THE DESIGN AND CONSTRUCTION OF A HOHLRAUM SPECTRAL ANALYZER

PROGRESS REPORT
on NGR 19-001-024 for
September, 1970 to September, 1971

by
Dupree Maples
C. A. Whitehurst

Prepared Under Grant NGR 19-001-024
The Division of Engineering Research
Louisiana State University
Baton Rouge, Louisiana

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NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

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I. Introduction

Public concern over environmental pollution is increasing at a geometric rate. The concern stems from an awareness of the threats to health and welfare from the wastes of our society; it leads inevitably to pressure on industry to reduce the discharge of contamination into the air and public waterways.

The implementation of an effective environmental improvement program depends on the coordinated efforts of the general public, government, and private industry. A problem confronting all agencies for environmental control is the lack of instrumentation to monitor pollutants. This research is an attempt to solve this instrumentation problem by developing a Spectral Analyzer for identifying and measuring pollutants.

The various interactions between air, water, and solid wastes must be evaluated by a plant or even an entire industry. The treatment of a water pollutant may create an air pollutant, for instance, an objectionable odor. And, of course, the collection and handling of the dust particles from a bag house will produce a solid waste disposal situation. This particular problem presents a dual instrumentation need, that is, the capability to monitor air and water contamination on a continuous basis.

Monitoring programs are established to ensure that certain regulatory requirements established by federal, state or local governing agencies are maintained and to evaluate potential hazardous conditions which may harm or endanger life or property. Monitoring may be done on a continual or part-time basis, depending upon the type of information desired.

Although some progress has been made in identifying and measuring general classes of pollutants, there is a real need for intensive research into methods for identifying and measuring these classes of pollutants. A need for intensive research into methods for identifying and measuring the individual substances that make up these classes is also sorely needed, as pointed out in the "Industrial Pollution Control Handbook." Simpler and less expensive procedures should be a prime requisite for this purpose. The Hohlraum Spectral Analyzer will facilitate research in areas where our knowledge is far from complete regarding air pollutants: their identity, specific sources from which they are derived, factors governing their dispersion, and chemical and physical changes in the atmosphere; their effects singly and in combinations.

Long-term effects of air pollution on man, on other biological systems, and on property must be quantitated accurately in order to establish the necessity, feasibility, and economic practicability of control measures designed to abate these effects. Research is still sorely handicapped by the lack of techniques sensitive enough to detect minimal changes.

Another problem area in which the Hohlraum Spectral Analyzer is applicable is the evaluation of the role fuel additives play in the composition of motor vehicle gasoline. The function of these additives are numerous. The most notable of these are the antiknock compounds. However, there are numerous others including detergents, deposit modifiers, corrosion inhibitors, upper cylinder lubricants, antioxidants, etc. As the use of the antiknock compounds diminishes, the effect of those additives and impurities on exhaust emissions may become more evident and significant. Some of these additives and impurities have the potential of producing air pollutants emitted in the exhaust, and although they are today present in the fuels at relatively low concentrations there is no way of knowing the level of their future usages. A program is needed to develop techniques and instrumentation for the determination of the effects that additives and impurities in gasoline have on exhaust emissions.

In this report the functional description of each component is discussed, followed by an outline of the experimental procedure. Also included is a statement of the proposed tasks for 1971-72.

II. Description of Instrument

The Hohlraum Spectral Analyzer, shown in Figure 1, consists of two sources and a double pass monochromator which supply radiation to the sample compartment. The sample compartment is equipped with three detectors to monitor the incoming radiation. The electrical signal from the detectors is first amplified and then recorded on a strip chart recorder. A McLeod gage is used to determine the pressure and quantities of pollutants. A vacuum pump is used to evacuate the sample compartment at the start of each test.

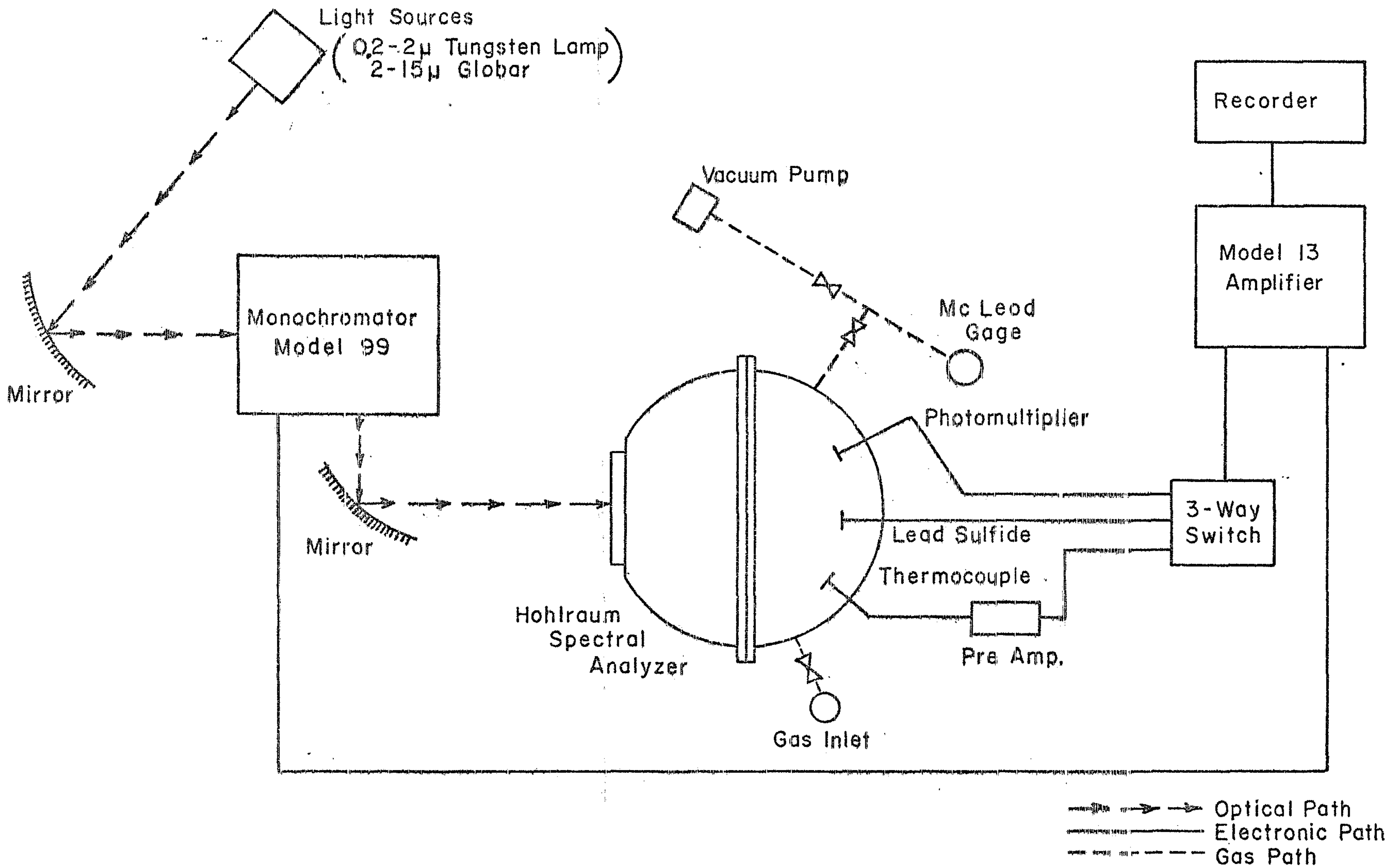


FIGURE I.
BLOCK DIAGRAM OF INSTRUMENTATION

Light Sources

Two light sources were used to provide radiant energy in the spectral range of 0.2 to 15μ. A tungsten lamp was used in the range of 0.2 to 2.0μ and a Globar was used for the region of 2.0 to 15.0μ.

The Sylvania tungsten lamp was placed on a stand with an aluminum reflector used to direct the radiant energy onto a spherical mirror. Power was supplied to the lamp by a powerstat (variable autotransformer).

A Perkin-Elmer Globar was operated at a temperature of 1100°C. The electrodes were water cooled to increase their useful life. Power was supplied at 40 volts and 5 amps to maintain the proper operating temperature.

Mirrors

In order to constrate the radiant energy from the light sources onto the entrance of the monochromator, a 108 mm diameter mirror with a radius of curvature of 0.56 meters was used. All mirrors were coated with Al-SiO for reflective purposes. A similar mirror received the monochromatic energy from the monochromator and reflected the radiation into the sample compartment.

Detectors

Three detectors give a useful energy range to the instrument from 0.2 to 20.0μ. Each detector (PM, PbS, and TC) is mounted through a separate outlet on the sample compartment. For simplicity the electronics associated with each detector were constructed separately.

Photomultiplier Detector

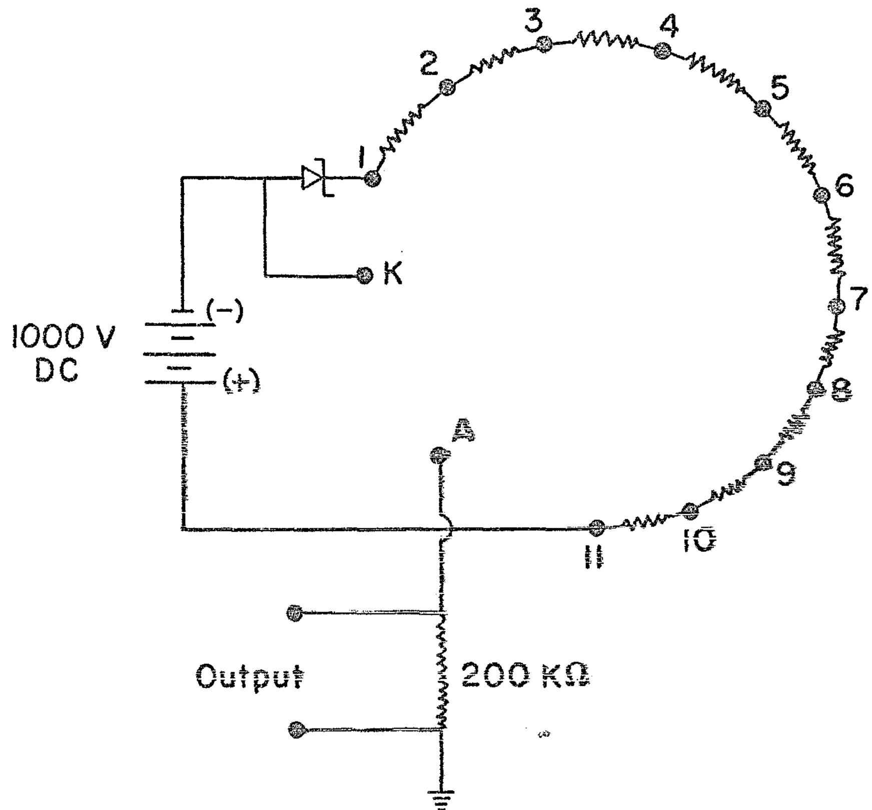
A photomultiplier tube (EMI type 9601-B) detects energy in the range 0.2 to 0.7μ. Its output is fed by a Perkin Elmer Model 13 Amplifier to a Leeds and Northrup type G recorder. Power is supplied at a voltage of 960 volts from a Perkin Elmer d.c. power supply. This detector has an anode sensitivity of 2000 A/L with a spectral response of S-11(c). The electrical circuit for the photomultiplier tube is shown in Figure 2.

Lead Sulphide Detector

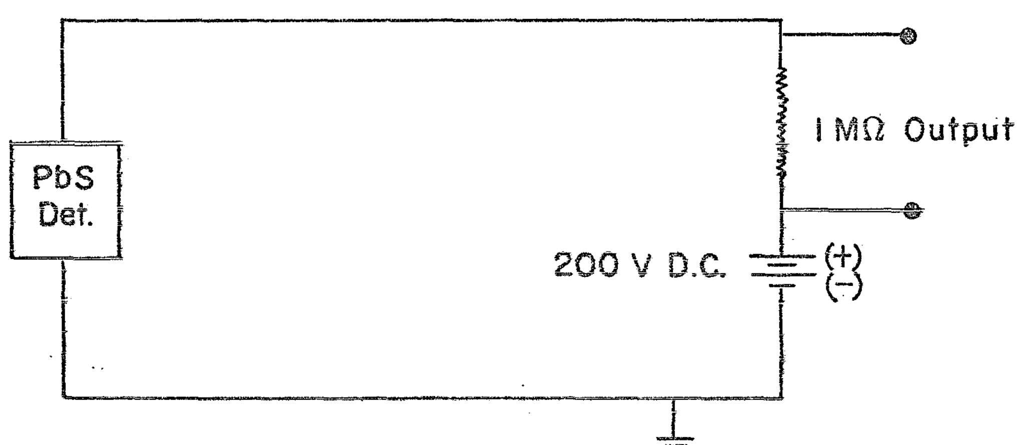
Placed inside the monochromator, a mechanical chopper interrupts the light beam at a frequency of 13 cycles per second. A photoconductive cell coupled to a tuned amplifier (Perkin Elmer Model 13) detects this radiation in the range 0.7 to 3.0μ. The cell (Optoelectronics Model KN2-12) has a sensitive area 10 mm x 10 mm and a dark resistance of 220 ohms. Power is applied to the cell at 200 volts d.c. and is supplied by a Fluke Model 301 c power supply. The electrical circuit for the lead suffide detector is shown in Figure 2.

Thermocouple Detector

For detection of infrared energy a thermocouple (Charles M. Reeder Model BL500) was employed in the range 3.0 to 15.0μ. The thermocouple has a sensitive area 8 mm x 8 mm with a K Br window. This type of window could not be operated where the relative humidity was above 40%. The output of the thermocouple was first passed to a pre-amplifier (Perkin Elmer Model 088-0054) which has an overall gain 6000 at 13 cycles per second, and then to the Model 13 amplifier.



PHOTOMULTIPLIER ELECTRICAL CIRCUIT



LEAD SULFIDE ELECTRICAL CIRCUIT

FIGURE 2. ELECTRICAL CIRCUITS FOR DETECTORS

Sample Compartment

A 10-inch inside diameter sphere was machined from two aluminum plates which were 13 inches in diameter and 6 inches thick. The sample compartment was constructed as two hemispheres which fit together so that no crack would result. All seals were "O" rings which were designed to seal at a high vacuum. The three detector ports were equipped with Ultra-torr fittings to seal against a vacuum. A K Br window allowed radiant energy to enter the compartment. The window material was 1 inch in diameter and 10 mm thick. The compartment also was equipped with a vacuum outlet and a gas filled port. A detail sketch is shown in Figure 3 and a photograph is shown in Figure 4 of the sample compartment.

Monochromator

The Perkin-Elmer Model 99 Double Pass Monochromator -- i.e., one which makes use of the same optical system twice -- is used to provide monochromatic radiation with high spectral purity. All the optics except the prism are reflecting and, since a number of prism materials are available, the instrument is usable throughout the visible, ultraviolet and infrared portions of the spectrum. A beam chopper driving motor is mounted in the base of the monochromator. The chopped radiation signal is rectified by synchronous breakers on the chopper shaft before passing to the recorder.

III. Experimental Procedure

The following is a step by step procedure to illustrate the method for operating the Hohlraum Spectral Analyzer.

1. The vacuum pump is turned on and allowed to evacuate the sample compartment to a pressure of 10^{-2} torr.

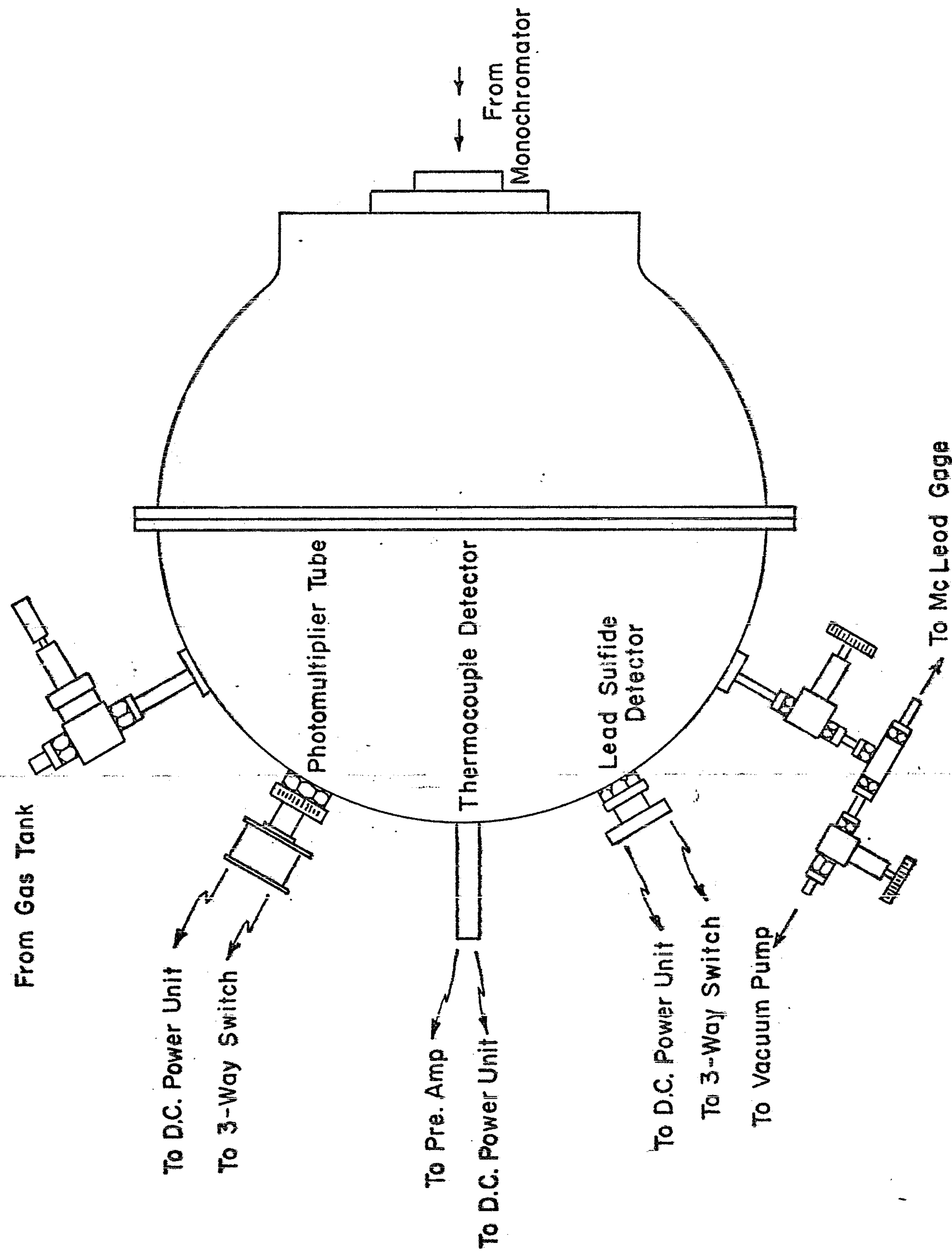


FIGURE 3. HOHLRAUM SPECTRAL ANALYZER

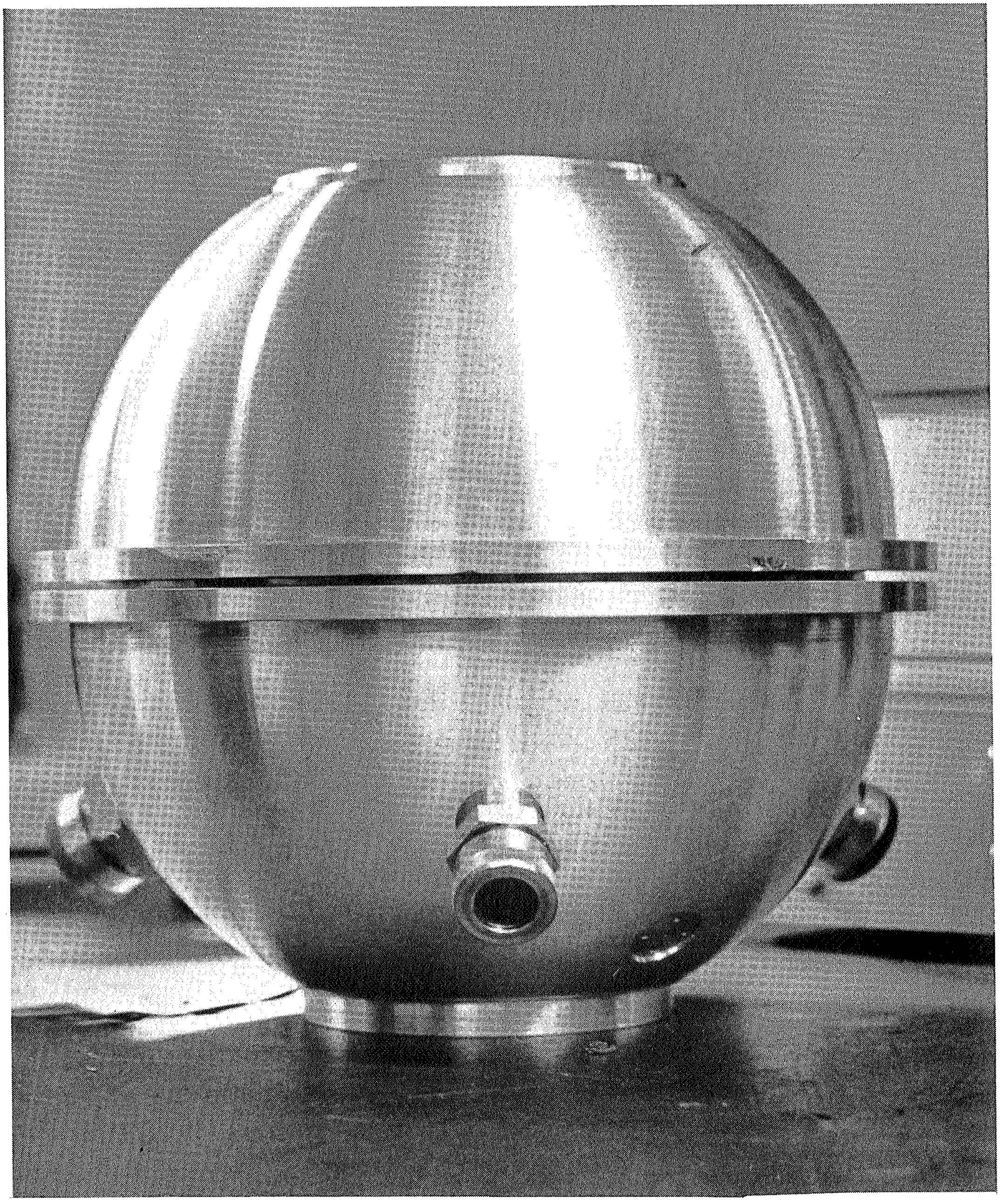


FIGURE 4 SAMPLE COMPARTMENT

2. The vacuum outlet valve is then closed and the pollutant tank valve is opened. The McLeod gage is used to monitor the partial pressure which is used to determine the PPm present of a particular pollutant. A mixture is formed by allowing the necessary quantity of high purity air to enter the sample compartment.
3. The power is turned on to the amplifier unit which in turn allows power to be supplied to the monochromator.
4. The sources are turned on independently depending on the region of the spectrum being analyzed. The tungsten lamp is used for the short wavelength region while the globar is used in the infrared region. Water is supplied to the globar for cooling.
5. The analyzer is now operational to determine the spectral data for the particular quantity of a given pollutant. The spectral variation is a resulting of turning the wavelength selector on the monochromator. The signal from the detectors is recorded on the strip-chart recorder. The spectometer operates on the principle that every mixture absorbs in distinctive spectral patterns.

IV. Scope of Work for the 1971-72 Year

The design and construction phase is near completion. The instrument development will be continued by performing the following task.

1. To obtain spectral characteristics for gas mixtures with known quantities of pollutants. The pollutants to

be tested are carbon monoxide, carbon dioxide, hydrogen sulfide, nitrogen dioxide, and sulfur dioxide. These gases are on order and should be delivered by the 15th of November.

2. To produce spectral traces of unknown samples. These unknown traces will be identified by comparison with traces obtained in Task 1.

3. To develop data handling equipment and software programs for data reduction from real problems.