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Long Term Drift Studies of Sandia H_2 Sensors in Reducing Atmospheres

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Long Term Drift Studies of Sandia H_2 Sensors in Reducing Atmospheres

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Abstract

A study of the drift in Pd/Ni alloy hydrogen sensitive resistor and transistor responses is presented. The sensors were monitored for a period of 6 months in a reducing atmosphere of 0.1% H_2 in N_2 with periodic calibration exposures. A comparison of a resistor film with an adhesion layer showed considerable improvement in diminishing the drift.

EXECUTIVE SUMMARY

Long term drift studies of over 200 days on five hydrogen sensors were conducted in a reducing atmosphere (1000 ppm of H_2 in N_2). This is the first time that any sensor of this type has been monitored in a reducing atmosphere for any length of time. The sensors had been stored in laboratory air (oxidizing atmosphere) for more than two years, so it is known that the sensors survive in that environment. All the sensors survived quite well, with some drift in the four that were on the Sandia Robust sensor platform. The best performance, with almost no measurable drift or change in sensitivity, was found in the experimental Wide Range Sensor (WRS) which is not yet in production. The WRS sensor would not require any recalibration over a six month time period, but the Robust sensors would require periodic recalibration with known hydrogen concentrations to provide accurate measurements.

INTRODUCTION

The characterization of Sandia National Laboratories' developed WRS (Wide Range Sensors) and Robust sensors in a long term exposure to a reducing atmosphere were performed at room and elevated temperatures respectively. The following is a preliminary report on the effects of long term exposure to 0.1% H_2 in N_2 on drift, hydrogen sensitivity, and speed of response.

EXPERIMENTAL SETUP

Hydrogen Sensors

Two types of hydrogen sensors that were developed at Sandia National Laboratories[1] were used in the long-term drift tests. The Robust hydrogen sensor, shown in figure 1, incorporates a hydrogen sensitive field effect transistor (FET), a hydrogen sensitive resistor, a temperature sensor, and two heaters all on a 7.4 mm x 3.3 mm ASIC silicon based platform. A wide range sensor (WRS), shown in figure 2, incorporates a hydrogen sensitive resistor, a temperature sensor, and a heater on a 6.7mm x 3.7mm area silicon wafer.

Two of the Robust sensors and one of the wide range sensors were mounted into plexiglass test fixtures, shown in figure 3, and connected to a controlled gas flow system (gas test bed). The Robust sensors used were from reticule set HS005 (TA770) 11A where the code 11A is an indication of the run number. Robust sensors 2386 and 2540 were processed at the same time, however 2386 came from wafer number 2 and 2540 came from wafer number 3. This particular lot was processed on 8/24/95 and completed processing on 1/16/96. It is not known when these individual devices were mounted into headers and wirebonded. However, as part of our test procedure both the Robust sensors and the WRS received a 250°C anneal at 2% H_2 in N_2 for 2 hours before being placed into operation.

Gas Test Bed & Long Term Test Systems

The gas test bed shown in figure 3, is a series of valves and mass flow controllers that mixes source gases to provide a consistent flow of 1 standard liter per minute (slm) of the desired gas mixture. The gas sources are provided by pre-mixed bottled gases and a high purity, nitrogen, cryo line. The bottled gases used are ultra high purity (uhp) grade gases and the cryo source has been tested and found to be of research grade quality (>99.9999%). Table 1 shows the concentration of contaminants found in the cryo nitrogen line a year after installation. Due to the superior quality of the cryo line, this source is used constantly and the uhp nitrogen bottle is used as a backup during cryo line outages. The desired gas concentration is obtained by using Brooks 5850E flow controllers to mix the oxygen, nitrogen and 1% H_2 in N_2 sources. A steady flow of 1 slm is supplied to the sensors throughout the duration of the test.

After a few weeks of initial drift the sensors were connected to a more permanent gas controlled flow system. Since gas test bed #1 was needed to satisfy the calibration requirements of the program, a dedicated gas controlled flow system was built for the long-term tests. This system was similar to the one shown in figure 2, except two Unit UFC-1100A mass flow controllers were used to mix the nitrogen (SN

C11-93025) and the 1% H_2 in N_2 (SN C11-93669). These mass flow controllers were calibrated for the respective source gases and delivered a total of 1 slm flow to the sensors.

Robust Circuit Board

The Robust and WRS were controlled by separate circuit boards that provided the capabilities of individually monitoring the sensor's responses and temperatures. The circuit[2] used for the Robust hydrogen sensors is shown in figure 4. An ac to dc converter, AD 902-2, supplies the $\pm$15 volt/ 100mamp source, and the + 5 volt/5 amp source is supplied by an AD977. An additional $\pm$ 5 volt reference is supplied by two AD584 voltage regulators. Starting at the top of the circuit and working down we first encounter the Silicon Gate Power Transistor CKT. There are two power transistors, one at each end of the ASIC, which supplies heat to the sensors. The drains, PWR_TX_VDD, are supplied with +5V and the amount of voltage applied to the gate and subsequently the heat delivered to the sensors, is determined by the resistance setting of the potentiometer, R3. The temperature obtained is measured by a temperature sensitive diode, transistor emitter to base, and labeled the VBE Temperature. This voltage signal is compared to the desired temperature setting and the resultant amplified signal supplied to the power transistor gates, PWR_TX_G. When a temperature setting is initially dialed in, voltage is applied to the power transistor gates and this causes the transistors to turn on and provide heat to the wafer. As the temperature increases the voltage dropped across the diode, amp1-pins 6 to 7, increases and the voltage applied to the gate decreases until equilibrium is reached. The temperatures corresponding to a given Vbe are shown in figure 5.

The next part of the circuit provides a constant current to the hydrogen sensitive FET and measures the V_{TH} (threshold voltage). Resistor R13 determines the amount of current that flows from the drain to source, which are labeled H2_TX_D and H2_TX_S respectively. The variable resistor R14 is used to adjust the FET's initial baseline, VTH, to zero. When the FET is exposed to hydrogen, the amount of gate voltage needed to maintain the current through R13 changes. As the gate voltage, H2_TX_G, changes, the amount of change is reflected in the VTH signal output. This signal has a factor of 10 gain, which can be corrected for later if accurate FET voltage changes are required.

The last portion of the circuit provides a constant current to the hydrogen sensitive resistor and measures the change in resistance with respect to the initial resistance. This feat is accomplished by a two-stage amplifier shown at the bottom of the schematic and labeled amp2. The constant current is provided by connecting the resistor to the output and input of the first amplifier. Pin 6 of this amplifier is latched to the constant voltage, 2.5V, of pin 5. The amplifier now outputs whatever voltage is required to maintain a constant current across the sensor. Therefore, a change in resistance due to hydrogen exposure will result in a change in the output voltage, pin 7. The initial resistance setting is accomplished by adjusting R19 until the output, Delta_R, is zero. By applying Kirchhoff's current and voltage laws to the first amplifier the following equation is obtained:

$$\mathrm{Vopin7} := \frac{10 \cdot \mathrm{Rsensor}}{\mathrm{Ro}} - 10$$

Ro is the combination of R19 and R19A and Rsensor is the resistance of the sensor between pins 6 and 7. By applying Kirchhoff's current and voltage laws to the second amplifier we obtain:

$$\mathrm{Vopin1} := -4\ \mathrm{Vopin7}$$

When R19 is adjusted to give zero voltage out, Vopin1, the value of Ro is equal to Rsensor as is realized in the first equation. Now any changes in Rsensor, due to hydrogen exposure, can be seen as a new value of Rsensor=Ro + ΔRsensor. If this new value of Rsensor is inserted into the first equation and the result plugged into the second we obtain:

$$Vopin1 = 10 \cdot \frac{\Delta Rsensor}{Ro}$$

The output is therefore proportional to the change in resistance divided by the initial resistance, which is stored by the value of R19 and R19A. To determine the magnitude of a signal output for a typical change in resistance, set Ro=1000 ohms and assume the Rsensor changes from 1000 to 1010 ohms. The change in Rsensor is 1% and the output will change from zero to 100mvolts. Therefore the output signal at pin 1 of amp2 is 0.01%/mV.

WRS Circuit Board

The wide range sensor control circuit utilizes both a palladium nickel (Pd/Ni) alloy film and a nickel (Ni) film to compensate for the temperature effects in the hydrogen response. The Pd/Ni film is sensitive to hydrogen and temperature while the nickel film is only sensitive to temperature. Therefore, the temperature response of the Pd/Ni film can be subtracted out by the temperature response of the Ni film. To accomplish this, the sensors are placed in the feedback loop of separate op-amps and a selectable constant current is flowed through each film at a level that provides cancellation. The signal from both sensors is monitored along with the compensated, differential, signal. Another advantage to this circuit is the four terminal resistance measurements, which eliminates any contact resistance changes with temperature or drift. The wide range sensor also has a nickel film heater that allows the sensor to be operated at elevated temperatures. However, for this project the effects of long-term drift at room temperature were desired and the heater portion of the circuit was disconnected.

DATA

The three devices used in the long-term drift test were annealed at 250°C for 2 hours in 2% H_2 in N_2. The anneal is a standard process that cleans the surface and in turn speeds up the response. For new devices, the anneal also removes the majority of undershoot problems possibly by reducing the number of grain boundaries in the Pd/Ni alloy films. Undershoot is a condition where the resistor's response purges past the baseline after seeing a hydrogen pulse. In unannealed devices this offset in the baseline can be as much as 40% of a 0.1% H_2 in N_2 signal level. The devices were annealed on 6/2/98 and placed into the test chamber on 6/10/98. The Robust devices were "zeroed" at room temperature by adjusting the control circuits offsets until the responses showed less then 500 mV.

Once it was determined the devices were responding to hydrogen, they were heated to ~50°C and given several calibration pulses and monitored for 12 days. Figures 7, 8, and 9 shows the temperature dependence, level of responses to 0.1%, 1%, and 10% H_2, and the initial drift due to heating for the Robust sensors 2386 Resistor, 2540 resistor, 2386 & 2540 FETs respectively. Expanded views of the calibration tests performed on the first day are shown in figures 10, 11, and 12 for resistors and FETs. After the devices were heated to 50°C, they were exposed to 1% H_2 in N_2 and 10% H_2 in N_2 with synthetic air purges in between and then stepped from 0.1% H_2 to 1% H_2 and back to 0.1% H_2. The resistors responded quickly and came back to their original baseline with almost no undershoot and showed no hysteresis in stepping to a higher concentration and back. Although it takes longer for the FETs to reach a saturated value, they also responded as quickly as previous devices of this type. From comparing the resistors' responses to the FETs one can see that at 0.1% H_2 in N_2 the resistors are at the lower end of its sensitivity while the FETs. are showing their normal logarithmic response to hydrogen partial pressure. With only about 20 mV change for each decade of hydrogen partial pressure, the accuracy of the FETs in reading the higher partial pressures is seen to be much less than the resistors

Although the heaters try to maintain a constant 50°C across the sensors, the surface temperature varies slightly with the ambient temperature and causes the resistors' responses to change slightly. Figure 13 shows how a half a degree change in the substrate temperature causes a 0.08% change in the 2386 resistor's response. This variation can be corrected by using the following temperature compensation formula:

$$R(\text{temp. corr.}) = R - (T - To) * \Delta R / \Delta T$$

Where To is the initial temperature to correct the resistance, R, to and $\Delta R/\Delta T$ is the ratio of the change in resistance to the change in temperature. Once the temperature effects are subtracted out, the amount of drift can be seen in the temperature corrected resistance, R(temp. corr.) in figure 13. The temperature corrected responses for figures 7 and 8 are shown in figures 14 and 15. The temperature corrected resistive response of the WRS, Vgas resistor, is shown in figure 16. The WRS device was operated at room temperature throughout the test. These slight changes in temperature do not effect the Robust FETs response and therefore does not need temperature correction.

After the first 12 days of drift the devices were connected to a new dedicated gas flow controlled system to allow the main flow controller system to be free for other characterization tests. The change-over induced some offsets in the baseline responses, which may be due to a slight difference in the hydrogen concentration (i.e. the new system uses a different set of mass flow controllers) and the possibility exists that the front panel offset adjustments could have been bumped. Notes at the start of the tests show a slightly different setting than current settings. The devices were monitored for the next 40 days in a 0.1% H_2 in N_2 atmosphere with one calibration test on Day 26. The results are shown in figures 17,18,19, and 20. Although the WRS resistor showed no drift, less than $\pm$ 100 ppm, Robust resistor 2386 showed a change in baseline that is equivalent to a 0.08% H_2 exposure and Robust resistor 2540 showed a change equivalent to a 0.4% H_2 exposure. The FETs also showed only slight drift after settling in.

On day 66 the building power was turned off for a period of 8 hours. During this time the sensors lost power and cooled to room temperature and were exposed to ambient air. Once the power was restored the sensors were reheated to 50°C and exposed to nitrogen before restarting the 0.1% H_2 in N_2 flow. The power outage effected the baselines on all the devices as can be seen in figures 21,22,23,24, and 25. The response for the Robust resistor 2540, figure 22, and Robust FET 2386, figure 24, showed a large increase in the drift rate which appears to be a result of the outage. Figures 23 and 25 shows very little drift for the Robust 2540 FET and WRS respectively.

RESULTS/DISCUSSION

The initial results of long term exposure to 0.1% H_2 in N_2 does not harm the sensitivity of the sensors or the speed of their responses. Although some of the sensors' baselines drifted with time, their responses to 0.1% H_2 and 1% H_2 remained the same. The same film on two different platforms showed different characteristics. The Pd/Ni resistive film on the WRS showed virtually no drift, the Pd/Ni resistive films on the Robust devices drifted. It is not known if the elevated temperature of 50°C contributed to the films drift in the form of accelerated aging or if elements of the control circuit may be changing with time. One of the devices, Robust 2540 FET, tended to indicate that the device drifts much less after an initial settling period.

To determine the source of the drift would require monitoring the currents through the sensors, and placing a secondary temperature indicator on the devices or periodic temperature calibrations. A change in the components used in the circuit would cause a change in voltage and therefore the current supplied to the hydrogen sensitive FET and resistor. This change in current would appear as long term baseline drift in the sensors. By monitoring the voltages and currents it could be determined if the drift was due to the Pd/Ni films or changes in the control circuit. Another possibility is the aging of the Robust temperature sensitive diode. By operating at elevated temperatures for long period of times, the diode characteristics may change and result in an increase or decrease in the heat applied to the wafer. To measure this type of effect would require placing another more durable temperature sensor on the device or performing temperature calibrations. Placing a secondary temperature indicator on the wafer may be possible but not practical. The alternative requires placing the sensors in an oven and reading the responses at a given temperature to a given gas concentration. The consistency of reading room temperature by these on-chip diodes means that the likelihood of drifting temperature readings under power is low.

Initial post mortem tests did reveal the source of the sudden base line change for the wide range sensor but not for the Robust sensor. A measurement of the supply voltage for the wide range sensor circuit showed a slight change had occurred. A careful measurement of the supply voltage and resultant signal was made before and after resetting to its previous value. The change in supply voltage resulted in a change in signal that exactly matched the change seen during the long-term tests. Therefore, it can be concluded that at the time of the baseline change someone had bumped the power supply voltage control knob. However, tests on the Robust circuits could not definitely determine the same results. Values recorded for the potentiometers setting before and after the tests showed no change for all but one. The change in signal for the slight change on the one potentiometer did not account for the changes seen during the long-term drift tests. Power failure tests were also simulated by removing power to both the Robust control circuits. Voltages were recorded at various tests points throughout the circuit and then power was removed. After the power was restored voltages were recorded again. This was done several times and a comparison of the test point voltages showed only slight changes which do not account for the changes observed during the long term tests.

A possible cause of the sudden shifts and drift could be due to the slow delamination of the films. It is known that at high hydrogen concentrations films can suddenly peel off due to a lack of an adhesion layer. This delamination may be slowly occurring at lower hydrogen concentrations. A comparison of the resistors with an adhesion layer (WRS) and the resistors without an adhesion layer (Robust) clearly show a superior performance in stability with adhesion layers. Currently no one has developed a way to improve the adhesion of the alloy films in transistors without eliminating the hydrogen response. However, we have several ideas on how to accomplish this task.

These preliminary results are very encouraging for the long-term use of the Robust and wide range sensors in an environment that has a primarily reducing atmosphere. This may be the case for most hermetically sealed components, where oxygen is excluded and corrosion tends to produce hydrogen gas. The Robust hydrogen sensor is now available from a company, DCH Technology in Valencia, CA (phone 805-775-8120). The commercial sensor is manufactured under a license agreement with SNL by Allied Signal. Almost any electronic part (transistor, diode, IC) that is offered as a commercial part will have reliability and performance testing of thousands of parts in many environmental conditions. Unfortunately, this is not the case for the Robust hydrogen sensor. Sandia never performed anything more than the testing of a few parts and the DCH company has very limited resources. Therefore, there has been no testing or burn-in of the commercial parts, other than a few days of head-to-head testing at Sandia which showed that the performance was comparable to Sandia produced parts. The aging test that is reported here is the first performed in a reducing atmosphere. For serious applications, one would want to perform similar aging, reliability and performance tests on several dozen parts to get statistical information. It would also be useful to correlate the drift phenomena with processing variations to produce more reliable parts.

ACKNOWLEDGEMENTS

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FIGURE CAPTIONS

Figure 1 A Robust hydrogen sensor ASIC mounted in a 20mm x 8mm dual inline package. The ASIC incorporates 4 active sensors, a hydrogen sensitive field effect transistor (FET), a hydrogen sensitive resistor, a nickel film temperature sensor, and a pn junction temperature sensor with two heaters. The Robust hydrogen sensor can sense from 10ppm to 100% hydrogen.

Figure 2 A wide range sensor (WRS) mounted in a dual inline package. The WRS incorporates a hydrogen sensitive resistor, a temperature sensor, and a heater on a 6.7mm x 3.7mm area silicon wafer. It can sense hydrogen concentrations from 100ppm to 100% hydrogen.

Figure 3 A schematic diagram of the Gas Test Bed used to expose the WRS and Robust sensors to mixed gases. The source gases are mixed via mass flow controllers to deliver the desired concentration to the sensors which are mounted in test fixtures as shown in bottom photo. Flow indicators monitor the gas flow to and from the sample chamber to insure no leaks are present.

Table 1 A time lapse measurement of the concentration of contaminants found in the cryo nitrogen line a year after installation.

Figure 4 A schematic of the control circuitry used to bias the sensors and heaters and monitor the sensors' responses. The connector diagrams show the pins used to supply power to the circuit board and for sending and receiving signals to the sensors.

Figure 5 The temperature of the Robust hydrogen sensor ASIC as function of V_{be} setting. The V_{be} setting is the value displayed by the dial mounted on the potentiometer. This potentiometer is the variable resistor R3 shown in Figure 4.

Figure 6 A schematic of the control circuitry for the Wide Range Sensor (WRS). The sensor elements are shown as Rgas, Rtemp, and Heater. Rgas is the hydrogen and temperature sensitive Pd/Ni film, Rtemp is the temperature sensitive Ni film, and Heater is a low resistance Ni film that can be used to heat the sensor. The temperature response of the Pd/Ni film is subtracted out by comparing signal Vg & Vt. The Differential Output is then the temperature independent hydrogen response.

Figure 7 Initial calibration of Robust 2386 resistor at 24°C and 49°C. The resistor was zeroed at room temperature in nitrogen and tested for response to 0.1% H2 in N2. The temperature was then elevated to the long-term test operating temperature of 50°C (actual temperature was 49°C). The resistor was allowed to stabilize in air environment and then exposed to 1% and 10% H2 in N2 before settling into a long term test condition of 0.1% H2 in N2. After 5 days the device's response to 1% H2 in N2 was checked again and returned to the 0.1% H2 in N2 test condition.

Figure 8 Initial calibration of Robust 2540 resistor at 24°C and 49°C. The resistor was zeroed at room temperature in nitrogen and tested for response to 0.1% H2 in N2. The temperature was then elevated to the long term test operating temperature of 50°C (actual temperature was 49°C). The resistor was allowed to stabilize in air environment and then exposed to 1% and 10% H2 in N2 before settling into a long term test condition of 0.1% H2 in N2. After 5 days the device's response to 1% H2 in N2 was checked again and returned to the 0.1% H2 in N2 test condition.

Figure 9 Initial calibration of Robust 2386 & 2540 transistors at 24°C and 49°C. The transistors were zeroed at room temperature in nitrogen and tested for response to 0.1% H2 in N2. The temperature was then elevated to the long-term test operating temperature of 50°C (actual temperature was 49°C). The transistors were allowed to stabilize in air environment and then exposed to 1% and 10% H2 in N2 before settling into a long term test condition of 0.1% H2 in N2. After 5 days the transistors' responses to 1% H2 in N2 were checked again and returned to the 0.1% H2 in N2 test condition.

Figure 10 An expanded view of figure 7 showing the initial calibration tests on Robust 2386 resistor after adjusting the temperature to 50°C.

Figure 11 An expanded view of figure 8 showing the initial calibration tests on Robust 2540 resistor after adjusting the temperature to 50°C.

Figure 12 An expanded view of figure 9 showing the initial calibration tests on Robust 2386 and 2540 transistors after adjusting the temperature to 50°C.

Figure 13 The temperature response of a Robust resistor reacting to changes in the room temperature cycles. The temperature in the room can vary as much as 8°C overnight, however the feedback control for the Robust circuit limits these excursions to less than 0.6C. The limited temperature change that the Robust resistor undergoes still causes a small change in the response of 0.1%. If the temperature response is removed the resultant signal, R(temp. corr.), can be used to monitor long term variations.

Figure 14 The temperature corrected signal of figure 7 showing the Robust 2386 resistor's response to the initial calibration gas mixtures.

Figure 15 The temperature corrected signal of figure 8 showing the Robust 2540 resistor's response to the initial calibration gas mixtures.

Figure 16 The temperature corrected signal of the Wide Range Sensor's response to the initial calibration gas mixtures. The Wide Range Sensor was operated at 24°C through the duration of the test.

Figure 17 Drift of the Robust 2386 resistor in 0.1% H2 in N2 for an additional 40 days with calibration tests around the 26^{th} day.

Figure 18 Drift of the Robust 2540 resistor in 0.1% H2 in N2 for an additional 40 days with calibration tests around the 26^{th} day.

Figure 19 Drift of the Robust 2386 and 2540 transisters in 0.1% H2 in N2 for an additional 40 days with calibration tests around the 26^{th} day.

Figure 20 Drift of the Wide Range Sensor's response to 0.1% H2 in N2 for an additional 40 days with calibration tests around the 26^{th} day.

Figure 21 Drift of the Robust 2386 resistor's response to 0.1% H2 in N2 for the next 125 days. At about day 65 power to the building was lost and subsequently the gas flow. The sensors therefore were exposed to ambient air in the room. After power was regained the devices were purged with nitrogen and then the 0.1% H2 in N2 gas mixture continued. During the 125 days of monitoring drift the device's responses to nitrogen and 1% H2 in N2 were checked.

Figure 22 Drift of the Robust 2540 resistor's response to 0.1% H2 in N2 for the next 125 days. At about day 65 power to the building was lost and subsequently the gas flow. The sensors therefore were exposed to ambient air in the room. After power was regained the devices were purged with nitrogen and then the 0.1% H2 in N2 gas mixture continued. It should be noted that the power outage caused a dramatic change in the slope of resistance change with respect to time. During the 125 days of monitoring drift the device's responses to nitrogen and 1% H2 in N2 were checked.

Figure 23 Drift of the Robust 2540 transistor's response to 0.1% H2 in N2 for the next 125 days. At about day 65 power to the building was lost and subsequently the gas flow. The sensors therefore were exposed to ambient air in the room. After power was regained the devices were purged with nitrogen and then the 0.1% H2 in N2 gas mixture continued. During the 125 days of monitoring drift the device's responses to nitrogen and 1% H2 in N2 were checked. The amount of drift seen in the transistor appears to be less with time.

Figure 24 Drift of the Robust 2386 transistor's response to 0.1% H2 in N2 for the next 125 days. At about day 65 power to the building was lost and subsequently the gas flow. The sensors therefore were exposed to ambient air in the room. After power was regained the devices were purged with nitrogen and then the 0.1% H2 in N2 gas mixture continued. During the 125 days of monitoring drift the device's responses to nitrogen and 1% H2 in N2 were checked.

Figure 25 Drift of the Wide Range Sensor's response to 0.1% H2 in N2 for the next 125 days. At about day 65 power to the building was lost and subsequently the gas flow. The sensors therefore were exposed to ambient air in the room. After power was regained the devices were purged with nitrogen and then the 0.1% H2 in N2 gas mixture continued. During the 125 days of monitoring drift the device's responses to nitrogen and 1% H2 in N2 were checked. Note that the WRS's baseline shifted at the time of the power outage. Post mortem tests revealed that the power supplied to the circuit had changed slightly. When the circuit was supplied with the correct voltage, the baseline returned to the previous value.

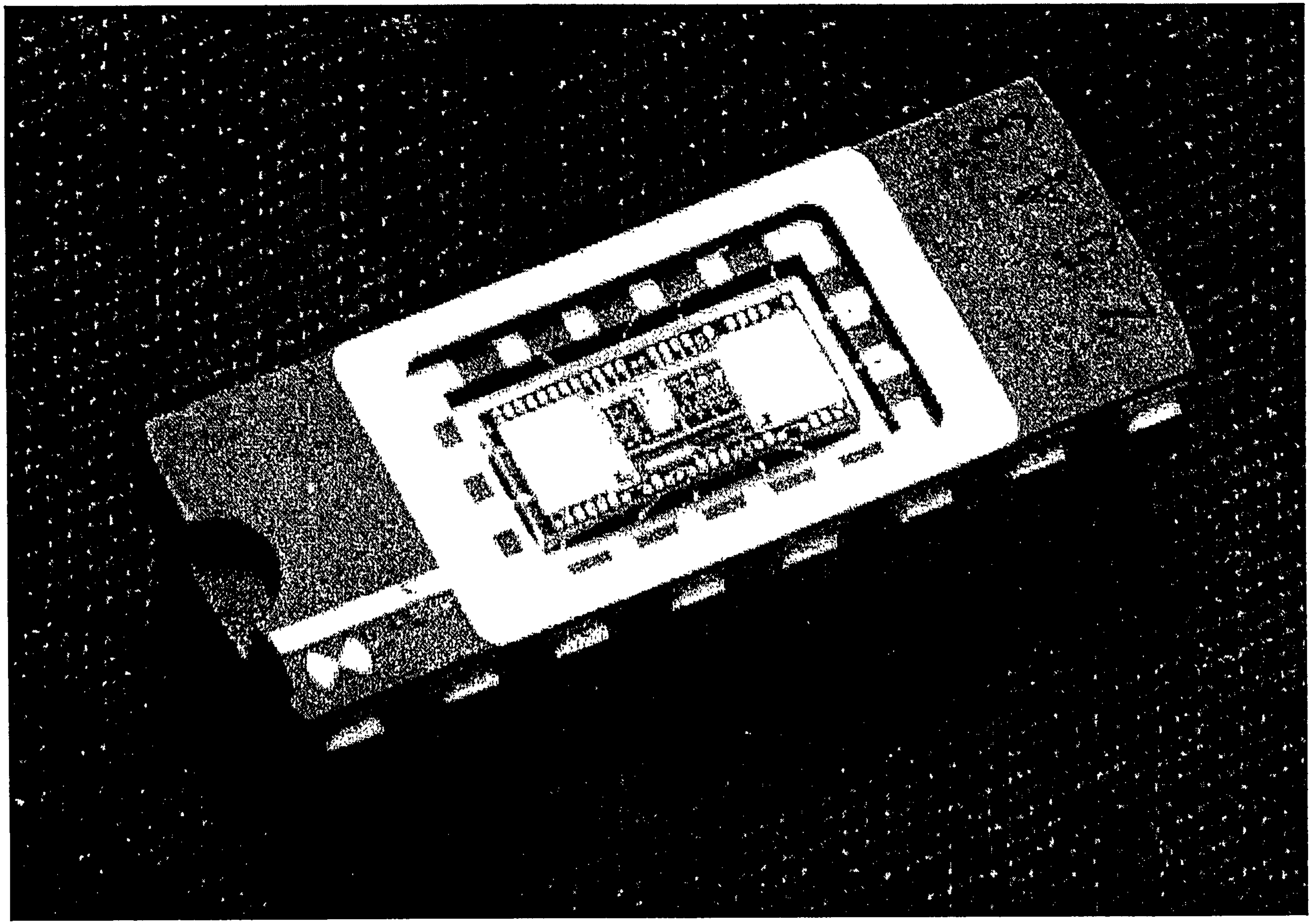


Figure 1

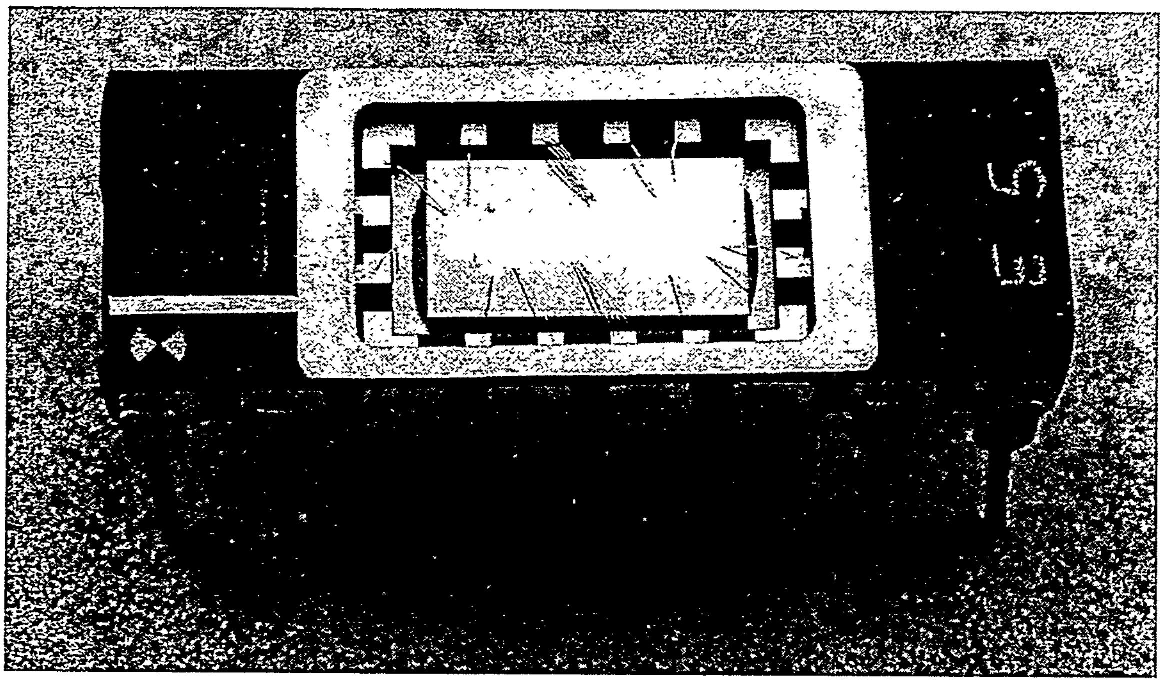
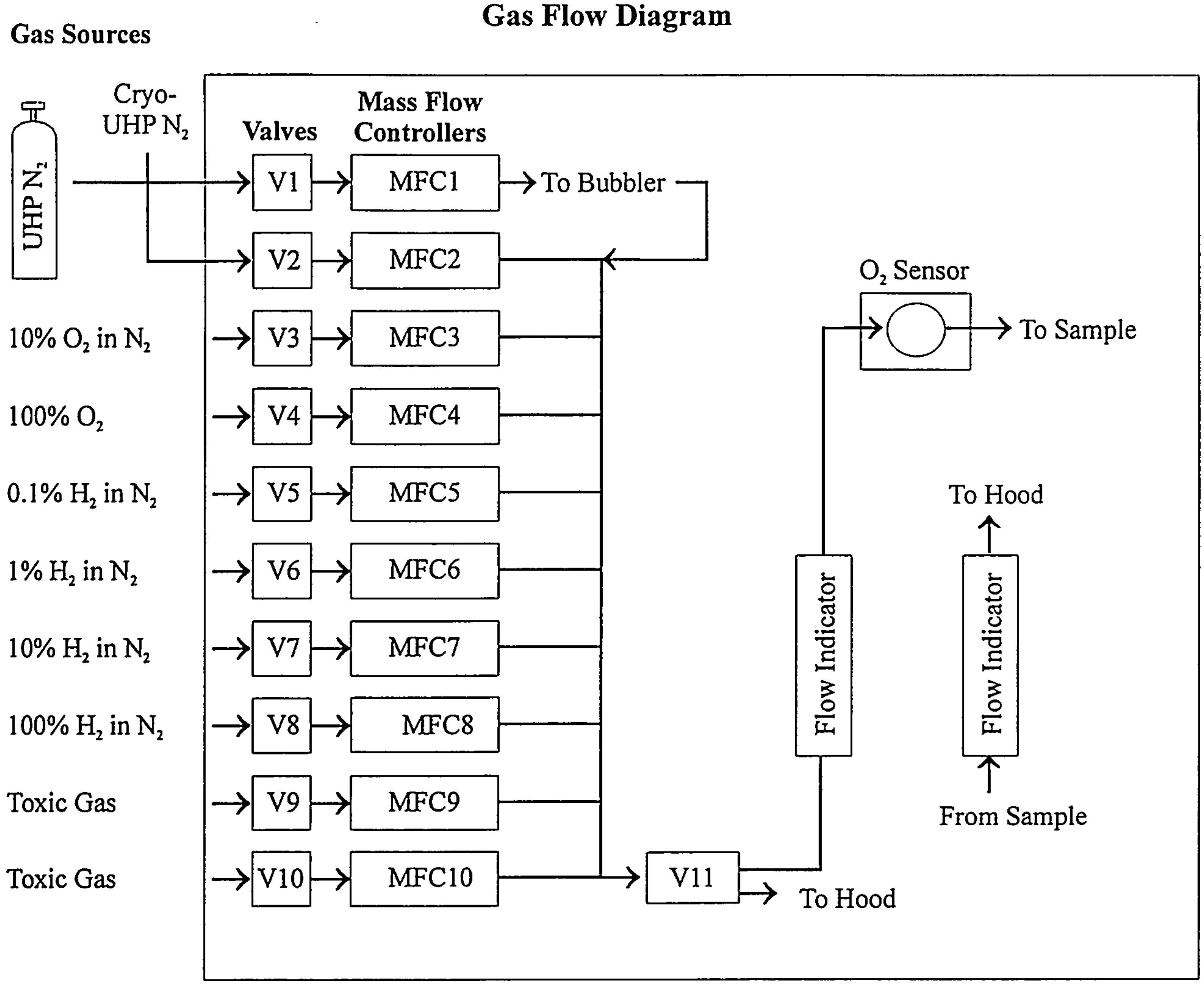


Figure 2

Gas Test Bed #1
Gas Flow Diagram



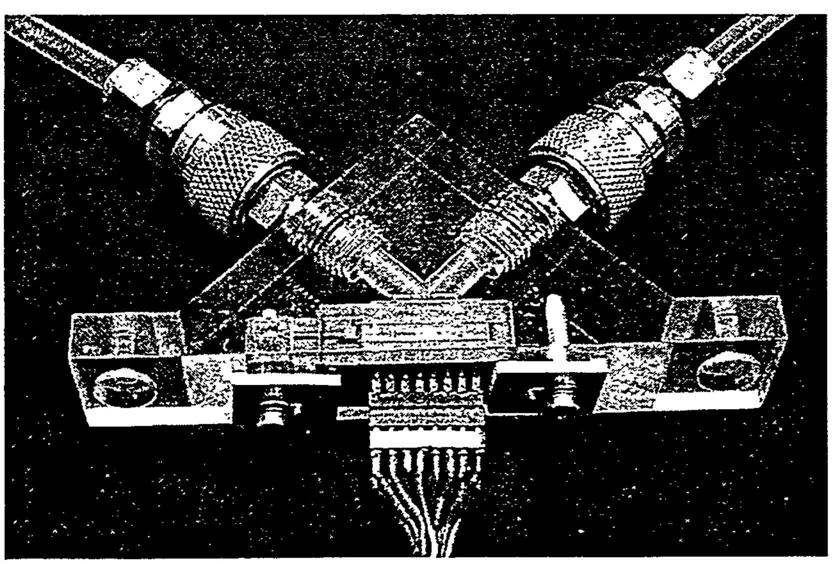


Figure 3

3N2 - Unpurified N2 (ppm)
5-7-97
Measured Impurities

Time	O2	H2O	THC	CO
2pm	0.02	0.06	0.01	0.01
1pm	0.06	0.06	0.01	0.01
12pm	0.06	0.06	0.01	0.01
11am	0.05	0.06	0.01	0.01
10am	0.01	0.06	0.01	0.01
9am	0	0.06	0.01	0.01
8am	0.01	0.06	0.01	0.01
7am	0	0.06	0.01	0.01
6am	0	0.06	0.01	0.01
5am	0	0.06	0	0.01
4am	0.01	0.06	0	0.01
3am	0	0.06	0	0.01
2am	0.01	0.06	0	0.01
1am	0.01	0.06	0	0.01
12pm	0	0.06	0.01	0.01
11pm	0	0.07	0	0.01
10pm	0.02	0.06	0	0.01
9pm	0.02	0.06	0	0.01
8pm	0.02	0.06	0.01	0.01
7pm	0.02	0.06	0	0.01
6pm	0.02	0.06	0	0.01
5pm	0.02	0.06	0	0.01
4pm	0.02	0.06	0	0.01
3pm	0.06	0.06	0	0.01

Tabl

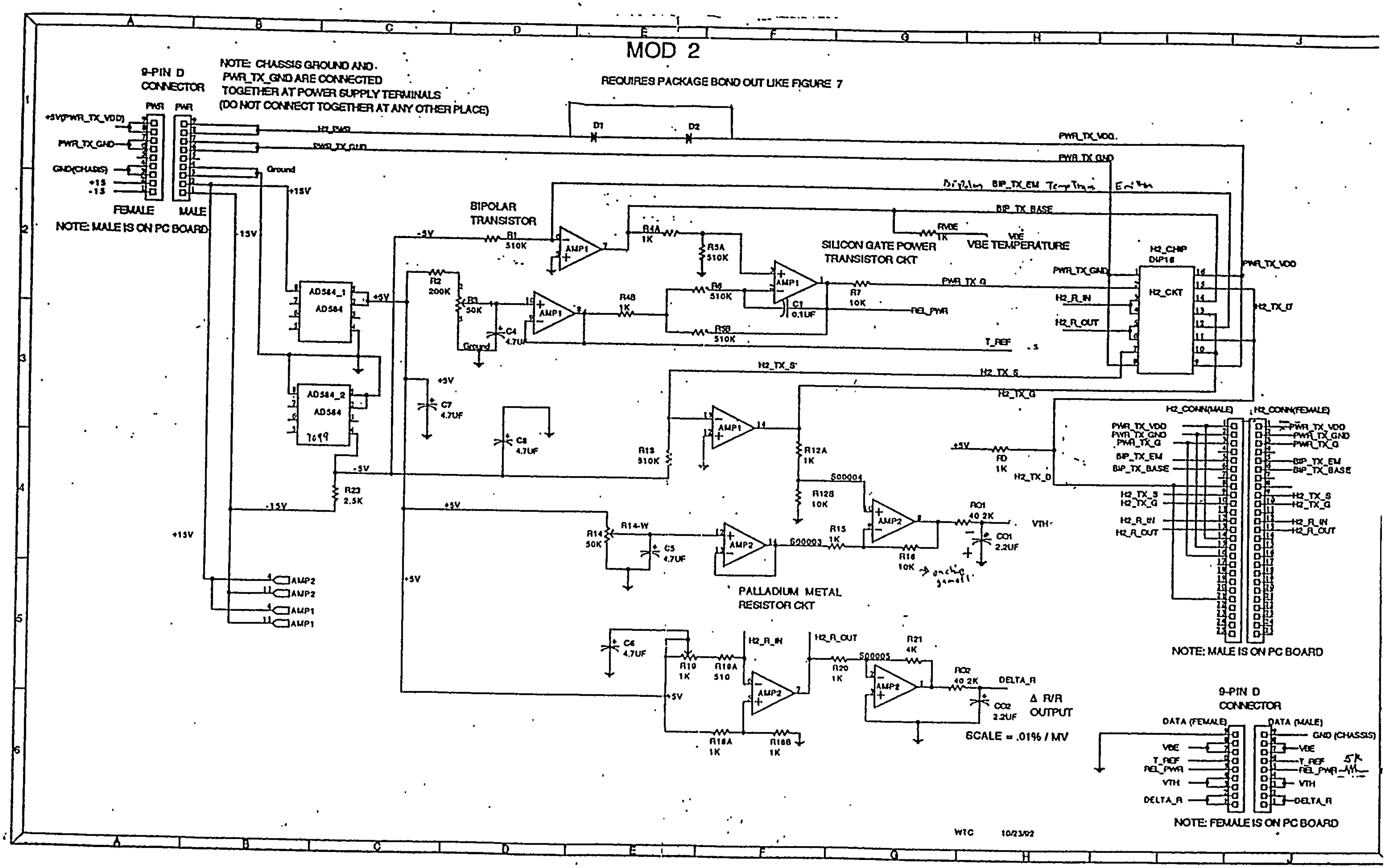

MOD 2
REQUIRES PACKAGE BOND OUT LIKE FIGURE 7
9-PIN D CONNECTOR
NOTE: CHASSIS GROUND AND PWR_TX_GND ARE CONNECTED TOGETHER AT POWER SUPPLY TERMINALS (DO NOT CONNECT TOGETHER AT ANY OTHER PLACE)
FEMALE
MALE
NOTE: MALE IS ON PC BOARD
BIPOLAR TRANSISTOR
SILICON GATE POWER TRANSISTOR CKT
VBE TEMPERATURE
PALLADIUM METAL RESISTOR CKT
Δ R/R OUTPUT
SCALE = .01% / MV
NOTE: MALE IS ON PC BOARD
9-PIN D CONNECTOR
NOTE: FEMALE IS ON PC BOARD
WTC
10/23/02

Figure 4

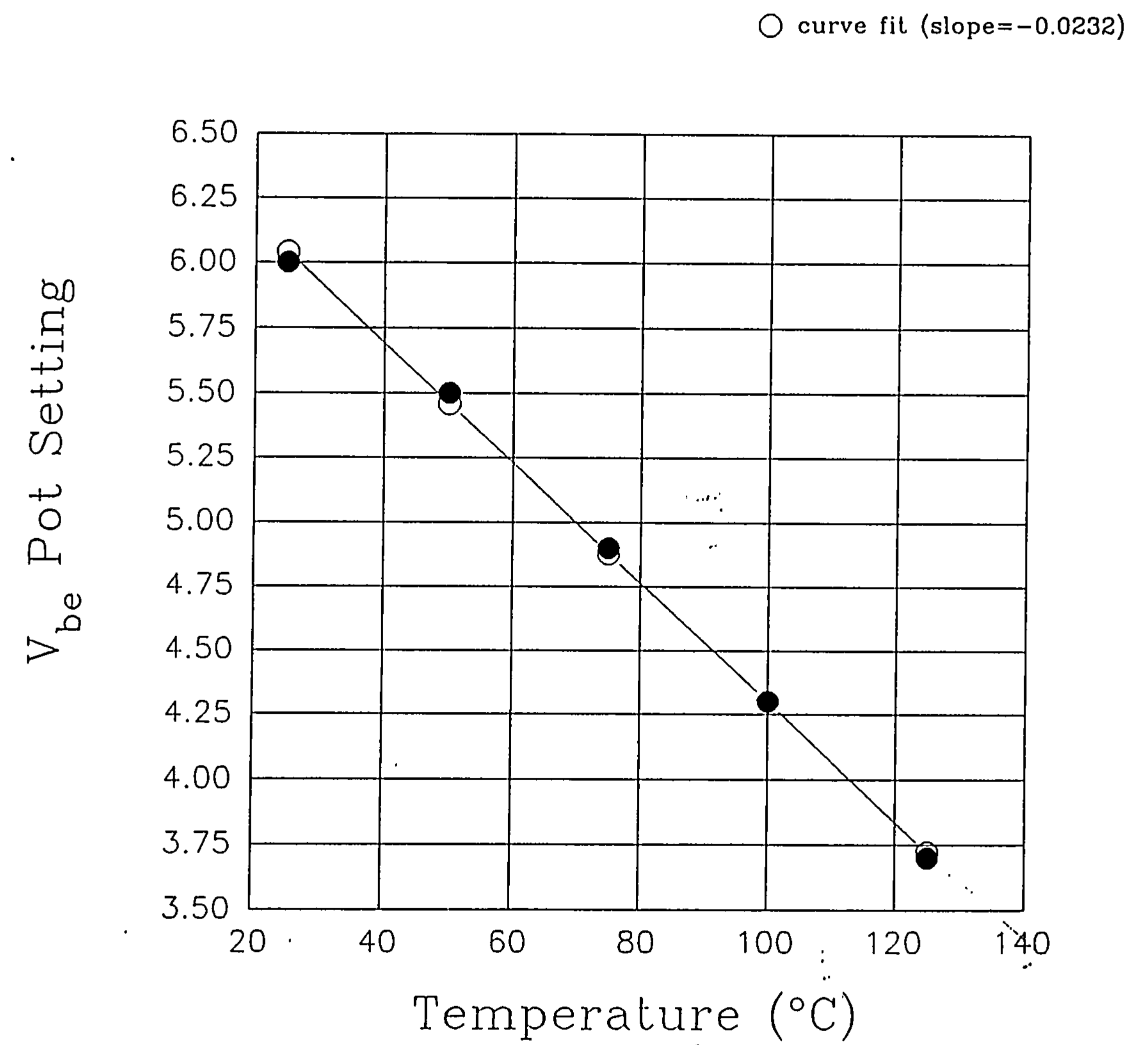
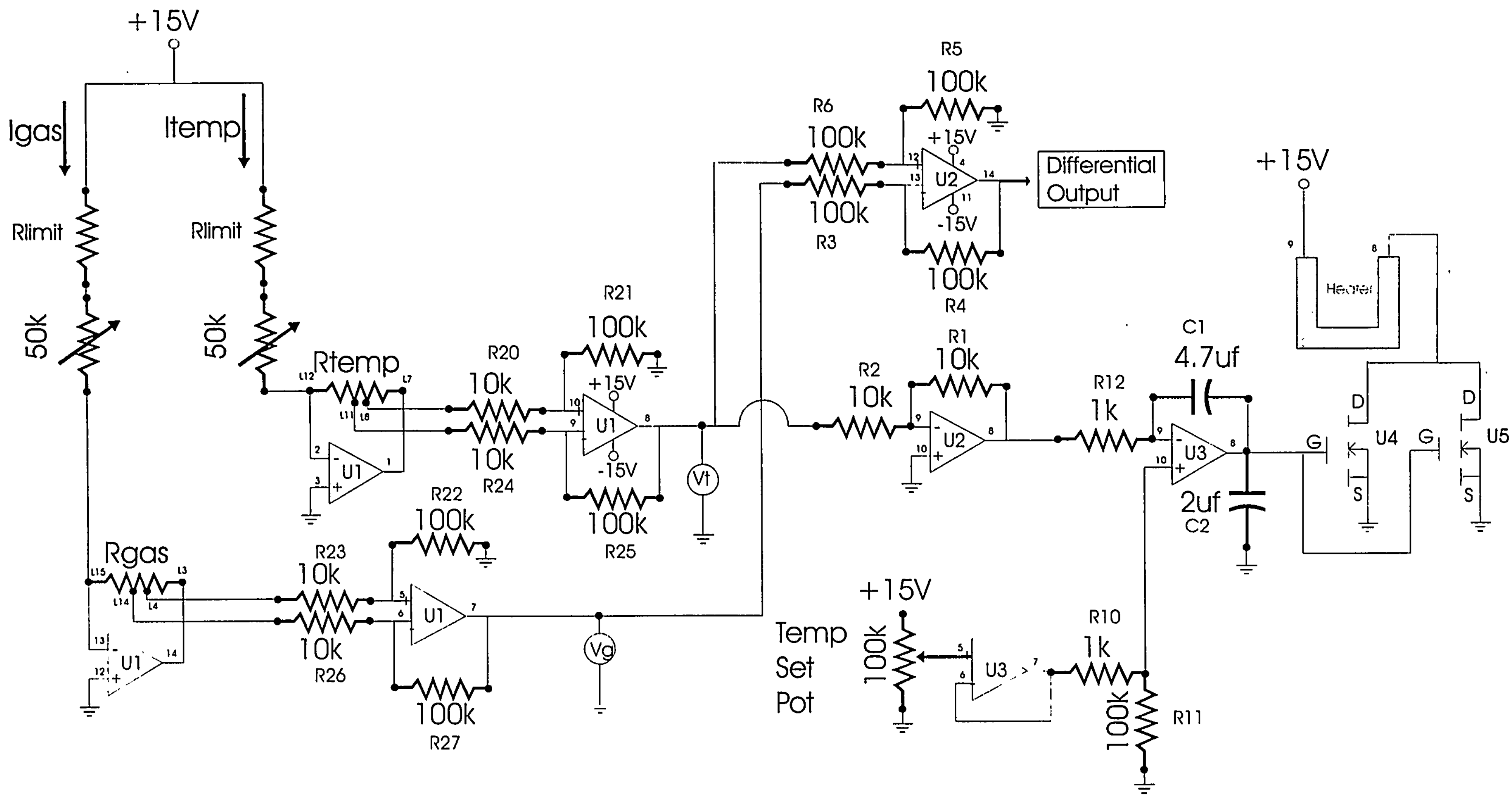
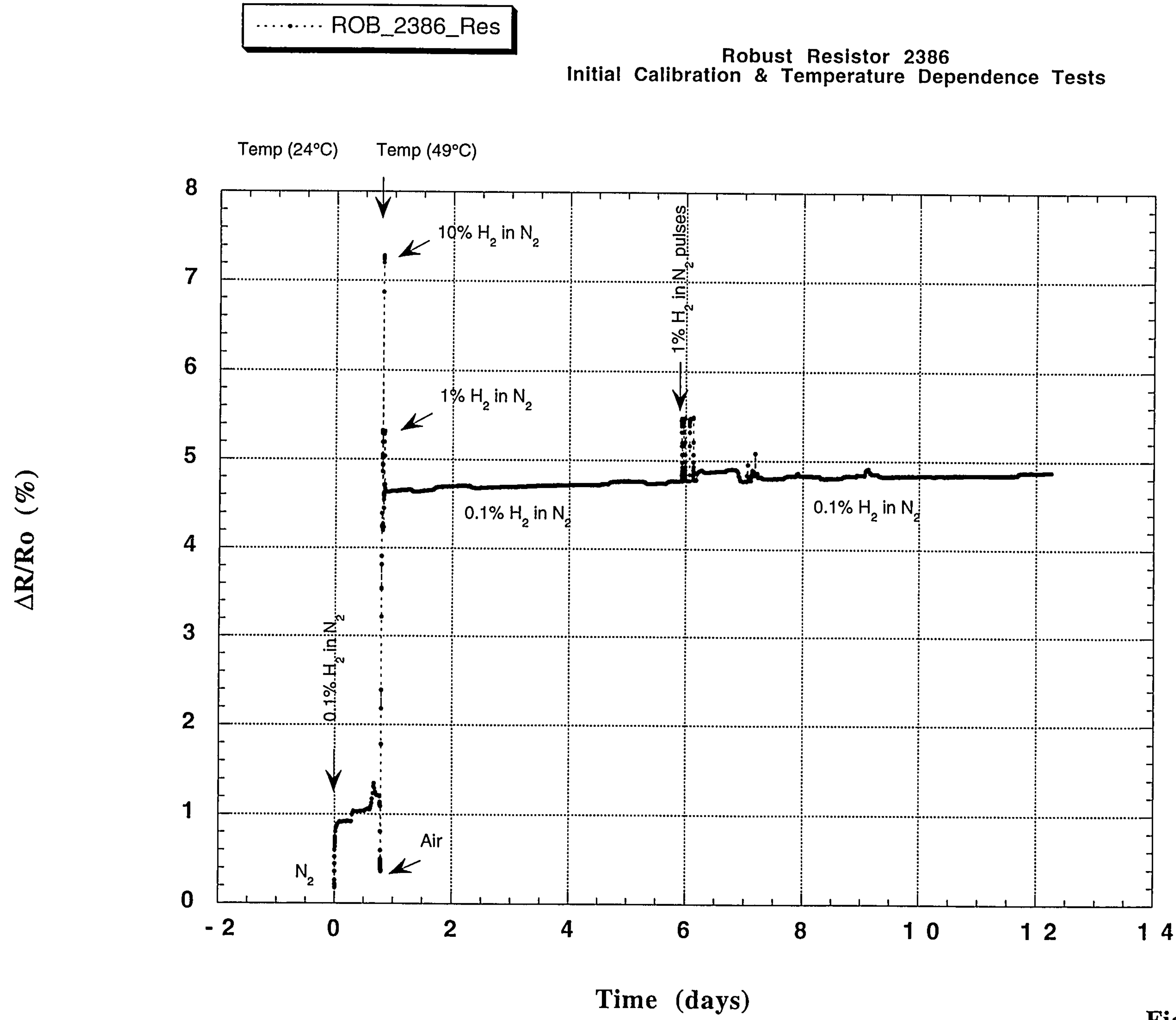

○ curve fit (slope=-0.0232)
6.50
6.25
6.00
5.75
5.50
5.25
5.00
4.75
4.50
4.25
4.00
3.75
3.50
V_{be} Pot Setting
20
40
60
80
100
120
140
Temperature (°C)

Figure 5



Battery Powered, Differential Output, Temperature Compensation Circuit
Mark Jenkins and Daniel Moreno
2/29/96 (revision 1) U1-U3 LP324, U4&U5 MMSF5N02HDR2

Figure 6



ROB_2386_Res
Robust Resistor 2386
Initial Calibration & Temperature Dependence Tests
Temp (24°C)
Temp (49°C)
10% H2 in N2
1% H2 in N2
1% H2 in N2 pulses
0.1% H2 in N2
0.1% H2 in N2
0.1% H2 in N2
Air
N2
ΔR/Ro (%)
Time (days)
8
7
6
5
4
3
2
1
0
-2
0
2
4
6
8
10
12
14

Figure 7

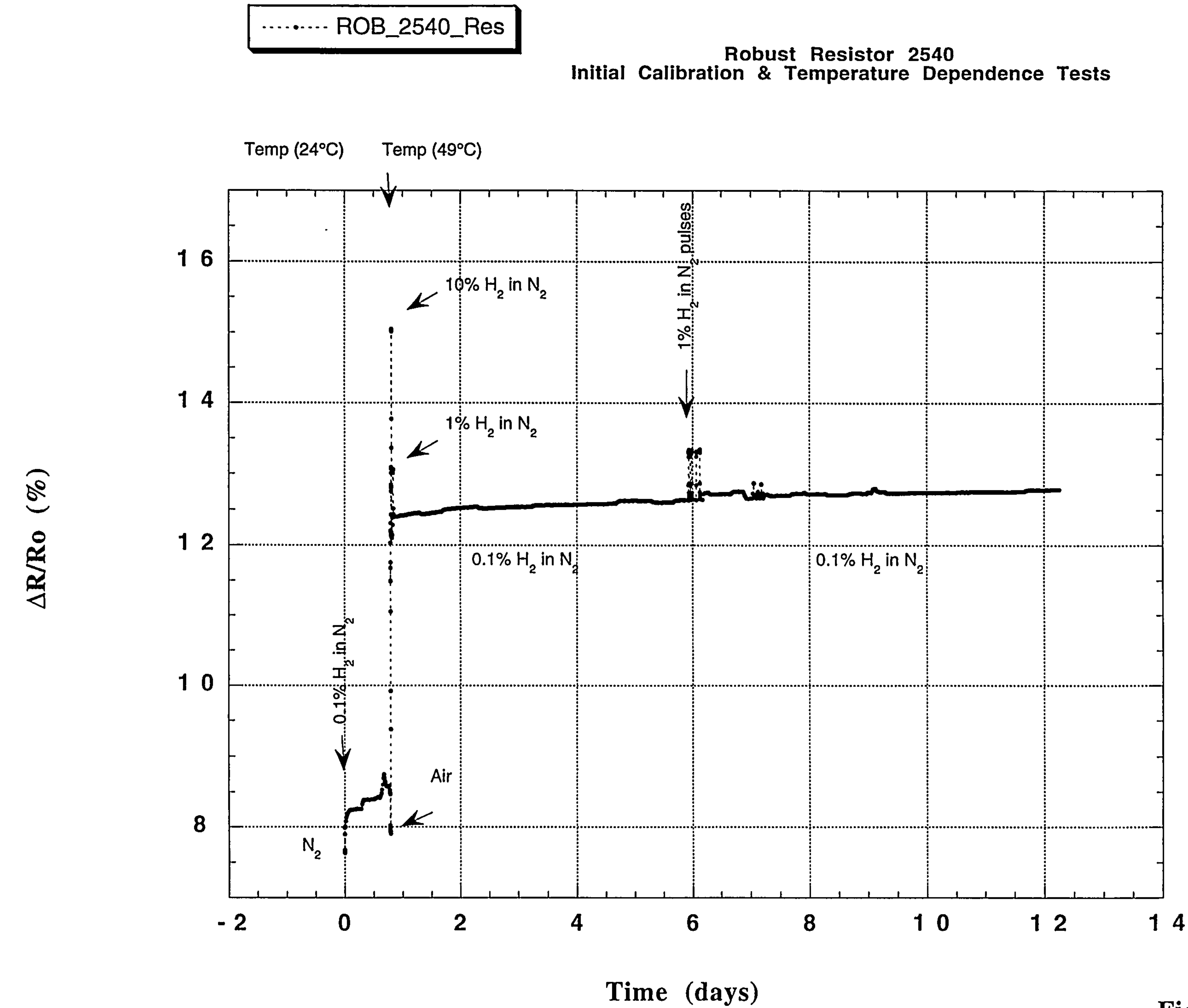
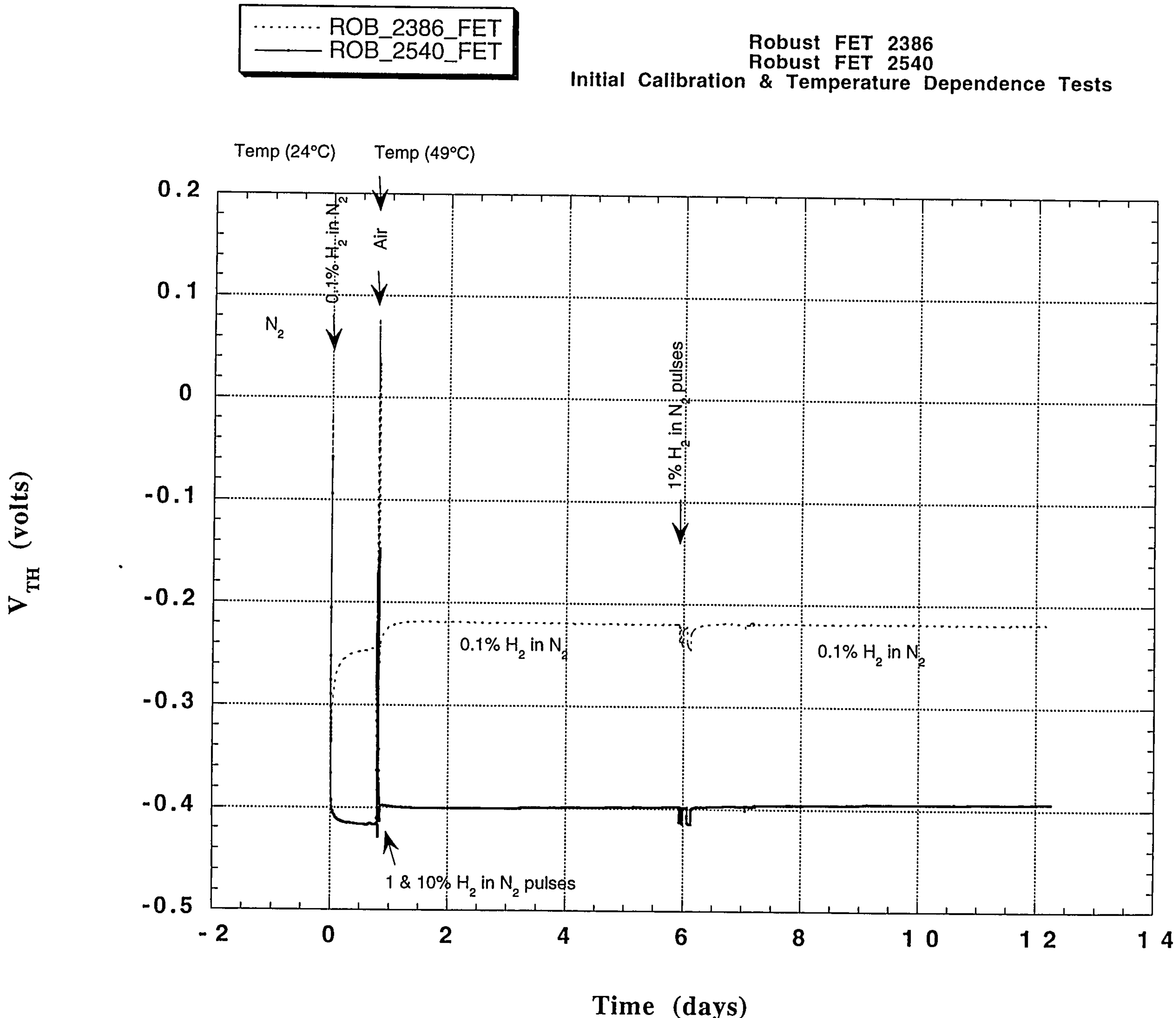

ROB_2540_Res
Robust Resistor 2540
Initial Calibration & Temperature Dependence Tests
Temp (24°C)
Temp (49°C)
10% H_2 in N_2
1% H_2 in N_2
1% H_2 in N_2 pulses
0.1% H_2 in N_2
0.1% H_2 in N_2
0.1% H_2 in N_2
Air
N_2
16
14
12
10
8
-2
0
2
4
6
8
10
12
14
ΔR/Ro (%)
Time (days)

Figure 8



ROB_2386_FET
ROB_2540_FET
Robust FET 2386
Robust FET 2540
Initial Calibration & Temperature Dependence Tests
Temp (24°C)
Temp (49°C)
0.1% H_2 in N_2
Air
N_2
1% H_2 in N_2 pulses
0.1% H_2 in N_2
0.1% H_2 in N_2
1 & 10% H_2 in N_2 pulses
0.2
0.1
0
-0.1
-0.2
-0.3
-0.4
-0.5
-2
0
2
4
6
8
10
12
14
V_{TH} (volts)
Time (days)

Figure 9

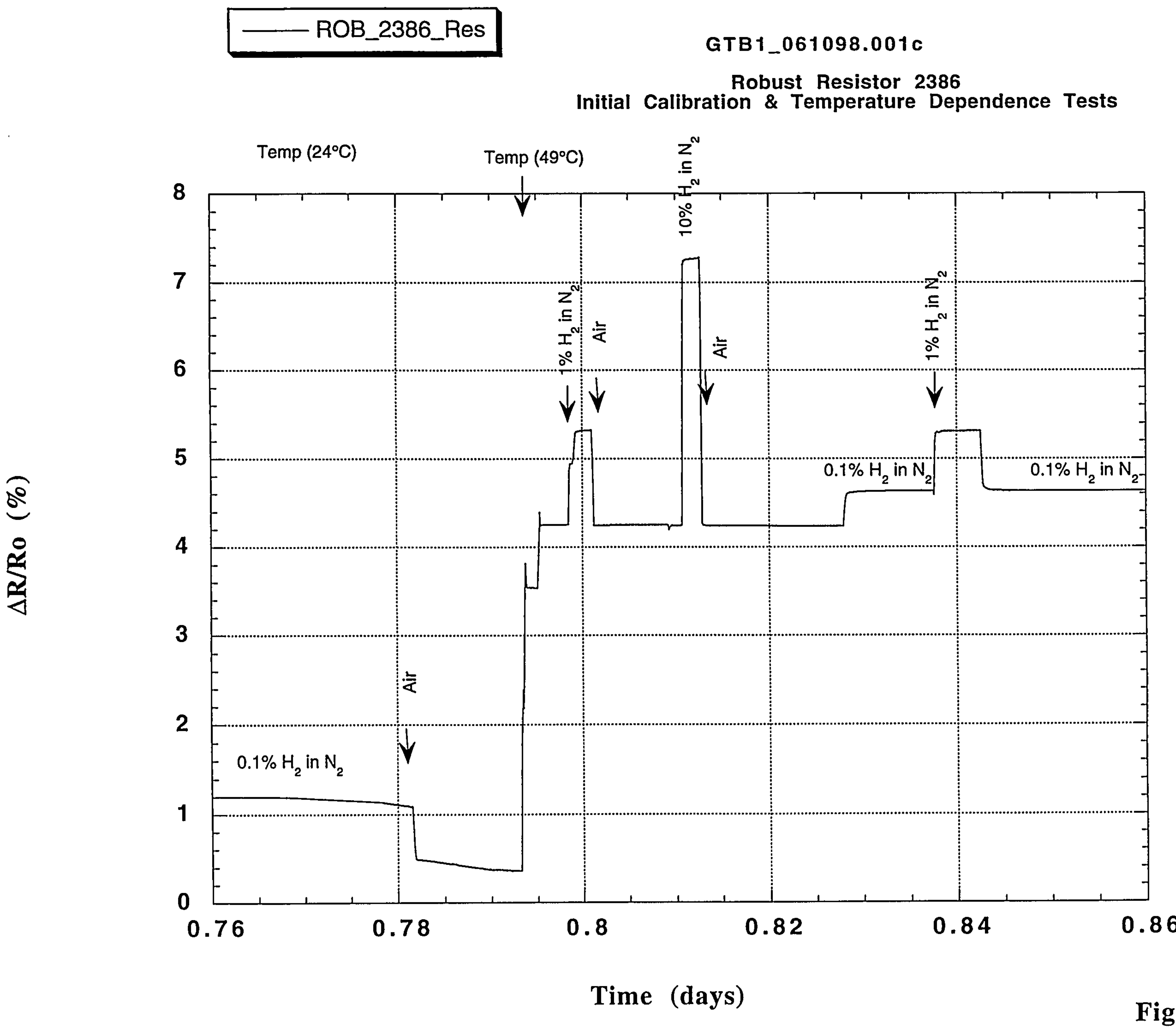

ROB_2386_Res
GTB1_061098.001c
Robust Resistor 2386
Initial Calibration & Temperature Dependence Tests
Temp (24°C)
Temp (49°C)
0.1% H_2 in N_2
Air
1% H_2 in N_2
Air
10% H_2 in N_2
Air
0.1% H_2 in N_2
1% H_2 in N_2
0.1% H_2 in N_2
ΔR/Ro (%)
8
7
6
5
4
3
2
1
0
0.76
0.78
0.8
0.82
0.84
0.86
Time (days)

Figure 10

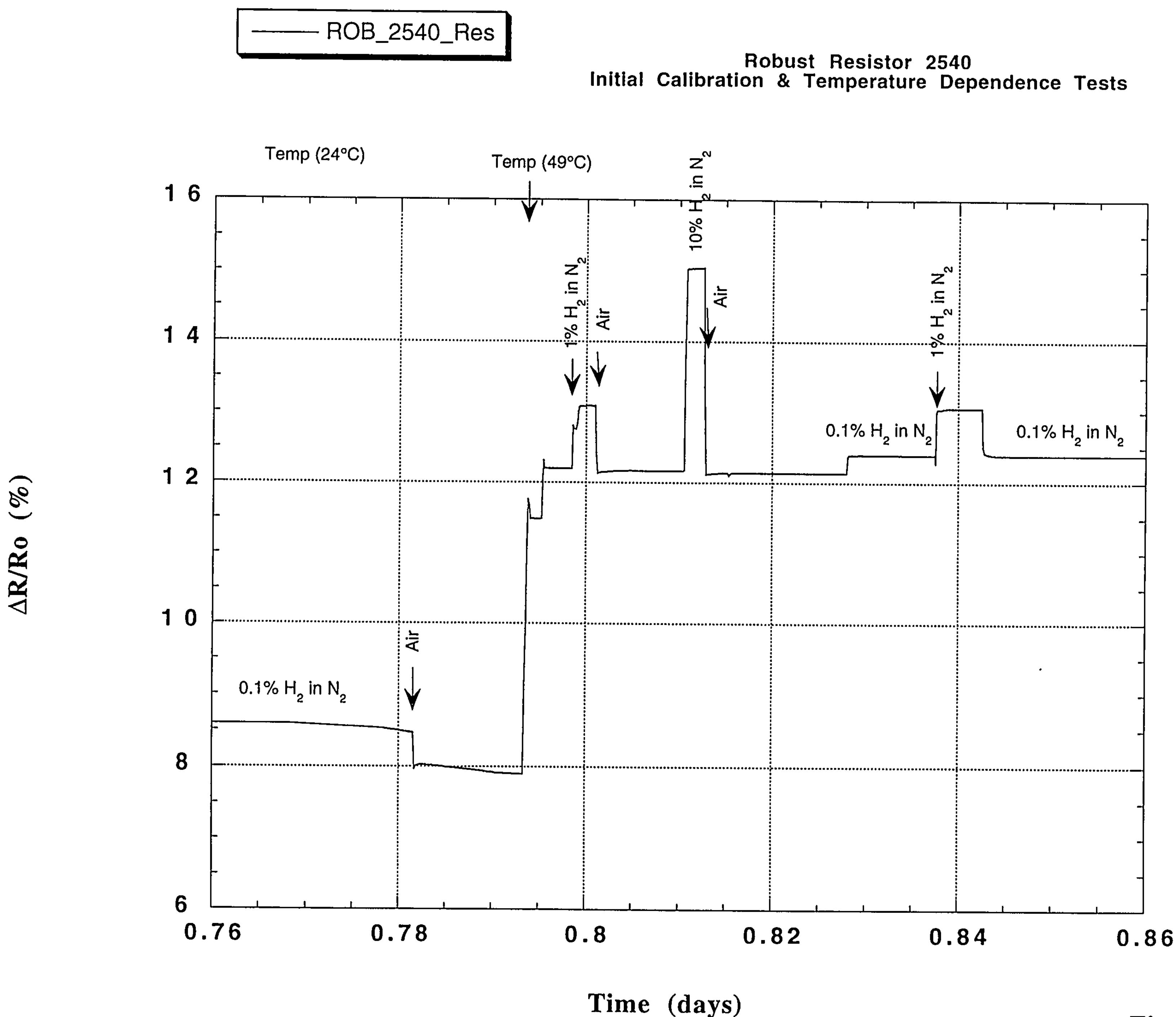

ROB_2540_Res
Robust Resistor 2540
Initial Calibration & Temperature Dependence Tests
Temp (24°C)
Temp (49°C)
1% H_2 in N_2
Air
10% H_2 in N_2
Air
1% H_2 in N_2
0.1% H_2 in N_2
0.1% H_2 in N_2
0.1% H_2 in N_2
Air
ΔR/Ro (%)
16
14
12
10
8
6
0.76
0.78
0.8
0.82
0.84
0.86
Time (days)

Figure 11

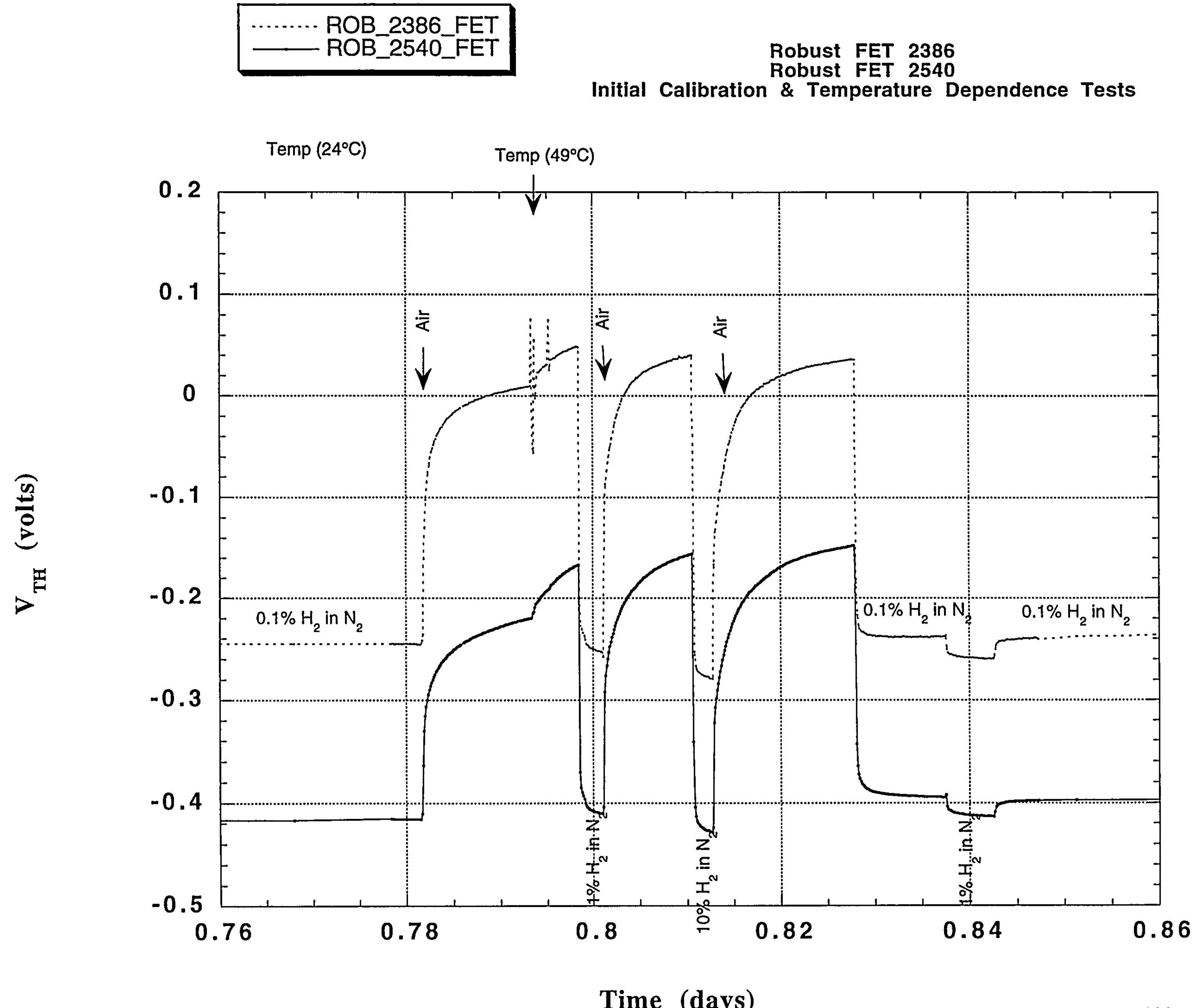

ROB_2386_FET
ROB_2540_FET
Robust FET 2386
Robust FET 2540
Initial Calibration & Temperature Dependence Tests
Temp (24°C)
Temp (49°C)
Air
Air
Air
0.1% H_2 in N_2
0.1% H_2 in N_2
0.1% H_2 in N_2
1% H_2 in N_2
10% H_2 in N_2
1% H_2 in N_2
0.2
0.1
0
-0.1
-0.2
-0.3
-0.4
-0.5
0.76
0.78
0.8
0.82
0.84
0.86
V_{TH} (volts)
Time (days)

Figure 12

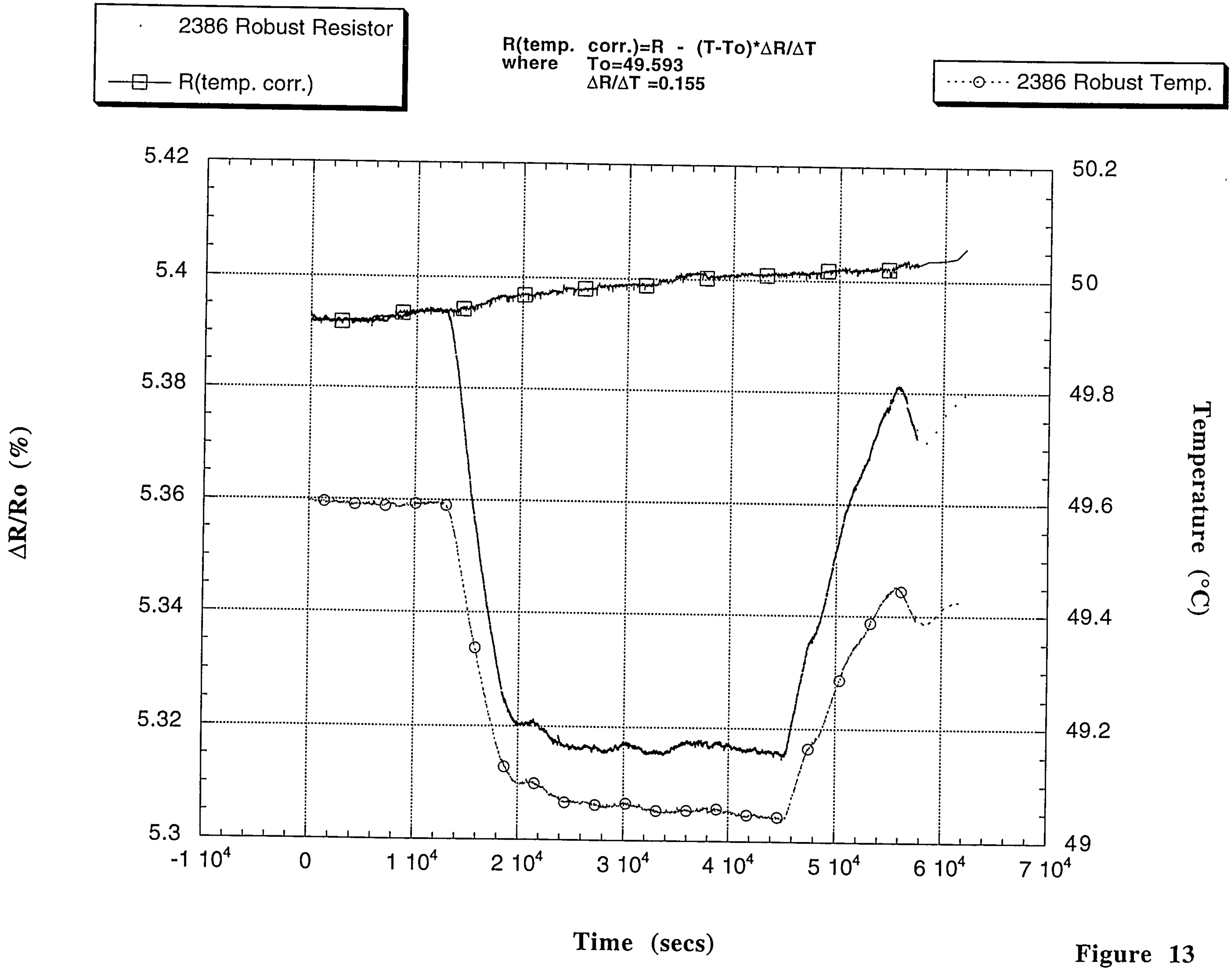
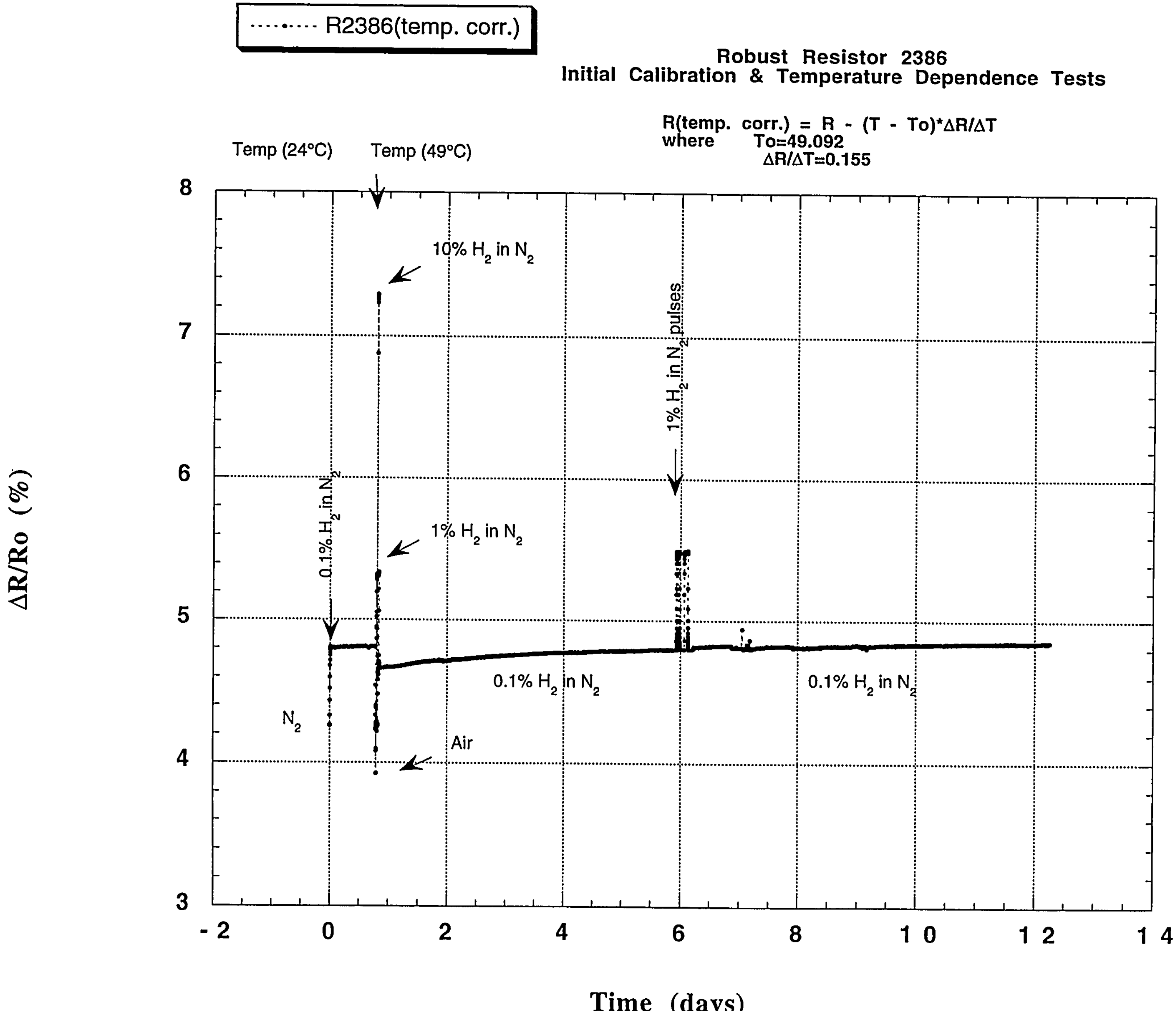

2386 Robust Resistor
R(temp. corr.)
R(temp. corr.)=R - (T-To)*ΔR/ΔT
where To=49.593
ΔR/ΔT =0.155
2386 Robust Temp.
5.42
5.4
5.38
5.36
5.34
5.32
5.3
ΔR/Ro (%)
50.2
50
49.8
49.6
49.4
49.2
49
Temperature (°C)
-1 10^4
0
1 10^4
2 10^4
3 10^4
4 10^4
5 10^4
6 10^4
7 10^4
Time (secs)

Figure 13



R2386(temp. corr.)
Robust Resistor 2386
Initial Calibration & Temperature Dependence Tests
R(temp. corr.) = R - (T - To)*ΔR/ΔT
where To=49.092
ΔR/ΔT=0.155
Temp (24°C)
Temp (49°C)
10% H2 in N2
1% H2 in N2 pulses
0.1% H2 in N2
1% H2 in N2
0.1% H2 in N2
0.1% H2 in N2
N2
Air
ΔR/Ro (%)
Time (days)
8
7
6
5
4
3
-2
0
2
4
6
8
10
12
14

Figure 14

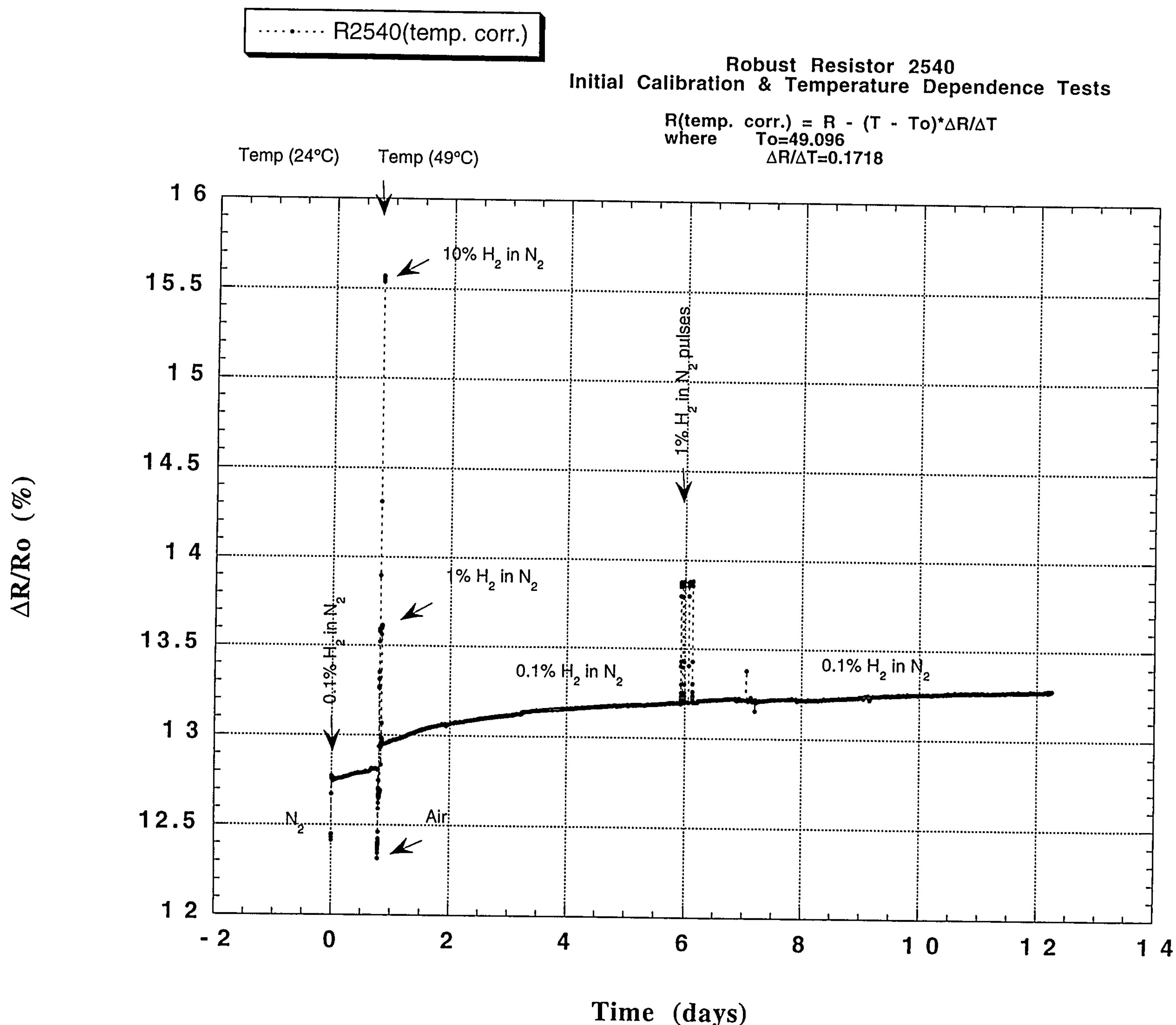
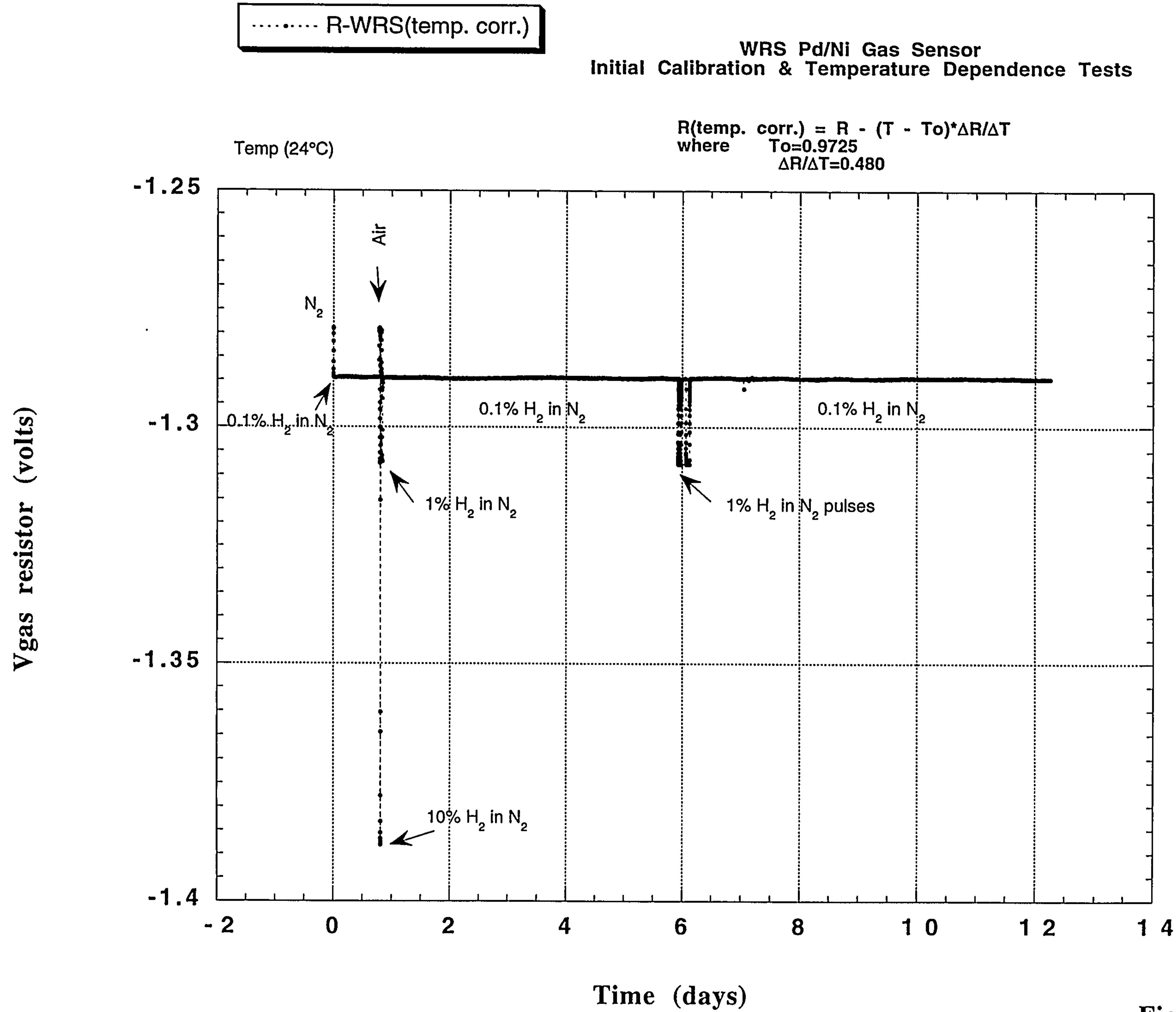

R2540(temp. corr.)
Robust Resistor 2540
Initial Calibration & Temperature Dependence Tests
R(temp. corr.) = R - (T - To)*ΔR/ΔT
where To=49.096
ΔR/ΔT=0.1718
Temp (24°C)
Temp (49°C)
10% H2 in N2
1% H2 in N2 pulses
1% H2 in N2
0.1% H2 in N2
0.1% H2 in N2
0.1% H2 in N2
N2
Air
16
15.5
15
14.5
14
13.5
13
12.5
12
-2
0
2
4
6
8
10
12
14
ΔR/Ro (%)
Time (days)

Figure 15



R-WRS(temp. corr.)
WRS Pd/Ni Gas Sensor
Initial Calibration & Temperature Dependence Tests
R(temp. corr.) = R - (T - To)*ΔR/ΔT
where To=0.9725
ΔR/ΔT=0.480
Temp (24°C)
-1.25
-1.3
-1.35
-1.4
-2
0
2
4
6
8
10
12
14
Vgas resistor (volts)
Time (days)
N_2
Air
0.1% H_2 in N_2
0.1% H_2 in N_2
0.1% H_2 in N_2
1% H_2 in N_2
1% H_2 in N_2 pulses
10% H_2 in N_2

Figure 16

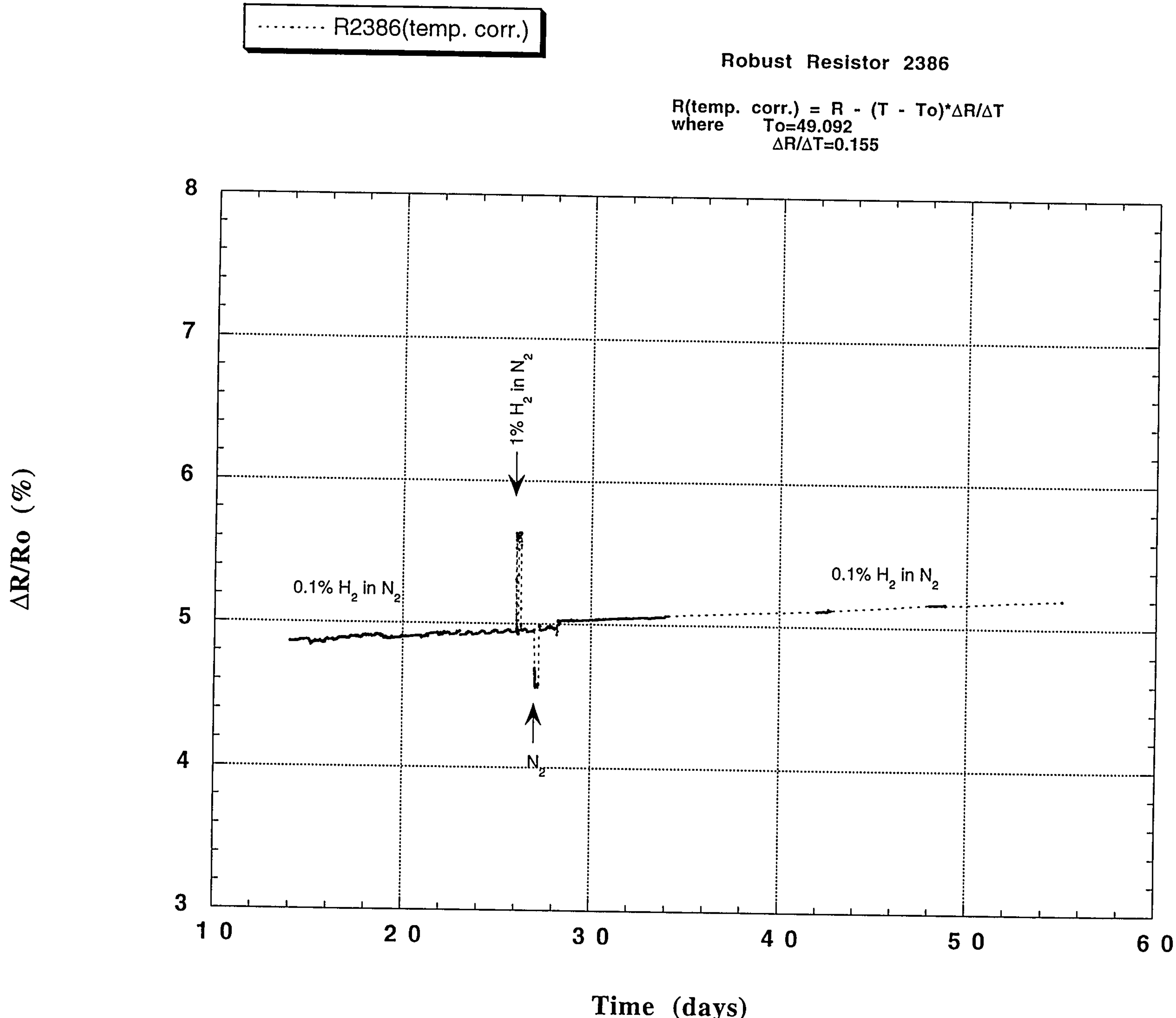
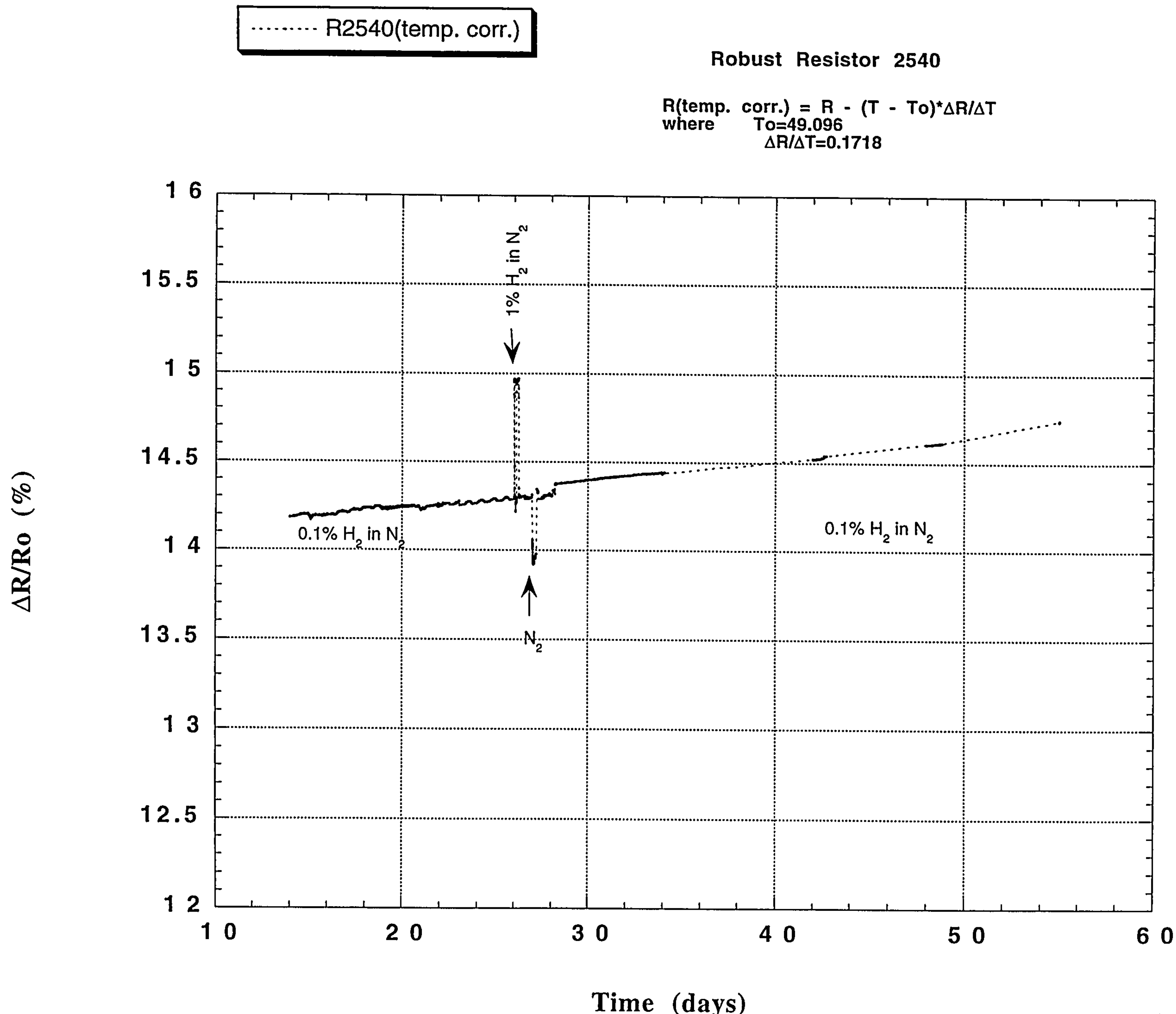
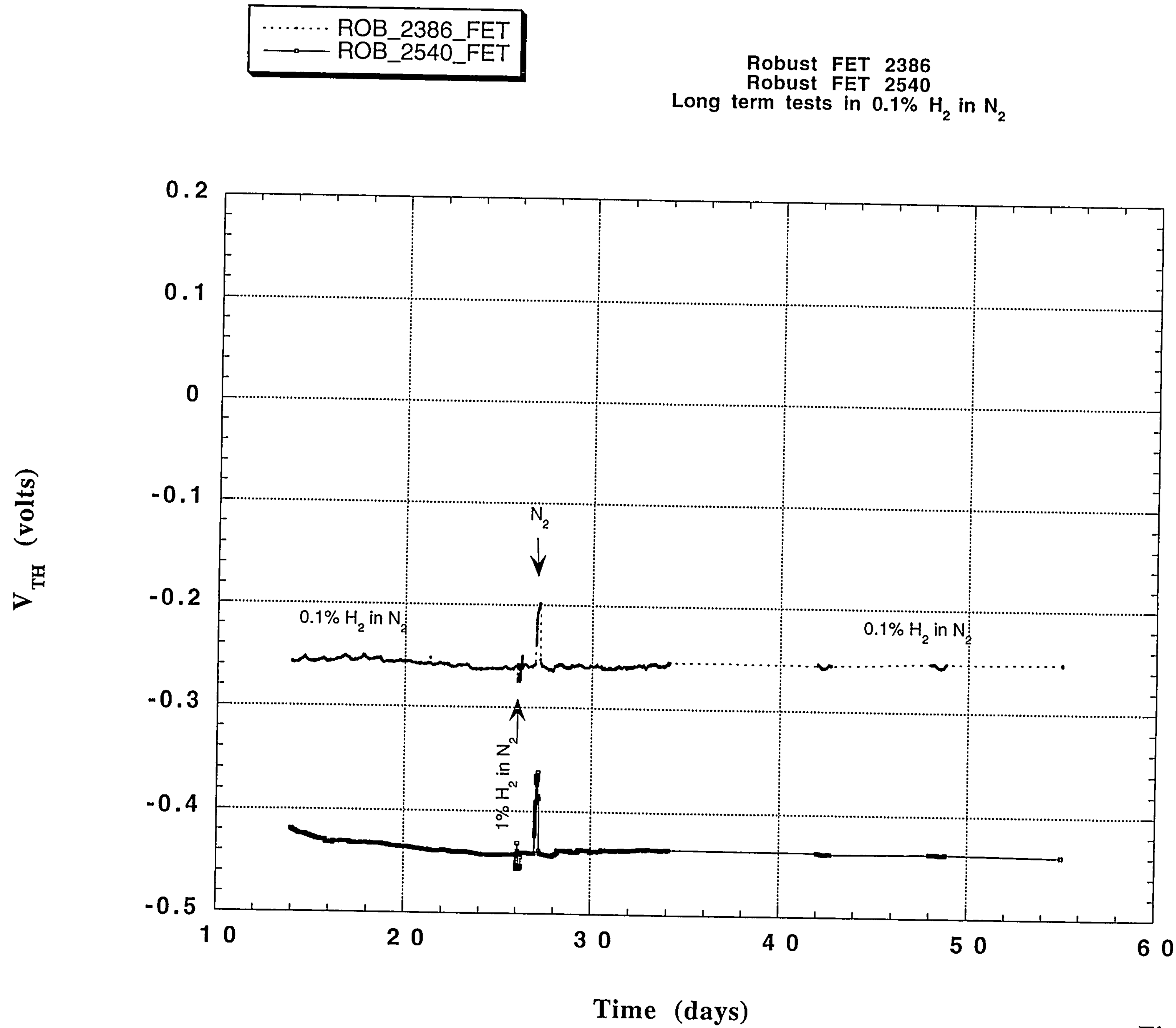

R2386(temp. corr.)
Robust Resistor 2386
R(temp. corr.) = R - (T - To)*ΔR/ΔT
where To=49.092
ΔR/ΔT=0.155
8
7
6
5
4
3
1 0
2 0
3 0
4 0
5 0
6 0
0.1% H2 in N2
1% H2 in N2
N2
0.1% H2 in N2
ΔR/Ro (%)
Time (days)

Figure 17



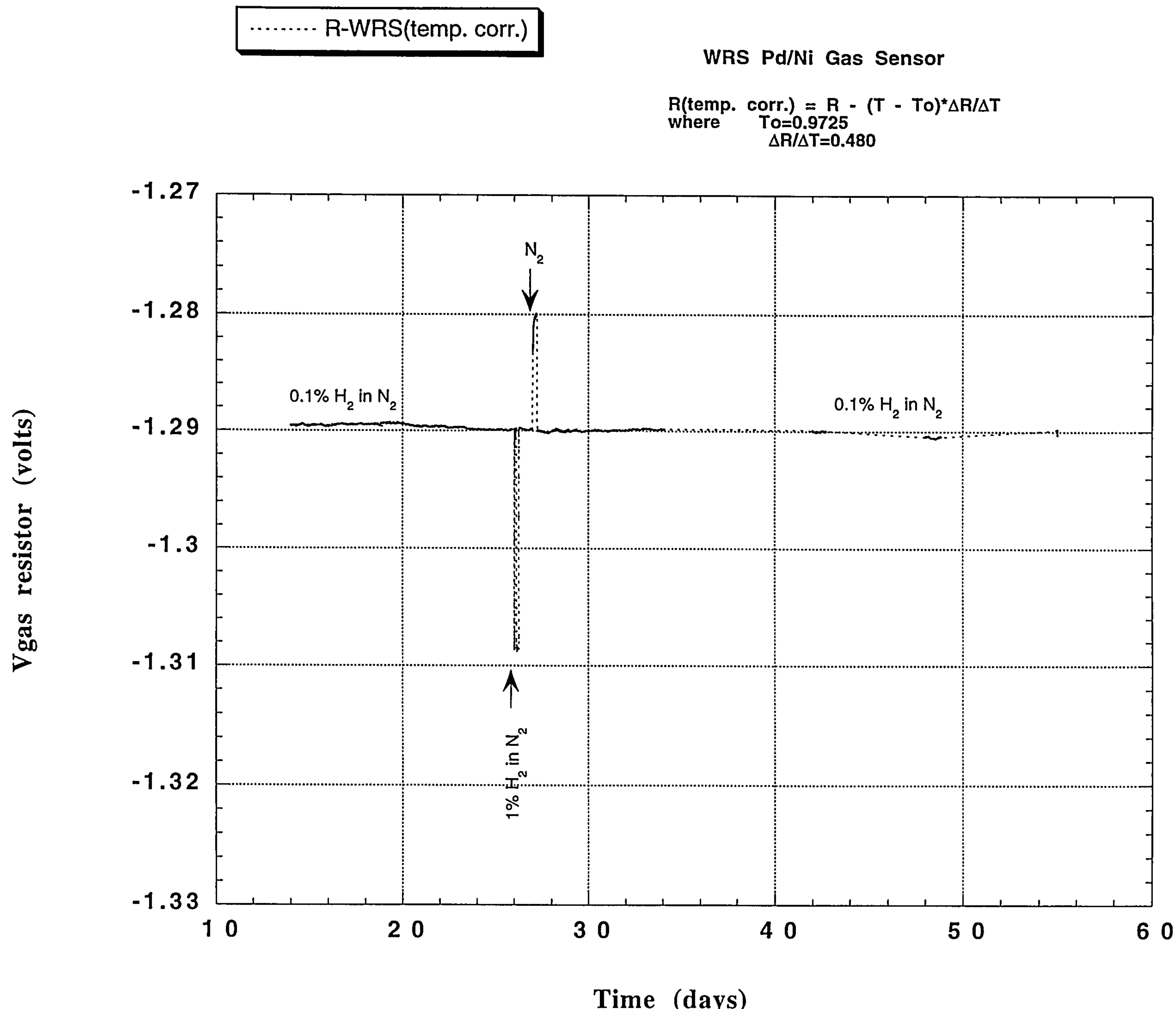
R2540(temp. corr.)
Robust Resistor 2540
R(temp. corr.) = R - (T - To)*ΔR/ΔT
where To=49.096
ΔR/ΔT=0.1718
1% H_2 in N_2
N_2
0.1% H_2 in N_2
0.1% H_2 in N_2
ΔR/Ro (%)
Time (days)

Figure 18



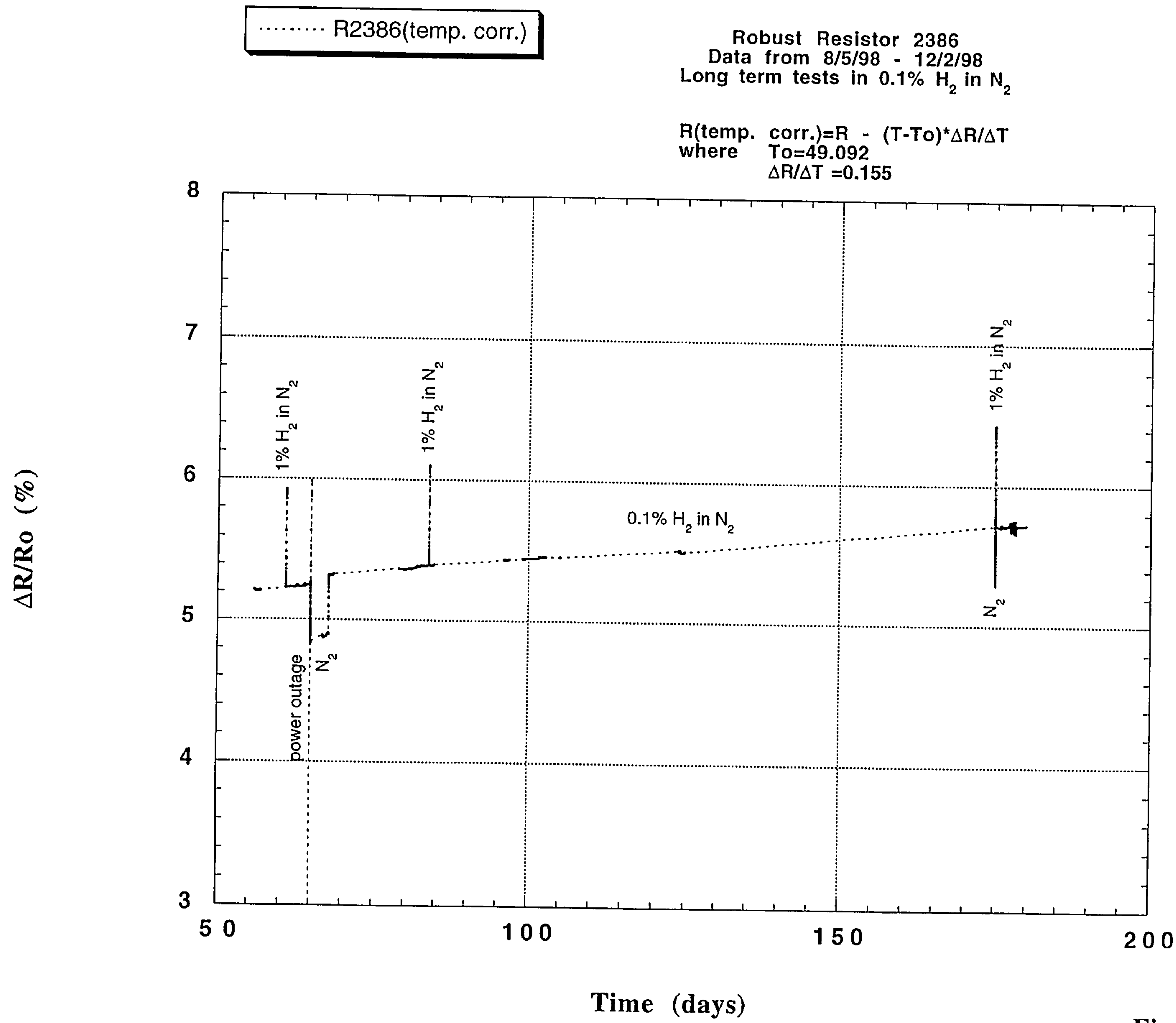
ROB_2386_FET
ROB_2540_FET
Robust FET 2386
Robust FET 2540
Long term tests in 0.1% H_2 in N_2
0.2
0.1
0
-0.1
-0.2
-0.3
-0.4
-0.5
V_{TH} (volts)
N_2
0.1% H_2 in N_2
0.1% H_2 in N_2
1% H_2 in N_2
10
20
30
40
50
60
Time (days)

Figure 19



R-WRS(temp. corr.)
WRS Pd/Ni Gas Sensor
R(temp. corr.) = R - (T - To)*ΔR/ΔT
where To=0.9725
ΔR/ΔT=0.480
-1.27
-1.28
-1.29
-1.3
-1.31
-1.32
-1.33
10
20
30
40
50
60
N2
0.1% H2 in N2
0.1% H2 in N2
1% H2 in N2
Vgas resistor (volts)
Time (days)

Figure 20



R2386(temp. corr.)
Robust Resistor 2386
Data from 8/5/98 - 12/2/98
Long term tests in 0.1% H_2 in N_2
R(temp. corr.)=R - (T-To)*ΔR/ΔT
where To=49.092
ΔR/ΔT =0.155
1% H_2 in N_2
1% H_2 in N_2
1% H_2 in N_2
0.1% H_2 in N_2
power outage
N_2
N_2
8
7
6
5
4
3
50
100
150
200
ΔR/Ro (%)
Time (days)

Figure 21

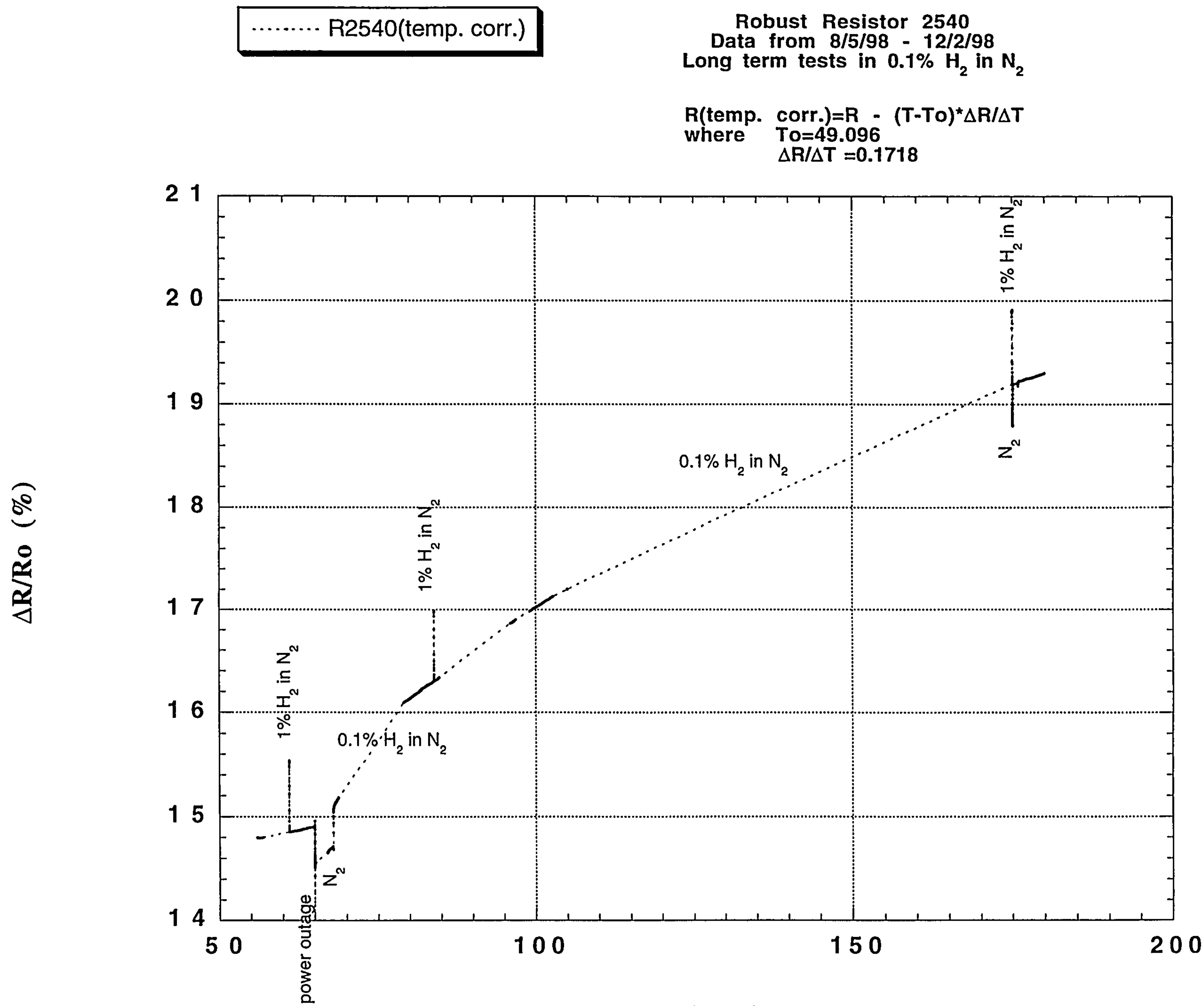

R2540(temp. corr.)
Robust Resistor 2540
Data from 8/5/98 - 12/2/98
Long term tests in 0.1% H_2 in N_2
R(temp. corr.)=R - (T-To)*ΔR/ΔT
where To=49.096
ΔR/ΔT =0.1718
1% H_2 in N_2
power outage
N_2
0.1% H_2 in N_2
1% H_2 in N_2
0.1% H_2 in N_2
1% H_2 in N_2
N_2
ΔR/Ro (%)
Time (days)
21
20
19
18
17
16
15
14
50
100
150
200

Figure 22

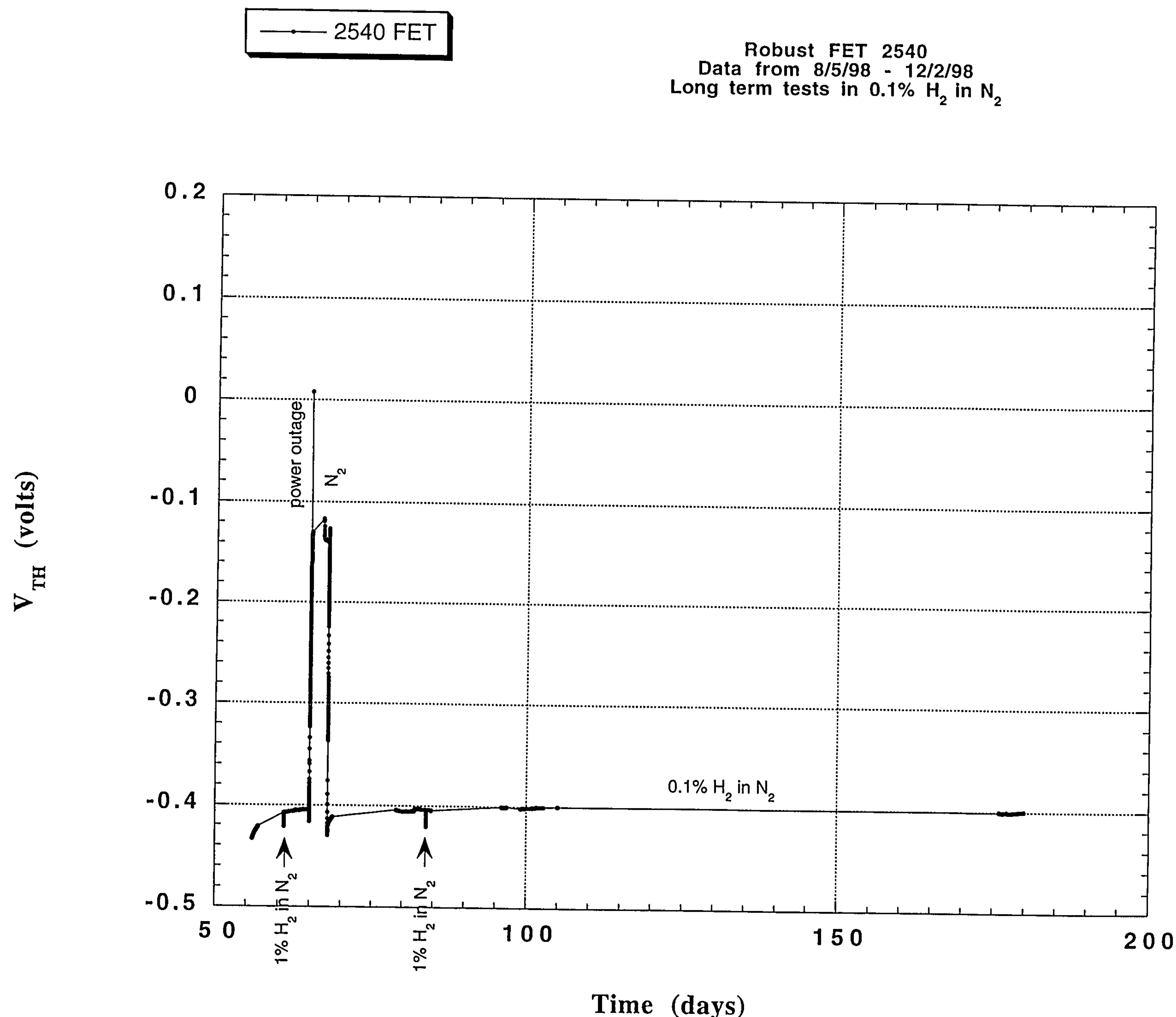

2540 FET
Robust FET 2540
Data from 8/5/98 - 12/2/98
Long term tests in 0.1% H_2 in N_2
0.2
0.1
0
-0.1
-0.2
-0.3
-0.4
-0.5
power outage
N_2
0.1% H_2 in N_2
1% H_2 in N_2
1% H_2 in N_2
50
100
150
200
V_{TH} (volts)
Time (days)

Figure 23

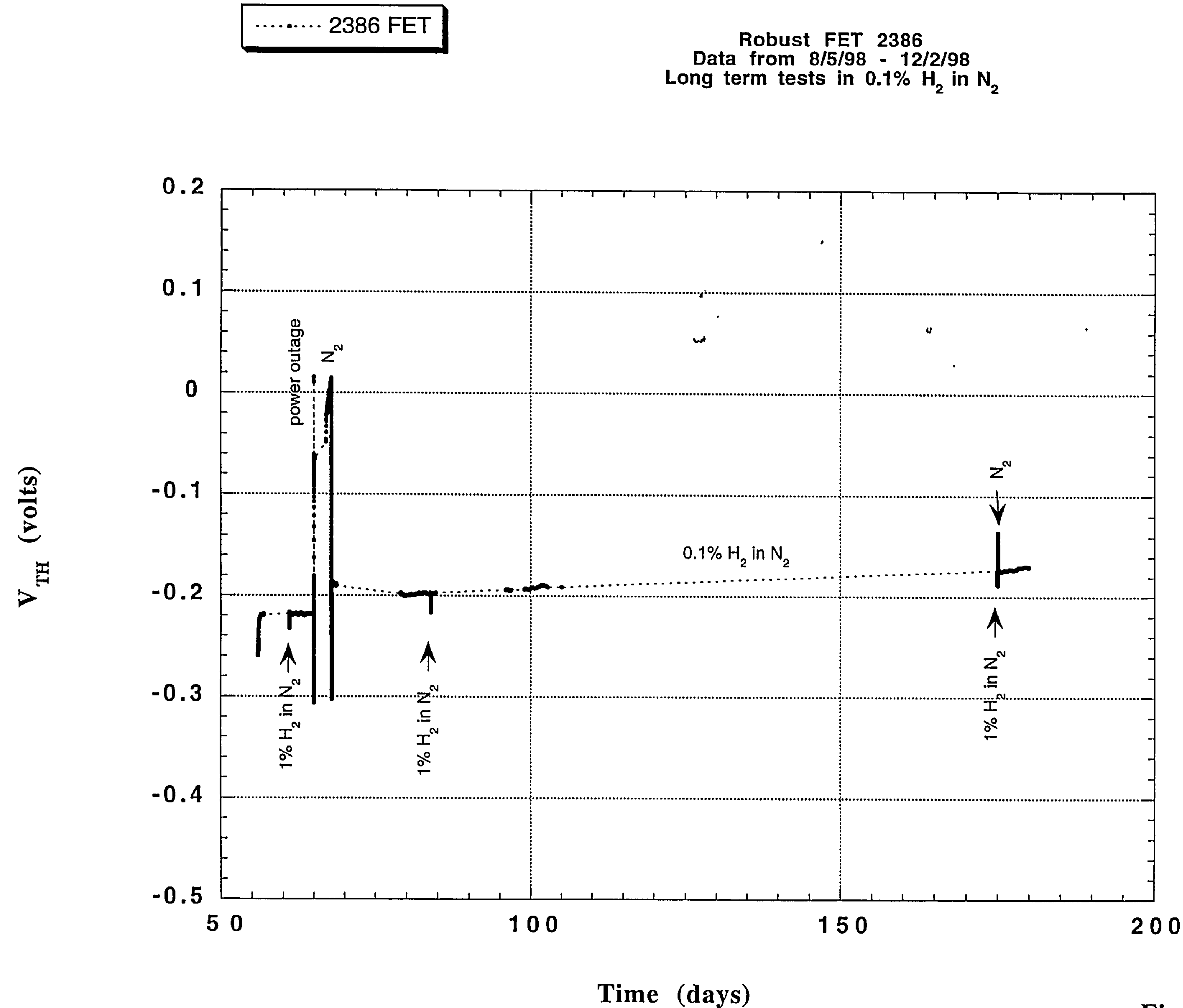

2386 FET
Robust FET 2386
Data from 8/5/98 - 12/2/98
Long term tests in 0.1% H_2 in N_2
power outage
N_2
1% H_2 in N_2
1% H_2 in N_2
0.1% H_2 in N_2
N_2
1% H_2 in N_2
V_{TH} (volts)
0.2
0.1
0
-0.1
-0.2
-0.3
-0.4
-0.5
50
100
150
200
Time (days)

Figure 24

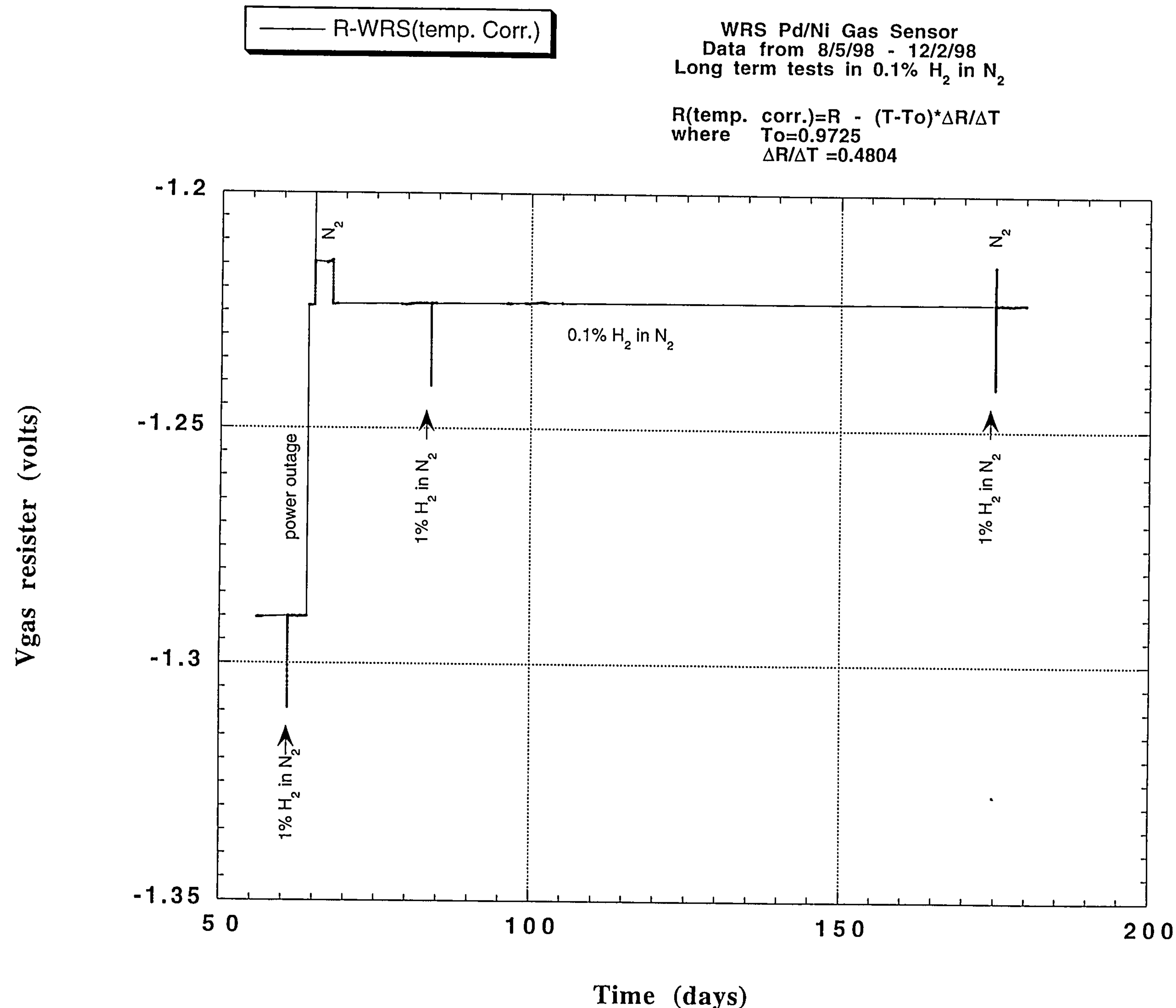

R-WRS(temp. Corr.)
WRS Pd/Ni Gas Sensor
Data from 8/5/98 - 12/2/98
Long term tests in 0.1% H_2 in N_2
R(temp. corr.)=R - (T-To)*ΔR/ΔT
where To=0.9725
ΔR/ΔT =0.4804
-1.2
-1.25
-1.3
-1.35
5 0
1 0 0
1 5 0
2 0 0
N_2
power outage
1% H_2 in N_2
0.1% H_2 in N_2
1% H_2 in N_2
N_2
1% H_2 in N_2
Vgas resister (volts)
Time (days)

Figure 25

Distribution List

Copies	Mail Stop	Addressee
5	MS 9014	Scott Faas, 2251
10	MS 1425	Mark Jenkins, 1715
1	MS 9018	Central Technical Files, 8940-2
2	MS 0899	Technical Library, 9616
1	MS 0612	Review & Approval Desk, 9612 For DOE/OSTI
1	MS 0161	Patent and Licensing Office, 11500