

Adaptation of a standard thermal analyzer for the characterization of porous materials vs. their volatile organic compounds sorption

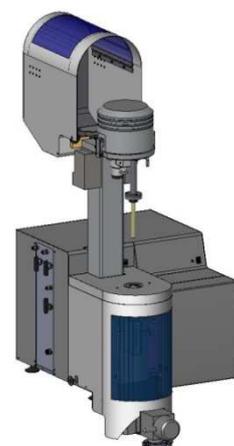
Introduction

The emissions of volatile organic compounds (VOC) has become one of the main concerns of numbers of industries. Due to their major impact on the environment and human health, international policies have become more and more restrictive with regard to VOC emissions. Moreover, their poor chemical compatibilities with many materials may lead to damages when they are evolved in confined systems. Nowadays, several methods are available to control the release including condensation and catalytic and thermal oxidation. These methods are well adapted when the concentration of vapours in the air is very significant. On the other hand, the use of porous adsorbent proved to be a very efficient treatment method for the low concentrations.

However, because of the large diversity of the VOC involved (e.g. aliphatics, aromatics, halogenated) and the number of porous materials available, most of the adsorption properties are still undiscovered. Thermogravimetry is a key technique for the assessment of sorption capacities and kinetics of a couple adsorbent / vapour from vacuum to atmospheric pressure.

Key elements

A specific adaptation was made to the SETSYS Evolution so as to integrate the possibility of working in 3 fully automated modes: The flow mode allows controlling a flow of inert gas charged with a controlled VOC vapors concentration. The dose mode allows injecting customizable volumes of pure VOC vapor in the Setsys to measure adsorption capacity versus VOC partial pressure. Finally, the desorption mode allows measuring TPD coupled with a gas analyzer (e.g. MS, FTIR, GC/MS).

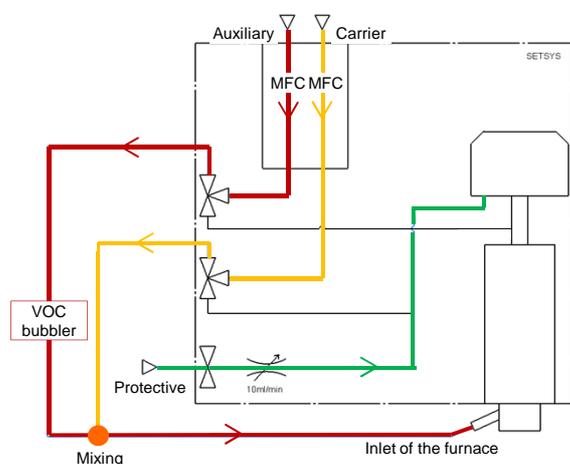


SETSYS evolution with VOC sorption adaptation

Technical achievements

The designed system includes a specific gas handling system fully integrated to the Setsys Evolution structure deeply simplifying the user's operations. A series of 3 way valves allows switching between the 3 measurement modes.

Gas flow mode



Gas configuration for Flow mode

The Auxiliary gas line goes to an external VOC bubbler to saturate the gas with vapor. The carrier gas is connected downstream the bubbler so as to mix saturated and inert gases and control the vapor concentration. Both flowrates are software controlled and programmable. A constant flow of inert gas protects the weighing module from the vapors.

www.setaram.com
sales@setaram.com



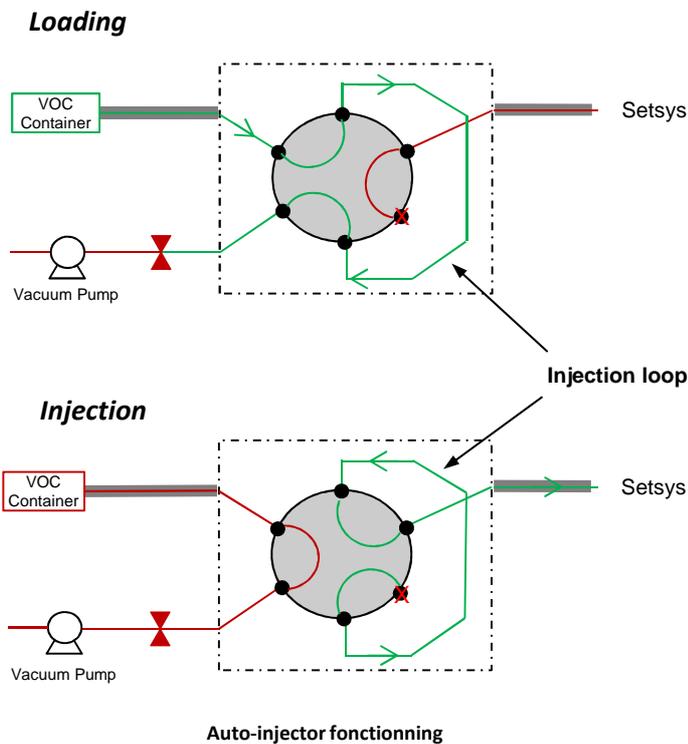
Dose mode

A fully automated injector is connected between a VOC container and the Setsys. It is based on an injection loop connected to a 6-way valve. The whole assembly is temperature controlled and the vapors are carried through the system *via* two heated transfer lines.

At the beginning of a test, the loop and the VOC container are pulled under vacuum to remove any trace of air. The adsorbent sample is also degassed thanks to a secondary vacuum pump.

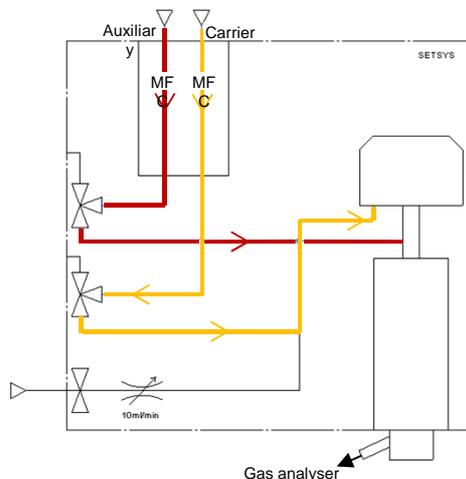
For each dose, the loop is initially loaded with an amount of VOC vapor. When the 6-way valve turns the volume of vapor contained in the loop is released in the Setsys. A limitless number of doses can be programmed. The mass gain due to sorption and equilibrium pressure are measured.

In this purely static mode, there is no flow of protective gas in the weighing module. To prevent any risk of condensation, the balance housing is heated at a temperature slightly above room temperature (typically 40°C but could be heated to 80°C).

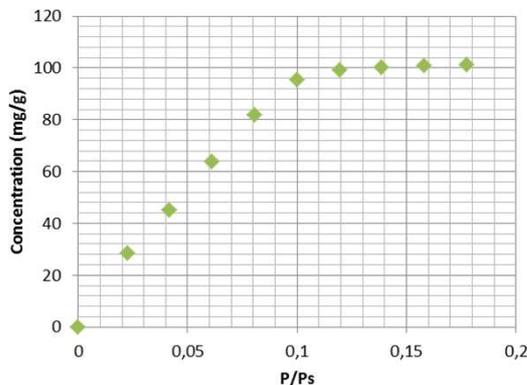
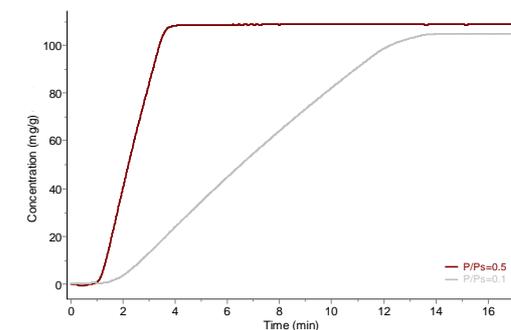


Desorption mode

The system can easily be switched into the desorption mode by driving the 3-way valves of the VOC option. This mode allows programming a standard TGA measurement coupled with an evolved gas analyzer (MS, FTIR, GC-MS).



Gas configuration for Desorption mode



n-hexane adsorption on MFI-type zeosil (silicate-1 zeolite) at 30°C - Concentration vs time (up) or equilibrium pressure (down) plots obtained under flow and dose modes respectively. Results are comparable to literature values [1].

[1] Daou et al, Microporous and Mesoporous Materials 173 (2013) 147–154