

Carbon number at 5% distillation point

ASTM D 6352 – 04

Adapted, with permission, from the Annual Book of ASTM Standards, copyright American Society for Testing and Materials, 100 Harbor Drive, West Conshohocken, PA 19428.

Copies of the complete ASTM standard may be purchased directly from ASTM, phone: +1 610-832-9585, fax: +1 610-832-9555, e-mail: service@astm.org, <http://www.astm.org>

"Carbon number" is number of carbon atoms in a molecule. Determine the boiling point distribution of the sample by gas chromatography. Individual hydrocarbons are separated on a non-polar open tubular capillary column using a linear temperature program in the order of their increasing boiling points. Detector response for each paraffin shall be close to unity.

Gas chromatography

Column: Non-polar wall-coated open tubular column (5 m x 0.53 - 0.75 mm, i.d.) stationary phase, 100% dimethylpolysiloxane, 0.1 μ m, or equivalent
Carrier gas: Helium, at a flow rate of 18 ml/min
Detector: FID; temperature 450°
Oven program: 50° - 10°/min - 400°
Injector: On-column or temperature programmable vaporizing injector.
Injection volume: 0.5 μ l
Calibration mixture: Prepare a mixture of hydrocarbons with known boiling points covering the range of the sample (e.g. from C10 to C90). Each component should be present at approximately 0.5 - 2.0%, dissolved in a suitable viscosity-reducing solvent such as carbon disulfide or cyclohexane.

Procedure:

Column resolution: Resolution is determined using C50 and C52 paraffins from a calibration mixture. Resolution shall be between 2 and 4 for satisfactory column performance.

Sample analysis: Using the schedule and temperature program used for the calibration mixture, cool the column and injector to the initial starting temperature. Inject the sample, diluted to approximately 1% in Carbon disulfide or hexane, and record the chromatogram. Inject a baseline blank, standard mixtures and samples in a predetermined order. Use the baseline blank to determine baseline drift and perform baseline subtraction from runs of samples and standards.

Calculation: Collect data, calculate the sample total area, normalise to area percent after background subtraction. Determine the initial and final boiling points by calculating 0.5% and 99.5% of the area counts, respectively. Use linear interpolation to determine the retention time associated with 5% and read the corresponding boiling temperature from the calibration curve.