

XYLENES (*o*-, *m*-, *p*-isomers)  
ETHYLBENZENE



Method number: 1002

Target concentration:  
 Xylenes: 100 ppm (435 mg/m<sup>3</sup>)  
 Ethylbenzene: 17 ppm (73 mg/m<sup>3</sup>)

OSHA PEL:  
 Xylenes: 100 ppm (435 mg/m<sup>3</sup>) (TWA)  
 Ethylbenzene: 100 ppm (435 mg/m<sup>3</sup>) (TWA)

ACGIH TLV:  
 Xylene: 100 ppm (TWA)  
 150 ppm (STEL/C)  
 Ethylbenzene: 100 ppm (TWA)  
 125 ppm (STEL/C)

Procedure: Active samples are collected by drawing workplace air through coconut shell charcoal sampling tubes with personal sampling pumps. Diffusive samples are collected by exposing SKC 575-002 Passive Samplers to workplace air. Samples are extracted with carbon disulfide and analyzed by GC using a flame ionization detector.

Recommended sampling time and sampling rate:  
 Charcoal tubes: 240 min at 50 mL/min  
 SKC 575-002  
 Passive Samplers: 240 min

Reliable quantitation limit (RQL) and Standard error of estimate (SEE):	<u>xylenes</u>			<u>ethylbenzene</u>		
	RQL	SEE		RQL	SEE	
	(ppb)	(µg/m <sup>3</sup> )	(%)	(ppb)	(µg/m <sup>3</sup> )	(%)
charcoal tubes	20.6	89.3	5.5	8.3	35.9	5.4
SKC 575-002 Passive Samplers	194.1	842.7	9.3*	72.8	316.0	9.4*

\*For samples when sampling site pressure and temperature are known. See Section 4.4.2 for applicable SEEs when either or both of these values are unknown.

Special requirement: Sampling site temperature and barometric pressure (station pressure) must be reported when diffusive samplers (such as SKC 575-002) are used to sample workplace air.

Status of method: Evaluated method. This method has been subjected to the established evaluation procedures of the Methods Development Team.

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## 1. General Discussion

### 1.1 Background

#### 1.1.1\_ History

Xylenes is a collective term for a mixture of *m*-, *o*-, and *p*- isomers of xylene. These isomers differ only in placement of two methyl groups on a benzene ring. Technical and commercial grades of xylenes often contain substantial amounts of ethylbenzene (10-50%), and perhaps minor amounts of other solvents as well. Mixtures of xylenes and ethylbenzene are occasionally termed mixed xylenes.<sup>1,2</sup>

Most occupational exposure to xylenes also results in exposure to ethylbenzene because technical and commercial grades of xylenes are often used by industry. Therefore, test atmospheres used in this work were prepared with a commercial source of xylenes to simulate workplace environment. This source of xylenes contained 43% *m*-xylene, 20% *o*-xylene, 19% *p*-xylene, and 15% ethylbenzene. Xylenes target concentration in test atmospheres were approximately 100 ppm for the sum of the three isomers. These xylenes air concentrations resulted in approximately 17 ppm ethylbenzene because of its level in the commercial xylenes. Xylenes and ethylbenzene can be present in the workplace in any combination and level, and this method should be satisfactory to monitor exposures to xylenes, individual xylene isomers, and ethylbenzene. The method recommends charcoal tubes for active sampling, and SKC 575-002 Passive Samplers for diffusive sampling. Samples are extracted with carbon disulfide, and are analyzed by GC using a flame ionization detector.

Determination of xylenes is well documented in the literature<sup>3,4</sup>, and one may question why this work was necessary. SLTC has begun to develop sampling and analytical methods which permit the use of diffusive, as well as, active sampling. One criterion for selection of chemicals for this evaluation program is the number of sample requests. Analysis of xylenes is one of the most requested solvent determinations performed at SLTC.

#### 1.1.2 Toxic effects (This section is for information only and should not be taken as the basis of OSHA policy.)

##### Xylenes

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<sup>1</sup> *Kirk-Othmer Encyclopedia of Chemical Technology*; 3<sup>rd</sup> ed.; Grayson, M Ed.; John Wiley & Sons: New York, 1984, Vol. 24; pp. 709-744.

<sup>2</sup> *Documentation of the Threshold Limits Values and Biological and Indices*, 6<sup>th</sup> ed., American Conference of Governmental Industrial Hygienists, Inc: Cincinnati, OH, 1991, Vol. III, pp. 187-193.

<sup>3</sup> *OSHA Analytical Methods Manual*; Vol. 1; U.S. Department of Labor, Occupational Safety and Health Administration; Directorate for Technical Support, OSHA Salt Lake Technical Center: Salt Lake City, UT, 1990; Method 7: Organic Vapors; American Conference of Governmental Hygienists (ACGIH): Cincinnati, OH; Publication No. 4542.

<sup>4</sup> *NIOSH Manual of Analytical Methods*, 4<sup>th</sup> ed.; U.S. Department of Health and Human Services, Public Health Service, Centers for Disease Control and Prevention, National Institute for Occupational Safety and Health: Cincinnati, OH, 1996, Method 1501: Hydrocarbons, Aromatic; (NIOSH) Cincinnati, OH; Publication No. 94-113.

There is no appreciable difference in the toxicological effects of the individual xylene isomers and those of mixed isomers. Xylenes are eye, skin, and mucous membrane irritants. They can cause narcosis at high levels. Xylenes can cause liver and kidney damage. There is little (if any) evidence for the carcinogenicity of xylenes in experimental animals. The ACGIH TLV-TWA was set at 100 ppm, and the STEL at 150 ppm, for mixed xylene isomers and for individual isomers. It was anticipated that irritant effects would be minimal, and that neither narcosis nor chronic injury would result from exposures at these levels.<sup>5</sup>

### Ethylbenzene

Ethylbenzene is a skin and mucous membrane irritant. It has acute and possibly chronic central nervous system effects that include vertigo, unconsciousness, tremors, and changes in respiration. Animal experiments suggest that ethylbenzene causes damage to the liver, kidneys, and testes. It was the opinion of the ACGIH TLV Committee that no systemic effects would be expected at concentrations which produce skin and eye irritation. The ACGIH TLV-TWA was set at 100 ppm and the STEL at 125 ppm to prevent such irritation.<sup>6</sup> ACGIH published in the 1998 TLVs and BEIs booklet<sup>7</sup> a "Notice of Intended Changes" to add the A3 notation to ethylbenzene. A3 is defined as "Confirmed Animal Carcinogen with Unknown Relevance to Humans".

#### 1.1.3 Workplace exposure

The main source of mixed xylenes since World War II has been reformed petroleum fractions. Earlier, xylenes were produced from coal. Coal may again become an important source as the large coal reserves in the United States are developed for petrochemical uses.<sup>8</sup> U.S. production of xylenes in 1995 was 9.4 billion pounds, and that for ethylbenzene was 13.7 billion pounds.<sup>9</sup>

Most mixed xylenes are used to blend gasoline. Mixed xylenes are also used in the paint and coatings industry. *m*-Xylene is used to produce isophthalic acid, which is used in polyesters; *o*-xylene is used to produce phthalic anhydride, which is used in plasticizers; *p*-xylene is used to produce terephthalic acid and dimethyl terephthalate, both of which are

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<sup>5</sup> *Documentation of the Threshold Limits Values and Biological and Indices*, 6<sup>th</sup> ed., American Conference of Governmental Industrial Hygienists, Inc: Cincinnati, OH, 1991, Vol. III, pp. 1732-1740.

<sup>6</sup> *Documentation of the Threshold Limits Values and Biological and Indices*, 6<sup>th</sup> ed., American Conference of Governmental Industrial Hygienists, Inc: Cincinnati, OH, 1991, Vol. I, pp. 581-584.

<sup>7</sup> *1998 TLVs and BEIs, Threshold Limit Values for Chemical Substances and Physical Agents*, ISBN: 1-882417-23-2; American Conference of Governmental Industrial Hygienists, (ACGIH): Cincinnati, OH, 1998.

<sup>8</sup> *Kirk-Othmer Encyclopedia of Chemical Technology*, 3<sup>rd</sup> ed.; Grayson, M Ed.; John Wiley & Sons: New York, 1984, Vol. 24; pp. 709-744.

<sup>9</sup> ACS Publications; <http://pubs.acs.org/hotartcl/cenear/960624/prod.html> (accessed March 1999).

used to produce polyesters. *o*-Xylene and *p*-xylene are used in vitamin and pharmaceutical synthesis, and to produce insecticides. Ethylbenzene is used to produce styrene.<sup>10, 11</sup>

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<sup>10</sup> *Kirk-Othmer Encyclopedia of Chemical Technology*, 3<sup>rd</sup> ed.; Grayson, M Ed.; John Wiley & Sons: New York, 1984, Vol. 24; pp. 709-744.

<sup>11</sup> *The Condensed Chemical Dictionary*, 8<sup>th</sup> ed.; Revised by Hawley, G., Ed., Van Nostrand Reinhold: New York, 1971, p. 358, 942, 943.

#### 1.1.4 Physical properties (<sup>12</sup> unless otherwise noted)

	xylene	<i>m</i> -xylene	<i>o</i> -xylene	<i>p</i> -xylene	ethylbenzene
CAS number <sup>13</sup>	1330-20-7 <sup>14</sup>	108-38-3	95-47-6	106-42-3	100-41-4
IMIS number <sup>15</sup>	2590				1080
molecular weight <sup>16</sup>	106.17	106.17	106.17	106.17	106.17
boiling point (°C)	137-145	138.8	144	138.5	136.19
melting point (°C)		-47.4	-25	13.2	-95.01 <sup>17</sup>
density (°C)	about 0.86	0.868 (15)	0.880 (20/4)	0.861 (20)	0.867 (20)
molecular formula	C <sub>8</sub> H <sub>10</sub>	C <sub>8</sub> H <sub>10</sub>	C <sub>8</sub> H <sub>10</sub>	C <sub>8</sub> H <sub>10</sub>	C <sub>8</sub> H <sub>10</sub>
flash point (°F)	81-115	85	115	81	59
vapor pressure (kPa, (°C)) <sup>18, 19</sup>		1.1 (25)	0.9 (25)	1.2 (25)	0.9 (20)

Xylenes (dimethylbenzene, xylol) are soluble in alcohol and ether, but insoluble in water. Each of the mixed xylenes is a clear, colorless liquid at room temperature, however, *p*-xylene forms crystals at a relatively high temperature. The xylene isomers: *m*-xylene (1,3-dimethylbenzene), *o*-xylene (1,2-dimethylbenzene), and *p*-xylene (1,4-dimethylbenzene) are soluble in alcohol and ether; but they are insoluble in water. Ethylbenzene (phenylethane) is soluble in alcohol, benzene, carbon tetrachloride, and ether; it is but almost insoluble in water.<sup>20</sup>

<sup>12</sup> *The Condensed Chemical Dictionary*, 8<sup>th</sup> ed.; Revised by Hawley, G., Ed., Van Nostrand Reinhold: New York, 1971, p. 358, 942, 943.

<sup>13</sup> *Kirk-Othmer Encyclopedia of Chemical Technology*, 3<sup>rd</sup> ed.; Grayson, M Ed.; John Wiley & Sons: New York, 1984, Vol. 24; pp. 709-744.

<sup>14</sup> OSHA Computerized Information System Database, Chemical Sampling Information, Salt Lake Technical Center, Occupational Safety and Health Administration, Salt Lake City, UT March 1999.

<sup>15</sup> OSHA Computerized Information System Database, Chemical Sampling Information, Salt Lake Technical Center, Occupational Safety and Health Administration, Salt Lake City, UT March 1999.

<sup>16</sup> *Kirk-Othmer Encyclopedia of Chemical Technology*, 3<sup>rd</sup> ed.; Grayson, M Ed.; John Wiley & Sons: New York, 1984, Vol. 24; pp. 709-744.

<sup>17</sup> *Documentation of the Threshold Limits Values and Biological and Indices*, 6<sup>th</sup> ed., American Conference of Governmental Industrial Hygienists, Inc: Cincinnati, OH, 1991, Vol. II, pp. 581-584.

<sup>18</sup> *Documentation of the Threshold Limits Values and Biological and Indices*, 6<sup>th</sup> ed., American Conference of Governmental Industrial Hygienists, Inc: Cincinnati, OH, 1991, Vol. III, pp. 1732-1740.

<sup>19</sup> *Documentation of the Threshold Limits Values and Biological and Indices*, 6<sup>th</sup> ed., American Conference of Governmental Industrial Hygienists, Inc: Cincinnati, OH, 1991, Vol. I, pp. 581-584.

<sup>20</sup> *The Condensed Chemical Dictionary*, 8<sup>th</sup> ed.; Revised by Hawley, G., Ed., Van Nostrand Reinhold: New York, 1971, p. 358, 942, 943.

Structural formulas:

This method was evaluated according to OSHA SLTC "Evaluation Guidelines for Air Sampling Methods Utilizing Chromatographic Analysis"<sup>21</sup>. The Guidelines define analytical parameters and specify required laboratory tests, statistical calculations and acceptance criteria. The analyte concentrations throughout this method are based on the recommended sampling and analytical parameters. Air concentrations in ppm and ppb are referenced to 25 °C and 101.3 kPa (760 mmHg).

## 1.2 Limit defining parameters

### 1.2.1 Detection limit of the analytical procedure

The detection limits of the analytical procedure (DLAP) are shown in Table 1.2.1. These are the amounts of analyte that will give detector responses significantly different from the response of a reagent blank. (Section 4.1)

Table 1.2.1  
DLAP (pg per sample)

xylenes	<i>m</i> -xylene	<i>o</i> -xylene	<i>p</i> -xylene	ethylbenzene
14.5	2.1	8.4	14.0	5.7

### 1.2.2 Detection limit of the overall procedure

#### Charcoal tubes

The detection limits of the overall procedure (DLOP) are shown in Table 1.2.2.1. These are the amounts of analyte spiked on the samplers that will give detector responses significantly different from the response of a sampler blank. (Section 4.2)

Table 1.2.2.1  
DLOP for Charcoal Tubes

analyte	ng	ppb	µg/m <sup>3</sup>
xylenes	322	6.2	26.8
<i>m</i> -xylene	159	3.0	13.2
<i>o</i> -xylene	239	4.6	19.9
<i>p</i> -xylene	100	1.9	8.3
ethylbenzene	129	2.5	10.8

#### SKC 575-002 Passive Samplers

The detection limits of the overall procedure (DLOP) are shown in Table 1.2.2.2. These are the amounts of analyte spiked on the samplers that will give detector responses significantly different from the response of a sampler blank. (Section 4.2)

Table 1.2.2.2  
DLOP for SKC 575-002  
Passive Samplers

analyte	ng	ppb	µg/m <sup>3</sup>
xylenes	847	58.2	252.8
<i>m</i> -xylene	448	31.1	134.9
<i>o</i> -xylene	325	21.9	95.0
<i>p</i> -xylene	437	30.1	130.8
ethylbenzene	315	21.9	94.9

### 1.2.3 Reliable quantitation limit

#### Charcoal tubes

The reliable quantitation limits (RQL) are shown in Table 1.2.3.1. These are the amounts of analyte that will give detector responses that are considered the lower limits for precise quantitative measurements. (Section 4.2)

Table 1.2.3.1  
RQL for Charcoal Tubes

analyte	ng	ppb	µg/m <sup>3</sup>
xylenes	1072	20.6	89.3
<i>m</i> -xylene	531	10.2	44.2
<i>o</i> -xylene	795	15.3	66.2
<i>p</i> -xylene	334	6.4	27.8
ethylbenzene	431	8.3	35.9

<sup>21</sup> Burreight, D.; Chan, Y.; Eide, M.; Elskamp, C.; Hendricks, W.; Rose, M. "Evaluation Guidelines for Air Sampling Methods Utilizing Chromatographic Analysis", OSHA Salt Lake Technical Center, U.S. Department of Labor: Salt Lake City, UT, 1999 (in-revision).

## SKC 575-002 Passive Samplers

The reliable quantitation limits (RQL) are shown in Table 1.2.3.2. These are the amounts of analyte that will give detector responses that are considered the lower limits for precise quantitative measurements. (Section 4.2)

Table 1.2.3.2  
RQL for SKC 575-002 Passive Samplers

analyte	ng	ppb	µg/m <sup>3</sup>
xylene	2823	194.1	842.7
<i>m</i> -xylene	1495	103.7	450.3
<i>o</i> -xylene	1084	73.0	317.0
<i>p</i> -xylene	1456	100.4	435.9
ethylbenzene	1049	72.8	316.0

## 1.2.4 Instrument calibration

The coefficients of determination ( $r^2$ ) and of nondetermination ( $k^2$ ) for the calibration curves are shown in Table 1.2.4. The calibrated range was 0.5 to 2 times the OSHA PEL. (Section 4.3)

Table 1.2.4  
Coefficients of Determination ( $r^2$ )  
and Nondetermination ( $k^2$ )

analyte	charcoal tubes		SKC 575-002 Pass Samplers	
	$r^2$	$k^2$	$r^2$	$k^2$
<i>m</i> -xylene	0.9994	$6 \times 10^{-4}$	0.9998	$2 \times 10^{-4}$
<i>o</i> -xylene	0.9995	$5 \times 10^{-4}$	0.9998	$2 \times 10^{-4}$
<i>p</i> -xylene	0.9994	$6 \times 10^{-4}$	0.9998	$2 \times 10^{-4}$
ethylbenzene	0.9994	$6 \times 10^{-4}$	0.9998	$2 \times 10^{-4}$

## 1.2.5 Precision (overall procedure)

### Charcoal tubes

The precision of the overall procedure at the 95% confidence interval for the ambient temperature 16-day storage test (at the target concentration) are shown in Table 1.2.5.1. Each precision includes an additional 5% for sampling pump variability. (Section 4.4)

Table 1.2.5.1  
Precision of the  
Overall Procedure for Charcoal Tubes

analyte	precision (±%)
xylene	10.8
<i>m</i> -xylene	10.7
<i>o</i> -xylene	11.0
<i>p</i> -xylene	11.1
ethylbenzene	10.6

### SKC 575-002 Passive Samplers

The precision of the overall procedure at the 95% confidence interval for the ambient temperature 16-day storage tests (at the target concentration) are given in Table 1.2.5.2. They each include an additional 8.7% for sampling rate variability. There are different values given, depending on whether both, either, or neither temperature or atmospheric pressure are known at the sampling site. If the sampling-site temperature ( $T$ ) is unknown, it is assumed to be  $22.2 \pm 15$  °C ( $72 \pm 27$  °F) and a variability of  $\pm 7.7\%$  is included. If the atmospheric pressure ( $P$ ) is unknown, it is estimated from sampling-site elevation and a variability of  $\pm 3\%$  is included. (Section 4.4)

Table 1.2.5.2  
Precision of the Overall Procedure for SKC 575-002 Passive Samplers (±%)

condition	xylene	<i>m</i> -xylene	<i>o</i> -xylene	<i>p</i> -xylene	ethylbenzene
both $T$ and $P$ known	18.2	18.3	18.1	18.3	18.4
only $T$ known	19.1	19.2	19.0	19.2	19.3
only $P$ known	23.7	23.7	23.6	23.7	23.8
neither $T$ nor $P$ known	24.4	24.4	24.3	24.4	24.5



### 1.2.6 Recovery

The recoveries from samples used in 16-day ambient temperature storage tests remained above those shown in Table 1.2.6. (Section 4.5)

Table 1.2.6  
Recovery (%)

analyte	charcoal tubes	SKC 575-002 Passive Samplers
xylene	99.1	97.7
<i>m</i> -xylene	99.6	98.2
<i>o</i> -xylene	98.5	96.6
<i>p</i> -xylene	98.5	97.8
ethylbenzene	100.2	99.5

### 1.2.7 Reproducibility

Twelve samples (six active and six diffusive) collected from test atmospheres were submitted for analysis by SLTC. The samples were analyzed according to instructions presented in a draft copy of this method after 16 and 22 days of storage at ambient temperature for the active and diffusive samplers, respectively. No individual result deviated from its theoretical value by more than the precision reported in Section 1.2.5. (Section 4.6)

## 2. Sampling procedure

All safety practices that apply to the work area being sampled should be followed. The sampling equipment should be attached to the worker in such a manner that it will not interfere with work performance or safety.

### 2.1 Apparatus

#### 2.1.1 Charcoal tubes

Samples are collected with a personal sampling pump calibrated, with the sampler attached, to within  $\pm 5\%$  at 50 mL/min.

Samples are collected with 7-cm  $\times$  4-mm i.d.  $\times$  6-mm o.d. flame sealed glass sampling tubes containing two sections of coconut shell charcoal. The front section contains 100 mg and the back section contains 50 mg of charcoal. The sections are held in place and separated with glass wool and polyurethane plugs. Commercially prepared sampling tubes were purchased from SKC for this evaluation (SKC Catalog no. 226-01, Lot 2000).

#### 2.1.2 SKC 575-002 Passive Samplers

Samples are collected with SKC 575-002 Passive Samplers. These samplers contain 500 mg of Anasorb 747. Lot numbers 347, 764, and 872 were used in this evaluation.

A thermometer and a barometer are needed to determine sampling site temperature and pressure.

### 2.2 Reagents

None required.

### 2.3 Technique

#### 2.3.1 Charcoal tubes

Immediately before sampling, break off both ends of the flame sealed sampling tube to provide openings approximately half the internal diameter of the tube. Wear eye protection when breaking tubes. Use sampling tube holders to shield the employee from the sharp, jagged ends of the sampling tubes. All sampling tubes should be from the same lot.

Use the smaller charcoal section of the sampling tube as a back-up and position it nearest the sampling pump. Attach the sampling tube to the sampling pump so that the tube is in an approximately vertical position with the inlet down during sampling. Position the sampling tube so that it does not impede work performance or safety.

Draw air to be sampled directly into the tube inlet. Sampled air is not to pass through any hose or tubing before entering the sampling tube.

Remove the sampler and seal the tube with plastic end caps after sampling for the appropriate time. Seal each sample end-to-end with an OSHA-21 form as soon as possible.

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

Record sample air volume in liters for each sample, and record any potential interference.

Submit the samples to the laboratory for analysis as soon as possible after sampling. Store the samples at reduced temperature if delay is unavoidable. Ship any bulk samples separate from air samples.

#### 2.3.2 SKC 575-002 Passive Samplers (In general, follow the manufacturer's instructions)

Remove the sampler from the clear package just before sampling. CAUTION - The monitor begins to sample as soon as it is removed from this package. Retain the O-ring, press-on cover, cover retainer, port plugs, and PTFE tube for later use.

Record the start time on the sampler label, or on the Form OSHA-91A.

Attach the sampler near the worker's breathing zone with the perforations in the sampler facing out. Assure that the area directly in front of the sampler is unobstructed throughout the sampling period.

Remove the sampler from the worker immediately at the end of the sampling period. Attach the cover with the O-ring in place onto the sampler using the cover retainer. Inspect the O-ring to be sure it is forming a good seal around the entire circumference of the sampler. Record the stop time on the sampler label, or on the Form OSHA-91A.

Prepare a blank sample by removing it from its clear package, and then immediately attaching a cover with the O-ring in place onto it.

Seal each sample with an OSHA-21 form.

Verify that sampling times are properly recorded on Form OSHA-91A for each sample. Identify blank samples on this form.

#### **Record sampling site temperature and atmospheric pressure (station pressure) on Form OSHA-91A.**

List any chemicals that could be considered potential interferences, especially solvents, that are in use in the sampling area.

Submit the samples to the laboratory for analysis as soon as possible. Store the samples in a refrigerator if delay is unavoidable. Include the port plugs and PTFE tubes which will be used in the laboratory analysis.

Ship any bulk samples separate from air samples.

## 2.4 Sampler capacity

### 2.4.1 Charcoal tubes

The sampling capacity of SKC Lot 2000 charcoal tubes was tested by sampling from a dynamically generated test atmosphere of mixed xylenes (1027 mg/m<sup>3</sup> or 237 ppm). Samples were collected at 50 mL/min and the relative humidity was about 78% at 21 °C. No breakthrough from the front to the back section was observed, even after sampling for ten hours. The 5% breakthrough sampling time was determined to be in excess of 600 min. (Section 4.7.1)

### 2.4.2 SKC 575-002 Passive Samplers

The sampling rate and capacity of SKC 575-002 Passive Samplers were determined by sampling from dynamically generated test atmospheres of mixed xylenes (1027 mg/m<sup>3</sup> or 237 ppm, at 78% relative humidity and 21 °C) for increasing time intervals. Sampling rates of 13.82 mL/min for *m*-xylene, 14.24 mL/min for *o*-xylene, 13.94 mL/min for *p*-xylene, and 13.83 mL/min for ethylbenzene at 760 mmHg and 25 °C were obtained from these tests. Sampler capacity was never exceeded, even after sampling for ten hours. (Section 4.7.2)

## 2.5 Extraction efficiency

It is the responsibility of the analytical laboratory to occasionally determine or confirm extraction efficiency because the adsorbent material, reagents, or technique may be different from those presented in this method.

### 2.5.1 Charcoal tubes

Mean extraction efficiencies (EE) of the analytes from SKC Lot 2000 charcoal are presented in Table 2.5.1. The range studied was from the RQL to 2 times the 100 ppm OSHA PEL for each xylene isomer, and for ethylbenzene. The extraction efficiency was not affected by the presence of water. (Section 4.8.1)

<i>m</i> -xylene	<i>o</i> -xylene	<i>p</i> -xylene	ethylbenzene
96.3	93.8	96.1	97.2

### 2.5.2 SKC 575-002 Passive Samplers

Mean extraction efficiencies (EE) of the analytes from SKC Anasorb 747 (the adsorbent in SKC 575-002 Passive Samplers) are presented in Table 2.5.2. The range studied was from the RQL to 2 times the 100 ppm OSHA PEL for each xylene isomer, and for ethylbenzene. The extraction efficiency was not affected by the presence of water. (Section 4.8.2)

<i>m</i> -xylene	<i>o</i> -xylene	<i>p</i> -xylene	ethylbenzene
96.1	89.4	95.3	99.1

## 2.6 Recommended sampling time and sampling rate

### 2.6.1 Charcoal tubes

Sample for up to 4 hours at 50 mL/min when using SKC 226-01 charcoal tubes to collect long-term samples. Sample for more than 5 min at 50 mL/min to collect short-term samples.

## 2.6.2 SKC 575-002 Passive Samplers

Sample for up to 4 hours when using SKC 575-002 Passive Samplers to collect long-term samples. Sample for more than 5 min to collect short-term samples.

Table 2.6.2  
Sampling Rates for SKC 575-002 Passive Samplers (mL/min) at 760 mmHg and 25 °C

<i>m</i> -xylene	<i>o</i> -xylene	<i>p</i> -xylene	ethylbenzene
13.82	14.24	13.94	13.83

2.6.3 The air concentration equivalent to the reliable quantitation limit becomes larger when short-term samples are collected. For example, the reliable quantitation limit for xylenes is 733 ppb (3180 µg/m<sup>3</sup>) when 0.25 L of air is sampled using charcoal tubes.

## 2.7 Sampling interferences (Section 4.9)

### 2.7.1 Charcoal tubes

#### Retention

The ability of the sampler to retain the analytes following collection was tested. The retention efficiency of all analytes for all samples was above 100.8% when three charcoal tubes containing 3 mg of mixed xylenes were used to sample 9 L of contaminant-free air having a relative humidity of 80% at 20 °C.

#### Low relative humidity

The ability of the sampler to collect and retain the analytes at low relative humidity was tested. The collection efficiency of all analytes for all samples was above 99.2% when three charcoal tubes were used to sample 12 L of air containing two times the target concentration of mixed xylenes and having a relative humidity of 5% at 20°C.

#### Low concentration

The ability of the sampler to collect and retain the analytes at low concentration was tested. The collection efficiency of all analytes for all samples was above 94.6% when three charcoal tubes were used to sample 12 L of air containing 0.1 times the target concentration of mixed xylenes and having a relative humidity of 80% at 22 °C.

#### Interference

The ability of the sampler to collect and retain the analytes in the presence of potential sampling interferences was tested. The collection efficiency of all analytes for all samples was above 101.2% when three charcoal tubes were used to sample 12 L of air containing one times the target concentration of mixed xylenes, 365 mg/m<sup>3</sup> toluene, 372 mg/m<sup>3</sup> butyl acetate, and a relative humidity of 81% at 21 °C.

### 2.7.2 SKC 575-002 Passive Samplers

#### Reverse diffusion

The sampling method was tested for reverse diffusion. The retention efficiency of all analytes for all samples was above 99.6% when three SKC 575-002 Passive Samplers containing 0.9 mg of mixed xylenes were used to sample contaminant-free air having a relative humidity of 80% at 20 °C for three hours.

#### Low relative humidity

The sampling method was tested to determine if the sampling rates remained constant at low relative humidity. The sampling rate of all analytes for all samples was above 97.8% of the sampling rate reported in Section 2.6.2 when three SKC 575-002 Passive Samplers were used to sample air containing two times the target concentration of mixed xylenes and having a relative humidity of 5% at 20°C for four hours. Low humidity did not affect the sampling rates.

#### Low concentration

The sampling method was tested to determine if the sampling rates remained constant at low concentration. The sampling rate of all analytes for all samples was above 95.0% of the sampling rate reported in Section 2.6.2 when three SKC 575-002 Passive Samplers were used to sample air containing 0.1 times the target concentration of mixed xylenes and having a relative humidity of 80% at 22 °C for four hours.

#### Interference

The sampling method was tested to determine if the sampling rates remained constant in the presence of sampling interferences. The sampling rate of all analytes for all samples was above 94.3% of the sampling rate reported in Section 2.6.2 when three SKC 575-002 Passive Samplers were used to sample air containing one times the target concentration of mixed xylenes, 365 mg/m<sup>3</sup> toluene, 372 mg/m<sup>3</sup> butyl acetate, and a relative humidity of 81% at 21 °C for four hours.

### 3. Analytical procedure

Adhere to the rules set down in your Chemical Hygiene Plan (which is mandated by the OSHA Laboratory Standard). Avoid skin contact and inhalation of all chemicals.

#### 3.1 Apparatus

- 3.1.1 A GC equipped with a flame ionization (FID) detector. A Hewlett-Packard Model 5890 Series II GC equipped with a ChemStation, an automatic sample injector, and an FID were used in this evaluation.
- 3.1.2 A GC column capable of separating mixed xylenes from the extraction solvent, internal standards, and potential interferences. A J&W Scientific 60-m × 0.32-mm i.d. DB-Wax (0.5µm df) capillary column was used in this evaluation.
- 3.1.3 An electronic integrator or other suitable means of measuring GC detector response. A Waters Millennium Chromatography Manager system was used in this evaluation.
- 3.1.4 Two and four-milliliter glass vials with PTFE-lined septum caps.
- 3.1.5 One and two-milliliter volumetric pipets.
- 3.1.6 A SKC Desorption Shaker with rack (226D-03K) was used to extract SKC 575-002 Passive Samplers in this evaluation.

#### 3.2 Reagents

- 3.2.1 Xylenes, Isomers plus ethylbenzene, 98.5+%, A.C.S. reagent, Aldrich Chemical Co., Lot TR 02505LR, was used in this evaluation.
- 3.2.2 *m*-Xylene, 99+%, anhydrous, Aldrich Chemical Co., Lot 00249MQ, was used in this evaluation.

- 3.2.3 *o*-Xylene, 98%, Spectrophotometric Grade, Aldrich Chemical Co., Lot 07946PN, was used in this evaluation.
- 3.2.4 *p*-Xylene, 99+%, anhydrous, Aldrich Chemical Co., Lot TQ 25949MQ, was used in this evaluation.
- 3.2.5 Ethylbenzene, 99.8%, anhydrous, Aldrich Chemical Co., Lot DR 03249JQ, was used in this evaluation.
- 3.2.6 Carbon disulfide (CS<sub>2</sub>), 99.9+%, low benzene content, Aldrich Chemical Co., Lot 07546PN, was used in this evaluation.
- 3.2.7 1-Phenylhexane (hexylbenzene), 97%, Aldrich Chemical Co., Lot 03006PZ, was used as an internal standard for **SKC 575-002 Passive Samplers** in this evaluation.
- 3.2.8 *p*-Cymene, 99%, Aldrich Chemical Co., Lot 11703TR, was used as an internal standard for **charcoal tube** samples in this evaluation.
- 3.2.9 The extraction solvent used for this evaluation consisted of 1µL of the appropriate internal standard per milliliter of CS<sub>2</sub>. **CAUTION:** extraction efficiency of the internal standard from the sampling medium has an effect on sample results. This effect is especially significant for SKC 575-002 Passive Samplers. Do not substitute internal standards unless extraction efficiencies are confirmed. Both internal standards can be present in the same extraction solvent if the appropriate internal standard is used to calibrate the GC, and to calculate sample results.
- 3.2.10 GC grade nitrogen, air, and hydrogen were used in this evaluation.

### 3.3 Standard preparation

- 3.3.1 Prepare stock mixed standards by weighing 1-mL aliquots of all four analytes into the same container. For example: a neat mixed standard was prepared that contained 212.7 mg/mL of *m*-xylene, 212.6 mg/mL of *o*-xylene, 213.4 mg/mL of *p*-xylene, and 215.3 mg/mL of ethylbenzene.
- 3.3.2 Prepare working range standards for the analysis of charcoal tubes by injecting microliter quantities of the stock mixed standard into 1-mL aliquots of extraction solvent (containing 1 µL *p*-cymene internal standard per milliliter of CS<sub>2</sub>). For example, a working range standard was prepared by injecting 6.0 µL of the stock mixed standard into extraction solvent. This standard contained 1276 µg/mL of *m*-xylene, 1276 µg/mL of *o*-xylene, 1280 µg/mL of *p*-xylene, and 1292 µg/mL of ethylbenzene.
- 3.3.3 Prepare working range standards for the analysis of SKC 575-002 Passive Samplers by first diluting the stock mixed standard (Section 3.4.1) 1 to 4 with CS<sub>2</sub>, and then injecting microliter quantities of the diluted stock mixed standard into 2-mL aliquots of extraction solvent (containing 1 µL 1-phenylhexane internal standard per milliliter of CS<sub>2</sub>). For example, a working range standard was prepared by injecting 6.0 µL of the diluted stock mixed standard into extraction solvent. This standard contained 319.0 µg/mL of *m*-xylene, 318.9 µg/mL of *o*-xylene, 320.1 µg/mL of *p*-xylene, and 323.0 µg/mL of ethylbenzene.
- 3.3.4 Prepare a sufficient number of standards so that sample results will likely be bracketed with standards. If sample results are outside the range of prepared standards, prepare and analyze additional standards, or dilute high samples with extraction solvent and then reanalyze the diluted samples.

### 3.4 Sample preparation

#### 3.4.1 Charcoal tubes

Remove the plastic end caps from the sampling tube and carefully transfer each section of the adsorbent into separate 2-mL glass vials. Check to be certain that no charcoal is trapped in the glass-wool plug. Discard the end caps, glass tube, glass wool plug, and foam plugs.

Add 1.0 mL of extraction solvent to each vial and immediately seal each vial with a PTFE-lined septum cap.

Shake the vials vigorously several times during the one-hour extraction time.

#### 3.4.2 SKC 575-002 Passive Samplers

Cut off the ends of the two protruding tubes of each sampler with a razor blade or a sharp knife.

Secure the sampler by clipping it to a rail of the detachable SKC Desorption Shaker rack. Carefully and slowly add 2.0 mL of extraction solvent through the protruding tube nearest the outside edge of the sampler using a volumetric pipet. The tip of the pipet should fit just inside the sampler tube. Immediately seal the sampler tubes with the plugs supplied by the manufacturer.

Replace the rack onto the SKC Desorption Shaker and shake the samples for one hour.

Do not allow the extracted sample to remain in the sampler. Transfer the extracted sample into 2-mL glass vials by removing the plugs from the protruding tubes, inserting the tapered end of the PTFE tube supplied by the manufacture into the protruding tube nearest the outside edge of the sampler, and carefully pouring the solution into a 2-mL glass vial. Immediately seal the vials with PTFE-lined septum caps.

### 3.5 Analysis

#### 3.5.1 GC conditions

zone temperatures:

column 40 °C, hold 1 min, program at 4 °C/min to 140 °C, and hold as necessary to clear column

injector 220 °C

detector 220 °C

gas flows: hydrogen (carrier) 4.4 mL/min (115 kPa head pressure)

nitrogen (makeup) 38 mL/min

hydrogen (FID) 35 mL/min

air (FID) 455 mL/min

signal range: 3

injection volume: 1 µL (20:1 split)

column: 60 m × fused silica 0.32-mm i.d. DB Wax 0.5-µm df

3.5.2 Measure peak areas with an electronic integrator or other suitable means.

3.5.3 An internal standard (ISTD) method is used to calibrate the instrument in terms of micrograms of analyte per sample. Prepare a calibration curve by analyzing standards, and construct calibration curves by plotting ISTD-corrected detector response versus mass of analyte. Bracket sample results with standards.





### 3.6 Interferences (analytical)

- 3.6.1 Any chemical that produces an FID response and has a similar retention time as any of the analytes or internal standard is a potential interference. Any reported potential interferences should be considered before samples are extracted. Generally, chromatographic conditions can be altered to separate an interference from an analyte or an internal standard.
- 3.6.2 The identity or purity of an analyte peak can be confirmed with additional analytical data. (Section 4.10)

### 3.7 Calculations

#### 3.7.1 Charcoal tubes

Obtain separate amounts of each analyte (*m*-xylene, *o*-xylene, *p*-xylene, ethylbenzene) per sample from the appropriate calibration curve in terms of micrograms per sample. These amounts are uncorrected for extraction efficiency. Be certain that the correct internal standard was used to calculate results (See Section 3.2.9). The back section of the sampling tube is analyzed primarily to determine the extent of sampler saturation. If any analyte is found on the back section, it is added to the amount found on the front section. This amount is then corrected by subtracting the total amount (if any) found on the blank. The air concentrations are then calculated using the following formulas. Calculate xylenes exposure by summing the individual xylene isomer results.

where 24.46 is the molar volume at 25°C and 101.3 kPa (760 mmHg)  
106.17 is the molecular weight of *m*-xylene, *o*-xylene, *p*-xylene, and ethylbenzene

#### 3.7.2 SKC 575-002 Passive Samplers

Obtain separate amounts of each analyte (*m*-xylene, *o*-xylene, *p*-xylene, ethylbenzene) per sample from the appropriate calibration curve in terms of micrograms per sample. These amounts are uncorrected for extraction efficiency. Be certain that the correct internal standard was used to calculate results. This amount is then corrected by subtracting the amount (if any) found on the blank.

Sampling time, sampling site temperature (°C), and sampling site pressure (mmHg) is information given by the person submitting the samples. Sampling rates at 760 mmHg and 25 °C ( $SR_{NTP}$ ) are given in Table 3.7.2. These sampling rates must be converted to their equivalent ( $SR_{amb}$ ) at sampling site temperature ( $T$ ) and sampling site pressure ( $P$ ) by the following formula:

Table 3.7.2  
Sampling Rates for SKC 573-002 Passive Samplers (mL/min) at 760 mmHg and 25 °C

<i>m</i> -xylene	<i>o</i> -xylene	<i>p</i> -xylene	ethylbenzene
13.82	14.24	13.94	13.83

Assume sampling site temperature is 22.2 °C if it is not given. If sampling site pressure is not given, it can be calculated by the following formula:

where:

$P$  is the approximate sampling site barometric pressure.  $E$  is the sampling site elevation.  $E$  can be estimated from airports near the sampling site location using a web site such as <http://www.airnav.com>

Liters of air sampled is calculated by multiplying the appropriate  $SR_{amb}$  by sampling time.

Air concentrations are then calculated using the following formulas. Calculate xylenes exposure by summing the individual xylene isomer results.

where: 24.46 is the molar volume at 25 °C and 101.3 kPa (760 mmHg)  
106.17 is the molecular weight of *m*-xylene, *o*-xylene, *p*-xylene, and ethylbenzene

#### 4. Backup Data

General background information about determination of detection limits and precision of the overall procedure is found in OSHA SLTC "Evaluation Guidelines for Air Sampling Methods Utilizing Chromatographic Analysis"<sup>22</sup>. The Guidelines define analytical parameters and specify required laboratory tests, statistical calculations and acceptance criteria.

##### 4.1 Detection limit of the analytical procedure (DLAP)

The DLAP is measured as the mass of analyte introduced into the chromatographic column. Ten standards were prepared in equal descending increments of analyte, such that the highest standard produced a peak approximately 10 times the response of a reagent blank. These standards, and a reagent blank, were analyzed using the recommended analytical parameters (1- $\mu$ L injection with a 20:1 split), and the data obtained were used to determine the required parameters ( $A$  and  $SEE_{DL}$ ) for the calculation of the DLAP. Xylenes DLAP was calculated by summing masses and areas for individual xylene isomers. The extraction solvent contained a contaminant that eluted at the same time as *p*-xylene. The amount of this contaminant was small, but sufficient to cause a higher DLAP for *p*-xylene than for the other analytes.

Table 4.1  
Detection Limit of the  
Analytical Procedure

analyte	DLAP (pg)
xylenes	14.5
<i>m</i> -xylene	2.1
<i>o</i> -xylene	8.4
<i>p</i> -xylene	14.0
ethylbenzene	5.7

<sup>22</sup> Burreight, D.; Chan, Y.; Eide, M.; Elskamp, C.; Hendricks, W.; Rose, M. "Evaluation Guidelines for Air Sampling Methods Utilizing Chromatographic Analysis", OSHA Salt Lake Technical Center, U.S. Department of Labor: Salt Lake City, UT, 1999 (in-revision).

Table 4.1.1  
DLAP for Xylenes

concn (ng/mL)	mass on column (pg)	area counts ( $\mu$ V-s)
0	0	150
502	25.6	196
1026	51.3	384
1548	76.9	569
2062	102.6	805
2584	128.2	102
3106	153.78	526
3624	179.25	646
4198	205.81	698
4666	230.38	769
5128	256.94	858
1708	85.4	240

Table 4.1.3  
DLAP for *o*-Xylene

concn (ng/mL)	mass on column (pg)	area counts ( $\mu$ V-s)
0	0	0
172	8.6	21
342	17.1	37
514	25.7	81
684	34.2	85
856	42.8	120
1028	51.4	143
1198	59.9	171
1370	68.5	174
1540	77.0	231
1712	85.6	235

Table 4.1.4  
DLAP for *p*-Xylene

concn (ng/mL)	mass on column (pg)	area counts ( $\mu$ V-s)
0	0	142
170	8.5	143
342	17.1	195
512	25.6	208
684	34.2	208
854	42.7	239
1024	51.2	278
1196	59.8	297
1366	68.3	327
1538	76.9	323
1708	85.4	368

Table 4.1.5  
DLAP for Ethylbenzene

concn (ng/mL)	mass on column (pg)	area counts ( $\mu$ V-s)
0	0	0
174	8.7	26
346	17.3	38
520	26.0	66
692	34.6	92
866	43.3	120
1040	52.0	146
1212	60.6	165
1386	69.3	177
1558	77.9	206
1732	86.6	233

#### 4.2 Detection limit of the overall procedure (DLOP) and reliable quantitation limit (RQL)

The DLOP is measured as mass per sample and expressed as equivalent air concentrations, based on the recommended sampling parameters. Ten 100-mg portions of SKC Lot 2000 charcoal, and ten 500-mg portions of SKC Anasorb 747, (representing SKC 575-002 Passive Samplers) were spiked with equal descending increments of analyte, such that the highest sampler loading would produce a peak approximately 10 times the response for a sample blank. These spiked samples, and sample blanks were analyzed with the recommended analytical parameters, and the data obtained used to calculate the required parameters (A and  $SEE_{DL}$ ) for the calculation of DLOP and RQL.

Xylenes DLOP and RQL were calculated by summing masses and areas for individual xylene isomers. Sample air volume and extraction efficiency for xylenes is the mean of those for individual xylene isomers. Table 4.2 is a summary of DLOP results and is presented for quick reference.

Table 4.2  
Detection Limit of the Overall Procedure Summary

analyte	charcoal tubes			SKC 575-002 Passive Samplers		
	ng	$\mu$ g/m <sup>3</sup>	ppb	ng	$\mu$ g/m <sup>3</sup>	ppb
xylenes	322	26.8	6.2	847	252.8	58.2
<i>m</i> -xylene	159	13.2	3.0	448	134.9	31.1
<i>o</i> -xylene	239	19.9	4.6	325	95.0	21.9
<i>p</i> -xylene	100	8.3	1.9	437	130.8	30.1
ethylbenzene	129	10.8	2.5	315	94.9	21.9

Table 4.2.1  
DLOP and RQL  
for Xylenes from Charcoal Tubes

mass per sample (ng)	area counts ( $\mu$ V-s)
0	139
513	238
1025	272
1538	357
2051	423
2563	485
3076	524
3590	624
4101	691
4614	763
5128	835

Table 4.2.2  
DLOP and RQL for Xylenes  
from SKC 575-002 Passive Samplers

mass per sample (ng)	area counts ( $\mu$ V-s)
0	136
1025	247
2051	278
3076	334
4101	452
5128	497
6152	588
7179	615
8204	696
9228	769
10255	844

Table 4.2.3  
DLOP and RQL  
for *m*-Xylene from Charcoal Tubes

mass per sample (ng)	area counts ( $\mu$ V-s)
0	0
171	39
341	54
512	74
683	109
853	116
1024	134
1195	168
1365	200
1536	215
1707	239

Table 4.2.4  
DLOP and RQL for *m*-Xylene from  
SKC 575-002 Passive Samplers

mass per sample (ng)	area counts ( $\mu$ V-s)
0	0
341	46
683	56
1024	61
1365	110
1707	116
2048	158
2390	175
2731	194
3072	226
3414	237

Table 4.2.5  
DLOP and RQL  
for *o*-Xylene from Charcoal Tubes

mass per sample (ng)	area counts ( $\mu$ V-s)
0	0
171	31
343	39
514	70
685	85
857	121
1028	115
1200	161
1371	168
1542	210
1714	239

Table 4.2.6  
DLOP and RQL for *o*-Xylene from  
SKC 575-002 Passive Samplers

mass per sample (ng)	area counts ( $\mu$ V-s)
0	0
343	29
685	35
1028	69
1371	96
1714	112
2056	126
2399	147
2742	164
3084	190
3427	223





Table 4.2.7  
DLOP and RQL  
for *p*-Xylene from Charcoal Tubes

mass per sample (ng)	area counts (μV-s)
0	139
171	168
341	179
512	213
683	229
853	248
1024	275
1195	295
1365	323
1536	338
1707	357

Table 4.2.8  
DLOP and RQL for *p*-Xylene from  
SKC 575-002 Passive Samplers

mass per sample (ng)	area counts (μV-s)
0	136
341	172
683	187
1024	204
1365	246
1707	269
2048	304
2390	293
2731	338
3072	353
3414	384

Table 4.2.9  
DLOP and RQL  
for Ethylbenzene from Charcoal Tubes

mass per sample (ng)	area counts (μV-s)
0	0
173	35
346	44
519	76
692	88
865	118
1038	136
1211	153
1384	182
1558	198
1731	231

Table 4.2.10  
DLOP and RQL for Ethylbenzene from  
SKC 575-002 Passive Samplers

mass per sample (ng)	area counts (μV-s)
0	0
346	25
692	52
1038	71
1384	90
1731	111
2077	143
2423	153
2769	186
3115	212
3461	251

The RQL is considered the lower limit for precise quantitative measurements. It is determined from the regression line parameters obtained for calculation of DLOP, providing the extraction efficiency (EE) is  $100 \pm 25\%$  at the RQL.

Table 4.2.11  
Reliable Quantitation Limits

analyte	charcoal tubes				SKC 575-002 Passive Samplers			
	ng	μg/m <sup>3</sup>	ppb	EE (%)	ng	μg/m <sup>3</sup>	ppb	EE (%)
xylene	1072	89.3	20.6	98.4	2823	842.7	194.1	93.6
<i>m</i> -xylene	531	44.2	10.2	98.9	1495	450.3	103.7	96.4
<i>o</i> -xylene	795	66.2	15.3	95.6	1084	317.0	73.0	84.9
<i>p</i> -xylene	334	27.8	6.4	99.9	1456	435.9	100.4	95.6
ethylbenzene	431	35.9	8.3	99.4	1049	316.0	72.8	97.6

### 4.3 Instrument calibration

The instrument was calibrated for xylene isomers and ethylbenzene over a range of from 0.5 to 2 times the 100 ppm PEL for each analyte. Calibration was performed at concentrations appropriate for both active and diffusive samplers. Calibration curves were constructed from the tabulated data and are shown in Section 3.5.3. Coefficients of determination ( $r^2$ ) and of nondetermination ( $k^2$ ) are shown in Table 4.3.

Table 4.3  
Coefficient of Determination ( $r^2$ )  
and of Nondetermination ( $k^2$ )

analyte	charcoal tubes		SKC 575-002 Pass Samplers	
	$r^2$	$k^2$	$r^2$	$k^2$
<i>m</i> -xylene	0.9994	$6 \times 10^{-4}$	0.9998	$2 \times 10^{-4}$
<i>o</i> -xylene	0.9995	$5 \times 10^{-4}$	0.9998	$2 \times 10^{-4}$
<i>p</i> -xylene	0.9994	$6 \times 10^{-4}$	0.9998	$2 \times 10^{-4}$
ethylbenzene	0.9994	$6 \times 10^{-4}$	0.9998	$2 \times 10^{-4}$

Table 4.3.1  
Instrument Response to  
*m*-Xylene for Charcoal Tubes

× OSHA PEL ( $\mu\text{g}/\text{sample}$ )	0.5×	0.75×	1×	1.5×	2×
area ( $\mu\text{V}\cdot\text{s}$ )	2568	3852	5136	7704	10272
	164794	269791	375905	593689	783759
	164754	269445	375908	592710	784944
	164983	269434	376753	593187	786776
	164992	269351	376901	592430	784541
	165047	269191	375901	591644	784130
	165182	268965	375624	593035	783251

Table 4.3.2  
Instrument Response to  
*o*-Xylene for Charcoal Tubes

× OSHA PEL ( $\mu\text{g}/\text{sample}$ )	0.5×	0.75×	1×	1.5×	2×
area ( $\mu\text{V}\cdot\text{s}$ )	2590	3884	5179	7769	10358
	167753	274897	383480	606183	801612
	167760	274708	383480	605143	802500
	167934	274660	384309	605576	804231
	167902	274497	384433	605029	802144
	167961	274406	383472	604460	801744
	168094	274212	383231	605477	800999

Table 4.3.3  
Instrument Response to  
*p*-Xylene for Charcoal Tubes

× OSHA PEL ( $\mu\text{g}/\text{sample}$ )	0.5×	0.75×	1×	1.5×	2×
area ( $\mu\text{V}\cdot\text{s}$ )	2559	3839	5118	7678	10237
	164552	268846	374277	590454	779090
	164539	268538	374271	589602	780366
	164734	268527	375116	590062	782170
	164768	268449	375271	589284	779941
	164807	268265	374253	588468	779572
	164923	268051	374021	589848	778654

Table 4.3.4  
Instrument Response to  
Ethylbenzene for Charcoal Tubes

× OSHA PEL ( $\mu\text{g}/\text{sample}$ )	0.5×	0.75×	1×	1.5×	2×
area ( $\mu\text{V}\cdot\text{s}$ )	2608	3912	5216	7824	10432
	164275	269560	375888	593883	783759
	164292	269199	375846	593063	785278
	164473	269224	376676	593502	787146
	164518	269089	376857	592685	784787
	164581	268885	375780	591749	784405
	164665	268679	375508	593173	783391

Table 4.3.5  
Instrument Response to *m*-Xylene  
for SKC 575-002 Passive Samplers

× OSHA PEL ( $\mu\text{g}/\text{sample}$ )	0.5×	0.75×	1×	1.5×	2×
area ( $\mu\text{V}\cdot\text{s}$ )	749	1177	1455	2140	2889
	29838	47626	58960	86200	117788
	29955	47689	59183	86119	117903
	29857	47832	59046	86097	117765
	29887	47847	59397	85956	118128
	29836	47796	59004	85933	117561
	29842	47774	59284	85854	117760

Table 4.3.6  
Instrument Response to *o*-Xylene  
for SKC 575-002 Passive Samplers

× OSHA PEL ( $\mu\text{g}/\text{sample}$ )	0.5×	0.75×	1×	1.5×	2×
area ( $\mu\text{V}\cdot\text{s}$ )	755	1187	1467	2158	2913
	30464	48643	60209	88024	120324
	30579	48697	60434	87973	120516
	30508	48848	60309	87915	120317
	30521	48880	60692	87801	120670
	30486	48780	60248	87757	120081
	30483	48795	60531	87695	120298

Table 4.3.7  
Instrument Response to *p*-Xylene  
for SKC 575-002 Passive Samplers

× OSHA PEL (µg/sample)	0.5×	0.75×	1×	1.5×	2×
area (µV-s)	29754	47433	58707	85808	117229
	29867	47507	58961	85716	117319
	29774	47680	58809	85692	117199
	29817	47672	59157	85590	117578
	29771	47625	58765	85545	116981
	29772	47597	59041	85468	117192

Table 4.3.8  
Instrument Response to Ethylbenzene  
for SKC 575-002 Passive Samplers

× OSHA PEL (µg/sample)	0.5×	0.75×	1×	1.5×	2×
area (µV-s)	29797	47592	58904	86193	117774
	29920	47668	59197	86089	117857
	29824	47822	59043	86057	117737
	29882	47826	59390	85970	118148
	29834	47764	58958	85917	117530
	29816	47738	59261	85830	117767

#### 4.4 Precision (overall procedure)

##### 4.4.1 Charcoal tubes

The precision at the 95% confidence level is obtained by multiplying the SEE by 1.96 (the z-statistic from the standard normal distribution at the 95% confidence level). In Section 4.5, 95% confidence intervals are drawn about their respective regression lines in the storage graph figures. The precisions of the overall procedure were obtained from the ambient temperature storage tests and are shown in Table 4.4.1.

Table 4.4.1  
SEEs and Precisions of the  
Overall Procedure for Charcoal Tubes

analyte	SEE (%)	precision (±%)
xylenes	5.50	10.8
<i>m</i> -xylene	5.45	10.7
<i>o</i> -xylene	5.59	11.0
<i>p</i> -xylene	5.67	11.1
ethylbenzene	5.41	10.6

##### 4.4.2 SKC 575-002 Passive Samplers

The precision at the 95% confidence level is obtained by multiplying the SEE by 1.96 (the z-statistic from the standard normal distribution at the 95% confidence level). In Section 4.5, 95% confidence intervals are drawn about their respective regression lines in the storage graph figures. Each precision includes an additional 8.7% for sampling rate variability. There are different values given, depending on whether both, either, or neither temperature or atmospheric pressure are known at the sampling site. If the sampling-site temperature (*T*) is unknown, it is assumed to be 22.2 ± 15 °C (72 ± 27 °F) and a variability of ±7.7% is included. If the atmospheric pressure (*P*) is unknown, it is estimated from sampling-site elevation and a variability of ±3% is included. The precisions of the overall procedure are shown in Table 4.4.2.

Table 4.4.2  
SEEs and Precisions of the Overall Procedure for SKC 575-002 Passive Samplers

condition	<u>xylenes</u>		<u><i>m</i>-xylene</u>		<u><i>o</i>-xylene</u>		<u><i>p</i>-xylene</u>		<u>ethylbenzene</u>	
	SEE (%)	precision (±%)	SEE (%)	precision (±%)	SEE (%)	precision (±%)	SEE (%)	precision (±%)	SEE (%)	precision (±%)
both <i>T</i> and <i>P</i> known	9.29	18.2	9.32	18.3	9.24	18.1	9.33	18.3	9.39	18.4
only <i>T</i> known	9.76	19.1	9.79	19.2	9.71	19.0	9.80	19.2	9.86	19.3
only <i>P</i> known	12.07	23.7	12.09	23.7	12.03	23.6	12.10	23.7	12.14	23.8
neither <i>T</i> nor <i>P</i> known	12.43	24.4	12.46	24.4	12.40	24.3	12.46	24.4	12.51	24.5

#### 4.5 Storage tests

##### 4.5.1 Charcoal tubes

Storage stability samples were prepared by sampling (at 50 mL/min for four hours) dynamically generated test atmospheres of mixed xylenes with SKC 226-01 sampling tubes. These samples were collected simultaneously along with diffusive samples. The concentrations of the test atmospheres were 207 mg/m<sup>3</sup> (48 ppm) for *m*-xylene, 96 mg/m<sup>3</sup> (22 ppm) for *o*-xylene, 90 mg/m<sup>3</sup> (21 ppm) for *p*-xylene, and 73 mg/m<sup>3</sup> (17 ppm) for

ethylbenzene at 83% relative humidity and 20 °C. Xylenes concentration was the sum of the individual isomers and was 393 mg/m<sup>3</sup> (91 ppm). These air concentrations were approximately one times the target concentration for xylenes and were in the same proportions as were the analytes in the mixed xylenes used to generate the test atmospheres. Xylenes results were calculated from summed individual isomers results. Sample results are corrected for extraction efficiency.

Table 4.5.1.1  
Storage Tests for Xylenes

time (days)	ambient storage recovery (%)			refrigerated storage recovery (%)		
0	97.0	98.9	96.8	97.0	98.9	96.8
3	101.1	102.5	100.3	103.1	101.3	100.3
7	102.2	97.4	103.1	96.3	100.2	100.8
10	96.5	97.9	99.2	100.4	98.2	98.8
14	101.6	96.7	97.9	101.3	99.4	100.2
16	99.8	97.0	101.1	101.7	93.8	102.0

Table 4.5.1.2  
Storage Tests for *m*-Xylene

time (days)	ambient storage recovery (%)			refrigerated storage recovery (%)		
0	97.3	99.1	97.2	97.3	99.1	97.2
3	101.4	102.8	100.6	103.3	101.6	100.6
7	102.5	97.8	103.3	97.6	100.7	101.2
10	97.0	98.3	99.5	100.7	98.7	99.2
14	101.9	97.2	99.8	101.6	99.7	100.5
16	100.6	98.7	101.9	102.0	93.7	102.3

Table 4.5.1.3  
Storage Tests for *o*-Xylene

time (days)	ambient storage recovery (%)			refrigerated storage recovery (%)		
0	96.1	98.4	95.9	96.1	98.4	95.9
3	100.7	101.8	99.5	102.5	100.7	99.6
7	101.5	96.3	102.6	95.6	99.2	100.0
10	95.2	97.0	98.4	99.6	96.9	97.7
14	101.0	95.6	98.5	100.6	98.8	99.5
16	99.5	95.8	100.6	101.1	94.4	101.4

Table 4.5.1.4  
Storage Tests for *p*-Xylene

time (days)	ambient storage recovery (%)			refrigerated storage recovery (%)		
0	97.0	98.8	96.8	97.0	98.8	96.8
3	101.1	102.5	100.3	103.0	101.3	100.3
7	102.1	97.5	103.0	96.4	100.3	100.9
10	96.6	98.0	99.2	100.4	98.3	98.9
14	101.6	96.8	93.1	101.3	99.4	100.2
16	99.6	97.0	101.1	101.7	93.5	101.9

Table 4.5.1.5  
Storage Tests for Ethylbenzene

time (days)	ambient storage recovery (%)			refrigerated storage recovery (%)		
0	98.2	99.7	98.3	98.2	99.7	98.3
3	102.1	103.6	101.6	104.0	102.3	101.3
7	103.1	99.0	103.9	97.5	101.9	102.2
10	98.2	99.3	100.3	101.6	100.1	100.3
14	102.5	98.6	100.8	102.3	100.6	101.2
16	100.0	97.5	101.4	102.6	93.5	103.0





#### 4.5.2 SKC 575-002 Passive Samplers

Storage stability samples were prepared by sampling dynamically generated test atmospheres of mixed xylenes with SKC 575-002 Passive Samplers. The face velocity of the test atmosphere was about 0.4m/s past the diffusive samplers. The samplers were orientated parallel to the flow direction. These samples were collected for four hours simultaneously along with active samples under conditions described in Section 4.7.1. Xylenes results were calculated from summed isomers results. Sample results are corrected for extraction efficiency.



Table 4.5.2.1  
Storage Tests for Xylenes

time (days)	ambient storage recovery (%)			refrigerated storage recovery (%)		
0	96.6	97.3	97.1	96.6	97.3	97.1
3	101.0	101.6	102.5	101.2	102.5	101.4
7	104.6	93.5	99.7	100.9	100.0	98.6
10	101.1	102.8	97.2	100.5	99.5	100.7
14	98.0	92.9	99.7	100.6	99.4	100.5
16	97.4	98.9	94.4	102.4	100.9	97.9

Table 4.5.2.2  
Storage Tests for *m*-Xylene

time (days)	ambient storage recovery (%)			refrigerated storage recovery (%)		
0	96.8	97.5	97.4	96.8	97.5	97.4
3	101.5	102.2	103.1	101.8	103.1	101.9
7	105.1	93.9	100.2	101.3	100.4	99.0
10	101.6	103.3	97.6	100.9	100.0	101.1
14	98.4	93.3	100.1	101.1	99.9	101.0
16	97.9	99.4	94.9	102.9	101.4	98.4

Table 4.5.2.3  
Storage Tests for *o*-Xylene

time (days)	ambient storage recovery (%)			refrigerated storage recovery (%)		
0	96.2	96.8	96.6	96.2	96.8	96.6
3	99.7	100.4	101.1	99.7	101.1	100.4
7	103.4	92.4	98.6	99.9	98.8	97.3
10	99.9	101.5	96.1	99.4	98.4	99.6
14	96.9	92.1	99.0	99.3	98.2	99.3
16	96.2	97.7	93.1	101.1	99.6	96.7

Table 4.5.2.4  
Storage Tests for *p*-Xylene

time (days)	ambient storage recovery (%)			refrigerated storage recovery (%)		
0	96.6	97.2	97.0	99.6	97.2	97.0
3	101.2	101.8	102.8	101.5	102.8	101.5
7	104.8	93.6	99.9	101.0	100.2	98.7
10	101.3	103.1	97.3	100.6	99.7	100.8
14	98.1	92.9	99.6	100.7	99.5	100.7
16	97.5	99.1	94.5	102.5	101.0	98.0

Table 4.5.2.5  
Storage Tests for Ethylbenzene

time (days)	ambient storage recovery (%)			refrigerated storage recovery (%)		
0	97.4	98.1	98.0	97.4	98.1	98.0
3	103.0	103.6	104.4	103.2	104.4	103.5
7	106.5	95.2	101.5	102.6	101.7	100.4
10	103.0	104.7	98.8	102.2	101.3	102.4
14	99.6	94.3	101.0	102.7	101.2	102.4
16	99.1	100.8	96.1	104.4	102.9	99.7





#### 4.6 Reproducibility

Twelve samples (six charcoal tubes and six SKC 575-002 Passive Samplers) were collected from controlled test atmospheres similar to that used to collect storage stability samples. The samples were submitted to SLTC for analysis. The charcoal tube samples were analyzed after 16 days of storage at ambient temperature, and the SKC 575-002 Passive Samplers after 22 days of storage at ambient temperature. Sample results were corrected for extraction efficiency. No sample result had a deviation greater than the precision of the overall procedure reported in Section 4.4. Xylenes results were calculated by summing results for individual isomers.

Table 4.6.1  
Reproducibility Data for Xylene Isomers on Charcoal Tubes

<i>m</i> -xylene				<i>o</i> -xylene				<i>p</i> -xylene			
theo µg/samp	reported µg/samp	recovery (%)	deviation (%)	theo µg/samp	reported µg/samp	recovery (%)	deviation (%)	theo µg/samp	reported µg/samp	recovery (%)	deviation (%)
2555	2552	99.9	-0.1	1189	1245	104.7	4.7	1112	1140	102.5	2.5
2595	2593	99.9	-0.1	1207	1266	104.9	4.9	1129	1158	102.6	2.6
2623	2427	92.5	-7.5	1221	1158	94.8	-5.2	1142	1082	94.7	-5.3
2557	2580	100.9	0.9	1190	1262	106.1	6.1	1129	1152	102.0	2.0
2564	2593	101.1	1.1	1193	1263	105.9	5.9	1116	1159	103.9	3.9
2623	2610	99.5	-0.5	1221	1275	104.4	4.4	1142	1166	102.1	2.1

Table 4.6.2  
Reproducibility Data for Xylenes and Ethylbenzene on Charcoal Tubes

xylenes				ethylbenzene			
theo µg/sample	reported µg/sample	recovery (%)	deviation (%)	theo µg/sample	reported µg/sample	recovery (%)	deviation (%)
4856	4937	101.7	1.7	904.8	929.4	102.7	2.7
4931	5017	101.7	1.7	918.9	944.2	102.8	2.8
4986	4667	93.6	-6.4	929.0	895.4	96.4	-3.6
4876	4994	102.4	2.4	905.6	939.1	103.7	3.7
4873	5014	102.9	2.9	911.8	945.6	103.7	3.7
4986	5051	101.3	1.3	929.0	944.3	101.6	1.6

Table 4.6.3  
Reproducibility Data for Xylene Isomers on SKC 575-002 Passive Samplers

<i>m</i> -xylene				<i>o</i> -xylene				<i>p</i> -xylene			
theo µg/samp	reported µg/samp	recovery (%)	deviation (%)	theo µg/samp	reported µg/samp	recovery (%)	deviation (%)	theo µg/samp	reported µg/samp	recovery (%)	deviation (%)
847.6	859.1	101.4	1.4	401.3	430.8	107.4	7.4	372.8	404.9	108.6	8.6
847.6	823.9	97.2	-2.8	401.3	417.1	103.9	3.9	372.8	389.1	104.4	4.4
847.6	811.5	95.7	-4.3	401.3	408.8	101.9	1.9	372.8	383.0	102.7	2.7
847.6	806.0	95.1	-4.9	401.3	399.0	99.4	-0.6	372.8	381.0	102.2	2.2
847.6	805.5	95.0	-5.0	401.3	406.3	101.2	1.2	372.8	380.2	102.0	2.0
847.6	803.3	94.8	-5.2	401.3	398.9	99.4	-0.6	372.8	380.4	102.0	2.0

Table 4.6.4  
Reproducibility Data for Xylenes and Ethylbenzene on SKC 575-002 Passive Samplers

xylenes				ethylbenzene			
theo µg/sample	reported µg/sample	recovery (%)	deviation (%)	theo µg/sample	reported µg/sample	recovery (%)	deviation (%)
1622	1695	104.5	4.5	302.3	312.2	103.3	3.3
1622	1630	100.5	0.5	302.3	298.3	98.7	-1.3
1622	1603	98.8	-1.2	302.3	293.9	97.2	-2.8
1622	1586	97.8	-2.2	302.3	295.0	97.6	-2.4
1622	1592	98.2	-1.8	302.3	291.7	96.5	-3.5
1622	1583	97.6	-2.4	302.3	293.7	97.2	-2.8

## 4.7 Sampler capacity

### 4.7.1 Charcoal tubes

The sampling capacity of charcoal tubes was tested by sampling dynamically generated test atmospheres of mixed xylenes with SKC 226-01 (Lot 2000) sampling tubes. These samples were collected simultaneously along with diffusive samples. The sampling times were 5, 10, 15, and 30 min; and 1, 2, 3, 4, 6, 8, and 10 hours. Three active and three diffusive samples were collected for each time period. The mean concentrations of the test atmospheres were 456 mg/m<sup>3</sup> (105 ppm) for *m*-xylene, 212 mg/m<sup>3</sup> (49 ppm) for *o*-xylene, 198 mg/m<sup>3</sup> (46 ppm) for *p*-xylene, and 161 mg/m<sup>3</sup> (37 ppm) for ethylbenzene at 78% relative humidity and 21 °C. These air concentrations were approximately two times the target concentration for xylenes and were in the same proportions as were the analytes in the mixed xylenes used to generate the test atmospheres. No breakthrough from the front to the back section of the sampling tubes for any of the analytes was observed even when samples were collected for ten hours at 50 mL/min. Sampler capacity was never exceeded. Nearly 31 mg of mixed xylenes had been collected after ten hours. The recommended sampling time was set at four hours and the recommended sampling rate at 50 mL/min. These tests also showed that samples can be collected for as short a time as 5 min at 50 mL/min and still provide excellent results.

### 4.7.2 SKC 575-002 Passive Samplers

The sampling rate and sampler capacity of SKC 575-002 Passive Samplers were determined with samples collected at the increasing time intervals from the controlled test atmospheres described in Section 4.7.1. The face velocity of the test atmosphere was approximately 0.4 m/s, and the samplers were orientated parallel to the flow direction. Three samples were collected at each time interval. Sampler capacity has been defined to be exceeded when the "apparent" sampling rate decreases rapidly. The sampling rate only appears to decrease because the sampler can collect no additional analyte at the point when capacity is exceeded. Sampling rates are presented in mL/min at 760 mmHg and 25 °C.

Table 4.7.2  
Determination of Sampling Rate and Recommended Sampling Time

time (h)	<i>m</i> -xylene		<i>o</i> -xylene		<i>p</i> -xylene		ethylbenzene	
	mL/min	RSD	mL/min	RSD	mL/min	RSD	mL/min	RSD
0.083	13.75	2.2	14.25	3.8	13.99	2.8	13.85	2.3
0.167	13.53	1.4	13.97	1.3	13.71	1.8	13.68	1.5
0.25	13.91	0.9	14.38	0.9	14.01	0.9	13.96	0.8
0.5	13.94	1.6	14.39	2.4	14.07	1.7	13.93	1.2
1	13.81	2.1	14.17	2.4	13.94	2.2	13.86	2.2
2	13.55	1.7	13.79	1.8	13.59	1.7	13.50	2.0
3	14.01	1.4	14.27	1.4	14.04	1.4	13.96	1.5
4	13.60	0.6	14.93	0.9	13.67	0.7	13.59	0.6
6	14.20	5.0	14.40	5.0	14.48	5.0	14.19	5.0
8	13.60	0.2	13.70	0.1	13.70	0.2	13.59	0.2
10	14.11	2.6	14.35	2.5	14.16	2.7	14.06	2.6
mean	13.82		14.24		13.94		13.83	
RSD	1.7		2.4		1.9		1.6	

The preliminary sampling rate was determined by averaging the values for the 0.5, 1, and 2 hour samples. Horizontal lines were constructed 10% above and 10% below the preliminary sampling rate. All the sampling rates were included in the calculated mean sampling rates because all were between the two horizontal lines.

#### 4.8 Extraction efficiency and stability of extracted samples

Each laboratory must determine and confirm extraction efficiency periodically. Other solvents can be used in conjunction with this method provided the new solvent is tested. The new solvent should be tested as described below and the extraction efficiency must be greater than 75%.

A summary of the extraction efficiency results over the range of RQL to 2 times the target concentration is presented in Table 4.8 for quick reference.

Table 4.8  
Extraction Efficiency (%) Summary

analyte	charcoal tubes	SKC 575-002 Passive Samplers
<i>m</i> -xylene	96.3	96.1
<i>o</i> -xylene	93.8	89.4
<i>p</i> -xylene	96.1	95.3
ethylbenzene	97.2	99.1

#### 4.8.1 Charcoal tubes

The extraction efficiencies (EEs) of the analytes were determined by liquid-spiking 100-mg portions of SKC Lot 2000 charcoal with the analytes at levels from the RQL to 2 times the OSHA PEL for each analyte. These samples were stored overnight at ambient temperature, and then extracted with 1mL of CS<sub>2</sub> (containing 1 µL of *p*-cymene per mL of CS<sub>2</sub>) for 1 hour. The samples were vigorously shaken periodically over the extraction time.

The EEs of the analytes at the target concentration were also determined from “wet” charcoal to confirm that EE remained constant. Wet charcoal was prepared by collecting samples from a humid (about 80% RH and 22 °C) atmosphere at 50 mL/min for 4 hours. Only the front section of these samples was used to prepare wet EE samples. The stability of extracted samples was investigated by reanalyzing the 1× PEL samples a day after the original analysis. Three vials were immediately resealed with new septa caps and three vials retained their punctured septa following the original analysis.

Table 4.8.1.1  
Extraction Efficiency of *m*-Xylene from SKC Lot 2000 Charcoal

× OSHA PEL	level	sample number						mean
	µg per sample	1	2	3	4	5	6	
RQL	0.548	107.1	104.6	97.9	92.7	95.4	95.7	98.9
0.05	256	96.9	96.1	95.3	94.7	94.6	95.8	95.6
0.1	512	96.5	99.7	96.3	94.0	96.7	94.0	96.2
0.2	1024	96.5	95.2	94.6	96.5	95.6	94.4	95.5
0.5	2560	97.7	96.0	94.6	93.9	95.0	95.3	95.4
1.0	5136	97.0	96.5	96.5	97.6	98.1	97.9	97.3
2.0	10486	93.7	95.6	94.3	97.6	94.5	95.5	95.2
wet (1.0)	5136	98.0	96.4	97.7	97.4	96.6	97.9	97.3

The mean EE at concentrations from the RQL to 2 times the PEL (excepting the wet EE) is 96.3%.

Table 4.8.1.2  
Extraction Efficiency of *o*-Xylene from SKC Lot 2000 Charcoal

× OSHA PEL	level	sample number						mean
	µg per sample	1	2	3	4	5	6	
RQL	0.785	99.6	96.9	100.8	88.4	92.8	95.0	95.6
0.05	257	93.8	93.0	92.4	91.6	91.6	92.7	92.5
0.1	514	93.5	96.7	93.3	91.2	93.7	91.2	93.3
0.2	1028	93.6	92.3	91.8	93.6	92.6	91.4	92.6
0.5	2570	94.9	93.2	92.0	91.3	92.4	92.7	92.8
1.0	5179	94.6	94.0	94.0	95.1	95.4	95.4	94.8
2.0	10575	93.2	94.6	93.9	98.5	93.9	94.5	94.8
wet (1.0)	5179	95.2	93.8	95.1	94.8	93.9	95.1	94.7

The mean EE at concentrations from the RQL to 2 times the PEL (excepting the wet EE) is 93.8%.

Table 4.8.1.3  
Extraction Efficiency of *p*-Xylene from SKC Lot 2000 Charcoal

× OSHA PEL	level	sample number						mean
	µg per sample	1	2	3	4	5	6	
RQL	0.788	102.1	96.9	100.7	103.3	105.6	90.8	99.9
0.05	256	96.5	95.7	95.0	94.3	94.2	95.3	95.2
0.1	512	96.1	99.3	95.9	93.7	96.3	93.3	95.8
0.2	1024	96.2	94.8	94.2	96.1	95.1	94.0	95.1
0.5	2560	97.3	95.6	94.2	93.6	94.6	94.9	95.0
1.0	5118	96.6	96.0	96.0	97.2	97.7	97.5	96.8
2.0	10450	93.2	95.2	93.8	96.9	94.1	95.1	94.7
wet (1.0)	5118	97.5	95.9	97.3	96.9	96.2	97.5	96.9



The mean EE at concentrations from the RQL to 2 times the PEL (excepting the wet EE) is 96.1%.

Table 4.8.1.4  
Extraction Efficiency of Ethylbenzene from SKC Lot 2000 Charcoal

× OSHA PEL	level	sample number						mean
	µg per sample	1	2	3	4	5	6	
RQL	0.43	96.9	97.1	96.2	100.7	109.1	96.6	99.4
0.05	260	98.3	97.4	96.6	95.9	95.9	97.1	96.9
0.1	519	97.8	101.2	97.6	95.4	98.0	95.2	97.5
0.2	1038	97.9	96.5	95.8	97.9	96.8	95.8	96.8
0.5	2596	99.0	97.3	95.8	95.1	96.2	96.5	96.7
1.0	5216	98.2	97.7	99.0	98.8	99.3	99.1	98.7
2.0	10650	93.0	95.7	93.7	95.3	94.2	95.6	94.6
wet (1.0)	5216	99.3	97.7	99.0	98.6	97.9	99.3	98.6

The mean EE at concentrations from the RQL to 2 times the PEL (excepting the wet EE) is 97.2%.

Table 4.8.1.5  
Stability of *m*-Xylene Extracted from SKC Lot 2000 Charcoal

punctured septa replaced			punctured septa retained		
initial EE (%)	EE after one day (%)	difference (%)	initial EE (%)	EE after one day (%)	difference (%)
97.0	97.4	0.4	97.6	96.1	-1.5
96.5	97.1	0.6	98.1	96.7	-1.4
96.5	98.2	1.7	97.9	95.9	-2.0
	mean			mean	
96.7	97.6	0.9	97.9	96.2	-1.6

Table 4.8.1.6  
Stability of *o*-Xylene Extracted from SKC Lot 2000 Charcoal

punctured septa replaced			punctured septa retained		
initial EE (%)	EE after one day (%)	difference (%)	initial EE (%)	EE after one day (%)	difference (%)
94.6	95.1	0.5	95.1	93.6	-1.5
94.0	94.6	0.6	95.4	94.1	-1.3
94.0	95.8	1.8	95.4	93.2	-2.2
	mean			mean	
94.2	95.2	1.0	95.3	93.6	-1.7

Table 4.8.1.7  
Stability of *p*-Xylene Extracted from SKC Lot 2000 Charcoal

punctured septa replaced			punctured septa retained		
initial EE (%)	EE after one day (%)	difference (%)	initial EE (%)	EE after one day (%)	difference (%)
96.6	97.0	0.4	97.2	95.6	-1.6
96.0	96.7	0.7	97.7	96.2	-1.5
96.0	97.8	1.8	97.5	95.3	-2.2
	mean			mean	
96.2	97.2	1.0	97.5	95.7	-1.8

Table 4.8.1.8  
Stability of Ethylbenzene Extracted from SKC Lot 2000 Charcoal

punctured septa replaced			punctured septa retained		
initial EE (%)	EE after one day (%)	difference (%)	initial EE (%)	EE after one day (%)	difference (%)
98.2	98.6	0.4	98.8	97.2	-1.6
97.7	98.2	0.5	99.3	97.8	-1.5
99.0	99.4	0.4	99.1	97.0	-2.1
	mean			mean	

98.3

98.7

0.4

99.1

97.3

-1.7

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#### 4.8.2 SKC 575-002 Passive Samplers

The extraction efficiencies (EE) of the analytes were determined by liquid-spiking 500-mg portions of SKC Anasorb 747 (the sorbent in SKC 575-002 Passive Samplers) with the analytes at levels from the RQL to 2 times the OSHA PEL for each analyte. These samples were stored overnight at ambient temperature, and then extracted with 2 mL of CS<sub>2</sub> (containing 1 µL of 1-phenylhexane per mL of CS<sub>2</sub>) for 1 hour. The samples were vigorously shaken periodically over the extraction time. The EEs of the analytes at the target concentration were also determined from “wet” samplers to confirm that EE remained constant. Wet SKC 575-002 Passive Samplers were prepared by sampling from a humid (about 80% RH and 22 °C) atmosphere for 4 hours. These samples were extracted with 2 mL of CS<sub>2</sub> (containing 1 µL of 1-phenylhexane per mL of CS<sub>2</sub>) for 1 hour on a SKC 226D-03K Desorption Shaker. The stability of extracted samples was investigated by reanalyzing the 1× PEL samples a day after the original analysis. Three vials were immediately resealed with new septa caps and three vials retained their punctured septa following the original analysis.

Table 4.8.2.1  
Extraction Efficiency of *m*-Xylene from SKC Anasorb 747

× OSHA PEL	level		sample number						mean
	µg per sample		1	2	3	4	5	6	
RQL	1.422		93.0	90.1	99.0	89.9	105.4	100.9	96.4
0.05	73		98.6	97.7	97.6	97.2	98.2	97.6	97.8
0.1	145		95.9	95.9	93.7	95.0	95.8	95.3	95.3
0.2	290		93.2	93.6	94.2	94.1	94.9	95.6	94.3
0.5	725		94.1	95.1	95.6	96.3	94.7	97.1	95.5
1.0	1451		97.2	96.1	101.1	95.8	96.0	97.0	97.2
2.0	2902		99.3	95.3	96.0	95.8	96.4	95.8	96.4
wet (1.0)	1498		96.6	96.7	93.8	98.0	96.7	95.3	96.2

The mean EE at concentrations from the RQL to 2 times the PEL (excepting the wet EE) is 96.1%.

Table 4.8.2.2  
Extraction Efficiency of *o*-Xylene from SKC Anasorb 747

× OSHA PEL	level		sample number						mean
	µg per sample		1	2	3	4	5	6	
RQL	1.075		83.9	76.4	86.3	87.8	86.6	88.1	84.9
0.05	73		92.3	90.9	91.0	90.4	91.8	91.5	91.3
0.1	146		89.8	89.6	87.5	89.0	89.9	89.3	89.2
0.2	292		87.7	88.0	88.4	88.4	89.0	89.8	88.6
0.5	728		88.4	89.2	89.8	90.4	88.8	91.2	89.6
1.0	1456		91.5	90.5	95.3	90.1	90.4	91.3	91.5
2.0	2913		93.7	88.7	90.7	90.5	90.9	90.4	90.8
wet (1.0)	1511		91.0	90.8	88.3	92.2	91.1	89.9	90.6

The mean EE at concentrations from the RQL to 2 times the PEL (excepting the wet EE) is 89.4%.

Table 4.8.2.3  
Extraction Efficiency of *p*-Xylene from SKC Anasorb 747

× OSHA PEL	level		sample number						mean
	µg per sample		1	2	3	4	5	6	
RQL	1.542		98.2	95.7	92.2	90.9	97.3	99.3	95.6
0.05	73		97.6	97.2	96.9	96.0	97.4	96.7	97.0
0.1	145		95.0	94.8	93.0	94.3	95.1	94.7	94.5
0.2	290		92.4	92.9	93.4	93.3	94.2	94.7	93.5
0.5	725		93.3	94.3	94.8	95.4	93.9	96.2	94.7
1.0	1451		96.3	95.2	100.2	94.9	95.1	96.1	96.3
2.0	2902		99.7	94.5	95.2	95.0	95.6	94.9	95.8

wet (1.0)	1493	95.7	95.7	92.9	97.0	95.8	94.4	95.3
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The mean EE at concentrations from the RQL to 2 times the PEL (excepting the wet EE) is 95.3%.

Table 4.8.2.4  
Extraction Efficiency of Ethylbenzene from SKC Anasorb 747

× OSHA PEL	level	sample number						mean
	µg per sample	1	2	3	4	5	6	
RQL	1.058	99.7	105.0	93.7	88.5	95.2	103.4	97.6
0.05	74	102.2	101.2	101.1	100.2	101.2	100.8	101.1
0.1	147	99.1	99.1	97.3	98.6	99.3	98.6	98.7
0.2	294	96.3	96.8	97.5	97.2	98.4	98.8	97.5
0.5	736	97.3	98.4	98.9	99.5	98.3	100.5	98.8
1.0	1471	100.5	99.3	104.4	99.0	99.1	100.3	100.4
2.0	2942	102.6	98.3	99.1	98.8	99.6	98.9	99.6
wet (1.0)	1521	99.8	100.1	97.0	101.3	100.0	98.4	99.4

The mean EE at concentrations from the RQL to 2 times the PEL (excepting the wet EE) is 99.1%.

Table 4.8.2.5  
Stability of *m*-Xylene Extracted from SKC Anasorb 747

punctured septa replaced			punctured septa retained		
initial EE (%)	EE after one day (%)	difference (%)	initial EE (%)	EE after one day (%)	difference (%)
97.2	97.8	0.6	95.8	94.2	-1.6
96.1	95.4	-0.7	96.0	94.0	-2.0
101.1	100.0	-1.1	97.0	95.7	-1.3
	mean			mean	
98.1	97.7	-0.4	96.3	94.6	-1.6

Table 4.8.2.6  
Stability of *o*-Xylene Extracted from SKC Anasorb 747

punctured septa replaced			punctured septa retained		
initial EE (%)	EE after one day (%)	difference (%)	initial EE (%)	EE after one day (%)	difference (%)
91.5	91.9	0.4	90.1	88.8	-1.3
90.5	89.7	-0.8	90.4	88.7	-1.7
95.3	94.2	-1.1	91.3	90.2	-1.1
	mean			mean	
92.4	91.9	-0.5	90.6	89.2	-1.4

Table 4.8.2.7  
Stability of *p*-Xylene Extracted from SKC Anasorb 747

punctured septa replaced			punctured septa retained		
initial EE (%)	EE after one day (%)	difference (%)	initial EE (%)	EE after one day (%)	difference (%)
96.3	96.9	0.6	94.9	93.5	-1.4
95.2	94.6	-0.6	95.1	93.3	-1.8
100.2	99.3	-0.9	96.1	94.9	-1.2
	mean			mean	
97.2	96.9	-0.3	95.4	93.9	-1.5

Table 4.8.2.8  
Stability of Ethylbenzene Extracted from SKC Anasorb 747

punctured septa replaced			punctured septa retained		
initial EE (%)	EE after one day (%)	difference (%)	initial EE (%)	EE after one day (%)	difference (%)
100.5	101.0	0.5	99.0	97.3	-1.7
99.3	98.7	-0.6	99.1	97.1	-2.0
104.4	103.4	-1.0	100.3	98.8	-1.5
	mean			mean	

101.4

101.0

-0.4

99.5

97.7

-1.7

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## 4.9 Interferences (sampling)

### 4.9.1 Charcoal tubes

#### Retention

The ability of charcoal tubes to retain mixed xylenes after collection was tested by sampling a test atmosphere containing 454 mg/m<sup>3</sup>, 211 mg/m<sup>3</sup>, 198 mg/m<sup>3</sup>, and 161 mg/m<sup>3</sup> *m*-xylene, *o*-xylene, *p*-xylene, and ethylbenzene, respectively (at 80% RH and 20 °C) with six samplers for one hour at 50 mL/min. Three samples were analyzed immediately and three were used to sample contaminant-free humid air for an additional three hours, and then analyzed. All the samples in the second set retained at least 101.0, 101.2, 100.8, 101.0% of the means of the first set for *m*-xylene, *o*-xylene, *p*-xylene, and ethylbenzene, respectively.

Table 4.9.1  
Retention of Mixed Xylenes on Charcoal Tubes

	<i>m</i> -xylene (mg/m <sup>3</sup> ) (% mean)		<i>o</i> -xylene (mg/m <sup>3</sup> ) (% mean)		<i>p</i> -xylene (mg/m <sup>3</sup> ) (% mean)		ethylbenzene (mg/m <sup>3</sup> ) (% mean)	
1 <sup>st</sup> set								
1	443.9		203.9		192.6		158.6	
2	428.5		194.6		185.7		154.8	
3	447.4		206.1		194.1		159.5	
mean	439.9		201.5		190.8		157.6	
2 <sup>nd</sup> set								
1	444.3	101.0	203.9	101.2	192.4	100.8	159.2	101.0
2	453.4	103.1	208.3	103.4	196.7	103.1	161.9	102.7
3	449.5	102.2	207.2	102.8	195.0	102.2	159.5	101.2

#### Low relative humidity

The ability of charcoal tubes to collect mixed xylenes at low humidity was tested by sampling a test atmosphere containing 468 mg/m<sup>3</sup>, 218 mg/m<sup>3</sup>, 204 mg/m<sup>3</sup>, 166 mg/m<sup>3</sup> *m*-xylene, *o*-xylene, *p*-xylene, and ethylbenzene, respectively (at 5% RH and 20 °C) with three samplers for four hours at 50 mL/min. The samples were analyzed immediately. The sample results (when compared to theoretical concentrations) were 102.0, 100.4, and 99.8% for *m*-xylene; 101.4, 100.4, and 99.2% for *o*-xylene; 101.6, 100.1, and 99.6% for *p*-xylene; and 102.5, 100.8, and 100.4% for ethylbenzene.

#### Low concentration

The ability of charcoal tubes to collect mixed xylenes at low concentrations was tested by sampling a test atmosphere containing 22 mg/m<sup>3</sup>, 10 mg/m<sup>3</sup>, 10 mg/m<sup>3</sup>, 8 mg/m<sup>3</sup> *m*-xylene, *o*-xylene, *p*-xylene, and ethylbenzene, respectively (at 80% RH and 22 °C) with three samplers for four hours at 50 mL/min. The samples were analyzed immediately. The sample results (when compared to theoretical concentrations) were 95.2, 97.6, and 102.2 for *m*-xylene; 94.6, 96.1, and 101.2% for *o*-xylene; 95.0, 97.2, and 101.8% for *p*-xylene; and 96.1, 98.6, and 102.5% for ethylbenzene.

#### Interference

The ability of charcoal tubes to collect mixed xylenes in the presence of sampling interferences was tested by sampling a test atmosphere containing 230 mg/m<sup>3</sup>, 107 mg/m<sup>3</sup>, 100 mg/m<sup>3</sup>, 82 mg/m<sup>3</sup>, 365 mg/m<sup>3</sup>, 372 mg/m<sup>3</sup> *m*-xylene, *o*-xylene, *p*-xylene, ethylbenzene, toluene, and butyl acetate respectively (at 81% RH and 21 °C) with three samplers for four

hours at 50 mL/min. The samples were analyzed immediately. The sample results (when compared to theoretical concentrations) were 103.3, 102.9, and 101.8 for *m*-xylene; 102.8, 102.5, and 101.2% for *o*-xylene; 103.2, 102.6, and 101.6% for *p*-xylene; and 104.1, 103.4, and 102.6% for ethylbenzene.

#### 4.9.2 SKC 575-002 Passive Samplers

##### Reverse diffusion

The ability of SKC 575-002 Passive Samplers to retain mixed xylenes after collection was tested by sampling a test atmosphere containing 454 mg/m<sup>3</sup>, 211 mg/m<sup>3</sup>, 198 mg/m<sup>3</sup>, and 161 mg/m<sup>3</sup> *m*-xylene, *o*-xylene, *p*-xylene, and ethylbenzene, respectively (at 80% RH and 20 °C) with six samplers for one hour. Three samples were analyzed immediately and three were used to sample contaminant-free humid air for an additional three hours, and then analyzed. Sampling rates from Section 4.7 were converted to their equivalents under experimental temperature and pressure and used to calculate results in Table 4.9.2. All the samples in the second set retained at least 100.2, 99.6, 99.8, and 100.0% of the means of the first set for *m*-xylene, *o*-xylene, *p*-xylene, and ethylbenzene, respectively.

Table 4.9.2  
Retention of Mixed Xylenes on SKC 575-002 Passive Samplers

	<i>m</i> -xylene		<i>o</i> -xylene		<i>p</i> -xylene		ethylbenzene	
	(mg/m <sup>3</sup> )	(% mean)	(mg/m <sup>3</sup> )	(% mean)	(mg/m <sup>3</sup> )	(% mean)	(mg/m <sup>3</sup> )	(% mean)
1 <sup>st</sup> set								
1	450.6		206.9		195.7		161.2	
2	449.4		208.0		197.0		162.8	
3	419.5		191.9		182.0		149.9	
mean	439.8		202.3		191.6		158.0	
2 <sup>nd</sup> set								
1	440.7	100.2	201.4	99.6	191.2	99.8	157.9	100.0
2	449.0	102.1	206.0	101.8	195.0	101.8	160.8	101.8
3	449.4	102.2	205.9	101.8	195.3	101.9	161.4	102.2

##### Low relative humidity

The ability of SKC 575-002 Passive Samplers to collect mixed xylenes at low humidity was tested by sampling a test atmosphere containing 468 mg/m<sup>3</sup>, 218 mg/m<sup>3</sup>, 204 mg/m<sup>3</sup>, 166 mg/m<sup>3</sup> *m*-xylene, *o*-xylene, *p*-xylene, and ethylbenzene, respectively (at 5% RH and 20 °C) with three samplers for four hours. The samples were analyzed immediately. Sampling rates (760 mmHg and 25 °C) were 13.92, 13.63, and 13.94 mL/min for *m*-xylene; 14.23, 13.93, and 14.24 mL/min for *o*-xylene; 14.05, 13.77, and 14.08 mL/min for *p*-xylene; and 14.01, 13.73, and 14.02 mL/min for ethylbenzene.

##### Low concentration

The ability of SKC 575-002 Passive Samplers to collect mixed xylenes at low concentrations was tested by sampling a test atmosphere containing 22 mg/m<sup>3</sup>, 10 mg/m<sup>3</sup>, 10 mg/m<sup>3</sup>, 8 mg/m<sup>3</sup> *m*-xylene, *o*-xylene, *p*-xylene, and ethylbenzene, respectively (at 80% RH and 22 °C) with three samplers for four hours. The samples were analyzed immediately. Sampling rates (760 mmHg and 25 °C) were 13.44, 13.68, and 13.16 mL/min for *m*-xylene; 13.90, 14.28, and 13.53 mL/min for *o*-xylene; 13.32, 13.69, and 13.36 mL/min for *p*-xylene; and 13.71, 13.76, and 13.37 mL/min for ethylbenzene.

##### Interference

The ability of SKC 575-002 Passive Samplers to collect mixed xylenes in the presence of sampling interferences was tested by sampling a test atmosphere containing 230 mg/m<sup>3</sup>, 107 mg/m<sup>3</sup>, 100 mg/m<sup>3</sup>, 82 mg/m<sup>3</sup>, 365 mg/m<sup>3</sup>, 372 mg/m<sup>3</sup> *m*-xylene, *o*-xylene, *p*-xylene, ethylbenzene, toluene, and butyl acetate respectively (at 81% RH and 21 °C) with three samplers for four hours. The samples were analyzed immediately. Sampling rates (760 mmHg and 25 °C) were 13.73, 13.12, and 13.77 mL/min for *m*-xylene; 14.06, 13.43, and 14.08 mL/min for *o*-xylene; 13.86, 13.25, and 13.90 mL/min for *p*-xylene; and 13.80, 13.18, and 13.86 mL/min for ethylbenzene.

#### 4.10 Qualitative analysis

The identity of suspected mixed xylenes can be confirmed by GC/mass spectrometry. Mass spectra for the analytes are presented below.