

INJECTORS

Heated Static Headspace Injector

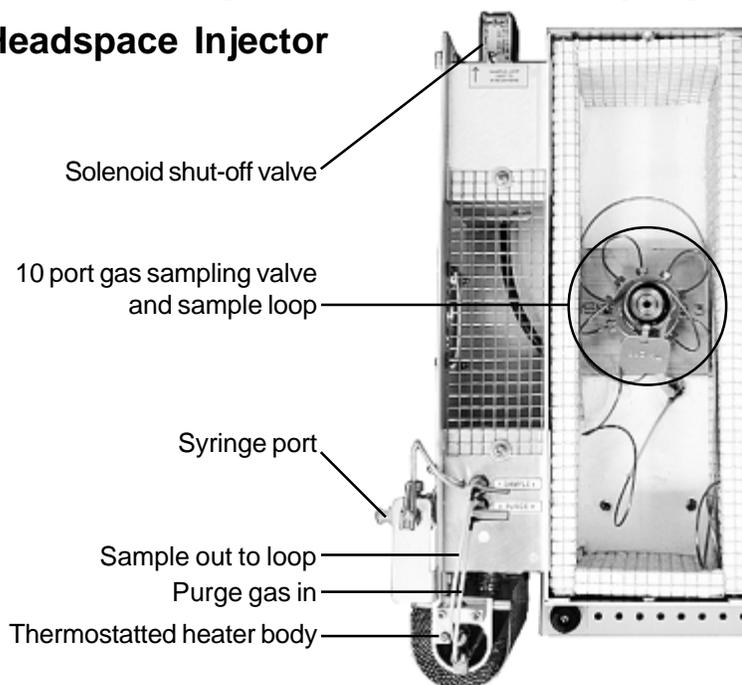
Overview



The Heated Static Headspace Injector is installed on the left side of the GC chassis

The Heated Static Headspace Injector is useful for analysis of the light volatiles that can be partitioned into the headspace of a vial containing either a liquid or a solid matrix sample. It can be used with dirty or complex samples, such as blood, urine, powders, foods and flavors. The SRI Heated Static Headspace Injector is built into the 8610C GC, on the left side of the chassis, eliminating the need for transfer lines and reducing dead volume. Thermostatted from ambient to 90°C, a heater body accepts a 40mL VOA vial containing 10-20mL of sample. Covered with a protective heat shield, the heater body is heated and mechanically agitated under control of the PeakSimple data system. Two needles puncture the vial's septum top upon insertion into the heater body. Purge gas to pressurize the vial is delivered through one needle. The other needle carries the headspace vapors to the sample loop on the 10 port gas sampling valve, located in the valve oven. On the downstream end of the loop is a solenoid shut-off valve, also controlled through the data system. This solenoid shut-off valve opens to fill the sample loop with the sample headspace from the pressurized vial. A syringe port allows the addition of internal standards, spikes, etc. into the vial without exposing the sample to ambient air.

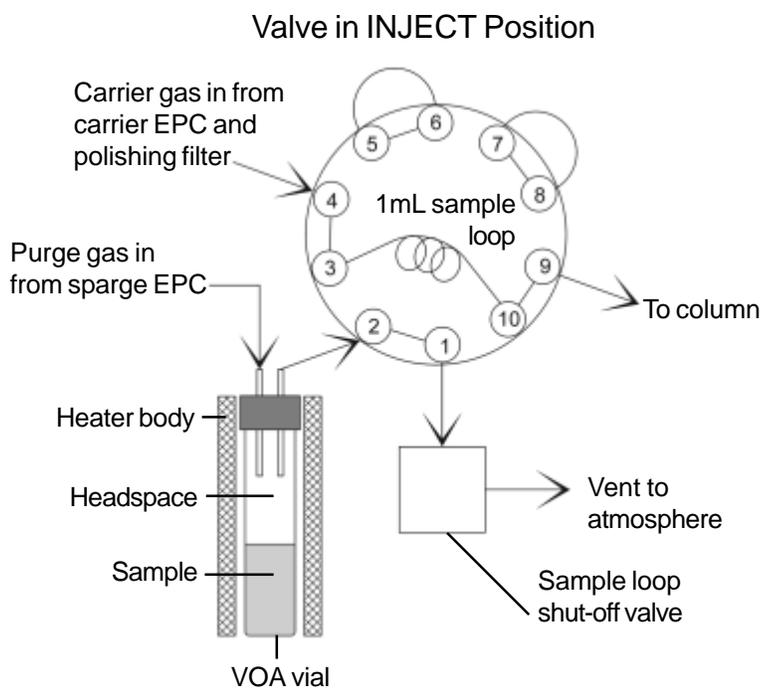
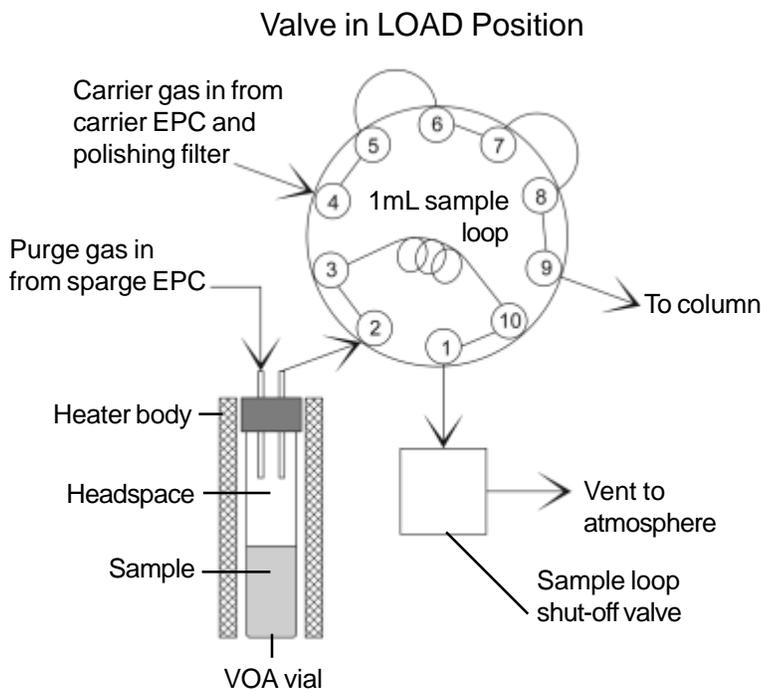
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Theory of Operation



The SRI Heated Static Headspace Injector uses a mechanically agitated heater body for sample equilibration, a 10 port gas sampling valve with a 1mL sample loop, and a sample loop shut-off solenoid valve. The headspace analysis begins with the sample equilibration period, during which the gas sampling valve is in the LOAD position. A 40mL VOA vial containing 10-20mL of sample matrix is inserted into the heater body, then heated and agitated (the time it takes to achieve equilibration depends on the sample matrix and the target analytes). Two needles in the upper part of the heater body puncture the septum top of the vial in order to deliver purge gas into and route the sample out of the vial. After the equilibration period, the vial is pressurized so that the sample will escape when the solenoid shut-off valve is opened. The amount of purge gas needed to pressurize the VOA vial depends upon the type of sample. A sample with high liquid content can create sufficient pressure during the equilibration period to fill the sample loop with headspace. A dry sample will require that the purge gas be turned ON, up to 10psi, to pressurize the sample vial. The solenoid shut-off valve at the downstream end of the 1mL loop is then opened briefly, and the pressurized headspace sample fills the loop as it exits through the valve. After the shut-off valve closes, the gas sampling valve is actuated to the INJECT position, placing the sample loop in the carrier gas stream to sweep the headspace sample into the GC column, and on to the detector(s).

INJECTORS

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General Operating Procedures

1. The Headspace injection technique requires little sample preparation, since just the sample headspace, not the sample itself, is run through the GC. Insert 10-20mL of sample into a clean VOA vial and seal it.

2. The purge gas pressure is controlled with an Electronic Pressure Controller (EPC). Set the purge gas to 0-10psi, depending on the liquid content of the sample. SRI recommends helium purge gas.

3. Using the trimpot on the top edge of the GC's front control panel, set the heater body temperature between ambient and 90°C. Pressure builds as the vial is heated. The temperature setting depends upon the target analytes and the liquid content of the sample.

4. Create or load an event table. **Hdspace.evt**, shown at right and on the **Expected Performance** page, is included in version 2.66 (and higher) of the PeakSimple software. A typical event table heats and agitates the vial for 20 minutes; it may take more or less time to achieve headspace equilibration.

5. Create or load a temperature program. The column oven is typically held at the initial temperature (usually 40°C) for the duration of the sample equilibration period, plus 2-4 more minutes.

6. Set the valve oven temperature to 100°C or higher to avoid water condensation (120°C is a typical setting).

7. Activate and energize your detectors as necessary. Consult the manual sections for your particular detector(s).

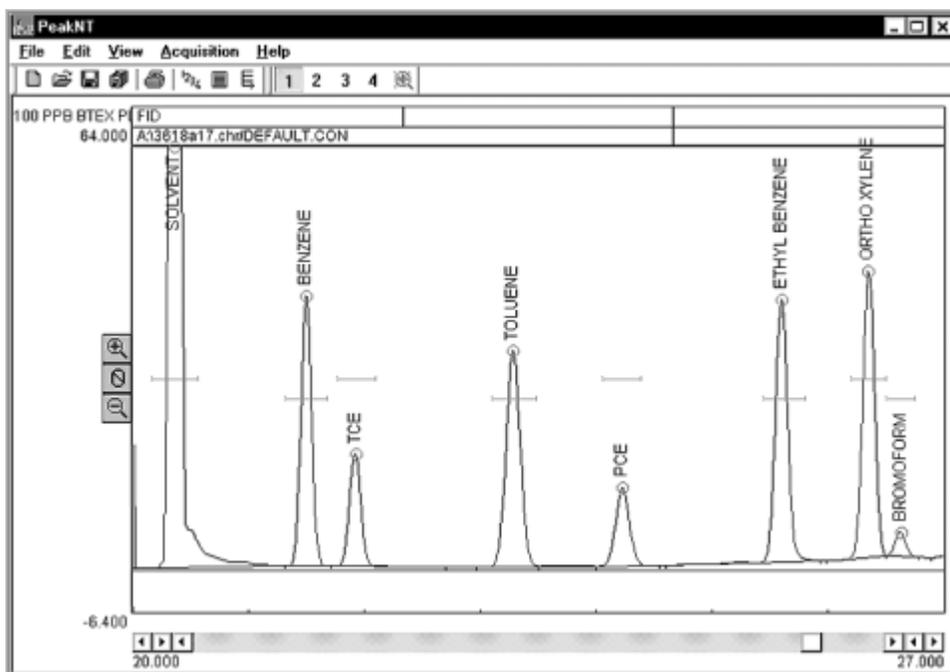
8. Insert a VOA vial filled with 10-20mL of sample into the headspace heater body: slide the vial into the heater body from the bottom. You will feel some resistance as the needles meet the vial septum lid, and once the needles have penetrated the septum, the vial will stop against the top of the heater body interior. The needles will hold the vial in place. Begin the analysis by pressing the RUN button on the GC or the spacebar on your computer keyboard.

Hdspace.evt		
EVENT TIME	EVENT	EVENT FUNCTION
0.000	ZERO	Zero signal
0.100	F "ON"	VOA Vial Heater
0.200	D "ON"	Shaker solenoid "ON"
19.400	D "OFF"	Shaker solenoid "OFF"
19.500	F "OFF"	VOA Vial Heater
19.600	E "ON"	Purge "ON"
19.700	E "OFF"	Purge "OFF"
19.800	A "ON"	Sample loop exit solenoid open
19.900	A "OFF"	Sample loop exit solenoid closed
20.000	G "ON"	Valve in INJECT
27.000	G "OFF"	Valve in LOAD

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Expected Performance



Sample: 1µL 100ppb BTEX Plus (vial 25% full of sample solution with 10µL of 100ppb BTEX Plus) VOA vial set to heat from ambient temperature to 50°C
 Column: 15m MXT-VOL
 Carrier: helium @ 10mL/min

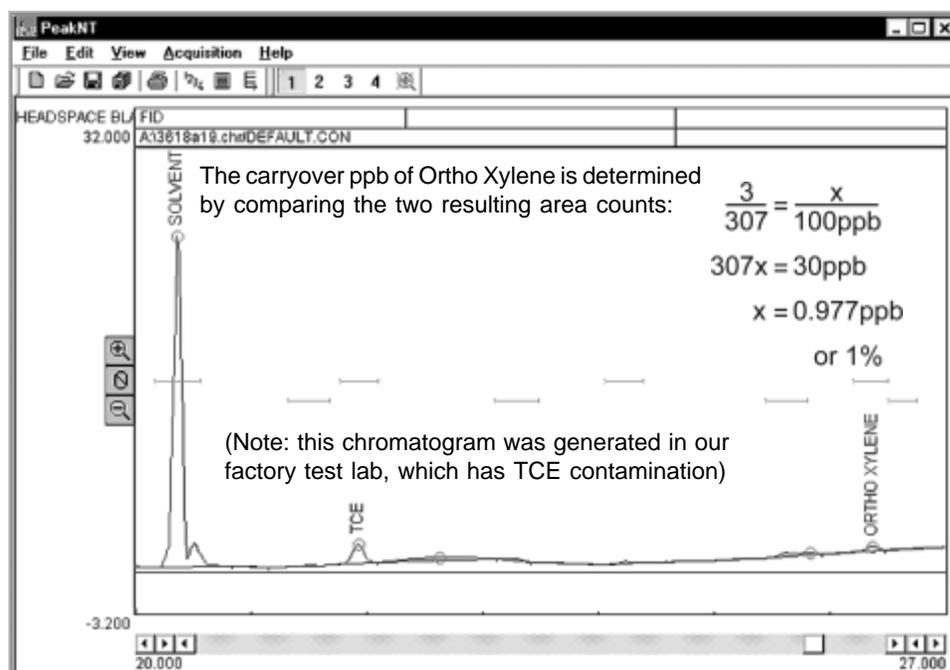
FID gain: HIGH
 FID temp: 300°C
 FID ignitor: -400
 Valve temp: 120°C

Column oven temperature program
(Hdspace.tem):
 Initial Hold Ramp Final
 50°C 23.00 10.00 160°C

The following two chromatograms were produced in series by an SRI GC equipped with a static headspace injector and an FID detector. The first chromatogram is a 100ppb BTEX Plus sample, and the second is a water blank. Both were run under identical conditions. Magnified for visibility, the water blank shows the carryover level of the Headspace injection system.

Events:

Time	Event
0.100	F ON (VOA vial heater)
0.200	D ON (shaker solenoid)
19.400	D OFF
19.500	F OFF
19.600	E ON (purge gas)
19.700	E OFF
19.800	A ON (sample loop exit solenoid)
19.900	A OFF
20.000	G ON (valve actuator)
27.000	G OFF



BTEX sample results:

Component	Retention	Area
Solvent	20.350	7332.0805
Benzene	21.483	266.2780
TCE	21.916	119.6645
Toluene	23.266	285.2310
PCE	24.216	98.6710
Ethyl Benzene	25.583	298.6540
Ortho Xylene	26.333	306.6115
Bromoform	26.616	24.4815
Total		8731.6720

Water blank results:

Component	Retention	Area
Solvent	20.350	137.0385
TCE	21.916	10.0770
Ortho Xylene	26.350	3.1305
Total		150.2460