

## Resonant photoacoustic cell for low temperature measurements

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### Abstract

This paper describes a resonant photoacoustic cell for low temperature measurements. It has the shape of the Helmholtz resonator, with the microphone separated from the sample by a resonant tube. In this way the microphone is kept at room temperature while the sample temperature is lowered. When the sample is placed in a liquid nitrogen tank it is possible to change its temperature from 77 K to 300 K, with the aid of a furnace, which surrounds the sample holder. Two optical fibres lead the laser light to both sides of the sample, making possible the determination of the thermal diffusivity in all temperature intervals, by using the photoacoustic phase-lag method. © 1999 Elsevier Science Ltd. All rights reserved.

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### 1. Introduction

A photoacoustic cell is a compartment that enables the study of the photoacoustic effect. This effect consists basically of the conversion of light energy into sound energy. When a beam of periodically modulated light falls onto a sample, enclosed in a compartment filled with gas, it is absorbed and transformed into heat. This heat propagates through the sample, warming up the surrounding gas. The periodic heating of the gas produces pressure oscillations, which are detected by a microphone coupled to the photoacoustic cell.

The coupling of the microphone to the cell defines the photoacoustic cell type and its applications. If the microphone is connected to the cell by a resonance tube, the photoacoustic cell is called resonant and is commonly used in experiments with temperature variations.

A common application is in the study of phase transitions [1].

If the microphone is connected directly to the gas chamber, the cell is called non-resonant and is the most commonly used in photoacoustic spectroscopy experiments, with many types of applications. If the microphone is placed directly over the sample, the gas chamber is formed by the separation between the sample and the diaphragm. This type of cell, called open cell [2], is commonly used in the study of biological materials “in vivo” and produces the most intense photoacoustic signal due to the minimum gas volume of the cell.

The photoacoustic effect has several different applications in physics, chemistry, biology, engineering and medicine. Some examples in physics are: spectroscopy, de-excitation processes, thermal properties and material surface studies.

In the study of thermal properties, mainly for a wide range of temperatures, the photoacoustic cell must be resonant to keep the microphone away from the temperature variations. In this way, the photoacoustic cell described in the next section allows the study of the materials thermal properties from 77 K to 300 K, using the photoacoustic phase-lag method [3].

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### 2. Description of the photoacoustic cell

The photoacoustic cell described in this paper has the shape of a Helmholtz resonator which permits the sample temperature to be held at cryogenic temperature

while the microphone remains at room temperature. This type of photoacoustic cell, besides being used at various temperatures, can be operated at its resonance frequency to increase the photoacoustic signal in the case of poorly-absorbing materials.

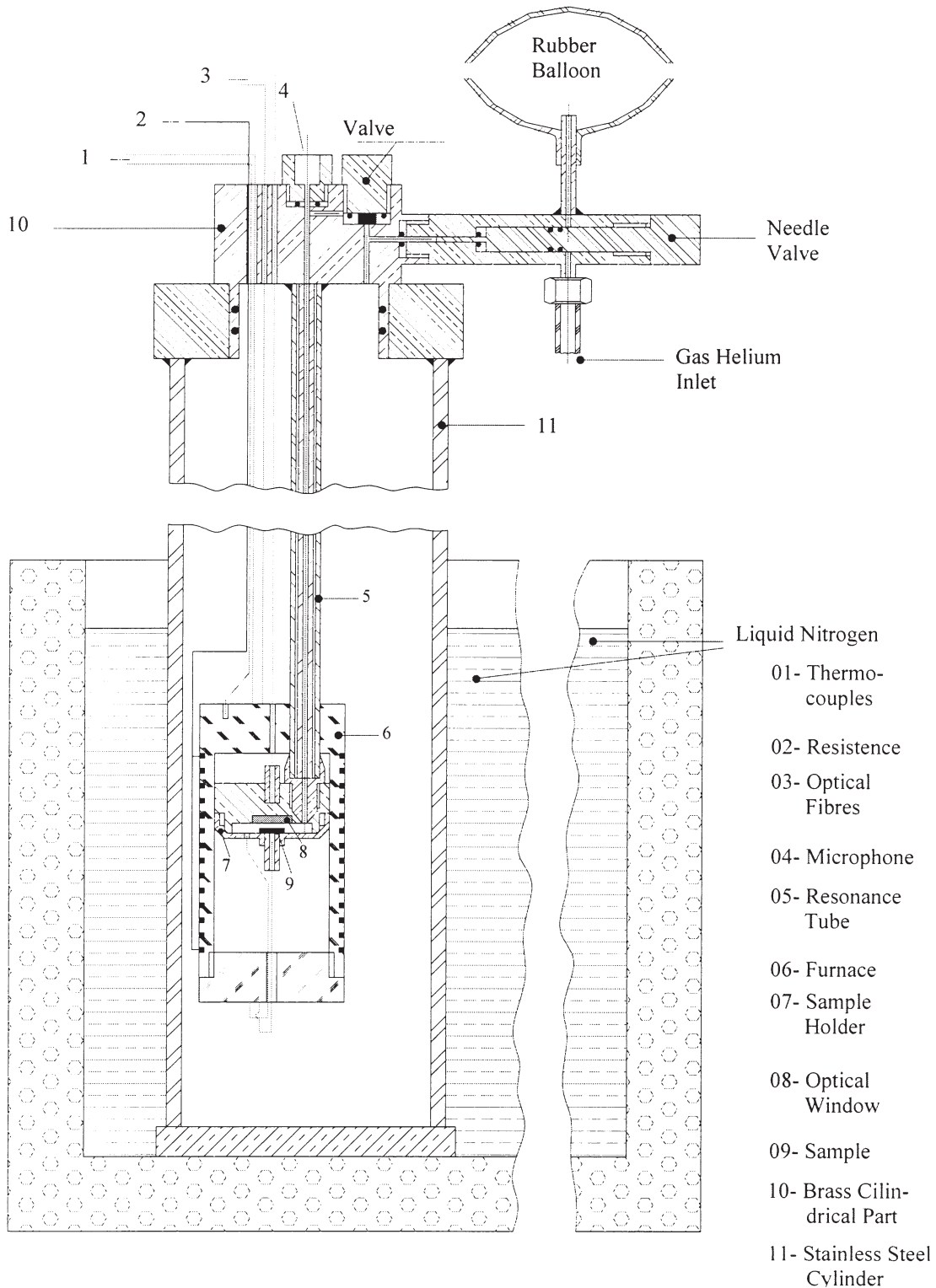


Fig. 1. Schematic cross-sectional view of the photoacoustic cell.

Fig. 1 presents a longitudinal cutting of the photoacoustic cell showing all its components.

The sample holder and the microphone compartment are connected by a stainless steel tube (No. 5) that is strong enough to sustain the sample holder. This tube has inside of it another tube of german-silver (or nickel-silver) 0.1 cm in diameter and 55 cm long, which is the Helmholtz resonance tube.

German-silver is a metallic alloy with 5–33% of nickel, 50–70% of copper and 13–35% of zinc. This material has good mechanical properties that make the construction of tubes with small diameters (of the order of mm) easy. Its thermal properties, similar to the stainless steel, make it possible to keep the two extremities of the tube at very different temperatures.

The tube dimensions determine the resonance frequency of the cell [4], as can be seen in Eq. (1).

$$w_0 = c_0(A/LV_r)^{1/2} \quad (1)$$

where:  $w_0$  is the resonance frequency,  $c_0$  is the sound velocity,  $A$  is the cross section of the tube,  $L$  is the length of the tube,  $V_r = V_1V_2/(V_1 + V_2)$ , where  $V_1$  is the volume of the microphone compartment and  $V_2$  is the volume of the sample holder.

The resonance frequency of the photoacoustic cell was obtained experimentally and theoretically from Eq. (1). For the cell filled with air at room temperature, the value obtained was 296 Hz, and for the cell filled with helium at room temperature, the value obtained was 860 Hz.

Long and/or narrow tubes reduce the resonance frequency to values inside the operation range of most modulators and avoid those signals produced by scattering light reaching the microphone.

The sample holder (No. 7) of the photoacoustic cell is formed by two parts which are sealed by one ring. This ring makes it possible to close the sample holder without any twisting, thus avoiding any damage to the elements fixed on it.

The upper part of the sample holder, which is fixed to the resonance tube, has one quartz window (No. 8) stopping up one hole destined for the optical fibre. In the lower part of the sample holder there is a second hole for the other optical fibre, which is stopped up by the sample (No. 9) with vacuum grease. Since the physical properties of the grease change for temperatures below 200 K, the best conditions of sealing depend on how the sample accommodates the grease.

The sample holder is enclosed by a furnace (No. 6) made of soap-stone which is used to change the sample temperature from 77 K to 300 K. In this furnace there is one hole to fix a thermocouple for the reference temperature.

In the other extremity of the resonant tube there is one

cylindrical part made of brass (No. 10), which has two o-rings that make possible the sealing of the stainless steel cylinder (No. 11). This part contains the microphone compartment (No. 4); holes for the optical fibres (No. 3); thermocouples (No. 1) and nickel-chrome resistance of the furnace (No. 2); one valve that seals the volume formed by sample holder, resonance tube and microphone; and one connection composed of one needle valve, and one rubber balloon to control the pressure and one helium gas inlet, all specified in Fig. 1.

Helium gas was employed for the low temperature experiments to avoid condensation of the water present in the air and to improve the thermal coupling between the sample, the furnace and the nitrogen liquid. The helium gas thermal conductivity is  $34.3 \times 10^{-5} \text{ cal cm}^{-1} \text{ s}^{-1} \text{ K}$  (at 273 K and 760 Torr), which is six times greater than the thermal conductivity of air,  $5.76 \times 10^{-5} \text{ cal cm}^{-1} \text{ s}^{-1} \text{ K}$  (at 273 K and 760 Torr).

The helium gas inlet is connected to the vacuum pump, which removes the air present inside the photoacoustic cell, and to the helium cylinder. The rubber balloon is an extra volume to keep the cell pressure constant as the temperature changes. Using the needle valve it is possible to isolate and/or to connect the photoacoustic cell to the rubber balloon and to the helium gas inlet, as needed.

The valve that separates the sample holder, resonance tube and the microphone from the rest of the cell is opened during the cell cleaning, the helium gas filling and variation of the temperature to avoid microphone damage. After the temperature is stabilized, the valve is closed in order to have a minimum volume and consequently a maximum photoacoustic signal.

### 3. Conclusion

The photoacoustic cell described in this paper was designed to conduct low temperature photoacoustic experiments. When the cell is placed in the liquid nitrogen bath it is possible to change the temperature from 77 K to 300 K, but using a liquid helium bath this range could be increased considerably. In this way, the physical properties of several materials could be determined, besides the possibility of observation of phase transitions.

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