TRAJAN

TECHNICAL ARTICLE

Syringe tips for improving manual GC injection reproducibility

Improving precision of manual injections

For many years, gas chromatographers have considered the normal variability of manual injections to be in the range of 5% RSD for an individual, with 10% RSD common between individuals. It is entirely possible however to reduce this variability to less than 1% RSD for one person and easily to less than 2% RSD for two or more individuals. The "secret" is in reducing the individual sources of variability by a systematic injection technique.

The difficulty of setting the plunger repeatedly to the same volume is the greatest source of error. Plunger throw (the distance the plunger moves during the injection) is often short, hence a small change in plunger position represents a large volume difference. Parallax (viewing the plunger other than 90º from the bore axis) is also a large source of error. Ideally use a repeating adaptor to overcome all parallax reading error. If your technique does not allow for a repeating adaptor, choose a syringe with graduation lines that extend to the rear side of the syringe to enhance readability.

Use a straight wall, well deactivated liner, with quartz wool packing positioned to wipe the needle (ie. FocusLiner®) where possible. This liner configuration give both good vaporization and precision on most gas chromatographs. Cup liners usually give poorer reproducibility than straight liners. Also a hint, don't overtighten the septum cap and make sure the system is leak free.

Systematic procedure for split injection using hot needle - rapid injection method without a repeating adaptor

1. Immerse the needle tip into the solution to be sampled.
2. Withdraw the plunger to aspire one syringe volume of liquid.
3. Remove the needle from the solution.
4. Expel the contents of the syringe into a waste vessel.
5. Repeat steps 1- 4.
6. Immerse the needle into the liquid again. Take up and expel the sample at least 5 times, being careful to keep the needle tip submerged.
7. Fill the syringe past the intended delivery volume mark by at least 4 mm.
8. Using a magnifying lube, carefully position the plunger to the intended volume mark. Decide where you intend to stop the plunger tip - whether at the front or back edge (or middle) of the calibration etching on the syringe barrel. For all measurements, use the position you decide on to set the plunger.
9. Check the liquid volume for and microbubbles. If you find any, repeat Steps 1-8.
10. Draw the plunger back to draw at least 1µL of air into the syringe and to empty the needle
11. Hold the syringe with the calibration marks facing you. Place your forearms on the front corners of the GC top to maintain the syringe orientation. This puts the needle bevel in the same position injection after injection and reduces coring the septum.
12. Insert the needle through the septum to its full needle depth.
13. Hold the syringe vertical and allow the needle to heat up for about 5 seconds. Keep this time as constant as possible once you determine how long you want to use. This allows the needle to be at a constant temperature for each injection and minimizes sample discrimination.
14. Make the injection rapidly and smoothly by depressing the plunger.
15. Leave the needle in place for at least 15-20 seconds. As long as you leave the needle in place at least 15 seconds, precision is enhanced.
16. Withdraw the syringe cleanly.

These steps should give precision on the order <1% RSD on a repeated basis.

Care should be taken using the hot needle injection technique with compounds that are thermally labile in the presence of a metal needle (eg. organochlorine pesticides). Compounds like these may decompose during the injection process.

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Information and support

Visit www.trajanscimed.com or contact techsupport@trajanscimed.com

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