



SERVOPRO Plasma (k2001) Trace N₂ Analyser

User Manual

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1.0 CONCERNING THIS MANUAL

This analyzer was designed for simple use, according to the "Plug and play" principle and so was this manual. For the benefits of clarity, all electronics, software and physical details not necessary for the operation of the unit are omitted. It is the way we want it.

We understand that you want to put your new analyzer to use as soon as possible. To achieve this goal, take the time to read **all this manual in its entirety**. Every section is based on the assumption that you have read and understood the preceding one, and every section has important comments for the user. This analyzer is very simple to install and to use; also, it is maintenance-free. No special technical knowledge is required to operate the unit.

We hope that you will enjoy working with the SERVOPRO PLASMA Trace Nitrogen Analyzer. In the spirit of progress and continuous improvement, we would appreciate any comments you may have, negative or positive - as long they are constructive.

Servomex believes that the information in this manual is accurate. The document has been carefully reviewed for technical accuracy. If there should be any error, Servomex reserves the right to make changes to subsequent editions of this document without prior notice to holders of this edition. The reader should contact Servomex if errors are suspected. In no event shall Servomex be liable for any damages arising out of or related to this document or the information contained in it.

2.0 CAUTIONS AND WARNINGS

2.1 Symbols description



2.2 Caution

Improper installation, operation or service of this analyzer may cause damage to the analyzer and void the manufacturer's warranty.

2.3 Electrical shock hazard

Do not operate unless the cabinet is securely closed. Servicing this instrument implies possible exposure to shock hazard level voltages which can cause death or serious injury.

For both safety and proper performance, this instrument <u>must</u> be connected to a properly grounded three-wire source of electrical power.

Both alarm switching relay contacts and digital output contacts wired to a separate power source must be disconnected before servicing.

Tampering or unauthorized substitution of components may adversely affect the safety of this product. Use only factory-approved components for repair.

2.4 Possible explosion hazard

Never introduce other gases than argon in this analyzer. If explosive, flammable or corrosive gases or mixtures are allowed to flow in the analyzer, fire or explosion may result. This analyzer is not designed to be used in hazardous areas.

User Manual

This analyzer must be installed in laboratory environments: moisture- and vibration-free, with stable temperatures.

2.5 General precautions for handling and storing high pressure cylinders

This analyzer is frequently applied to verify the contents of high-pressure cylinder gases. Mishandling of gas cylinders could result in death, serious injury or property damages. Handle gas cylinders with extreme care. Refer to general precautions for handling and storing high-pressure cylinders. Here are some precautions from the <u>COMPRESSED GAS ASSOCIATION'S</u> <u>HANDBOOK</u>.

- 1. Never drop cylinders or permit them to strike each other violently.
- 2. Cylinders may be stored in the open, but in such cases, should be protected against extreme weather. To prevent rusting, keep away from the dampness of the ground.
- 3. The valve protection cap should be left on each cylinder until it has been secured against a wall or a bench, or placed in a cylinder stand until it is ready to be used.
- 4. Avoid dragging, rolling, or sliding cylinders, even for a short distance; they should be moved by using a suitable hand-truck.
- 5. Never tamper with safety devices in valves or cylinders.
- 6. Do not store full and empty cylinders together. Serious suck back can occur when an empty cylinder is attached to a pressurized system.
- 7. No part of a cylinder should be subjected to a temperature higher than 125 °F (52 °C). A flame should never be permitted to come in contact with any part of a compressed gas cylinder.
- 8. Do not place cylinders where they may become part of an electric circuit. When electric arc welding, precautions must be taken to prevent striking an arc against the cylinder.

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Edited from selected paragraphs of the Compressed Gas Association's

"Handbook of Compressed Gases" published in 1981.

Compressed Gas Association

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2.6 Gas hazard

Argon and helium are member of the rare gas family which consist of helium, argon, krypton, xenon or neon.

All of these gases are monoatomic and are characterized by their extreme chemical inactivity. They are colorless, odorless, tasteless and NON TOXIC.

However, these gases can act as a simple asphyxiate by displacing the necessary amount of oxygen to support life. Proper ventilation must be done to provide safe area.

The nitrogen is a diatomic molecule and is colorless, odorless and non toxic. However, nitrogen can also act as a simple asphyxiate by displacing the necessary amount of oxygen to support life. Proper ventilation must be done to provide safe area.

2.7 General safety instructions

To avoid the risk of electric shock, do not remove the casing or open the back. There are no user serviceable parts inside. Leave servicing to the experts!

To prevent fire or the risk of electric shock, keep this unit out of the rain and away from moisture. The lightning symbol inside an equilateral triangle means that there are live, uninsulated parts inside this unit that may give you a dangerous electric shock if touched.

- 1. Instructions: Read all the safety instructions and all the operation instructions thoroughly before using the unit for the first time. Keep these safety instructions and operating instructions somewhere safe in case you need to refer to them again in the future.
- 2. Safety warnings: In your own interest pay heed to all the safety warnings on the unit and in the operating instructions. Follow the instructions on operation and use of the unit in every respect.
- 3. Water and moisture: Never use the unit near water, for example near a bath, a wash basin, a sink a washing machine, in a damp cellar or near a swimming pool.
- 4. Ventilation: Wherever you put the unit, always ensure there is sufficient ventilation. Never put the unit on a bed, for example, or a sofa. a carpet or similar surface that might block the vents. Make sure there is proper ventilation to avoid overheating.
- 5. Effects of heat: Do not put the unit anywhere near sources of heat, such as radiators, hot-air shafts, ovens, etc.
- 6. Power source: Connect the unit only to the power source indicated on the operating instructions or on the unit.
- 7. Protecting the power cord: Run the power cord so that no one can step on it and nothing can rest on or against it. The power cord is particularly at risk in the area of the plug, the socket and where it comes out of the unit.
- 8. Cleaning: Follow the manufacturer's recommendations for cleaning the unit.
- 9. Unit not in use: If you are not going to use the unit for some time, remove the plug from the socket.
- 10. Foreign bodies: Take great care to ensure that no liquids or other foreign bodies can find their way inside the unit through the openings in the casing.
- 11. Repair in the event of damage: The unit should only be repaired by qualified personnel. Never try to do more in the way of maintenance to your unit than the operating instructions allow. Beyond that, always consult an expert for repair work.

2.0 CAUTIONS AND WARNINGS

RETURNING A PRODUCT FOR REPAIR

Upon determining that repair services are required, the customer must :

- 1. Obtain an RMA (Return Material Authorization) number.
- 2. Supply a purchase order number or other acceptable information.
- 3. Include a list of problems encountered along with your name, address and telephone, and RMA number.
- 4. Ship the analyzer in its original packaging or equivalent. Failure to properly package the analyzer will automatically void the warranty.
- 5. Every gas connection must be capped with appropriate metal caps. Failure to do so will automatically void the warranty.
- 6. Write RMA number on the outside of the box.
- 7. Use a Servomex approved carrier. The delivery must be sent to the regional Servomex Repair Centre. Servomex will not accept airport to airport delivery.
- 8. Servomex will not cover transport fees.

Other conditions and limitations may apply to international shipments.

NOTE: Seller applies to SERVOMEX and/or authorized distributors.

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| 4.0 | SPECIFICATIONS |) |
|-----|-----------------------|---|
|-----|-----------------------|---|

| Detector type: | Plasma Emission Detector (PED). Material: Quartz, single element, vacuum tight to 69 kPa (10 Psig) |
|--------------------------------|---|
| Range: | X1: $0 - 1$ ppm display resolution to 10 ppb X10: $0 - 10$ ppm display resolution to .1 ppm X100: $0 - 100$ ppm display resolution to 1 ppm |
| Accuracy: | $\pm 1\%$ full scale ⁽¹⁾ |
| Drift: | $\pm 1\%$ over 24 hours |
| Noise: | ≤ 2 ppb |
| Sample flow requirements: | 25 ml min^{-1} to 150 ml min^{-1} |
| Recommended calibration | |
| gas: | |
| for $0 - 1$ ppm range: | Zero: purified gas with a heated purifier (ex: GP-200)Span: 0.7 to 0.9 ppm |
| for 0-10, 0-100 ppm range: | - 20 % for zero and 80% of full scale you will be using. |
| Flow accuracy: | 0 to 200 ml min ⁻¹ \pm 1% full scale |
| Operating pressure: | Sample pressure: 27.6 to 55.2 kPa gauge (max) |
| | (5 to 10 Psig(max)) |
| Operating temperature: | $+5^{\circ}C$ to $+40^{\circ}C$ |
| Sample and vent connections: | 1/8" Swagelok tube fittings |
| Supply: | Electrical Supply: |
| | Voltage: 100 to 120 Vac or 220 to 240 $Vac^{(2)}$ |
| | Frequency: 50 to 60 Hz |
| | Maximum power consumption: |
| _ | Power: 45 W |
| Fuses: | Two fuses 1 amp / 250 V, Littelfuse type "F", fact-acting |
| - | miniature fuses 5 x 20 mm |
| Response time: | - X10 and X100: 20 sec. for 90% of a step change at $75 \text{ m} \text{ step}$ |
| | 75 ml min^{-1} |
| | - X1: 40 sec. for 90% of a step change $/5$ ml min ⁻ |
| Dimensions (Height x width x | $133 \text{ mm} \times 483 \text{ mm} \times 457 \text{ mm} (5.25 \text{ ins} \times 19 \text{ ins} \times 18 \text{ ins})$ |
| Depth): Weighte | $15 \ln (22 \text{lbs})$ |
| Weight: Operating altitude: | 13 kg (33 108) |
| Operating annual: | 2000 III (IIIAX) 0 to 05% PH non-condensing |
| range. | 0 to 35 /0 Km non-condensing |
| range. Ingress protection. | IP20 |
| ingi coo pi otection. | II 20 |

(1) The accuracy may be decreased by an additional 4% full scale at some frequencies under the influence of radiated RF fields specified for industrial environments.

(2) The analyzer is supplied configured for operation with one of these voltage ranges. You must specify the voltage range when you order the analyzer.

- Fully microprocessor-controlled, with 4 x 40 LCD display
- Self-diagnostic system software
- Auto-zero, auto-span calibration system (optional). Provides maintenance-free system
- Digital output: 4 digital outputs: 3 are for range indication and1 for system status monitoring Relay contact rating: 30 Vrms, 42.4 V peak or 60 Vdc
- Alarm output: 2 digital outputs for a specified value chosen by the user Relay contact rating: 30 Vrms, 42.4 V peak or 60 Vdc
- 4-20 mA isolated output Maximum load impedance: 600 Ω at 20 mA DC Isolation: 750 VDC or AC peak maximum
- Serial port: RS-232 (optional)
- Custom options: contact factory
- CE and Y2000 compliant
- CSA approval on demand
- The analyzer is rated in accordance to Over Voltage Category II, Pollution Degree 2
- Ingress protection: IP20.

The SERVOPRO PLASMA analyzer is only suitable to measure nitrogen in helium and argon.

5.0 DESCRIPTION

5.1 General description

The SERVOPRO PLASMA Trace Nitrogen in argon Analyzer is an instrument for measuring 0 to 100 ppm nitrogen in argon. This analyzer is designed to work with pure gas. Avoid introduction of air into the sampling system. If this happens, let clean gas flow in it: the unit will recover and normally will not require recalibration. The overall functions of the analyzer are controlled by the microprocessor boards. Different functions are accessed by selecting the proper menu items on the display with the help of the membrane keypad.

PLEASE READ THE ENTIRE MANUAL BEFORE OPERATING THE ANALYZER.

You will find important comments in all sections of this manual.

5.2 Sampling

The argon sample is introduced through a 1/8" tube Swagelok fitting at the rear of the cabinet. The sample is directed to a particle filter and goes through a thermofluidic flow control valve. From there, the argon sample gas is introduced into the plasma cell. The sample coming out from the cell passes through an electronic mass flow transducer before it is vented into the atmosphere.

The mass flow transducer gives flow information to the microprocessor system. This information is used to control the intensity of signal to be sent to the thermofluidic flow control valve. The microprocessor executes a P.I.D. algorithm (proportional - integral - derivative) to maintain the flow at the set point value. <u>Never</u> block the vent, or permanent damage will result to flow transducer. Leave vent at atmospheric pressure.

5.3 Power supply

The utility power supply must be stable and transient-free for a reliable operation of the analyzer. The system has two fuses.

The analyzer has one power supply module delivering D.C. regulated voltage to the signal conditioning module, the microprocessor system display and the flow control module. If you have a built-in option, the power supply module may also be connected to it, depending on the option installed.



5.4 Analysis section

The sample gas, which at atmospheric pressure, flows through a proprietary design; a pure quartz cell is submitted to a high-frequency, high-intensity electromagnetic field.

The SERVOPRO PLASMA Analyzer is based on a spectroscopic emission cell, which in itself is not a new technique. On the other hand, the characteristics which make this system stable and selective are the frequency, the intensity, the regulation, as well as the coupling technique and focusing (stabilizing) electrodes used to keep the plasma stable.

Under these conditions, the plasma becomes the center of a luminous phenomenon (electroluminescence). In fact, the plasma is electromagnetically induced. Plasma is a collection of charged particles; In this case, the plasma consists of a stream of sample gas. This process being an emission technique, it is very useful for quantitative analysis. This is not a new technique. These recent advances make the development of new instruments based on plasma technology easier; this without the cost normally associated with this category of analytical instruments.

Once the sample gas is ionized (charged), many spectral lines are emitted.

Here, we think a few words regarding the technique used to create the plasma are necessary. There are many ways of producing light from a gas stream for analytical purposes. The electroluminescence phenomenon includes luminescence from all kinds of electrical discharges, such as sparks, arcs or tubes of different kinds, operating on direct or alternating current of low or high frequency. Recently, some experiments were conducted in the microwave range by surface-wave-induced plasma.

Excitation, in these cases, results mostly from electron or ion collisions; that is, the kinetic energy of electrons or ions accelerated in an electric field in which the atoms or molecules of a gas are subjected that causes the emission of light.

By any of the above mentioned methods, a characteristic emission spectra can be obtained for the plasma gas and each substance in it. The emission usually varies for a given substance, depending on the mode of excitation.

We developed our plasma generator and plasma cell in such a way as to minimize the heat generation inside the cell to get a clear spectral line for the substance of interest, i.e. nitrogen.

Once the intense line is identified, we must use a system to filter it out. In our case, instead of using a defraction grating, prism or tunable filter, we use an custom made optical filter with a special coating that lessens background noise considerably and reduces spectral interference. This filter shows an exceptional blocking range. The filter's main characteristic is its great stability: temperature and humidity and time will not affect its performance. The filter is coupled with a special lens that focuses the optical beam.

The resulting light is directed to a special photodiode, where an electrical current is generated. This current is proportional to the nitrogen level in the argon stream.

By regulating the power of the plasma and having close control of plasma position with the focusing electrodes; we can optimize the sensitivity for a particular spectral ray.

5.5 Electronics

The current from the photodiode is directed to a low bias current switched integrator, followed by a precision floating-point programmable gain amplifier, and ending with an analog to digital converter. This data is read by the microprocessor circuit, where a digital signal processing algorithm is executed to determine the equivalent parts per million value.

The microprocessor board converts this value back into an analog one; then an optional 4-20 mA isolator transmits it to a remote PLC, computer or recorder.

The microprocessor board also manages operator interventions and validates data input from the keypad. The data is displayed on a LCD of 4 lines x 40 characters. Also, the microprocessor board controls flow with a P.I.D. algorithm.

The microprocessor board uses a multitasking kernel to control the operation of the analyzer.

On option, the analyzer may be equipped with digital output, for remote range indication. Also, a serial port may be installed with the appropriate protocol for remote data transmission.

5.6 Description of controls and I/O ports



6.0 INSTALLATION

6.1 Electrical

Connect the line cord to the proper line voltage according to the model you have (i.e. Voltage: 100 to 120 Vac or 220 to 240 Vac; Frequency: 50 to 60 Hz).

For optimum unit performance this voltage must be stable, transient-free and have a stable frequency. Also, the unit must be properly grounded.

Check the earth (ground) continuity between your electrical supply outlet earth (ground) and the functional earth (ground) terminal on the rear of the analyzer.

6.2 Gas circuit

For all gas line connections (including calibration gas and sample gas), we recommend the use of 1/8" stainless steel tubing in full length, with no fittings. You must avoid pipe thread connections: they are usually sealed with Teflon tape and some particles can get into the lines. By using compression-type tube fittings, the venturi aspiration of outside contaminants is virtually eliminated.

If you suspect occasional contamination with air, it is a good idea to install a small molecular sieve trap (50 cc to 100 cc) at the sample inlet. This minimizes pollution with moisture. Moisture or air will not damage the cell, but will give noise and instability. Again, this analyzer is designed to operate with clean gas.

Tube quality is often overlooked. For a 1/8" O.D. tube, use a minimum wall thickness of .028". The tube must be purchased to meet ASTM69 specifications. Inferior quality tubes may have irregularities on their circumference, causing inadequate sealing with compression tube fittings.

Install a by-pass rotometer on your sample line. Install the rotometer near the analyzer and close to the stream selection valve. This increases gas velocity and allows a faster purging of the sample line, before selection. You will also get a faster response time this way. Also adjust your sample line pressure to a value which will bring proper flow into the system: higher sample line pressure results in longer response time.

All lines must be cleaned and purged to remove any trace of moisture or particles. Particles can damage your stream selection valves. Also, particles in the inlet filter will trap moisture.

After all these precautions are taken, the pressure regulator on the calibration cylinder must also have a S.S. diaphragm and compression tube fitting on the low pressure side, and CGA fitting on the high pressure side. A regulator with discharge pressure adjustable from 0 to 207 kPa (30 PSI) is recommended. Apply equal pressure for the zero, the span gas, and the sample gas.

Regulator purging is critical. When you connect the regulator to a cylinder, it is full of air (790 000 ppm of N_2). If you open the valve on the cylinder and leave it open even for a short period of time, the air inside the regulator will diffuse into the cylinder. It is impossible to know exactly how much nitrogen you may have in your cylinder. Naturally, because this cylinder has no more correspondence to its analyzed certificate, it must not be used as a reference.

Very often (too often!!), problems related to this type of analyzer (and this applies also for trace oxygen analyzers) occur from an inefficient purge of the pressure regulator, resulting in a bad calibration gas. Regulator purging is an operation that is not always given the attention it deserves when using calibration gases.

The end user should understand that what happens to the gas between the cylinder and its end use is controlled by the quality of connecting lines and the efficiency of the purging procedures.

Please refer to appendix 1 for more details about sampling system and regulator purging. This is what we recommend and this is what we approve. Our performances are guaranteed if installation is done as per drawing CAI-4003. Nothing else will be better than this.

The vent of the analyzer must be at atmospheric pressure. If you suspect vent blocking, install a back-pressure regulator or a relief valve with a pressure setting of 7 kPa (1 PSI). This will prevent over pressurization of the system. Remember, the analyzer will perform better if the vent is working at atmospheric pressure. You may use 1/8" S.S. tubing for a length of 10 feet. At this point use 1/4" - or bigger - tubing to vent the analyzer.

6.3 Analyzer cabinet installation

This unit is designed for a rack mounted cabinet. If you install it in a different type of cabinet without side support bracket, **you must install a metal bracket to support the rear** side of the cabinet.

Like every analytical equipment, it <u>must</u> be installed properly. The unit should not be installed in direct sunlight and exposed to any vibrations. The ideal room temperature is around 25° C and, most important of all, the temperature must be stable; it is essential to avoid excessive excursion in temperature swings.

<u>Never</u> install the analyzer in an area where a strong electromagnetic field is present. Never use radio transmitters near the analyzer. Also, it is a good idea to eliminate fluorescent lights near sensitive electronic circuits.

WARNING !!!

- 1. TO AVOID DAMAGE TO ANALYZER, ALWAYS LEAVE THE PROTECTION CAPS ON GAS CONNECTIONS UNTIL <u>CLEAN GAS</u> IS READY TO FLOW IN THE ANALYSER.
- 2. THE VENT CONNECTION OF THE ANALYZER MUST BE AT ATMOSPHERIC PRESSURE **ALL THE TIME.** PRESSURIZING THE VENT SIDE OF ANALYZER COULD CAUSE THE QUARTZ CELL TO CRACK AND ALSO DAMAGE THE FLOW MODULE. FOR EXAMPLE, IF YOU HAVE TO CHECK YOUR SAMPLE LINES FOR LEAKS, DON'T DO IT WITH THE ANALYZER CONNECTED TO SAMPLE LINE WHILE THE VENT PROTECTION CAP IS STILL INSTALLED. SO PLEASE DO YOUR LEAK TEST AND CONNECT YOUR SAMPLE LINE TO SAMPLE INLET ONLY AFTER ALL LEAK TESTS ARE DONE. ANY FAILURE TO FOLLOW THESE RECOMMENDATIONS WILL VOID THE WARRANTY.
- **3.** THE ANALYZER CABINET IS NOT DESIGNED TO BE SUPPORTED BY FRONT PANEL ONLY. A BRACKET SUPPORTING THE REAR OF THE ANALYZER CABINET MUST BE INSTALLED. SEE USER MANUAL FOR DRAWING OF SUCH INSTALLATION.





PR1 is a miniature stainless steel high purity pressure regulator. The internal volume must absolutely be at it's minimum. Any unswept dead volume will cause noise and instability and, will cause poor recovery time. PR1 should be installed close to the sample point. PR1 should be installed only if there is large pressure excursion (over 69 kPa (10 PSIG)).



Some users wish to monitor the inlet pressure. Connecting a pressure gauge at the inlet will result in big unswept volume. This will cause poor response time, drift and noise. DON'T DO THIS! Problems caused:

- long response time

- will eventually read below the scale because of error of calibration.

✓ NO

ANALYZER FRONT END SYSTEM



Some users wish to monitor the inlet flow with a falling ball rotometer. Those rotometers (or flowmeters) are calibrated at atmospheric pressure. The flow indicated will be wrong, it must be compensated for the line pressure. Furthermore, these types of rotometers are absolutely not leak tight. Air will diffuse into the system, there will also be some permeation through the material used to build it. There is also a risk that someone tries to control the flow with the valve mounted on this rotometer (if any). This will become in competition with the analyzer internal flow control system.

Problems caused:

- line pollution
- flow instability
- reading will be noisy
- reading will definitely drift.



ANALYZER FRONT END SYSTEM



Some users wish to avoid back flow from a vent header into the analyzer vent. Doing so will cause sample flow and cell pressure variation. The cell pressure will follow header pressure variation. Check valve cracking pressure is not constant. The plasma cell must work at atmospheric pressure with no backdraft. Problems caused: - noisy signal

- risk of cracking the cell on the event of check valve failure.

SAMPLE STREAM SELECTION SYSTEM ACCEPTABLE



All V valves are diaphragm or bellow type valves made of stainless steel. The connections are 1/8" double ferrule compression type. The inlet by-pass rotometer • could be installed to provide faster response time. By-pass flow must be constant. By-pass rotometer • could also be installed, it's flow must be constant. This will speed up purge and response time since the flow in the analyzer is small (25 sccm). The drawback of this system is the dead unswept volume introduced by each unselected valve. The discharge branch • will slow down response time and introduce some noise and long term drift. The length of each branch must be made as small as possible to minimize this problem. Furthermore, the length of section • is also a problem if there are many V valves. For example if V1 is selected (open) all other valves are closed, based on number of valves the unswept dead volume can become too high. The next drawing shows a way to overcome this situation.

SAMPLE STREAM SELECTION SYSTEM BETTER



The loop eliminates the problem introduced by the length of section **④**. Here only dead volume introduced by section **⑤** is present. By making these lengths as small as possible, the problem still is tolerable.





7.0 OPERATION

All analyzer functions are controlled through different menu options. The following figures in this section show the overall menu structure. You must become familiar with it.

At power on, the analyzer displays the MAIN MENU.



The **MAIN MENU** is shown in figure 1.

The following pages give a complete description of each menu and sub-menu.

Note the screen displays at the bottom right the software version programmed into the analyzer.

7.1 Configuration

Pressing **F1** from the **MAIN MENU** will bring you to the CONFIGURATION MENU. The CONFIGURATION MENU prompts you to enter three different parameters:

You have to decide whether or not to use "auto ranging". Choosing F1 "yes" allows the analyzer to "switch" automatically between the three possible ranges (0 - 1 ppm, 0 - 10 ppm, 0 - 100 ppm). F1 "toggles" the selection between "yes" and "no".



FIGURE 2: CONFIGURATION MENU

Desired sample flow: you enter your desired sample flow value simply by using the number keys and confirming your value with the "E" key for enter. The allowed numeric value for the flow is any integer number between 0 and 200. The flow unit is cc/m.

The new input value becomes immediately active and the flow valve will react accordingly.

3. Analog output mode: by pressing the "**F2**" key, the analog 4 - 20 mA output will be in either "track" or "hold" mode.

Hold mode: "hold" will maintain analog output at last "on run" gas value when exiting run mode.

Track mode:"track" will cause analog output to track input gas value during all modes.

Pressing the"F3" key will bring you to the second page of the CONFIGURATION MENU.



FIGURE 3 : CONFIGURATION MENU - Page 2

4. mA Failure mode F1 : in the case of an "Underscale", "Over scale", "Low Flow" or "Plasma off", this option will set the 4-20 mA output : Off (always remain between 0 and 20 mA, even if an error occurs), Low (below 4 mA), or High (higher than 20 mA).

If you have any options, you will find them described in the appropriate appendix of this manual.

You exit the configuration section by pressing **F4**. The analyzer will display the first page of the **CONFIGURATION MENU**. Pressing **F4** again brings up the **MAIN MENU**.

This is the standard configuration. Other menus may appear if your analyzer has other optional features (serial port, fully automatic calibration, digital I/O, etc.).

NOTE: When you select the automatic ranging feature, the scale selection is based on analog to digital count value or ppm value, based on which one reaches the maximum value for the scale in use.

7.2 Calibration

The calibration menu is shown in Fig. 4. In this section, you have to enter the value in ppm of your zero calibration gas and your span calibration gas. The recommended values are between 0 and 20% of the full scale for the zero value, and between 80 and 95% of the full scale for the span value.

For example, a zero gas of 2.0 ppm and a span gas of 8.0 ppm are excellent values for X10 range. Once the span value is entered, the analyzer will automatically select the proper operating scale. For this reason, you must wait for signal stabilization before sampling the value for calibration.

From MAIN MENU, when you press the F2 key, the CALIBRATION MAIN MENU is displayed on the analyzer. See Fig. 4.



FIGURE 4: MAIN CALIBRATION

You enter your zero calibration gas value in ppm, as well as your span calibration gas value. This is done by selecting F1 for the zero value, and F2 for the span value. Figures 5 and 6 show the display for each selection.

<CALIBRATION MENU>> Input ZERO value:0.0 Actual value:0.0 Value must be in PPM F4:RET

FIGURE 5: DISPLAYED WHEN ENTERING ZERO VALUE



FIGURE 6: DISPLAYED WHEN ENTERING SPAN VALUE

The analyzer will automatically select the appropriate range based on the span value entered.

When you have entered a value and pressed the "E" key (E for enter), the processor checks the format, evaluates it and validates it. If everything is OK, your value will be redisplayed in the actual value field. If not, the value is rejected, the input field is cleared and the actual value is left unchanged.

If you made an error when entering your value, just press enter and start over.

Once you have entered the zero and the span values, select F3 to get the "CALIBRATION SUB-MENU".

The following figure shows this sub-menu:

```
<CALIBRATION FACTORS CALCULATION>>
Measure: 9 PPM Sample Flow: 25 CC
F1:Zero F2:Span F4:RET
Calibration status:
```

FIGURE 7: CALIBRATION SUB MENU

The display shows the actual ppm of nitrogen value and the actual flow in cc. If the unit is started up for the first time or you replace the calibration gas with a new value, the ppm display has no meaning. This can be zero or off scale.

In order to calibrate the unit properly, all lines and regulators on the calibration cylinders must be completely purged. Allow the zero gas to flow into the analyzer. After 10 minutes of the zero gas flowing into the analyzer, select "F1" on the keypad. You will be asked for a confirmation to rezero, select "YES", at this moment this message will be replaced by "PLEASE WAIT", indicating the sampling of the analog input. This message is displayed for 5 seconds.

NOTE: an indicator is displayed on the right of the confirmation message informing the user if the re-span or the re-zero has not been performed yet.

After this delay, the "ZERO DONE" message is displayed. Now, allow the span gas to flow into the analyzer for 10 minutes or until the displayed measure value is very stable. Select "F2" on the keypad. Again, you will be asked for a re-span confirmation, select "YES", at this moment, again, this message will be replaced by "PLEASE WAIT" on the display for a certain amount of time. When this step is done, the "SPAN DONE" message is displayed; the span gas value is shown on the left corner of the display. Recheck zero and, if necessary, calibrate a second time.

When the calibration procedure is finished, go back to the **CALIBRATION MAIN MENU** by pressing "F4" on the keypad. From the **CALIBRATION MAIN MENU**, select "F4" again to go back to the **MAIN MENU** ("TOP LEVEL MENU").

7.2.1. Important facts concerning calibration

With today's technology of Argon production, total impurities in argon are below 1 ppm. It is difficult to obtain reliable calibration gases with low ppm value. For example, to calibrate in range 1 with a calibration cylinder one would require a zero gas containing around 0.2 ppm and a span gas containing 0.8 ppm. In practice, many users have discovered that zero gases were giving higher cell counts than the span gases. There are many reasons for this: the mixture can be wrong or the cylinder may have been contaminated when installed. Bad purging procedures will shift the calibration cylinder by many orders of magnitude. The SERVOPRO PLASMA analyzer can be calibrated in the 0-10 range or in the 0-100 ppm range; and this without affecting the accuracy on the 0-1 ppm range if the zero gas is really zero. To achieve this, we recommend

7.0 OPERATION

the use of a getter to generate the zero gas and a span gas having 7 to 9 ppm for calibration if the 0-1 and 0-10 ppm ranges are the most frequently used. There is no offset between ranges with the SERVOPRO PLASMA; therefore, no error is generated when switching ranges.

A 70 to 90 ppm of N_2 can be used if working in the 0-100 ppm range. Again, the accuracy will be correct in the 0-1 ppm range. Please see next figure on "How to install a getter to generate the zero gas". It is an excellent idea to use a regulator at the outlet of the purifier. This way the purifier can be mounted at 552 kPa (80 PSIG) to 621 kPa (90 PSIG), reducing the risk of inboard contamination. Higher working pressure results in better performance for the purifier. Adjust the analytical regulator at 69 kPa (10 PSIG).

HERE IS HOW TO INSTALL THE PURIFIER FOR ZERO GAS GENERATION



7.3 Diagnosis

Pressing "F3" from the MAIN MENU will bring you to the DIAGNOSTIC MENU.

The diagnostic feature of the analyzer gives you the opportunity to verify the output of the analog to digital converter count value and check the 4 - 20 mA output.

If you have any options other than standard, you may also verify them (digital and/or fully automatic calibration option). Please refer to the appropriate appendix of this manual.

```
<<DIAGNOSTIC MENU>> F3:NEXT
Cell counts: 13120
Flow counts: 1666432 Temperature: 24.45
F2:Error and Alarm historic F4:RET
```

FIGURE 8: DIAGNOSTIC MAIN MENU

In the diagnostic menu the cell counts and flow counts are displayed. It is very useful to verify stability and cell offset.

By selecting "F2" it is possible to see the error and alarm historic menu.

"F3" brings you to the second diagnostic menu.

By pressing the **"F2"** key, it is possible to control the analog output manually. By selecting **"F4: RET"**, the system displays the **DIAGNOSTIC MAIN MENU** again. The display looks like the following figure.



FIGURE 9: DIAGNOSTIC MENU



FIGURE 10: DIAGNOSTIC MENU

You need only enter any integer number value between 0 and 100 % and press the "E" key for enter. Zero (0) is for 4 mA and one hundred (100) for 20 mA. This makes it easier to calibrate

remote monitoring system and also makes it faster to verify the hardware part for the 4 - 20 mA signal isolation module.

NOTE: the analog output mode selected from the configuration menu ("track" or "hold") will affect the 4 - 20 mA output. Set the analog output mode in "hold" for 4 - 20 mA output verification. If not, the 4 - 20 mA will still track input gas value.

7.3.1 Error and Alarm Historic

This menu is very useful. You can see the last 25 alarms with the time and the date. Pressing "F1" from the **DIAGNOSTIC MAIN MENU** brings you the newest errors and F2 the oldest. For example, if a low flow error occurs at 9:34:37, the error and alarm historic menu will display "Low Flow" message with the hour and the date. When the flow is restored, the same message is displayed with ":OK". For example, if the low flow is restored at 10:23:01, the error and alarm historic menu will display "Low Flow" DK" like in Figure 11 with the date and the time.



FIGURE 11: DIAGNOSTIC MENU

Here possible errors thus their significance:

LOW FLOW: The flow on sample is smaller than 10 ml min⁻¹ (10 cc/min).

PLASMA OFF: The plasma is shut down. Occur when there is low flow since 30 seconds or when the starting count is not reached.

STARTING: The plasma is trying to start.

WAITING: The system is waiting to preserve the valve. This error occurs 1 minute after the plasma is turning off. The system waits 2 minutes before trying reopen the valve.

OVER SCALE: The impurities value is greater than scale.

UNDER SCALE: The impurities value is smaller than scale.

ALARM 1: Alarm 1 set point is reached.

ALARM 2: Alarm 2 set point is reached.

AUTCAL FAIL: This error occurs when the auto calibration failed.

Pressing "F1" brings you the newest errors and "F2", the oldest.

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7.3.2 Ethernet info



FIGURE 12: DIAGNOSTIC MENU

This menu is accessible by pressing F3 from the verification page of the 4-20 mA outputs. It shows different information concerning the Ethernet connection of the analyzer. The TCP/IP address of the analyzer is displayed in front of "IP" and can be used to control the analyzer from the Internet and through a simple network. Simply enter, for example, http://192.168.0.111 in your web browser to see exactly what is currently displayed on the analyzer screen. Use the computer mouse to click on the keyboard displayed in your browser in order to control the analyzer normally through the network. The MAC address, the mask and the gateway can be pertinent information for your network administrator. The MAC address can't be changed. It is a hardware characteristic set by Servomex. But to configure the mask, the gateway or change the Ethernet password, go to the Ethernet pages of the **HIDDEN MENU**. Refer to the **WEB INTERFACE** section for a connectivity procedure.

7.3.3 Serial number



FIGURE 13: SERIAL NUMBER MENU

This menu is accessible by pressing F3 from the Ethernet page. It displays the analyzer serial number, which corresponds to the one written at the back of the analyzer. This can be useful if the back of the analyzer is not accessible.

7.4 Run

Most of the time, you will be in this mode, which displayed the following screen that is accessible by pressing F4 from the **MAIN MENU**.
```
<<RUN MODE>> F4:RET
Measure(PPM): 10.0
Sample flow(CC): 75.0 Scale: 0-10 PPM
System status:OK
```

```
FIGURE 14: RUN MODE
```

In this mode, the following information is displayed: measurement in ppm, flow in cc, system status, and range in use.

The 4 - 20 mA output is refreshed to reflect the level of impurities according to the scale in use. If you have the digital output option, it can be connected to a remote system to indicate the scale in use.

If from the "**CONFIGURATION MENU**" you have selected auto-ranging, the scale will change according to the ppm value. If you have selected manual ranging, you must select the desired scale by pressing the "1" key for 0 - 1 ppm, the "2" key for 0 - 10 ppm and the "3" key for 0 - 100 ppm.

In both cases, i.e. manual or auto ranging, the actual scale in use is shown on the display.

The gas sample flow is always displayed. Normally, the value displayed is the value entered in the **CONFIGURATION MENU** for the sample flow set point. The value may change when you select different gas streams or if you get too much inlet pressure variation. The inlet pressure must be stable - between 34 kPa (5 PSI) to 69 kPa (10 PSI) for best results.

In the run mode, you may get a status message. Normally, the "**OK**" message is always displayed. Below is a list of messages and their corresponding meaning:

OK: No problem.
 STARTING: Boosted power is applied to the plasma generator for start up. Normally displayed for a short period of time, but it may take as long as 30 to 45 minutes to start the plasma, depending on gas condition or if cell temperature is too cold.

Other messages can be displayed where the ppm value is normally displayed. You may get "Under scale" if the cell count or the calculated ppm value is too low for that scale. "Over scale" will be displayed if the cell count or calculated ppm value is too high for the scale in use. "Plasma off" will be displayed if the plasma is off. "Waiting" will be displayed if the plasma stays off for about 1 minute. In this case, the sample flow will be stopped to protect the valve. This is a short time operation, however, the sample must always be connected and flow must pass thru the analyzer.

Other messages can appear depending on options or custom features ordered (auto-zero, auto-span).

When an error is detected, the status (ST) digital output will be deactivated (contact open).

NOTICE: When the flow is lower than 20 cc for longer than 30 seconds, an internal relay will shut down the plasma generator and the appropriate message will be displayed. When the flow comes back to normal, the generator restarts.

7.5 Special consideration about the 0 - 1 ppm range

When the 0 to 1 ppm range is selected, the system is very sensitive. The display resolution is fixed at 10 ppb. In order to use this range efficiently, it is very important that the connections be leak-free, that there is no moisture and no quick connector and that the unit is installed and started up properly. It may take some time to get a stable reading. On this range, the response time is longer. The by-pass flow must be constant, and vent line pressure must be at atmospheric pressure. Do not forget: <u>NEVER USE LINES OTHER THAN STAINLESS STEEL FOR THAT</u> TYPE OF MEASUREMENT.

It is normal to see the display reading wander slowly on this range. If higher sensitivity is required, you must select our ppb analyzer. Of course, the cost of such systems is much higher: it is normally used by electronic wafer fab industries for end-of-life argon purifier verification.

If you analyze pure argon on this scale, you will get a good approximation of its quality.

It is possible to verify and calibrate this range using the flow dilution technique instead of a gas cylinder. It is very difficult to use gas cylinders for calibration on this range, because very careful handling is required. We use the dilution technique.

7.6 Hidden Menu

By pressing 1,2,3 and E (enter) from the Main Menu, the **HIDDEN MENU** opens.

7.6.1 Starting Count

The starting count value is the threshold value where the analyzer switches from starting mode to normal mode.

If the cell counts (value from the analog to digital converter; this value may be observed under diagnostic menu) is under the starting count value, and higher power is applied to the cell by the plasma generator. Once the plasma is started, the cell counts should become higher than the starting count value and the power will come back to normal. If for any reason, the cell count value with the normal power applied to the cell is too close or oscillates around the starting count value, the analyzer will go back and forth between starting and normal mode.

Too get rid of such situation, you should enter a new (lower) threshold value. To enter a new value just use the numerical keys on the keypad followed by enter key (E key). The value must be between 0 and 16,777,215 counts. The default value is normally just what the system needs and under normal conditions this value should not be changed.



FIGURE 15: HIDDEN MENU, FIRST PAGE Minimum: 0 counts Maximum: 16 777 215 counts

Resolution: 1 count

This value is set by Servomex, and may need to be re-adjusted over the year.

7.6.2 Temperature Coefficient

This menu gives the possibility to enter coefficient that will be used for system temperature variation. Using these factors, the software will do a temperature compensation to minimize temperature drift. There are two factors, one for the offset and another for the gain. The temperature affects the baseline of the instrument and the sensitivity. These factors are entered at the factory during temperature drift tests. Don't change them, except if you suspect that they are not good. There is a procedure to follow to do temperature tests. If you have to do it, call Servomex to get the exact procedure. It must be done by trained personnel. Most of the time there is no compensation and these factors are then set to 1.000.

Press F1 to access the temperature offset menu.

<<<TEMPERATURE OFFSET>>> Offset:1.000 Actual:1.000 F4:RET

FIGURE 16: TEMPERATURE COEFFICIENT Minimum: 0 Maximum: 2 Resolution: 0.001

If you have to change the offset compensation factor, enter the value with the numeric keypad and then, press E to validate the new value.

Press F2 to access the temperature gain menu.

| <<<' | TEMPERATURE | GAIN>>> | |
|-------------|--------------------|---------|--------|
| Gain :1.00 |) | | |
| Actual:1.00 |) | | |
| | | | F4:RET |
| | | | |
| | | | |

FIGURE 17: TEMPERATURE GAIN

Minimum: 0 Maximum: 2 Resolution: 0.001

If you have to change the gain compensation factor, enter the value with the numeric keypad and then, press E to validate the new value. The menu to enter the compensation factor for the offset looks the same as the one for gain.

N.B: The ambient temperature is saved at each calibration. This value is compared with the actual ambient temperature to calculate the new "compensated" ppm value.

7.6.3 Time and date setting

When you enter in the time and date setting menu, the following menu appears. Enter time first, using the numbers on the keypad. For example, if you wish to enter 15:45:02, you only have to do the following steps.



FIGURE 18: TIME AND DATE SETTING

- 1- Enter the number 15 on the keypad. Then, press enter.
- 2- Enter the number 46 on the keypad. Then press enter.
- 3- Enter the number 23 on the keypad. Then press enter.

Now, the menu is supposed to look like the following screen.

```
<<TIME AND DATE SETTING>>>

Time 15:46:23 Actual: 00:00:00

Date 00/MM/DD Actual: 00/00/00

F3:NEXT F4:RET
```

FIGURE 19: TIME AND DATE SETTING

When you enter the time, you are obliged to enter the date. You only have to enter the year, month and day like you did in the preceding steps for the time.

<<<TIME AND DATE SETTING>>>
Time 15:46:23 Actual: 00:00:00
Date 05/06/27 Actual: 00/00/00
F3:NEXT F4:RET

FIGURE 20: TIME AND DATE SETTING

When you enter the day and then press enter, the analyzer refreshes the actual time and date and the menu is supposed to look like the following screen.

```
<<<TIME AND DATE SETTING>>>
Time HH:MM:SS Actual: 15:46:23
Date YY/MM/DD Actual: 05/06/27
F3:NEXT F4:RET
```

FIGURE 21: TIME AND DATE SETTING

If you enter a bad value, the analyzer clears the field and you are asked to enter a new one.

7.6.4 System Gain

This value is used to adjust the last stage of amplification. If for any reason the same amount of impurities gives fewer signals, you may adjust this value to set the amplification. You may see it as a span potentiometer.

N.B.: You must recalibrate the analyzer each time you change the system gain.

The smallest gain is 2 and the highest is 1000



FIGURE 22: SYSTEM GAIN

7.6.5 PID values

```
<<<PID values>>>
1-Proportional 2- Integral 3-Derivative
Sample flow(CC):25.0
F3:NEXT F4:RET
```

FIGURE 23: GAS FLOW PID VALUES

The PID values are the proportional, integral and derivative constant of the loop controlling the gas flow. Depending on the gas flowing in the analyzer, the PID values could have to be changed in order to obtain a stable flow. To modify any of the three constant, press 1, 2 or 3 respectively for the proportional, integral or derivative value.



FIGURE 24: CHANGING A PID VALUE

The screen will display the preceding figure. Type a new value on the keypad and press E (Enter) to update the current value. Press F4 to modify another value or the leave the menu.

7.6.6 Lock Range



FIGURE 25: LOCK RANGE

Pressing F3 from the System Gain Menu will bring you to the Lock Range Menu. The lock range can be set "ON" or "OFF".

If the lock range is set 'OFF'', the user is allowed to manually change the range in the run menu. If it is set ''ON'' the range cannot be changed.

NOTE: To lock the range, you must disable the auto ranging in the configuration menu.

NOTE: You cannot activate the auto ranging if the range is locked.

7.6.7 Ethernet configuration 1

Pressing F3 from the Lock range menu will bring you to the Ethernet Configuration 1 page.





The Ethernet connectivity can be turn off or on by pressing F1. If this option is set to NO, the analyzer can't be controlled through a network. To control it through a network, this option must be set to Yes.

Pressing F2 will enable you to change the default password required to access the analyzer from a network using Ethernet. The default password is "KONTROL" and will be used in your web browser.

| | <<< | <input string=""/> >>> | |
|---------|-------------|------------------------|--------|
| Ne 0 | w Password: | | |
| F1 | UP | F2:DOWN | F4:RET |

FIGURE 27: SETTING THE WEB PASSWORD

To modify the first letter of the password, press F1 to increase the value (from 0 to 9 and then from A to Z) or F2 to decrease it. Press E (enter) to fix the first letter to this value and to modify the second letter. Modify the second letter by increasing or decreasing the value with F1 and F2. Press E (enter) to keep this value and to change the next letter. Continue to modify the letters until the desired password is set and press F4. A maximum of 12 letters is allowed. The new password, in other words, the actual password, is displayed new to "Actual:" in the **ETHERNET CONFIGURATION 1 PAGE**.

7.6.8 Ethernet configuration 2

Press F3 from the first page of the Ethernet configuration 1 menu to access the second page.



FIGURE 28: ETHERNET CONFIGURATION 2 PAGE with DCHP on

If the analyzer is connected to the Internet via a DHCP server, the network router assigns a TCP/IP address automatically to the analyzer. For this to happen, the DHCP option must be enabled in the analyzer. To do this, press F1 in this menu in order to set the DHCP to "Yes". This corresponds to the easiest way to connect the analyzer to the network because the router manages the Ethernet connectivity automatically. All the useful information is then displayed in the Ethernet page of the **DIAGNOSTIC MENU**.

If the network does not have the DHCP option, a TCP/IP address must be entered manually in the analyzer. To do this, first turn off the DHCP option by pressing F1 in order to see "No" next to "DHCP Enabled?". The menu will look like that:



FIGURE 29: ETHERNET CONFIGURATION 2 PAGE with DHCP off

To set the IP address and the other parameters related to the Ethernet connectivity, press F2.

<<<IP PARAMETERS>>> F1:Local IP:192.167.0.5 F2:Mask: 255.255.255.0 F3:Gateway:192.167.0.1

FIGURE 30: IP PARAMETERS

F4:RET

Contact your network administrator to set-up this aspect of the analyzer. You must first know the Gateway of your network. Set it by pressing F3. Then, enter a value of 1 to 3 digits and press E (enter). Enter the second value of 1 to 3 digits and press E. Enter the third value of 1 to 3 digits and press E. Finally enter the fourth value of 1 to 3 digits and press E. The Gateway is then updated. Do the same to change the Mask to the value given to you by your network administrator by first pressing F2. The Mask is then updated. The Local IP is the address of the analyzer that is used in your browser to remotely access the analyzer. It generally begins by the three first values of the Gateway. In general, only the last value (the fourth one) is not equal to the last value of the Gateway. When everything is set, exit this menu with F4. All the information is then displayed in the **DIAGNOSTIC MENU**.

7.6.9 Averaging Number



FIGURE 31: AVERAGING NUMBER

Minimum: 1 Maximum: 25 Resolution: 1

The software may execute a moving average on the calculated ppm value. This will also affect the response time of the system. The software uses this parameter to determine the number of sample PPM values that are used in its moving average sub-routine. The number of samples ranges from 1 to 25. When entering 1, there is no moving average.

For example, in a 5 point moving average filter, the displayed actual process valve will be:

$$Ppm = \underline{p(1) + p(2) + p(3) + p(4) + p(5)}_{5}$$

Where p(5) is the new measure done and p(1) to p(4) are the four previous ones.

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7.6.10 Calibration done parameters

```
<<<CALIBRATION DONE PARAMETERS>>>
Slope (x10K): 0.0000 Offset: 0.000
Zero: 0 Span: 0
F4:RET
```

FIGURE 32: CALIBRATION DONE PARAMETERS

This menu displays the calibration values calculated by the analyzer. In front of "Zero", we find the analog to digital converter counts of the signal measured with the zero gas. In front of "Span", we find the analog to digital converter counts of the signal measured with the span gas. The slope and the offset of the equation generated from these two values for further calculations are displayed in front of "Slope (x10K):" and "Offset:".

7.7 SERVOPRO PLASMA web interface

This section explains the general steps to follow in order to control the analyzer through a local network or from Internet.

STEP 1, the cable: the first step is to connect the analyzer to the local network. If the analyzer has the Ethernet connectivity option, connect a RJ45 cable from the analyzer rear panel to a router or a hub of the network.

STEP 2, the IP address: then, the IP address of the analyzer must be set. This address could be, for example, 198.123.0.1. It corresponds to the address that is used in your web browser to access the analyzer, as you would do to access any other web site on the Internet. From the browser point of view, the analyzer will act as a server.

It is important to know first if your network works with DHCP. If so, activates the DHCP in the Ethernet configuration 2 page of the **HIDDEN MENU** (refer to the appropriate section) and restart the analyzer without the cable connected. Then, turn off the analyzer, plug the cable and restart the analyzer.

If your network does not have the DHCP option, you must set the IP address, the mask and the Gateway manually. All those are network parameters. To set these parameters in the analyzer, go to the Ethernet page of the **DIAGNOSTIC MENU**. When the parameters are set, restart the analyzer.

Contact your network administrator first for further help about your network characteristics.

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STEP 3, the router: the analyzer now acts as a server over the network. If you want to access it from outside the local network, the network router must allow the IP address to be accessed from the Internet on <u>TCP port 3076</u>. Setting this aspect refers to your router specifications.

STEP 4, the browser: now, in the address bar of your web browser, enter the IP address that is displayed in the Ethernet page of the **DIAGNOSTIC MENU** preceded by "http://".

If you have Windows Service Pack 2 installed on your computer, a security tooltip bar will appear on your browser. As shown below, click on it, then click "Allow Blocked Content..."



A security warning popup should then appear. Click on Yes.



Then, the Java applet will be loading for about 10 to 20 seconds if it is the first time ever you load it:



STEP 5, the browser: the last step before being able to completely control the analyzer through a network is to type in the password that you can see and change in the Ethernet Configuration 1 page of the **HIDDEN MENU** and to click on the **Submit** button. The password is case insensitive.



FIGURE 33: ENTERING PASSWORD

If you enter an incorrect password, you will be asked to enter it again 10 seconds later. Otherwise, you gain control of the analyzer and see the screen that is currently displayed on the analyzer.

| | K A | (on Nai | TRO LYTI | L IK |
|---|--------|--------------|-------------|---------|
| KONTROL ANALYTIK K2002 nnb N2 ANALYZER | 1 | 2 | 3 | F1 |
| < <main menu="">></main> | 4 | 5 | 6 | F2 |
| Fl:Configuration F3:Diagnostic F2:Calibration F4:Run V3.63 | 7 | 8 | 9 | F3 |
| | • | 0 | Е | F4 |
| | | K20(K20(| 01n: 02 | |

FIGURE 34: SERVOPRO PLASMA WEB INTERFACE

STEP 6, controlling the analyzer: from now, use the mouse to click on the displayed keyboard to operate the analyzer, as you would do with the physical SERVOPRO PLASMA keypad.

The analyzer uses a 128 bits AES encryption key to send and receive data, so it is very secure.

WARNING: take your time while controlling the analyzer through a network; the analyzer answer to your command can take a while to reach back your computer due to the network transmission delay.

WARNING: be aware that anyone with the IP address of the analyzer can control it from all around the world. The web password is a good protection. But for improved security, control the analyzer within a local network protected by a router that locks the IP address of the analyzer so that it could not be accessed from outside the local network (i.e. Internet is outside your local network). For an even better safety, unplug the Ethernet cable that connects the analyzer to the router/hub when the analyzer does not need to be remotely controlled. Therefore, it is recommended to use two networks in your factory, one for general purposes (computers connected to the Internet as usual) and another one for your analyzers and industrial components. When necessary, this second network can be connected to the first one in order to be accessed from the Internet, by Servomex Canada support team for example.

NOTE: the SERVOPRO PLASMA web interface has been tested on a computer equipped with Internet Explorer 5.5 to 6.0 on Windows XP with Service Pack 1, with no Antivirus software and firewall. If the page cannot be displayed message is displayed in Internet Explorer after having typed the password, try to turn off any software on your computer that controls the ethernet connection.

7.0 OPERATION

First try to turn off the "Windows Firewall". Go in the "Control Panel", click on the "Network Connections", right-click on your "Local Area Connection" and select "Properties". Then, select the "Advanced" tab. Click on "Settings" and select "Off" as the Windows Firewall option. By sure that any other firewall is turned off. If you have the "Windows Service Pack 2", open the "Security Center" from the "Control Panel" and by sure that the firewalls are "OFF". Reset the analyzer and try again. Note that a router can act as a firewall if the Windows's firewall is off.

If it does not solve the problem, turn off anti-virus protections such as worm protection. Refer to your anti-virus manual for further explanation on how to turn off worm protection. Reset the analyzer and try again.

Still experiencing problems? Lower the Internet Security of your browser. In Internet Explorer, open "Internet Options" from the "Tools" menu. Select the security tab. Click on "Local Intranet", then on "Default Level" and lower the slide to "Low".

If you want to use the SERVOPRO PLASMA web interface from outside your network, click on the "Internet" icon that is next to "Local Intranet" and lower the security as you did for the "Local Intranet".

8.0 START-UP

Once all lines are purged and cleaned, put the power "**ON**" with the ON-OFF switch at the rear of the analyzer. The display will come on and show the **MAIN MENU**.

When the system is powered up, the flow set point has a default value of 75 cc; it normally does not require further setting, except for faster purging. In this case, increase the flow to 190 cc to purge the unit for one or two hours.

NOTE: The flow control valve is a miniature thermal valve. On power up the valve is cold. It may take up to two minutes before to have flow trough this valve. Once the valve is warm the flow will stabilize. If you put the flow set point to 0 cc for a while the valve will cool down again. Never leave the flow set point higher than zero cc if gas is not available, there is a risk to burn the valve, and flow module replacement will be necessary. For this reason make sure that gas is available before powering up the analyzer.

But, again, <u>before putting the power on</u>, be sure that your gas lines are properly purged with good quality argon. In this way, the analyzer will be ready to use much faster. Also, and more importantly, you do not send higher level of impurities in the plasma.

After the purge time is elapsed, you may adjust down the by-pass rotometer to an acceptable flow, depending on the speed of response required and the distance between the analyzer and the source of gas to be analyzed. The analyzer flow set point may be returned to 75 cc/m.

You are now ready to calibrate the analyzer. We strongly recommend you our intelligent gas purifier (GP-200-I) in line with a Grade 5.0 or better Argon UHP for the zero calibration. By using our gas purifier you will avoid negative reading caused by polluted low ppm cylinder. More over, you will save money since the price for low ppm N_2 in Argon is very expensive and not reliable.

In case you use our gas purifier for the zero calibration, enter 0.0 ppm in the calibration menu for the zero gas.

For the span calibration, choose a cylinder between 7.0 and 9.0 ppm. The analyzer will automatically select the scale X10, when you go to sub-menu calibration (see the operation section for the detailed procedure). To enter your calibration gas value and calibrate, follow the steps in the operation section of this manual.

Once the calibration is done, the analyzer is now functional; it will take a few days for the analyzer to stabilize itself. For this reason, it may be necessary to recalibrate.

8.1 Routine and operational verification

From time to time, it is a good idea to go in the "**DIAGNOSTIC MENU**", check cell counts with a reference gas and record this value for comparison with a later value. This gives an indication of the degree of cell offset.

Also, check the flow count stability. Too much variation is an indication of line clogging.

The frequency of calibration is normally once a week.

8.2 Hints and tips

When the analyzer is used to verify gas cylinders, there are no special considerations. But, when you use it for argon in the liquid phase, or for trucks in loading stations, you must be aware of a few facts.

Analysis is made with a gaseous sample of the cryogenic liquid. For trucks, a common procedure is to use a quick connector. Then, the liquid vaporizes inside the line in the direction of the flow. Other installations use a flash vaporizer for tanks, and a distillation column.

A special difficulty arises in obtaining a gas sample which has the same molar concentration of trace contaminants as the liquid being sampled.

For example, liquid argon may typically contain traces of oxygen, nitrogen, hydrogen, methane and water. When the temperature of liquid argon is raised, the vaporization of argon and its different contaminating gases take place at different temperatures and thus at different points in time.

This way, fractionation of the sample occurs: the analytical composition of the gas sample obtained varies continuously and does not accurately represent the analytical composition of the cryogenic liquid. For example, this phenomenon is very apparent when analyzing hydrocarbons in pure oxygen.

If your reading varies a lot on the liquid sample, you must think of a way to present a true sample of the liquid to the analyzer. Normally, this is not a problem for the customer in the electronics industries, foundries or specialty gas laboratories.

When mounting a quick connector on a truck, moisture and air get into the sample line. Allow some time, depending of line length, to purge the sample line.

Often (very often) quick connectors can leak, when the lines attached to them are moving. Be aware of this.

Before ending this section, there is one more thing to consider. Calibration cylinders come with a certificate attached, confirming the amount of impurities in the cylinder. It is a good idea to find out how this analysis was done.

Servomex is a good customer of such cylinders coming from different sources of specialty gases. Sometimes, with three cylinders containing the same quantity of impurities (less than .2 ppm total difference), each coming from different suppliers, we obtained some surprising readings. In some cases, there was a difference of more than three times the quantities stipulated on the certificate from cylinder to cylinder. **Beware of this**.

See our application note "RECOMMENDED GAS SAMPLING HARDWARE AND TECHNIQUE FOR LOW TRACE N_2 MEASUREMENTS IN ARGON".

9.0 MAINTENANCE AND TROUBLE SHOOTING

9.1 Maintenance

This analyzer is maintenance-free. You only need to calibrate at the frequency determined by your experience. Normally, if you use a clean gas and a good sampling system, a calibration every two weeks is enough.

The unit has a two micron inlet filter. Depending on the conditions of use, you may need to replace this filter. Normally, it is a good idea to replace it once a year. This filter traps particles that may accumulate moisture. This moisture is released at different rates, depending on ambient temperature and particle size. For this reason, instability may occur if your filter has any accumulation of dirt. See "Appendix 1".

9.2 Trouble-shooting

Generally, it is strongly recommended to limit your interventions to the replacement of defective modules. If you experience trouble during normal operation, use the **DIAGNOSTIC MENU** to verify cell counts and flow counts. Before referring to the trouble-shooting procedure, you have to make sure that the flow is constant and the vent pressure is at atmospheric pressure.

You cannot open the cover of the module because it is filled with special putty material. For example, there is silicon base putty for the cell, around the quartz to allow for thermal expansion. Surrounding this silicon, a special sealing putty with appropriate dielectric properties fills the rest of the modules and cures under vacuum. The result is a leak-free, vibration-resistant, thermally stable system.

The cell module, the signal conditioner with optical separation system and the flow module are not serviceable. Under normal conditions, they will last for years.

The analyzer is modular; therefore module replacement is fast and easy.

| We recommend the following spare parts: | One (1) flow module |
|---|-------------------------|
| | One (1) DC power supply |
| | One (1) I/O board |
| | One (1) set of fuses |

The following table will help you identify possible causes of problems. If you cannot fix your problem after these suggestions, please contact the factory and be ready to give details regarding the installation of your analyzer.

9.3 **Problem causes**

| PROBLEM | POSSIBLE CAUSE | REMEDY |
|----------------------------|---------------------------|------------------------------|
| Power is on but display is | • Fuses, or power cord | Connect power cord or |
| still off | not connected | replace fuse |
| Display is off, fuses are | • DC power supply | Replace each module |
| OK, power cord is | • LCD contrast power | sequentially until |
| connected | supply | problem is fixed |
| | • LCD display damaged | |
| Display comes on but | • LCD controller display | Replace each module |
| nothing displays | • CPU board | sequentially until |
| | Contrast potentiometer | problem is fixed |
| | has been affected | • Re-adjust contrast potent. |
| | | on CPU board |
| ppm reading still at zero | • Cell module | Replace cell module |
| or negative, flow OK | • I/O board | (call factory) |
| | | Replace I/O board |
| No flow reading | • Flow valve or flow | Replace flow module |
| | transducer | |
| Flow reading is erratic | • Inlet pressure too low | Increase inlet pressure |
| | • Flow module | until flow is stable |
| | | • Set flow higher than 20 cc |
| | | Replace flow module |
| ppm reading going on | • Flow oscillating around | • Set flow higher than 20 cc |
| and off scale | 20 cc | |
| ppm reading wandering | • Moisture in sample | Replace sample trap |
| or drifting, on X10 or | Sample line leaking | • Check sample line for |
| X100 range | • Unstable flow | leaks |
| | • Vent not at | • Check flow and vent |
| | atmospheric pressure | pressure |
| | | |

APPENDIX 1 / HARDWARE AND TECHNIQUE

RECOMMENDED GAS SAMPLING HARDWARE AND TECHNIQUE

FOR LOW TRACE N₂ MEASUREMENTS IN ARGON Revision 1, 200307

By Servomex, Québec, Canada.

<u>1.0</u> Introduction

Servomex produces very sensitive equipment for low level measurements in the range of ppm and ppb. In such types of measurements, the stability, accuracy and response time of the system are greatly dependent on the gas sampling lines, regulators, valves and cylinders in use; operator manipulations are also crucial, one must be careful . Servomex guarantees the performance within analyzer specifications, if the system is installed according to this application note.

The cost of such gas sampling systems is much lower than the cost entailed by loss of production or by shipping bad products to customers, - especially for customers operating electronic wafer plants.

This application note is based on our experience in the use of our equipment. The hardware listed here was tested by us.

1.1 Leaks

Before going further we must explain what "leak" means. The most frequent cause of malfunctions in analytical systems is leakage. Here, by a leak we mean introduction of outside contaminants originally not present in the system.

Introduction of outside contaminants inside a gas line may happen through *permeation*. For example, different types of plastic lines, polymers, neoprene, etc. are permeable to oxygen, nitrogen and moisture. You will not detect any leak, but, oxygen and other contaminants will in fact get into your line, even if the line pressure is much higher than atmospheric pressure. We may define a leak in the following way: a leak occurs in a system where the mass flow velocity is less than the molecular velocity.

When this situation occurs, gas molecules move in both directions through the leak. The net flow of a particular gas (contaminant) will depend on the relative partial pressure of that gas on each side of the leak. In a sample line of argon having only 0.5 ppm of nitrogen, there will be a net flow of nitrogen inward unless the sample pressure is several thousands of pounds.

To avoid such leaks, **never** use plastic lines. Use stainless steel lines with compression-type fittings or better (VCR type). Avoid pipe thread fittings in analytical equipment.

2.0 Selection of material

2.1 Introduction

Special care must be taken in selecting the distribution equipment that will remove the gas from the cylinder and transport it to the analytical instrumentation.

Bad selection of equipment can result in unwanted oxygen, nitrogen or moisture diffusing into the system. Outgassing of monomers and dimers from an elastomer can result in system drift and instability. For example, outgassing from neoprene diaphragms in a pressure reducing regulator can cause excessive drift.

For maintaining gas purity stainless steel is preferred, since oxygen, nitrogen and water do not adsorb into steel as much they adsorb in copper. Furthermore, as an option, you can have stainless steel degreased and passivated. This process removes all traces of oil, grease and dirt, thus ensuring optimal performance.

For all your gas lines and pressure regulators, stream selection valves and fittings, use <u>stainless</u> <u>steel</u>. The use of pipe thread fittings is not recommended by Servomex, and the performance of the analyzer is not guaranteed under this condition of use.

2.2 Pressure regulators selection

Industrial general purpose regulators are often constructed with either Buna-N or neoprene diaphragms. You must not use this type of regulators in analytical systems. Both Buna-N and neoprene are permeable to oxygen.

The only type of pressure regulator that we recommend is the one found at the end of this application note. Any real equivalent will be acceptable.

2.3 Sample lines selection

From the outlet of the pressure regulator you must use a stainless steel line. Stainless steel lines of 1/8" diameter, clean and passivated are commercially available at moderate cost. No other material is approved by Servomex. Plastic tubing often found in older air separation plants is absolutely not recommended. These types of tubing are permeable to oxygen, nitrogen and moisture.

The following table gives some permeation factors of the most commonly used so-called "plastic tubing".

| | PERMEABILITY (approx.) AT 25 ^o C | | |
|----------------------------|--|----------------|--|
| TUBING | PERMEABILITY (approx.) | | |
| FORMULATION TYPE | Units : $\left[\frac{cc-mm}{\sec-cm^2-cm\ Hg}\right]x\ 10^{-10}$ | | |
| | O ₂ | N ₂ | |
| Gum rubber | 171-307 | 80-118 | |
| NORPRENE food | 80 | 200 | |
| Nylon | 2.0-5.4 | 0.2-1.1 | |
| PHARMED [®] | 80 | 200 | |
| Polyethylene | 3.6 | 0.09 | |
| Polypropylene | 25 | 4 | |
| Polyurethane | 10.5 | 17.1 | |
| PTFE | | 1.0 | |
| PVC (BUBBLE [®]) | 1345 | | |
| Reinforced PVC | 0.13-36 | | |
| Reinf. silicone, platinum | 7961 | 2763 | |
| Reinf. silicone, peroxide | 7961 | 2763 | |
| Silicone, platinum-cured | 7961 | 2763 | |
| Silicone, peroxide-cured | 7961 | 2763 | |
| TYGON, food/beverage | 30 | 60 | |
| TYGON, fuel/lubricant | 12 | 22 | |
| TYGON, high-purity | 135 | 45 | |
| TYGON, lab vacuum | 40 | 80 | |
| TYGON, ultra-resistant | 980 | 350 | |
| Vinyl | 40 | 80 | |
| VITON | 13-15 | 4.3 | |

2.4 Isolation valves

On figure 2, V1 to V4 represent isolation and purging valves. The key specifications for those valves are:

- metal bellow or diaphragm valves (no packing)
- 1/8" tube compression fitting for port connections
- leak rate : 4×10^{-9} atm cc/sec
- stainless steel

This type of valve is available from Parker or NUPRO Company. The V1 valve is used to isolate the H_2O trap when replacing the gas cylinder and the purging regulator. V2 is used for purging the regulator. See section 3.0 for procedures on purging the pressure regulator.

2.5 Sample traps

Nearly all gas cylinders have trace amounts of water. We found some cylinders with more than 10 ppm of water! Moisture in the detector will not damage it, but will cause drift and instability, which is something that we don't want and neither does the user. Another problem with that moisture is the nitrogen may dissolve in it or may be retained by it. When the ambient temperature of a sample line is increased the nitrogen is released, giving a temporarily higher reading.

The only type of trap that we recommend is the T-3A-R available from Servomex. Almost all of commercially available traps use molecular sieves with 5A or 13X. The use of such traps will introduce a lot of drift and a long response time. Some of this nitrogen will be released when the ambient temperature is increased. Conversely, when the ambient temperature is decreased the nitrogen will be adsorbed by the trap, giving a lower reading.

We recommend one trap per cylinder and one on the sample line. The cylinder traps will last longer than the one on the sample line; normally, calibration cylinders are replaced once or twice a year. The trap is normally good for four cylinders. The one installed on the sample line may be bigger. Your traps must not be exposed to temperature variations.

The traps are easily regenerated by putting them in an oven and reversing the gas flow direction. See technical information included with your trap, or contact Servomex.



3.0 Regulator purging

Regulator purging is an operation that is not always given the attention it deserves in the use of both high-purity gases and calibration gases. It is easy to understand that special precautions are necessary when using theses types of gases.

In order to maintain cylinder integrity and obtain the best results possible, the end user should purge all regulators. It should be remembered that what happens to the gas between the cylinder and its end use is controlled by the quality of the connecting lines and the efficiency of the purging procedure.

Regulator purging is often not done at all, or is done by simply allowing an arbitrary amount of gas to flow through the regulator. However, there is a shortcoming to this method. In virtually all regulators, there are internal "dead" pockets which tend to hold contaminants.

The internal "dead" pockets in a regulator tend to be unaffected by the flow of a purge gas. Better results will be achieved by alternately pressurizing and depressurizing the regulator with the purge gas. This is called dilution purging, or static purging.

The most effective means of purging connecting lines and regulators is by using the dilution purging method. The following procedure refers to figure 3. The first step in dilution purging is to attach the regulator to the specialty gas cylinder. A tee with a valve on the side branch should then be located in the line between the regulator and the instrument (figure 3). This branch should be connected to a vent, while the main trunk runs to the instrument.

The second step is to turn the regulator adjustment knob to the fully closed position (fully clockwise). V1 and V2 must be closed. V1 will stay in the closed position to keep the sample trap and the line filled with clean gas.

Third, open and quickly close the cylinder valve: that will pressurize the inlet side of the regulator to cylinder pressure. It is necessary to quickly close the cylinder valve after each cycle in order to keep downstream contaminants from entering the cylinder until the regulator is fully purged. Wait approximately one minute, and proceed to the next step.

The fourth step is to open V2 to bleed regulator pressure. To avoid introduction of air do not depressurize completely. For example, if your pressure regulator has a maximum outlet pressure of 207 kPa (30 PSI) or 690 kPa (100 PSI), depressurize it to 34 kPa (5 PSI). Then close V2.

Go back to the third step and repeat steps three and four. This cycle should be repeated 7 to 10 times to ensure that the regulator and the connecting line are both properly purged.

When this is done, readjust the outlet pressure of the regulator between 34 kPa (5 PSI) and 69 kPa (10 PSI), venting the excess pressure through V2. Close V2, open V1 and allow flow through the line into the analyzer.

After this procedure, you should have clean gas in your system, and your calibration gas cylinder was not polluted by air.

If you are not using your calibration gas cylinder for a long period of time, for any reason, close the cylinder valve.



Figure 3

4.0 Avoiding pitfalls

Intended as practical guidelines, here are some rules of thumb.

Use stainless steel lines. "Plastic" type lines must not be used when you are working with ppm level gases.

- 1. Use stainless steel compression-type fittings. Avoid pipe threads with Teflon tape sealant. When installing compression-type fittings, be sure that the outside diameter of the S.S. tubing has no scratches. Clean the outside O.D. of the tube and ferrules with acetone. Do not overtighten the fitting. Normally 3/4 to 1 1/4 turn are enough for Swagelok fittings.
- 2. Use stainless steel pressure regulators. Neoprene and Buna-N type diaphragms, will allow atmospheric contaminants to diffuse in your system.
- 3. Avoid the use of rotary stream selection valves. If you have no choice, use a valve with the smaller orifice available to avoid cross flow port contamination; furthermore, install on particle filter on each inlet port. However, please remember that this is not recommended by Servomex Canada.

- 4. If you are installing a SERVOMEX ppb analyzer, all compression tube fittings must be replaced with metal-to-metal seal fittings, (so called VCR[®]-type fittings). Tubing size will be 1/4" electropolished stainless steel. All accessories (i.e. sample trap, valves, filters) must have VCR[®] fittings. The ppb trace N₂ in argon analyser is supplied with such fittings.
- 5. Avoid the use of quick connectors to temporarily connect cryogenic trucks or other sample points. Use CGA-type fittings and reduce to 1/8" O.D. stainless steel tubing. Keep your sample line on purge with DRY and particle-free gas when they are not in use.
- 6. Don't use SNOOP® type liquid for finding leaks on such systems. If you have leaks, the liquid will go inside the system. It will take a long time to desorb that moisture from the lines.

APPENDIX 2 / ANALOGUE AND DIGITAL OUTPUTS

DIGITAL OUTPUT / ALARM OUTPUT OPTION

The digital output permits the transmission of the range in use and, also, the transmission of the analyzer status (internal alarms).

This is done by using four (4) dry contact outputs, labeled S1, S2, S3, ST. S1 is for 0-1 ppm, S2 is for 0-10 ppm and S3 for 0-100 ppm. The contact is closed to indicate the range in use.

The status (ST) contact output is always closed when the power is on, and no internal alarm is active. The contact will open when a problem occurs in the analyzer (see user's manual for more details), or if the power is off.

See attached drawing on the next page for the diagram of the digital output board. There is a 1 amp fuse on the digital board.

If you only have the standard digital output, there is no relay installed on the board for alarms or auto calibration options. One side of each relay is connected to a terminal identified "C" on the analyzer back panel terminal strip. The other side of the contact relays are labeled S1 (0-1 ppm), S2 (0-10 ppm), S3 (0-100 ppm).

There are snubber circuits installed on the digital board to protect the contact of relays.

See drawings on the following pages for external wiring information.

The rating of the relays is: 30 Vrms, 42.4 V peak or 60 Vdc, 1 Amp maximum

To comply with immunity and emissions requirements of the European Community Directive on EMC:

- The mains cable shall be unscreened.
- A multi-core cable with overall screen shall be used for connections to the relay output terminals;
- A twin core cable with overall screen shall be used for connection to the mA output terminals;
- The screens of the relay and mA output cables shall be connected to analyzer enclosure. The remote end of screens shall be open circuit.
- All I/O cables and sample pipes are formed into a bundle and routed away from analyzer enclosure.

ALARM OUTPUT OPTION

With this option, two digital dry contact outputs are available for process alarms. These contacts are always closed for fail safe purposes. They are connected to a terminal strip labeled "AL1" for alarm #1 and "AL2" for alarm #2. One side of each relay the output is connected to the "C" terminal, on the rear panel terminal strip. They share the same fuse used by other digital outputs. The total current of all loads connected to digital output i.e. status, range in use; and alarms must not exceed: 30 Vrms, 42.4 V peak or 60 Vdc, 1 Amp maximum. The alarms output contacts are protected with snubber circuits mounted on the digital output board inside the analyzer.

When the ppm value of the sample gas exceeds the set point of alarm #1 or alarm #2 their respective contact will open.

To enter the set point value for alarms you should go in the CONFIGURATION MENU, at page 2 (to access page 2, press **F3** labeled NEXT on page 1).



FIGURE 1 : CONFIGURATION MENU, page 2

Pressing F2 will bring up the line for entering the alarm set point for alarm #1. See figure 2 below.

<<CONFIGURATION MENU>> Page 2
F1:mA failure mode (High/Low/Off): HIGH
Input Alarm#1:95.0 Actual:100.0
F3:Alarm #2 Set Point:100.0 PPM F4:RET

FIGURE 2: ENTERING ALARM #1 SET POINT

Enter your value in ppm with the numerical keys and confirm with the "E" key for ENTER. Once the ENTER key is pressed, the value will be activated immediately and this value will be displayed under ACTUAL. To exit the alarm #1 input function, press F4. This will bring up the original page 2 of CONFIGURATION MENU (fig. 1).

To enter the set point for alarm #2, follow the same procedure as above, but press **F3**, this will bring up the following display.



FIGURE 3: ENTERING ALARM #2 SET POINT

When you have finished entering your value for alarm #2, pressing F1 brings up the original page 2 of the CONFIGURATION MENU (fig. 1). Pressing F4 brings the first page of the CONFIGURATION MENU and pressing F4 again brings back to MAIN MENU.

The values you enter must be in ppm. You may enter any value from 0 to 100 ppm, for any alarm. Alarm #1 may have a set point higher or lower than alarm #2 and vice versa.

The digital contact output will open when the ppm value is equal or higher than the set point value. The contact will close when the actual ppm value will go under the set point minus 0.1 ppm. This hysterisis will avoid oscillation.



External connections



WARNING

The milliamp output, and Ethernet and RS-232 terminals (where fitted) are separated from the analyzer mains circuits by reinforced insulation. The terminals must only be connected to circuits that are themselves separated from mains voltages by at least reinforced insulation.

Circuits connected to terminals S1, S2, S3, ST, AL1 and AL2 shall only be powered by supply connected to terminal C.



WARNING

The digital output terminals are separated from analyzer mains circuits by reinforced insulation. The terminals must only be connected to circuits that are themselves separated from mains voltages by at least reinforced insulation.

To comply with immunity and emissions requirements of the European Community Directive on EMC:

- The mains cable shall be unscreened.
- A multi-core cable with overall screen shall be used for connections to the relay output terminals;
- A twin core cable with overall screen shall be used for connection to the mA output terminals;
- The screens of the relay and mA output cables shall be connected to analyzer enclosure. The remote end of screens shall be open circuit.
- All I/O cables and sample pipes are formed into a bundle and routed away from analyzer enclosure.

WARNING



The cable connected to the Ethernet port shall not be longer than 30 m, nor shall it leave the building in which the analyzer is installed without suitable isolation, nor shall it be routed outside of the building in which the analyzer is installed.

WARNING



The cable connected to the relay output terminal shall not be longer than 30 m, nor shall it leave the building in which the analyzer is installed without suitable isolation, nor shall it be routed outside of the building in which the analyzer is installed.

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WARNING



The cable connected to the mA output terminal shall not be longer than 30 m, nor shall it leave the building in which the analyzer is installed without suitable isolation, nor shall it be routed outside of the building in which the analyzer is installed.



WARNING

The rear terminal connector is protected against ESD by a protective plastic cover. When the plastic cover is removed to connect a wire to the terminal, operator shall be properly grounded to earth.

All relay output terminals are rated: 30 Vrms, 42.4 V peak or 60 Vdc.
APPENDIX 4 / RS-232C

RS-232C SERIAL COMMUNICATION OPTION

Introduction

With this option installed, this analyzer retransmits the operating parameters and process values to a remote computer through a serial link. The computer is connected to the analyzer by the mean of a DB-9 RS-232C (Null-modem) cable. The computer must have appropriate software to read the data transmitted by the analyzer. Any commercial software can be used to receive the data.

1. Description

1.1. Hardware

The RS-232 port on the analyzer is driven by an opto-isolator/convertor.

It is necessary to isolate the analyzer C.P.U. board and its power supply from spurious interferences and transients otherwise these sources of interference could interact with the analyzer and could even damage sensitive parts.

Another reason to provide isolation is to break any ground loop between the remote computer and the analyzer C.P.U. board. Such ground loops could cause interference and noise to the highly sensitive analogic electronic circuits of the analyzer.

The DB-9 serial connector mounted on the analyzer rear panel is a special one.

All precautions are taken to give a reliable serial transmission, even if the opto isolator/converter system provides a barrier against noise, transient and ground loop, it cannot support high voltage differences between two different floating systems. For this reason, the remote computer and the analyzer must be properly grounded (ground at same potential).

The standard RS-232C specifications stipulate a maximum distance of 15 meters between two systems. But, if you are using good communications cable (good shielding and low capacitance) longer distances may work successfully.

If you want to use a remote computer located far away from the analyzer (up to 1200 meters), a RS-422 converter should be used to drive the communication lines.

A RS-232C null modem DB-9 cable is supplied with the analyzer. This allows direct connection to a personal computer serial port.

The communication parameters of the analyzer are:

- Baud rate: 9600
- Parity bits: none
- Data bits: 8
- Stop bits: 1

1.2. Transmission protocol

The different parameters are transmitted in the following order:

ppm value sign, ppm value, **TAB**, flow, **TAB**, flow counts, **TAB**, cell counts **TAB**, status and range, **TAB**, checksum, **CR**.

Each parameter is formatted as followed:

- **PPM value sign**: 1 byte equal to "+" or "-" in ASCII, depending on the polarity of the ppm value;
- **PPM value**: 6 bytes in ASCII (3 digits before the dot and two after);
- **Flow**: 6 bytes in ASCII (3 digits before the dot and two after);
- Flow counts: 8 bytes in ASCII;
- Cell counts: 8 bytes in ASCII;
- **Status and range**: bitwise (logical on 8 bits);

| bit 7: alarm 2 | 1 if alarm 2 is ON |
|----------------------------|---|
| bit 6: alarm 1 | 1 if alarm 1 is ON |
| bit 5: low flow | 1 if there is a low flow error |
| bit 4: plasma status | 1 if there is a plasma off error |
| bit 3: system status | 1 if there is a low flow, plasma off, underscale or |
| | overscale error |
| bit 2, and 0: range in use | 001 = range 1 (ex: 0-1 ppm) |
| | 010 = range 2 (ex: 0-10 ppm) |
| | 100 = range 3 (ex: 0-100 ppm) |

Each value is separated by a TAB character. The end of one transmission (packet or frame) is indicated with a carriage return.

The checksum is the arithmetic sum of every transmitted byte (excluding TAB bytes). The checksum may be used to verify the data integrity.

Example : the ppm value is 40.1, the flow is 75.0 ml min⁻¹ (75 cc/min), the flow counts are 8388600, the cell counts are 190011, the range used is the first one (0-1 ppm), there are no alarm, but there is a low flow error.

The data sent in ASCII is:

| <u>Data</u> | Check sum |
|---|---------------------|
| "+", "NUL", "4", "0", ".", "1", "0", | 286 |
| "TAB", "NUL", "7", "5", ".", "0", "0", | 250 |
| "TAB", "NUL" "8" "3" "8" "8" "6" "0" "0" | 369 |
| "TAB", "NILL" "NILL" "1" "0" "0" "0" "1" "1" | 200 |
| "TAB", | 300 |
| ")", "TAB", | <u>41</u> |
| «1», «)», « <i>A</i> », «6», | Total : 1246 |
| "CR". | |

APPENDIX 4

APPENDIX 5 / WEEE

Product disposal in accordance with the Waste Electrical and Electronic Equipment (WEEE) Directive

The label shown in Figure 1 is fitted to the analyser.



Figure 1 The WEEE label

This label identifies that:

- The analyser is considered to be within the scope of the Waste Electrical and Electronic Equipment (WEEE) Directive.
- The analyser is not intended for disposal in a municipal waste stream, but shall be submitted for material recovery and recycling in accordance with the local regulations which implement the WEEE Directive.
- For additional information and advice on the disposal of the analyser in accordance with the requirements of the WEEE Directive, contact Servomex at <u>info@servomex.com</u> or your local Servomex agent.
- If you send the analyser to Servomex or your local Servomex agent for disposal, the analyser must be accompanied by a correctly completed decontamination certificate.

APPENDIX 6 / APPLICATION NOTES

SAMPLING LINE SIZE, (AN-01)

It is a common practice in air separation plants to use 1/4" O.D. line for sampling system. In older installation it is also common to have sample pressure regulator close to the analyzer sample inlet. This can lead to long lag time limiting the speed of response of an analytical system. For example, sample line of 100 feet of 1/4" O.D. with .190" I.D. (typical copper line) has an internal volume of .02 cubic feet. If the sample flow is 1 SCFH (475 sccm) and we assume the line to be at atmospheric pressure, it will take 1 minute and 12 sec. to travel down this line. If the same line is made of 1/8" O.D. with .085" I.D., the internal volume becomes equal to .004 cubic feet. This is 5 times less volume. It will take 24 seconds to travel down the line with 1 SCFH. We have assumed a line pressure equals to the atmospheric one. If the line is pressurized at 1 atmosphere there will be twice the volume of gas into the line so, twice the time will be required for a sample to go through this line.

One may thought to increase the sample flow through the $\frac{1}{4}$ " O.D.line to overcome this problem. If a bypass flow is set to 10 SCFH the time will be decrease by a factor of 10. But after one year of operation this result in 87600 cubic feet of gas thrown away. If this gas is pure argon, this gives around 97 cubic feet of liquid. This is also equivalent to 350 gas cylinders (250 cubic feet size cylinder). The older type of analyzer system for trace nitrogen measurement uses 2 to 4 SCFH of sample flow (for silent electric discharge type) or 1.48 cubic feet (700 sccm) for ion mobility type. Servomex Canada analyzer works with a default sample flow of 75 sccm. The flow can be set as low an 25 sccm if required.

In conclusion, we are recommending the use of 1/8" O.D. stainless steel line for sampling lines. Furthermore, 1/8" O.D. line are available in coil of 500 or 1000 feet for long run. There is no need for fittings or welding. Also, installation cost is minimum since 1/8" O.D. line are easily installed. A sample line of 100 feet made of 1/8" O.D. and .085" I.D. connected to a nitrogen source at 1 PSIG and vented to the atmospheric pressure will have a flow of 475 sccm (1 SCFH). Well enough to supply sample gas to a K3000. It will require 10 psig for 1000 feet long sample line.

The sample pressure regulator must be installed as close as possible from the sample connection point. The pressure will be adjusted to the minimum value required to have the proper flow into the analyzer. Such sampling system will have a faster response time, **<u>better leak integrity</u>**, less operation cost.



THE IMPORTANCE OF REGULATOR PURGING, (AN-01)

Here are some quick calculations to help you understand why it is so important to have some techniques to evacuate the air from pressure regulators when replacing calibration cylinders.

For example, let's take a pure argon cylinder of size 44 (i.e. $6m^3$ of gas). On this cylinder there is a double stage pressure regulator with two pressure gauges, CGA connector, and an outlet isolation valve. Lets assume that the internal volume of this pressure regulator is 100 CC ($\pm 10\%$). When installing this pressure regulator on the cylinder, the internal volume is occupied by the atmospheric air i.e. 78.2% N₂, 20.9% O₂, 0.9% Ar, moisture, CO₂, etc.

When the regulator is screwed in place on the pressure regulator, the air still is trapped inside the regulator. If you open the valve on the cylinder to pressurize the regulator, and there is no or little flow through the regulator, the air trap inside the regulator will diffuse inside the argon cylinder. The shock caused by the quick pressure build up inside the regulator helps to speed up the diffusion process.

So, assume no flow (worst case), we have the following situation:

100 CC of air and atmospheric impurities added to 6 m³ of pure argon (assuming perfect argon i.e. no impurities at all). This leads to the following calculation:

$$\frac{100 \text{ x } 10^{-6} \text{ m}^{3} \text{ (i.e. 100 CC) of Air}}{6 \text{ m}^{3} \text{ argon}} = 16.66 \text{ x } 10^{-6}$$

and

So the dilution ratio is 16.66×10^{-6}

 $16.66 \text{ x } 10^{-6} \text{ x } 78.2\% \text{ N}_2 = 13 \text{ ppm of } \text{N}_2$

and 16.66 x 10^{-6} x 20.8% $O_2 = 3.5$ ppm of O_2

So starting from a pure argon cylinder and just by a bad pressure regulator purging procedure, we've got an argon cylinder with 13 ppm of N_2 and 3.5 ppm of O_2 . These impurities will be added to any other impurity in the cylinder. This situation makes it difficult or even impossible to get accurate calibration In some cases, we received phone calls from people claiming that the zero cylinder had higher readings than the span cylinder....

So Be Aware !!!!!

LEAK FINDING PROCEDURE (AN-05)

Experience has shown that bad analysis results often come from inboard contamination following from leaks in the tubing bringing the sample to the analyzer's detector.

Using the right procedure, the trace Nitrogen analyzer SERVOPRO PLASMA can self diagnose the presence or absence of contaminating leaks.

We first have to understand that the gas circuit is divided in two major zones. The proportional valve located inside the flow module is the boundary separating these two zones. So the first zone is constituted of all the tubing, valves, sampling system, pressure regulators etc. located between the gas source (gas cylinder, gas tank, truck tank, etc) and the inlet off the flow module located inside the analyzer. The second zone is constituted of everything located between the flow module outlet and the detector (the cell module) inlet, including the gas conditioning module. There is a specific procedure for checking leaks of each zone. The main difference between our two zones is that the gas pressure in the first zone (upstream the flow module) is pretty high (usually 5 to 15 PSIG) while the gas pressure in the second zone is just a little bit higher than atmospheric pressure (0.1 or 0.2 PSIG).

The main difference between the two leaks finding procedure will be the following one:

- In zone 1, we will play with the gas pressure (generally with a gas regulator) and check changes in analyzer reading.
- In zone 2, we will play with the analyzer flow and watch for changes in the analyzer reading.

We recommend doing both tests before trying to fix leaks.

To run both tests, we will watch the changes in ppm value. Of course, the results will be reliable providing that the analyzer already has a reliable calibration. New analyzers are shipped pre-calibrated so we can use this pre-calibration to run the tests. If the calibration has been fouled up by calibrating with contaminated calibration gas, we will need to watch the raw signal from the detector i.e. the <u>cell counts</u> in the diagnostic menu.

TEST FOR ZONE 1

This test will mainly consist in changing the line pressure from normal operating pressure (usually somewhere between 5 to 15 PSIG) to a pretty low pressure i.e. < 1 PSIG. To achieve this, you drop the pressure low enough in such a way that the analyzer flow will slightly drop from its normal 75 cc flow to, let's say 70 cc. The analyzer flow should stay that much low, \approx 70 cc, due to the fact that the inlet pressure is not high enough to supply the whole normal flow. We know at that moment that the line pressure is well below 1 psi, usually around 0.6 psi. If there is no leak, there will be no noticeable change in analyzer reading or cell counts. If the signal (ppm or cell counts) goes high and after a while resumes to a value close to the one we had before dropping the pressure, this is symptomatic of a dead leg or dead volume. If the signal goes high (could be a 5 or 10 ppm step or many thousand cell counts) and stays high, there is a leak for sure.

Before trying to fix leaks, this test can be done using different gas sources i.e. zero calibration gas, span gas, normal sample etc. Of course if the same leak is observed for any of the gas sources we will look for the source of this leak in a part of the gas circuit which is common to all the streams and so on. We have to notice that during this test, the conditions have not changed in the zone 2 i.e. downstream the flow module; except if we have caused an important flow change by dropping the pressure too low. A good system will not show a noticeable change in signal (<0.5 ppm) while running it at low or high pressure. Of course we easily understand that presence of leaks will bring unreliable calibrations, erratic sample analysis results and all the nightmares that come with all that. The only solution is a good tubing and sampling system.

TEST FOR ZONE 2

Prior to run this test, make sure that the analyzer is running under gas since at least 2 or 3 days. Doing this test on a newly installed analyzer could give false results since the analyzer's dry down is not done yet. Therefore this test will be simply done by changing the flow and checking for signal change. If there is a leak we will observe mainly a leak dilution phenomenon. Usually a leak brings in a certain amount of impurity, no matter how high or low is the flow in the tubing. Since we will run this test with the zero gas, presence of a leak will be confirmed by an increase in reading when dropping the flow (less diluted contaminant) or decrease in reading when increasing the flow.

N.B.: The inlet pressure should be normal (between 5 and 15 PSIG) when running this test otherwise with a low inlet pressure we would observe the dilution of a leak that could be located in zone 1.

N.B.: to change the flow, you have to go to configuration menu (F1), push the digit keys ex. 2, 5, and push E for Enter to get a flow of 25 ml min⁻¹ (25 cc/min).

When changing the flow from 75 ml min⁻¹ (75 cc/min) to 25 ml min⁻¹ (25 cc/min) an increase in the reading of no more than 0.25 ppm should be observed. If the presence of a leak is detected try to retighten each fitting one by one (4 nuts total), and wait 10 seconds between each tightening to see if there is a change in the reading.

One could ask how come he should check these fittings since the analyzer's manufacturer should have installed them correctly. It is a fact that when a SERVOPRO CHROMA leaves the factory it has been thoroughly checked and there was no leak inside since we are aware of leak problems and we know very well how to track them. But here is what experience shows about compression fittings:

- When fittings are newly installed according to manufacturer's specification (Swagelok, Parker, Valco, etc) they most of the time show no leak, except if some irregularities are present (scratched tubing, dirt or dust on the ferrule, etc.). Anyway, these possible problems have been checked and solved at Servomex Canada factory (regarding the analyzer itself).

So a properly installed fitting, when tightened, is preloaded i.e. there is a permanent pressure applied on the front ferrule against its seat providing therefore a good sealing. Overtime, in the real life, what happens?

During shipping, transport, installation, operation, if too much vibration occurs, the ferrule preload can release and the leak appears. Other factors also affect this phenomenon like using tubing having too much thin wall, which accelerates apparition of leaks. However, let's not be too pessimist. Experience has shown us that easily 95% of the fittings will work great for a very long time. We just have to be aware that presence of leak is always possible. The only important point is to know how to check if leaks are present and how to solve the problem.

P.S.: Servomex also manufactures a patented sampling system using a principle that eliminates almost 100 % of the leaks inside it.

LEAK FINDING TEST

COMPANY NAME: ______TEL:_____

CONTACT PERSON:______FAX:_____

ANALYZER'S SERIAL NUMBER: _____E-MAIL: _____

APPROXIMATE TIME ELAPSED SINCE START-UP: _____

ZONE 1 TEST

WE SUGGEST TO USE THE ZERO CALIBRATION GAS TO RUN THE TEST PPM VALUE OF THE GAS BEING USED: _____ ppm

| | PRESSURE READ ON REGULATOR | FLOW DISPLAYED BY ANALYZER | PPM DISPLAYED | CELL COUNTS |
|--------------------|-------------------------------|-------------------------------|---------------|-------------|
| NORMAL PRESSURE | | | | |
| LOW PRESSURE | | | | |

ZONE 2 TEST

WE AGAIN SUGGEST TO RUN THIS TEST WITH THE ZERO CALIBRATION GAS PPM VALUE OF THE GAS BEING USED: _____ ppm

| | PRESSURE READ | FLOW DISPLAYED | PPM DISPLAYED | CELL COUNTS |
|------------|---------------|----------------|---------------|-------------|
| | ON REGULATOR | BY ANALYZER | | |
| 75 CC FLOW | | | | |
| 25 CC FLOW | | | | |

OVERALL PERFORMANCE VERIFICATION

| | PPM VALUE | CELL COUNTS |
|-----------------------|-----------|-------------|
| ZERO CALIBRATION GAS: | | |
| SPAN CALIBRATION GAS | | |

It is also a good idea to verify for possible changes in analyzer reading using the span gas and do again the tests for zone 1 and zone 2. So you can optionally fill in the two following tables.

ZONE 1 - TEST WITH SPAN GAS

PPM VALUE OF THE GAS BEING USED: _____ ppm

| | PRESSURE READ ON REGULATOR | FLOW DISPLAYED BY ANALYZER | PPM DISPLAYED | CELL COUNTS |
|--------------------|-------------------------------|-------------------------------|---------------|-------------|
| NORMAL PRESSURE | | | | |
| LOW PRESSURE | | | | |

ZONE 2 - TEST WITH SPAN GAS

PPM VALUE OF THE GAS BEING USED: _____ ppm

| | PRESSURE READ ON REGULATOR | FLOW DISPLAYED BY ANALYZER | PPM DISPLAYED | CELL COUNTS |
|------------|-------------------------------|-------------------------------|---------------|-------------|
| 75 CC FLOW | | | | |
| 25 CC FLOW | | | | |

APPENDIX 6

APPENDIX 7 / AUTO-CALIBRATION OPTION

AUTOMATIC CALIBRATION OPTION

NOTE TO THE READER:

Before proceeding with this section you must first have read and understood the User Manual. Furthermore the analyzer must be properly started-up. Good calibration depends on hardware used in sampling system.

1) Gas Circuit Description

Analyzers with this option are equipped with 3 solenoid valves. These valves are made of stainless steel and have excellent characteristics for inboard contamination. These valves are used to select between sample, zero and span gas. All gas connections are made on the analyzer rear panel. All gas inlets are protected with a 2 micron frit mounted inside the bulkhead. These are acting as particle filters. Please see the gas flow path drawing on page 6.

When the analyzer is "ON", there is always one and only one valve, over the gang of three, being "ON". So the gas flows through this valve to the common outlet manifold and then to the bulkhead labeled "Trap In". At the same time, the manifold also supplies gas to the two other unselected valves and the gas exits the valves through the purge port, to a common header, this one being connected to the bulkhead labeled "Purge Vent". Purging the valves this way ensures that there is no unswept dead leg that would bring contamination of the sample and even eliminates the effect (contamination) of an eventual leaking valve. So, as soon as the analyzer is "ON", the three valves are swept by the selected gas and there is a flow going to the "Purge Vent" bulkhead. The amount of this flow is as the following table:

| Inlet Gas Pressure | Purge Gas Flow |
|--------------------|--|
| 34 kPa (5 PSIG) | 40 ml min ⁻¹ (40 cc/min) |
| 69 kPa (10 PSIG) | 125 ml min^{-1} (125 cc/min) |

We recommend keeping the inlet pressure between 34 kPa (5 PSIG) to 69 kPa (10 PSIG).

2) System Operation

Calibration of the analyzer may be done manually or automatically at a time interval defined by the user. In both cases calibration gases are selected with SOV1, SOV2 and SOV3. This section explains how to execute a calibration manually and automatically.

NOTE: Zero and span gas values must be set prior to any calibration with a span value bigger than the zero value.

2.1 Entering Zero and span gas values

From the main menu, press F2 to enter the calibration menus of the analyzer.



FIG 1: ANALYZER MAIN MENU

This will bring you to the CALIBRATION MODE SELECTION MENU as shown below. For the moment, proceed to the CALIBRATION MAIN MENU by pressing F3.

| < <calibration menu="">></calibration> | |
|---|--------|
| F1:Calibration Mode: MANUAL | |
| F2:ACTUAL GAS: SAMPLE | |
| F3:NEXT | F4:RET |
| | |

FIG 2: CALIBRATION MODE SELECTION MENU

| < <calibration menil="">></calibration> | |
|--|---------|
| F1:Zero | F2:Span |
| F3:Calibrate | F4:RET |
| | |

FIG 3: CALIBRATION MAIN MENU

From the CALIBRATION MAIN MENU, pressing F1 will enable you to enter the zero gas value.

<<CALIBRATION MENU>> Input ZERO value: 0.00 Actual Value:0.00 Value must be in PPM F4:RET

FIG 4: SETTING THE ZERO GAS VALUE

| < <calibration menu="">></calibration> | |
|---|--------|
| Input SPAN value: 0.00 | |
| Actual Value:0.00 | |
| Value must be in PPM | F4:RET |
| | |

FIG 5: SETTING THE SPAN GAS VALUE

With the numeric keys, enter the zero gas value in PPM followed by the E key. The actual value of the zero gas is then updated in front of "Actual value:". To set the span gas value, go back to the CALIBRATION MAIN MENU by pressing F4 and then press F2. With the numeric keys, enter the span gas value in ppm followed by the E key. The actual value of the span gas is then updated in front of "Actual value:". The span and zero gas values for calibration are now set and a manual or an automatic calibration can be performed. Press F4 to get out of this menu and to go back to the CALIBRATION MAIN MENU.

2.2 Manual Calibration

You have to select the "MANUAL" mode if you want to perform a manual calibration. To do so, go to the CALIBRATION SELECTION MODE MENU (FIG 2) by pressing F2 from the MAIN MENU (FIG 1). Toggle between calibration modes by pressing F1 until "MANUAL" is displayed. Then, toggle between gases by pressing F2 until "ZERO" is displayed (SOV2 is energized).

Be sure that the zero and the span gas values are set (see the previous section) and let the zero gas flow in the instrument for a while. The zero gas must flow long enough to be sure that the previous gas is purged away and that the system has returned to equilibrium. When working with Argon, the purge time is quite fast. However, Helium is a poor purge gas. 20 to 30 minutes is then recommended for a purge in helium. You may watch the cell counts in the DIAGNOSTIC MENU to make sure that the signal is stable before executing a calibration.

When done, press F3 from the CALIBRATION MAIN MENU (FIG 3) to enter the CALIBRATION FACTOR CALCULATION MENU.



FIG 6: CALIBRATION FACTOR CALCULATION MENU

When the cell counts are stable, press F1 to re-zero the analyzer with the gas currently flowing. Selecting F2 will confirm the rezero and calculate the calibration factors. The analyzer will display the message "PLEASE WAIT" in front of "CALIBRATION STATUS" and finally, this message will be replaced by "ZERO DONE" to confirm the zero calibration. If the zero and span values were not set properly, "WRONG CAL. GAS VALUE" will be displayed. Change the zero and span values, with the span value bigger than the zero value, and try again. If a right re-span was previously performed before this re-zero, "CALIBRATION DONE" will be displayed. <<CALIBRATION FACTOR CALCULATION>> Measure:0.00 PPM Sample Flow:75.0 CC RE-ZERO(F2-Yes F1-No) SPAN NOT SET Calibration status:

FIG 7: CALIBRATION FACTOR CALCULATION MENU

To re-span the instrument, a re-zero must have been performed before. You must then allow the span gas to flow in the analyzer. If your analyzer has the auto-calibration option, select "SPAN" as the gas source from the CALIBRATION MODE SELECTION MENU (FIG 2) by using the F1 key.

When this is done, let the gas stabilize, go back to the CALIBRATION FACTOR CALCULATION MENU and press F2 to respan the instrument. Selecting F2 will confirm the re-span of the instrument and will start the factor calculation with the gas currently flowing. "CALIBRATION DONE" must be displayed next to "CALIBRATION STATUS". From that point, you may press F4 several times to exit the calibration menus.

NOTE: "WARNING: actual selected gas is" followed by the gas currently selected is displayed if this gas is not the zero gas (SOV2 energized) when a re-zero is about to be performed or if it is not the span gas (SOV3 energized) when a re-span is about to be performed.

The previous procedure was intended for a manual calibration in which the user controls the various steps of calibration. To enable the analyzer to recalibrate by itself, read the following section about automatic calibration.

2.3 Automatic Calibration Mode

In this mode, the analyzer executes calibration (i.e. re-zero and re-span) at fixed intervals defined by the user. This duration is called "TIME BETWEEN CALIBRATIONS". When this time interval is reached, the analyzer opens the status contact and energizes solenoid SOV2 to allow the zero gas to flow into the instrument. This gas flows during "TIME ON CAL.GAS" which is also defined by the user. When the "TIME ON CAL. GAS" is reached, a re-zero (with the zero gas) is automatically performed. The span gas is then selected for a "TIME ON CAL. GAS" duration after which a re-span is automatically executed (with the span gas). Finally, the analyzer automatically re-selects the sample gas and closes back the status contact. Calibration timers are re-settled at zero and the cycle is repeated again and again.

If the 4-20 mA analog output of the analyzer is set to "HOLD" mode in the CONFIGURATION MENU, the analyzer waits for the "TRANSFER DELAY" before refreshing the 4-20 mA after the re-span has been performed. This delay gives the time to purge the span gas out of the system and avoid undesired bumps on the 4-20mA trending. The analog output remains at the value calculated just before the beginning of the calibration.



FIG 8: TRANSFER DELAY

To configure the analyzer to perform automatic calibrations, first set the different timers by going to the HIDDEN MENU. To access the HIDDEN MENU, go to the MAIN MENU and enter the secret code by pressing 1, 2, 3, and E consecutively on the keyboard. This brings up the HIDDEN MENU. Press F3 repetitively until the screen of figure 10 is displayed.

| < <starting count="">></starting> |
|--------------------------------------|
| Actual starting count value:25000 |
| Input new value: |
| F3:NEXT |
| |



<<AUTO CALIBRATION PARAMETERS>>> F1:Time between calibration: 168.0 Hrs F2:Time on cal. gas:10.0 Min F3:NEXT F4:RET

FIG 10: AUTOMATIC CALIBRATION TIMERS

Minimum: 0 Maximum: 90000.0 (between calibration) and 25000.0 (on calibration gas)

Set the "TIME BETWEEN CALIBRATIONS" by pressing F1 and entering the value in hours followed by the E key. Press F2 and enter a value in minutes followed by the E key to set the "TIME ON CAL. GAS".

To set the "TRANSFER DELAY", advance in the HIDDEN MENU by pressing F3 until this page is displayed:



FIG 11: ANALOG OUTPUT HOLD TO TRACK TRANSFER DELAY

Minimum: 0 Maximum: 90000.0

Enter a realistic value based on your system configuration, typically around 5 minutes. You should make sure that this value is big enough to allow the sample gas to purge the span gas in the system.

When the proper timing parameters are entered, automatic calibration can be turned on from the CALIBRATION SELECTION MODE MENU (FIG 2).

Toggle the calibration mode until "AUTOMATIC" is selected by pressing F1 in the CALIBRATION SELECTION MODE MENU. The calibration timer ("TIME BETWEEN CALIBRATIONS") begins to count down from the moment you put the analyzer in automatic calibration mode. You may track the automatic calibration process and monitor both timers ("TIME BETWEEN CALIBRATIONS") from the DIAGNOSTIC MENU. The value displayed next to

"ELAPSED TIME SINCE LAST CAL." is the number of hours since the analyzer was put in automatic calibration mode. The value displayed next to "MINUTES ON CAL. GAS" is the number of minutes the analyzer has spent on calibration gas (zero or span) since the zero or span gas was selected. "0.0" is displayed if the analyzer is not currently performing a calibration.

Furthermore, in the RUN MENU, the gas currently selected is displayed next to "Gas: ". Note that during an automatic calibration sequence, the status contact remains OPEN.

When the analyzer is in the RUN MENU during an automatic calibration, the word "AUTOCALIB" will be displayed instead of the measure in PPM.

NOTE: In order to get accurate readings when using auto-calibration mode, the user must enter proper timing values in accordance with the sampling system. Longer purging time are better than shorter ones.

NOTE: Only one alarm can be used with the auto-calibration option.



APPENDIX 8 / TRACE NITROGEN IN HELIUM VERSION

1. User Manual

This User Manual applies to both versions of SERVOPRO PLASMA analyzers, i.e. Argon or Helium. Since the most popular version is Argon, all published manuals refer to Argon as background gas. You have to replace the word Argon by Helium when you are reading this manual.

2. Flow Control System

The flow control valve used in the SERVOPRO PLASMA trace Nitrogen analyzer for Helium background is the same as used in the Argon version. This valve is not a tight shut off valve. Its purpose is to maintain the flow at the default flow set point, i.e. 75 sccm. It is possible to have a little flow (usually not more than 10 sccm for inlet pressure of 10 psig) with a flow set point value of 0 sccm. You don't have to worry about that, it is normal phenomenon. Also, please notice that the plasma is shut off when the real flow is below 10 sccm. The analyzer should be operated and calibrated with a flow set point of 75 sccm and stable inlet pressure. The recommended inlet pressure range is between 5 to 15 psig, ideal being 10 psig.

3. Leaks

The most critical point in trace Nitrogen analyzer is to avoid the pollution of the sample stream. The leak integrity of the sampling and calibrating system is critical for the performance of the analyzer, no doubt can subsist about that. Be aware that leaks are more critical with Helium than Argon. Furthermore, the sample must be as dry as possible. You should use a molecular sieve 3A trap to keep the sample dry.

4. Working with Helium

It is a challenge to work with Helium in an on line continuous measurement of ppm N_2 . The Helium molecule size and weight are so small compared to N_2 that special attention must be paid to the sampling system in order to get a better stability.

With Argon, the sampling system operating condition may produce good results. The same condition applied with Helium can lead to unacceptable performance.

Most of the time, when sampling Helium, if line temperature pressure and flow vary, reading will also vary. The stabilization time with Helium is much longer than with Argon. Here is why; an often overlooked factor in response speed and signal stability (or the statistic distribution of N_2 molecule in Helium) is the molecular weight of the carrier gas (Helium in this case) relative to the impurity to be measured (Nitrogen). Because Helium gas has a small molecular mass, it is difficult matter for them to move heavier Nitrogen molecules out of the system. So as a rule of thumb, when sampling Helium, do the following:

- ▶ Keep a constant and high flow rate in the sampling line. Exhaust the excess flow through a by-pass rotometer.
- Close to sample point tap or connection, use a small stainless steel electropolish with VCR connection only pressure regulator. The by-pass rotometer with its valve will fix the line flow and the sample point pressure regulator will maintain the line pressure constant.
- The third parameter: temperature. If the line temperature varies, reading will also vary due to change in equilibrium conditions, change in temperature when sampling Helium will cause reading drift and recovery time will be long if sample flow is low. The best result is achieved if you can heat trace the sampling line and maintain the temperature between 60°C to 75°C. The temperature must be constant.
- > Install the supplied trap at the analyzer inlet.

Use only electropolish stainless steel. Electropolishing is an electrochemical procedure that satisfies the deficient ionic sites of surface metallic molecules. These ionic sites strongly attract polar molecules causing a stronger adsorption effect at that site.

APPENDIX 9 / DUAL BACKGROUND ANALYZERS

NOTE TO THE READER

The SERVOPRO PLASMA trace nitrogen in argon or helium analyzer version is shipped configured for argon.

We strongly recommend you read the standard User Manual followed by this addendum. Reading this addendum supposes that you have read and understood the User Manual.

1. User Manual

This User Manual applies to both versions of the K2001 analyzer, i.e argon or helium. Since the most popular version is argon, all published manuals refer to argon as the background gas.

You must read this addendum to know how to select argon or helium backgrounds.

2. Flow control system

The flow control valve used in the SERVOPRO PLASMA trace nitrogen analyzer for helium background is the same as the one for the argon version. This valve is not a tight shut off valve. Its purpose is to maintain the flow at the default flow set point, i.e. 75 sccm. It is possible to have a little flow (usually not more than 10 sccm for inlet pressure of 10 psig) even if the flow set point value is 0 sccm. You don't have to worry about that, it is a normal phenomenon. Also, please notice that the plasma is shut off when the real flow is below 20 sccm for more than 30 seconds. The analyzer should be operated and calibrated with a flow set point of 75 sccm and stable inlet pressure. The ideal inlet pressure range is between 5 to 15 psig.

3. Leaks

The most critical point in trace nitrogen analysis is to avoid the pollution of the sample stream. The leak integrity of the sampling and calibrating system is critical for the performance of the analyzer, no doubt can subsist about this. Be aware that leaks are more critical with helium than with argon.

Furthermore, the sample must be as dry as possible. You should use the supplied trap to keep the sample dry. It is also recommended to have one trap for each calibration cylinder.

4. Configuration and operation

4.1 Introduction

All configuration, calibration, diagnostic and run menus described in the standard User Manual also apply to the argon/helium version of the SERVOPRO PLASMA series analyzer.

There is a supplementary "HIDDEN" menu to select the starting count value and the background operating gas.

4.2 HIDDEN menu access

From the main menu (fig. 1), the one displayed when the analyzer is powered up or simply when you come back to this menu by exiting any other sub menu, enter the following key sequence with the help of the keypad: 1, 2, 3, E. This will bring up the menu showed on fig. 2.

SERVOMEX CANADA K2001 TRACE N₂ ANALYZER MAIN MENU

F1: configuration **F2**: calibration **F4**: run

FIG. 1 MAIN MENU

OPERATING PARAMETERS

ACTUAL STARTING COUNT VALUE : 500 INPUT NEW VALUE :

F3 : NEXT

F4: QUIT

FIG. 2 OPERATING PARAMETERS MENU

The starting count value is the threshold value where the analyzer switches from starting mode to normal mode.

If the cell count value (value from the analog to digital converter; this value may be observed under <u>diagnostic</u> menu) is under the starting count value, a higher power is applied to the cell by the plasma generator. Once the plasma is started, the cell count will be higher than the starting count value and the power will come back to normal. If, for any reason, the cell count value with the normal power applied to the cell is too close or oscillates around the starting count value, the analyzer will go back and forth between starting and normal mode.

To get rid of such a situation, you should enter a new (lower) threshold value.

To enter a new value, just use the numerical key on the keypad followed by enter key ("E" key). The value must be between 0 and 1,000,000 counts.

The default value is normally just what the system needs and, under normal conditions, this value should not be changed.

From this menu, by selecting "F3" for next sub-menu, the following menu is brought up on the display.

BACKGROUND GAS SELECTION ACTUAL GAS : HELIUM Use F1 key for selection F3 : NEXT

F4 : QUIT

FIG. 3 BACKGROUND GAS SELECTION MENU

This menu is used for display and selection of the background gas type. The background gas may be helium or argon.

Using "F1" key toggles between argon and helium. *NOTE*: When selecting helium, the sample flow will be set at 75 sccm. Selecting back argon will set the sample flow set point at 75 sccm. You may change later the sample flow set point in the configuration menu. See User's Manual. After the selection is done, use F3 to go in the next menu or F4 to quit.

Figure 4 below shows the next menu.

| TEMPERATURE COEFFICIENTS | |
|--------------------------|----------|
| F1: OFFSET | |
| F2: GAIN | |
| F3: NEXT | F4: QUIT |

FIG.4 TEMPERATURE COEFFICIENT MENU

The temperature coefficients are defined during bench testing at the factory. These values are used to do temperature compensation for ambient temperature change if required. You should not change this value unless you know how to proceed for temperature bench marking. A value of 1.000 cancels the temperature compensation.

You may enter any value between $\underline{0}$ and $\underline{2}$ with a resolution of 0.001. The offset value will compensate for the baseline drift. If for any reason you suspect excessive temperature drift, please contact us for exact procedures to be followed.

4.3 Operating consideration

WARNING:

Before changing the operating background gas, make sure that you have the selected gas available. It is not recommended to operate the analyzer under argon background gas selected when helium is flowing into the analyzer. The flow loop will try to maintain the flow, but helium does not have the same thermal conductivity as argon. So, in fact, there will be a much higher flow, the velocity may come too high in the flow module (thermal bridge), and damage may occur to this sensor.

5. Calibration and alarms data

Any value entered under calibration menu and all calculated operating parameters will be saved in the memory when you change from argon to helium or vice versa.

Same thing applies for alarm set points if you have this option and temperature coefficient entered in the hidden menu.

So, most of the time, the calibration will stay valid when changing the background gas selection. Of course, a purging time is necessary to clean out argon or helium.

When you change the background gas selection, a new flow table in the software is used. The tuning of the flow loop is also changed. Calibration and alarms are saved in the memory. The signal processing algorithm is also modified accordingly.

There is also some hardware change in the cell design in this version of SERVOPRO PLASMA.

6. <u>Conclusion</u>

To resume, your dual background version analyzer is in fact like two analyzers in the same cabinet. However, there is some differences between argon and helium backgrounds.

- 1) Starting count value will be different
- 2) Default sample flow will be different. Recommended sample flow:

Argon \rightarrow 75 sccm Helium \rightarrow 75 sccm

To get the same system overall response time, the by-pass flow, when in helium will have to be higher.

3) The dual background version is shipped with a metal based moisture trap. If you operate in argon for a long period, get a molecular sieve trap from Servomex Canada .

IMPORTANT

The metal based trap is made with $\frac{1}{4}$ " O.D. S.S. tubing. Don't allow air or high level of O₂ to flow in it because excessive heat would be generated. Install the trap only when all lines are purged. This trap <u>must</u> be installed to get good results when operating under helium. This trap can be shipped back to Servomex Canada for proper regeneration. We strongly recommend to keep one in stock.

4) When changing background gases from argon to helium, or vice et versa, all the previously in use background gases must be purged out before attempting any calibration or expecting any reliable reading. Normally this is done in 24 hours.

APPENDIX 10 COMPLIANCE AND STANDARDS INFORMATION

- The analyzer complies with the European Community "Electromagnetic Compatibility Directive":
 - Emissions: Class B Equipment suitable for use in domestic establishments and in establishments directly connected to a low voltage supply which supplies buildings for domestic purposes.
 - Immunity: "Basic" Considered appropriate to equipment intended for use in domestic, commercial and light industrial environments.
- The analyzer complies with the European Community "Low Voltage Directive", by the application of EN 61010-1 and rated for Over Voltage Category II, Pollution Degree 2.
- The analyzer complies with the Class B digital apparatus requirements of ICES-003 of Canada through the application of EN 55011:2007.
- L'analyseur est conforme aux Conditions B numériques d'appareillage de classe de NMB-003 du Canada par l'application du EN 55011:2007.
- This analyzer complies with Part 15 of the US FCC Rules for Class B equipment. It is suitable for operation when connected to a public utility power supply that also supplies residential environments.
- The analyzer has been assessed to IEC 61010-1 for electrical safety including any additional requirements for US and Canadian national differences.