RESTEK Pure Chromatography

ASTM D3606 Capillary Options: Products and tips for getting started

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Abstract

ASTM method D3606 is used to separate benzene and toluene in spark ignition fuels. Analysts may use capillary (option A) or packed (option B) columns for this method. The capillary option uses two columns connected in series, where the first column is nonpolar dimethylpolysiloxane phase (Rxi-1ms, cat# 13338) and the second is a polar 'wax' phase (Stabilwax, cat# 10657) column. After toluene is eluted from the first column, the first column is backflushed while an auxillary EPC maintains flow through the second column. The complicated setup associated with the ASTM D3606 capillary option may make the method unappealing, but the results show excellent separation of target analytes. We used Restek's Rxi-1ms and Stabilwax to separate target analytes in D3606 standard 8.5.2 and a sample of gasoline, and generated helpful tips for getting started and troubleshooting this method. Future work may consider performance of this column set for a full method validation, and potential limitations.

Featured products

Rxi-1ms
30m x 0.25mm x 0.5um
(cat# 13338)

Stabilwax
60m x 0.32mm x 1um
(cat# 10657)

Restek Electronic Leak Detector
(cat# 28500)

Restek ProFLOW 6000 Electronic Flowmeter
(cat# 22656)

Tips for setting up

Controlling Leaks: More connection points means more leaks. Use a leak detector to pin-point leaks!

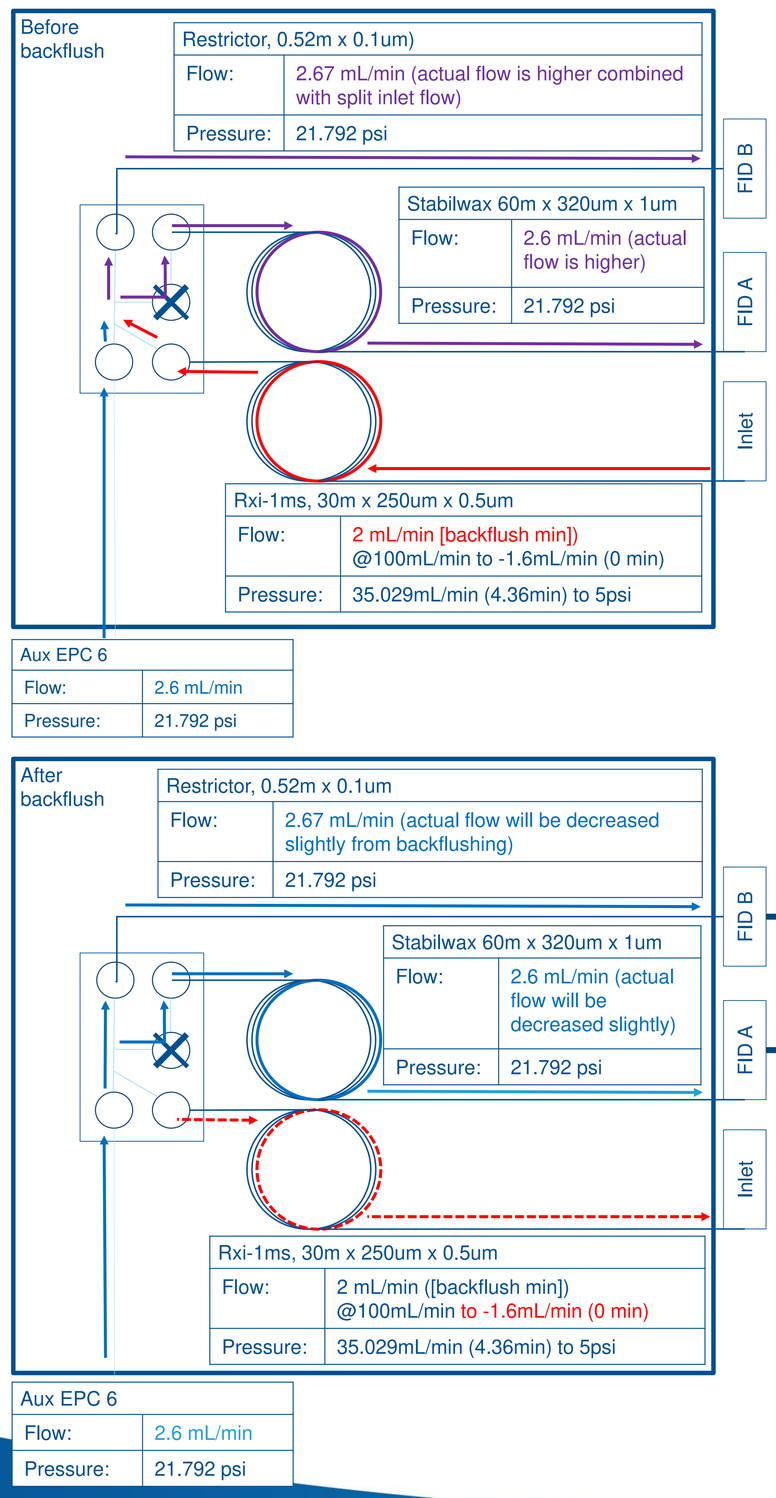
Is my system backflushing?: Use a flowmeter to check that your system is backflushing. Reduce your split to 2:1 and measure flow from the split vent at your backflush time. Flow should jump by your backflush rate.

ex: 2 mL/min split flow + 1.6 mL/min backflush =
3.6 mL/min out the split vent at the backflush time

Monitoring toluene: Run a blank between samples to make sure you are backflushing *completely.* If pressure settings are off, toluene may be caught in the restrictor or pre-column.

Splitting you flow evenly with a restrictor (optional): The column length listed in the method is a good starting point, but length will have to change depending on your exact column dimensions. Check if your auxiliary EPC comes with a calculator to help.

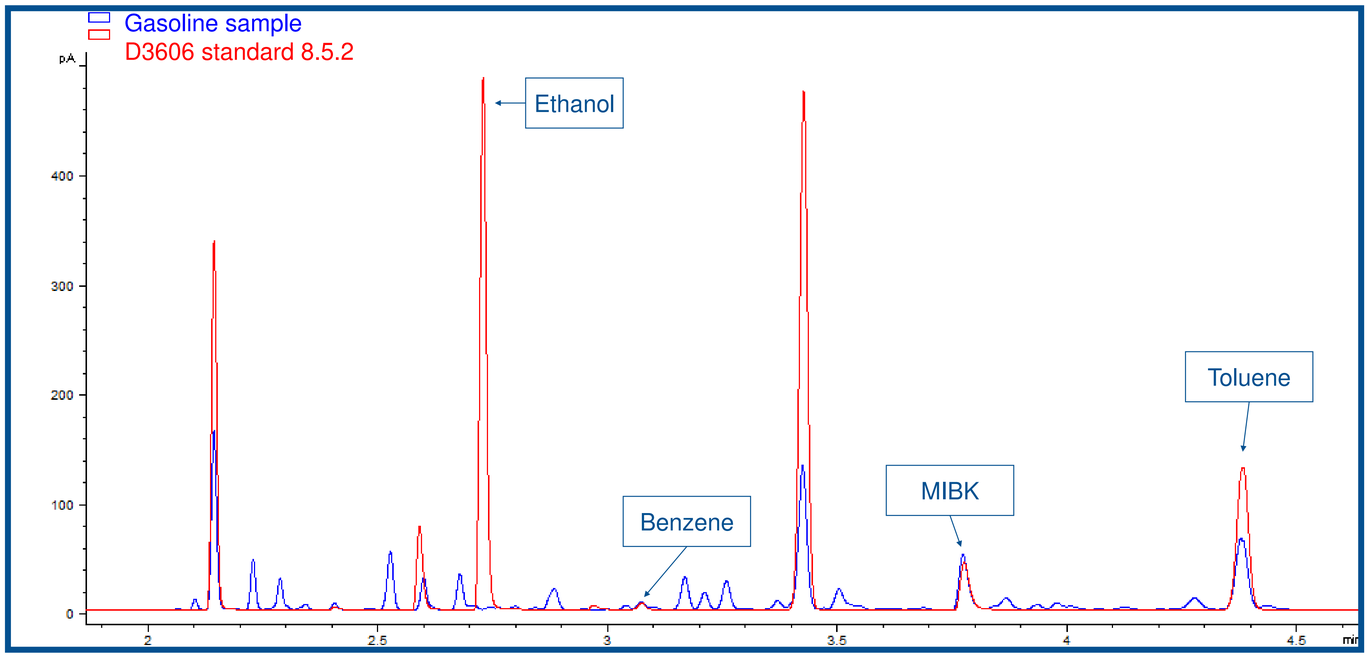
Method Setup



Separation of benzene and toluene using the Rxi-1ms and Stabilwax

Restrictor (Rxi-1ms)

- The restrictor monitors flow from the Rx-1ms
- Nonpolar dimethyl-polysiloxane phase recommended
- Retains larger hydrocarbons
- Polar volatiles elute early
- Small hydrocarbons clutter the baseline



Stabilwax

- Analytical column is a polar Stabilwax
- 'Wax' phase column recommended
- Nonpolar hydrocarbons elute early
- Polar volatiles are retained
- Target compounds are well separated
- Backflushing keeps the method <20 min