

DETERMINATION OF SPECIATED PERMEATION RATES FOR POLYMERS

Application

This method is used to determine the permeation rates of fuel components through a polymer film using the trap and desorption method. Using a gas chromatograph the collected vapors are speciated to provide permeation rates in grams/m² day for each component.

Principle

The small scale solvent permeability test determines the permeation rate of a solvent mixture through a test film (e.g. polymer, elastomer or complex layers). The film is sandwiched between two chambers of a sealed small scale test cell. The test solvent/solution (in liquid or gas phase) is in contact with one side of the test film, and high purity nitrogen purge gas is in contact with the opposite side of the film.

The volatile organic compounds (VOC) permeating through the test film from the solvent side to the nitrogen purge side are trapped onto Trap and Desorption (T&D) tubes. The composition of the speciated permeated vapor matrix is quantitatively measured by subsequent desorption and analysis of the trapped VOC from the T&D tubes using a two-stage Automated Thermal Desorption Unit attached to a Gas Chromatograph with a flame ionization detector (GC/FID).

Equipment Required

Film test apparatus:

1. **Test cells,**
2. **Ovens,** (air circulating, mechanical convection, e.g. FREAS 625 or equivalent),
3. **T&D sampling tubes,** containing appropriate trapping material,
4. **Gas flow controllers,** (e.g. MKS 1459C or equivalent), one per test cell,
5. **Gas flowmeter,** (e.g. J&W ADM 2000 or equivalent),

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6. **Stopwatch**,
7. **Balance**, (0.1g accuracy; to 1000g),
8. **Precision thickness gage**, (e.g. Logitech CG-10 or equivalent),

T&D Analytical Apparatus:

9. **Desorber**, Two-stage automated thermal (e.g. Perkin Elmer ATD 400, or equivalent),
10. **Gas Chromatograph**, with Flame Ionization Detector (e.g. Perkin Elmer AutoSystem GC/FID or equivalent) with appropriate analytical gas chromatographic separation column, GC controlling, acquisition and analysis software, and computer file management and storage,
11. **T&D sampling tubes**, $\frac{1}{4}$ " glass containing appropriate trapping material for the VOC.

Reagents Required

1. Purge Gas, (high quality Nitrogen),
2. Test solvents (should be at least LC quality or better): isooctane (ISO), toluene (TOL), ethanol (ET); or mixtures of these, or other liquid solvents. The solvents should be stored in a flammable solvent cabinet.
3. Certified VOC mixture in vapor gaseous Nitrogen matrix: isooctane (ISO), toluene (TOL), ethanol (ET), or mixtures of these, or others (Scott Certified Gases or equivalent).

Procedure

Test Apparatus:

The test cells can be operated with either vapor or liquid contact with the film. The cell should be vented through a 1.8m long, 1/16" wide metal tubing which drops pressure to atmospheric, but minimizes diffusive loss of solvent vapors. The typical test is conducted with the solvent mixture in vapor contact with the film. The flow of the purging gas is controlled by a gas flow controller. The typical purge gas used is high purity Nitrogen at about 20 cc/min flow. The cells are contained in an air circulating oven with uncertainty better than $\Delta T = \pm 0.5^{\circ}\text{C}$. The test can be run in an isothermal mode, or with a temperature ramp. The typical test temperature is 40°C isothermal.