PYROJECTOR II

Installation and Operation Manual

The Projector Mk 2 was tested in accordance with the EN55011 Group 1, Class B emission requirements and EN50082-1 immunity requirements, and EN61010-1. The test sample complied with both the EMI and EMC test requirements as required by the Eurpoean EMC Directive 89/336/EEC, and Low Voltage Directive as required by 72/23 EEC.

CE

PYROJECTOR II

Index

1.0 WARRANTY

The Pyrojector II control module is guaranteed against faults in materials and workmanship for a period of twelve months from the date of invoice.
The Pyrojector II furnace assembly and components are subject to three months warranty from the date of invoice.

This warranty implies free repair and/or replacement of defective goods, only upon proper written proof and (where authorized) return of the defective product.

This unit has been designed to fulfill the purpose of pyrolysis of small samples for gas chromatography. This warranty is void if the instrument is used for any unrelated purposes. SGE reserves the right to refuse free service under warranty on any unit that has been abused or tampered with in any way.

No other warranty or representation is expressed or implied by SGE for its products with respect to merchantability, fitness for any particular use or purpose or any other matter. SGE shall not, under any circumstances, be liable for any incidental, consequential, or compensatory damages arising from use of, or in conjunction with, its products. The maximum liability for breach of warranty shall be the invoice price of said products. SGE acknowledges that there are many service engineers in the field who have expertise to service the Pyrojector II and to assist them, we have enclosed the necessary data in this manual. To protect your interest however, we respectfully suggest that any failures be attended to by SGE service personnel or by SGE authorized service people.

Note: SGE does not advocate the use of hydrogen with this accessory due to potential problems associated with its inflammable nature.

2.0 INTRODUCTION

Pyrolysis Gas Chromatography is a specialized sample introduction technique in which a high molecular weight sample is heated rapidly to pyrolysing temperatures up to 900˚C in a non-oxidizing atmosphere.

Under these conditions the high molecular weight sample will fragment or "unzip" into lower molecular weight fragments, which can be chromatographed in the conventional manner.

The result is normally a complex GC chromatogram (or pyrogram), whose profile is characteristic of the particular parent sample under the particular pyrolysis conditions. This technique is especially valuable for the rapid identification of samples, which are not chromatographable by conventional GC techniques.

Examples of some of the areas of application are:
Identification of synthetic polymers
Bacteriological identification
Characterization of natural polymers
Forensic applications such as fiber or paint cross-matching

There are two major types of pyrolysis units:
1. Continuous–mode pyrolyser – Essentially an on-line inlet furnace which is continuously heated to the desired pyrolysis temperature. SGE's Pyrojector II is an example of this type of system. The sample is injected directly into the furnace using special types of syringes. The advantages of this system are that the pyrolysis conditions can be controlled very accurately and reproducible sample sizes are more easily achieved.
2. Pulse-mode pyrolyser – The sample is placed directly onto the cooled element at the pyrolysis probe tip. The probe is then inserted and sealed into the inlet and the element is rapidly heated (at a uniform rate), to the pyrolysis temperature range. Pulsed systems have the disadvantage that the exact temperature is not measurable and pyrograms are more hardware dependent. In addition, small sample sizes are more difficult to control.

General Considerations
The pyrolysis profile obtained is a function of the individual type and design of the pyrolyser and the gas chromatograph conditions. There are several factors that will affect the pyrogram profile and reproducibility. Among these are:
Temperature control: Dependent on pyrolyser design and hardware considerations. (Furnace ID, length of heated zone, etc).

Pyrolysis furnace gas flow: The gas flow rate determines the residence time of the sample pyrolysis products in the furnace and may affect secondary pyrolysis.
Sample size: Best results are achieved with very small sample sizes, which minimize the occurrence of secondary reactions.

Pyrolyser/GC Interface: Choice of the GC inlet type is important, primarily due to factors such as discrimination and the width and size of the sample band as it is introduced into the column.

SGE's Pyrojector II has been developed with all these considerations in mind to give reproducible pyrolysis.

3.0 INSTALLATION

Before commencing installation, please check all items listed on the packing list (page 12) have been supplied. Contact your local SGE office or supplier if any parts are missing.

3.1 FURNACE ASSEMBLY

For shipment, the furnace is packed in a number of pieces and requires assembly. Refer to Figure 1 for the diagram of the furnace assembly.

Insert a plug of quartz wool (6) into one of the quartz furnace liners (3) to form a bed approximately 5mm thick. This plug should be positioned to be in the centre of the furnace once installed.

This plug of quartz wool is required to prevent the sample from passing through the furnace into the cooler, lower part of the tube without undergoing satisfactory pyrolysis. The quartz wool is also of use to prevent particulate matter falling down into the transfer line, resulting in blockages.

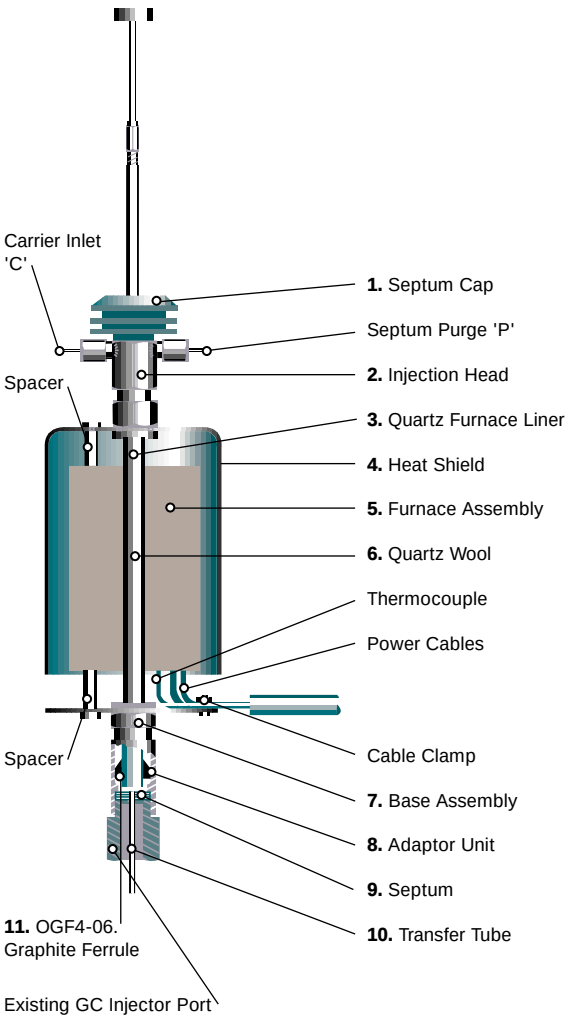
The Pyrojector II is supplied with three different sample injection heads:
- Septum injection head
- Septumless injection head
- Pelletizer injection head

See section 4.5 on page 7 for a guide to selecting the most suitable injection head for your application.

Lay the furnace on its side and slide the quartz furnace liner packed with quartz wool into the furnace. (The liners are symmetrical and can be inserted either way).

Fit a 1/4" graphite ferrule with 6mm ID (11) onto the outlet end of the quartz furnace liner (3). The tapered end of the ferrule should face into the baseplate assembly (7). (See Figure 1)
Insert the transfer tube (10) into the GC instrument specific adapter (8) and screw the adapter onto the baseplate to lock the quartz furnace liner into position. (It is essential that the quartz furnace liner is fully located into the end of the transfer tube end fitting to avoid creating a large dead volume.) Tighten the adapter finger-tight, then one half of a turn with a spanner.

Figure 1



Slide a 1/4" graphite ferrule with 6mm ID (11), taper first over the quartz liner at the top of the furnace and screw on the selected sample injection head. Finger tighten at first and then a further half turn with a spanner.

CAUTION: Do not overtighten the fittings as damage to the furnace liner may result.

3.2 INSTALLATION ON THE GC INJECTOR

There are a range of instrument specific adapters (8) available to suit the injector of most commercial gas chromatographs.

> Before proceeding any further, ensure that the gas chromatograph has been turned off and that the injection port has cooled to room temperature. Turn off all gas flows and allow any pressure in the system to equilibrate to pressure.

(See section 7.0). The type of GC injector that the Pyrojector II is to be used with should have been specified at the time of ordering the Pyrojector II.

Remove the GC injector's septum cap and septum. (Store these items as they will be required when the pyrolyser is not being used.)

Fit a new triple layer septum (9) into the injector and slide it over the transfer tube (10) on the furnace. (The septum size should be the same as normally used on the injector). Slide the transfer tube into the injector port of the gas chromatograph. Fasten the furnace by screwing down the instrument specific adapter (8).

Note: Be careful to allow the injection head assembly on top of the furnace to turn freely.

3.3 MAIN CONTROL MODULE

3.3.1 Main Voltage Selection

Before connecting the control module to mains power, ensure the correct voltage to suit the supply in the laboratory has been set. This is indicated on the voltage selector on the rear panel of the instrument. Insert the correct fuse:

– 800mA for 240 volt systems
– 1.25A for 110 volt systems

MAIN ELECTRICAL CABLE

The power cable must be fitted with the correct plug to suit your electrical power outlet.

Note: Local electrical regulations for power cabling must be complied with. It is the responsibility of the user to ensure that the correct power plug with an earth connection is attached to the mains lead.

3.3.2 Gas Connections

Locate the Pyrojector II control module at a convenient place on or near the gas chromatograph.

Connect a 1/8" gas supply line from the main carrier line to the rear of the control module. Use a 1/8" graphite front ferrule for sealing.

Two 1/16" lines should be connected to the PYROLYSIS GAS OUT and SEPTUM PURGE IN on the rear of the control module using 1/16" vespel sealing rings. These lines are then connected to the injection head (2) on the furnace.
(Ensure the pyrolysis zone of the injection head is blocked off.

- in the septum injection head a septum should be in place;
- with the pellitizer injection head the supplied plug should be in place;
- with the septumless injection head the seal is closed.

When the gas connections are complete, and before heating of the furnace is commenced, pressurize the system and check for leaks around all connections.
(It is recommended that the outlet of the GC injector be blocked and the system pressurized. Then turn off the gas supply and monitor the supply pressure – any leak will result in a drop in the pressure reading).

If hydrogen is to be used as the carrier gas it is the responsibility of the user to take all necessary safeguards with respect to safety. SGE does not recommend the use off hydrogen in this system. Hydrogen leaks will result in a fire and it should be noted that hydrogen burns with an invisible flame.

> Gas Leaks must be eliminated before proceeding with the operation of the system.

3.3.2.1 Septum Injection Head and Pelletizer Injection Head
The connections are made as follows using 1/16" vespel sealing rings provided.

Module Injection Head
Pyrolysis gas out to "C"
Septum purge in to "P"

3.3.2.2 Septumless Injection Head
The PYROLYSIS GAS OUT from the rear of the control module is connected to the "carrier in", as shown in Figure 2.

The SEPTUM PURGE IN on the module is connected to the purge point on the pneumatic head.

To operate the pneumatic seal in the septumless injection head a means of switching gas to the pneumatic head needs to be installed.

Connect a 1/16" OD compressed gas line to the pneumatic head (16) using a 1/16" stainless steel nut (14) and 1/16" vespel sealing ring (15). The line is then connected using a 1/16" vespel sealing ring to the MTV-3 toggle valve as illustrated in Figure 3.

A 1/16" line is then connected to the base port of the MTV-3 valve to a compressed gas source of not more than 100psi, but not less than 50 psi. Again use a 1/16" vespel sealing ring. The MTV-3 should be installed in a convenient position.

3.3.3 Furnace Power Cable
The power cable from the furnace incorporates the low voltage heater power lines and the thermocouple wires. Plug the cable into the rear of the control unit.

Figure 2

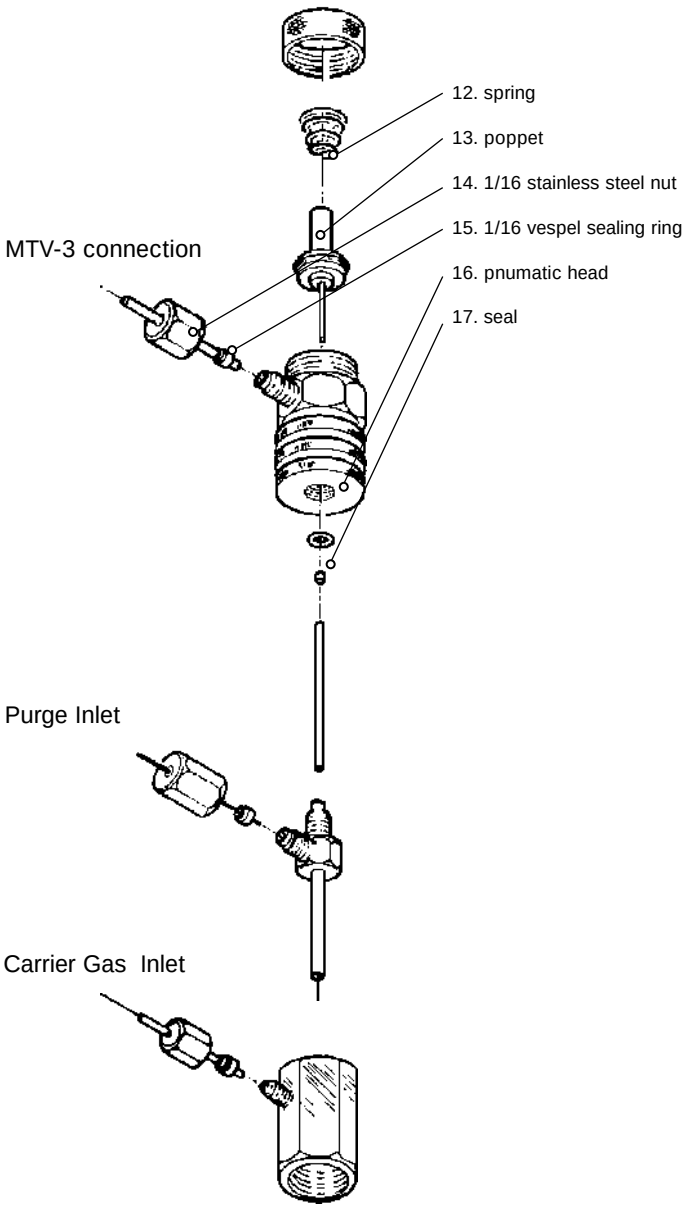
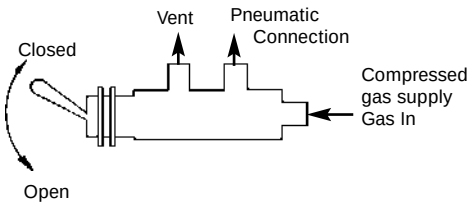


Figure 3



4.0 OPERATION

4.1 FRONT PANEL CONTROLS

PURGE Used to set the septum purge flow to 0.5 - 1 mL/min. Flow may be measured at the rear panel.

PRESSURE Regulator for setting the pressure in the pyrolysis furnace. Usually set to 5psi above the column pressure.

FURNACE PRESSURE Indicates the pressure at the inlet to the furnace. (psi)

TEMPERATURE SET
Pressing either the up or down TEMPERATURE SET buttons displays the set point temperature of the temperature control circuit (˚C).

4.2 REAR PANEL CONNECTIONS

PYROLYSIS GAS IN Inlet carrier gas line for furnace pyrolysis gas.

PYROLYSIS GAS OUT Outlet carrier gas line to be connected to the furnace purge head connection "C".

SEPTUM PURGE IN Septum purge line connector from the septum purge outlet on the furnace purge head connection "P".

SEPTUM PURGE OUT Outlet port for checking the purge flow and venting the septum purge gas to a safe disposal area.

MAIN SWITCH/FUSE Main power inlet and fuse holder. Use the following fuse:

- 220/240V 500mA
- 110/120V 1.0amp

FOUR PIN SOCKET Power socket for furnace supply and temperature sensor.

VOLTAGE SELECTOR Allows selection between 120V and 240V.

Figure 4

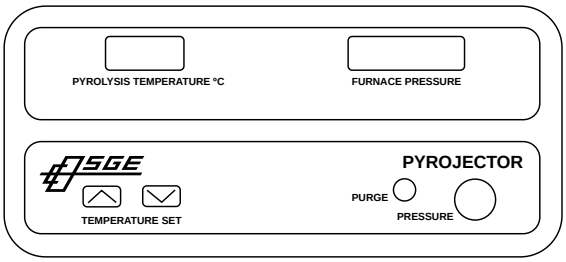
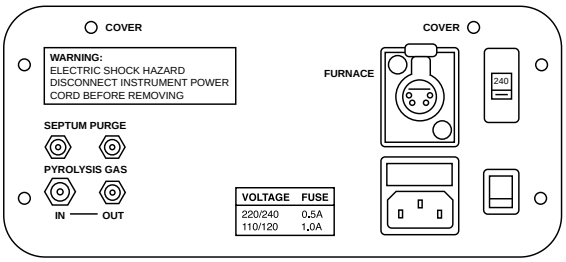


Figure 5



4.3 GAS FLOWS

Optimize the gas flows on the GC to those settings required for the particular analysis and allow the oven, detector and injector temperatures to reach their set point.

Adjust the pressure control on the Pyrojector II's control unit to give a pressure reading on the control unit pressure gauge of 5psi above that of the column head pressure. Refer to the operating hints section to provide an indication of the effect of gas flow.

4.4 TEMPERATURE SETTING

The digital temperature controller used in the Pyrojector II is a microprocessor developed specifically for the SGE range of instruments. The "temperature set" facility built into this controller incorporates both a fast and slow scroll modes to allow for easy temperature setting over large temperature ranges.

To set the desired temperature, press either the up or down button labeled "TEMPERATURE SET" on the lower left side of the front panel. It will automatically display the set temperature. Press the set button once and the temperature controller will be activated in the fast scroll mode. Press the set button a second time and the temperature controller changes into the slow scroll mode. With the set buttons released, the temperature controller will display the set temperature for a few seconds and then automatically change back to displaying the actual temperature.

4.5 SAMPLE INJECTION

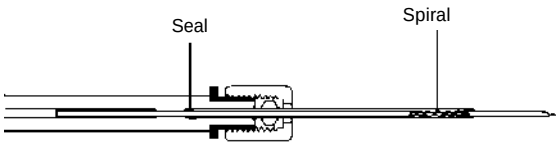
Three different sample injection heads are

supplied with the Pyrojector II. Each is intended for a particular type and size of sample.

4.5.1 Conventional Septum Injection
The sample is injected through a septum. The pyrolysis zone is always sealed from external air. A conventional syringe, with a needle diameter 0.75mm or less, may be used for injecting liquid samples or samples in solution.

4.5.1.1 Low Viscosity Liquid Samples

Figure 6



For low viscosity liquids, or samples dissolved in volatile solvents, a conventional syringe is recommended. (SGE does not recommend using plunger-in-needle syringes due to the high temperatures in the furnace, which could cause the plunger to bind in the needle.) When using the septum injection head, a needle of 7.5cm length is recommended.

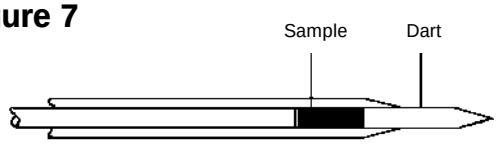
4.5.1.2 High Viscosity Liquid Samples
For liquids that have a high viscosity, or contain a high level of suspended material, we recommend the use of the SGE Solids Injector (7.5cm) with a spiral plunger (p/n 009982).

Samples in a solution are applied to the spiral section of the plunger. The solvent is evaporated depositing the solid on the spiral section of the plunger, which is then retracted into the needle. The injection port is then penetrated and on depressing the plunger fully the sample is carried into the heated injection port.

4.5.1.3 Solid Samples
Solid samples in the form of granules, fibers and powders can be packed into the bore of the solids injector needle with the plunger retracted. Alternatively, samples can be cut out with the sharp tip of the needle or pressed into pellets using the Pelletizer.

When solid samples are being introduced, the supplied solids injector (7.5cm) with the flat tipped plunger may be used. It is recommended that a dart is used in the end of a solids injector syringe when injections through a septum are being performed. This guides the needle through the septum, preventing septum material being deposited in the pyrolysis zone. After penetrating the septum, the plunger is depressed, and both the sample and the dart ejected. Loose darts can be recovered at a convenient time from the furnace when cleaning or replacing the liner.

Figure 7



4.5.1.4 Pelletizer P-1
Withdraw the long plunger from the Pelletizer barrel and ensure the short plunger is inserted into the narrow end of the barrel. Insert (or pour) sample into the wider bore of the glass barrel forming a pellet by compacting the sample between the ends of both plungers.

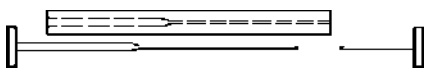
At this stage, for certain samples e.g. Thermoplastics, a denser pellet may be moulded by gently heating the barrel while the sample is under compression between the two plungers.

Withdraw both plungers and insert the needle of the solids injector, with its plunger retracted, as far as possible into the large bore of the Pelletizer barrel.

Transfer the pellet to the solids injector by inserting the longer plunger into the small bore of the Pelletizer and forcing the pellet into the needle.

Insert a dart into the end of the needle, after withdrawing it from the Pelletizer, and inject the sample into the furnace.

Figure 8



4.5.2 Septumless Injection

Solid samples may be introduced using the solids injector (Needle & Plunger_11cm) kit (supplied), by changing over the needle and plunger. (This is to ensure the sample is ejected into the centre of the furnace).

Because the seal is opened pneumatically as the syringe needle is inserted into the injector, there can be no damage to the seal by the needle. This has the advantage that it is not necessary to cool the pyrolysis zone regularly to remove accumulated darts.

The septumless injection head may be used with needles up to 0.7mm diameter. For preparation of the sample for injection the instructions as for the septum injection head should be followed in sections 4.5.1.3 and 4.5.1.4. The exception is that darts are not required when the solids injector is being used to introduce solid samples. (See Figure 2)

4.5.2.1 Operation of Septumless Injector

1. Set the required conditions for the chromatograph and the Pyrojector II.
2. After the pyrolysis temperature has stabilized, take the sample into the solids injector as described in section 4.5.1.3 or if a liquid sample, into a conventional syringe with a needle diameter less than 0.7mm.
3. Gently introduce the syringe needle into the poppet (13) until it meets the resistance of the seal.
4. Actuate the toggle valve to introduce the compressed gas into the pneumatic head. The poppet will lift against the spring (12) allowing the seal (17) to open which allows the needle to be passed through into the injection area.
5. Immediately close the toggle Figure 3, make the injection swiftly and withdraw the needle.

NOTE: It is not necessary to reopen the seal to withdraw the needle.

4.5.3 Pelletizer Injection

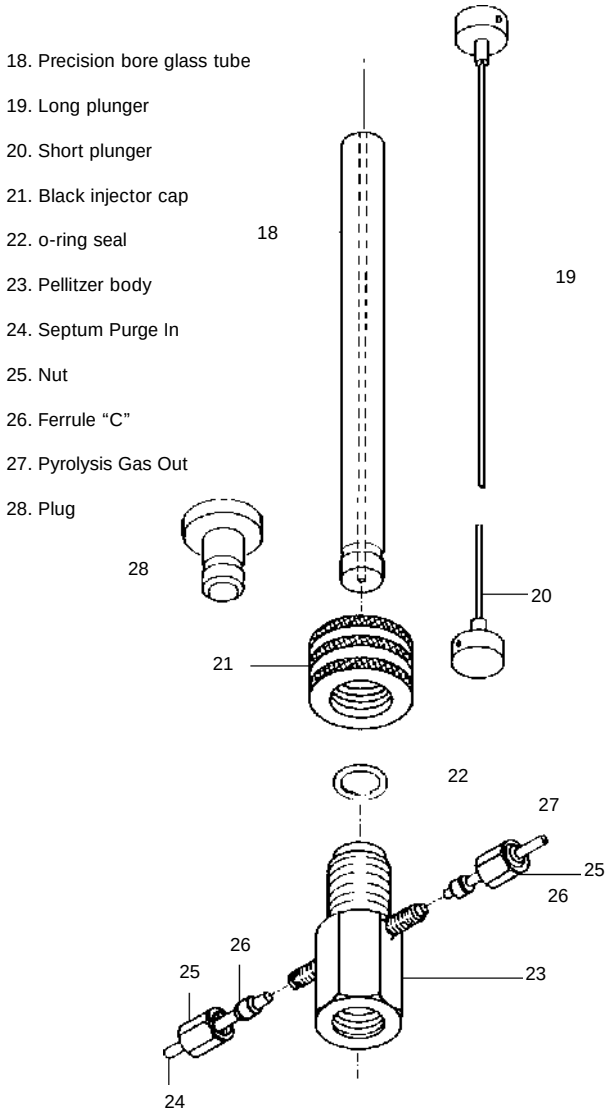
The sample size of solids introduced by the above methods is limited to a few milligrams. In some instances it is necessary to introduce larger sample sizes than this. This is most likely when the sample under investigation has a low level of organic matter present e.g. when geochemical samples such as coals, petroleum source rocks and recent sediments are being examined.

SGE has designed the Pelletizer Injector (P-3), for the introduction of this type of sample, with an inside diameter of 1.6mm. It is ideal for sample sizes up to 10 milligrams.

The P-3 Pelletizer consists of a precision bore glass tube (18), a short plunger (20) to help compress the sample and a long plunger (19) for expelling the solid sample into the Pyrojector II furnace. (See Figure 7)

The Pelletizer Injection Head is installed as detailed in Section 3.1.

Figure 9



4.5.3.1 Operation of Pelletizer Injector

1. The solid sample to be analyzed needs to be in a cut up or ground state, so that it can be introduced into the P-3 glass pelletizer tube (18). Do not attempt to deposit too much sample in the tube so that the plug cannot be moved easily when pushed with the plunger.

2. When the sample has been placed in the tube it should be gently compressed using the two plungers. The shorter of the two plungers should be inserted in the end of the glass that is grooved. Do not overcompress the sample, as this will make it difficult to expel the sample from the pelletizer into the furnace. (Overcompression will also affect the dispersion and rapid heating of the sample in the furnace.) Remove the short plunger from the glass tube.3. With the furnace carrier gas turned off, loosen the black injector cap (21) and slide the grooved section of the pelletizer tube into the o-ring seal (22) in the injector head. Firmly tighten the black cap and set the correct pyrolysis gas pressure at the control module. Check that the septum purge is approximately 1-2mL/min.

4. When the conditions in the gas chromatograph are ready for injection, quickly depress the P-3 plunger so that the sample is rapidly expelled from the tube into the hot quartz furnace liner. It is important that the sample is sent all the way into the middle of the hot furnace zone and caught in the quartz wool.

5. Leave the glass pelletizer with the plunger depressed in the injector head until the GC run is finished and the GC column is cooling down. Then the pyrolysis gas can be turned off and the black septum cap can be loosened and the pelletizer removed.

6. A plug (28) is provided to block the open end of the quartz lined pyrolysis zone while the P-3 pelletizer has been removed for cleaning or sample loading.

7. Before the next sample is prepared ensure that the pelletizer tube is clean and free of particulate material. Because the plunger is a very accurate fit in the glass barrel any remaining material may jam the plunger and possibly crack the glass.

4.6 FURNACE REMOVAL AND CLEANING

Periodic cleaning of the quartz furnace liners is required to prevent the build-up of excess carbon residues in the liner and for the removal of accumulated darts from the solids injector. (A deterioration of peak shape will begin to become evident when approximately 10 darts have accumulated in the quartz furnace liner).

4.6.1 Quartz Furnace Liner Removal

Caution: Before removing the furnace turn off the electrical power to the control module and allow the temperature of the furnace to cool. When the furnace is sufficiently cool, turn off the gas flow to the furnace from the control module.

Turn off the gas flow to the injector of the gas chromatograph. Use the heat resistant gloves supplied, to prevent burns. Loosen the instrument specific adapter (8) by unscrewing it half of a turn from the baseplate assembly (7). Undo the injection head (2). Carefully lift the injection head up from the quartz liner and lean it to the side. Gently pull the furnace liner out of the furnace.

Note: Even with the protective gloves, make sure the furnace liner has cooled before attempting to hold onto it.

4.6.2 Quartz Furnace Liner Cleaning

Push the quartz wool out of the furnace and discard it in a safe receptacle. Reclaim the darts and clean by washing with detergent and water. (Or a proprietary cleaning agent and an ultrasonic bath.) Clean the quartz tube by rinsing with detergent and scrubbing with a pipe cleaner or thin brush. (It may be necessary to clean it with a proprietary laboratory cleaning agent and an ultrasonic bath.)

Replace the quartz wool plug (using fresh quartz wool every time), ensuring that it is located approximately half way down the liner.

4.6.3 Quartz Furnace Liner Replacement

Re-insert the clean quartz furnace liner by pushing it through the furnace and locating it in the instrument specific adapter (8). It is most important that the furnace liner is located correctly at the top of the transfer tube (10). Tighten the instrument specific adapter (8) half a turn into the baseplate assembly (7) to secure the liner and make a gas tight seal. Reposition the injection head (2) and tighten the nut with a spanner a half turn to seal the assembly to the liner.

4.7 SYRINGE MAINTENANCE

4.7.1 PTFE Seal Replacement

Each Syringe and Repair Kit is supplied with a spare PTFE Seal Tube and Insertion Tube assembly – see Figure 8.

As illustrated, a small PTFE tube seal (6) is located inside the glass barrel, around the plunger, and located at the back end of the needle. Generally, this seal should not require replacement, but if it becomes dislodged or damaged it can be replaced.

Is The Seal In Position?

To check, simply remove nut (33) and spring (31) leaving the needle (30) and plunger assembly still inside the glass barrel (35). Gently push needle into the barrel (35). If the 1.5mm long PTFE Tube Seal (34) is missing, the needle stop (32) will rest flush against the face of the glass barrel. If the seal is in position the needle stop (32) will protrude about 1.5mm from the face of the glass barrel as illustrated in the schematic drawing.

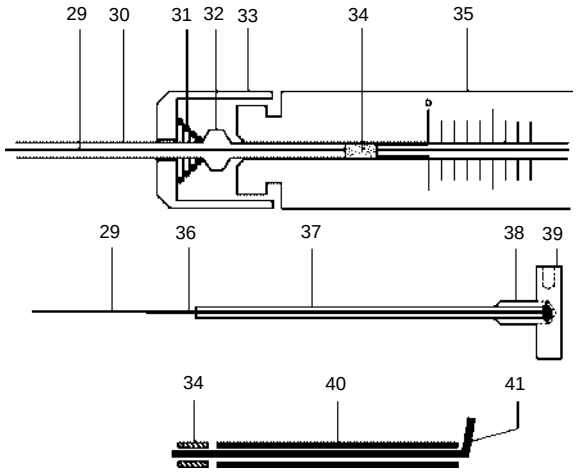
Seal replacement can be made as follows:-

1. Loosen syringe nut (33) and withdraw plunger assembly.
2. Remove nut (33), needle (30) and spring (31) as illustrated.
3. The seal insertion tool is supplied assembled as illustrated, and consists of a stainless steel tube (40) wire (41) and PTFE seal (34).
4. Hold Insertion Tool assembly in one hand and syringe barrel in the other and insert the seal (34) and tube (40) into the small hole in the glass barrel where the needle normally fits. Then withdraw wire (41) about 5mm, which will leave the seal in position. Remove the insertion tool
5. Replace needle (30) and nut assembly ensuring spring (31) is located against stop (32).6. Refit plunger and then tighten nut.

4.7.2 Plunger Replacement

1. Loosen nut (33) and withdraw plunger assembly from syringe.
2. Firmly hold threaded section (38) on guide tube (37) and unscrew button (39). Pliers may be required.
3. Push out plunger wire (29) and strengthening sheath (36) through threaded section of guide tube (37).
4. Fit new plunger and lock into position with button (39).
5. Insert the plunger assembly down level with the zero mark on the scale.
6. Tighten nut.
7. Thoroughly clean needle before use

Figure 10 Type B Syringe



PFTE Seal Insertion Tool

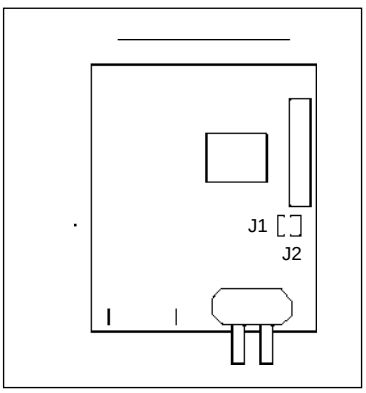
4.8 DIGITAL PRESSURE METER.

Optimum performance in pyrolysis capillary chromatography requires accurate and precise measurement of furnace gas pressure. The Pyrojector II 's Digital Pressure Meter implements a solid state pressure transducer capable of measuring gas pressure from 0 to 100psi
(0 to 690kPa) with a resolution of 0.1psi (1kPa).

To change the pressure units from psi to kPa, remove the covers to gain access to Digital Meter Circuit Board. Find the jumper block labeled J1 & J2 (see wiring diagram). Removal of jumper J1 allows measurement in kPa.

CAUTION: Ensure no power is supplied to the unit when removing the covers.

Figure 11
Pressure Meter Circuit Board



5.0 OPERATING HINTS

The pyrolysis products obtained primarily depend on two things:

- The temperature in the pyrolysis furnace.
- The residence time of the sample in the furnace.

With the Pyrojector II, both of these parameters may be adjusted to obtain the required pyrolysis profile.

As an example:
The chromatograms on page 18 show the effect of changing the pyrolysis temperature on the breakdown of n-Butyl Benzene dissolved in pentane.
For reproducible pyrolysis, the flow of gas through the furnace must be well controlled. In the Pyrojector II, this is achieved with a restrictor in the transfer tube between the furnace and the GC's injector. The pressure in the furnace is controlled separately from the pressure in the gas chromatograph's injector. The pressure in the furnace should be set above the pressure in the GC injector.

A differential of approximately 5psi (35 kPa) is a good starting point but it can be altered to change the pyrolysis products.
Increasing the pressure differential decreases the sample residence time in the furnace because the flow rate through the furnace increases. In this situation the degree of pyrolysis is decreased. The degree of pyrolysis can be increased by reducing the pressure differential to increase the sample residence time in the furnace.

Where non-reconcentrating gas chromatography injection techniques are being used such as split or direct injection, the pressure differential must not be too low. For non-reconcentrating injection techniques it is a requirement that the sample is introduced onto the chromatography column in a narrow band so that there is a minimal contribution to peak broadening from the sampling technique. If the flow in the pyrolysis furnace is too low the sample takes an excessive time to transfer onto the column and is spread along the column, resulting in broad peaks and loss of resolution. (When reducing the pressure differential, watch for signs of peak broadening).

5.1 TEMPERATURE PROFILES

If any changes are made to the column head pressure, the pressure in the pyrolysis furnace must also be adjusted to maintain the same pyrolysis conditions.
Where a split type injector is used, the split flow may be changed within a range without affecting the pyrolysis conditions. Depending on the design of the injector, if the split flow is increased excessively, the pressure in the injector may drop slightly, increasing the pressure differential and thus increasing the flow in the pyrolysis furnace, which may slightly change the pyrolysis conditions.

If identification of pyrolysis products is required, standards can be injected into the Pyrojector II using the following technique. The furnace temperature should be dropped to a level at which breakdown of the standard will not occur. The standard may be injected as a liquid or in solution and can be injected using a conventional microliter capacity syringe. The retention time of the resulting peak can be compared with the pyrogram.

5.2 CRYOGENIC OPERATION

For some injection modes, and where the residence time of the sample in the pyrolysis furnace is likely to be excessive, it will be necessary to focus the pyrolysis products at the start of the column. If the products of interest are non-volatile, it may be possible to do this simply by using a reduced initial temperature in the GC column oven. It will then be necessary to do a temperature programmed GC run.

If it is necessary to focus more volatile components on the column, then cryofocussing will be necessary using liquid carbon dioxide or nitrogen.

SGE has available the following cryogenic accessories to allow cryofocussing to be carried out.

1. The cold trap kit-CO_2

(p/n 093346) is for use with liquid CO_2 and is suitable for the majority of applications where cryofocussing isrequired with the Pyrojector II. The coolant flow is controlled by a special valve, which is pneumatically actuated. The actuation of the valve is controlled through a pneumatic switch (supplied with the cold trap kit) or a solenoid valve which can be operated through the external events of an integrator or gas chromatograph.

2. The cold trap kit-N_2

(p/n 093345) is used where lower temperatures are required and uses liquid nitrogen for cooling. This option would be required where cryofocussing of volatile components in ovens having a relatively high temperature is required. Operation of this system is similar to the CO_2 cold trap system.

3. Indirect Cryogenic Cold Trap
(p/n 0933451), the indirect system allows the choice of coolant to suit the application. Anything from liquid nitrogen to alcohol/dry ice or even ice/brine mixture may be used.

For further information, request PD-0044-A. "Cryogenic Cold Trap – Easily installed kits for Cryofocusing in Gas Chromatography."

6.0 SERVICE

6.1 PACKING & REORDER LIST

This packing list is to be used as a reference when checking the contents of your kit. Any items missed during shipment can be requested from this list.

DESCRIPTION	QUANTITY	REORDER PART NO.
Control Module	1	0950301
Furnace	1	0950220
Power Lead (no plug)	1	
Installation and Operation Manual	1	MN-0280-E
Kevlar Gloves	1	
Pyrojector Accessories Kit:		
- 1/8" Graphite Ferrules (Pkt 5)	1	072602
- Spanner, (1/4" x 5/16"AF)	1	
- Spanner, (1/2" x 9/16"AF)	2	
- 1.25A Slo Blo Fuse	1	18030003
- 800mA Slo Blo Fuse	1	18030001
- 10mL Syringe	1	002060
- Pellitizer Injector (P1)	1	009986
- Pellitizer Injector (P3)	1	009988
- Solids Injector (SI-1)	1	009982
- Pre-Drilled 10mm Triple Layer Septa (Pkt 10)	1	041855
- 1/16" Graphite Vespel Ferrules/0.8mm ID (Pkt 5)	2	072655
- 1/16" Vespel Sealing Rings (Pkt 10)	2	072653
- O-Ring, Silicone (Pkt 2)	1	040110
- O-Ring, Nitrile (11mm)	1	040108
- O-Ring, Nitrile (3mm)	1	040103
- Transfer Tube	1	0950205
- Needle Seal (Pkt 5)	1	09321640
- Quartz Tube for Furnace	2	0950120
- Pellitizer Injection Adaptor	1	
- Septum Injection Adaptor	1	
- Septumless Injection Adaptor	1	
- MTV-3 Toggle Valve	1	
- Deactivated Quartz Wool	1	18060001
- Seat Removal Tool	1	1236101
- Solids Injector (Needle & Plunger Kit_11cm)	1	03162891
- 1/4" Graphite Ferrules with 6mm ID (Pkt 10)	1	0726212
- Steel Darts (Pkt 5)	1	031765
Installation Accessories Kit:		096030
- Stainless Steel Tubing (1/16" x 0.8mm ID x 5m)	1	
- Copper Tubing (1/8" x 1/16" ID x 5m)	1	
- 1/16" Graphite Ferrules (Pkt 10)	1	072603
- 1/8" Graphite Ferrules (Pkt 5)	1	072602
- 1/8" Graphite Vespel Ferrules/1/16" ID (Pkt 2)	1	
- 1/8" Extended Stainless Steel Nut (Pkt 2)	1	
- 1/16" x 1/16" x 1/16" Stainless Steel Union Tee	1	
- 1/8" x 1/8" x 1/8" Stainless Steel Union Tee	3	
- 1/8" x 1/16" Reducing Union	1	
-1/16" x 1/16" Stainless Steel Union	1	
Solid Sampling Kit:		0950204
- Sample Injector Adaptor	1	
- Quartz Tube Adaptor	1	
- Punch/Probe Adaptor	1	
- Injector Head	1	
- Injector Head Cap	1	
- Quartz Tubes (Pkt 50)	2	0950095
- 1/16" Extended Stainless Steel Nuts (Pkt 2)	1	
- 1/16"/1.0mm ID, PTFE Ferrules (Pkt 2)	1	
- O-Ring, Nitrile (Pkt 2)	1	
- 1/16" Graphite Vespel Ferrules (Pkt 2)	1	072661

The Pyrojector II is supplied with one adapter for interfacing the pyrolysis furnace to the GC injection system.

6.2 FAULT FINDING

6.2.1 Electrical Faults

Failure of the thermocouple or the furnace element are the most likely areas where faults can develop. The following table should assist in the location of a problem.

Fault	Possible Cause	Action Required
No readout	No power to control module	Check power is available to unit Check main cable Check fuse for rating and continuity Check voltage selector
	Transformer failure	Test continuity of transformer wingdings
	Thermocouple failure	Replace thermocouple
Temperature Controller readout shows excessive in temperature display	Thermocouple not located correctly in furnace	Refer to thermocouple replacement instructions

6.3 FURNACE REPLACEMENT

Failure of the furnace element requires complete replacement of the furnace. The normal DC resistance of the furnace element is approximately 7-9 ohms, but this will increase with time due to erosion of the filament.
It is recommended that the resistance be measured before purchasing a replacement furnace to ensure that the furnace has in fact failed.

1. Caution: before attempting to replace the furnace make sure that the gases are turned off.
2. Unplug the connector from the rear of the control module.
3. Disconnect the carrier gas and purge lines to the injection head. Loosen the adapter by unscrewing the nut half of a turn. Then undo the injection head. Carefully lift the injection head up from the quartz liner and place it to the side. Gently pull the furnace liner out of the furnace.

NOTE: Make sure the furnace has cooled sufficiently before handling it.

4. Remove the instrument specific adapter unit (8) from the baseplate assembly (7).
The replacement furnace assembly (part number 0950220) includes the heatshield (4), the baseplate assembly (7) and the thermocouple/power cable and plug.
The furnace assembly can be reinstalled by fitting the quartz furnace liner, adapter unit and injector head as detailed in the installation instructions in section 3.1.

6.4 THERMOCOUPLE REPLACEMENT

Thermocouple failure is indicated by 0 or 1 displayed on the temperature controller display. The thermocouple can be replaced and is available upon request.

Caution: before attempting to replace the thermocouple, make sure that the whole system including the gas chromatograph is turned off and all electrical power and gases are turned off.

1. Unplug the connector from the rear of the control module.
2. Disconnect the carrier gas and purge lines to the injection head. Loosen the adapter by unscrewing the nut half of a turn. Then undo the injection head. Carefully lift the injection head up from the quartz liner and place it to the side. Gently pull the furnace liner out of the furnace.

   NOTE: MAKE SURE IT HAS COOLED SUFFICIENTLY BEFORE ATTEMPTING TO HANDLE IT.

3. Place the furnace assembly on the bench and undo the cable clamp and the other screws on the base assembly.
4. Remove the faulty thermocouple from the furnace being careful to ensure that all the wire is removed from the surface.
5. Push the rubber sheath back from the body of the cannon plug and unscrew the cable clamp screw.
6. Loosen the screw from the body of the plug by turning clockwise into the plug. Push the cable assembly through the body until the rear of the pins are exposed.
7. Unsolder the thermocouple from pins 2 and 3.
8. Pull the faulty thermocouple wires out of the cable sheath and discard them.
9. Push the welded end of the new thermocouple wires through the plug, rubber sheath, and then through the cable sheath.
10. Solder the loose ends of the thermocouple wires to pins 2 and 3 of the cannon plug.

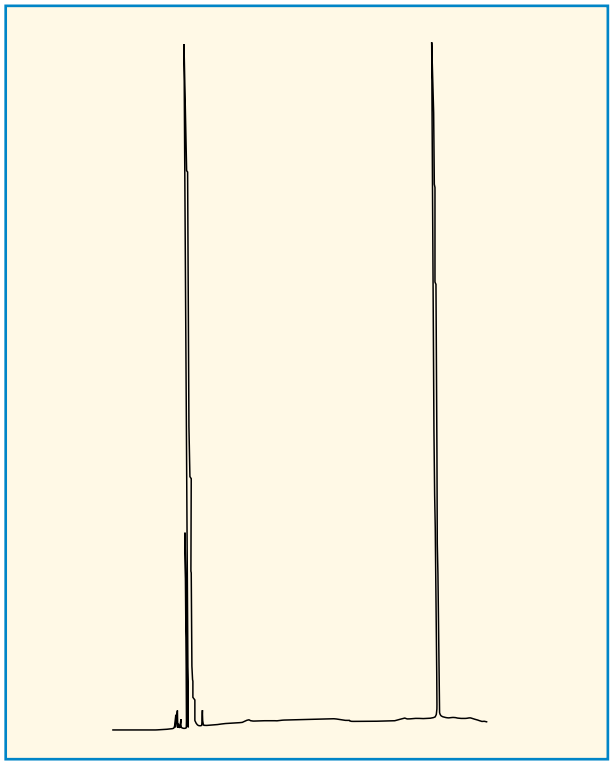
Note the following polarities:
PIN 2: RED BRAID
PIN 3: YELLOW BRAID
Check resistance is 3.5 – 5 ohms

11. Push the pin assembly into the plug body and secure with the screw. Screw in the cable clamp screw to hold the cables and relieve the strain on the pin connections.
12. Replace the rubber sheath on the end of the plug.
13. Push the thermocouple up into the furnace until it is located approximately in the middle of the furnace.
14. Replace the baseplate making sure the three spacers are correctly located on the spacer rods between the furnace and the baseplate and that the cable clamp is located directly below the cables. Screw the spacer rods into the baseplate and lock into position with the locknuts.
15. Replace the cable clamp ensuring that the thermocouple wire is located next to the screw and held in place by the power cable.
16. Replace the furnace as detailed in the installation instructions in section 3.1.

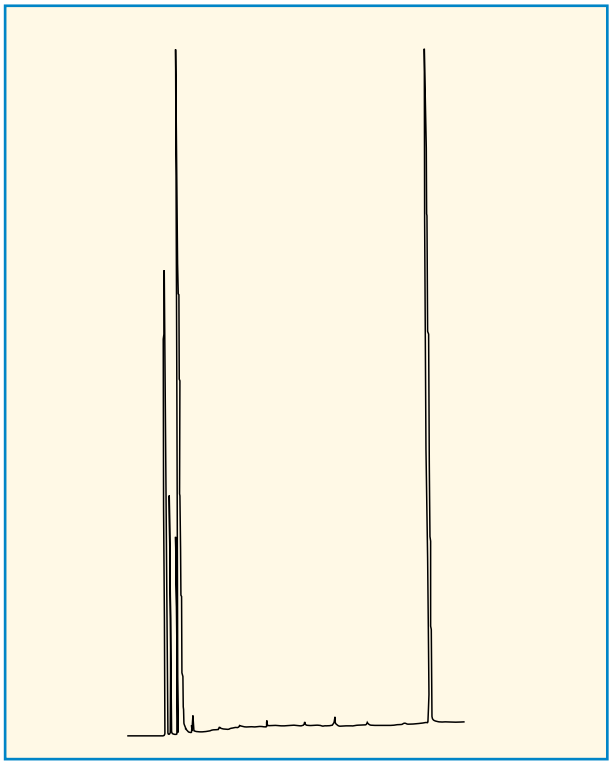
7.0 PYROJECTOR II - GC INJECTOR ADAPTORS

Instrument Description	Part No.
Antek 3000	0950021
PE 8300, 8500	0950021
PE F30, 900/10/90	0950021
Sigma	0950021
Konik	0950021
Pack Beck 437-439	0950021
Pack Beck 436	0950021
Varian 3700 - Packed	0950042
Vista 6000 - Packed	0950042
Varian 3700 - Capillary	0950043
Vista 6000 - Capillary	0950043
Tracor 560/65/70	0950043
Varian 33/34/3500	0950043
Varian 3700-1075 opt	0950045
Vista 6000-1075 opt	0950045
Unijector Mk II	0950051
HP–5830-90, 5700-Capillary	0950013
HP–5830/40, 5700-Packed	0950021
HP–5880/90-Packed	0950011
Girdel 3000	0950052
Delsi DI700, DI200	0950053
Shimadzu 8A-17A	0950071
Hitachi 163	0950071
Finnigan Tremetrics	0950080
Phillips 4400 GC	0950082
Shimadzu SPL-14	0950085
Carlo Erba	0950090
Dani 3000,6500,84/8500	0950091
Carlo Erba 4160	0950091

Pyrolysis Temperature 600°C



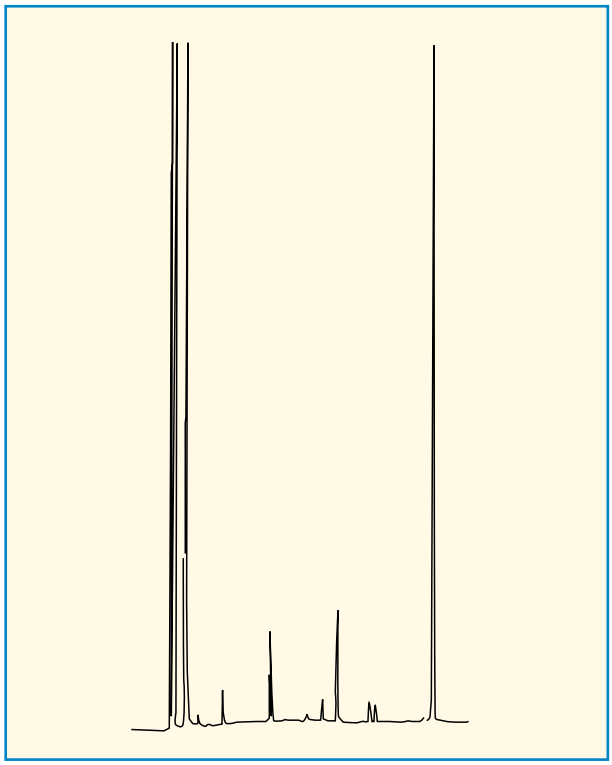
Pyrolysis Temperature 700°C



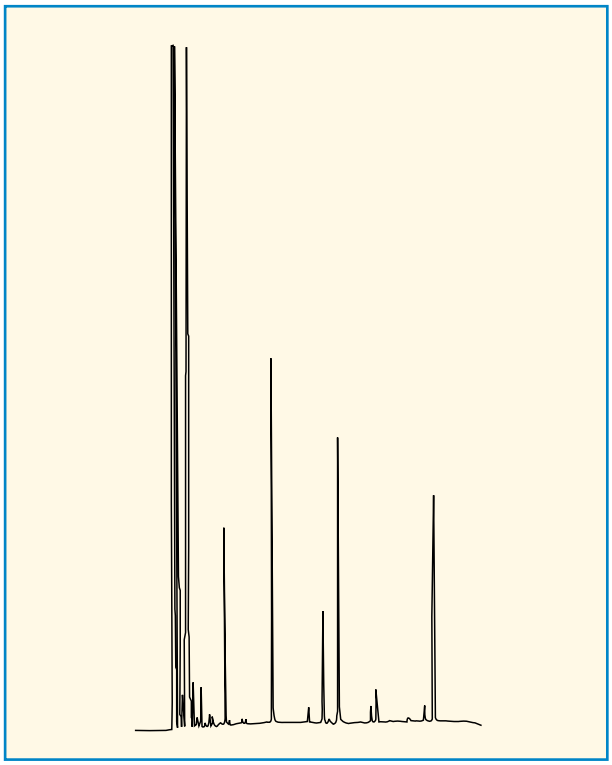
Components:

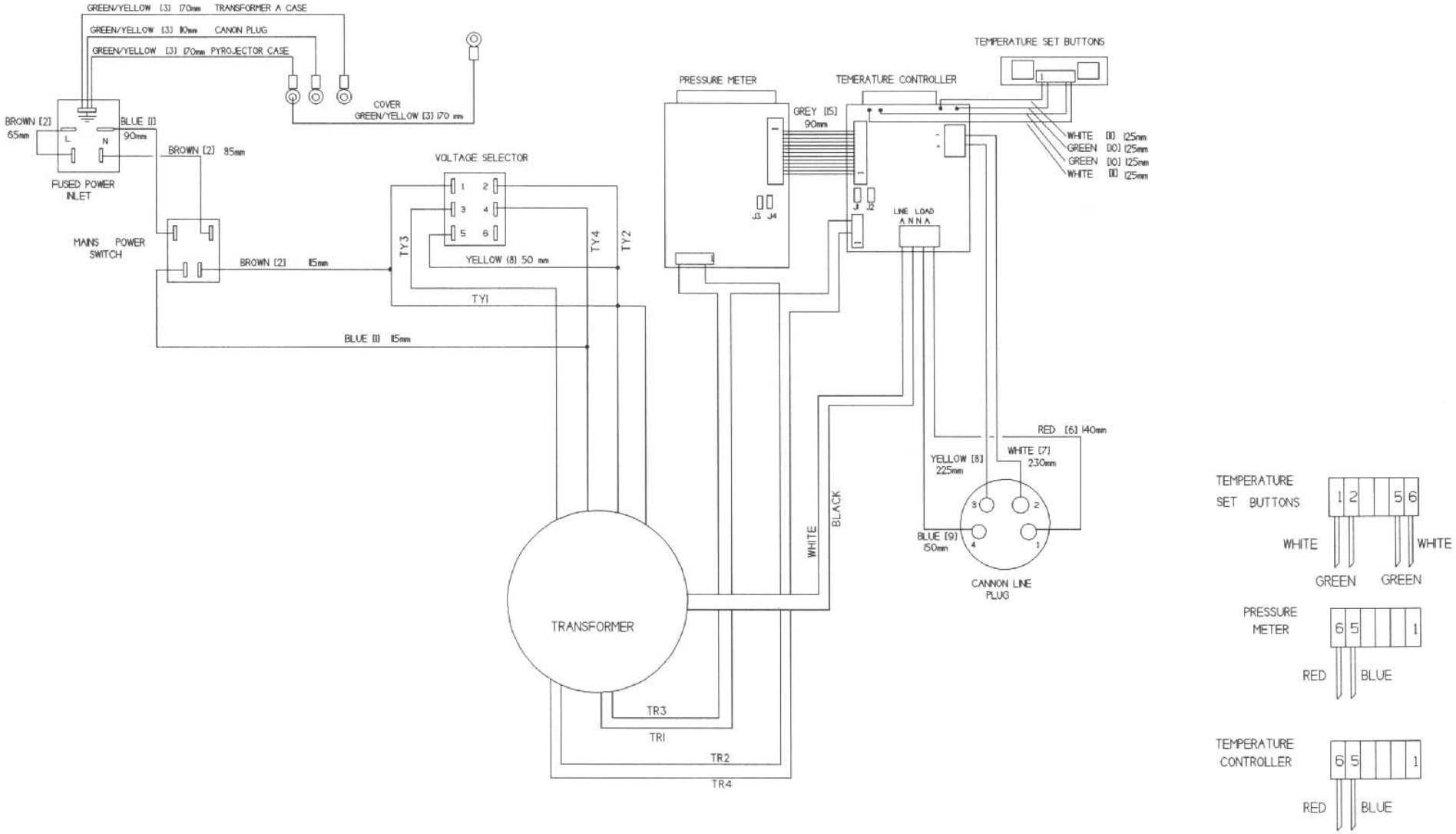
1. Pentane
2. Benzene
3. Toluene
4. Ethylbenzene
5. Styrene
6. n-Propylbenzene
7. n-Butybenzene

Pyrolysis Temperature 800°C



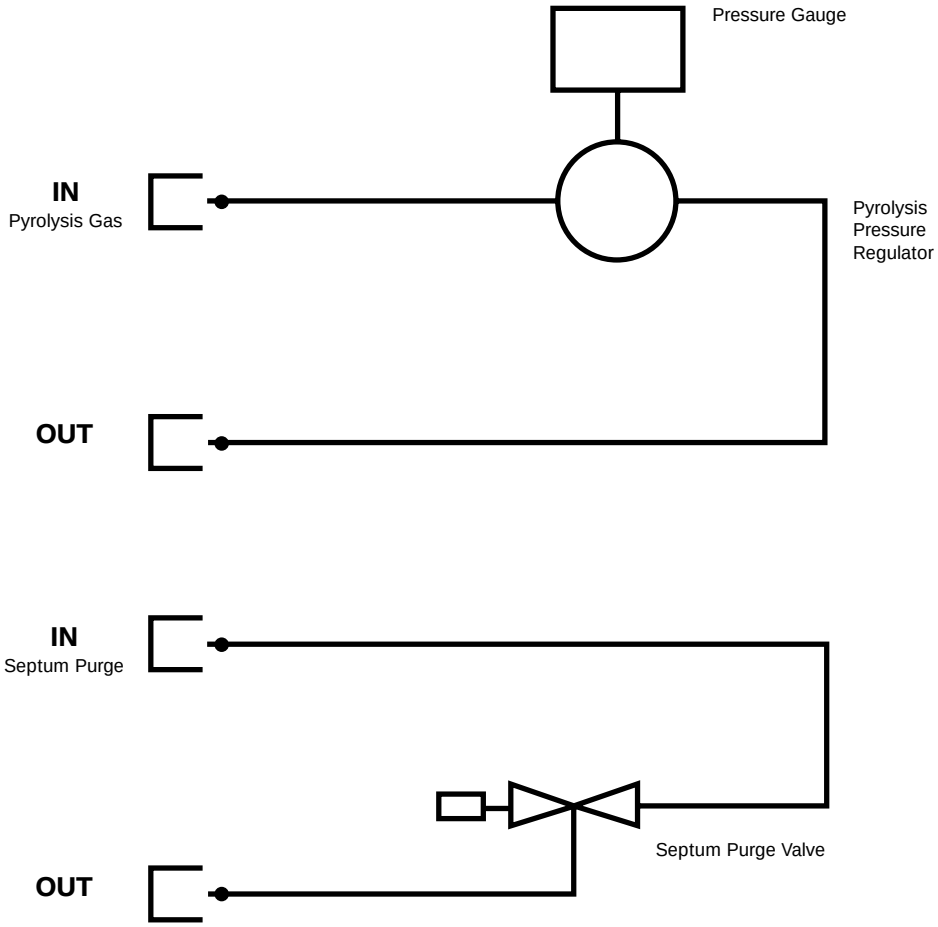
Pyrolysis Temperature 900°C




GREEN/YELLOW [3] 170mm TRANSFORMER A CASE
GREEN/YELLOW [3] 110mm CANON PLUG
GREEN/YELLOW [3] 170mm PYROJECTOR CASE
COVER
GREEN/YELLOW [3] 170 mm
BROWN [2] 65mm
BLUE [1] 90mm
L
N
FUSED POWER INLET
BROWN [2] 85mm
MAINS POWER SWITCH
BROWN [2] 115mm
VOLTAGE SELECTOR
1 2
3 4
5 6
TY3
TY4
TY2
TY1
YELLOW (8) 50 mm
BLUE [1] 115mm
PRESSURE METER
TEMERATURE CONTROLLER
TEMPERATURE SET BUTTONS
GREY [15] 90mm
WHITE [11] 125mm
GREEN [10] 125mm
GREEN [10] 125mm
WHITE [11] 125mm
J3 J4
J1 J2
LINE LOAD
A N N A
RED [6] 140mm
YELLOW [8] 225mm
WHITE [7] 230mm
BLUE [9] 150mm
CANNON LINE PLUG
WHITE
BLACK
TRANSFORMER
TR3
TR1
TR2
TR4

TEMPERATURE SET BUTTONS
1 2 5 6
WHITE
WHITE
GREEN
GREEN
PRESSURE METER
6 5 1
RED
BLUE
TEMPERATURE CONTROLLER
6 5 1
RED
BLUE

Figure 12 Pneumatic Connections



www.sge.com

SGE International Pty. Ltd.
Toll Free: 1800 800 167
Tel: +61 (0) 3 9837 4200
Fax: +61 (0) 3 9874 5672
email: support@sge.com

SGE, Incorporated (USA)
Tel: (800) 945 6154
Fax: (512) 836 9159
email: usa@sge.com

SGE Europe Ltd. (UK)
Tel: +44 (0) 1908 568844
Fax: +44 (0) 1908 566790
email: uk@sge.com

SGE (France) Sarl
Tel: +33 (0) 1 69 29 80 90
Fax: +33 (0) 1 69 29 09 25
email: france@sge.com

SGE Italia Srl.
Tel: 06 4429 0206
Fax: 06 4429 0724
email: sge.italia@tin.it

SGE (Deutschland) GmbH
Tel: +49 (0) 6151 860486
Fax: +49 (0) 6151 860489
email: germany@sge.com

SGE China
Tel: +86 (10) 6588 8666
Fax: +86 (10) 6588 6577

SGE Japan Inc.
Tel: +81 45 222 2885
Fax: +81 45 222 2887
email: japan@sge.com

SGE India
Tel: +91 (022) 471 5896
Fax: +91 (022) 471 6592
email: sgeindia@vsnl.com

Publication No. MN-0280-E Rev:08 10/01