

# GC/MS Analysis of Semi-volatile Organic Compounds in Drinking Water: Productivity Solution for US EPA Method 525.2

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## Overview

### Summary

The United States Environmental Protection Agency (US EPA) Method 525.2 is a general purpose method for the gas chromatograph/mass spectrometer (GC/MS) analysis of organic compounds in source water, finished drinking water, or drinking water at any stage of treatment.<sup>1</sup> This method presents a number of analytical challenges, including target compounds of varying polarity and with wide ranges of stability. To address these challenges and to streamline method development and validation processes, a Productivity Solution was developed for US EPA Method 525.2 using the DSQ™ II single quadrupole mass spectrometer, along with a TRACE GC Ultra™ and an AS 3000 liquid autosampler. This Productivity Solution (Thermo Scientific PN 120296-KIT) incorporates the appropriate hardware, software, methodologies and consumables required to meet the demanding quality control (QC) criteria laid out by the method. The solution provides optimal performance of Method 525.2 by balancing sample throughput, robustness, and ease of use. Rigorous method development and validation processes ensure that the Productivity Solution for US EPA Method 525.2 for the DSQ II GC/MS system satisfies the demanding requirements of this method.

### Methods

An instrument method was developed for the DSQ II GC/MS system. A surged splitless injection with an injection volume of 1 µL allowed for sample introduction, and the oven was programmed to ensure that the chromatography met the method requirements for resolution of critical compound pairs. All compounds eluted in less than 25 minutes. The DSQ II mass spectrometer acquired EI full scan data, with a mass range of  $m/z$  45 to 450. Maintenance included daily replacement of the inlet septum but no other routine maintenance was required due to the relative cleanliness of the samples. For sample identification and quantification, processing methods and EnviroLab™ Forms 2.0 methods were also developed and integrated into the Productivity Solution. During the method development phase of the project, the robustness of the hardware was evaluated by running the application for several months. Finally, consumables and standards were carefully evaluated and selected based upon their performance for the method.

### Results

By combining the Productivity Solution for US EPA Method 525.2 with the DSQ II GC/MS system, detection limits were achieved that far surpass those described in the method, even for challenging compounds. Across a calibration range from 0.1 to 10.0 ng/µL, the average % RSD across the entire target list was 9.2% and all compounds met average response factor or linear fit calibration criteria. Instrument detection limits ranged from 0.014 µg/L for stirofos to 0.49 µg/L for pentachlorophenol, with a GC run time of approximately 30 minutes. EnviroLab Forms 2.0 software provided data acquisition, processing and reporting capabilities that streamlined data review, ensured QC criteria were met and allowed for reporting of all study results. The 525.2 Productivity Solution simplified method development and validation by providing pre-developed templates for Initial Calibration, Method Detection Limit determinations, and daily batches. A Standard Operating Procedure and How To Manual with a Quick Start Guide facilitated installation of the application.<sup>2,3,4</sup>

### Introduction

US EPA Method 525.2 "...is a general purpose method for the analysis of organic compounds in finished drinking water, source water, or drinking water in any stage of treatment."<sup>1</sup> The method requires a one liter sample volume, which is prepared for GC/MS analysis, typically through solid phase extraction, with a final extract in ethyl acetate. To avoid any adverse effects caused by calibration in a different solvent from that of the extracts, calibration standards were prepared in ethyl acetate. For the 525.2 Productivity Solution, the Initial Calibration curve was established using the following concentrations: 0.1, 0.5, 1.0, 2.0, 5.0 and 10 ng/µL. Because the Productivity Solution includes six standard solutions for this calibration curve, the standard dilution/preparation step is eliminated, simplifying calibration for the method. Replicates for instrument detection limits (IDLs) were performed at 0.5 ng/µL and the method validation study utilized 5 ng/µL samples. These standards were included in the productivity solution for ease of use.

## Results

In order to effectively illustrate the performance of the DSQ II and the Productivity Solution, the results will be discussed following the normal workflow in the laboratory, with an emphasis on the 525.2 method-specific criteria and the performance of the solution relative to those requirements.

- Evaluation of GC/MS performance criteria
- Meeting the calibration curve requirements
- Initial demonstration of laboratory capability
- Determination of MDLs or Instrument Detection Limits (IDLs)
- Meeting the Continuing Calibration Check criteria
- Identification of analytes
- Identification of multi-component analytes

## Evaluation of GC/MS Performance Criteria

Typically, the first injection of a 12-hour shift tests mass spectrometer tuning as well as overall system performance. The GC/MS Performance Check Solution, which contains 5 ng/μL of DFTPP, endrin, and 4,4'-DDT in iso-octane, is used to assess both tune and system performance in a single injection. The mass spec must meet the tuning criteria of the method for DFTPP as listed in Table 1 of US EPA Method 525.2.<sup>1</sup> System performance is checked by evaluating the breakdown products for endrin and 4,4'-DDT. Breakdown for these components is calculated by summing up the TIC area counts for the breakdown products and dividing by the total areas of both the primary pesticide and its breakdown products. This total breakdown percentage must be < 20% for endrin and < 20% for 4,4'-DDT. The breakdown products for endrin are endrin aldehyde and endrin ketone. The

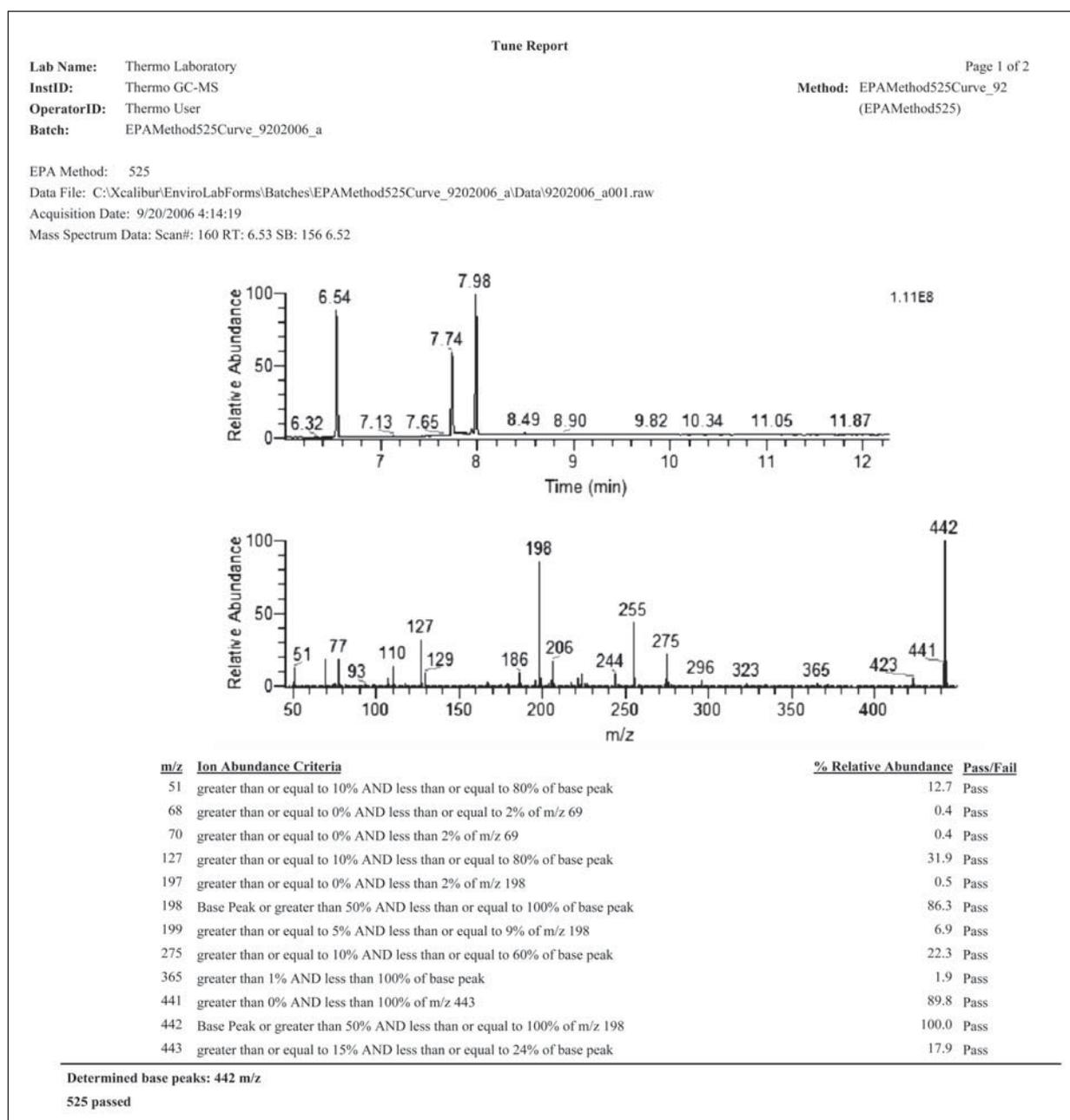


Figure 1: EnviroLab Forms report for DFTPP tune check, showing TIC of performance solution, DFTPP spectrum, and tune check results

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breakdown products for 4,4'-DDT are 4,4'-DDE and 4,4'-DDD. EnviroLab Forms software automatically generates reports that include the results for both the tune check and the breakdown results, and this report is shown in Figure 1. Using Autotune tune settings for the DSQ II, the tuning criteria for DFTPP were easily met. Breakdown was minimized by using the deactivated liners included in the Productivity Solution, along with the analytical column, both of which minimized breakdown within the system. Total endrin breakdown was 1.2%, and 0.2% breakdown was found for 4,4'-DDT.

In addition to these criteria for tune and breakdown, Method 525.2 also specifies chromatographic performance requirements that need to be achieved during method development. Required baseline separation of anthracene and phenanthrene and a valley of < 25% of the averaged peak height for benzo(a)anthracene and chrysene are mandated and must be achieved through a combination of column selection and oven temperature ramping. The DSQ II GC/MS system easily met these resolution requirements, and in fact, baseline separation was achieved on both compound pairs (Figures 2 and 3).

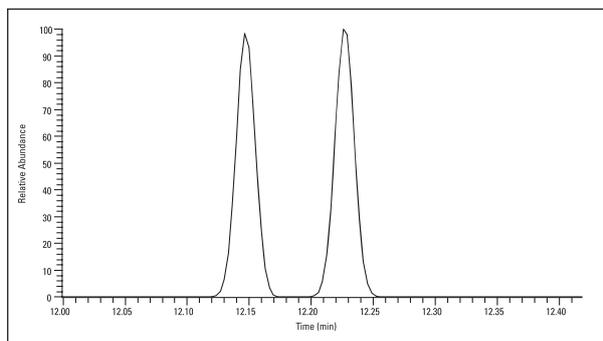


Figure 2: Baseline separation of anthracene and phenanthrene

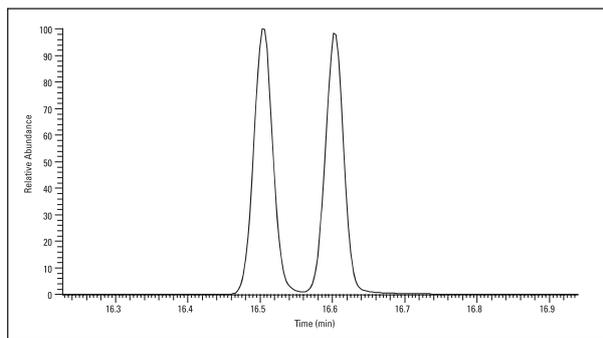


Figure 3: Separation of benzo(a)anthracene and chrysene

### Meeting the Calibration Curve Requirements

A six-point initial calibration curve was established using the following levels: 0.1, 0.5, 1, 2, 5, and 10 ng/ $\mu$ L. Internal standards and surrogates were added at 5 ng/ $\mu$ L. The Productivity Solution provided an electronic version of a batch template for initial calibration designed for use with EnviroLab Forms: *EPAMethod525Curve.utm*. Method 525.2 specifies that for initial calibration, the average percent relative standard deviation (% RSD) must be < 30% for all target compounds. If the average % RSD requirement is not met, a linear least squares fit may be

applied at  $r^2 > 0.99$ . For the DSQ II and the 525.2 Productivity Solution, the initial calibration results are listed in Table 1. The average %RSD across the entire target list was 9.2% RSD, far exceeding the minimum 30% criteria.

Because the list of target analytes in Method 525.2 is comprised of compounds with highly varied polarities – from non-polar polynuclear aromatics (PNAs) to very polar organophosphorus pesticides – adjustments from standard conditions were made to optimize separation and performance. A surged pressure injection was selected to minimize the time that the more thermally labile pesticides spent in the injection port liner. The stationary phase for the analytical GC column was a low polarity proprietary phase, a 30 m x 0.25 mm i.d. x 0.25  $\mu$ m film thickness TRACE™ TR-525 (Thermo Scientific PN 26RX142P), which yielded excellent resolution and sensitivity for the Aroclors, PNAs, and pesticides. This column was selected for its superior performance, as found during the evaluation of different stationary phases. Other columns from various vendors were evaluated, and each demonstrated a level of irreversible adsorption of some of the analytes, resulting in loss of detection below 5 ng and poor linear fits. The TRACE TR-525 analytical column, by comparison, showed minimal adsorption and yielded the best analytical results.

### Initial Demonstration of Laboratory Capability

In the environmental laboratory, the precision and accuracy of sample preparation and analysis are typically validated by running seven replicates of a laboratory fortified blank containing target compound at 5  $\mu$ g/L, a mid-level concentration for the calibration range. The 525.2 Productivity Solution contains five 1 mL ampoules of Method Validation (MVD) solutions at 5 ng/ $\mu$ L, which would be equivalent to 1 liter of water spiked to contain 5  $\mu$ g/L, extracted and concentrated to 1 mL. These method validation solutions were acquired and processed using the EnviroLab Forms batch template *EPAMethod525MVD.utm*. Method 525.2 requires that the relative standard deviation (RSD) for each compound must be < 30% and the percent recoveries must be > 70 and < 130%. The results of the method validation study are shown in Table 1. All targets met the method criteria, with an average of 1.9% RSD for all targets analyzed. A total ion chromatogram (TIC) of the 5 ng/ $\mu$ L method validation sample is shown in Figure 4.

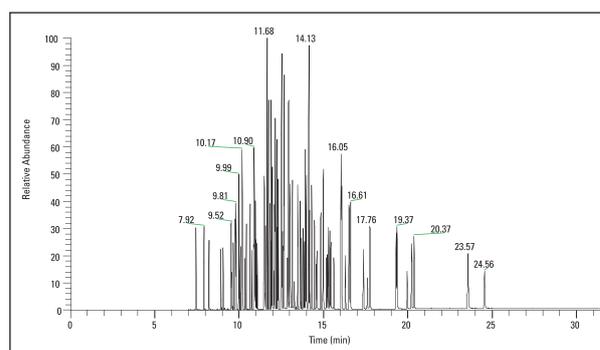


Figure 4: Method Validation at 5 ng/ $\mu$ L

## Determination of MDLs

As with the method validation study, the method detection limit study is typically performed by running seven replicates of a laboratory fortified blank containing target compound at 0.5 µg/L. For this study, an approximation of the Method Detection Limit (MDL) was made by performing seven replicates at a concentration of 0.5 ng/µL, using the Instrument Detection Limit (IDL) standard solution. The Productivity Solution contains five 1-mL ampoules of MDL standard solution, and these standards are equivalent to extraction of 1 liter of spiked organic-free water concentrated to 1 mL. Because this solution is not taken through sample preparation procedures, the study is an IDL rather than an MDL. The IDL studies are run using the EnviroLab Forms batch template: *EPAMethod525MDL.utm*. Based on this study, the average IDL was 0.04 µg/L. The complete IDL results are shown in Table 1.

## Meeting the Continuing Calibration Check Criteria

A continuing calibration check standard is used to verify the MS tune and initial calibration at the beginning of each 12 hour shift. Method 525.2 requires that the absolute areas of the quantitation ions for both the internal standards and the surrogates must be within ± 50% from the most recent curve. The Response Factors (RF) must be within ± 30% of the mean values measured in the most recent calibration curve. Alternately if a linear

regression is applied, the calculated amount for each analyte must be ± 30% of the true value. The 525.2 Productivity Solution contains five 1-mL ampoules of this continuing calibration check solution, at a concentration of 5 ng/µL. For the continuing calibration check performed as part of the study, the average percent difference for all targets was 8% difference. These results are shown in Table 2.

## Identification of Analytes

Target analytes are identified by both retention time and a mass spectrum as compared to user library reference spectra generated from the standard. To ensure accurate identification and quantification, an Xcalibur processing method is created that contains a single quantitation ion, and the qualifier ions as listed in Table 2 of the US EPA Method 525.2 were added to facilitate ion ratio confirmation.<sup>1</sup> Retention time windows in the processing method provided accurate identification, and a co-elution retention time window for the qualifier ions enabled further qualification of identifying features. The target list with retention times, quantitation, and qualifier ions are listed in Appendix 1 of the *Productivity Solution: SOP Based on EPA Method 525.2* (Thermo Scientific PN 120295-001).<sup>2</sup> Selected instrument parameters for the DSQ II, TRACE GC Ultra and AS 3000 are included in Table 3.

<b>DSQ II:</b> Source Temperature: 250 °C Filament Delay: 7.0 min Chrom Filter Width: 2.5 sec Detector gain: 0.8 (1192 V) Emission current: 25 µA Full Scan: 45-450 <i>m/z</i> Scan Rate: 5 scans/sec Electron Energy: -70 eV	<b>TRACE GC Ultra:</b> <b>For DFTPP:</b> Oven Initial Temp: 40 °C Initial Time: 0.5 min Ramp 1: 40 °C/min Temp 1: 150 °C Time 1: 0 min Ramp 2: 13 °C/min Final Temp 310 °C Final Time: 15 min SSL Injection Port Temp: 250 °C Surge Pressure: 120 kPa Surge Duration: 0.5 min Splitless Time: 0.5 min Split flow: 50 mL/min Carrier Flow (He): 1 mL/min Flow Mode: Constant Flow Vacuum compensation: On Transfer Line Temperature: 310 °C	<b>For Target Analysis:</b> Oven Initial temp: 40 °C Initial Hold Time: 2.0 min Ramp 1: 18 °C/min Temp 1: 270 °C Time 1: 0.0 min Ramp 2: 5 °C/min Final Temp: 310 °C Final Hold Time: 5 min SSL Injector Temperature: 250 °C Surge Pressure: 120 kPa Surge Duration: 0.5 min Splitless Time: 0.5 min Split Flow: 50 mL/min Carrier Flow (He): 1 mL/min Flow Mode: Constant Flow Vacuum compensation: On Transfer Line Temperature: 310 °C
<b>AS3000 (5 µL):</b> Sample volume: 1 µL Plunger strokes: 10 Viscous sample: YES Sampling depth in Vial: Bottom Injection depth: Standard Pre-inject dwell time: 0 seconds Post-inject time: 0 seconds Post-Inject Wash Solvent: B Rinse Cycles: 10		

Table 3: Selected instrument parameters for the Method 525.2 Productivity Solution. The use of two different GC methods for performance mix evaluation and target compound analysis streamlines workflows.

Compound	Curve (0.1,0.5,1,2.5,10 ng/ $\mu$ L)		IDLs*		MVD (5 ng/ $\mu$ L)	
	% RSD	% RSD	$\mu$ g/L*	% DIF	% RSD	% RSD
isophorone	7	3	0.038	10	2	2
1,3-dimethyl-2-nitrobenzene	2	1	0.163	0	1	1
dichlorovos	10	5	0.069	11	2	2
hexachlorocyclopentadiene	7	2	0.021	7	2	2
EPTC	7	2	0.031	4	1	1
butylate	6	3	0.041	10	2	2
mevinphos	12	3	0.034	10	2	2
vernolate	5	2	0.033	3	1	1
pebulate	7	2	0.025	5	1	1
etridiazole	10	3	0.048	9	2	2
dimethyl phthalate	3	2	0.027	3	1	1
acenaphthylene	3	2	0.033	5	1	1
fluorene	10	5	0.071	6	3	3
2,6-dinitrotoluene	9	3	0.034	10	2	2
acenaphthene-d10(IS)	4	5			3	3
2-chlorobiphenyl	2	1	0.016	2	2	2
chloroneb	2	3	0.041	3	2	2
tebuthiuron	17	2	0.03	15	1	1
molinate	5	2	0.029	4	1	1
diethyl phthalate	4	2	0.029	9	1	1
mevinphos	12	3	0.034	10	2	2
vernolate	5	2	0.033	3	1	1
pebulate	7	2	0.025	5	1	1
etridiazole	10	3	0.048	9	2	2
dimethyl phthalate	3	2	0.027	3	1	1
acenaphthylene	3	2	0.033	5	1	1
fluorene	10	5	0.071	6	3	3
2,6-dinitrotoluene	9	3	0.034	10	2	2
acenaphthene-d10(IS)	4	5			3	3
2-chlorobiphenyl	2	1	0.016	2	2	2
chloroneb	2	3	0.041	3	2	2
tebuthiuron	17	2	0.03	15	1	1
molinate	5	2	0.029	4	1	1
diethyl phthalate	4	2	0.029	9	1	1
2,4-dinitrotoluene	14	2	0.029	14	1	1
propachlor	5	3	0.038	6	1	1
ethoprop	14	3	0.035	13	2	2
cycloate	5	2	0.029	8	2	2
trifluralin	10	3	0.047	14	2	2
chlorpropham	4	3	0.041	6	2	2
2,3-dichlorobiphenyl	2	1	0.022	1	2	2
atraton	10	2	0.024	11	2	2
prometon	11	4	0.054	9	1	1
alpha-BHC	4	3	0.04	6	1	1
hexachlorobenzene	3	3	0.042	0	3	3
propazine	3	3	0.04	3	2	2
simazine	10	3	0.045	6	2	2
diazinon	4	3	0.045	5	2	2
terbufos	9	3	0.037	11	3	3
pronamide	10	2	0.029	7	2	2
pentachlorophenol	25	13	0.493	16	2	2
disulfoton	8	2	0.027	6	1	1
beta-BHC	3	4	0.056	4	2	2
terbacil	20	3	0.033	15	1	1
phenanthrene-d10(IS)	3	4			3	3
methyl paraoxon	17	2	0.02	22	2	2
phenanthrene	4	2	0.031	-2	1	1
anthracene	3	1	0.014	0	1	1
gamma-BHC	4	4	0.052	4	2	2
2,4,5-trichlorobiphenyl	2	2	0.038	0	2	2
alachlor	10	2	0.024	7	1	1
prometryne	9	3	0.042	6	2	2
atrazine	5	4	0.05	6	3	3
metribuzin	7	1	0.02	6	2	2
delta-BHC	2	4	0.058	1	2	2

Table 1: Results of Calibration Curve, IDLs, and MVDs

Curve (0.1,0.5,1,2,5,10 ng/μL)		IDLs*		MVD (5 ng/μL)	
Compound	% RSD	% RSD	μg/L*	% DIF	% RSD
simetryn	8	2	0.029	4	1
chlorothalonil	6	2	0.036	3	2
heptachlor	6	2	0.025	4	1
di-n-butylphthalate	5	1	0.015	5	2
terbutryn	11	5	0.07	2	1
bromacil	17	2	0.029	14	1
chlorpyrifos	4	3	0.042	2	3
metolachlor	8	1	0.013	6	2
DCPA	5	3	0.047	-3	1
2,2',4,4'-tetrachlorobiphenyl	5	4	0.057	-2	2
aldrin	5	3	0.05	-1	2
triadimefon	10	4	0.058	7	2
cyanazine	10	3	0.042	9	2
MGK-264	14	2	0.023	15	1
diphenamid	7	2	0.035	5	1
merphos	24	14	0.132	33	7
ametryn	14	4	0.071	-1	2
2,2',3,4,6-pentachlorobiphenyl	4	9	0.131	14	5
heptachlor epoxide	4	4	0.054	6	2
butachlor	14	2	0.023	13	1
stirofos	18	1	0.014	18	2
fenamiphos	24	3	0.032	21	2
alpha-chlordane	1	4	0.063	2	2
gamma-chlordane	6	4	0.056	3	3
napropamide	13	3	0.035	8	1
trans-nonachlor	4	4	0.068	1	2
endosulfan I	4	6	0.088	5	1
4,4'-DDE	5	3	0.045	-3	3
pyrene	5	2	0.038	-5	2
2,2',4,4',5,6-hexachlorobiphenyl	5	4	0.06	-2	2
carboxin	17	2	0.027	14	1
dieldrin	4	2	0.029	5	2
chlorobenzilate	14	3	0.04	9	2
tricyclazole	26	6	0.061	26	2
endrin	7	5	0.074	7	2
4,4'-DDD	8	2	0.028	8	2
bis(2-ethylhexyl)adipate	16	2	0.028	14	2
butyl benzyl phthalate	13	3	0.04	12	2
endosulfan II	5	8	0.119	6	1
endrin aldehyde	11	5	0.071	7	3
norflurazon	19	2	0.028	17	2
4,4'-DDT	13	4	0.054	12	3
triphenylphosphate(surr)	4	3	0.507	1	3
hexazinone	13	3	0.042	8	2
endosulfan sulfate	8	3	0.045	7	2
bis(2-ethylhexyl)phthalate	15	3	0.038	13	2
methoxychlor	10	2	0.03	10	2
2,2',3,3',4,5,6,6'-octachlorobiphenyl	5	4	0.069	-3	2
2,2',3,3',4,4',6'-heptachlorobiphenyl	8	5	0.09	-4	2
endrin ketone	3	3	0.044	2	2
benzo(a)anthracene	3	2	0.027	2	1
chrysene-d12(I)	6	6			4
chrysene	3	2	0.026	4	1
fenarimol	14	2	0.028	15	2
cis-permethrin	16	3	0.033	19	2
trans-permethrin	17	4	0.048	15	2
beno(b)fluoranthene	9	3	0.035	9	2
benzo(k)fluoranthene	12	3	0.045	9	2
fluridone	0.9989	5	0.049	31	2
benzo(a)pyrene	14	2	0.027	14	2
perylene-d12(surr)	4	3	0.412	3	1
dibenzo(a,h)anthracene	27	5	0.055	24	4
indeno(1,2,3-cd)pyrene	22	5	0.053	21	3
benzo(g,h,i)perylene	18	4	0.047	16	3

\*IDL: Instrument Detection Limit, replicates at 0.5 ng/μL if one liter water extracted and concentrated to 1 mL = 0.5 μg/L

Table 1 continued: Results of Calibration Curve, IDLs, and MVDs

**CONTINUED CALIBRATION CHECK**

Compound	RF % DIF	Pass/Fail	Compound	RF % DIF	Pass/Fail
1,3-dimethyl-2-nitrobenzene	2	Pass	DCPA	0.5	Pass
dichlorovos	12	Pass	2,2',4,4'-tetrachlorobiphenyl	0.6	Pass
hexachlorocyclopentadiene	16	Pass	aldrin	2	Pass
EPTC	7	Pass	triadimefon	9	Pass
butylate	11	Pass	cyanazine	14	Pass
mevinphos	11	Pass	MGK-264	15	Pass
vernolate	5	Pass	diphenamid	5	Pass
pebulate	6	Pass	merphos	28	Pass
etridiazole	11	Pass	ametryn	15	Pass
dimethyl phthalate	3	Pass	2,2',3,4,6-pentachlorobiphenyl	0.9	Pass
acenaphthylene	6	Pass	heptachlor epoxide	5	Pass
fluorene	2	Pass	butachlor	15	Pass
2,6-dinitrotoluene	9	Pass	stirofos	15	Pass
2-chlorobiphenyl	5	Pass	fenamiphos	22	Pass
chloroneb	4	Pass	alpha-chlordane	3	Pass
tebuthiuron	13	Pass	gamma-chlordane	2	Pass
molinate	3	Pass	napropamide	8	Pass
diethyl phthalate	11	Pass	trans-nonachlor	3	Pass
2,4-dinitrotoluene	15	Pass	endosulfan I	5	Pass
propachlor	5	Pass	4,4'-DDE	3	Pass
ethoprop	12	Pass	pyrene	-3	Pass
cycloate	7	Pass	2,2',4,4',5,6-hexachlorobiphenyl	0.4	Pass
trifluralin	14	Pass	carboxin	12	Pass
chlorpropham	7	Pass	dieldrin	5	Pass
2,3-dichlorobiphenyl	3	Pass	chlorobenzilate	13	Pass
atraton	11	Pass	tricyclazole	21	Pass
prometon	10	Pass	endrin	6	Pass
alpha-BHC	6	Pass	4,4'-DDD	9	Pass
hexachlorobenzene	4	Pass	bis(2-ethylhexyl)adipate	13	Pass
propazine	5	Pass	butyl benzyl phthalate	13	Pass
simazine	8	Pass	endosulfan II	8	Pass
diazinon	5	Pass	endrin aldehyde	10	Pass
terbufos	12	Pass	norflurazon	19	Pass
pronamide	10	Pass	4,4'-DDT	13	Pass
pentachlorophenol	16	Pass	triphenylphosphate(surr)	3	Pass
disulfoton	5	Pass	hexazinone	7	Pass
beta-BHC	6	Pass	endosulfan sulfate	12	Pass
terbacil	14	Pass	bis(2-ethylhexyl)phthalate	17	Pass
methyl paraoxon	19	Pass	methoxychlor	14	Pass
phenanthrene	-3	Pass	2,2',3,3',4,5,6,6'-octachlorobiphenyl	1	Pass
anthracene	2	Pass	2,2',3,3',4,4',6-heptachlorobiphenyl	3	Pass
gamma-BHC	5	Pass	endrin ketone	5	Pass
2,4,5-trichlorobiphenyl	1	Pass	benzo(a)anthracene	0.2	Pass
alachlor	10	Pass	chrysene	1	Pass
prometryne	6	Pass	fenarimol	15	Pass
atrazine	9	Pass	cis-permethrin	20	Pass
delta-BHC	5	Pass	trans-permethrin	16	Pass
simetryn	6	Pass	beno(b)fluoranthene	11	Pass
chlorothalonil	6	Pass	benzo(k)fluoranthene	10	Pass
heptachlor	7	Pass	fluridone	29	Pass
di-n-butylphthalate	5	Pass	benzo(a)pyrene	14	Pass
terbutryn	3	Pass	perylene-d12(surr)	2	Pass
bromacil	15	Pass	dibenzo(a,h)anthracene	26	Pass
chlorpyrifos	1	Pass	indeno(1,2,3-cd)pyrene	21	Pass
metolachlor	7	Pass	benzo(g,h,i)perylene	18	Pass

Table 2: Continued Calibration Check

## Identification of Multi-component Analytes

Identification of multi-component analytes such as toxaphene and the Aroclors is made by recognition of characteristic patterns based on the relative amounts of each component. An individual stock solution has been included for the multi-component analytes. An Xcalibur processing method has been built for each of the following:

- Toxaphene
- Aroclor 1016
- Aroclor 1221
- Aroclor 1232
- Aroclor 1242
- Aroclor 1248
- Aroclor 1254
- Aroclor 1260

Characteristic TICs are shown in Figures 5 for Aroclor 1254 and Figure 6 for toxaphene. Typical calibration curves are shown in Figure 7 for Aroclor 1254 and Figure 8 for toxaphene. Identification and quantification of these multi-component analytes is facilitated by EnviroLab Forms software, which allows for grouping of compounds to streamline this complex analysis.

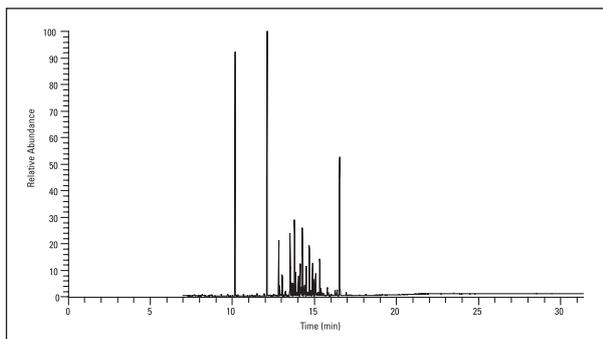


Figure 5: TIC for Aroclor 1254 at 25 ng/μL

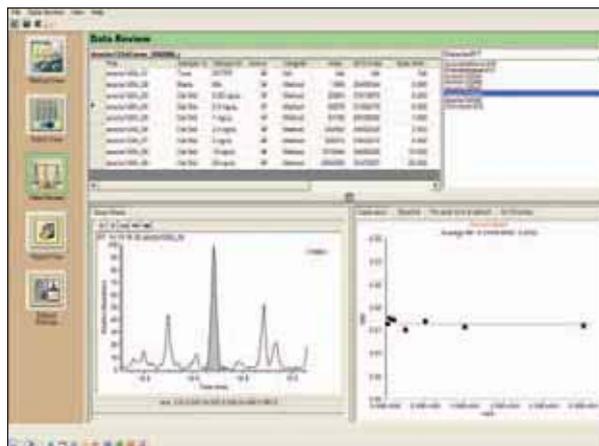


Figure 7: TIC for Aroclor 1254 at 25 ng/μL

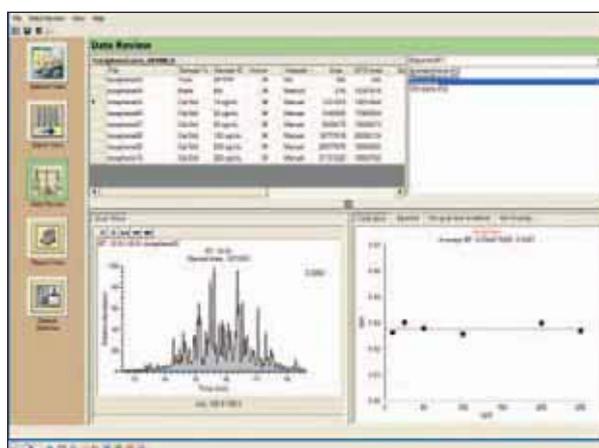


Figure 8: TIC for toxaphene at 250 ng/μL

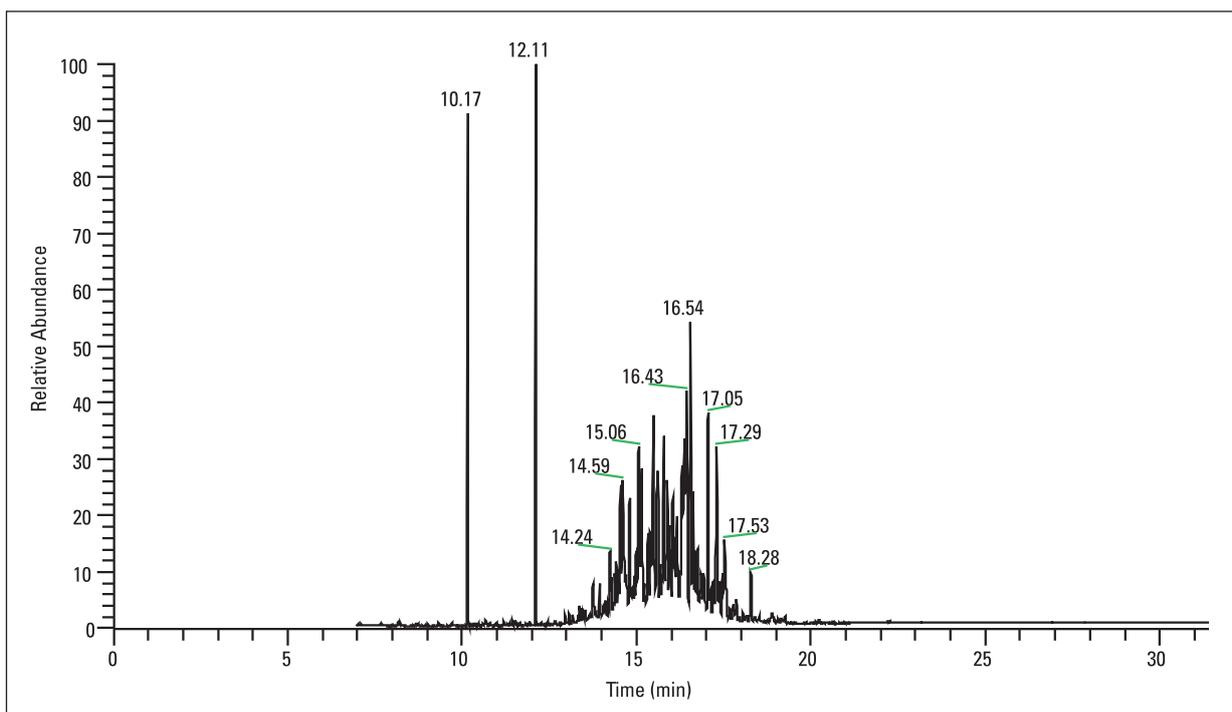


Figure 6: TIC for Toxaphene at 250 ng/μL

## Productivity Solution Contents

The 525.2 Productivity Solution is designed to incorporate the hardware, software, methodologies, and consumables to ensure optimal performance of US EPA Method 525.2 using the DSQ II GC/MS system. In addition to the consumables and standards as described above, the Productivity contains two books: *Standard Operating Procedure* and *How To Manual* with a *Quick Start Guide*, and the following three CDs:

1. EnviroLab Forms 2.0 – application software for method management, batch programming, data review, and report generation

2. Interactive Reference CD – automated downloading of instrument/processing methods for EPA Method 525.2, along with electronic versions of documents listed above
3. Validation Data CD – proven performance with results for calibration curve, instrument detection limits, daily calibration check standard and performance mix

This Productivity Solution includes all of the consumables required for two weeks of operation, excluding sample preparation. The contents of the Productivity Solution are listed in Table 4, while Figure 9 displays the complete set of components included in the US EPA Method 525.2 Productivity Solution.

Description	Quantity	Part Number
<b>EPA Method 525 Productivity Solution</b>		<b>120296-KIT</b>
<i>This kit contains:</i>		
EnviroLab Forms 2.0	1	120265-0001
525 Productivity Reference CD	1	120296-CD
525 Validation Data CD	1	120296-VCD
SOP EPA Method 525	1	120296-0001
How To Guide EPA Method 525	1	120296-0002
Getting Productive Poster	2	120041-ENV
Quick Start Guide EPA Method 525	1	120296-0003
Ion Volume EI Closed DSQ II	1	119650-0221-T
Ion Volume Holder DSQ II	1	70001-20532-T
Graphite ferrule 0.1-0.25	10	29053488
TRACE TR-525 GC Column 30 m x 0.25 mm ID x 0.25 µm	1	26RX142P
Septum	50	31303211
Liner Seal	1	29033406
Splitless Liner 5 mm ID, 105 mm	5	45350033
Silver Seal	10	29033629
Ferrule 0.4 mm ID 1/16 G/V	10	29033496
Blank Vespel Ferrule	10	290VT155
2 mL Amber Silanized Glass Vial	100	60180-567
Blue cap with Ivory PTFE/Red Rubber Seal	100	60180-569
Syringe 5 µL 50 mm – Al/AS3000	2	36500505
Method 525 CAL KIT, 30 Ampoules	1	60181-301
<i>This kit contains:</i>		
Calibration Curve Standard 0.1 µg/mL, C1, 1 x 1.2 mL ampoules	1	60181-302
Calibration Curve Standard 0.5 µg/mL, C2, 1 x 1.2 mL ampoules	1	60181-303
Calibration Curve Standard 1.0 µg/mL, C3, 1 x 1.2 mL ampoules	1	60181-304
Calibration Curve Standard 2.0 µg/mL, C4, 1 x 1.2 mL ampoules	1	60181-305
Calibration Curve Standard 5.0 µg/mL, C5, 1 x 1.2 mL ampoules	1	60181-306
Calibration Curve Standard 10 µg/mL, C6, 1 x 1.2 mL ampoules	1	60181-307
Calibration Curve Standard (0.1, 0.5, 1, 2, 5, 10 µg/mL) C1-C6, 6 x 1.2 mL ampoules	1	60181-308
Calibration Check Standard 2nd Source 5 µg/mL C5, 5 x 1.2 mL ampoules	5	60181-309
IDL Standards 0.5 µg/mL C2, 5 x 1.2 mL Ampoules	5	60181-310
Performance Check Solution 5 µg/mL, 5 x 1.2 mL ampoules	5	60181-311
Internal Standard/surrogates 500 µg/mL, 1 x 1.2 mL ampoule	1	60181-312
Aroclor 1016, 1000 µg/mL, 1 x 1 mL ampoules	1	60181-313
Aroclor 1221, 1000 µg/mL, 1 x 1 mL ampoules	1	60181-314
Aroclor 1232, 1000 µg/mL, 1 x 1 mL ampoules	1	60181-315
Aroclor 1242, 1000 µg/mL, 1 x 1 mL ampoules	1	60181-316
Aroclor 1248, 1000 µg/mL, 1 x 1 mL ampoules	1	60181-317
Aroclor 1254, 1000 µg/mL, 1 x 1 mL ampoules	1	60181-318
Aroclor 1260, 1000 µg/mL, 1 x 1 mL ampoules	1	60181-319
Toxaphene, 5000 µg/mL, 1 x 1.2 mL ampoule	1	60181-320

Table 4: Productivity Solution PN 120296-KIT



Figure 9: Photo of Method 525.2 Productivity Solution

### EnviroLab Forms 2.0

EnviroLab Forms 2.0 software is a layered application that works with Xcalibur to provide a comprehensive tool for method management, batch programming, data review, and report generation. This software is a central component of the 525.2 Productivity Solution. When EnviroLab Forms is used in conjunction with the Interactive Reference CD, the user can be productive immediately by running pre-developed methods and batch templates. These templates and methods are configured for ease of use and optimal performance. For example, one of these templates is used to develop the first sequence for running the initial curve, and the template is linked to the appropriate Xcalibur instrument and processing methods. After programming the batch and placing the sample vials onto the system, the batch is exported to Xcalibur and initiated by selecting *Run*. As each acquisition finishes, the appropriate QC report comes off of the printer. The use and application of other templates is described above. A set of application-specific templates have been created in EnviroLab Forms to facilitate the creation of the batch sequences to be run on the instrument.

- EPAMethod525Curve.utm (calibration curve)
- EPAMethod525MDL.utm (Method Detection Limit Determination)
- EPAMethod525MVD.utm (Method Validation)
- EPAMethod525Daily.utm (Typical Daily Batch with unknowns)

By providing instrument and processing methods, along with the relevant EnviroLab Forms methods and templates, the 525.2 Productivity Solution ensures streamlined integration of the DSQ II GC/MS system into the laboratory.

### Conclusion

Dedicated analyzers ensure high sample throughput in the lab. When the entire GC/MS system has been proven to produce the performance required in the EPA Method, tight QC criteria can easily be met on a day to day basis. The Productivity Solution for US EPA Method 525.2 has been validated in the factory to meet all of the QC criteria listed in the method. Pre-validated consumables make daily operation of the system routine. The DSQ II showed excellent sensitivity and linear range for the analytes studied. EnviroLab Forms reporting software produced all of the required QC reports automatically at the end of each run.

### References:

1. *Method 525.2 Determination of Organic Compounds in Drinking Water by Liquid-Solid Extraction and Capillary Column Gas Chromatography/Mass Spectrometry*, Rev 2.0, National Exposure Research Laboratory Office of Research and Development U. S. Environmental Protection Agency Cincinnati, Ohio 45268
2. *Productivity Solution: SOP Based on EPA Method 525.2 Determining Semi-volatile Organics in Drinking Water Using the DSQ II*, PN 120296-001
3. *Productivity Solution: How To: Based on EPA Method 525.2 Determining Semi-volatile Organics in Drinking Water Using the DSQ II*, PN 120296-001
4. *Quick Start Guide for EPA Method 525 Analyzer PN 120296-003 TRACE GC Ultra, DSQ II, and AS3000*