# A newly developed attachment to the Lambda 9 spectrometer for in situ reflectance measurements of catalysts at temperatures up to 623 K

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## Abstract

The spectroscopic investigation of catalyst powders under reaction conditions is generally possible only in reflectance.

Two problems occur:

1. Low signal level

Normally the sample is positioned directly at the integrating sphere and all diffusely scattered light from the sample is reflected back into the integrating sphere.
A hot reactor cell cannot be attached directly, it must be positioned as far as possible from the integrating sphere to avoid its heating.
If the reactor cell is attached at some distance from the measurement window of the integrating sphere only a portion of the diffusely scattered light will be reflected back into the sphere. At a distance of 12 mm, for example, this will only be 20%.
In a first setup we bridged the distance with highly reflecting ceramics, to increase the part of light reflected into the sphere (Fig.1).

2. Thermal radiation

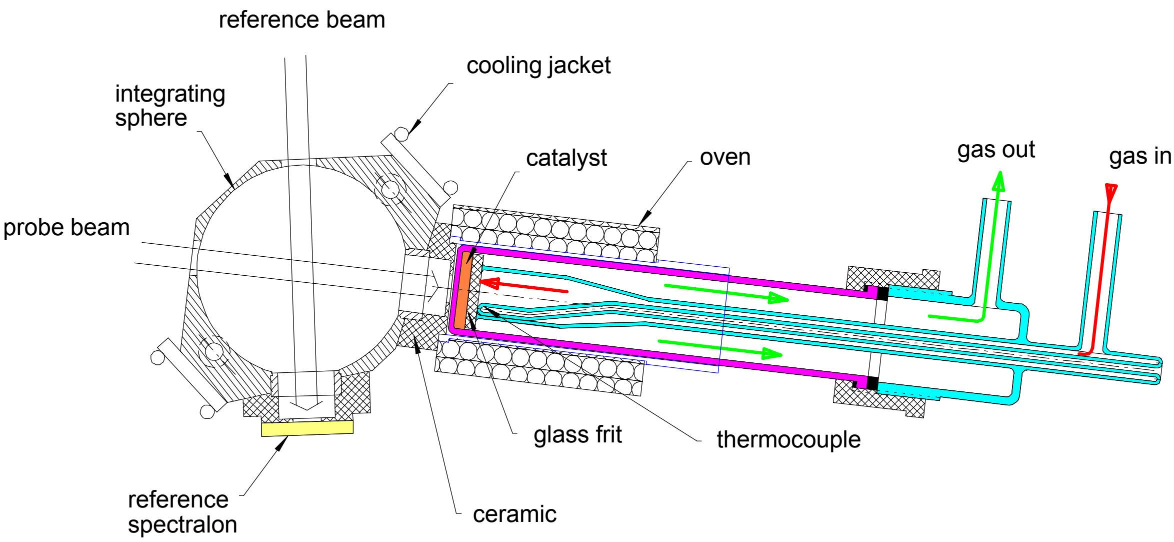
The thermal radiation of the hot reactor cell also enters the integrating sphere.At higher temperatures it is significantly stronger than the measurement light from the spectrometer itself. This leads to increased noise and saturation of the detector.

In the newly developed setup we tried

a. to make the distance between integrating sphere and reactor cell with oven as large as possible;
b. to increase the signal level
c. to decrease the surface which causes the thermal radiation, and
d. to bring reactor cell and oven into a vertical position which facilitates the work with powder samples.

These requirements could be fulfilled by the application of a specially formed light conductor made of quartz, in which the light is conducted to the sample and back into the integrating sphere by total reflectance. At the same time, the surface which causes the thermal radiation was reduced (Fig.2).

In this way, we succeeded in improving the signal-to-noise ratio by a factor of 4-5 compared with the first setup.


reference beam
cooling jacket
integrating sphere
catalyst
oven
gas out
gas in
probe beam
glass frit
thermocouple
reference spectralon
ceramic

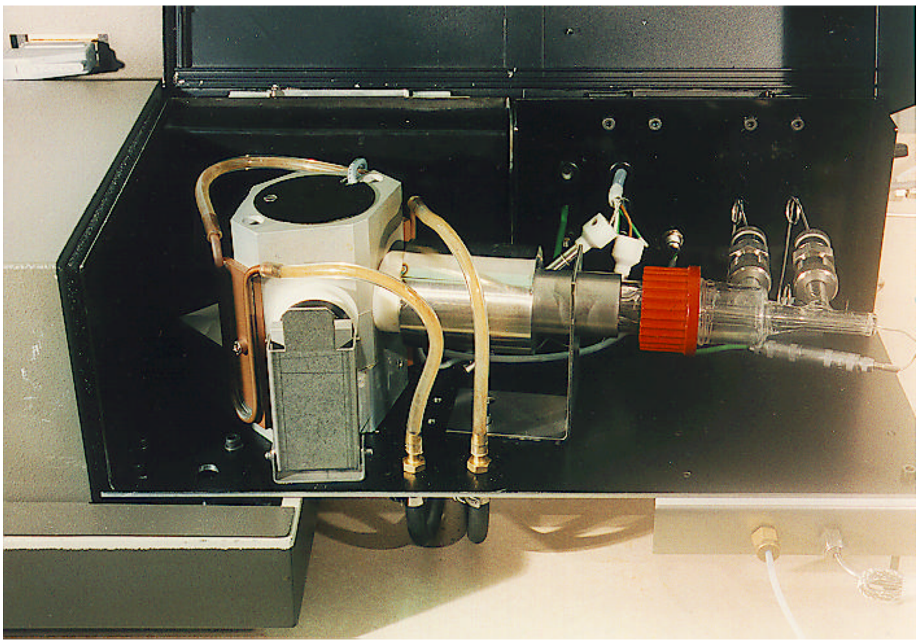
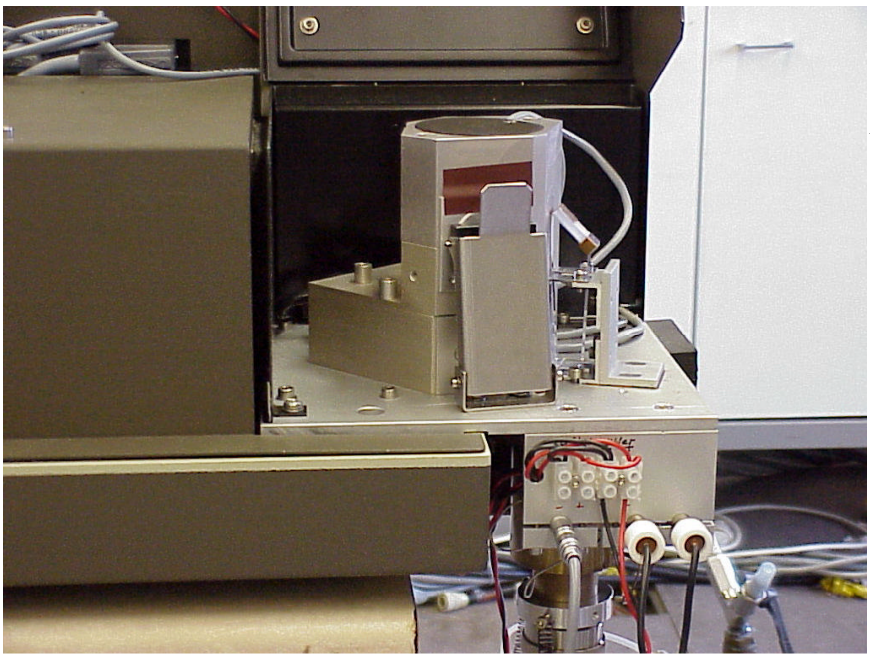


Fig. 1



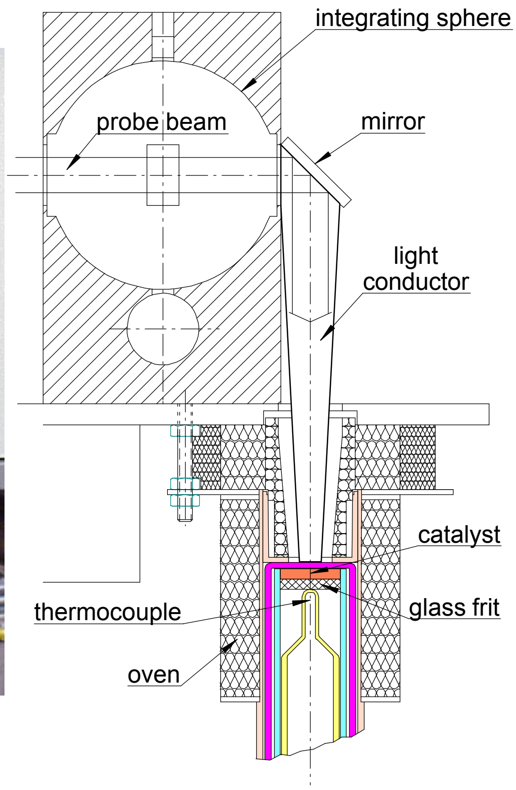

integrating sphere
probe beam
mirror
light conductor
catalyst
glass frit
thermocouple
oven

Fig. 2