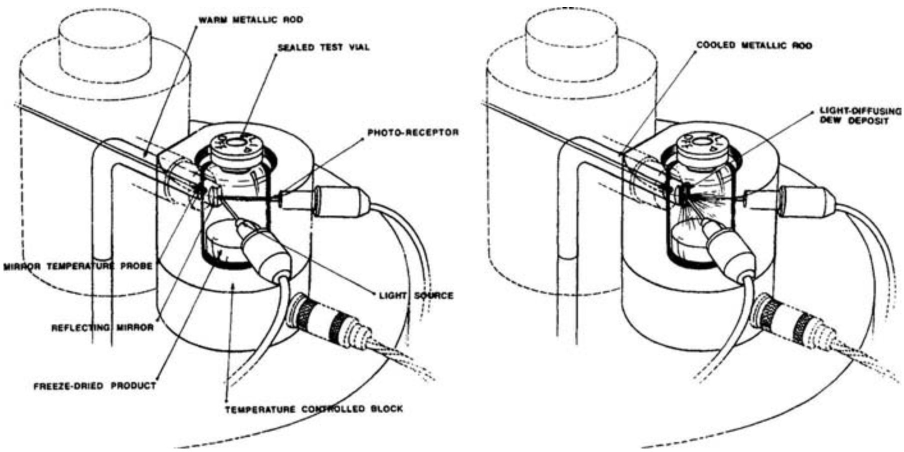

FIGURE 15 Operating principle of the device made to measure the equilibrium water vapor pressure inside a sealed vial.

very accurate standardized techniques to execute these determinations by chemical Karl Fischer titration or thermo gravimetric techniques. The only drawback is that, in both cases, the material is destroyed.

It is, in fact, because of that specific point that our approach has followed a different route.

Use a nonintrusive method leaving the product intact.
Be able, under those conditions, to do repetitive measurements on the *same vial* in the course of time to assess its evolution during storage.

Figure 15 shows the principle of the method we developed jointly with J. Mosnier (deceased). The selected vial or ampoule is placed in a thermostatic metallic block where temperature can be maintained constant from −10°C to +60°C. On one side of the block a metallic "finger," having at its end a tiny polished stainless steel cylindrical mirror (2 × 6 mm), is pushed in close contact with the outside wall of the glass vessel and the connection is made optically and thermally tight, thanks to a trace of silicon grease. On the other side is placed a near-infrared diode that beams light throughout the vial on the mirror, which, in turn, reflects that light on a phototransistor placed in a symmetrical position.

When the temperature of the mirror is equal to or higher than that of the metallic block (hence the temperature of the sample) the level of reflected light is steady. To start measurement the temperature of the mirror is progressively decreased, thanks to combined Peltier and cold nitrogen gas. Because of the good thermal contact on a very limited surface (12 mm^2) the internal wall of the flask is equally cooled on a small isolated spot. When, finally, the temperature of the mirror (and the temperature of the spot on the internal surface) reaches the value corresponding to the saturated water vapor pressure within the flask, some dew (or ice) is deposited on the internal wall that immediately becomes diffusive. The reflected light drops sharply. The measurement is done. The mirror can thus be warmed again, dew evaporates, light is restored, and the sample can be placed back into the storage cabinet for further determination. Figure 16 gives an