ExtraSolution

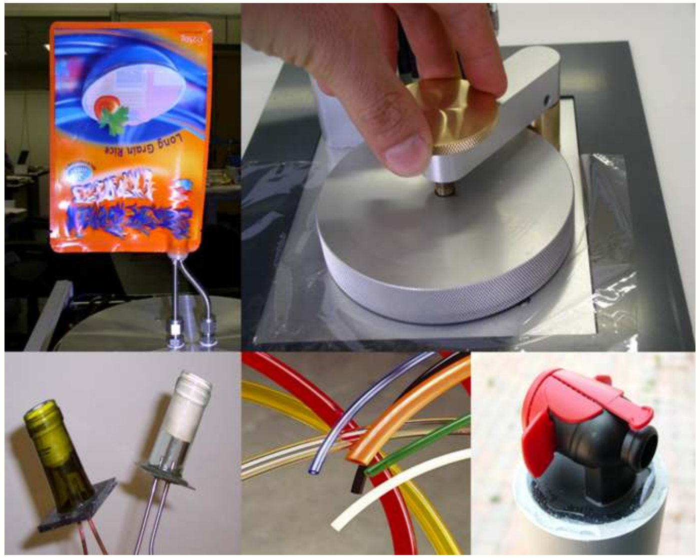
New techniques for measuring the gas permeability

Summary

- Company overview
- Typical measurement times for permeability tests
- OTR tests through 3D packages
    - Prior Art
    - Invention
    - Advantages
- GTR tests through films and sheeting
    - Prior Art
    - Invention
    - Advantages

Permeability testers

We can test every objects that makes barrier.



TotalPerm

- **OTR, WVTR and CO_2TR**
- **Three sensors**
- **Sequential tests**
- **Both films & packages**

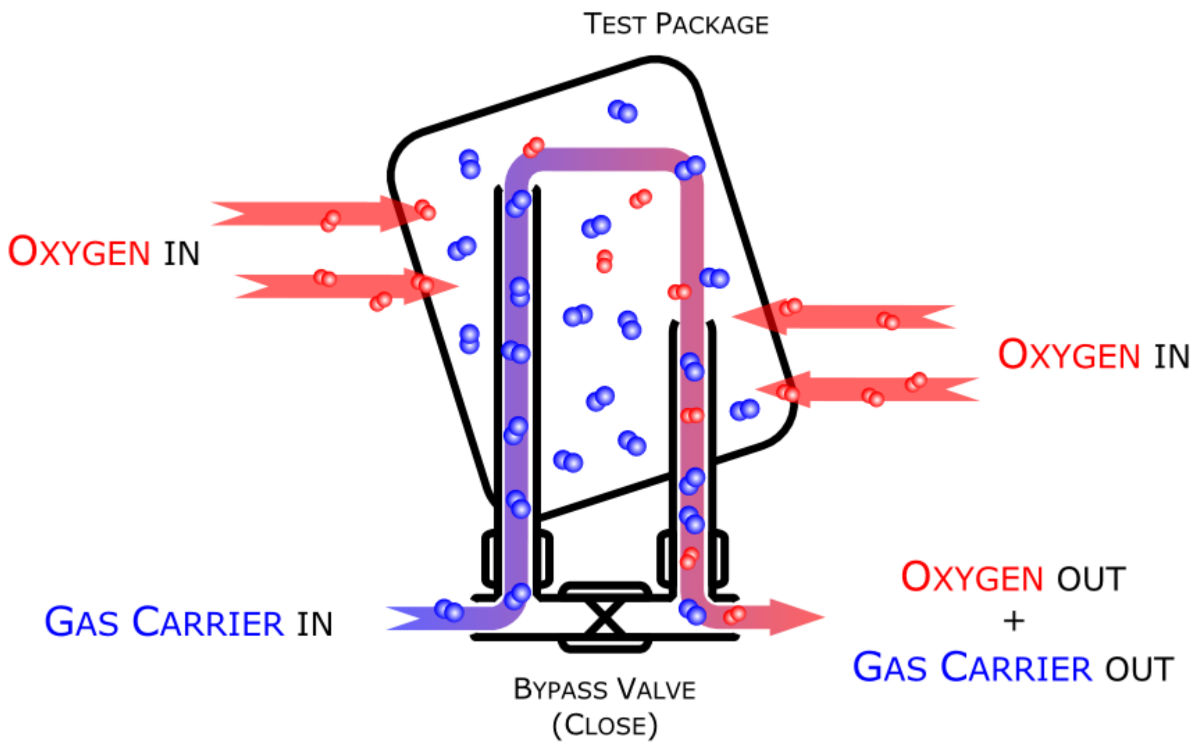
Time

Table indicating the typical duration of the gas permeation process through some widely used plastic materials for food packaging.

Sample	Size	Time (approx.)
Film PET biaxial	23 µm	1 hour
Film PET biaxial	250 µm	4 days
Film LDPE	60 µm	0,5 hour
Alu film (bag-in-box)	60 µm	5 hours
Syntethic cork	Ø18 x 40 mm	10-20 days

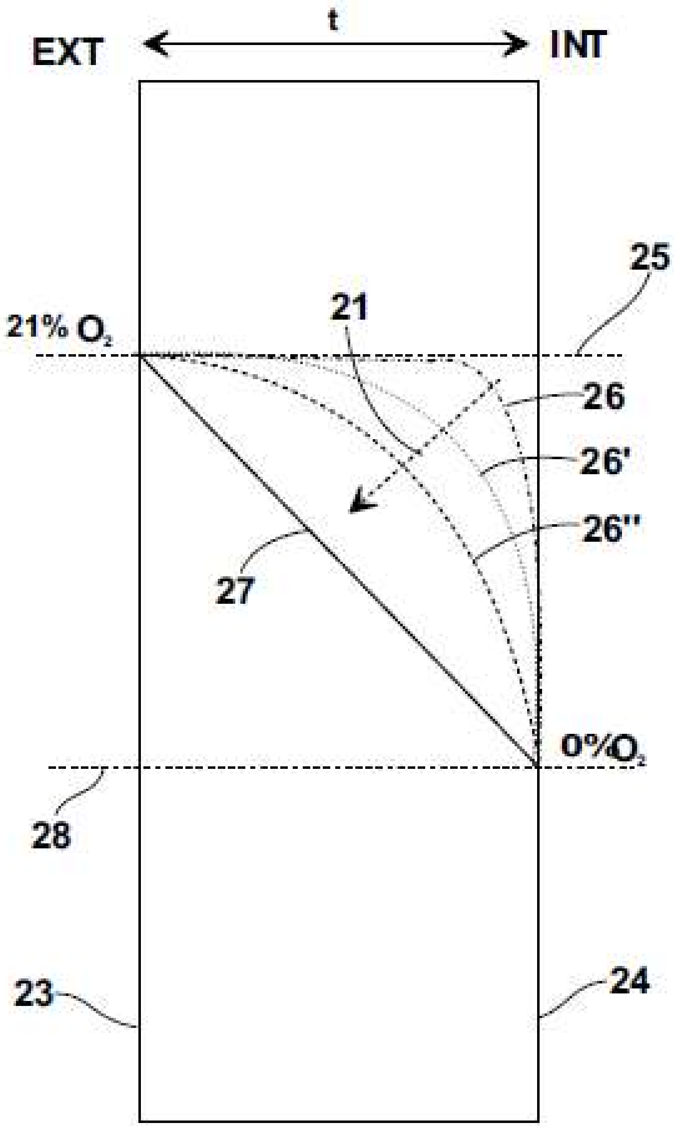
Oxygen Transmission Rate (OTR) tests through 3D dry packages.

Prior Art (1)



An electronic controlled nitrogen flow starts flowing through the sample. The O_2 present in the sample is purged by the carrier gas and it is transported to the sensor.

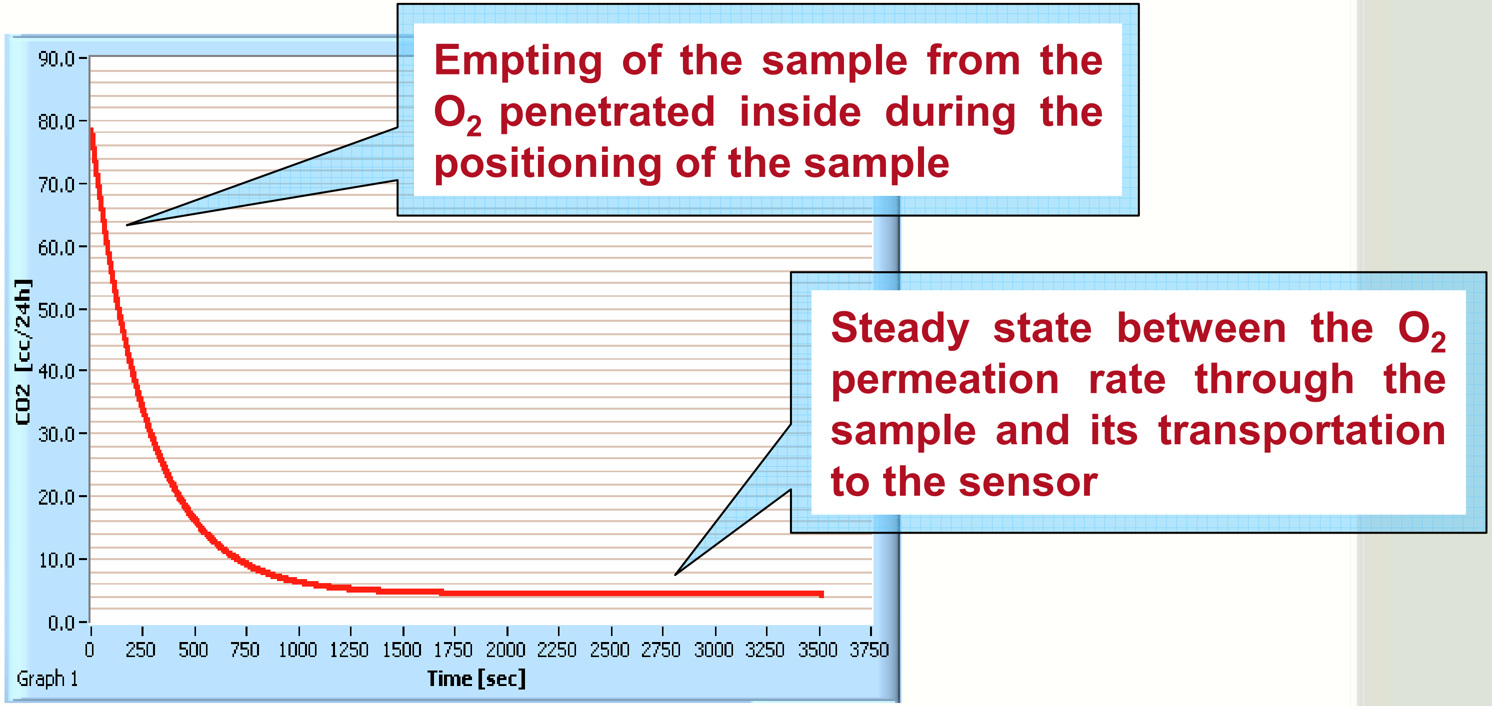
Prior Art (2)



Transient phase of the permeation through a wall cross section of a container having a generic thickness t whose external surface comes into contact with air (21% O_2) and the internal one with the carrier gas (0% O_2).

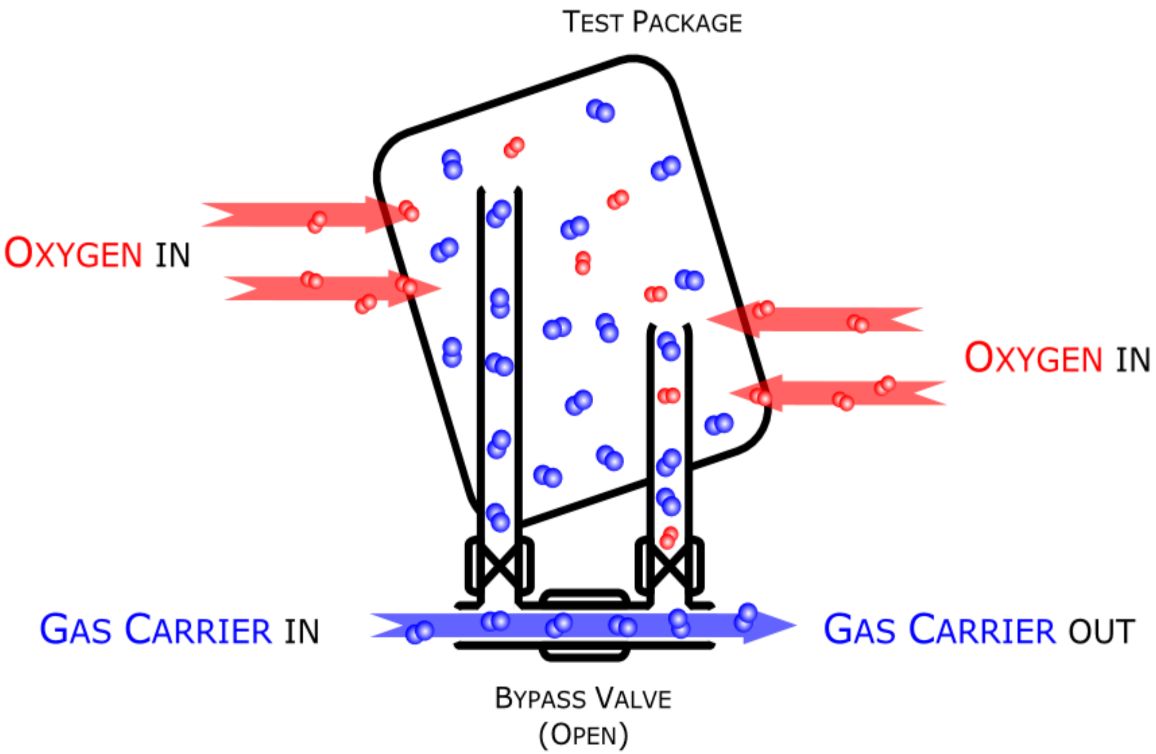
Prior Art (3)

The graph shows a decreasing curve until it stabilizes to a value of plateau.



The value of the plateau indicates the amount of O_2 permeated through the sample in addition with any other amount of O_2 present in N_2 carrier before getting into the sample.

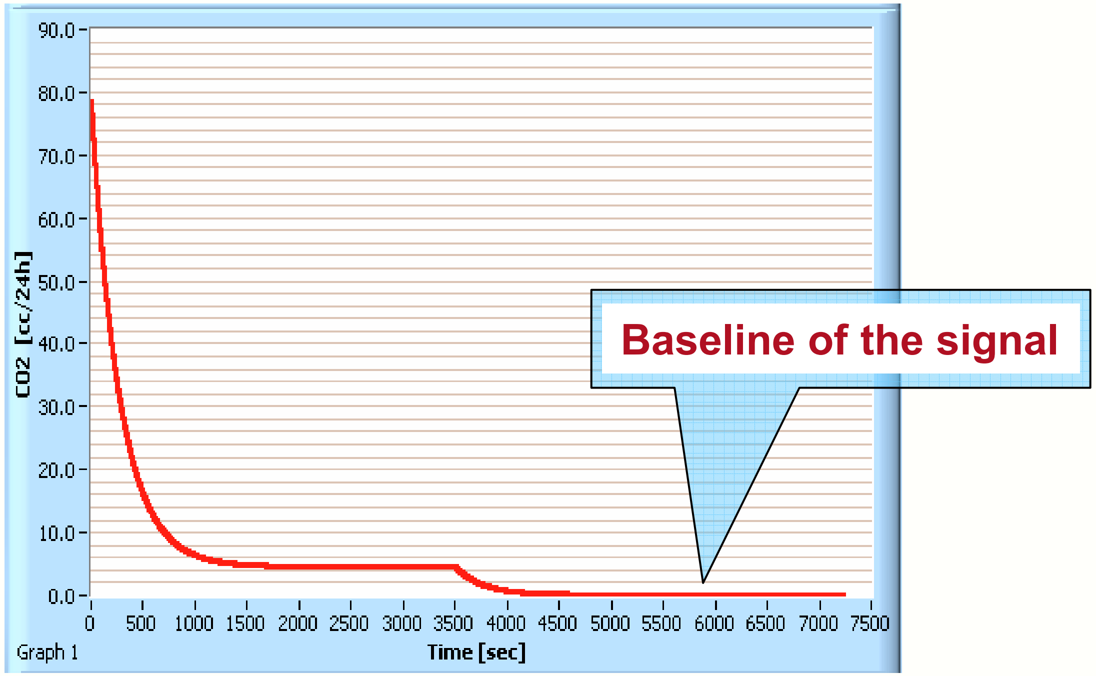
Prior Art (4)



Then the gas carrier by-passes the sample and starts flowing directly to the sensor.

Prior Art (5)

The graph shows again a decreasing curve that stabilizes to a new plateau.



The "zero value", the baseline of the signal, takes in account both any possible leaks present in the gas line or any impurities present in the carrier gas.

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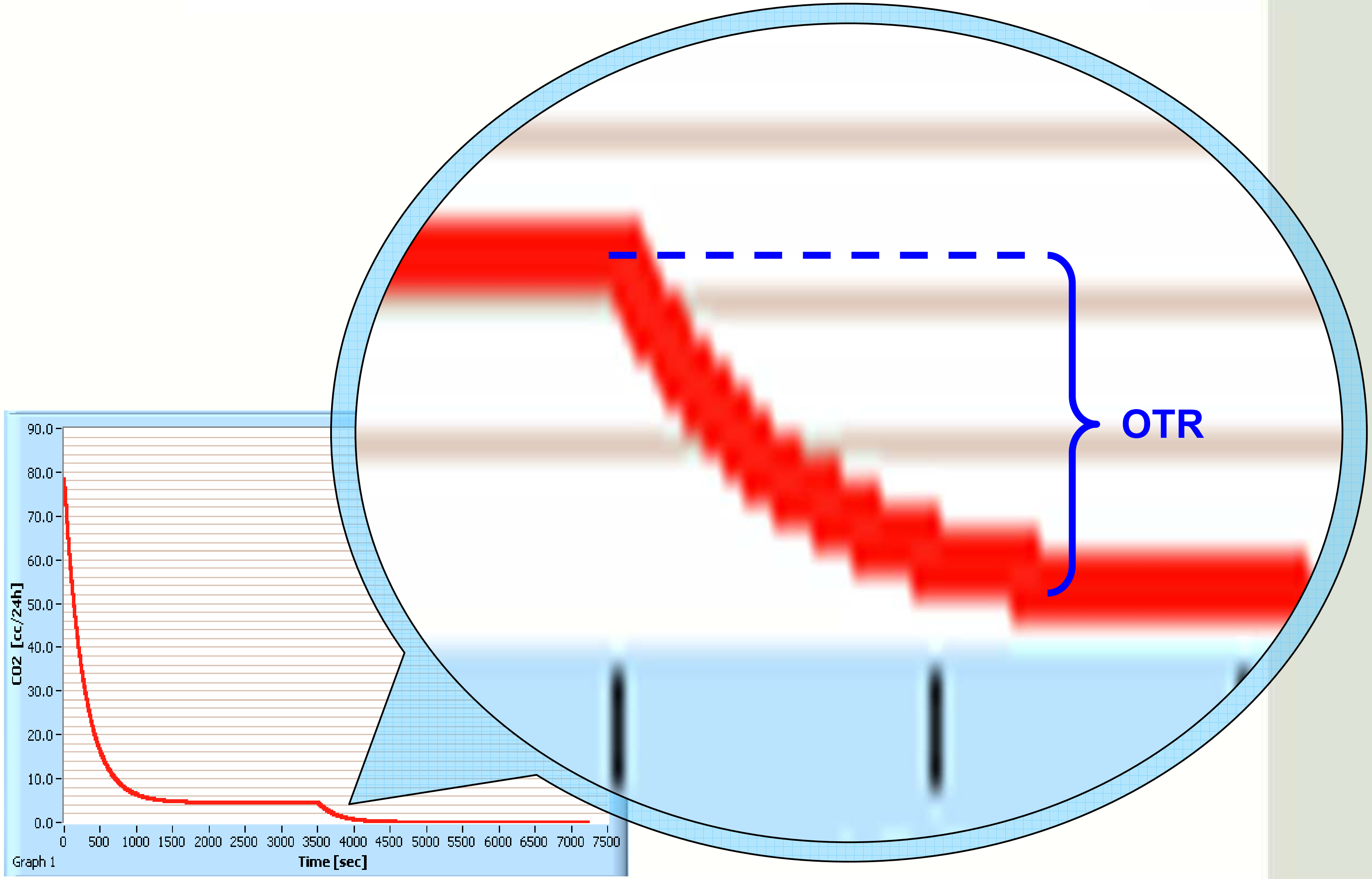
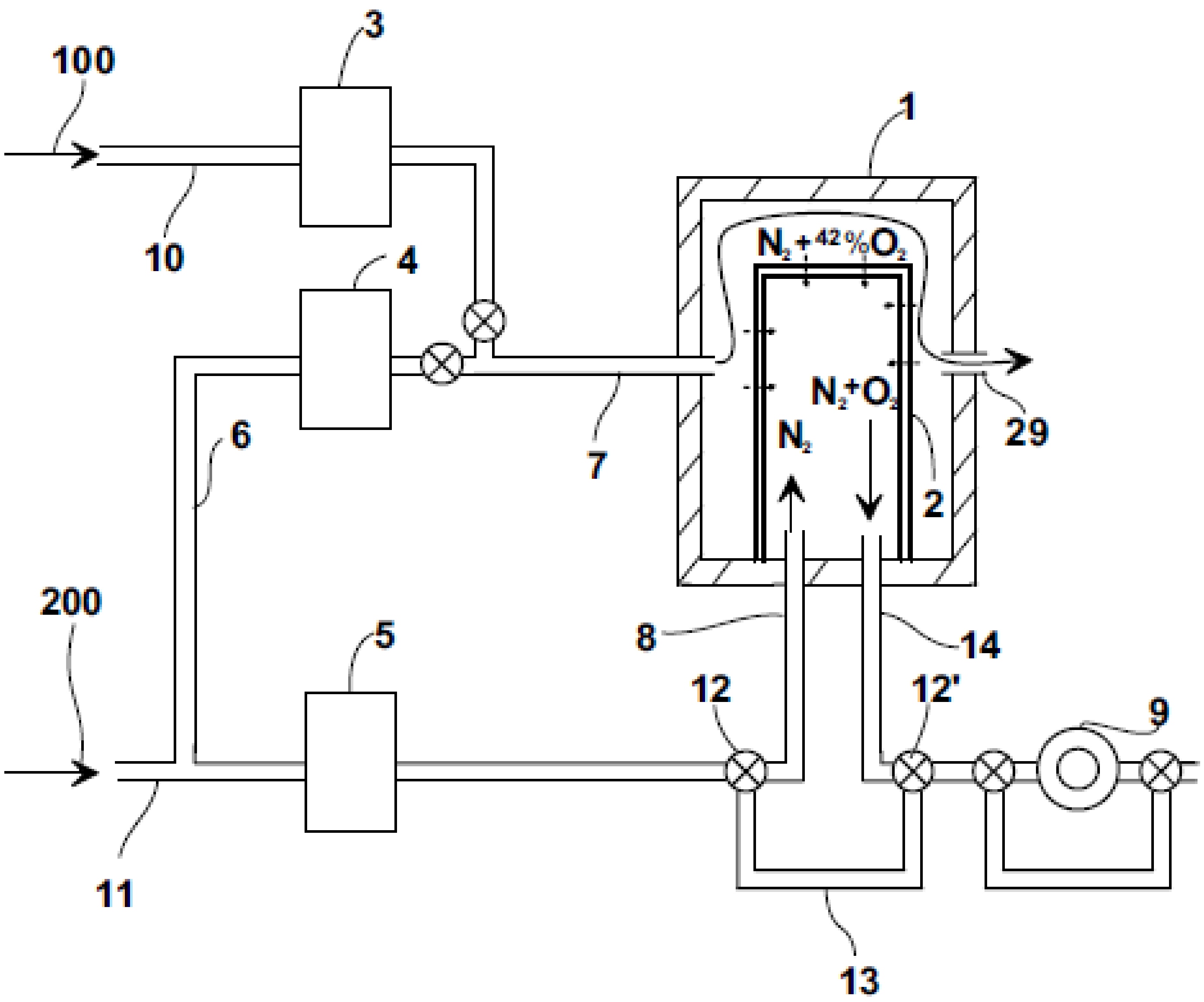
Prior Art (6)

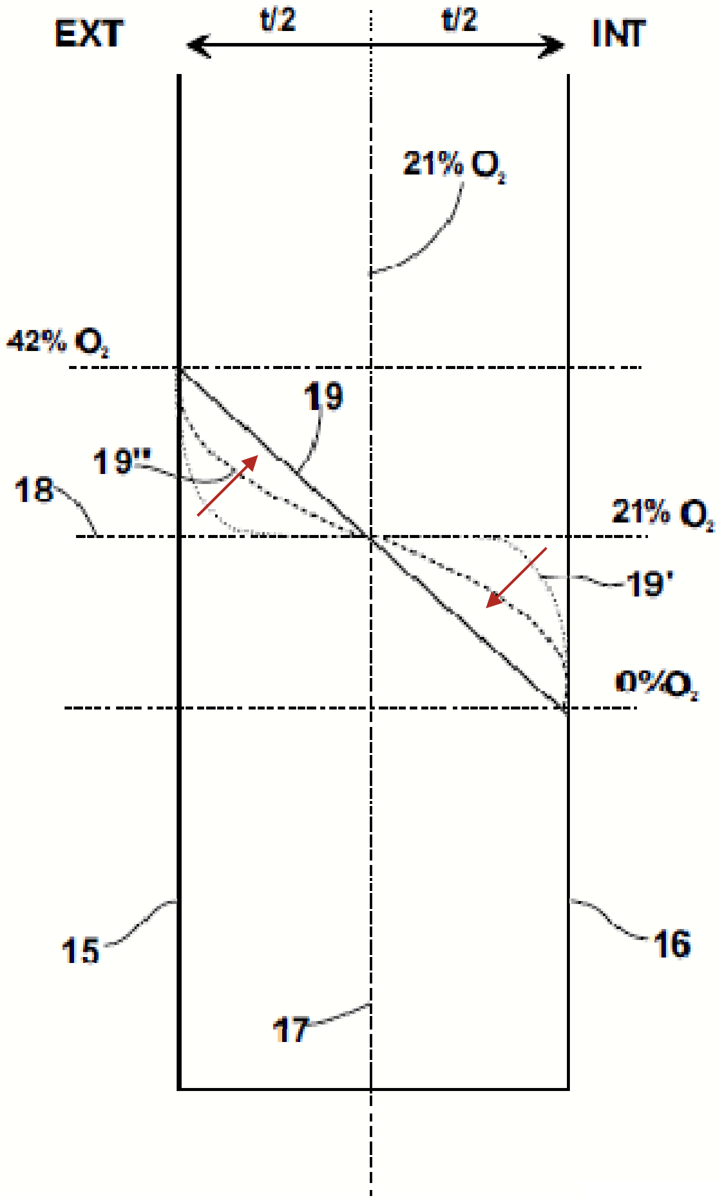


The software automatically calculates the differential between the two values and it gives the result.

Patent "Double concentration" (1)



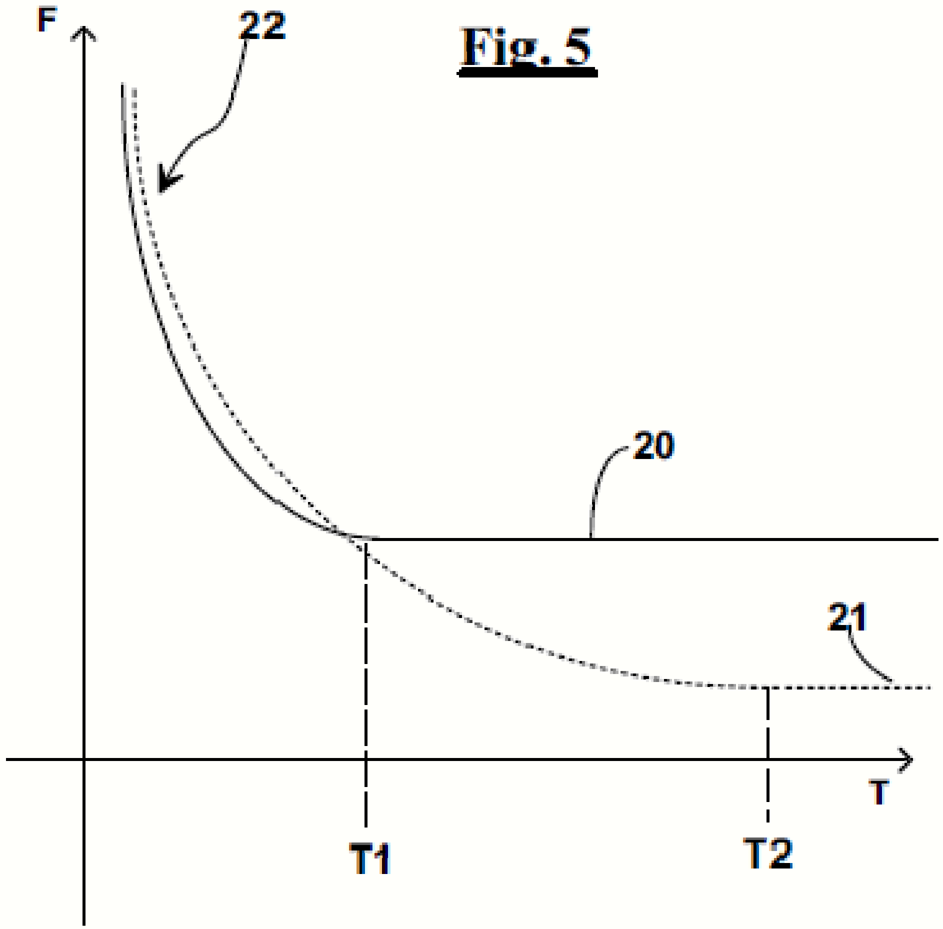
Patent "Double concentration" (2)



Transient phase of the permeation through a wall cross section of a container having a generic thickness t whose external surface comes into contact with gas mix (42% O_2) and the internal one with the carrier gas (0% O_2).

Patent "Double concentration" (3)

Fig. 5



Comparison of a chart of the O_2 flow that permeates with time through the cross section of the container according to the invention (——) and according to the prior art (·······).

Patent "Double concentration" (4)

It is possible to demonstrate analytically that the minimum diffusion time through a monolithic material with constant thickness is obtained with $[O_2]_{ext}$ = 42% and $[O_2]_{int}$ = 0%.

The method also works with multi-layer materials and/or different thicknesses. In these cases, to minimize the diffusion times, $[O_2]_{ext}$ will have to be optimized each time.

<u>In any case the diffusion time according to the invention is going to be shorter than according to the prior art.</u>

Patent “Double concentration” (5)

Main Advantages

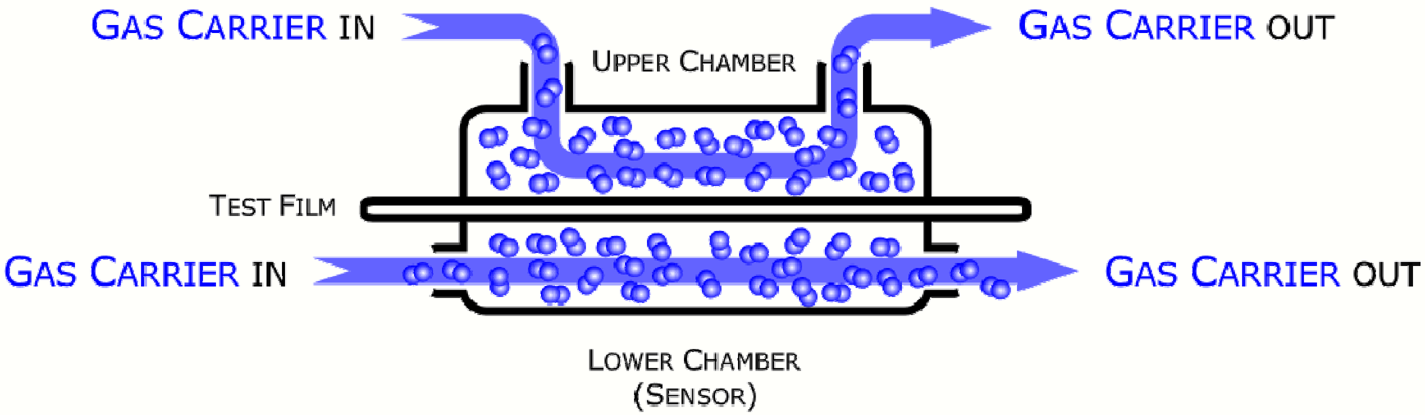
Measurement time reduced to a quarter.

Amplification of the measured signal ($O_2TR \propto \Delta[O_2]$).

Elimination of the need for a possible preliminary conditioning of the sample.

Gas Transmission Rate (GTR) tests through plastic films and sheeting.

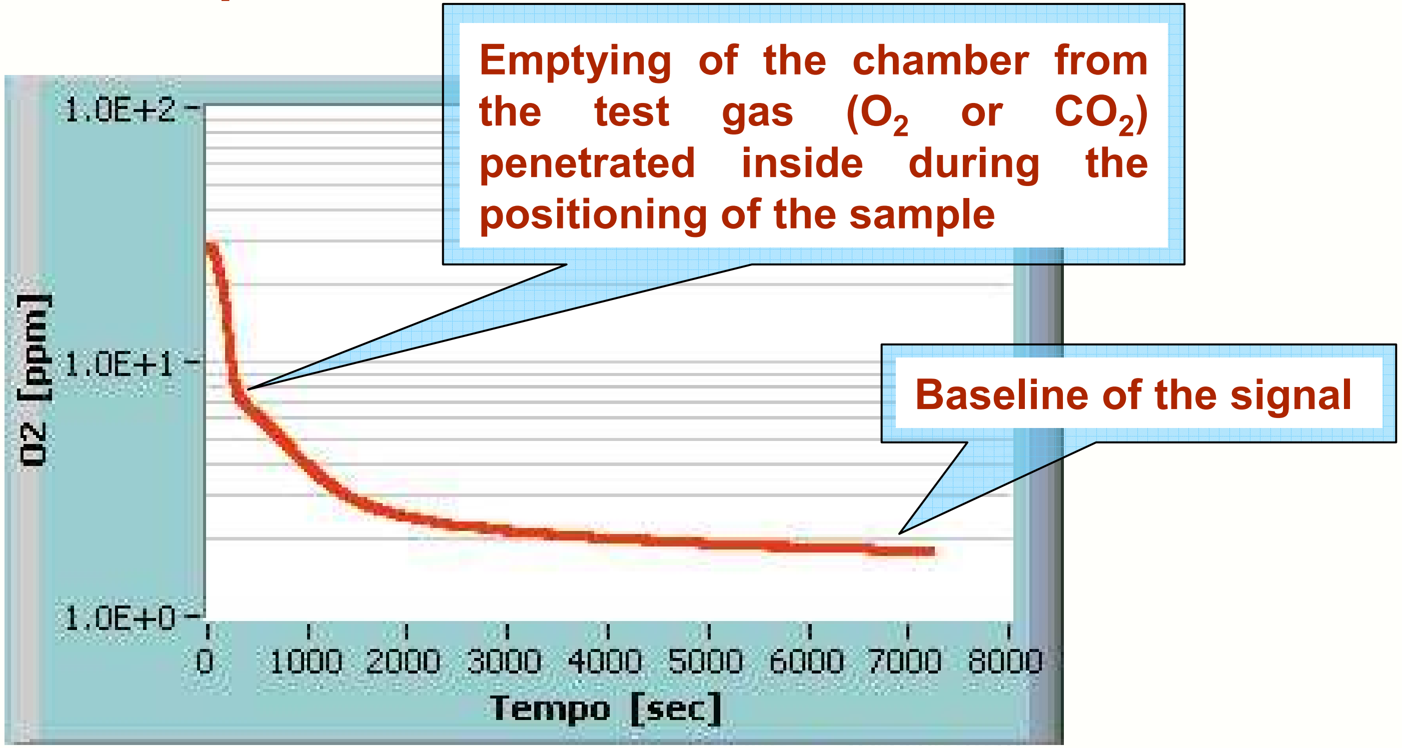
Prior Art (1)



An electronic controlled nitrogen flow starts flowing in both the chambers (purging phase). Residual traces of gas (O_2 or CO_2) present in the chamber are swept away by the carrier gas and transported to the sensor.

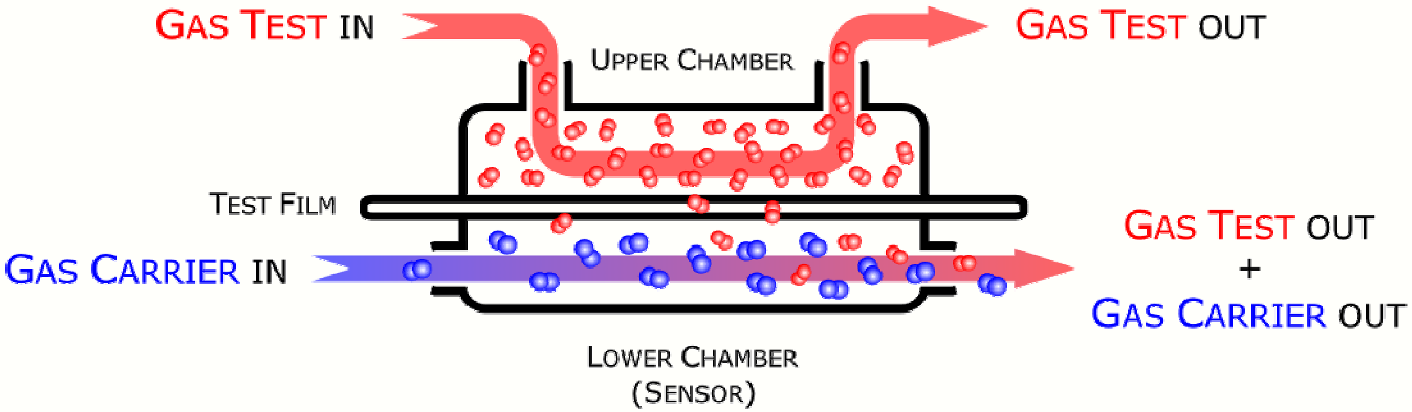
Prior Art (2)

The graph shows a decreasing curve until it stabilizes to a value of plateau.



The "zero value", the baseline of the signal, will take in account all possible leaks present in the gas line and all impurities present in the carrier gas.

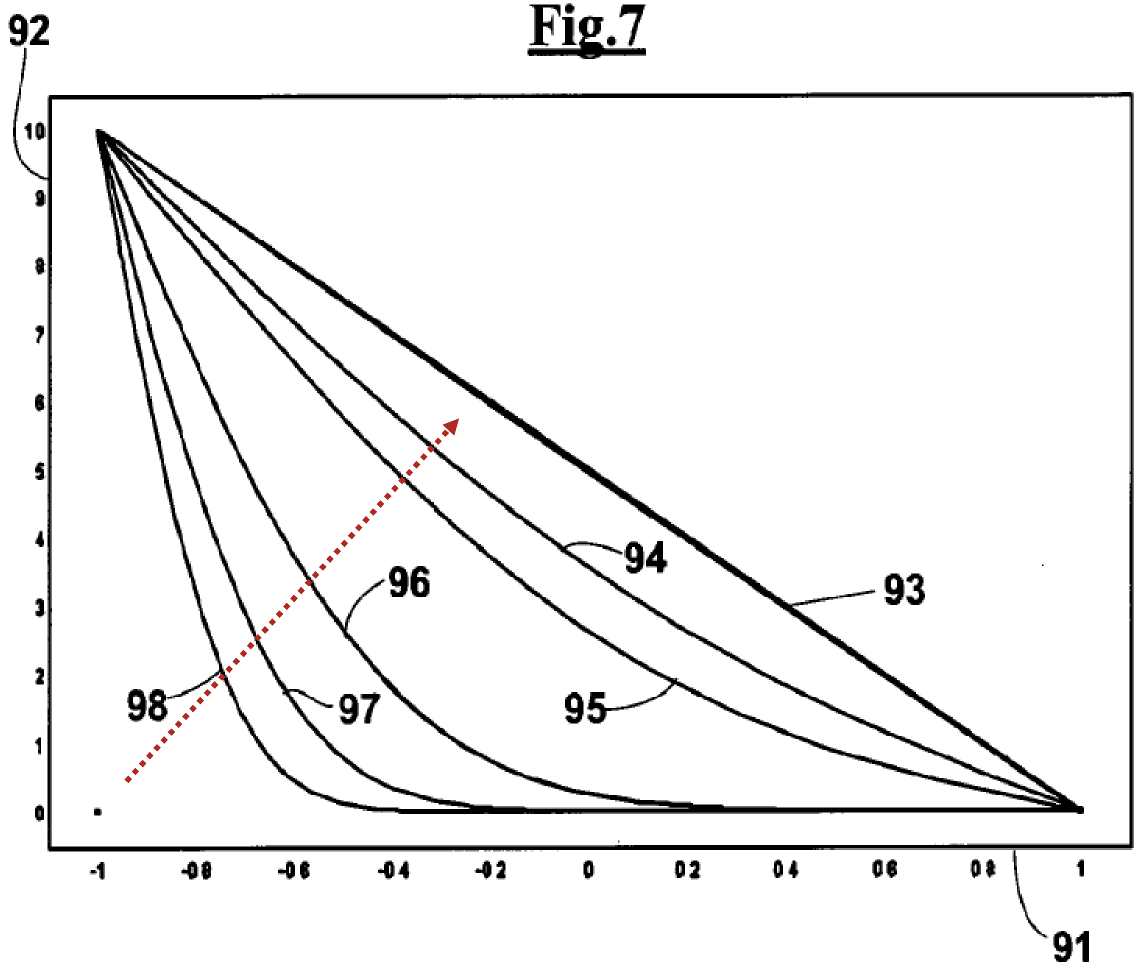
Prior Art (3)



At this point the pure test gas (O_2 or CO_2) starts flowing in the upper chamber. Test gas permeates through the sample and the carrier gas brings it along to the sensor.

Prior Art (4)

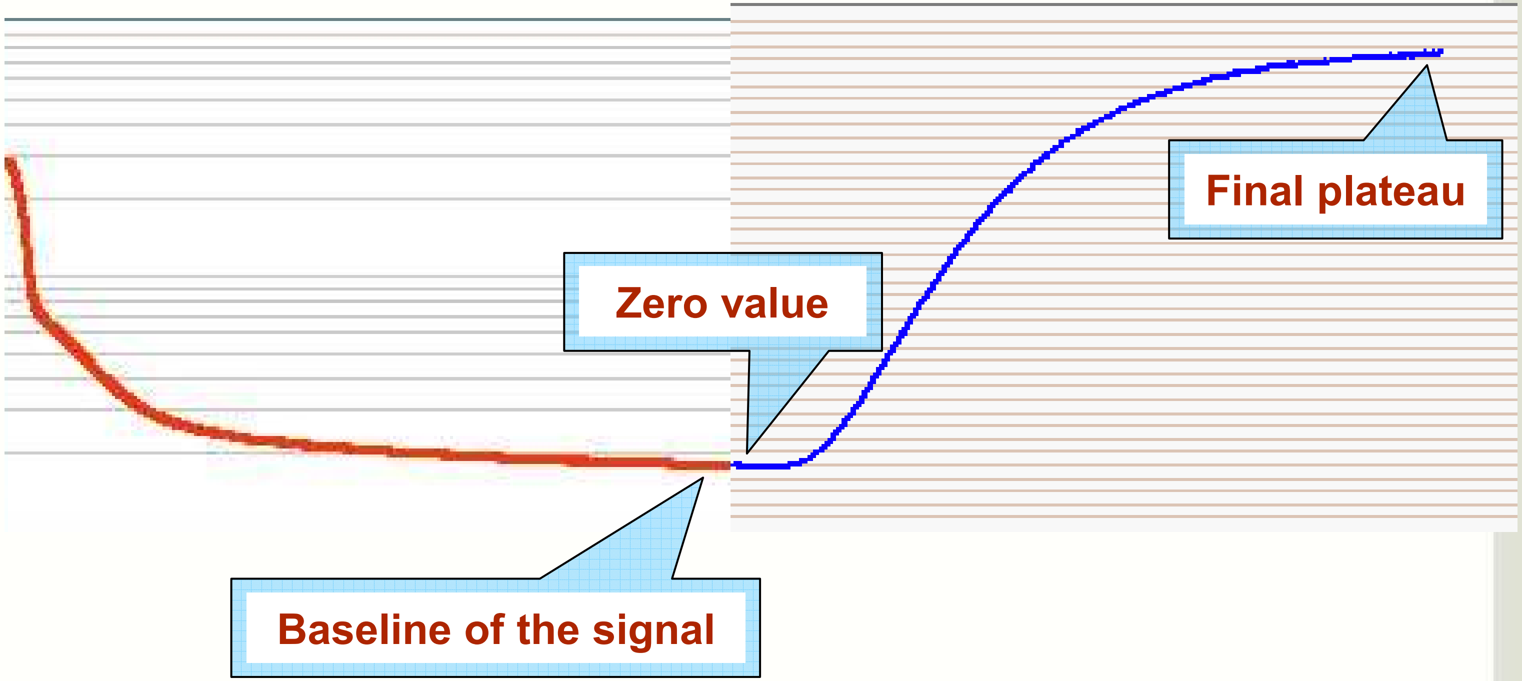
Fig.7



Evolution time of the concentration of the gas in the thickness of the film according to the traditional technique.

Prior Art (5)

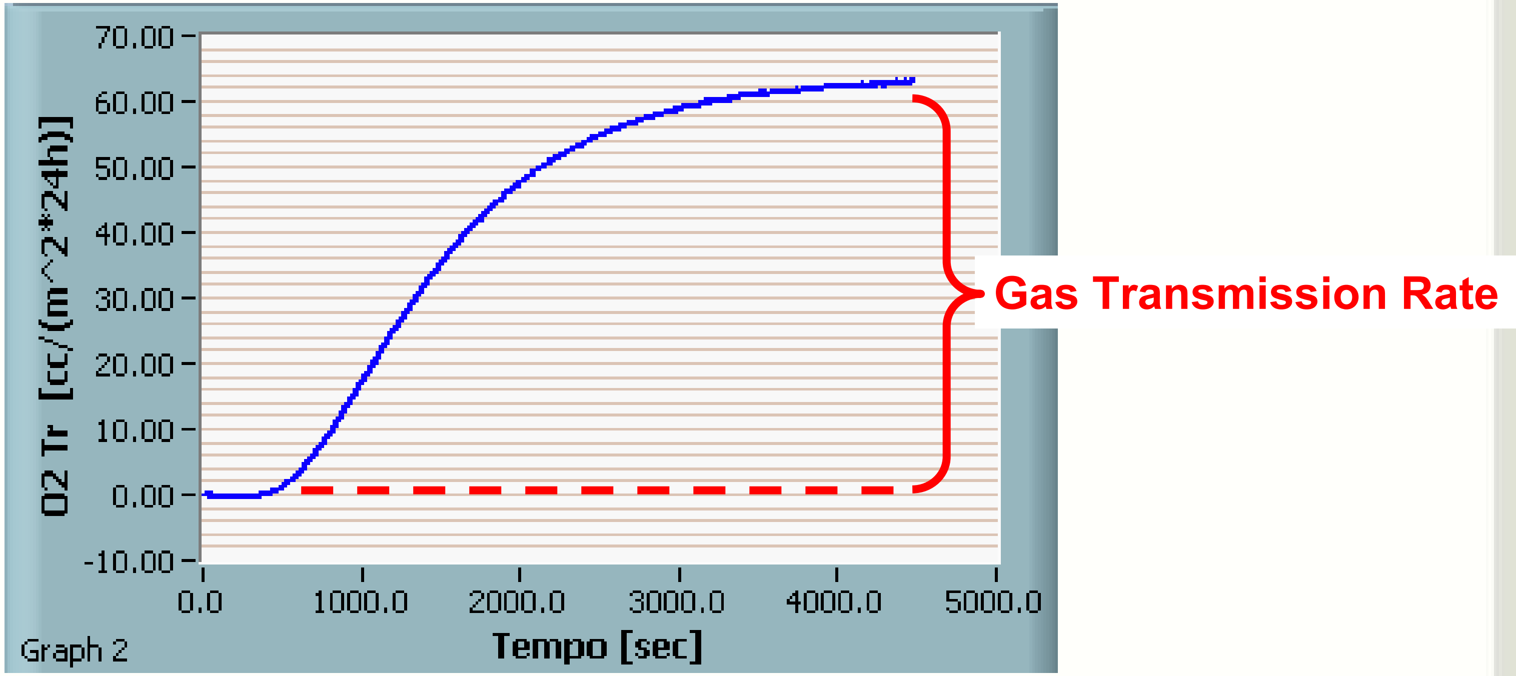
The second graph shows an increasing curve that then stabilizes to a final plateau.



The final plateau indicates that an equilibrium between the gas permeation through the film and the transportation of the permeated molecules to the sensor has been reached .

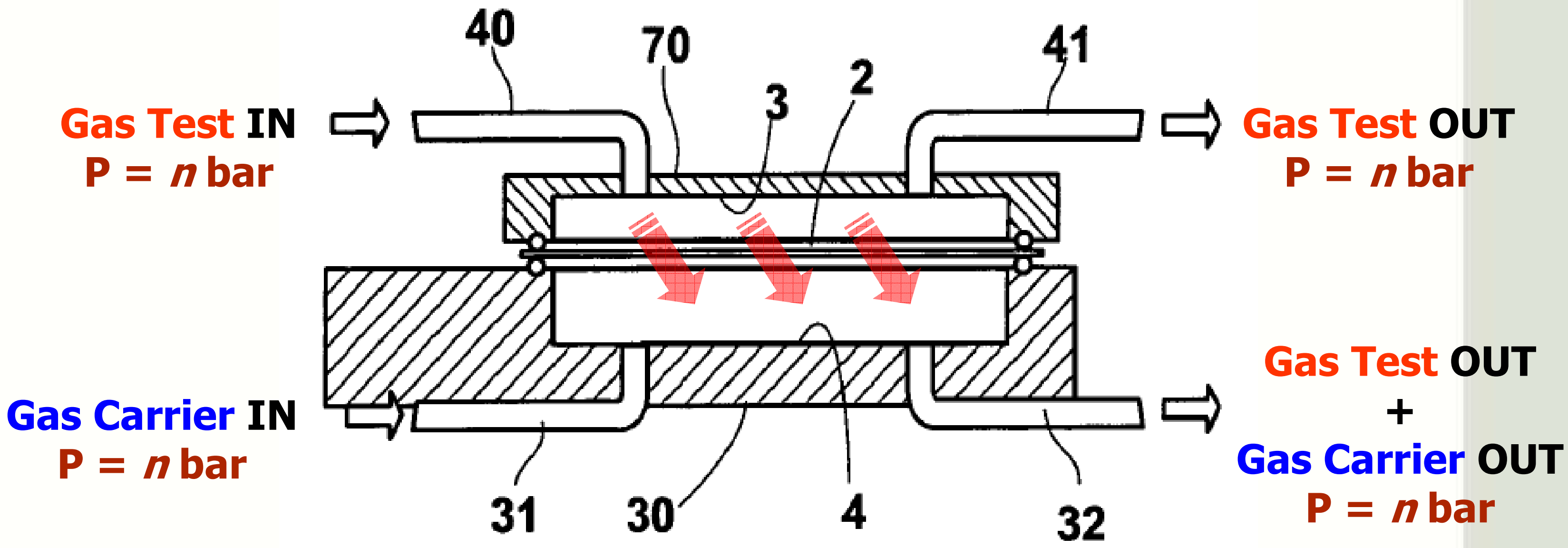
Prior Art (6)

The software automatically calculates the difference between the two values and transforms the result into standard units (e.g. $cm^3/(m^2 \cdot day \cdot bar)$).



Patent "Pressure" (1)

Fig. 3



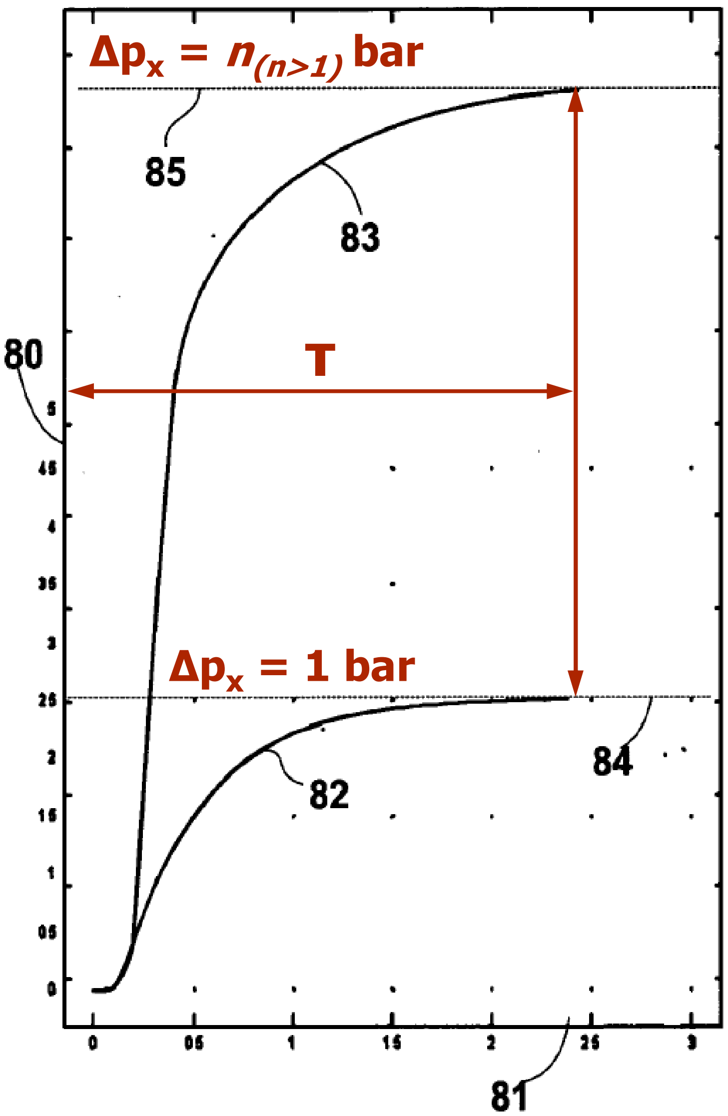
$$\Delta P = 0$$

$$\Delta p_x = n \text{ bar}$$

Cross sectional view of a portion of a measuring device according to the invention.

Patent "Pressure" (2)

Fig.4



Comparative diagram of the course of gas permeability versus time between the known devices (curve 82) and the device according to the invention (curve 83), where the measurement permeability is carried out at high pressure.

$$GTR_{83} = n\, GTR_{82}$$

$$T_{83} = T_{82}$$

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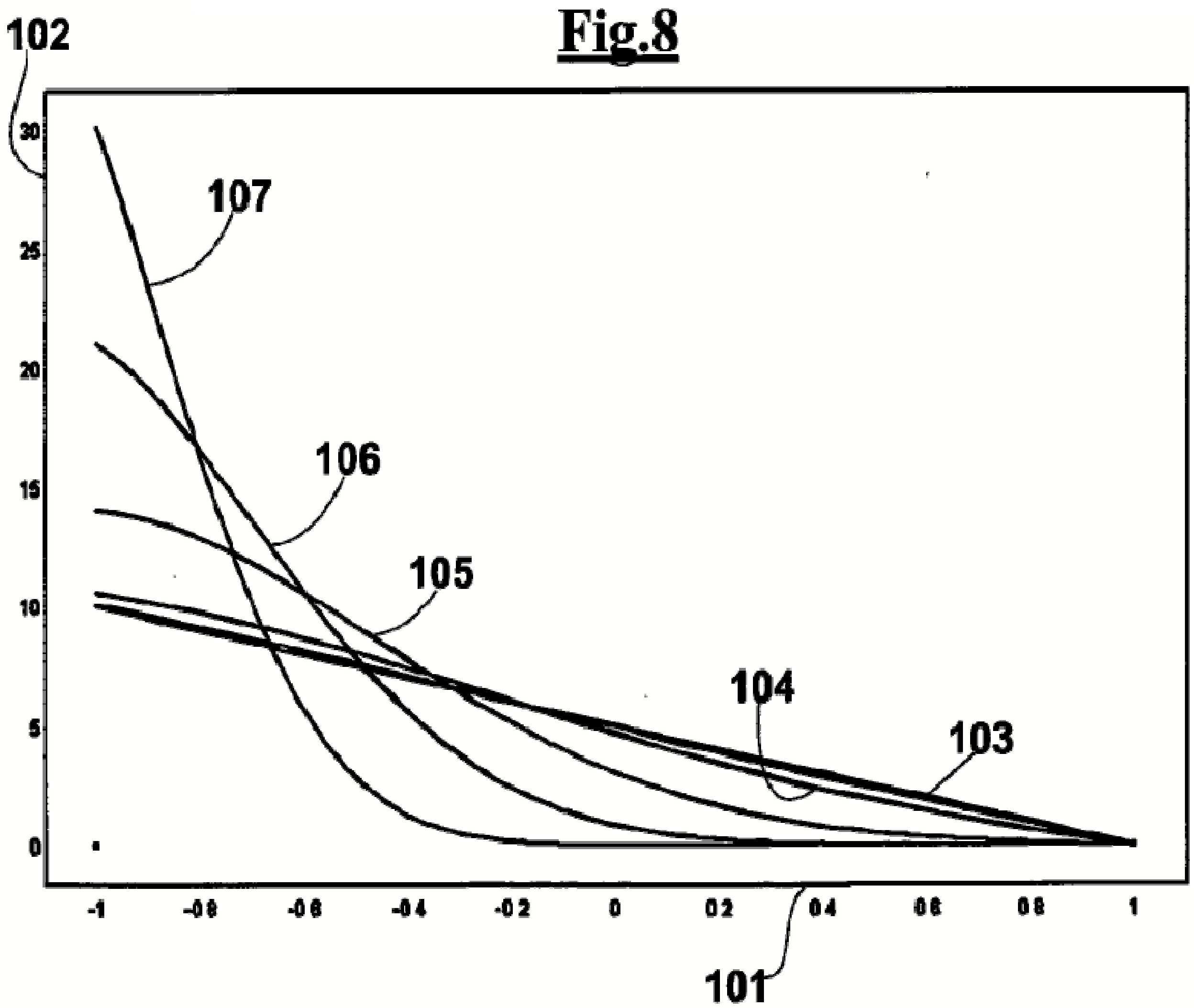
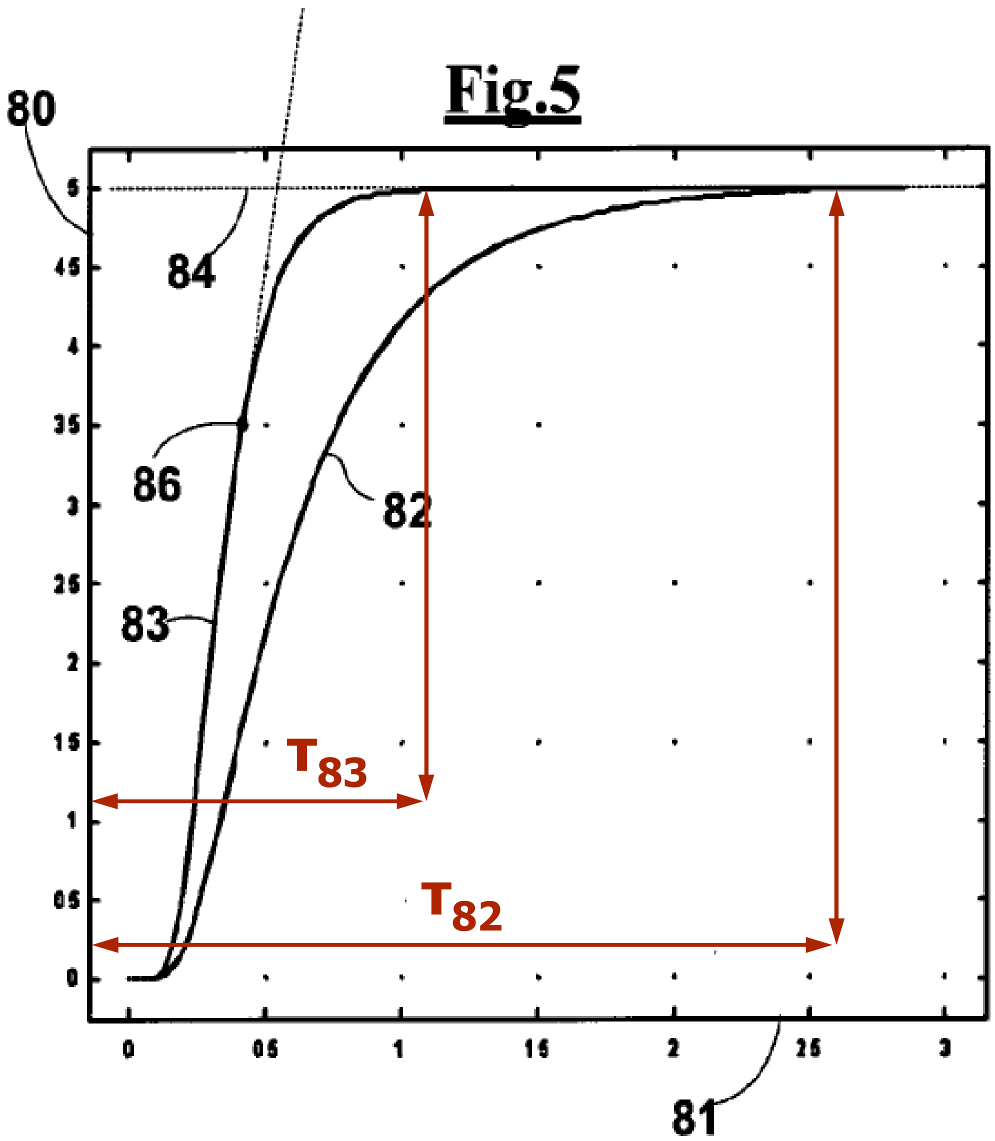
Patent "Pressure" (3)



Evolution time of the gas concentration in the thickness of the film according to the present invention.

Patent “Pressure” (4)

Fig.5



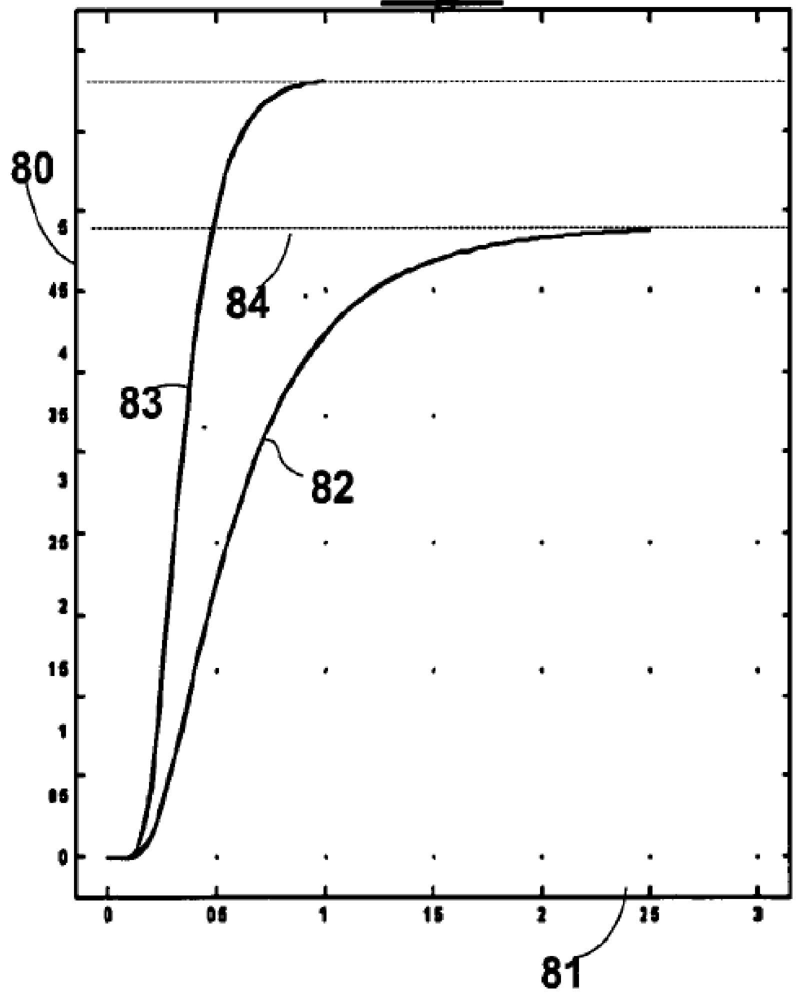
Comparative diagram of the course of permeability obtained with the known systems (curve 82) with respect to the course obtained with the present invention (curve 83), where after a first measuring step at a high pressure, the pressure is reduced to a value close to atmospheric pressure.

$$GTR_{83} = GTR_{82}$$

$$T_{83} < T_{82}$$

Patent "Pressure" (5)

Fig.6



Comparative diagram of the course of permeability obtained with the known systems (curve 82) with respect to the course obtained with the present invention (curve 83), wherein after a first measuring step at a high pressure, the pressure is reduced partially up to a value in any case larger than atmosperic pressure.

Main Advantages

Shortening of the measurement time.

Amplification of the measured signal.

Possibility to run quickly «threshold» tests, useful for Quality Control.

Thanks for your attention!

ExtraSolution

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