Variable Temperature Control with Joule-Thomson Temperature Stages

... the experts in variable temperature solid state characterization!

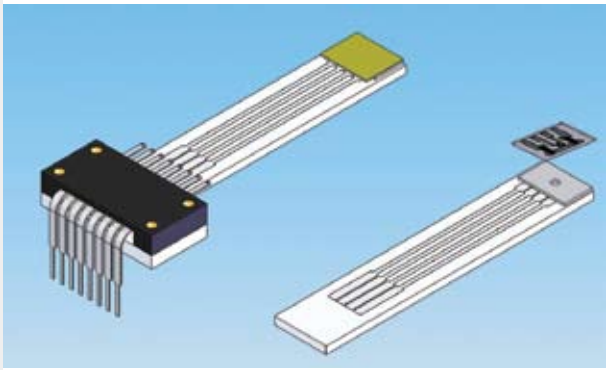
MMR Technologies, Inc

The Joule-Thomson Effect

When a non-ideal gas expands from a high pressure to a low pressure, there is a temperature change even though no other work is being done by the gas. This is the Joule-Thomson expansion of gas. The ratio of the temperature change to the pressure change is known as the Joule-Thomson Coefficient. When this ratio is positive, a drop in pressure results in a drop in temperature. At MMR Technologies, this principle; the expansion of compressed gas to atmospheric pressure generating liquid cryogen, is utilized in the microminiature refrigerator.

High pressure gas (blue arrow) enters the heat exchanger at approximately 1800 psig. The gas passes through capillary channels and undergoes two pressure drops (red circle and black circle) in short succession resulting in an overall temperature drop of 15 to 20 degrees. Roughly 80% of this precooled gas follows a second set of capillary channels out of the microminiature refrigerator, pre-cooling the new incoming gas. Repeated cycles of gas coming in, pre-cooling of the incoming gas by the outgoing, and the pressure drops results in liquefaction (blue circle) of the incoming gas when the thermal stage is under vacuum. The gas exits the thermal stage at less than 10 psig in pressure (red arrow).

Joule-Thomson Thermal Stages



Joule-Thomson Thermal Stages come in three basic temperature configurations: room temperature only, hot stages, or combination cold and hot stages designed for operation over extended temperature ranges. The basic construction of a thermal stage is the same, regardless of the temperature range or the specific instrument. The stage material is either a special laminated glass plate constructed with micro channels for cooling or it is a ceramic stage for room temperature or hot stage applications. One one end of the microminiature refrigerator, a connector is mounted which is unique to each specific instrument. The connector also contains electrical leads and gas connections necessary for the operation of the thermal stage. At the other end of the stage is a sample mounting pad. Heating is provided by a small resistive heater mounted beneath the stage. Leads from the temperature sensors run from beneath the mounting pad to the connector.

Within the combination heating and cooling thermal stages, there are two types; thermal stages capable of temperatures down to 80K and thermal stages capable of temperatures down to 70K, which requires using nitrogen gas. The second type of thermal stage requires a vacuum assist at the refrigerator port to obtain temperatures below 80K.

A Typical Microminiature Thermal Stage System

A typical variable temperature Joule-Thomson thermal control system includes:

- High purity high-pressure gas (typically nitrogen or argon)
- A filter/dryer apparatus
- The thermal stage
- A temperature controller
- A vacuum chamber
- High pressure gas line tubing

Available Temperature Ranges on Thermal Stages

When a system is held under a vacuum pressure of at least 8 milliTorr, the following temperature ranges are available on the MMR Technologies' instruments:

- Room Temperature
- 70K to 580K*
- 80K to 580K
- 70K to 730K*
- 80K to 730K
- Room temperature to 730K

* Vacuum assist Joule-Thomson thermal stages require an auxiliary vacuum pump at the thermal stage gas exhaust. These thermal stages are not available on ultra high vacuum or scanning electron microscope systems.

When a thermal stage is used within an ambient pressure setup, with a well controlled atmosphere, as in atomic force microscopy, the following temperature ranges** are available using the appropriate thermal stage setup:

- -10 °C to 200 °C (using nitrogen gas) or -30 °C to 200 °C (using argon gas)
- -10 °C to 350 °C (using nitrogen gas) or -30 °C to 350 °C (using argon gas)
- Room temperature to 350 °C

** These are the maximum temperature ranges under ideal conditions like a glove box where there is no humidity and a dry, clean gas environment.

Specifications for Joule-Thomson Thermal Stages***

Operating Temperature Range:	Available between 70K and 730K
Minimum Temperature with no heat load:	80K with a nitrogen pressure of 1800 psi and 5 milliTorr vacuum 70K with vacuum assist at stage exhaust on "70K" thermal stages
Temperature Accuracy:	< 0.5K at 80K; +/- 0.5K between 80K and 400K; < 1.5K from 400K to 730K
Temperature Stability:	+/- 0.05K
Cooling Capacity:	250mW at 85K (nitrogen gas); 500mW at 95K (argon gas)
Maximum Cooling Rate:	No load cool down time from 300K to 80K in less than 20 minutes No load cool down time from 730K to 300K in less than 20 minutes
Maximum Heating Rate:	15 K/minute No load warm-up time from 80K to 300K is less than 5 minutes. No load warm-up time from 300K to 730K is less than 5 minutes.
Additional Power Supply Required:	Only on stages that go up to 730K
Temperature Sensor:	Platinum Resistance Thermometer
Sample Mounting Surface Size:	10 mm x 12 mm
Maximum Sample Weight Allowed:	No more than 5 grams
Sample Stage Material:	Aluminum Oxide
Thermal Stage Dimensions:	Approximately 35 mm x 100 mm (dependent on thermal stage model)
Weight of Thermal Stage:	Between 10 gram and 60 grams, depending on the model
Temperature Controller Requirements:	K-20 Programmable Temperature Controller
Filter/Dryer Requirements:	Either the standard filter dryer or the reversible filter dryer system
Gas Requirements:	99.998% Pre-Purified Nitrogen or Argon Gas of at least 1800 psi delivery pressure
Vacuum Requirements:	8 milliTorr or less within the vacuum chamber

*** The Joule-Thomson Thermal stages have similar specifications across all offered temperature ranges. Any differences are noted under the specification concerned. Thermal stage specifications may be changed at any time by the manufacturer. Please contact the manufacturer for the latest information and specifications.

Features and Benefits

Microminiature refrigerators or Joule-Thomson thermal stages provide many advantages in scientific applications:

- Fast cool-down and warm-up.
- Precise temperature control: +/- 0.1K
- Excellent temperature setability, stability, and reproducibility.
- Absence of mechanical, acoustic, or electrical noise.
- Small, compact size.
- Wide range of operation: 70K to 730K
- Non-magnetic electrical feedthroughs facilitate electrical connections directly to samples on the thermal stage.
- Low cost of operation: $0.50/hour
- No liquid cryogens to handle. All cryogens are produced and consumed within the stage, making this safe and easy to use
- No maintenance required
- Very low power consumption - less than 12 watts on any stage.

Applications

MMR Technologies provides many different microminiature refrigerators or Joule-Thomson thermal stages for use in chemistry, physics, and materials science - as well as other special applications, including:

- Hall Effect Measurement Systems
- Seebeck Effect Measurement Systems
- Micro manipulation and Microprobe Systems
- Deep Level Transient Spectroscopy Studies
- Optical Microscopy
- Electron Microscopy
- Atomic Force Microscopy
- Optical Transmission Spectroscopy
- X-Ray Diffraction
- Raman and Fluorescence Studies
- Interface with customized vacuum and environmental control chambers

These systems are flexible, modular, and highly adaptable to fit into most applications, giving variable temperature control as a valuable addition to any experimental setup.

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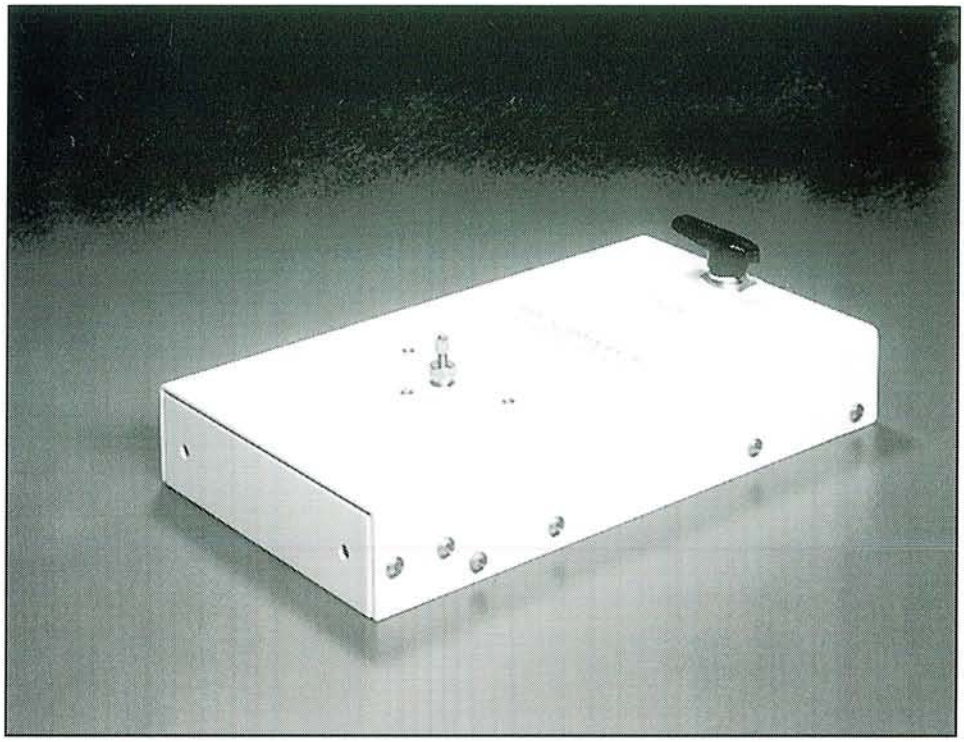
Model F2105

Product Description



The Model F2105 Reversible Gas Filter-Dryer has been designed to remove water vapor and other condensible contaminants from high pressure gas used with the company's line of microminiature Joule-Thomson refrigerators. The unique design of this filter-dryer makes it unnecessary, in normal operation, to replace the contents of the drying cartridges.

Applications

The Model F2105 Reversible Filter-Dryer can be used for a variety of applications, including:

- The drying of argon, nitrogen, hydrogen, helium and many other gases to a Dew Point below -75°C.
- Use of this dryer with MMR's line of microminiature refrigerators allows continuous opera tion of the refrigerators for up to hundreds of hours without clogging.
- The dryer may be used to provide point-of-use, dry gas at a purity level, previously attainable only with dryers of much greater cost. In addition, unlike other such dryers which must be replenished periodically, the drying cartridges of the Model F2105 never need to be replaced or refilled.

Features

Several unique features of the F2105 provide significant user benefits.

Simple compact design — Requires only 7" x 12.5" of bench space, or may be wall mounted.

Dew Points below -75°C — Attainable without the use of expensive prepurified gases.

Leak free operation — 1/8" Swagelock fittings on input and outlet assures leak-free operation.

High pressure operation — Designed for

Inlet pressures to 2,500 psi.
Outlet pressures to 2,500 psi.
Exhaust at atmospheric pressure.

High throughput — Continuous flow to 4 1/m. (8.8 scfh)

Small bypass flow — Less than 0.3 1/m. (0.66 scfh)

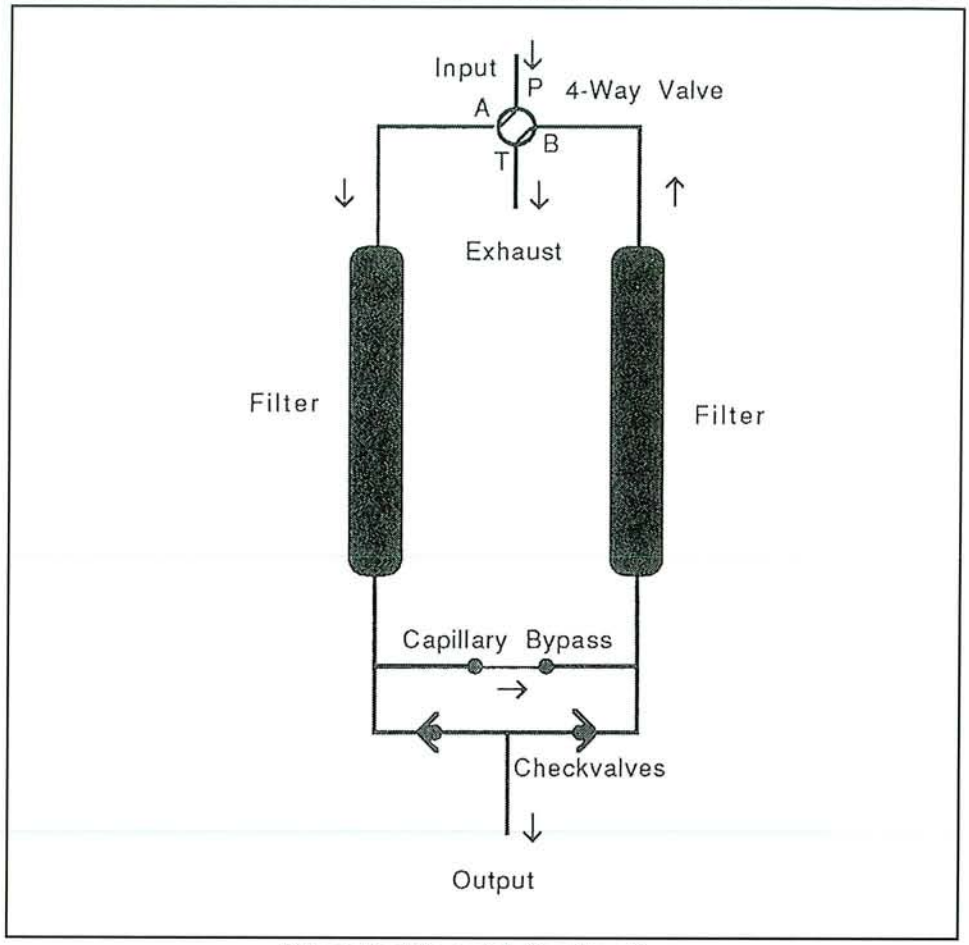
Simple operation — Active filter selected manually. Cartridges never need replacement.

No electrical power required — System is self purging, operation requires only high pressure gas.

Multiple filters — 3 micron filters on inlet and outlet lines prevents particulate contamination of gas lines or refrigerators used with the filter-dryer.

Model F2105

Specifications



Schematic of Reversible Gas Filter-Dryer

Size — 12.5" x 7" x 2".
Weight — 4 pounds.
Electrical Power Requirements — None.
Outlet Gas Dew Point — <-75°C typical.
Fittings — Input and Output Fittings - 1/8" Stainless Steel Swagelock.
Mounting — Wall or Benchtop. (Mounting Bracket supplied)

Principle of Operation

The Model F2105 Gas Filter-Dryer uses the principle of the heaterless dryer to achieve an extraordinary purity of outlet gas. The filter-dryer shown schematically above contains two drying cartridges which are selected for use through the four way valve shown. In use, moderately dry gas from a high pressure cylinder is passed through the four way valve to the first drying cartridge. After being purified by this cartridge the gas passes through a check valve and then to the outlet, to the refrigerator. In addition, a small fraction of the gas from the first cartridge is allowed to expand through a fine capillary constriction to atmospheric pressure and then passes back through the second cartridge, then through the four way valve to the exhaust port. This dry gas purges impurities and water vapor from the second cartridge. After 4 to 24 hours of operation, the four way valve is reversed, reversing the flow of gas through the two cartridges. With each reversal, the gas at the high pressure outlet becomes more and more highly purified, eventually reaching a purity level, containing less than a few tens of parts per billion of water vapor.

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Model K-20-1

Product Description

The Model K-20-1 Programmable Temperature Controller is intended for use with the cryogenic cooling and thermal stage systems of MMR Technologies. It provides accurate temperature measurement, precise and very stable temperature control and easy to use data acquisition functions over the temperature range of 20K to 730K. Controlled cycling, temperature ramping and changing temperature operation under software control are possible. The unit is compatible with a variety of auxiliary computers or smart keyboards through either an IEEE-488 or RS-232C interface. Computer interface software is supplied for use under Windows 3.1. A LabView Program is now available

Applications

The Model K-20-1 can be used for a variety of applications including:

- Provide very precise, very stable controlled temperatures anywhere within the range of 20K to 730K.
- Record temperature, system operating parameters and experimental data as a function of time
- Record system operating parameters and experimental data as a function of temperature
- Serve as data acquisition system
- Provide programmable voltage source

Features

Several unique features of the K-20-1 provide significant user benefits.

Excellent temperature setability and stability - the electronic temperature controller enables the user to set temperature with an accuracy of ± 0.5K, achieves a temperature stability of better than ± 0.05K when used with MMR's cooling systems in the range of 70K to 400K.

Temperature monitor - temperature readout is in Kelvin. The temperature monitor may also be used as a 16 bit voltage monitor.

Monitors Pt RTD and Silicon diode temperature sensors - cover operating temperatures of 70K-730K and 20K-400K respectively.

Heater power monitor - refrigerator cooling capacity or sample power dissipation on the sample mounting stage can be measured to within 5%.

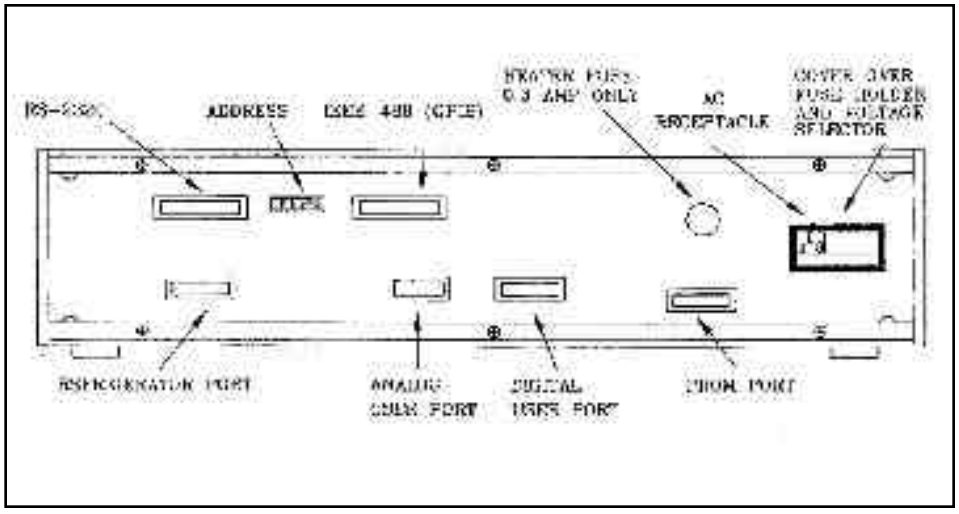
Analog signal output port - the analog output port can supply a programmable voltage of ±1.250 VDC, ±0.002 Volts.

Model K-20-1

Features

Analog signal input ports - two analog input ports are provided for use in data acquisition. Each can measure a voltage of ±2.50 VDC, with a precision of ±0.010 VDC under operator or software control.
Digital signal input port - an 8-bit digital input port is provided to read switch positions, etc.
Digital signal output port - an 8-bit digital output port is provided for the control of system support devices such as gas valve, solenoid, compressor, relay, etc.
Computer interface - the Model K-20-1 can be interfaced to a smart keyboard (RS-232C) or a computer (IEEE-488 or RS-232C) to place the unit under manual and software control respectively.
Data as function of time - the Model K-20-1 can be supplied with data acquisition and control software which logs and displays data as a function of time.
Data as a function of temperature - ThermoPlot® software is available which logs and displays data as a function of temperature rather than time.
PID constant setting - a single PID constant can be varied from 1 to 255, default setting is 100.

Specifications



Operating Temperature Range — 20K-400K - Silicon Diode
70K-730K - Pt RTD
Temperature Setability — ±0.5K (20K to 400K), ±1% (400K to 730K)
Temperature Resolution — 0.01K
Temperature Stability — ±0.05K or better (80K to 400K)
Temperature Rate Change — 20K/sec or faster when heating
6K/sec or faster when cooling
Power Requirements — 110V, 60Hz (Optional: 100V, 60Hz; 120V, 50Hz; 220V, 50Hz)
Weight — 12 lbs
Shipping Weight — 18 lbs
Size — 3 1/2" x 17" x 14"
Rack Mounts — Optional
IEEE-488 Compatibility — Capital Equipment Board

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MMR Technologies Refrigerator Vibration Measurements

Performed by Dr. Hunt at Ohio University

January 1993

Figure 1: Scanning Tunneling Microscope apparatus with MMR refrigerator installed to sample mount.

Figure 2: Closeup of MMR refrigerator mounted to beam which is attached to the sample mount of the STM.

Figure 3: Graphite film mounted on cold pad of MMR refrigerator and installed under the STM probe.

Figure 4: MMR refrigerator covered with cotton to isolate it from vibration in the surroundings.

Figure 5: Photo shows the high pressure filter and gas line feeding to the refrigerator.

Figure 6: STM image of graphite film with no gas flowing through the refrigerator. The carbon molecules have a spacing of 2.4 angstrom.

Figure 7: STM image with gas flowing, inlet pressure of 500 psi. Vibration amplitude is less than 2.4 angstrom.

Figure 8: STM image with gas flowing, inlet pressure of 1000 psi. Vibration amplitude is less than 2.4 angstrom.

Figure 9: STM image with gas flowing, inlet pressure of 1200 psi. Vibration amplitude is less than 2.4 angstrom.

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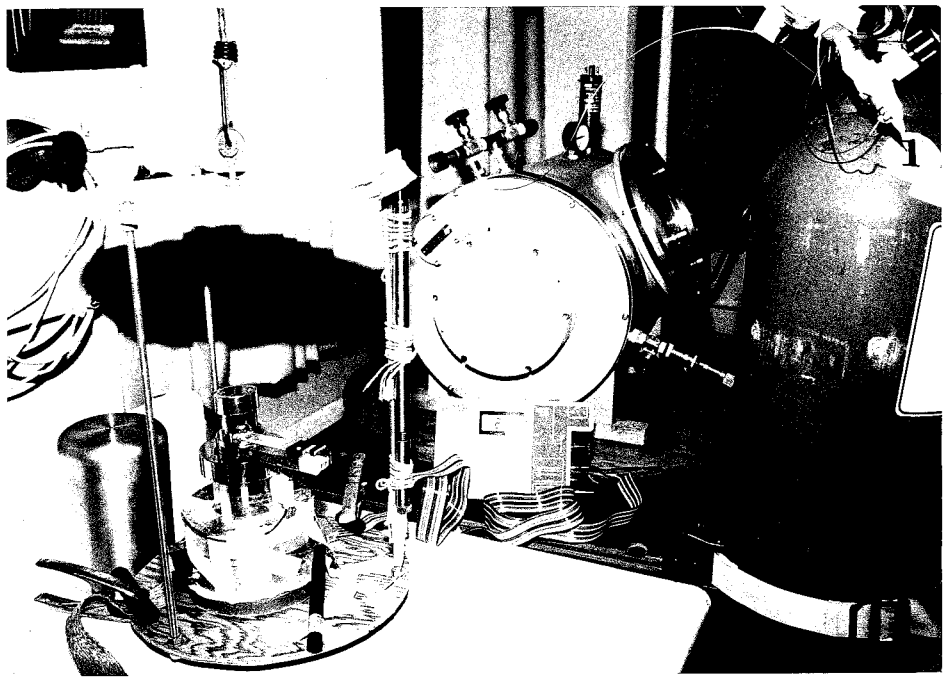

FIGURE 1

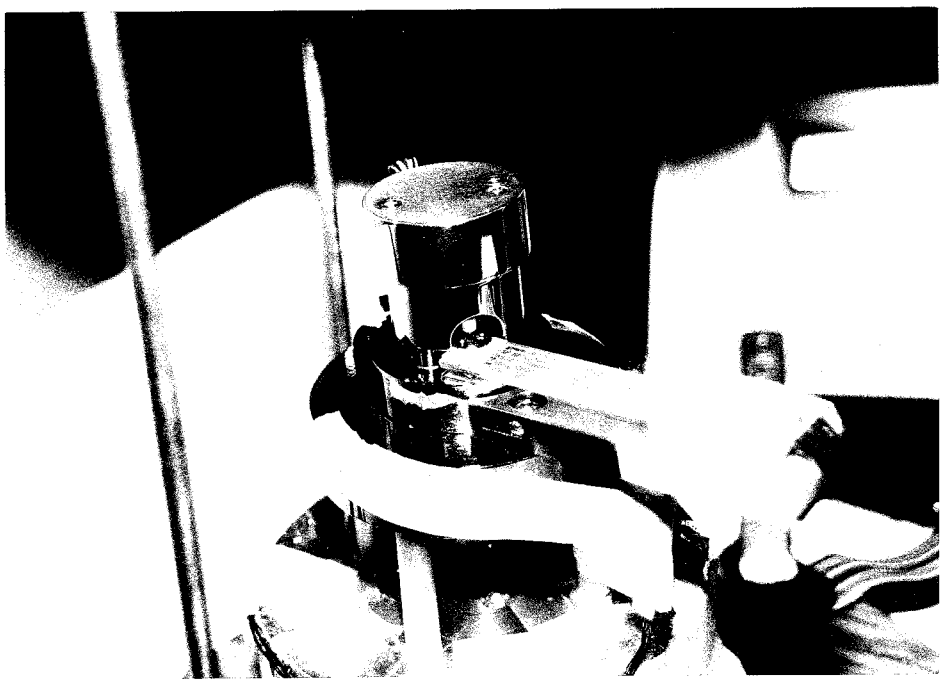

FIGURE 2

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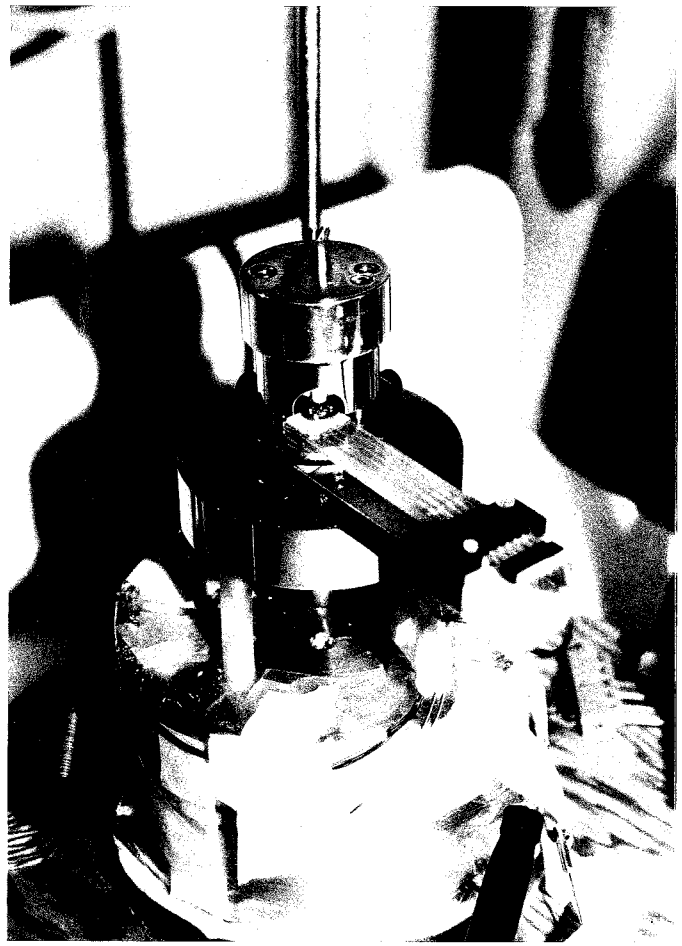

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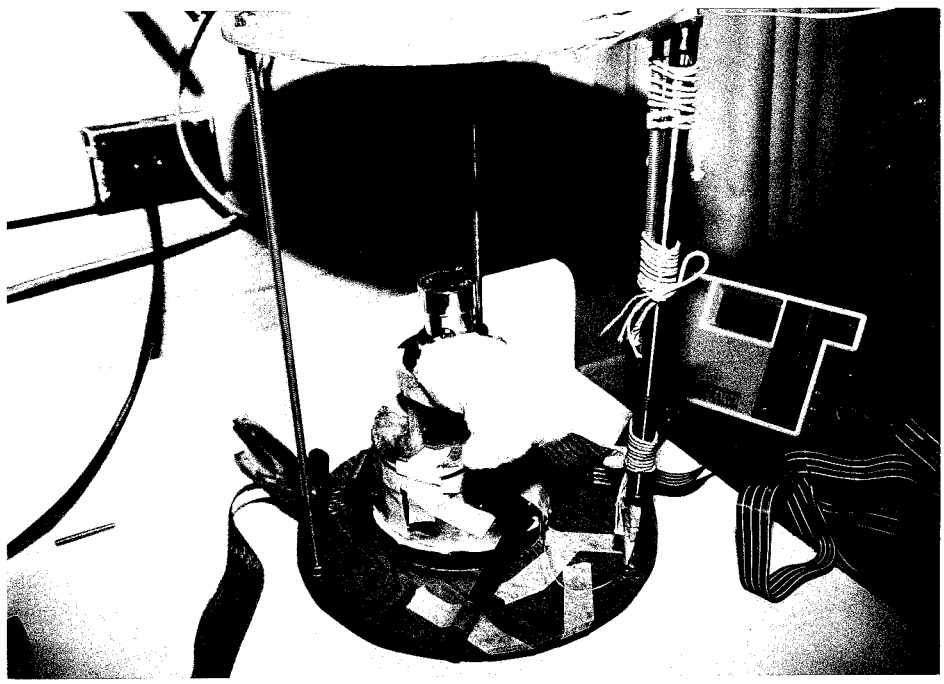

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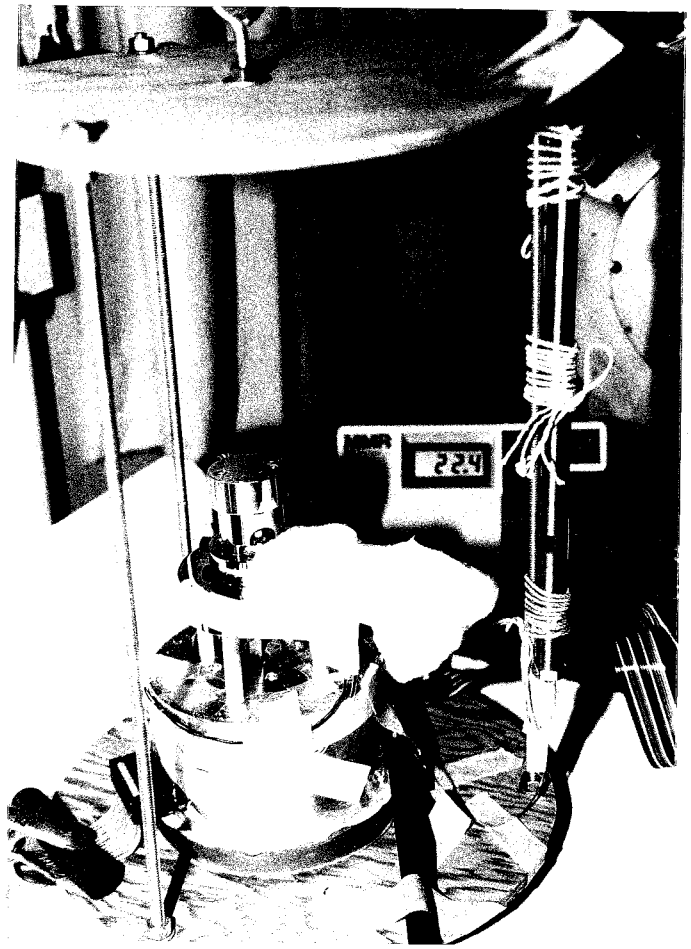

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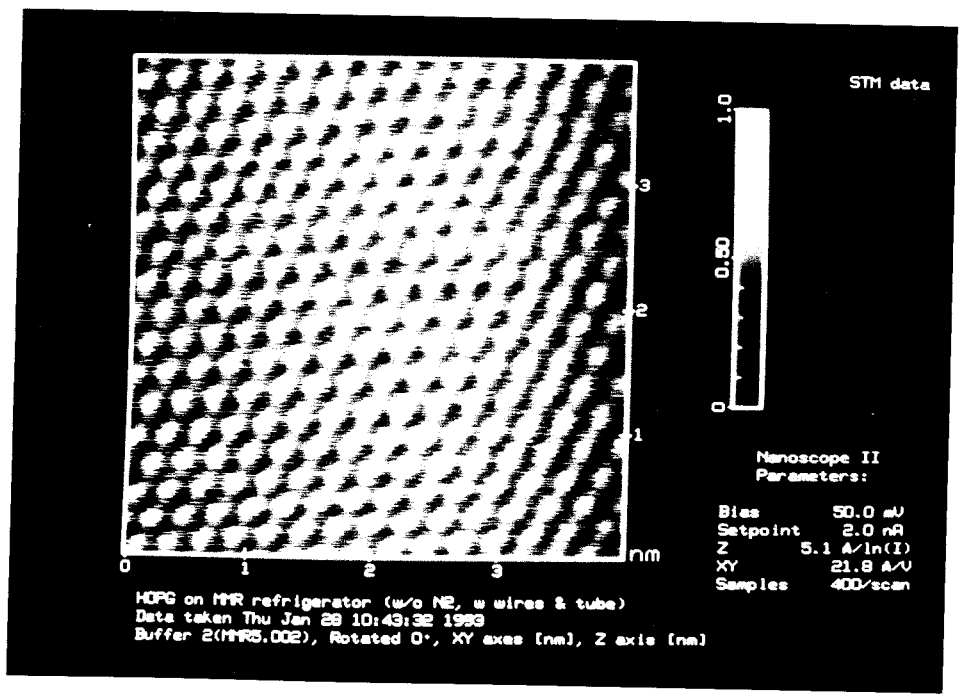

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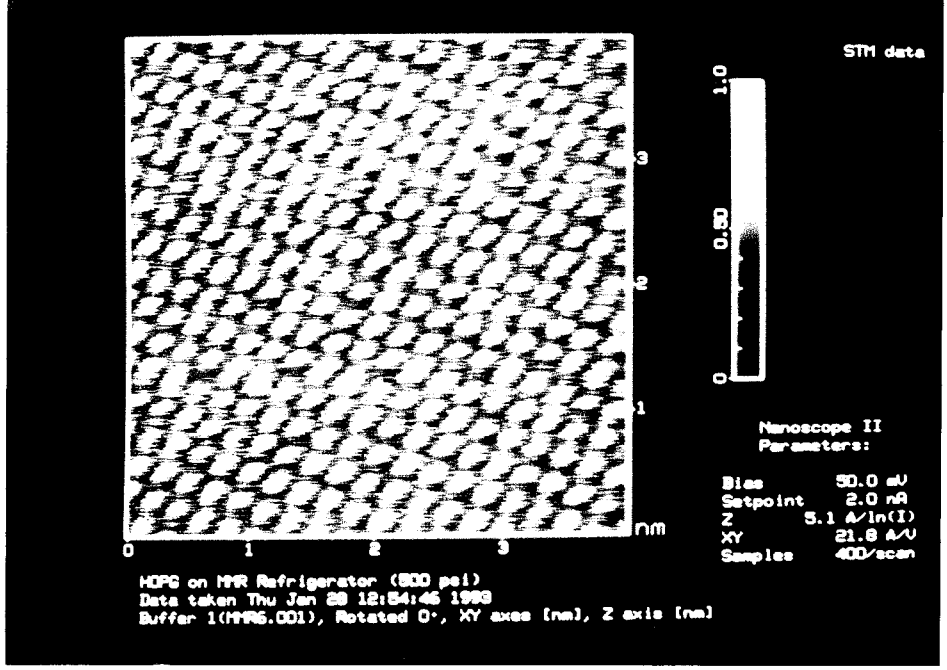

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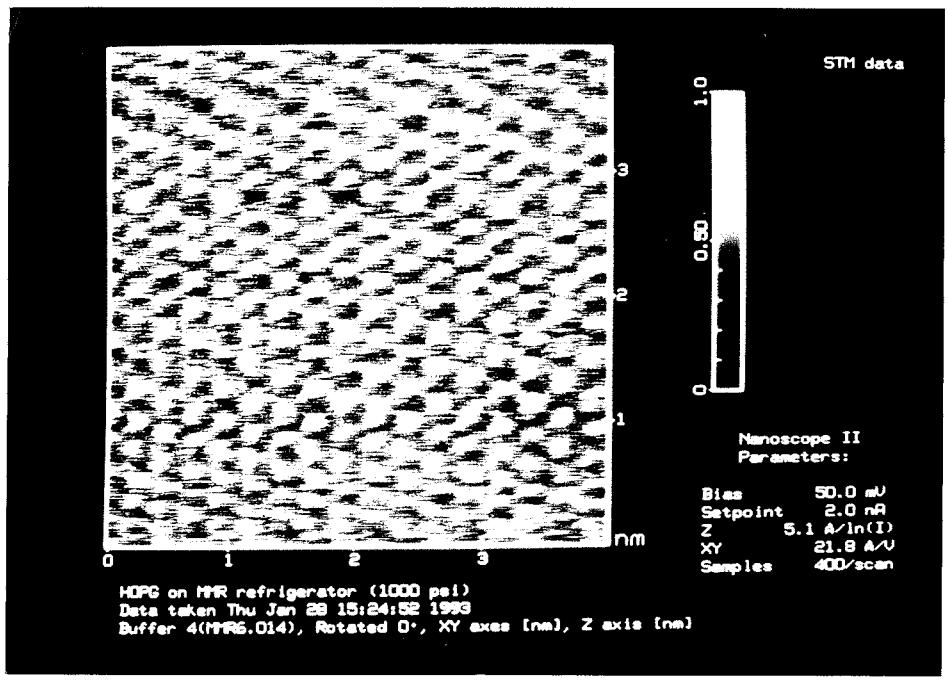

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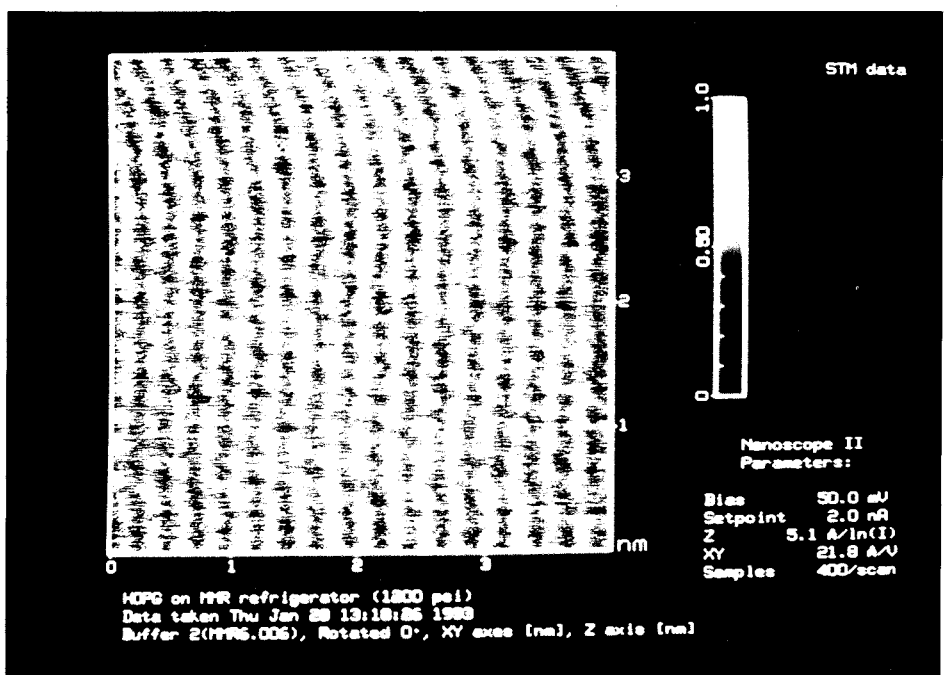

FIGURE 9