

Qualitative Screening for Volatile Organic Compounds Collected on Thermal Desorption Tubes

Procedure:	Collect air samples by drawing workplace air containing organic volatile compounds (up to C ₂₈) through stainless steel thermal desorption (TD) tubes using a hand-operated pump. Extract samples with a two-stage thermal desorption system and analyze by gas chromatography mass spectrometry (GC/MS).
Recommended sampling volume:	Collect 100 mL of workplace air for suspected analytes with permissible exposure limits above 5 ppm. Collect up to 500 mL for suspected analytes with permissible exposure limits at or below 5 ppm.
Limits of detection:	0.53 to 65 ng/sample for analytes tested, see Section 4.1.
Uncertainty (<i>u</i>):	Qualitative method, uncertainty not determined.
Special requirements:	Seal tubes with brass storage caps immediately after sampling to prevent diffusive contamination and sample loss. Avoid any physical impact that may damage the tube. Do not write on or apply any adhesive onto the tube. Do not refrigerate samplers before or after sampling. Return TD tubes to the OSHA Technical Center immediately after sampling.
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OSHA Method 1031, Qualitative Screening for Volatile Organic Compounds Collected on Thermal Desorption Tubes

1 Introduction

The methodologies described in this method are designed to help identify unknown volatile organic compounds (up to C₂₈) in workplace air. This method is specific to the sorbent and cold trap described in Sections 2 and 3.

2 Sampling Procedure

Follow all safety practices that apply to the work area being sampled. Use sampling equipment in a manner that will not interfere with work performance or safety. Uncap sampling tubes only when sampling and properly re-cap tubes immediately after sampling.

2.1 Apparatus

- A 1/4-inch O.D. x 3.5-inch stainless steel or inert thermal desorption (TD) tube with a dual-bed sorbent of Tenax TA and Carbograph 5TD^a (i.e., Markes International C3-AXXX-5304 or C3-CXXX-5304 or equivalent).
- A hand-operated pump capable of accurately and precisely sampling 50 mL and 100 mL interval volumes (i.e., Markes International C-EZVOCPO or equivalent).
- Two 1/4-inch I.D. brass long-term storage caps (i.e., Markes International C-CF020 or equivalent) for each TD tube fitted with two PTFE ferrules (i.e., Markes International C-FP020 or equivalent).
- Tools for capping and uncapping tubes with brass long-term storage caps (i.e., Markes International CapLok or equivalent).
- Sampling kits are provided to OSHA field activities through official sampling media procurement channels. Sampling kits consist of a carrying case with ten conditioned and sealed TD tubes, one CapLok tool, one hand-operated pump, and a user quick-guide.

2.2 Reagents

None required

2.3 Technique

Immediately before sampling, uncap a sealed TD tube on both ends and securely position the non-sampling end of the tube into the pump inlet with the flow direction arrow pointing towards the pump. Hold the apparatus with one hand on the shaft of the pump while operating the plunger handle of the pump with the other hand. Place the open sampling end of the tube inside the area of interest or close to suspected off-gassing sources without contact.

Select a sample volume of 50 mL or 100 mL by aligning the specific volume-labeled red dot on the plunger with the red dot on the shaft. Pull out the plunger slowly and evenly for 5 or 10 seconds until it latches to complete a half-stroke for 50 mL or a full stroke for 100 mL, respectively. To return the plunger to its starting point, rotate it a quarter turn clockwise when set to 100 mL or a quarter turn counterclockwise when set to 50 mL, then push the plunger back until it fully stops. To make multiple strokes on a single tube, keep the tube in place and repeat pulling and returning plunger steps.

Collect 100 mL of workplace air (one 100-mL stroke or two 50-mL strokes) for suspected analytes with permissible exposure limits above 5 ppm. Collect up to 500 mL (five 100-mL strokes or ten 50-mL strokes) for suspected analytes with permissible exposure limits at or below 5 ppm.

After sampling the appropriate volume, remove the tube from the pump and immediately cap both ends with brass long-term storage caps by inserting each tube end all the way passing through the cap nut and the PTFE ferrule then finger-tightening the cap bolt onto the top of the tube end. Use the CapLok tool or wrenches to tighten further one quarter turn. Avoid overtightening that may damage the PTFE ferrule and compromise the seal. Ensure an effective seal is achieved by firmly gripping both end caps and attempting to pull them off the tube. Re-seal the tube if tightened end caps can be removed without effort.

^a TD tubes are conditioned, cleaned, and replaced per manufacturer instructions. Before shipping TD tubes, all conditioned tubes are screened using the analytical conditions described in Section 3.5 to verify the elimination of contaminants. All pre-screening chromatograms are documented to compare to sample chromatograms.

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Submit at least one blank sample with each set of samples. Handle the blank sample in the same manner as the other samples except draw no air through it.

Record sample air volume (mL) along with any potential interference on the Form OSHA-91A.

Place all sealed tubes along with all other received items into carrying case and seal the handle of the case with a Form OSHA-21. Do not apply any label or markings to the tubes or caps due to the risk of contamination during high-temperature desorption.

Submit the entire kit, including any unused tubes, to the laboratory for analysis by overnight shipping. Do not refrigerate the samples before, during, or after shipping.

3 Analytical Procedure

3.1 Apparatus

- TD tubes
- Diffusion-locking (DiffLok) caps and ferrules
- CapLok Tool
- Hand-operated pump with 50 mL/100 mL intervals
- Calibration solution loading rig (CSLR)
- Tube conditioner equipped with nitrogen gas purging
- Cold traps (i.e., Markes International Air Toxics Analyzer U-T15ATA-2S or equivalent)
- Two-stage TD-GC/MS instrument

3.2 Reagents

Use analytical grade standards and reagents.

3.3 Standard Preparation

Use methanol and carbon disulfide as solvents for preparation of polar and non-polar standards, respectively. Prepare single or multiple analyte-containing standard solution with a 1:2000 or higher dilution factor from respective standards to avoid saturating the cold trap. Purge the CSLR with a conditioned TD tube connected using ultra-high purity nitrogen at a flow rate of 50-100 mL/min for 5 minutes prior to spiking. Spike 1 to 2 μL of the standard solution onto the sorbent while purging for 5 minutes then immediately cap the tube for subsequent analysis.

3.4 Sample Preparation

Check tightness of storage caps on each received TD tube. Record tubes with significantly loose caps for potential sample loss and contamination. Recap tubes with DiffLok caps immediately after removing storage caps. Analyze DiffLok-capped tubes within 24 hours to prevent sample loss and contamination.

3.5 Qualitative Analysis

Analyze samples using a TD-GC/MS instrument and the analytical conditions described below. Obtain an optimal mass spectrum by subtracting a background spectrum from a data point extracted from the apex of a single analyte peak. Identify unknown analytes using the National Institute of Standards and Technology (NIST) Mass Spectral Library. Verify identity of an unknown analyte by matching the retention time of a candidate reference standard peak. If a reference standard is not available, verify the identity using the NIST Retention Index (RI) Database for normal alkane RI on a similar column to the one used in analysis. Normal alkane retention indices can be calculated using Equation 1 shown below.

$$I = 100 \left[n + \frac{(t - t_n)}{(t_{n+1} - t_n)} \right] \quad (1)$$

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where I is the retention index of an analyte, t is the retention time of the analyte, t_n and t_{n+1} are retention times of reference normal alkanes eluting immediately before and after the analyte, and n is the carbon number of the normal alkane eluting immediately before the analyte.¹

Retention times for sixty-three selected analytes are listed below.

TD conditions

cold trap:	Air Toxics Analyzer
mode:	Two-stage tube desorption
carrier gas:	Helium
standby split flow:	15 mL/min
flow path temperature:	180 °C
GC cycle time:	45 min
dry purge time:	3 min
dry purge flow:	50 mL/min
tube desorption time:	10 min
tube desorption temperature:	280 °C
tube desorption trap flow:	25 mL/min
tube desorption split flow:	225 mL/min
trap purge time:	4 min
trap purge flow:	100 mL/min
trap low temperature:	25 °C
trap high temperature:	300 °C
trap desorb time:	4 min
trap split flow:	12 mL/min (Total split ratio: 110:1)

GC conditions

oven temperature:	35 °C (hold 5 min), ramp to 280 °C at 7 °C/min (hold 1 min)
run time:	41 min
inlet temperature:	180 °C
column:	Agilent HP-5MS UI capillary column, 30-m × 0.25-mm i.d., d_f = 0.25- μ m, or equivalent
column mode/initial flow:	constant flow/1.2 mL/min (helium)
retention times:	1.66 min - ethyl alcohol 1.75 min - acetonitrile 1.75 min - acetone 1.76 min - isopropyl alcohol 1.77 min - <i>n</i> -pentane 1.85 min - <i>tert</i> -butyl alcohol 1.88 min - methyl acetate 1.91 min - methylene chloride 1.95 min - allyl alcohol 2.00 min - <i>n</i> -propyl alcohol 2.13 min - 1,1-dichloroethane 2.25 min - methyl ethyl ketone 2.26 min - <i>n</i> -hexane 2.27 min - <i>sec</i> -butyl alcohol 2.40 min - ethyl acetate 2.43 min - chlorobromomethane 2.43 min - chloroform 2.51 min - 1-bromopropane 2.52 min - isobutyl alcohol 2.77 min - ethylene dichloride 2.93 min - benzene 2.93 min - cyclohexane 2.94 min - carbon tetrachloride 2.95 min - <i>n</i> -butyl alcohol 2.96 min - isopropyl acetate 3.53 min - <i>n</i> -heptane

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3.54 min - trichloroethylene
3.68 min - *tert*-butyl acetate
4.01 min - epichlorohydrin
4.04 min - methyl cyclohexane
4.41 min - isoamyl alcohol
4.54 min - methyl isobutyl ketone
5.24 min - *sec*-butyl acetate
5.35 min - toluene
5.53 min - 1,1,2-trichloroethane
5.73 min - isobutyl acetate
6.52 min - *n*-octane
6.76 min - tetrachloroethylene
7.12 min - *n*-butyl acetate
7.95 min - diacetone alcohol
7.99 min - 2-methoxyethyl acetate
7.99 min - chlorobenzene
8.48 min - methyl isoamyl ketone
8.53 min - ethyl benzene
8.77 min - *m*-xylene
8.79 min - *p*-xylene
9.15 min - isoamyl acetate
9.40 min - ethyl butyl ketone
9.50 min - *o*-xylene
9.51 min - cyclohexanone
9.79 min - *n*-nonane
9.82 min - *sec*-hexyl acetate
9.95 min - 2-butoxyethanol
10.16 min - 2-ethoxyethyl acetate
10.28 min - *n*-amyl acetate
10.73 min - α -pinene
11.72 min - 1,3,5-trimethylbenzene
11.81 min - diisobutyl ketone
12.26 min - ethyl amyl ketone
12.39 min - 1,2,4-trimethylbenzene
12.92 min - benzyl chloride
13.14 min - 1,2,3-trimethylbenzene
13.42 min - 1,2-dichlorobenzene

mass spectrometer conditions

mode:	electron ionization (EI)
acquisition mode:	scan, m/z 20-350
EMV mode:	gain factor (1)
temperatures:	230 °C (source), 150 °C (quadrupole assembly), 280 °C (transfer line)

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Table 1. Analytes with Mass Spectral Data.

analyte	characteristic ions (<i>m/z</i>)	analyte	characteristic ions (<i>m/z</i>)
acetone	43, 58*	ethyl butyl ketone	57, 72, 85, 114*
acetonitrile	39, 40, 41*	ethylene dichloride	49, 62, 64, 98
allyl alcohol	39, 57, 58*	<i>n</i> -heptane	43, 57, 71, 100*
<i>n</i> -amyl acetate	43, 55, 61, 70	<i>n</i> -hexane	43, 57, 86*
benzene	77, 78*	<i>sec</i> -hexyl acetate	43, 69, 84, 87
benzyl chloride	65, 89, 91, 126*, 128	isoamyl acetate	43, 55, 70
1-bromopropane	41, 43, 122, 124	isoamyl alcohol	42, 55, 57, 70
2-butoxyethanol	41, 45, 57, 87, 100	isobutyl acetate	43, 56, 73
<i>n</i> -butyl acetate	41, 43, 56, 73	isobutyl alcohol	41, 42, 43, 74*
<i>sec</i> -butyl acetate	43, 56, 87	isopropyl acetate	43, 61, 87
<i>tert</i> -butyl acetate	41, 43, 56, 57, 59	isopropyl alcohol	43, 45, 59
<i>n</i> -butyl alcohol	41, 42, 43, 55, 56, 57	2-methoxyethyl acetate	43, 45, 58
<i>sec</i> -butyl alcohol	45, 59	methyl ethyl ketone	43, 57, 72
<i>tert</i> -butyl alcohol	41, 43, 57, 59	methyl isoamyl ketone	43, 58, 71
carbon tetrachloride	82, 117, 119, 121	methyl isobutyl ketone	43, 57, 58, 85, 100*
chlorobenzene	50, 51, 77, 112*, 114	methyl acetate	43, 59, 74*
chlorobromomethane	49, 51, 93, 95, 128, 130, 133	methyl cyclohexane	41, 55, 83, 98*
chloroform	47, 83, 85, 87	methylene chloride	49, 51, 84*, 86
cyclohexane	41, 55, 56, 69, 84*	<i>n</i> -nonane	43, 57, 71, 85
cyclohexanone	42, 55, 98*	<i>n</i> -octane	43, 57, 71, 85
diacetone alcohol	43, 58, 59, 101	<i>n</i> -pentane	43, 57, 72*
1,1-dichloroethane	63, 65, 83, 85, 98*	α -pinene	93, 105, 121, 136*
1,2-dichlorobenzene	50, 75, 111, 146, 148	<i>n</i> -propyl alcohol	42, 59, 60*
diisobutyl ketone	41, 57, 58, 85, 142*	tetrachloroethylene	129, 131, 133, 164, 166, 168
2-ethoxyethyl acetate	43, 59, 72	toluene	65, 91, 92*
epichlorohydrin	49, 51, 57, 62	1,1,2-trichloroethane	97, 99, 132, 134
ethyl acetate	43, 45, 61, 70, 88*	trichloroethylene	60, 95, 97, 130, 132, 134
ethyl alcohol	43, 45, 46*	trimethylbenzene	77, 91, 105, 120*
ethyl amyl ketone	43, 57, 71, 72, 99	xylene	77, 91, 105, 106*
ethyl benzene	65, 77, 91, 106*		

* Indicates molecular ion

3.6 Interference

The initial air peak and TD tube artifacts may contribute to masking analytes of interest. Any overlapping or co-eluting compound can potentially result in an indiscernible mass spectrum pattern leading to failure of mass spectral library matching. Identification of co-eluting components might be achieved by using spectral deconvolution-supported software, such as Agilent MassHunter 10. Decomposition of diacetone alcohol into acetone was observed during the storage stability study in Section 4.2.2, which may lead to an inaccurate conclusion on the presence of acetone.

3.7 Reporting

Compare each sample chromatogram to its corresponding pre-screening chromatogram and the field blank chromatogram. Do not report an analyte found in the pre-screening chromatogram or the field blank chromatogram with similar or larger analytical response. For each analyte reported, provide the CAS number, Integrated Management Information System (IMIS) number if applicable, and the confirmation method used. Confirmation methods include matching retention time and mass spectra to a standard reference, matching RI and mass spectra to the NIST Retention Index Database and NIST Mass Spectral Library, or matching mass spectra to the NIST Mass Spectral Library.

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4 Method Validation

Validation focused on retention times, mass spectral library matching, detection limit of the overall procedure (DLOP) and post-sampling storage stability.

4.1 Detection Limit of the Overall Procedure

The DLOP is the analyte mass per sample that produces a response greater than $3.3 \times$ the standard error of estimate ($S_{y/x}$) divided by the slope of the linear regression equation. Ten TD tubes were spiked with approximately equal descending increments of analyte using CSLR, such that the highest loading would produce a peak approximately 10 times the response of a sample blank at or near the chromatographic retention time of the analyte. These spiked tubes and a sample blank were analyzed with the analytical parameters. The data obtained were used to calculate the required parameters ($S_{y/x}$ and the slope) for the calculation of the DLOP. Due to the interference of the initial air peak, artifacts from the TD tubes and incompatibility of spiking solvent, only a group of analytes were selected for determination of DLOP as shown in Table 2. The least sensitive characteristic ion was used for determination of DLOP to obtain a reliable mass spectrum. Results obtained for the sample blank and the ten spiked tubes are listed below in Tables 3 through 48 and plotted in associated Figures 1 through 46.

Table 2. Selected analytes for DLOP data.

analyte	ng/sample	analyte	ng/sample
allyl alcohol	4.0	ethylene dichloride	0.72
<i>n</i> -amyl acetate	3.6	sec-hexyl acetate	2.1
benzyl chloride	3.9	isoamyl acetate	4.0
1-bromopropane	1.3	isoamyl alcohol	4.9
<i>n</i> -butyl acetate	3.1	isobutyl acetate	3.5
sec-butyl acetate	1.3	isobutyl alcohol	2.8
<i>tert</i> -butyl acetate	6.4	isopropyl acetate	1.4
<i>n</i> -butyl alcohol	2.5	2-methoxyethyl acetate	65
sec-butyl alcohol	2.1	methyl isoamyl ketone	2.3
<i>tert</i> -butyl alcohol	1.3	methyl isobutyl ketone	1.1
carbon tetrachloride	1.5	methylene chloride	0.95
chlorobenzene	0.88	α -pinene	3.5
chlorobromomethane	0.87	<i>n</i> -propyl alcohol	1.2
chloroform	0.77	tetrachloroethylene	0.65
cyclohexanone	2.2	1,1,2-trichloroethane	0.91
1,1-dichloroethane	1.5	trichloroethylene	0.53
1,2-dichlorobenzene	1.8	1,3,5-trimethylbenzene	1.1
diisobutyl ketone	1.9	1,2,4-trimethylbenzene	1.1
2-ethoxyethyl acetate	9.7	1,2,3-trimethylbenzene	1.3
epichlorohydrin	5.9	<i>m</i> -xylene	1.0
ethyl acetate	1.7	<i>o</i> -xylene	1.1
ethyl amyl ketone	1.9	<i>p</i> -xylene	1.1
ethyl benzene	0.75		
ethyl butyl ketone	3.2		

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Table 3. DLOP data for allyl alcohol (m/z 58).

mass per sample (ng)	response
0.00	0.00
3.84	728
7.68	1386
11.5	2138
15.4	3197
19.2	3487
23.0	5046
26.9	5080
30.7	6125
34.6	6603
38.4	7492

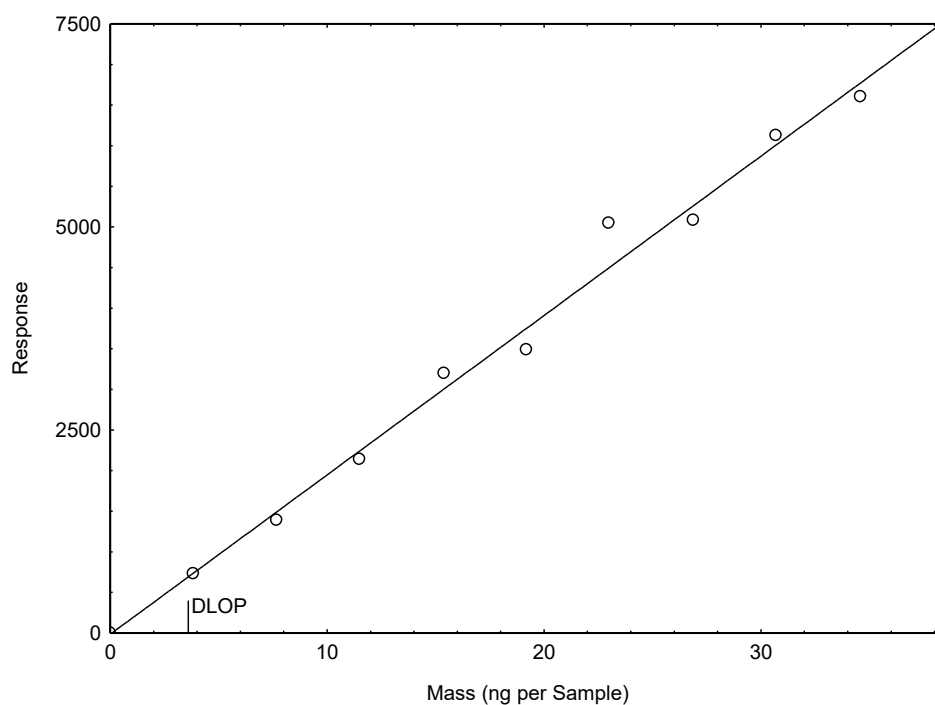


Figure 1. Plot of data used to determine the DLOP for allyl alcohol ($y = 196x - 14.9$, $S_{y/x} = 237$, DLOP = 4.0 ng/sample).

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Table 4. DLOP data for *n*-amyl acetate (*m/z* 61).

mass per sample (ng)	response
0.00	0.00
1.77	0.00
3.54	268
5.31	595
7.08	1186
8.85	1123
10.6	1519
12.4	2312
14.2	2585
15.9	3180
17.7	3317

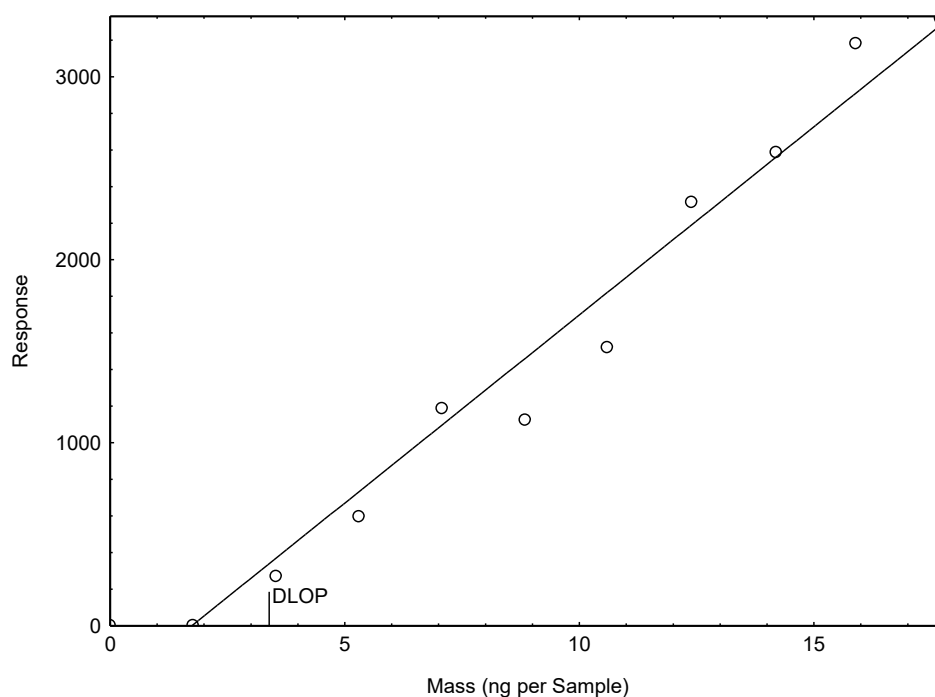


Figure 2. Plot of data used to determine the DLOP for *n*-amyl acetate ($y = 206x - 357$, $S_{y/x} = 226$, DLOP = 3.6 ng/sample).

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Table 5. DLOP data for benzyl chloride (m/z 89).

mass per sample (ng)	response
0.00	0.00
1.97	0.00
3.94	277
5.91	500
7.88	595
9.85	968
11.8	1369
13.8	1739
15.8	1890
17.7	2161
19.7	2095

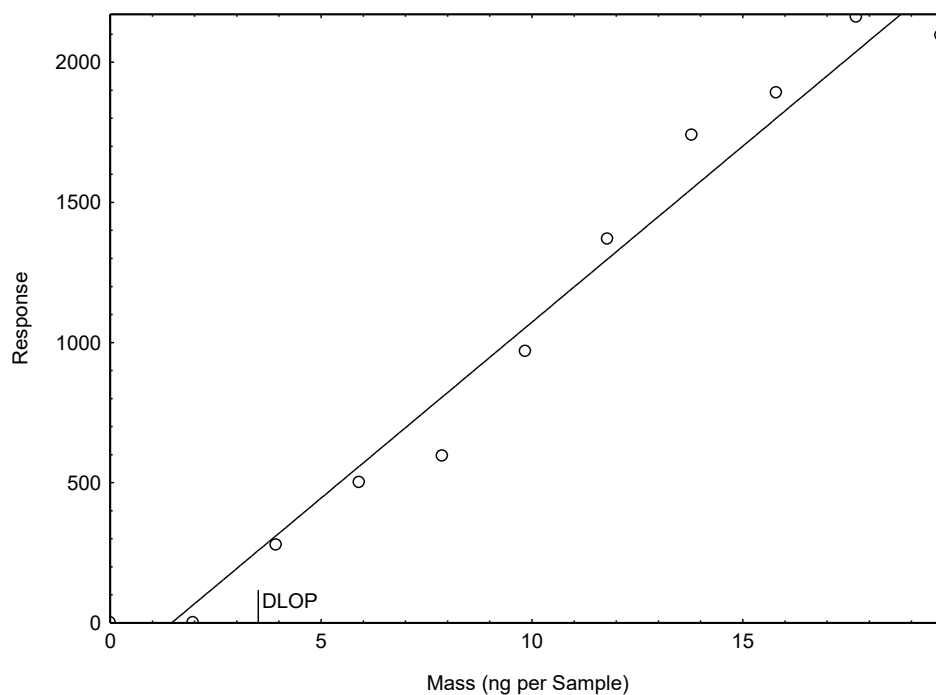


Figure 3. Plot of data used to determine the DLOP for benzyl chloride ($y = 126x - 183$, $S_{y/x} = 148$, DLOP = 3.9 ng/sample).

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Table 6. DLOP data for 1-bromopropane (m/z 124).

mass per sample (ng)	response
0.00	0.00
0.541	48.7
1.08	276
1.62	548
2.16	729
2.71	615
3.25	1162
3.79	1091
4.33	1374
4.87	1476
5.41	1981

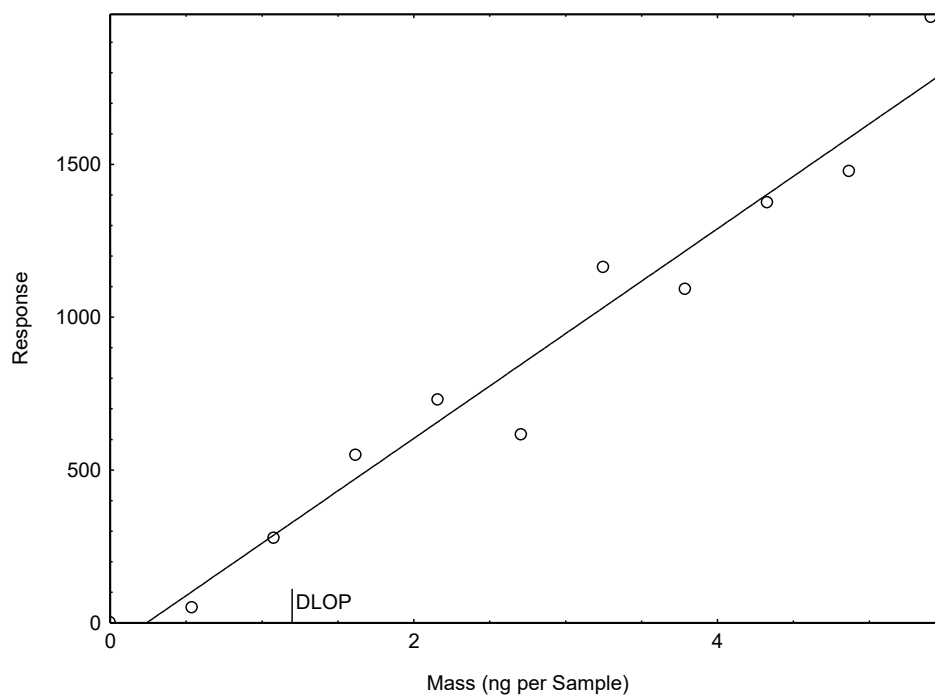


Figure 4. Plot of data used to determine the DLOP for 1-bromopropane ($y = 343x - 82.5$, $S_{y/x} = 135$, DLOP = 1.3 ng/sample).

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Table 7. DLOP data for *n*-butyl acetate (*m/z* 73).

mass per sample (ng)	response
0.00	0.00
1.59	0.00
3.18	494
4.77	891
6.36	1140
7.95	1266
9.54	1842
11.1	2059
12.7	2105
14.3	2857
15.9	2650

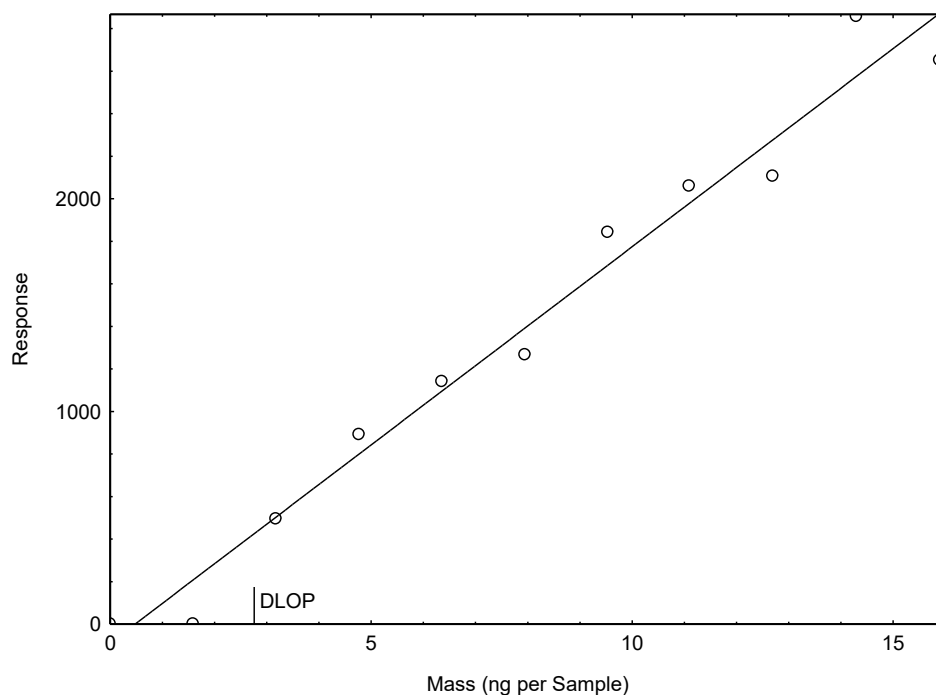


Figure 5. Plot of data used to determine the DLOP for *n*-butyl acetate ($y = 186x - 88.5$, $S_{y/x} = 172$, DLOP = 3.1 ng/sample).

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Table 8. DLOP data for *sec*-butyl acetate (*m/z* 87).

mass per sample (ng)	response
0.00	0.00
0.902	34.3
1.80	761
2.71	1066
3.61	1489
4.51	1536
5.41	2059
6.31	2463
7.22	2601
8.12	3221
9.02	3236

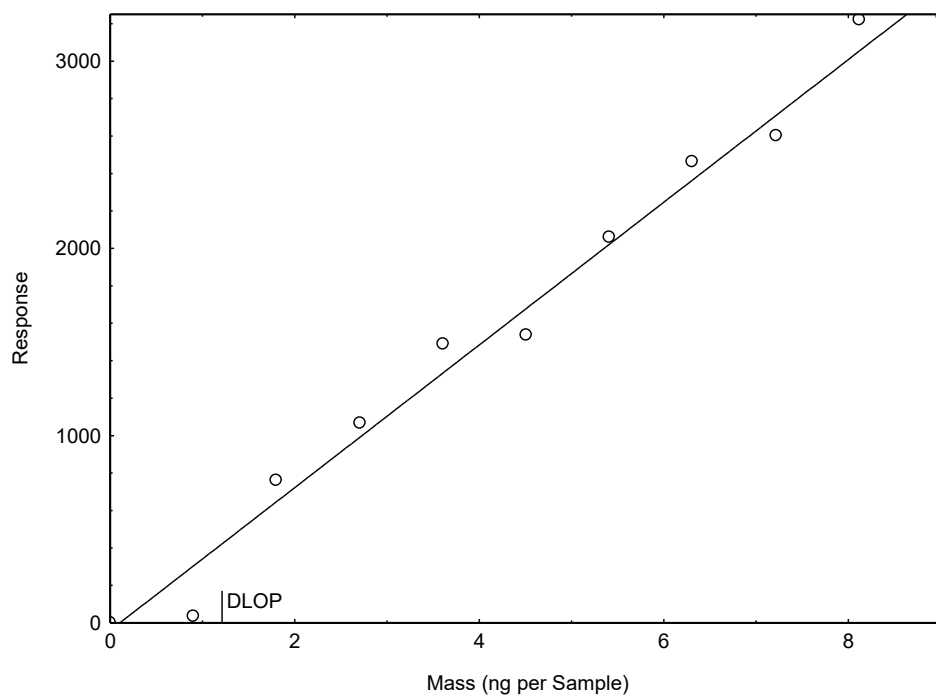


Figure 6. Plot of data used to determine the DLOP for *sec*-butyl acetate ($y = 381x - 39.8$, $S_{y/x} = 154$, DLOP = 1.3 ng/sample).

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Table 9. DLOP data for *tert*-butyl acetate (*m/z* 56).

mass per sample (ng)	response
0.00	0.00
1.85	0.00
3.70	44.0
5.55	514
7.40	761
9.25	457
11.1	840
12.9	1542
14.8	1435
16.7	1531
18.5	2063

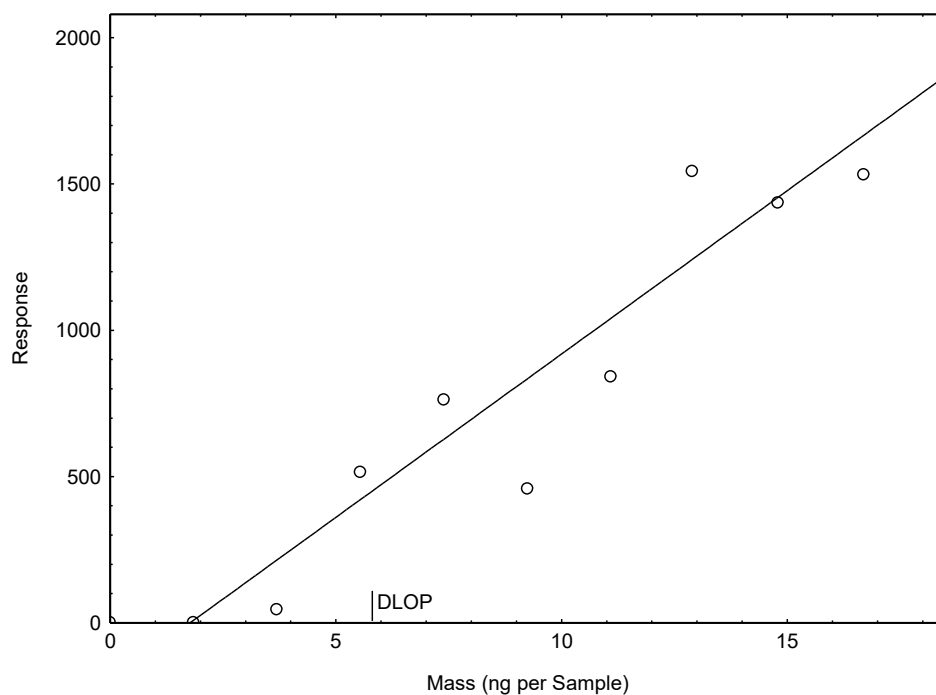


Figure 7. Plot of data used to determine the DLOP for *tert*-butyl acetate ($y = 112x - 198$, $S_{y/x} = 217$, DLOP = 6.4 ng/sample).

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Table 10. DLOP data for *n*-butyl alcohol (*m/z* 57).

mass per sample (ng)	response
0.00	0.00
2.02	392
4.04	494
6.06	826
8.08	1033
10.1	1131
12.1	1435
14.1	1831
16.2	2038
18.2	2370
20.2	2390

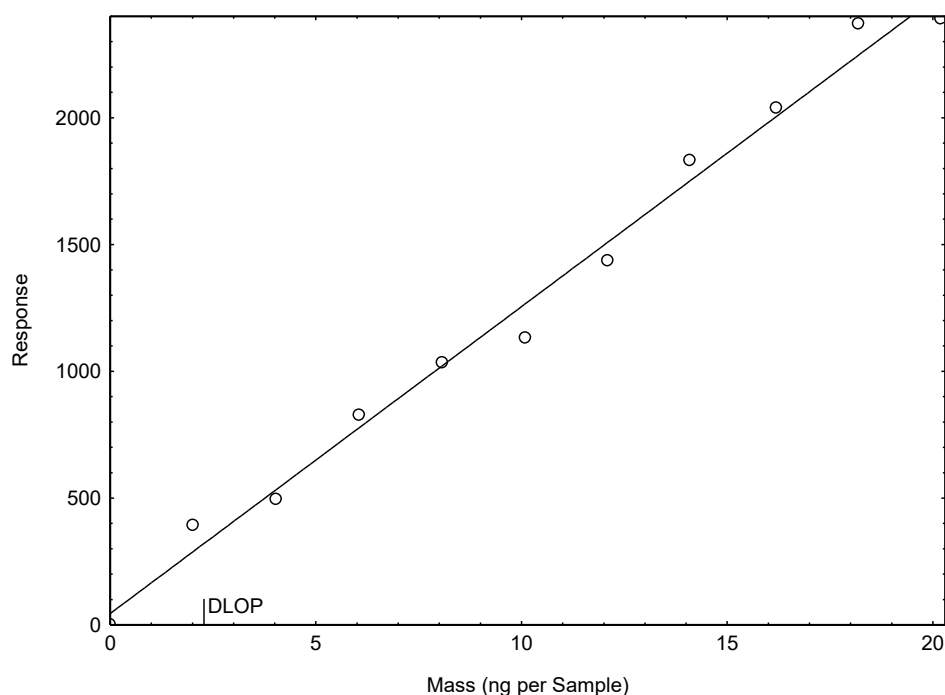


Figure 8. Plot of data used to determine the DLOP for *n*-butyl alcohol ($y = 121x + 44.8$, $S_{y/x} = 90.0$, DLOP = 2.5 ng/sample).

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Table 11. DLOP data for *sec*-butyl alcohol (m/z 59).

mass per sample (ng)	response
0.00	0.00
1.61	457
3.22	1166
4.83	1912
6.44	2716
8.05	2790
9.66	3903
11.3	4248
12.9	5073
14.5	5259
16.1	5616

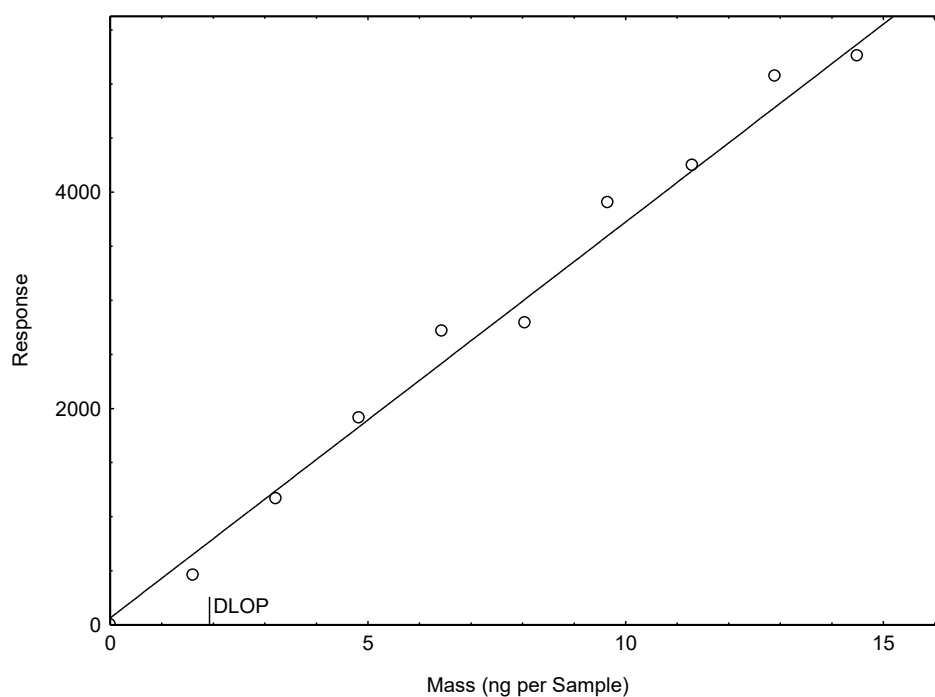


Figure 9. Plot of data used to determine the DLOP for *sec*-butyl alcohol ($y = 366x + 64.7$, $S_{y/x} = 235$, DLOP = 2.1 ng/sample).

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Table 12. DLOP data for *tert*-butyl alcohol (*m/z* 57).

mass per sample (ng)	response
0.00	0.00
1.37	367
2.74	512
4.11	933
5.48	1219
6.85	1359
8.22	1785
9.59	2239
11.0	2418
12.3	2669
13.7	2879

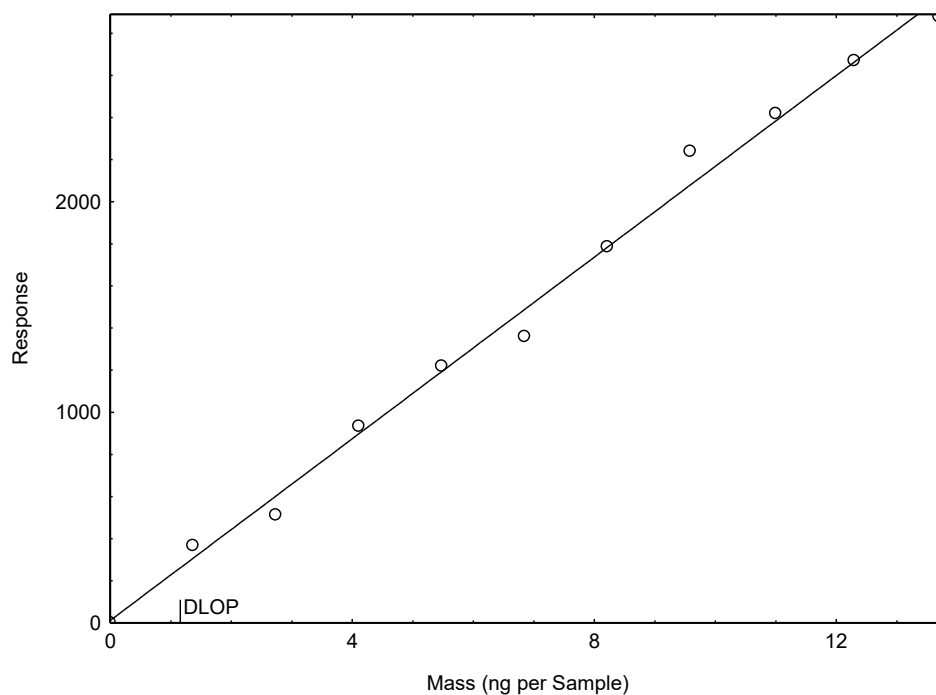


Figure 10. Plot of data used to determine the DLOP for *tert*-butyl alcohol ($y = 216x + 11.6$, $S_{y/x} = 84.9$, DLOP = 1.3 ng/sample).

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Table 13. DLOP data for carbon tetrachloride (m/z 82).

mass per sample (ng)	response
0.00	0.00
0.708	275
1.42	705
2.12	1117
2.83	1336
3.54	1577
4.25	2073
4.96	2372
5.66	2716
6.37	3270
7.08	2812

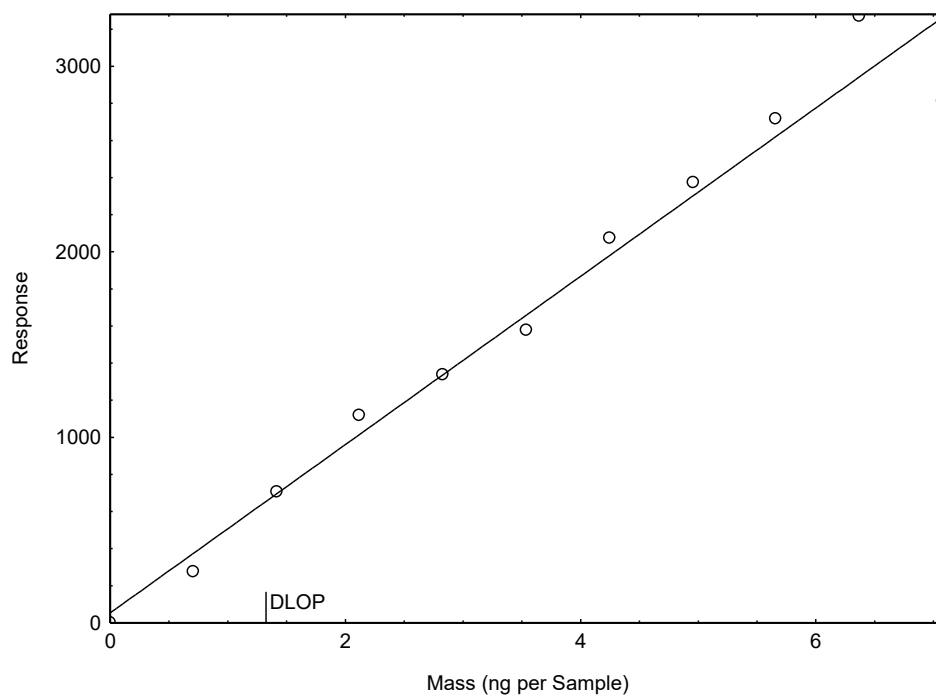


Figure 11. Plot of data used to determine the DLOP for carbon tetrachloride ($y = 454x + 53.8$, $S_{y/x} = 201$, DLOP = 1.5 ng/sample).

OSHA Method 1031, Qualitative Screening for Volatile Organic Compounds Collected on Thermal Desorption Tubes

Table 14. DLOP data for chlorobenzene (m/z 50).

mass per sample (ng)	response
0.00	0.00
0.498	33.9
0.996	233
1.49	420
1.99	464
2.49	591
2.99	905
3.49	1093
3.98	1139
4.48	1511
4.98	1686

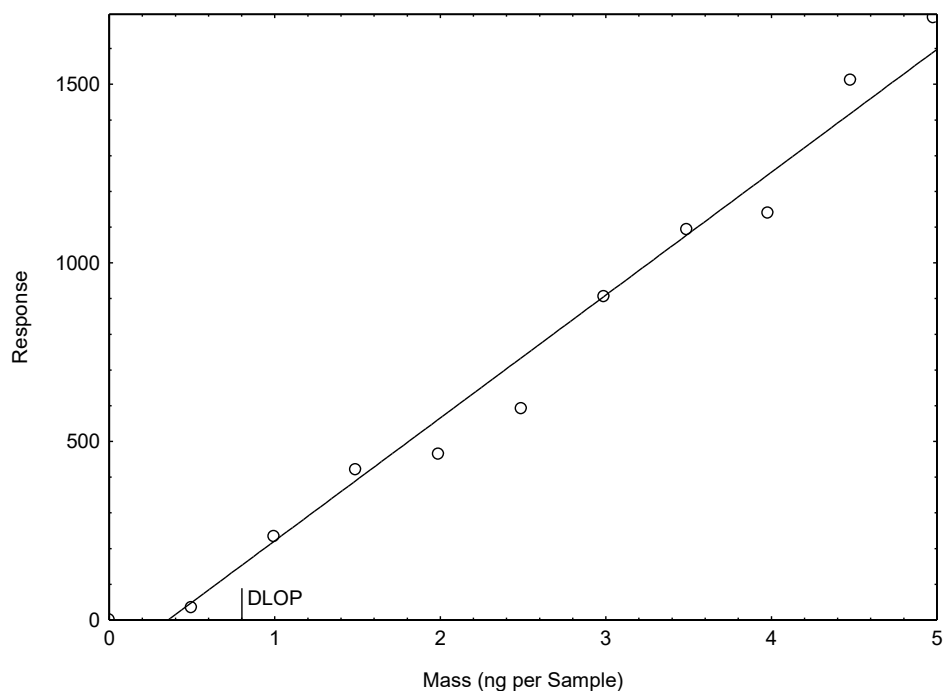


Figure 12. Plot of data used to determine the DLOP for chlorobenzene ($y = 344x - 122$, $S_{y/x} = 91.8$, DLOP = 0.88 ng/sample).

OSHA Method 1031, Qualitative Screening for Volatile Organic Compounds Collected on Thermal Desorption Tubes

Table 15. DLOP data for chlorobromomethane (m/z 95).

mass per sample (ng)	response
0.00	0.00
0.386	0.00
0.772	79.0
1.16	298
1.54	373
1.93	463
2.32	519
2.70	685
3.09	885
3.47	870
3.86	851

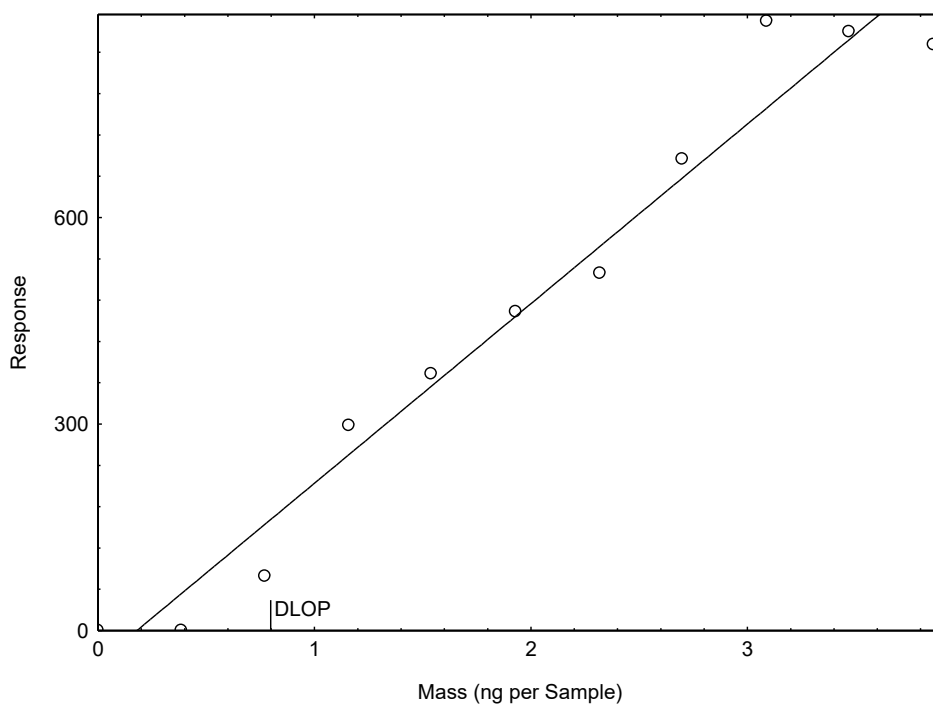


Figure 13. Plot of data used to determine the DLOP for chlorobromomethane ($y = 261x - 46.8$, $S_{y/x} = 69.2$, DLOP = 0.87 ng/sample).

OSHA Method 1031, Qualitative Screening for Volatile Organic Compounds Collected on Thermal Desorption Tubes

Table 16. DLOP data for chloroform (m/z 87).

mass per sample (ng)	response
0.00	0.00
0.297	0.00
0.594	116
0.891	268
1.19	459
1.48	438
1.78	636
2.08	588
2.38	949
2.67	950
2.97	918

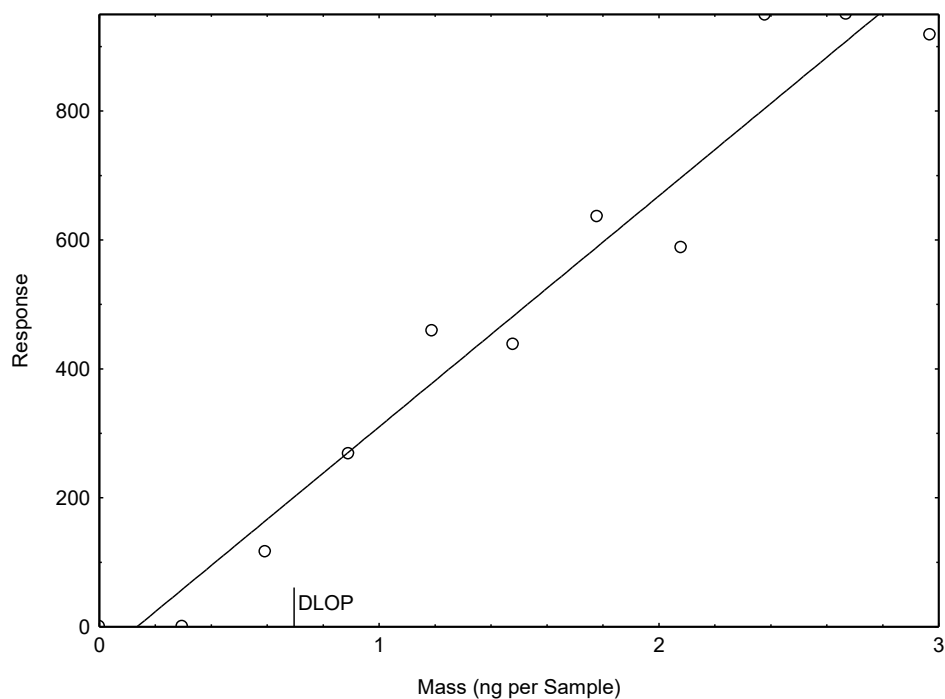


Figure 14. Plot of data used to determine the DLOP for chloroform ($y = 358x - 48.3$, $S_{y/x} = 83.5$, DLOP = 0.77 ng/sample).

OSHA Method 1031, Qualitative Screening for Volatile Organic Compounds Collected on Thermal Desorption Tubes

Table 17. DLOP data for cyclohexanone (m/z 98).

mass per sample (ng)	response
0.00	0.00
1.92	143
3.84	683
5.76	1465
7.68	1850
9.60	2461
11.5	3455
13.4	3422
15.4	4126
17.3	4813
19.2	5402

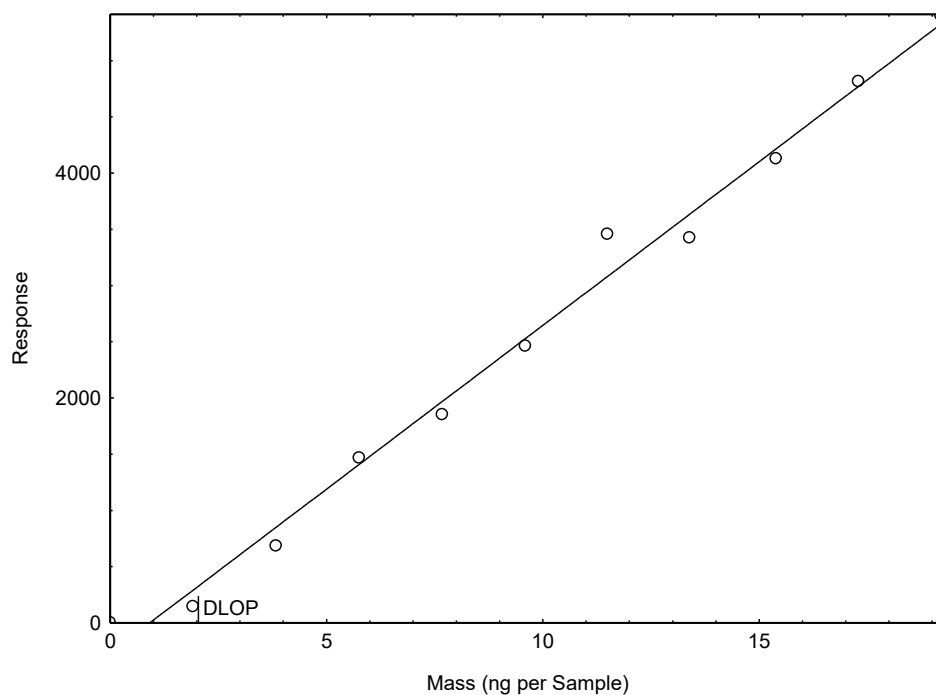


Figure 15. Plot of data used to determine the DLOP for cyclohexanone ($y = 291x - 267$, $S_{y/x} = 196$, DLOP = 2.2 ng/sample).

OSHA Method 1031, Qualitative Screening for Volatile Organic Compounds Collected on Thermal Desorption Tubes

Table 18. DLOP data for 1,1-dichloroethane (m/z 98).

mass per sample (ng)	response
0.00	0.00
0.524	0.00
1.05	36.6
1.57	210
2.10	239
2.62	444
3.14	669
3.67	690
4.19	764
4.71	799
5.24	801

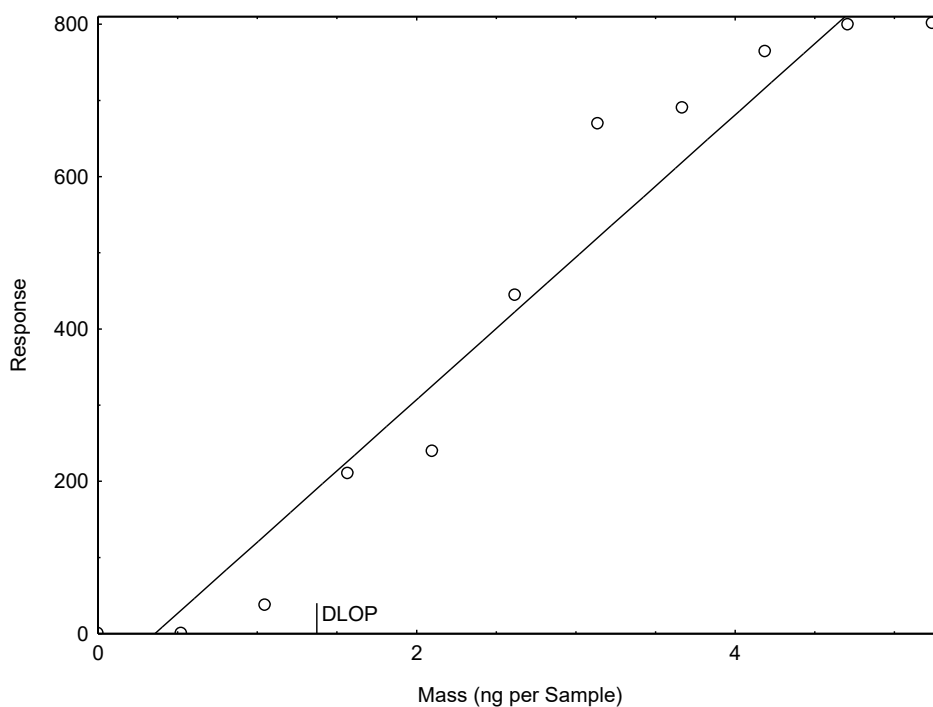


Figure 16. Plot of data used to determine the DLOP for 1,1-dichloroethane ($y = 187x - 66.8$, $S_{y/x} = 84.6$, DLOP = 1.5 ng/sample).

OSHA Method 1031, Qualitative Screening for Volatile Organic Compounds Collected on Thermal Desorption Tubes

Table 19. DLOP data for 1,2-dichlorobenzene (m/z 50).

mass per sample (ng)	response
0.00	0.00
0.976	0.00
1.95	86.7
2.93	281
3.90	428
4.88	579
5.86	895
6.83	1045
7.81	1119
8.78	1409
9.76	1691

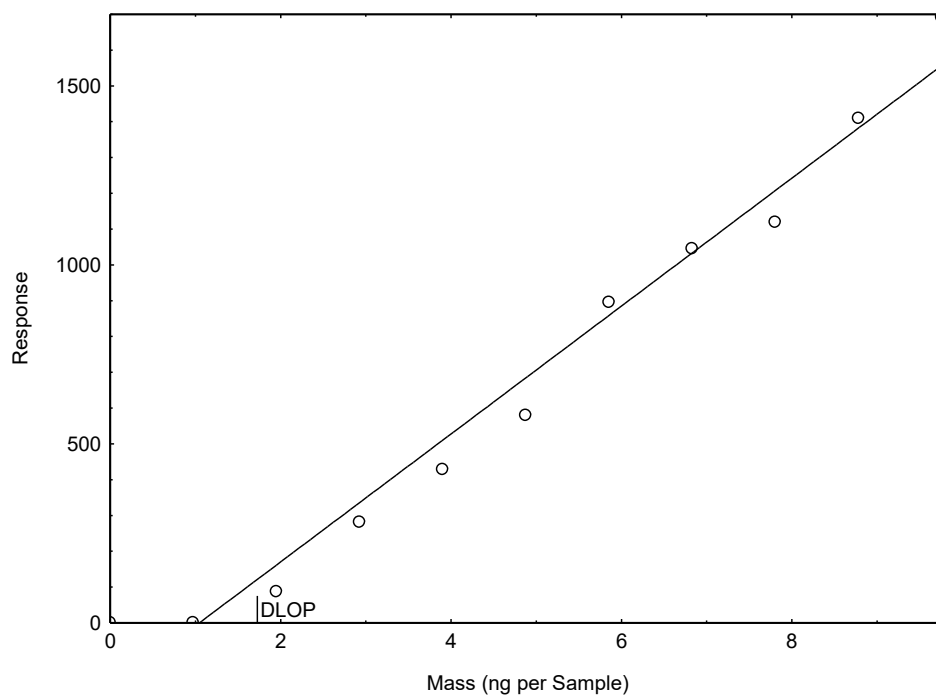


Figure 17. Plot of data used to determine the DLOP for 1,2-dichlorobenzene ($y = 179x - 187$, $S_{y/x} = 99.9$, DLOP = 1.8 ng/sample).

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Table 20. DLOP data for diisobutyl ketone (m/z 142).

mass per sample (ng)	response
0.00	0.00
1.44	0.00
2.88	176
4.32	364
5.76	483
7.20	625
8.64	771
10.1	862
11.5	1149
13.0	1257
14.4	1476

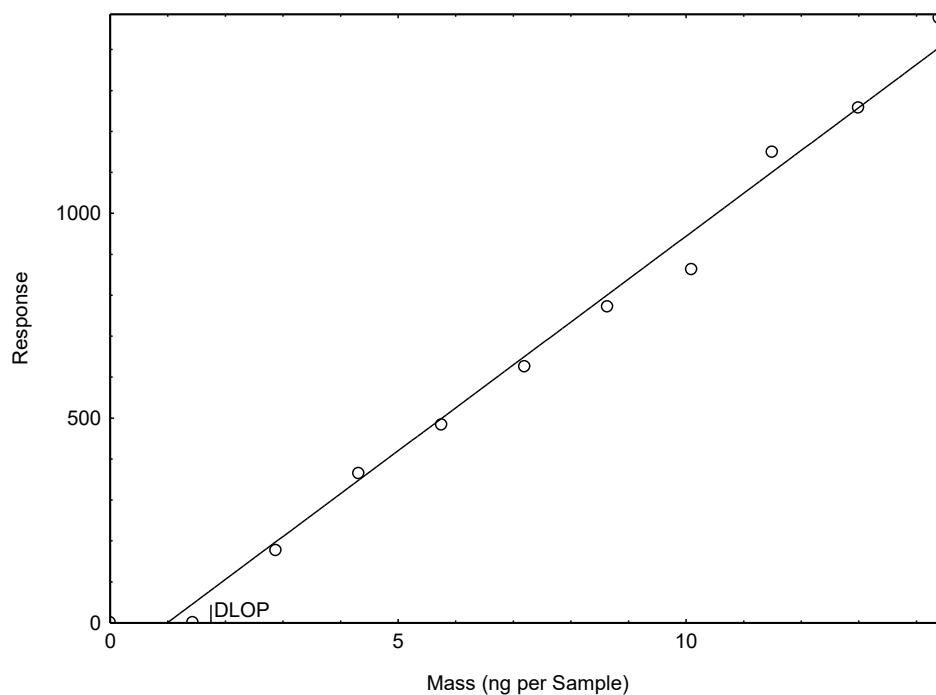


Figure 18. Plot of data used to determine the DLOP for diisobutyl ketone ($y = 105x - 103$, $S_{y/x} = 59.1$, DLOP = 1.9 ng/sample).

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Table 21. DLOP data for 2-ethoxyethyl acetate (m/z 72).

mass per sample (ng)	response
0.00	0.00
3.56	45.8
7.12	520
10.7	1440
14.2	2300
17.8	1870
21.4	2408
24.9	4135
28.5	4451
32.0	5692
35.6	6642

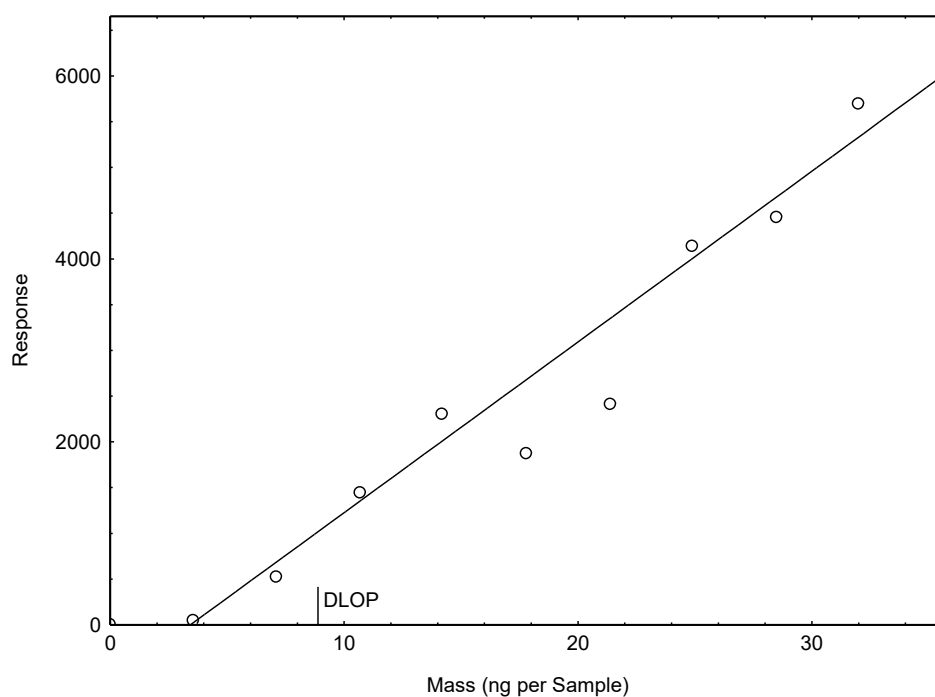


Figure 19. Plot of data used to determine the DLOP for 2-ethoxyethyl acetate ($y = 187x - 639$, $S_{y/x} = 547$, DLOP = 9.7 ng/sample).

OSHA Method 1031, Qualitative Screening for Volatile Organic Compounds Collected on Thermal Desorption Tubes

Table 22. DLOP data for epichlorohydrin (m/z 51).

mass per sample (ng)	response
0.00	0.00
2.95	0.00
5.90	222
8.85	574
11.8	984
14.8	947
17.7	1539
20.6	1641
23.6	1747
26.6	1837
29.5	2285

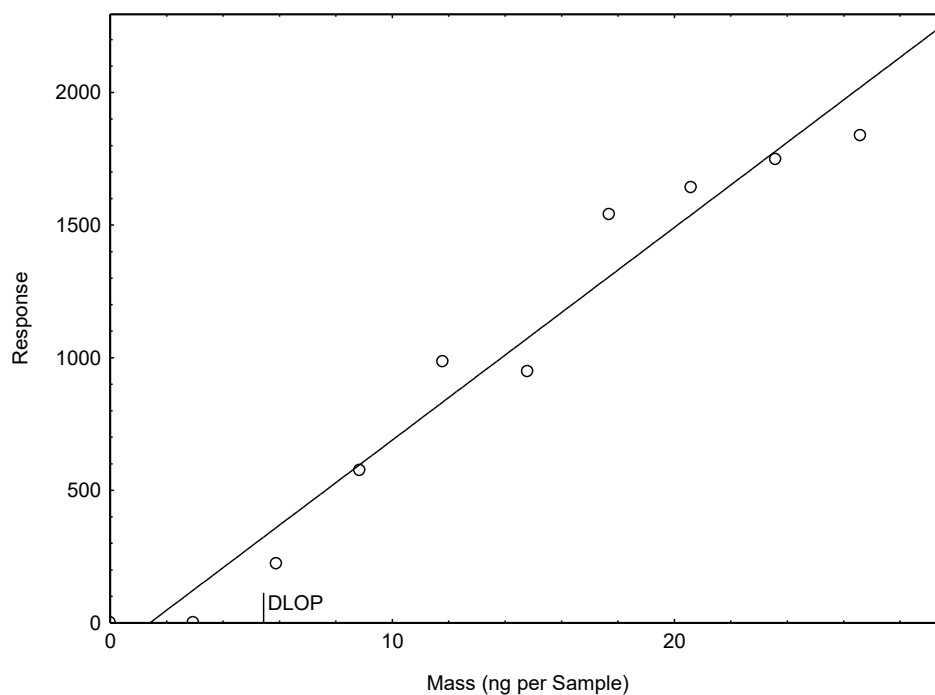


Figure 20. Plot of data used to determine the DLOP for epichlorohydrin ($y = 80.2x - 113$, $S_{y/x} = 144$, DLOP = 5.9 ng/sample).

OSHA Method 1031, Qualitative Screening for Volatile Organic Compounds Collected on Thermal Desorption Tubes

Table 23. DLOP data for ethyl acetate (m/z 88).

mass per sample (ng)	response
0.00	0.00
0.720	0.00
1.44	117
2.16	275
2.88	446
3.60	459
4.33	572
5.05	860
5.77	691
6.49	1041
7.21	1177

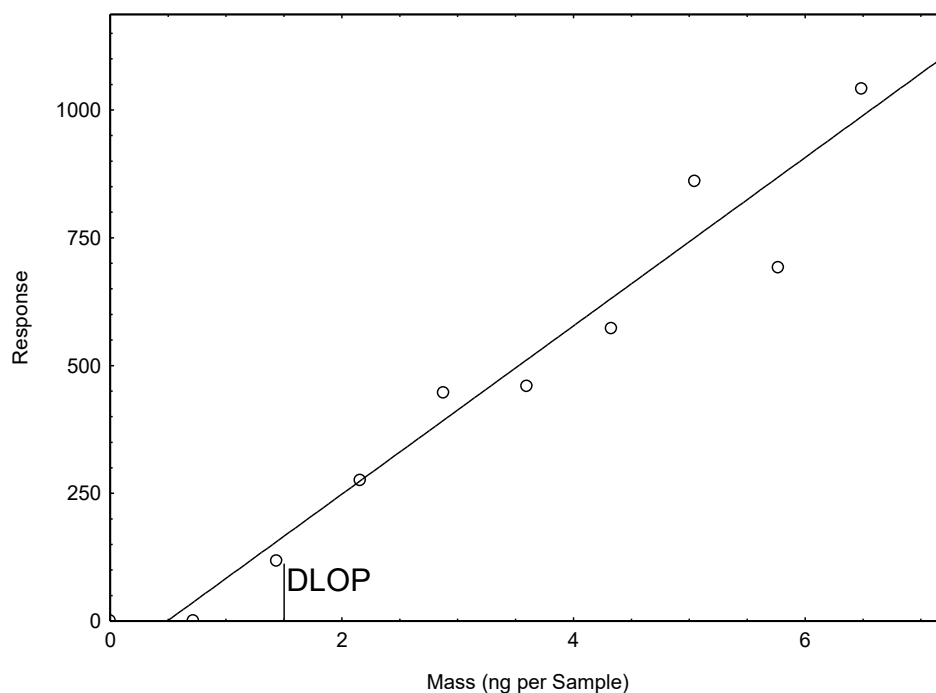


Figure 21. Plot of data used to determine the DLOP for ethyl acetate ($y = 165x - 81.0$, $S_{y/x} = 88.3$, DLOP = 1.8 ng/sample).

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Table 24. DLOP data for ethyl amyl ketone (m/z 71).

mass per sample (ng)	response
0.00	0.00
1.76	0.00
3.52	456
5.28	959
7.04	1169
8.80	1516
10.6	1914
12.3	2066
14.1	2616
15.8	3008
17.6	3275

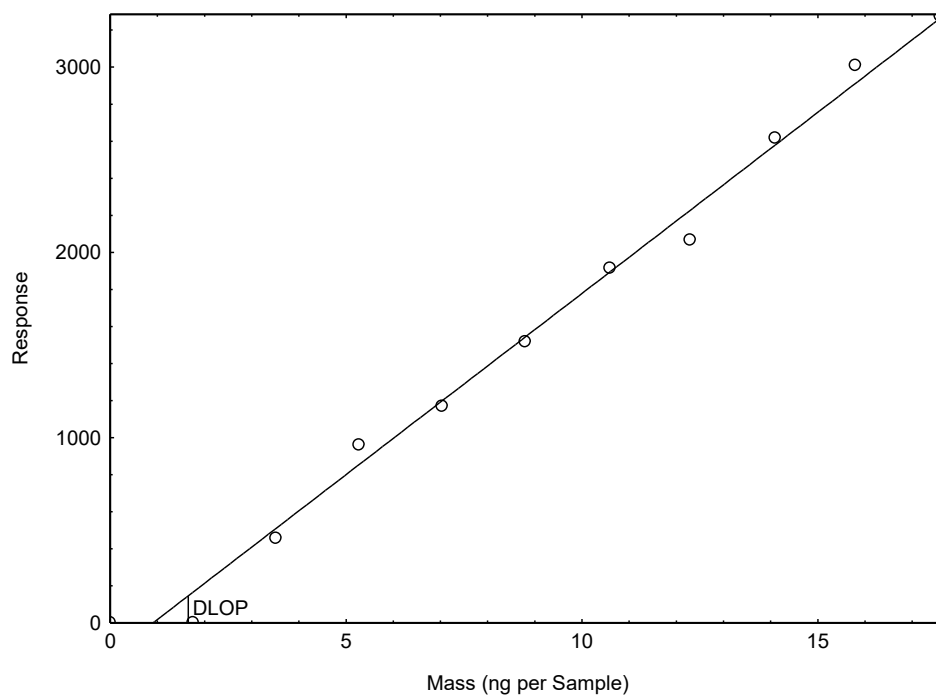


Figure 22. Plot of data used to determine the DLOP for ethyl amyl ketone ($y = 196x - 178$, $S_{y/x} = 112$, DLOP = 1.9 ng/sample).

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Table 25. DLOP data for ethyl benzene (m/z 77).

mass per sample (ng)	response
0.00	0.00
0.433	0.00
0.866	161
1.30	297
1.73	461
2.16	601
2.60	629
3.03	691
3.46	901
3.90	1149
4.33	1138

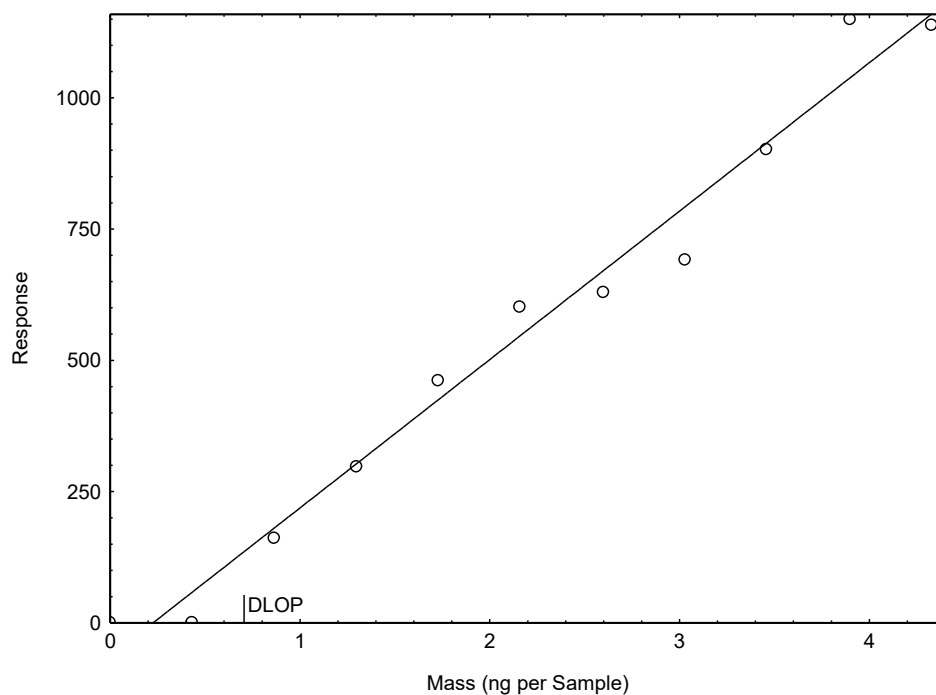


Figure 23. Plot of data used to determine the DLOP for ethyl benzene ($y = 283x - 63.8$, $S_{y/x} = 64.2$, DLOP = 0.75 ng/sample).

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Table 26. DLOP data for ethyl butyl ketone (m/z 114).

mass per sample (ng)	response
0.00	0.00
1.43	0.00
2.86	0.00
4.29	195
5.72	453
7.15	621
8.58	890
10.0	806
11.4	1141
12.9	1419
14.3	1495

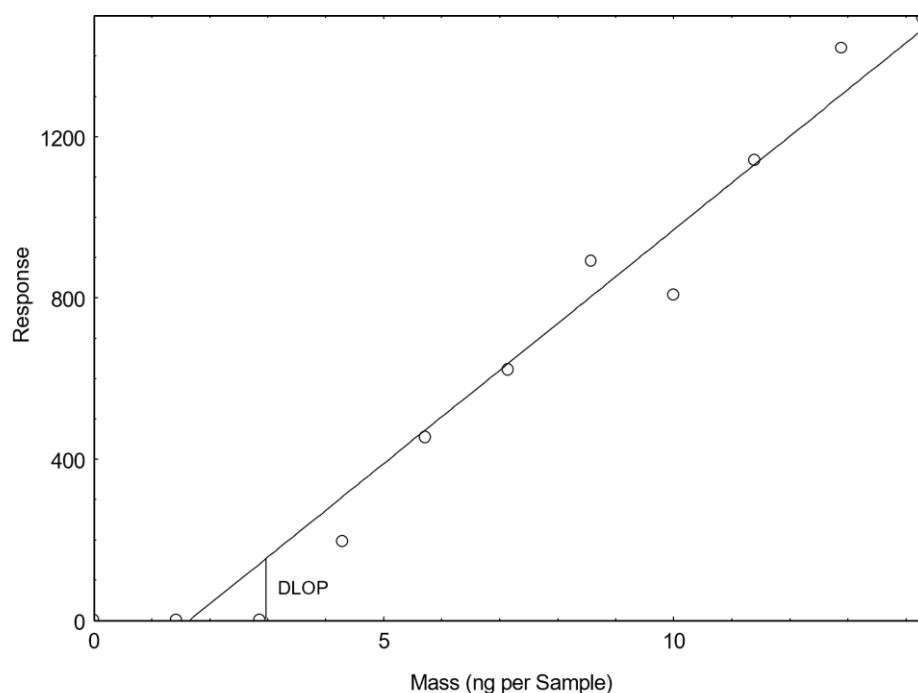


Figure 24. Plot of data used to determine the DLOP for ethyl butyl ketone ($y = 116x - 191$, $S_{y/x} = 114$, DLOP = 3.2 ng/sample).

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Table 27. DLOP data for ethylene dichloride (m/z 98).

mass per sample (ng)	response
0.00	0.00
0.502	0.00
1.00	272
1.51	381
2.01	648
2.51	641
3.01	797
3.51	1011
4.02	1203
4.52	1240
5.02	1393

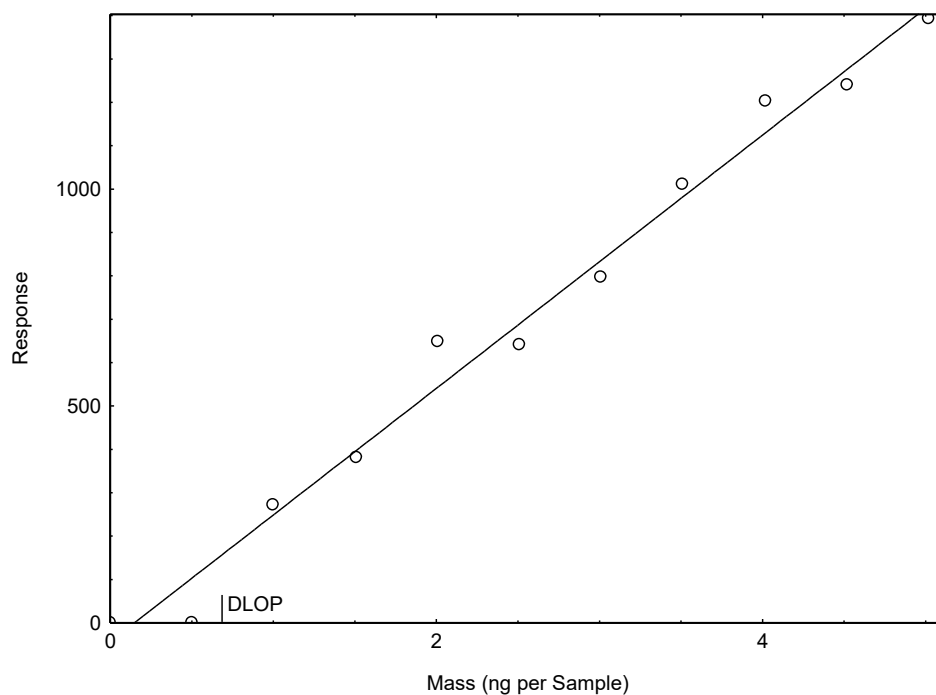


Figure 25. Plot of data used to determine the DLOP for ethylene dichloride ($y = 292x - 43.3$, $S_{y/x} = 63.6$, DLOP = 0.72 ng/sample).

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Table 28. DLOP data for *sec*-hexyl acetate (m/z 84).

mass per sample (ng)	response
0.00	0.00
1.39	0.00
2.78	309
4.17	902
5.56	1177
6.95	1255
8.34	1661
9.73	2108
11.1	2302
12.5	2922
13.9	3249

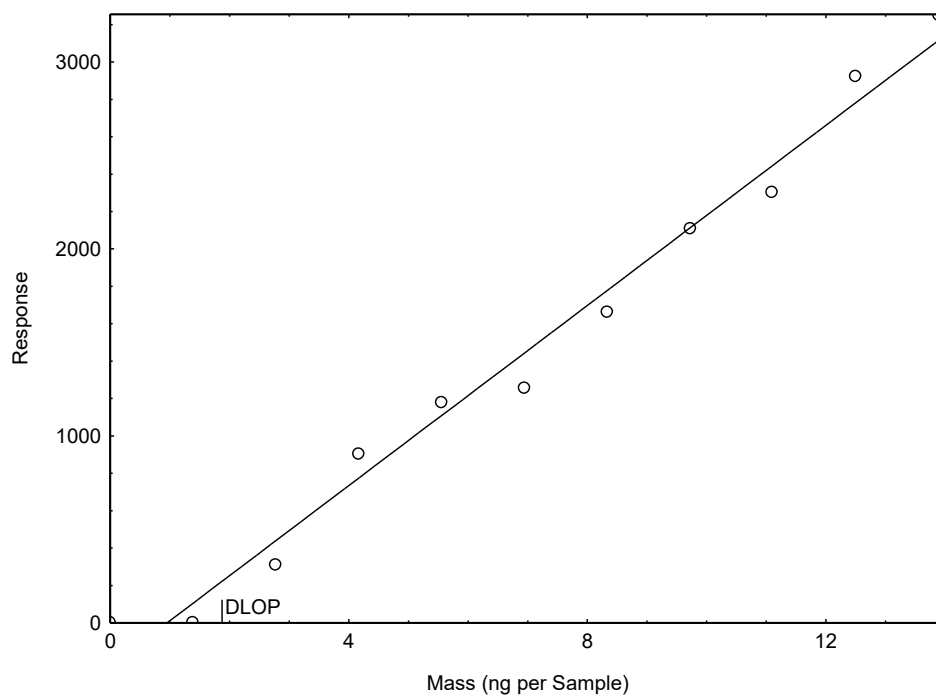


Figure 26. Plot of data used to determine the DLOP for *sec*-hexyl acetate ($y = 241x - 229$, $S_{y/x} = 152$, DLOP = 2.1 ng/sample).

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Table 29. DLOP data for isoamyl acetate (m/z 55).

mass per sample (ng)	response
0.00	0.00
1.45	81.8
2.90	791
4.35	2016
5.80	2477
7.25	2307
8.70	3822
10.2	4817
11.6	4033
13.0	6288
14.5	5803

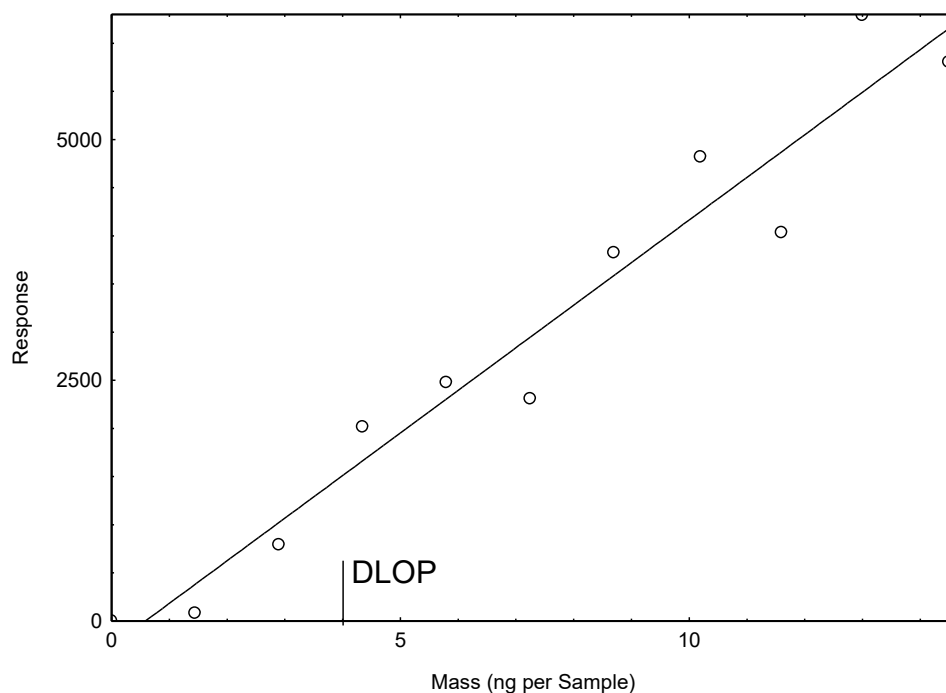


Figure 27. Plot of data used to determine the DLOP for isoamyl acetate ($y = 442x - 258$, $S_{y/x} = 538$, DLOP = 4.0 ng/sample).

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Table 30. DLOP data for isoamyl alcohol (m/z 57).

mass per sample (ng)	response
0.00	0.00
3.05	212
6.10	702
9.15	1169
12.2	2029
15.2	2330
18.3	3466
21.4	3920
24.4	4525
27.4	5815
30.5	5656

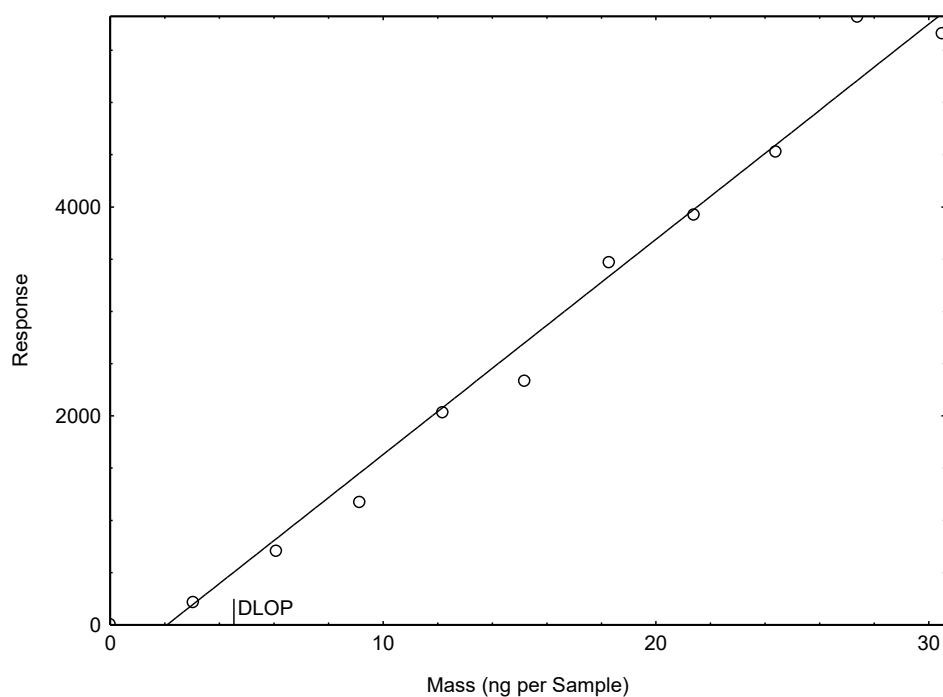


Figure 28. Plot of data used to determine the DLOP for isoamyl alcohol ($y = 206x - 429$, $Sy/x = 307$, DLOP = 4.9 ng/sample).

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Table 31. DLOP data for isobutyl acetate (m/z 73).

mass per sample (ng)	response
0.00	0.00
0.910	0.00
1.82	692
2.73	1192
3.64	1283
4.55	1493
5.46	2344
6.37	3010
7.28	2286
8.19	3740
9.10	2915

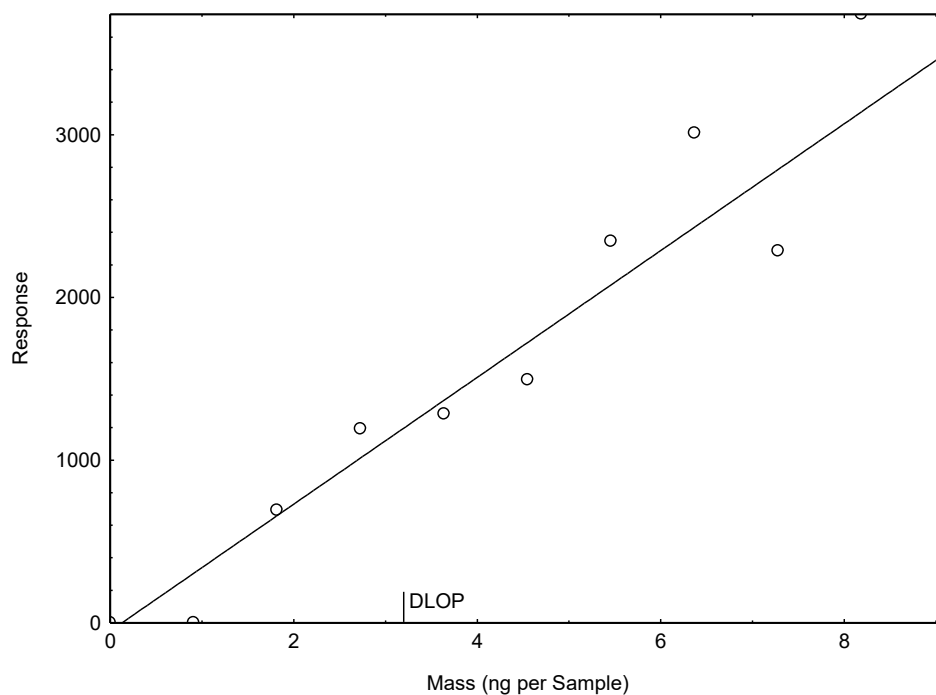


Figure 29. Plot of data used to determine the DLOP for isobutyl acetate ($y = 390x - 50.2$, $S_{y/x} = 414$, DLOP = 3.5 ng/sample).

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Table 32. DLOP data for isobutyl alcohol (m/z 74).

mass per sample (ng)	response
0.00	0.00
2.22	87.0
4.44	278
6.66	713
8.88	918
11.1	945
13.3	1313
15.5	1491
17.8	1716
20.0	1890
22.2	2050

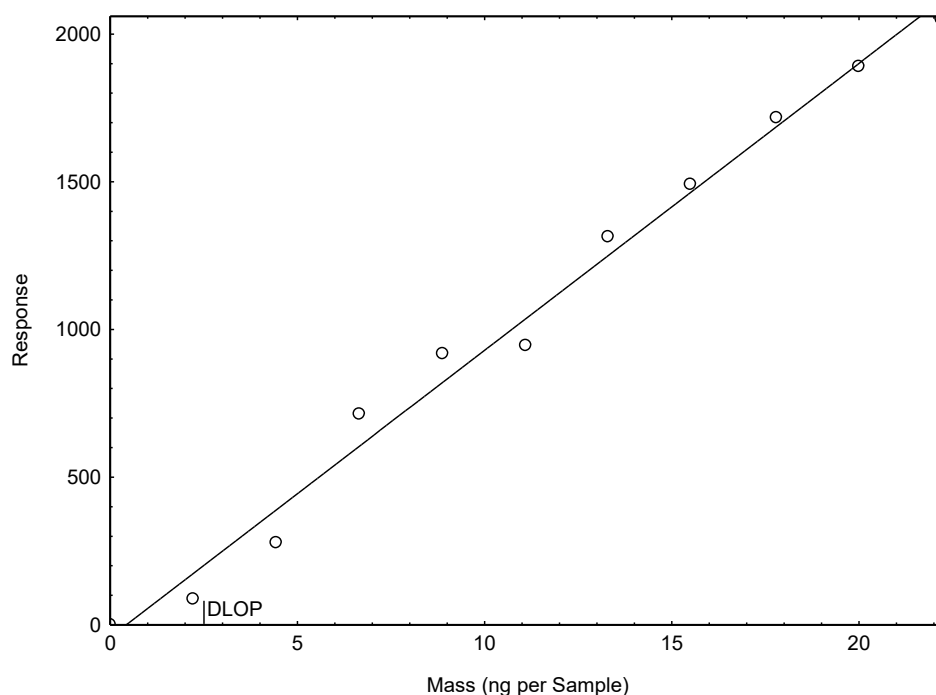


Figure 30. Plot of data used to determine the DLOP for isobutyl alcohol ($y = 97.1x - 41.6$, $S_{y/x} = 82.3$, DLOP = 2.8 ng/sample).

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Table 33. DLOP data for isopropyl acetate (m/z 87).

mass per sample (ng)	response
0.00	0.00
0.61	0.00
1.22	404
1.84	618
2.45	646
3.06	925
3.67	1584
4.28	1378
4.90	1585
5.51	1872
6.12	1975

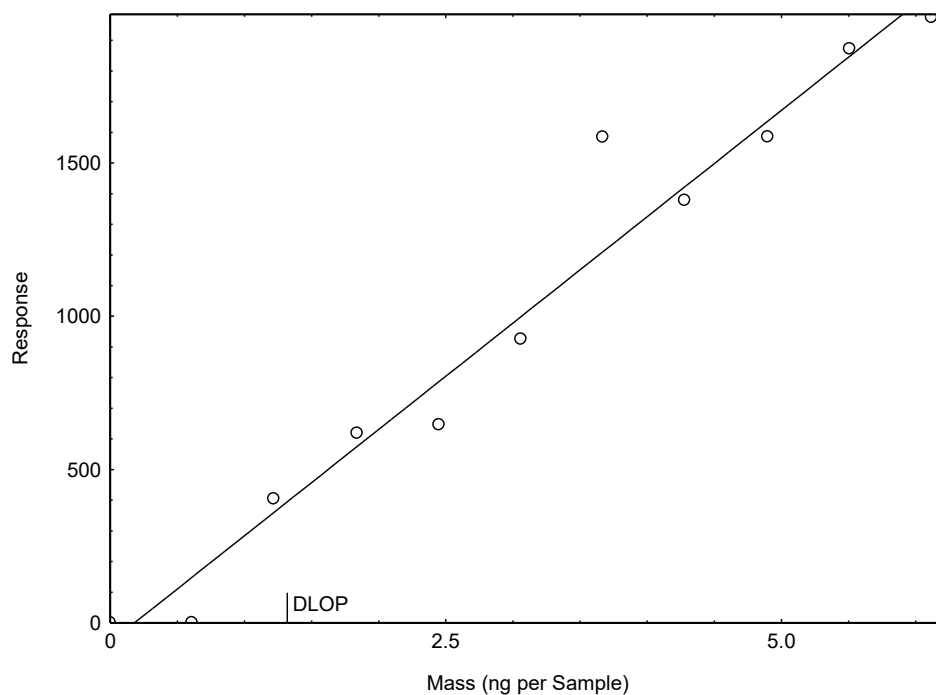


Figure 31. Plot of data used to determine the DLOP for isopropyl acetate ($y = 347x - 62.8$, $S_{y/x} = 152$, DLOP = 1.4 ng/sample).

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Table 34. DLOP data for 2-methoxyethyl acetate (m/z 73).

mass per sample (ng)	response
0.00	0.00
18.4	0.00
36.8	0.00
55.2	248
73.6	284
92.0	781
110	915
129	1805
147	2332
166	2454
184	2629

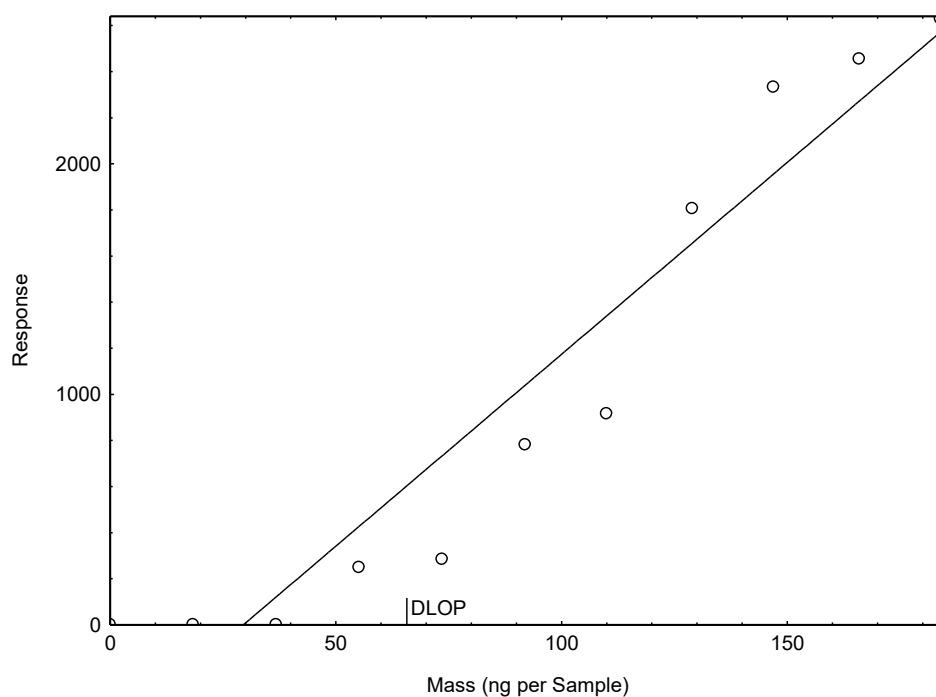


Figure 32. Plot of data used to determine the DLOP for 2-methoxyethyl acetate ($y = 16.6x - 491$, $S_{y/x} = 329$, DLOP = 65 ng/sample).

OSHA Method 1031, Qualitative Screening for Volatile Organic Compounds Collected on Thermal Desorption Tubes

Table 35. DLOP data for methyl isoamyl ketone (m/z 71).

mass per sample (ng)	response
0.00	0.00
1.09	0.00
2.18	0.00
3.27	364
4.36	421
5.45	512
6.54	635
7.63	684
8.72	972
9.81	1006
10.9	1097

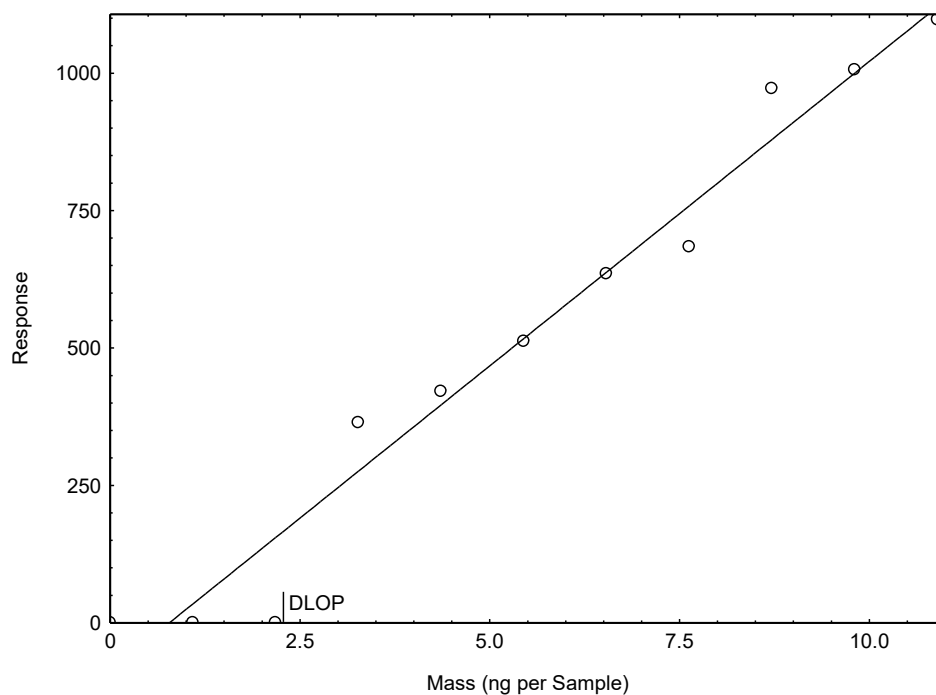


Figure 33. Plot of data used to determine the DLOP for methyl isoamyl ketone ($y = 111x - 86.2$, $S_{y/x} = 78.8$, DLOP = 2.3 ng/sample).

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Table 36. DLOP data for methyl isobutyl ketone (m/z 100).

mass per sample (ng)	response
0.00	0.00
0.539	0.00
1.08	158
1.62	501
2.16	458
2.70	799
3.23	773
3.77	908
4.31	1099
4.85	1128
5.39	1366

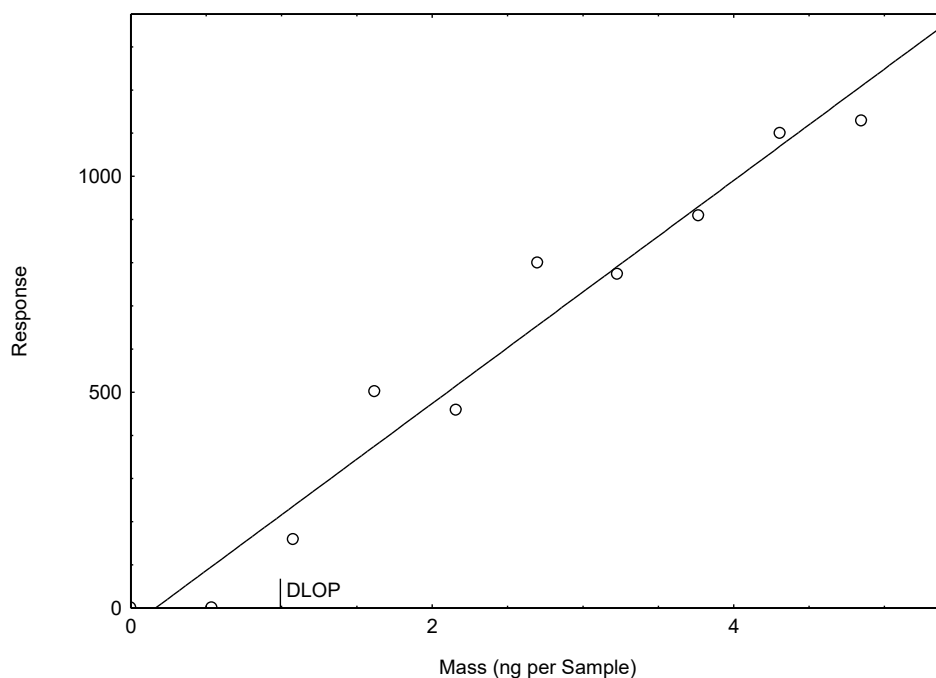


Figure 34. Plot of data used to determine the DLOP for methyl isobutyl ketone ($y = 258x - 42.1$, $S_{y/x} = 85.5$, DLOP = 1.1 ng/sample).

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Table 37. DLOP data for methylene chloride (m/z 51).

mass per sample (ng)	response
0.00	0.00
0.497	0.00
0.994	379
1.49	667
1.99	1178
2.48	1343
2.98	1801
3.48	1797
3.98	2002
4.47	2225
4.97	2426

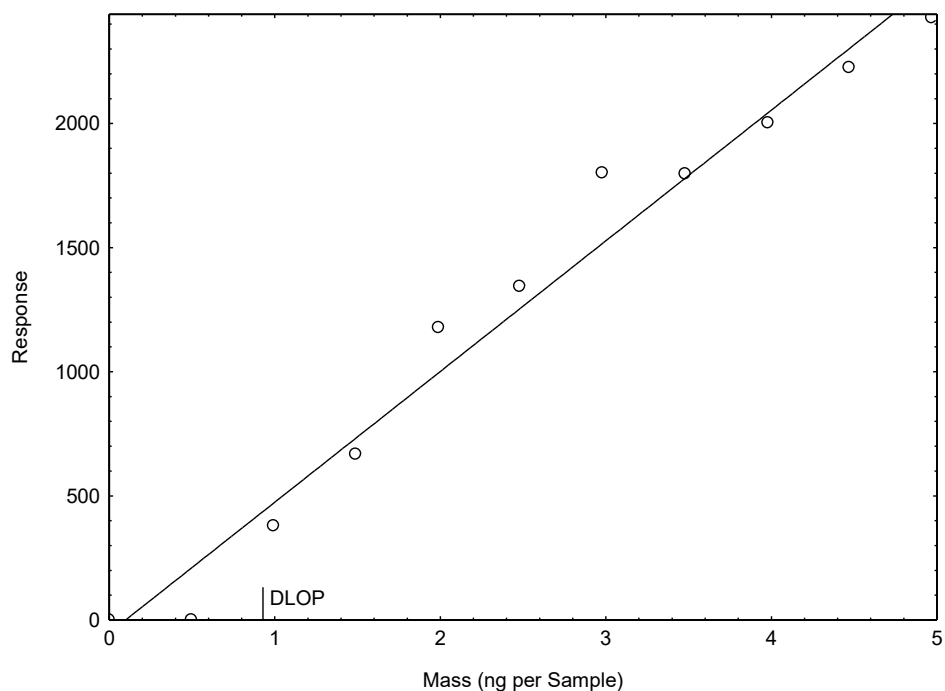


Figure 35. Plot of data used to determine the DLOP for methylene chloride ($y = 526x - 51.9$, $S_{y/x} = 152$, DLOP = 0.95 ng/sample).

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Table 38. DLOP data for α -pinene (m/z 136).

mass per sample (ng)	response
0.00	0.00
1.69	0.00
3.38	36.0
5.07	148
6.76	278
8.45	387
10.1	623
11.8	771
13.5	811
15.2	968
16.9	1004

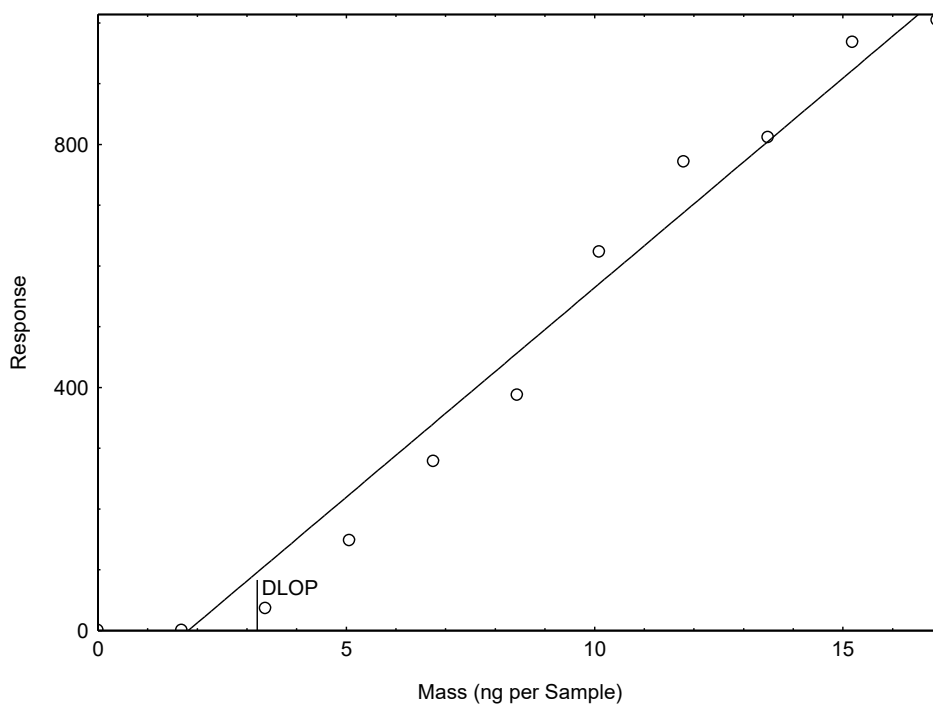


Figure 36. Plot of data used to determine the DLOP for α -pinene ($y = 69x - 125$, $S_{y/x} = 73.4$, DLOP = 3.5 ng/sample).

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Table 39. DLOP data for *n*-propyl alcohol (*m/z* 60).

mass per sample (ng)	response
0.00	0.00
0.644	193
1.29	406
1.93	438
2.58	705
3.22	608
3.86	962
4.51	1159
5.15	1320
5.80	1426
6.44	1451

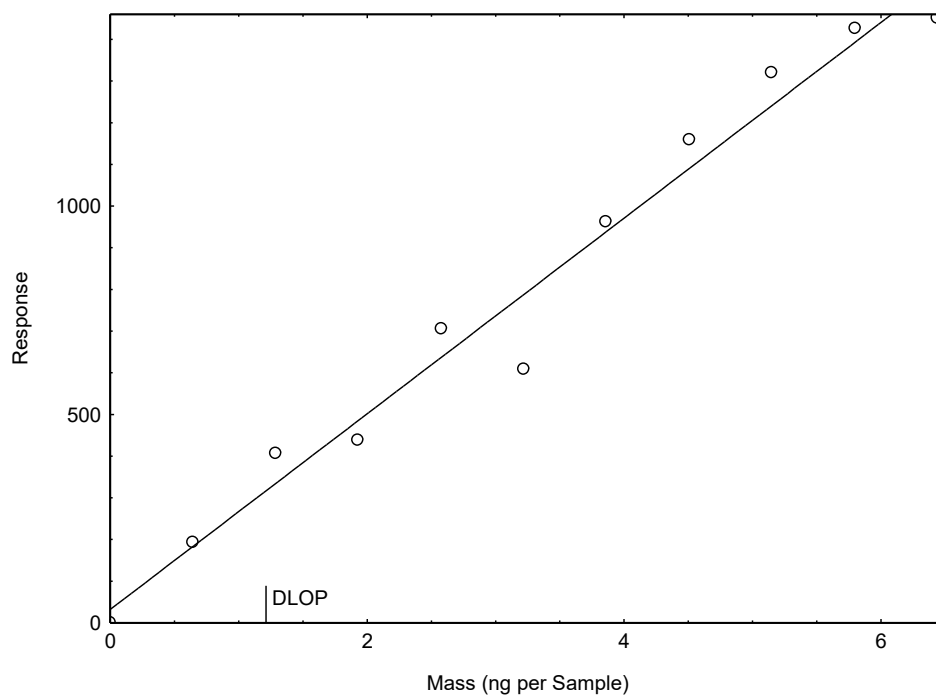


Figure 37. Plot of data used to determine the DLOP for *n*-propyl alcohol ($y = 235x + 32.1$, $S_{y/x} = 85.9$, DLOP = 1.2 ng/sample).

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Table 40. DLOP data for tetrachloroethylene (m/z 133).

mass per sample (ng)	response
0.00	0.00
0.365	0.00
0.730	200
1.10	499
1.46	513
1.82	766
2.19	1185
2.56	1287
2.92	1511
3.28	1538
3.65	1718

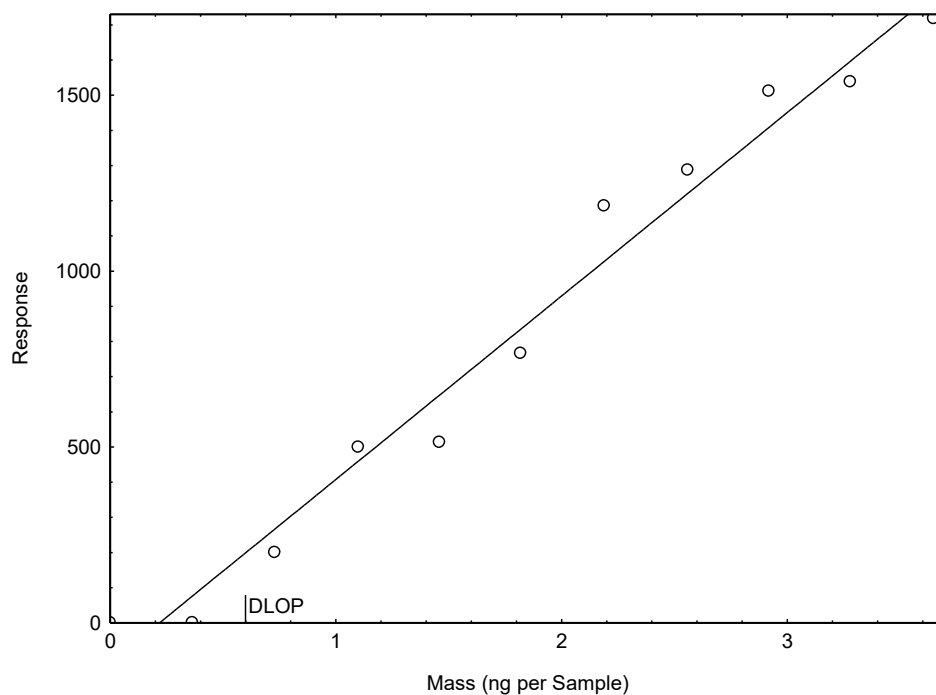


Figure 38. Plot of data used to determine the DLOP for tetrachloroethylene ($y = 521x - 114$, $S_{y/x} = 103$, DLOP = 0.65 ng/sample).

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Table 41. DLOP data for 1,1,2-trichloroethane (m/z 132).

mass per sample (ng)	response
0.00	0.00
0.425	0.00
0.850	0.00
1.28	141
1.70	291
2.12	379
2.55	643
2.98	669
3.40	768
3.82	868
4.25	947

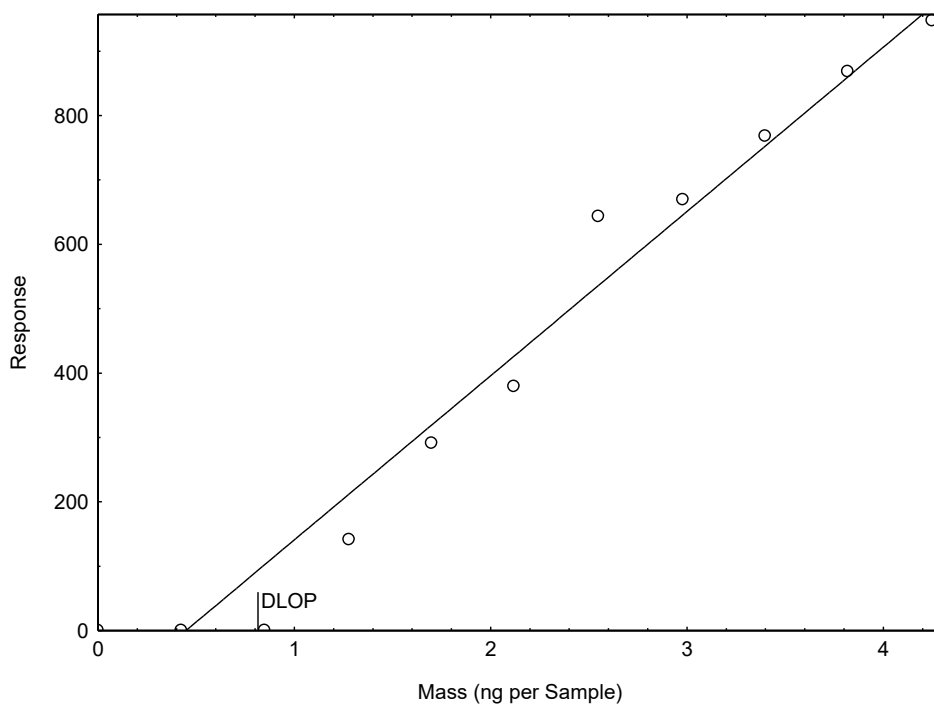


Figure 39. Plot of data used to determine the DLOP for 1,1,2-trichloroethane ($y = 255x - 114$, $S_{y/x} = 70.3$, DLOP = 0.91 ng/sample).

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Table 42. DLOP data for trichloroethylene (m/z 134).

mass per sample (ng)	response
0.00	0.00
0.256	38.0
0.512	279
0.768	588
1.02	793
1.28	867
1.54	1149
1.79	1396
2.05	1471
2.30	1439
2.56	1639

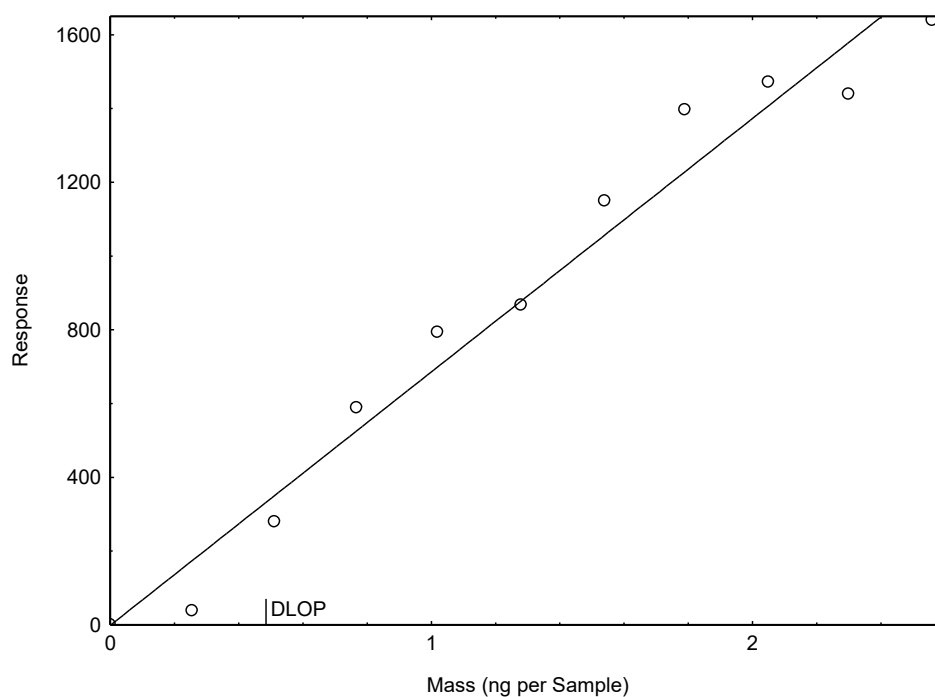


Figure 40. Plot of data used to determine the DLOP for trichloroethylene ($y = 687x - 1.32$, $S_{y/x} = 111$, DLOP = 0.53 ng/sample).

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Table 43. DLOP data for 1,3,5-trimethylbenzene (m/z 91).

mass per sample (ng)	response
0.00	0.00
0.857	0.00
1.71	208
2.57	328
3.43	532
4.28	775
5.14	901
6.00	1037
6.86	1292
7.71	1329
8.57	1635

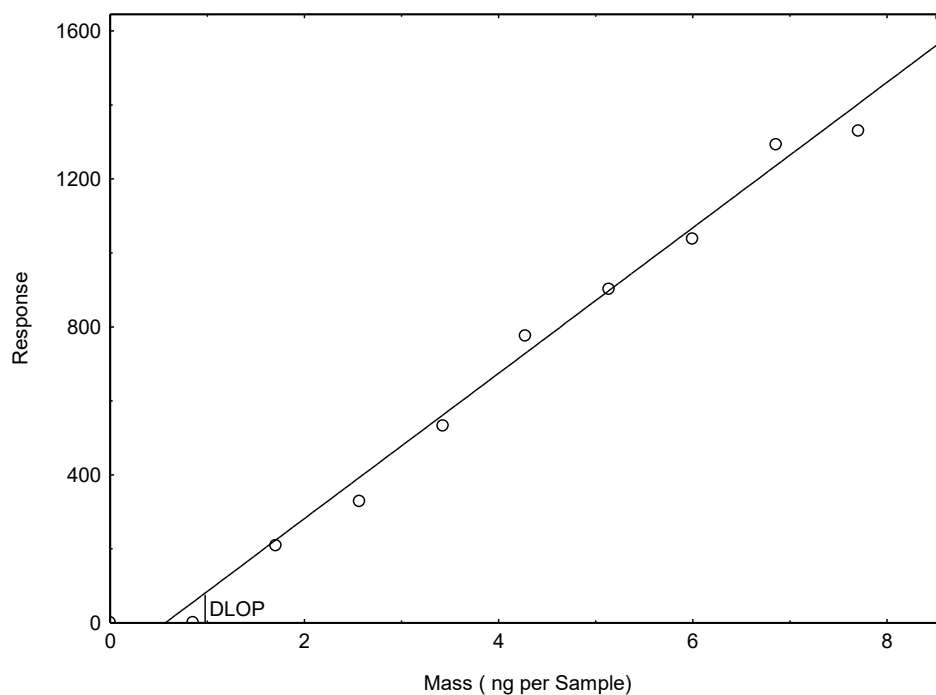


Figure 41. Plot of data used to determine the DLOP for 1,3,5-trimethylbenzene ($y = 197x - 111$, $S_{y/x} = 63.9$, DLOP = 1.1 ng/sample).

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Table 44. DLOP data for 1,2,4-trimethylbenzene (m/z 91).

mass per sample (ng)	response
0.00	0.00
0.865	0.00
1.73	293
2.60	373
3.46	500
4.32	736
5.19	1001
6.06	1175
6.92	1330
7.78	1549
8.65	1779

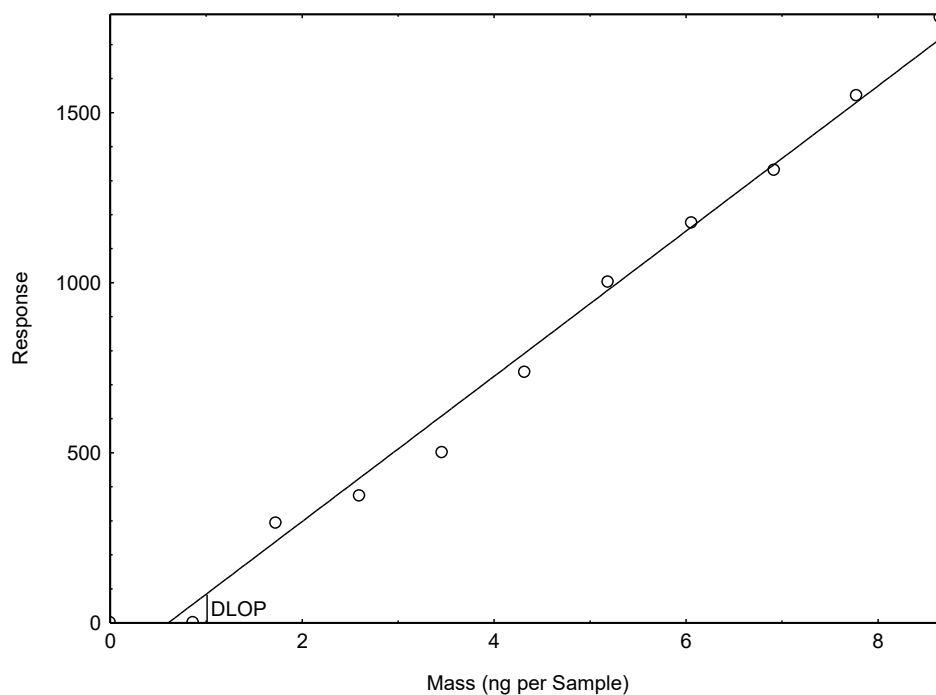


Figure 42. Plot of data used to determine the DLOP for 1,2,4-trimethylbenzene ($y = 213x - 129$, $S_{y/x} = 71.2$, DLOP = 1.1 ng/sample).

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Table 45. DLOP data for 1,2,3-trimethylbenzene (m/z 91).

mass per sample (ng)	response
0.00	0.00
0.956	0.00
1.91	156
2.87	457
3.82	566
4.78	687
5.74	884
6.69	1051
7.65	1237
8.60	1299
9.56	1650

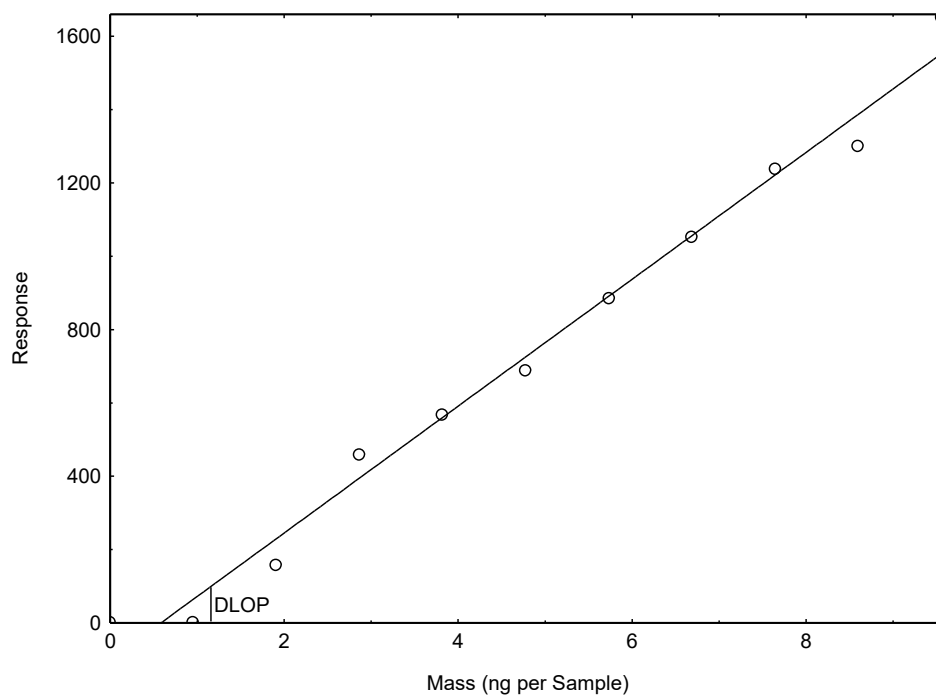


Figure 43. Plot of data used to determine the DLOP for 1,2,3-trimethylbenzene ($y = 173x - 101$, $S_{y/x} = 68.7$, DLOP = 1.3 ng/sample).

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Table 46. DLOP data for *m*-xylene (*m/z* 77).

mass per sample (ng)	response
0.00	0.00
0.429	0.00
0.858	0.00
1.29	363
1.72	334
2.14	632
2.57	657
3.00	843
3.43	822
3.86	986
4.29	1222

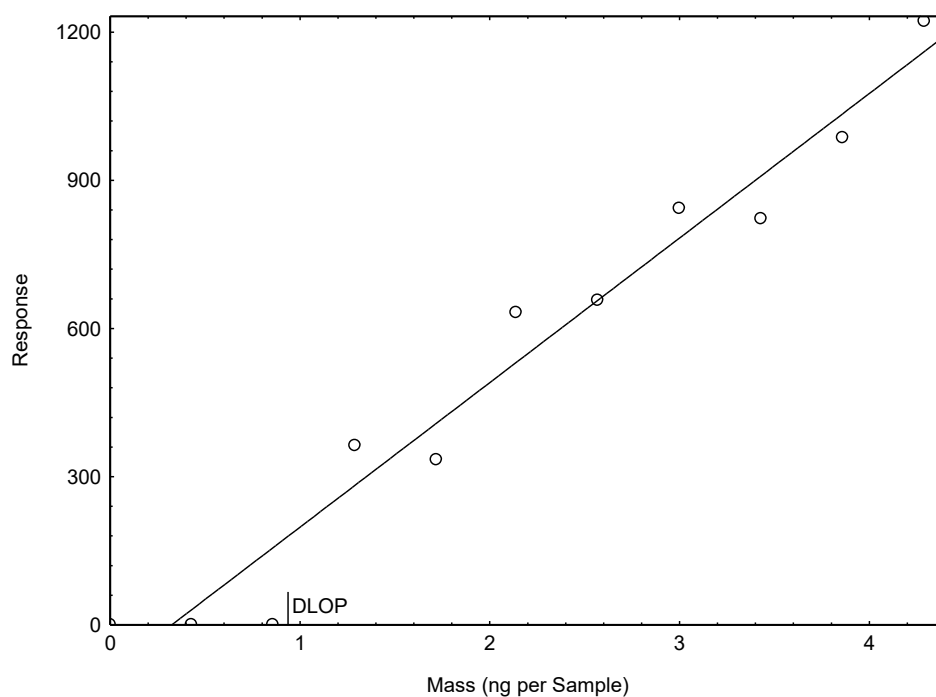


Figure 44. Plot of data used to determine the DLOP for *m*-xylene ($y = 293x - 94.9$, $S_{y/x} = 90.5$, DLOP = 1.0 ng/sample).

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Table 47. DLOP data for *o*-xylene (*m/z* 77).

mass per sample (ng)	response
0.00	0.00
0.440	0.00
0.880	34.5
1.32	83.7
1.76	432
2.20	582
2.64	521
3.08	674
3.52	786
3.96	945
4.40	1086

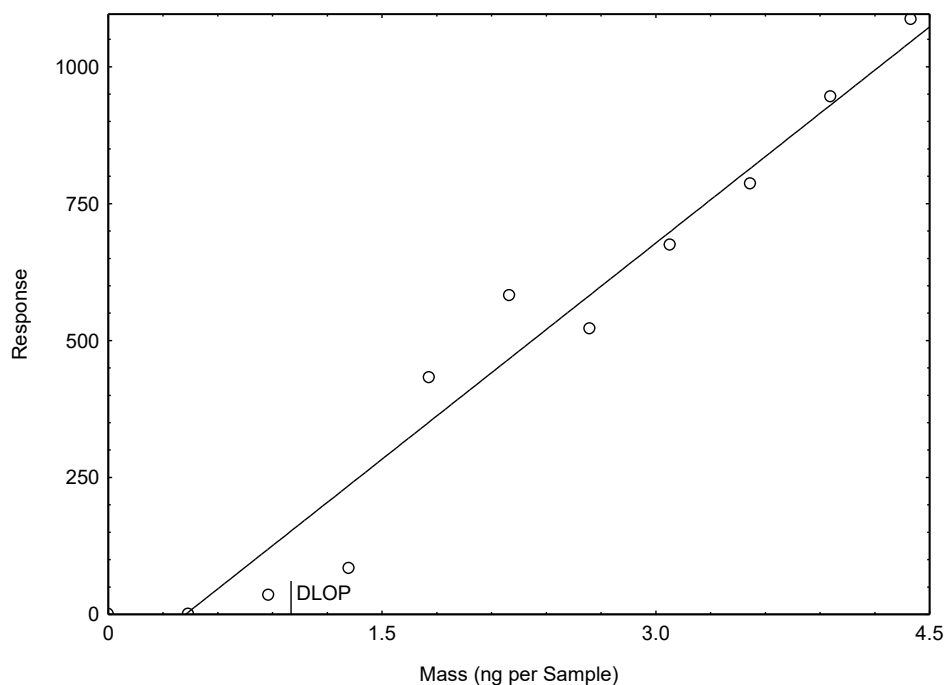


Figure 45. Plot of data used to determine the DLOP for *o*-xylene ($y = 263x - 111$, $S_{y/x} = 87.9$, DLOP = 1.1 ng/sample).

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Table 48. DLOP data for *p*-xylene (*m/z* 77).

mass per sample (ng)	response
0.00	0.00
0.866	0.00
1.73	287
2.60	471
3.46	744
4.33	1052
5.20	1142
6.06	1300
6.93	1489
7.79	1842
8.66	1886

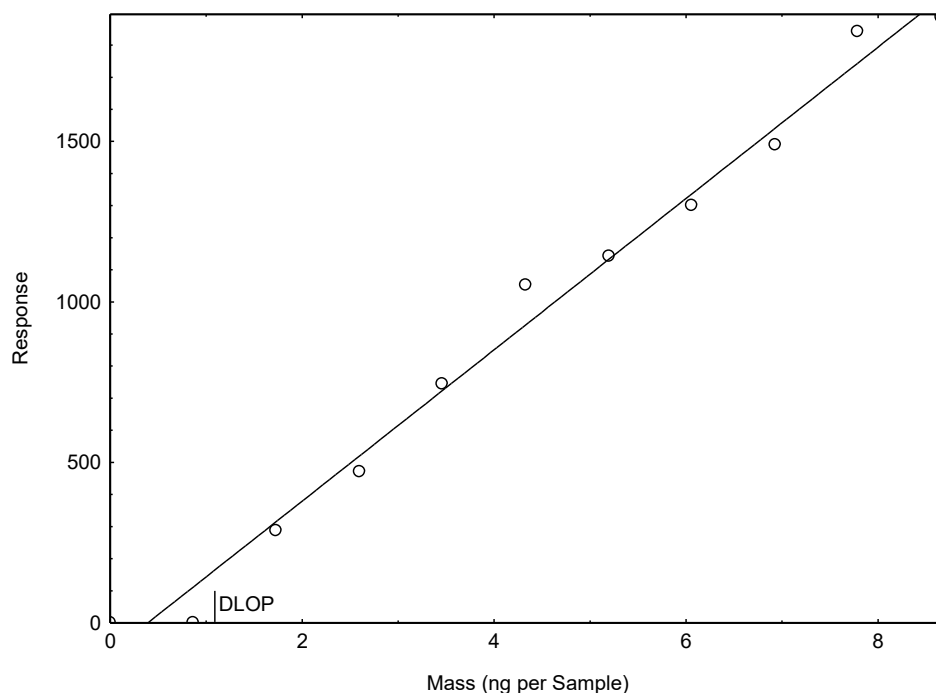


Figure 46. Plot of data used to determine the DLOP for *p*-xylene ($y = 236x - 92.4$, $S_{y/x} = 80.1$, DLOP = 1.1 ng/sample).

4.2 Storage Stability

Sealing TD tubes with long-term brass storage caps combined with PTFE ferrules can minimize external contamination and sample loss over storage periods up to 27 months.² However, storage stability evaluations were conducted to ensure the TD tube storage stability meets the requirements of daily sampling operations.

4.2.1 Long-Term Storage Stability of Sealed Sampling Tubes

Ten material emissions TD tubes were conditioned and screened immediately before long-term storage using brass caps and PTFE ferrules. All tubes were stored under ambient conditions for seven months. Compared with the initial screening result of each sample tube, results after the long-term storage as shown in Figure 47 indicated that long-term storage caps prevent contaminations from the external environment for up to seven months.

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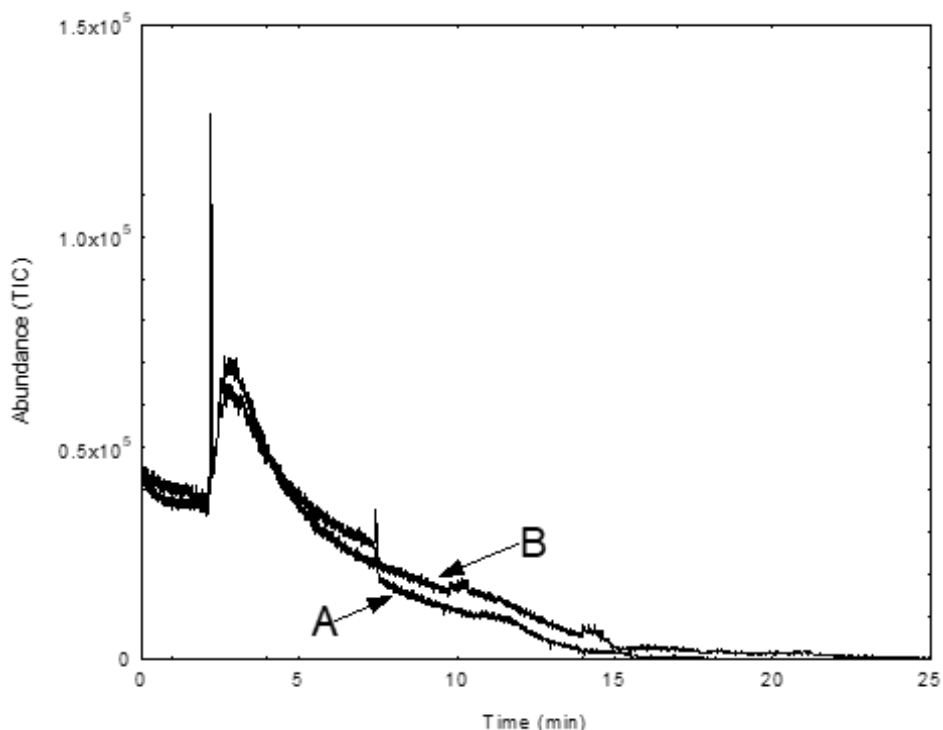


Figure 47. Response from one of ten TD tubes analyzed (A) immediately after conditioning and (B) after storage for 212 days.

4.2.2 Post-Sampling Storage Stability

Twelve samples were collected by spiking an amount equivalent to a 100-mL sampling volume of an analyte at 0.5 ppm or a 500-mL sampling volume at 0.1 ppm. The maximum sampling volume (500 mL) of humid air with 80% relative humidity at 22 °C was drawn through each sample. Three samples were analyzed immediately after sampling. The remaining samples were placed on a bench at ambient temperature (about 22 °C). Three samples were selected and analyzed at the intervals noted below in Tables 49 through 111. The recovery of each sample was evaluated by dividing the response area of a significant characteristic ion peak by the mean response area of corresponding ion peaks of three samples on Day 0. Results listed in Tables 49 through 111 indicate that reliable mass spectra of all tested analytes were obtained within 10 days. However, it is recommended to return the sampling kit by the end of the sampling day to avoid unexpected sample loss.

Table 49. Ambient sampler storage stability data for acetone (m/z 58).

time (days)	sample 1 (%)	sample 2 (%)	sample 3 (%)
0	97.0	98.9	104.1
3	99.6	99.3	98.1
6	99.6	105.9	98.1
10	106.5	105.2	101.2

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Table 50. Ambient sampler storage stability data for acetonitrile (m/z 41).

time (days)	sample 1 (%)	sample 2 (%)	sample 3 (%)
0	99.7	105.2	95.1
3	109.4	110.4	111.1
7	116.6	107.5	129.8
10	99.1	108.1	118.3

Table 51. Ambient sampler storage stability data for allyl alcohol (m/z 57).

time (days)	sample 1 (%)	sample 2 (%)	sample 3 (%)
0	102.8	99.6	97.6
3	70.4	103.8	76.9
7	103.2	109.8	108.4
10	99.9	107.1	109.0

Table 52. Ambient sampler storage stability data for *n*-amyl acetate (m/z 70).

time (days)	sample 1 (%)	sample 2 (%)	sample 3 (%)
0	100.1	98.1	101.7
3	100.6	101.4	103.4
7	98.9	99.1	97.0
10	100.6	102.2	105.0

Table 53. Ambient sampler storage stability data for benzene (m/z 78).

time (days)	sample 1 (%)	sample 2 (%)	sample 3 (%)
0	98.5	98.1	103.4
3	105.0	103.9	101.3
7	105.5	101.9	102.2
10	100.9	103.2	99.5

Table 54. Ambient sampler storage stability data for benzyl chloride (m/z 91).

time (days)	sample 1 (%)	sample 2 (%)	sample 3 (%)
0	96.1	104.2	99.8
4	98.5	101.9	111.4
7	94.1	97.1	101.6
11	90.8	94.8	96.9

Table 55. Ambient sampler storage stability data for 1-bromopropane (m/z 122).

time (days)	sample 1 (%)	sample 2 (%)	sample 3 (%)
0	100.7	99.6	99.7
3	96.5	94.0	94.2
7	97.8	96.2	93.8
10	94.9	91.6	92.0

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Table 56. Ambient sampler storage stability data for 2-butoxyethanol (*m/z* 57).

time (days)	sample 1 (%)	sample 2 (%)	sample 3 (%)
0	100.0	94.9	105.2
3	96.7	112.9	99.1
6	116.2	113.7	117.1
10	259.0	285.6	258.4

Table 57. Ambient sampler storage stability data for *n*-butyl acetate (*m/z* 56).

time (days)	sample 1 (%)	sample 2 (%)	sample 3 (%)
0	95.5	98.8	105.6
3	95.8	102.4	107.9
7	95.1	101.1	102.7
10	99.1	105.9	111.1

Table 58. Ambient sampler storage stability data for *sec*-butyl acetate (*m/z* 56).

time (days)	sample 1 (%)	sample 2 (%)	sample 3 (%)
0	97.7	98.5	103.8
3	109.9	110.2	110.2
7	99.6	100.7	100.7
10	111.7	111.9	114.3

Table 59. Ambient sampler storage stability data for *tert*-butyl acetate (*m/z* 57).

time (days)	sample 1 (%)	sample 2 (%)	sample 3 (%)
0	103.6	86.7	109.8
3	105.9	111.6	114.1
7	107.3	113.0	99.9
10	101.6	121.4	120.6

Table 60. Ambient sampler storage stability data for *n*-butyl alcohol (*m/z* 74).

time (days)	sample 1 (%)	sample 2 (%)	sample 3 (%)
0	100.4	100.6	99.0
3	110.1	105.9	105.3
7	103.2	108.0	109.6
10	105.5	105.7	110.0

Table 61. Ambient sampler storage stability data for *sec*-butyl alcohol (*m/z* 59).

time (days)	sample 1 (%)	sample 2 (%)	sample 3 (%)
0	100.8	101.3	97.9
3	101.0	105.2	102.1
7	105.0	106.8	118.1
10	108.6	114.9	120.2

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Table 62. Ambient sampler storage stability data for *tert*-butyl alcohol (*m/z* 59).

time (days)	sample 1 (%)	sample 2 (%)	sample 3 (%)
0	105.3	98.3	96.5
3	93.3	99.7	98.0
7	101.6	108.4	109.9
10	107.2	113.2	114.3

Table 63. Ambient sampler storage stability data for carbon tetrachloride (*m/z* 117).

time (days)	sample 1 (%)	sample 2 (%)	sample 3 (%)
0	100.4	100.3	99.3
3	97.8	98.5	97.7
7	95.7	96.5	94.5
10	96.6	92.5	91.0

Table 64. Ambient sampler storage stability data for chlorobenzene (*m/z* 112).

time (days)	sample 1 (%)	sample 2 (%)	sample 3 (%)
0	97.5	100.2	102.2
3	104.7	97.8	100.5
7	106.1	105.4	111.3
10	103.8	105.6	104.1

Table 65. Ambient sampler storage stability data for chlorobromomethane (*m/z* 130).

time (days)	sample 1 (%)	sample 2 (%)	sample 3 (%)
0	100.9	98.9	100.2
3	98.1	98.1	97.2
7	97.3	96.0	93.4
10	96.7	92.1	92.8

Table 66. Ambient sampler storage stability data for chloroform (*m/z* 83).

time (days)	sample 1 (%)	sample 2 (%)	sample 3 (%)
0	98.6	97.7	103.7
3	95.3	96.5	94.2
6	95.8	93.0	95.0
10	103.0	104.1	99.2

Table 67. Ambient sampler storage stability data for cyclohexane (*m/z* 84).

time (days)	sample 1 (%)	sample 2 (%)	sample 3 (%)
0	91.6	97.4	111.0
3	102.0	89.9	68.1
7	109.1	89.7	82.5
10	100.7	102.2	112.0

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Table 68. Ambient sampler storage stability data for cyclohexanone (*m/z* 55).

time (days)	sample 1 (%)	sample 2 (%)	sample 3 (%)
0	97.6	98.0	104.4
3	95.2	101.8	98.0
6	99.7	99.5	99.7
10	105.7	106.9	103.6

Table 69. Ambient sampler storage stability data for diacetone alcohol (*m/z* 58).

time (days)	sample 1 (%)	sample 2 (%)	sample 3 (%)
0	106.0	97.7	96.3
3	133.1	395.9	161.6
6	595.8	492.2	346.5
10	222.2	352.1	218.7

Table 70. Ambient sampler storage stability data for 1,1-dichloroethane (*m/z* 63).

time (days)	sample 1 (%)	sample 2 (%)	sample 3 (%)
0	101.0	99.0	100.0
3	98.7	97.5	97.9
7	98.7	97.6	96.5
10	99.7	96.2	97.1

Table 71. Ambient sampler storage stability data for 1,2-dichlorobenzene (*m/z* 146).

time (days)	sample 1 (%)	sample 2 (%)	sample 3 (%)
0	99.9	102.5	97.6
4	107.1	106.4	109.4
7	103.1	103.4	105.3
11	102.9	103.5	105.2

Table 72. Ambient sampler storage stability data for diisobutyl ketone (*m/z* 57).

time (days)	sample 1 (%)	sample 2 (%)	sample 3 (%)
0	93.4	96.7	109.9
4	115.4	117.7	116.3
7	119.0	116.7	118.1
11	108.6	116.3	112.1

Table 73. Ambient sampler storage stability data for 2-ethoxyethyl acetate (*m/z* 72).

time (days)	sample 1 (%)	sample 2 (%)	sample 3 (%)
0	98.8	97.3	103.9
3	100.8	106.8	110.6
7	104.4	108.8	108.4
10	105.6	112.6	116.4

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Table 74. Ambient sampler storage stability data for epichlorohydrin (*m/z* 57).

time (days)	sample 1 (%)	sample 2 (%)	sample 3 (%)
0	98.3	100.5	101.2
3	94.2	94.2	92.7
7	79.9	89.3	95.1
10	92.7	83.0	86.5

Table 75. Ambient sampler storage stability data for ethyl acetate (*m/z* 61).

time (days)	sample 1 (%)	sample 2 (%)	sample 3 (%)
0	97.6	98.3	104.1
3	100.1	104.6	103.6
7	96.8	96.9	96.5
10	101.2	102.7	106.2

Table 76. Ambient sampler storage stability data for ethyl alcohol (*m/z* 46).

time (days)	sample 1 (%)	sample 2 (%)	sample 3 (%)
0	100.8	109.4	89.8
3	87.1	93.6	92.7
7	92.7	83.6	86.1
10	70.7	91.3	106.5

Table 77. Ambient sampler storage stability data for ethyl amyl ketone (*m/z* 57).

time (days)	sample 1 (%)	sample 2 (%)	sample 3 (%)
0	96.7	97.7	105.7
3	92.2	108.0	104.4
6	106.6	107.7	108.1
10	107.9	111.3	107.2

Table 78. Ambient sampler storage stability data for ethyl benzene (*m/z* 91).

time (days)	sample 1 (%)	sample 2 (%)	sample 3 (%)
0	98.2	100.0	101.8
3	101.9	95.6	98.8
7	101.8	102.8	105.4
10	102.9	103.6	101.8

Table 79. Ambient sampler storage stability data for ethyl butyl ketone (*m/z* 57).

time (days)	sample 1 (%)	sample 2 (%)	sample 3 (%)
0	95.2	98.0	106.8
3	107.6	113.3	109.5
6	114.1	111.9	113.6
10	108.3	112.7	109.1

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Table 80. Ambient sampler storage stability data for ethylene dichloride (*m/z* 62).

time (days)	sample 1 (%)	sample 2 (%)	sample 3 (%)
0	100.5	99.9	99.6
3	97.7	98.1	97.2
7	102.3	101.8	96.7
10	101.0	125.3	97.7

Table 81. Ambient sampler storage stability data for *n*-heptane (*m/z* 71).

time (days)	sample 1 (%)	sample 2 (%)	sample 3 (%)
0	92.3	96.6	111.1
3	102.4	90.9	66.3
7	110.0	89.8	81.4
10	97.5	98.5	108.5

Table 82. Ambient sampler storage stability data for *n*-hexane (*m/z* 86).

time (days)	sample 1 (%)	sample 2 (%)	sample 3 (%)
0	92.1	96.3	111.7
3	103.5	91.8	71.1
7	111.8	92.1	86.8
10	101.0	102.4	111.1

Table 83. Ambient sampler storage stability data for *sec*-hexyl acetate (*m/z* 69).

time (days)	sample 1 (%)	sample 2 (%)	sample 3 (%)
0	100.7	98.1	101.2
3	100.6	100.5	103.6
7	96.7	96.2	95.7
10	99.2	100.6	104.1

Table 84. Ambient sampler storage stability data for isoamyl acetate (*m/z* 70).

time (days)	sample 1 (%)	sample 2 (%)	sample 3 (%)
0	99.4	97.3	103.3
3	97.4	99.0	103.1
7	96.9	99.9	99.9
10	100.7	102.9	106.6

Table 85. Ambient sampler storage stability data for isoamyl alcohol (*m/z* 70).

time (days)	sample 1 (%)	sample 2 (%)	sample 3 (%)
0	104.5	99.2	96.3
3	87.1	95.3	93.6
7	95.1	99.0	100.4
10	103.3	108.1	107.6

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Table 86. Ambient sampler storage stability data for isobutyl acetate (*m/z* 56).

time (days)	sample 1 (%)	sample 2 (%)	sample 3 (%)
0	97.7	97.2	105.1
3	104.8	105.8	108.6
7	98.9	100.0	101.3
10	108.6	106.5	113.1

Table 87. Ambient sampler storage stability data for isobutyl alcohol (*m/z* 74).

time (days)	sample 1 (%)	sample 2 (%)	sample 3 (%)
0	98.9	100.5	100.6
3	102.4	99.1	95.9
7	97.8	94.2	92.5
10	104.1	105.4	103.8

Table 88. Ambient sampler storage stability data for isopropyl acetate (*m/z* 61).

time (days)	sample 1 (%)	sample 2 (%)	sample 3 (%)
0	94.7	98.9	106.4
3	98.9	105.4	108.0
7	99.1	102.9	104.3
10	104.5	108.5	114.0

Table 89. Ambient sampler storage stability data for isopropyl alcohol (*m/z* 45).

time (days)	sample 1 (%)	sample 2 (%)	sample 3 (%)
0	103.3	99.7	97.1
3	101.2	103.2	94.5
7	103.9	107.9	107.5
10	109.2	114.0	117.0

Table 90. Ambient sampler storage stability data for 2-methoxyethyl acetate (*m/z* 58).

time (days)	sample 1 (%)	sample 2 (%)	sample 3 (%)
0	95.4	97.1	107.6
3	97.6	106.2	109.9
7	101.6	109.8	112.5
10	106.9	115.7	123.0

Table 91. Ambient sampler storage stability data for methyl ethyl ketone (*m/z* 72).

time (days)	sample 1 (%)	sample 2 (%)	sample 3 (%)
0	97.2	97.8	105.0
3	108.4	110.6	106.8
6	111.1	108.3	108.9
10	99.9	111.2	105.0

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Table 92. Ambient sampler storage stability data for methyl isoamyl ketone (*m/z* 58).

time (days)	sample 1 (%)	sample 2 (%)	sample 3 (%)
0	92.1	97.4	110.6
3	120.0	126.6	122.8
6	124.5	122.8	123.7
10	108.6	118.4	113.4

Table 93. Ambient sampler storage stability data for methyl isobutyl ketone (*m/z* 58).

time (days)	sample 1 (%)	sample 2 (%)	sample 3 (%)
0	97.9	98.7	103.4
3	115.6	116.3	109.9
6	116.9	108.6	111.4
10	96.1	100.5	95.5

Table 94. Ambient sampler storage stability data for methyl acetate (*m/z* 74).

time (days)	sample 1 (%)	sample 2 (%)	sample 3 (%)
0	97.8	98.4	103.9
3	97.6	99.7	97.7
7	97.4	95.1	93.2
10	98.0	99.5	101.5

Table 95. Ambient sampler storage stability data for methyl cyclohexane (*m/z* 83).

time (days)	sample 1 (%)	sample 2 (%)	sample 3 (%)
0	92.1	97.0	110.9
3	103.6	91.8	68.8
7	110.9	91.0	82.6
11	97.6	98.8	107.6

Table 96. Ambient sampler storage stability data for methylene chloride (*m/z* 84).

time (days)	sample 1 (%)	sample 2 (%)	sample 3 (%)
0	100.0	99.1	100.9
3	99.9	99.2	96.8
7	94.2	95.7	95.9
10	101.9	97.0	101.3

Table 97. Ambient sampler storage stability data for *n*-nonane (*m/z* 57).

time (days)	sample 1 (%)	sample 2 (%)	sample 3 (%)
0	89.1	94.5	116.4
3	106.3	91.5	63.2
7	117.2	94.9	82.7
10	111.1	114.4	124.9

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Table 98. Ambient sampler storage stability data for *n*-octane (*m/z* 85).

time (days)	sample 1 (%)	sample 2 (%)	sample 3 (%)
0	90.5	94.7	114.7
3	105.3	92.3	60.2
7	113.5	87.3	76.3
10	122.2	124.8	136.3

Table 99. Ambient sampler storage stability data for *n*-pentane (*m/z* 72).

time (days)	sample 1 (%)	sample 2 (%)	sample 3 (%)
0	91.5	97.3	111.2
3	102.8	93.3	76.5
7	110.5	93.3	88.4
10	83.9	85.3	94.5

Table 100. Ambient sampler storage stability data for α -pinene (*m/z* 93).

time (days)	sample 1 (%)	sample 2 (%)	sample 3 (%)
0	94.2	103.2	102.6
4	105.3	108.7	117.3
7	104.2	106.9	114.9
11	103.0	110.8	114.7

Table 101. Ambient sampler storage stability data for *n*-propyl alcohol (*m/z* 59).

time (days)	sample 1 (%)	sample 2 (%)	sample 3 (%)
0	103.2	101.0	95.8
3	92.2	107.8	91.0
7	107.4	109.9	111.4
10	109.8	110.9	117.0

Table 102. Ambient sampler storage stability data for tetrachloroethylene (*m/z* 166).

time (days)	sample 1 (%)	sample 2 (%)	sample 3 (%)
0	100.2	98.9	100.9
3	100.5	99.2	99.5
7	101.0	100.8	102.0
10	104.4	98.7	98.9

Table 103. Ambient sampler storage stability data for toluene (*m/z* 91).

time (days)	sample 1 (%)	sample 2 (%)	sample 3 (%)
0	96.9	103.6	99.4
4	114.7	114.6	116.5
7	116.4	116.2	115.0
11	109.4	110.5	111.6

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Table 104. Ambient sampler storage stability data for 1,1,2-trichloroethane (*m/z* 97).

time (days)	sample 1 (%)	sample 2 (%)	sample 3 (%)
0	100.9	99.4	99.8
3	95.2	94.4	94.3
7	99.2	97.8	97.6
10	102.1	96.2	96.5

Table 105. Ambient sampler storage stability data for trichloroethylene (*m/z* 130).

time (days)	sample 1 (%)	sample 2 (%)	sample 3 (%)
0	101.2	100.1	98.7
3	93.6	89.4	92.1
7	105.8	103.3	99.7
10	103.8	97.7	97.7

Table 106. Ambient sampler storage stability data for 1,3,5-trimethylbenzene (*m/z* 105).

time (days)	sample 1 (%)	sample 2 (%)	sample 3 (%)
0	95.8	103.3	100.8
4	107.1	111.8	119.6
7	104.6	109.7	116.1
11	104.3	110.0	116.6

Table 107. Ambient sampler storage stability data for 1,2,4-trimethylbenzene (*m/z* 105).

time (days)	sample 1 (%)	sample 2 (%)	sample 3 (%)
0	97.0	103.1	99.9
4	106.3	107.8	119.2
7	107.5	107.9	112.5
11	105.4	107.8	113.2

Table 108. Ambient sampler storage stability data for 1,2,3-trimethylbenzene (*m/z* 105).

time (days)	sample 1 (%)	sample 2 (%)	sample 3 (%)
0	99.5	103.4	97.1
4	106.9	106.1	110.7
7	105.1	104.4	107.0
11	104.4	104.4	107.1

Table 109. Ambient sampler storage stability data for *m*-xylene (*m/z* 91).

time (days)	sample 1 (%)	sample 2 (%)	sample 3 (%)
0	97.8	98.2	104.0
3	97.6	101.6	107.1
7	96.0	99.9	101.6
10	99.9	105.2	111.2

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Table 110. Ambient sampler storage stability data for *o*-xylene (*m/z* 91).

time (days)	sample 1 (%)	sample 2 (%)	sample 3 (%)
0	100.3	98.0	101.7
3	100.7	101.5	104.0
7	97.1	97.5	97.1
10	99.2	101.2	105.1

Table 111. Ambient sampler storage stability data for *p*-xylene (*m/z* 91).

time (days)	sample 1 (%)	sample 2 (%)	sample 3 (%)
0	99.1	99.8	101.1
3	98.0	96.1	97.0
7	100.0	100.2	101.5
10	103.8	104.5	100.8

4.3 Validation of Vapor Generation System

Injection volume rates were set to 0.3, 1.3, 6.5, 32.5 and 52.2 $\mu\text{L/h}$ with a constant flow rate of 1 L/min to generate 1.15, 5, 25, 125 and 200 ppm of toluene vapors, respectively. A total of fifteen air samples were collected on active sampling charcoal tubes (SKC 226-01) and subsequently analyzed on GC-FID using OSHA 5000 method.³ Results obtained for the fifteen samplers are listed below in Table 112 and plotted in Figure 48. The back sections of all samplers were non-detected, indicating no breakthrough occurring.

Table 112. Analytical data on GC-FID for toluene.

concentration of generated vapor (ppm)	1.15	5	25	125	200
concentration determined by GC-FID	1.17	5.11	24.52	120.63	201.43
(ppm)	0.98	4.77	24.46	125.32	203.45
	1.00	4.76	24.35	126.78	201.73

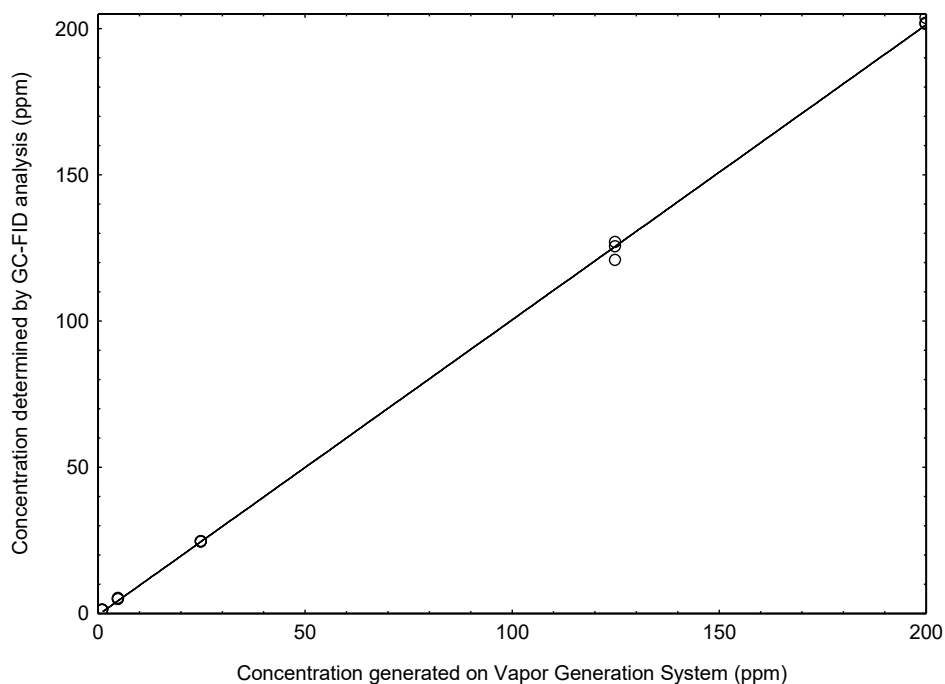


Figure 48. Plot of data used to validate the vapor generation system ($y = 1.01x - 0.52$, $S_{y/x} = 1.58$, $R^2 = 0.9998$).

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4.3.1 Spiking Standards Using Vapor Generation System

Calibrate a calibrator (i.e., Gasmet Calibrator) with a parameter-registered syringe as shown in Figure 49. Determine total injection volume rate to generate an organic vapor at a concentration of 5 ppm. Prepare a single/multiple analyte-containing standard solution from miscible neat reagents. Set temperature of the heating chamber to 5-10 °C above the highest boiling point of the above standard solution. Allow the system to equilibrate for 5 minutes after initializing liquid injection. Connect a conditioned TD tube to a sampling outlet of the vapor chamber then immediately collect 100 mL using a hand-operated pump.

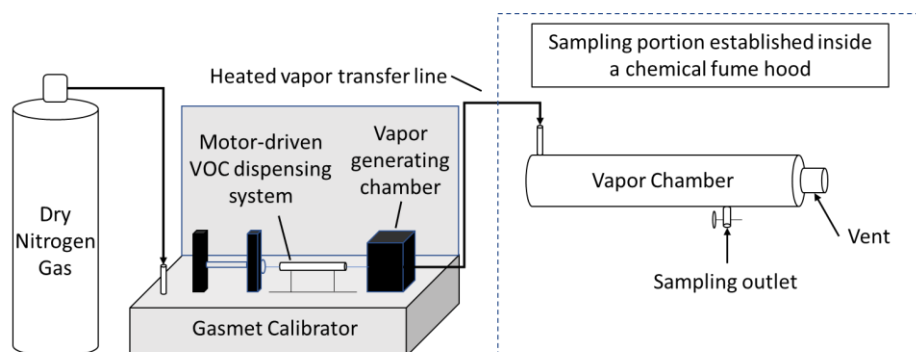


Figure 49. Illustration of the vapor generation system.

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3. Pearce, D. Organic Vapor Sampling Group 1 (OVSG-1), Carbon Disulfide-Extracted Analytes Collected on Coconut Charcoal Sorbent Tubes (OSHA 5000 Method), 2021. United States Department of Labor, Occupational Safety & Health Administration website. <http://www.osha.gov/> (accessed May 2026).