

**MODEL 4000A**  
**CHEMILUMINESCENT**  
**OXIDES OF NITROGEN ANALYSER**  
**OPERATING MANUAL**

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## **ADDENDUM TO 4000A OPERATION MANUAL**

The following caution applies to all issues of this manual,  
up to and including issue 2.01

### **SAFETY INSTRUCTION**

Customers wishing to refill the Ozone trap on the  
Signal Model 4000 Series must take note:

1. The chemical process used to remove the ozone is exothermic and the carbon material becomes hot while the analyser is working.
2. If the trap is opened when hot, the contents may catch fire.
3. At least one hour should be allowed after the instrument is switched off, for the trap to cool. Alternatively, the trap may be removed intact from the analyser and immersed in water. Once cold, the trap may be safely opened and cleaned, and the carbon material replaced.

## INDEX

	<b>Page</b>
1. Unpacking Instructions	5
<b>2. Introduction</b>	<b>6</b>
2.01 The Principle of Chemiluminescence	6
2.02 The Detector/Reaction Chamber	7
2.03 The Ozoniser Assembly	7
2.04 The NO <sub>2</sub> to NO Converter	8
2.05 The Sample Inlet	9
2.06 Electronics Section	9
2.07 Flow Control Schematic Diagram	11
2.08 Photomultiplier Tube Voltage PSU	12
<b>3. Installation</b>	<b>13</b>
3. Installation Overall Diagram	13
3.01 General	14
3.02 High Temperature Environment	14
3.03 Sample Connections	14
3.04 Bypass Flow Output	14
3.05 Detector Vent	15
3.06 Oxygen/Air Connection	15
3.07 Calibration Gas Connection	15
3.08 Output of Remote Range Change	16
3.09 Remote Control of Range Change	16
3.10 Remote Control of Sample/Cal. Gas Selector	17
3.11 Power Input Connection	17
3.12 Input/Output Ports	17
<b>4. Operation</b>	<b>18</b>
4.01 Front Panel Controls	18
4.02 Front Panel Control Diagram	19
4.03 Setting Up Instructions	20
4.04 Diagram of Rear Panel	22
4.05 Shutting Down Procedure	22
4.06 Service Instructions	22
4.07 Spare Parts	23
4.08 Routine Services	23
<b>5. Converter Efficiency</b>	<b>24</b>
5.01 NO <sub>x</sub> Generator Method	24
5.02 Converter Efficiency Detector Diagram	25
5.03 Linearity	26
5.04 Model 4000 Check List	27

## 1. UNPACKING INSTRUCTIONS

This instrument is packaged for general freight purposes. It should withstand the occasional "bumps and knocks" which occur during transit.

Please check the instrument for damage however, and report any damage within 24 hours to the factory or its Sales Office or Distributor.

1. Before any connection is made, unscrew the 4 cross head screws on instrument cover lid.
2. Slide lid back to reveal the internal assemblies of the instrument.  
NOTE: Do not take the cover off with power connected.
3. Check that all PCB's are firmly in their mating connectors on the front of each PCB.
4. Check for any loose or broken parts which may have occurred during transit.
5. Slide lid back and re-do the screws (do this before connecting power).
6. Read through the rest of this manual thoroughly and then carry out the installation.

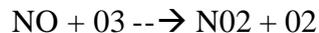
## 2. INTRODUCTION

This manual is for operational use only and is not a maintenance handbook.

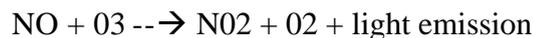
Maintenance handbooks are normal issue to distributors and service agents only, but can be purchased by customers if they wish to carry out their own servicing. All warranty will cease however, if a customer carries out his own servicing during the specified warranty period unless special arrangements have been made in writing. Servicing should be carried out every 2700 hours of use. This instruction book covers the operation of the Model 4000A.

### 2.01 The Principle of Chemiluminescence

The Chemiluminescent method used in the Model 4000A is based upon the Chemiluminescent gas phase reaction between Ozone and Nitric oxide to give Nitrogen dioxide and Oxygen:-



About 10% of the Nitrogen dioxide produced is in an electronically excited state, and the transition from this state to normal, as the molecules lose energy, gives rise to a light emission, varying in wavelength between 0.6 and 0.3 micrometers :-



The intensity of this emission is proportional to the mass flow rate of Nitrogen dioxide into the reaction chamber, and the emission is measured by means of a photomultiplier tube and associated electronics.

In order to ensure that the performance of the Signal non vacuum Model 4000A is similar to those of the vacuum based units special flow rates are set together with a sample dilution arrangement to reduce the CO<sub>2</sub> and H<sub>2</sub>O quenching effects normally associated with non vacuum instruments.

There are separate sections which form the Model 4000A NO<sub>x</sub> analyser. They are as follows:-

1. The detector/reaction chamber assembly.
2. The Ozoniser assembly.
3. The NO<sub>2</sub> to NO converter assembly.
4. The sample inlet.
5. The electronics section.
6. The flow control assembly.
7. Photomultiplier power supply.

## 2.02 The Detector/Reaction Chamber

Sample gas and ozone are passed at an accurately controlled flow rate to the reaction chamber. The reaction chamber is a precision machined part with specially designed mixing ports for maximum light emission and reflection.

An optical window is fitted to the reaction chamber which allows only light emission from the  $\text{NO} + \text{O}_3$  reactions (0.3 to 0.6 microns) and excludes any other interference which may emit light such as olefinic hydrocarbons

The reaction chamber is fitted to the photomultiplier tube housing whereby the reaction light emitted is detected by the photomultiplier tube.

Photomultiplier units are carefully selected for optimum sensitivity to the emitted light coming from the reaction but they are very sensitive to ambient temperature changes especially when the instrument is being used on the most sensitive range and also especially at higher ambient temperatures.

In order to eliminate this effect, Signal offers an optional cooled PM tube which houses the photomultiplier in a special housing which is fitted onto a thermo electric Peltier cooler.

The photomultiplier is connected to a light-tight box where the wire connections are made. The signal output cable carries the signal from the photomultiplier tube to the amplifier PCB and high voltage (approx. 1,000v DC) is connected to the photomultiplier tube.

## 2.03 The Ozoniser Assembly

The Nitric oxide (NO) + Ozone ( $\text{O}_3$ ) reaction, of course, needs a constant supply of Ozone. Incorporated in the Model 4000A therefore, is a very sophisticated Ozoniser device. The device is fully enclosed in an enclosure where dangerously high voltages exist.

The device consists of an ionization tube and a high voltage pulsed electronic circuit. Air or Oxygen is supplied (see 3.06 Oxygen /Air Connection to Ozoniser) to the analyser for the Ozoniser and some of the Oxygen is ionized to Ozone. The Ozone is then constantly controlled in both magnitude and flow rate to the reaction chamber.

It is essential that only qualified service engineer's deal with this unit as the high voltage inside, even when power is off could be deadly.

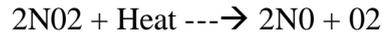
Normally only DRY air is needed to supply the Ozoniser as the analyser can be used for measurements up to the maximum range of 10,000 without any cut-off in reading. Oxygen is therefore not required and indeed, as the oxygen will flow through the analyser to the oil-filled vacuum pump, it would be advisable not to use Oxygen if possible.

**NEVER USE THE OZONISER WITHOUT GAS FLOW**

## 2.04 The NO<sub>2</sub> to NO Converter

As only Nitric oxide (NO) can be detected by chemiluminescence a converter is incorporated within the Signal 4000 Series analysers, to convert any Nitrogen dioxide to Nitric oxide.

Conversion of Nitrogen dioxide to Nitric oxide is carried out by passing the gas through a heated tube containing a carbon material. Part of the conversion is accomplished thermally:-



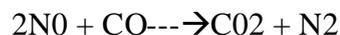
The remainder of the conversion is carried out by Carbon chemically reducing Nitrogen dioxide:-



When gas is passing through the converter the analyser is said to be in NO<sub>x</sub> mode, when gas is by-passing the converter, the instrument is said to be in NO mode.

It is important, when designing a converter, that operating conditions and materials are chosen to prevent undesirable side reactions occurring which may destroy the Nitric oxide in the sample gas.

Carbon monoxide is known to cause problems in certain types of converters due to the reaction between Nitric oxide and Carbon monoxide: -



The reaction takes place in the absence of Oxygen and is catalysed by stainless steel. Also, some higher temperature converters convert Ammonia to NO thus producing erroneous NO<sub>x</sub> reading.

The Model 4000A converter has been designed to overcome this undesirable side reaction by the use of a titanium furnace tube and a special Carbon material in place of stainless steel.

Following prolonged use of the Carbon converter it will be necessary to replace the Carbon material as this is slowly eroded by Oxygen and Nitrogen dioxide. Replacement of this Carbon will therefore be necessary after about six months of continuous use. Replacement is a simple job and a spare charge of Carbon is available in Spares Kit No. 102990.

(In order to comply with the EPA and European emissions regulations, you must be able to prove the efficiency of the NO<sub>2</sub> to NO converter. Signal manufacture a specially designed instrument called the NOXGEN 3 for this purpose.)

The converter temperature is controlled by a platinum resistance probe and pulse width modulation electronics circuit, located within the electronics module. The temperature of the converter may be altered by adjusting the potentiometer situated behind the left side door panel (see Operation section for details).

### **Model 4000A**

The two modes 'NOx' and NO, are selected on this model by pressing the buttons marked NOx and NO (see 4.01), on the right hand front panel. These switches operate a solenoid valve which diverts sample either through the converter or directly to the detector. NO<sub>2</sub> can be found by subtracting the NO reading from the NOx reading.

### **Model with Optional NO/NOx/NO<sub>2</sub>**

Operation of this instrument in 'NOx' and NO mode is the same as the standard unit but it has a third button marked 'NO<sub>2</sub>' (see 4.01). Pressing this switch causes the instrument to automatically cycle through the NOx and NO modes and also provide a subtraction for NO<sub>2</sub>. Three outputs are provided for NOx, NO and NO<sub>2</sub> and these are updated through sample and hold circuits every 30 seconds. The front panel display reads the channel which is selected, i.e. NO, NOx or NO<sub>2</sub>.

## **2.05 The Sample Inlet**

The Model 4000A is designed for use with dry cool samples (for measurements of raw hot dirty samples the Model 4000 vacuum based analyser will be needed).

The sample is brought into the instrument from an external pump (not installed within the instrument) and is connected to the rear panel tube connection.

An internal filter will remove any particulate in the sample (but heavy particulate laden samples may need a separate level of filtration prior to the sample entering the instrument).

The sample is bypassed through the fast flow bypass flow controller whilst a constant back pressure is controlled by the back pressure regulator. Following the bypass control the sample is split into two separate streams whereby flow control to the NO and NOx modes is controlled by two independent matched capillary tubes.

From these capillary tubes connection to the NOx converter and the NOx/NO balance control is made.

## **2.06 Electronics Section**

### **Electronic Arrangement**

The photomultiplier tube produces an extremely small current in response to light emitted from the reaction chamber. This current is very low, in the order of approximately 10<sup>-6</sup> amp and is amplified using the very latest in electronic circuitry.

A high impedance FET op amplifier is used and all the range change switching utilizes solid state FET analogue switches. The range change is facilitated with "up/down" push buttons on

the front panel. Autorange change is also incorporated as standard and the ranges can be selected remotely with three lines of TTL to the appropriate rear panel connection pins. (See operation instructions).

Due to the resolution of the 3 1/2 digit liquid crystal (LED) display, on the Model 4000A a multiplier is used when the 4000 ppm and 10,000 ppm ranges are selected. This is simplified by the illumination of the LED on the front panel marked "PPM x 1000". When this light is illuminated, simply multiply the digital display by 1000.

### **Data Acquisition and Computer Interfacing**

When using data acquisition, both a 0-7V and a 0-7 mA step change output signifies range selection. An output can be connected to a spare chart recorder pen and the step change records the range whilst another pen records the detector output.

A three-line BCD coded output is also available to signify range selection for more intelligent data acquisition systems.

For computerised data acquisition, you may wish to have the computer change range rather than rely on the auto range change function. This may be necessary if you are using an 8 Bit A-D converter and the ranges need only be changed twice to cover the entire range. A facility for this is incorporated where the computer sends a logic 0 TTL signal which is connected to the remote range function rear panel pin.

This acquires a remote range function and a further three lines of BCD TTL logic 0 will select the range required.

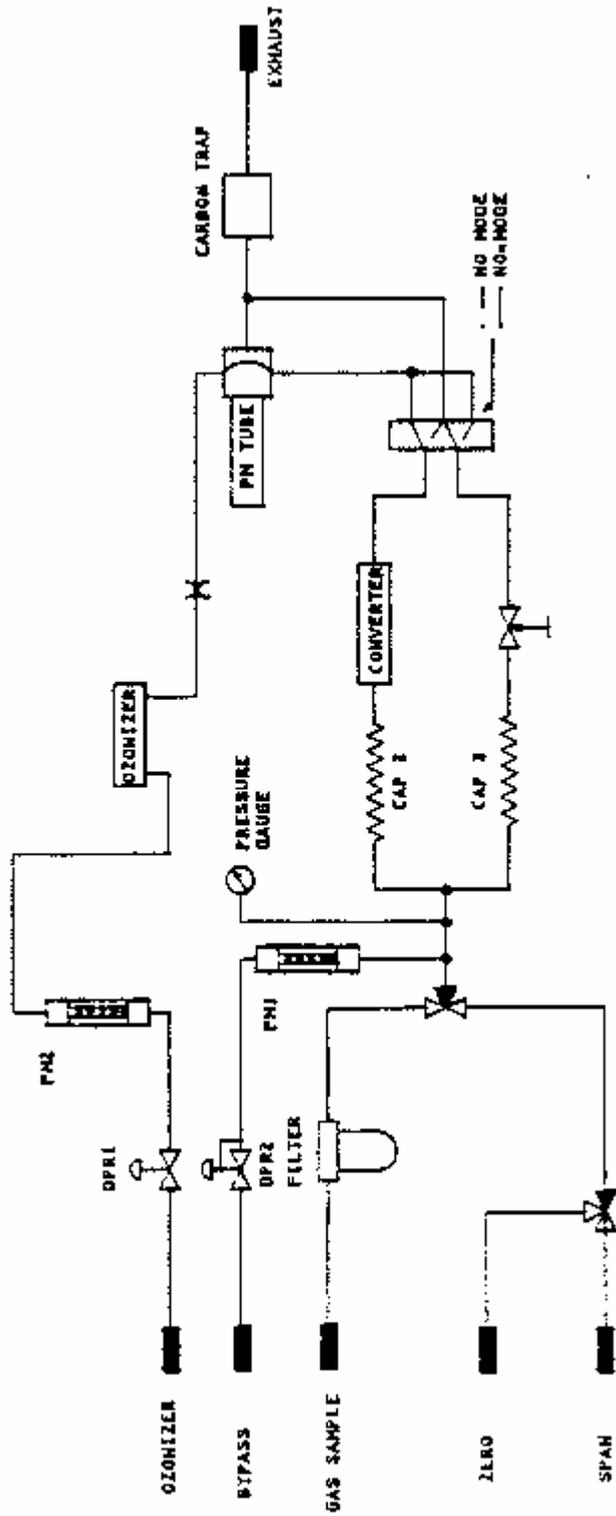
When the computer has acquired the remote range change function, the light illuminates in the "REM" front panel button

If for any reason the operator at the instrument wishes to take back local range switching, then pushing the "REM" button takes back to local, the range change function.

Therefore, the computer can always take remote range function prior to sending remote range change instructions. (For actual PIN and logic data, see the Operations section)

The zero gas/span gas/sample gas/selector valve is arranged so that a remote logic 0 signal to the rear 'D' connector will energise the solenoid valve situated inside the oven. Energising the solenoid will select the sample gas, de-energising it will select the calibration gases.

### 4000 A FLOW DIAGRAM



## 2.08 Photomultiplier Tube Voltage Power Supply Unit

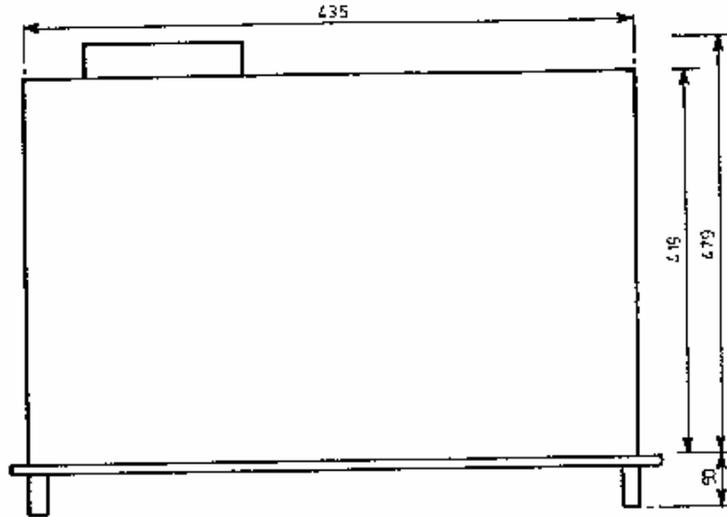
This unit provides high voltage of up to 1000V via a self-contained enclosed switched mode power supply unit. Each photomultiplier tube requires a maximum voltage with which it produces a photon to current output. The maximum voltage is preset to individual photomultiplier tubes, but below this maximum level, it can be adjusted for optimum use using the adjustment situated behind the left side door panel. (See Operation for details)

The voltage set to the PM tube is normally about 600V (6.00 on the PM tube voltage adjust dial). Normally, the gain of the detector signal is taken care of by the amplifier span control, if, however, a dirty sample gas slowly obscures the optical window and lowers the response signal, you may (at the expense of increased baseline noise) increase the PM tube voltage to add more gain to the system. When the noise becomes intolerable, or when you have no more gain left in the system, a strip down of the sample reaction cell and cleaning of the optics etc. will be needed.

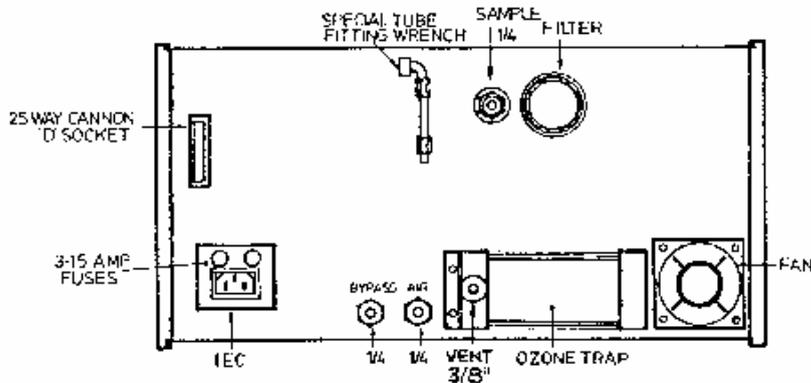
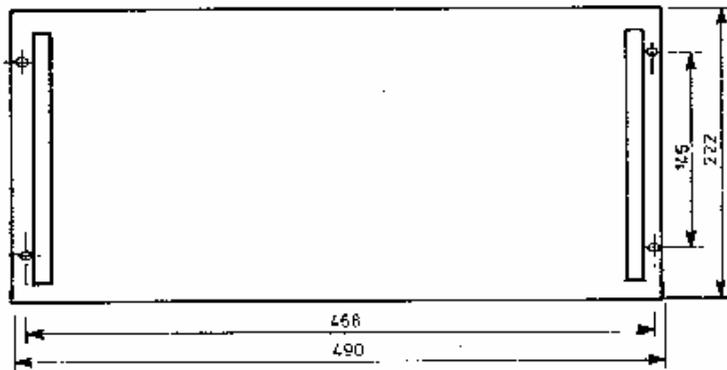
For cleaning fluid and spare gaskets etc. order Spares Kit No. 102980.

### 3. INSTALLATION

#### 3. Installation Overall Diagram



WEIGHT: 15Kg NET



### **3.01 General**

This section is very important and should be read carefully.

The instrument is designed for bench top use or 19" rack. It measures 220 mm high (5 units) 480 mm deep and 19" rack wide. There are ventilation louvers on the top and bottom panels and a fan on the rear panel which draws fresh air in through the bottom panel.

When used on a bench top there are feet on the bottom to prevent damage to work surfaces and the front feet have extension legs which allow the instrument to be tilted up for easier control. Normally, when used on a bench, no special precautions need to be taken, except to make sure that the ventilation louvers are not covered over, otherwise overheating will occur and this may damage the instrument. It is best to tilt the analyser on its front legs in order to allow better ventilation through the bottom.

Installation in a rack will firstly need the feet of the instrument to be removed. To do this, rest the instrument on the front handles, unscrew the four retaining screws on the bottom cover and slide cover backwards. When you have removed the bottom cover you can easily unscrew the four feet.

### **3.02 High Temperature Environment**

When this instrument is used in a 19" rack type console, great care will be needed to ensure that cool air is allowed to be drawn in through the bottom cover. A problem will exist if this analyser is placed above a heated oven or Hydrocarbon analyser, or other similar heated component. If it is unavoidable to place this instrument above a heated unit, at least 175 mm space should be left between, and then this space should also have a louvered front panel, preferably with fan air blowers being fitted.

The cooled PM tube housing will ensure that temperatures of up to 35 deg.C. will have little effect on the performance of the unit.

### **3.03 Sample Connection**

An optional sample pump can be fitted to the instrument but otherwise, a pressure of sample of 10 psi 0.75 bar will produce a flow of approximately 5 l/min going through the bypass flow control system.

### **3.04 Bypass Flow Output**

The bypass from this instrument will contain sample gas, it is therefore important that you connect a bypass vent tube to the rear panel in order to allow sample bypass to vent away from the instrument.

It is important that an adequate size of tubing is used in order to ensure that no further back pressure builds up in the tubing and causes the back pressure control system to malfunction. (i.e. 1/4 inch tube upto 5 metres or 3/8" tube upto 20 metres)

### 3.05 Detector Vent

The detector sample gas is exhausted through a rear panel mounted carbon trap which removes ozone and NO gas.

A suitable 3/8" tube will need to be connected to this and piped to a safe ventilation area. Ensure that no back pressure is allowed to build up.

### 3.06 Oxygen/Air Connection to Ozoniser

Air or Oxygen (see 4.04) is connected via a 1/4" compression fitting to the rear panel to the bulkhead fitting marked "Air/O2 Input".

The Ozoniser unit fitted to the 4000 Series instruments is powerful enough to allow measurement of NO<sub>x</sub> levels to 10,000 ppm with no spurious noise readings caused by NO<sub>x</sub> production in the Ozoniser.

The Ozoniser air supply flows right through the instrument to the oil filled vacuum pump. If oxygen is used, you may need to change the oil to a fluorocarbon-based lubricant in order to prevent the possibility of a flame occurring when the Oxygen meets the oil. For this reason air is preferred.

When using an air source you must be sure that the air used is dry. (Moisture quickly destroys Ozone and the analyser readings will vary) Use an air bottle preferably.

Set 30 psig (2 bar gauge) on the cylinder regulator and you will instantly see a flow on the front panel left side flow meter. This is fixed by a capillary and cannot be adjusted.

### 3.07 Calibration Gas Connections

There is a facility in the model 4000A to allow selection of both zero reference gas and span reference gas. The rear panel has an electrically activated valve to which you should connect these two gases. Looking at the instrument from the rear, the zero gas is the left hand side connection of the valve and the span the right hand side. The analyser is generally linear and calibrating at one point is generally sufficient. To meet with certain emission legislation work however, the linearity must be checked at several points in the analyser's range.

Signal produce the Signal model 821S for this purpose.

To select each calibration reference gas, use the selector switch located on the front recessed panel marked "zero span sample". Set the zero point first and then the span point.

### 3.08 Output of Remote Range Change

TTL level loads can be connected to the appropriate pins for remote range coded information:-

000 = Range 1  
001 = Range 2  
010 = Range 3  
011 = Range 4  
100 = Range 5  
101 = Range 6  
110 = Range 7  
111 = Range 8

0 = 0v  
1 = +5v

Also, for analogue recorders, both 0-7V DC and 0-7 mA output changes from zero to full scale in eight equal steps corresponding to the eight ranges.

0v = Range 1  
1v = Range 2  
2v = Range 3 etc.

### 3.09 Remote Control of Range Change

In order to facilitate remote control of range changing, connect a logic 0 level to the pin No. 6 on the rear panel D connector. This will take the control of the range change from the front panel. To select the desired range, connect three lines of logic in the following order:-

000 = Range 1  
001 = Range 2  
010 = Range 3  
011 = Range 4  
100 = Range 5  
101 = Range 6  
110 = Range 7  
111 = Range 8

0 = Open Circuit  
1 = Closed Circuit to Return Line Pin No. 4.

### 3.10 Remote Control of Sample/Calibration Gas Selector

The facility to select sample gas or calibration gas does not allow you to select the positions of 'Zero', 'Span' and 'Sample'. The remote logic level input only controls the selection of the sample. To operate this, firstly decide which gas you require selection of, i.e. Zero gas or Span gas, and select this manually using the gas selector switch (Q) behind the left side door panel. DO NOT leave this selector switch in the sample position as this will override the remote actuation. The application of a logic level to pin 1, or a short circuit link from pin 1 to pin 4, will select sample, the removal of this link will select calibration gas.

### 3.11 Power Input Connection

The Model 4000A is designed for use on 240V 50Hz or 110V 60Hz. This will be specified on the rear panel power input connection. The power input is protected by two 3.15A fuses. The power should be connected via a standard mains power plug fitted with an earth connection and a 5A fuse.

### 3.12 Input/Output Ports

1. Remote TTL logic 0.
2. Make no connection to this pin.
3. NO<sub>x</sub> output 0-10 mA - Model 4100 only.
4. Return line (ground).
5. Remote range input TTL logic line 1.
6. Remote range function enable TTL logic 0 input (see Data Acquisition section for explanation).
7. Remote range indication output TTL logic line 1.
8. NO output 0-by DC. (This is used on all models for NO detector output and Model 4000 for NO<sub>x</sub> when selected.)
9. NO analyser output as No.8 but 0-10 mA.
10. Remote converter temperature input. 0-500 deg.C can be set by applying a continuous voltage of 0-5V DC. 10 mV per deg.C for setting required temperature. (When remote temperature enable is actuated). Do not exceed 5V under any circumstances.
11. Remote temperature enable TTL logic 0 (or link to ground).
12. Make no connections to this pin.
13. NO<sub>2</sub> output 10 mA - Model 4100 only.
14. Make no connection to this pin.
15. Make no connection to this pin.
16. NO<sub>x</sub> output 0-by - Model 4000 only.
17. NO<sub>2</sub> output 0-by - Model 4000 only.
18. Remote range indication input TTL logic line 0.
19. Remote range indication input TTL logic line 2.
20. Remote range indication output TTL logic line 2.
21. Remote range indication output TTL logic line 0.
22. Remote range indication analogue 0-7V DC.
23. Remote range indication analogue 0-7 mA.
24. Make no connection to this pin.
25. Make no connection to this pin.

**NOTE:**

**All logic TTL levels may use a short circuit to ground (if TTL levels are not available) connected to return line pin No. 4.**

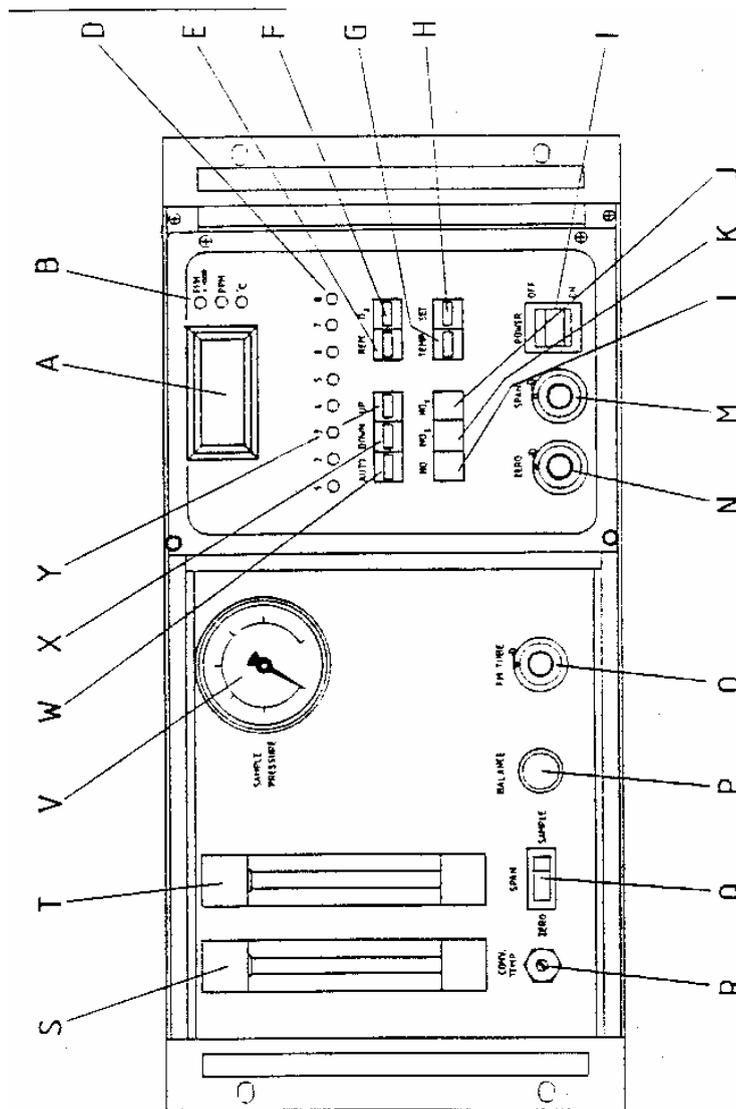
All TTL logic 1 outputs will drive a transistorised relay (prior to load) connected to return line pin No. 4.

**4. OPERATION****4.01 Front Panel Controls**

- A. Digital display directly in PPM (or PPM times 1000 when "PPM x 1000" light is on).
- B. Display x 1000 multiplier light (when this is on, multiply the reading on the display by 1000).
- C.
- D. Range selected indicator. 1=4 ppm, 2=10 ppm, 3=40 ppm, 4=100 ppm, 5=400 ppm, 6=1000 ppm, 7=4000 ppm, 8=10,000 ppm.
- E. Local/Remote Control (see Data Acquisition section for explanation).
- F. Ozone On/Off control. (Switch off the Ozone a minute before switching off the analyser to allow the Ozone to flush out. Also do not use without gas flow).
- G. Converter actual temperature indicate selector.
- H. Converter temperature set point selector.
- I. Power on/off switch.
- J. NO<sub>x</sub> mode switch.
- K. NO<sub>2</sub> mode switch. (This optional feature automatically sets off the sample and hold timer and displays the subtracted NO<sub>x</sub>-NO = NO<sub>2</sub> reading). Model 4100 only.
- L. No mode switch.
- M. Span calibration control (sets the gas).
- N. Zero calibration control (sets the zero).
- O. Photomultiplier adjustment (reads directly in volts, i.e. 6.00 = 600 volts.)
- P. Balance valve. (Used to set NO and NO<sub>x</sub> reading the same)
- Q. Zero/span/sample gas selector switch.

- R. Converter temperature adjustment. (Press button H marked "set" to see the set temperature)
- S. Ozoniser air flow meter.
- T. Bypass flow meter (Do not exceed 5)
- V. Sample vacuum gauge (should read approx. 4 psig).
- W. Auto range change switch.
- X. Range downward button.
- Y. Range upward switch button.

**4.02 Front Panel Control Diagram**



### 4.03 Setting up Instructions

1. Undo sample filter in rear panel and check that a fresh filter is fitted.
2. Check that the installation procedure has been carried out in accordance with this instruction book.
3. Switch on mains power and wait one hour for the instrument to warm up. You can observe the temperature of the N<sub>2</sub> to NO converter by using the front panel temperature indicator marked "Temp". When this is about 350-400 deg.C the instrument will be ready for use, but for low level measurements on the lowest range the analyser will need temperature equalization for a longer period before stability will be reached.
4. Set 30 psi (2 bar gauge) on the Ozoniser air supply. The flow meter should register at the flow tube.
5. Switch on Ozoniser using switch on front panel marked "O3".
6. Select the NO<sub>x</sub> channel.
7. Switch on sample pump. Note this may be fitted separately to the instrument.
8. Select zero gas using the selector switch located on the left side recessed panel.
9. Set the range switch to the range suitable for the span calibration gas.
10. Using the zero dial, set the display to read 0.00 (ensure you have selected the NO<sub>x</sub> channel).
11. Select span gas using the gas selector switch.
12. Adjust the display to read the concentration which is in the span gas cylinder.
13. Select the NO channel.
14. Using the balance valve, carefully adjust the display until the reading is the same as with the NO<sub>x</sub> channel.

**NOTES:**

- A. After setting this balance valve, you may find a problem on zero gas in getting the NO and NOx channels to read the same, particularly when using air as the zero gas. This is largely due to N<sub>2</sub>O contamination in the gas which is converted to read NOx in the NOx channel, but not in the NO channel. Using Nitrogen will reduce this effect.
- B. The gain in the detector amplifier is set by both the photomultiplier voltage as well as the amplifier span control. Normally you should find plenty of gain in the span control but as the optics in the sample reaction cell become opaque (caused by the dirty sample gas) you will need to increase this span setting. If you finally run out of gain in this setting, the photomultiplier voltage adjust will allow you to increase further gain (at the expense, of course, of increased baseline noise). When the baseline noise is intolerable, or when you finally run out of gain with both controls, you will need to have the detector stripped down and cleaned. Call your local Signal service dealer or the Signal factory for this service.

**Setting Up Instruction for Model with NO/NOx/N<sub>2</sub>O option**

These models operate in exactly the same way as described before in paragraph 4.02, page 25, but have additionally, an automatic control which operates a cycle between NO and NOx, with N<sub>2</sub>O being automatically subtracted.

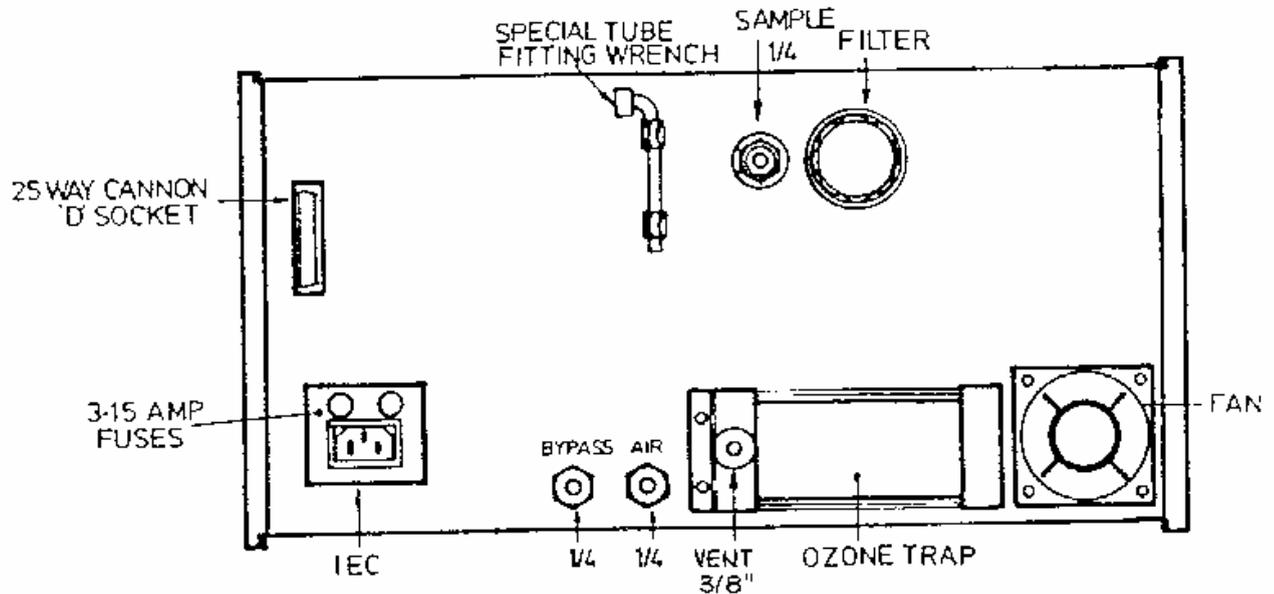
To operate the choice of NOx, NO only or N<sub>2</sub>O, the following procedure must be followed:-

- A. **To measure NOx**, simply press the button marked NOx (J) on the front panel. Operating this button makes the sample gas flow through the N<sub>2</sub>O to NO converter before going to the detector. The NOx analogue output is produced continuously with the front panel reading but the NO and N<sub>2</sub>O outputs will not be valid because the sample and hold circuits will soon fade away.
- B. **To measure NO only**, simply press the button marked N<sub>2</sub>O (L) on the front panel. Operating this button makes the sample gas flow directly to the detector where NO only is measured. The NO analogue output is produced continuously with the front panel reading but the NOx and N<sub>2</sub>O outputs will not be valid because the sample and hold circuits will soon fade away.
- C. **To measure N<sub>2</sub>O**, simply press the button marked N<sub>2</sub>O (K) on the front panel. Operating this button makes the sample gas automatically cycle through the use of an electronic control, 30 seconds on NOx and 30 seconds on NO only. The sample and hold circuits are each updated every cycle and the difference between NOx and NO is automatically obtained and this N<sub>2</sub>O signal is displayed on the front panel as well as fed to the N<sub>2</sub>O analogue output.

**NOTE:**

The only peculiarity with the set up is that the Autorange change is only operated from the NO signal, therefore when NOx is selected, the automatic range change cannot be used because the NO signal fades away and is not updated.

#### 4.04 Diagram of Rear Panel



#### 4.05 Shutting Down Procedure

The Signal model 4000 is generally designed to have no real idiosyncrasies when shutting down.

It is good practice, however, to switch off the Ozoniser for a few minutes prior to switching the analyser off, (this allows the Ozone to flush out).

Also, you should remember to switch off the Ozoniser air cylinder.

**Never use the Ozoniser without gas flow.**

#### 4.06 Service Instructions

In order to maintain this instrument in a fully operational manner and to keep it working in accordance with the published specifications, the instrument must be fully serviced every 2700 hours of use. This is 4 months at continuous operation.

Servicing must be carried out by the factory or alternatively an authorised service dealer. A service maintenance workshop manual is available for those customers who wish to carry out their own servicing.

If 2700 hours elapse during the first 12 months, warranty for this instrument is only applicable if the instrument has been serviced by the factory or an authorised service dealer or if prior arrangement in writing has been made with the user for customers wishing to carry out their own routine servicing. The following parts should be ordered with the service workshop manual, and a list of special instruments and tools is also available upon request.

#### 4.07 Spare Parts

##### **Spare Parts Kit - Suitable for one year's operation (Part No. 102990) comprises:-**

Vacuum pump oil (only for vacuum units)  
Ozone trap refill  
Diaphragm pump kit  
Converter refill  
Box of 10 filters  
Fuses kit  
Spare tube fitting nuts and ferrules.

##### **Spare Parts Kit - Additional (Part No. 102980) comprises:-**

102990 spares kit as above  
Ozoniser tube  
Ozoniser tube 'O' rings  
Reaction chamber gasket kit  
Optical window cleaning fluid  
Filter 'O' ring  
Solenoid valve repair kit  
Spare vacuum hose connectors (2)  
Spare vacuum hose seals (2)  
Spare pair sample capillaries.

#### 4.08 Routine Services

The following routine servicing will be necessary.

1. The carbon converter will chemically decay dependent upon the amount of N<sub>2</sub> present and the time it is used, i.e. Nitric acid plants with around 1000 ppm N<sub>2</sub> can expect about 2-3 months use. Normal combustion emission applications should expect about a years use. A very special vitreous carbon is used and must be replaced by genuine Signal replacement Carbon. Spare Carbon refills are available in spare kit no. 102990. These are available separately, part no. CHEM/001.
2. Occasionally it has been known for the matched sample capillaries to become blocked, particularly if the pumps are left running with the analyser oven switched off. This causes condensation to occur. To replace these capillaries, use the matched pair available in spare kit no. 1029980. These are available separately, part no. MC/837

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## 5. CONVERTER EFFICIENCY

### 5.01 NOx Generator Method

This method is recommended by various legislative bodies such as the EPA. The 'NOXGEN', a ready built NOx Generator is available from Signal. A simple schematic of a NOx Generator or 'Converter Efficiency Detector' is shown in Fig. 5.02.

The principle of operation is based upon the fact that Ozone very rapidly oxidises Nitric oxide to Nitrogen dioxide, whereas Oxygen oxidises Nitric oxide very slowly. By blending a small Oxygen flow with a rapid Nitric oxide/Nitrogen flow, a reduction in concentration of Nitric oxide will be seen, purely due to a dilution effect. Virtually no Nitrogen dioxide will be formed under these conditions. On switching on an Ozoniser in the Oxygen line, Ozone will be quickly generated thereby giving rise to Nitrogen dioxide.

**Procedure:** (Owners of the 'Noxgen' should also refer to the 'Noxgen' manual).

1. Attach the Nitric oxide/Nitrogen supply to the 'Noxgen' and connect the gas outlet to the sample inlet of the analyser. Attach an oxygen supply to the Noxgen. Air may be used in place of Oxygen to facilitate better control if low concentrations of Nitrogen dioxide are required.
2. With the Noxgen Ozoniser switched off, place the 4000 analyser in NO mode and close the valve in the Oxygen line. Open the valve controlling the Nitric oxide/Nitrogen supply until sufficient flow and stable readings are obtained. Zero and span the analyser to indicate the value of the Nitric oxide concentration being used. Record this concentration (A).
3. Open the Oxygen valve and adjust the Oxygen flow until sufficient Oxygen is flowing to lower the Nitric oxide concentration (A) by 10%. Record this concentration (B).
4. Turn on Ozoniser in Noxgen and adjust the Ozone generation rate until the Nitric oxide concentration (B) is reduced to about 20% of (A). Nitrogen dioxide is now being formed from the Nitric oxide/Ozone reaction. There must always be at least 10% un-reacted Nitric oxide at this point. Record this concentration (C).
5. When a stable reading has been obtained from 4, place the 4000 analyser in NOx mode. The analyser will now indicate the total NOx concentration. Record this concentration (D).
6. Turn off the Ozoniser in the Noxgen and allow the analyser to stabilize. The mixture Nitric oxide plus Oxygen is still passing through the converter. This reading is the total NOx concentration of the diluted Nitric oxide span gas used at step 3. Record this concentration (E).
7. Close the Oxygen valve in the Noxgen. The Nitric oxide concentration should be equal to or greater than the reading A, indicating whether the Nitric oxide contains any Nitrogen dioxide.

**Calculation:**

$$\text{Method 1} \quad \% \text{ Efficiency} = \frac{(D - C)}{(E - C)} \times 100$$

$$\text{Method 2} \quad \% \text{ Efficiency} = \frac{(1 + (D - E))}{(B - C)} \times 100$$

This method (2) does not depend on the amount of Nitrogen dioxide in the span gas, nor on the equivalence of flows in NO and NO<sub>x</sub> modes. However, to be consistent with good operating practice, flows should be nominally the same and the Nitrogen dioxide concentration 5% or less of the Nitric oxide concentration.

**Typical Example**

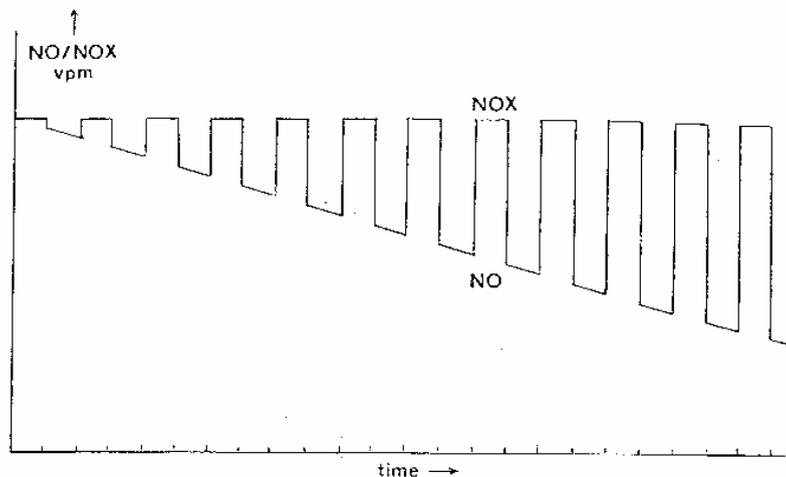
$$\begin{aligned} A &= 110 \\ B &= 100 \\ C &= 25 \\ D &= 97 \\ E &= 102 \end{aligned}$$

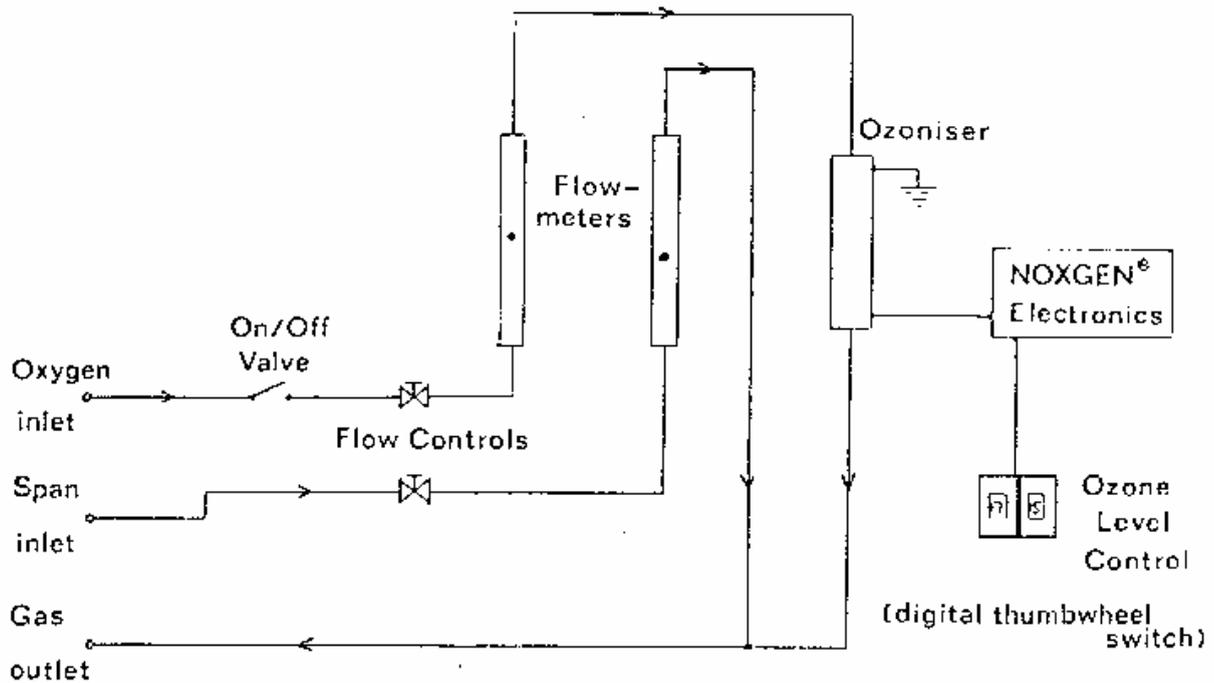
$$\text{Method 1} \quad \% \text{ Efficiency} = \frac{(97 - 25)}{(102 - 25)} \times 100 = 93.5\%$$

$$\text{Method 2} \quad \% \text{ Efficiency} = \frac{(97 - 102)}{(100 - 25)} \times 100 = 93.3\%$$

**NOTE:**

If the value of E is the same as B, i.e. no Nitrogen dioxide in span gas, or mismatch of flows in analyser, then Method 1 and Method 2 yield the same numerical result.

**5.02 Converter Efficiency Detector Diagram**



### Converter Efficiency Detector

#### 5.03 Linearity

This can be established using the Signal 821S gas divider available from your local sales office.

The span and zero gases are connected to the Signal 821S, and eleven points, including zero and full scale, are available by selection from the 821S. These points are at 10% intervals and allow linearity to be measured to within 0.5% of point.

The Signal 821P5S Pre-diluter can also be used to provide the span gas connection for routine work. Full details are available from Signal Instruments or your local sales office.

