

## A Simple System for Low-Temperature Spectral Measurements in the UV-Vis Range

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Index Headings: Spectroscopic techniques; UV-visible spectroscopy.

### INTRODUCTION

Because of its importance in the study of molecular structure and kinetics, the measurement of electronic absorption spectra in the UV-Vis region at variable temperatures, particularly at low temperatures, has always been of great interest to the spectroscopist. Many excellent cryostats are available in the market, but they are so expensive that ordinary laboratories cannot afford to buy them. If damaged, these instruments are often difficult to repair without sophisticated shop facilities.

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A simple system has been proposed<sup>1</sup> for this purpose, but in our work we have been able to construct a still simpler assembly.

### DISCUSSION

In the proposed system, both the reference and the sample cells are simultaneously cooled at a similar rate by the passage of dry nitrogen gas through a coil of copper tubing immersed in liquid nitrogen. The whole assembly is made up of perspex sheet. As shown in Fig. 1, it mainly consists of three parts (a1, b, and a2). Parts a1 and a2 are 162 × 62 × 5 mm sheets, both with two 25 × 6 mm slits 10 cm apart (this is the separation between the two beams provided in a standard spectrophotometer), and b is a 162 × 62 × 12.5 mm sheet cut at two places in the way shown in Fig. 1. There is a groove joining the two cuts. Sheet a1 is permanently fixed by using locally available adhesive, Alphy (based on  $\alpha$ -cyanoacrylate), whereas sheet a2 is mounted by screws so that it can be removed when thorough cleaning is required. After a1, b, and a2 are joined, two cell chambers are created in which a standard 1-cm quartz cell can be tightly inserted and can rest on "stop hedges" projecting from inside panel a2 below the slits. Both of the cell chambers are painted black inside, to avoid any stray light.

The cooled gas enters the chambers (from the side nozzles N1 and N2 connected to a T-junction by Tygon tubing), cools the cells, and leaves the compartment

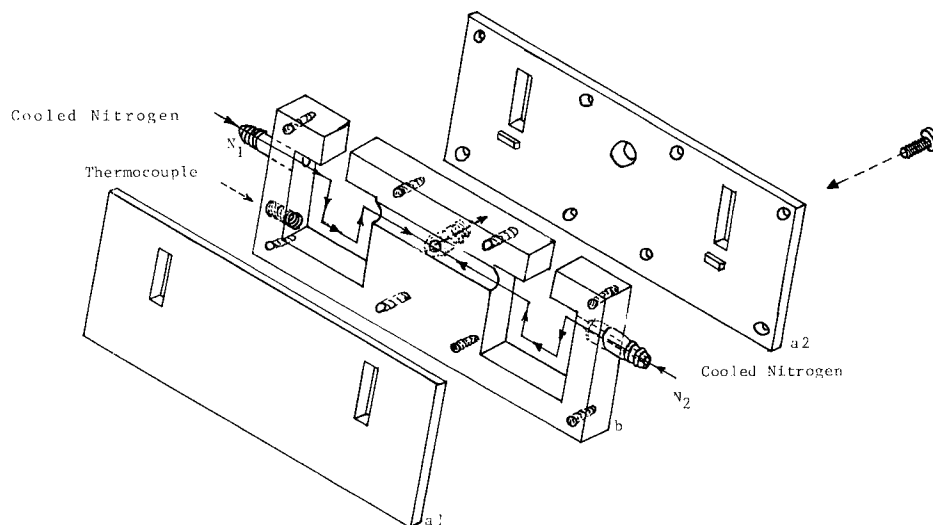


FIG. 1. Cut-away view of assembly.

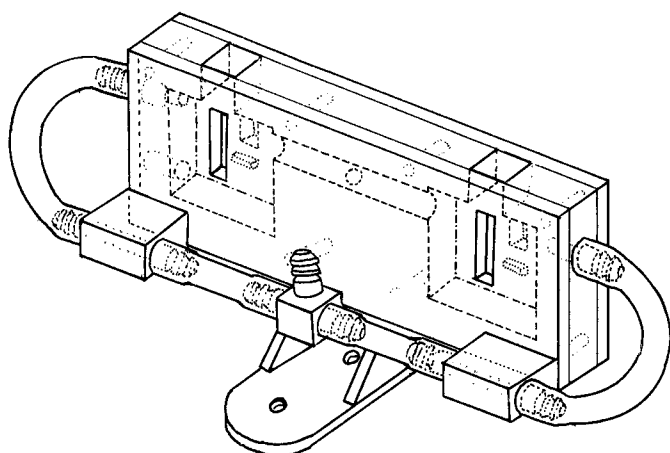


FIG. 2. System in assembled form.

through an outlet. The inlet and outlet for the gas are provided on a separate perspex sheet which fits in the space created by leaving the shutter of the compartment open accordingly. Possible fogging is avoided by a procedure of flushing the compartment with dry nitrogen before cooling it and by the use of anhydrous silica gel in the compartment. The whole assembly can be placed in the compartment and mounted by the screws already provided by the manufacturer for the standard cell-holder. By controlling the gas flow, one can maintain temperatures down to  $-170^{\circ}\text{C}$  (with an accuracy of  $\pm 2$ ). An iron-constantan thermocouple connected to a 3.5-digit millivoltmeter with ice water as the reference junction,<sup>2</sup> or any suitable commercial device, can be used for temperature measurements. The system in assembled form is shown in Fig. 2.

1. A. W., Burgstahler and D. D., McBain, *J. Chem. Educ.* **61**, 469 (1984).
2. *CRC Handbook of Chemistry and Physics* (CRC Press, Boca Raton, Florida, 1981), 62nd ed. p. E-107.

## Sample Handling of Gold at Low Concentration in Limited-Volume Solutions Preconcentrated from Seawater for Inductively Coupled Plasma Mass Spectrometry

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### INTRODUCTION

Since the early 1900s, investigators have attempted to measure the gold content of seawater. After devoting ten years to quantifying its concentration, Haber concluded that insufficient gold existed in seawater ( $20 \text{ pg g}^{-1}$ – $20 \text{ ng g}^{-1}$ ) to merit its economic extraction.<sup>1-2</sup> Subsequently, gold has been determined in seawater, often with values ranging from those reported by Haber to those orders of magnitude smaller ( $0.02$ – $0.2 \text{ pg g}^{-1}$ ).<sup>3-7</sup> A comprehensive study of its distribution and behavior in the oceans has yet to be completed, because the measurement techniques are tedious. The determination of extremely low gold levels encountered in marine waters is difficult and generally requires chemical preconcentration. A method for determination of gold in seawater after preconcentration with activated charcoal was reported recently by Hall *et al.*<sup>8</sup> A relatively convenient technique for the preconcentration of gold from seawater has been recently revised,<sup>4</sup> and in this note we report precautions needed for inductively coupled plasma mass spectrometry (ICP-MS) to determine gold in seawater after this preconcentration step.

### EXPERIMENTAL

The preconcentration scheme is presented on Fig. 1. Aqua regia was made with  $\text{HNO}_3$  and trice distilled in a Vycor still. Samples were spiked with  $^{195}\text{Au}$  tracer (New England Nuclear Corp., Boston, MA). KCN was purified by being passed through an anion-exchange resin prior to complexing of the gold. Ammonia was redistilled, and distilled deionized water ( $18 \text{ M}\Omega$ ) was used throughout. The gold complex was collected on an anion-exchange resin (AG 1-X2, 50-100 mesh) and subsequently eluted with  $\text{HCl}$  at  $90^{\circ}\text{C}$ . Silica was removed as  $\text{SiF}_4$ . If residual

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