INSTRUCTION MANUAL

903 & 903H MOISTURE EVOLUTION ANALYZER

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Thermal Analyzers

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PN 902117.001 Rev.H
June 1989
184198A

This manual is a guide to the use of

903 and 903H MOISTURE EVOLUTION ANALYZER

Data herein has been verified and validated and is believed adequate for the intended use of the instrument. If the instrument or procedures are used for purposes over and above the capabilities specified herein, confirmation of their validity and suitabiltiy should be obtained, otherwise Du Pont does not guarantee results and assumes no obligation or liability. This publication is not a license to operate under, or a recommendation to infringe upon, any process patents.

Notes, cautions, and warnings within the text of this manual are used to emphasize important and critical instructions.

WARNING: An operating procedure, practice, etc, which, is not correctly followed, could result in personal injury.

CAUTION: An operating procedure, practice, etc, which, if not strictly observed, could result in damage of equipment.

NOTE: An operating procedure, condition, etc, which it is essential to highlight.

Health hazards precaution data. If and when hazardous chemicals adverse or health factors affect the environment or use of the equipment, appropriate precautions are provided.

WARNING

The ac receptacle must be a 3-wire grounded circuit to minimize the hazard of electrical shock. There is no on-off switch, since removal of ac would allow the ambient moisture to saturate the cell.

WARNING

The oven may be still hot when the COMPLETE lamp lights.

WARNING

Line voltage is present throughout the equipment. Only qualified personnel must troubleshoot this analyzer.

WARNING

Use normal precautions with nitric acid. Read label!

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Figure 1-1. 903 Moisture Evolution Analyzer.

Section 1. DESCRIPTION

1-1. SCOPE.

This manual provides the information necessary to install, operate, and maintain the 903 and 903H Moisture Evolution Analyzers (figure 1-1). This section provides the purpose, description, and theory of operation. A glossary of terms is included at the end of the section.

1-2. PURPOSE.

The 903 and 903 H Moisture Evolution Analyzers (moisture analyzers), quantitatively analyze moisture (H_2O) content in solid samples and display the results. The 903H analyzer is the same as the 903, except that it has a higher oven temperature capability. Refer to paragraph 1-4 for the difference in models.

1-3. PHYSICAL DESCRIPTION.

The moisture analyzer (figure 1-2) is a compact unit for bench or table-top mounting containing electronic control circuits, an electrolytic cell, and an

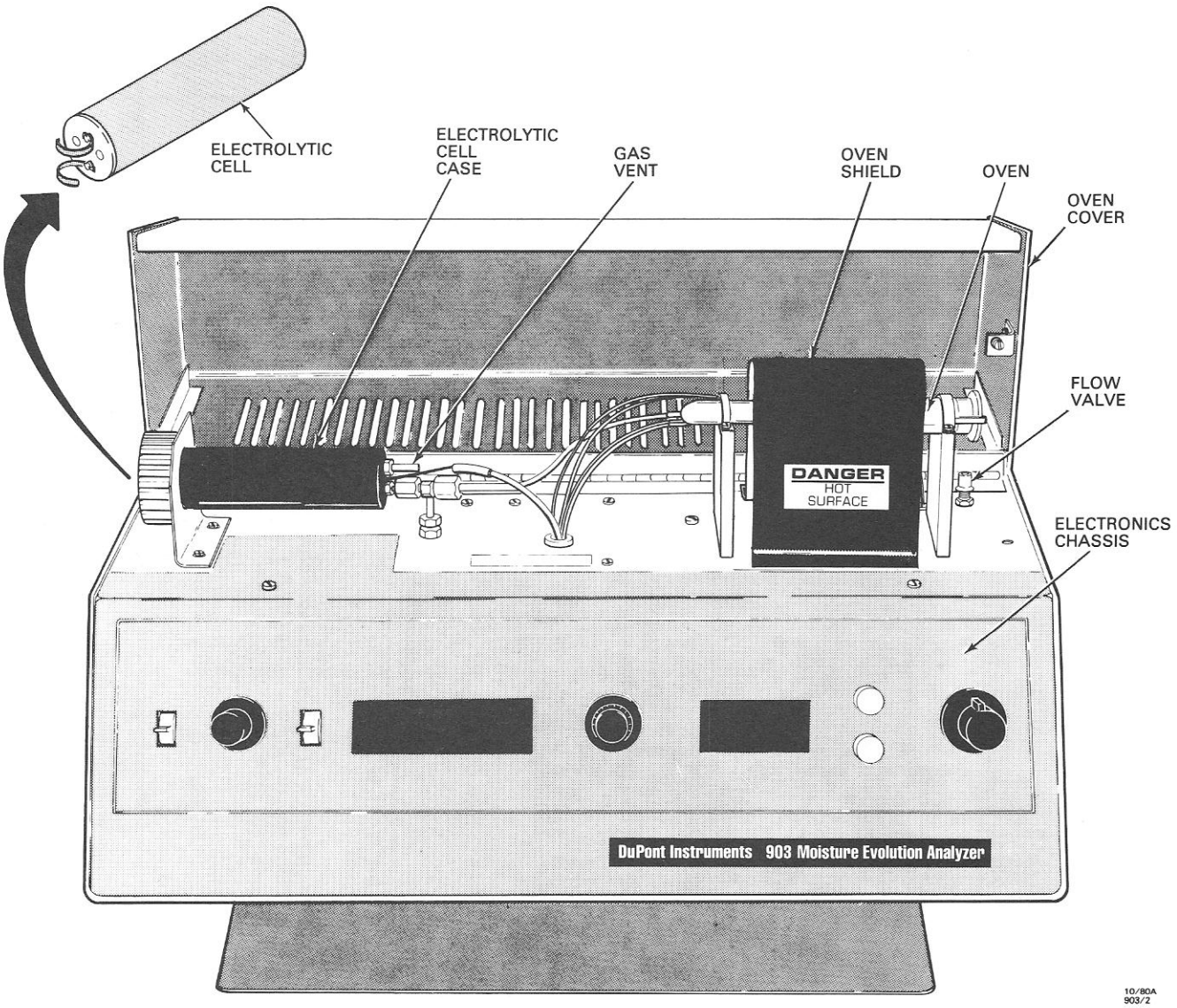


Figure 1-2. Moisture Analyzer, Oven Cover Open.

oven. A molecular sieve dryer is supplied for the carrier gas (figure 1-3). Also, a source of extra dry nitrogen gas is required for operation. The analyzer has terminal jacks for connecting a recorder or voltmeter.

1-4. DIFFERENCE IN MODELS.

The two analyzers operate and function identically. The 903H temperature capability is 1000°C while the 903 temperature capability is 300°C.

1-5. EQUIPMENT SPECIFICATIONS.

Table 1-1. Specifications

Performance	
Output:	Digital readout 0.0 to 99,999.9 μg ±0.1 0-10 mV for recorder
Sensitivity:	0.1 μg
Accuracy:	2% of reading or ±20 μg H_2O, whichever is greater.
Sample Size:	3.5 cm^3 (maximum volume of sample boat)
Timing Unit:	0 to 60 min
Maximum Oven Temperature:	Model 903 — 300°C Model 903H — 1000°C

General	
Power:	100 V ac ±10%, 50, 60 Hz, 500 V•A 120 V ac ±10%, 50, 60 Hz, 600 V•A 240 V ac ±10%, 50, 60 Hz, 600 V•A
Dimensions:	31 cm (12.2 in.) high, 48 cm (18.9 in.) wide, 24 cm (9.4 in.) deep
Weight:	Net 11 kg (24 lb)
Flow Rate (carrier gas):	70 cm^3/min ±20 at 35 kPa (5 psig)

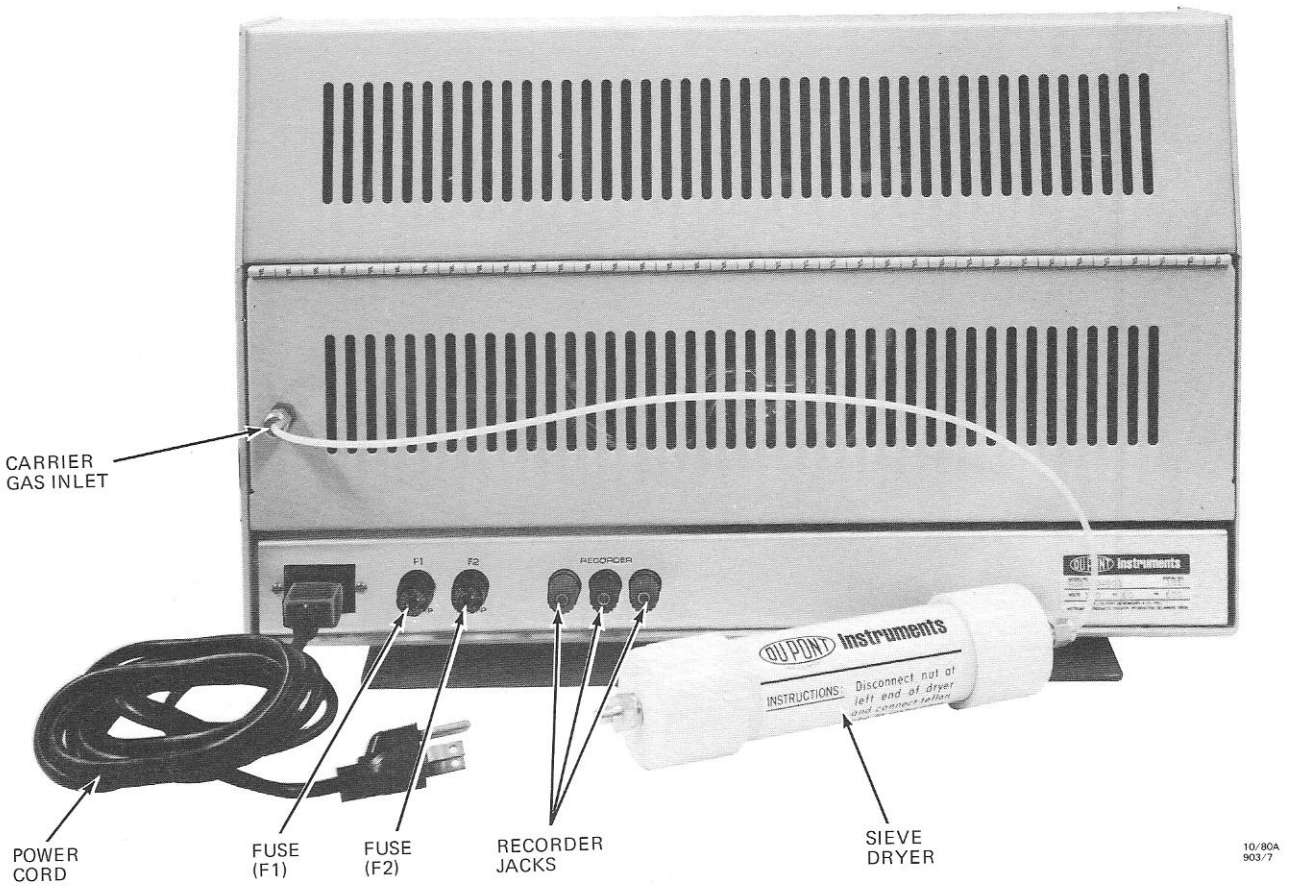


Figure 1-3. Moisture Analyzer, Rear View.

1-6. THEORY OF OPERATION.

a. General (figure 1-4).

The sample to be analyzed is weighed and placed in the oven. The oven heat vaporizes any moisture in the sample. The nitrogen carrier gas flows through the flowmeter and the dryer into the oven where it transports the moisture to the electrolytic cell. A thin film of phosphorous pentoxide (P_2O_5) deposited between two helically-wound electrodes absorbs the water. The absorbed water is electrolyzed by the current flowing between the electrodes. Electrolysis changes the water to hydrogen and oxygen gases which discharge through the vent with the carrier gas, coulometrically regenerating the phosphorous pentoxide. The charge required to completely regenerate the P_2O_5 is integrated and displayed on the front of the instrument as total moisture content in micrograms.

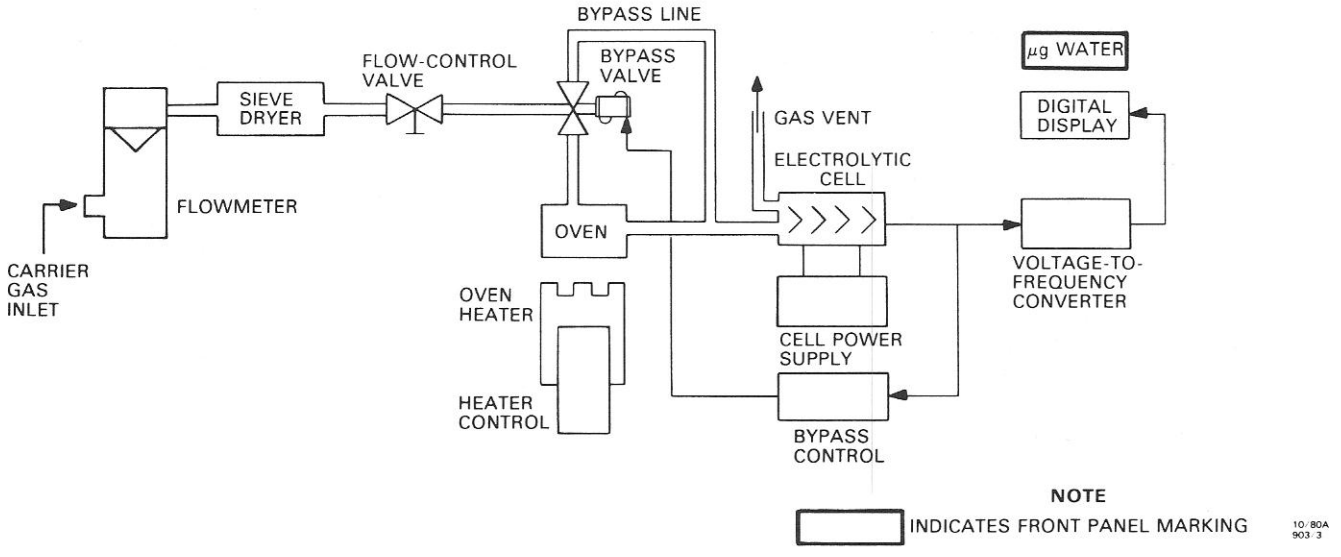
A bypass control automatically opens and closes to prevent saturation of the P_2O_5 by excessive moisture.

b. Functional Description (figure 1-5).

The oven temperature control is set to the required temperature. A section of the solid to be analyzed is weighed and quickly placed in the oven. The timer control is set to the time required for the analysis. A cam switch on the timer closes for the period of time selected and completes a circuit energizing relay K2. K2 has two sets of contacts; one set applies power to the heater and heater control circuits, the other set lights the PROCEED lamp. The oven heat vaporizes the moisture in the sample. The carrier gas flows through the dryer, regulator valve, and bypass valve into the oven where it picks up moisture and transports it into the electrolytic cell. The cell (figure 1-6) contains a U-shaped tube, with two parallel, helically-wound, platinum electrodes. A thin film of phosphorous pentoxide is deposited between the electrodes. Phosphorous pentoxide, a nonconductor when dry, is extremely hygroscopic and readily absorbs moisture from a carrier gas, transforming it to phosphoric acid, a conductor. The platinum electrodes have a 67-Volt potential on them, causing a current to flow through the cell and electrolyzing the moisture to hydrogen and oxygen. The quantity of charge (coulombs) required to electrolyze 0.1 μg of moisture is a constant which is the basis of the electronic measurement. The electrolytic current produces a voltage which is applied to an integrator circuit (ramp generator U1A). The integrator output voltage, a function of time and input voltage, is applied to comparator U2. When the integrated output reaches a preset level (10 V), the comparator produces an output (0—5 V) which triggers one-shot multivibrator U3. The integrator is adjusted to produce one pulse from the multivibrator for each 0.1 μg of moisture electrolyzed.

c. Ramp Reset.

Each positive pulse turns on ramp-reset trigger Q2. The output of Q2 turns on field-effect transistor



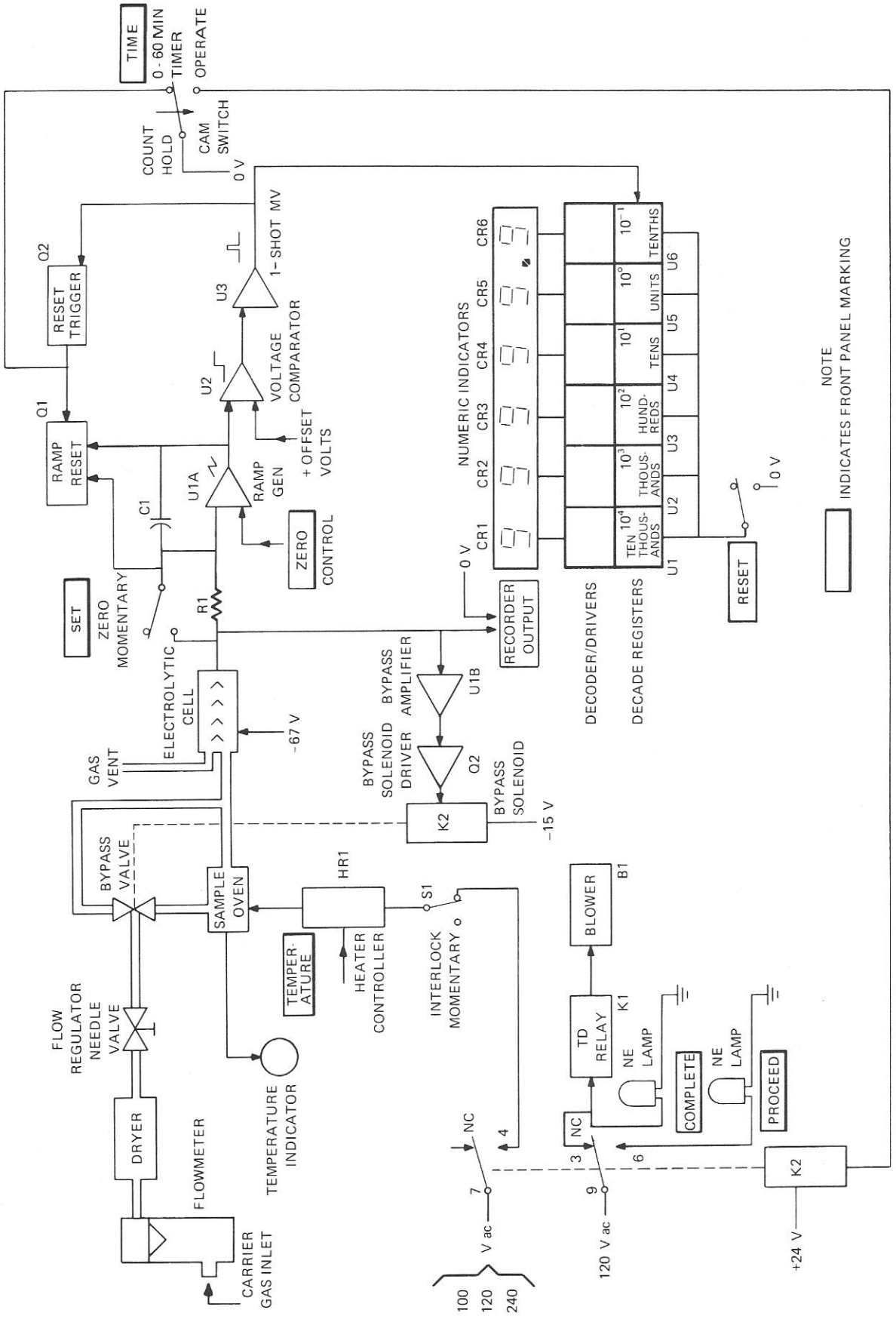


Figure 1-5. Functional Block Diagram,Moisture Analyzer.

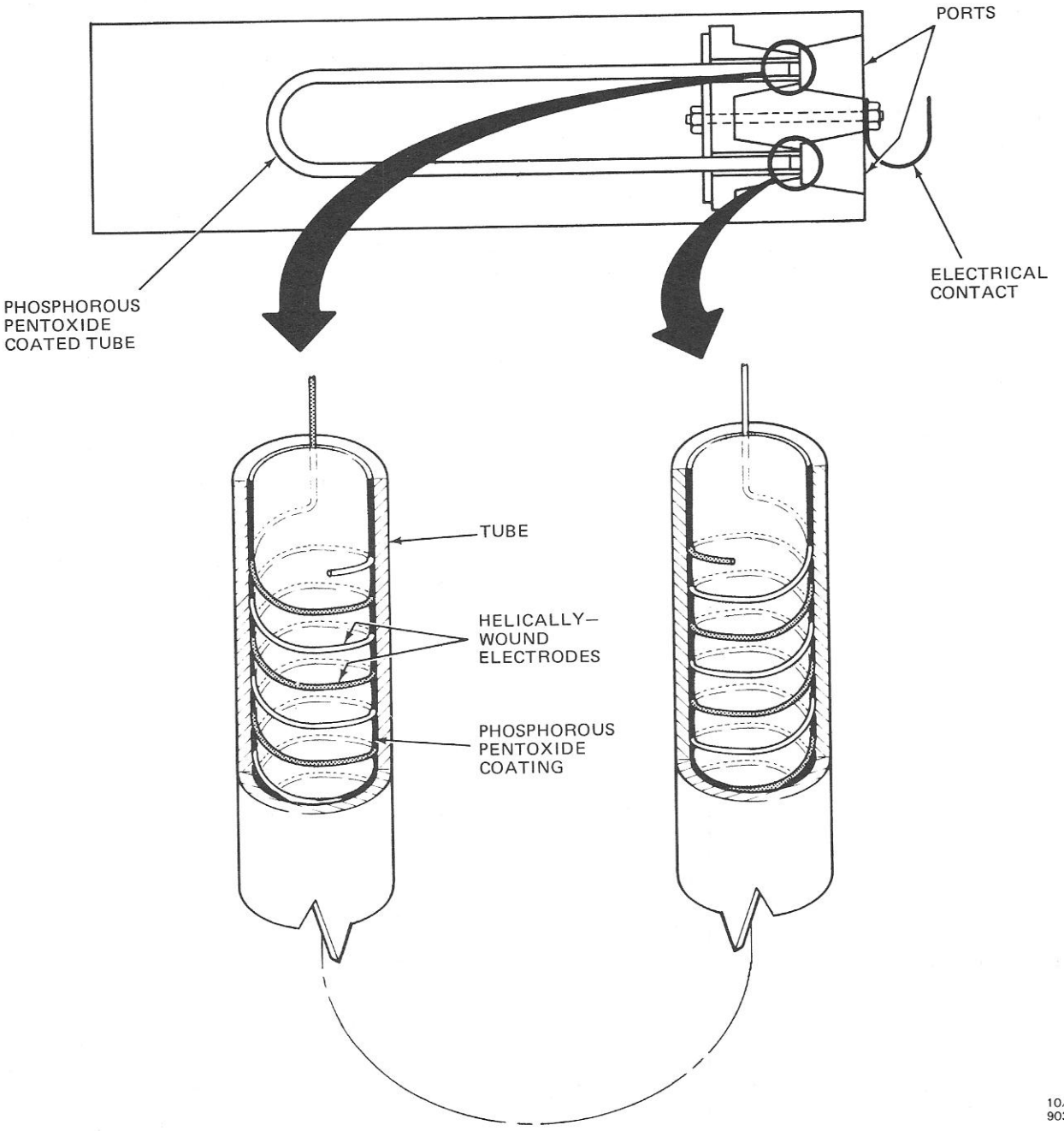


Figure 1-6. Electrolytic Cell Construction Details.

Q1 (ramp reset). Q1, when conducting, provides a low resistance discharge path for C1 to reset the ramp generator. The ramp generator is reset after each pulse of the one-shot multivibrator.

d. Decade Registers (counters).

The positive pulses from the multivibrator trigger a register consisting of six decade counters. Each pulse increments the first decade counter (tenths). After 10 pulses, a carryover triggers the second decade counter. Each succeeding counter (units, tens, hundreds, thousands, and ten thousands) receives a carryover after the preceding counter reaches maximum (9). The maximum count which can be accumulated is 99999.9. The pulse following the maximum resets the counter to 0.

e. Decoder Driver.

The decoder consists of 10 NAND gates for each decade counter. The binary number in each counter is decoded by enabling a unique arrangement of gates. The low output of the enabled gates inhibits an arrangement of eight NAND-gate drivers. Each inhibited NAND-gate driver applies a high output to a light-emitting diode (LED). These outputs are designated a, b, c, d, e, f, g, and dp. The high output forward biases the LED and it conducts (lights).

f. Numeric Indicator.

There is a numeric indicator for each decoder. The numeric indicator consists of seven LED's arranged in segments (figure 1-7). An eighth LED functions as a decimal point (dp). Each segment also is designated a, b, c, d, e, f, g, and dp corresponding to the designation of the NAND-gate drivers. A binary seven (0111) in the decade counter energizes LED a, b, and c to display the number 7. The contents of each decade register is displayed by its numeric indicator in the same manner. The digital display continues to increment as the analysis proceeds.

g. Bypass Valve.

To prevent the cell from saturating, a bypass valve (figure 1-5) energizes when the electrolyzing current approaches the cell's maximum moisture handling capacity. When the current approaches 30 mA (≈2000 μg/g), bypass amplifier U1B produces an output which energizes bypass-solenoid driver Q2 to close the oven inlet and open the bypass. With the oven inlet closed, the carrier gas cannot enter it and therefore absorbs no moisture. It flows through the cell and escapes through the vent without contributing to the moisture count. When the cell electrolyzing current decreases, the bypass amplifier turns off the solenoid driver, deenergizing the valve. The carrier gas again flows through the oven, absorbs moisture, and continues into the cell for electrolysis. This continued bypass action prevents the cell from saturating without losing any measurable amount of sample moisture. The digital count continues to accumulate and to be displayed until the timer times out and its cam contact T3 closes. T3 completes ground (0 V) to the base of Q2 (ramp reset trigger) to inhibit any further pulses to the decade register which then holds the final count. Timer contact T1 opens to deenergize relay K2. Relay contacts 7 and 4 open to remove power from the heater. Contacts 9 and 3 close to apply power to blower motor B1 through time-delay relay K1. The blower motor runs to cool the oven until K1 times out (2.5 min). With relay K2 deenergized, power is switched from the PROCEED indicator (contacts 9 and 6) to the COMPLETE indicator (contacts 9 and 3).

This completes the analysis and the total moisture content of the sample under analysis is displayed on the digital readout.

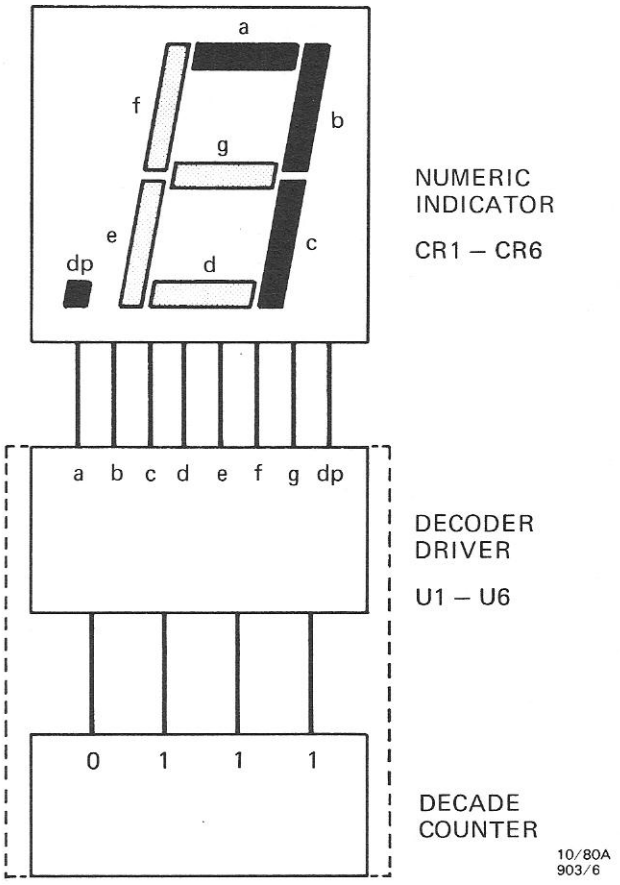


Figure 1-7. LED Segments, Display Indicator.

h. Interlock Switch.

An interlock switch opens the oven heater circuit when the oven cover is lifted.

i. Blank Count.

Five factors introduce additional moisture counts in the accumulated total.

- Carrier gas contains some moisture — less than 5 μg/g.
- Oven cap removal introduces moisture from ambient atmosphere.
- Moisture adheres to sample boat.
- ZERO control is adjusted for 6 to 10 seconds between pulses which adds 10 to 6 counts for each minute of analysis.
- Analysis continuing beyond the precise time required to vaporize the total moisture content of the sample. For example, when the time to vaporize the total moisture content of the sample is extended by 1 minute, 6 to 10 counts are added.

NOTE

The analysis is considered complete when a 1-minute reading changes the total accumulated value by less than 1%.

These factors are readily determined by placing an empty sample boat in the oven and running the same (time and temperature) analysis without the sample. The blank count is subtracted from the total count accumulated during analysis with the sample. The result is an accurate measure of the moisture content of the sample.

1-7. GLOSSARY

Saturated Cell — A condition in which the phosphorous pentoxide coating has absorbed more water than can be readily electrolyzed.

Flooded Cell — A condition of extreme wetness, which dissolves the phosphorous pentoxide coating in a cell.

System Equilibrium — Condition within the system when water is absorbed in the walls of the system at the same rate as it is given off.

Entrained Dirt Particles — Dirt carried by a sample stream.

Dry Carrier Gas — An inert gas (nitrogen) containing less than 5 μg/g water.

Section 2. INSTALLATION

2-1. GENERAL.

This section provides information necessary to inspect and install the moisture analyzer.

2-2. PRE-INSTALLATION INSPECTION.

a. Inspect the instrument for mechanical damages, scratches, dents, or other defects. Check the cushioning material for signs of severe stress. If the instrument is damaged in transit, or fails to meet specifications upon receipt, notify the carrier and the nearest Du Pont service office.

b. Open the accessories carton and check items against the following list:

Item	Quantity	Part No.
Sample Boat	5	901083-901
Dryer Assy	1	901030-901
Power Cord	1	202231
Electrolytic Cell*	2	902002
Cell Filter	10	902121-901
Tweezers	1	203636
Shredded Teflon® TFE Fluorocarbon polymer (package)	1	901025
Flowmeter	1	901032
Connectors, Swagelok®	2	203333-012
Tubing, Teflon®	1	204914
Instruction Manual	1	902117-901

*Refer to Section 5 for electrolytic cell exchange plan.

c. After unpacking and inspection, proceed as follows:

(1) Lift the oven cover as shown in figure 1-2.

(2) Remove the two screws and pull the front panel fully forward to access the interior, and lay the oven deck back (this requires removal of two additional screws) (figure 2-1).

NOTE

If necessary, unplug the cable connector shown to lay the oven deck on the bench.

(3) Check that the printed circuit boards and relays are firmly seated in their sockets.

(4) Inspect the interior for loose or broken wires and any other obvious damage.

(5) Connect the temperature indicator plug.

CAUTION

Check input transformer taps for position of spade lug connectors. The taps are marked 110, 120, and 220. The spade lug must be on the tap corresponding to the available primary power.

(6) Close the panel and secure with the four screws.

(7) Close the oven cover.

2-3. RECOMMENDED OPTIONAL EQUIPMENT.

A kit, part number 901034-901, containing an electronic calibrator (902089) and capillary micropipets (901046-901) is available for standardizing the moisture analyzer.

A recorder aids in determining optimum time and temperature setting for solids when these factors are unknown. It graphically indicates the rise and decay of the electrolytic cell current in relation to time. The optimum time for analysis can be determined from this graph. Similarly, the optimum temperature can be determined through experimentation.

2-4. REQUIRED ADDITIONAL EQUIPMENT.

To accurately analyze the moisture contents of various solids, two additional items are required:

- Source of extra-dry nitrogen with a maximum of 5 μg/g of moisture, pressure regulated at 35 kPa (5 psig).

NOTE

Prepurified grade of bottled nitrogen is acceptable.

- Analytical balance readable to 10 μg.

2-5. INSTALLATION.

WARNING

The ac receptacle must be a 3-wire grounded circuit to minimize the hazard of electrical shock. There is no on-off switch, since removal of ac would allow the ambient moisture to saturate the cell.

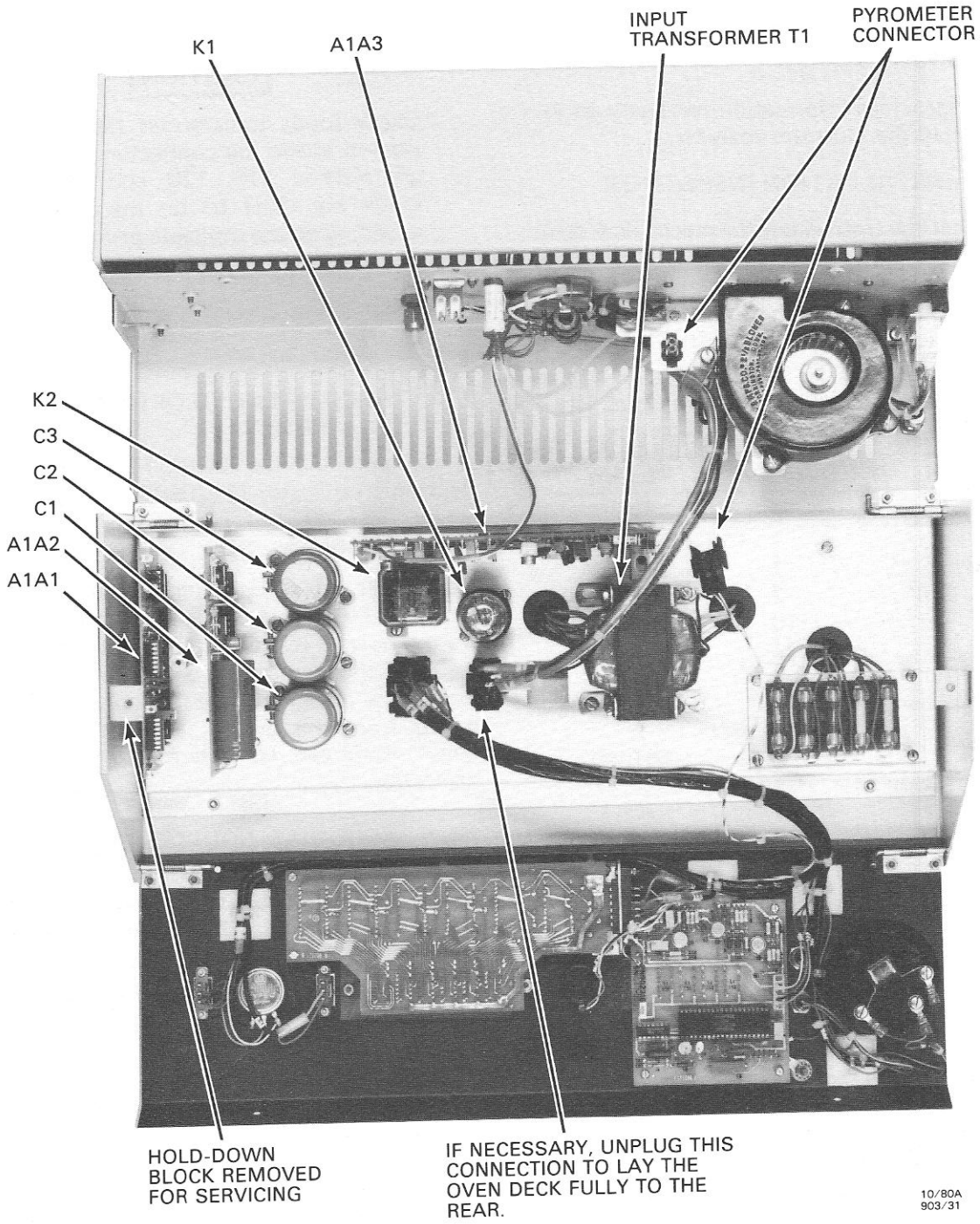


Figure 2-1. Moisture Analyzer, Interior View.

a. Install the electrolytic cell as follows:

(1) Unpack the electrolytic cell and remove its protective cap.

(2) Take a cell filter disc from the envelope and place it in the bottom port as shown in figure 2-2.

(3) Remove the cap from the cell case and carefully insert the cell into the cell case, port end first.

(4) Gently push the cell to the rear of the case while slowly turning it. When you feel the cell ports engage the intake and exhaust nozzles, continue the pressure until the ports fully engage the nozzles. The nozzles must be flush against the cell ports (figure 2-2).

NOTE

No electrical alignment (polarity) need be observed when installing the cell.

(5) Install the cap on the cell case.

b. Check the electronics as follows:

(1) Connect the power cord to the analyzer and to a receptacle of proper voltage (110, 120, or 240 Volts ac).

(2) Turn the ZERO control fully clockwise. See figure 3-1 for location of controls, if necessary.

(3) Turn the TEMPERATURE control clockwise to 1.0

NOTE

The oven cover must be down to close the heater interlock switch.

(4) Turn the TIME control beyond 10.

(5) Observe the μg WATER indicator. The digital count should start accumulating.

(6) After 3 or 4 minutes, check that the TEMPERATURE indicator reads near 100°C.

If the conditions of steps (5) and (6) are met, the electronic circuits are functional.

c. Install the dryer as follows:

(1) Install the flowmeter at the nitrogen source (figure 2-3).

(2) Remove the protective cap from the gas inlet at the rear of the analyzer.

(3) Read the instructions on the dryer and connect the reference tube to the fitting on the rear of the analyzer.

(4) Connect the dryer inlet to the flowmeter outlet using only metal or fluorocarbon tubing supplied.

(5) Adjust the pressure regulator to 35 kPa (5 psig).

(6) Remove the protective cap from the vent on the cell case.

(7) Adjust nitrogen flow to 50 to 90 cm^3/min with the flow valve (figure 1-2).

NOTE

Analyzer flow rate has been factory set at 70 cm^3/min ±20 at 35 kPa (5 psig) at the gas vent and should not be changed.

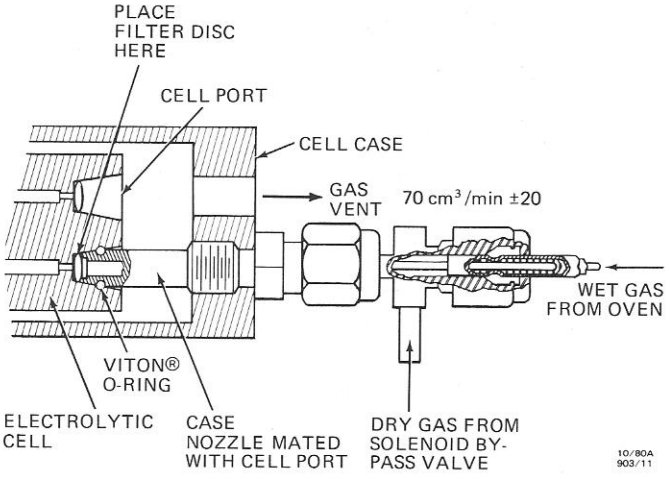


Figure 2-2. Cell Ports Mated with Nozzles and Position of Filters.

d. Contaminant Preventive.

It has been found that cell life between coatings often can be increased by putting shredded Teflon® in the oven beyond the heated zone.

CAUTION

Teflon® TFE Fluorocarbon polymer degrades above 500° C. Do not use it in the 903H unit.

Remove the oven cap and place a 13-mm (0.5-in) wad of Teflon® TFE Fluorocarbon polymer in the oven beyond the heater as shown in figure 2-4. Use caution to prevent damage to the thermocouple.

e. Moisture Purge.

(1) With the analyzer installed, check that the oven cap is in place and turn on the gas.

(2) Let the carrier gas purge the system for 10 minutes.

(3) Set the TIME to 20 minutes and TEMPERATURE controls to 0.

(4) Permit the gas to purge the system until the µg WATER indicator increments at a rate of 10 counts per minute or less. After the system has dried out, it is ready for moisture analysis (Section 3).

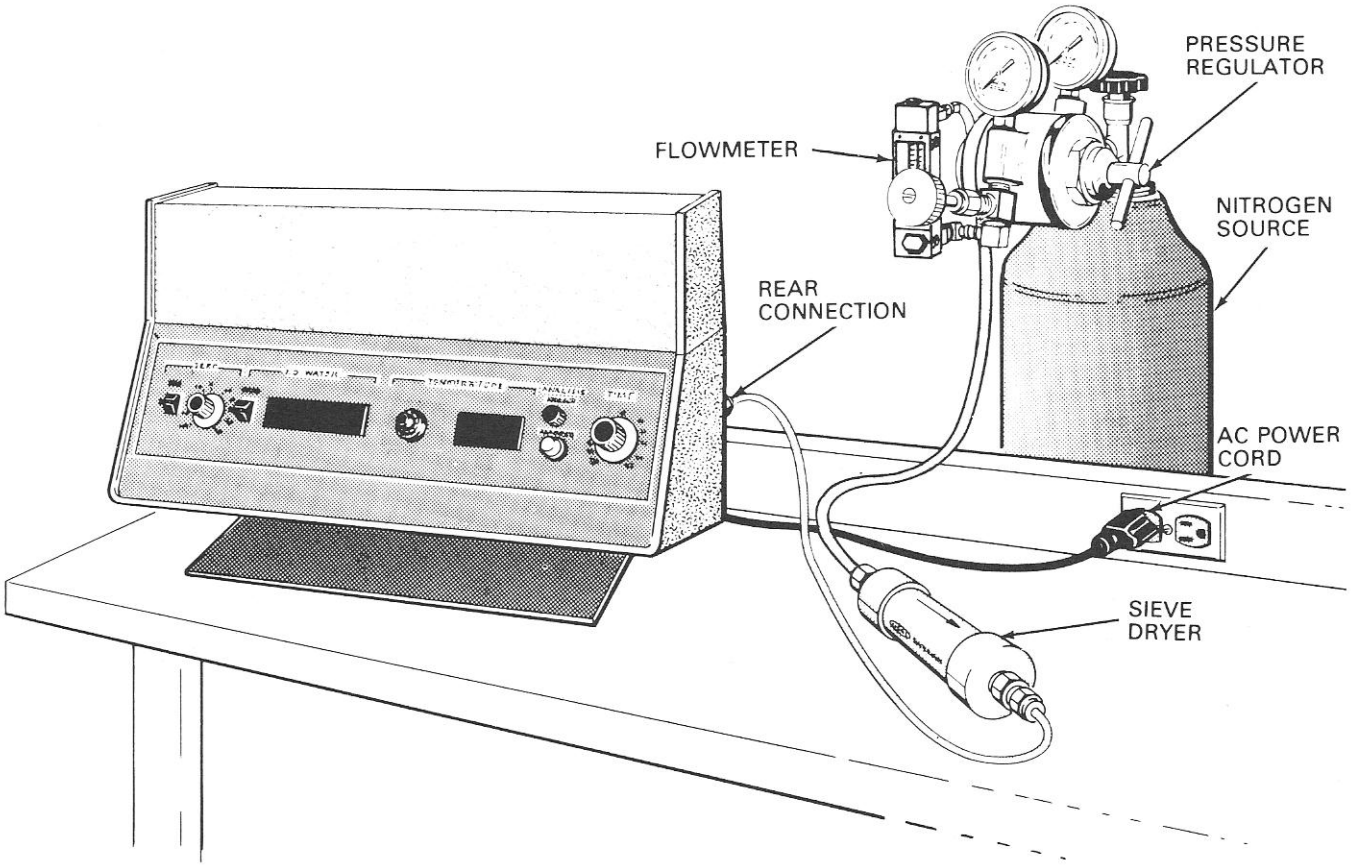


Figure 2-3. Installation Diagram.

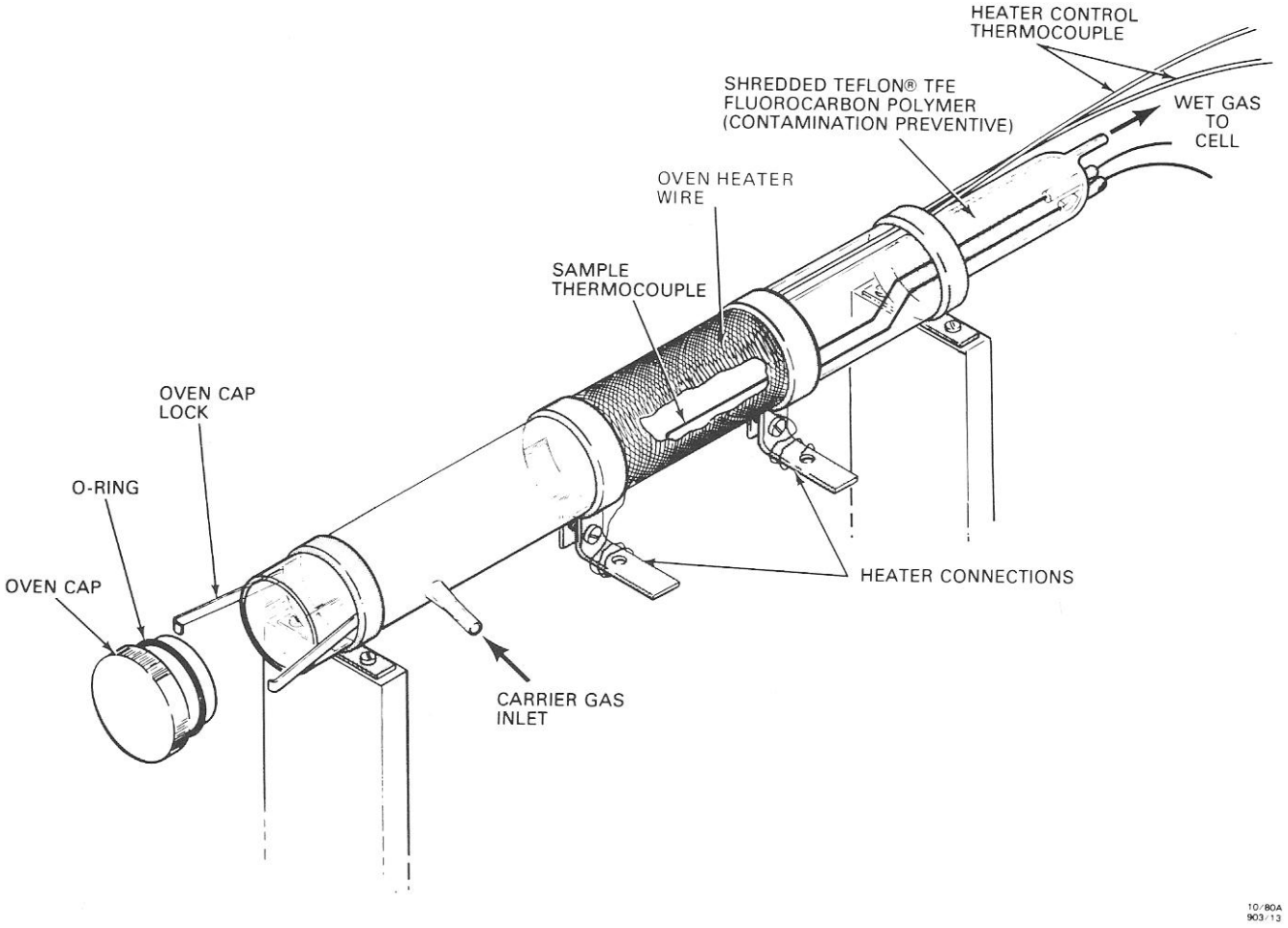


Figure 2-4. Placement of Contaminant Preventive.

Section 3. OPERATION

3-1. GENERAL.

This section lists and describes controls and indicators and gives the procedure for routine moisture analysis.

It is suggested that a time and temperature log book or record be kept of all experimental and routine analyses performed with the instrument.

Appendix D provides some application briefs which should be helpful until experimental data is accumulated. If the time and temperature requirements are unknown, they can be determined by running a trial analysis.

CAUTION

Appendix B contains a list of contaminants. Refer to the list if there is a question regarding the reaction of the solid to be analyzed with the phosphorous pentoxide. Solids that sublime must be analyzed at low temperatures to prevent their vaporization and ultimate condensation within the cell. Coating of the phosphorous pentoxide by other solids prevents absorption of the moisture from the sample under analysis and necessitates cleaning the cell.

3-2. CONTROLS AND INDICATORS.

Table 3-1 lists the controls and indicators and briefly describes their function. See figure 3-1 for their locations.

Table 3-1. Controls and Indicators.

Name	Function
TEMPERATURE control (5)	Selects oven temperature.
TEMPERATURE indicator (6)	Indicates the oven temperature in °C.
μg WATER indicator (4)	Six, 8-segment numeric readouts indicate total moisture content of the specimen in micrograms at the end of the analytical test cycle.
RESET switch (3)	When pressed, resets the μg WATER indicator to 0.
TIME control (9)	Sets the timer from 0 to 60 minutes in 5-minute increments to initiate and time moisture analysis. The timer must always be set beyond 10 to ensure that the associated switch is actuated. When the analysis time is less than 10 minutes, the control is set beyond 10 then back to the desired time.
ANALYSIS PROCEED lamp (8)	When lit, indicates that moisture analysis is in progress, i.e., timing sequence not yet complete. When the timing sequence is complete, this lamp goes out.
ANALYSIS COMPLETE lamp (7)	When lit, indicates the analysis is complete.
ZERO control (2)	Provides an offset voltage adjustment to compensate for inherent moisture. It is a coarse or fine adjustment, depending on the position of ZERO SET switch.
ZERO SET switch (1)	When pressed, changes the pulse repetition rate by a factor of approximately 8. This permits coarse zero adjustment to minimize the error count. When released, it provides a fine zero adjustment to further minimize the error count produced by the inherent moisture.

3-3. OPERATING THE MOISTURE ANALYZER.

The moisture analyzer has been calibrated and its performance checked against specific standards by Du Pont. To ensure that cell efficiency has not been lost by previous samples, check calibration at least once a shift or more often if unknown samples are being run or if you suspect something is wrong with the cell by one of the standard procedures in paragraph 4-3.

The moisture analyzer must be installed and prepared for operation as described in Section 2. Since delay in loading the sample can affect the analysis, read and understand the instructions completely before starting. Prior to moisture analysis, purge the analyzer as follows:

a. Turn the TIME and TEMPERATURE controls to 0.

b. Make sure that the oven is empty and oven cap is in place.

c. Adjust the carrier gas to 50 to 90 cm^3/min at 35 kPa (5 psig).

d. Allow the carrier gas to flow 5 to 10 minutes.

e. Adjust electronics to compensate for inherent moisture as follows:

(1) Set TIME control beyond 10 to start a counting sequence.

(2) Hold the ZERO SET switch down and adjust the ZERO control for an interval of 1 second between pulses (coarse adjustment).

(3) Release the ZERO SET switch and adjust the ZERO control for an interval of 6 to 10 seconds between pulses (fine adjustment).

(4) If it is difficult to adjust the pulse count to 1 per 6 to 10 seconds, allow the carrier gas to

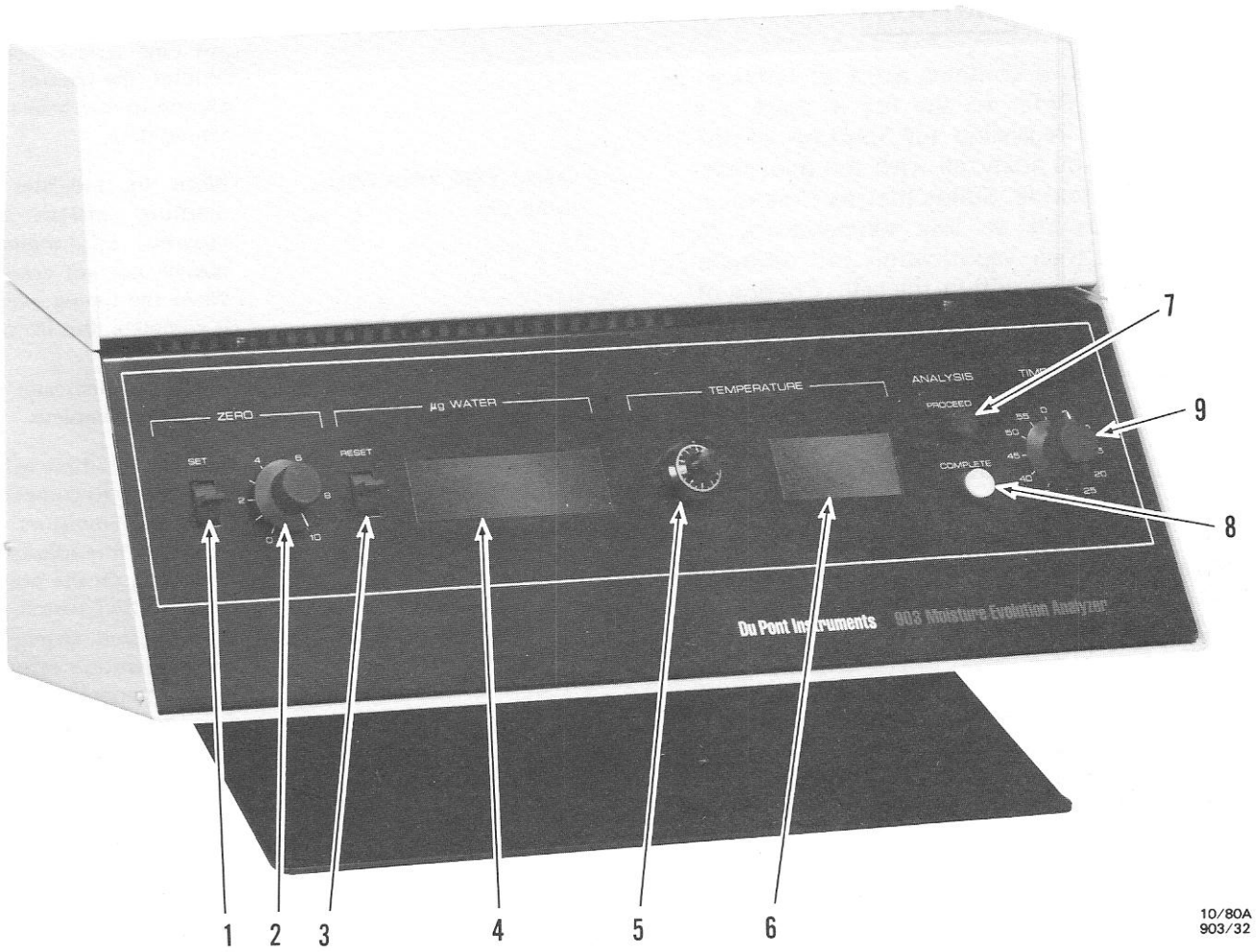


Figure 3-1. Location of Controls and Indicators.

flow for an additional 5 to 10 minutes and repeat step (2) as required until count is 1 per 6 to 10 seconds.

f. Determine the blank count as follows:

(1) Remove the oven cap and use tweezers to place the empty sample boat in the oven (figure 3-2). Install the oven cap immediately.

(2) Adjust the TEMPERATURE control as required for sample analysis.

(3) Press the RESET switch to reset the μg WATER indicator to 0.

(4) Adjust the TIME control as required for sample analysis.

(5) Wait until the timing sequence is complete (COMPLETE lamp lights) and record the numbers from the μg WATER indicator (blank count).

g. Prepare the sample for analysis as follows:

(1) Select and accurately weigh the sample on the analytical balance. Place the sample in the boat.

NOTE

If some knowledge of the expected moisture level is available, select a sample size to yield approximately 2 μg of moisture.

(2) Remove the oven cap and place the sample boat in the oven with the tweezers (figure 3-2).

(3) Install oven cap immediately.

h. Adjust the TEMPERATURE control as required. If the required temperature is unknown, determine it through experimentation.

NOTE

Many materials may be analyzed at 110°C.

i. Adjust the TIME control as required. If the required time is unknown, determine it through experimentation.

NOTE

The analysis is complete when counts obtained in one minute increase the total count by less than 1%.

j. When the time sequence is complete and the COMPLETE lamp lights, read and record the moisture content directly from the μg WATER indicator. The COMPLETE lamp indicates the analyzer is ready for another analysis.

WARNING

The oven may be still hot when the COMPLETE lamp lights.

k. Determine the correct total moisture content of the sample by subtracting the blank count (step f) from the accumulated count (step j). The percentage of moisture of the solid sample equals total moisture content (μg) times K divided by the weight of the sample (mg).

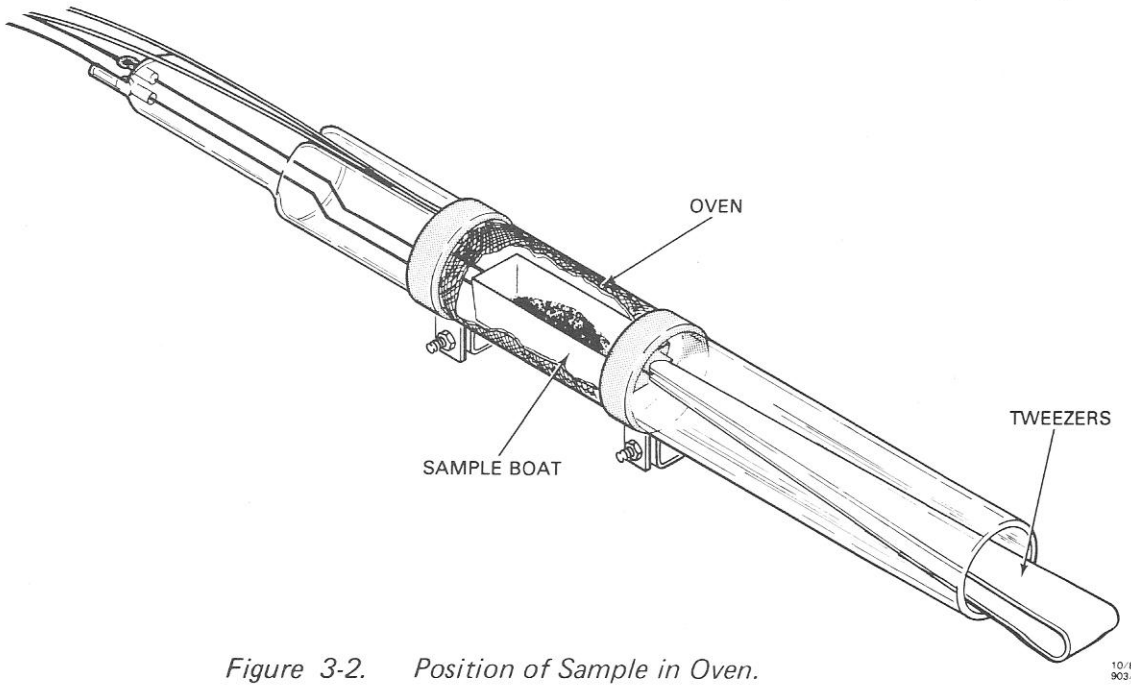


Figure 3-2. Position of Sample in Oven.

$$\% \text{ Moisture} = \frac{K(C \text{ sample} - C \text{ blank})}{\text{Wt. sample (mg)}}$$

and

$$K = \frac{F \times \text{Wt. std.} \times 100}{(C \text{ std.} - C \text{ blank})}$$

Where:

C Sample = count obtained for sample

C blank = count obtained for blank

C std. = count obtained for standard material

Wt. sample = weight of sample in milligrams

Wt. std. = weight of standard material in milligrams

F = fraction of standard material attributable to water.

K = calibration factor in milligrams per count (should be near 0.1).

l. Remove the cap and boat from the oven and install the cap.

m. Leave the carrier gas running and the analyzer plugged in to keep the cell dry so that the analyzer is always ready for use.

Section 4. MAINTENANCE

4-1. GENERAL.

This section contains maintenance, troubleshooting, and repair data for the moisture analyzer. The scheduled maintenance consists of routine cleaning while unscheduled maintenance includes cleaning, troubleshooting, adjustments, and parts replacement.

The troubleshooting chart provides an approach to locating a faulty or deteriorating component. Repair, replacement, or adjustment procedures are given to replace or correct the faulty component. Standardization and calibration procedures are given to maintain the analyzer in optimum working condition.

4-2. SCHEDULED MAINTENANCE.

a. Daily.

(1) Cleaning. Clean the analyzer by wiping with a moist, lint-free cloth. Inspect and, when required, vacuum accumulated dust from inside the analyzer and remove any deposits using a cloth moistened with a suitable solvent.

NOTE

A suitable solvent is dependent upon the material deposit. Discretion must be used in its selection.

(2) Standardization Test. Refer to paragraph 4-3 and perform one of the standardization tests.

b. Weekly.

Cleaning. Inspect the oven for spills and remove them with a swab and suitable solvent. Take care not to damage the thermocouple when swabbing the oven.

4-3. STANDARDIZATION.

Run a standardization test at least once each shift using one of the following methods and calculate the calibration factor using the formula:

$$K = \frac{F \times W_{std} \times 100}{C_{std} - C_{blank}}$$

where: K = Calibration factor in μg % water per count (should be near 0.1)

F = Fraction of standard material attributable to water

- Method a: 1.000
- Method b: 0.1566
- Method c: 0.1092

W_{std} = Standard material weight in mg

C_{std} = Count obtained for standard material

C_{blank} = Count obtained for blank

If K is within 10% of 0.1, use the factor in your final calculations. If K differs by more than 10% of 0.1, calibrate the instrument as directed in paragraph 4-9.

a. Capillary Dip Method.

The capillary dip method uses a glass capillary (micropipet) to measure a standard quantity of water by weight. Proceed as follows:

(1) Prepare the analyzer for use (paragraph 3-3).

(2) Adjust the TEMPERATURE control to 150°C.

(3) Fill the micropipet to contain 2 mg by holding it at midpoint with a pair of tweezers and dipping it lightly in distilled water.

(4) Remove the moisture from the side of the pipet with absorbent tissue. Take care not to touch the end of the pipet with tissue because it will absorb the moisture from the capillary.

NOTE

If there is difficulty in filling the micropipet, tap the tweezers while holding it in the water. If this does not help, clean the micropipet with chromic acid solution. Rinse thoroughly.

(5) After filling the pipet, place it in the sample boat and set the boat in the oven.

(6) Set the TIME control to 20 minutes and run the analysis. Record the readout.

(7) Remove the boat and run another analysis to determine the blank count.

b. Sodium Tartrate Method.

This method uses reagent grade sodium tartrate dihydrate ($Na_2C_4O_6 \cdot 2H_2O$) as the standard. Sodium tartrate's theoretical water content is 15.66% by weight which is the basis of standardization.

Proceed as follows:

(1) Prepare the analyzer for use (paragraph 3-3).

(2) Adjust the TEMPERATURE control to 150°C.

CAUTION

Since the organic portion of sodium tartrate begins to decompose at higher temperatures and may deteriorate the cell, do not exceed 220°C.

(3) Weigh 8 to 10 mg of sodium tartrate and place it in the sample boat. Accurately record the weight.

(4) Place the sample boat and contents in the oven.

(5) Adjust the TIME control to 20 minutes and run the analysis. Record the readout.

(6) Remove the boat and run another analysis to determine blank count.

c. Sodium Tungstate Dihydrate Method

This method uses reagent grade sodium tungstate dihydrate ($Na_2WO_4 \cdot 2H_2O$) as the standard. Sodium tungstate dihydrate's theoretical water content is 10.92% by weight, which is the basis of standardization.

Proceed as directed for the sodium tartrate method in step b. Sample quantity and analyzer settings are the same.

4-4. UNSCHEDULED MAINTENANCE.

4-5. TROUBLESHOOTING.

WARNING

Line voltage is present throughout the equipment. Only qualified personnel must troubleshoot this analyzer.

There are two separate systems involved in the moisture analyzer: the electronic and the gas carrier. Incorrect results could be caused by either system.

To determine which system has failed, remove the electrolytic cell and use controlled current from the optional calibrator for calibration. If the analyzer can be electronically calibrated (paragraph 4-9) without the cell, then the gas carrier system, including the cell, is suspect. If the analyzer cannot be calibrated, then the electronic circuits are suspect. To troubleshoot these circuits, refer to table 4-1.

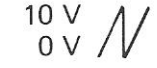
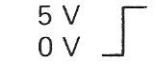
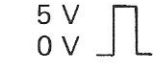
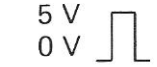
Table 4-1. Troubleshooting Chart

Indication	Possible Cause	Procedure
a. Moisture reading high.	1. ZERO not properly adjusted.	1. Refer to paragraph 3-3 e and adjust.
	2. Excessive moisture in the system.	2. Refer to paragraph 2-5 e and purge the system.
	3. Analyzer out of calibration.	3. Refer to paragraph 4-3 and run a standardization test. Calibrate if necessary.
	4. Faulty carrier or electronic system.	4. Perform calibration in paragraph 4-9 to determine which system is at fault.

Table 4-1. Troubleshooting Chart (continued)

Indication	Possible Cause	Procedure
		NOTE If system can be calibrated with cell removed, then the carrier system including the cell is suspected. If it cannot be calibrated with the cell removed, then the electronics is suspected. If the carrier system is suspected, proceed to step 5. If the electronics is suspected, proceed to step c 3.
	5. Shorted cell.	5. Measure voltage and resistance across the cell. The dry voltage and resistance should be -67 volts dc ±5 and 400 MΩ ±100. Refer to paragraph 4-7 for circuit repair.
	6. Saturated sieve dryer.	6. Refer to paragraph 2-5 c and replace dryer.
	7. Faulty bypass valve.	7. Check bypass solenoid, driver, and amplifier and replace as necessary.
b. Moisture reading low.	1. Dirty, contaminated, or washed out cell.	1. Refer to paragraph 4-6 and 4-7 and clean, flush, and/or recoat cell as required.
	2. ZERO not properly adjusted.	2. Refer to paragraph 3-3 e and adjust.
	3. Analyzer out of calibration.	3. Refer to paragraph 4-3 and run a standardization test. Calibrate if necessary.
		NOTE If analyzer can be calibrated, proceed to step 4. If analyzer cannot be calibrated, proceed to step c and troubleshoot the electronics.
	4. Faulty carrier or electronic system.	4. Perform the calibration procedure in paragraph 4-9 to determine which system is at fault.
	5. Faulty electronic circuits.	5. Refer to step c 3 and troubleshoot the electronic circuits.
c. No digital output plus the following indications:		

Table 4-1. Troubleshooting Chart (continued)

Indication	Possible Cause	Procedure
1. Numeric readout does not increase as it normally does with no sample under analysis.	1. Unplugged power cord, blown fuse, or faulty power supply.	1. Check power cord, fuse, or refer to test points listed below and measure the power supply voltages. See figure 4-1 for test point locations. Location (assembly) / Test Point TP- / Reading (Vdc) A1A2 / 1 / -67 A1A2 / 2 / 0 A1A2 / 3 / + 5 A1A2 / 4 / -15 A1A2 / 5 / +15
2. Oven does not heat and PROCEED lamp is not on.	2. Faulty relay K2 or unregulated 24-volt supply.	2. Measure the 24-volt supply between C1 (+) and ground. If voltage is present, check relay K2.
3. Oven heats and PROCEED lamp is on.	3. Faulty V/F converter board.	3. Use an oscilloscope and check for the waveform shown below at the test points indicated. See figure 4-1 for test point location. Location (assembly) / Test Point TP- / Waveform A1A1 / 1 / 10 V, 0 V  A1A1 / 2 / 5 V, 0 V  A1A1 / 3 / 5 V, 0 V  A1A1 / 4 / 5 V, 0 V 
d. No digital display although waveform data is correct.	1. Faulty LED display. 2. Faulty drivers.	1. Replace the LED display. 2. Replace drivers.
e. Temperature indication low; i.e., control setting gives low temperature reading.	1. Thermocouple junction not touching oven wall. 2. Resistor R2 misadjusted.	1. Move thermocouple junction until it is against oven wall. 2. Adjust R2 (figure 4-2).

Either system would indicate failure if its associated power supply failed. The troubleshooting procedures list power supply test points and voltages. Figure 4-1 shows the location of test points. Since it is relatively simple, first check the fuse and make certain the power cord is plugged in. Measure power supply voltages at the test points given unless the symptoms indicate other than a faulty power

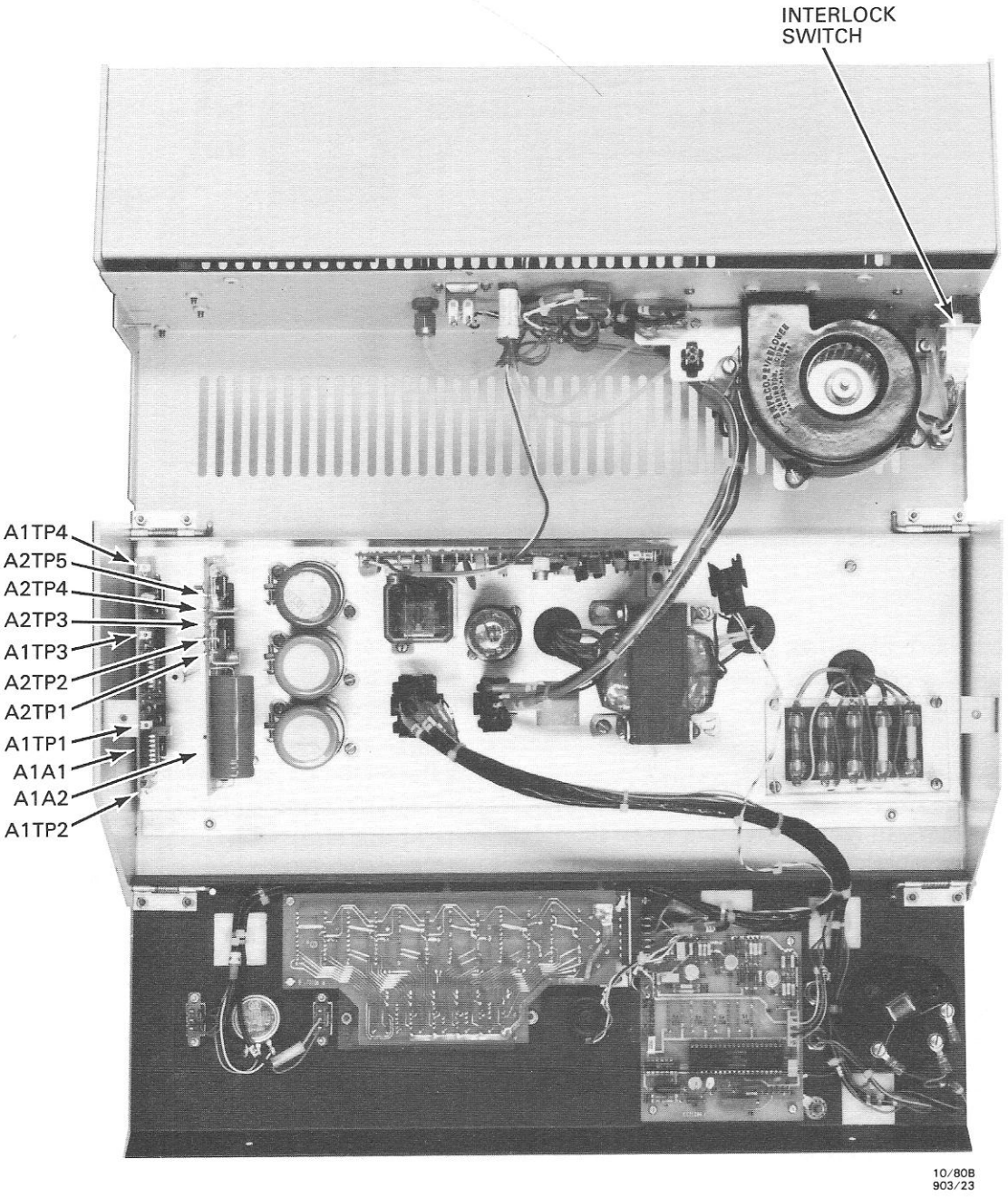


Figure 4-1. Test Point Locations.

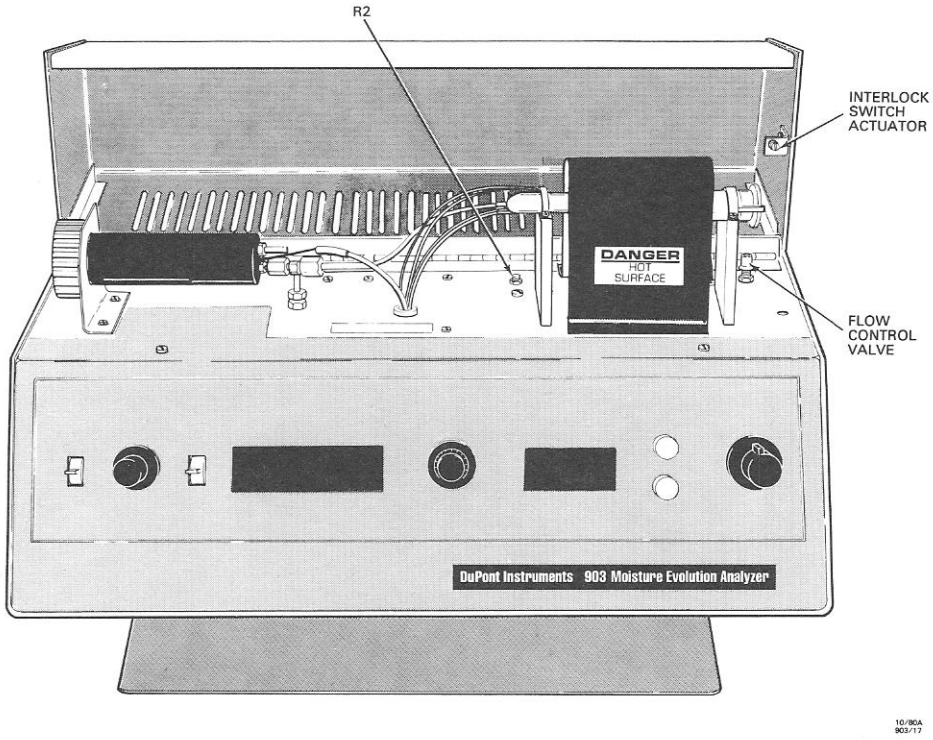


Figure 4-2. Resistor R2 Location.

supply. Figure 5-1 identifies the major units for troubleshooting. A schematic diagram (figure 4-6) is included for diagnostic purposes.

4-6. CLEANING AFTER CONTAMINATION.

The carrier system will require cleaning when there has been an accidental introduction of a contaminant or when a solid under analysis has sublimed in the cell. If this occurs, disassemble the carrier system and clean with a solvent that will not attack glass, brass, Teflon® TFE Fluorocarbon polymer or Viton® Fluoroelastomer. After cleaning, thoroughly flush parts with acetone and dry with carrier gas. Replace the shredded Teflon® as directed in paragraph 2-5. Clean the cell as follows:

CAUTION

Acetone will attack the cell case. Remove any parts from case before using acetone to clean them.

NOTE

Cleaning and flushing the cell will render it inactive and it must be recoated.

a. Place two 0.64-mm (0.25-in.) O-rings on a syringe as shown in figure 4-3, or preferably use an aspirator to suck the liquid through the cell. Do not force the end of the syringe against the bottom of the port opening.

b. Use the syringe or, preferably, the aspirator and flush the cell with distilled water.

c. Flush with acetone, alcohol, or other solvent as required.

d. Flush solvent from cell with distilled water and give it a final flush with acetone.

e. Suck the remaining moisture from the cell with dry air and install the cell in its case.

f. Turn on carrier gas and check the cell before recoating.

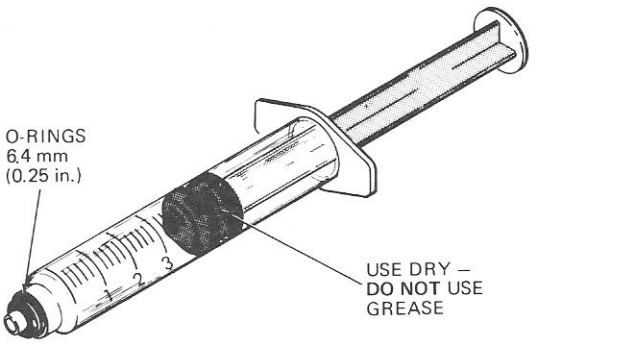


Figure 4-3. Syringe for use in Cell Flushing.

g. Adjust the ZERO control for 1 pulse per 6 to 10 seconds.

NOTE

If it is difficult to adjust for 1 pulse per 6 to 10 seconds or if the pulse rate increases after adjustment, there is a leak or a short.

h. If a leak or a short exists, repeat steps b thru g. If a second flushing does not clear the leak or short, repair the circuit as directed in paragraph 4-7.

i. If there is no leak or short, recoat the cell as directed in paragraph 4-8.

4-7. SHORT CIRCUIT REPAIR.

If it is suspected that the cell is shorted, repair it by one or both of the following procedures:

a. Using a Tesla Coil (figure 4-4).

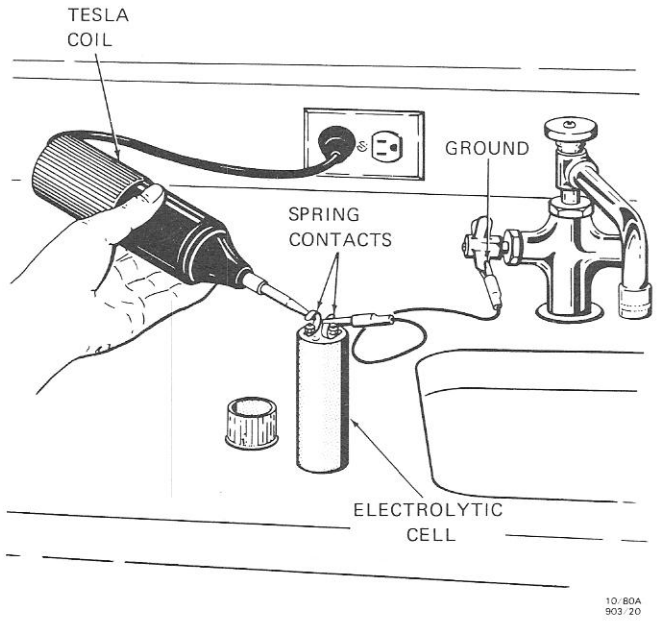


Figure 4-4. Use of Tesla Coil to Remove Short.

(1) Connect one cell terminal to an electrical ground such as a water pipe or an electrical conduit box.

(2) Connect the power cord of a Tesla coil into an ac outlet.

(3) Adjust the Tesla coil to produce a 25 to 40-mm (1 to 1.5-in.) arc when its probe is held close to the electrical ground.

(4) Touch the probe to the ungrounded cell terminal while looking into the port opening. A visible arc indicates that the short has probably been removed.

(5) Measure the cell resistance to see if the short is removed (400 MΩ ±100, dry).

(6) If the short remains, repeat step (4) several times.

(7) If the high voltage does not remove the short, proceed to step b.

b. Using Concentrated Nitric Acid.

WARNING

Use normal precautions with nitric acid. Read label!

(1) Fill the cell with concentrated nitric acid and let it stand 30 minutes.

(2) Drain the acid, flush several times with distilled water followed by acetone (paragraph 4-6), and dry.

(3) Measure cell resistance to see if the short is removed.

(4) After the short is removed, recoat the cell as directed in paragraph 4-8.

4-8. CELL RECOATING.

The cell may be recoated locally or it may be exchanged (paragraph 5-2). Although the recoating process is simple, the procedure must be followed precisely and under laboratory conditions. Cleanliness is extremely important. The solvents and solutions used must be reagent grade and be free of any suspended particulate matter. Any accessories used in the coating process must be clean and free from contamination. Recoat the cell as follows:

a. Clean the cell as directed in paragraph 4-6.

b. Prepare a coating solution. Mix two parts of concentrated reagent grade phosphoric acid (85% H_3PO_4 in water) and eight parts acetone by volume.

c. Use the syringe (figure 4-3) and fill the cell with the coating solution.

d. Withdraw the coating solution and, with vacuum applied to one port, gently dry the cell for several minutes. Return the cell to its case.

NOTE

Disconnect the ac power cord to prevent applying voltage to the cell until it has dried down (20 to 30 min).

e. Purge the cell with dry carrier gas for 20 to 30 minutes.

f. Stabilize the new coating by operating the moisture analyzer for a few hours.

g. Run a standardization test as directed in paragraph 4-3 and calibrate, if necessary.

4-9. CALIBRATION.

Calibration consists of adjusting the voltage-to-frequency circuit for 1000 pulses (counts) per minute with the equivalent of a 17.83-mA electrolytic current. This procedure uses the recommended optional electronic calibrator. An alternate calibration procedure is given in paragraph b.

a. Calibrate with the electronic calibrator as follows:

(1) Prepare the analyzer for operation as directed in paragraph 3-3.

(2) Remove the electrolytic cell.

(3) Press the RESET switch to set the μg WATER indicator to 0.

(4) Adjust the TIME control to an arbitrary setting above 10 to start a digital readout.

(5) Adjust the ZERO control for 10 counts per minute, 6 seconds between pulses, using a stopwatch.

(6) Place the electronic calibrator in the cell case and install the cap. The calibrator completes the cell circuit and a 17.85-mA current will flow.

(7) Reset the TIME control to provide sufficient time for the timing count.

(8) Press the RESET switch to reset the μg WATER indicator to 0.

(9) Start the stopwatch immediately at the 100th pulse (10.0 μg) and stop it at the 1110th pulse (111.0 μg).

NOTE

The count of 1110 actually is a count of 1000 since timing started at 100 plus a no-current count of 10 per minute. Properly calibrated, there should be 1000 pulses per minute for a 17.85-mA cell current.

(10) If the count is not 1000 per minute, adjust R3 (figure 5-3). Repeat steps (7) thru (10) after adjusting R3. Turn R3 clockwise to increase the count.

b. Calibrate without the electronic calibrator as follows:

(1) Prepare moisture analyzer for operation as directed in paragraph 3-3.

(2) Remove the electrolytic cell.

(3) Press the RESET switch to set the μg WATER indicator to 0.

(4) Adjust the TIME control to an arbitrary setting above 10 to begin a digital readout.

(5) Adjust the ZERO control for 10 seconds between counts.

(6) Connect either of the circuits shown in figure 4-5.

(7) Adjust the 2-kΩ resistor for 25 mA ±0.05 (figure 4-5 a) or for 250 mV ±0.2 (figure 4-5 b).

NOTE

Regardless of which circuit is used, you are adjusting the cell current for the equivalent of 25 mA ±0.05.

(8) Reset the TIME control to provide sufficient time for timing count.

(9) Press the RESET switch to reset the μg WATER indicator to 0.

(10) Start timing using a stopwatch at any convenient count but note the starting count.

(11) Adjust calibrate potentiometer R3 (figure 5-3) for a total count of 2346 ±5 (234.6 μg ±0.5) in 100 seconds or a count of 10 000 ±22 (1000.0 μg ±22) in 426.3 seconds. Turn R3 clockwise to increase the count.

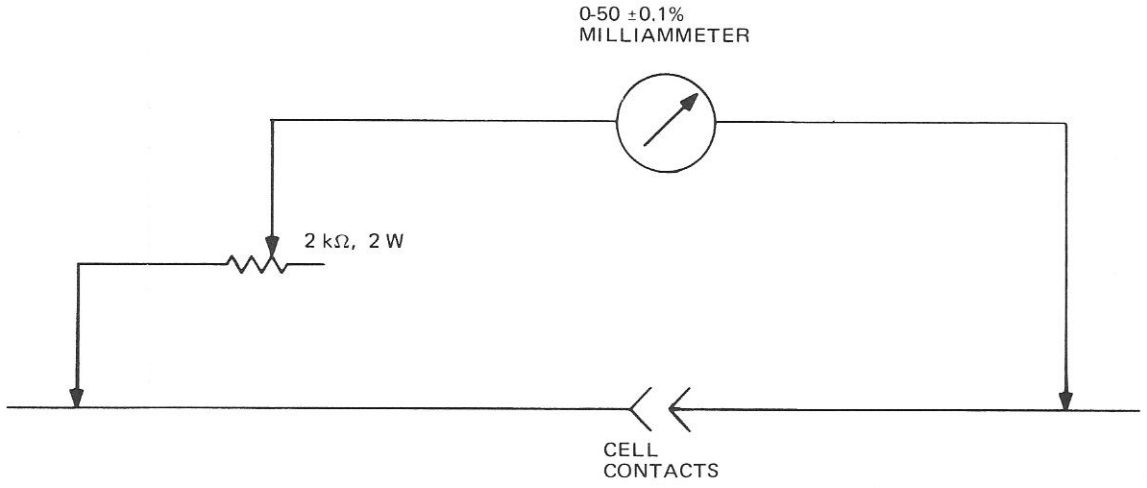
NOTE

In step (5), the no-current adjustment was for 10 counts in 100 seconds. Determine the total count by multiplying this count (10) times the number of 100-second increments of the timing period (423.6 seconds equals 4.236 increments x 10 = 42.36) and subtracting this number (42.36) from the indicated number.

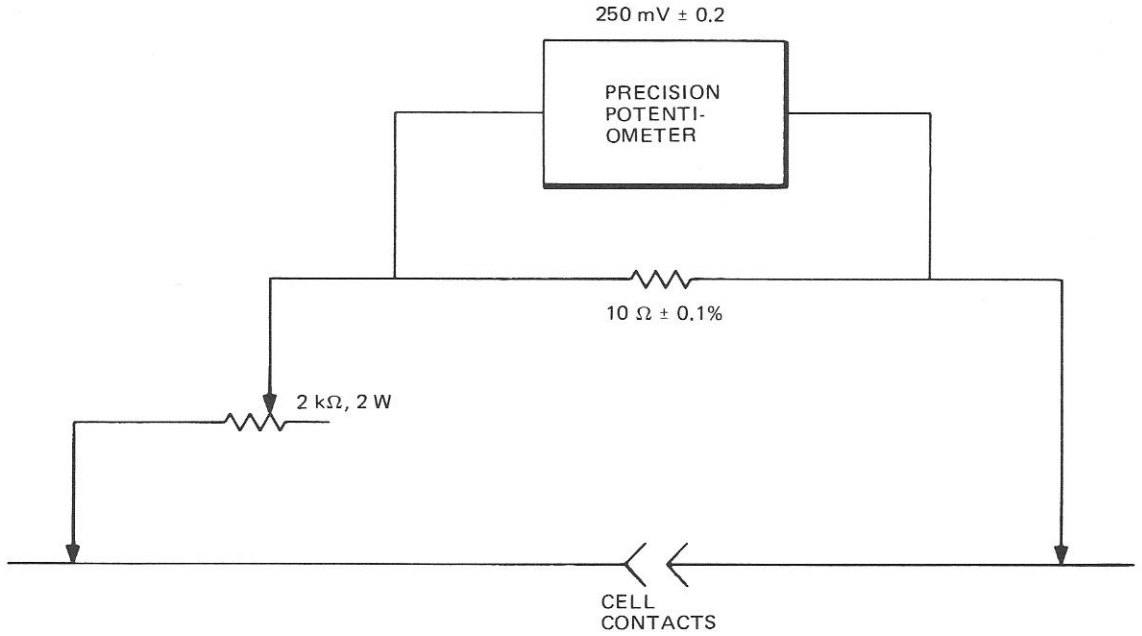
In addition, subtract the starting point from the total: e.g.,

1142.3	indicated μg
- 42.3	total no-current count
-100.0	starting point
1000.0	total count

The starting point should be the smallest possible number to minimize the no-current count.



a. CURRENT METHOD



b. VOLTAGE METHOD

10/80A
903/21

Figure 4-5. Calibration Connections.

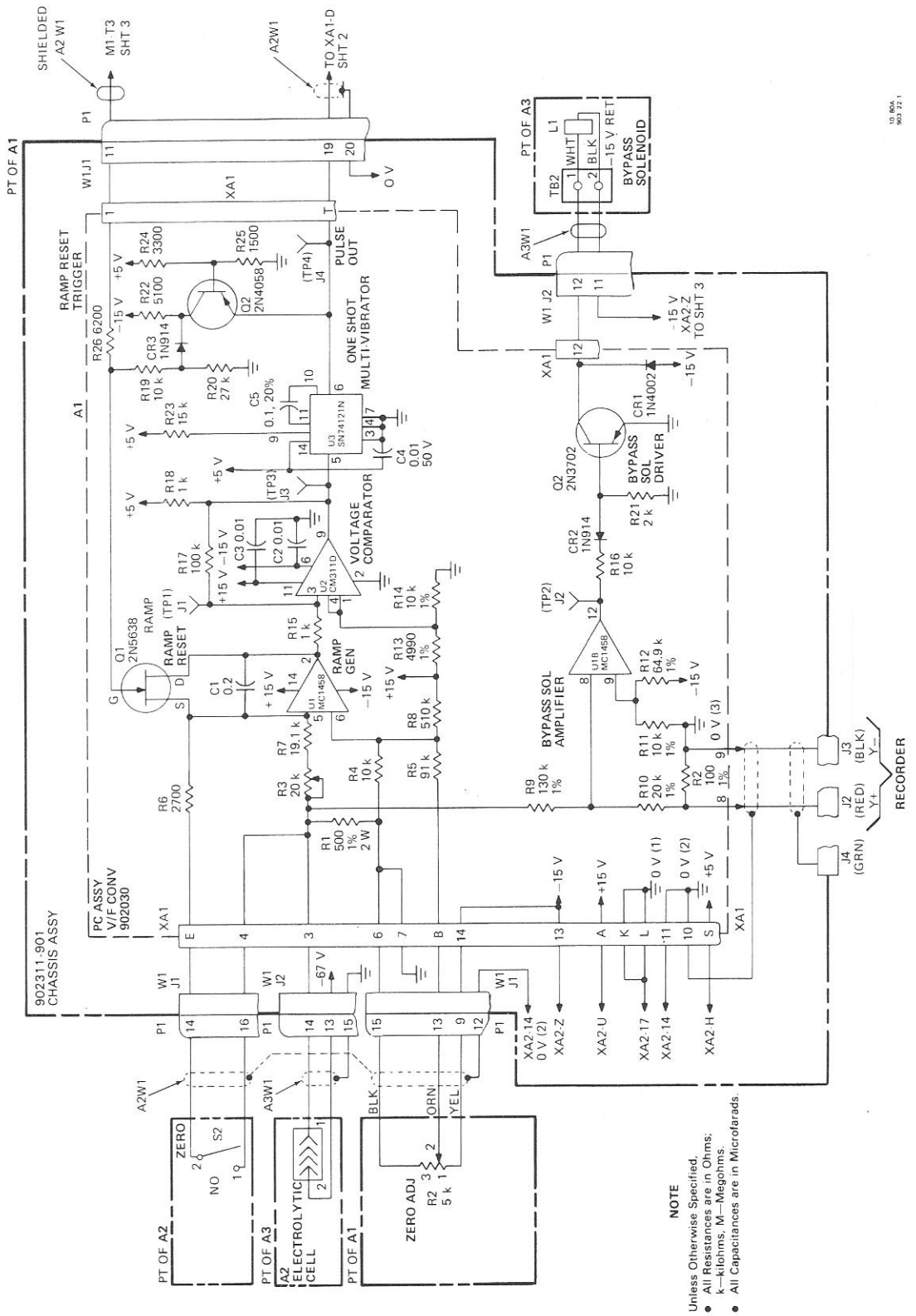


Figure 4-6. Schematic Diagram, Moisture Analyzer (Sheet 1 of 5)

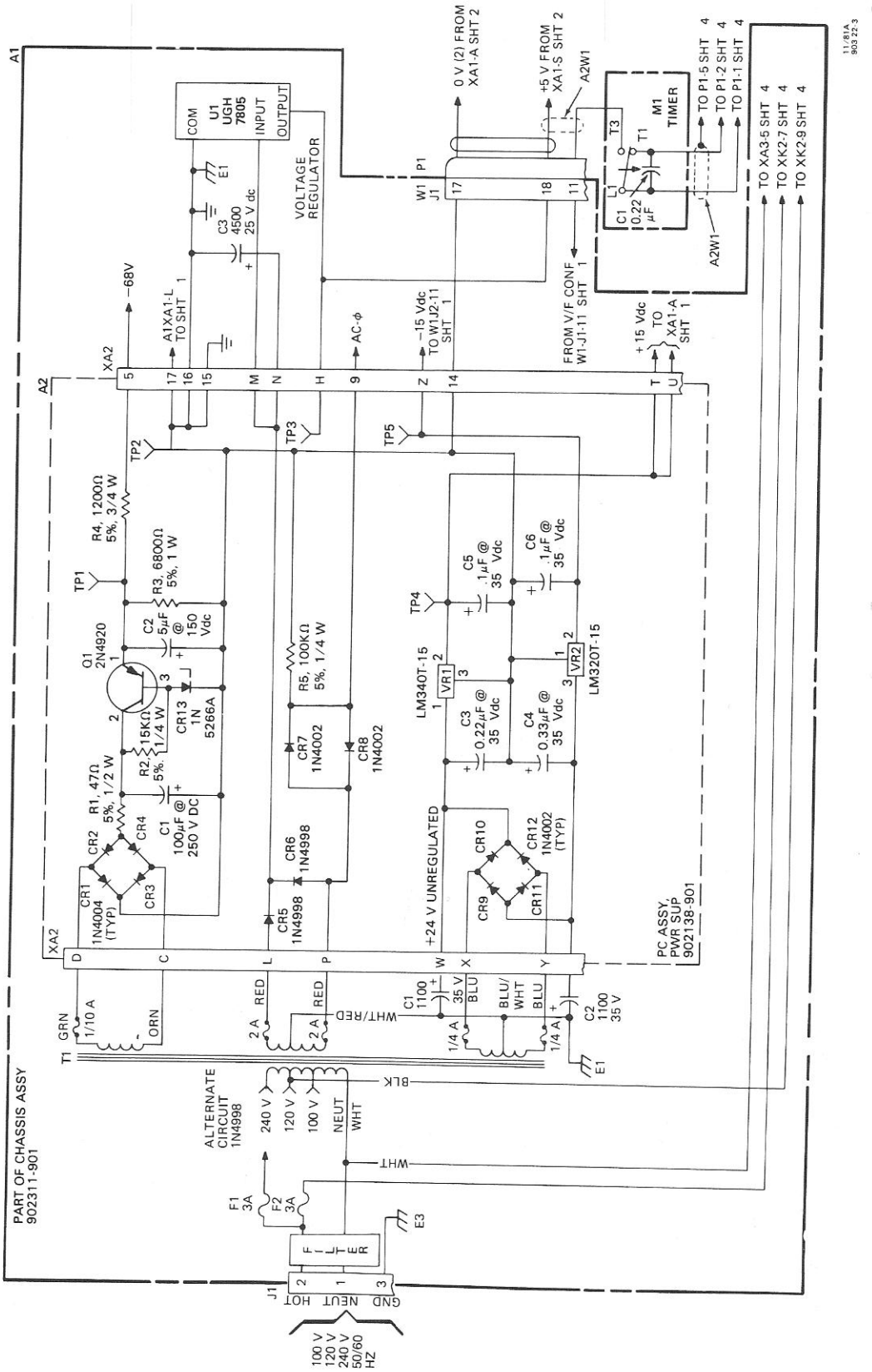


Figure 4-6. Schematic Diagram, Moisture Analyzer (Sheet 3 of 5)

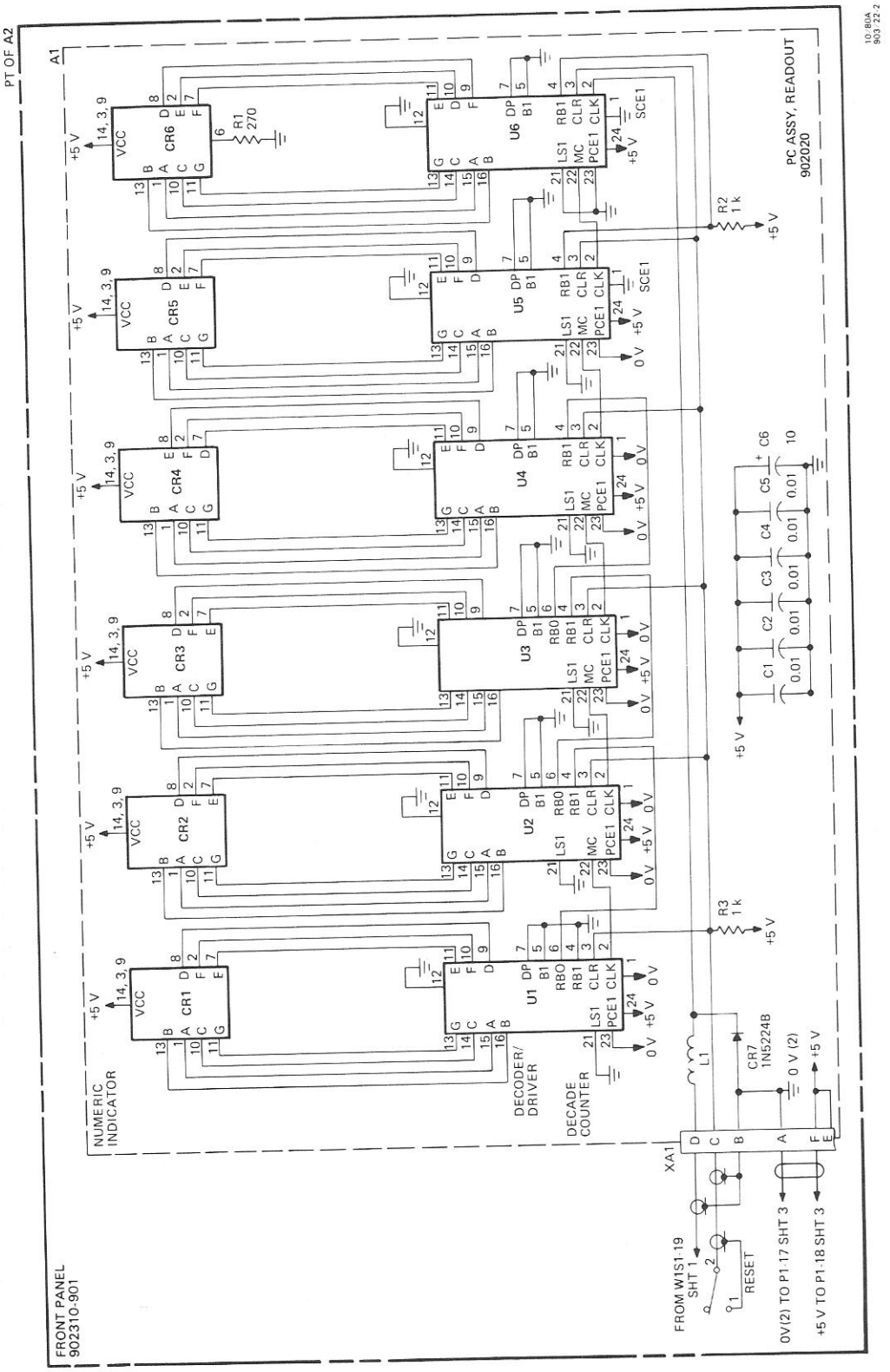
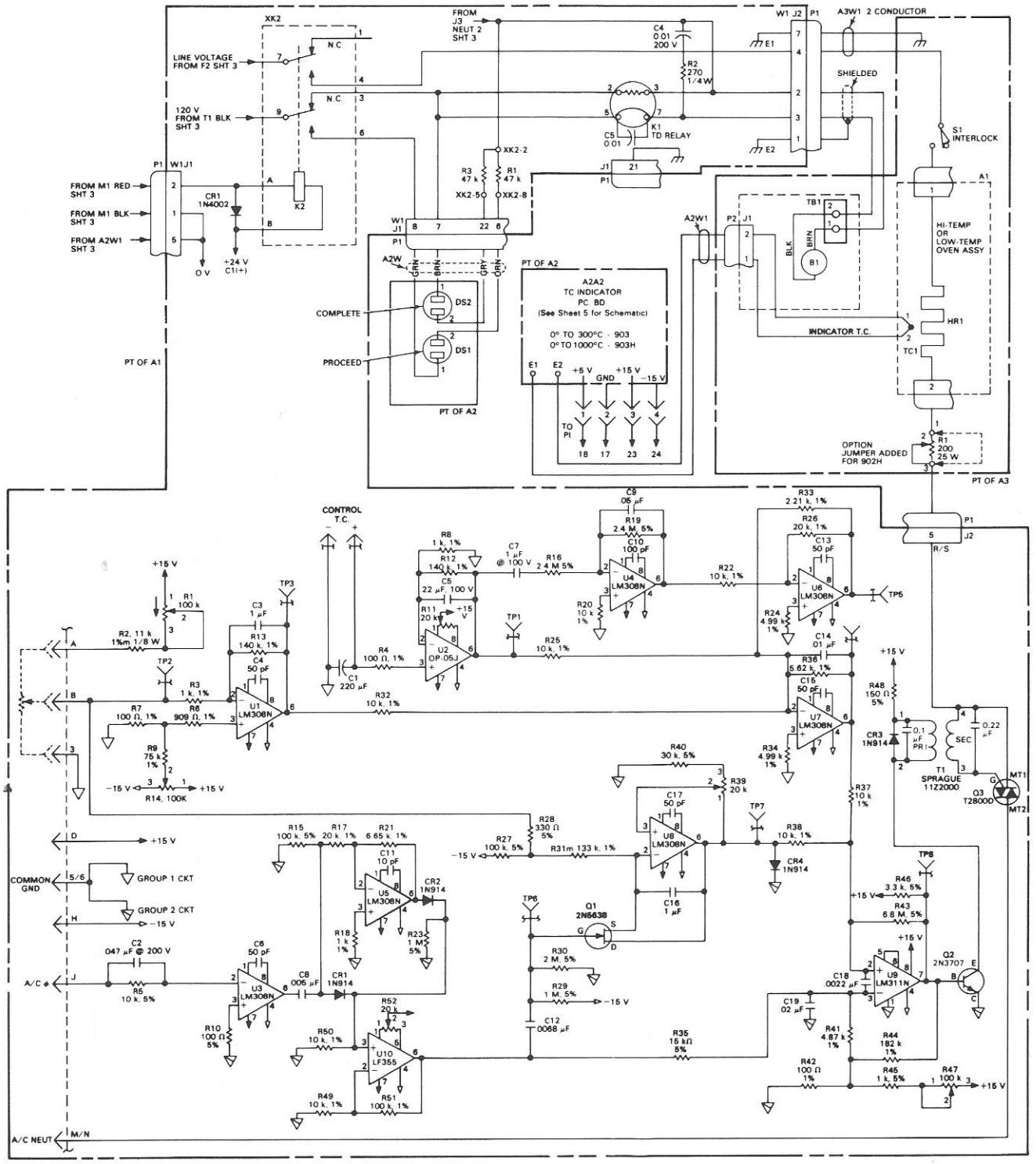


Figure 4-6. Schematic Diagram, Moisture Analyzer (Sheet 2 of 5)



NOTES:

1. ALL COMPONENTS U1, U2, U4, U6 &U7 SHOULD BE AWAY FROM THE TRANSFORMER.
2. ALL COMPONENTS U3, U5, U8 & U9 CAN BE AT HIGH A/C PICK-UP POINT — TRANSFORMER SIDE.
3. GROUP 1 CKT & GROUP 2 CKT GROUNDS SHOULD BE HELD SEPARATE WITH ONE COMMON POINT, AT PIN CONNECTION (PINS 5/6).
4. ALL TEST POINTS (T.P.) BROUGHT OUT TO DISCONNECT PINS.
5. ALL RESISTANCE VALUES ARE IN OHMS ARE 1/4 W UNLESS OTHERWISE SPECIFIED.

11/80A
903/22-4

Figure 4-6. Schematic Diagram, Moisture Analyzer (Sheet 4 of 5)

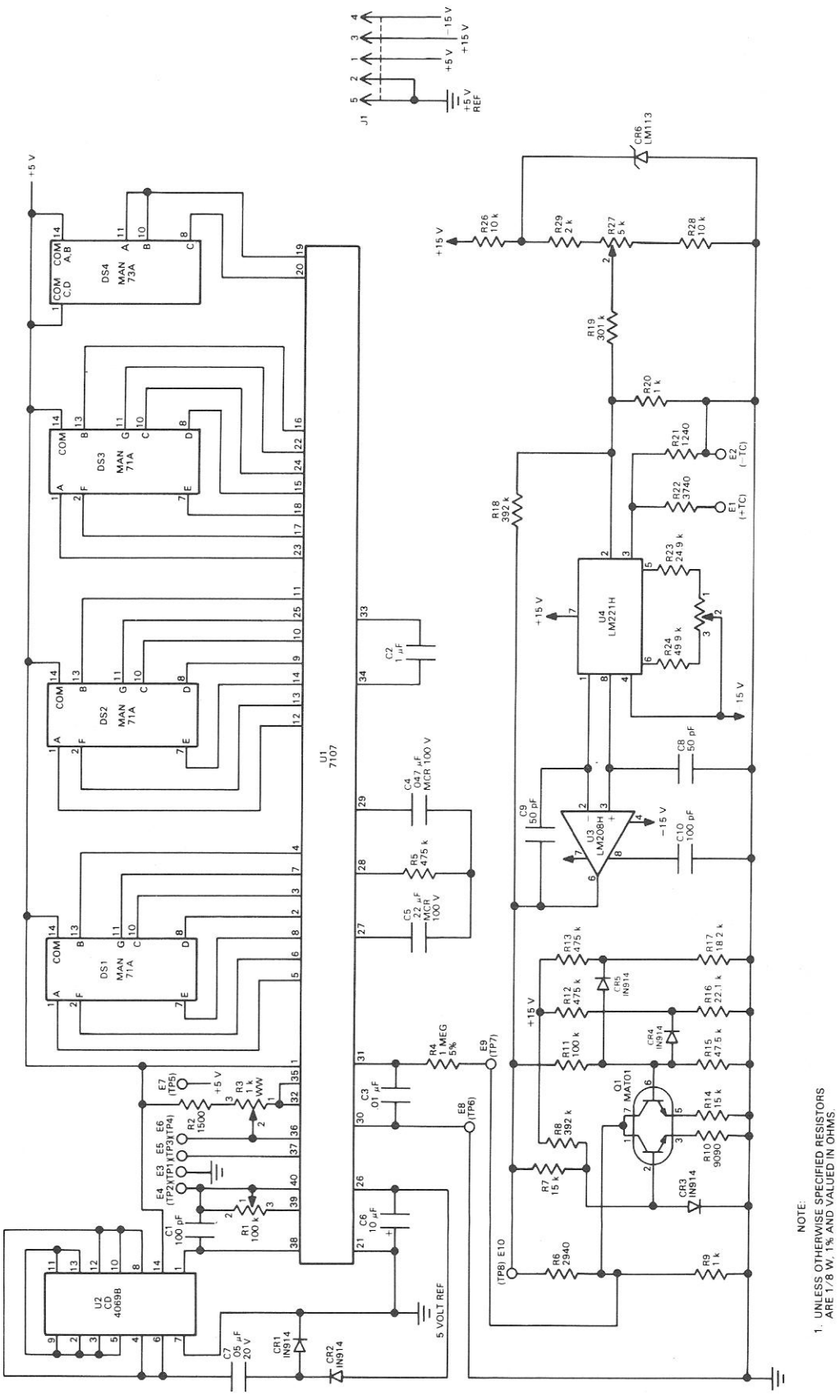


Figure 4-6. Schematic Diagram, Moisture Analyzer (Sheet 5 of 5)

Section 5. PARTS LIST

5-1. GENERAL.

This section lists the parts which Du Pont considers practical to stock and supply for replacement. The parts are identified by reference designation or item number in figures 5-1 through 5-8; figures in other sections of the manual are also referenced.

Table 5-1 lists the parts. Included in the table are item number or reference designation, part number, description, and figure reference.

5-2. CELL EXCHANGE PLAN.

If you do not want to recoat your own cells, you may exchange them for NEW cells. Send inactive cells to:

E. I. du Pont de Nemours and Co., (Inc.)
Analytical Instruments Division
TA Service Department — Glasgow
Wilmington, DE 19898

Normally, only special attaching hardware is listed. Except for any special item, all hardware is stainless steel in standard sizes that can be obtained locally.

5-3. PARTS ORDERING INFORMATION.

To obtain replacement parts, contact any of the regional offices listed below and include the part number and description. To ensure that you receive the correct part for your instrument, include the instrument type or number and its serial and/or model number.

Du Pont Company
Analytical Instruments Division
Concord Plaza — McKean Building
Wilmington, DE 19898
Phone: (302) 772-5500

Du Pont de Nemours Deutschland GMBH
Instrument Products Division
Dieselstrasse 18
P.O. Box 1509
D-635 Bad Nauheim 1
Federal Republic of Germany
Phone: (6032) 3961
Telex: 691576

Du Pont U.K. Ltd
Instrument Products Div.
Wedgwood Way, Industrial Bldg. No. 2
Stevenage, Hertfordshire
England SG1 4QN
Phone: (438) 727181
Telex: 825591

Table 5-1. Parts List

Item No. or Ref Des	Part No.	Description
A1 (fig. 5-1)	902311-901	Chassis Assembly
F1 (fig. 5-2)	205225-027	Fuse 3A, 250 V
F2 (fig. 5-2)	205224-055	Fuse, 3A, Slo-Blo, 250 V
F3 (fig. 5-2)	205224-009	Fuse, 0.1A, Slo-Blo, 250 V
F4, F5 (fig. 5-2)	205224-050	Fuse, 2A, Slo-Blo, 250 V
F6, F7 (fig. 5-2)	205224-021	Fuse, 0.25A, Slo-Blo, 250 V
A1A1 (fig. 5-1)	902030-901	Printed Circuit Board Assy, Voltage-to-Frequency Converter
A1A2 (fig. 5-1)	902138-901	Printed Circuit Assy, Power Supply
A1A3 (fig. 5-1)	902129-901	Printed Circuit Assy, Heater Controller, T/C Feedback
A2 (fig. 5-1)	902310-901	Panel Assy, front, Moisture Evolution Analyzer, 903 (0°-3000°C)
	902310-902	Panel Assy, front, Moisture Evolution Analyzer, 903-H (0°-1000°C)
A2A1 (fig. 5-1)	902020-901	Printed Circuit Assy, Readout
A2A2 (fig. 5-1)	902316-901	Printed Circuit Assy, Linearizer CKT, T.C. Indicator, MEA
M1 (fig. 5-6)	902118	Timer
A3 (fig. 5-1)	902326-901	Deck Assy, Cell and Oven, LO Temp.
	902326-902	Deck Assy, Cell and Oven, HI Temp.
1 (fig. 5-8)	303033	Cell Cap
2 (fig. 5-8)	901040	Oven Plug
3 (fig. 5-8)	202813-014	Seal, O-ring Viton® Fluoroelastomer
4 (fig. 5-8)	902333-901	Sample Oven
5 (fig. 5-8)	901051-905	Cell Case
A4 (fig. 1-3)	901030-901	Sieve Dryer Assy
E1 (fig. 1-2)	902002-901	Electrolytic Cell
1 (fig. 2-2)	902121-901	Filter Disc
2 (fig. 2-2)	202813-006	Seal, O-ring Viton® Fluoroelastomer, 2.9 mm (0.114)
CONSUMABLES:		
	901025-000	Shredded Teflon® TFE Fluorocarbon polymer
	901083-901	Sample Boat
	901046-901	Micropipetes

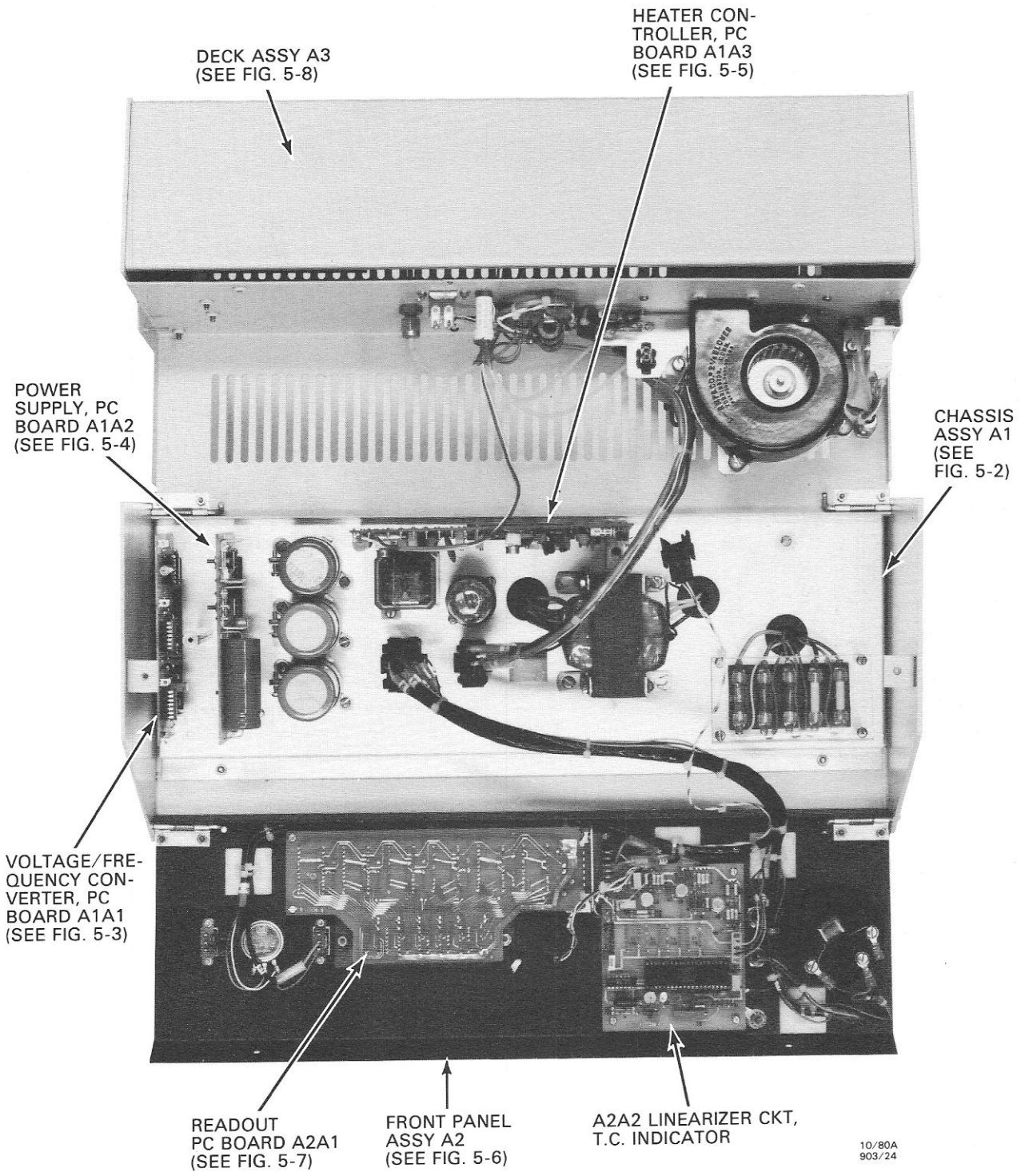


Figure 5-1. Location of Major Assemblies and Subassemblies.

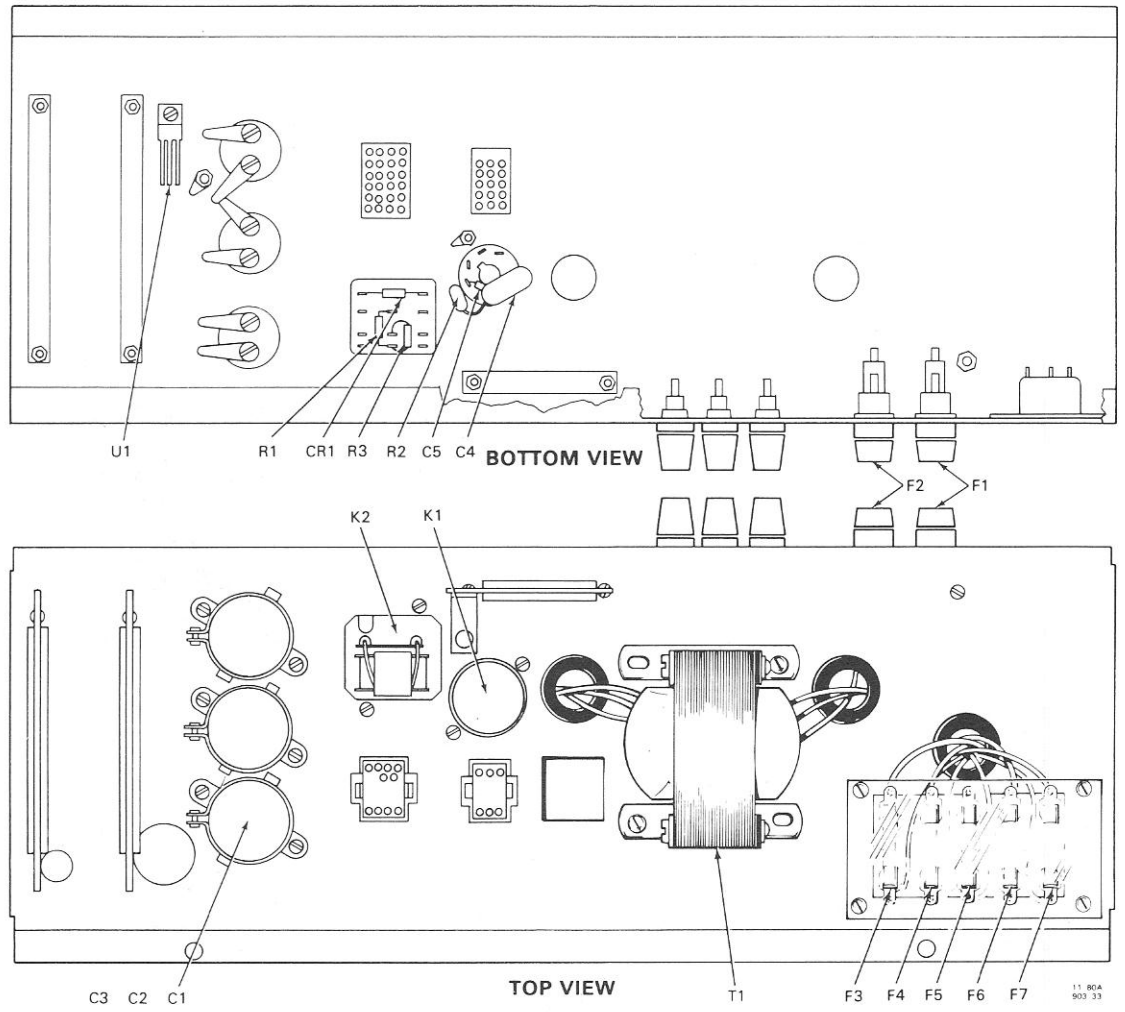


Figure 5-2. Chassis Assembly.

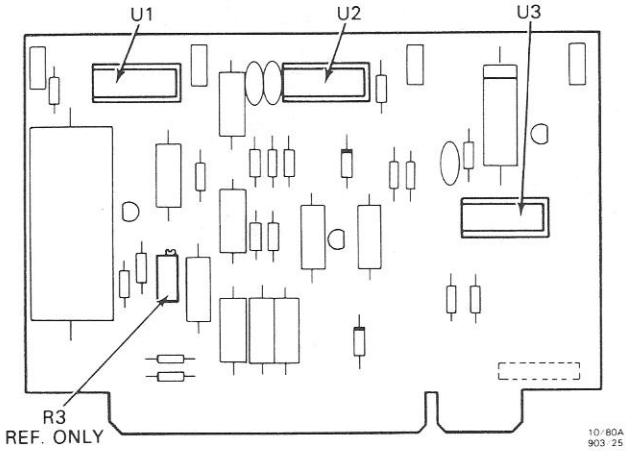


Figure 5-3. Voltage-to-Frequency Converter Assembly.

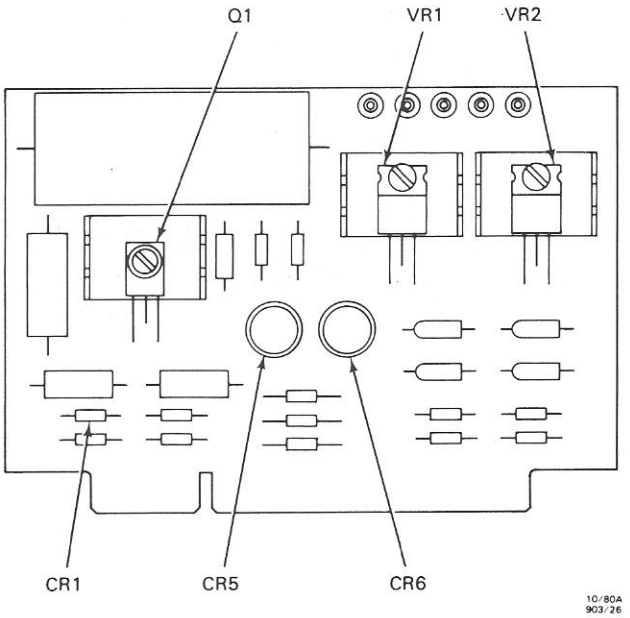


Figure 5-4. Power Supply Assembly.

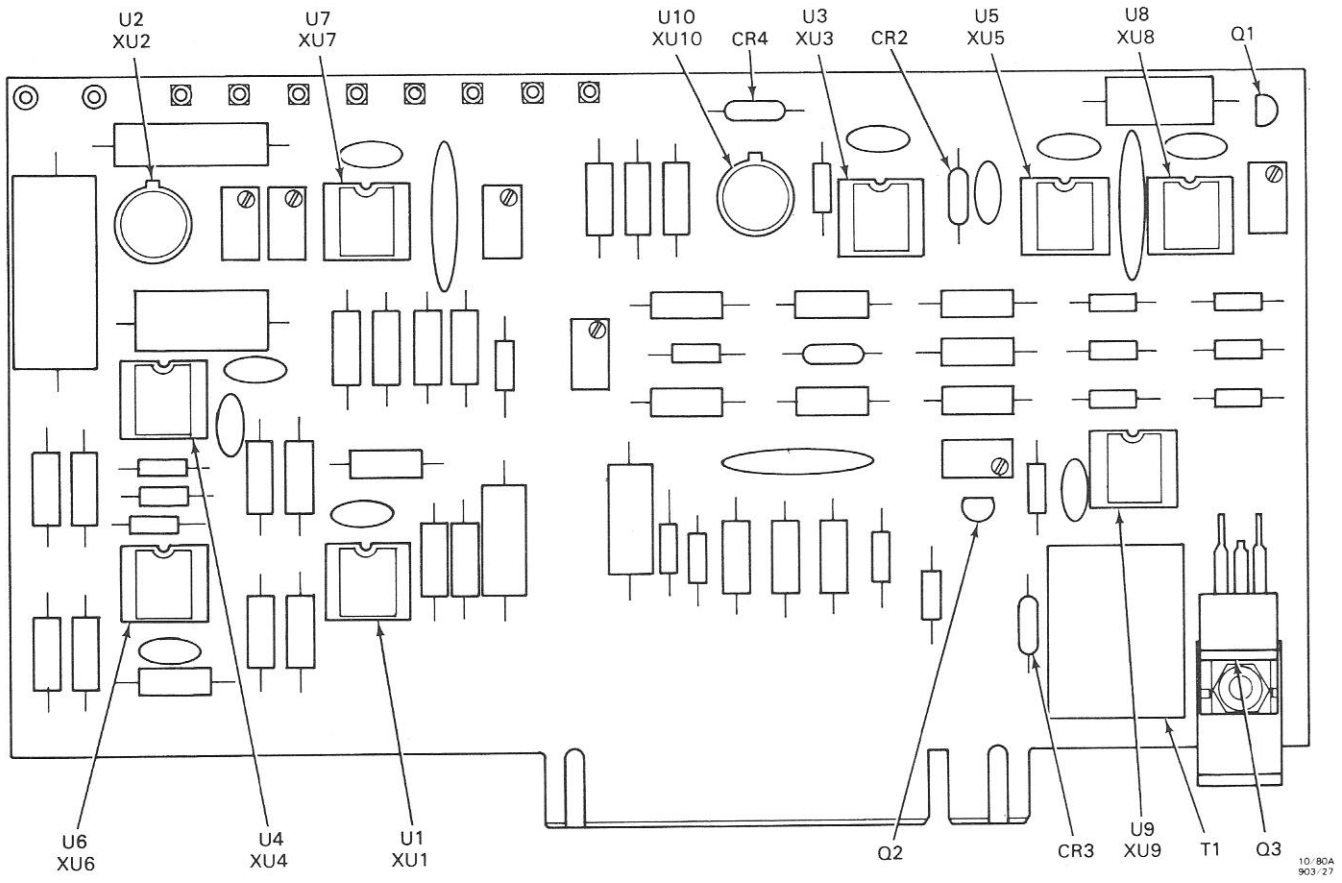


Figure 5-5. Heater Controller Assembly

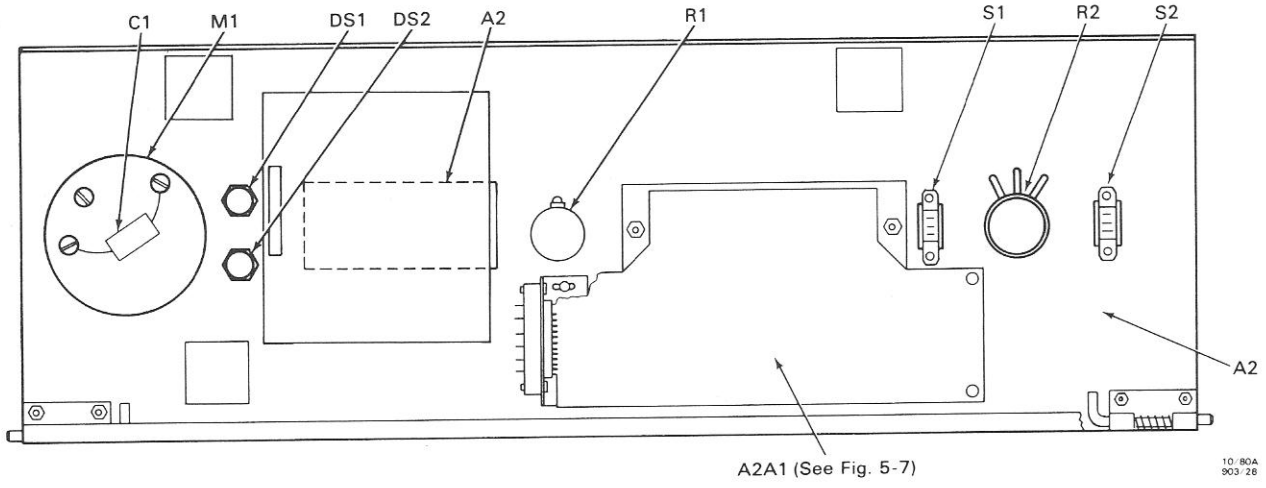


Figure 5-6. Front Panel Assembly.

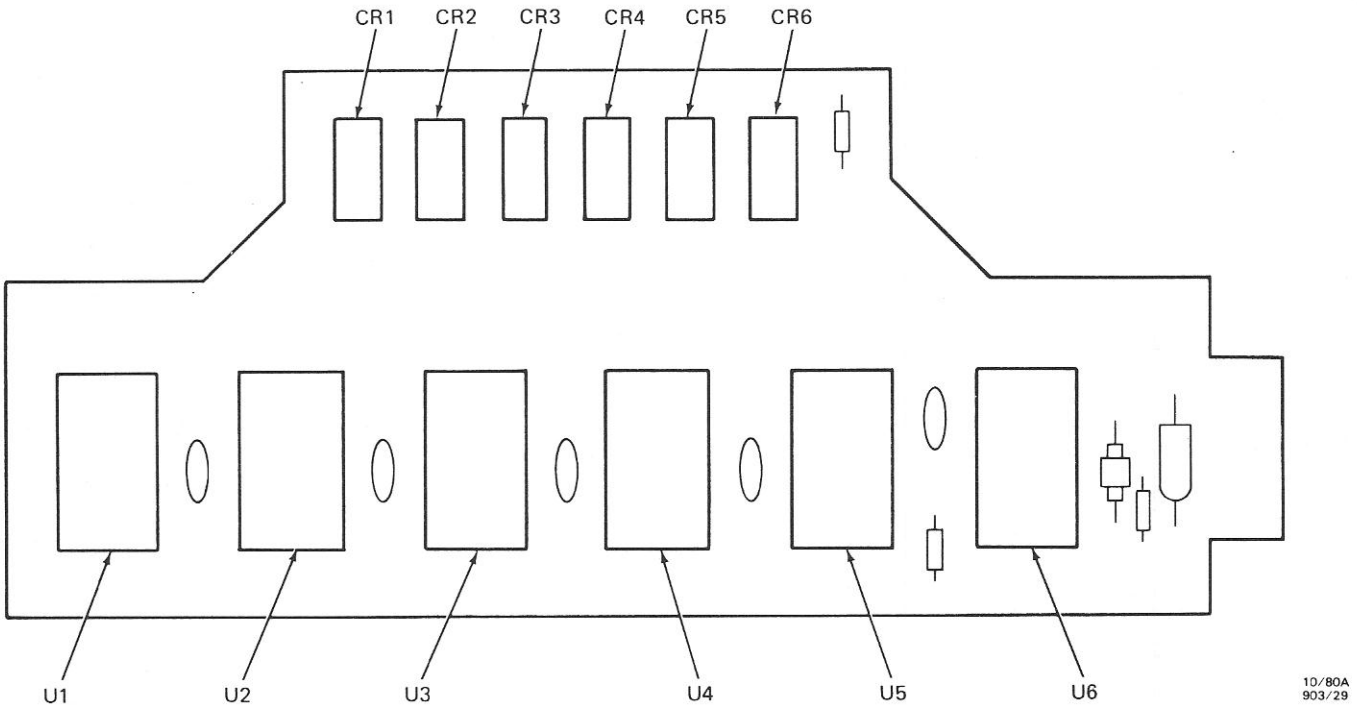


Figure 5-7. Readout Assembly.

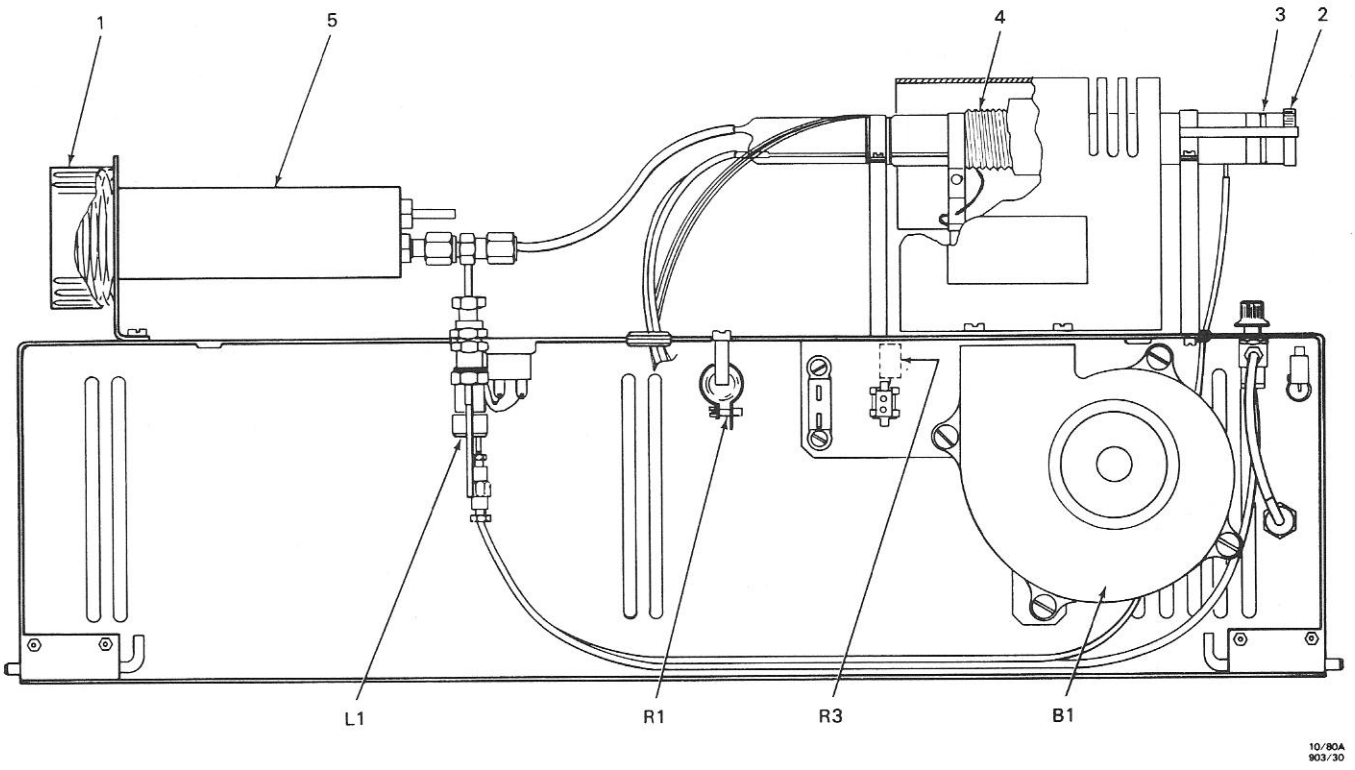


Figure 5-8. Deck Assembly.

APPENDIX A

THE ELECTROLYTIC CELL

The physical construction of the Du Pont electrolytic cell is shown in figure 1-6. The carrier gas with the moisture enters the cell through one of the ports in the end piece of Teflon® TFE Fluorocarbon polymer, passes through the length of the glass tubing inside the cell, and exits through the other port. Secured inside the glass tubing are two parallel, helically-wound platinum wires. Between these wires, the electrodes, a thin layer of phosphorous pentoxide is deposited on the glass. The wires are brought out through leak-tight seals to the electrical contacts in the Teflon® end piece.

The assembly is cased in epoxy resin for mechanical protection. This normally excludes damage from any cause except objects which may enter through the cell ports. For this reason, never insert any object such as a probe, syringe needle, wire, etc., into the small hole at the bottom of the port leading into the cell. This invariably makes the cell useless.

In an analyzer, the cell converts the sample water-content of the gas to an electrical current. The cell determines the moisture by absorbing this gaseous water on the thin film of phosphorous pentoxide deposited between the two electrodes. A dc voltage on the electrodes electrolyzes the absorbed water into molecular hydrogen and oxygen. Since phosphorous pentoxide is extremely hygroscopic and the cell is designed for intimate contact of gas with the absorbing film, all of the water vapor is absorbed and electrolyzed. This accounts for the high degree of accuracy and specificity, since the process is virtually unique for water and consumes at least 99.9% of the gaseous water at levels as low as 1 μg/g or as high as 100 percent.

Expressed simply as possible, the chemical and electrochemical reactions occurring are:

$$H_2O\,(g) + P_2O_5 \rightarrow P_2O_5 \bullet H_2O \rightarrow P_2O_5 + H_2\,(g) + 1/2\,O_2\,(g)$$

Step one is a simple chemical absorption of gaseous water by phosphorous pentoxide, which renders the film electrically conductive. Step two is electrolysis of the water into its component elements of hydrogen and oxygen, accompanied by a transfer of charge from one cell electrode to the other. If water enters at a given rate, as in an equilibrium state, it is absorbed and electrolyzed at a constant rate, resulting in a steady rate of charge transfer, which is synonymous with a constant current. Thus, current is directly and linearly related to mass flow-rate of water, and the magnitude of the resultant current may be calculated from Faraday's Law, the flow rate of gas and the relationship between mass flow-rate and volume flow-rate.

When these factors are considered for water being electrolyzed by a moisture cell, the current in microamperes per μg/g of water is given by 1 = (0.132 x flow rate) μA where the flow rate is the sample flow rate in cm^3/min, measured at 101 kPa (1 atm) pressure and 25°C. Thus, for example, at 20 cm^3/min, 1 μg/g water produces 2.64 μA; 100 cm^3/min, 13.2 μA.

Phosphorous pentoxide in its dry state is a very poor conductor of electricty, so the background signal not due to water is extremely low. The lowest readings ever obtained with the electrolytic cell correspond to just less than 0.01 μg/g, and indicate an internal dc cell resistance of more than 500 MΩ. Therefore, the error due to leakage in the cell must be, at most 0.01 μg/g.

APPENDIX B

ELECTROLYTIC CELL CONTAMINATION

B-1. INTERFERING AND CONTAMINATING SUBSTANCES.

In this discussion, a distinction is made between interfering and contaminating substances on the following basis: interfering substances are those causing an incorrect instrument reading, whether or not the cell is damaged; contaminating substances are those which cause damage to the cell whether or not the value of the reading is affected.

B-2. SUBSTANCES WHICH ONLY INTERFERE.

Hydrogen and, to a lesser extent, oxygen or air may cause a high reading due to recombination in the cell. Basic gases, such as volatile amines or ammonia are classified as interfering.

B-3. SUBSTANCES WHICH BOTH CONTAMINATE AND INTERFERE.

- Unwanted liquids and solids in the gas stream may have both effects.
- Alcohols and glycols, particularly the lower ones, are dehydrated and respond like water. They may also esterify with the cell coating and destroy it, necessitating recoating.

B-4. SUBSTANCES WHICH DO NOT INTERFERE OR CONTAMINATE.

These substances are typified by the inert gases, nitrogen, air, and oxygen (except as noted above), natural gases, and many aliphatic and aromatic hydrocarbons.

B-5. SUBSTANCES WHICH ONLY CONTAMINATE.

- Corrosive acid gases will corrode the instrument tubing and flow control system, but will not harm the cell, barring a few exceptions noted below. Typical examples are chlorine and hydrogen chloride.
- Some hydrocarbons do not interfere, but gradually coat the cell with a polymeric coating. Butadiene is an example. This coating will render the cell inactive, often in a relatively short period of time.
- Entrained liquids and solids can also cause contamination without interference, resulting in cell failure. Certain materials, such as graphite dust, usually cause irreparable damage. Always take precautionary measures in doubtful cases.
- Hydrogen fluoride will cause permanent damage and should not be run under any condition. Hydrogen fluoride will inevitably destroy the cell and usually the sample system.
- Ammonia and other basic elements react with the acidic cell coating, rendering the cell unresponsive in a short time but usually is not permanent.

B-6. When samples contain relatively high-boiling volatile materials, such as plasticizers, they may carry over and coat the cell, reducing efficiency. It has been found that cell life between coatings can often be increased (as much as tenfold) in such cases by putting shredded Teflon® TFE Fluorocarbon polymer in the oven beyond the heated zone. A bag of shredded Teflon® is provided with the instrument for this purpose. The Teflon® should be removed and cleaned with solvent or discarded when it becomes covered with trapped material.

APPENDIX C

MOISTURE ANALYSIS PARAMETERS

Due to the high sensitivity of the 903, the relationship of four parameters must be considered for accurate, repetitive analyses:

- Sample size
- Flow rate
- Temperature
- Time

Sample size and flow rate are closely related as are temperature and time. Sample size also is directly related to H_2O content. For example; a 15-mg sample with $\approx$ 15% H_2O at 100 cm^3/min may allow a small amount of undetected moisture, but 8 mg at the same flow or 15 mg at 80 cm^3/min should produce an accurate readout.

The following sample sizes are suggested for various expected moisture content percentages.

H_2O Content	Sample Size
$\mu g/g$	1 – 2 g
0.5%	$\approx$300 mg
1.0%	$\approx$150 mg
10.0%	$\approx$ 15 mg

For most samples, a 70-cm^3/min flow rate is recommended. Very large ($>$ 15 mg) samples at high moisture content ($>$ 10%) may require a lower flow rate. A high flow rate coupled with a high moisture laden sample may saturate the cell or pass some H_2O undetected. Also, a low flow may result in an overly long analysis time. A readout of approximately 2000 μg after a 20 minute interval is ideal. With a higher readout, some moisture may be undetected depending on the flow rate. One way to check the accuracy is to halve the sample size and make another run in the same time frame. If the readout is halved, you can be assured of the accuracy. If the readout is not halved (within $\approx$ 5%), cell calibration should be checked.

Since temperature affects the rate of H_2O volatilization, it also must be considered. Temperature must be high enough for the sample to volatilize the H_2O but yet not too high that organic material would be volatilized from the sample which may condense on the cell electrodes and cause cell efficiency to decrease. Cell efficiency does not necessarily have to be 100%, since the cell calibration factor (K) is determined in the standardization procedures and used in calculation of moisture content of samples analyzed. But, if K becomes less than $\approx$ 85%, the cell should be cleaned and recoated.

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