An integrated GC-MS workflow solution for the determination of (semi)volatiles in drinking water and solid waste according to the U.S. EPA guidelines

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INTRODUCTION

Worldwide there are many regulatory organizations safeguarding the environment by the issuance of health and environmental standards, such as the United States (U.S.) Environmental Protection Agency (EPA) and the European Committee for Standardization (CEN). These standards include many standardized guidelines for the analysis of hazardous environmental contaminants.

The challenge for today's laboratories is to correctly execute these guidelines, such as those described in the EPA methods, including calculating and assessing the final results. In many labs the chromatographic results generated while executing these methods are transferred to spreadsheets in order to perform the calculations and assess the final results. This process can be time-consuming and transcription errors are a common problem. As a result laboratories may generate results that are hard to compare, are inconsistent, and can even be misleading. In order to establish a system for compliance with the regulatory methodology, environmental laboratories develop internal guidelines, such as standard operating procedures (SOPs), for sequence creation, sample analysis, and result calculation.

Mass spectrometry (MS) is becoming a more common chromatography detection technique in environmental laboratories and is also used in a wide variation of environmental methods. However, MS instruments often use specific software, without the flexibility of a chromatography data system (CDS) which forces users to learn another software package.

This poster will show an integrated GC-MS workflow solution for the determination of volatiles and semi-volatiles in drinking water and solid waste following the U.S. EPA guidelines using one CDS, including all required calculations and assessment of the final result. This solution helps ensure that analysts follow SOPs consistently and accurately, resulting in high quality and reliable results, saving valuable time and ensuring data integrity.

MATERIALS AND METHODS

All experiments were performed with the Thermo Scientific™ TRACE 1300 Gas Chromatograph in combination with the Thermo Scientific™ ISQ™ Series Single Quadrupole GC-MS system. The minimum software version for instrument control, MS data processing, and calculation and reporting of the results with the environmental GC-MS extension pack (Table 1) was Thermo Scientific™ Chromeleon™ CDS version 7.2 Service Release 5.

U.S. EPA Method	Scope
524.2, 524.3, 524.4	Volatile organics in Drinking Water
525.1, 525.2, 525.3	Semi-volatile organics in Drinking Water
8260B, 8260C	Volatile organics in Solid Waste
8270C, 8270D	Semi-volatile organics in Solid Waste

Table 1. Overview of methods supported by the environmental GC-MS extension pack

The examples shown are based on U.S. EPA method 8270D[1], for the determination of the concentration of semi-volatile organic compounds in extracts prepared from many types of solid waste matrices, soils, air sampling media and water samples.

Workflow Automation

Even though all steps to execute the U.S. EPA methods are outlined down to the smallest detail, in a typical lab they still have to be executed manually by the analysts, thereby introducing the risk of human-generated errors. Chromeleon CDS solves these problems using built-in eWorkflows™, a set of rules capturing all the unique aspects of a chromatography workflow, guiding the users through a minimum number of choices needed to create a complete, correct sequence with predefined files and a well-defined structure.

The environmental GC-MS extension pack within Chromeleon CDS provides predefined eWorkflows to execute analysis as defined in U.S. EPA method (Figure 1A). With the available method and report templates, these eWorkflows can be configured to suit any of the U.S. EPA method mentioned in Table 1 and to your laboratories requirements (Figure 1B).

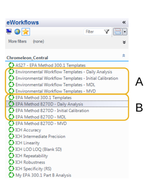

Figure 1. eWorkflows for environmental GC-MS analysis

Figure 2 shows the preconfigured sequence layout of the daily analysis (A), including additional custom columns for identification of the analysis type and spike groups, and how this looks for 8 samples (B).

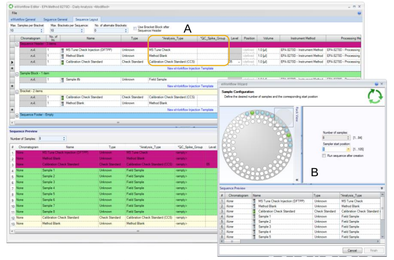

Figure 2. Sequence layout configuration for the daily analysis

RESULTS

DFTPP Tune Verification

MS instruments are tuned to assure accuracy of masses and maximized intensity. U.S. EPA method 8270 prescribes verification of the MS tune using DFTPP according to the criteria as described in Figure 3. Figure 4 shows a typical mass spectrum of DFTPP.

TABLE 3
DFTPP KEY IONS AND ION ABUNDANCE CRITERIA[A5]

Mass	Ion Abundance Criteria
51	10-80% of Base Peak
68	< 2% of mass 69
70	< 2% of mass 69
127	10-80% of Base Peak
197	< 2% of mass 198
198	Base peak, or > 50% of Mass 442
199	5-9% of mass 198
275	10-60% of Base Peak
365	> 1% of mass 198
441	present but < 24% of mass 442
442	Base Peak, or > 50% of mass 198
443	15-24% of mass 442

[A] The majority of the data are taken from Reference 13 (Method 525.2).

[B] The criteria in this table are intended to be used as default criteria for quadrupole instrumentation if optimized manufacturer's operating conditions are not available. Alternate tuning criteria may be employed (e.g., CLP or Method 625), provided that method performance is not adversely affected. See Sec. 11.3.1.

Figure 3. Ion abundance criteria as described in U.S. EPA method 8270

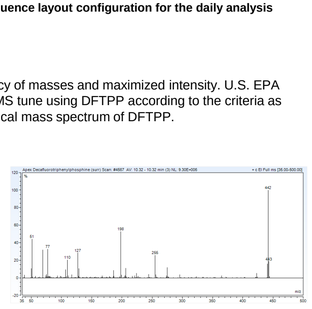

Figure 4. Mass Spectrum of DFTPP in tune injection

To automate the assessment of these criteria, they can be translated to system suitability tests (SST) in the Chromeleon software (Figure 5). This provides a consistent evaluation of the data and automatic reporting of the results (Figure 6), and allows to setup in-run actions if the test criteria are not met.

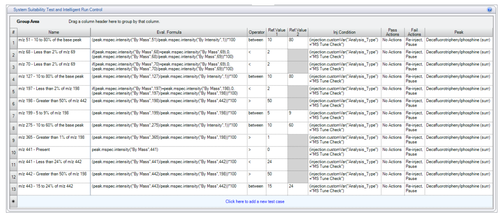

Figure 5. Ion abundance criteria translated to SST

No.	Name	Eval. Result	Operator	Ref. Value 1	Ref. Value 2	Result
1	m/z 51 - 10 to 80% of the base peak	48.6	between	10	80	Passed
2	m/z 68 - Less than 2% of m/z 69	0.5	<	2		Passed
3	m/z 70 - Less than 2% of m/z 69	0.4	<	2		Passed
4	m/z 127 - 10 to 80% of the base peak	31.1	between	10	80	Passed
5	m/z 197 - Less than 2% of m/z 198	0.1	<	2		Passed
6	m/z 198 - Greater than 50% of m/z 442	55.0	>	50		Passed
7	m/z 199 - 5 to 9% of m/z 198	5.7	between	5	9	Passed
8	m/z 275 - 10 to 60% of the base peak	11.9	between	10	60	Passed
9	m/z 365 - Greater than 1% of m/z 198	4.3	>	1		Passed
10	m/z 441 - Present	1433459.1	>	0		Passed
11	m/z 441 - Less than 24% of m/z 442	16.2	<	24		Passed
12	m/z 442 - Greater than 50% of m/z 198	181.9	>	50		Passed
13	m/z 443 - 15 to 24% of m/z 442	18.1	between	15	24	Passed
					Overall Result:	Passed

Figure 6. Reported results of check against ion abundance criteria

Initial Calibration

To calculate the component amount in a field sample, for each component a calibration is required at a minimum of five levels and is based on the average relative response factor (RRF) or an alternative curve fit if the acceptance criteria for the average RRF are not met. In addition the average RRF must not be below the minimum response factor as specified in the method. This represents a lot of requirements just for the calibration, with an additional need to enter method specific criteria.

The processing method is set up with additional columns to enter the required criteria (Figure 7), such as the minimum response factor or the maximum relative percent difference (RPD). A selection of sheets in the report template provides all calculations and in addition checks the results against the criteria as entered in the processing method.

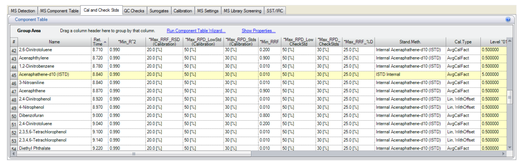

Figure 7. Processing Method setup to enter calibration and check standard parameters and acceptance criteria

Figure 8 shows two examples of calibration reports, one for the calibration results and check these results for minimum relative response factor or coefficient of determination (A), the other one checking the relative response factors in all calibration injections against the minimum required response factors.

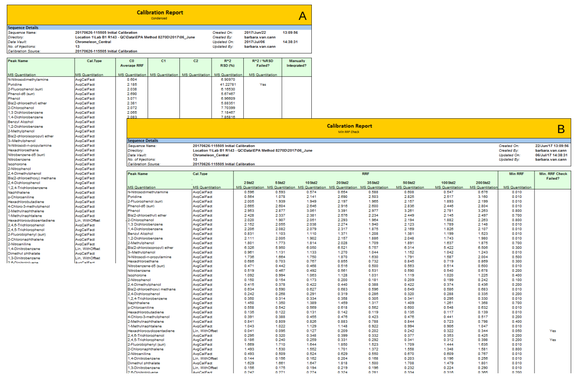

Figure 8. Reported results including checks for (A) minimum RRF/r^2 and (B) for minimum ion abundance

Daily Analysis

After the initial calibration the daily analysis is a sequence with a fixed structure. Due to the automation delivered via the eWorkflows the correct sequence structure and number of method blanks and calibration check standards are automatically added, depending on the number of samples analyzed.

The report template provides reports for each aspect required in the daily analysis. This results in instant report creation without the need to transfer data to an external spreadsheet, thus eliminating the risk of error due to human data transcription.

Beside the MS Tune Check and calibration reports, the environmental template provides reports for all daily analysis experiments; Sequence overview, breakdown, blanks, check standards, quantitation, tentatively identified peaks, internal standard summaries, surrogate recovery, duplicates and so on. Figure 9 shows some examples of the reports; A is the general quantitation report, B the internal standard summary.

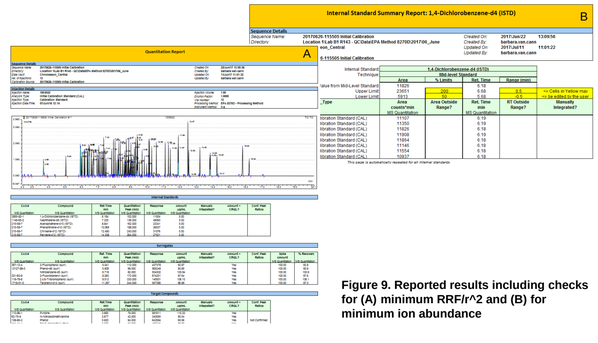

Figure 9. Reported results including checks for (A) minimum RRF/r^2 and (B) for minimum ion abundance

Automated report creation

The data can be automatically reported in order to create the final environmental report of the entire analysis. Chromeleon CDS also supports the creation of an electronic version of the report using the Adobe portable document file format (.pdf) and can be directly integrated with a laboratory information management system (LIMS), making it very easy to distribute the report to all stakeholders.

CONCLUSIONS

Chromeleon CDS offers a fast and simple way to run analyses and create environmental reports according to the extensive requirements as described in the U.S. EPA methods. Benefits include:

- Increased reliability of results – eWorkflows ensure that SOPs are followed and transcription errors and errors caused by manual calculations are eliminated.
- Increased laboratory productivity – users can spend more time running experiments and less time learning different software packages and performing manual data transcription and calculations.
- Faster creation of environmental reports – the flexible reporting tool enables the environmental reports to be created through three mouse clicks.

REFERENCES

1. U.S. EPA. July 2014. Method 8270D (SW-846): Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS), Revision 5.

TRADEMARKS/LICENSING

PO72449-EN 0717S