



K41 100 MICRO CONRADSON CARBON RESIDUE TESTER

OPERATION AND INSTRUCTION MANUAL

REV A

Koehler Instrument Company, Inc.

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Petroleum Testing & Analysis Instrumentation • Custom Design & Manufacturing

CERTIFICATE OF CONFORMANCE

Micro Carbon Residue Apparatus K411XX

This certificate verifies that part number K411XX, Micro Carbon Residue Apparatus, was manufactured in conformance with the applicable standards set forth in this certification.

Specifications: **ASTM D4530**

This unit is tested before it leaves the factory, to ensure total functionality and compliance to the above specifications and ASTM standards. Test and inspection records are on file for verification.

June Kelly

Jesse Kelly
Application Engineer
Koehler Instrument Company

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1 - GENERAL

1.1 - Safety rules

Basic precautions must be observed when using Koehler Instrument Company, Inc. instruments and their accessories.

1. Read and understand in full these instructions prior to use.
2. In the case of damage or dropping of the appliance, have it examined by a qualified technician.
3. Do not touch parts of the appliance which become extremely hot.
4. Do not use with a defective connection cable.
5. Do not allow the connection cable to hang over the edge of a table or touch a hot surface.
6. If an extension lead is necessary, use a cable suitable for the current intensity that will be used. Using a cable of too small a cross-section may cause overheating. The cable must not be twisted or stretched.
7. Disconnect the appliance from the mains power when it is not in use. Never pull on the cable to disconnect it from the mains. Take hold of the plug and remove it from the power socket.
8. Allow the appliance to cool completely before moving it. Wind the cable loosely around it for storage.
9. To avoid danger of electrocution, do not immerse the appliance in water or any other liquid.
10. Do not disassemble the appliance, but have it repaired by a qualified technician when intervention is necessary. Any incorrect assembly may result in electric shocks on restoring it to service. Before using the appliance, the operator must have:
 - * Knowledge of manual methods and experience in the types of tests to be performed.
 - * Knowledge of the risks resulting from handling products and samples.

Users are advised not to handle containers of hot samples. If it is necessary, this operation must be performed with extreme caution.



In all cases, the operator must wear the necessary protective clothing: gloves, goggles, protective garments.

1.2 - Instructions for use

The appliance must be used solely:

- ▶ In conformity to the methods described in the instructions
- ▶ With the accessories supplied and designated in the instructions
- ▶ In accordance with the test and commissioning procedures described in the instructions

During operation, the appliance must under no circumstances be left unsupervised.

The appliance must imperatively be located under an extraction hood with a smoke extraction system: the work room must be adequately ventilated.



An inspection of the gas connections must be carried out at least one a month in order to check satisfactory condition of pipes and their fastenings.



In the case of an incident that could result in damage to the appliance (excess gas pressure, abnormal heating of the appliance, leaking or spillage of a product), an internal and external examination of the appliance must be performed by a competent person.



All maintenance interventions must be performed exclusively by an authorized technician with the appliance at room temperature and switched off (except when it must be switched on: e.g. calibration) and disconnected from the electricity and gas mains supplies.

The maintenance functions described and detailed in the instructions must only be performed by an authorized technician.

The functions and controls linked to the keyboard are clearly explained in the instructions; the user must become familiar with these before handling the appliance.

1.3 - After-sales service guarantee

KOEHLER INSTRUMENT CO. guarantees its products for a period of twelve months from the date of dispatch. During this period, any part with defects in the materials of construction or in operation and which is returned to KOEHLER INSTRUMENT CO. will be replaced free of charge, except for consumables (lamps, printing paper, sample bowl, etc.). Work under guarantee will be performed by KOEHLER INSTRUMENT CO.

If the guarantee work must be performed on site, the travel and subsistence costs will be paid by the user.

Service under guarantee does not apply to an appliance of which the defect is the result of incorrect use, connection to an unsuitable electricity supply, absence of ground, an act of vandalism or caused by the forces of nature (thunder, flooding, earthquake, etc.).

KOEHLER INSTRUMENT CO. does not accept any liability for physical injury or financial loss caused by a defective appliance during the guarantee period, whether under a Service Contract or otherwise.

This guarantee replaces all other official or implicit agreements.

The guarantee will become null and void if the equipment is transported outside the country where it was initially supplied by KOEHLER INSTRUMENT CO.

1.4 - Contact in case of emergency

KOEHLER INSTRUMENT CO. 1595 Sycamore Ave. Bohemia, NY 11716

Ph: 1 631 589 3800 - e-mail: service@koehlerinstrument.com

1.5 - Symbols used



Warning

To be respected scrupulously to avoid any physical injury or damage to your appliance



Maintenance

Supplies important information concerning the maintenance of your appliance



Remark

Provides useful advice for optimum use of your appliance

2 - TECHNICAL ASPECT

2.1 - Scope of application

This appliance is intended for the treatment of samples in compliance with the following standards:

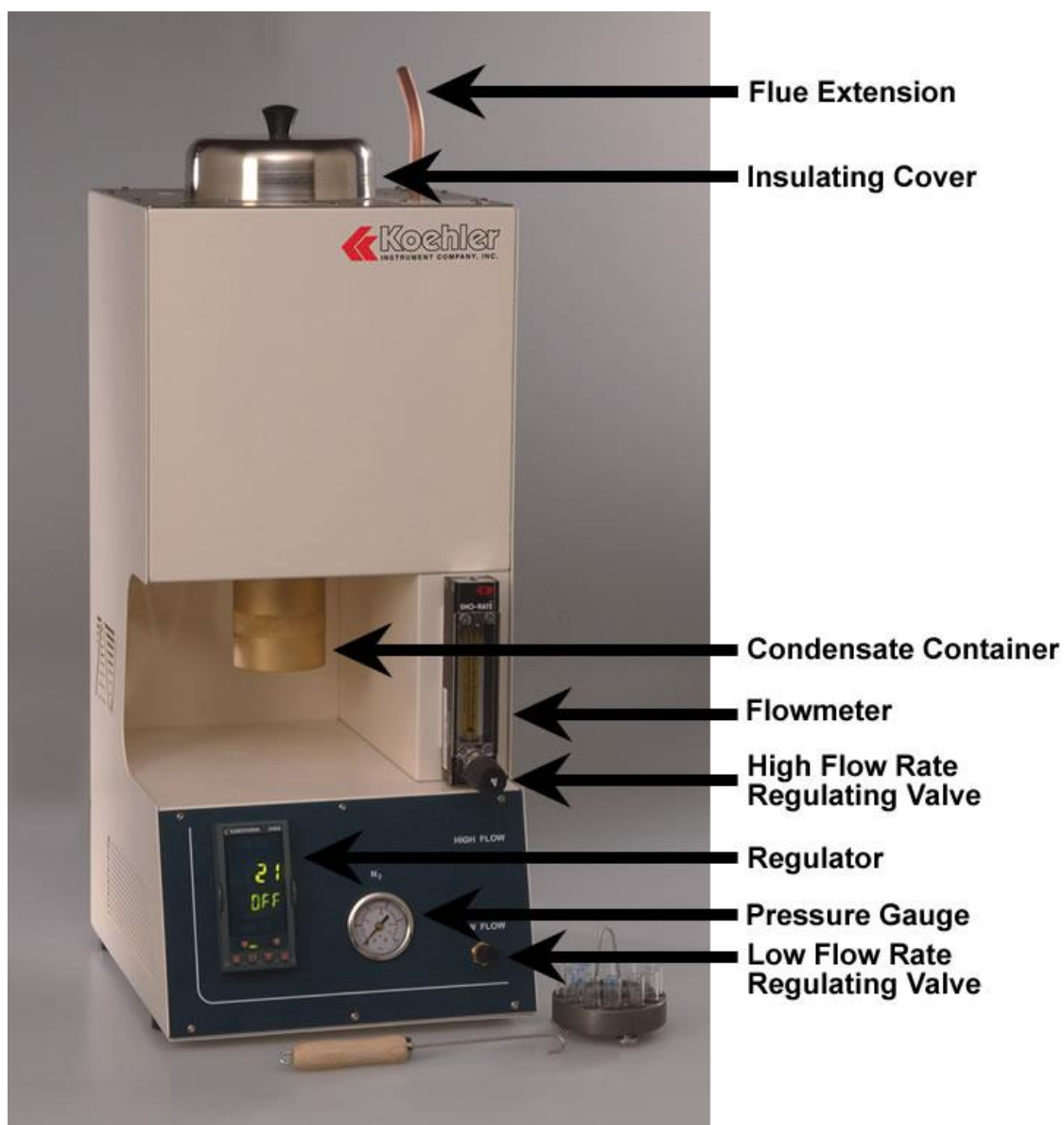
ASTM D 4530

2.2 - Technical characteristics

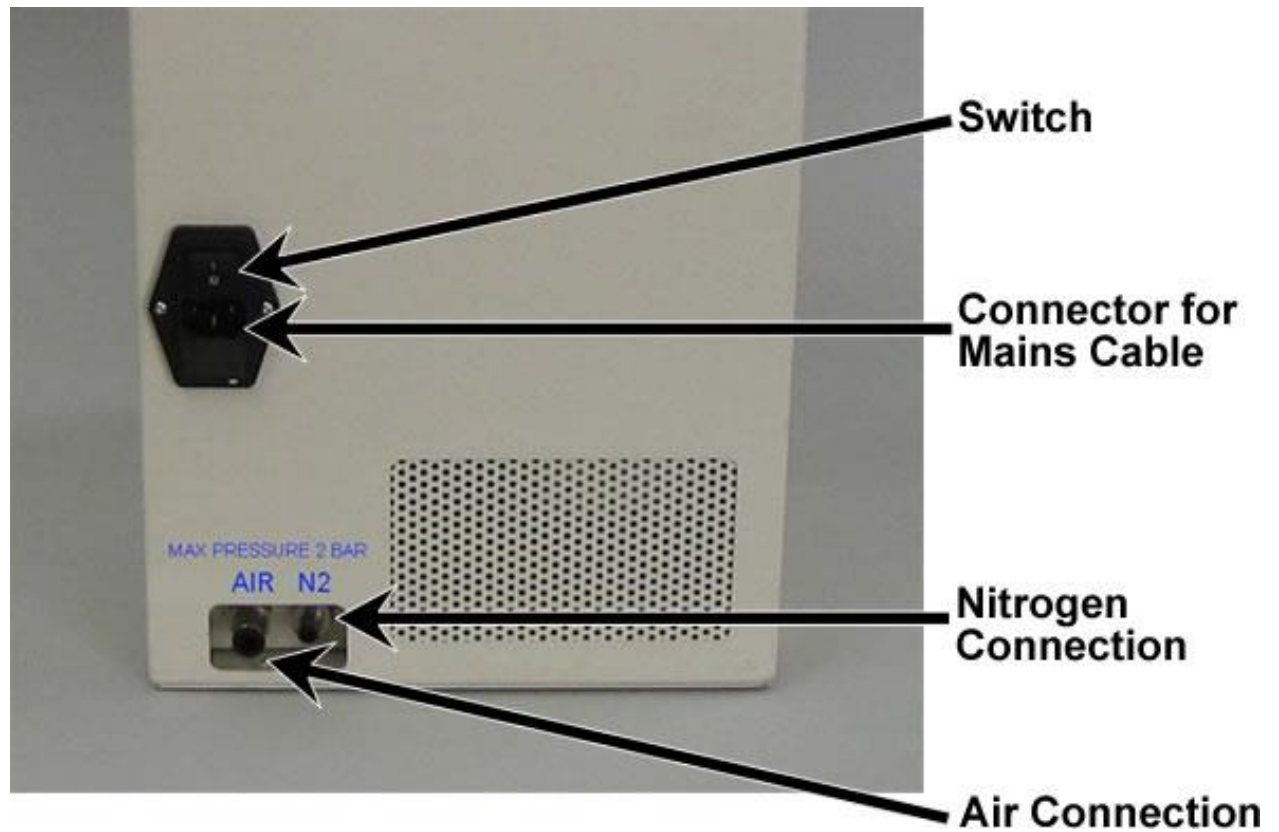
Ambient temperature for use	15 to 30° C
Ambient relative humidity for use	Max 85% non-condensing
Oven temperature range	Ambient T° to 550° C
Oven temperature probe	Thermocouple J
Resolution of temperature measurement	1° C
Type of oven	Conform to the standard ASTM D 4530
Cooling of oven	Accelerated by injection of compressed air for forty minutes
Gas connections at the rear of the appliance	Instant connections dia. 4 mm for nitrogen and dia. 8 mm for compressed air
Maximum upstream gas pressure	2 bar
Type of gas authorised	Air for cooling the oven - nitrogen for the oven
Maximum sample load	12 vials each of 2 ml
Maximum heating kinetic	15° C/min
Use voltage	230 V 50/60 Hz
Power	1500 W
Temperature safety device	By separate J thermocouple set at 550° C
Electrical safety of electronic unit	By fuse (10 A)
Supply voltage	230 V - 50 Hz
Dimensions (L x W x H)	250 x 300 x 600 mm
Weight	19 kg

2.3 - Description of the appliance

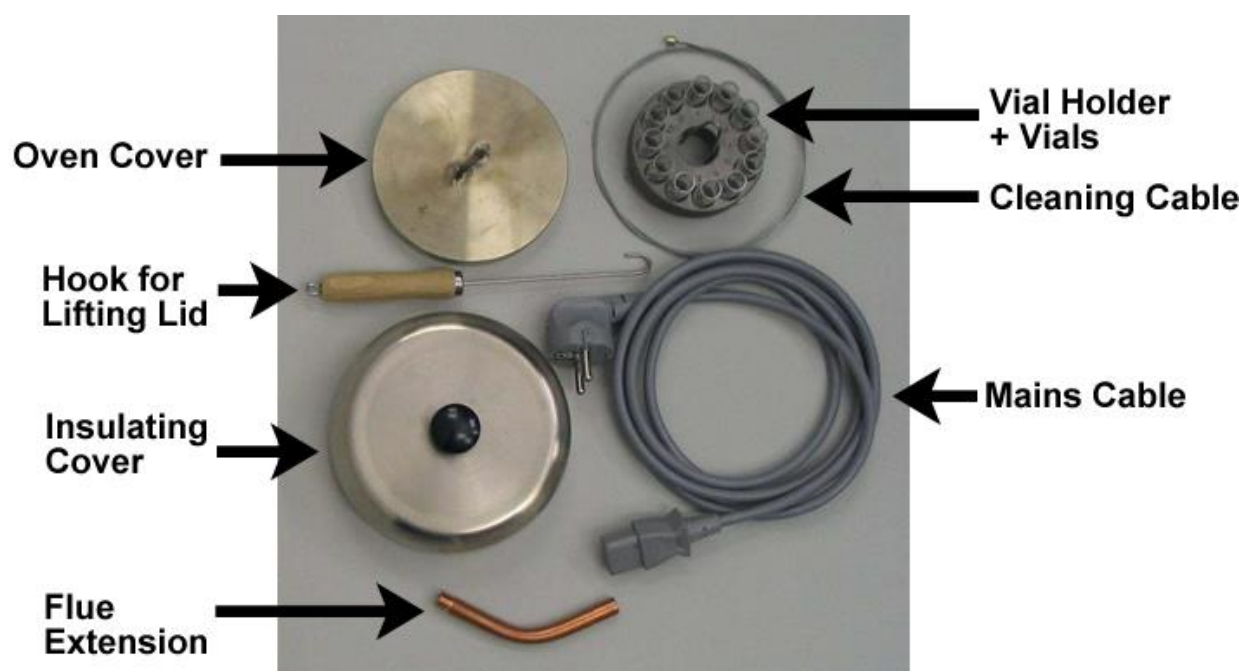
2.3.1 Front panel



2.3.2 Rear Panel



2.3.3 Accessories



3 - COMMISSIONING

3.1 - Installation

Install the appliance on a horizontal and stable surface.

The environment must be clean and free from dust.

Avoid locations that would expose the appliance to direct sunlight or draughts. Unpack and remove the protective packaging from the appliance and its accessories.

Check the equipment and ensure it suffered no damage during transport.

Check the delivery note and make sure all accessories are present.

Check that the electrical specifications marked on the manufacturer's plate are the same as those that will be used. Check that the supply line is protected by fuses and grounded (green/yellow wire).

An appliance with traces of impacts or apparent defects after transport or unpacking must not be commissioned.

To avoid risks of condensation, the appliance must be left at room temperature for several hours before it is powered on.

3.2 - Appliance connections

Depending on the desired use, the appliance must be connected to a source of nitrogen supplying a maximum pressure of 2.0 bar (200 KPa) and to an air source of maximum pressure 2.0 Bar (200 KPa). In order to obtain a stable flow rate during the test, the gas must be supplied through a pressure relief device to stabilize the upstream pressure.

During the first test, which must be a no-load test (without samples), it is vital to check the value of the gas flows and to adjust these depending on the upstream gas pressure.

For guidance, for a flow rate of 150 ml/min, the flow meter should be set to 20.62 mm and for a flow rate of 600 ml/min, at 58.20 mm. Always refer to the abacus received with the appliance.

Position the flue extension.



No connection for smoke evacuation should be made on the flue or its extension. The flue must remain free to ensure optimum exhausting. Any connection on the flue or its extension risks causing:

- condensates on the appliance pipework
- a counter-pressure that would interfere with the test by modifying the flow rates and causing leaks through the hot cover of the oven.



From the side of the appliance take the cover handling hook and the sample vial (see photo opposite)

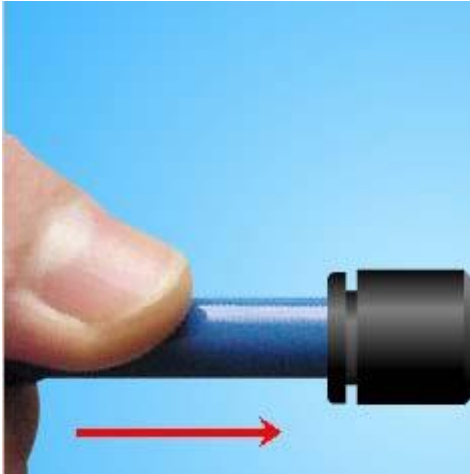
Use the mains cable to supply power to the appliance (check the mains voltage and power are suitable for the appliance).

Reminder: Irrespective of the type of tests and the method used, the appliance must be placed under an extractor hood to allow evacuation of the smoke generated by the test.

3.2.1 Use of instant connectors

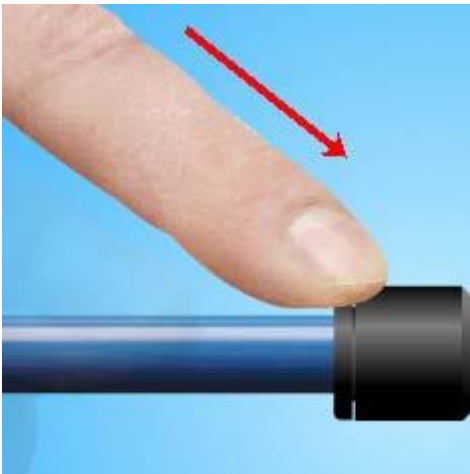
3.2.1.1 Connection of a pipe

Simply push the pipe totally home in the connector. The connection and leak tightness are made instantaneously.

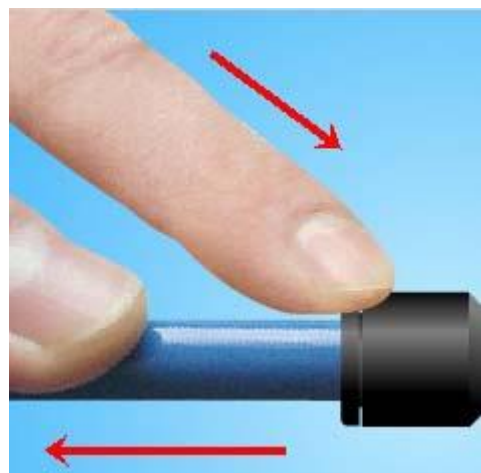


3.2.1.2 Disconnection of a pipe

Press the push button on the outside of the connector.



Pull the pipe to disconnect.

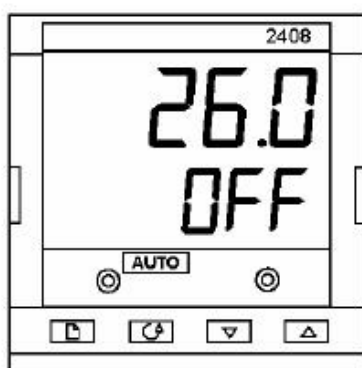


3.3 - Starting up

Power on the appliance by pressing the switch on the rear panel of the appliance.

When the temperature regulator is powered on, for three seconds it runs a series of automatic tests. After each sequence, the display shows the temperature measured on the upper display and displays 'OFF' on the lower display.

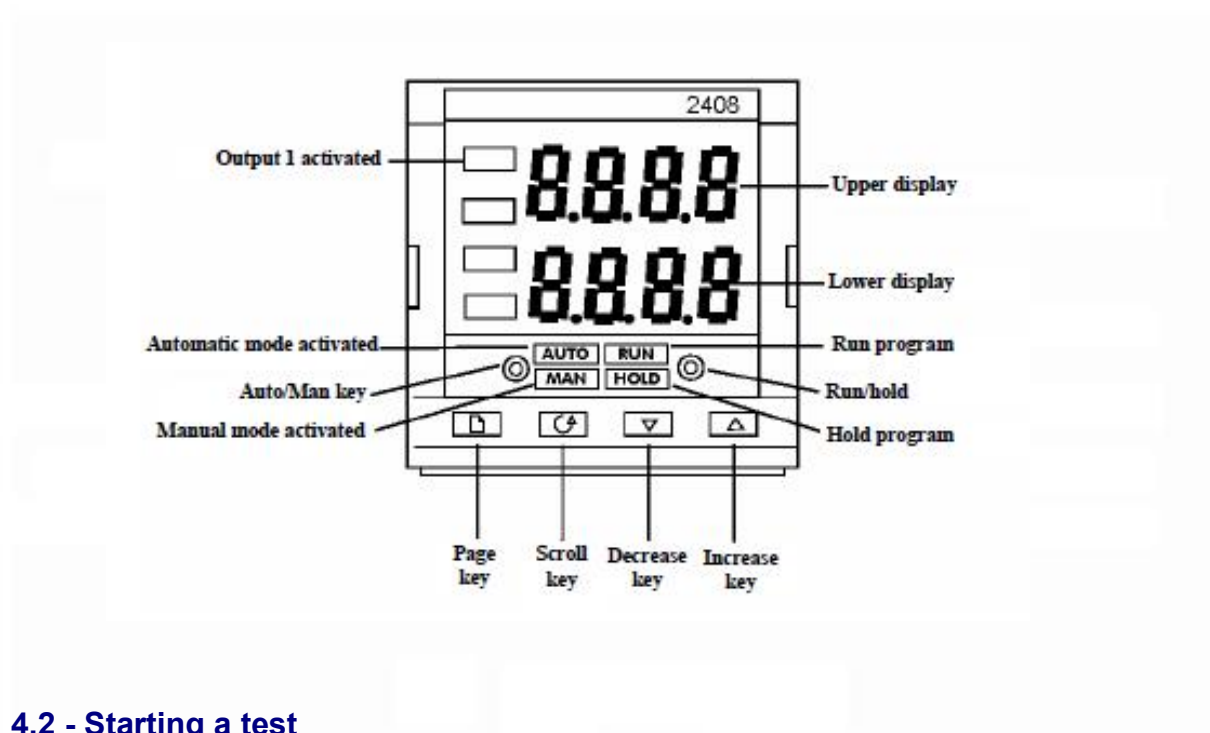
This display is called the At Rest page.



The appliance is ready for a test.

4 - USE OF THE APPLIANCE

4.1 - Description of the temperature regulator



4.2 - Starting a test



The appliance must be placed under an evacuation hood to allow evacuation of smoke generated by the test.

Reminder: It is vital to be familiar with the standards concerning the tests to be initiated.

Reminder: The installing and removal of samples after the tests requires working with a hot oven and hot samples.

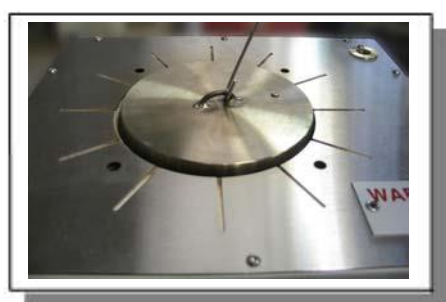
The operator must wear safety equipment (thermal insulating gloves, protective clothes and goggles) and use the appropriate accessories supplied with the appliance.

- ⌚ Prepare the sample or samples in compliance with the standard (marking, weighing, etc.), and place on the vial holder.

- ⌚ Check that the condensates container is empty.



This operation can only be performed on an oven where the temperature is below 100 °C

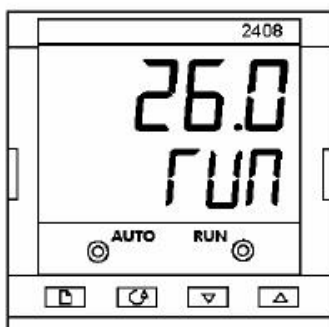


- ⌚ Remove the insulating cover
- ⌚ Remove the oven cover with the tool supplied (see photo).

Caution: the oven may be hot, prudence is vital including at the start of the test.

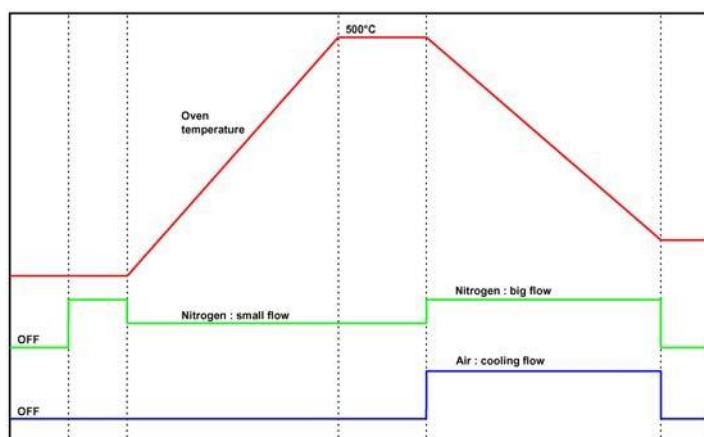
- ⌚ Check that the oven outlet is not blocked by carbon deposits (see Para. Maintenance)
- ⌚ Place the vial holder in the oven using the tool supplied (see photo)
- ⌚ Replace the oven cover (with the tool), placing it correctly to ensure satisfactory air tightness
- ⌚ Replace the insulating cover on the oven
- ⌚ Ensure the gas supply valves are open (mains gas)
- ⌚ Check that the extractor hood is operating and that the extension is correctly positioned on the oven flue
- ⌚ To start the test, press the key (key on the right). The RUN LED will light up. The appliance begins to heat up. 'RUN' is displayed on the lower display.





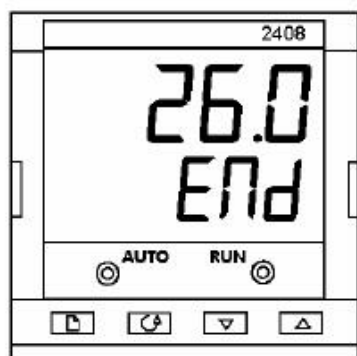
During a test, the regulator follows five stages:

1. Hold time of ten minutes at the oven temperature at the start of the test. Entry of nitrogen to the oven (high flow rate).
2. Heating according to a ramp of $12.5^{\circ}\text{C}/\text{min}$ up to 500°C . Entry of nitrogen in the oven (low flow rate).
3. Hold time of fifteen minutes at 500°C . Entry of nitrogen in the oven (low flow rate).
4. Cooling with compressed air for forty minutes. Entry of nitrogen in the oven (high flow rate).
5. End of test. Switch off heating and gas supply.



During the test, check that the nitrogen flow rates are correct. For guidance, for a flow rate of 150 ml/min , the flow meter should be set at 20.62 mm and for a flow rate of 600 ml/min , at 58.20 mm . Always refer to the abacus received with the appliance.

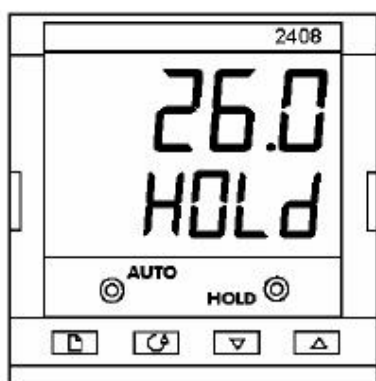
- ⌚ When the test is completed, the RUN LED will flash. 'END' is indicated on the lower display. The temperature regulator will remain in this status until it is reinitialized.



- ⌚ To reinitialize the program, press and hold the key for 2 seconds (the RUN LED goes out). The regulator is then ready to restart a test.

4.3 - Interruption of a test

- ⌚ To stop a test in progress, press and hold down the key for 2 seconds (the RUN LED goes out).
- ⌚ To put the program on hold, press the key (the RUN LED goes out and the HOLD LED lights up). 'HOLD' is indicated on the lower display.



The temperature regulator will remain in this status until it is restarted or stopped.

- ⌚ To restart, press the key (the RUN LED will light up and the HOLD LED will go out). 'RUN' is indicated on the lower display.
- ⌚ To stop the test, press the key in for two seconds (the HOLD LED will go out).

4.4 - Alarm

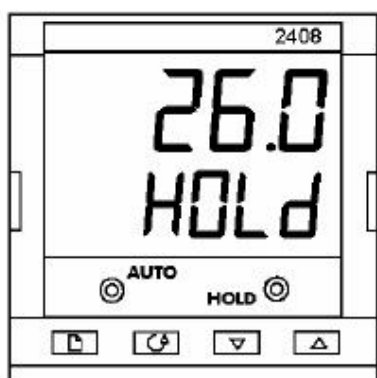
This appliance is equipped with a high temperature safety device. When the temperature of the oven exceeds 550° C, the heating is cut off.



**The heating power supply will be restored when the oven is cold.
ALWAYS INVESTIGATE THE CAUSE OF OVERHEATING AND
REMEDY IT**

4.4.1 Alarm during a test

If a test is in progress, the safety device will switch the regulator to HOLD (the RUN LED goes out and the HOLD LED comes on).
'HOLD' is indicated on the lower display.



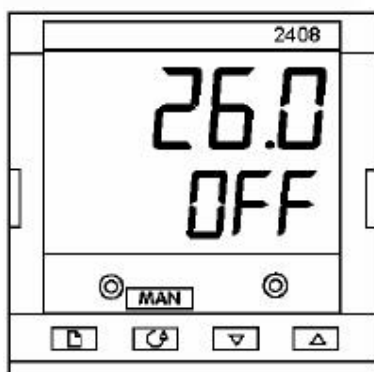
The temperature regulator will remain in this status until it is restarted or stopped.
To restart, press the key (the RUN LED lights up and the HOLD LED goes out).
'RUN' is indicated on the lower display.



**Before restarting the program, it is necessary to investigate the
cause of the overheating and remedy it.**

4.4.2 Alarm in wait mode

If the regulator is waiting (no test in progress) the safety device switches the regulator to manual mode (the AUTO LED goes out and the MAN LED lights up).



When the oven is cold, the regulator reverts to wait mode. In the case of an alarm, it is always necessary to investigate the cause.

4.5 - Important remark

Refer to the regulator manual for additional information.

5 - INSPECTIONS AND GENERAL MAINTENANCE OF THE APPLIANCE

Koehler Instrument Co. has reduced the maintenance requirements of the appliance to the strict minimum. However, in order to ensure reliability of results, it is vital to carry out basic maintenance of the appliance at regular intervals.

5.1 - Inspections before each test

Before any test, it is mandatory to check:

- ① The condensates container: to avoid any overflow or blocking.



Handling of residues must be done with extreme precaution. These residues are highly toxic and the operator must be equipped with the necessary individual protective clothing (gloves, goggles and work clothes).

- ① The oven outlet must be free of carbon deposits. If this is not the case, it is recommended running the cleaning cable through to clean off any deposits (see next paragraph).

5.2 - General maintenance

Depending on the number of analyses and the type of samples used, periodic maintenance is necessary. The frequency will be adapted to the rate and type of use of the appliance and must be determined by observation.

5.2.1 Maintenance of the oven

5.2.1.1 Cleaning the cover seal

Air tightness of the oven mouth is by metal-metal contact between the oven and its cover. Thus it is imperative:

- ✦ that the cover is free of any shock or impacts ✦ that the cover and the oven mouth are free of any carbon deposits.

At the time of manufacture, each oven is paired with its own cover. A number at the base of the oven and on the cover allow checking the pairing.



To remove all deposits from the cover and the oven mouth, use wire wool. **Do not damage the metal.**

Do not use emery cloth which would wear away the metal and cause leaks.

5.2.1.2 Removing carbon deposits from the oven

The outlet at the base of the oven may be a point for deposits of carbon, which by accumulation could block the oven exhaust.

To avoid total blockage of the oven, it is preferable to clean this part regularly, using the cable supplied.



To do this:

- ① With the oven cold and disconnected from the power supply, remove the condensates container.
- ① Remove the insulating cover and the metal cover of the oven.
- ① Pass the cable through the oven mouth and push out through the exhaust pipe.



- ① With a backwards and forwards movement, orientating the cable in all directions, clean the pipe by removing all deposits of carbon.

- ① Check the condition of the flue pipe and its extension. Perform the same operation in the case of deposits of carbon.

5.2.2 Checking the oven temperature

To check the oven temperature, a control thermocouple must be placed in the oven.

For this purpose, the cover has a hole blocked with a small stud. This hole allows inserting a thermo-couple to monitor and measure the oven temperature. This stud is simply forced into the hole. By pushing it can be ejected.

If the oven temperature is not satisfactory, it is recommended to check the calibration of the oven thermo-couple (see Para. Calibration).



5.2.3 Monitoring the gas flows

To monitor the gas flows, it is vital to start a no-load test.

Calibration of gas flow rates must be performed using the abacus received with the appliance.

For guidance, for a flow rate of 150 ml/min, the flow meter should be regulated at 20.62 mm and for a flow rate of 600 ml/min, at 58.20 mm.

Always refer to the abacus received with the appliance.

6 - MAIN CAUSES OF POOR RESULTS

Since the function of the appliance is to process a sample, the cause of poor results may not be attributable to the appliance.

Poor manipulation of samples (with absorption of humidity at the end of the test), inaccurate weighing, an error in entering the weight, an error in identifying the samples may all cause errors in the expression of results.

In the case of a doubtful result, it is appropriate to:

- ① Check the condition of the cover after the test. If leaks have occurred at that location there will be traces on the cover. Also, check there are no deposits on the oven mouth and the oven cover that could adversely affect oven Air tightness.
- ① Check that the exhaust of the oven is not partially or completely blocked.
- ① Check that the gas system is properly open and that the flow rates are correct.
- ① Ensure that the load placed in the vials is not excessive and that there has been no loss by overflowing.
- ① Finally, check calibration of the oven thermo-couple.

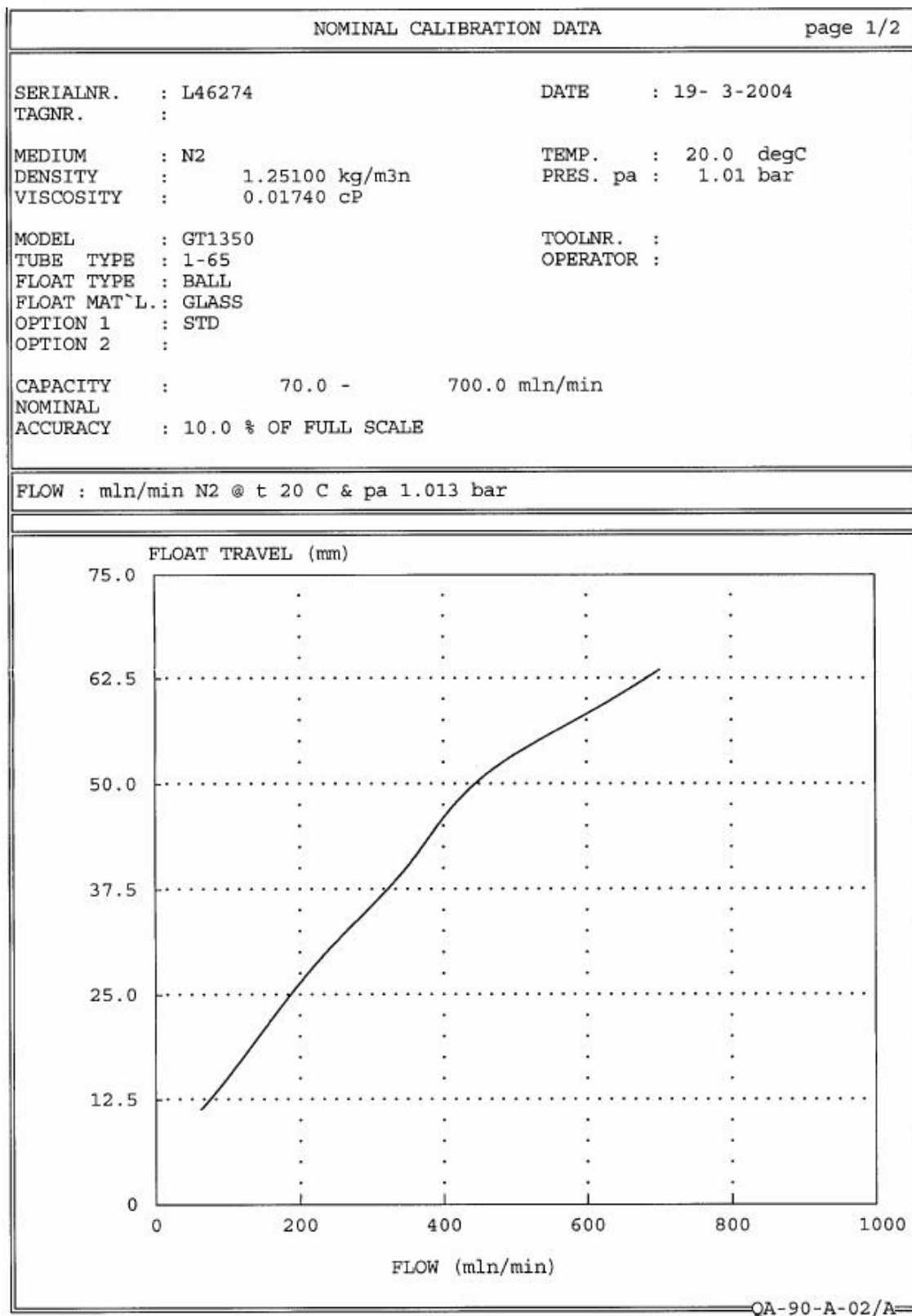
7- LIST OF ACCESSORIES AND SPARE PARTS

Designation

Cleaning cable
Mixed vial holder
Small hole vial holder
Large hole vial holder
Flue extension
Handling hook
Complete oven
Oven thermo-couple
Safety thermo-couple
Insulating cover
Glass vial, 2 ml
Glass vial, 16 ml
Pressure gauge
Brass condensates container
Glass condensates container
Condensates container seal
Flow Meter
Air electro-valve
Nitrogen electro-valve
1 nitrogen connection pipe dia. 2.7 X 4 mm
1 air connection pipe dia. 6 X 8 mm
Eurotherm regulator[^]
Static relay
Temperature safety device
Valve
Pair of heat-resistant gloves (kevlar)
Accessories support

8 - ANNEXES

8.1 Flow meter abacus



NOMINAL CALIBRATION DATA

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SERIALNR. : L46274

TAG NR. :

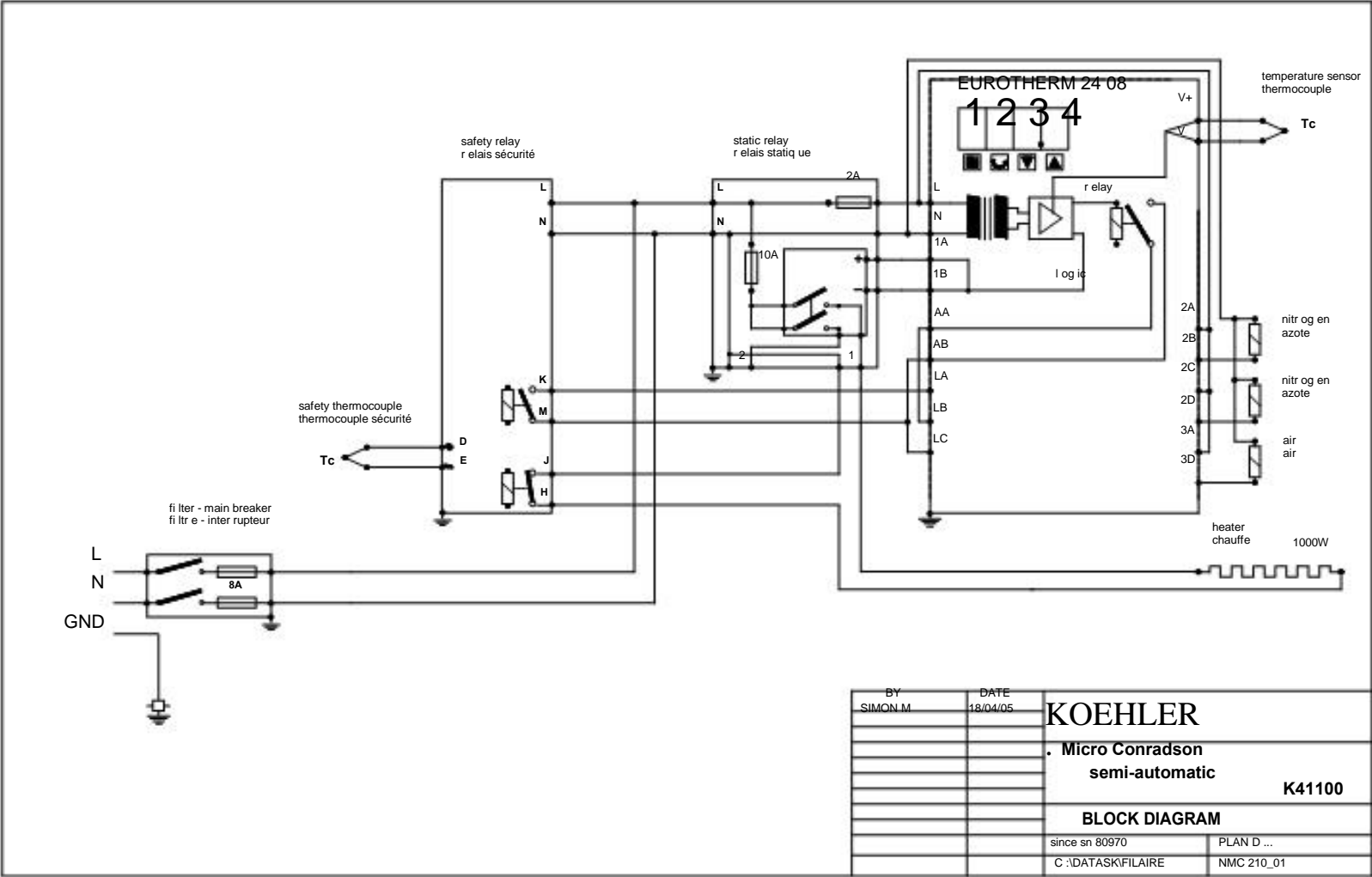
FLOAT TRAVEL mm	FLOW mln/min	FLOAT TRAVEL mm	FLOW mln/min	FLOAT TRAVEL mm	FLOW mln/min	FLOAT TRAVEL mm	FLOW mln/min
		26.0	197.8	51.0	460.5		
		27.0	207.4	52.0	476.4		
		28.0	217.2	53.0	494.2		
		29.0	227.5	54.0	513.5		
		30.0	238.1	55.0	533.9		
		31.0	249.1	56.0	555.0		
		32.0	260.5	57.0	576.4		
		33.0	272.1	58.0	597.7		
		34.0	283.8	59.0	618.5		
		35.0	295.5	60.0	638.4		
		36.0	307.0	61.0	657.6		
12.0	71.6	37.0	318.3	62.0	676.2		
13.0	81.5	38.0	329.3	63.0	694.3		
14.0	91.1	39.0	339.7				
15.0	100.4	40.0	349.6				
16.0	109.4	41.0	358.8				
17.0	118.3	42.0	367.5				
18.0	127.1	43.0	376.0				
19.0	135.8	44.0	384.4				
20.0	144.4	45.0	393.0				
21.0	153.1	46.0	402.0				
22.0	161.7	47.0	411.6				
23.0	170.5	48.0	422.0				
24.0	179.4	49.0	433.5				
25.0	188.5	50.0	446.2				

FLOW mln/min	FLOAT TRAVEL mm	FLOW mln/min	FLOAT TRAVEL mm	FLOW mln/min	FLOAT TRAVEL mm	FLOW mln/min	FLOAT TRAVEL mm
		260.0	32.04	510.0	54.17		
		270.0	32.93	520.0	54.71		
		280.0	33.81	530.0	55.21		
		290.0	34.68	540.0	55.68		
		300.0	35.55	550.0	56.13		
		310.0	36.42	560.0	56.55		
70.0	11.91	320.0	37.30	570.0	56.97		
80.0	12.95	330.0	38.19	580.0	57.38		
90.0	14.00	340.0	39.10	590.0	57.79		
100.0	15.08	350.0	40.04	600.0	58.20		
110.0	16.17	360.0	41.01	610.0	58.63		
120.0	17.27	370.0	41.99	620.0	59.07		
130.0	18.39	380.0	43.00	630.0	59.54		
140.0	19.50	390.0	44.01	640.0	60.02		
150.0	20.62	400.0	45.03	650.0	60.53		
160.0	21.74	410.0	46.03	660.0	61.05		
170.0	22.85	420.0	47.03	670.0	61.59		
180.0	23.96	430.0	48.00	680.0	62.14		
190.0	25.05	440.0	48.94	690.0	62.70		
200.0	26.12	450.0	49.84	700.0	63.27		
210.0	27.17	460.0	50.70				
220.0	28.21	470.0	51.51				
230.0	29.21	480.0	52.26				
240.0	30.18	490.0	52.95				
250.0	31.12	500.0	53.58				

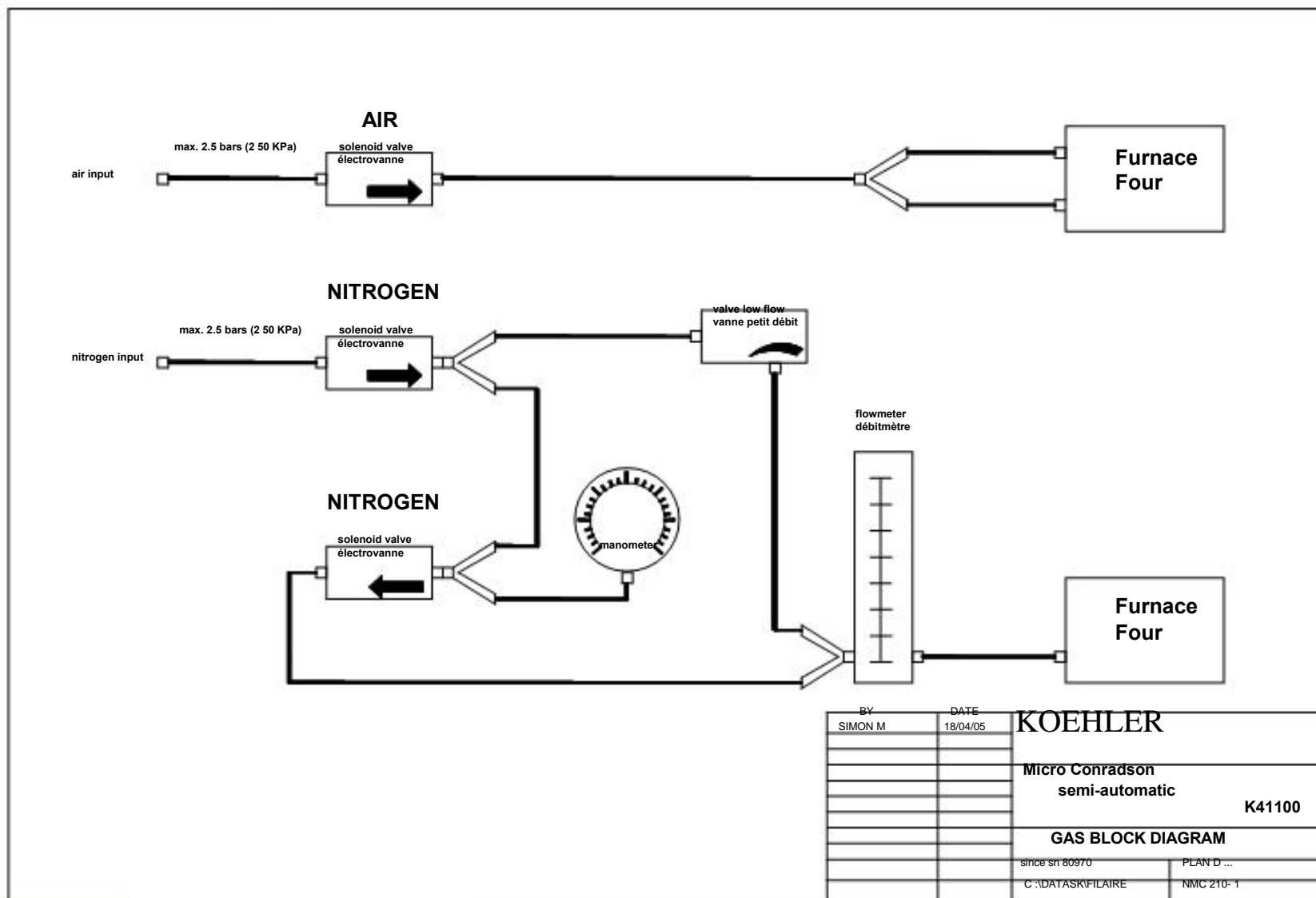
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K41100

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