default classification option includes P Class (Phosphates), N Class (Nitrates), S Class (Sulfates) and CHO class (phthalates, etc.). The user can remove classes and add new ones, as well as add and remove class restrictions as seen fit. NearQuant also displays carbon-binned data, with customizable carbon number for each bin.

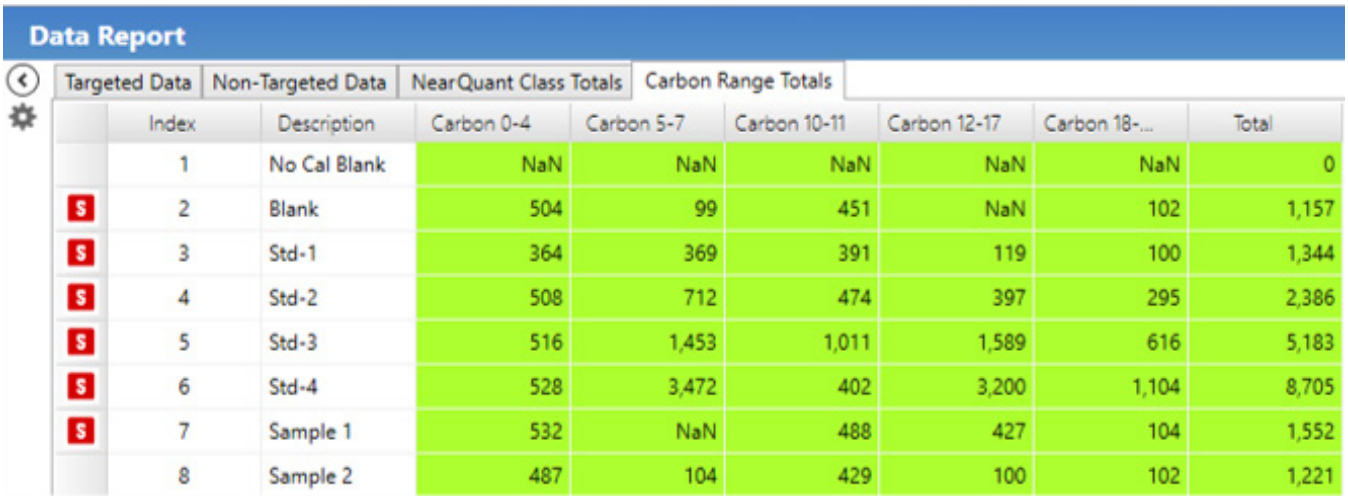


Data Report

Targeted Data | Non-Targeted Data | NearQuant Class Totals | Carbon Range Totals

	Index	Description	P Class	N Class	CHO Class	S Class	Total
	1	No Cal Blank	NaN	NaN	NaN	NaN	0
S	2	Blank	504	99	451	102	1,157
S	3	Std-1	479	254	391	220	1,344
S	4	Std-2	708	511	474	285	1,979
S	5	Std-3	987	981	1,511	715	4,194
S	6	Std-4	1,540	2,460	1,402	1,299	6,701
S	7	Sample 1	532	NaN	488	531	1,552
	8	Sample 2	487	104	429	202	1,221

Figure 10. Report showing contaminants grouped by NearQuant classification



Data Report

Targeted Data | Non-Targeted Data | NearQuant Class Totals | Carbon Range Totals

	Index	Description	Carbon 0-4	Carbon 5-7	Carbon 10-11	Carbon 12-17	Carbon 18-...	Total
	1	No Cal Blank	NaN	NaN	NaN	NaN	NaN	0
S	2	Blank	504	99	451	NaN	102	1,157
S	3	Std-1	364	369	391	119	100	1,344
S	4	Std-2	508	712	474	397	295	2,386
S	5	Std-3	516	1,453	1,011	1,589	616	5,183
S	6	Std-4	528	3,472	402	3,200	1,104	8,705
S	7	Sample 1	532	NaN	488	427	104	1,552
	8	Sample 2	487	104	429	100	102	1,221

Figure 11. Report showing contaminants binned by carbon number

Qualitative Analysis

prep*FAST* CARBON can also be used to obtain information on qualitative differences between samples. The software displays mass spectra of samples as well as difference spectra of the two selected samples. The difference spectra enable the user to quickly highlight the qualitative differences between the two samples. This is valuable when comparing samples from different batches, different manufacturers, different sampling points, etc.

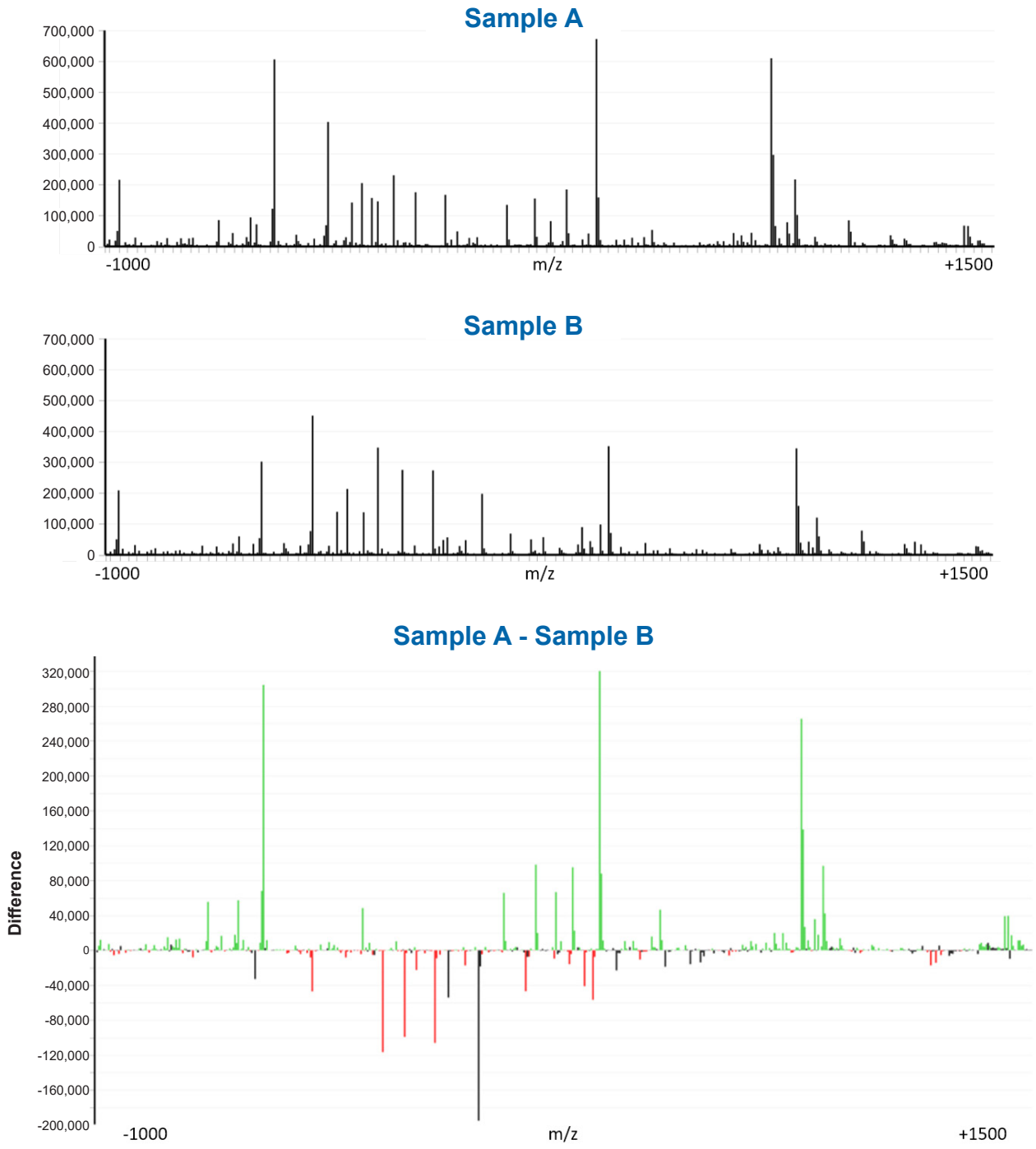


Figure 12. Difference plot comparing two samples, in which Sample B was subtracted from Sample A. Black bars indicate unique compounds, green bars indicate a greater signal for the source on top (Sample A), and red bars indicate a greater signal for the bottom source (Sample B). While hovering over a peak in the difference plot, the software displays mass-to-charge ratio, formula, compound ID, and relative intensity.

CarbonTrends

CarbonTrends is a part of the prep*FAST* CARBON software which shows how compounds change over time across samples. It enables detection of changing contaminant levels, exposure to potential environmental contaminants, or systematic errors. CarbonTrends can track contaminants over long time periods, up to months of consistent tracking.

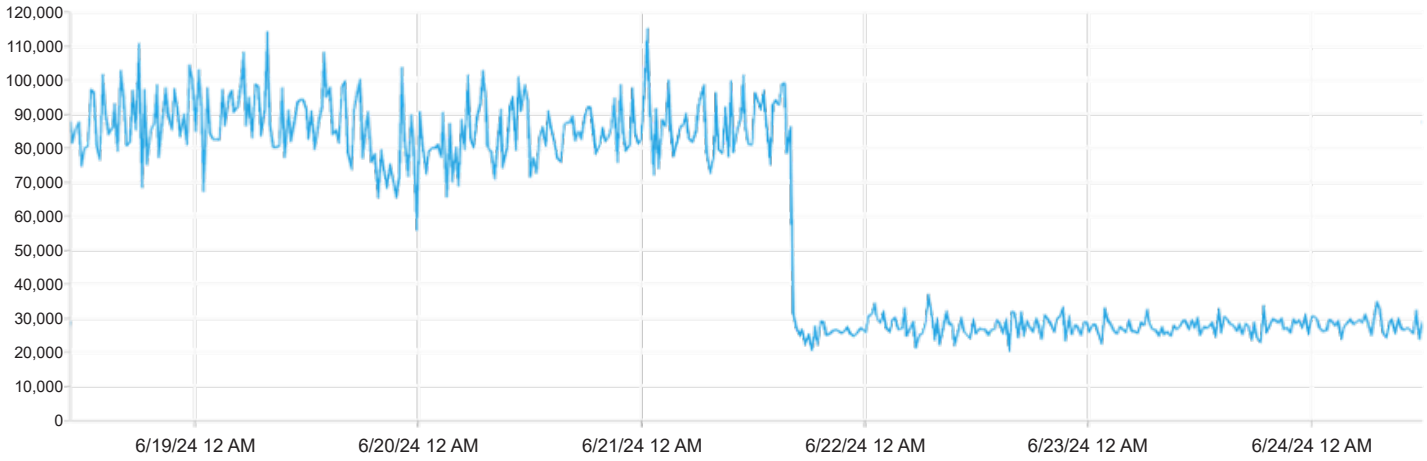


Figure 13. A Trends chart showing change in concentration of a single compound over time. In this instance, a sample contaminated with Triditertbutylphenylphosphate was replaced with a clean sample.

Contamination Alerts

Alerts are a highly customizable system that trigger various effects, such as re-running a sample using the same *FAST* method with new parameters or washing a dirty system. prep*FAST* CARBON can automate the identification and resolution of issues that can happen during analysis by setting limits on common issues such as saturated compounds, high levels of selected contaminants, or low I.S. recovery.

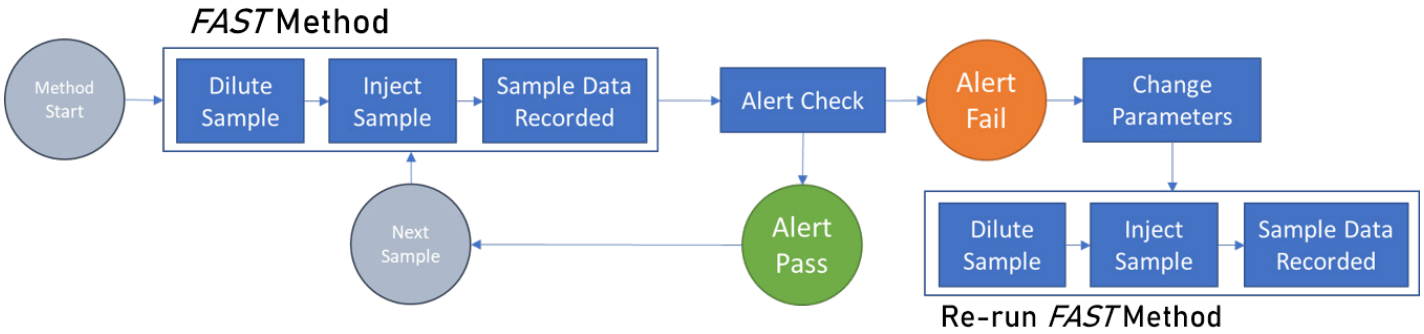


Figure 14. A flowchart demonstrating a general alert setup

Conclusion

prep*FAST* CARBON is a lab system with innovative and advanced automation, offering the solution for low level organic compound detection and analysis with low levels of hands-on lab time. It has ppt detection of contaminants, intuitive and highly customizable software, NearQuant algorithm for quantifying unknown organics, CarbonTrends for longer term sample monitoring, a reactive alert system, and more. prep*FAST* CARBON is essential for laboratories looking to elevate their analysis of organics.

Notes