



**Detailed list of components of  
the BIPM high accuracy  
reference facility for ongoing  
comparisons of carbon dioxide  
in air standards.**

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## 1. Title: Detailed list of components of the BIPM high accuracy reference facility for on-going comparisons of carbon dioxide in air standards.

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Abstract: The article *A high accuracy reference facility for on-going comparisons of CO<sub>2</sub> in air standards* (J. Viallon et al.) [1] in which the BIPM PVT-CO<sub>2</sub> system characterization is reported also contains a description of the facility. This technical supplement includes additional details of the components used in this facility.

## 2. Disclaimer

Commercial equipment, instruments and materials are identified hereafter in order to specify experimental procedures as completely as possible. This does not imply a recommendation or endorsement by the Bureau International des Poids et Mesures (BIPM) nor does it imply that any of the materials, instruments or equipment identified are necessarily the best available for the purpose.

## 3. Introduction

This report aims to provide an extensive parts list of the BIPM PVT-CO<sub>2</sub> system including parts that are not shown on the facility schematics presented in the article. Some elements present in the facility are not available commercially and were manufactured at the BIPM. Details will be given of these elements so that they can be duplicated.

## 4. Facility schematics

Figure 1 shows a schematic of the facility, which is also displayed in the article. The elements which are part of the facility can be grouped into five ensembles according to their purpose. Each ensemble of parts is then further detailed as follows:

- Temperature controlled enclosure.
- Pressure measurement elements.
- Temperature measurement elements.
- Vessels, tubing and valves.
- Mass flow control.
- Cryogenic elements.
- Pumps.
- Gas analysers.
- Computer and computer add-ons for communication with all of the above.

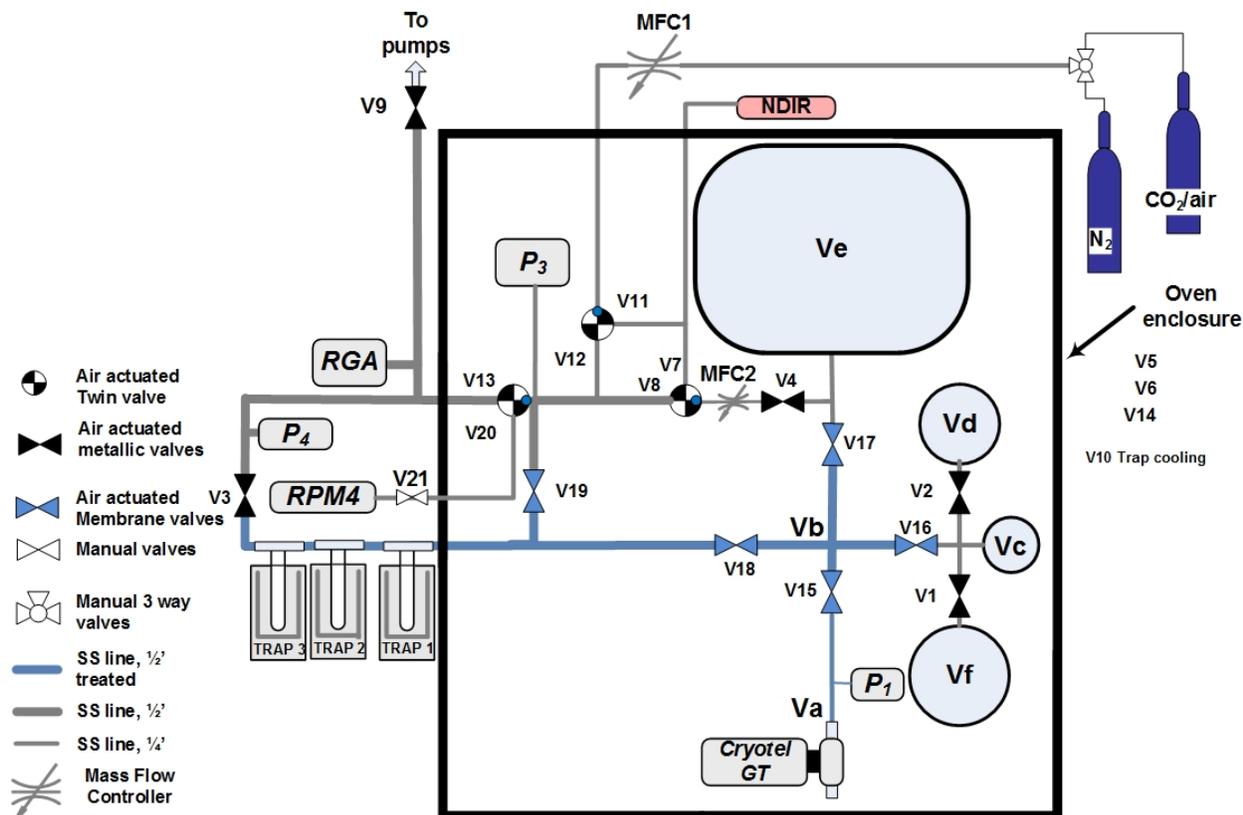


Figure 1. Measurement setup, including: a temperature-controlled chamber (indicated as oven enclosure) which itself contains six vessels Va to Vf. Other parts are: RGA – Residual Gas Analyser (Quadrupole mass spectrometer); TMP – Turbo Molecular Pump; Vn – Valve; MFC – Mass Flow Controller; NDIR – Non Dispersive Infrared Detector; Pn – Pressure Gauge n; TRAPn – Cryogenic trap ; Cryotol GT – Stirling Cryocooler

## 5. Temperature controlled enclosure

The temperature controlled enclosure is a bacteriological incubator (BINDER Incubator BD 720) modified to gain access from the top through a  $\varnothing 100$  mm port and from the sides through  $\varnothing 50$  mm ports to pass tubes and wires. The exact position of these access ports is shown in Figure 2. The enclosure is maintained at a nominal temperature of 308 K during measurements. In addition, fans, deflectors and insulation shields have been positioned inside the enclosure to improve the temperature uniformity and stability around the vessels, ensuring that temperature gradients across vessels are no greater than 0.03 K. The fans are remotely controlled from the computer as they must be stopped when needed during the extraction procedure. The fan remote control is detailed in section 13. Equipment references are listed in Table 1.

Quantity	Part description	Manufacturer	reference
1	bacteriological incubator	Binder	BD 720
8	Fans 12 Vdc 119 mm 1.8 m <sup>3</sup> /min	NMB	4710KL-04W-B10
1	AC/DC 12 Vdc, 3.5 A, 42 W power supply	RS PRO	175-3277

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Table 1: Temperature controlled enclosure elements

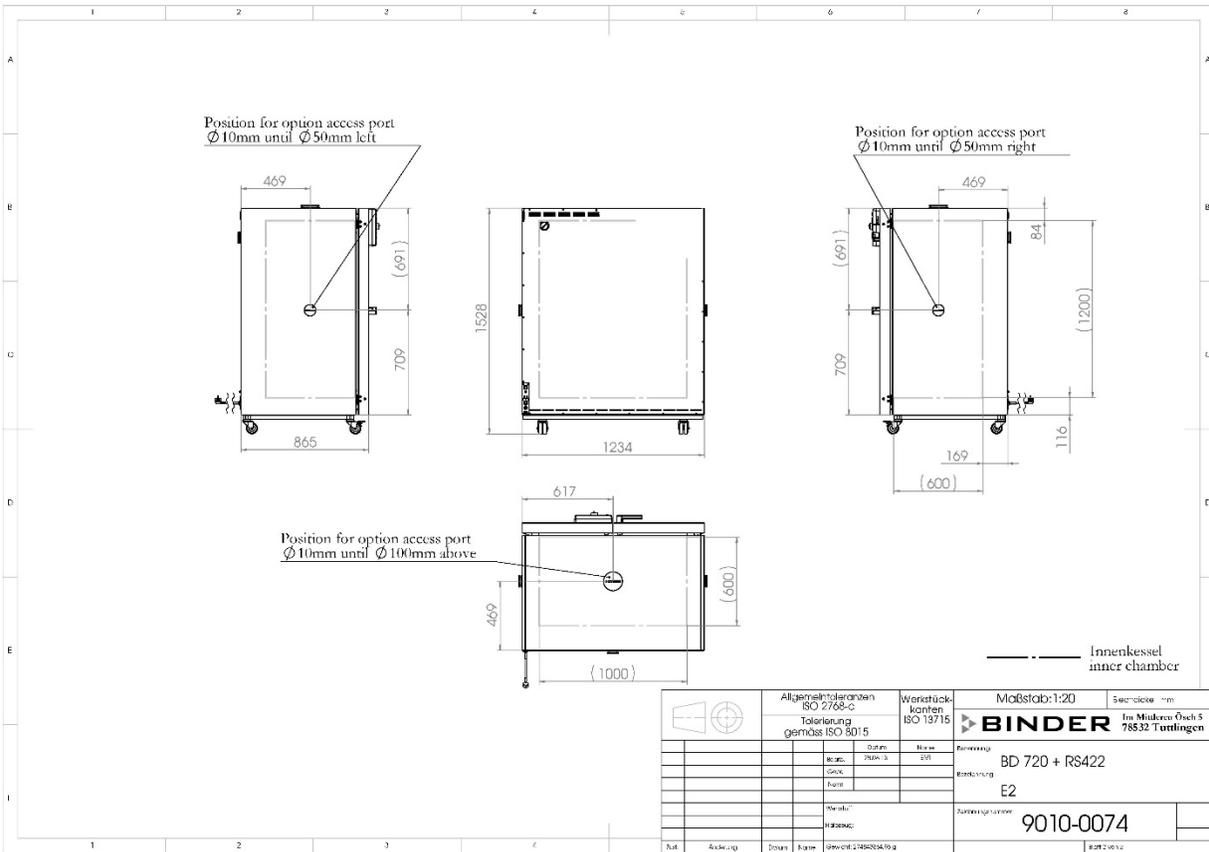


Figure 2. Position of the BD 720 additional access ports (all dimensions in mm)

## 6. Pressure measurement elements

Four pressure measuring elements are shown in the schematics: P1, P3, P4 and RPM4 (the reference P2 referred to a pressure transducer that has been removed from the system). Their references are listed in Table 2. P1, which is used to measure the air sample pressure and the final CO<sub>2</sub> pressure, is regularly calibrated against RPM4 which is traceable to an NMI pressure reference over the range 50 Pa to 110 kPa. P3 and P4 are for indication only. They are located before and after the external traps on the gas stream, to indicate the pressure present in the traps.

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Schematics label	Part description	Manufacturer	reference
P1	DPS 80HA High Accuracy Digital Pressure Sensor	General Electric	E80HA-TA-A2-CC-H0-PA
P3	Convection enhanced pirani gauge	KJ Lesker	300 series
P4	Convection enhanced pirani gauge Benchtop Gauge Controller	KJ Lesker KJ Lesker	275 series 375 Series
RPM4	Base pressure monitor Hi rpt,110 kpa absolute gas, std	Fluke Fluke	D3-RPM4-BASE D3-RPM4-HI-A100KS

Table 2: Pressure measurement element references

P1 has a nominal pressure resolution of 1 Pa and is declared as being compensated for temperature over the full pressure measurement range. The commands do not give direct access to the sensor temperature or the maximum resolution of the probe. To gain access to these, the calibration coefficients stored in the instrument (command E,? and L,?), the raw pressure signal in Hz and the temperature signal in mV are recovered (command Z) then the pressure conversion performed externally following the instructions mentioned in the operating manual. This gives access to the sensor temperature with a resolution of 0.1 °C and a slightly improved pressure signal resolution.

### 7. Temperature measurement elements

Twenty temperature measurements are monitored within the system. Ten are NTC sensors (calibrated in the range 20 °C to 40 °C) which are placed on the vessels (four on Ve, two on Va and one on each Vb, Vc, Vd, Vf) and used in computations. The other ten are not used for computation but are used either for action triggering, regulation or simply for trouble shooting purposes. Out of these ten sensors, four are PT100 sensors B class uncalibrated and are used to monitor the temperature of the cold traps and six are temperature measurements provided by other instruments and are discussed in relevant sections. The list of equipment is given in Table 3.

Quantity	Part description	Manufacturer	reference
1	ALMEMO® 8036-9 acquisition unit	Ahlborn	MA80369
1	USB connection cable		ZA1919DKU
4	Pt100 sensor B class		FP0802
10	NTC sensor (range -20 °C to +100 °C)		FN0001
4	ALMEMO connector PT100 4 wires		ZA9030-FS2
5	ALMEMO connector 2 x NTC		ZA9040-FS2

Table 3: Temperature measurement element references

### 8. Vessels, tubing and valves

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The chamber contains six stainless-steel vessels (Va to Vf) of different sizes that are connected using stainless-steel tubes and isolated from each other using fourteen pneumatically actuated membrane valves. An additional valve can be closed to isolate the system from the pumps after the connection to the residual gas analyser (RGA). Elements that could not be purchased with Silconert® or Silonite® treatment were treated afterwards, whenever it was possible, to minimize the adsorption of CO<sub>2</sub>. The only surfaces inside Va which could not be treated are the wetted surfaces of the pressure gauge P1, and it was estimated that this represented less than 14 % of the internal surfaces in the volume Va.

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Schematics label	Part description	Manufacturer	reference
Vc	Silonite treated minican 100 cc	Entech instruments	SKU: 01-29-MC100SQT
Vd	Silonite treated minican 450 cc	Entech instruments	SKU: 01-29-MC450SQT
Ve	Canister Silcocan 6 L	RESTEK	22092
Vf	Silonite treated minican 1400 cc	Entech instruments	SKU: 01-29-MC14SQT
Va	Obtured ¼" electropolished tube (15 cm)	Deltron	ULTUA03.35*0.89_.13
Vb	Union cross ½" 4x adaptors ½"-1/4" 3x ¼" tube – ¼" male VCR	Swagelok Swagelok Swagelok	SS-810-4 SS-811-PC-4 SS-4-VCR-6-400
V15	Silconert treated valve	Rotarex	M4SIV BPNF 2V1 K A/B:V1/4F
V7-V8 V11-V12 V13-V20	316L VIM-VAR UHP Diaphragm Sealed 2-Valve 3-Port Monoblock	Swagelok	6LVV-P1V222P-AA
V16 V17 V18 V18	316L VIM-VAR UHP Diaphragm Sealed Valve, 1/4 in. Female VCR Fitting	Swagelok	6LVV-DPFR4-P1-C
V1 V2 V3 V4	316L VIM-VAR UHP Diaphragm Sealed Valve, 1/4 in. Swagelok Tube Fitting	Swagelok	6LVV-DPS4-C
V9	Vacuum valve	Edwards	IPV16PKA B
<b>Other miscellaneous parts</b>			
	½" electroponished transfer line	DOCKWEILER	ULTUA012.70*1.22_.18
	¼" electropolished transfer line	DOCKWEILER	ULTUA03.35*0.89_.13
	8x ¼" tube – ¼" male VCR	Swagelok	SS-4-VCR-6-400
	½" T union		SS-810-3
	2x adaptors ½"-1/4"		SS-811-PC-4
	2 x KF16 to ¼" tube adaptor	Neyco photonics	KSWA167S
	2 x KF-16 Metal seal	Neyco photonics	KF10/16MS
	2 x KF-16 clamp	Neyco photonics	KF10/16MSC

Table 4: Vessels, tubing and valve references

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## 9. Mass flow control

There are two mass flow controllers installed in the system. MFC1 is used to control the flow of gas introduced from the cylinder under analysis into the system and MFC2 to control the flow when the large volume is emptied through the external traps. The references of both pieces of equipment are given in Table 5

Schematics label	Part description	Manufacturer	Reference
MFC1	Red-Y flow 0-1 L/Min	Vögtlin	GSC-B9TS-DD23
MFC2	Red-Y flow 0-0.2 L/Min		GSC-B4TS-BB12

Table 5: Mass flow controller references

## 10. Cryogenic elements

The system contains four cryogenic traps: three home-designed cold traps to extract the CO<sub>2</sub> from air, located outside the temperature-controlled enclosure, and the final volume Va, which is also a cold finger, located inside the temperature controlled enclosure.

The final volume Va is a single ended ¼” electropolished Silconert treated tube cooled by a Stirling cryo-cooler of which the heat rejector is cooled by a cold-water circulation from a Lauda Microcool (not represented in the diagram).

The cryo-cooler and the Microcool both report a temperature reading.

The coupling between the cold head of the cryocooler and Va is a copper block machined at the BIPM (Figure 6).

The circulation fans placed inside the temperature-controlled enclosure are switched off while the Stirling cryo-cooler is cooling Va, otherwise the forced air flow around Va prevents its temperature from dropping below the CO<sub>2</sub> freezing point.

The external traps labelled Trap 1 Trap 2 and Trap 3 are identical and home designed. Their temperature can be set to any temperature between 78 K and 350 K. The cooling is performed with liquid nitrogen circulation into a heat exchanger, which surround each trap. The liquid nitrogen flow is controlled by an actuated on/off valve placed after each heat exchanger. The actuation is controlled by a PID controller.

The references of commercial equipment are given in. Table 5.

Drawings of non-commercial pieces are supplied in Figure 4 to Figure 6.

Each PID reports the temperature reading used for regulation purposes.

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Va Trap			
Schematics label	Part description	Manufacturer	reference
Cryotel GT	Stirling cooler with water cooling jacket	Sunpower	Cryotel-GT-W
	Cryotel GT power supply 50 V 20 A	Digimess	SM5020
	Cold water circulation	Lauda	MC250
	Copper adaptation bloc	BIPM	GT-Va block
Trap 1, Trap 2 and Trap 3			
Quantity	Part description	Manufacturer	reference
3	PID controller	Lumel	RE72 122200E0
3	SSD relay	Finder	34.81.7.005.9024
3	Heater	Watlow	Firerod 2110M E1A-621
3	PT100 sensor	RS PRO	611-7817
1	24 V power supply 300 W	XP Power	VES300PS24
1	RS485-USB converter	FTDI	USB-RS485-WE-1800-BT
3	Silconert treated traps	BIPM	PVT-Trap
3	Copper heat exchanger	BIPM	PVT-Heat exchanger
3	Cryogenic valves	ASCO	E262K232S1TD0H1
1	60 L Dewar	Air Liquide	TR60 (113529)
4	Isolation ¼ ball valves	Swagelok	SS-43GS4
1	safety valve set at 0.25 bars g	NORGEN	1002/BM000
1	KF50 modified flange	BIPM	PVT-KF50 flange

Table 6: Cryogenic cooling commercial parts references

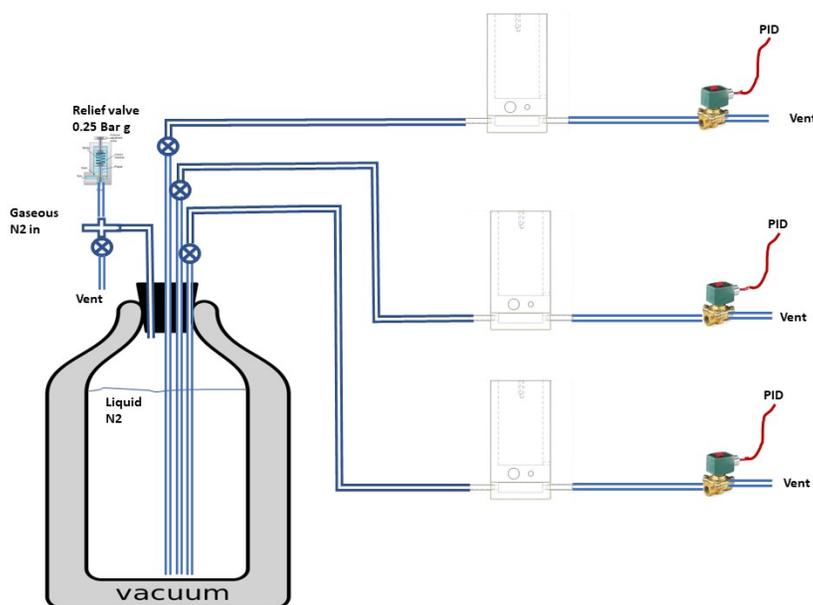


Figure 3. PVT heat exchanger cooling flow chart



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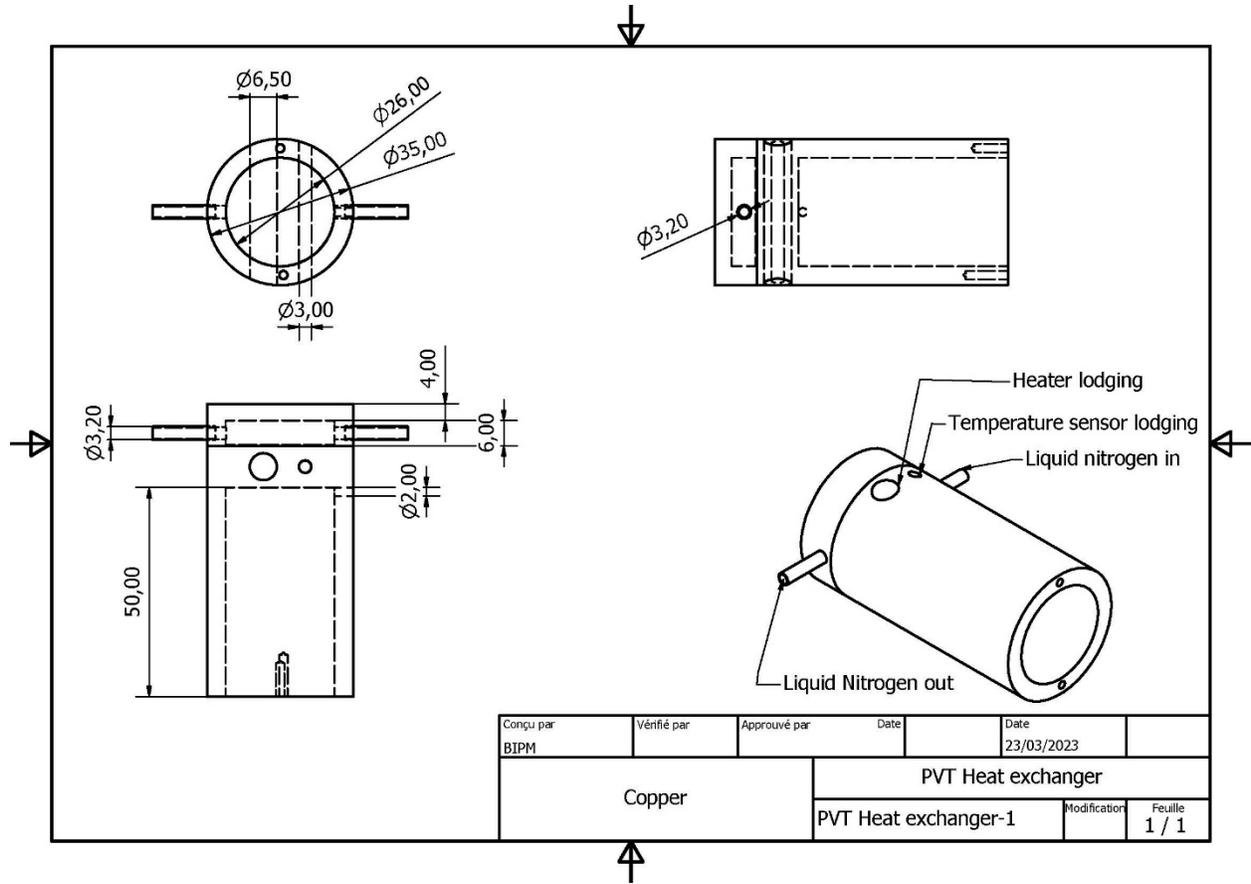


Figure 5. drawing of the heat exchanger surrounding each trap (all dimensions in mm)

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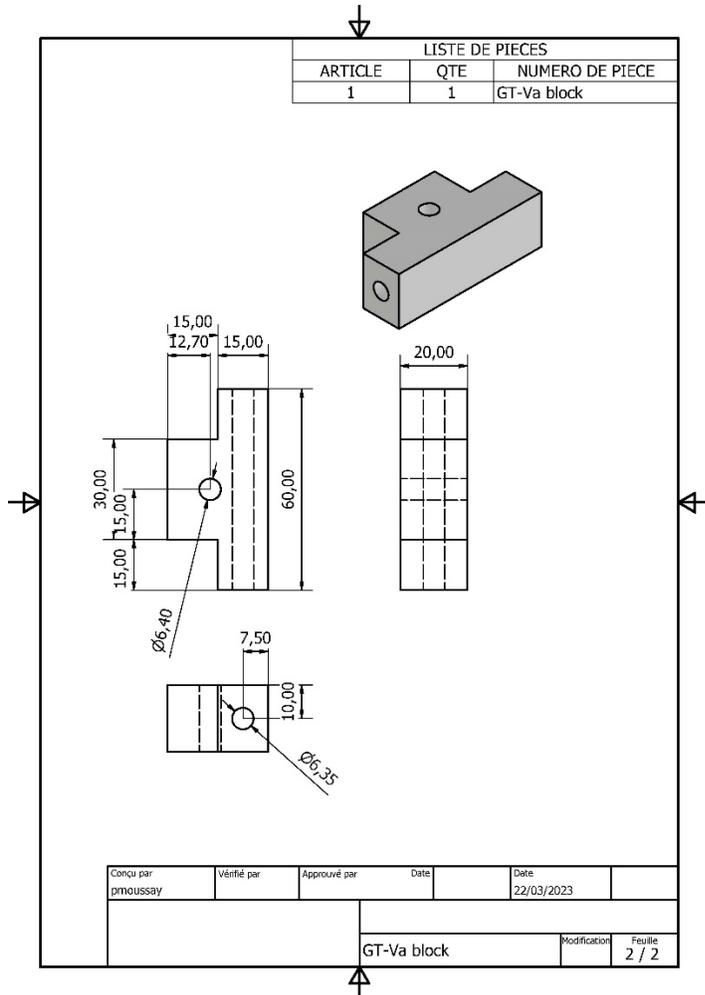


Figure 6 : GT-Va block drawing (all dimensions in mm)

## 11. Pumps

The facility is equipped with a scroll pump and a turbo pump. The turbo pump can be switched on and off from the computer.

Quantity	Part description	Manufacturer	reference
1	Scroll pump XDS 10	Edwards	A72601903
1	Turbo pump STP-301 C		B74871010
1	SCU-301/451 STP Turbo Pump Controller		PT21Z0Z04

Table 7: Pump references

Vacuum adaptors/elements are not listed as they are regular off the shelf elements.

## 12. Gas analysers

The facility is equipped with two gas analysers, which are present for quality control.

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A nondispersive infrared sensor (NDIR) that measures CO<sub>2</sub> amount fraction is located outside the chamber and connected by tubing to valve V7 and V11, to provide an approximated value in the first minutes of the measurement process.

A quadrupole mass spectrometer (RGA model MKS Vision 1000 C) is included to check the composition of the gas at various steps in the measurement process (initial air sample, condensed gases, pure CO<sub>2</sub>). Its purpose is to detect anormal components (for example high levels of water or CO<sub>2</sub> in the external traps after transfer to Va).

Schematics label	Part description	Manufacturer	reference
NDIR	CO <sub>2</sub> analyser	LI-COR	LI-820
RGA	Residual gas analyser	MKS	LM76 - V1000C

Table 8: Gas analysers references

### 13. Computer and computer addons for communication with all of the above

The elements of the facility are connected to a 64 bit computer operated under windows 7 professional.

This PC is equipped with add-ons to increase the number of RS232 and RS485 communication ports as well as the number of digital input/output lines necessary to control the actuated valves, the status of the Turbo pump and the operation of the enclosure temperature homogenization fans.

The reference of commercial equipment is given in Table 9.

Computer and addons			
Quantity	Part description	Manufacturer	reference
1	64 bit computer With PCI socket	DELL	Precision T5610
1	Rs232 port extender	National instrument	PCIe-8430/8
1	Valves control	National instrument	PCI-6517
16	Valves solenoid	SMC	VZ110-5MZ-M5-X51
1	Control of Pump and fans	National instrument	USB-6525
1	SSD relay	Finder	34.81.7.005.9024
2	RS485 communication for Red-y and Lumel	FTDI	USB-RS485-WE-1800-BT
1	USB to 8 port RS232	Brainbox	US-279

Table 9: computer and add-on parts references