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Determination of Perchloroethylene Content of Waste Materials from Filters and Still Bottoms

Conditional Test Method

control technology center

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DETERMINATION OF PERCHLOROETHYLENE CONTENT OF WASTE MATERIALS FROM FILTERS AND STILL BOTTOMS

CONDITIONAL TEST METHOD

by

John D. Jeffrey
Alliance Technologies Corporation
213 Burlington Road
Bedford, Massachusetts 01730

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Project Officers

Robert Judge
Air Management Division, Region I
U.S. Environmental Protection Agency
John F. Kennedy Federal Building
Boston, Massachusetts 02203

and

Anthony P. Wayne
Technical Support Division
Office of Air Quality Planning and Standards
U.S. Environmental Protection Agency
Research Triangle Park, North Carolina 27711

Prepared for

Control Technology Center
U.S. Environmental Protection Agency
Research Triangle Park, North Carolina 27711

PREFACE

The "Determination of Perchloroethylene Content of Waste Materials from Filters and Still Bottoms - Conditional Test Method" was funded as a cooperative project by the Control Technology Center (CTC) and the Air Management Division, EPA Region I. The project was implemented by Region I and the EPA's Emission Measurement Branch, Technical Support Division, Office of Air Quality Planning and Standards (OAQPS).

The CTC was established by EPA's Office of Research and Development (ORD) and OAQPS to provide technical assistance to State and local air pollution control agencies and EPA regional offices. Three levels of assistance can be accessed through the CTC. First, a CTC HOTLINE has been established to provide telephone assistance on matters relating to air pollution control technology. Second, more in-depth engineering assistance can be provided when appropriate. Third, the CTC can provide technical guidance through publication of technical guidance documents, development of personal computer software, and presentation of workshops on control technology matters.

The technical guidance projects, such as this one, focus on topics of national or regional interest that are identified through contact with State and local agencies or regional offices. In this case, the CTC joined EPA Region I to develop this conditional test method as part of Region I's 1990 Rule Effectiveness Study. The Test Support Section, Emission Measurement Branch, OAQPS, was responsible for project oversight. The Emission Measurement Branch supports and is an integral part of the Emission Measurement Technical Information Center (EMTIC). The purpose of this project was to evaluate a test method for determining the perchloroethylene content of wastes from dry cleaning facilities. The result is a conditional test method that is now available for use by State an local agencies in their development of ozone non-attainment State Implementation Plans.

DETERMINATION OF PERCHLOROETHYLENE CONTENT OF WET WASTE MATERIALS FROM FILTERS AND STILL BOTTOMS

1.0 APPLICABILITY AND PRINCIPLE

1.1 Applicability. This method is applicable to the sampling and determination of perchloroethylene in wet waste material from diatomaceous earth filters and solvent stills at perchloroethylene dry-cleaners on a weight percent basis.

1.2 Principle. Samples are obtained from waste material at a perchloroethylene dry-cleaner. A known sample mass is mixed with water and placed in a glass still equipped with a Liebig straight-tube type reflux condenser and a Bidwell-Sterling type graduated trap. Water and perchloroethylene in the sample are seperated through repeated distillation until all of the perchloroethylene has been recovered in the trap and the volume recorded. The mass of perchloroethylene collected is determined from the product of its volume and specific gravity. The total weight of perchloroethylene obtained is divided by the total weight of sample analyzed to obtain the perchloroethylene content of the wet waste residue.

2.0 APPARATUS

2.1 Flask. Round bottom, short-necked flask having a nominal capacity of 500 ml. Figure 1 shows recommended designs of glass connections.

2.2 Condenser. Liebig straight-tube type, with a jacket not less than 400 mm long and with an inner tube having an outside diameter of 10 to 13 mm. Figure 1 shows recommended designs of glass connections.

2.3 Trap. Bidwell-Sterling type, graduated from 0 to 5 ml in 0.1-ml divisions. Calibrate at four or more points by first filling the trap with water and then adding a hydrophobic solvent with a specific gravity greater than water from a standard buret having a calibrated capacity at least equal to that of the trap. The error of the indicated volume shall not exceed 0.05 ml. Figure 2 shows characteristic details of a suitable trap.

2.4 **Heater.** Any suitable gas burner or electric heater for the glass flask.

2.5 **Sample container.** Metal can with a leak proof closure, 150 ml.

3.0 PROCEDURE

3.1 Sampling.

3.1.1 **From distiller (cooker).** After a cycle of the perchloroethylene distilling and the still bottoms have come to approximately room temperature (i.e., 21 to 38°C), obtain three 150 ml samples of the wet waste residue from the distiller (cooker) drain. Completely fill each sample container (three total) to avoid evaporation loss. Immediately close the sample container lid securely.

3.1.2 **From wet waste containers.**

3.1.2.1 **Large unmixed containers.** Using a clean sampling spoon, spatula, or other appropriate device, obtain three 150 ml samples. Each sample shall be comprised of an approximately 50 ml sub-sample from the top, bottom, and mid-point locations of the container. Transfer the three sub-samples to each sample container. Completely fill the sample container to avoid evaporation loss. Immediately close the sample container lid securely .

3.1.2.2 **Small containers.** If the waste container can be thoroughly mixed prior to sampling, mix the container contents thoroughly and obtain the sample by pipetting. The pipette should have at least a 150 ml capacity and should be long enough to reach within 2 cm of the bottom of the container. Obtain three 150 ml samples. Transfer the sample to each sample container. Completely fill the sample container to avoid evaporation loss. Immediately close the sample container lid securely.

3.1.3 **Label the containers.** Use waterproof and oil-proof ink for labeling.

3.1.4 **Storage.** Store the sample in a cool dry atmosphere.

3.1.5 **Shipment.** Transfer the sample to the appropriate laboratory for analysis within 48 hours of obtaining the sample. The sample shall remain sealed until the time of analysis.

3.2 **Analysis.** Conduct duplicate analyses of each sample container and record the recovered perchloroethylene from each analysis.

3.2.1 For each analysis, weigh and record the weight of an empty flask and stopper, W_i, to the nearest 0.1 mg. Mix each unopened sample container by shaking. Open the sample container and immediately transfer

approximately 20 ml of wet waste material to the flask. Stopper the flask and reseal the sample container. Weigh and record the weight of the flask plus added portion, d_i, to the nearest 0.1 g. The mass added to the flask shall not exceed 35 g. Add water to the flask to make a total mixture volume of approximately 250 ml. Fill the trap with cold water. Connect the flask to the distillation trap.

3.2.2 Assemble the apparatus as shown in Figure 1 or 2 so that the tip of the condenser is directly over the indentation in the trap.

3.2.3 Heat the flask so that refluxing starts within 7 to 10 minutes. Adjust the rate of boiling so that the condensed distillate is discharged from the condenser at a rate of 1 to 3 drops per second.

3.2.4 From the time refluxing starts, obtain readings of the amount of perchloroethylene collected after 5, 15, and 30 minutes, and each following 15 minutes. End the test when the volume of perchloroethylene is increased by not more than 0.1 ml in a 15 minute period or the amount of perchloroethylene exceeds the trap capacity.

3.2.6 At the end of the test run turn off the heater. Allow the equipment to stand at least 30 minutes to allow the distillate to settle clear and to cool to room temperature. Read the volume of perchloroethylene collected in the trap. If the amount of perchloroethylene exceeded the calibrated capacity of the trap, report the volume of perchloroethylene as 5.0 ml plus.

4.0 CALCULATIONS

4.1 Nomenclature.

D = Density of perchloroethylene at 20°C, 1.6227 g/ml.
f_i = Weight of perchloroethylene in the portion, g.
s_i = Weight of the wet waste portion, g.
V_i = Volume of perchloroethylene collected in the trap, ml.
w_i = Weight of the empty flask and stopper, g.
d_i = Weight of the flask plus portion, g.

4.1 Calculate the total mass of the portion in the flask:

$$s_i = w_i - d_i \qquad \text{Eq. 1}$$

4.2 Calculate the total mass of perchloroethylene, f_i, collected in the trap from each analysis:

$$f_i = V_i \times D \qquad \text{Eq. 2}$$

4.3 The perchloroethylene content of the wet waste (R), expressed in kg per 100 kg wet waste material, is equal to the total weight of the perchloroethylene obtained from the portions divided by the total weight of the analyzed portions, multiplied by 100.

$$R = 100 \times \frac{\sum_{i=1}^{n} f_i}{\sum_{i=1}^{n} s_i} \qquad \text{Eq. 3}$$

where: n = the total number of analyses.

5.0 PRECISION AND ACCURACY

5.1 **Accuracy.** Concentrations of audit samples obtained by the analyst shall agree within 10 percent of the actual concentrations. If the 10-percent specification is not met, reanalyze the compliance samples and audit samples, and include initial and reanalysis values in the test report.

5.2 **Precision.** Duplicate results produced by the same analyst should be considered suspect if they differ by more than 5 percent.

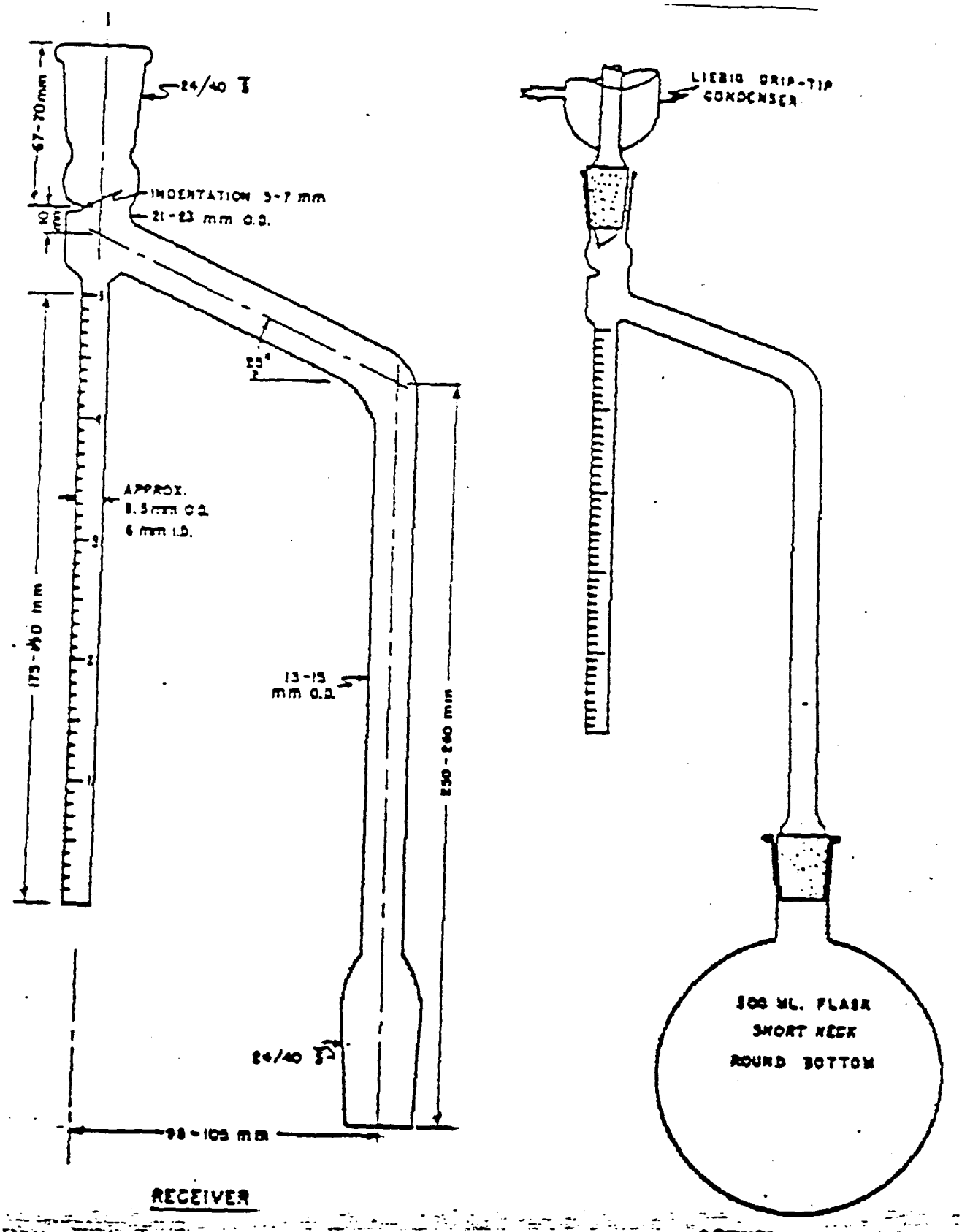

Fig. 2 Apparatus for Determining Perchloroethylene Content of Wet Waste Materials

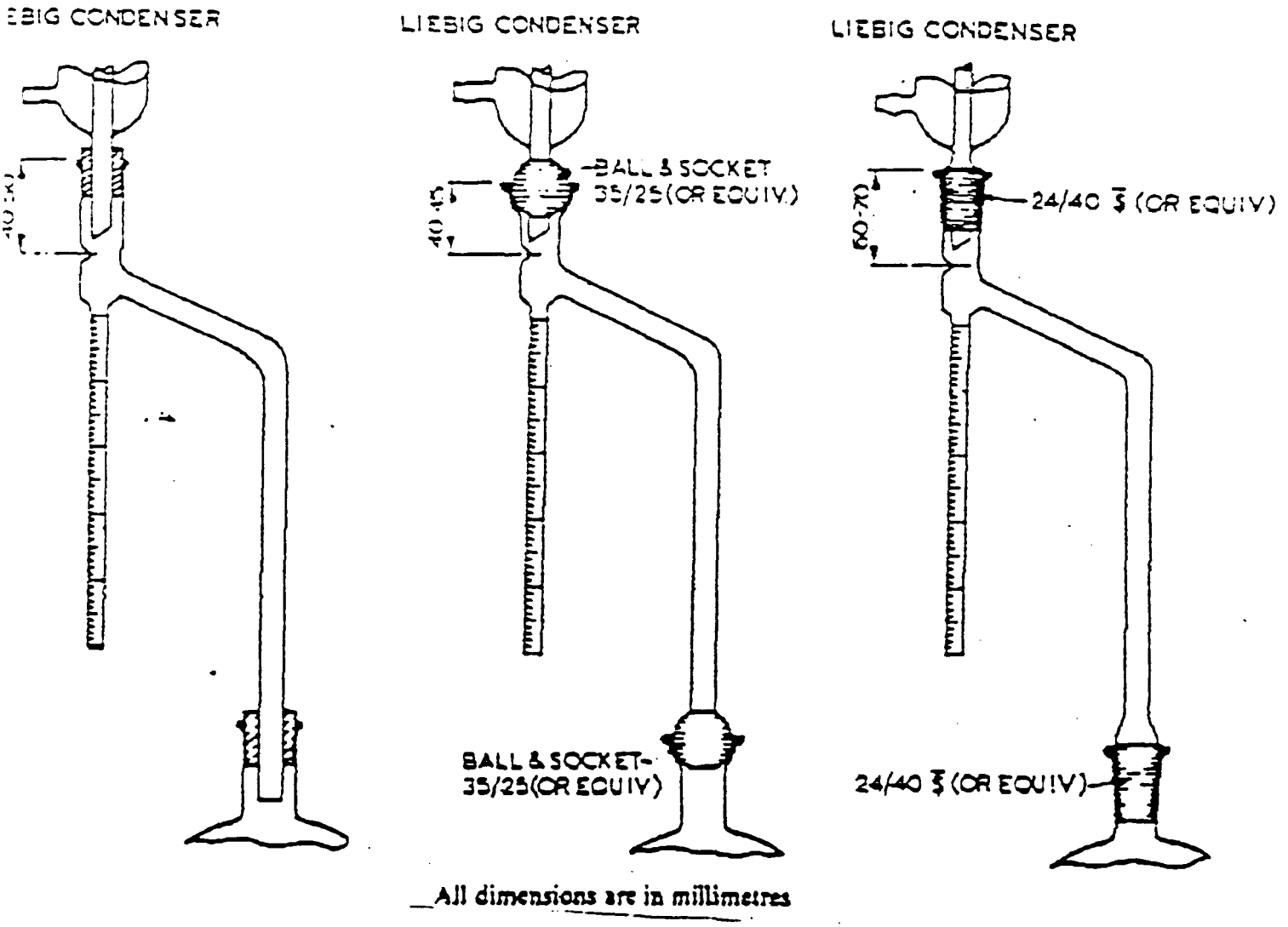

Fig. 1 Alternate Closures for Trap