

Adaptation of a standard thermobalance for measurements using multiple simultaneous analytic methods

INTRODUCTION

Coupling analytical methods can be a way to draw more information from a single sample to understand more into depth its chemical / physical behavior, or to obtain simultaneous data under the exact same conditions. But it requires the evolution of standard analyzers so that they can physically fit and that they are not disturbed by their simultaneous operation.

A good way to determine the adsorbed species on the surface of a catalyst is infrared spectroscopy, while thermogravimetry is a good tool for the quantification of those adsorbed species. The Laboratory of Catalysis and Spectrochemistry of Caen (France) approached KEP Technologies to modify a standard microbalance in order to fit it with operando IR spectroscopy. The goal was to develop a TG-IR coupling with an IR beam directly oriented to the surface of the catalyst being weighed by the balance.

Key elements

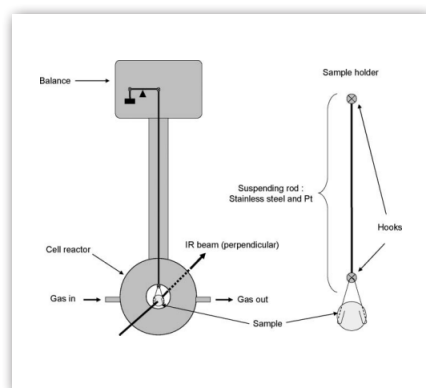
Our skills in customized instrument design, our expertise in thermobalance development, and the organization of our R&D service being structured to manage specific projects have been applied for a long time in such cases. The microbalance had thus to be adapted to fit with the furnace developed by a third party. The latter was equipped with a KBr window, allowing the focus of the IR beam on the sample. The selected weighing module was based on the principle of the articulated beam on a torsion wire successfully applied in Setaram TGA instruments.

Technical achievements

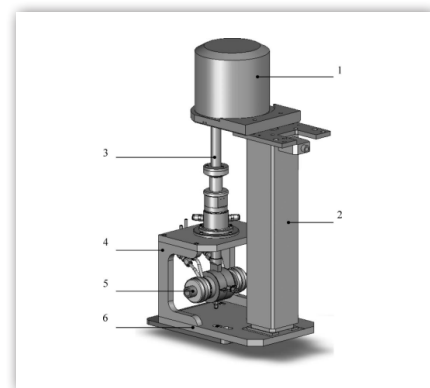
The provided setup was based on the following elements:

1. The microbalance with its electronic controller, data acquisition and treatment software
2. A lifting device meant to place and remove the sample in the furnace
3. A series of metal parts and O-rings tightly connecting the balance with the furnace

Moreover, the sample shape being a disk, a specific holding part had to be developed in order to expose one of its surfaces to the IR beam and keep the angle between the beam and the disk steady during the experiments. It was based on several suspensions whose top and bottom had the shape of hooks and a platinum wire on which the sample could be inserted. The whole setup is described in [1] together with typical application examples.



Schematics of the setup after microbalance integration (reproduced from [1])



Going further

A number of microbalances have been adapted to cover specific needs :

- measurement of humidity uptake in climatic chambers
- multiple (5) samples weighed and tested simultaneously against their resistance to oxidation in very fast temperature scanning furnaces

[1] P. Bazin and al, Dalton Trans., 2010, 39, 8432–8436