ASSOCIATION CONNECTING
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Number	
2.3.41	
Subject **Test Method for Total Halogen Content in Base Materials**	
Date **04/06**	Revision
Originating Task Group **Halogen Free Materials Subcommittee (4-33)**	

IPC-TM-650
TEST METHODS MANUAL

1 Scope The purpose of this test method is to measure the amount of chlorine and bromine compounds in base materials. This test method is applicable to reinforced base materials with a minimum thickness of 0.3 mm [0.012 in] and to un-reinforced base materials with a minimum thickness of 0.08 mm [0.0031 in].

A combustion flask is used to extract ionic and covalent halogen from the sample, and Ion Exchange Chromatography is used for the quantitative analysis of halogen content.

2 Applicable Documents All terms and definitions in this document conform to IPC-T-50, *Terms and Definitions for Interconnecting and Packaging Electronic Circuitry.*

The IPC Test Method is generated and written, verbatim, from the **IEC 61189-2 TEST 2C12: Total Halogen Content in Base Materials.**

3 Test Specimens Rigid or flexible base materials shall be used for the test provided they meet the minimum thickness requirements described in the scope. The copper foil (if applicable) shall be removed from the test specimens by etching by any industry acceptable etching method or by mechanical peeling before test.

For reinforced base materials, the number of test specimens shall not be less than five (5) with a minimum size of 1 cm x 1 cm [0.39 in x 0.39 in]. For unreinforced base materials, the number of test specimens shall not be less than five (5) with a minimum size of 1 cm x 1 cm [0.39 in x 0.39 in].

Wash the specimens thoroughly in distilled or deionized water.

For reinforced base materials, dry the sample at 105 °C [221 °F] +5/- 0 °C [+9 °F/-0 °F] for one hour + 0.25 hour /-0 hour.

For unreinforced base materials, wipe off the water with a lint-free cloth or paper wiper and leave them to dry at room temperature and atmospheric pressure for a minimum of one hour.

4 Apparatus & Materials

a. Ion exchange chromatograph with a detection limit of 10 ppm or better.
b. Analytical balance with an accuracy of approximately 1 mg in weight or better.
c. Knife.
d. Tweezers.
e. Vinyl gloves.
f. Lint-free cloth, paper wipers or equivalent.
g. Quantitative filter paper.
h. Combustion flask, or equivalent.
i. Oxygen (99.9 % purity or better).
j. Gas pressure regulator.
k. Flint striker, or another ignition device.
l. Wash bottle.
m. Beaker.
n. Micro-pipette.
o. Flasks (various sizes).
p. Potentiometric titrator for silver nitrate, if necessary.
q. Platinum basket for holding sample.
r. Alkali solution.
s. Ethyl alcohol.
t. Chlorine ion standard solution for chromatograph.
u. Bromine ion standard solution for chromatograph.
v. Silver nitrate titrant, if potentiometric titration using silver nitrate is used.

5 Procedure

5.1 Combustion Procedure Weigh the specimen using the analytical balance and record the result.

Place approximately 50 ml alkali solution in a combustion flask to act as an absorbent of combustion gas.

Fill the combustion flask with oxygen.

Insert a test specimen into the combustion flask as shown in Figure 1.

Insert a piece of filter paper into the combustion flask as shown in Figure 1 to act as a fuse/flame starter.

Apply several drops of ethyl alcohol on the test specimen for improving the ignition of the specimen and then ignite the specimen.

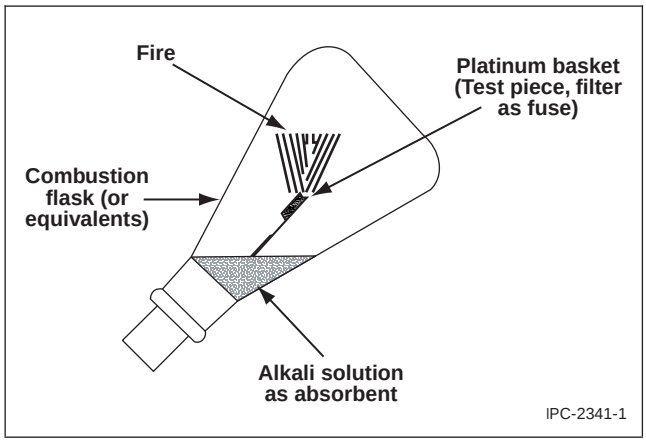

Figure 1 Absorb Combustion Gas Using a Combustion Flask

Leave the flask at room temperature for 30 min ± 2 min after combustion. The generated gas is absorbed into the alkali solution to produce Cl^-/Br^- ion solution.

Transfer the solution from the combustion flask to a volumetric flask. Introduce deionized water in the flask until the total amount is 100 ml of the test solution.

Remove the suspended subjects from the test solution by filtration or centrifugation if necessary.

Follow the same procedure outlined above without the test specimen to prepare a reference test solution (blank) without combustion of a test sample.

Note: Chlorine and bromine contents shall be measured beforehand without any test specimen in the combustion flask.

5.2 Chemical Analysis Inject the test fluid gathered from the flask in 5.1 into the inlet of an ion exchange chromatograph shown in Figure 2.

Analyze the peak area/height of the Cl^-/Br^- conductivity on the recorder and obtain Cl^-/Br^- concentration from a calibration curve.

Obtain Cl^-/Br^- ion concentrations of the reference by comparing its conductivity using the standard solution. The test solution is compared to the reference solution (blank). Any contaminant in the reference solution will be subtracted from the test solution.

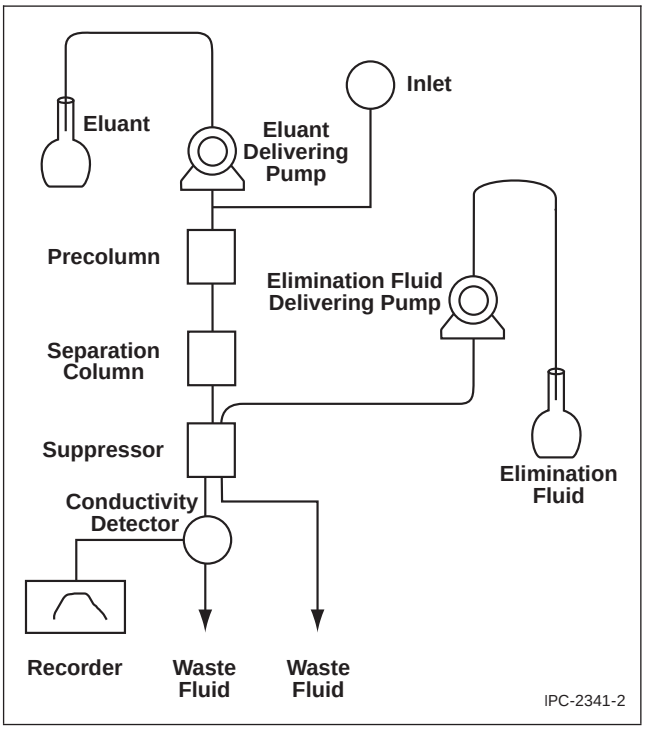

Figure 2 Composition of Ion Exchange Chromatograph

Titrate test solution using silver nitrate if concentration of Cl^-/Br^- ions is higher than 1 wt%. An example of analyzing conditions for the ion exchange chromatography is given in Table 1.

Table 1 Example of Analyzing Conditions for the Ion Exchange Chromatography

Item/Material	Definition/Quantity
Eluant	Alkali solution
Eluant delivering rate	1.0 ml/min to 2.0 ml/min
Elimination fluid	H_2SO_4
Elimination fluid and delivering rate	1.0 ml/min to 2.0 ml/min
Column	Precolumn, separation column
Suppressor	Suppression for anion
Detector	Conductivity meter

Analyzing conditions depends on the test devices, test specimens, their composition and environment.

5.3 Calculation of Halogen Content Insert the concentration of halogen ion (Cl^-/Br^-) obtained in 5.2 in the following formula to obtain halogen contents in the specimens:

Chlorine (wt%) =
{[Cl^- concentration in the test fluid (ppm) x
Cl^- Dilution ratio in the fluid] -
[Cl^- concentration in the reference (ppm) x
Cl^- Dilution ratio in the reference]} x
{quantity of test solution (ml) /
mass of the test specimen} x 10^{-7}

Note: For the bromine content, use the same equation but insert Br^- values in the place of Cl^-.

Note: The dilution ratio is the amount of added water as compared to the total amount of solution in the test flask. If the amounts used were 50 ml each, the dilution ratio would be 2.

6 Report In addition to the general requirements for reporting, the report shall include:

a. Test method number and revision.
b. Date of the test.
c. Identification and description of the specimen.
d. Average chloride content of the five (5) specimens in ppm.
e. Average bromide content of the five (5) specimens in ppm.
f. Average total halogen content of the five (5) specimens in ppm.
g. Any deviation from this test method.
h. Date of the test.
i. Name of the person conducting the test.