SCAQMD METHOD 310-91

DETERMINATION OF PERCHLOROETHYLENE

1. Principle

A sample is cleaned up, if necessary, by vacuum distillation and analyzed by gas chromatography (GC) with thermal conductivity detection.

2. Equipment

2.1 Vacuum distillation apparatus with heated oil bath and liquid nitrogen cold trap. See SCAQMD Method 302.

2.2 Gas Chromatograph equipped with thermal conductivity detector and auto injector system

2.3 Integrator

2.4 Columns: 10% SP^{R}-1000, 20'X1/8" or a DB-Wax^{R}, 30 m X 0.53 mm, 1 um film thickness

2.5 Analytical balance capable to weighing to 0.1 mg

2.6 Flasks, volumetric, 50 mL, Class A

2.7 Pipettes, volumetric, 1 mL and 5 mL, Class A

2.8 Vials, 15 mL, screw thread

3. Reagents

3.1 Perchloroethylene, >99% purity

3.2 Methyl ethyl ketone (MEK), >99% purity

3.3 Isooctane, >99% purity

3.4 Helium, carrier gas, 99.5% purity

4. Procedure

4.1 Sample preparation

4.1.1 If the sample is a homogeneous liquid, determine the density of the sample (Ds) by ASTM D 1475.

4.1.2 If the sample is not a homogeneous liquid, distill a portion of the sample by SCAQMD Method 302.

4.1.2.1 For a homogeneous distillate, record the distillate volume, (Vdis), and transfer the distillate to a clean vial.

4.1.2.2 For a two-phase distillate, record the volume of the organic phase, (Vo), and transfer the organic phase to a clean vial.

4.1.3 Pipette 5 mL of sample* and 1 mL MEK into a tared 50 mL volumetric flask. Tare weight of 50 mL volumetric flask is Wx. Fill to the mark with isooctane. Record the total weight, (Wy).

4.1.3.1 *Sample in 4.1.3 is either undistilled sample, homogeneous distillate, or the organic portion of a multi-layer distillate.

4.2 Preparation of Perchloroethylene Standard

4.2.1 Standard: Accurately pipette 1 mL each perchloroethylene, (Vps), and MEK into a tared, 50 mL volumetric flask. Tare weight of 50 mL volumetric flask is Wa. Fill to the mark with isooctane. Record the total weight, (Wb).

4.3 Gas Chromatography

4.3.1 GC Parameters

4.3.1.1

Column:	10% SP^{R}-1000, 20' X 1/8"
Flow:	20.7 mL/min He
Injector Temp:	200^{o}C
Detector Temp:	190^{o}C
Oven Temp:	120^{o}C Isothermal
Injection Volume:	1 ul

4.3.1.2

Column: DB-Wax^{R}, 30 m X 0.53 mm, 1 um film thickness	
Column Flow:	15 mL/min He
Reference flow:	22 mL/min He
Injector Temp:	200^{o}C
Detector Temp:	190^{o}C
Oven Temp:	80^{o}C Isothermal
Injection Volume:	1 ul

4.3.2 Inject separately, 1 uL standard and 1 uL samples in duplicate. After a maximum of 10 samples (twenty injections), inject the standard.

4.3.3 The area response of duplicate injections must be within $\pm$10% of each other.

4.3.4 The calibration factor obtained from standards injected in duplicate must be within $\pm$10% of each other.

4.3.5 If the area response of the perchloroethylene in the sample preparation is not greater than 20% of the area response of the standard, adjust the sample preparation accordingly.

5. Calculations

5.1 Weight percent of perchloroethylene in the standard, (Wps), is calculated by:

$$Wps = \frac{Vps \times Dp}{Wb - Wa} \times 100$$

Vps = Volume of perchloroethylene added, mL, (Sec. 4.2.1)
Dp = Density of perchloroethylene, g/mL
Wb-Wa = Weight of perchloroethylene standard solution, g, (Sec. 4.2.1)

5.2 Response factor

$$R = \frac{(Wps)(Ais)}{Aperc}$$

Ais = Average area of MEK in standard
Aperc = Average area of perchloroethylene in standard
Wps = Weight percent perchloroethylene in standard (See 5.1)

5.3 Weight percent perchloroethylene in the prepared sample (Wpp):

$$Wpp = \frac{Asmpl}{Aisp} \times R$$

Asmpl = Average area of perchloroethylene in preparation
Aisp = Average area of MEK in prepared sample
R = Response factor (See 5.2)

5.4 Weight percent perchloroethylene in original sample.

5.4.1 Samples which are <u>not</u> distilled

$$\text{Wt. \% perc} = \frac{(Wpp) \times (Wy-Wx)}{V \times Ds}$$

5.4.2 Samples with homogeneous distillate

$$\text{Wt. \% perc} = \frac{(Wpp) \times (Wy-Wx) \times (Vdis)}{V \times Wsd}$$

5.4.3 Samples with multi-layer distillate

$$\text{Wt. \% perc} = \frac{(Wpp) \times (Wy-Wx) \times (Vo)}{V \times Wsd}$$

Wpp = Weight percent perchloroethylene in the prepared sample (See 5.3)
Wy-Wx = Total weight of preparation, g (See 4.1.3)
V = Volume of sample used in preparation, mL (See 4.1.3)
Ds = Density of sample, g/mL (See 4.1.1)
Vdis = Volume of the distillate, mL (See 4.1.2.1)
Wsd = Weight of sample distilled, g (See SCAQMD Method 302)
Vo = Volume of the organic layer in the distillate, mL (Sec 4.1.2.2)

SOUTH COAST AIR QUALITY MANAGEMENT DISTRICT

APPLIED SCIENCE & TECHNOLOGY DIVISION

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SCAQMD METHOD 310-91

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DETERMINATION OF PERCHLOROETHYLENE

This method is applicable for samples suspected of containing perchloroethylene. This method is used to show compliance with Regulation XI Rules, (Rule 1102 specifically).

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