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CONCAP IV:
A COMPLEX AUTONOMOUS PAYLOAD (CAP) FOR GROWING ORGANIC THIN FILMS IN MICROGRAVITY

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INTRODUCTION TO THE SCIENTIFIC MISSION

The Consortium for Materials Development in Space and the Center for Microgravity and Materials Research at the University of Alabama in Huntsville are collaborating to grow Nonlinear Optical (NLO) organic crystals and thin films. NLO materials play a key role in the fields of optics and optoelectronics. Loosely, they can be said to play the same role in optics that semiconductors play in electronics. There are many advantages to using organic materials over inorganic materials for NLO applications. The two advantages we are concerned with are that organic NLO materials frequently have a much higher nonlinearity than inorganic materials do and in some cases can transmit higher power densities.

One of the major drawbacks to organic NLO materials lies in the preparation of samples to be used in devices. Organic crystal growth is made difficult by weak crystal binding forces, low thermal conductivity, and, frequently, thermal instability at growth temperatures. A ground-based crystal and thin film growth facility has been developed which addresses these limitations. After carefully fine-tuning the ground-based facility it was adapted for use in the Get Away Special Canister. By growing crystals in space we hope to obtain more uniform compositions and avoid defects caused by plastic deformation of the crystals under their own weight at the elevated growth temperature. By growing thin films in space it may be possible to obtain more uniformly ordered films in terms of both crystallinity and composition.

GAS-105: THE FIRST NLO MISSION

The first microgravity NLO crystal/thin film experiment was GAS-105, flown in June 1991 on the STS-40 GAS bridge. The results were both encouraging and disappointing. Four crystal growth cells and two thin film growth cells were flown with a four-day growth time. Neither thin film cell produced a thin film, two of the crystal seeds shrank instead of growing, another decomposed, and the remaining crystal grew well. In most cases the quality of the starting material proved to be of prime importance. The temperature controller and software provided extremely high stability (±0.01°C) in some cases. The insulative technique we employed was very efficient, using approximately 3W of power to operate two growth cells at 125°C in a single oven chamber. This hardware will be described in more detail in a later section of this paper.

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The thin films are being grown from a diacetyline monomer starting material, with polymerization being induced after the thin film is grown. Each of the thin film cells on GAS-105 experienced different problems, both due to the presence of impurities. In one case the monomer polymerized prior to thin film transport. Vapor transport is impossible once polymerization has taken place due to the subsequent low vapor pressure. Although the elevated temperature contributes to polymerization once the process has begun, it was the presence of impurities which initiated polymerization at the growth temperature. The other thin film cell did undergo vapor transport, but when the cell was opened after the mission it was observed that the transported material was a brown, viscous liquid instead of a white (monomer) or purple (polymerized) fibrous film. It was determined that the material decomposed to a diol after transport due to the presence of impurities.

Four crystal growth cells were flown. Two contained a substituted nitroaniline compound (DAN), the other a proprietary material from our industrial sponsor, Teledyne Brown Engineering (TBE-1). Each crystal growth cell was equipped with small starting seeds on the growth sting to avoid nucleation problems. In both of the DAN cells the seeds actually shrank during the mission, rather than growing. This was due to improper selection of growth temperatures. In the case of the first TBE-1 sample, the source material and the seed crystal decomposed. This decomposition was stimulated by the presence of impurities in the sample material. The second TBE-1 sample exhibited excellent growth in terms of both transparency and faceted morphology. The seed crystal, however, was polycrystalline to begin with, so the grown crystal was a polycrystal also. However, even if the crystal had been a single crystal, it still would have been too small to characterize optically. TBE-1 grows so slowly that it takes nearly three weeks to obtain a crystal large enough on which to perform NLO characterization.

FUTURE NLO MISSIONS

The nature of crystal and thin film growth has led to the separation of follow-up missions into two categories, short-term and long-term. Short-term missions will be dedicated to growing thin films, which takes only a few hours. Long-term missions will be dedicated to growing crystals, which takes from two to four weeks. The short-term experiment platform will be Complex Autonomous Payload (CAP) missions on the Space Shuttle. Vapor transport is not disturbed by gravity levels less than 10^{-2}g's. It is not too difficult to find Shuttle missions which provide five hours of stability at 10^{-3}g's or less.

The long-term platform will be the Commercial Experiment Transporter COMET. This is a free-flying satellite which is expected to provide 30 days of microgravity at 10^{-5}g's. The COMET NLO crystal growth experiment is currently scheduled for launch in the first half of 1993. No further discussion will be made of this aspect of the program since the focus of this paper is the CAP payload.

CONCAP IV: SPACE SHUTTLE NLO THIN FILM GROWTH

The Consortium for Materials Development in Space is developing a series of CAP missions, called CONCAP. The NLO project is the CONCAP IV series. The first NLO mission, CONCAP IV-01, is currently slated for launch on STS-57 in the spring of 1993.

CONCAP IV-01 Requirements

Power Requirements

Each oven, containing two growth cells, consumes approximately two watts of power when operating at 95°C. CONCAP IV-01 will accommodate six pairs of cells, yielding a total steady state power consumption of 12W by the heaters during thin film growth. Initial heat-up demands will, of course, be higher, roughly a total of 40W for one hour. In addition to this, the controller and other electronics draw about 3W. Thus the total average power draw during thin film growth is about 15W with a total growth time of 3 hours. The total battery requirements then are 40W-hrs plus 45W-hrs, or roughly 85 W-hrs.

Vacuum Requirements

The CONCAP IV experiments require access to the vacuum of space for three purposes: 1) vacuum is one of the insulative techniques used to obtain the highly efficient thermal conditions, 2) vacuum enhances vapor transport process , and 3) exposure of the source materials to non-inert gases (i.e. oxygen) increases the likelihood of decomposition at elevated temperatures.

Experiment Time

There are three distinct phases in the thin film growth experiment: 1) heat-up, 2) thin film growth, and 3) cooldown. The exact nature of each phase will be discussed in a later section, but the times required are one hour for heat-up, three hours for thin film growth, and one hour for cooldown.

Microgravity

It has been shown that, while melts and fluids are subject to gravitationally-driven convection at 10^{-5}g's to 10^{-6}g's, vapors tolerate g-levels as high as 10^{-2}g's. Therefore the CONCAP IV missions require gravity levels of 10^{-2}g's or greater.

Gas Backfill

After thin film growth is complete it is necessary to backfill the growth cells with enough inert gas to quench vapor transport, roughly two orders of magnitude more pressure than the vapor pressure of the grown thin films. This turns out to be roughly 2 psi.

Experiment Hardware

The overall payload layout is shown in Figure 1. The experiment subsystems are: 1) the primary experiment support structure, 2) the vacuum vent valve assembly, 3) the inert gas backfill assembly, 4) the primary battery, 5) the controller, and 6) the physical vapor transport (PVT) hardware.

Primary Experiment Support Structure

Figure 1 shows the primary experiment support structure. It consists of three parts: 1) a round baseplate, to which is attached 2) a rectangular mounting plate, and 3) bumpers to secure the free end of the rectangular plate. The assembly is aluminum and weighs about 50lb.

Vacuum Vent Assembly

The vacuum vent assembly is shown in Figure 2. The valve consists of a bellows vacuum valve coupled to a polarized gear motor. The motor is controlled by the experiment controller and can be operated in either direction via polarity switching. Venting is done through the modified battery vent turret supplied by GSFC. The modification consists of a 9/16-18 thread into the side of the top of the turret. Into this thread is attached the vacuum vent tube, consisting of a corrugated metal hose with a 9/16-18 straight thread/o-ring terminus.

Gas Backfill Assembly

Figure 3 shows the gas backfill assembly. It consists of a 500ml DOT-rated pressure vessel, a motor/valve assembly, and a connecting stainless steel tube. Use of the DOT cylinder greatly eased the safety process. The cylinder is held in place by an aluminum bracket on either end. The connecting tube is welded into the valve port and swaged into the cylinder connector.

Primary Battery

The primary battery is undergoing a design change as of the deadline for this paper. The original experiment configuration called for a 13-day crystal and thin film growth mission. With this mission requirement in mind we selected a 200A-hr 15-cell silver zinc battery (4500W-hr). Subsequently, however, we obtained the COMET free-flyer long duration mission opportunity and changed the focus of CONCAP IV-01 to thin films only. This greatly reduced our power needs to less than 200W-hr. Currently we are investigating the use of lead-acid cells for this application.

Controller

The controller consists primarily of three printed circuit cards: 1) the smart card, 2) the power switching card, and 3) the thermistor signal conditioning card. The smart card is built around a Z80-based microprocessor chip. The signal conditioning card has 64 8-bit A/D channels for temperature acquisition. The current configuration uses a gain of twenty to achieve better than 0.01°C resolution in a 4°C window around the set-point. We also have ungained resolution of about 0.2°C over the range from 0°C to 130°C. The power switching card uses P-FETS to provide 24 channels of 28V on-off supply. A control algorithm using proportional PID on/off control is capable of maintaining the temperature to within ±0.012°C.

Figure 4 shows the controller assembly. The mounting structure consists of two aluminum angles with slots running down their lengths into which the cards are placed. Expanding wedges are used to hold them in place. A bottom plate is used to hold the assembly together during mounting and bench-top activities. The top and end are for mechanical and dust protection and can be eliminated from the flight configuration if weight is a problem.

PVT Hardware

The PVT hardware is shown in Figure 6. It consists of the insulative assembly, referred to as the NLO ovens, and the thin film growth cells. the NLO oven is made of two polished aluminum concentric cylinders. The inner cylinder is held into place with small kevlar strings. The strings are the only mechanical contact between the two cylinders, thus minimizing conductive heat loss. The concentric cylinder arrangement minimizes radiative heat loss and the vacuum between the two eliminates air as a heat path. In this way the primary heat loss path is the heater and thermistor wires leading from the growth cells. In this way the highly efficient thermal behavior (125°C at 3W) is obtained. Each NLO oven is approximately 12 inches long and houses two PVT (thin film or crystal) growth cells.

The PVT cells consist of three parts: 1) the source chamber, 2) the growth sting, and 3) the flange. The source chamber is a glass tube 2.5cm diameter by 5cm long closed off at one end. The source material for thin film growth is placed into this chamber and is held in place by a stainless steel screen epoxied in place across the middle of the tube. The heater is a 165Ω kapton thin film heater wrapped around the glass cell with shrink band. The thermistor is epoxied directly onto the shrink band.

The growth sting is made out of a copper rod 2.5cm long by 1cm diameter. The heater, which is a 1.6KΩ .5W resistor, and a thermistor are epoxied into a hole drilled through the center of the copper rod.

The flange is made of teflon and serves three purposes: 1) mechanical support, 2) thermal isolation, and 3) leak to vacuum. Mechanically, the teflon flange serves to hold the copper sting in place relative to the glass cell. Thermally, the flange separates the sting and the cell so that significant (16°C) temperature differences can be maintained over a relatively short distance.

The third role of the teflon flange is providing a leak to vacuum through 4 small holes drilled through it. The leak to vacuum is necessary to provide more efficient vapor transport and to remove decomposition impurities which would otherwise accumulate in front of the growing thin film.

SUMMARY

A GAS facility has been developed for carrying out low temperature experiments. The arrangement being used on CONCAP IV-01 is designed for thin film growth in microgravity and can process up to twelve samples per mission. The NLO oven hardware can also be made suitable for other low temperature applications, such as gradient freeze and solution growth of crystals.

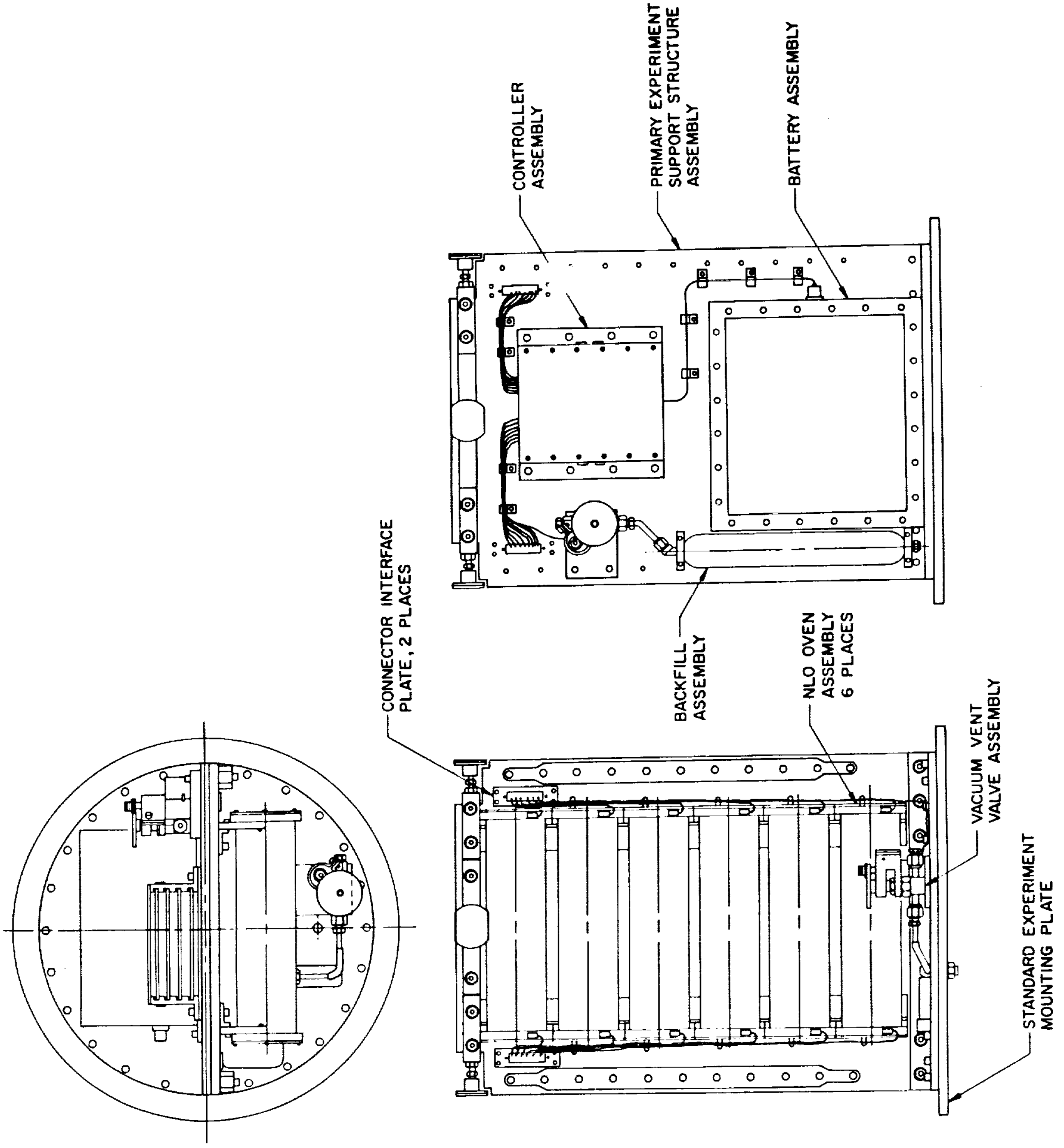

Figure 1: CONCAP IV Payload Layout

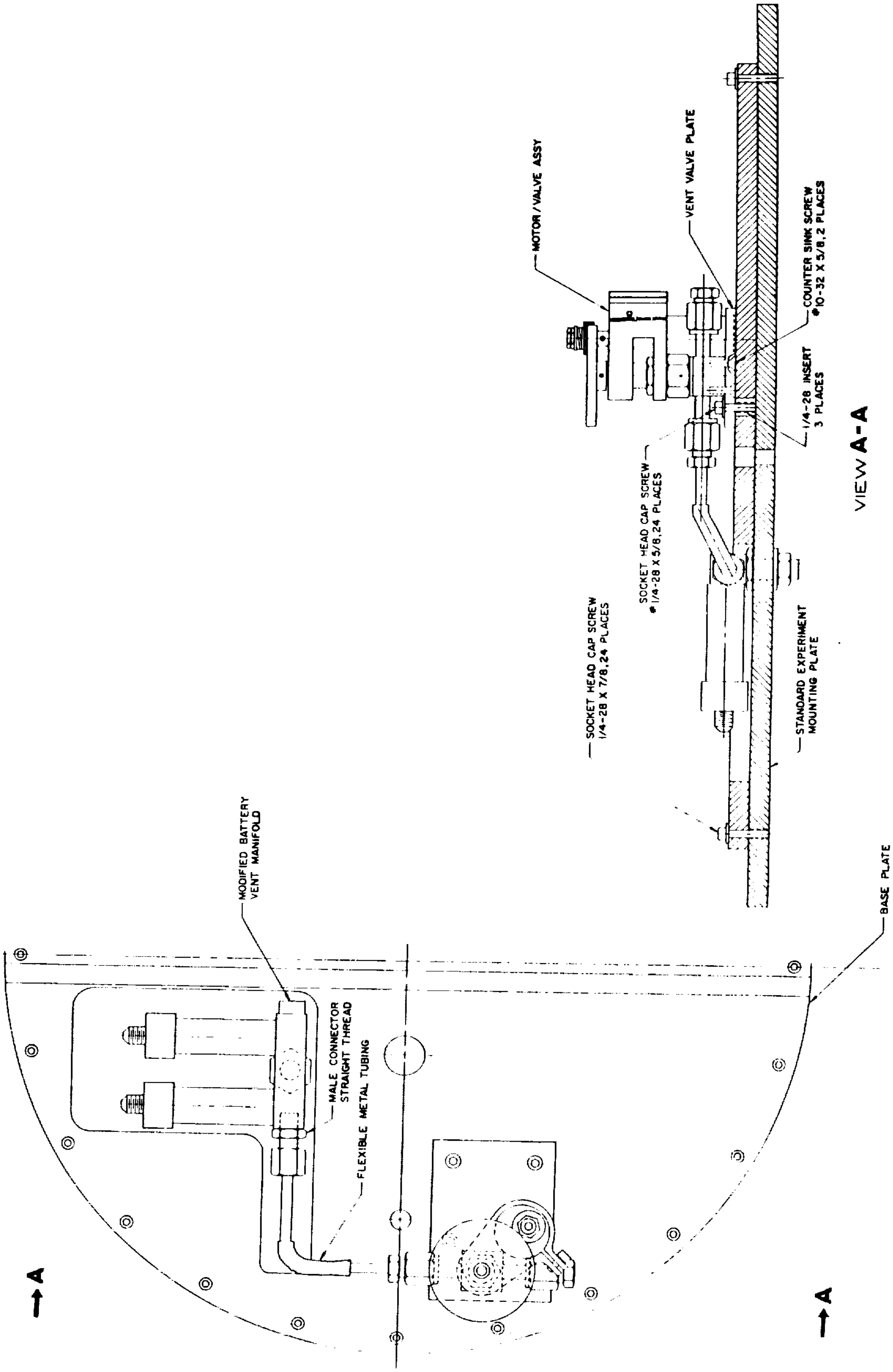

Figure 2: CONCAP IV Vent Valve Assembly

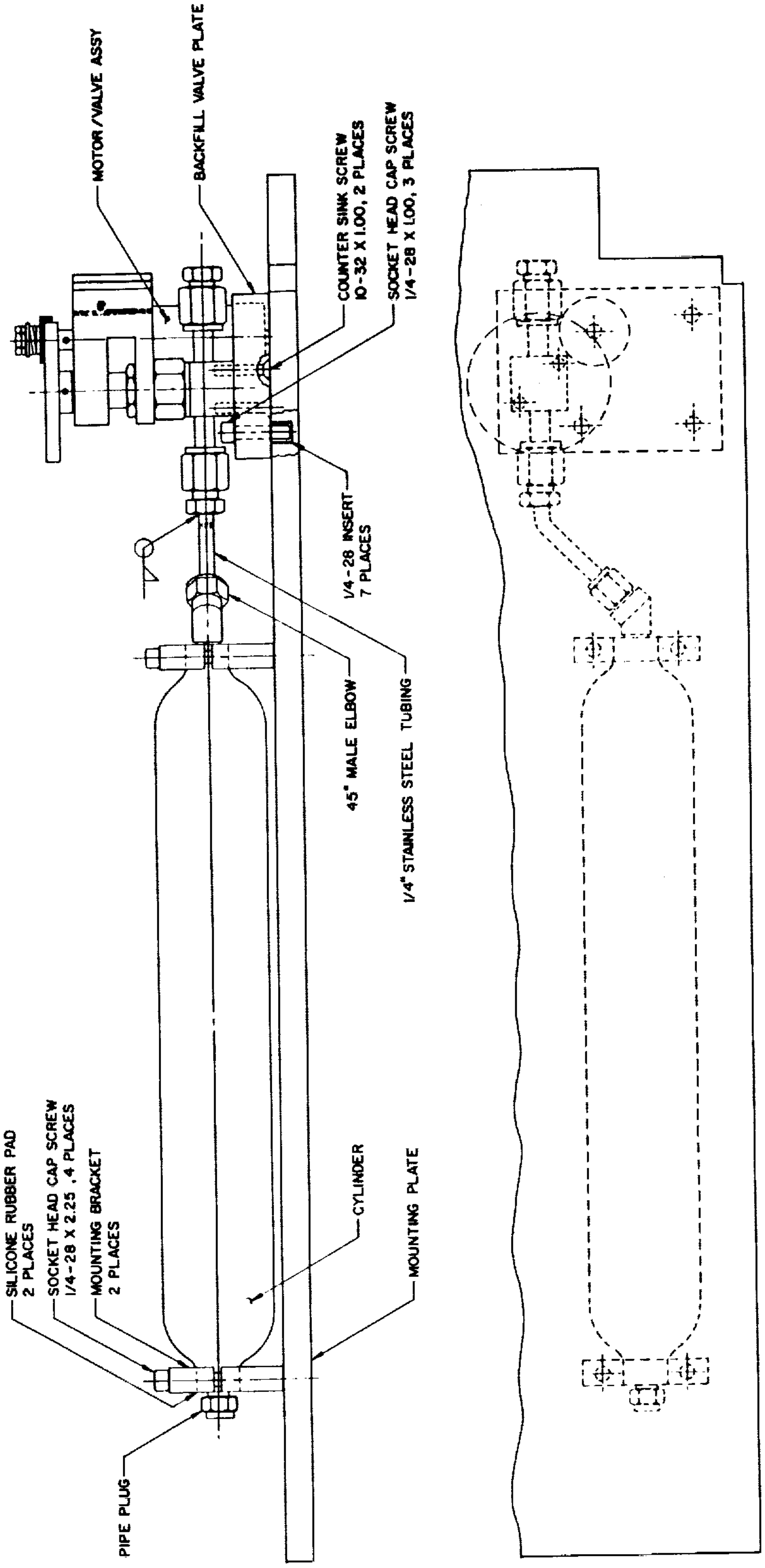

Figure 3: CONCAP IV Backfill Valve Assembly

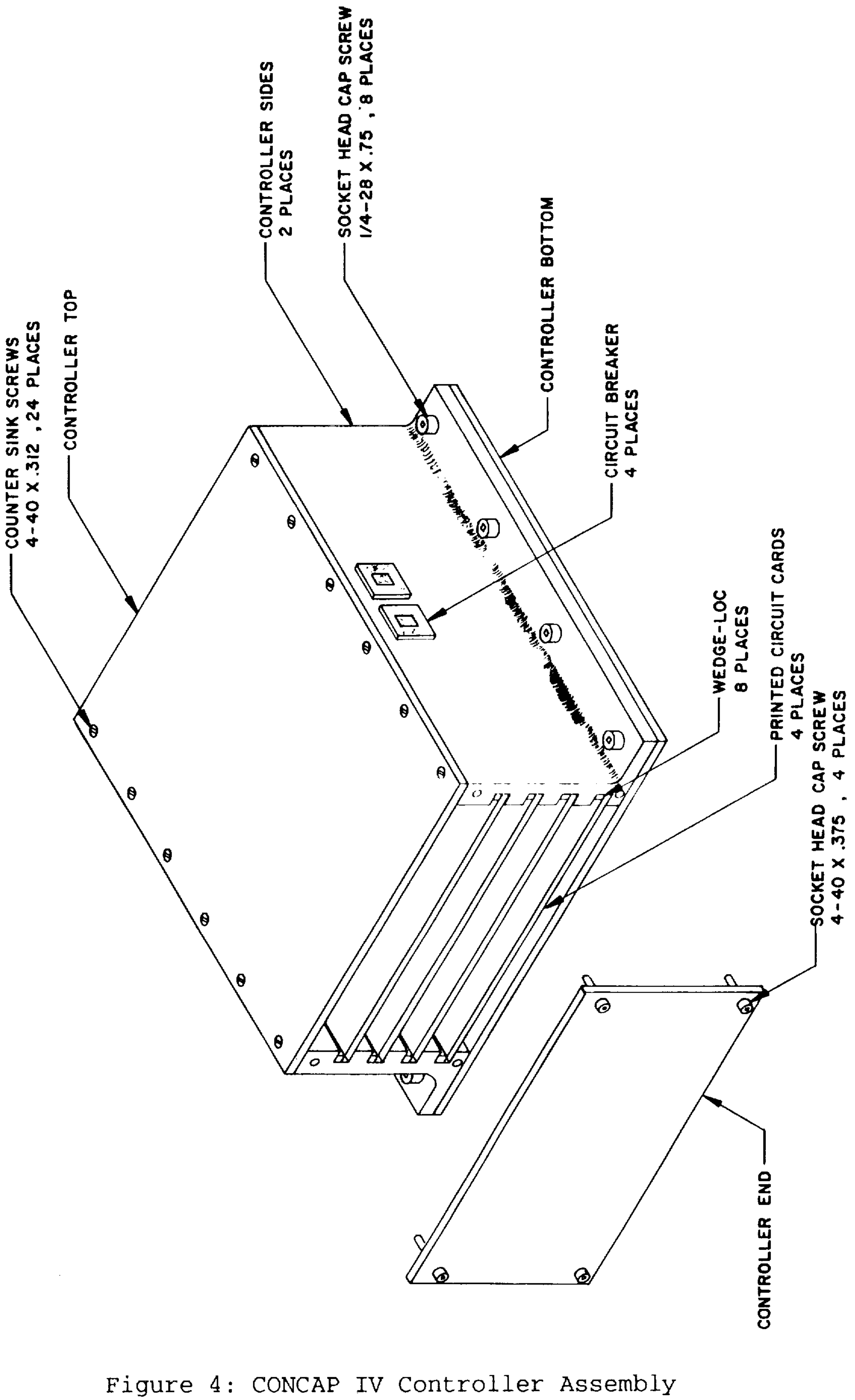

Figure 4: CONCAP IV Controller Assembly

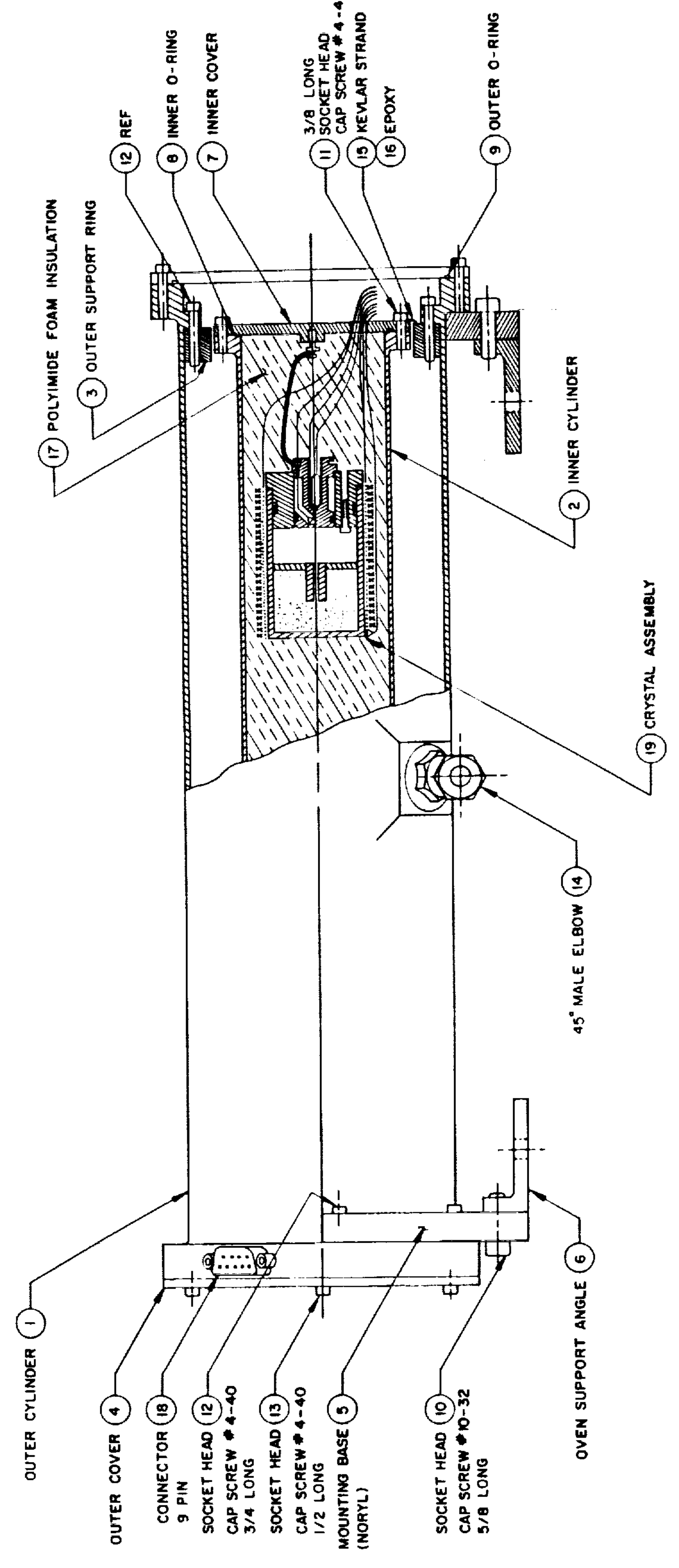

Figure 5: NLO Oven assembly with PVT cell