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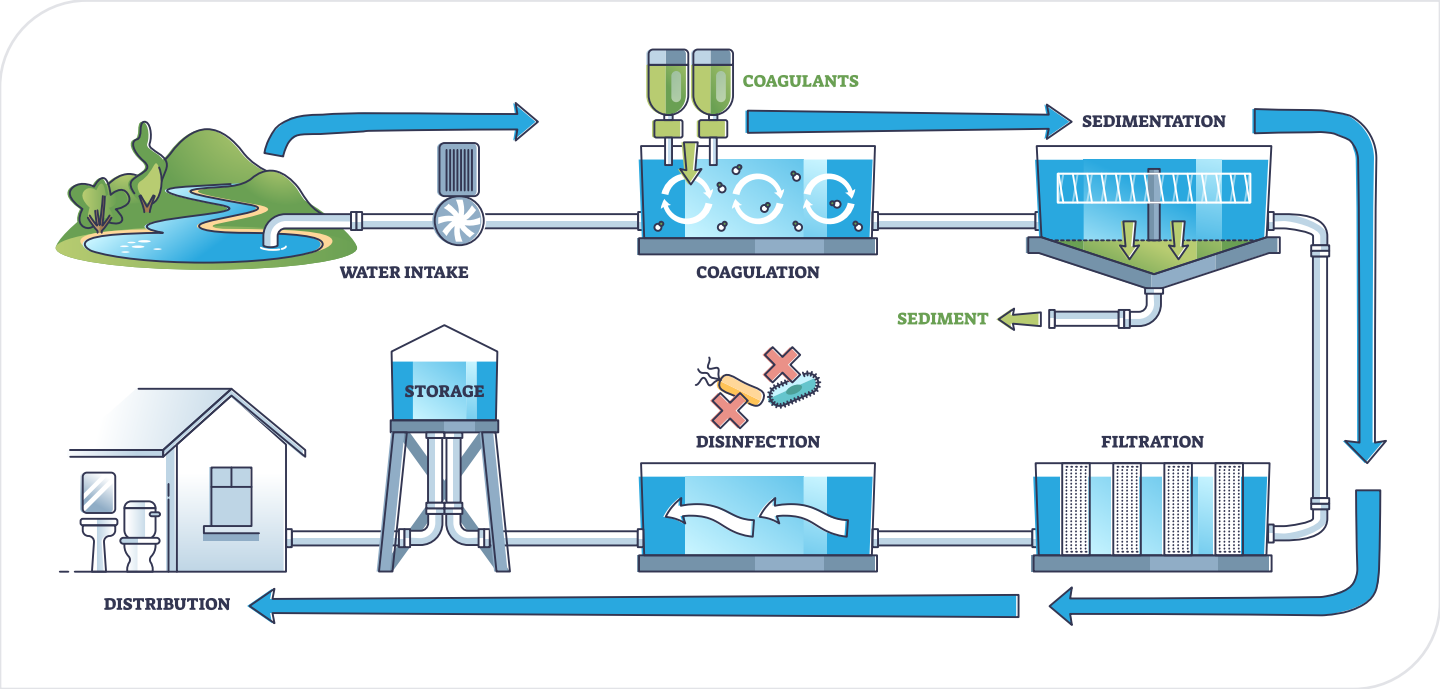
Application Note

THM Analysis with N_2 as Purge and Carrier Gas

Analyze for trihalomethanes using purge and trap concentration with gas chromatographic separation and surface acoustic wave detection



Introduction

During water treatment and delivery, organic compounds react with free chlorine to form trihalomethanes (THMs). THMs are part of a group of compounds known as disinfection by-products (DBPs) and include chloroform, dichlorobromomethane, dibromochloromethane, and bromoform. These are considered a health risk when elevated levels are present. Formation of these compounds can vary by such factors as the water source, pH, temperature, coagulant treatment, and disinfectant dose. THMs are regulated by the Safe Drinking Water Act (SDWA) under the Environmental Protection Agency (EPA). The SDWA contains National Primary Drinking Water Regulations where maximum contaminant levels are set. The limit for total trihalomethanes is 80 ppb.

Water treatment plants and distribution systems are required to monitor THMs at various monitoring sites to ensure compliance and keep the treatment process optimized. Plant operators need real time data to adjust treatment processes, especially the impact of disinfectant dosage, that affect THM formation.

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The classic way to determine THMs typically includes concentration by purge and trap (P&T) with separation by gas chromatography and detection by mass spectrometry (GC/MS). A common solution for testing THMs includes the collection of a sample and sending it to a commercial laboratory for testing. This process may take days to weeks to obtain the results. This delay in reporting is not practical since a THM event may have occurred, and no proper mitigation strategies could be implemented. Bringing P&T-GC/MS testing to the municipal lab bench has been cost prohibitive and a very complex technique to learn and maintain.

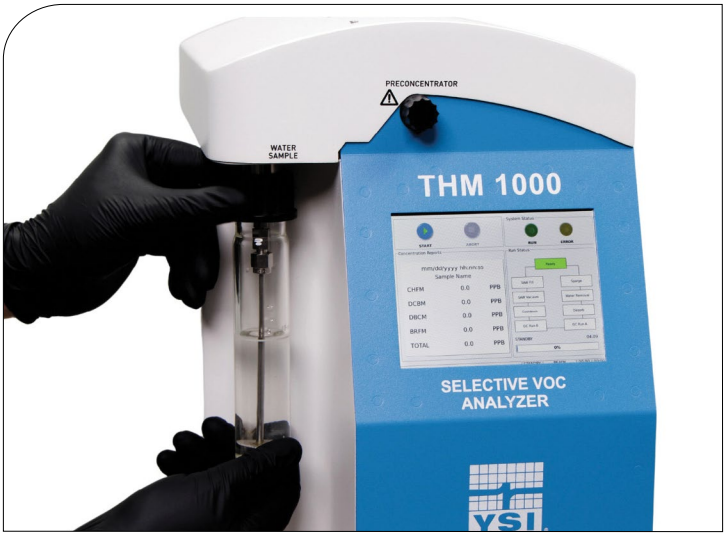
A new solution for effective monitoring of THMs now exists that is fast, simple, and reliable. The THM 1000 Selectable VOC Benchtop Analyzer has combined the P&T, GC, and detector into one simple analyzer. The innovative portion of this solution is the SAW (surface acoustic wave) detector. This unique monolithic detector is coated with a nonporous carbon layer and provides almost instant and extremely sensitive responses. Developed in conjunction with Sandia National Labs, this technology has simplified the detector portion to ensure routine and maintenance free operation.

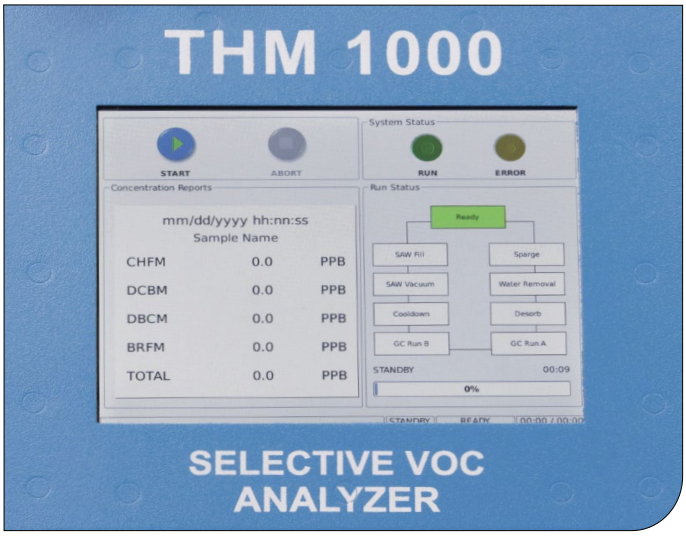
This application note demonstrates calibration, linearity, reproducibility, and method detection limit results using the THM 1000 with Nitrogen (N_2) purge and carrier gas instead of Helium (He).

The THM 1000 can help operators optimize water treatment at the plant and evaluate water age in the distribution system for improved monitoring of the formation of THMs, an EPA-regulated disinfection by-product.

Instrumentation

The instrument used for analysis was the THM 1000 Selectable VOC Benchtop Analyzer.





Methodology

EPA Method 501 was used as guidance for the THM 1000 analysis. All QC and samples were analyzed using a 40 ml sample volume for purging. A system monitoring compound, difluorobenzene, at 20 ppb was added to all samples to monitor purging efficiency. A calibration was run using 5, 10, 20, 40, 60, 80, and 100 ppb THM standards. External standard calibration using peak height and linear regression was performed. A limit for correlation coefficient of >/= 0.995 was used for calibration. Initial demonstration of capability (IDC) was performed by analyzing six replicates at 40 ppb.

The method detection limit (MDL) study was performed by analyzing nine replicates at 2 ppb over a three-day period. Please see Table 1 for THM 1000 instrument parameters.

Table 1. THM 1000 Method Parameters

Parameter	Setting
Trap sorbent	#Tenax®
Purge gas	Zero grade nitrogen
Safety vent 1	25 seconds
Sparge/purge time	600 seconds
Water removal time	150 seconds
Safety vent 2	5 seconds
Desorb time	100 seconds
Bake time	655 seconds
Trap temperature	Ambient during purge 35 ˚C during water removal 240 ˚C during desorb 150 ˚C during bake
GC column	Agilent LTM DB-624 30 meter, 0.32 mm ID, 1.8 µm film
GC column carrier gas	Zero grade nitrogen
GC column flow rate	1 mL/min
GC oven program	Oven before desorb 30 ˚C Hold at 30 ˚C for 200 seconds 160 ˚C/minute to 100 ˚C 40 ˚C/minute 200 ˚C hold 180 seconds 160 ˚C/minute 75 ˚C hold 150 seconds 160 ˚C/minute to 30 ˚C hold 250 seconds

Results

Calibration criteria, precision and accuracy of IDCs, and MDL criteria were easily met on the THM 1000.

Table 2. THM 1000 QC

Compound	Calibration Correlation	MDL Spike (ppb)	MDL % Recovery	MDL (ppb)	IDC Spike (ppb)	IDC % Recovery	IDC % RSD
Chloroform	0.998	2	82.2	0.33	40	100	2.48
Dichlorobromomethane	0.997	2	91.1	0.32	40	99.8	3.66
Dibromochloromethane	0.996	2	96.1	0.28	40	101	4.61
Bromoform	0.997	2	78.3	0.29	40	99.9	4.34

Figure 1. 50 ppb THM from THM 1000

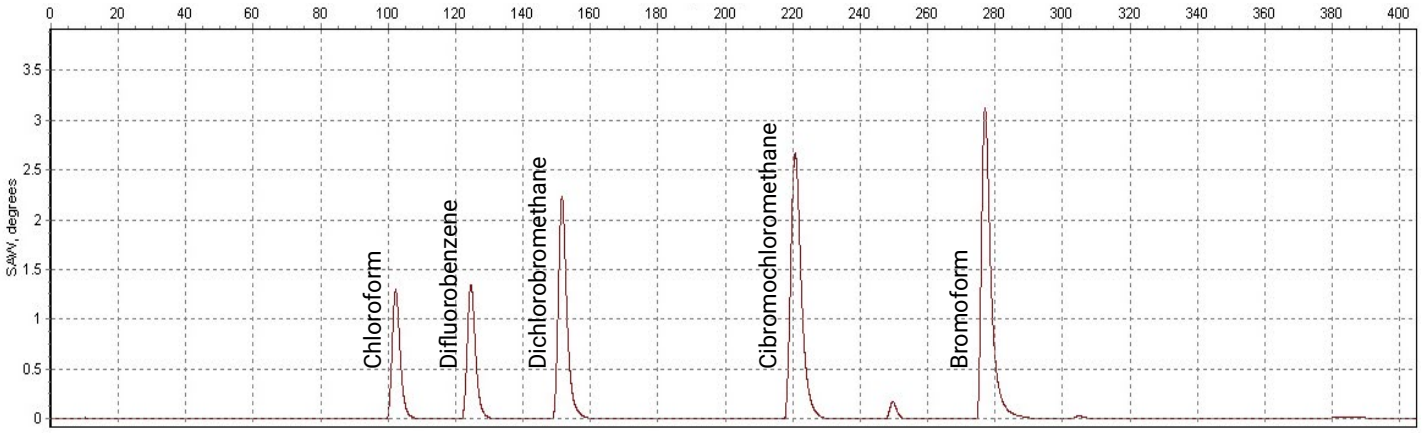
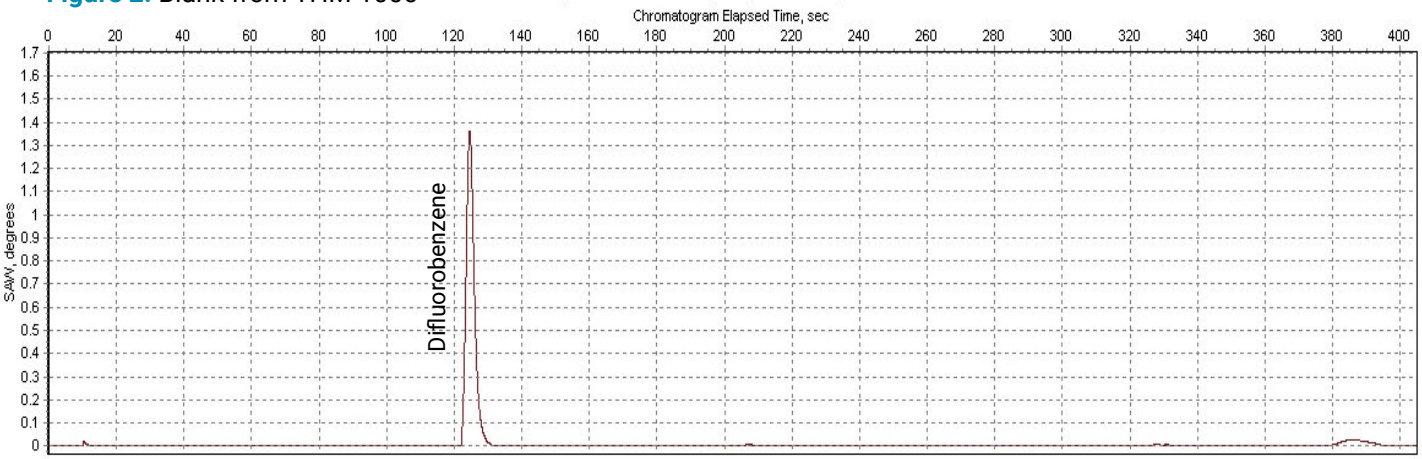


Figure 2. Blank from THM 1000



Conclusions

Reliable and timely THM data is essential for water treatment process optimization. The analysis using the THM 1000 offers a fast, accurate alternative to GC/MS for in-house monitoring of THMs in drinking water plants and distribution systems. The instrument can save facilities time and money as well as offer flexibility in monitoring the treatment process. Using N_2 instead of He saves the lab money without sacrificing quality.

References

EPA Method 501.3. Measurement of Trihalomethanes in Drinking Water with Gas Chromatography/Mass Spectrometry and Selected Ion Monitoring. U.S. Environmental Protection Agency, Office of Ground Water and Drinking Water: Cincinnati, Ohio, 1996.

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