Gas flow management and THEMYS

INTRODUCTION

Gas flow at normal pressure is required for most analyses performed with a thermogravimetric analyzer or a simultaneous thermal analyzer.
The main purpose of a gas flow is to renew the atmosphere by carrying away the vapors released by the sample. However, depending on the nature of the gas, it may have other functions:
• An inert gas (helium, argon, nitrogen, etc.) protects the sample from oxidation during heating.
• An active gas (oxygen, hydrogen, etc.) reacts with the sample and the corresponding transformation can then be studied.
Accurate gas control, including composition and flow rate, is therefore of the utmost importance for many Themys measurements, as measurement quality relies on it.

1. Themys gas management system

Atmosphere and flow control

Themys is available with three different gas management configurations: PureGas, GasBlend and MultiGasBlend. What they have in common is a software-based control system, which enables gas types, flow rates or mixing ratios to be changed sequentially, either during the experiment or during sample pre-treatment.

• PureGas configuration

In this configuration, a single gas, called the carrier gas, is introduced into the Themys balance and then flows downwards through the furnace, vented via a one-way valve.

The line is equipped with a mass flow controller, located upstream of the balance. It has a flow control capacity of up to 200 milliliters per minute.

The system is also equipped with three gas connectors and solenoid valves. This means that three gas cylinders or lines of different natures can be permanently connected to the Themys structure. At any time, the solenoid valves can be activated to change the nature of the carrier gas before, during or after experimentation.

It is important to note that in this configuration, the nature of the gases used must be compatible with the equipment present in the balance, especially with the electronics. This configuration therefore applies to experiments with inert, oxidizing or reducing gases that are not corrosive to the balance components and that are dry.

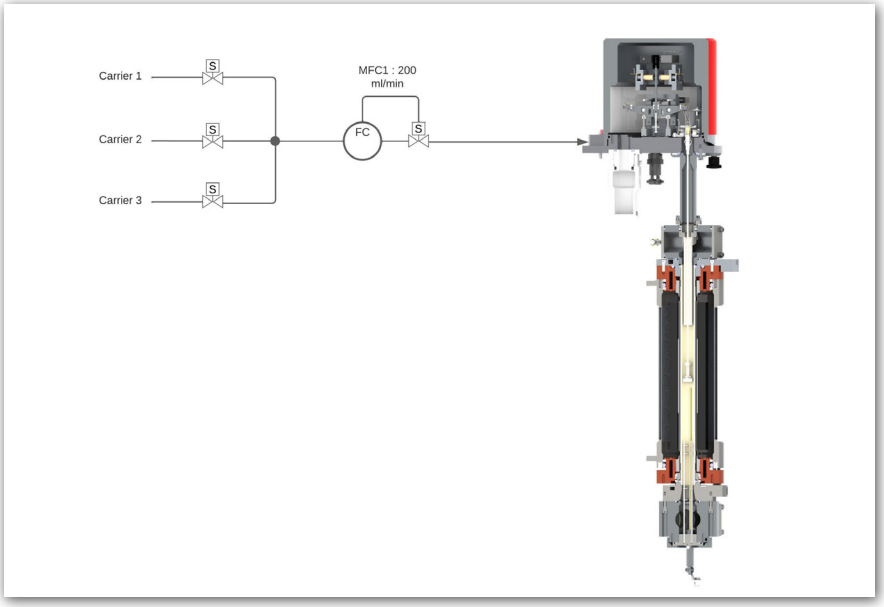

Figure 1 – schematics of the gas flow management system of Themys in its PureGas version.

• GasBlend configuration

The GasBlend configuration adds a second gas line to the carrier gas line. This gas is referred to as auxiliary or reactive.

This auxiliary gas line is equipped with a mass flow controller, a solenoid valve and a three-way valve, all controlled by Calisto software. The maximum auxiliary gas flow rate is limited to 16 milliliters per minute.

Auxiliary gas is either introduced directly between the balance and the oven, or mixed with the carrier gas in proportions chosen by the user and injected into the balance. In the first case, the auxiliary gas may be potentially corrosive to the balance. In the second case, its nature is subject to the same restrictions as the carrier gas.

At any time, solenoid valves and mass flowmeters can be activated to change the nature of the carrier gas, inject auxiliary gas or not, change mass flow rates or the carrier/auxiliary gas ratio. These actions can be programmed to take place before, during or after the experiment.

Auxiliary gas is vented from the system via the same non-return valve as the carrier gas.

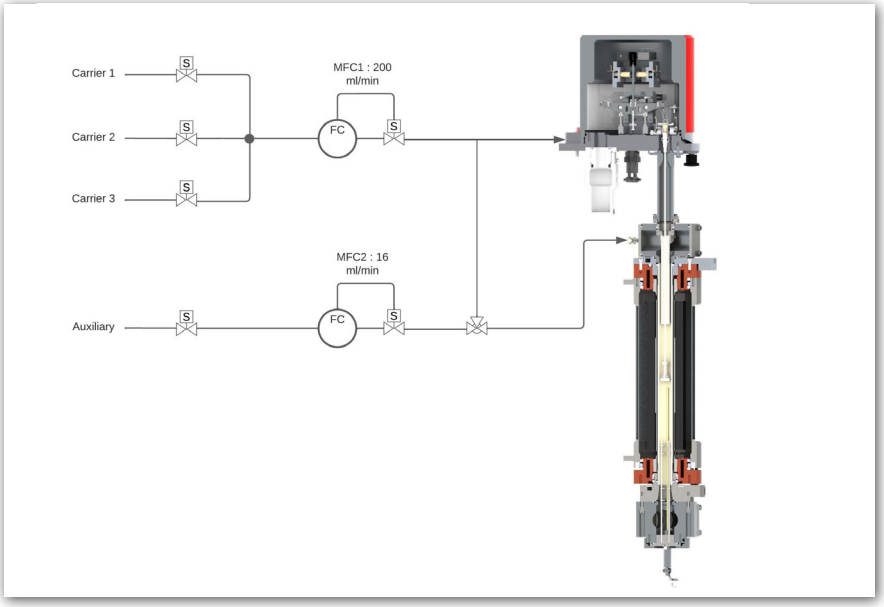

Figure 2 – schematics of the gas flow management system of Themys in its GasBlend version.

• **MultiGasBlend configuration**

An external gas panel called FLEXI GAS is used for this gas management configuration, the most versatile of the Themys range. In addition to all the functions offered by the PureGas or GasBlend configuration, it can be used to add up to 2 additional gases to the gas flow. The FLEXI GAS external gas panel comprises two gas lines, each equipped with a mass flow meter.
• The first gas line has three connectors and solenoid valves for three different types of gas cylinders, as well as a mass flow controller. Maximum flow on this line is limited to 200 milliliters per minute.
• The second is equipped with a connector for one gas cylinder, a mass flow meter and a solenoid valve. Maximum flow on this line is limited to 16 milliliters per minute.
The flowmeters and valves on this external panel are controlled by Calisto software, just like their internal counterparts. The user can change the type of gas, adjust or stop the mass flow rates of all or some of the gases connected to the Themys structure. These actions can be programmed to take place before, during or after the experiment.
This external gas panel can be connected upstream of the balance, if the gas is compatible with the balance, or directly to the furnace inlet, if the gas is potentially corrosive to the balance.

Note: A THEMYS equipped with PureGas and GasBlend configuration can be upgraded at any time to MultiGasBlend configuration by adding the FLEXI GAS external gas panel

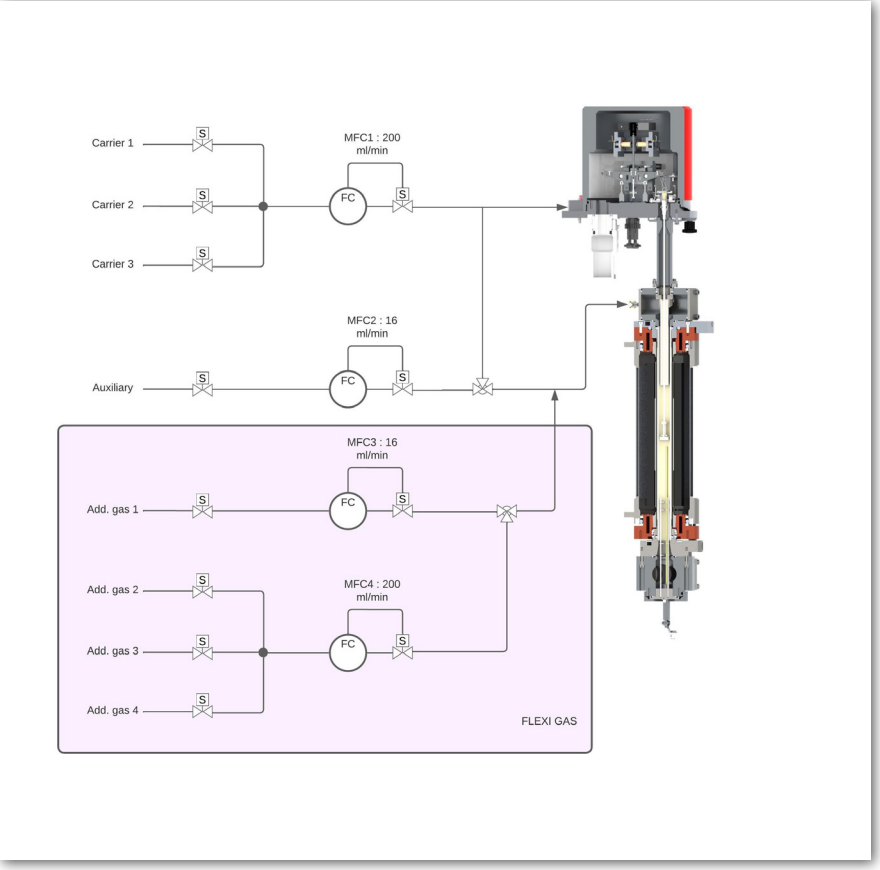

Figure 3 – schematics of the gas flow management system of Themys in its MultiGasBlend version.

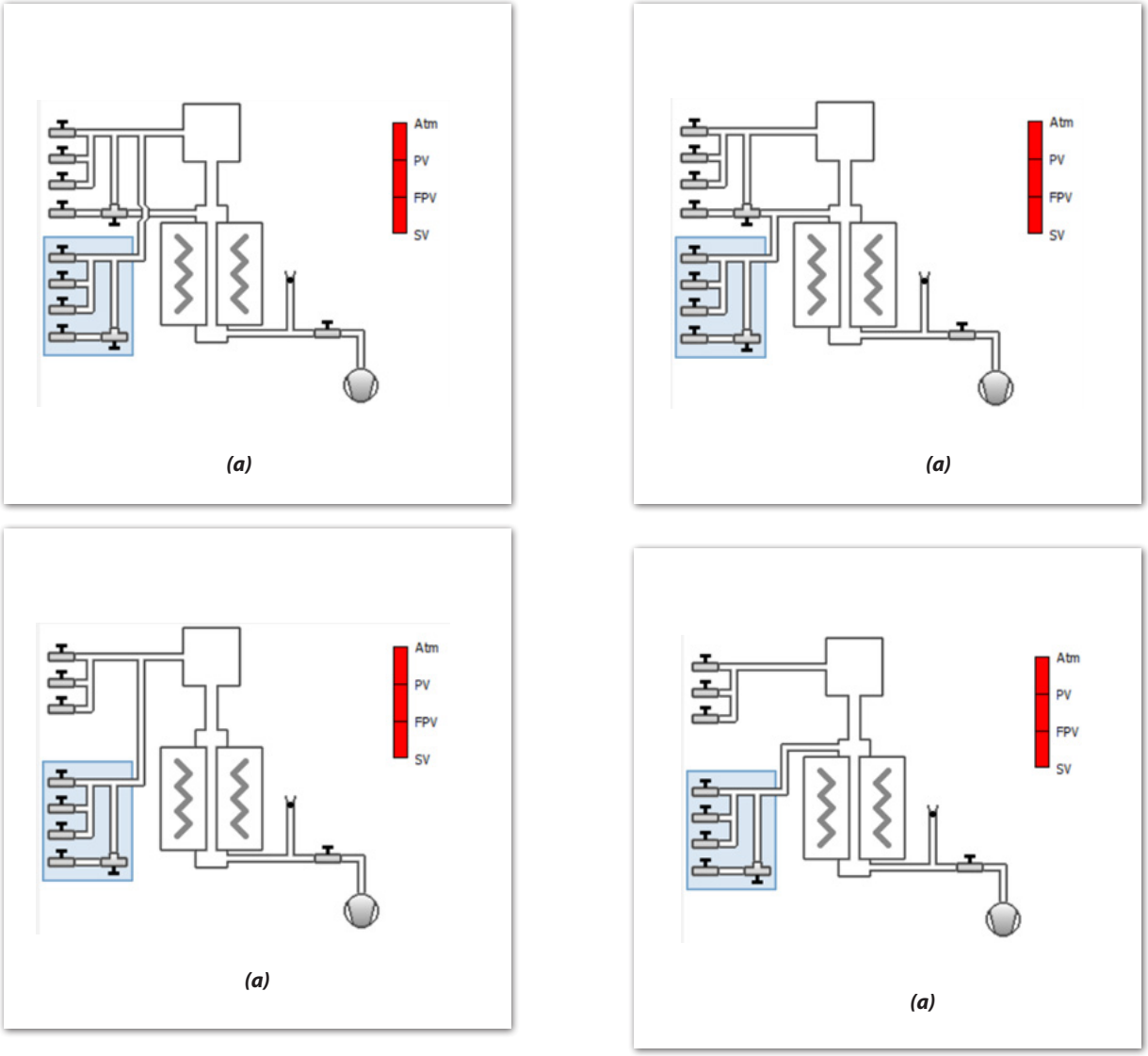

Figure 4 – simplified schematics of the various uses of the FLEXI GAS external gas panel with Themys thermal analyzer.

		Option		
Features		**PureGas**	**GasBlend**	**MultiGasBlend**
Carrier gases (typically inert)	Connected to Themys	Up to 3	Up to 3 Connected to Themys	Up to 6[a,c]
	Flowing the furnace	1 among the connected ones	1 among the connected ones	Up to 3 among the connected ones[a,c]
Auxiliary gas (typically reactive)	Connected to Themys	-	1	Up to 5[b]
	Flowing the furnace	-	1	1 pure[a] OR a blend of 2[d] or 3[b] among the connected ones
Mass Flow Controllers		1 inside the Themys platform	2 inside the Themys platform	1 or 2 inside and 2 outside the Themys platform
Sequential programming		Yes	Yes	Yes

[a] : case (a) on Figure 4 ; [b] : case (b) on Figure 4 ; [c] : case (c) on Figure 4 ; [d] : case (d) on Figure 4

Vacuum level control and monitoring

For many experiments, renewing the atmosphere in the analysis chamber by flushing carrier gas is not enough to ensure perfectly inert conditions. In addition, some measurements have to be carried out under vacuum, for example to simulate processes at sub-atmospheric pressure. In such cases, it is necessary to evacuate the sample chamber.

For example, to remove all traces of oxygen to prevent sample oxidation, or to degas the sample (especially its surface) before introducing a reactive gas.
Depending on the needs of the experiment, different vacuum configurations are available with the THEMYS.
• Primary vacuum with a rotary vane pump placed at the furnace outlet.
• High primary vacuum, with the same rotary vane pump placed at the furnace outlet, but connected to a larger diameter tube than for primary vacuum.
• Secondary vacuum, with an additional turbomolecular pump placed between the balance and the furnace.

Calisto software enables predefined vacuum sequences to be programmed. Opening of the valves for the different vacuum levels is triggered when different programmable pressure thresholds are reached.
A pressure sensor indicates the internal pressure in the analysis chamber. It operates over a range from 0 to 1500mbar. In addition, dedicated vacuum gauges are available for measuring high primary and secondary vacuum.

Furnace protection gas

It should be noted that the furnace heating element must be permanently protected by argon gas flow during all heating and cooling operations. The furnace is designed to allow this gas to be introduced and circulated. The argon is evacuated through an outlet fitted with a one-way valve to prevent any backflow into the device and any overpressure in the enclosure.

2. Corrosive gases

Although corrosive gases are prohibited from flowing in the Themys measurement module (balance or TMA module), it is possible, under certain conditions and with specific configurations, to flow corrosive gases in your furnace. Consult your Setaram contact for more details on these configurations.

In all cases, it is essential to analyze beforehand the compatibility of the intended atmospheres with the materials to be used. Experimental parameters (flow rates, humidity levels) must also be adjusted to avoid concentrating reactive atmospheres (wet, corrosive) in sensitive areas of the instrument.