Gas Chromatographic Injectors

Introduction

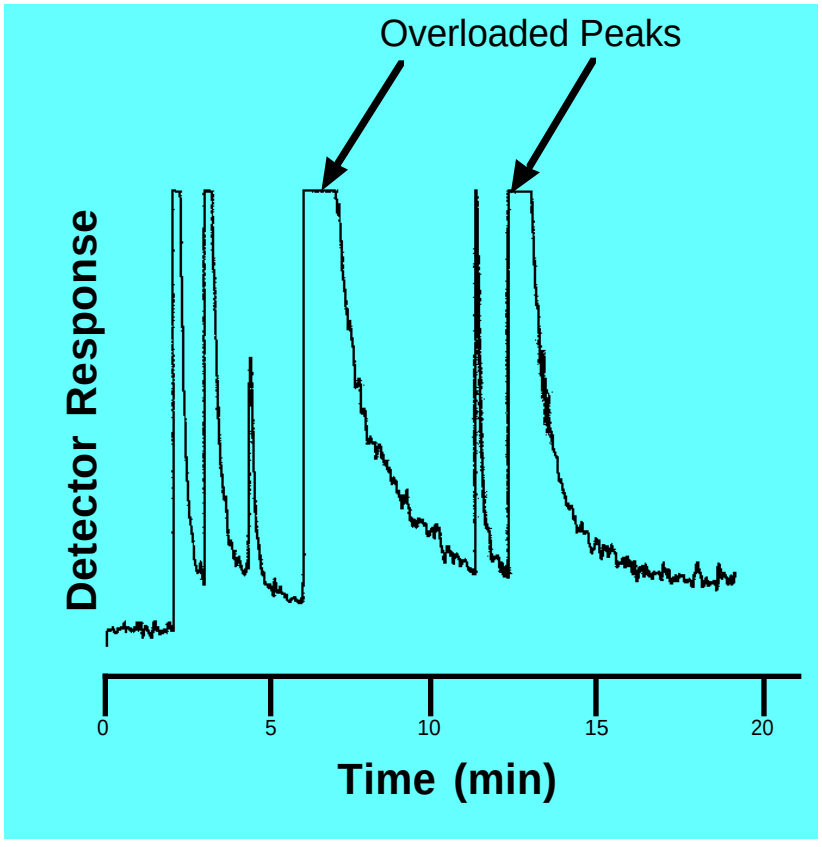
The great analytical strength of capillary gas chromatography lies in it high resolution. Capillary columns have 1) more theoretical plates (a measure of column resolving power or efficiency) per meter as compared to packed columns and 2) since they have less resistance to flow they can be longer than packed columns. This means that the average capillary column (30 meters long) has approximately 100,000 theoretical plates while the average packed column (3 meters) has only 2500 plates.

But with this separation power comes some limitations: 1) Capillary columns, because they have smaller diameters (0.05 to 0.53 mm) than packed columns (2 to 4 mm), require relatively specialized injectors and ancillary flow and pressure controllers and 2) capillary column require a smaller amount of sample than packed columns. While the average sample mass of each component in a mixture that is separable by packed column GC can be in the microgram range (10^{-6} grams) per injection, capillary columns routinely only handle 50 nanograms (10^{-9} grams) of a particular component or less.

Overloaded Chromatography

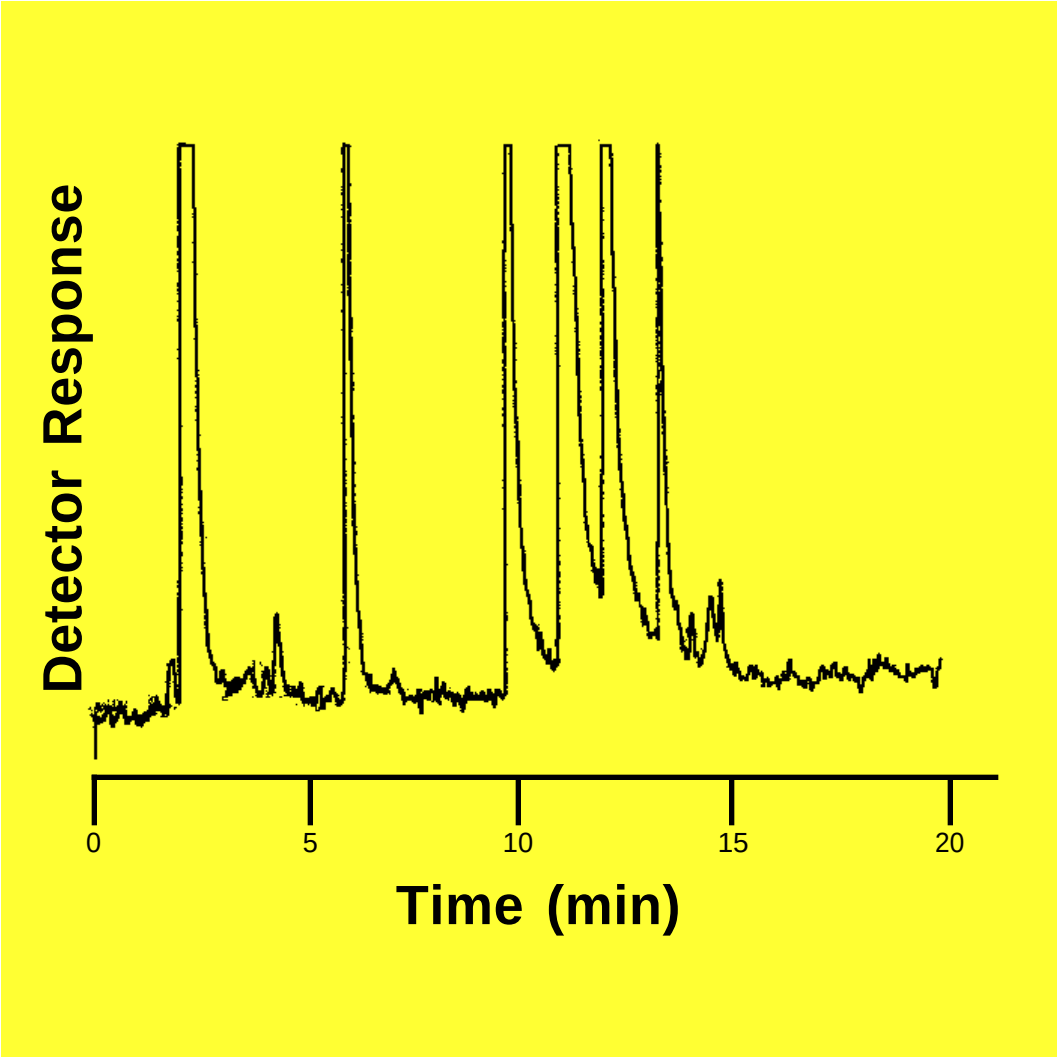
This sample size requirement initially meant that if samples contained components that were too concentrated for a capillary chromatographic analysis, the sample had to be diluted before it was analyzed. Otherwise the column would be **overloaded** by those high concentrated components. An example of this appears in the first figure below. The clearly overload peaks are indicated. And while some of the other components are in the resolvable (not overloaded) range, having large masses of components can also distort the peak shape of some of the lower mass components.

An Overloaded Chromatogram



The following figure shows a little better chromatography with fewer overloaded peaks. The second eluting peak (about 6 minutes) is clearly not overloaded while the group between 10 and 14 minutes still shows overloading characteristics: long drawn-out tailing and much less than baseline separation with peaks that elute nearby (the 11 and 12 minute peaks, for instance).

A "not so" Overloaded Chromatogram

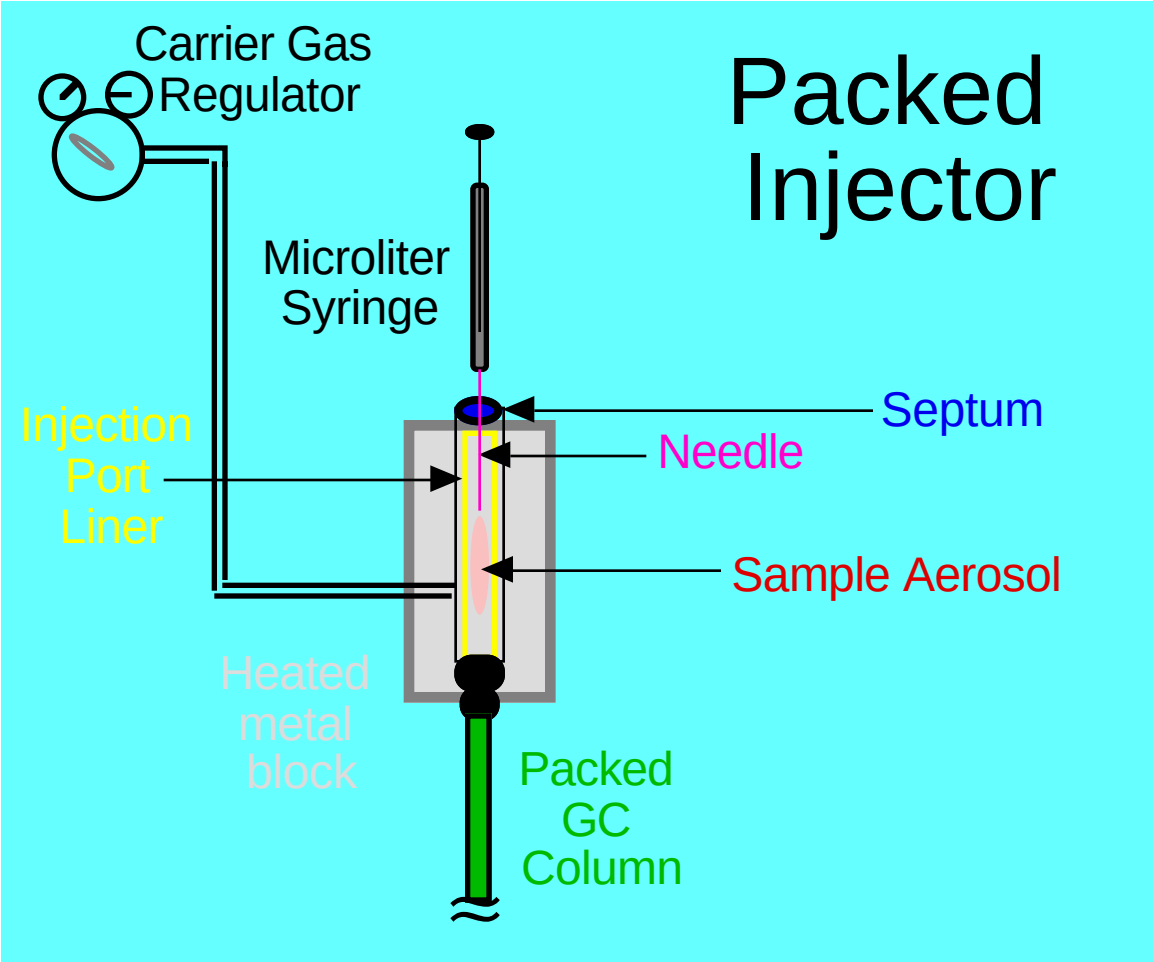


Normal Packed Column Injector

The normal sequence of events in a GC injection is as follows. We will assume in this explanation that some analytes are dissolved in a (liquid) solvent although much of this process also holds for gas GC injections too: A small amount of liquid (microliters) is injected through a silicon rubber septum (using a special microliter syringe) into the hot (usually 200 ^{o}C) GC injector that is lined with an inert glass tube. The injector is kept hot by a relatively large, metal heater block that is thermostatically controlled. The sample is immediately vaporized and a pressurized, inert, carrier gas-which is continually flowing from a gas regulator through the injector and into the GC column-sweeps the gaseous sample, solvent, analyte and all, onto the column. In the packed column injector, ALL the vaporized sample enters onto the column. This is how all packed column injectors work; everything that is injected goes onto the column. One modification of this is a small ancillary flow of carrier gas that bathes the underside of the injector's septum so that hot vaporized sample gases can't interact and possibly stick to the septum. This improves peak shape and reproducibility. This last feature is called the septum

purge. The following figure is a schematic of a packed column injector, sometimes called a direct or flash injector. The septum purge is not shown here although the carrier gas regulator and inlet, a septum, and an injection port liner ARE detailed. Last by not least, the packed GC column itself is connected at the bottom of the injector via metal fittings.

Schematic of packed GC column injector



One last subtle point about the configuration of the carrier gas inlet: notice that it enters the injector at about the middle of the heating block and its gas has to travel along the outside of the injector before it enters the injection port liner AT THE TOP. This is so the carrier is preheated before it enter the liner where the sample is vaporized. This helps to prevent a cold spot at the top of the injector where the carrier gas enters.

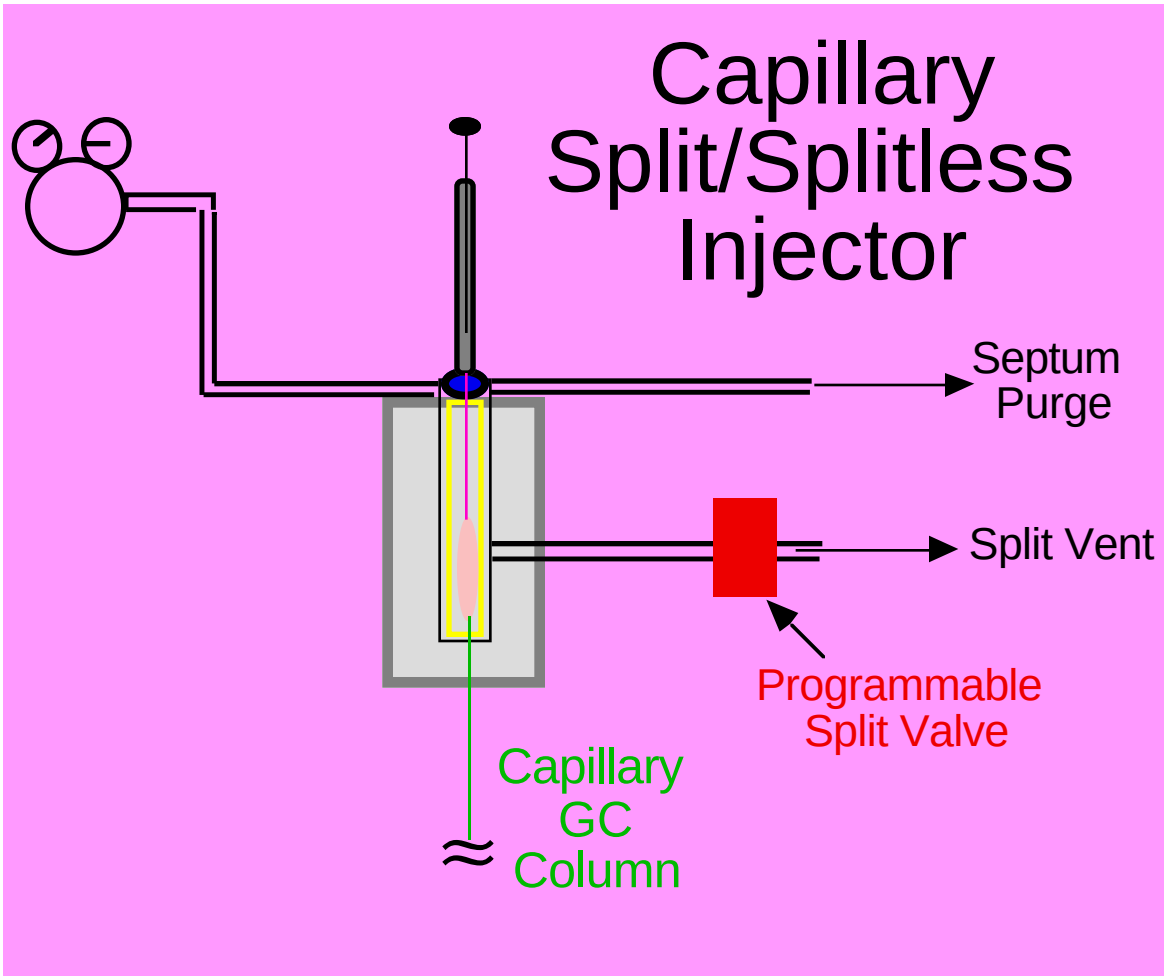
Now remember that the size of the capillary column limits the amount of analyte that can be injected, otherwise, chromatographic overloading occurs. Therefore this packed column injector design, if used with a capillary column, would require that samples with high concentrations of analytes be diluted. Unless... what other alternative is there to get the amount of analytes that are injected onto the column smaller without having to dilute concentrated samples? The solution is the split/splitless capillary GC injector.

The Split/Splitless Capillary Injector

OK so far so good. But how does the split/splitless injector work? It starts with the same requirements as the packed column injectors: carrier gas inlet, a septum, septum purge, injector insert, heater block, and column connection; but the heart of this technological feat is another set of gas lines **out** of the injector-another path that the vaporized sample can take. This is called the

split line or vent. The manufacturers of these systems design them so that the carrier gas flow onto the column is constant-to maintain the chromatographic requirements of the column and yield reproducible retention times for analytes. At the same time, the amount of gas that goes out the split vent controls the amount of sample that enters the column. If the split vent is closed, via a computer controlled split valve, then all of the sample introduced into the injector goes on the column. If the split vent is open then most of the vaporized sample is thrown away to waste via the split vent and only a small portion of the sample is introduced to the column. The following diagram illustrates a split/splitless injector with the split vent **on** so that only a small portion of the sample injected goes on the column.

Split/splitless GC injector



And finally, a very neat aspect of this is that the amount of gas exiting the split vent can be varied while keeping the flow onto the column constant. This means that the AMOUNT of the split (called the split ratio) can be varied. A common split ratio is 50 to 1. That is, for every 50 units of gaseous sample that are thrown away to waste, 1 unit goes on the column. The analyst keeps careful control of the split ratio so that results from the chromatography can still be quantified. Chromatographic peaks that show up as, say 2.5 ng of compound X really represent 2.5 x 50 = 125 ng of analyte X in the original sample. Also notice that this mass (125 nanograms) would have overloaded the column if all of it ended up on the capillary column. Voila! A split injection, and no sample dilution required.

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