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To cite this version:


HAL Id: jpa-00223253
https://hal.archives-ouvertes.fr/jpa-00223253
Submitted on 1 Jan 1983

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LASER PHOTOACOUSTIC DETERMINATION OF TRACE SUBSTANCES


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Abstract – An experimental apparatus for the photoacoustic determination of trace Co in the form of PAN-Co(III) is described. A good working curve is obtained for quantitative detecting for trace Co.

The photoacoustic spectroscopy using laser as light source has very high sensitivity. Its principle and theory were reviewed by several authors (1-3). The method is being applied increasingly widely to the analysis of trace substances (4-20). In this paper, the quantitative determination of trace substances by solid-state photoacoustic spectroscopy is studied, using a He-Ne laser and a He-Cd laser as light sources and a solid complex PAN-Co(III) as a research object.

The block diagram of the experimental apparatus used in this work is shown in Fig. 1.

Fig. 1 - Block diagram of apparatus for laser photoacoustic determination.

1. He-Cd laser ; 2. He-Ne laser ;
3. pinhole ;
4. total reflective mirror for He-Ne laser ;
5. beam splitter ; 6. lens ;
7. chopper ;
8. power source for the chopper ;
9. photoacoustic cell and microphone ;
10. preamplifier for the electric signal from microphone ;
11. lock-in amplifier ;
12. X-Y recorder with double pen ;
13. laser powermeter
14. power source for He-Ne laser
15. power source for He-Cd laser.

It is arranged in such a way that it is capable of making both the single-beam and double-beam determination. The photoacoustic cell used are made of pyrex glass. The schematic diagram of single-microphone cell structure is shown in Fig. 2.
In the single microphone photoacoustic cell a sensitive microphone that is used to detect the photoacoustic signal (PA) is fixed on the sample side of cell. Double microphones are also used, but they are fixed on the opposite sides vertical to the incident laser beam. In order to reduce the interference of ambient noise the photoacoustic cells is buried in sand. The background noise of the cells is 15-60 nV, depending on the strength of ambient noise.

![Diagram of single-microphone cell structure](image)

**Fig. 2** - Schematic diagram of single-microphone cell structure.

The pA signal produced by the exciting light can be detected by the microphone and translated into an electric signal which is fed into the lock-in amplifier through the preamplifier. The pA signal is picked up from noise, and then fed into the y-axis of the X-Y recorder. The strength of pA signal \( S_{pA} \) stands for the amount of the trace substance detected.

The following aspects are studied by the above apparatus.

1. The effect of modulated frequency \( f \) and laser power on \( S_{pA} \). The results obtained show that there is a linear relationship between \( S_{pA} \) and \( f^{-1} \) when \( f > 60 \) Hz. If \( f \) is less than 60 Hz, the relationship isn't linear. This is due to the range of frequency response of the microphones used (60 Hz to 10 KHz). The results are shown in Fig.3.

The \( S_{pA} \) measured is directly proportional to the power of incident laser, as shown in Fig.4.

![Graph of \( S_{pA} \) vs. \( f^{-1} \)](image)

**Fig. 3** - \( S_{pA} \propto f^{-1} \) relationship.

![Graph of \( S_{pA} \) vs. power](image)

**Fig. 4** - \( S_{pA} \propto p \) relationship.
The increase of laser power can therefore raise the sensitivity of photoacoustic detection.

2. The effect of sample backing and carrier on $S_{PA}$. In our work the two kinds of substrate are used: one is a glass rod with a polished end covered by a layer of carrier, and another is a piece of plain glass adhered to a glass tube, which is also covered by a layer of carrier. In each case, the thickness of carrier is 0.1-0.2 mm. The $S_{PA}$ produced by a same substrate are 150-240 $\mu$V (for 8 mW He-Ne laser) and 200-300 $\mu$V (for He-Cd laser), corresponding to that produced by 2-3 ng Co in the form of PAN-Co(III). In the determination of trace Co, the background signal is removed by zero adjustment of the lock-in amplifier. The carrier used are silica gel (SiO$_2$), BaSO$_4$ and Al$_2$O$_3$, which are carefully treated before use and are of homogenized grain size of about 5 $\mu$m. In the cases of PAN-Co(III)-SiO$_2$ and PAN-Co(III)-BaSO$_4$ systems larger $S_{PA}$ can be produced (about 100 $\mu$V/ng Co). The $S_{PA}$ produced by PAN-Co(III)-Al$_2$O$_3$ systems is smaller, and the layer made of Al$_2$O$_3$ is also rougher. Therefore, it isn't used in further work.

3. The quantitative determination of trace Co. In this case the requirements of heat-thinness and good light transparency for the sample to be determined can be met, and $S_{PA}$ can be expressed as a approximate formula:

$$S_{PA} = K \cdot B_1 = K' \cdot C$$

where $B_1$ is the light absorption of sample, and $C$ is the content of substance to be determined. A good linear relationship between the $S_{PA}$ and contents of Co is found, as shown in Fig.5.

![Graph](Fig_5.png)

Fig. 5 - working curve for Co.8 mW He-Ne laser, f=32 Hz, silica gel carrier, single microphone cell.

4. The comparison of the detection sensitivity of double-microphone cell with that of single-microphone cell. The results show that the double-microphone cell has an additive effect, that is, the detected $S_{PA}$ is the sum of that from the two microphones, and it raises not only the detection sensitivity, but also $S/N$.

5. The comparison of the $S_{PA}$ produced by double-beam light with that by single-beam light. The absorption curve of PAN-Co(III)-CHCl$_3$ solution is shown in Fig.6. It has maximal absorbance at 452 nm and 585 nm. In our work, a He-Ne laser (633 nm, near 585 nm) and a He-Cd laser (442 nm, near 452 nm) are used to excite coaxally the sample to be detected. The results measured are shown in Fig.7.

It is seen from Fig.7 that the $S_{PA}$ produced by double-beam light is equal to the sum of that produced by each light beam, corresponding to the effect of increasing laser power.

In our experimental condition, it is possible to detect Sub-ng Co in the form of PAN-Co(III). This shows that the laser photoacoustic spectroscopy is a very effective tool for the determination of trace substances.
**Fig. 6** - Absorption curve of PAN Co(III)-CHCl₃ solution.

**Fig. 7** - SpA of double-beam light.

Curve 1: He-Ne laser (~5.3 mW)
Curve 2: He-Cd laser (~5 mW)
Curve 3: He-Ne laser + He-Cd laser
f = 40 Hz, silica gel carrier, single microphone cell.

Reference