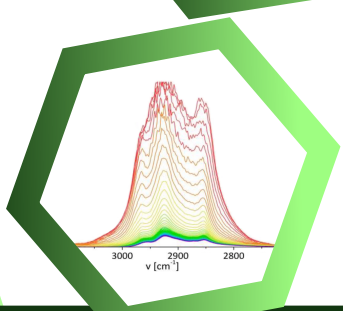
MEASLINE
ADVANCED SOLUTIONS

The HT-IRS 01 Reactor for spectroscopic studies



The device adapted to IR spectrometers working in transmission mode.

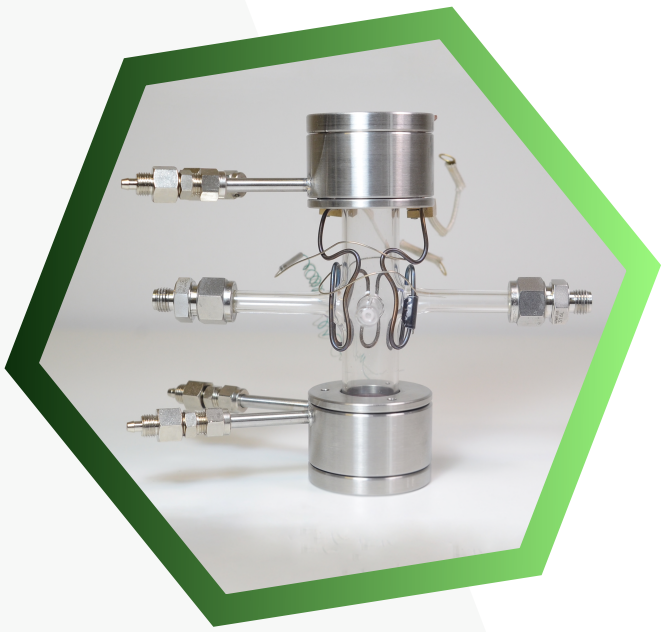
The HT-IRS 01 reactor is an extension of spectrometers working in infrared, and it is fully compatible with all commercially available IR spectrometers. The device is dedicated to working in the transmission mode of optical spectroscopy. Furthermore, its unique construction allows it to be easily combined with other analytical methods like mass spectroscopy, gas chromatography, and more.

Motivation to create the HT-IRS 01 Reactor

Infrared spectroscopy is an universal, non-destructive technique used to identify the molecular structure. Spectroscopic IR studies of the phenomena occuring on the working-catalysts provides both qualitative and quantitative information in the nature of the surface centers, binding the reagent molecule to the catalyst surface and the successive transormation of the latter. Quantitative IR studies enable the determination of the concentration of centers responsible for the course of a specific reaction. Such studies are possible only through the transmission technique, in which the light passing through the sample is analyzed.

The currently available apparatus for spectroscopic examinations has several limitations, including too narrow ranges of reactor operating temperatures, often the inability to experiment both in vacuum and reagent flow conditions, as well as disturbing the results through interactions with substances released from metal and polymer reactor structures.

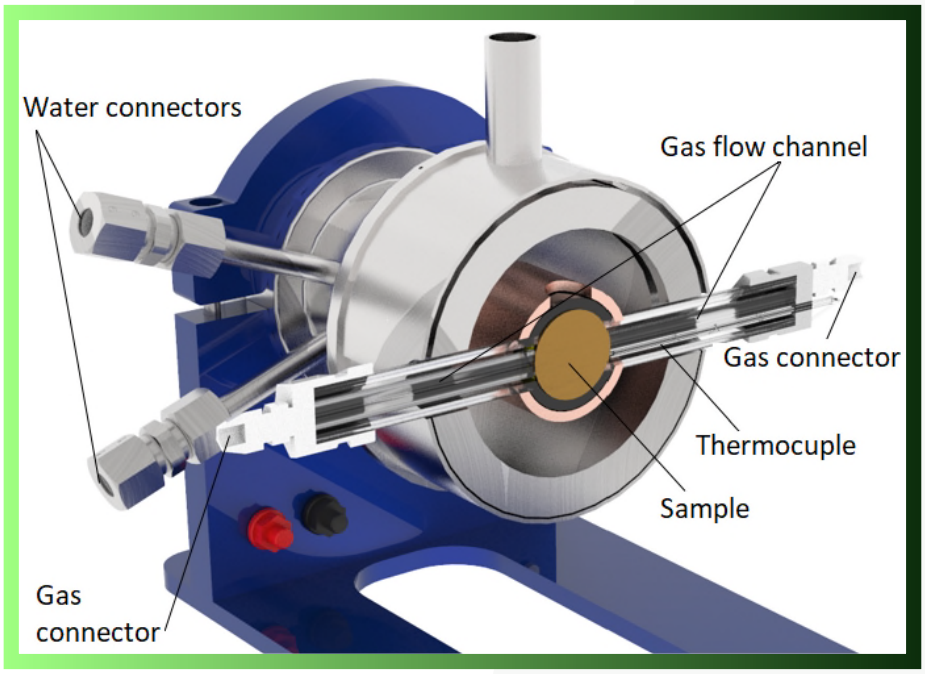
The most important features of the device

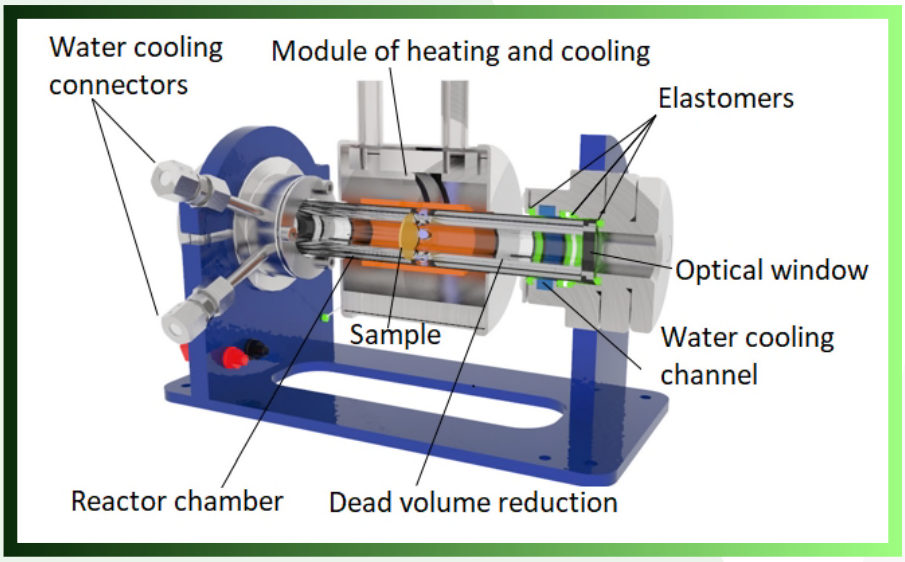


- ensuring the chemical passivity of the analytical chamber by making it entirely of quartz;
- wide range of sample activation temperatures from room temperature to 1000 K;
- wide range of operating temperatures from 100 to 1000 K;
- working temperature stabilized by the use of a thermocouple located just above the sample, coupled with the cooling and heating system. Contact of the thermocouple with the gaseous medium is eliminated by placing it in a quartz tube;
- increased cooling efficiency due to the direct contact of the cooling liquid with the reaction chamber in the area of seals;
- chamber tightness at the level of 10^{-9} mbar*l/s, enabling the experiment to be carried out in vacuum conditions;
- the ability to work also into gas flow;
- elimination of the undesirable phenomenon of water adsorption and the corrosion of the system components induced by the reagent. Above is ensured by elastomeric seals kept at the temperature of the cooling water. The location of the seals provides an almost complete absence of contact with the gaseous medium filling the reactor.

Construction of the HT-IRS 01 Reactor

The essential part of the HT-IRS 01 reactor is the reaction chamber, comprising a cylindrical quartz tube with at least two perpendicular outlets that allow for providing the gaseous working medium to the reaction area with the test sample placed. The third outlet, perpendicular to the others, ensures the thermocouple placement in the reaction chamber just above the sample. The thermocouple is placed in a quartz tube to avoid interaction with the reactants. At the ends of the reaction chamber, optical windows sealed with elastomeric seals separated from the reactants are placed.





The desired temperatures are obtained using a heating and cooling system located directly on the reaction chamber and surrounding it. The thermocouple coupling with the heating and cooling system ensures the fine temperature adjustment.

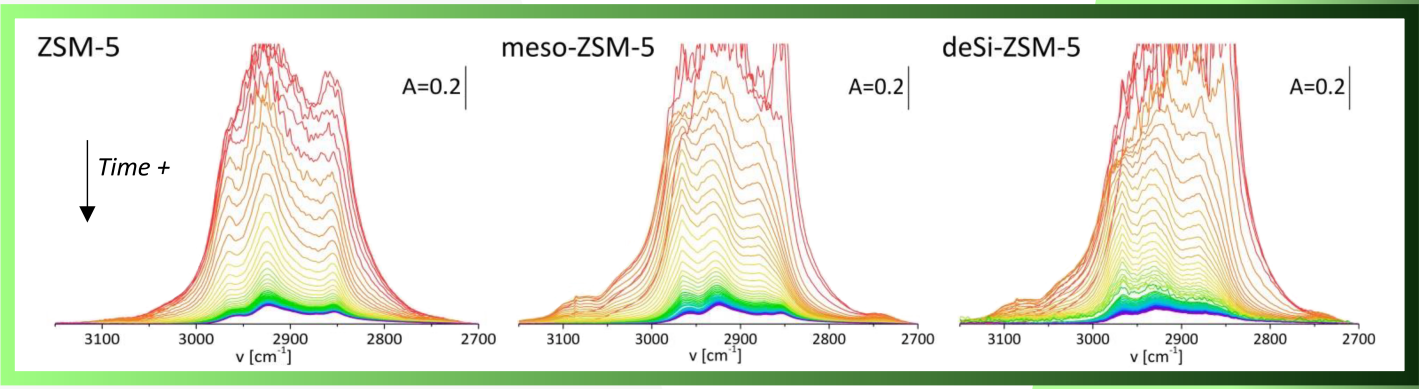
The reaction chamber is placed on an aluminum frame using fixing systems holding the two opposite ends of the chamber. The chamber with the heating and cooling system is placed in an internal and external thermal shield.

The temperature inside the reactor is adjustable by the TCT 02 controller. TCT 02 is equipped with a temperature regulator connected to a thermocouple integrated with the heater. The device also has a temperature display that allows the temperature from the thermocouple reading. The controller can be connected to a computer via USB input.

Additional advantages of the HT-IRS 01 Reactor

- minimized dead volume of the reactor chamber ensures real-time recording of phenomena. The dead volume is reduced by thick wall quartz fittings, and it does not exceed 1 cm^3 ;
- simple disassembly and assembly during changing the sample or cleaning the chamber;
- possibility of cooling the quartz chamber with liquid nitrogen;
- minimization of costs related to the operation of the device in the field of optical components;
- reactor equipped with a dedicated temperature controller.

Example of results of tests with the HT-IRS 01 Reactor



FTIR spectra obtained during measurements in operando mode (in the flow of reaction gases), resulting from the stretching vibrations of C-H bonds of compounds present on the catalyst surface during LDPE cracking at a temperature of 230 ° C.
These results were published in a publication available on https://doi.org/10.1002/cssc.201802190.

The device was developed in cooperation with the Jagiellonian University in Krakow.

The reactor is covered by a patent: Pat.232633.

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