

25400-700

## A SYRINGELESS INJECTION DEVICE FOR THE INTRODUCTION OF SOLIDS AND LIQUIDS INTO A SPLIT/SPLITLESS CAPILLARY INJECTION PORT

Gary Lavigne, University of Connecticut, Institute of Materials Science 97 North Eagleville Road, Storrs Mansfield, CT 06268-2536, Myer Ezrin

A syringeless injection device has been developed for Gas Chromatographs to expand the capabilities of Split/Splitless Capillary Injection Ports to include solid sampling. By utilizing the carrier flow and temperature controls of a Gas Chromatograph, a disposable glass sample vial can be moved into and out of the injection port, for controlled heating and transfer of volatile materials directly into a capillary column.

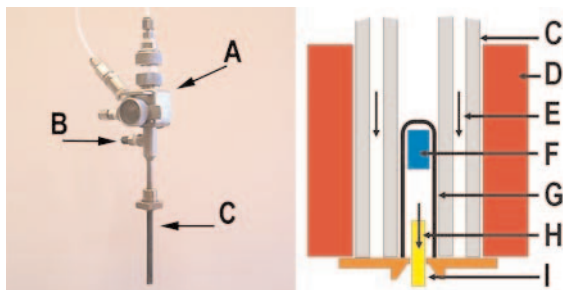
Once a sample has been placed within a sample vial, the vial is then inserted into the syringeless injector by rotation of the load / run valve. In the load position sample can be inserted or removed from the device without depressurizing the injection port. In the run position the sample vial is allowed to move into or out of the injection port. The movement of the sample vial is controlled by the injection ports Split/Splitless control valve. Operating the injection port in the Split mode causes the sample vial to be pushed up and out of the injection port by a high flow rate of carrier gas. By switching to Splitless operation the carrier gas supporting the vial is removed and the vial falls into the injection port. During the injection process the sample vial surrounds the inlet of the capillary column allowing the volatile gases to be swept into the column for collection and separation. This unique arrangement allows for dynamic thermal desorption of solids and liquids directly within the injection port at temperatures from ambient to 400C without the need for transfer lines, external heaters, pressure / flow controllers or solvents.

The syringeless injection technique can be utilized in a number of unique ways. The sample vial will accept 100 microliters of a liquid, which can then be evaporated to dryness leaving a residue of less volatile organics on the inner walls of the sample vial. This can be repeated to provide adequate sample. The vial can then be analyzed directly for semivolatile materials. The sample vial can also be filled with a solid absorbent trapping material to collect volatiles, in either static or dynamic mode, for subsequent thermal extraction directly within the injection port. Microscopic particulates or liquid contaminants in the microgram to nanogram range can be analyzed directly without prior solvent dilution. The result is greatly enhanced detectability of volatile and semivolatile compounds for both qualitative and quantitative analysis.

Keywords: Gas Chromatography, Gas Chromatography/Mass Spectrometry, Headspace, Sample Introduction

Application Code: Materials Characterization

Methodology Code: Gas Chromatography/Mass Spectrometry



A: Syringeless Injector B: Carrier gas inlet C: Injection port liner  
D: Injection port E: Carrier gas flow preheating F: Solid Sample  
G: Sample Vial H: Volatiles Collection flow I: Capillary Column