POTENTIOMETRIC MICROSENSORS AND TELEMETRY

by

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To Alexis

POTENTIOMETRIC MICROSENSORS AND TELEMETRY

ABSTRACT

The use of ion-selective field-effect transistors (ISFETs) as potentiometric microsensors was investigated. In the first stage, an instrument was designed and built to operate an array of ISFETs. A microcomputer was used for instrument control and acquisition of data.

The second phase of research focussed on the development of a pH sensitive radiotelemetric device that could eventually be used for the noninvasive monitoring of gastric pH. The first attempt used an ISFET as a variable resistor in a simple telemetry circuit. The drift in the pH dependant signal from this device was significant. The use of a differential sensor was studied as a possible way to minimize the effect of signal drift. This system measured the differential output of a pH ISFET and a pH insensitive ISFET. The pH insensitivity was due to an alkanethiol monolayer at the ISFET|solution interface.

It was shown that ISFETs are well suited for use as sensors in telemetry devices. The union of these previously independent research areas has been achieved.

POTENTIOMETRIQUES MINIATURISES ET TELEMETRIE

RESUME

Nous avons étudié l'âpplication de transistors à effet de champ modifiés chimiquement (ISFETs) en tant que détecteurs potentiométriques miniaturisés. La première étape fût de concevoir et construire un instrument capable contrôler un réseau de ces transistors (ISFETs). Nous avons employé un microordinateur pour l'opération de l'instrument et l'acquisition des données.

Durant la deuxième étape de ces travaux, nous nous sommes penché sur l'élaboration d'un détecteur télémétrique pour le dosage du pH. Le but de ce projet était d'en arriver à un détecteur non effractif pour le dosage du pH gastrique en milieu clinique. Notre première approche fût d'employer l'ISFET comme résistance variable dans un cicuit de télémétrie. Cependant, la dérive du signal analytique de ce circuit était considérable. Nous avons dont étudié l'application d'un détecteur comparatif afin de minimiser la dérive de ce signal. Ce systéme mesurait la différance entre le signal d'un ISFET sensible au pH et un autre rendu insensible par l'application d'une mince couche thiolalcane sur sa surface en contact avec la solution.

Nous avons démontré que les ISFETs sont des détecteurs adéquats pour la télémétrie. Nous avons aussi accomplis le marriage de ces deux domaines autrefois distincts.

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GLOSSARY

A	analyte ion
a_A	activity of the analyte in the test solution
$a_{A(int)}$	activity of the analyte in the internal solution
AC	alternating current
A/D	analog-to-digital
AM	amplitude modulation
ASCII	American Standard Code for Information Interchange
B	interfering ion
BF	broadcast frequency
C1	RC circuit capacitor
C18	octadecane
ChemFET	chemically sensitive field-effect transistor
C_{ox}	oxide capacitance of FET
Ct	tank circuit capacitor
CWE	coated-wire electrode
D/A	digital-to-analog
DC	direct current
E	battery voltage
E	measured potential
E_{asym}	asymmetry potential of membrane
E_{const}	collection of constant potentials
E_{int}	internal reference electrode potential
E_{lj}	liquid junction potential
E_{mem}	potential developed across a membrane
EnFET	enzyme-modified field-effect transistor
E_{ref}	external reference electrode potential
F	Faraday constant
FC1-FC5	FET controller circuit boards
FET	field-effect transistor
FM	frequency modulation
GasFET	gas sensitive FET
GI	gastrointestinal
GPIB/IEEE-488	parallel interface standard
IC	integrated circuit
I_D	drain current
IGFET	insulated-gate field-effect transistor
ImFET	immunologically-modified FET
ISE	ion-selective electrode
ISFET	ion-selective field-effect transistor
IUPAC	International Union of Pure and Applied Chemistry
JFET	junction field-effect transistor
K	collected physical constants of FET
$k_{A,B}$	selectivity coefficient
L	length of the FET channel
L1, L2	inductors
LB	Langmuir-Blodgett

L_{L1}, L_{L2}	inductance of L1 and L2
LOD	limit of detection
L_T	total inductance
M	mutual inductance
μ_n	electron mobility
MOSFET	metal-oxide-semiconductor field-effect transistor
op-amp	operational amplifier
optrode	optical chemical sensor
PC	printed circuit
PF	pulse frequency
PF	pulse period
PL	pulse length
φ_o	surface potential
PT1-PT3	prototype telemetry devices
R	gas constant
Rb	ISFET base resistor
RC	resistance-capacitance
R_{DS}	channel resistance between the source and drain
REF0	reference electrode output
ReFET	"reference" ISFET
RF	radiofrequency
R_S	source resistor
RS232C	serial interface standard
SAW	surface acoustic wave device
SCE	saturated calomel electrode
T	temperature
T	transistor
UU03	ISFET chip
Val/PVC	valinomycin/poly(vinyl chloride) membrane
V_B	base terminal voltage
V_D	drain terminal voltage
VD0	drain voltage in FET controller
$V_{D(sat)}$	saturation drain voltage
V_G	gate terminal voltage
V_S	source terminal voltage
VS0	set voltage in FET Controller
V_T	threshold voltage
V_T^*	modified V_T for ISFET
VZ0	zero offset voltage in FET Controller
W	width of the FET channel
X_C	reactance of capacitor C
z_A	charge of the principle ion A
z_B	charges of the interferent ion B

PREFACE

As the title indicates, the research presented in this thesis focuses on two distinct, and previously separate, fields of study. Ion-selective field-effect transistors (ISFETs) have been used as potentiometric microsensors for twenty years now, however they have not yet achieved widespread commercial use. The applications which successfully utilize ISFETs take advantage of the physical and chemical characteristics inherent in ISFETs which differentiate them from conventional potentiometric sensors. Our initial investigations into the use of ISFETs led to the conclusion that they would also be very well suited to be employed as the sensor component in a telemetric device. The absence of any previous work involving ISFETs in telemetry can be attributed to the fact that the majority of telemetry research predates the more recent work on potentiometric microsensors. This thesis describes our efforts to incorporate ISFETs into the field of telemetry. The specific objective was to employ a pH ISFET in a biotelemetry device which could eventually be used for gastric pH monitoring.

Chapter 1 serves as an introduction to chemical sensors. A cursory overview of the different types of chemical sensors presently available is given. The balance of the chapter outlines the parameters which must be considered when evaluating any sensor.

Due to the fact that the ISFET and telemetry research fields were previously independent, these areas are reviewed separately. In Chapter 2, the history and relevant theories of operation for ISFETs are presented. In order to study the operation of ISFETs, it was necessary to design and construct customized instrumentation. To test and evaluate ISFETs, a FET Controller was built which is discussed in Chapters 3 and 4. This instrument was also used, in a modified form, for the experiments summarized in Chapter 7.

Chapter 5 covers the history of telemetry research as well as possible modes of operation. This chapter also outlines the need for gastric pH determination and reviews the methods presently available.

Chapter 6 describes the initial attempt to use a pH ISFET in a simple telemetry device. The results from these tests presented further problems, which led to the consideration of a differential sensing circuit. As shown in Chapter 7, in this circuit the signal from a pH ISFET is compared to the signal from a pH-insensitive ISFET.

CHAPTER 1

CHEMICAL SENSORS

1.1 Introduction

Today's society is becoming more information oriented, with particular emphasis placed on quick, if not immediate, access to such information. One manifestation of this is the need for direct-reading chemical sensors in a wide variety of environments such as clinical laboratories, process-control systems, and mobile or on-site testing facilities. A chemical sensor provides an instantaneous readout which allows the analyst to establish the presence, or concentration, of an atomic, molecular or ionic species in either an aqueous or gaseous medium. There should be no sample pretreatment or separation steps required. Although a direct, on-line readout is achieved, most chemical sensors require some preliminary setup before use (*e.g.* calibration). Nevertheless the information obtained can be invaluable. The use of blood electrolyte sensors can help to increase the safety and well-being of a patient during a medical operation. Similarly, a chemical sensor could alert the operator when a batch reactor is malfunctioning, allowing the problem to be corrected online. Chemical sensors are also used as specific detectors in flow-injection analysis.

A chemical sensor can be said to consist of three distinct, but integrated elements: chemical recognition, transduction, and signal processing. The chemical recognition stage is the heart of the sensor, and is largely responsible for the ultimate performance. This stage determines the chemical selectivity of the sensor. For a sensor which is used in a liquid solution, chemical recognition is often achieved by an ion-selective membrane or an enzyme reaction. This element must also produce a physical signal due to the selective chemical interaction. For electrochemical sensors the signal is a voltage or current change, while an optical sensor relies on a change in intensity of the optical signal. The transducer element monitors the physical signal produced. This step amplifies the signal, and usually converts it to a voltage output. The ideal transducer extracts all the raw data available from the chemical recognition stage without creating any error due to the transduction process. The sensitivity of the sensor is dependant on both the recognition and transduction elements. The final step, signal processing, is not usually considered an integral part of the sensor, however it is important to the commercial viability of any chemical sensor. This provides for the conversion of the transducer voltage output into a more convenient readout quantity such as concentration. While this may require some work on the part of the user, it is preferable to perform this solely in hardware. Although this may appear trivial, it is this last step which presents the desired information and determines the ease of use of a chemical sensor. Improvements in signal processing are essential to the further development of smart sensors capable of multiplexing several sensors, automatic calibration, drift adjustment, *etc.*[1].

The term *microsensor* is difficult to define unambiguously. There are specific criteria which must be met in order for an amperometric sensor to be classified as micro. In this case the electrode must be small enough to comply with the spherical diffusion model [2]. This is the exception however. For other types of chemical sensors the differentiation between conventional sensors and microsensors is much more vague. Microsensors generally incorporate some sort of modern technology such as microelectronic engineering or fiber-optic spectroscopy, and have at least one dimension in the sub-millimeter range. A more useful approach might be to consider the minimum sample volume required; a microsensor should be able to function in microliter volumes. Finally, one could look at the possible applications. For example, most sensors that can be used *in vivo* can be considered to be microsensors. While this terminology is useful at present, it can be safely assumed that future developments in sensor research will make today's microsensors appear rather large and poorly labelled.

1.2 Types of Sensors

This thesis examines the use of potentiometric microsensors; nevertheless there is a variety of other types of chemical sensors which should also be mentioned. Sensor type is determined by both the chemical recognition and transduction elements, however some sensors could be said to belong to more than one type. Table 1.1 lists the different sensor types which are discussed here. For this introduction it is impossible to review all types of sensors in detail, therefore the following sections are limited to a discussion of the basic principles involved and are accompanied by a few examples of each type of sensor.

Table 1.1
Classification of Sensors

Class	Medium (1)	Transducer	Physicochemical Property Measured
Optical	liquid gas	spectrometer	optical properties
Thermal	gas liquid	thermistor or pyroelectric crystal	temperature
Piezoelectric	gas liquid	bulk wave or surface acoustic wave devices	surface adsorption of matter - mass change
Electrochemical			
Amperometric	liquid gas (2)	potentiostat	oxidation/reduction current
Conductometric	gas/vapor liquid	conductivity meter	chemically modulated resistance
Potentiometric	liquid	potentiometer or ISFET	interfacial potentials
	gas	MOSFET	change in work function of gate

(1) For each class, the media are listed in order of importance
(2) Self-contained aqueous sensor covered with gas permeable membrane

There are several recently published books which do cover all chemical sensors in detail [3,4,5], and the biennial reviews in Analytical Chemistry [6,7] present an overview of recently published sensor research.

1.2.1 Optical Sensors

Optical chemical sensors, commonly known as optrodes, combine optical fiber technology and the highly specific interaction of electromagnetic radiation with matter. An optical fiber consists of a cylindrical, transparent core of quartz, plastic, or glass, surrounded by a cladding of lower refractive index whereby light entering the fiber undergoes total internal reflection. These fibers range from a few micrometers to several hundred micrometers in diameter and are quite flexible. They act as a light conduit between the chemical recognition element and the instrumentation, usually consisting of a source and a detector, which act as the transducer. Four of the common designs are shown in Figure 1-1. These sensors measure the fluorescence due to the chemical interaction in the analysis area. Other optical properties that can be similarly measured include absorbance, reflectance, refractive index, chemiluminescence and Raman effects.

An example of the optrode in Figure 1-1(a) can be found in the paper by Luo and Walt [8]. They have constructed a pH sensor for the range of pH values from 5.5 to 8.0 using pH sensitive fluorescent dyes. To increase the lifetime of the device, the dyes were entrapped in controlled-released polymers that were incorporated in the tip of the optrode but out of the light path. Sustained release of the dye enabled reported lifetimes of at least three months in a laboratory. A penicillin optrode which used a single fiber (Figure 1-1(a)) with the enzyme penicillinase and a pH sensitive fluorescent dye immobilized onto the fiber tip as in Figure 1-1(b) was reported by Kulp *et al.*[9]. By co-immobilizing the enzyme and dye directly on the tip of the fiber, a decrease in response time was obtained compared to membrane encased enzyme optrodes. Figure 1-1(c), used mainly for absorbance measurements, is similar to (a) but uses two fibers.

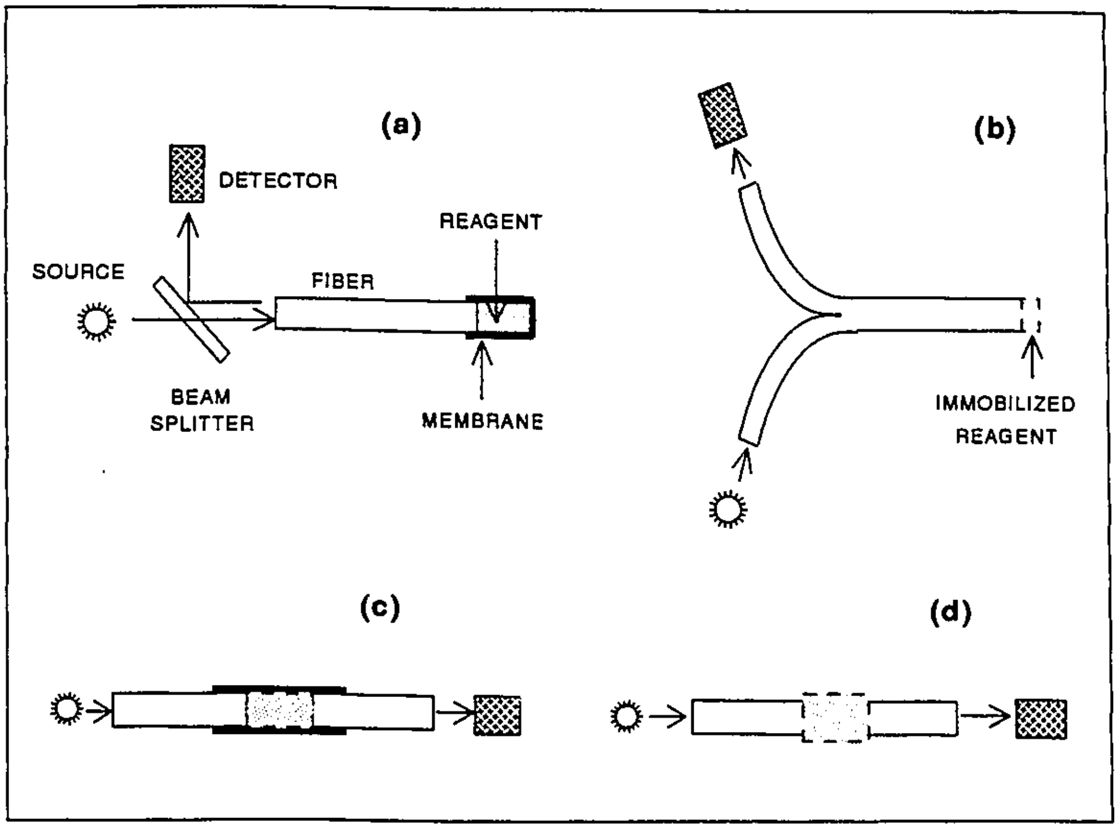

Figure 1-1. Optrode designs
(a) single fiber with reagent encased in membrane at tip
(b) bifurcated fiber with reagent coated on tip
(c) dual fiber with reagent encased in membrane
(d) reagent coated on surface of fiber

Carroll *et al.*[10] described an optrode consisting of a bifurcated fiber (Figure 1-1(b)) with the reagent encased in a membrane at the distal tip of the fiber (Figure 1-1(a)) to simultaneously determine Al^{3+} and either Ga^{3+} or In^{3+}. The reagent was a chelate which forms strongly fluorescent complexes with these trivalent ions. To differentiate between ions, signal processing in the form of time resolved fluorometry was used.

Figure 1-1(d) illustrates an alternative sensor design where the chemically selective coating interacts with the evanescent wave at the surface of the fiber. The evanescent wave is a standing wave on the fiber surface created by interference between the incident and reflected internal beams. Carey *et al.*[11] described an optrode where a small section of bare fiber was coated with a Hammett indicator entrapped in a polymer. This was used to measure the molarity of concentrated acids (1-10 M). This surface interaction method, known as attenuated total reflectance, is particularly well suited for affinity sensors based on immunological binding [12,13].

Fiber optic chemical sensors are well suited to be used as microsensors. Selectivity can be excellent with the proper chemical recognition element such as an enzymatic reaction, and they can be quite sensitive (low detection limit) due to the lack of noise pickup from stray electromagnetic radiation. They also provide electrical isolation of the sample, or patient, which is important particularly for *in-vivo* sensing.

1.2.2 Thermal Sensors

There are a variety of thermally sensitive detectors or sensors. For this review, only those thermal sensors which meet the definition of a chemical sensor, as outlined in the introduction, will be considered. Thermal sensors detect the evolution of heat during a chemical reaction due to enthalpy changes. The keys to the success of these devices are the specificity of the chemical reaction that is monitored and, to a lesser extent, the magnitude of the enthalpy change. In practice this has limited the reactions used for chemical recognition to enzymatic processes.

Of the two types of transducers listed in Table 1.1, the thermistor is used far more often than pyroelectric crystals. These types of transducers can be used directly in microsensors without any adaptation. A thermistor is a small bead of sintered oxide semiconductor encased in a glass shield. The response is due to the effect of temperature on the bandgap energy of the semiconductor, and can range from -80 ^{o}C to +350 ^{o}C [14]. A pyroelectric crystal responds to temperature changes by changing its polarization vector thereby inducing a surface charge which can be measured at the electrodes. These crystals can give three orders of magnitude greater sensitivity than thermistors [15], however they have been rarely used to date due to the increased complexity compared to thermistors.

Enzyme thermistors either have the enzyme immobilized onto the glass casing of the thermistor, or on a larger scale, the thermistor is simply placed in a column of immobilized enzymes. Both devices are usually used in conjunction with a second reference thermistor and the output is taken from a Wheatstone bridge circuit. A review of enzyme thermistors [16] describes sensors for urea using urease, and for glucose, oxalate and ethanol using the appropriate oxidase, as well as other substrate/enzyme systems.

1.2.3 Piezoelectric Sensors

Piezoelectric gravimetric sensors are based on the same phenomenon as pyroelectric thermal sensors. Instead of responding to heat, piezoelectric crystals react to applied stress by changing their natural frequency of oscillation. Physical sensors such as the quartz crystal microbalance and various pressure sensors use piezoelectric crystals. Chemically selective mass sensors using piezoelectric transducers have a selective layer on the crystal surface which specifically adsorbs molecules or ions resulting in a change in mass [17].

The two types of piezoelectric transducers are shown in Figure 1-2. In these diagrams only a portion of the crystal is coated and exposed, however in many cases the entire surface of the device is used. Bulk wave devices were developed first and have since been used mainly for gas or humidity sensing with or without a chemically selective coating [18]. Surface acoustic wave devices (SAWs) were first described by White and Voltmer [19] in 1965, when they reported how interdigital arrays could be used to propagate waves across the surface of a crystal. Over the last ten years SAWs have become more popular than bulk wave devices because: several SAWs can be placed on a single crystal; SAWs can be made much smaller; higher fundamental frequencies of SAWs allow for lower absolute limits of detection; and it is easier to expose the coated area on a SAW to the solution [20,21].

McCallum's review [22] in 1989 on mass and chemical piezoelectric sensors is quite comprehensive. It lists 137 references and

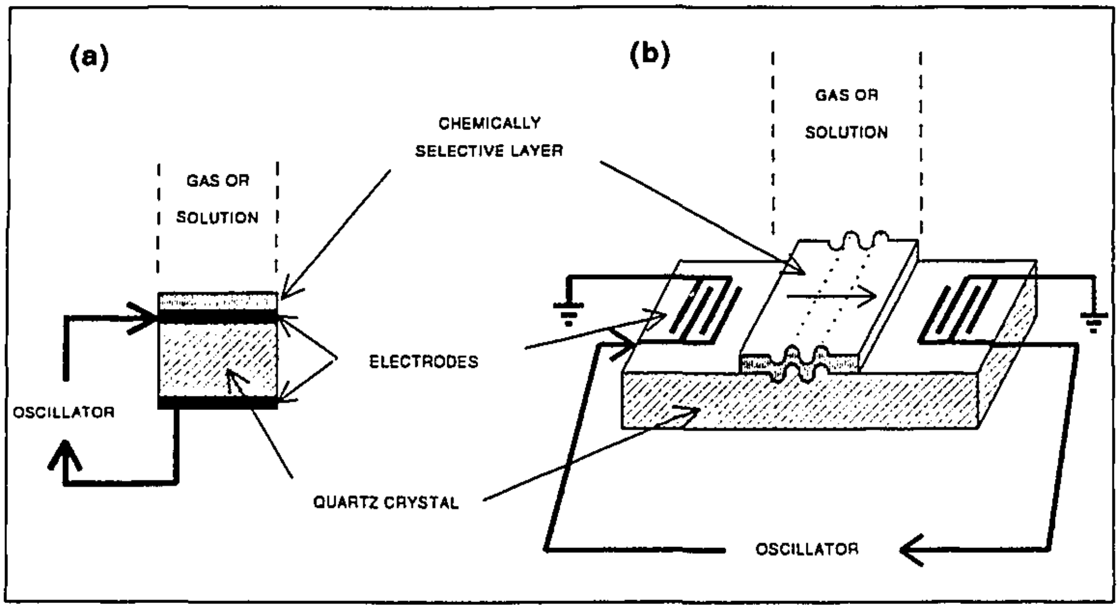

Figure 1-2. Piezoelectric transducers
(a) bulk wave oscillator
(b) surface acoustic wave device

summarizes reports of sensors for over 25 chemical compounds. This review, and other literature [6,17,18,21], indicate that many research groups have attempted to use coated SAWs as chemical sensors in solution with varying degrees of success.

A driving force in the development of solid state microsensors is the possibility of sensor arrays. This advantage was exploited by Rose-Pehrsson *et al.*[23] in their use of an array of ten SAWs, each coated with a different polymer, to detect nine vapors. The individual polymers were not chemically specific, however the authors were able to identify the vapors by use of pattern recognition analysis.

Because of the mass sensitivity of piezoelectric devices, they have great potential as an immunochemical assay transducer. An example of this is the paper by Muramatsu *et al.*[24] where Protein A was immobilized on a bulk wave device to detect immunoglobulin G.

Piezoelectric sensors with chemical recognition have become firmly established, and research interest continues to expand. Their utility as gas phase sensors has been proven, and the range of applications will continue to grow. As for their use in solution, there are still problems to be solved, but the future appears bright.

1.2.4 Amperometric Sensors

Since the Clark electrode was first described in the early 1950's [25], many amperometric sensors have been developed and have proven to be useful. Amperometric sensors have benefited from the large amount of research involving classical electrochemical techniques, such as voltammetry, which can be used to investigate the recognition-transduction process.

The potentiostat and electrode system combine to act as the transducer. The working electrode (sensor) is held at a constant potential with respect to a reference electrode. The measured current is due to electron transfer between the electrode and the species undergoing oxidation or reduction in solution.

The Clark electrode consists of a pair of electrodes exposed to an electrolyte solution which is separated from the sample solution by a gas permeable membrane. Oxygen diffuses from the sample to the inner electrolyte where it is detected amperometrically as it is reduced at the cathode. This is a simple, yet quite elegant chemical sensor. It can be operated in aqueous or gaseous samples, and by changing the membrane it can modified to detect other electrochemically active gases. It has also been used in a miniaturized form as a non-invasive probe for transcutaneous oxygen monitoring [26].

While the choice of working electrode potential and the use of selective membranes offer some selectivity, the majority of research in amperometric sensors is now focussed on the use of enzymes to increase selectivity and broaden the range of possible applications. The enzyme acts upon a substrate, usually electrochemically inert, to produce an electroactive product (*i.e.* H_2O_2), or consume an electro-active reagent (*i.e.* oxygen). Perhaps the most successful amperometric sensor is the pen-sized glucose meter developed by Cardosi and Turner [27]. This sensor uses glucose oxidase, but ferrocene is substituted for oxygen as the mediator to eliminate sample matrix effects due to varying oxygen levels.

1.2.5 Conductometric Sensors

Conductometric sensors have not enjoyed widespread use but they can be quite useful in certain applications. Almost all conductometric sensors are constructed similarly. Usually they consist of a pair of interdigitated metal electrodes on a substrate of insulating material. A surface layer spread across the electrodes provides the chemical selectivity. Conductometric sensors may use either direct or alternating current.

Chemiresistors are the simplest of these sensors. They are used for the detection of various gases and vapors in air. A variety of selective layers have been used including metal, organic, and polymeric thin films [6,7]. Chemiresistors are operated in the direct current mode; usually a commercial conductivity meter is used. Dielectrometers are an extension of chemiresistors, the main difference being the use of an alternating excitation signal. These are used almost exclusively for humidity sensing.

The last five years have seen the introduction of enzyme-based conductometric sensors for use in solution. In this case the enzyme reaction must change the conductance of the solution. Once again, an alternating excitation signal is used and an alternating current response is observed. Mikkelsen and Rechnitz [28] report sensors for the determination of urea and D-amino acids. They also point out that because these sensors respond to changes in ionic strength, buffers must be used in sample preparation to maintain constant ionic strength.

1.2.6 Potentiometric Sensors

This thesis is concerned with potentiometric sensors, the last class of chemical sensors listed in Table 1.1. In particular, the use of ion-selective field-effect transistors (ISFETs) as potentiometric microsensors was studied. This section examines the general principles of potentiometry and compares sensors using ISFETs with other potentiometric sensors. ISFETs will be reviewed in detail in Chapter 2.

There are certain types of potentiometric electrodes (*e.g.* redox electrodes), often used in titrations, which are not considered to be chemical sensors due to the lack of chemical specificity. Reference 29 covers all areas of potentiometry. A *sensor* must be specific and you must be able to use it directly in the sample solution. This limits our consideration to membrane-based, ion-selective electrodes (ISEs).

A potentiometric sensor measures the potential difference across the interface between a chemically selective layer and the sample solution. The potential difference can arise from surface adsorption, absorption into the bulk layer or selective transport through a membrane. While there is constant ionic flux across the interface, there is zero net current flow in potentiometry. For conventional ISEs, the membrane acts as the chemical recognition element and a potentiometer is the transducer.

In Figure 1-3 a typical configuration for an ISE is shown. The internal and external reference electrode potentials (E_{int} and E_{ref} respectively) must be constant in order to observe the potential changes at the membrane-solution interface. The liquid junction potential (E_{lj}) is also considered to be constant. The potential developed across the membrane (E_{mem}) is dependant on the activities of the analyte in the test solution (a_A) and in the internal solution

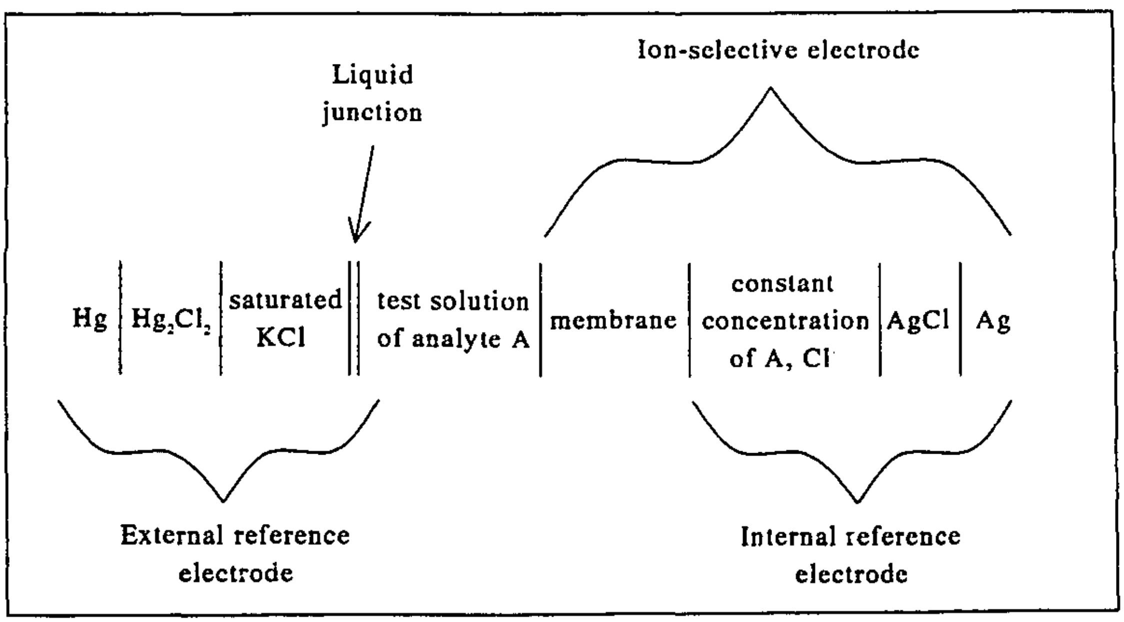

Figure 1-3. Conventional ISE configuration.

($a_{A(int)}$) according to Equation 1.1

$$E_{mem} = \frac{RT}{z_A F} \ln \left[\frac{a_A}{a_{A(int)}} \right] \tag{1.1}$$

where R is the gas constant, T is temperature in Kelvin, z_A is equal to the charge of the principal ion A, and F is the Faraday constant. The measured potential (E) is the sum of all the potentials as shown in Equation 1.2.

$$E = E_{ref} + E_{lj} + E_{int} + E_{asym} + E_{mem} \tag{1.2}$$

where E_{asym} is the asymmetry potential. This potential is due to defects in the membrane. Although it changes slowly with time, it can be considered constant if frequent calibration is used.

Because $a_{A(int)}$ is constant, it can be removed from Equation 1.1 and combined into one constant (E_{const}) with all the other constant terms in Equation 1.2 to give Equation 1.3.

$$E = E_{const} + \frac{RT}{z_A F} \ln a_A \tag{1.3}$$

Unfortunately, the membranes used in ISEs are selective rather than specific. An ion-selective membrane will respond to interfering ions as well as the principal ion, the response being greatest for ions with similar charge and size. The selectivity coefficient ($k_{A,B}$) is a measure of how selective the ISE is for the interfering ion, B, compared to the analyte ion, A. This effect is taken into account by the Nikolsky-Eisenman equation as follows:

$$E = E_{const} + \frac{2.303\, RT}{z_A F} \log \left[a_A + \sum_{B=1}^{B=n} k_{A,B} \left(a_B\right)^{z_A / z_B} \right] \tag{1.4}$$

where z_A and z_B are the charges on the analyte and the interferent ions, respectively, and there are n interfering species. A low value for the selectivity coefficient is desired. The actual $k_{A,B}$ value which is necessary for a ISE to be effective is dependant on the relative levels of the analyte and interfering ions.

The preceding equations indicate that potentiometric sensors respond to changes in activity of the analyte ion, while it is preferable to use concentrations for simplicity. In order to use standards of varying concentration as the basis for a calibration curve, the activity coefficients of the sample and standard solutions should be equal. This is usually achieved either by using a buffer to maintain constant ionic strength in sample and standards, or the composition of the standard is made to be as similar to that of the sample as possible.

It is beyond the scope of this thesis to even begin to summarize research in the field of ISEs. It is safe to say that, at a minimum, there have been thousands of ISEs reported, and the number of possible analytes must be in the hundreds. The range of analytes includes non-ionic compounds due to the coupling of ISEs with enzymes and gas permeable membranes. A series of reviews by Koryta [30] reports on approximately 400 ISE papers per year.

Aside from work on the actual ion-selective membrane, a great deal of research in the last twenty years has focussed on the physical construction of ISEs. A primary goal was to minimize their size. This led to efforts to eliminate the internal solution and the development of coated-wire electrodes (CWEs) and ISFETs. The conceptual relationship between ISEs, CWEs and ISFETs can be seen in Figure 1-4. In Figure 1-4(a) a conventional ISE is hooked up to a potentiometer. The input stage is a metal-oxide-semiconductor field-effect transistor (MOSFET) which monitors the voltage without drawing any current. The CWE uses the same instrumentation, but now the membrane is coated directly on the tip of the wire. Finally in Figure 1-4(c) the wire is eliminated. Here the membrane replaces the metal gate on the MOSFET and the device, now called an ISFET, is placed in the solution. The aim of our research is represented in Figure 1-4(d), where the ISFET sensor circuit broadcasts a signal to an external recording instrument. It should be noted that the reference electrode

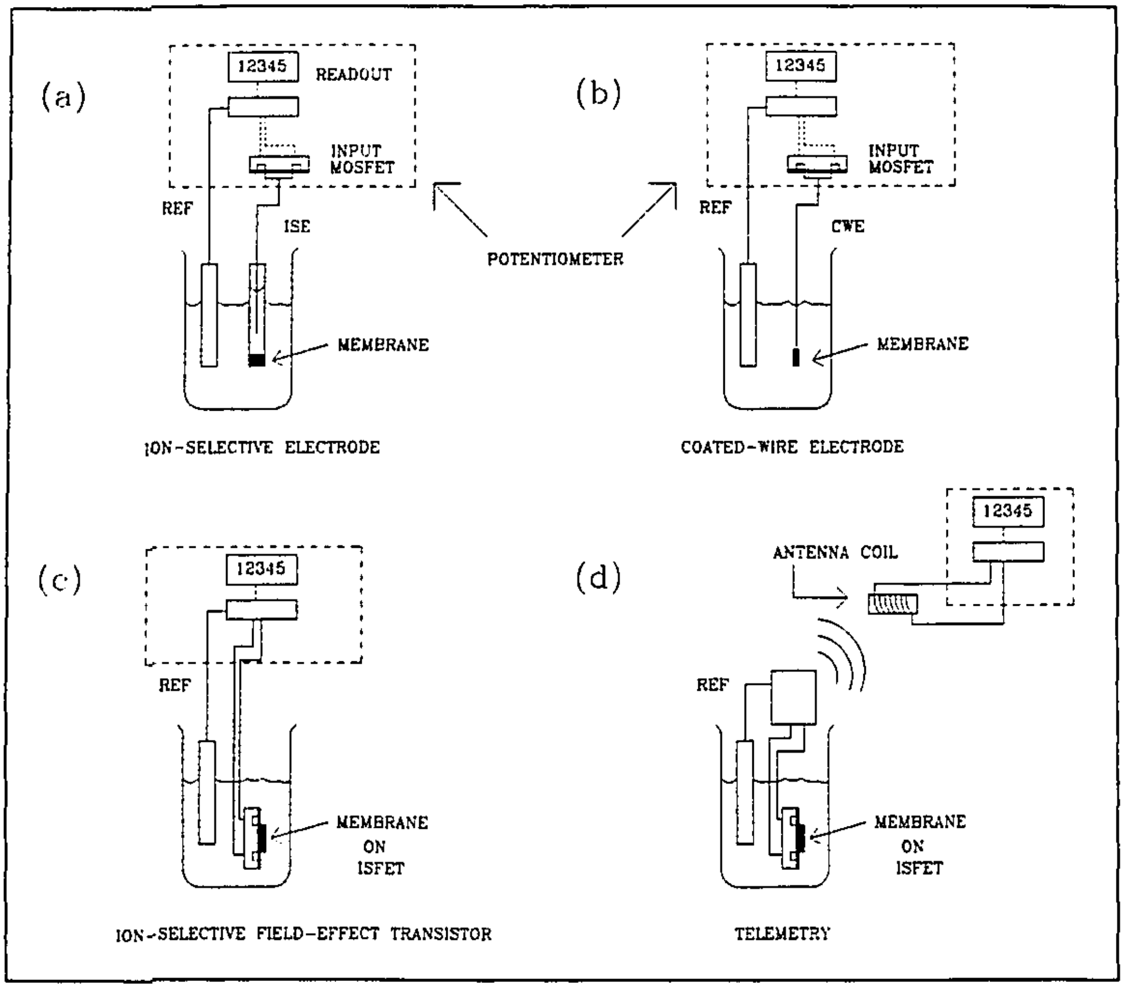

Figure 1-4. Potentiometric sensors.
(not to scale)

does not change in Figure 1-4. This is still a hindrance in the development of potentiometric microsensors and will be addressed in Chapters 2 and 7.

1.3 Criteria for the Evaluation of Sensors

In the evaluation of any type of chemical sensor there are certain criteria which must be addressed. The relative importance of these factors varies depending upon the application. The reliability of a chemical sensor can be said to be the sum of all the performance and physical characteristics. These can be divided into two areas of concern as follows:

Performance	Physical
Selectivity	Lifetime
Sensitivity	Size
Limit of detection	Durability
Response time	Biocompatibility
Reversibility	Uniformity
Drift	Cost

Each of these criteria will be discussed in the balance of this chapter, with the focus on potentiometric sensors.

1.3.1 Performance Characteristics

The performance of a chemical sensor is dependant on all three elements of the sensor: chemical recognition, transduction, and signal processing. In practice most researchers concentrate on only one type of transducer. In our case the transducer is a potentiometric micro-sensor. This leaves the chemical recognition element, the ion-selective membrane, as the main determinant of the performance. Signal processing can be used to compensate for deficiencies of the recognition element.

In order to reduce confusion in the area of ISE research, IUPAC put forward in 1975 a set of recommendations for the nomenclature of ion-selective electrodes [31]. Most of the performance characteristics were defined. When reporting values for these performance characteristics, all the experimental details should also be stated; including electrode conditioning and history, stirring rate, temperature, and solution composition(s).

1.3.1.1 Selectivity

The selectivity of a sensor is a measure of the ability of the sensor to accurately determine the concentration of the analyte in the presence of interfering ions which can also affect the measured potential. The selectivity of a sensor should be determined for all possible interferents. This requires knowledge of the composition of the test solution. If this information is not available, the selectivity coefficients should be determined for all ions similar to the analyte as well as the more common ions (H^+,Na^+,Ca^{2+},Cl^- *etc.*).

There are two methods for the determination of $k_{A,B}$ [31]. The first is the ***separate solution method.*** For this method the slope of the calibration curve must be known. Two solutions are used, one containing only the analyte and the other with only interferent present. The second method is the *fixed interference method.* This method uses a series of solutions containing a constant concentration of interferent but varying concentrations of analyte. The fixed interference method is preferred over the separate solution method because it is a more accurate representation of the actual conditions of use, and is not affected by variations in slope.

Typical selectivity coefficients range from 1×10^{-6}, where the interferent has very little effect, to 1×10^{3}, in which case the ISE actually has a greater response to the interferent than the analyte, but this still could be used as long as the ratio of analyte to interferent is at least 10^5. The greatest selectivity is obtained by coupling an enzyme reaction which alters the concentration of an ion (usually H^+ or NH_4^+) that can be monitored with an ISE or ISFET.

1.3.1.2 Sensitivity

Sensitivity is basically the minimum change of analyte concentration that can be observed. The slope of the calibration curve, noise, and readout resolution combine to determine the sensitivity of a sensor. Sensitivity is usually discussed in combination with the linear response range.

If we consider Equation 1.4, the theoretical slope for a singly charged ion ($z_A=1$) is equal to 2.303RT/F, or 59.16 mV per decade at 25 °C. One of the advantages of ISEs compared to other sensors is that they respond logarithmically and can be used over a wide linear response range, often up to 5-6 orders of magnitude. This requires a varying degree of sensitivity. For instance, if we consider a typical ISE system where readout resolution is 1 mV, and noise is negligible, then an observed change of 1 mV corresponds to concentration change of roughly 4×10^{-5} M in a 1×10^{-3} M solution while in a 1×10^{-2} M solution the change is 1×10^{-4} M.

1.3.1.3 Limit of Detection

While the term "limit of detection" is often confused with "sensitivity", they are not interchangeable. By definition the limit of detection (LOD) is lowest concentration of analyte that can be discriminated, within a specified degree of confidence, from a blank solution under specified conditions.

In ISE research, the reporting of LODs is confusing due to the existence of several different conventions. Three methods for calculating the LOD are shown in Figure 1-5. The highest LOD value is just the lower limit of the linear response range, and is usually determined graphically from the calibration curve. Typical values range from 10^{-5} to 10^{-7} M, however much lower LODs are possible (*e.g.* pH electrode; enzyme coupled ISEs). The IUPAC recommendations [31] define the LOD to be equal to the activity of the analyte at the point of intersection of the two linear portions of the calibration curve. This gives a LOD value which is lower than the limit of the linear response range. The lowest LOD can be calculated statistically using signal-to-noise parameters according to the definition in the preceding paragraph. This corresponds better to the methods used for LOD calculations in other areas of chemical analysis. When reporting or comparing reported LODs, one must be conscious of the method used.

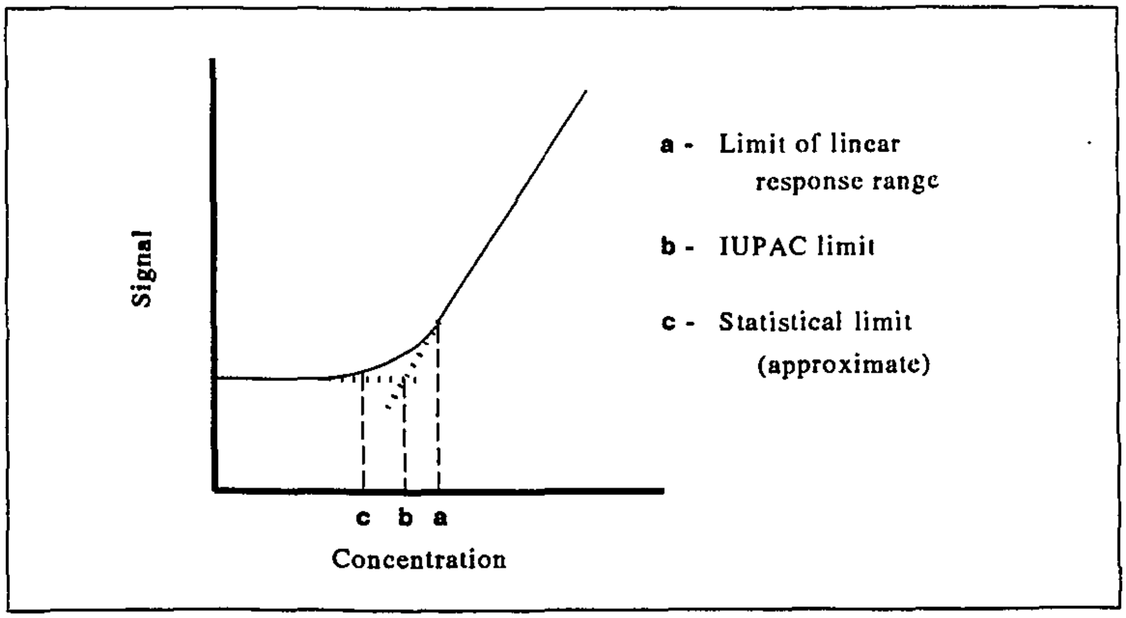

Figure 1-5. Determination of limit of detection from calibration curve.

1.3.1.4 Response Time

In general the response time of a chemical sensor is defined to be the length of time it takes after immersion in a new solution for a sensor to settle to within ±1% of the final signal. This is very dependant on the experimental details listed in the introduction (Section 1.3.1). For ISEs, IUPAC has altered this definition. They recommend that the length of time be measured from immersion to the instant that the signal approaches within ±1 mV of the steady state signal.

Response times for ISEs are largely determined by the choice of membrane. Thin, solid state membranes give the fastest response, in the order of seconds. The longest times are seen for polymer membranes and enzyme coupled ISEs which can take up to several minutes to settle. Compared to ISEs, ISFETs have quicker response times due to the thinner membranes used.

1.3.1.5 Reversibility

Unlike the previous characteristics, reversibility is not usually measured or quantified. A sensor is said to be reversible if it can be reused repeatedly with or, preferably, without an intermediate clean-up cycle. Ideally the sensor should exhibit no memory effects upon changing of solutions.

When a potentiometric sensor is changed from solution X to solution Y and back to X, the two potentials for solution X should be identical, otherwise hysteresis is said to have occurred. This effect is worst if solution Y is either extremely dilute or concentrated. In certain cases reconditioning may restore proper behavior.

1.3.1.6 Drift

Any type of drift in the output of a chemical sensor is undesirable. Short term drift generally renders the sensor useless. Long term drift can sometimes be accommodated by either frequent recalibration or some type of drift correction in the signal processing stage. For ISEs drift can be due to a variety of physical or chemical parameters such as temperature fluctuations, leaching of electroactive material from the membrane, sorption of interferents or a variation in the potentials assumed to be constant (E_{int}, E_{ref} and E_{lj}). A second sensor, insensitive to the analyte, is sometimes used to try to eliminate drift. This is easily achieved with ISFETs and other sensors that can be produced in compact arrays.

1.3.2 Physical Characteristics

The physical properties of a chemical sensor are determined primarily by the choice of the transducer. While the performance characteristics of a sensor determine whether or not a sensor is viable, the physical characteristics often are the deciding factors as to commercial success.

1.3.2.1 Lifetime

The lifetime of a sensor can be considered both a performance and a physical parameter. Sensor life can be terminated by failure of the chemical recognition element or electronic malfunction. Some sensors have lifetimes up to several years. Other sensors have been designed for single-use applications which may only require lifetimes of hours or even minutes.

1.3.2.2 Size

The concept and size of microsensors have already been discussed in the Introduction (Section 1.1). There is a drive to the miniaturization of all types of chemical sensors even though most sensors are used in macroscopic samples. The two main areas of use for microsensors are low volume flow stream detectors and *in-vivo* sensing.

For the evaluation of any sensor both the overall size of the sensor and the actual dimensions of the sensing area should be considered. The application establishes which measurement is most important.

During the development of a sensor, the size is often much larger than necessary to facilitate manual processing. The final dimensions will usually be smaller, especially if automated manufacturing methods are used.

1.3.2.3 Durability

A chemical sensor may be subjected to many different environments. It should be resistant to chemical attack by the constituents of the sample, variable temperatures, and physical shock. It should also be capable of undergoing cycles of dry storage, reconditioning, and use without loss of performance.

1.3.2.4 Biocompatibility

This criteria is important only if the sensor is to used for *in-vivo*, extra-corporeal or undiluted biological fluid testing. Two aspects of biocompatibility must be considered. The first is the effect of the sensor on the safety and well-being of the subject. *In-vivo* sensors for use in bloodstreams must be non-thrombogenic, and all sensors must be non-toxic to the subject.

The second aspect which must be considered is the effect of the biological matrix on the performance of the sensor. Surface adsorption of proteins and immunochemical attack are the two biggest problems. In some case an additional membrane, permeable to smaller ions and molecules, can be used to shield the sensing area from the larger biomolecules.

1.3.2.5 Uniformity

This is simply an indication of how reproducible the manufacturing process is. Ideally all sensors of a particular type should be perfectly interchangeable. In practice the performance characteristics are consistent, but each sensor usually has to be individually calibrated. The use of automated production methods greatly enhances reproducibility.

1.3.2.6 Cost

Cost is primarily determined by the manufacturing method, the expense decreasing with the use of automation. The raw materials are cheap, especially for any process using conventional microelectronic or fiber optic technology. For sensors using biological materials, the small volumes required for microsensors reduces the cost compared to conventional sensors.

1.4 Summary

Several different types of chemical sensors have been discussed in this chapter. From among these sensors, we decided to focus on the use of ISFETs as potentiometric microsensors. ISFETs possess the necessary characteristics for use in a pH sensitive telemetry device. The history, theory and use of ISFETs is discussed in greater detail in the next chapter.

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CHAPTER 2

ION-SELECTIVE FIELD-EFFECT TRANSISTORS

2.1 Introduction

Although the essential concepts of field-effect transistors (FETs) were patented in the late 1920's by Lilienfeld [1], it wasn't until the 1950's that the technology was available to actually construct functional devices. The production of both junction field-effect transistors (JFETs) [2] and metal-oxide-semiconductor field-effect transistors (MOSFETs) [3] at Bell Laboratories was, in a sense, a re-invention of the original patents. The creation and subsequent applications of FETs was delayed, in part, by the earlier success of the bipolar transistor. The inherent differences between the various types of transistors (bipolar, JFET, MOSFET) are distinct enough such that all three types are currently in use.

The MOSFET has a sandwich style construction where an insulating oxide layer separates the metal input gate from the semiconductor channel (Figure 2-1). The input gate voltage creates a electric field across the oxide layer into the semiconductor channel thereby controlling the current through the channel. In this manner a MOSFET acts as a transconductance device with a theoretically infinite input impedance and very low output impedance.

The impedance characteristics of MOSFETs make them an ideal input stage device for potentiometric measurements; indeed all potentiometers utilize MOSFETs for this purpose. The ISFET was created by moving the input MOSFET into the sample solution. In Section 1.2.6 the relationship between ISEs, CWEs, and ISFETs was

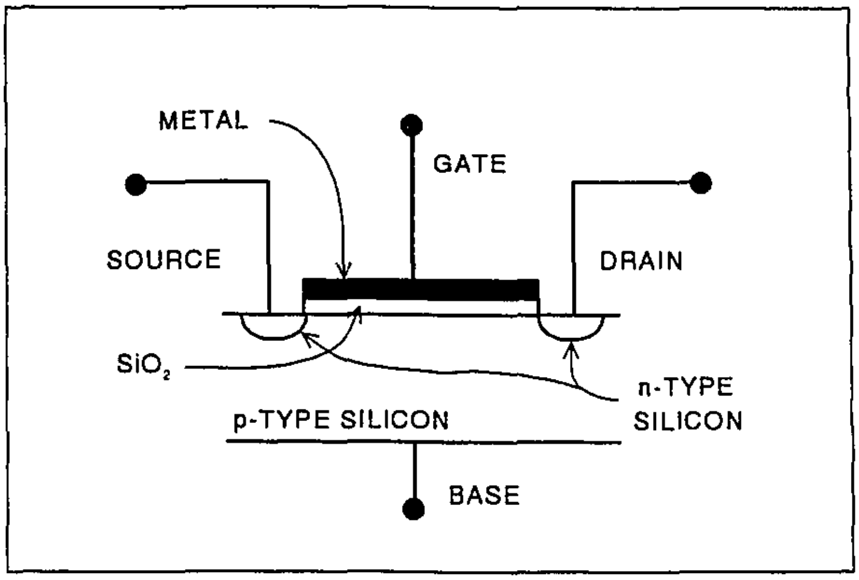

Figure 2-1. Simple model of a MOSFET

examined. While this conceptual relationship is convenient, it does not accurately portray the chronological order of development. In fact, ISFETs and CWEs were developed around the same time, but independently of each other.

In this chapter we will first summarize the history of the development of ISFETs and discuss the different types of FET-based sensors available. Following this, the operation of MOSFETs will be examined, and the theories extended to the use of ISFETs. The last part of this chapter looks at the importance of the reference electrode and introduces the concept of differential sensing.

2.2 Development of ISFETs

The first report of an ISFET was by Bergveld [4] in 1970 when he exposed the silicon dioxide insulating layer directly to solutions of varying Na^+ activity. In his next paper [5], the response for H^+ was described. Matsuo and Wise [6] were simultaneously investigating the operation of a pH ISFET which had a silicon nitride layer exposed to the solution, and their results were published in 1974. Although Bergveld's initial work focussed on the measurement of ionic flux in order to observe variations in electrophysiological potentials, subsequent research has concentrated on the selective determination of ionic activities mainly in biological fluids. ISFETs have also been used as selective detectors in flow-injection analysis, as gas sensors, for micro-titrations, and in process control systems.

Following the initial work, several university groups started research on ISFETs in the 1970's. These include the Universities of Utah, North Carolina, Pennsylvania, and also Stanford and Case Western Reserve in the United States. The University of Twente and, later, the University of Newcastle-upon-Tyne were active in Europe. In Japan there was Tohoku University. In the 1980's additional university, governmental and industrial labs also started research programs. In particular there was a lot of activity in Japanese government and industrial labs. There is a substantial body of work published in Japanese which will not be referenced here.

The development of ISFETs has been well documented. The proceedings from two meetings in 1977 [7] and 1980 [8] cover the early history of ISFETs as well as underlying principles and design considerations. Comprehensive reviews appear routinely which cover the complete field of ISFETs [9-12]. Bergveld and Sibbald's [13] recent book also encompasses the history, theory, construction, and applications of ISFETs. In addition, the fundamental reviews in Analytical Chemistry initiated a Chemical Sensors section in 1988 [14,15] which includes the most recent advances in ISFETs.

A selection of 30 research papers representing innovations in the development of FET-based sensors is given in Table 2.1. Omission from this list should not be taken as a indication of lesser importance. In particular, papers focussing on the theory and response mechanisms of ISFETs were not included.

2.3 FET-Based Sensors

In Table 2.1 there are sensors listed which are selective for non-ionic as well as ionic species. The principles are very similar and research often overlaps amongst the different types. All these sensors

Table 2.1
Development of FET-based Sensors (1)

Author (year)	Analyte	Membrane	Comments	Reference
Bergveld (1970)	Na^+	SiO_2	Constant pH; no reference used	4
Bergveld (1972)	Na^+, H^+	SiO_2	Selectivity varied with each device	5
Matsuo & Wise (1974)	H^+	Si_3N_4	Greater stability; reference was used	6
Moss, Janata & Johnson (1975)	K^+	Val/PVC	First composite membrane Solvent cast polymer over Si_3N_4	16
Lundstrom et al. (1975)	H_2	Pd	First gas sensitive FET	17
Buck & Hackleman (1977)	Ag^+, Br^-	AgBr	Vacuum deposited solid state membrane	18
Esashi & Matsuo (1978)	H^+ Na^+	Si_3N_4 Aluminosilicate	Dual ISFET microprobe structure (200 micron tip)	19
Moss, Johnson & Janata (1978)	Ca^{++}	Orion Ca^{++} ion exchanger/PVC	Also discusses H^+ and K^+ ISFETs	20
Comte & Janata (1978)	H^+	Buffered gel over Si_3N_4	Microreference electrode	21
Bos et al. (1979)	H^+	SiO_2	Optimized for fast titrations	22
Abe, Esashi & Matsuo (1979)	H^+	SiO_2, Si_3N_4, Al_2O_3	Comparision of membranes	23
Caras & Janata (1980)	Penicillin H^+	Penicillinase Si_3N_4	Enzyme FET; differential measurement Enzyme cross-linked with albumin	24
Blackburn & Janata (1982)	K^+	Val/PVC	Suspended polyimide mesh used to aid adhesion of membrane	25
Covington, Harbinson & Sibbald (1982)	Phenobarbital anion	Adogen 464 /PVC	Direct extension of CWE work	26
Nakajima, Esashi & Matsuo (1982)	-	Parylene	Ion-insensitive polymer coated FET	27
Akiyama et al. (1982)	H^+	Ta_2O_5	Different membrane; increased sensitivity	28
Ho et al. (1983)	-		Solid-state, automated encapsulation methods	29
Bergveld (1984)	H^+	Al_2O_3	Dual ISFET chip with multiplexing circuit	30
Sibbald, Covington & Carter (1984)	H^+, Na^+, K^+ & Ca^{++}	-	Extracorporeal blood monitor for 4 ions	31
Brown (1984)	K^+	Val/LB film	New method to immobilize ionphore	32
Winquist et al. (1984)	Urea NH_3	Urease IrPd	Ammonia vapour gas FET suspended above enzyme solution	33
Caras, Petelenz & Janata (1985)	Glucose H^+	Glucose oxidase Si_3N_4	Differential measurement Enzyme entrapped in polyacrylamide gel	34
Miyahara, Moriizumi & Ichimura (1985)	Urea Glucose	Urease/PVA Glucose oxidase/PVA	Silicon-on-sapphire FET multisensor using a photosensitive polymer	35
Sobczynska et al. (1985)	H^+	BN	Better selectivity	36
Sibbald (1985)	H^+ or K^+	Si_3N_4 or Val/PVC	Control circuitry and ISFET combined	37
Satchwill & Harrison (1986)	K^+	Val/PVC	Derivitized PVC to increase membrane adhesion	38
Wakida et al. (1986)	$SeCN^-$	Ionophore/Urushi	Natural lacquer (Urushi) used as matrix	39
Josowicz & Janata (1986)	Alcohols	Polypyrrole/Pt	Suspended-gate work-function gas FET	40
Bataillard et al. (1987)	Ag^+	Silanized SiO_2	Covalent bonding to oxide surface	41
Moritz et al. (1988)	F^-	LaF_3	Vapour deposition of membrane	42

(1) Abbreviations are defined in the glossary (page ix).

share the same transduction element (a FET) as ISFETs; however, the chemical recognition element differs. The variety of FET-based sensors has led to a abundance of acronyms, some of which are defined in the following sections.

2.3.1 Ion-Selective Field-Effect Transistors

A wide variety of gate surfaces, both inorganic and organic, have been used to construct ISFET sensors. Some of these are just adaptations of conventional ISE membranes to an ISFET device such as silver halide solid-state membranes, aluminosilicates for Na^+, or thick film ionophore-doped polymeric membranes (*e.g.* valinomycin/ poly(vinyl chloride) (Val/PVC) for K^+). Of particular interest are ISFETs which incorporate novel sensing mechanisms that cannot be used in conventional ISEs. Langmuir-Blodgett (LB) films can be fashioned to include an ionophore, thereby providing a very thin selective membrane; however there are several difficulties yet to be resolved [32]. Similarly, several groups have used ion-implantation into inorganic insulators, usually to try to make an alkali metal sensor. This technique has met with mixed results.

2.3.1.1 pH ISFETs

The pH ISFET has been the subject of many studies, far more than any other ISFET, and therefore merits special attention. There are three major driving forces for this popularity: 1) extensive use and demand for pH sensors, 2) pH sensitivity of many inorganic "solid-state" surfaces, and 3) they provide a common focal point for different theoretical treatments.

The first devices of Bergveld [4,5] used an SiO_2 gate dielectric exposed to the solution, and while these showed a useful response there were some drawbacks. In the original paper the pH was kept constant while the response to Na^+ was measured; it was only in the second paper that the greater sensitivity to H^+ was described. The SiO_2 acts as a glass electrode because the oxide hydrates; however this eventually leads to the breakdown of the device. Due to the poor stability, durability, and lack of specificity of this surface, a variety of other inorganic films have been investigated. Matsuo and Wise [6] initially used a Si_3N_4 layer on top of the SiO_2 insulator. The Si_3N_4 surface forms a surface hydration layer only. It was shown to be more stable and it also exhibited increased sensitivity and selectivity. Further work led to the use of Al_2O_3 [23], Ta_2O_5 [28] and ZrO_2 [43]. While these compounds have been used successfully as ISFET membranes, they could not be employed as conventional ISE membranes due to their insulating properties. These surfaces can be roughly rated in order of stability, sensitivity, and selectivity: $SiO_2 < Si_3N_4 < Al_2O_3 = Ta_2O_5$. These membranes have also been used in pairs (*e.g.* Si_3N_4 and Ta_2O_5) to provide for differential sensing based on the variation in response sensitivity where the difference in response current is monitored [44]. This method can increase the selectivity and stability.

2.3.2 Gas Sensitive Field-Effect Transistors

In 1975 Lundstrom *et al.*[17] were the first to describe a gas sensitive MOSFET device. This device consisted of a MOSFET with a palladium gate exposed to the gas sample, which responded selectively to H_2. Later work showed that an ammonia vapour sensor could be achieved by adding a catalytic iridium layer to the palladium [33]. Janata's group has focussed on work-function sensors which use a suspended gate of catalytic metal. The selectivity was modified to allow detection of the lower aliphatic alcohols by adding a polypyrrole layer to a suspended platinum gate [40].

Many of the gas sensitive FETs have been given specific names and acronyms; however they can all be considered to fall into the generic category of GasFETs.

2.3.3 Enzyme-Modified Field-Effect Transistors

In order to broaden the range of possible applications and increase the selectivity of ISFETs, enzymes have been incorporated into the chemical recognition stage of ISFETs. The resulting devices are usually termed EnFETs.

The majority of EnFETs reported in the literature utilize a pH ISFET combined with an enzyme either immobilized on the surface or entrapped in a gel layer. The enzyme alters the local pH at the pH sensitive surface. The first functioning device was a penicillin responsive sensor that gave a differential output from a cross-linked albumin/penicillinase film versus a blank cross-linked albumin film [24]. Similar sensors have been constructed for urea, glucose, and acetylcholine. Recent papers involving EnFETs describe the use of a LB film to immobilize penicillinase [45], and a F^- ISFET used in conjunction with co-immobilized glucose oxidase and peroxidase, in the presence of organo-flouro compounds, to monitor glucose [46].

Lundstrom's group has produced a different sort of EnFET [33]. They used a NH_3 sensitive GasFET which was either held above the enzyme solution or separated from the solution by a gas permeable membrane.

An oft-quoted advantage of the first type of EnFET described above is that the small gate area provides for the use of extremely small amounts of enzyme. This is not the case for the gas-sensing FETs.

2.3.4 Miscellaneous FET-Based Sensors

There are a couple of other types of sensors which are not covered in this review that should be mentioned briefly. First there are the immunologically-modified FETs (ImFETs). While several attempts have been made to manufacture such a device, there has been a marked lack of success. These are still very much a "future" possibility. Next there are the modified geometry FETs such as the extended-gate ISFET which facilitates the encapsulation procedure [47], or the micro-ISFET where integrated circuit construction technology is used to simultaneously shape the probe and fabricate the ISFET [19].

Another term which is commonly encountered is chemically sensitive field-effect transistor (ChemFET). Unfortunately this has taken on two different meanings. Sometimes the definition encompasses all of the FET-based sensors described above. A more limited definition narrows the scope to just the FET-based sensors which respond to non-ionic compounds.

2.4 Operational Theory

The relationship between MOSFETs and ISFETs was examined briefly in Section 1.2.6. In this section the basic theories concerning the operation of MOSFETs will be first examined using a simple model, and these theories are then extended to the use of ISFETs.

2.4.1 MOSFETs

The treatment presented here is drawn from Chapter 9 of Muller and Kamins [48]. The reader should consult this reference, or any other text covering field-effect electronics, for a more detailed discussion of the physical electronics of semiconductors.

2.4.1.1 Simple Model

The MOSFET is a four-terminal device as seen in Figures 2-1 and 2-2. A voltage, usually ground, is applied to the base terminal (V_B) which is connected to the body of the device which is *p*-type silicon in this example. Current is measured across the terminals connected to the source and drain junctions which are *n*-type silicon diffused regions. The gate is the input terminal. The voltage present at the gate (V_G) modulates the drain current (I_D), while the insulating oxide layer between the metal gate and the body of the device prevents any current from being drawn from the input gate.

Another name for the MOSFET is insulated-gate field-effect transistor (IGFET). This term is more generic; it covers devices that have insulating layers of silicon dioxide as well as other materials.

2.4.1.2 Modes of Operation

In order for the channel between the source and drain to be

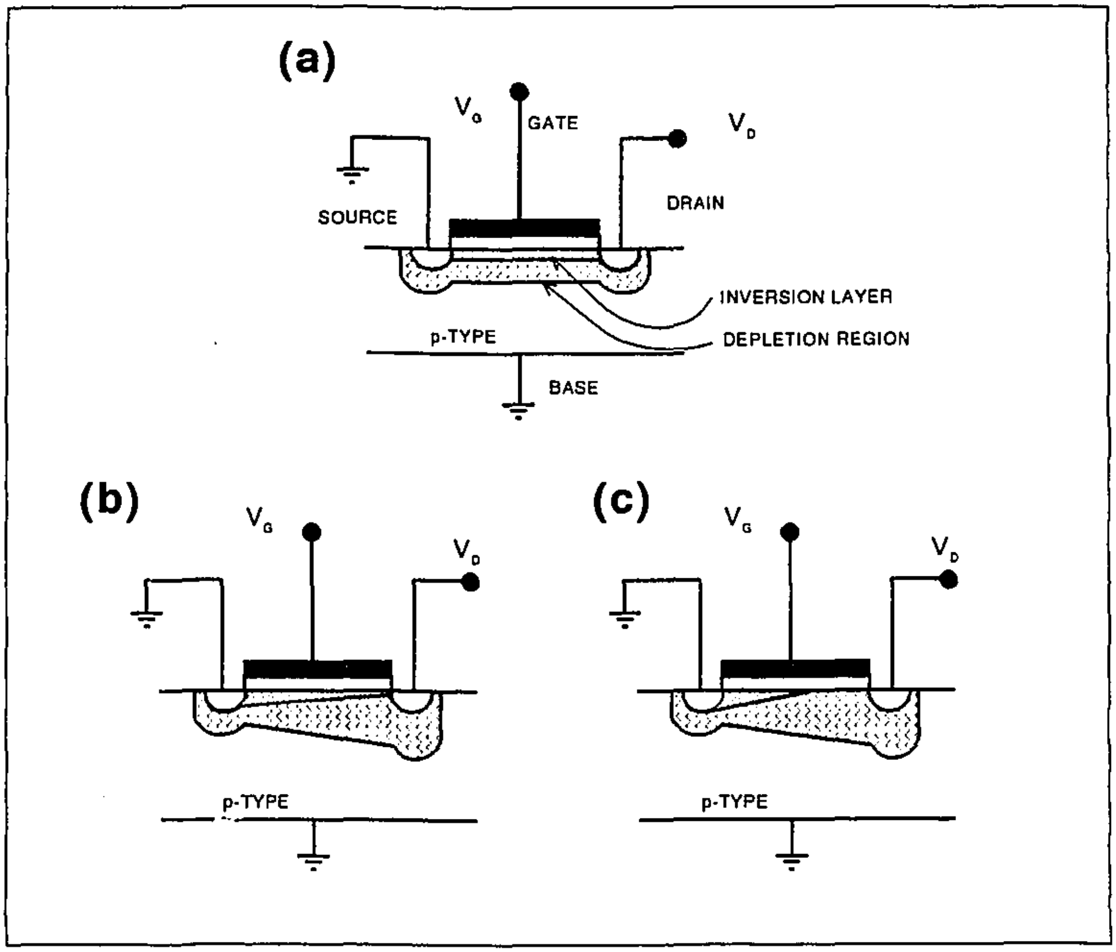

Figure 2-2. MOSFET cross sections with inversion and depletion regions.
(a) $V_G > V_T$, V_D is zero
(b) $V_G > V_T$, V_D is small
(c) $V_G > V_T$, V_D is large

conductive, the field due to the input gate voltage must cause an inversion layer in the *p*-type silicon. The inversion layer is shown in Figure 2-2. (In this discussion and in Figures 2-2 and 2-3, the base and source terminals are grounded ($V_B = V_S = 0$ V). This is the standard setup. All voltages are relative to V_B.)

When V_G is increased from zero volts, a depletion region is formed first as the positive holes are repelled from the Si|SiO_2 interface. As V_G is increased further to a voltage greater than the threshold voltage (V_T), the inversion layer is created at the interface (Figure 2-2(a)). This is effectively a channel of *n*-type silicon. The V_T of a MOSFET is characteristic of the particular device and is dependant on the physical and chemical design parameters. When V_G is greater than V_T, any variations in V_G alter the electron density in the channel thereby modulating I_D. This output current flows from the source through the channel to the drain when a voltage is applied to the drain (V_D) (Figure 2-2(b)). In Figure 2-2(c) V_D has been increased to the point that the electron density around the drain approaches zero and the channel is pinched off. Current still flows but is not affected by any further changes in V_D. The transistor is said to be saturated.

There are thus three distinct states possible for a MOSFET. If V_G is less than V_T, the transistor is turned off. When the transistor is turned on it can operate in either an unsaturated or a saturated mode. This can be seen in Figure 2-3. The dashed line separates the unsaturated region to the left from the saturated region to the right. This line indicates the drain voltage at the onset of saturation ($V_{D(sat)}$) which can be calculated according to Equation 2.1.

$$V_{D(sat)} = V_G - V_T \tag{2.1}$$

In the unsaturated region the current response follows Equation 2.2:

$$I_D = \frac{\mu_n C_{ox} W}{L} \left[\left(V_G - V_T \right) V_D - \frac{V_D^2}{2} \right] \tag{2.2}$$

where μ_n is the electron mobility, C_{ox} is the oxide capacitance, W is the width of the channel and L is the length of the channel (distance between source and drain).

In the saturated region V_D is greater than $V_{D(sat)}$, however the transistor acts as if only $V_{D(sat)}$ is applied. The value of $V_{D(sat)}$ from Equation 2.1 can therefore be inserted into Equation 2.2 to give:

$$I_D = \frac{\mu_n C_{ox} W}{2\,L} \left(V_G - V_T \right)^2 \tag{2.3}$$

for the current response in the saturated region.

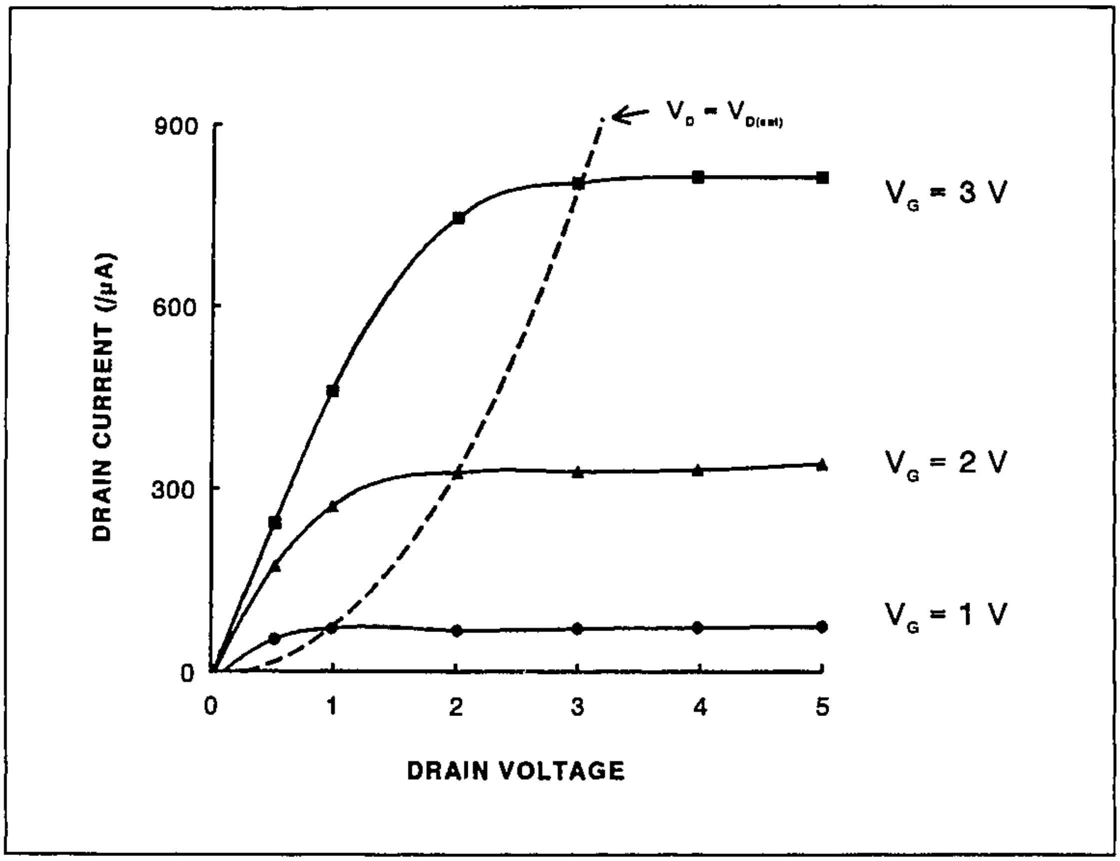

Figure 2-3. MOSFET response curves.
$V_T = 0$ V; $V_B = V_S = 0$ V
(This data is actually from a Ph ISFET in 0.1 M KCl versus a SCE reference electrode (File CH36-S2.TST))

There are distinct advantages to working in both regions. In the unsaturated region I_D responds linearly to changes in V_G, but is susceptible to any variation or noise in V_D. Operation in the saturated region gives a larger I_D which is unaffected by V_D but the response is non-linear.

2.4.1.3 Types of MOSFETs

There are four possible types of MOSFETs. The above example has a *p*-type silicon base with a source and a drain of *n*-type silicon. This is called a *n*-channel MOSFET. In a *p*-channel MOSFET the base is *n*-type and the diffused junctions are *p*-type. The theory for *p*-channel MOSFETs is analogous to the theory given for *n*-channel devices.

Both *n*-channel and *p*-channel MOSFETs can be further classified as either enhancement-mode or depletion-mode. A *n*-channel enhancement mode MOSFET requires that a positive voltage be applied to the gate in order for the inversion layer to exist ($V_T > 0$). In a *n*-channel depletion-mode device the channel is created by ion-implantation. The channel is present even with a negative V_G applied ($V_T < 0$), and the gate is used to reduce the channel conductance. Similar enhancement and depletion-mode devices also exist for *p*-channel MOSFETs.

2.4.2 ISFETs

The operational theory for a MOSFET can be easily extended, in an empirical sense, to describe the response of an ISFET. A comparison of the MOSFET and ISFET reveals many similarities. The ISFET of Figure 2-4 differs from the simple model of Figure 2-1 only in the presence of a reference electrode|solution|insulator interface in place of the metal gate|insulator interface. The metal electrode of the reference can be considered as the gate, and the potential contributions due to E_{ref}, E_{lj} and the pH-dependant surface can all be considered as contributing to the threshold potential. All the constant terms can be collected in $V_T{}^*$, leaving the surface potential φ_o as the only variable. In the unsaturated region, Equation 2.2 can be rewritten as:

$$I_D = \frac{\mu_n C_{ox} W}{L} \left[\left(V_G - V_T^* - \varphi_o \right) V_D - \frac{V_D^2}{2} \right] \qquad (2.4)$$

and the same change can be made to Equation 2.2.

When conventional membranes such as Val/PVC are coated on an ISFET, Nernstian response can be achieved and the Nernst term can be used to replace φ_o:

$$\varphi_o = \frac{RT}{z_A F} \ln a_A \qquad (2.5)$$

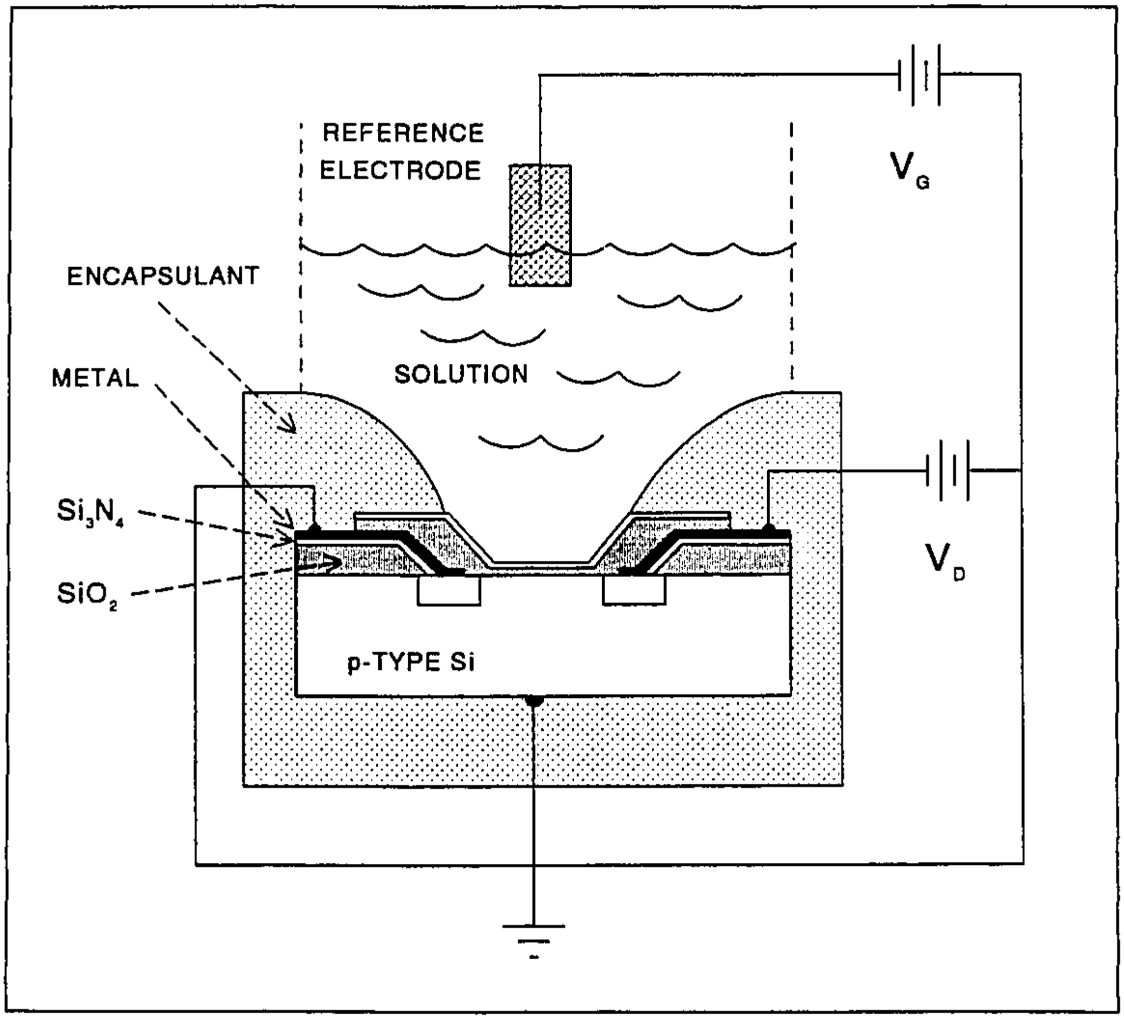

Figure 2-4. Schematic of an ISFET (not to scale).

For the inorganic membranes used for pH determination sub-Nernstian response is the norm and Equation 2.4 represents the response of the device more truthfully.

2.4.2.1 Mechanism of Response for the pH ISFET

The first ISFETs that were studied used a SiO_2 membrane. Because of the similarity to conventional ISE glass membranes, initial attempts at developing an operational mechanism for the ISFET were based on the theory for hydrated glass electrodes. This did not adequately explain the sub-Nernstian response observed for the SiO_2 ISFET, and was found to be inapplicable to ISFETs using other membranes such as Si_3N_4 and Ta_2O_5 which do not form a hydrated surface layer.

Siu and Cobbold [49] were the first to apply the site-binding model to ISFETs. This treatment was developed initially to study the oxide/electrolyte interface in colloid chemistry [50]. In this model the surface potential (ψ_o) is developed due to the acid/base chemistry of amphcteric surface hydroxyl groups. Electrolyte components bind reversibly to these surface sites and there is no transport or diffusion into the bulk of the membrane. The pH sensitivity of the insulating layer has been shown to depend on the site density and the reactivity of the site as expressed by the acid and base dissociation constants [51,52]. The membrane used in this thesis, Si_3N_4, forms an oxynitride surface layer which provides the necessary hydroxyl groups, and the response adheres to this model.

The site-binding model has been compared to other models involving diffusion into the bulk in a paper by Bousse and Bergveld [53]. They found that for gate materials which do not hydrate the site binding model is accurate. (In their work the term site-dissociation is used instead of site binding.) For SiO_2 the presence of buried OH sites was proposed as an explanation for behaviour which did not follow the site-binding model. Sandifer [54] has also examined this topic. His paper focuses on the effects of non-specific adsorption and its relationship with the different models.

2.4.2.2 Reference Electrodes for ISFETs

In Bergveld's original work [5] he introduced the possibility that an ISFET could be operated without a reference electrode. This idea was greeted with skepticism and several papers proved that a reference electrode was necessary if the ISFET was to function as a potentiometric sensor [16,55]. The thrust of these arguments was that in order to measure the potential across an interface, the potential on one side must be fixed with a reference electrode. This has been acknowledged by Bergveld [56]; however, he points out that his suggestion was that an ISFET without the gate could monitor the surface charge, not potential, which could be modulated by the solution composition. A device of this type could not accurately be called a FET. This direction of research was not pursued further.

All present ISFET research is conducted with some type of reference electrode. Unfortunately the best reference electrode is still a conventional style saturated calomel electrode (SCE) or silver-silver chloride electrode. This eliminates some of the benefits of ISFETs. To fully exploit the small size of ISFETs a micro-reference of similar dimensions is necessary.

Three approaches have been used to try to develop a micro-reference electrode. The most obvious route is to just make a micro version of a conventional electrode. For simplicity and safety, considering the possibility of *in vivo* use, the calomel electrode has been ignored and attention has focussed on the Ag/AgCl electrode. The main problem with these electrodes is that the internal solution is reduced by the size restrictions to microliter volumes. This considerably reduces the lifetime and stability of the device. Attempts to improve this type of reference have included the use of a gel as the filling solution and reduction of the porosity of the plug that allows solution contact. Neither of these improvements has overcome the inherent problems.

The second type of micro-reference electrode eliminates the internal solution completely and exposes the electrode directly to the sample. A bare Ag/AgCl electrode requires that the chloride activity of the sample be constant. This is not always a valid assumption. Nevertheless, this type of reference may be useful for specific situations where accuracy is not important.

The last micro-reference electrode we will consider uses differential sensing and three electrodes. An ISFET is paired with a ion-insensitive reference FET (ReFET) and the gate voltage is applied by the third electrode, known here as the gate electrode. The difference in the currents for the two FETs is monitored. While the gate electrode can be a regular reference electrode, ideally it would be replaced with a simple metal electrode. This ReFET configuration is well suited to be used in a microsensor; however there are definite problems with both the use of a metal gate electrode as well as the methods used to render the ReFET insensitive. This topic is investigated in detail in Chapter 7.

It is desirable that the micro-reference electrode be incorporated into the ISFET chip using microelectronic engineering techniques, rather than just using smaller tubing, wires *etc.*. This should enhance the stability, reproducibility and ease of handling of the overall sensor. A device of the first type has been constructed which had a micro-machined cavity with an internal Ag/AgCl electrode and a porous Si plug [57]; however it did not overcome the problem of reduced internal solution volume. The second type is the simplest to implement, but can only be used in a few specific instances. The last type is a viable micro-reference electrode only if a metal gate electrode can be used.

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CHAPTER 3

DESIGN AND CONSTRUCTION OF THE FET CONTROLLER

3.1 Introduction

ISFET research can be roughly divided into three branches. The most basic research focuses on the actual semiconductor device itself. This area requires access to integrated circuit (IC) design and production facilities. The next level of research involves the post-fabrication modification of ISFETs. Essentially, this is the study of selective membranes which are attached by chemical or physical means to the ISFET gate. The final research area is concerned with the control electronics and end-use applications of ISFETs.

The research undertaken for this thesis addressed the last two research areas. One of the first tasks was to decide how the ISFETs would be operated. Unlike ISEs and CWEs, which can be hooked up to a potentiometer, ISFETs require control instrumentation which is not commercially available. The instrument must control the voltages at the four ISFET terminals (base, source, drain and reference), while monitoring the output current.

An instrument was designed and fabricated in order to study the operation and modification of ISFETs. This instrument was named the FET Controller. Its design and construction is detailed in this chapter. The operation of the FET Controller is described in Chapter 4, along with the computer software which completes the system. The instrument is also used, after some modifications, in Chapter 7.

3.2 Design Considerations

Before designing an instrument, the desired capabilities must be clearly defined so that the instrument will be able to fulfill its purpose. This is difficult in basic research, however, because the results of each experiment may change the course of the research, or even alter the ultimate goal of the research. Thus, one of the prime requirements that a research instrument must meet is adaptability. It should be possible to modify the instrument in order to meet unforeseen demands, as well as the expected requirements.

One desired capability was that the instrument should be able to control an array of ISFETs. This was to facilitate testing of a series of membranes of varying composition. For example, an array of K^+ ISFETs could be tested simultaneously in a single solution. With this setup, the effects of a variety of external parameters, such as stirring rate and temperature, are reduced within the individual runs. An added benefit is a decrease in the experimental time.

An ISFET array may exist on a single IC chip, be composed of several chips containing single ISFETs, or some combination of these two extremes. The IC chips used in this research incorporated two ISFETs and two IGFETs on each chip. The use of several of these chips allows for concurrent monitoring of separate solutions with multiple ISFETs. This capability fits the requirements for dissolution testing, which was initially considered as a possible application. Dissolution testing is used in the pharmaceutical industry to determine the rate of solvation of a pill or capsule in an aqueous system. To conform to government and industry standards, the apparatus used for dissolution tests must have six separate vessels. With this application in mind, it was decided that the FET Controller should control 6 chips (containing 12 ISFETs and 12 IGFETs).

The simultaneous control of, and data acquisition from 12 ISFETs, with the analysis of the results by hand, would have been difficult and time consuming. To avoid these problems, the FET Controller had to be computer operated. The computer was used to acquire, display, and store the data in real time as well as for subsequent analysis. The use of computer control also increased the adaptability of the instrument system because the computer programs could be modified independently, or in conjunction with the FET Controller circuitry.

The ability to perform comprehensive tests on a single ISFET, or IGFET, was also desired. This was to enable the determination of the various electronic parameters that apply to all FETs, such as the characteristic drain current versus drain voltage curves seen in Figure 2-3. For this mode, the signal had to be sampled as directly as possible while the gate and drain voltages were varied.

3.2.1 The ISFET Chip

Before the FET Controller could be designed and constructed, the ISFET chips it was to control had to be considered. The ISFET chips were obtained from Dr. Jiri Janata of the University of Utah, Salt Lake City, UT. The chip that was used was the UU03 [1,2], shown in Figure 3-1. The equivalent circuit is given in Figure 3-2(b). The exposed gate consists of 80 nm of Si_3N_4 on top of 80 nm of SiO_2.

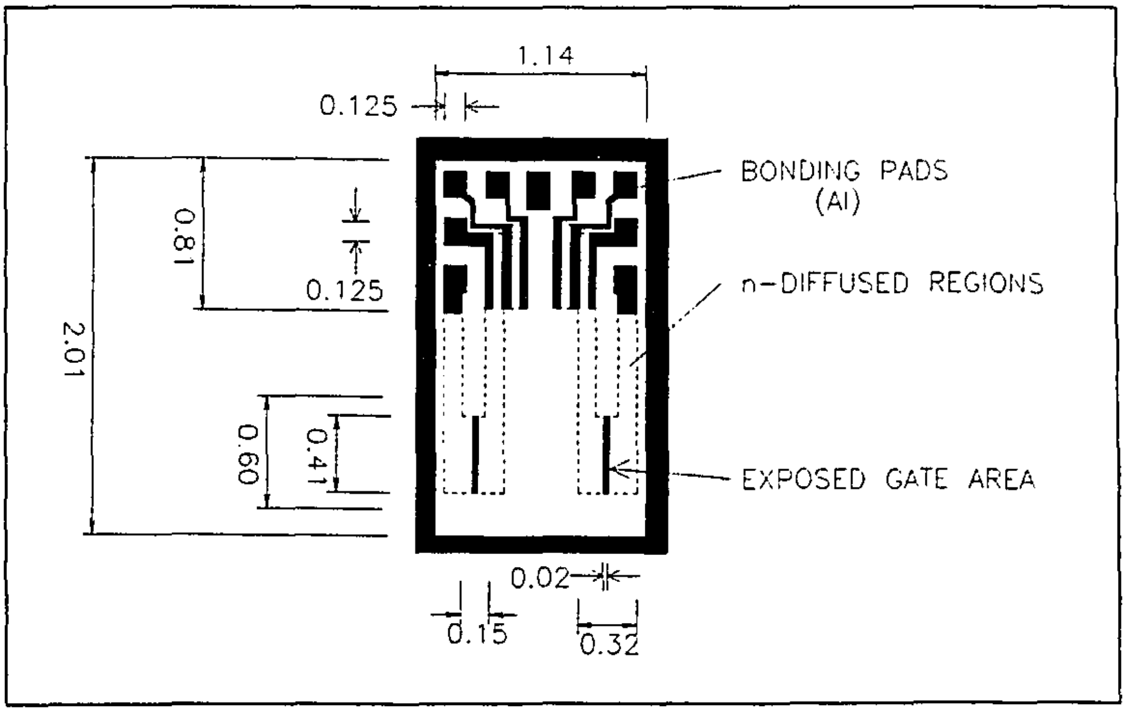

Figure 3-1. Schematic diagram of UU03 chip.
All dimensions are in mm. (scale = 2.54cm:1mm)

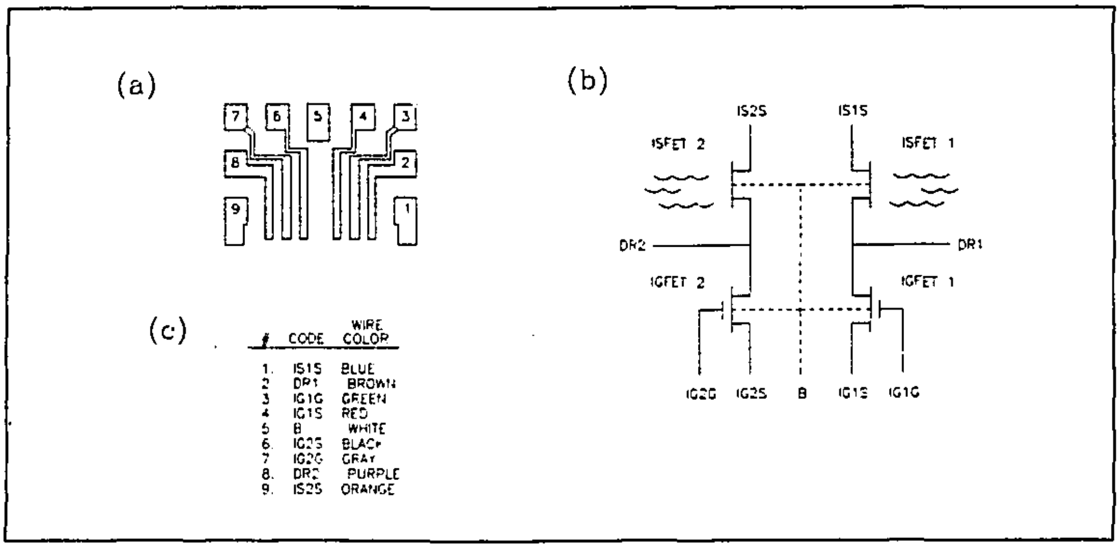

Figure 3-2. Electrical connections and equivalent circuit for UU03 chip.
(a) Bonding pads. (b) Equivalent circuit.
(c) Relationships between bonding pad #, function, and wire color for external connection

The ISFET chips had to be mounted, wired, and encapsulated before they could be used. The chip was mounted on a glass ceramic die on which a thick-film gold pattern had been previously inlaid. The chip was wire-bonded to the gold inlay. Figure 3-3 shows the die and chip at this point. External connection to the FET controller was achieved by soldering hook-up wires to the larger rectangular areas of the gold inlay. The device was then encapsulated in epoxy. The details of this entire process are given in Section 3.4

The final device consisted of an encapsulated ISFET at the end of a nine-wire cable. The free end of the cable was connected to the FET Controller according to the relationships shown in Figure 3-2(c). Each FET was identified by its chip number, CH##, followed by S1, S2, G1 or G2 to designate ISFET 1 or 2, or IGFET 1 or 2, respectively, according to Figure 3-2. For example, CH22-S2 designates the second ISFET on chip 22.

3.3 FET Controller Design

There are two possible modes of operation for an ISFET. In the

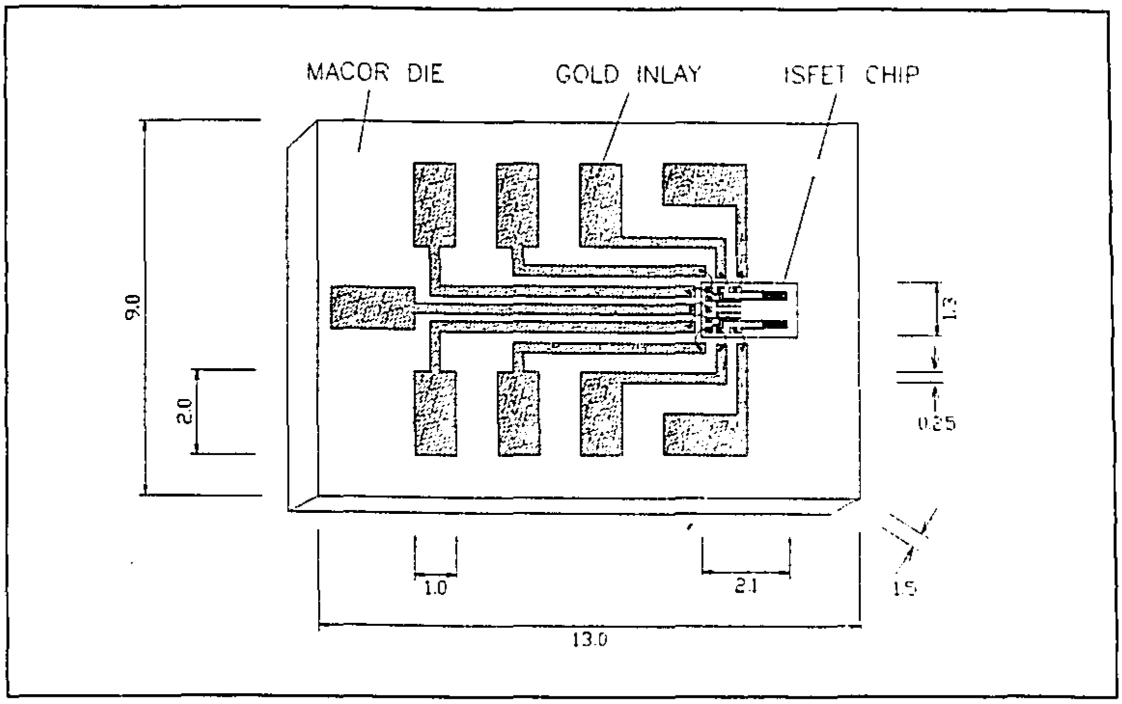

Figure 3-3. Schematic of ISFET chip mounted and wire-bonded on die.
All dimensions in mm.

first mode, all of the control voltages are held constant while the output current is monitored. This is known as *constant voltage* operation. The second mode is *constant current* operation. The output current, I_D, is held constant by using feedback to vary V_G (the reference voltage). Monitoring V_G provides a direct measure of the changes in the surface potential. The operational theory given in Section 2.4 was based on constant voltage operation. The theory is also valid for constant current operation, however the equations can be rewritten to solve for V_G.

Constant current operation is preferred because of the direct correspondence between the output and the surface potential. Unfortunately, this mode cannot be used to operate an array of ISFETs which all share the same reference electrode. In this situation, the reference electrode potential must be held constant.

An alternative approach was used by Haemmerli *et al.*[3] which combined features of constant voltage and constant current operation. This was the mode of operation chosen for use in the FET Controller. The circuit is detailed in the following section. Once the ISFET control circuit was finalized, the demands that the rest of the FET Controller components had to meet were also known. These were satisfied by use of a digital-to-analog (D/A) system to provide the necessary control voltages, and an analog-to-digital (A/D) system to acquire the data.

3.3.1 ISFET Control Circuit

The circuit of Haemmerli *et al.* was designed to use either one of the two matched ISFET/IGFET pairs on the UU03 chip (described in Section 3.2.1). This circuit was modified slightly for use in the FET Controller. The complete ISFET control circuit for a single ISFET is shown below (Figure 3-4). Although this is a constant voltage circuit, it does provide a direct measure of the changes in surface potential, as is found in constant current circuits. It achieves this effect by using feedback to the IGFET as follows.

The source terminals of the ISFET (S0S) and IGFET (G0S) form

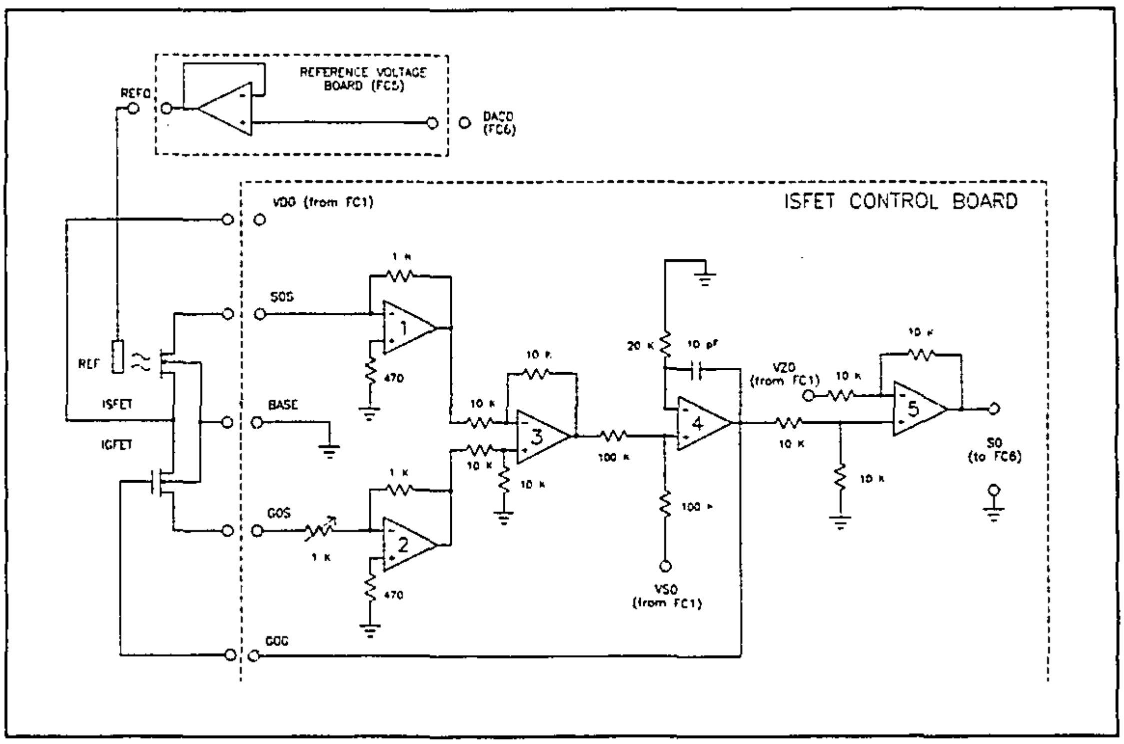

Figure 3-4. Schematic diagram of ISFET control circuit.
Function of operational amplifiers: 1, 2 - current-to-voltage converters; 3 - differential amplifier; 4 - feedback stage; 5 - output buffer.

the inputs to a matched pair of current-to-voltage converters. The source voltages are zero because the inverting inputs to the operational amplifier (op-amp) are virtual grounds. The voltage outputs from these two converters are then fed into a differential amplifier. The noninverting input of the feedback stage is also a virtual ground. Therefore the output of the differential amplifier must be equal to -VS0. The feedback stage must produce an output voltage which, when fed back to the IGFET, makes it possible to maintain this condition. This voltage is the output of the circuit. The last op-amp merely serves as an output buffer to isolate the control circuit from the A/D circuit input.

The effect of this circuit is to keep the difference in the drain currents constant. The magnitude of the difference is dependant on VS0 and, of course, the ISFET and IGFET themselves. In this manner, the feedback voltage, or IGFET gate voltage, mirrors the changes in the surface potential of the ISFET. The other voltages that must be controlled are VD0, the drain voltage, and VZ0 which is present to offset the output voltage. (The output offset was necessary to keep the output within the voltage range of the A/D converter.)

For the direct testing of single ISFETs or IGFETs the output of op-amp 1 was monitored. (For the IGFET, the metal gate, G0G, was connected to REF0.) In this fashion the output of the FET passes through only a single current-to-voltage stage before being input to the data acquisition board. This is not shown in Figure 3-4.

3.3.2 Digital-to-Analog Control Circuit

There were 8 voltages that had to be controlled in the ISFET control circuit. The circuit itself controlled the source voltages (S0S and G0S), the common base terminal, and the IGFET gate (G0G). The reference voltage (REF0) was set directly by a D/A converter included on the commercial data acquisition board.

This left the common drain voltage (VD0), the set voltage (VS0) and the zero offset voltage (VZ0) to be controlled for each ISFET. The 12-ISFET array therefore required a total of 36 voltage inputs. The data acquisition board had only 2 D/A converters, with one already assigned to control REF0. A possibility that was considered was to use the acquisition board's remaining D/A converter combined with a series of sample-and-hold circuits. This idea was rejected because of the excess computer time that would have to be spent on periodically refreshing the voltages. A hardware intensive method was used instead, whereby a series of 36 D/A converters, built into the FET Controller, were used to individually control the voltages. Because of their ability to hold a constant output voltage, these converters had to be set only when the voltage needed to be changed. This allowed more computer time to be spent on data acquisition, display and storage.

The D/A converters were controlled by the digital input/output section of the data acquisition board.

3.3.3 Analog-to-Digital Data Acquisition

The requirements for this subsystem were fairly simple. The outputs from the 12 ISFET control circuits (S0-S11) and the test circuit output were all referenced to a common ground, therefore either single-ended or differential inputs could have been used. The A/D converter inputs could have been bipolar or unipolar. This was dependant on the set voltages and offset voltages used.

Most commercial pH/ion potentiometers have a readout resolution of 1 mV. This was taken as the maximum acceptable value for the A/D converter used for the FET Controller. A 12-bit converter was found to be satisfactory. This gave a resolution range from 4.88 mV on a ±10V scale to 0.30 mV on a +1.25V scale. While a 14- or 16-bit converter would have been useful, these would have had to be purchased while a 12-bit board was already available in the laboratory.

3.4 Processing of ISFET Chip

3.4.1 Preparation of Die

The dies were fashioned from a 7.6 x 7.6 x 0.16 cm sheet of Macor, a machinable glass ceramic made by Corning Glass Works (Corning, NY). The process for the metallization of the Macor was derived from previous work in our laboratory [4].

The Macor was cut into individual dies measuring 13.0 x 9.0 x 0.16 mm with a diamond blade saw. The surface was ground and polished on an Ecomet II polisher/grinder (Buehler Ltd., Lake Bluff, IL), using water as a lubricant. All grinding was done with Norton Tufbak Durite T421 diamond grit sandpaper (Pascal Hardware, Montreal, Que.). Polishing was performed with various grades of Micropolish II aluminum oxide on a Microcloth (all from Buehler Ltd.). The Macor was polished, degreased, and dried as follows:

1. Grind with 400 grit paper for 1 min.
2. Grind with 600 grit paper for 1 min.
3. Sonicate in distilled water for 5-10 min.
4. Sonicate in acetone for 5-10 min.
5. Dry in 130 °C oven for 10 min.
6. Polish with 14.5 μm aluminum oxide for 30 s.
7. Polish with 1.0 μm aluminum oxide for 30 s.
8. Polish with 0.3 μm aluminum oxide for 1 min.
9. Sonicate in 50:50 acetone:trichloroethylene for 15-30 min.
10. Rinse with distilled water.
11. Dry in 130 °C oven for 1 h. Dies should be vertical.

The die was then coated with a positive photoresist made by MG Chemicals (Toronto, Ont.). One side of the die was dipped in a 1:2 emulsion:thinner (MG#416:MG#417) solution. Excess photoresist was drained off by touching the corner of the die to a Nalgene surface. The resist was then air dried for 2 h in the dark, at room temperature. The other side of the die was coated in the same manner, and the die was dried overnight.

The resist was exposed to light in order to define the gold inlay pattern. The inlay pattern can be seen in Figure 3-3. A positive image of the inlay (*i.e.* gold area was black) was photo-reduced (1/5), and the negative was used as the exposure mask. The mask was positioned on the die. The photoresist was exposed to a 275W sunlamp at a distance of 23 inches for 3 min. The photoresist was developed in a 6:1 water: developer(MG#418), room temperature solution for 30 s. The die was removed and swabbed with a Kimwipe soaked with the developing solution to remove the remaining resist from the inlay areas. The die was washed with distilled water and patted dry with a Kimwipe.

The next step was to etch the inlay pattern using hydrofluoric acid to dissolve the glass component of the exposed Macor. The dies were placed in a 5% HF solution, swirled for 15 min, sonicated for 1 min, and swirled for 2 min more. The dies were then rinsed with distilled water, followed by acetone to remove the resist, water again, then dried for 1 h at 130 °C. The acid etch left a ceramic residue. This was scraped out, using needle probes and a small knife, to leave the recessed inlay pattern. The dies were cleaned and dried by repeating steps 9,10, and 11.

The gold inlay was formed by depositing a gold ink (#A3360, Englehard Industries, East Newark, NJ) on the surface of the die, followed by grinding and polishing of the surface. The die was covered with gold ink, and a glass slide was drawn across the surface to remove excess. The dies were placed in a muffle furnace at room temperature, then the furnace was turned on and heated at the maximum rate to a final temperature of 900 °C. This temperature was held for 15 min, then the door was opened slightly to allow the furnace to cool at a rate of approximately 30 °C/min. A second coat of ink was applied and fired in the same manner.

Steps 2 through 11 of the grinding and polishing procedure were repeated, leaving the thick-film gold inlay on the die surface.

3.4.2 Mounting and Wire-Bonding of the ISFET Chip

The ISFET chip was glued onto the die using Epo-Tek H62 epoxy (Epoxy Technology Inc., Billerica, MA). This epoxy was also used for all subsequent encapsulation steps. The epoxy is a single component, black, thixotropic paste. A small dab of the epoxy was placed on the die and the chip was seated. The epoxy was cured at 130 °C for 1 h.

Wire-bond connections, between the chip and the gold inlay on the die, were made with a hybrid thermosonic wire-bonder, Model 2402-2, equipped with a 2-channel ultrasonic generator, Model 4320A, both made by Kulicke & Soffa Ind. Inc. (Horsham, PA). A 25-μm gold wire was first ball-bonded to the gold inlay, then stich-bonded to the aluminum pads on the chip. The instrument settings were:

stage temperature	170 °C
1st bond power	9
time	6
force	4
2nd bond power	6
time	1
force	7
ball size	6
manual flame	off

The continuity was checked between the gold inlay and the aluminum bonding pads on the chip. Any breaks were repaired with a silver epoxy, Epo-Tek H21-D, which was cured at 130 °C for 15 min.

3.4.3 Encapsulation and External Connection

The ISFET chip was encapsulated with the Epo-Tek H62 epoxy, before the external connections were made, to prevent damage to the wire-bond connections. A very small amount of epoxy was applied to a particular area on the chip or die, *i.e.* one end of the gold wire, and the epoxy was quickly cured by placing the die on a flat surface 5 cm below the nozzle of a heat gun for 5-10 min. This cycle was repeated until the entire chip and wire-bond wires were encapsulated, taking care to leave the ISFET gate areas exposed.

A 1 meter, 9 conductor (multi-strand) shielded cable was used to connect the ISFET chip to the FET Controller. The ends of these wires (1-2 mm) were stripped, tinned, and bent into the proper shape. They were then soldered to the wiring pads, also pre-tinned, on the periphery of the die (Figure 3-5). The electrical connections were tested again.

The die was then completely encapsulated, except for the gate areas. Small windows, approximately 0.8 by 0.5 mm, around each gate allowed the ISFET gates to be exposed to the solution. This was again done by applying small amounts of the epoxy at a time, with curing under the heat gun. The whole cable, die, and ISFET assembly was placed in an 80 °C oven overnight for final curing.

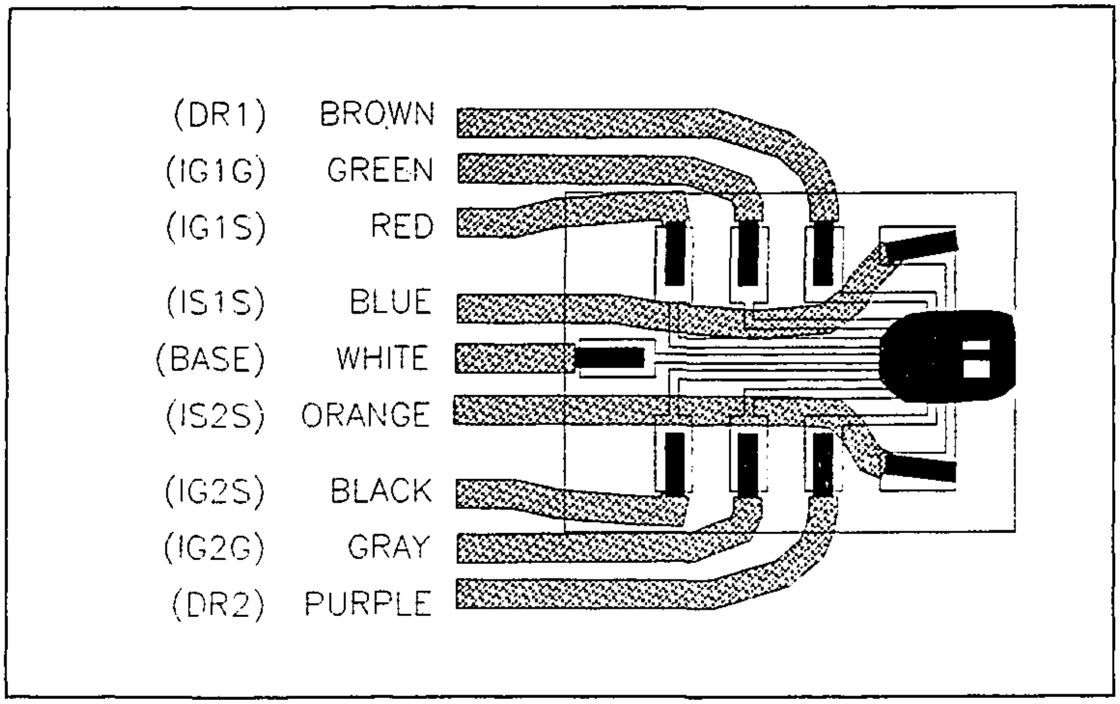

Figure 3-5. ISFET and die prior to final encapsulation.

3.5 Construction of the FET Controller

The FET Controller was contained in an instrument case separate from the microcomputer. The ISFETs and reference electrodes were hooked up to the FET Controller via front panel connectors. All of the control circuits were fabricated on five wire-wrap circuit boards (FC1-FC5) which were plugged into a series of edge connectors that were permanently installed and hardwired in the instrument case. A terminal strip board (FC6), supplied with the data-acquisition board, was also hardwired into the FET Controller. A ribbon cable enabled communication between the instrument and the data-acquisition board in the computer. The power supply and front panel connectors were also permanently installed in the instrument.

A block diagram of the FET Controller appears on the next page (Figure 3-6). The connections between the D/A board (FC1) output voltages (V00 ... V83) and the input voltages (VZ0 ... VS11) on the ISFET control boards (FC2, FC3, and FC4) have been omitted from this diagram for clarity.

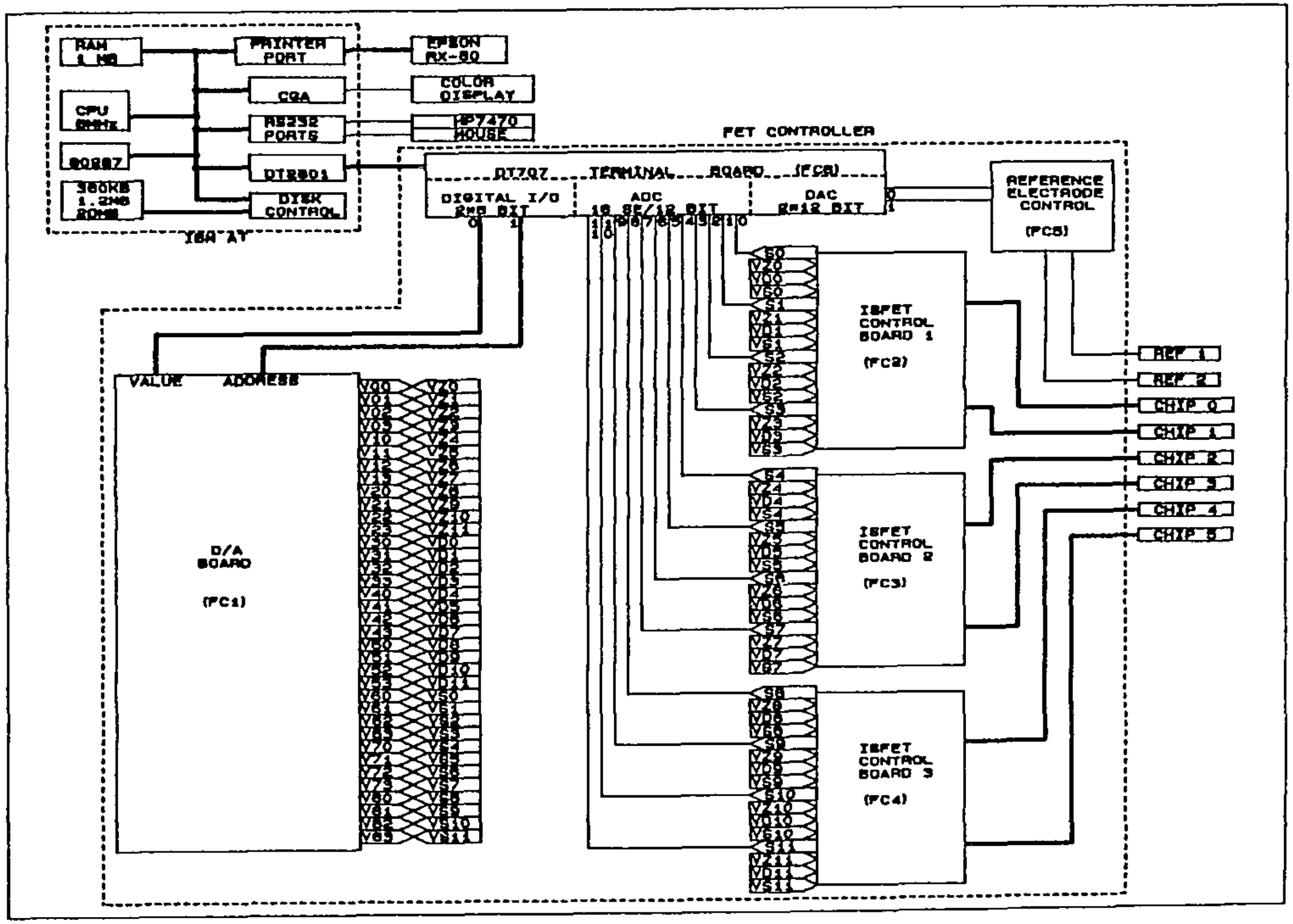

Figure 3-6. Block diagram of FET Controller.

The details of the parts and circuits used in the FET Controller are given in the balance of this section. The computer and data-acquisition board will be discussed in Section 3.6.

3.5.1 FET Controller Parts List

3.5.1.1 Part Nomenclature

Table 3.1 is a complete list of the parts used in the FET Controller. All of the parts on the wire-wrap circuit boards (FC1-FC5) were assigned a reference label. This was to allow cross reference between the schematics, layouts and parts list. The first part of this label is one or two letters describing the type of part (*i.e.* OA - operational amplifier, RP - resistor pack). Following this is a single number indicating which board the part is on (1-5). The number after the hyphen refers to the number of the part on the board. For example:

IC1-9	is the 9th integrated circuit on board FC1
C3-4	is the 4th capacitor on board FC3

The only exceptions to this nomenclature were the capacitors placed between the operational amplifier power supply terminals and ground (labelled CPS), and the power supply and case components.

3.5.1.2 Sources of Parts

The quad D/A converters (IC1-1 through IC1-9) were purchased from BBD Electronique, Pointe Claire, Que. The FC6 board was the remote terminal board (DT707) supplied with the data-acquisition board (model DT2801, Data Translation Inc., Marlborough, MA). The manufacturer's manual should be consulted for further details concerning these two boards.

All other parts were purchased from any of three electronic wholesalers: Electrosonic (Mississauga, Ont.); Active Electronics (Montreal, Que.); or Newark Electronics (Laval, Que.). Some parts came from more than one supplier, or were stocked in the laboratory. Therefore, only the original manufacturer is given in Table 3.1.

Table 3.1
Parts List for FET Controller

Reference	Part	Description	Rating	Manufacturer
FC1	3662-2	wire wrap circuit board	4.5x9.6 inches	Vector
IC1-1 to IC1-9	AD7226KN	quad D/A converter	8 bit	Analog Devices
IC1-10	SN74LS42N	4 to 10 decoder	---	Motorola
VG1-1	uA7905C	-5 V voltage regulator	5%, 100 mA	Fairchild
VG1-2	LM340-5T	+5 V voltage regulator	5%, 1.5 A	Motorola
VR1-1	MC1404U5	+5 V voltage reference	1%, 10 mA	Motorola
VR1-2	MC1404U10	+10 V voltage reference	1%, 10 mA	Motorola
T1-1 & T1-2	3252W	100 KΩ trimpot, 25 turn	5%, 1 W	Bourns
RD1-1	SN75473	dual relay driver	300 mA	Texas Instruments
RR1-1 & RR1-2	835C-1	SPDT reed relay	5 V, 200 Ω	Gordos
RP1-1 to RP1-9	4116R-001	8x10 kΩ dip resistor pack	2%, 2.25 W	Bourns
OA1-1 to OA1-9	TL075CN	quad operational amplifier	---	Texas Instruments
OA1-10 to OA1-12	TL071CP	single operational amplifier	---	Texas Instruments
T1-3 to T1-5[a]	3299W	10 kΩ trimpot, 25 turn	0.5 W	Bourns
FC2, FC3, FC4	3662	wire wrap circuit board	4.5x6.5 inches	Vector
R2-1 to R2-8 R3-1 to R3-8 R4-1 to R4-8	MR25F	470 Ω metal-film resistors	1%, 1/4 W	Philips
R2-9 to R2-16 R3-9 to R3-16 R4-9 to R4-16	MR25F	1 kΩ metal-film resistors	1%, 1/2 W	Philips
R2-17 to R2-32 R3-17 to R3-32 R4-17 to R4-32	MR25F	10 kΩ metal-film resistors	1%, 1/2 W	Philips
R2-33 to R2-36 R3-33 to R3-36 R4-33 to R4-36	MR25F	20 kΩ metal-film resistors	1%, 1/4 W	Philips
R2-37 to R2-44 R3-37 to R3-44 R4-37 to R4-44	MR25F	100 kΩ metal-film resistors	1%, 1/2 W	Philips
RP2-1, RP2-2 RP3-1, RP3-2 RP4-1, RP4-2	4116R-001	8X10 kΩ dip resistor pack	2%, 2.25 W	Bourns
C2-1 to C2-4 C3-1 to C3-4 C4-1 to C4-4	10 pF	NPO ceramic capacitors	---	Philips

(Continued on next page ...)

Continuation of Table 3.1
(Parts List for FET Controller)

T2-1 to T2-4 T3-1 to T3-4 T4-1 to T4-4	3299W	1 kΩ trimpot, 25 turn	0.5 W	Bourns
T2-5 to T2-12 [1] T3-5 to T3-12 [1] T4-5 to T4-12 [1]	3299W	10 kΩ trimpot, 25 turn	0.5 W	Bourns
OA2-1 to OA2-4 OA3-1 to OA3-4 OA4-1 to OA4-4	TL072CP	dual operational amplifiers	---	Texas Instruments
OA2-5 to OA2-8 OA3-5 to OA3-8 OA4-5 to OA4-8	TL071CP	single operational amplifier	---	Texas Instruments
OA2-9 to OA2-12 OA3-9 to OA3-12 OA4-9 to OA4-12	LM741CN	single operational amplifier	---	Motorola
OA2-13, OA3-13 & OA4-13	TL075CN	quad operational amplifier	---	Texas Instruments
FC5	3662	wire wrap circuit board	4.5x2.25 inches	Vector
T5-1, T5-2 [1]	3299W	10 kΩ trimpot, 25 turn	0.5 W	Bourns
OA5-1, OA5-2	TL071CP	single operational amplifier	---	Texas Instruments
---	1452 FF13	instrument case	9.5x9.5x13 inches	Hammond
---	W28	wire wrap wire	---	Vector
---	8529-100	hook-up wire	---	Belden
CPS [2]	10 nF	Mylar capacitors	---	Active Electronics
---	T68, T49	wire wrap posts	---	Vector
---	8 pin, 14 pin	wire wrap dip sockets	---	Texas Instruments
---	14, 16, 20 pin	wire wrap dip sockets	---	Cambion
---	3416	14 pin dip connector for ribbon cable	---	Scotchflex
---	---	44 pin card edge connector	---	EDAC
RELAY	KA11AT	power interruption protection relay	---	Potter&Brumfield
FUSE	AGC2	AC line fuse	2 A, 250 V	Buss
PANEL SWITCH	554-1131-211	front panel switch	---	Dialight
POWER SUPPLY	HPFD 015 006	---	±15 V at 0.6 A	Hammond

(1) Trimming potentiometers are shown in Figures 3-9, 3-11, and 3-12, but not in Figures 3-8 and 3-10. These were used to offset the operational amplifiers.

(2) CPS capacitors were placed across operational amplifier power supplies (±15V) and ground.

3.5.2 Case and Power Supply Connections

The data-acquisition terminal board (FC6), card edge connectors, front panel connectors, and the power supply were permanently installed and hardwired in the instrument case. The ±15 VDC power supply configuration is shown in Figure 3-7. A relay was placed on the incoming AC power line to prevent the FET Controller from turning back on after a power interruption. This was to avoid damage to the ISFET chips due to random voltage settings of the D/A, and surging, upon power up. The D/A board (FC1) included two voltage regulators (VG1-1 and VG1-2) to supply ±5 VDC to various IC chips.

The front panel connectors consisted of two sockets for the reference electrodes, and six snaplock 9-terminal strips for the ISFET chips. The card edge connectors were mounted vertically on an L-bracket of our own design. The wire-wrap circuit boards were plugged into these connectors. The hardwiring of the edge connectors is detailed in Tables 3.2, 3.3, and 3.4 for the D/A board, reference board, and ISFET control boards, respectively.

3.5.3 D/A Board

The D/A board (FC1) uses digital inputs and nine quad 8-bit D/A converters (IC1-1 ... IC1-9) to produce the 36 control voltages required to control the 12 ISFETs. Banks of resistors (RP1-1 ... RP1-9) and op-amps (OA1-1 ... OA1-9) were used to transform the D/A converter's unipolar analog output into a bipolar output. The circuit diagram in Figure 3-8 shows only the first and last banks for simplicity. The board layout in Figure 3-9 includes all the components.

The 36 D/A converters were controlled with the 16-bit digital input-output lines from the data-acquisition board. The D/A converters used 8 bits (Port0, D0 ... D7) for the digital input of the voltage value. Two more bits (Port1, D6 and D8) were used to select either a +5V or +10V reference voltage. (One bit, D6, set the reference voltage for all the zero offsets (VZ's); and the other bit, D7, set the reference

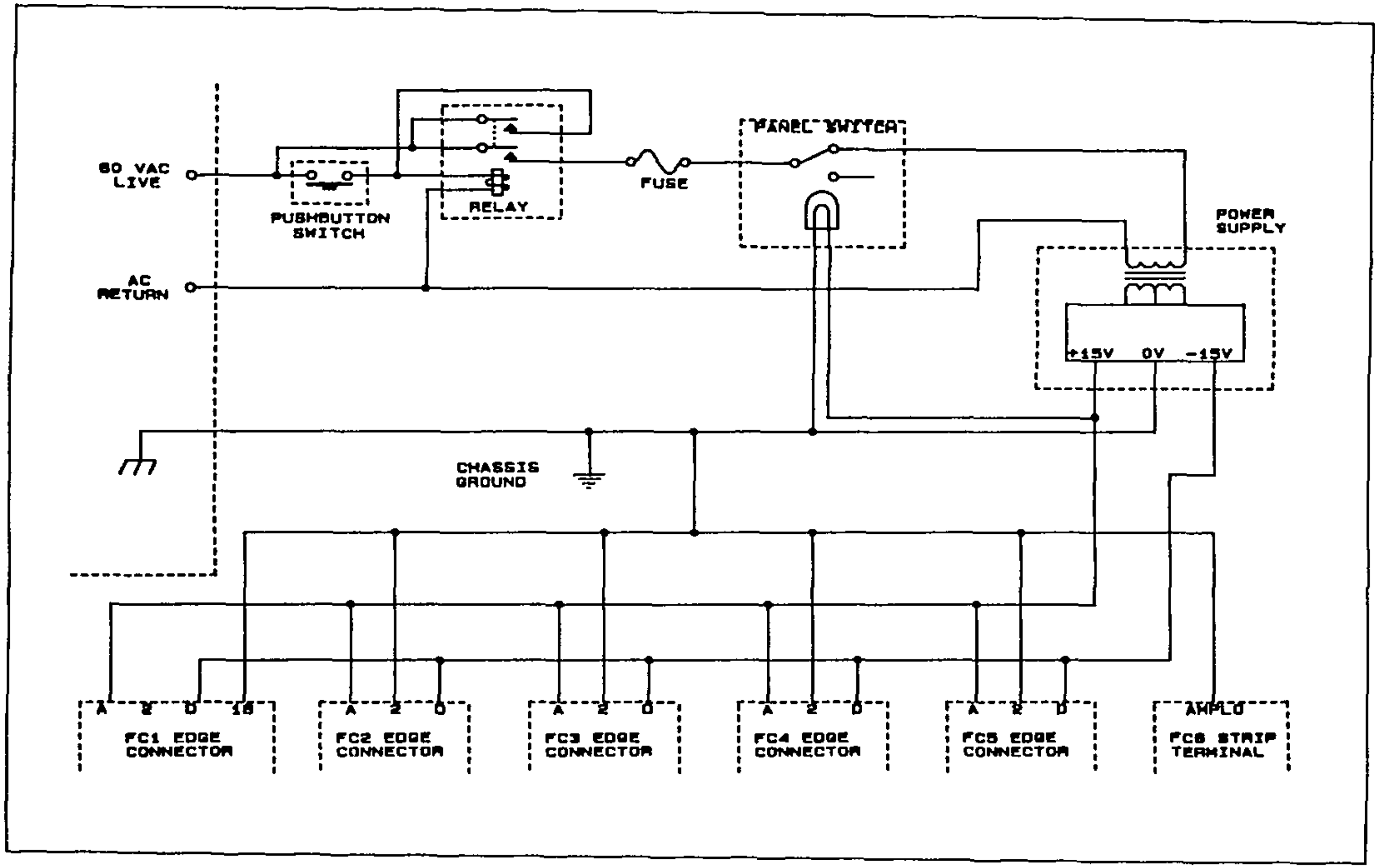

Figure 3-7. Schematic of power supply circuit.

Table 3.2
D/A Board (FC1) Edge Connections

FC1 Edge Connector	FC6 Terminal Strip Digital I/O	
7	Port 0, bit 7	
J	Port 0, bit 6	
L	Port 0, bit 5	
10	Port 0, bit 4	
12	Port 0, bit 3	
P	Port 0, bit 2	
S	Port 0, bit 1	
15	Port 0, bit 0	
H	Port 1, bit 0	
8	Port 1, bit 1	
9	Port 1, bit 2	
M	Port 1, bit 3	
N	Port 1, bit 4	
13	Port 1, bit 5	
14	Port 1, bit 6	
T	Port 1, bit 7	
18	GRD	Power Supply
A	+15 V	
D	-15 V	

Table 3.3
Reference Board (FC5) Edge Connections

FC5 Edge Connector	Connects to	
H	DAC0 on FC6	
L	DAC1 on FC6	
J	REF0 on front panel	
M	REF1 on fron panel	
2	GRD	Power Supply
A	+15 V	
D	-15 V	

Table 3.4
ISFET Control Boards - Edge Connections

Edge Connector	FC2 Board Function	FC2 Board Connects to	FC3 Board Function	FC3 Board Connects to	FC4 Board Function	FC4 Board Connects to
2		GRD		GRD		GRD
A		+15 V		+15 V		+15 V
D		-15 V		-15 V		-15 V
E	B0		B4		B8	
5	S0S		S4S		S8S	
F	S1S		S5S		S9S	
6	DR0		DR4		DR8	
H	DR1		DR5		DR9	
7	G0G		G4G		G8G	
J	G1G		G5G		G9G	
8	G0S	Front panel	G4S	Front panel	G8S	Front panel
K	G1S	connections to	G5S	connections to	G9S	connections to
L	B2	ISFETs 0,1,2,3	B6	ISFETs 4,5,6,7	B10	ISFETs 8,9,10,11
10	S2S		S6S		S10S	
M	S3S		S7S		S11S	
11	DR2		DR6		DR10	
N	DR3		DR7		DR11	
12	G2G		G6G		G10G	
P	G3G		G7G		G11G	
13	G2S		G6S		G10S	
R	G3S		G7S		G11S	
		FC6 A/D Inputs		FC6 A/D Inputs		FC6 A/D Inputs
V	TST	CH12				
W	S0	CH0	S4	CH4	S8	CH8
X	S1	CH1	S5	CH5	S9	CH9
Y	S2	CH2	S6	CH6	S10	CH10
Z	S3	CH3	S7	CH7	S11	CH11

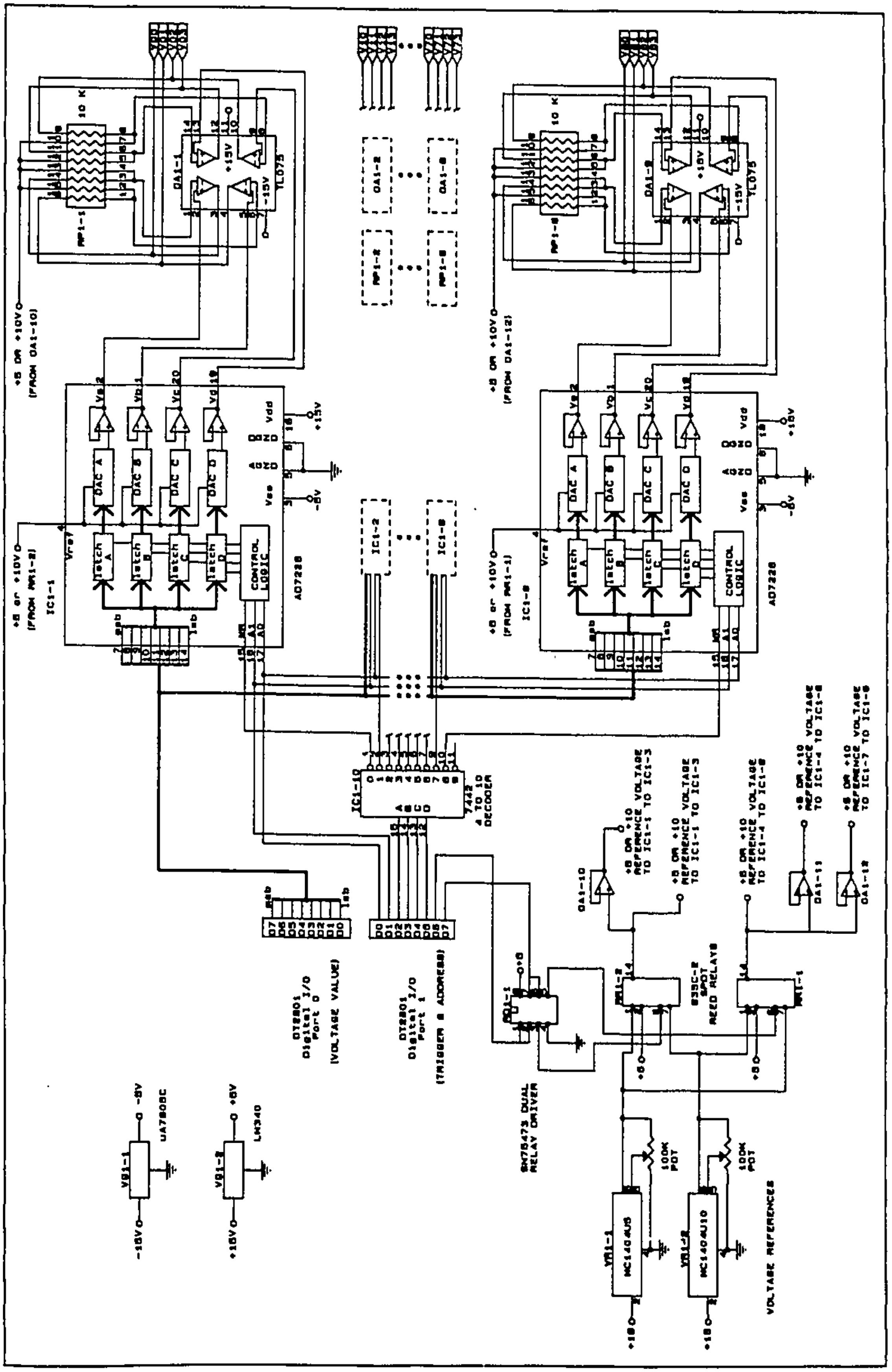

Figure 3-8. Schematic diagram of D/A board (FC1).

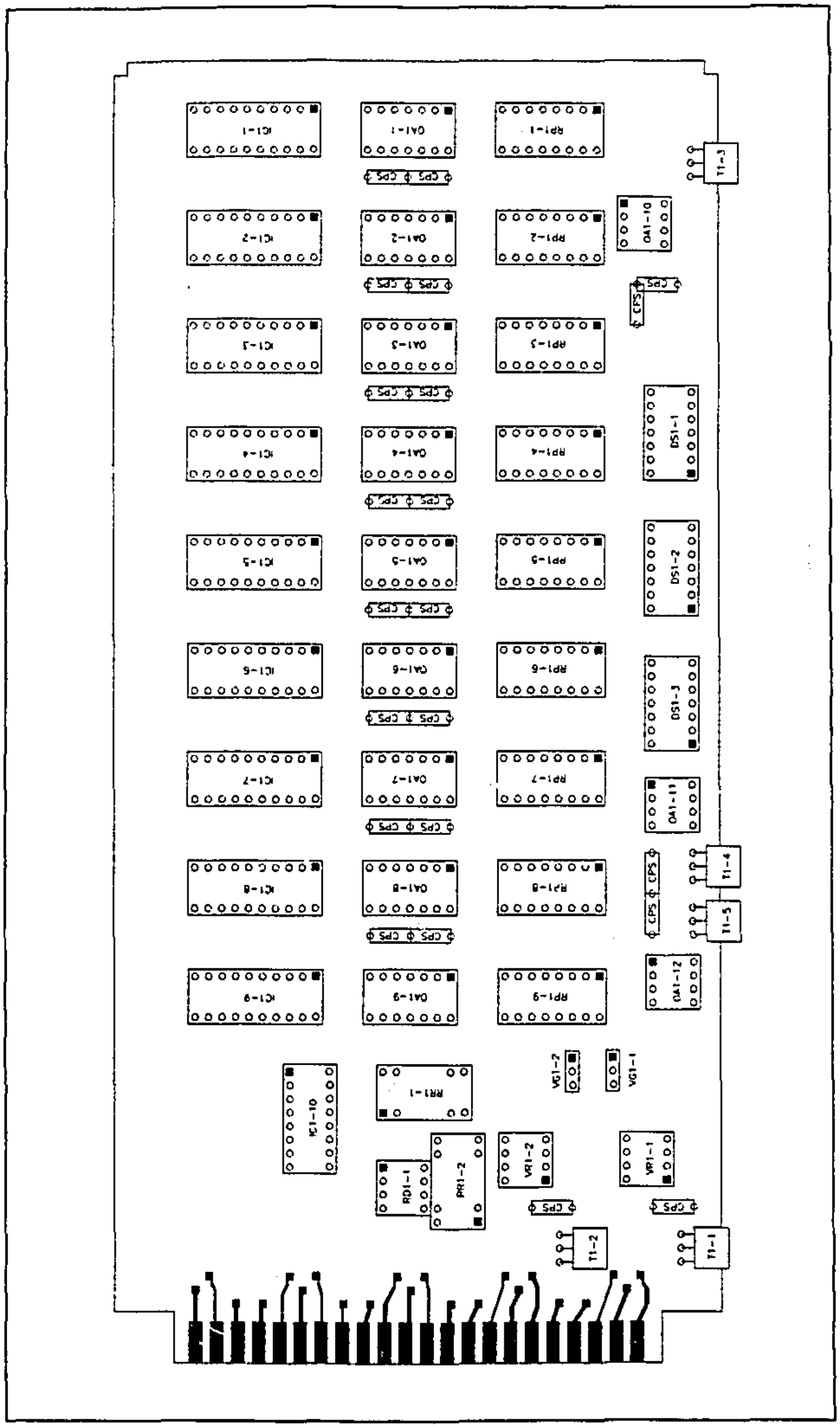

Figure 3-9. Component layout for A/D board (FC1).

for the drain (VD's) and set (VS's) voltages.) The remaining 6 bits (Port1, D0 ... D5) were used to address and trigger the desired D/A converter.

The output voltage specifications for this board are listed in Table 3.5. In practice, imperfections in the op-amps and resistors produced slight deviations from these specifications. The true values were determined experimentally for all 36 outputs and incorporated into the software.

Table 3.5
D/A Board (FC1) Output Specifications

Reference Voltage	Range (V)	Resolution (V)
+5	-5 TO +4.96	0.0390
+10	-10 TO +9.92	0.0781

3.5.4 ISFET Control Boards

The outputs of the D/A board were connected by ribbon cables to the three ISFET control boards (FC2, FC3, and FC4) according to Table 3.6. These boards contain 4 ISFET control circuits as described in Section 3.3.1. The schematic of a complete control board is given in Figure 3-10 and the board layout is shown in Figure 3-11. Each of these boards controls 4 ISFETs, and the matching 4 IGFETs, on two chips.

A dual op-amp was used to provide matching current-to-voltage input stages (op-amps 1 and 2 in Figure 3-4). The differential amplifier and feedback stages used individual op-amps which were trimmed to zero offset. A quad op-amp and two thick-film resistor networks were used for all four output stages (op-amp 5 and the four 10-KΩ resistors).

Table 3.6
Ribbon Cable Connection Between D/A Board (FC1) and ISFET Control Boards (FC2, FC3, FC4)

DAC Output from FC1(1)	DS1-1 Dip Socket Pin #	DS2-1 Dip Socket Pin #	Input Function on FC2(2)
V00	7	7	VZ0
V01	6	6	VZ1
V02	5	5	VZ2
V03	4	4	VZ3
V30	2	2	VD0
V31	1	1	VD1
V32	13	13	VD2
V33	14	14	VD3
V60	8	8	VS0
V61	9	9	VS1
V62	10	10	VS2
V63	11	11	VS3

DAC Output from FC1(1)	DS1-2 Dip Socket Pin #	DS3-1 Dip Socket Pin #	Input Function on FC3(2)
V10	7	7	VZ4
V11	6	6	VZ5
V12	5	5	VZ6
V13	4	4	VZ7
V40	2	2	VD4
V41	1	1	VD5
V42	13	13	VD6
V43	14	14	VD7
V70	8	8	VS4
V71	9	9	VS5
V72	10	10	VS6
V73	11	11	VS7

DAC Output from FC1(1)	DS1-3 Dip Socket Pin #	DS4-1 Dip Socket Pin #	Input Function on FC4(2)
V20	7	7	VZ8
V21	6	6	VZ9
V22	5	5	VZ10
V23	4	4	VZ11
V50	2	2	VD8
V51	1	1	VD9
V52	13	13	VD10
V53	14	14	VD11
V80	8	8	VS8
V81	9	9	VS9
V82	10	10	VS10
V83	11	11	VS11

(1) Voltage outputs from D/A board (see Figures 3-4 and 3-8).

(2) Voltage inputs to ISFET control boards (see Figures 3-6 and 3-10).

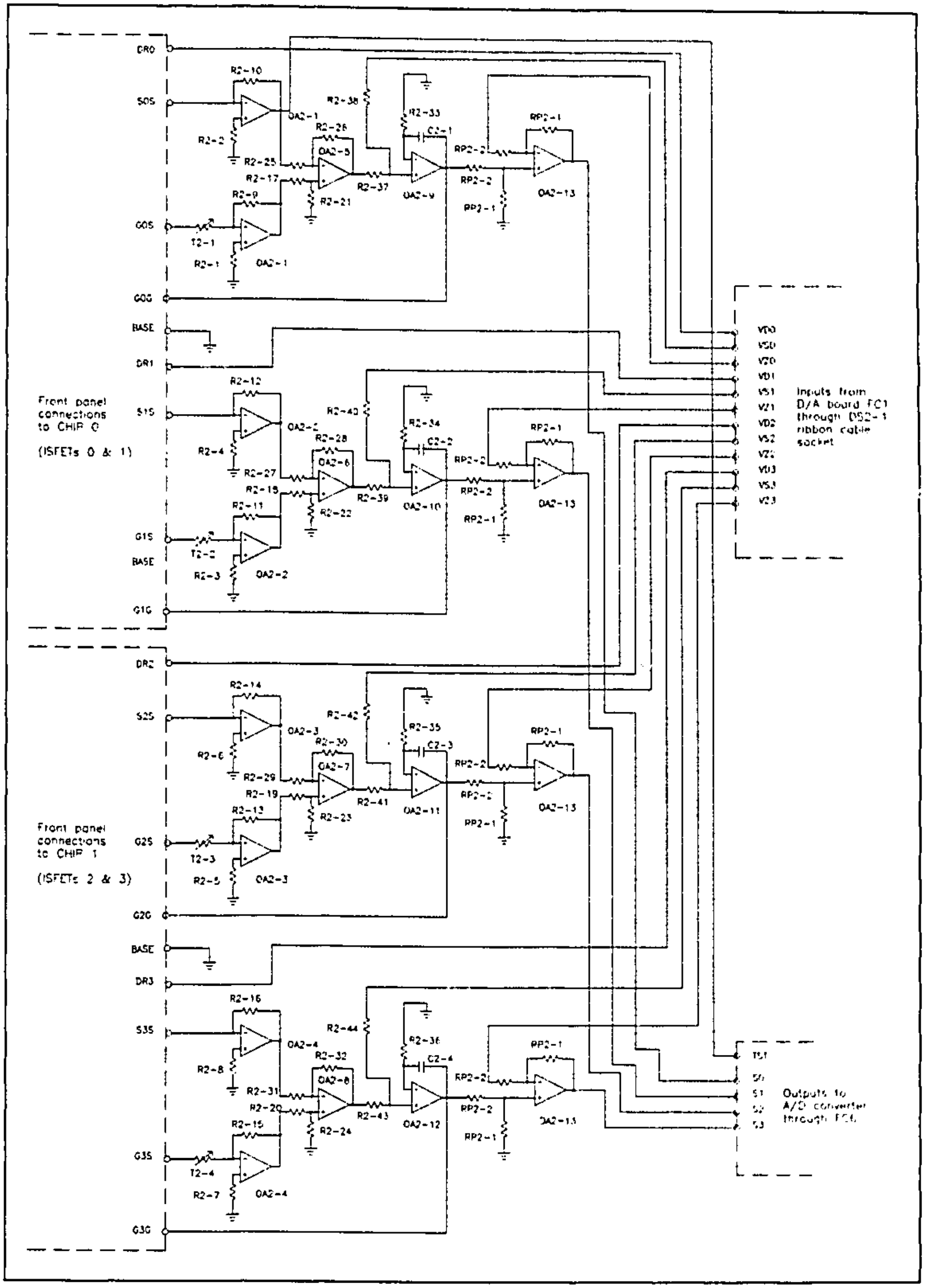

Figure 3-10. Complete schematic for ISFET control board 1 (FC2).
ISFET control boards 2 and 3 (FC3 and FC4) are identical except:
1- TST output is only present on FC2, not on FC3 or FC4
2- For FC3: substitute 4,5,6,7 for 0,1,2,3; CHIP 2 and 3 for CHIP 0 and 1
3- For FC4: substitute 8,9,10,11 for 0,1,2,3; CHIP 4 and 5 for CHIP 0 and 1

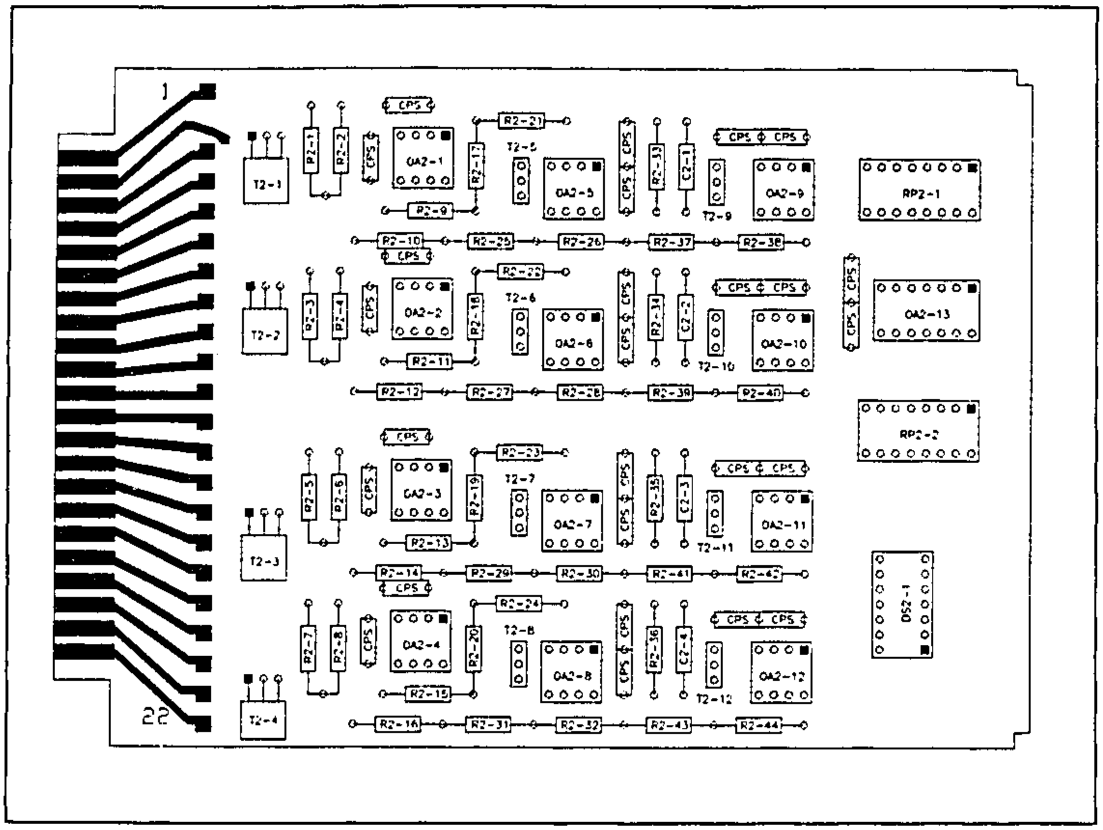

Figure 3-11. Component layout of ISFET control boards (FC2, FC3, FC4).

To allow for the comprehensive testing of individual ISFETs and IGFETs, the output of op-amp 1 on ISFET control board 1 (FC2) was wired to the card edge. Hardwiring in the instrument case connected this terminal (TST) to the A/D converter as the thirteenth input. Aside from this, all three ISFET control boards were interchangeable.

3.5.5 Reference Voltage Board

The reference board was the simplest of all, and it turned out to be unnecessary. The D/A converters on the data-acquisition board were used to supply the reference-electrode potentials. Both of the available D/A channels were used to allow for the possible use of two reference electrodes simultaneously, as well as to provide two different ranges (REF0: 0 - +5V, 1.22 mV resolution; REF1: ±10V, 4.88 mV resolution). An op-amp buffer was used to isolate the D/A output from the solution.

The circuit was exactly as shown at the top of Figure 3-4. The board layout can be seen in Figure 3-12.

In the end, it appeared that the D/A outputs could have been directly applied to the reference electrode. The main function of this board is to allow for future modifications if so desired.

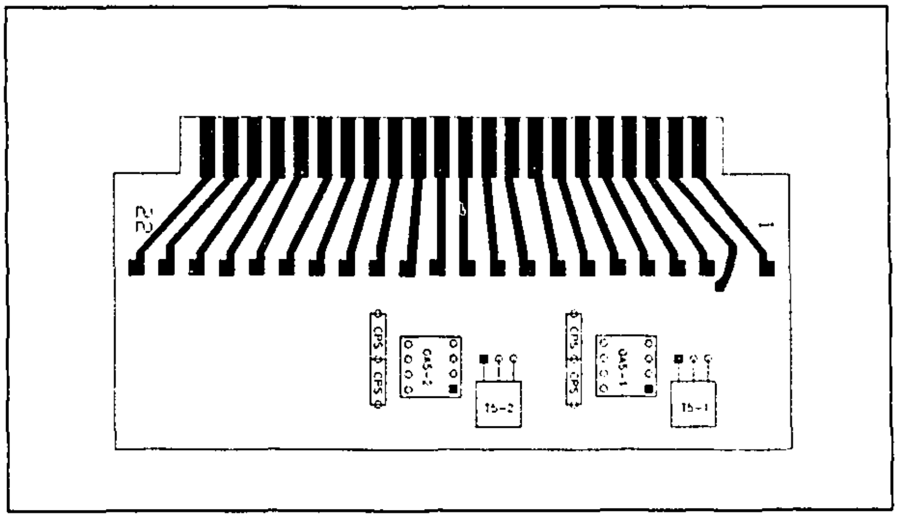

Figure 3-12. Component layout for reference board (FC5).

3.5.6 Data-Acquisition Terminal Board

This board (FC6) is actually just a screw terminal board that was connected via a ribbon cable to the data-acquisition board in the computer. It was fixed and hardwired inside the FET Controller. The connections for the digital signals, D/A and A/D channels were listed in Tables 3.2, 3.3, and 3.4, respectively. These hookups are quite straightforward: 16 digital lines to the FET Controller's D/A board, 2 D/A outputs to the reference board, and 13 inputs from the ISFET control boards. The only thing of note is that single-ended, or pseudo-differential, inputs were used. In this mode all the A/D inputs are compared to the AMPLO terminal. This was connected to the FET Controller supply ground.

3.6 Computer Configuration

The computer was an IBM AT running at 6 MHz with an IBM CGA color display. Storage media included a 20-MB hard disk, 1.2-MB and 360-kB floppy drives. Hard copy was printed on either an Epson RX-80 parallel printer or a Hewlett Packard HP7470A serial plotter on the COM1 port. The COM2 serial port was occupied by a Genius mouse.

The computer software required the presence of a 80287 math coprocessor chip. One MB total memory (640 kB + 384 kB expanded; expandable to 2 MB total) was provided by the 512 kB on the mother board plus 512 kB on an Intel AboveBoard 286. The expanded memory was used for data arrays by the programs in the next chapter.

3.6.1 Data-Acquisition Board

The Data Translation (Marlborough, MA) DT2801 (Rev A) acquisition board was used to provide the 13 single-ended 12-bit A/D channels (16 channels available), the 16 bits of digital input/output, and the 2 12-bit D/A channels. The accompanying screw terminal board, DT707, was discussed in Section 3.5.6.

The jumpers on the DT2801 board were set as listed in Table 3.7 to

Table 3.7
Data Acquisition Board DT2801 Jumper Settings

In	Out	Function
W1 W19, W23	W2, W17, W18, W20 W21, W22, W24	Set base address to HEX 2EC
W26 W27	W25, W28 W22, W24	Select DMA channel 1
W6, W8	W3, W7	Single-ended A/D operation
W4	W5	Bipolar A/D operation
W10, W12	W9, W11	D/A converter DAC0 - range: 0 to +5 V
W13, W15	W14, W16	D/A converter DAC1 - range: -10 to +10 V

enable the proper operation of the board. For further details about the data-acquisition board, the reader should consult the manufacturer's manual.

3.7 Summary

The design and construction of the FET Controller was described in this chapter. It is worthy to note that the instrument was designed before the end-use applications were determined. As such, there were certain capabilities and functions that were built into the instrument which were never fully utilized.

The FET Controller has no use as a stand-alone instrument. The software that is required to operate the instrument is described in the next chapter, along with some test runs. The modular construction and adaptability of the FET Controller was exploited in the experiments that are reported later in Chapter 7, where a slight hardware modification enabled the instrument to function in a totally different manner.

3.8 References

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CHAPTER 4

SOFTWARE CONTROL AND OPERATION OF THE FET CONTROLLER

4.1 Introduction

The software programs used to operate the FET Controller were written in the Asyst language. The programs were composed from modules of code. Some of these modules were used, as is, in more than one program, while others were easily modified for use in other programs. This method of programming, combined with the ability to control all of the experimental parameters from software and the modular construction of the instrument, provided the FET Controller system with a great degree of adaptability.

Two programs were written to control and acquire data from the FET Controller. A multiple ISFET data-acquisition program enabled the use of 1-12 ISFETs for a variable length of time. This program had to control the ISFETs, as well as acquire, process, display and temporarily store the data in real time. The data could be permanently stored after the run. The second program was used to test single FETs. This program had to cycle through the different V_D's and V_G's. The acquisition, processing and display of the data for a single FET was much simpler than the multiple ISFET program.

Another two programs were required for the post-run analysis and plotting of the data from the two acquisition programs.

The listings of the four programs can be found in Appendix A. In the balance of this chapter, each program is described and sample outputs are shown. First, however, we will look at the Asyst programming package in general, and its advantages which were exploited by the FET Controller software.

4.2 The Asyst System

Over the last 10 years a wide variety of sophisticated scientific software for microcomputers has been introduced [1,2]. The Asyst system (Keithley Asyst, Rochester, NY) has proven to be one of the best packages for use in the research laboratory due to its combination of data-acquisition, analysis and graphics capabilities. The Asyst language is similar to the Forth language in that it consists of a library of compiled words, from the simple, "+", to the complex, "SMOOTH", which can be used to create new words that are immediately compiled and added to the library. Other similarities to Forth are the use of stacks and reverse polish notation. All words can be executed interactively, or from within another word. The Asyst package combines high-level language characteristics such as conditional looping and multidimensional arrays, low-level execution speeds due to optimized coding of library words, and an interactive programming environment including array and text editors.

The real strength of Asyst lies in the comprehensive library of over 1100 words. It provides support for communication using RS232C and GPIB/IEEE-488 protocols, simplifying the process tremendously. Files can use several data formats including Lotus 1-2-3, ASCII and user-specified. The data-analysis module provides a variety of techniques for waveform processing, curve fitting, matrix mathematics and statistics which are too numerous to list, but it suffices to say that only the most exacting application would require the user to write further detailed routines. The graphics capabilities range from automatic X-Y plots to customized 3-D plots.

There were four features of Asyst which proved to be particularly useful. These are: 1) the interactive programming environment, 2) data acquisition capabilities, 3) multitasking acquisition mode, and 4) token variables. Interactive operation greatly facilitated program development. Small segments of code could be run before, or after, they were incorporated into larger segments. At the same time, the status of the number and symbol stacks could be monitored. Another area which benefited from interactive operation was post-run analysis. After the programs described in this chapter were loaded, the data could be easily manipulated further without having to write, or rewrite, a program. These interactive tests usually consisted of 1-10 lines of code. If a routine proved to be useful it could then be expanded and saved for future use. This is how the multiple ISFET and test FET analysis programs described later in this chapter were developed.

The second useful feature was the data-acquisition capabilities built into Asyst. The system uses a standardized set of words to control virtually all commercial data-acquisition boards. While knowledge of the principles of data acquisition and the characteristics of the board are still essential, the low level programming of the board is performed by Asyst. This makes the programmer's job much easier, and there is no decrease in performance. Indeed, the data-acquisition board's performance is enhanced by the higher level features of Asyst. One feature, used in the multiple ISFET data-acquisition program, is the ability to input A/D data into two buffer arrays cyclically. While buffer A is being filled, buffer B can be processed and cleared. After buffer A is full, the roles are reversed. This acquisition mode ties in with the third Asyst feature that was exploited, multitasking. This is not true multitasking in the sense that several programs could be run simultaneously, but rather a form of concurrent processing. In Asyst, several data-acquisition and control functions can be performed in the background while a regular program is running in the foreground. The background acquisition is controlled by the interrupts from the real time clock. In the multiple ISFET acquisition program, the acquisition of the A/D data and the filling and switching of cyclic buffers (A and B) was achieved in the background. The foreground program would check the status of the background operations and process the data when necessary. The use of cyclic buffers prevented the foreground and background operations from changing the same data simultaneously.

The last Asyst feature which proved invaluable was the ability to use token variables. A token variable is actually a pointer. It can point to an address(es) in memory which may contain a scalar, or an array, of any type. It may point to a named variable, in which case the variable and token names are interchangeable, or to a copy of a named or unnamed variable, leaving the token and variable independent of each other. There are two main benefits of token variables. One is that although the token has to be declared before its use in a word, the data type and size do not. Thus, a token can act as a dynamic array in that its size, shape and type can change before or after its use in a word definition, or during the execution of the word. Secondly, Asyst permits the token to point to expanded memory as well as main memory (<640 kB). These two benefits combined to allow the collection and processing of large amounts of data. The only disadvantage to using tokens is an increase in processing time, but this was not a problem because the FET Controller did not require fast acquisition rates.

4.2.1 Asyst Configuration

The programs in this thesis were written using Asyst 2.01 and the computer configuration described in Section 3.6. The two acquisition programs required the presence of Asyst modules 1 (base system, graphics and statistics) and 3 (data acquisition). The analysis programs needed modules 1 and 2 (analysis), and could be run without the data-acquisition board in place (*i.e.*- on another computer). The Asyst package is completed with module 4 (GPIB/IEEE-488).

The mouse was set up to mimic the cursor control keys by executing the driver MENU SMOUSE before entering Asyst. The driver was obtained from Smart Mouse Software (Houston, TX) and was originally developed for use with a different software package. The mouse was mainly used in the array and text editors.

Besides the hardware setup, the Asyst software also had to be configured to specify how the available memory was to be used and which system overlays were permanently loaded. Two versions of Asyst were used. The data-acquisition programs were run under the version JMASYST2, and the analysis programs under ANALYZE. The main differences between these versions are that ANALYZE has a larger space reserved for unnamed arrays, to allow for calculations, and that

JMASYST2 has the overlays and hardware configuration necessary for data acquisition. The software configuration specifics can be found in Appendix A (Table A.2).

4.3 Multiple ISFET Operation

4.3.1 Data Acquisition

This program shared a similar structure with all the other programs. The first part loaded was always a Declare module. This serves to initialize the data-acquisition interface, all the variables (including scalars, arrays and tokens) and the various text and graphics windows. The Declare file consists of words which are directly executed upon loading. All of the other modules, which are loaded after the Declare module, define new words that are executed later. These modules consist of groups of related word definitions. For example, Words5 contains all the words used for the permanent storage and retrieval of data.

Only two words, SETUP.FETS and RUN.FETS (File: Program1, line #'s 18 and 67, respectively), are needed to execute the program. Another word, TURN.ON (line # 5), was used to ensure that the ISFET control voltages were all zero before the ISFET leads were connected to the FET Controller. After the run was finished, the data and comments could be permanently saved with SAVE.MULTIPLE.DATA (File: Words5, line # 78). These four words were entered and executed interactively.

The complete listing of the multiple ISFET acquisition program is in Appendix A. A flowchart of the multiple ISFET data acquisition program is shown in Figure 4-1. The word SETUP.FETS must be executed before acquiring data. This word prompts the user to enter the necessary parameters using the two screens shown in Figure 4-2. The first screen was just a list of questions. The default parameters could be accepted by hitting return. The second screen was a customized array editor for the entry of all the control voltages values for the FET

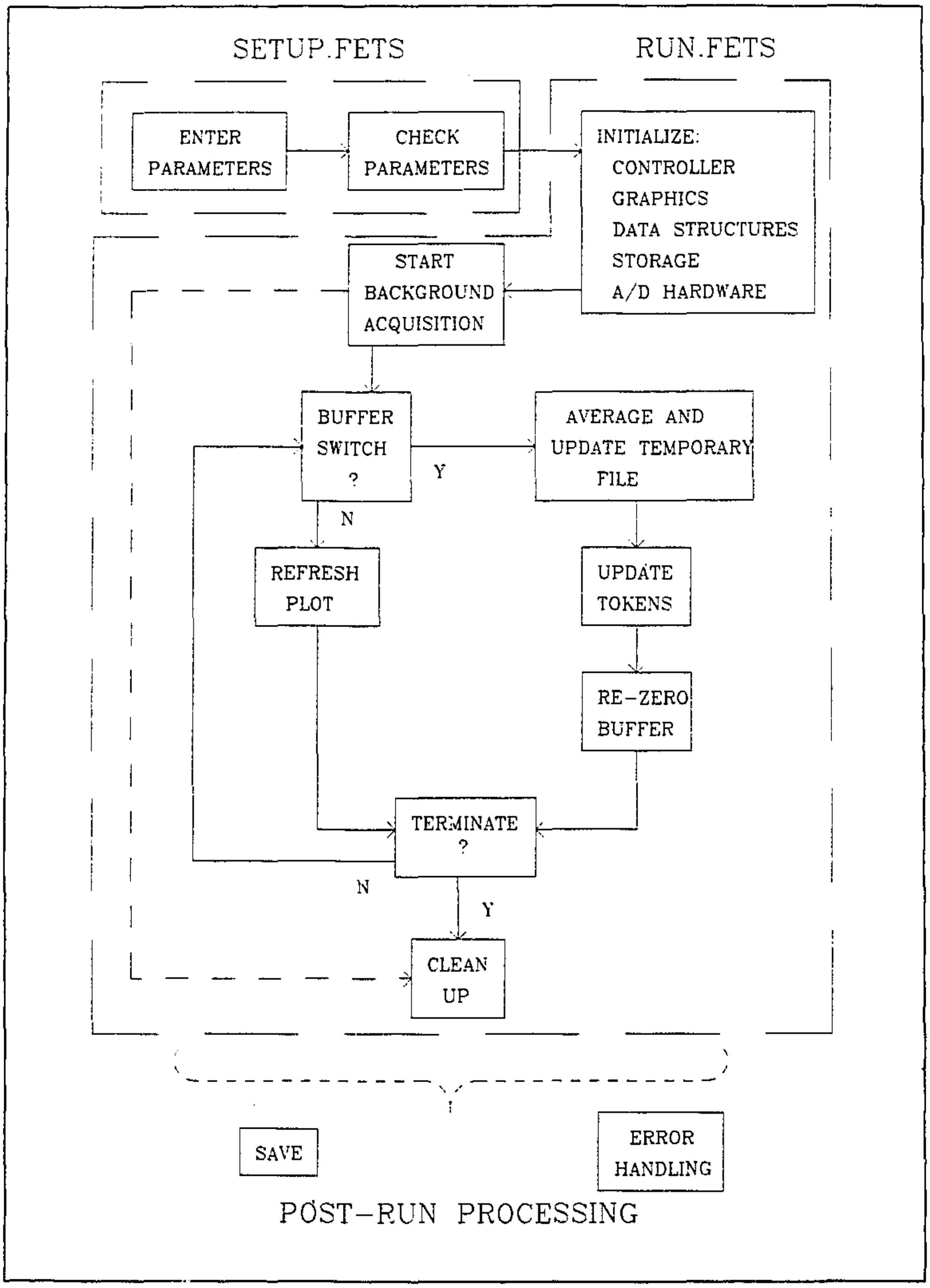

Figure 4-1. Flowchart for multiple ISFET data-acquisition program.

(a)

```
VZ ranges: -5 to +5
          -10 to +10
(Default value = 10 V)
Enter new range (5 or 10) : 10

VD/VS ranges: -5 to +5
             -10 to +10
(Default value = 5 V)
Enter new range (5 or 10) : 5

Enter REF0 value ( 0 ) : 1
REF0 = 1.0000 V

Enter REF1 value ( 0 ) : 2
REF1 = 2.0000 V


Enter number of FETs to be monitored (12) : 12

Enter number of samples to be averaged per point (8) : 8

Enter acquisition period in seconds (1) : .5

Enter gain (1) : 1
```

(b)

```
                 VZ            VD            VS

  0             .000          1.000         .500
  1             .000          2.000         .500
  2             .000          2.000         .500
  3             .000          2.000         .500
  4             .000          2.000         .500
  5             .000          2.000         .500
  6             .000          1.000         .500
  7             .000          1.000         .500
  8             .000          1.000         .500
  9             .000          1.000         .500
 10             .000          1.000         .500
 11             .000          2.000         .500

    Enter Value <CR>:  2.00

 Input or change voltage values.
 Present ranges are:  VD/VS = ± 5     VZ.RANGE = ± 10
 Note: VS0 cannot be set < 0 ; VS3 cannot be set < -4.99
       VD1, VD2, and VS9 cannot be set > 4.99
 Hit ESC when finished
```

Figure 4-2. Input screens for multiple ISFET data-acquisition program.
(a) First screen - entry of hardware and acquisition parameters
(b) Second screen - customized array editor for input of FET Controller D/A voltages

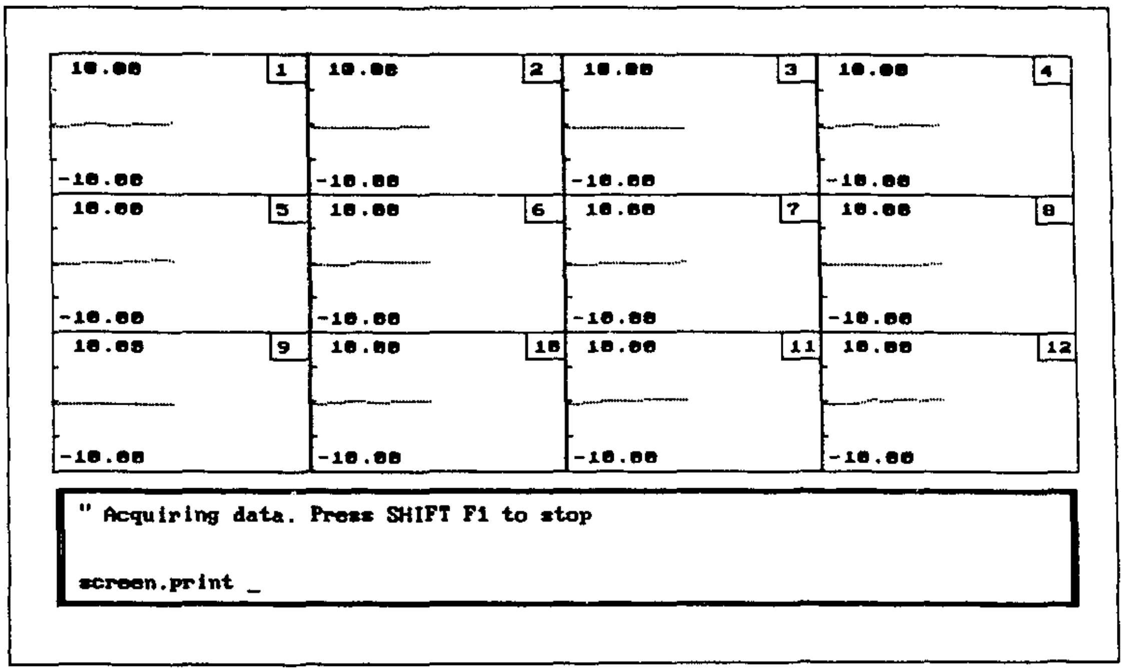

Figure 4-3. Multiple ISFET data acquisition screen image.

Controller D/A board. After the initial use, this word was only used if the parameters had to be changed.

The data acquisition was achieved by the word RUN.FETS. The hardware and software were first initialized according to the parameters that had been entered. It was at this point that the token variables' dimensions and size were set. The display was configured to provide separate windows for each ISFET (Figure 4-3) and the data acquisition was started in the background. Meanwhile, the foreground program started to cycle through the "refresh plot" branch. When one of the A/D buffers was filled, the second branch would execute. The averaged data would be appended to temporary file on the hard disk and also written to the appropriate token in expanded memory. The temporary file was a safety device to enable data recovery in case of a crash during an experiment. The program terminated on the operator's request or when the tokens were full. The 16-kB expanded memory tokens permitted data to be collected for 27 minutes at a 5-Hz acquisition rate or up to 136 hours for a rate of 1 point a minute. The run time could be increased by a maximum factor of 4 by adding another 576-kB expanded memory. Five Hz was the maximum acquisition rate for the default settings of 12

ISFETs, 8 samples per point and a 0.1-ms conversion delay.

The CLEAN.UP routine (File: Words3, line # 236) was invoked by the proper termination of the program, and also by minor crashes which did not hang up the entire system. In the second case the system status and error flags could be examined in the post-run processing stage.

The data could be saved permanently at this stage by the word SAVE.MULTIPLE.DATA. The data file was formatted to save the data for all 12 ISFETs, the parameters, the date and time, and comments.

4.3.2 Data Analysis

The multiple ISFET analysis program, listed in Appendix A, enabled multiple ISFET data to be read in and plotted out. The word PLOT (File: Words8, line # 52) was used for screen output, while the word HARD.PLOT (line # 115) produced a hard copy output on the plotter. These words shared similar characteristics including automatic scaling and labeling. Both words require the token data for a single ISFET to be present on the number stack. A set of smoothed data, offset from the original data, is plotted out as well. The Asyst SMOOTH function is a time-domain filter involving a convolution of the data with filter weights derived from a low-pass (0.05-Hz cutoff) Blackman window frequency response [3].

Post-run analysis was also performed interactively. The most frequent operation was the extraction of datum points at specified times. The data were read off the screen using the GRAPHICS.READOUT function of Asyst.

4.4 Testing of Single FETs

Every ISFET and IGFET was tested using this program before use in any other test or experiment. Later tests could be compared to the original data. Because this is meant to be a standard test, there are no user inputs. The data-acquisition parameters could be changed interactively or by altering the file Declare2.

4.4.1 Data Acquisition

The data-acquisition requirements were less stringent for this program compared to the multiple ISFET case. There was no need to store the data during acquisition because of the short experimental time. The test could be repeated if necessary. A dynamically sized token in expanded memory was still used to accommodate changes in the acquisition parameters in interactive operation; however the data were read directly into the token rather than passing through a buffer. The rate of total A/D conversions was much lower because only one FET was tested at a time and real time averaging was deemed unnecessary. This allowed the use of Asyst's software synchronization capability which is simpler than the background-tasking technique. Data were collected at a 10-Hz rate for 6 seconds.

Basically this program just monitored I_D, by measuring the output of the current-to-voltage converter stage, while V_G and V_D were varied stepwise. All tests were performed with the chip immersed in a 0.1 M KCl (reagent grade) stirred solution. All FETs were tested by hooking up the appropriate source lead wire to the front panel connector S0S, and the drain lead wire to DR0. The REF0 output was connected to the reference electrode for ISFETs, or to the gate lead wire for IGFETs.

The program was started by executing the word TEST.FET (File: Words6, line # 80). The program had a triple-nested loop structure as seen in Figure 4-4. The data were plotted on-screen as it was acquired and the values of V_G and V_D being tested were displayed. The data and experimental parameters were permanently stored by the word SAVE.TEST.DATA (File: Words7, line # 82). A table of I_D values for each combination of V_G and V_D was printed out by the word PRINT.TEST.RESULTS (line # 148).

4.4.2 Data Analysis

The main purpose of the test FET analysis program was to construct the characteristic I_D versus V_D curves at different V_G's. Test FET data were retrieved using the word RETRIEVE.TEST.DATA

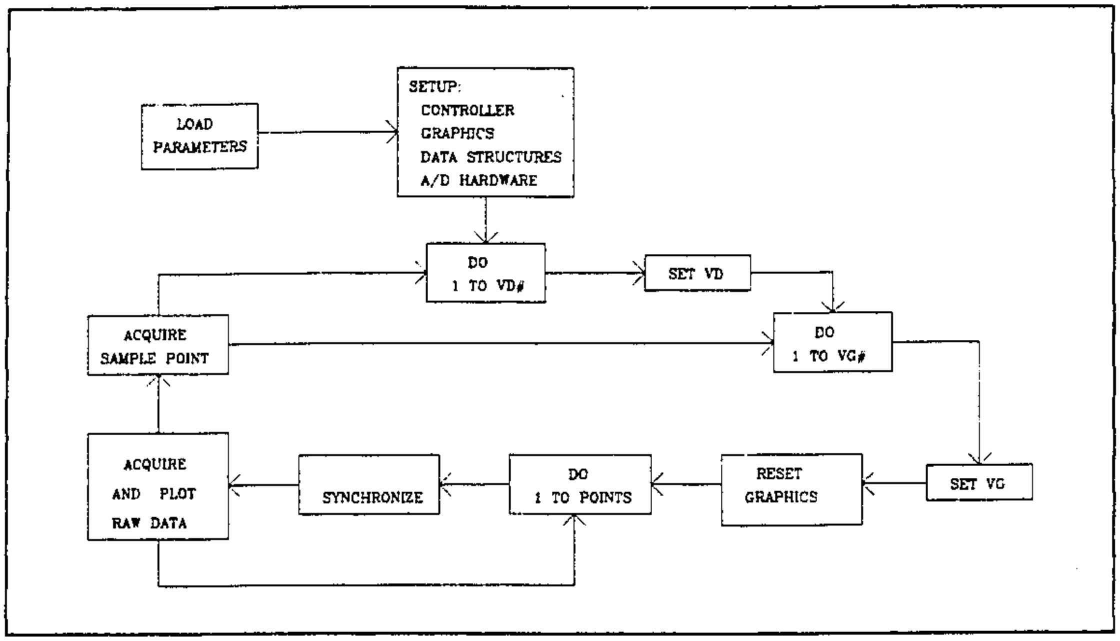

Figure 4-4. Loop structure of test FET acquisition program.

(File: Words7, line # 96). The word GO (File Words10, line # 17) printed out the file comments, the mean data values for each V_D-V_G combination, and the characteristic plot on the system printer. This printout indicated whether the FET was functional. A sample of the test FET analysis output is given in Figure 4-5.

4.5 Discussion

The FET Controller instrument has been fully described by the combination of this chapter, Chapter 3, and Appendix A. It should be noted that the software discussed in this chapter was presented in its final incarnation. These programs were in a constant state of flux. A menu system could be easily set up to control and execute the FET Controller programs; this would be expected in a commercial instrument. For our research, however, interactive programming provided a higher degree of adaptability and convenience.

Initially, the FET Controller was used as originally expected (see Section 3.2), that is to simultaneously test a series of ion-selective PVC-ionophore membranes on ISFETs in order to evaluate and

(a)

```
Enter filename for retrieval: ch38-s2.tst

Comments from C:\JEFF\DATA\CH38-S2.TST

1 )   06/25/90  22:47:38.08
2 )   FIRST TEST (IN KCL)
3 )   BOOK VI PAGE 75 CHIP 38 SENSOR 2   BARE GATE
4 )   LOOKS OK, CURRENTS COMPATIBLE WITH S1 AU/THIOL GATE
5 )
6 )
Retrieving data from C:\JEFF\DATA\CH38-S2.TST
Data retrieval finished.
```

(b)

VG VOLTAGES	VD VOLTAGES .00	.52	.99	2.01	3.00	3.98	5.01
-2.99	-.006	-.006	-.009	-.006	-.009	-.006	-.006
-2.00	-.006	-.009	-.006	-.009	-.006	-.006	-.006
-.99	-.009	-.006	-.006	-.006	-.006	-.006	-.006
.00	-.013	-.004	-.004	-.004	-.004	-.001	-.004
1.01	-.021	.060	.077	.084	.084	.087	.087
2.01	-.016	.226	.346	.404	.409	.416	.421
3.01	-.011	.382	.651	.932	.978	.995	1.005

02/27/91 16:30:33.50
FET ID: chip 38, sensor 2 - bare gate pH sensor

(c)

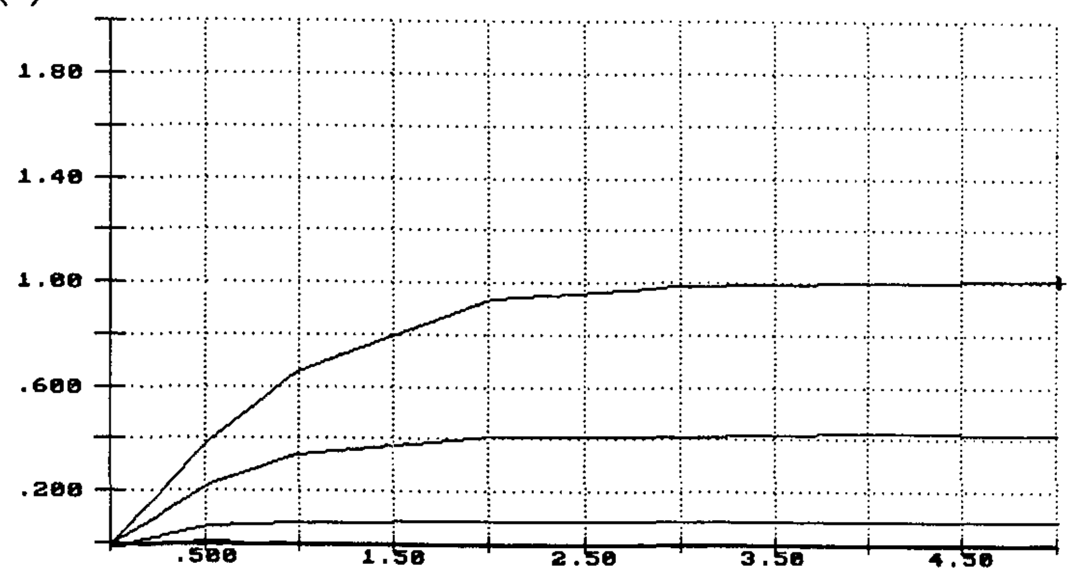

Figure 4-5. Output from test FET analysis program.
(a) data file comments printed out by RETRIEVE.TEST.DATA
(b) mean I_D currents (in mA) for each V_D-V_G combination
(c) characteristic plots at constant V_G's (from top: $V_G=3$, $V_G=2$, $V_G=1$ V)
Y axis: I_D (/mA)
X axis: V_D (/V)

improve the response. These experiments were useful in terms of the expertise gained in the handling and operation of ISFETs, and also served to establish that the FET Controller performed as expected. This line of research was not carried through to fruition, however, because the direction of research was changed to incorporate a new interest, namely the field of telemetry.

The balance of this thesis is concerned with the coupling of the ISFET and telemetry research areas. The FET Controller was used for work presented in Chapter 7. The software programs were used unchanged, while the hardware had to be slightly modified. This, in itself, indicates that the FET Controller did meet one of its primary requirements - adaptability.

4.6 References

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CHAPTER 5

TELEMETRY

5.1 Introduction

In the broadest sense, telemetry can be defined as *a process for measurement at a distance.* In its earliest incarnation in the mid-1800's telemetry was achieved by using wires to hook up a sensing device with an instrument. In today's vernacular this would be just a case of remote sensing. With the advent of telecommunications around the turn of the century new methods of transmitting the information from the sensor to the recording instrument were introduced, including the telephone, telegraph and radio. While the telegraph has fallen out of use, the telephone is still used for telemetry in specific cases. Radio, however, has become the dominant mode of transmission in modern telemetry. Other methods currently used include ultrasonic transmission for aqueous environments, and storage telemetry where the device is recovered and the data are then read.

The development of solid-state electronics was the next catalyst in the evolution of telemetry. In the 1950's the field of biotelemetry, also known as biomedical telemetry, was firmly established. (There were some scattered experiments in the first half of this century, but the crude electronics severely curtailed success [1].) Biotelemetry entails the sensing of physiological information and its transmission from the subject to a remote location. The sensor-transmitter can be used *in vivo* or attached externally to the subject. Usually the procedure is performed under clinical conditions which only require short range transmission. Long range transmission from remote emergency stations or free-ranging animals is also possibie but more extensive equipment is needed.

There are several advantages of using biotelemetry compared to a conventional system where the sensor and instrumentation are physically linked. The subject can enjoy greater comfort and ambulatory freedom, especially for *in vivo* sensing. The safety of the patient is increased due to the total electrical isolation and lower risk of infection. From the analyst's point of view, a possible benefit is an increase in the accuracy of the determination. With conventional systems the instrumental link, which may be an indwelling catheter or gastric tube, may perturb the system under study thereby altering the parameter being measured. For an *in vivo* test this can arise from a stress response or a reaction to the physical aggravation. The nature of a telemetry system, requiring no physical connection, may reduce this effect [2].

The main disadvantage of biotelemetry is the complexity of the equipment. While very simple, yet elegant, systems do exist, a large share of the research effort is devoted to advances in electronics. The receiver-recorder instrumentation is fairly straightforward. The problem lies in that for every new parameter to be monitored, a new sensor-transmitter must be developed.

There are three excellent sources of information concerning all aspects of biotelemetry. The pioneering work from the fifties and sixties by Mackay and coworkers, and other groups, is covered in his book [3]. The review by Topich in 1978 [4] focuses on the design and application of biotelemetry techniques. Design considerations were reviewed by Jeutter in 1982 [5], with 175 references given.

In this chapter, we will next look at the different transmission modes possible in telemetry. In Section 5.3 various biotelemetry applications are briefly discussed. A more detailed look at gastric pH determination is included. Finally, a review of the different types of sensors used in telemetry is given and the use of the ISFET is introduced.

5.2 Transmission Modes

The distinguishing characteristic of biotelemetry is the transmission of the desired information by radio signals. This section examines how the signal is transmitted.

5.2.1 Long Range Versus Short Range

Radio telemetry has been used to transmit information over distances ranging from centimeters to thousands of kilometers. There is no set distance which differentiates between short and long range telemetry. In his review [6], Fryer considers transmission over a distance greater than 50 feet as long range telemetry. He points out that in the long range, the system operates within the radiofrequency (RF) region controlled by government agencies. Short range transmission usually falls outside of such regulations due to the low power and limited range. For this thesis the goal is operation in a clinical laboratory, therefore short range telemetry is employed.

Telemetry devices can also be classified according to what type of field is detected by the receiver [7]. Radio waves are a combination of a magnetic field and an alternating electric field (RF radiation). The decrease in magnetic field strength is proportional to the cube of the distance between transmitter and receiver. This is called the "near field" due to its high power at very short distances which decreases rapidly. The RF radiation, or "far field", strength falloff is proportional to the first power of the distance. The near and far field strengths are equal at a distance of approximately one-sixth of a wavelength. Short range telemetry involves detection of the magnetic field. Long range telemetry uses only RF radiation.

Using the magnetic field in short range telemetry simplifies the requirements for propagation and detection of the radio signal. In long range telemetry an antenna is required for both the transmission and detection of the RF radiation. By using only the magnetic field in short range telemetry, the antenna is not needed. Transmission is achieved by inductive coupling between coils in the transmitter and receiver.

These coils are much smaller than any antenna and can be incorporated directly into the transmitter circuitry.

5.2.2 Frequency

The frequencies used for telemetry have ranged from a hundred kHz to several hundred MHz. There is no optimum frequency. Higher frequencies provide benefits which include smaller antennas, smaller components, stronger far field strength and more sensitive detection. A low frequency is attenuated less when passing through body tissue. For biotelemetry based on inductive coupling, only the near field is used and no antenna is needed so the lower frequencies are a better choice.

The other considerations in choosing a frequency are legal factors and equipment. The legal factor is skirted by using a short range, inductively coupled transmitter at low power. Otherwise the device must conform to the restrictions concerning allocated frequencies and bandwidths for the particular country.

The main consideration as to equipment is the choice of receiver. The transmitter is always custom designed, however the receiver may be obtained commercially and modified if necessary. While it is convenient to operate in the regular AM or FM band, the existence of commercial broadcast stations is a problem. Operating in the short wave bands (2-20 MHz) reduces this problem and still allows use of commercial receivers.

5.2.3 Modulation

The transmitter sends the transducer response to the receiver by modulating the radio signal. Quite a wide range of modulation techniques are possible. The simplest is amplitude modulation (AM), however this is not often used in telemetry because the observed signal varies with the signal strength which can change according to the position and orientation of both the transmitter and receiver. Frequency modulation (FM) is preferred because it is insensitive to signal strength variations. Direct FM can be used. In this case the frequency of the signal changes according to the sensor response. (Note the distinction between the commercial bands, AM and FM, designating wavelength regions, and the modulation techniques, also known as AM and FM. To wit: FM techniques can be used in the AM band.)

Blocking oscillators combine AM and FM advantages. In a blocking oscillator the AM signal is turned on and off at a rate proportional to the sensor response. This allows the use of simple circuitry. The desired information is encoded in the frequency of the pulsing, and variations in magnitude are not important. This technique will be discussed further in Chapter 6.

True FM requires more complex circuits in the transmitter. Usually a customized integrated or hybrid circuit must be designed and manufactured. Besides the decreased noise sensitivity, FM allows for the transmission of more information. This is necessary for multisensor systems.

5.2.4 Multiplexing

In this thesis only single sensor telemetry devices are studied, and simple modulation techniques are used. For a multisensor system the sensor signals must be multiplexed to be transmitted. This can utilize either time or frequency division formatted FM. Formatting methods that have been used include pulse width modulation, pulse frequency modulation and pulse amplitude modulation. References 4 and 5 examine this topic in greater detail.

5.3 Applications of Biotelemetry

5.3.1 Physical

Biotelemetry techniques have been used in the last forty years to monitor a variety of physical and chemical parameters. Among the first applications was the transmission of information concerning bioelectric potentials, including electrocardiograph and electroencephalograph signals. Telemetry is still used for this purpose, especially for clinical studies involving animals or exercise.

Other physical parameters of interest include temperature and pressure. Mackay and Jacobson [8] reported a device in 1957 for use in the gastrointestinal (GI) tract which measured pressure and temperature simultaneously. This device was in the shape of a small capsule which could be ingested. They called the device an *endoradiosonde*, however this term has since been displaced by a simpler term, *radio pill*, to describe any ingestible radio telemetric device. A radio pill is indigestible and can be recovered after passing through the entire GI tract. Generally the device is approximately the size and shape of a large gelatin capsule. Research has extended the use of *in vivo* biotelemetry based on physical parameters to more complex applications such as the cardiovascular system and intracranial measurements. These applications require smaller devices which are usually surgically implanted.

The last physical parameter that we will consider is position. On a large scale this includes animal tracking by long range telemetry, which is quite a large field of study in itself. For short range telemetry utilizing the near field, two distinct methods exist. In Section 5.2.3 we noted that amplitude modulation is not used due to its susceptibility to movement and orientation. This can be exploited. By using AM, the movement of the capsule can be detected. This has been used to monitor stomach motility as well as the activity of laboratory animals (*i.e.* sleep patterns). In the second method, the telemetry device transmits a signal when it identifies certain conditions, and then the location of the transmitter is determined. Hassan *et al.*[9] described a radio pill which used a radioactivity detector that could determine the site of bleeding in the GI tract. A radioisotope tracer would be injected into the bloodstream. The radio pill would then send a signal when blood was detected and the exact position of the pill could be found by taking an X-ray of the patient. This combines detection of both physical and chemical parameters.

5.3.2 Chemical

There are a limited number of biotelemetry applications that detect purely chemical parameters. The majority of devices employing chemical sensing focus on the determination of pH in the GI tract. This is discussed separately in the next section. The pH of dental plaque has also been determined by telemetry [10,11]. The only other ion that has been monitored successfully by telemetry is F^- in oral fluids [11,12].

Telemetry devices using amperometric sensing have been used to analyze blood for dissolved gases. A transcutaneous O_2 sensor-transmitter was described by Hartley *et al.*[13]. This concept was extended by Sansen and Lambrechts [14] who used glucose oxidase and a Clark electrode to monitor glucose.

5.3.2.1 Gastric pH

The GI tract provides the means for ingestion, digestion and absorption of food and the elimination of wastes. It is comprised of the mouth, esophagus, stomach, and the small and large intestines. The pH in the tract ranges from 1 in the stomach to around 8 in the intestines. The sensing of pH throughout the GI tract has benefitted from the introduction of telemetry.

Oral pH studies fall into the realm of dental research. Usually the pH device is fixed onto the teeth and retrieved from the mouth. To monitor the pH of the esophagus the sensor device must be tethered (usually to a tooth) and may be removed through the mouth or allowed to pass through the GI tract. Several different methods have been used to determine gastric pH. Before the development of the pH radio pill, gastric pH was measured either by sampling through a gastric tube or by use of a flexible pH probe. Both of these methods can be expected to alter the functioning of the GI system due to physical and emotional distress. The intestines were basically inaccessible, short of surgery, previous to the radio pill [15].

In all cases the use of telemetry increases the comfort and safety of the subject. By using telemetry the GI tract is perturbed to a lesser extent compared to other methods involving tubes. Therefore the pH in the GI tract is closer to the pH that exists when the system is undisturbed. The challenge in designing a telemetry device is to ensure that the observed pH value is an accurate representation of the pH being measured.

The pH radio pill can provide other information about the GI tract. Stomach motility can be determined by observing the variation in signal strength as mentioned above. Another parameter that can be measured is gastric residence time. A sharp increase in pH indicates that the pill has moved from the stomach into the small intestine.

The determination of GI pH was the first application of telemetry based on chemical sensing. Several commercial devices were developed which encouraged the use of telemetry in clinical laboratories. The first successful device was based on the work of Noller [16]. This device, known as the Heidelberg capsule, is still available in a modified form [17] (Heidelberg International Inc., Atlanta, Ga.). The other commercial device of note was developed by Colson *et al.*[18] and is distributed by Rigel Research (Sutton, Surrey). The pH sensors used in these devices are discussed the following section. Applications have included the determination of antacid effectiveness [19], evaluation of drug delivery systems [20] and diagnosis of GI disorders [21].

5.4 Chemical Sensors in Biotelemetry

In virtually every successful chemically selective telemetry device the chemical recognition element has been a miniaturized version of a conventional sensor. The first pH radio pill reported by Jacobson and Mackay [22] did use a nonconventional sensor, however the results were disappointing. They used a copolymer which exhibited pH dependant reversible mechanical expansion with a pressure sensor that gave slow response and poor selectivity.

The Heidelberg capsule is based on classical electrochemistry. It uses an antimony electrode exposed to the test solution combined with an Ag|AgCl|saturated KCl electrode which is separated from the test solution by a membrane. This forms a galvanic cell with an output voltage that is pH dependant. This battery drives an oscillator which has an oscillating frequency proportional to the pH dependant voltage.

This type of cell is not highly selective and long term stability can be a problem, especially in complex matrices. The manufacturer guarantees the accuracy to be ±0.5 pH units over 6 to 8 hours in the pH range 1-8. A recent independent test [23] indicated that the capsule just barely met these standards in simple aqueous buffers.

The problems with the Heidelberg capsule led to the development of the pH radio pill of Colson *et al.*[18], which uses an even more conventional sensor. Their device uses miniaturized pH glass and Ag|AgCl|saturated KCl electrodes at opposite ends of a radio pill. They report an accuracy of ±0.2 pH units, a range from 1-10, response time of 1 second (to 95% of final reading) and an *in vitro* lifetime of 4-5 weeks.

The F^- telemetry device for oral fluids [11,12] also uses conventional type sensors. In this case an LaF_3 ISE (with an internal reference) and external reference were used.

The problem with these potentiometric sensors for H^+ and F^- arises from the presence of the internal solutions necessary for both the indicator and reference electrodes. The small volume of the solution restricts the lifetime of the device. Further miniaturization, which would be necessary for an implantable device, is not possible due to this same problem.

The amperometric gas sensors rely on the membrane/enzyme combination for selectivity. This type of device has not found common use as a chemically selective *in vivo* sensor, but does show promise. The only amperometric telemetry devices that are currently in use are the simple transcutaneous blood gas probes.

All of the chemically selective telemetry devices considered here and reported in the literature use conventional sensor technology. In part, this is due to the fact that the origin and development of biotelemetry predates the growth of sensor research in the last few decades. The incorporation of novel sensors into telemetry has been slowed by the success of devices based on older technologies. Another obstacle is the difficulty in combining the different areas of research including chemical sensors, electronics and clinical chemistry.

The integration of solid-state sensors, developed over the last twenty years, and telemetry research is overdue. One device has been reported which combines a piezoelectric sensor and a telemetric integrated circuit for the measurement of intracranial pressure [24]. This thesis is more concerned with chemical sensors rather than physical. The characteristics of solid-state chemical sensors are highly compatible with the requirements for a telemetry sensor.

5.4.1 ISFETs as Telemetry Sensors

The combination of ISFETs and telemetry is the focus of this thesis. No reference could be found in the literature concerning the use of ISFETs, or any other novel chemical microsensor, in a telemetry device. In his 1978 review Topich [25] specifically mentions the future possibility of the use of ISFETs in telemetry. A recent paper reports the use of a pH-ISFET in a catheter to study esophageal function [26]. It is not known if this work is being extended to include telemetry. Finally, a variety of workers at Case Western Reserve have published research on either ISFETs or telemetry, but no indication can be found of any work combining the two fields.

The use of ISFETs in a telemetry device has certain advantages aside from the novelty of using a new type of sensor. The low power consumption and essentially zero leakage currents should extend the battery life, and therefore the device lifetime. The smaller size of the ISFET compared to a miniaturized ISE would reduce the size of the sensor-transmitter, thereby making implantation more feasible. Because the solid-state construction allows multiple ISFETs on a single chip it should be possible to design a multisensor telemetry device without a great increase in the overall size.

The final advantage to using the ISFET in a telemetry device is that the range of possible biotelemetry applications is greatly expanded. The variety of FET-based sensors reviewed in Chapter 2 could all be used in future telemetry devices, with little modification in the sensor-transmitter circuitry needed.

5.5 References

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CHAPTER 6

A TELEMETRY DEVICE INCORPORATING A pH ISFET CHEMICAL TRANSDUCER

6.1 Introduction

This chapter details the initial efforts to incorporate a pH ISFET into a simple telemetric device. While the ultimate goal is to use such a device to monitor the pH in the GI tract, the initial work was limited to *in vitro* studies. This work also served as an introduction to the laboratory techniques and instrumentation associated with telemetry.

6.2 Design Considerations

The immediate concern was to design and produce a device in prototype form that could be tested during operation *in vitro*. This implied that the device should be totally self-contained and powered by its own battery. Secondly, the available resources and equipment had to be considered. The prototype had to be made by hand using discrete components.

Even though the prototype was only to be used *in vitro*, we decided to try to keep the overall size and dimensions similar to what would be required for an ingestible radio pill. This meant that the circuitry had to be kept simple and use as few components as possible. To comply with these requirements we decided that the telemetry system should use inductive coupling for transmission. It was shown in Section 5.2.1 how this eliminates the need for an antenna. This simplifies the circuit considerably.

A reference electrode had to be included in the circuit to properly bias the ISFET gate potential. This was, and still is, a problem. To facilitate the work presented here, we decided to use a conventional reference electrode hardwired to the telemetry circuit. This could eventually be incorporated, in a miniaturized form, into a radio pill. While this option is viable, it still presents the same problems related to the internal solution as was discussed in Section 5.4.

6.3 Selection of Circuit

There are two possible ways of using an ISFET in a telemetric device. The first follows the divide and conquer strategy. The ISFET has its own control circuitry that produces an output which modulates a separate oscillator circuit. This is the method of choice for a multisensor system, but for a single sensor device the circuitry is more complicated than necessary. The other alternative is to integrate the ISFET directly into an oscillator circuit. This reduces the complexity of the circuit and minimizes the number of components needed. The second method was chosen for this work.

Several different circuits and modulation techniques were evaluated and tested on a breadboard. The investigation focussed on amplitude modulation and blocking oscillators with broadcast frequencies in the AM and short wave bands (0.5-18.0 MHz). FM techniques were considered but were found to be unnecessarily complex. Pure AM techniques were rejected because of the problems outlined in Section 5.2.3. The blocking oscillator, also known as the squegging oscillator, was chosen due to its simplicity and stable signal.

The ISFET was tested in two modes of operation. Initially the ISFET was used as an active transistor to drive the oscillator circuit. These efforts were not successful. The second configuration used a standard bipolar transistor to drive the oscillation while the ISFET was used as a variable resistor. The oscillator output was modulated by the pH dependant variable resistance of the ISFET.

The configuration of the final circuit was therefore a blocking oscillator incorporating an ISFET as a variable resistor. The next section details how a FET can be used as a variable resistor. This is followed by description of how a blocking oscillator actually works. In Section 6.4 the final circuit design and construction is detailed.

6.3.1 The FET as a Variable Resistor

A theoretical set of characteristic response curves for a FET are shown in Figure 6-1. The expanded view of the intersection of the axes illustrates how a FET can act as a variable resistor. In the unsaturated region, the FET responds linearly to changes in V_D as the drain voltage approaches zero. The slopes of these curves at $V_D=0$ are equal to the inverse of the channel resistance between the source and drain (R_{DS}). The relationship of R_{DS} to V_G can be derived from the equation for the current response in the unsaturated region (Equation 2.2). When V_D is

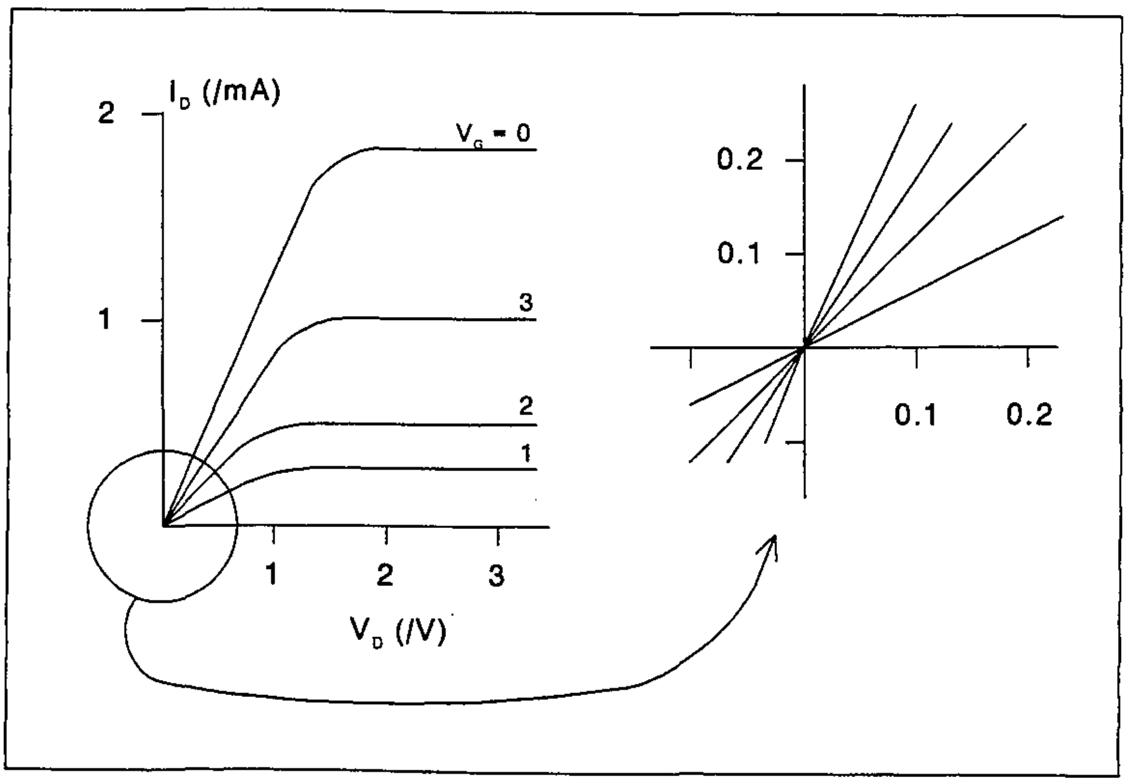


Figure 6-1. Theoretical response curves for a FET
Voltages relative to V_B. ($V_B = V_S = 0V$)

zero this gives:

$$R_{DS} = \frac{L}{\mu_n C_{ox} W \left(V_G - V_T \right)} \tag{6.1}$$

where all conditions and variables are as defined in Section 2.4.1.2. The only experimental variable in this equation is V_G.

While this equation is valid at $V_D=0$, there must be a finite voltage applied ($V_D>0$) during normal operation. As V_D increases the degree of nonlinearity also increases. Typical values are 2% deviation for $V_D < 0.1(V_G - V_T)$, and 10% when V_D approaches $0.25(V_G - V_T)$. This effect is minimized by either reducing V_D or increasing V_G.

Under these conditions a FET, or ISFET, can act as a variable resistor. The resistance of the channel is inversely proportional to the gate voltage. In this configuration a FET can be incorporated into a circuit in place of a resistor.

6.3.2 Blocking Oscillators

Much of the early work in telemetry used a blocking oscillator as the transmitter. The theory and practical use of this type of circuit was covered by Mackay [1]. All blocking oscillators have several common characteristics, however the actual circuits may differ. They are distinguished by their unique output waveform. A typical waveform is shown in Figure 6-2 in three different time scales. Figure 6-2(a) was acquired at the slowest sweep rate; (b) and (c) show an expanded single pulse at faster sweep rates. (The acquisition of these oscilloscope images is described in Section 6.4.3.)

Analysis of the waveform in Figure 6-2 provides three pieces of information. The pulse frequency (PF) can be measured from (a), the pulse length (PL) from (b) and the broadcast frequency (BF) from (c). The BF varies during the pulse so this is not useful in terms of transmitting information. Because the BF changes, this type of transmitter broadcasts over a wide frequency band. Information is usually encoded in either the length or frequency of the pulses.

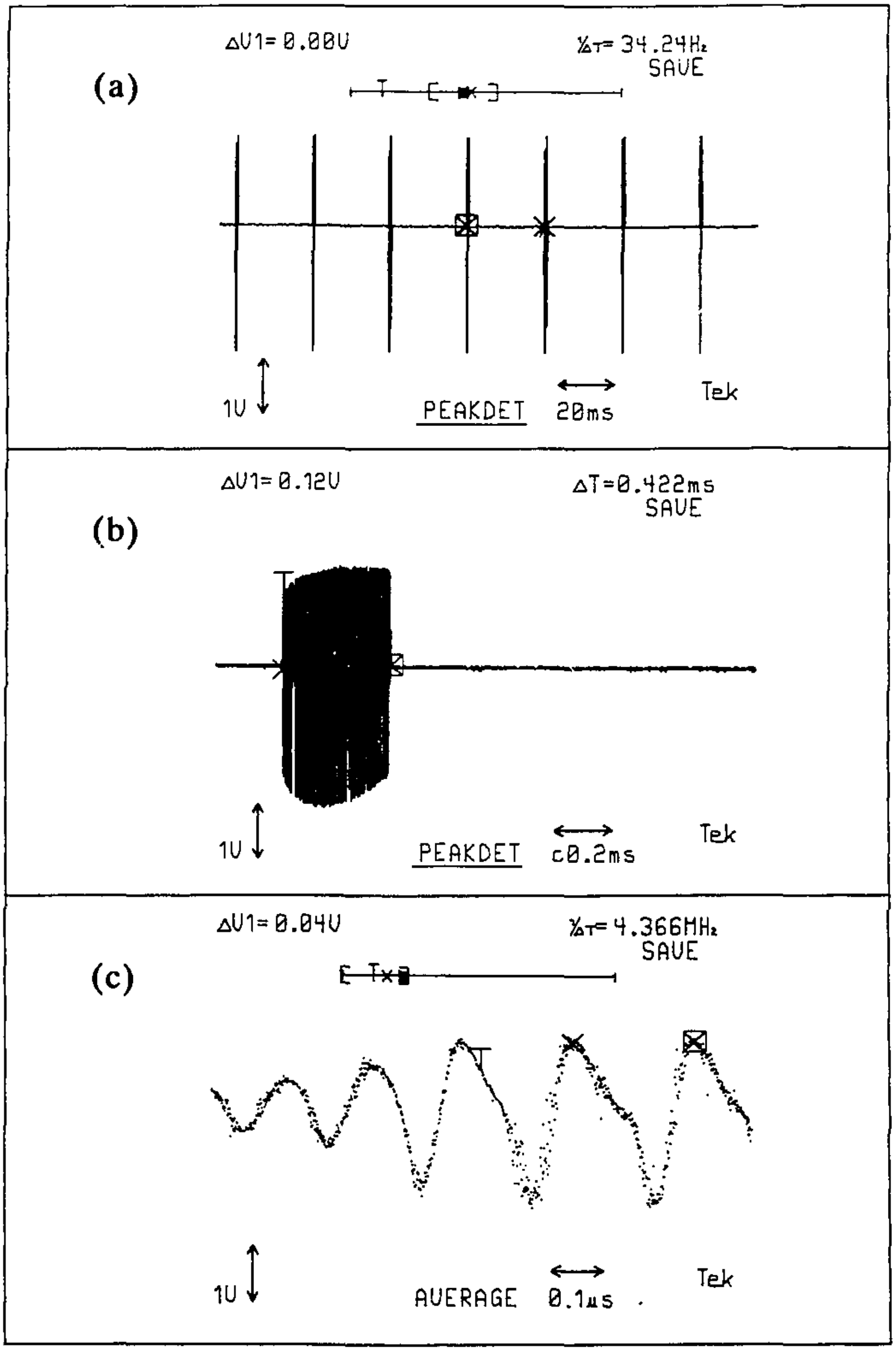
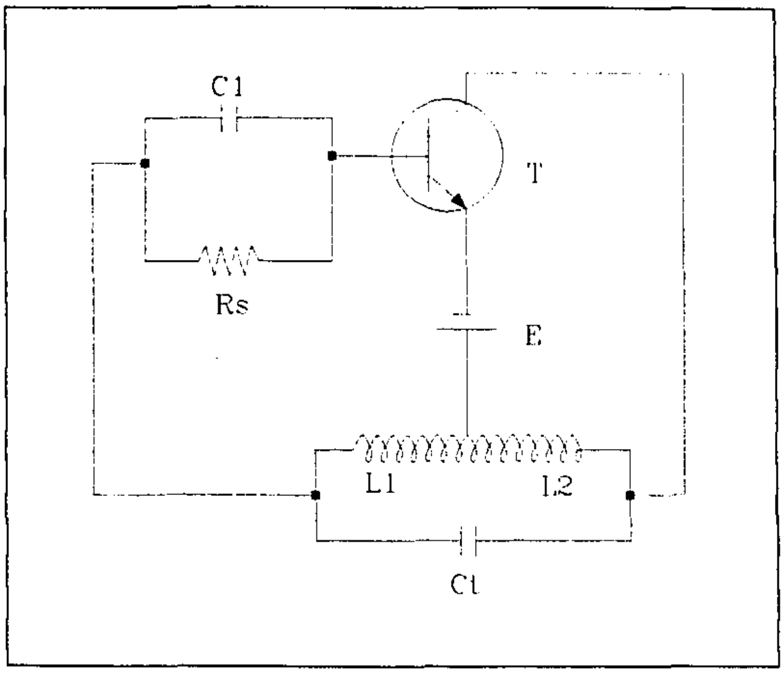


Figure 6-2. Sample output of a blocking (squegging) oscillator.

Oscillation across collecter and emitter of transistor. Images plotted from oscillscope (ground to emitter). Circuit is described in Section 6.4.
(a) series of pulses (pulse frequency = 34.24 Hz)
(b) expanded view of single pulse (pulse length = 0.422 ms)
(c) further expansion of single pulse (broadcast frequency = 4.366 MHz)



$C1 >> Ct$

$Rs >> |X_C|$
(X_C: reactance of C1)

Figure 6-3. Basic circuit of a blocking oscillator.

The blocking oscillator is based on the Hartley oscillator, which is distinguished by the presence of a tapped coil in parallel with a capacitor. Figure 6-3 shows a basic blocking oscillator circuit which was analyzed in detail by Lin and Ko [2]. The BF is determined by the component values of the capacitor Ct and the inductors L1 and L2. The other two parameters, PL and PF, are dependant on the transistor T, battery voltage E and the RC circuit consisting of C1 and Rs. Oscillation starts when the charge at the base of the transistor is sufficiently large enough to turn on the transistor. During oscillation this charge is dissipated and the transistor is eventually turned off. The base charge is then replenished at a rate determined by the time constant of the RC circuit until the cycle begins again.

The blocking oscillator can exist in three states. The quiescent state, where no oscillation occurs, and the sustained oscillation state are of no practical use. The components must be chosen to allow operation in the squegging, or pulsating, oscillation state.

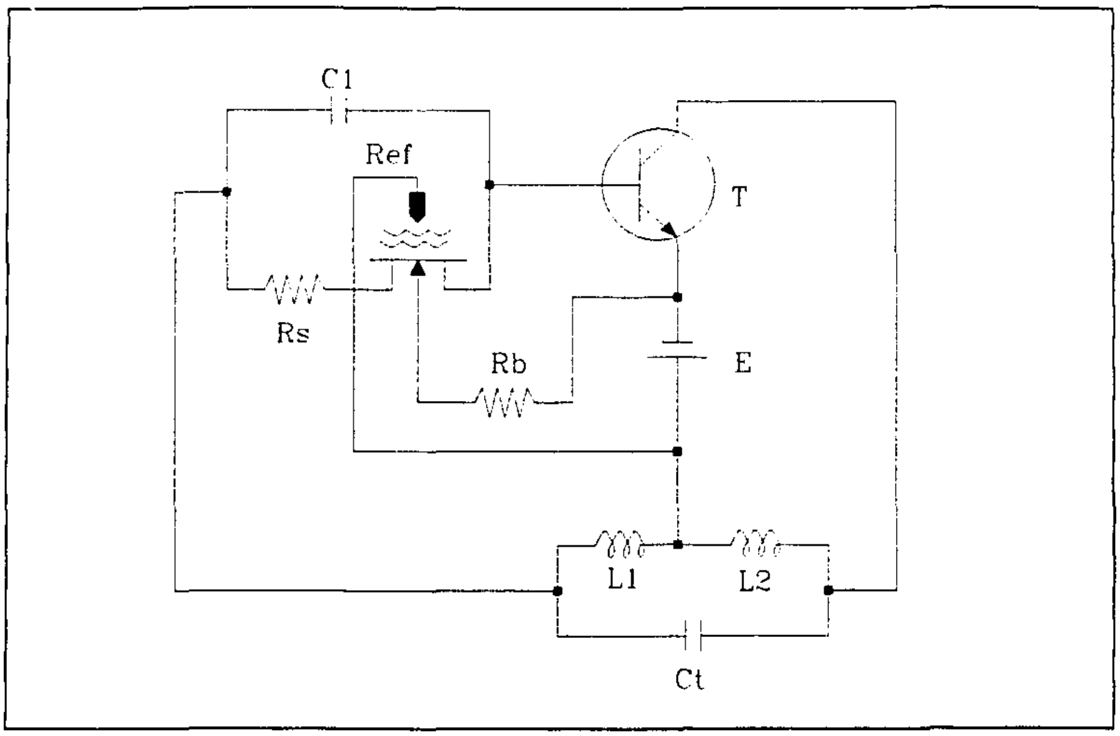


Figure 6-4. Final pH ISFET telemetry circuit.
Ref: Ag/AgCl reference, T: 2N3904(NPN), L1,L2: 47μH, Ct: 330 pF, C1: 100 nF, Rs: 249 kΩ, Rb: 249 kΩ, E = 1.60 V.

6.4 Telemetric Circuit Design and Manufacture

The telemetric device described in this chapter was based on the circuit in Figure 6-3 with a pH ISFET incorporated as a variable resistor in series with Rs. The final circuit is shown in Figure 6-4. The gate of the ISFET is biased by the reference electrode. The ISFET channel resistance varies in accordance with the pH dependant surface potential at the insulator|solution interface of the gate area. Changes in the combined resistance of Rs and the ISFET affect the pulsing rate of the device. Therefore the observed PF is directly related to the solution pH. The BF and PL are unaffected by any change in resistance.

This device was developed in three stages. First the circuit was tested on a regular breadboard and was powered by a Micronta variable DC power supply (model 22-8230, Radio Shack). In the next step the power supply was replaced with a battery and a miniature breadboard was used. The final prototype device used a printed circuit board powered by a battery. With each decrease in size, the performance and stability of the device was seen to improve. This can be attributed to the decrease of stray capacitances and inductances created by the long leads and breadboard connectors.

The balance of this section details the development of the prototype. The choice of the individual components is covered first, followed by the physical construction of the device. The last part of this section describes how the radio signal was received.

6.4.1 Choice of Components

The component values shown in Figure 6-4 were chosen after examining the role and effect of each component. These tests are summarized in the following sections.

6.4.1.1 Battery

The choice of battery was limited by what was commercially available. The battery had to be as small as possible and exhibit a flat discharge curve (*i.e.* constant voltage). Fortunately there are a number of batteries produced for watches, hearing aids, *etc.*, that meet these requirements.

A D379B silver oxide button-cell battery (Duracell, Mississauga, Ont.) was used in the prototype. This battery has a Zn anode and an AgO cathode. The initial voltage was measured as 1.61 V which dropped within a few minutes to a constant voltage of 1.58 V. The lifetime of the battery was listed as 11 mAh, and in the prototype the lifetime was 4-6 days. The dimensions were 5.8 mm in diameter and 2.2 mm thick. This is about the maximum size that could be used in an ingestible device. The connection to the circuit was made by using silver epoxy (Epo-Tek H21D, Epoxy Technology Inc., Billerica, MA) to glue on the lead wires. The epoxy was cured at 80 ^{o}C for 90 minutes or 50 ^{o}C for 12 hours. (At higher temperatures the battery may explode.)

There is a variety of other methods that can be used to power a biotelemetry device [3]. Other conventional batteries, such as lithium cells, are often used. Thin film batteries, using conducting polymers, which are presently in development will be very useful. Nuclear batteries have been mainly used in pacemakers where their long lifetimes offset the increase in cost. It is also possible to have a sensor-transmitter without a battery. In this case the device receives power from an external source by inductive coupling. After turning on, the device transmits the sensor information by inductive coupling on a different frequency.

6.4.1.2 Transistor

Perhaps surprisingly, the choice of the transistor was not critical. A low cost general purpose NPN switching transistor (2N3904, generic) was used initially. Later tests using higher priced audio and high frequency transistors showed no discernable improvement. The transistor does influence PL and PF, however it was easier to control these parameters by varying the other components. A generic 2N3904 was used in the prototype.

6.4.1.3 Inductors

The combination of L1, L2 and Ct determine the BF of the circuit according to Equation 6.2:

$$BF = \frac{1}{2\pi \sqrt{C_{Ct} L_T}} \tag{6.2}$$

where L_T is the total inductance given by:

$$L_T = L_{L1} + L_{L2} + 2M \tag{6.3}$$

The mutual inductance, M, between L1 and L2 is dependant on the type and relative position of the inductors. By measuring L_T when L_{L1} and L_{L2} are known, M can be calculated.

The BF increases during each pulse. In order to obtain consistent measurements, the BF was always measured at 1/10 PL. This gave the experimental relationship:

$$BF \approx \frac{0.8}{2\pi \sqrt{C_{Ct} L_T}} \tag{6.4}$$

which was determined from the data in Tables 6.1 and 6.2, in lieu of Equation 6.2.

Table 6.1 also shows that both PL and PF are controlled by L1 and L2. This occurs because when the inductance changes, the shift in BF results in a change in the impedance of C1. This mainly affects PL, but a slight change in PF is also seen. To keep things simple, L1, L2 and Ct were used to control BF while PF and PL were controlled by Rs and C1.

In the first test circuits, homemade inductors were used. Copper wire (28 or 40 gauge) was wrapped around either glass or plastic cores, both of which act as "air" cores. The geometry of a homemade coil could be easily customized to fit into an ingestible radio pill, however the difficulty in reproducing these coils defeated this advantage. Commercial inductors were found to be more convenient. Dale molded

Table 6.1
Effect of L1 and L2 on Signal
(L1 = L2)

L1/L2 (μH)	PF (Hz)	BF (MHz)	PL (μS)
1	could not	6.66	97
10	detect signal	2.15	157
22	14.28	1.40	236
47	14.38	1.01	343
68	14.51	0.85	374
100	14.73	0.69	430

FET: CH16-S2, pH: 2, Ref: Ag/AgCl, T: 2N3904, Ct: 120 pF, C1: 100 nF, Rs: 1 MΩ, Rb: 1 MΩ, E: 1.58 V.

Table 6.2
Effect of Ct on Signal

Ct (pF)	PF (Hz)	BF (MHz)	PL (μS)	PL (in air) (μs)
10	14.36	1.05	62	3625
22	14.17	1.01	81	732
33	13.07	1.66	426	344
68	13.24	1.05	200	826
120	12.94	1.00	331	221
220	12.87	0.83	310	279
330	12.78	0.71	299	296
470	12.74	0.62	296	308
1000	13.66	0.41	165	171

FET: CH11-S1, pH: 6, Ref: Ag/AgCl, T: 2N3904, L1,L2: 47 μH,
C1: 100 nF, Rs: 1 MΩ, Rb: 1 MΩ, E: 1.58 V.

inductors (Active Electronics, Montreal, Que.) from 0.1 μH to 100 μH (10% tolerance) were tested. These coils have a magnetic core of iron or ferrite and are produced in a tubular package (6.5 mm long, 2.5 mm diameter) similar to resistors. An added benefit of the molded coils was that the mutual inductance was insignificant in any configuration. This simplified the BF calculations. In the prototype, L1 and L2 were both 47 μH ferrite core inductors.

6.4.1.4 Ct

The relationship of Ct to BF is given above (Equations 6.2 and 6.4), and Ct has the same indirect effect on PF and PL as was noted for the inductors. An additional observation was that when Ct was less than 100 pF the stability of the circuit suffered. In particular, the PL in the air differed from the PL in solution (Table 6.2). This was attributed to the increased effect of the ISFET gate capacitance, and other stray capacitances, as Ct is lowered. By keeping Ct>100 pF this effect is eliminated.

Monolithic ceramic radial capacitors made by AVX Corporation (Active Electronics, Montreal, Que.) were used. These come in a small package (3.4x2.9x1.7 mm) and are rated as 5% tolerance up to 100 V. The Ct values tested ranged from 10 to 1000 pF. The value chosen for the prototype was 330 pF. Combined with the chosen inductors this gives a theoretical BF of 0.90 MHz according to Equation 6.2, and an experimental BF of 0.72 MHz according to Equation 6.4. This is located in the bottom half of the AM band.

6.4.1.5 C1

The timing capacitor C1 in the RC circuit was used to control PL and, in conjunction with Rs, was also used to control PF. The PL ranged from 23 μs for C1=0.47 nF to 307 μs for C1=100 nF (Table 6.3). Erratic behaviour was seen when C1 was less than 10 nF. While the shorter PL might seem advantageous in terms of prolonging the device lifetime, the PF increases at a faster rate; therefore the device is actually in oscillation a greater percentage of the time (*i.e.* higher duty cycle). These two factors suggest that a large capacitance be used. A value of 100 nF was chosen because larger value capacitors were also physically

Table 6.3
Effect of C1 on Signal

C1 (nF)	PF (Hz)	BF (MHz)	PL (μS)	Duty cycle (PF×PL)
0.47	1937	0.88	23	0.0474
1.0	1105	0.88	26	0.0285
4.7	271.1	0.77	40	0.0101
10	140.7	0.74	50	0.0073
33	41.60	0.75	115	0.0049
100	12.86	0.71	307	0.0039

FET: CH11-S1, pH: 6, Ref: Ag/AgCl, T: 2N3904, L1,L2: 47 μH, Ct: 330 pF, Rs: 1 MΩ, Rb: 1 MΩ, E: 1.58 V.

larger. The C1 capacitor was the same type as the Ct capacitor described above.

The relationship of C1 to PF is covered in the next section.

6.4.1.6 Rs

The resistor, Rs, connected to the ISFET source terminal is necessary because the resistance of the ISFET by itself is not sufficient. When the total resistance of Rs and the ISFET was below 100 kΩ, the circuit went into permanent oscillation (Table 6.4). As the ISFET resistance was only 1-10 kΩ, an Rs value greater than 100 kΩ was needed to ensure reliable operation.

Philips metal film resistors (Electrosonic, Mississauga, Ont.) rated as 1% tolerance, ¼W, were used. The prototype device used a Rs of 249 kΩ. These resistors are 2.3 mm in diameter and 7.0 mm long.

Table 6.4 indicates that the value of Rs has no bearing on either

Table 6.4
Effect of Rs on Signal

Rs (KΩ)	PF (Hz)	BF (MHz)	PL (μS)
10	permanent oscillation	0.87	
50		0.90	
87		0.89	
100	1346.0	0.81	59
158	866.9	0.77	54
249	584.2	0.75	52
412	364.2	0.76	51
499	301.6	0.78	50
634	241.4	0.75	47
866	178.8	0.74	50
1000	137.8	0.75	50

FET: CH11-S1, pH: 6, Ref: Ag/AgCl, T: 2N3904, Ct: 330 pF, C1: 10 nF, L1,L2: 47 μH, Rb: 1 MΩ, E: 1.58 V.

BF or PL. Both Rs and C1 determine the PF according to Equation 6.5:

$$PF \approx \frac{1}{0.7\ R_{Rs}\ C_{C1}} \tag{6.5}$$

which was derived experimentally. In the prototype the PF was approximately 57 Hz which corresponds to a pulse period of 17.5 ms.

6.4.1.7 Rb

Initially the ISFET base resistor, Rb, was omitted and the base was connected directly to the negative battery terminal. In this configuration the ISFET acts as a capacitor across the transistor's base and emitter terminals and this caused the device to go into permanent oscillation. By inserting Rb, the ISFET base was held at 0 V (relative to the negative battery terminal) while the AC current was decreased enough to prevent permanent oscillation.

It was observed that during the actual oscillation pulse, the voltage at the ISFET base was also seen to oscillate. This is due to capacitive coupling of the base with the source-drain channel. The ISFET base voltage returned to 0 V within 0.05 ms after termination of the pulse. With a pulse period of 17 ms this temporary deviation of base voltage is insignificant.

As expected, Rb had no effect on BF, PF or PL. The same type of resistor was used for Rb as for Rs. A value of 249 kΩ was used, as much for symmetry as any other factor.

6.4.2 Prototype Construction

The prototype device was designed and built in two parts (Figure 6-5). The telemetry circuit was fashioned on a custom-built printed circuit (PC) while the actual ISFET chip was mounted on a separate ceramic die. The halves were electrically connected by edge connectors on both the die and PC. The halves were held together physically by pincers for temporary use, or solder and epoxy for a permanent connection.

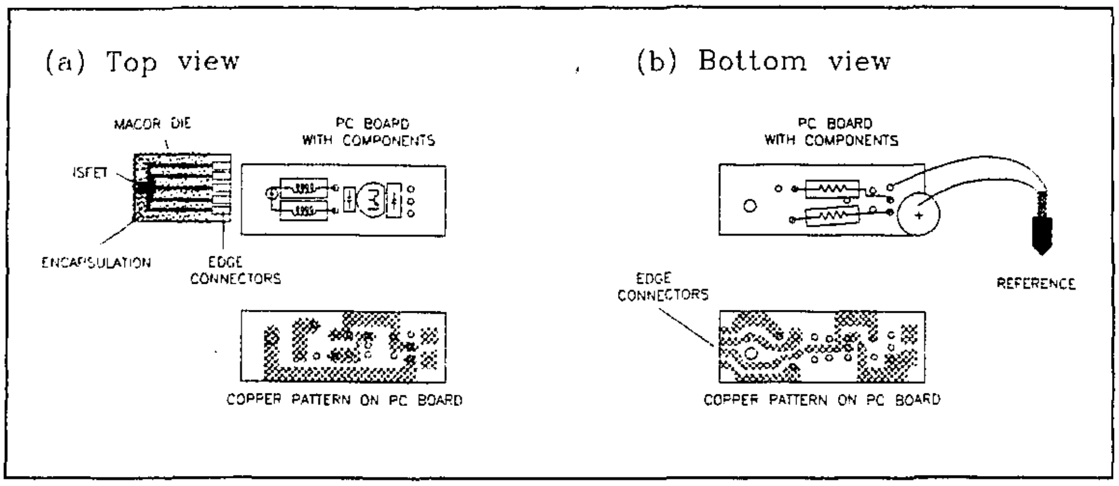


Figure 6-5. Prototype telemetry device.
Actual size except for reference electrode. The encapsulation forms a window on the ISFET to expose pH gate (not shown).

To use the prototype device in solution, the device was clamped vertically above the solution with just the encapsulated area of the die actually immersed in the test solution. In this manner the unencapsulated PC board was kept dry and accessible for circuit tests.

The reference electrode was an Ag/AgCl (3 M NaCl) Model RE-1 electrode (Bioanalytical Systems, West Lafayette, IN). The power to the circuit could be cut off by disconnecting the reference from the lead wires. This enabled each PC to be used over a much longer time (more than 3 months) than would have been possible if the battery was continuously discharging.

The ceramic dies and PCs were constructed in the laboratory by hand. The gold inlay pattern for the die and the copper circuit patterns for the PC were designed with the use of a computer-aided design (CAD) program (Autosketch, Autodesk Inc., Sausalito, CA). The dies were similar to the ISFET die described in Chapter 3, but a different gold inlay pattern was used. For the prototype device, only one ISFET was used at a time. The die was designed so that either of the two ISFETs could be used. This meant that 5 outputs had to be available: the sources and drains of both ISFETs, and the base. These were all lined up at the edge of the Macor die, opposite to the chip. The schematic of the die is shown in Figure 6-6. The procedure for the preparation of the die, mounting, and wire-bonding of the ISFET was

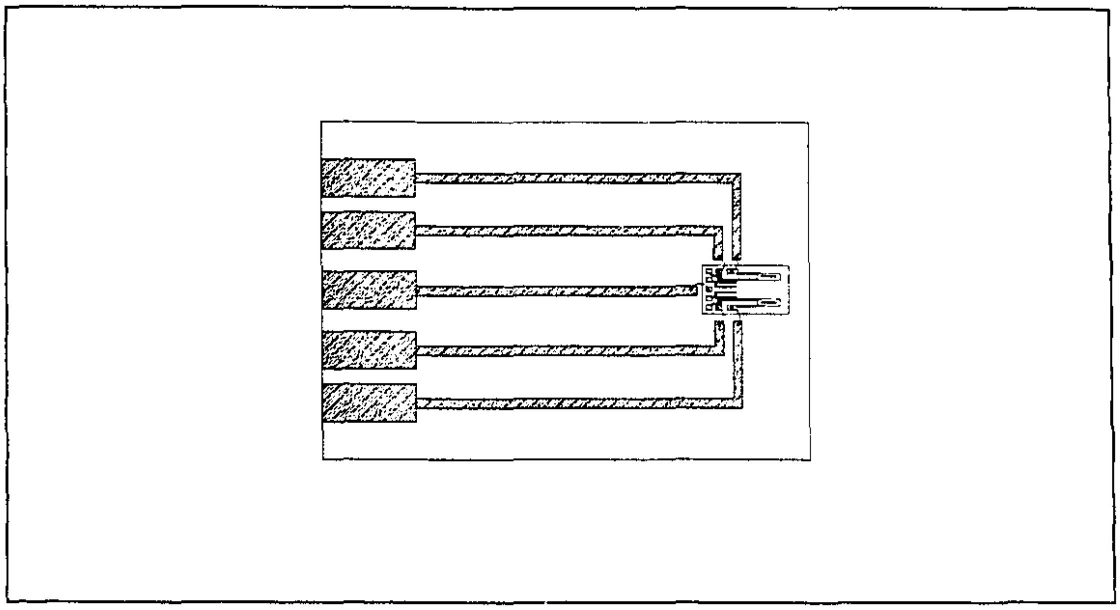


Figure 6-6. Schematic of Macor die used in the telemetry studies.

the same as in Section 3.4, except a new photoresist exposure mask was made, and there were fewer wire-bond connections to be made. No external wires had to be soldered to the gold inlay because the die was designed to be press-fitted or glued to the printed circuit telemetry device. This meant that the left edge of the die (when orientated as in Figure 6-6), had to be left free of epoxy encapsulation.

The PC, which controlled the ISFET and emitted the radio signal, was made from a dual-sided copper circuit board. Figure 6-7 shows the photoresist masks used to define the copper patterns on the PC. The edge connectors on the bottom of the board were used to connect the PC to the Macor die. Note that the PC has 4 connectors while the die has 5. In actual use, the die and PC are offset from each other, and only 3 connectors are used. When the base terminal on the die was lined up with base (1) terminal on the PC, the source and drain of ISFET 1 were connected to the PC. ISFET 2 was selected by connecting the base terminal of the die to base (2).

The PC was fabricated using a positive photoresist system from MG Chemicals (Toronto, Ont.). Each PC was cut, in a darkroom, from a 6x6 inch dual-sided copper circuit board pre-coated with MG#416 resist. The PC dimensions were 28x10 mm. The PC board was placed

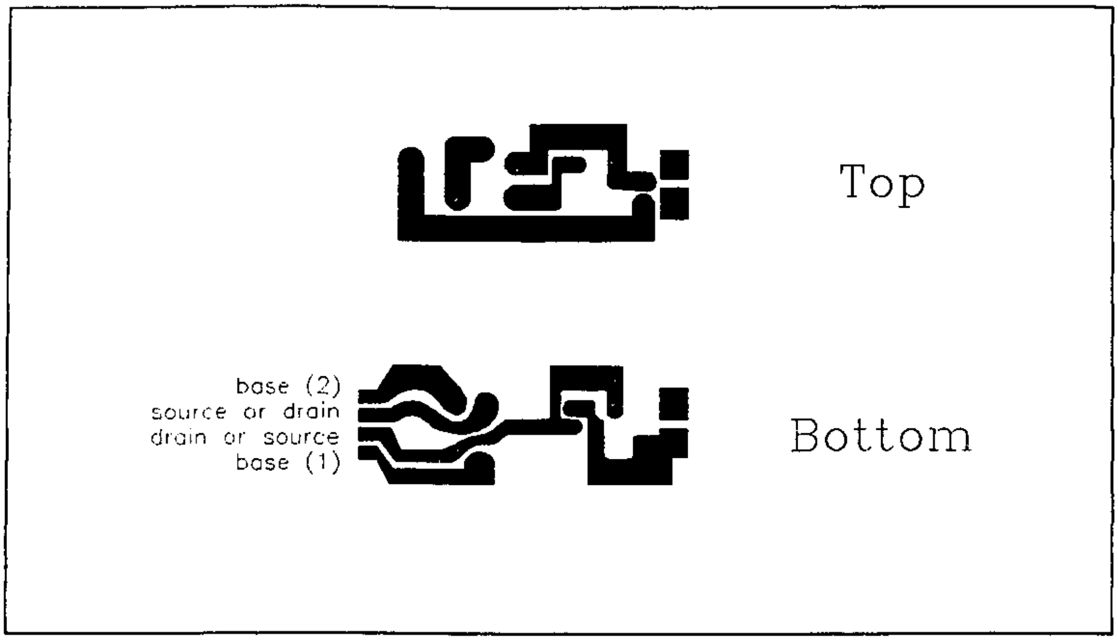


Figure 6-7. Photoresist masks for the prototype telemetry device.
(magnified, 0.6:1 scale)

between the 2 masks which were then taped together to maintain proper alignment. Each side of the PC board was exposed to a 275W sunlamp at a distance of 23 inches for 13 min. The resist was developed by swirling the PC for 5 min in a fresh solution of 1 part MG#418 developer and 4 parts warm tap water, then rinsed with distilled water. The copper was etched in a ferric chloride solution (MG#415), room temperature, for 15-25 minutes. After washing the PC with distilled water, the remaining resist was removed from the copper circuit by an acetone rinse.

Holes were then drilled in the PC board (Figure 6-8). The one large hole was made with a no. 60 bit (1.016 mm diameter). The rest of the holes were made with a no. 66 bit (0.838 mm diameter.

All components, except the battery, were mounted and soldered onto the PC board as shown in Figure 6-9. The negative terminal of the battery was glued to the copper circuit with silver epoxy (Epo-Tek H21-D). This was cured at 80 °C for 30 min. The silver epoxy was also used to attach a 6 cm lead wire to the positive battery terminal. The circuit was powered only when the two lead wires were connected at the reference electrode.

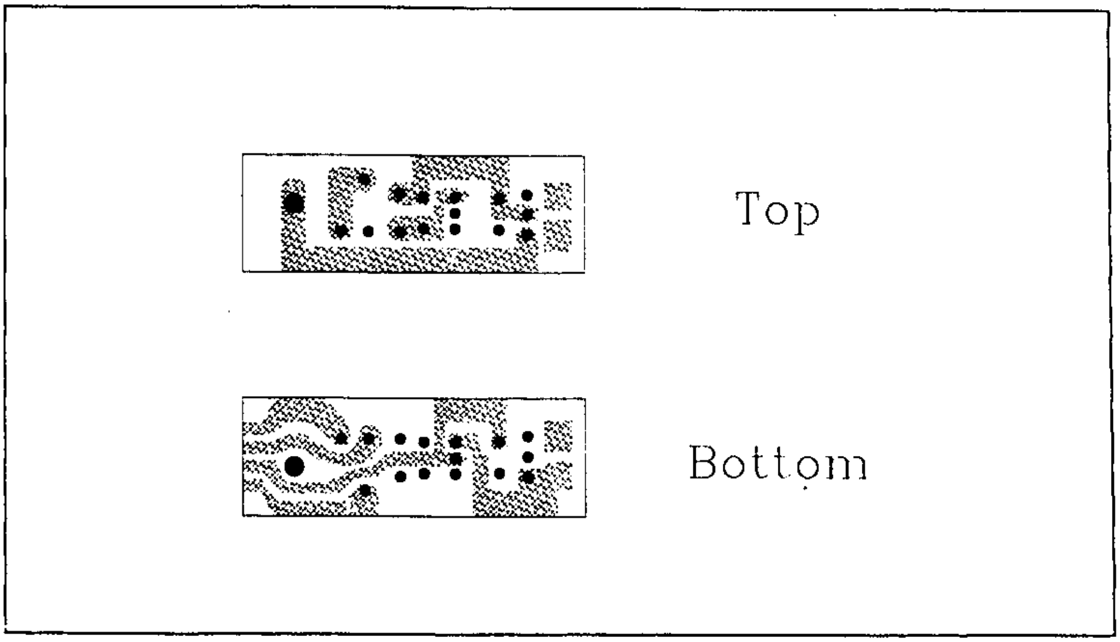


Figure 6-8. Copper printed circuit and holes.
(magnified, 0.6:1 scale)

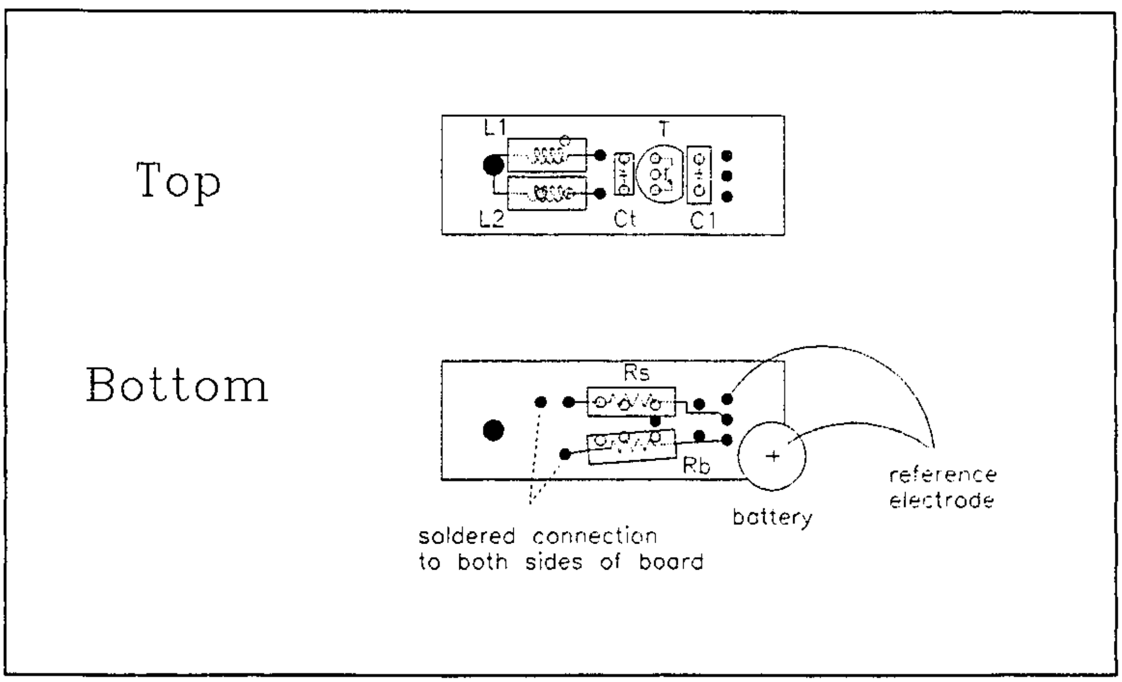


Figure 6-9. Components mounted on prototype telemetry device.
(magnified, 0.6:1 scale)

The prototype device was a compact sensor-transmitter for *in vitro* studies which was could be easily tested during actual operation. It was too large to be directly used as an encapsulated, ingestible radio pill, however it was on the proper scale. The necessary size reduction could be achieved by placing the ISFET on the PC board and the use of surface mounted resistors and capacitors or a thick film circuit. In this configuration the reference would have to be miniaturized and incorporated in the pill.

6.4.3 Signal Reception

The method of radio reception was kept in mind when the prototype telemetry device was being designed. Basically, the simpler the receiver, the better. The wideband, pulsing AM output of the prototype is one of the simplest radio signals to detect. Each pulse produces an audible buzz or "click" from the speaker. If the PF is low enough the signal can be recorded by listening to the clicking and timing a set number of pulses. At higher PFs the clicks fuse together to produce an audible tone with a pitch that varies with PF.

The simple requirements that the receiver had to meet enabled the use of an old commercial radio. The radio was a Silver Voyager 14 Transistor (model 14SF-38, Shin-Shirasuna Denki Mfg. Co., Nagoya, Japan). This radio was capable of receiving signals in the standard AM and FM bands as well as in the short wave region from 1.8 to 18 MHz. The use of an old radio was convenient because it was simpler to access the internal circuitry than a new one. The radio was powered by the variable DC power supply, set at 6 V, to eliminate the AC power line noise (mainly 60 Hz). Batteries could also be used for this purpose.

Either one of two instruments was used to measure the PF from the radio. Both instruments were set up to measure the voltage output to the speaker. The first instrument used was a Tektronix 100 MHz model 2230 digital storage oscilloscope (Tektronix, Beaverton, OR). The PF (or pulse period) could be read off the screen after manually positioning two cursors in the storage mode. Images could be stored and output to an Omnigraphic Series 2000 X-Y recorder model (Houston Instrument, Austin, TX) which was modified to accept the oscilloscope output. Appendix B describes the modifications that were made to the plotter. The hard copy output from the scope is illustrated in Figure 6-10. The problem with the oscilloscope was the time needed to store the image and adjust the cursors to get the PF. This led to the use of an electronic counter (Dana model 8010B, Dana Laboratories, Irvine, CA) which could provide a direct frequency or period readout. This was set up to trigger on the large, positive spike of the speaker voltage seen in Figure 6-10. The counter had an 8 digit readout and an 150 MHz limit.

The oscilloscope and plotter were also used to monitor the telemetry circuit directly during initial tests. The images in Figure 6-2 were obtained in this manner. Hooking up the oscilloscope, or any other instrument, to the circuit was observed to perturb the circuit slightly. Therefore, in all the actual pH tests the prototype was freestanding and signals were measured remotely.

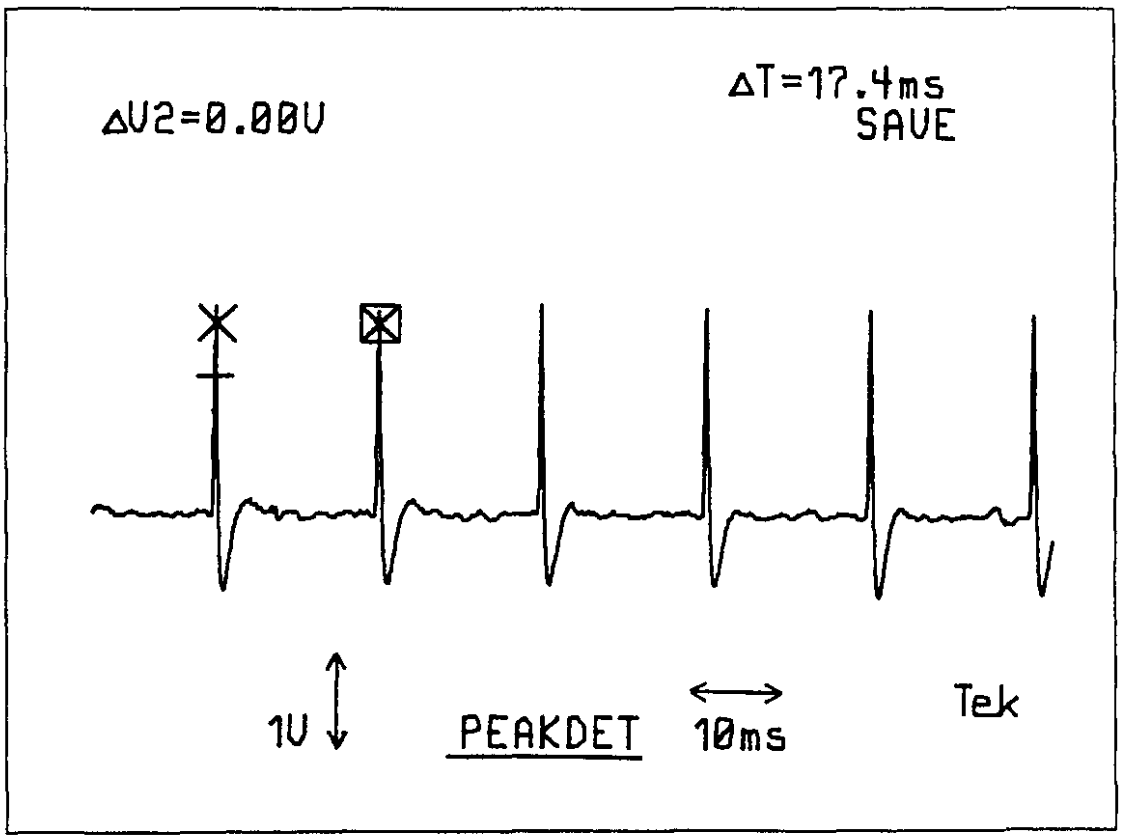


Figure 6-10. Pulsing output as seen at radio speaker.

The prototype was expected to have a BF of approximately 0.72 MHz. The signal was detected by the radio from the bottom of the AM band (0.55 MHz) up to 0.95 MHz. The signal peaked between 0.85 and 0.94 MHz. There was a sharp drop-off at 0.95 MHz. As expected, the signal strength was dependant on the orientation of the radio with respect to the transmitter. This effect can be eliminated by the use of a more sophisticated antenna and receiver. The signal was easily detected when the distance between the radio and transmitter was less than 25 cm, but was undetectable at distances greater than 50 cm.

6.5 Testing of the Prototype in Aqueous pH Buffers

6.5.1 Experimental

In these experiments the prototype devices were immersed in a series of pH buffers prepared in the laboratory according to the protocol set forth by the National Bureau of Standards (NBS - now known as the National Institute of Standards and Technology). The prototype was held vertically in a clamp with the encapsulated ISFET at the bottom. The ISFET was immersed in the test solution by raising the beaker until the ceramic die was half submerged. The experiments were performed in subdued lighting to prevent any possible photoelectric effects at the gate surface. This was achieved by using beakers covered with electric tape and placing a box over the sensor-transmitter and beaker during the measurements.

All of the prototypes used the UU03 ISFET chip obtained from Prof. J. Janata [4]. Either of the two ISFETs on the chip could be used in the prototype. The UU03 chip was described in Section 3.2.1.

Each ISFET was immersed in the tetroxalate buffer for 1 hour before use. The ISFET and die were washed with distilled water and patted dry between solutions. Upon immersion in a test solution, the initial reading and time were recorded, then readings were subsequently taken every 30 seconds. The length of the runs varied from 5 to 15 minutes. The chips were stored dry after being washed.

The radio was positioned approximately 10-15 cm from the prototype device. This gave a strong signal that was easy to trigger on. For these experiments the pulse period was read off the electronic counter. This mode was recommended in the counter's manual because the period measurement is more accurate than the frequency measurement at low frequencies. The counter averaged 100 periods for each reading. Therefore the readout was effectively the average period over approximately two seconds.

6.5.1.1 pH Buffers

The NBS procedure for the preparation and handling of standard aqueous buffers has been described by Bates [5], and further information can be found in the CRC Handbook [6]. The buffers used in this study were made following this procedure, but the NBS standards were replaced with commercial, high purity, reagents. All the buffers were made up at room temperature in distilled, deionized water and were stored in Nalgene bottles. The pH of each buffer was checked in the laboratory on a Accumet Model 805MP pH meter (Fisher Scientific Co., Montreal, Que.) using new pH and saturated calomel electrodes (models 13-620-256 and 13-639-52, Fisher Scientific Co.). The meter was calibrated with Fisher buffers B-80 and B-79 (pH of 9.18 and 4.01 respectively). The measured pH's of all the standard buffers were within ±0.05 units of the NBS standard buffer values.

The tetroxalate buffer (pH = 1.68) was made by dissolving 12.6134 g of potassium tetraoxalate (>99.5%, Fluka Chemical Corp., Ronkonkoma, NY) and diluting to 1 liter volumetrically.

The tartrate buffer (pH = 3.56) was a saturated solution of potassium hydrogen L-tartrate (>99%, Fluka Chemical Corp.). The solution was vacuum filtered before use. This buffer was not used after 2 days due to the possibility of mold growth.

For the phthalate buffer (pH = 4.01), 10.1257 g of potassium hydrogen phthalate (>99.5%, Fluka Chemical Corp.) was required per liter of solution. The salt was dried overnight at 130 °C before weighing.

The two phosphate buffers were made from potassium phosphate, monobasic (ACS reagent, 99%) and sodium phosphate, dibasic (certified ACS, 100.7%), both from Fisher Scientific Co.. The Na_2HPO_4 was dried overnight at 130 °C, and the KH_2PO_4 was dried for 30 minutes at 110 °C. The phosphate I buffer (pH=6.86) required 3.3886 g of KH_2PO_4 and 3.5348 g Na_2HPO_4 per liter. The phosphate II buffer (pH=7.41) used 1.1792 g of KH_2PO_4 and 4.3042 g Na_2HPO_4 per liter.

6.5.2 Results

There were three prototypes that were tested successfully. For the first two devices, the printed circuit PC1 was combined with the pH ISFETs CH22-S2 (PT1) and CH25-S2 (PT2). The third prototype (PT3) used the pH ISFET CH23-S2 which was permanently attached to PC2.

The three prototypes were tested in air before they were used in solution. Two devices, PT1 and PT3, had a pulse period of approximately 20 ms. The third, PT2, was tested soon after removal from the oven and showed a higher period of 33 ms. The pulse period for all three devices dropped after the initial conditioning in the tetroxalate buffer. The period decreased by 2 ms for PT1 and PT3, while the PT2 period dropped by 12 ms. The behaviour of PT2 can be attributed to the differences in temperature. After the prototypes were used once, the pulse period of the dry device in air was observed to be greater than the period in solution, but only by approximately 0.5 ms.

The effect of stirring was investigated. The stirring rate had to be kept low because only 5 mm of the device was immersed in the solution. Even at slow rates, stirring was seen to increase the fluctuations in the readings. The magnitude of this effect was not quantified. All test results reported here were obtained in quiescent solution.

The response of the prototype pH telemetry device in the aqueous buffers can be seen in Figures 6-11 and 6-12. These data were obtained by immersing device PT3 in each of the solutions for 5 minutes. The calibration curve, Figure 6-11, shows a non-linear response over the range of these buffers. In this experiment the device was conditioned in the tetroxalate buffer, then the solutions were tested in order of decreasing pH, starting with the phosphate II buffer. Similar results were seen

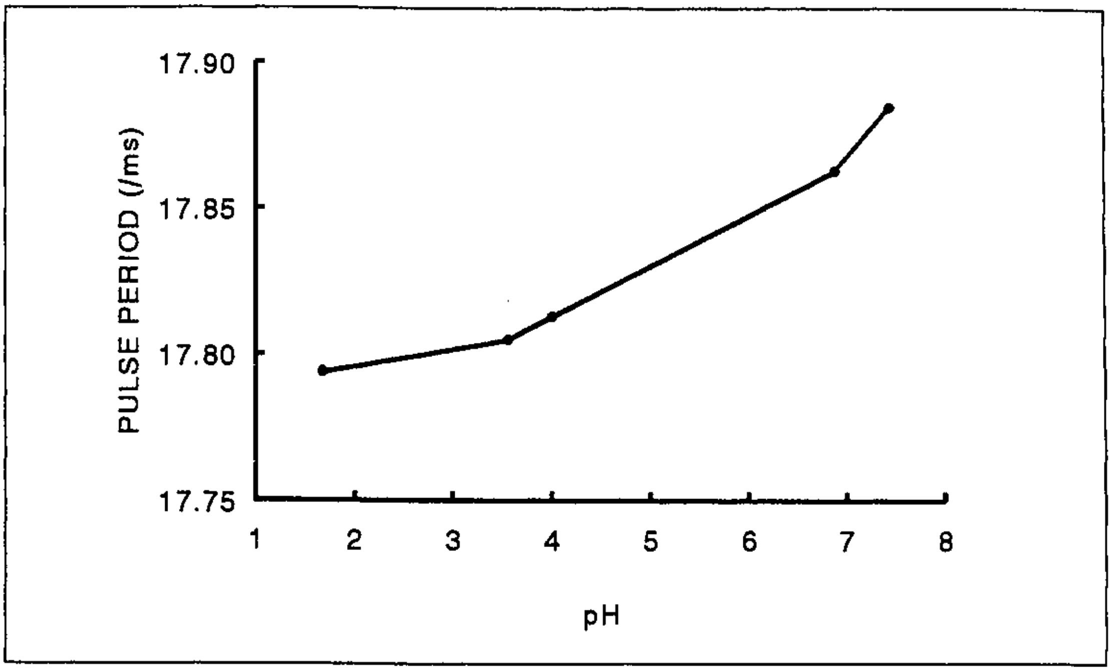


Figure 6-11. Calibration curve for prototype in aqueous pH buffers.
Data from prototype PT3, 2.5 minutes after immersion.
Buffers and conditions described in text.

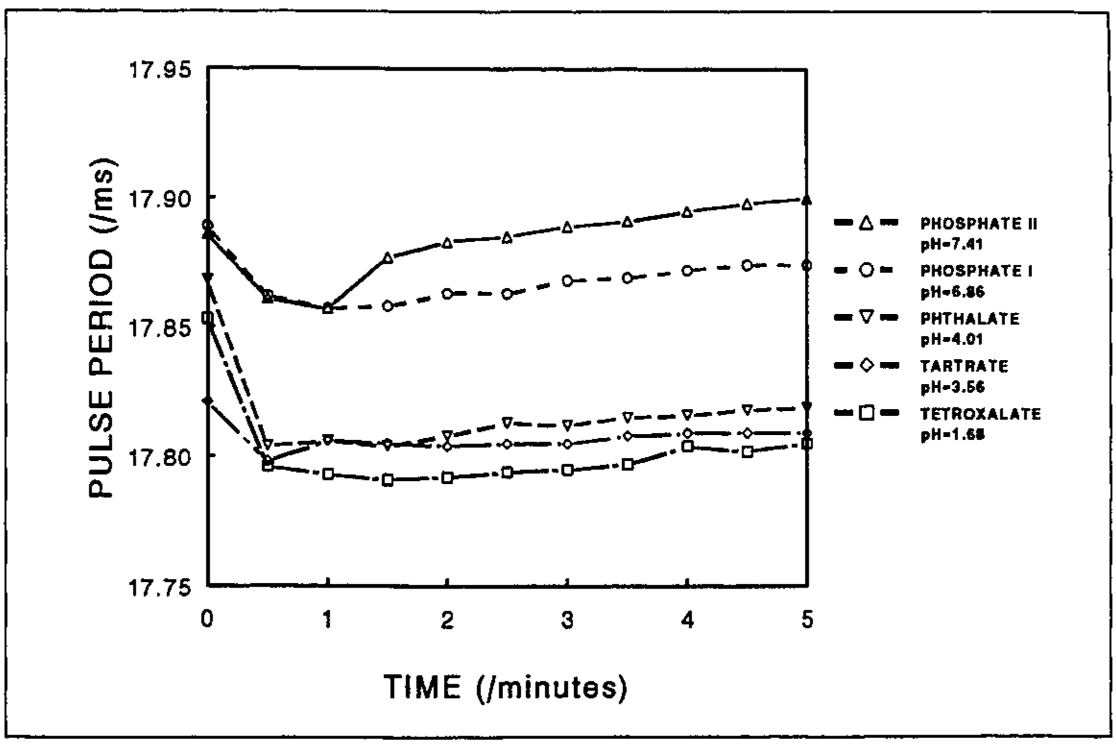


Figure 6-12. Time study of prototype in pH buffers.
Data from prototype PT3. Buffers and conditions described in text.

when the order was random for this and the other two prototypes.

The readings in Figure 6-11 were taken at the halfway point of the five-minute run. The importance of the elapsed time is illustrated in Figure 6-12, which shows the period readings for the five buffers over the entire run. It takes over a minute after immersion for the response to settle to a steady-state signal. After the first minute a drift in the response was seen, even in the short time frame of this experiment. Because of this drift, the shape and position (along the y-axis) of the calibration curve is dependant on the elapsed time.

The average magnitude of the drift was determined to be 0.005 ms/minute in the short term (*i.e.* over a span of less than 10 minutes).This short term drift rate may seem small, but it actually corresponds to a rate of roughly 0.3 pH units per minute for the data in Figure 6-11. The drift may be positive or negative. Over several hours the direction of the drift often reversed, resulting in a deceivingly lower rate of drift.

6.5.3 Discussion

The slow response seen on immersion is not surprising. In general, ISFETs are reported to have a response time of less than a second, but this is for a step change in the solution concentration. In the procedure used here, the dry ISFET is placed in a quiescent solution. There is, naturally, a time lag before the electrochemical double layer is formed at the gate surface. Stirring hastens the initial response, but introduces undesired fluctuations.

The procedure of taking a reading at a specific time after immersion in the test solution is commonly used in potentiometry. This helped to make the prototype data more presentable, however this alone cannot compensate for the problems presented by the data for the prototype. There are two very basic problems, which are partially related. The first is the lack of sensitivity, especially when the magnitude of the signal is considered. It is difficult to measure a small change in a large number. The change in the signal over the pH range tested was only 0.05%. By calculating backwards this indicates a change in R_{DS} of only 1 kΩ. This is too small relative to the combined resistance of R_S and R_{DS}. The value of R_S could be reduced, but not low enough to eliminate this problem. Ideally a new ISFET could be designed to give a high R_{DS} and, more importantly, a higher rate of change in resistance relative to the surface potential.

The second problem is the high rate of drift relative to the sensitivity. If a new ISFET was to increase the sensitivity while exhibiting the same drift rate, this problem would be solved. However, the drift may also increase. This could only be evaluated if a new ISFET was available. Another approach is to try to reduce the effect of the drift electronically. Feedback is commonly used to regulate the output of an FET. This would require a different, and more complex circuit. The drift can also be negated by using a pair of ISFETs to differentiate between the signal and drift. This method of common-mode rejection is the basis of the differential sensing technique used in Chapter 7.

As it stands, the drift is far too high compared to the sensitivity of the response for this prototype. Further efforts at developing an actual gastric pH pill would be unwarranted with the present circuit and components. If a new ISFET could be developed to solve the problems discussed, then the next step would be to further reduce the size and properly encapsulate the device.

6.6 Summary

The data presented here demonstrate that the prototype was successful as a telemetric pH sensor in standard solutions *in vitro*. Several problems have been identified which must be addressed before continuing the development of this device towards the ultimate goal of a gastric pH sensor. One solution to these deficiencies is the design and fabrication of a new ISFET specifically for this type of circuit, however this was not possible in this laboratory. An alternative course based on differential sensing from a pair of ISFETs was pursued. This work is presented in the next chapter.

6.7 References

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CHAPTER 7

DIFFERENTIAL SENSING

7.1 Introduction

The concept of differential sensing, first mentioned in Sections 2.3.1.1 and 2.4.2.2, is neither novel nor limited to ISFET applications. The same basic principle applies to many analytical methods, such as double-beam spectrophotometry. In a differential chemical sensor the signal of a chemical sensor is compared with the signal of another sensor, which exhibits a similar response but is chemically non-selective. The difference in the responses is monitored, and this represents the signal due to the chemical recognition of a particular species. This results in an enhancement of the selectivity and stability of the sensor due to the common-mode rejection of the effects of interfering species, temperature, voltage variations, and other uncontrollable parameters.

Differential sensing has been rarely used in conventional potentiometry. Its use in ISFET research is more common because of the ease of fabricating matched pairs of ISFETs on an IC chip, and the need for a reference electrode on the same scale as the ISFET. The ISFET pair requires a third electrode to bias the gates. While a conventional reference electrode can be used, it has usually been replaced with a simple noble metal electrode [1-4]. The metal electrode can be easily miniaturized; often it is just an evaporated metal film on the ISFET chip. The potential of the metal electrode cannot be expected to be constant, and the voltage fluctuations are seen by both ISFETs. As long as the outputs of the ISFETs vary by the same amount, the differential circuit eliminates the effect of the unstable gate potential. The metal electrode is called a *pseudo-reference* electrode. The onus of providing a stable "reference potential" is passed on to the reference ISFET, or ReFET.

There are two distinctly different types of ReFETs. The first type consists of ReFETs that *do* respond to changes in the solution, but respond differently than the ISFET. The electrolyte|ReFET interface in this type of ReFET is said to be ion-unblocked. The difference between the ISFET and ReFET is that the ISFET should show a greater selectivity to a particular ion. For example, a pair of pH ISFETs which have different pH sensitivities can be used [3]. A recent report describes a ReFET with a chemically attached polymer that exhibits a near zero pH sensitivity [5]. Another popular version uses an enzyme entrapped in a polymer matrix on the selective ISFET, while the ReFET is covered with just the polymer matrix alone [4]. In this type of differential sensor, the sensitivity of the observed response is usually less than what would be seen for the selective sensor by itself, but this is more than offset by the increases in stability and selectivity. This type of ReFET has been properly characterized and is well established.

The second type of ReFET uses an ion-blocking layer as the interface between the FET insulator and the solution. This approach has been viewed with some skepticism [6,7]. The argument against this type of ReFET is that an interfacial potential exists at any blocked interface, which cannot be stable because of the low exchange-current density (the passage of ionic or electronic charges, equal in both directions, across an interface at equilibrium). Indeed, if the interface is ideally blocked, then the exchange current would be zero, but the interfacial potential should also be zero and the interface should act as a capacitor. However, achieving an ideally blocked interface is very difficult. Deviations from ideal behaviour are due mainly to the surface adsorption of charged species. Unfortunately, this phenomenon is quite common [6,8].

The basis of the above argument is the assumption that the existence of an ideally ion-blocked layer is impossible, and that any deviation from ideal behaviour is fatal. Nevertheless, research in this area has continued, mainly in Japan [8-13] and Europe [14-16]. This research acknowledges, and attempts to correct, or compensate for, the problems due to non-ideal behaviour. The next section reviews the different layers that have been used previously in this area of research, and Section 7.3 introduces the blocking layer used in this chapter.

7.2 Ion-Blocking Layers

The ideal blocking layer must possess four qualities: 1) it must block the passage of all ions and electrons, 2) be physically stable, 3) be resistant to the adsorption of charged and neutral species, and 4) it should not alter the electrical characteristics of the FET. When these conditions are met, the interfacial and FET-insulator capacitances can be considered as a single gate capacitance.

The initial efforts at producing a blocking layer focussed on hydrophobic organic polymers films such as Parylene (Union Carbide Corp.) [8-11], Teflon (E.I. duPont de Nemours & Co. Inc.) [12], and polystyrene [13]. These were found to be inadequate because of surface adsorption and the thickness ($\geq$ 100 nm) of the film required to ensure the elimination of pinholes. These thick films adversely affect the FET's electronic characteristics. Later work has concentrated on altering the surface chemistry by chemical modification. The results indicate that it is impossible to produce an ideally ion-blocked layer by this method because of the difficulty of capturing enough of the active sites [14]. Bergveld *et al.* [5,16] have shifted their research towards non-blocking layers chemically bonded onto the gate surface.

It has been suggested that a Langmuir-Blodgett (LB) film could be used as an ion-blocking layer [17,18]. These films exhibit the required insulator characteristics, and are thin enough (5-100 nm) that they would not affect the FET performance. The problem of surface adsorption would still exist, and the mechanical fragility of the films is also a problem. An advantage of LB films is that chemically selective functionalities can be incorporated into the film [19,20]. The feasibility of ReFETs using LB films remains to be determined.

7.3 Self-Assembled Alkanethiol Monolayers

The consideration of LB films led to the investigation of an alternative type of blocking layer, namely self-assembled alkanethiol monolayers on gold surfaces, for use as a new ReFET surface. The monolayer is formed by the specific interaction between the sulfur atom and the gold surface, resulting in a densely packed, crystalline or liquid-crystalline film [21]. While the affinity of sulfur for gold has been known for some time [22], the bulk of research in this area has taken place in the last 5 years [21,23-29].

The adsorption process occurs spontaneously upon immersion of gold into a solution containing the alkanethiol. The resultant film is shown in Figure 7-1. The adsorbed species is thought to be a gold thiolate, $RS^{-}Au(I)$ [23]. The alkyl chains, or tails, form a closely packed organic film. The angle of the tails relative to the gold surface and the packing geometry are dependant on the alkanethiol and the adsorption conditions [21,28]. The film thickness for an octadecane (C18) thiol is approximately 2 nm. The stability of the monolayer is extremely high. Generally, it can be removed only by mechanical abrasion.

Electrochemical studies [24-26,29] have shown that alkanethiol monolayers strongly inhibit ion transport and electron transfer.

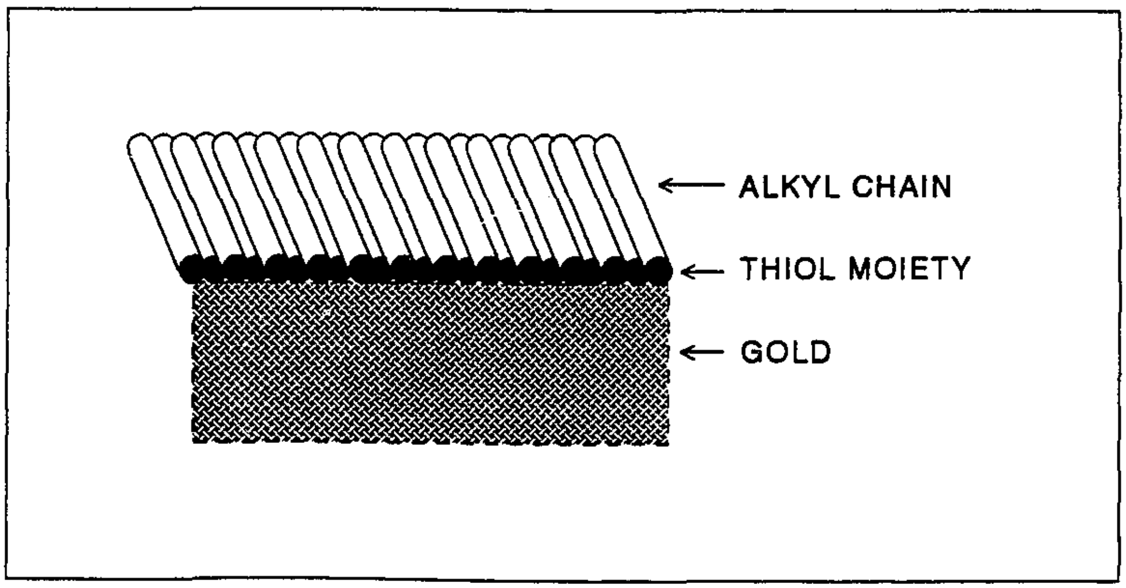

Figure 7-1. Self-assembled alkanethiol monolayer on gold.

Deviations from ideal behaviour have been attributed to the presence of pinholes in the film [26,29]. The fraction of the surface area occupied by pinholes ranges from 10^{-2} to 10^{-5} (99 to 99.999% coverage). Pinholes can be reduced by using a smooth, clean gold surface and a long-chain alkanethiol.

Self-assembled alkanethiol monolayers possess three of the four qualities required for an ideal blocking layer. The blocking action is excellent when the proper conditions and reagents are used. The physical stability is far greater than that of LB films, and the deposition is fairly straightforward. The monolayer is thin enough so that the FET electronic characteristics should not be adversely affected. The last desired quality, resistance to adsorption, remains a problem. Though not addressed in this thesis, it is reasonable to expect that the organic monolayer would be susceptible to adsorption, especially in biological matrices.

This chapter presents the first use of an alkanethiol monolayer on gold as a blocking layer on a potentiometric sensor. A schematic of the ReFET is given in Figure 7-2. The metal layers between the organic

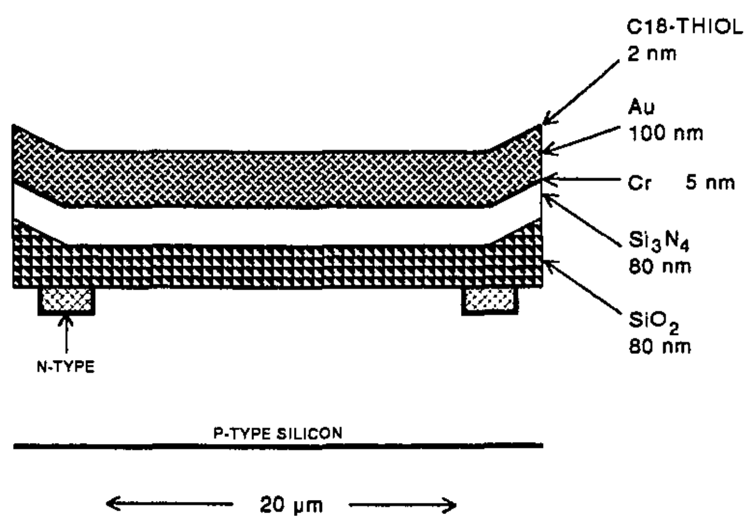

Figure 7-2. Schematic of ReFET incorporating alkanethiol monolayer.
(not to scale)

monolayer and the Si_3N_4 insulator were formed by vacuum sputtering (the thin layer of chromium is present to enhance adhesion). The gold layer is electrically floating and equipotential [30]. The alkanethiol| metal|insulator combination acts as a single gate capacitance.

In this work, an alkanethiol ReFET was used in conjunction with a regular pH ISFET on the same chip. In an optimal differential sensing setup, the surfaces of the ISFET and ReFET would be more closely matched. Other research groups are presently working on introducing chemical functionalities into alkanethiol monolayers [27,28]. It should be possible to modify the monolayer to confer selectivity. An ISFET using such a selective monolayer would then better match the ReFET.

7.4 Experimental

7.4.1 ReFET Construction

One of the two ISFETs on a UU03 chip (see Sections 3.2.1 and 6.5.1) was used, as received, as a pH sensor, and the other was used for the ReFET. The first step in the construction of the ReFET was to prepare the chips for the deposition of the Cr/Au metal film. The FET chips were placed in a 120 °C oven for two hours and subsequently stored in a desiccator. A layer of paper was glued (Scotch Brand 6065 spray mount glue, 3M Canada Inc., London, Ont.) onto a 2.5x7.6 cm glass slide. Thirteen chips were then seated in recesses cut out of the paper. Masks were prepared from 25.4 μm thick Teflon film (DF100, Dilectrix, Lockport, NY). The Teflon was taped onto a plastic block and 0.25x0.60 mm holes were made with a metal punch. The Teflon was then positioned on the glass slide so that the holes were lined up with the gate areas, and then taped in place.

Metallization of the gate area was performed by Guy Rodrigue in the Dept. of Electrical Engineering at McGill. A Key High Vacuum Products Model KV-301 vacuum sputterer was used, and the film thickness was monitored during sputtering with a Granville-Phillips Series 270 gauge controller. The base pressure before deposition was $4x10^{-8}$

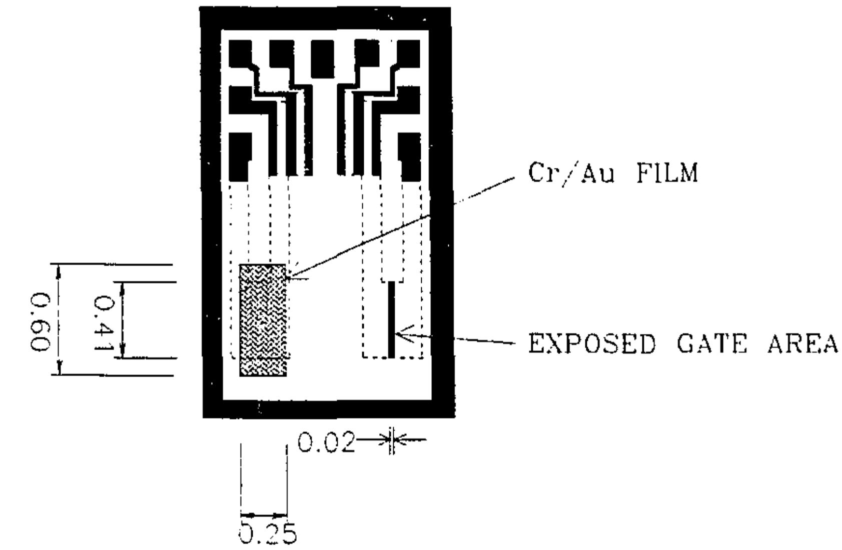

Figure 7-3. Schematic diagram of UU03 chip after metallization.
All dimensions are in mm. (scale=2.54cm:1mm)

torr. The chromium was melted at 60 A (amperes), then vaporized at 360 A. A 5-nm Cr layer was deposited at a rate of 3.5 nm/s. The gold was then melted at 150 A and vaporized at 400 A. The 100-nm Au film was deposited at a rate of 2.5 nm/s. Figure 7-3 shows the chip after removal of the Teflon mask.

The ISFET chips were die mounted, wired, and encapsulated according to the procedure described in Section 3.2.1. Extreme care was necessary when applying epoxy around the evaporated gold because the affinity of the epoxy for the gold caused it to flow over the entire gold surface. To avoid this, the epoxy was applied only to the top half of the chip (covering the wires and bonding pads) and around the edges of the bottom half (the gate area). No epoxy was used between the ReFET and ISFET gates.

The last step was to coat the gold surface with the alkanethiol layer. The encapsulated chips were washed with deionized, distilled water followed by absolute ethanol, then dried in an 120 °C oven for a minimum of one hour. A 1.0-mM solution of 1-octadecane thiol (98%, Aldrich Chemical Co., Milwaukee, WI) in absolute ethanol was prepared. After the chips cooled to room temperature, they were immersed into the fresh thiol solution in a Nalgene bottle, and the bottle was sealed with Parafilm. The chips were left in the solution for 18 hours at room temperature. After removal, the chips were washed with absolute ethanol followed by deionized, distilled water, then patted dry. The chips were usually stored dry.

The reagents and conditions for the alkanethiol monolayer deposition were chosen to try to maximize coverage and blocking ability [21,26,31]. A similar film was observed to have a fractional pinhole area of less than 6×10^{-6} [26]. Unfortunately, there was no way to test the extent of coverage on the ReFET. While total coverage is assumed, the presence of pinholes is certainly possible.

7.4.2 FET Controller Modification

The hardware of the FET Controller (Chapter 3) was modified to allow simultaneous operation of up to four ISFET/ReFET pairs using a differential circuit. The FET Controller software (Chapter 4) did not require any changes. The modifications had no effect on the testing of single FETs.

The first ISFET control board (FC2 - see Sections 3.3.1 and 3.5.4) was modified to give the circuit in Figure 7-4. The ISFET was hooked up to the FET Controller in the normal fashion. The ReFET was connected to the terminals originally used for the IGFET (the IGFET gate terminal, G0G, was not used). The FET sources are the inputs to a matched set of current-to-voltage converters. The final stage is the differential amplifier. The balance of the circuit seen in Figure 3-4 was deactivated by removing the chips RP2-1, RP2-2, and OA2-13. This allowed the output of op amp 3 to be connected to the ISFET control board output, S0. This was repeated for the other three ISFETs controlled by board FC2. Thus, the data acquired by the software for ISFETs 1 through 4 actually represents a voltage proportional to the difference in the drain currents of the four ISFET/ReFET pairs.

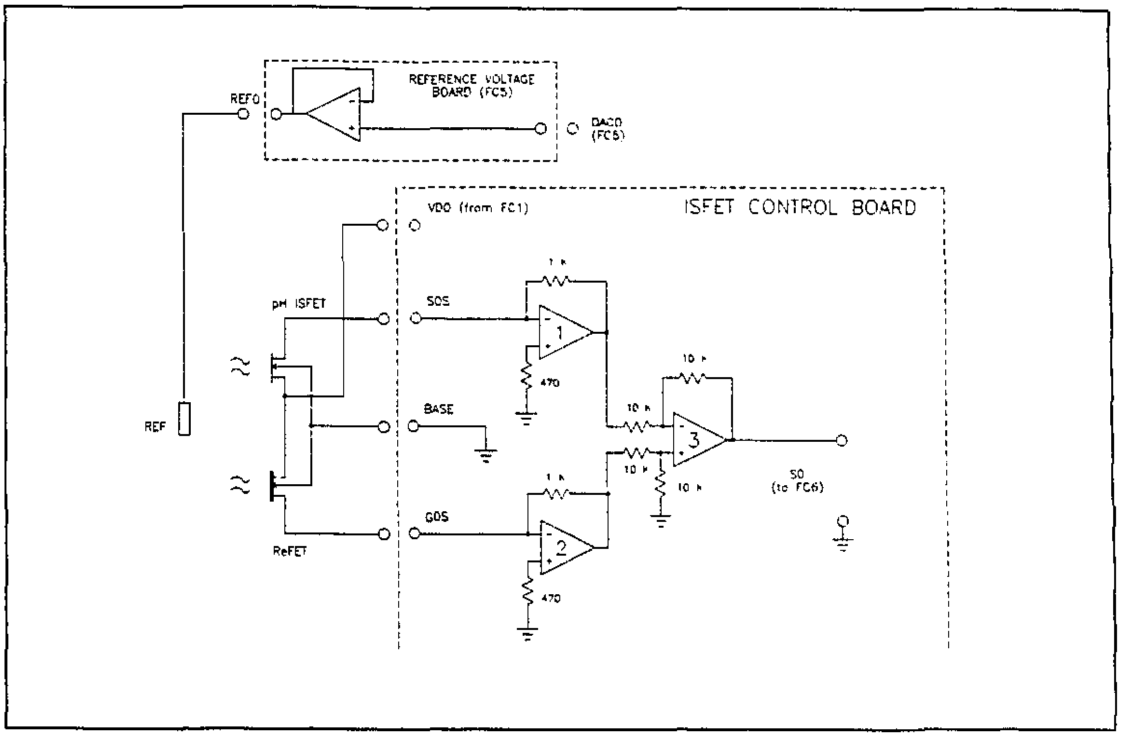

Figure 7-4. Schematic diagram of differential sensing control circuitry for one ISFET/ReFET pair

7.4.3 pH Buffers

The pH's of all the buffers were checked according to the protocol described in Section 6.5.1.1. For the initial experiments, the buffers were made following the procedure also given in Section 6.5.1.1.

The last set of experiments involved the titration of phosphoric acid with a sodium hydroxide solution. A solution of approximately 0.1 M H_3PO_4 was made by diluting reagent grade H_3PO_4 (assumed to be 85%). The titrant was reagent grade 0.1 M NaOH which was standardized against primary standard grade potassium hydrogen phthalate. Sample volume was 25.0 ml. The titrant volume and glass electrode pH readings were recorded manually during the titration. The differential output data was retrieved from the computer after the run and matched to the titrant volume and pH readings.

The ISFET and ReFET gates were biased with either an SCE reference or a platinum bullet pseudo-reference electrode (approximate surface area = 2.4 cm^2) connected to the front panel REF0 output.

7.5 Results

7.5.1 Preliminary Tests

All of the ISFETs and ReFETs were tested individually, using the test FET program, before use in the differential sensing configuration. The alkanethiol ReFETs exhibited normal response, however the drain currents appeared to be lower than usually seen for the pH ISFETs. This is a subjective observation, because the magnitude of the current response varies for all FETs and a limited number of ReFETs (6) were tested.

Analysis of the test FET data indicated that the ISFET threshold voltage, V_T^*, was approximately equal to 0 V for both the ISFETs and ReFETs.

As a preliminary experiment, an ISFET/ReFET pair was tested in different buffers using the test FET program. With this program, each FET/buffer combination constituted a separate run. After all the data were collected, the drain currents for the ISFET and ReFET were compared. The difference in the drain currents was plotted against the pH of the buffers (Figure 7-5). The results indicate that alkanethiol monolayer did suppress the pH response of the ReFET. The insensitivity at low pH for the saturated case may have been due to the large drain currents (>1000 μA) observed in both FETs. This may have limited the response range.

The next set of experiments used two ISFET/ReFET pairs in the differential sensing mode to establish the performance characteristics of the system, and determine the optimum settings for the software parameters.

It was quickly seen that the differential output signal was quite small - on the order of tens of millivolts. The highest gain (8) was used for the A/D converter, corresponding to a ±1.25V range and a resolution of 0.6 mV. Because the data acquisition system was being operated near its limits, we decided to take a closer look at the noise in the signal. The initial test is shown in Figure 7-6(a). With only one A/D conversion sample per point, the noise level was quite disturbing. The

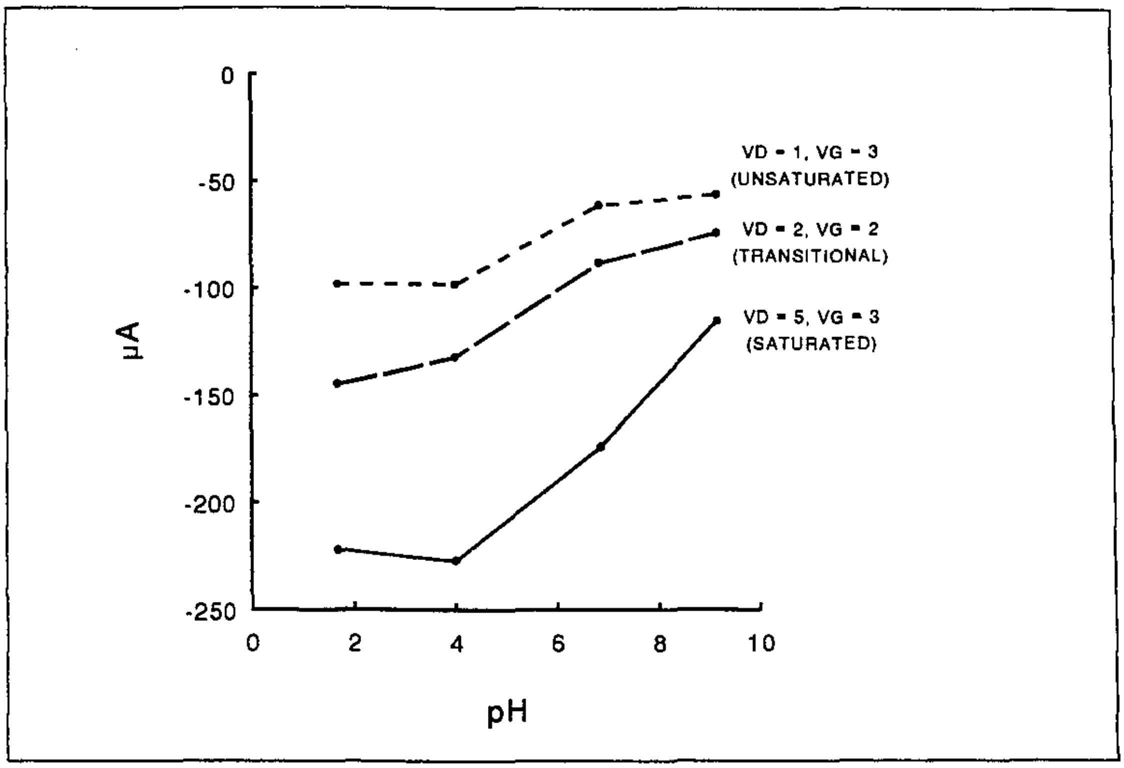

Figure 7-5. Difference in drain currents for ISFET/ReFET pair.
Plotted as $I_{D(ReFET)}-I_{D(ISFET)}$. Chip:Ch33.
Parameters set by test FET program.

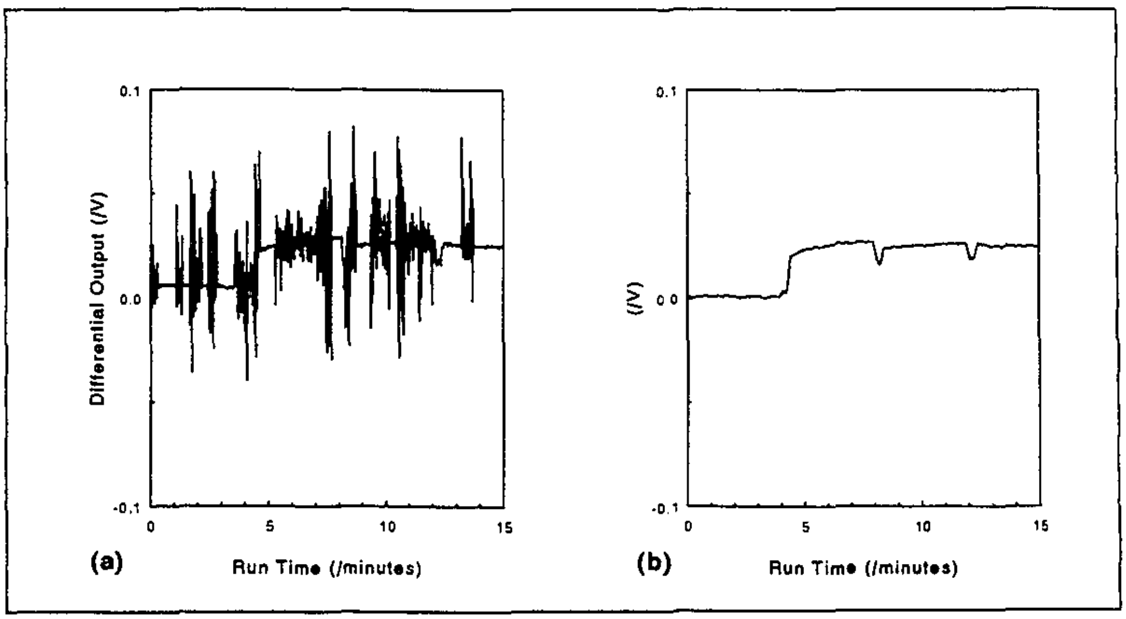

Figure 7-6. Effect of real-time averaging on differential sensing noise.
Chip: CH39; gain = 8; V_D = 3 V; V_G = 2 V; acquisition period = 1 s
(a) 1 A/D conversion sample per point (b) 50 A/D conversions per point
At time = 0 minutes: tetroxalate buffer (ph = 1.68)
At time = 4, 8, and 12 minutes: phosphate II buffer (pH = 7.41)

noise consisted of random pulses of high frequency noise. This was eliminated by increasing the number of samples per point to 50 (Figure 7-6(b)). Considering that the 50 samples were obtained in approximately 0.01 s, the frequency of the noise was apparently greater than 100 Hz. The peak-to-peak noise for the first four minutes in Figure 7-6(b) was measured as 2.1 mV, and, assuming a normal population, this corresponds a standard deviation of 0.42 mV. This indicates that the minimum detectable change in the signal is 1.26 mV (99.9% confidence, k=3), or roughly double the resolution of the A/D converter. A more precise A/D converter would be useful or, alternatively, the differential amplifier gain could be increased to boost the signal level.

The final preliminary test examined the effect of V_D and V_G on the pH response to three buffers (tetroxalate, phthalate, and phosphate II; pH=1.68, 4.01, and 7.41 respectively). It was found that operation in the saturation region was best in terms of linearity and sensitivity. The values chosen were: $V_D = V_G = 3$ V. These values are also in compliance with the battery voltage that would probably be used in a telemetry device.

7.5.2 Titration of Phosphoric Acid

The purpose of the phosphoric acid titrations was to monitor the progress of an acid-base titration with both a conventional pH glass electrode and the ISFET/ReFET differential sensor simultaneously. The differential output was then compared to the pH reading from the glass electrode, assumed to be true. The titration of H_3PO_4 was chosen because of the buffering action of the salts formed during the titration. This made it easy to obtain evenly spaced readings with regard to pH. The pH range of the titration (1.5 to $\geq$10) closely approximates the gastric pH range.

A set of representative data is shown in Figure 7-7. The titration curve in Figure 7-7(a) exhibits the classic shape. Of more interest is the comparison between the differential output and the true pH in Figure 7-7(b). The equation for this data set is: $y = 8.97x - 28.00$ (R=0.999) between pH 1.5 and 8. All of the titrations employing the SCE as the

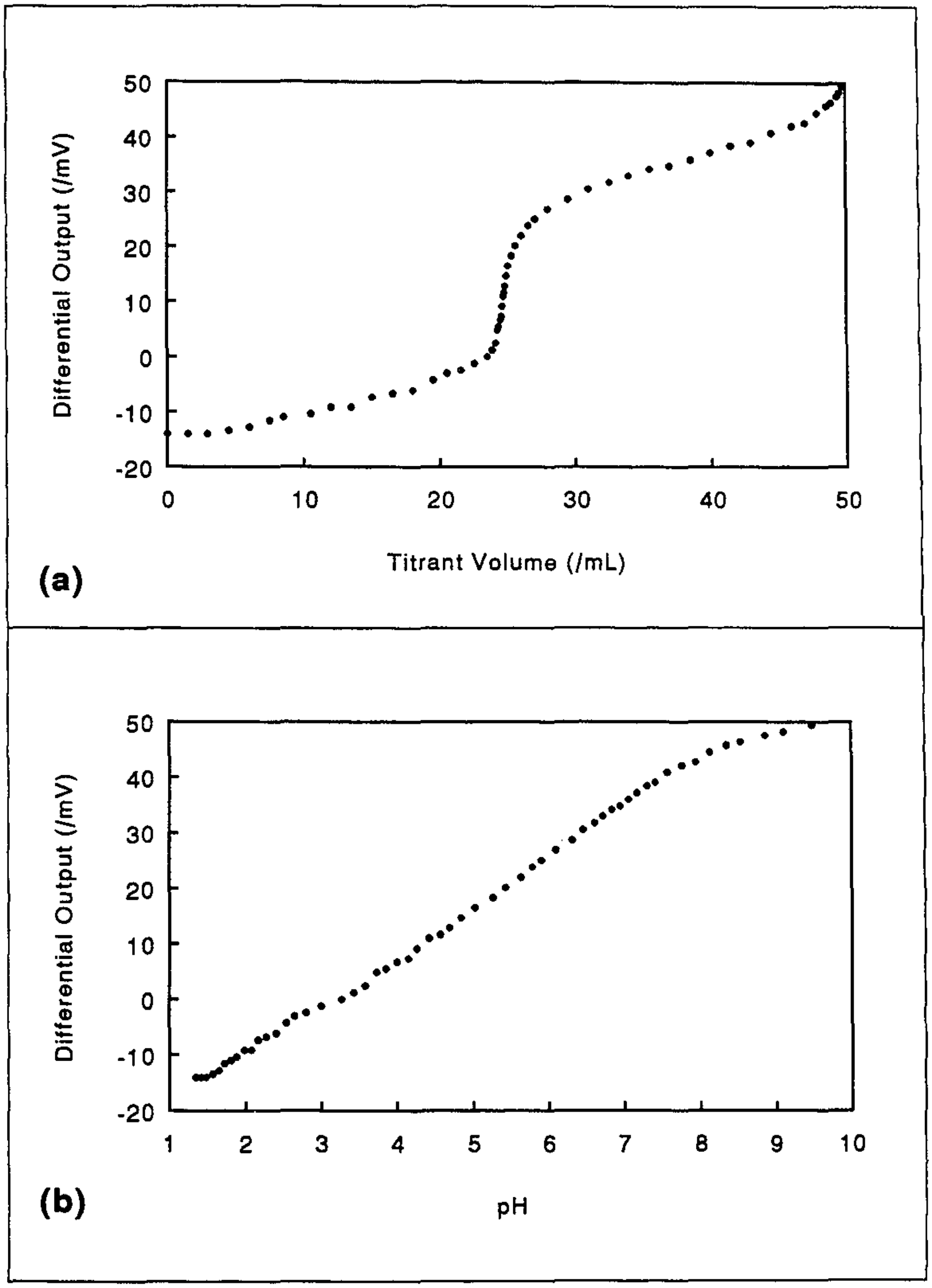

Figure 7-7. Titration of phosphoric acid with sodium hydroxide using differential sensing and an SCE reference.
Chip: CH38; gain = 8; V_D = V_G = 3 V; acquisition period = 1 s; samples/point = 50. Sample: 25 ml of 0.1 M H_3PO_4. Titrant: 0.1 M NaOH.

reference gave similar results.

The experiment was repeated with the Pt electrode replacing the SCE. The titration and calibration curves are given in Figure 7-8 for a sample titration. The data in Figure 7-8(b) fit the equation: $y = 8.25x - 13.02$ (R=0.998) between pH 1.5 and 8. While the curves in Figures 7-7 and 7-8 are similar, there are three distinct differences. In all cases, the use of a Pt pseudo-reference resulted in a shift in the differential output of approximately +10 mV. Secondly, the slope of the Pt calibration curve was usually 1 mV/decade less than the slope of the SCE curve. Finally, a loss of signal at high pH's (>7), as seen in Figure 7-8(b), was occasionally observed.

7.5.3 Performance Characteristics

The selectivity was not determined for the ISFET/ReFET pair. It can be assumed that the selectivity is high, with selectivity coefficients on the order of 10^{-7} for Na^+ and K^+ based on literature values [32].

With a minimum detectable change in signal of 1.26 mV, and a calibration curve slope of approximately 9 mV/decade, the sensitivity was 0.14 pH units. This is the minimum pH change that could be observed. The limit of detection can be considered to be the limit of the linear response range, pH=8 (see Section 1.3.1.3 for a discussion of the different LOD's).

The response time of the sensor varied with the method used and the pH level. When the device was placed in a new solution, the differential output would take 10-30 s to settle. In the titrations, where the sensor remained in solution, the output was steady within 2 s after addition of the titrant. These values are valid within the pH range 2-8. The response time increased at the pH extremes, particularly at low pH where it took up to 10-20 min to reach the final signal.

The average magnitude of the short term drift was 0.49 mV/minute, or 0.054 pH units per minute. Once again, the long term drift was deceivingly low due to reversals in the direction of the drift. This was exemplified by the calculated mean of the drift (-0.073 mV/minute, $n=10$, $\sigma=0.66$), which was much lower than the average magnitude.

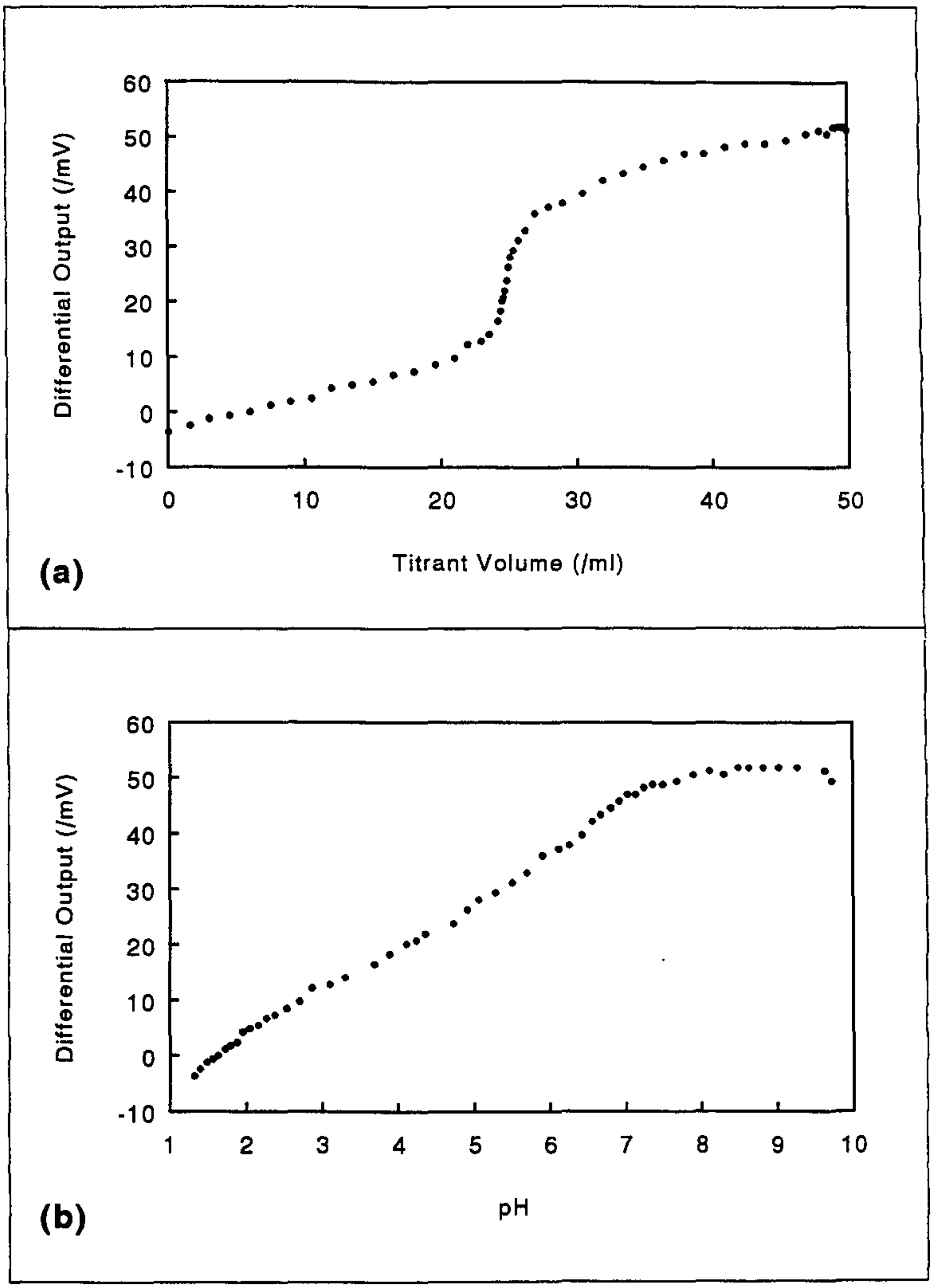

Figure 7-8. Titration of phosphoric acid with sodium hydroxide using differential sensing and a Pt pseudo-reference electrode. Chip: CH39; (all other parameters are the same as in Figure 7-7)

7.6 Discussion

Although the ISFET/ReFET differential sensor performed well, there are many questions yet to be answered concerning the utility of the alkanethiol monolayer and the possibility of using a pseudo-reference electrode. In order to understand the action of the ReFET, the differential output must be analyzed in terms of what is happening at the surfaces of the FETs. This can be achieved with the equation for the operation of an ISFET in the saturated region (see Section 2.4.2):

$$I_D = K \left(V_G - V_T^* - \varphi_o \right)^2 \tag{7.1}$$

where K contains all the physical constants associated with the device. The value of K was calculated using rough estimates for the other variables ($V_D = V_G = 3V$, $V_T^* = 0V$, $I_D = 1mA$), based on experimental data, giving $K = 1.6 \times 10^{-4}$. The slope of the differential output versus pH calibration curve, approximately 10 mV/decade, corresponds to a change in difference current of 10 μA/decade. This value, along with the K and Equation 7.1, was used to determine the change in φ_o per decade change in pH. This was found to be approximately 15 mV, well below both the ideal value of 59 mV dictated by the Nernst equation and the experimental value of around 50 mV reported for Si_3N_4 pH ISFETS [32]. This indicates that the alkanethiol monolayer is not acting as an ideal blocking layer.

This conclusion is also substantiated by the observation of the shift in the differential response when the Pt pseudo-reference electrode replaced the SCE reference. This demonstrated that changes in the gate bias, or reference, voltage were not completely eliminated by common-mode rejection. Therefore the Pt electrode could only be used in situations where the requirements concerning accuracy and precision are not stringent.

The question remains as to whether the non-ideal behaviour of the alkanethiol monolayer ReFET was due to flaws in the device design and construction, or to more serious concerns. Because of the limited number of devices constructed for this work (5) and the difficulties involved in testing of the monolayer *in situ*, it is probable that the results presented in this chapter can be improved upon. It would be particularly advantageous to be able to test the monolayer for defects before and after use. Another direction of research would be to replace the inorganic gate ISFET with a chemically selective alkanethiol monolayer ISFET. The alkanethiol coated FET could also find use in immunological sensors, where the lack of an ideal blocking layer continues to be an impediment.

Differential sensing was approached as a method to improve upon the telemetry device of Chapter 6. Is this possible? The answer is a qualified yes. In itself, differential sensing does improve on the performance of the sensor as a whole. The drift of the differential sensor was almost an order of magnitude better than that of the prototype telemetry device. Problems remain with the reference electrode and biocompatibility, neither of which is trivial. The alkanethiol monolayer ReFET produced some interesting and useful results, and with some improvements it could be incorporated successfully into a telemetry device.

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Contributions to Original Knowledge

1. A procedure for the construction of a glass ceramic die was developed. Two versions of the die were constructed. These dies were used for the mounting of ISFET chips.

2. A microcomputer controlled instrument (the FET Controller), which controlled an array of 1 to 12 ISFETs, was designed and constructed.

3. A software program was written to control and acquire data from 1 to 12 ISFETs using the FET Controller. The program used background multitasking to acquire data which was displayed, processed, and saved in the foreground in real time.

4. A software program was written to automatically test and characterize single FETs at varying drain and gate potentials.

5. A telemetric pH sensor was designed and built incorporating a pH ISFET. The suitability of ISFETs for use as the chemical recognition and transduction elements in a telemetric sensor was established. To the best of our knowledge, this represents the first use of an ISFET in a telemetry device. This opens up new avenues in both the ISFET and telemetry research fields.

6. The use of an ISFET as a pH dependant, variable linear resistor was described.

7. The pH telemetry device was tested in aqueous buffers. Signal drift was observed to be significant. The major problem with this device was determined to be the low ratio of analytical signal to the background signal.

8. A self-assembled alkanethiol monolayer on gold was investigated as a blocking, or ion-insentive, layer on an ISFET. This was successfully used as a ReFET in conjunction with an pH ISFET and an SCE as the reference electrode. This was the first use of such a layer in a potentiometric device.

9. The ISFET/ReFET pair was also used with a platinum pseudo-reference electrode with mixed results.

10. Differential sensing was confirmed to be a superior operational mode than the simpler design using the ISFET as a variable resistor.

APPENDIX A

ASYST PROGRAMS FOR FET CONTROLLER

This appendix contains the 4 programs which were discussed in Chapter 4, and used in Chapters 4 and 7. Table A.1 gives the details necessary to run each program. The program listings follow Table A.2.

The two ASYST versions were configured differently in terms of memory allocation and permanently loaded overlays. The configuration specifics for JMASYST2 and ANALYZE are given in Table A.2.

The individual modules that must be loaded for each program are listed in the 3rd column of Table A.1, and this was done by the first executable word for each program (*i.e.* LOAD.MULTIPLE.FETS). These "LOAD" words were saved as part of the applicable ASYST version. The other executable words listed are the highest level words, and are meant to be entered interactively. Lower level words can also be run interactively, but this is not advised.

APPENDIX A

Table A.1
FET Controller Program Details

Program	ASYST Version	Load, in order	Executable Words
Multiple ISFET Acquisition	JMASYST2	DECLARE WORDS0 WORDS1 WORDS2 WORDS3 WORDS4 WORDS5 PROGRAM1	LOAD.MULTIPLE.FETS TURN.ON SETUP.FETS RUN.FETS SAVE.MULTIPLE.DATA
Multiple ISFET Analysis	ANALYZE	DECLARE3 WORDS5 WORDS8	LOAD.MULT.ANALYZE RETRIEVE.MULTIPLE.DATA fet#.token PLOT fet#.token HARD.PLOT
Test FET Acquisition	JMASYST2	DECLARE2 WORDS0 WORDS6 WORDS7	LOAD.TEST.FET TURN.ON TEST.FET SAVE.TEST.DATA PRINT.TEST.RESULTS
Test FET Analysis	ANALYZE	DECLARE4 WORDS7 WORDS10	LOAD.TEST.ANALYZE RETRIEVE.TEST.DATA GO

Table A.2
ASYST Configuration
Status upon entry; minimized system

Version:	JMASYST2	ANALYZE
	Total bytes (% used)	
Base Dictionary	65536 (83)	65536 (81)
Code Segment	80400 (79)	78736 (74)
System Dictionary	134432 (84)	123392 (82)
Symbol Table	20480 (70)	20480 (67)
String Segment	6144 (56)	6144 (71)
DAS Buffer Segment	1024	0
GPIB Queue Segment	0	0
User Dictionary	30720 (01)	20480 (01)
Token Heap	34816	32768
Unnamed Array Heap	81920	163840
Array Storage Area	102016 (10)	46096 (47)
Keyboard Buffer	200	
System Buffer	65520 (in expanded memory)	
Expanded Memory		
Total Pages	24	
Total Bytes	393216	
Unallocated Pages	20	
Unallocated Bytes	327680	
Permanently Loaded System Overlays	Data Files Array Editor HP Plotter Driver	
	Editor Help System Data Acquisition Master Data Translation 2800	Software Scroller Statistics & Special Functions Waveform Operations

APPENDIX A

Multiple ISFET data acquisition program
File: Declare Page 1 of 3

```
DT2800                                      \ Data Translation board DT2801

0 0 D/A.TEMPLATE DAC0                       \ DAC0 and DAC1 to be used for reference
1 1 D/A.TEMPLATE DAC1                       \ electrode control

4 DIGITAL.TEMPLATE PORT0                    \ Digital templates will be used for con-
5 DIGITAL.TEMPLATE PORT1                    \ trol of FET Controller operatation.
6 DIGITAL.TEMPLATE WORD
0 12 A/D.TEMPLATE TASK.CHNLS                \ Used for multiple FET data acquisition

0.1 CONVERSION.DELAY                        \ DT2801 parameter ( 0.073 to 163.8 mS)

DAS.INIT

INTEGER DIM[ 36 ] ARRAY VZDS.COUNT          \ Integer value output to FET Control-
    128 VZDS.COUNT :=                       \ ler ( 0 to 255)

REAL DIM[ 36 ] ARRAY VZDS.INPUT             \ User input
    0 VZDS.INPUT :=
  DIM[ 36 ] ARRAY VZDS.VALUE                \ Actual voltage applied by FET Con-
    0 VZDS.VALUE :=                         \ troller

  DIM[ 36 ] ARRAY VZDS.MINIMUM              \ Experimentally determined parameters
  DIM[ 36 ] ARRAY VZDS.STEP

FILE.OPEN \JEFF\DATA\DACCONST.DAT                \ Fill minimum and step arrays with
3 SUBFILE VZDS.MINIMUM FILE>ARRAY                \ with default values for VZ = 10
4 SUBFILE VZDS.STEP FILE>ARRAY                   \ and VD/VS = 5
FILE.CLOSE

INTEGER SCALAR NUM          0 NUM :=                      \ Multipurpose input variable
    SCALAR GAIN          1 GAIN :=                        \ Hardware A/D gain
    SCALAR VZ.RANGE         10 VZ.RANGE :=                \ Zeroing voltage range
    SCALAR VD/VS.RANGE      5 VD/VS.RANGE :=              \ Drain and source volt-
                                                          \ age range ( 5 or 10)
    SCALAR MAX.BUFF.SIZE  16384  MAX.BUFF.SIZE :=         \ Should be a power of 2
    SCALAR NUM.FETS         12  NUM.FETS :=               \ Number of FETs
    SCALAR SAM.POINT         8  SAM.POINT :=              \ Number of samples ave-
                                                          \ raged per point
    SCALAR POINTS          85  POINTS :=                  \ Number of points taken
                                                          \ per FET per buffer
                                                          \ cycle
    SCALAR CYCLE#           0  CYCLE# :=                  \ Count of number of
                                                          \ data buffer cycles
    SCALAR INDEX           0  INDEX :=                    \ Used to access pro-
                                                          \ per FET#.TOKEN
                                                          \ position

REAL    SCALAR CONV.FACT        0  CONV.FACT :=           \ General use conversion
                                                          \ factor
    SCALAR REF0           0  REF0 :=                      \ Reference 0 voltage
    SCALAR REF1           0  REF1 :=                      \ Reference 1 voltage
    SCALAR ACQ.PERIOD        1  ACQ.PERIOD :=             \ Acquisition period in
                                                          \ seconds (0.2 to 60)
    SCALAR TASK.PERIOD.MS    50  TASK.PERIOD.MS :=        \ Period in mS between
                                                          \ task interrupts ( <55)

1 17 1 63 WINDOW {ARRAY.HORIZ.LABEL}             \ Custom array editor windows
1 14 14 15 WINDOW {ARRAY.VERTI.LABEL}
20 1 23 78 WINDOW {GRAPHICS.PROMPT}              \ Multiple FET acquisition text
                                                 \ window
186 205 201 200 188 187 BORDER.CHARS        \ Text window border

TOKEN BUFFER.A                              \ Cyclic double buffer tokens in real memory for
TOKEN BUFFER.B                              \ multiple FET acquisition.
TOKEN PRESENT.ARRAY                         \ Token to be EQUIV> to either BUFFER.A or .B
```

```
TOKEN TEMP    EXP.MEM > TEMP              \ Token for massaged but unsorted data

\ The following FET#.TOKENS will each occupy 16K page in expanded memory.
\ With default MAX.BUFF.SIZE = 16K these tokens will hold 8K of data points.
TOKEN FET1.TOKEN  EXP.MEM > FET1.TOKEN
  INTEGER DIM[ MAX.BUFF.SIZE 2 / ] UNNAMED.ARRAY BECOMES > FET1.TOKEN
  0 FET1.TOKEN :=
TOKEN FET2.TOKEN  EXP.MEM > FET2.TOKEN
  INTEGER DIM[ MAX.BUFF.SIZE 2 / ] UNNAMED.ARRAY BECOMES > FET2.TOKEN
  0 FET2.TOKEN :=
TOKEN FET3.TOKEN  EXP.MEM > FET3.TOKEN
  INTEGER DIM[ MAX.BUFF.SIZE 2 / ] UNNAMED.ARRAY BECOMES > FET3.TOKEN
  0 FET3.TOKEN :=
TOKEN FET4.TOKEN  EXP.MEM > FET4.TOKEN
  INTEGER DIM[ MAX.BUFF.SIZE 2 / ] UNNAMED.ARRAY BECOMES > FET4.TOKEN
  0 FET4.TOKEN :=
TOKEN FET5.TOKEN  EXP.MEM > FET5.TOKEN
  INTEGER DIM[ MAX.BUFF.SIZE 2 / ] UNNAMED.ARRAY BECOMES > FET5.TOKEN
  0 FET5.TOKEN :=
TOKEN FET6.TOKEN  EXP.MEM > FET6.TOKEN
  INTEGER DIM[ MAX.BUFF.SIZE 2 / ] UNNAMED.ARRAY BECOMES > FET6.TOKEN
  0 FET6.TOKEN :=
TOKEN FET7.TOKEN  EXP.MEM > FET7.TOKEN
  INTEGER DIM[ MAX.BUFF.SIZE 2 / ] UNNAMED.ARRAY BECOMES > FET7.TOKEN
  0 FET7.TOKEN :=
TOKEN FET8.TOKEN  EXP.MEM > FET8.TOKEN
  INTEGER DIM[ MAX.BUFF.SIZE 2 / ] UNNAMED.ARRAY BECOMES > FET8.TOKEN
  0 FET8.TOKEN :=
TOKEN FET9.TOKEN  EXP.MEM > FET9.TOKEN
  INTEGER DIM[ MAX.BUFF.SIZE 2 / ] UNNAMED.ARRAY BECOMES > FET9.TOKEN
  0 FET9.TOKEN :=
TOKEN FET10.TOKEN  EXP.MEM > FET10.TOKEN
  INTEGER DIM[ MAX.BUFF.SIZE 2 / ] UNNAMED.ARRAY BECOMES > FET10.TOKEN
  0 FET10.TOKEN :=
TOKEN FET11.TOKEN  EXP.MEM > FET11.TOKEN
  INTEGER DIM[ MAX.BUFF.SIZE 2 / ] UNNAMED.ARRAY BECOMES > FET11.TOKEN
  0 FET11.TOKEN :=
TOKEN FET12.TOKEN  EXP.MEM > FET12.TOKEN
  INTEGER DIM[ MAX.BUFF.SIZE 2 / ] UNNAMED.ARRAY BECOMES > FET12.TOKEN
  0 FET12.TOKEN :=

TOKEN PLOT.ARRAY                 \ Token to be EQUIV > to data to be plotted
VUPORT VUPORT.MAIN               \ Single graphics window to be used with the
0.0 0.25 VUPORT.ORIG             \ {GRAPHICS.PROMPT} text window
1.0 0.75 VUPORT.SIZE

VUPORT VUPORT.FET1               \ Multiple FET acquisition graphics windows
0.0 0.75 VUPORT.ORIG
0.24 0.25 VUPORT.SIZE
VUPORT VUPORT.FET2
0.25 0.75 VUPORT.ORIG
0.24 0.25 VUPORT.SIZE
VUPORT VUPORT.FET3
0.5 0.75 VUPORT.ORIG
0.24 0.25 VUPORT.SIZE
VUPORT VUPORT.FET4
0.76 0.75 VUPORT.ORIG
0.24 0.25 VUPORT.SIZE
VUPORT VUPORT.FET5
0.0 0.5 VUPORT.ORIG
0.24 0.25 VUPORT.SIZE
VUPORT VUPORT.FET6
0.25 0.5 VUPORT.ORIG
0.24 0.25 VUPORT.SIZE
VUPORT VUPORT.FET7
0.5 0.5 VUPORT.ORIG
0.24 0.25 VUPORT.SIZE
```

```
VUPORT VUPORT.FET8
0.76 0.5 VUPORT.ORIG
0.24 0.25 VUPORT.SIZE
VUPORT VUPORT.FET9
0.0 0.25 VUPORT.ORIG
0.24 0.25 VUPORT.SIZE
VUPORT VUPORT.FET10
0.25 0.25 VUPORT.ORIG
0.24 0.25 VUPORT.SIZE
VUPORT VUPORT.FET11
0.5 0.25 VUPORT.ORIG
0.24 0.25 VUPORT.SIZE
VUPORT VUPORT.FET12
0.76 0.25 VUPORT.ORIG
0.24 0.25 VUPORT.SIZE

DEF.VUPORT                              \ Reset to default vuport.

REAL DIM[ 1024 ] ARRAY PIX.BUFF.1       \ Graphic pixel buffers for fast
REAL DIM[ 512 ] ARRAY PIX.BUFF.2        \ erasing of data in multiple FET
REAL DIM[ 352 ] ARRAY PIX.BUFF.3        \ acquisition mode
REAL DIM[ 256 ] ARRAY PIX.BUFF.4
REAL DIM[ 256 ] ARRAY PIX.BUFF.5        \ Higher dimensions for 1, 2 and 3 are
REAL DIM[ 256 ] ARRAY PIX.BUFF.6        \ to accomodate larger array sizes when
REAL DIM[ 256 ] ARRAY PIX.BUFF.7        \ fewer FETs used
REAL DIM[ 256 ] ARRAY PIX.BUFF.8
REAL DIM[ 256 ] ARRAY PIX.BUFF.9
REAL DIM[ 256 ] ARRAY PIX.BUFF.10
REAL DIM[ 256 ] ARRAY PIX.BUFF.11
REAL DIM[ 256 ] ARRAY PIX.BUFF.12
```

APPENDIX A

Multiple ISFET data acquisition program
File: Words0 Page 1 of 5

```
\ Basic words for triggering and zeroing of FET Controller DACs.
\ (needs DECLARE or DECLARE2)

BINARY
: LATCH.V00                        \ [ - ]
  PORT1                            \ These latches trigger the individual
  11, DIGITAL.MASK                 \ DAC's to reset. PORT0 should contain
  00, WRITE.BITS                   \ the proper count and be initialized
  111100, DIGITAL.MASK
  PULSE.BITS
;
: LATCH.V01
  PORT1
  11, DIGITAL.MASK
  01, WRITE.BITS
  111100, DIGITAL.MASK
  PULSE.BITS
;
: LATCH.V02
  PORT1
  11, DIGITAL.MASK
  10, WRITE.BITS
  111100, DIGITAL.MASK
  PULSE.BITS
;
: LATCH.V03
  PORT1
  11, DIGITAL.MASK
  11, WRITE.BITS
  111100, DIGITAL.MASK
  PULSE.BITS
;
: LATCH.V10
  PORT1
  11, DIGITAL.MASK
  00, WRITE.BITS
  111000, DIGITAL.MASK
  PULSE.BITS
;
: LATCH.V11
  PORT1
  11, DIGITAL.MASK
  01, WRITE.BITS
  111000, DIGITAL.MASK
  PULSE.BITS
;
: LATCH.V12
  PORT1
  11, DIGITAL.MASK
  10, WRITE.BITS
  111000, DIGITAL.MASK
  PULSE.BITS
;
: LATCH.V13
  PORT1
  11, DIGITAL.MASK
  11, WRITE.BITS
  111000, DIGITAL.MASK
  PULSE.BITS
;
: LATCH.V20
  PORT1
  11, DIGITAL.MASK
  00, WRITE.BITS
  110100, DIGITAL.MASK
  PULSE.BITS
;
```

```
: LATCH.V21
  PORT1
  11, DIGITAL.MASK
  01, WRITE.BITS
  110100, DIGITAL.MASK
  PULSE.BITS
;
: LATCH.V22
  PORT1
  11, DIGITAL.MASK
  10, WRITE.BITS
  110100, DIGITAL.MASK
  PULSE.BITS
;
: LATCH.V23
  PORT1
  11, DIGITAL.MASK
  11, WRITE.BITS
  110100, DIGITAL.MASK
  PULSE.BITS
;
: LATCH.V30
  PORT1
  11, DIGITAL.MASK
  00, WRITE.BITS
  110000, DIGITAL.MASK
  PULSE.BITS
;
: LATCH.V31
  PORT1
  11, DIGITAL.MASK
  01, WRITE.BITS
  110000, DIGITAL.MASK
  PULSE.BITS
;
: LATCH.V32
  PORT1
  11, DIGITAL.MASK
  10, WRITE.BITS
  110000, DIGITAL.MASK
  PULSE.BITS
;
: LATCH.V33
  PORT1
  11, DIGITAL.MASK
  11, WRITE.BITS
  110000, DIGITAL.MASK
  PULSE.BITS
;
: LATCH.V40
  PORT1
  11, DIGITAL.MASK
  00, WRITE.BITS
  101100, DIGITAL.MASK
  PULSE.BITS
;
: LATCH.V41
  PORT1
  11, DIGITAL.MASK
  01, WRITE.BITS
  101100, DIGITAL.MASK
  PULSE.BITS
;
: LATCH.V42
  PORT1
  11, DIGITAL.MASK
  10, WRITE.BITS
```

```
 101100, DIGITAL.MASK
 PULSE.BITS
;
: LATCH.V43
 PORT1
 11, DIGITAL.MASK
 11, WRITE.BITS
 101100, DIGITAL.MASK
 PULSE.BITS
;
: LATCH.V50
 PORT1
 11, DIGITAL.MASK
 00, WRITE.BITS
 101000, DIGITAL.MASK
 PULSE.BITS
;
: LATCH.V51
 PORT1
 11, DIGITAL.MASK
 01, WRITE.BITS
 101000, DIGITAL.MASK
 PULSE.BITS
;
: LATCH.V52
 PORT1
 11, DIGITAL.MASK
 10, WRITE.BITS
 101000, DIGITAL.MASK
 PULSE.BITS
;
: LATCH.V53
 PORT1
 11, DIGITAL.MASK
 11, WRITE.BITS
 101000, DIGITAL.MASK
 PULSE.BITS
;
: LATCH.V60
 PORT1
 11, DIGITAL.MASK
 00, WRITE.BITS
 100100, DIGITAL.MASK
 PULSE.BITS
;
: LATCH.V61
 PORT1
 11, DIGITAL.MASK
 01, WRITE.BITS
 100100, DIGITAL.MASK
 PULSE.BITS
;
: LATCH.V62
 PORT1
 11, DIGITAL.MASK
 10, WRITE.BITS
 100100, DIGITAL.MASK
 PULSE.BITS
;
: LATCH.V63
 PORT1
 11, DIGITAL.MASK
 11, WRITE.BITS
 100100, DIGITAL.MASK
 PULSE.BITS
;
: LATCH.V70
```

```
  PORT1
  11, DIGITAL.MASK
  00, WRITE.BITS
  100000, DIGITAL.MASK
  PULSE.BITS
;
: LATCH.V71
  PORT1
  11, DIGITAL.MASK
  01, WRITE.BITS
  100000, DIGITAL.MASK
  PULSE.BITS
;
: LATCH.V72
  PORT1
  11, DIGITAL.MASK
  10, WRITE.BITS
  100000, DIGITAL.MASK
  PULSE.BITS
;
: LATCH.V73
  PORT1
  11, DIGITAL.MASK
  11, WRITE.BITS
  100000, DIGITAL.MASK
  PULSE.BITS
;
: LATCH.V80
  PORT1
  11, DIGITAL.MASK
  00, WRITE.BITS
  011100, DIGITAL.MASK
  PULSE.BITS
;
: LATCH.V81
  PORT1
  11, DIGITAL.MASK
  01, WRITE.BITS
  011100, DIGITAL.MASK
  PULSE.BITS
;
: LATCH.V82
  PORT1
  11, DIGITAL.MASK
  10, WRITE.BITS
  011100, DIGITAL.MASK
  PULSE.BITS
;
: LATCH.V83
  PORT1
  11, DIGITAL.MASK
  11, WRITE.BITS
  011100, DIGITAL.MASK

  PULSE.BITS
;
DECIMAL

: ZERO.DACS                         \ [ - ]
  PORT0                             \ Outputs 128 to all DAC's
  128 DIGITAL.OUT                   \ Sets bipolar DAC's to 0 V
  LATCH.V00
  LATCH.V01
  LATCH.V02
  LATCH.V03
  LATCH.V10
  LATCH.V11
```

```
    LATCH.V12
    LATCH.V13
    LATCH.V20
    LATCH.V21
    LATCH.V22
    LATCH.V23
    LATCH.V30
    LATCH.V31
    LATCH.V32
    LATCH.V33
    LATCH.V40
    LATCH.V41
    LATCH.V42
    LATCH.V43
    LATCH.V50
    LATCH.V51
    LATCH.V52
    LATCH.V53
    LATCH.V60
    LATCH.V61
    LATCH.V62
    LATCH.V63
    LATCH.V70
    LATCH.V71
    LATCH.V72
    LATCH.V73
    LATCH.V80
    LATCH.V81
    LATCH.V82
    LATCH.V83
  ;
```

APPENDIX A

Multiple ISFET data acquisition program
File: Words1 Page 1 of 2

```
\ Basic words for control of FET Controller DACs and reference voltages.
\ (needs DECLARE)

: SET.REF0                              \ [ - ]  Sets DAC0 to REF0 voltage.
  DAC0 D/A.INIT
  REF0
  0 5 D/A.SCALE D/A.OUT
  CR ." REF0 =" REF0 . ." V" CR
;

: SET.REF1                              \ [ - ]  Sets DAC1 to REF1 voltage.
  DAC1 D/A.INIT
  REF1
  -10 10 D/A.SCALE D/A.OUT
  CR ." REF1 =" REF1 . ." V" CR
;

: INPUT.REF0
  CR ." Enter REF0 value { 0 } : "
  BEGIN #INPUT
    IF REF0 :=
      REF0 0 <
      REF0 4.9993 > OR
    ELSE FALSE
    THEN
  WHILE CR ." REF0 value must be within 0 and +4.9993."
     CR ." Try again : "
     0 REF0 :=
  REPEAT
;

: INPUT.REF1
  CR ." Enter REF1 value { 0 } : "
  BEGIN #INPUT
    IF REF1 :=
      REF1 -10 <
      REF1 9.9975 > OR
    ELSE FALSE
    THEN
  WHILE CR ." REF1 value must be within -10 and +9.9975."
     CR ." Try again : "
     0 REF1 :=
  REPEAT
;

: SET.REFERENCES
  INPUT.REF0
  SET.REF0
  INPUT.REF1
  SET.REF1
;

: INITIAL.SET                           \ Set DT2801 outputs such that FET Controller can
  DAS.INIT                              \  be turned on safely.
  WORD                                  \ Outputs 1111 1111 1000 0000
  65408 DIGITAL.OUT
  0 REF0 :=
  SET.REF0
  0 REF1 :=
  SET.REF1
;

: INPUT.DAC.RANGES                      \ [ - ]
  CR CR ." VZ ranges: -5 to +5"         \ Prompts user to input values for
  CR ."        -10 to +10"              \  VZ and VD/VS ranges. Checks for
  CR ." {Default value = 10 V}"         \  acceptable input of either 5 or
  CR ." Enter new range (5 or 10) : "   \  10 then sets appropiate
```

```
 BEGIN #INPUT                                   \ variable when correct.
    IF VZ.RANGE : =
      VZ.RANGE 5 < >
      VZ.RANGE 10 < > AND
    ELSE FALSE
    THEN
 WHILE CR ." Invalid entry. Try again : "
    10 VZ.RANGE : =
 REPEAT

 CR CR ." VD/VS ranges: -5 to +5"
 CR ."        -10 to +10"
 CR ." {Default value = 5 V}"
 CR ." Enter new range (5 or 10) : "
 BEGIN #INPUT
    IF VD/VS.RANGE : =
      VD/VS.RANGE 5 < >
      VD/VS.RANGE 10 < > AND
    ELSE FALSE
    THEN
 WHILE CR ." Invalid entry. Try again : "
    5 VD/VS.RANGE : =
 REPEAT
;

: SET.DAC.RANGES                                \ [ - ] Outputs digital code to FET Control-
  DAS.INIT                                      \ ler to set hardware for VZ and VD/VS ranges.
  INPUT.DAC.RANGES
  PORT1
  64, DIGITAL.MASK                              \ Set the bit for VZ.RANGE = 10
  VZ.RANGE 10 =                                 \ or clear bit for VZ.RANGE = 5
  IF  1, WRITE.BITS
  ELSE 0, WRITE.BITS
  THEN
  128, DIGITAL.MASK                             \ Set the bit for VD/VS.RANGE = 5
  VD/VS.RANGE 10 =                              \ or clear bit for VD/VS.RANGE = 10
  IF  0, WRITE.BITS
  ELSE 1, WRITE.BITS
  THEN

  FILE.OPEN \JEFF\DATA\DACCONST.DAT
  VZ.RANGE 2 * VD/VS.RANGE +                    \ Calculation to determine which set
  CASE                                          \ of minimum and step values to load
    25 OF 3 SUBFILE VZDS.MINIMUM FILE>ARRAY     \ VZ = 10; VD/VS = 5
       4 SUBFILE VZDS.STEP FILE>ARRAY
     ENDOF
    30 OF 1 SUBFILE VZDS.MINIMUM FILE>ARRAY     \ VZ = 10; VD/VS = 10
       2 SUBFILE VZDS.STEP FILE>ARRAY
     ENDOF
    20 OF 5 SUBFILE VZDS.MINIMUM FILE>ARRAY     \ VZ = 5; VD/VS = 10
       6 SUBFILE VZDS.STEP FILE>ARRAY
     ENDOF
    15 OF 7 SUBFILE VZDS.MINIMUM FILE>ARRAY     \ VZ = 5; VD/VS = 5
       8 SUBFILE VZDS.STEP FILE>ARRAY
     ENDOF
  ENDCASE
  FILE.CLOSE
;
```

APPENDIX A

Multiple ISFET data acquisition program


```
\ Entering and setting of all FET Controller DAC voltages.
\ (needs DECLARE, WORDS1)

: SET.ARRAY.EDIT.WINDOWS                    \ [ - ] Set custom array editor
  -1 3 FIX.FORMAT                           \ windows for voltage inputs.
  {[]DATA}   3 18 15 63 SET.WINDOW
  {[]PROMPT} 18 9 23 70 SET.WINDOW
  {[]STATUS} 16 1 16 0 SET.WINDOW
  {[]INSERT} 16 15 16 79 SET.WINDOW
;

: ARRAY.EDIT.LABELS                         \ [ - ] Custom array editor labels
  {ARRAY.HORIZ.LABEL}
  SCREEN.CLEAR
   ."        VZ        VD        VS"
  {ARRAY.VERTI.LABEL}
  SCREEN.CLEAR
    ." "
    ." "
    ." 0"
    ." 1"
    ." 2"
    ." 3"
    ." 4"
    ." 5"
    ." 6"
    ." 7"
    ." 8"
    ." 9"
    ." 10"
    ." 11"
;

: INPUT.VOLTAGES                            \ [ - ] Prompt user to input VZ, VS, and
  INSTALL NOP IN PROMPT.XEQ                 \ VD voltages.
  SET.ARRAY.EDIT.WINDOWS
  VZDS.INPUT DIM[ 3 , 12 ] RESHAPE TRANS[ 1 , 2 ]    \ Reshapes 1 dimensional
  ARRAY.EDIT(MANUAL)                                 \ array to 2 D.
  ARRAY.EDIT.LABELS
  {[]PROMPT}
   {BORDER}
    CR ." Input or change voltage values."
  BEGIN
    CR ." Present ranges are: VD/VS = " 241 ASCII" "TYPE VD/VS.RANGE .
     ."   VZ.RANGE = " 241 ASCII" "TYPE VZ.RANGE .
    CR ." Note: VS0 cannot be set < 0 ; VS3 cannot be set < -4.99"
    CR ."      VD1, VD2, and VS9 cannot be set > 4.99"
    CR ." Hit ESC when finished"
    INSERT                                          \ Allow for user input.
    TABLE TRANS[ 1 , 2 ] DIM[ 36 ] RESHAPE          \ Reshapes 2 D array to 1 D
    VZDS.INPUT :=
    VZDS.INPUT VZDS.MINIMUM - VZDS.STEP /           \ Calculate digital output
    VZDS.COUNT :=                                   \ value (count)
    VZDS.COUNT 255 [>] []SUM                        \ Check digital values for
    VZDS.COUNT  0 [<] []SUM +                       \ proper range.
    0 >
    VZDS.COUNT [ 25 ] 128 < OR
  WHILE
    CR ." INPUT VOLTAGES(S) OUT OF RANGE. PLEASE CORRECT."
    CR
  REPEAT
  DEFAULT.PROMPT
;

: PRINT.VOLTAGES                                    \ [ - ] Display true applied volt-
  VZDS.COUNT VZDS.STEP * VZDS.MINIMUM +             \ ages and print out if desired.
  VZDS.VALUE :=                                     \ True values are calculated based
```

```
  INSTALL NOP IN PROMPT.XEQ                          \ on experimental parameters.
  SET.ARRAY.EDIT.WINDOWS
  VZDS.VALUE DIM[ 3 , 12 ] RESHAPE TRANS[ 1 , 2 ]
  ARRAY.EDIT(MANUAL)
  ARRAY.EDIT.LABELS
  {[]PROMPT}
   {BORDER}
   ."   ".DATE ."   " .TIME
   CR ." These are the actual voltages output from the control board."
   CR ." Use SHIFT-PRTSC to print, any other key to continue
  PCKEY DROP ?DROP
  DEFAULT.PROMPT
;

: SET.DACS                             \ [ - ] Set all FET Controller DACs
  1 36 DO                              \ Loop places all digital values on
   VZDS.COUNT [ I ]                    \ stack
  -1 +LOOP

  PORT0 DIGITAL.OUT LATCH.V00          \ Each DIGITAL.OUT command removes the
  PORT0 DIGITAL.OUT LATCH.V01          \ proper value from the stack and
  PORT0 DIGITAL.OUT LATCH.V02          \ presents it to the FET Controller.
  PORT0 DIGITAL.OUT LATCH.V03          \ The LATCH.V## triggers the appropiate
  PORT0 DIGITAL.OUT LATCH.V10          \ DAC.
  PORT0 DIGITAL.OUT LATCH.V11
  PORT0 DIGITAL.OUT LATCH.V12
  PORT0 DIGITAL.OUT LATCH.V13
  PORT0 DIGITAL.OUT LATCH.V20
  PORT0 DIGITAL.OUT LATCH.V21
  PORT0 DIGITAL.OUT LATCH.V22
  PORT0 DIGITAL.OUT LATCH.V23
  PORT0 DIGITAL.OUT LATCH.V30
  PORT0 DIGITAL.OUT LATCH.V31
  PORT0 DIGITAL.OUT LATCH.V32
  PORT0 DIGITAL.OUT LATCH.V33
  PORT0 DIGITAL.OUT LATCH.V40
  PORT0 DIGITAL.OUT LATCH.V41
  PORT0 DIGITAL.OUT LATCH.V42
  PORT0 DIGITAL.OUT LATCH.V43
  PORT0 DIGITAL.OUT LATCH.V50
  PORT0 DIGITAL.OUT LATCH.V51
  PORT0 DIGITAL.OUT LATCH.V52
  PORT0 DIGITAL.OUT LATCH.V53
  PORT0 DIGITAL.OUT LATCH.V60
  PORT0 DIGITAL.OUT LATCH.V61
  PORT0 DIGITAL.OUT LATCH.V62
  PORT0 DIGITAL.OUT LATCH.V63
  PORT0 DIGITAL.OUT LATCH.V70
  PORT0 DIGITAL.OUT LATCH.V71
  PORT0 DIGITAL.OUT LATCH.V72
  PORT0 DIGITAL.OUT LATCH.V73
  PORT0 DIGITAL.OUT LATCH.V80
  PORT0 DIGITAL.OUT LATCH.V81
  PORT0 DIGITAL.OUT LATCH.V82
  PORT0 DIGITAL.OUT LATCH.V83
;

: SET.VOLTAGES
  INPUT.VOLTAGES
  PRINT.VOLTAGES
  SET.DACS
;

: SET.DAC                              \ [ N1 N2 N3 - ]
  SWAP                                 \ Converts N2 and N3 to single characters
  " LATCH.V"                           \ and catenates them to form LATCH.VXX.
  "." ".".                             \ N1 is written as value to VXX.
```

```
"CAT "CAT 32 "COMPRESS                  \ 0 < = N1 < = 255
PORT0 DIGITAL.OUT "EXEC                 \ 0 < = N2 < = 8   0 < = N3 < = 3
;

: INPUT.VOLTAGE                          \ [ - ] Accepts input from keyboard for a
  BEGIN                                  \ single DAC voltage then calculates and
    #INPUT                               \ checks the digital value (count).
    IF VZDS.INPUT [ NUM ] :=
      VZDS.INPUT [ NUM ] VZDS.MINIMUM [ NUM ] -
      VZDS.STEP [ NUM ] / VZDS.COUNT [ NUM ] :=
      VZDS.COUNT [ NUM ] 255 >
      VZDS.COUNT [ NUM ]  0 <  OR
    ELSE TRUE
    THEN
  WHILE  CR ."      Invalid entry. Try again : "
  REPEAT
  VZDS.COUNT [ NUM ] VZDS.STEP [ NUM ] *
  VZDS.MINIMUM [ NUM ] + VZDS.VALUE [ NUM ] :=
;

: CHANGE.VOLTAGE                         \ [ - ] Prompts user to choose a single
  NORMAL.DISPLAY                         \ voltage to be changed, waits for the input
                                         \ then changes the voltage.
  CR ." Which control voltage would you like to change?"
  CR
  CR ."  1. VZ0     13. VD0     25. VS0"
  CR ."  2. VZ1     14. VD1     26. VS1"
  CR ."  3. VZ2     15. VD2     27. VS2"
  CR ."  4. VZ3     16. VD3     28. VS3"
  CR ."  5. VZ4     17. VD4     29. VS4"
  CR ."  6. VZ5     18. VD5     30. VS5"
  CR ."  7. VZ6     19. VD6     31. VS6"
  CR ."  8. VZ7     20. VD7     32. VS7"
  CR ."  9. VZ8     21. VD8     33. VS8"
  CR ." 10. VZ9      22. VD9     34. VS9"
  CR ." 11. VZ10     23. VD10     35. VS10"
  CR ." 12. VZ11     24. VD11     36. VS11"
  CR
  CR ."      Enter number : "
  BEGIN  #INPUT
    IF NUM :=
      NUM 1 <
      NUM 36 > OR
    ELSE TRUE
    THEN
  WHILE  CR ."      Invalid entry. Try again : "
  REPEAT
  NUM 13 <
  IF CR CR ."      Enter voltage for VZ" NUM 1 - .
    CR  ."      between -" VZ.RANGE . ." and +" VZ.RANGE . ." : "
    INPUT.VOLTAGE
    CR CR ."      Actual voltage applied for VZ" NUM 1 - .
      ." = " VZDS.VALUE [ NUM ] .
    CR  ."      Press any key to continue."
    PCKEY DROP ?DROP
  ELSE  NUM 25 <
    IF  CR CR ."      Enter voltage for VD" NUM 13 - .
      CR  ."      between -" VD/VS.RANGE .
        ." and +" VD/VS.RANGE . ." : "
      INPUT.VOLTAGE
      CR CR ."      Actual voltage applied for VD" NUM 13 - .
        ." = " VZDS.VALUE [ NUM ] .
      CR  ."      Press any key to continue."
      PCKEY DROP ?DROP
    ELSE
      CR CR ."      Enter voltage for VS" NUM 25 - .
      CR  ."      between -" VD/VS.RANGE .
```

```
      ." and +" VD/VS.RANGE . ." : "
    INPUT.VOLTAGE
    CR CR ."     Actual voltage applied for VS" NUM 25 - .
      ." = " VZDS.VALUE [ NUM ] .
    CR   ."     Press any key to continue."
    PCKEY DROP ?DROP
  THEN
THEN
VZDS.COUNT [ NUM ]
NUM 2 - 4.1 / FIX                 \ These two lines determine which DAC needs
NUM 1 - 4 MODULO                  \ to be latched.
SET.DAC
;
```

<u>*APPENDIX A*</u>

Multiple ISFET data acquisition program
File: Words3 Page 1 of 4

```
\ Words required for multiple FET acquisition and temporary storage.
\ (needs DECLARE)

: DECLARE.BUFFER.TOKENS                              \ Dynamically sizes BUFFER.A and .B
  TASK.CHNLS CLEAR.TEMPLATE.BUFFERS                  \ tokens according to MAX.BUFF.SIZE,
  MAX.BUFF.SIZE 2.0                                  \ NUM.FETS, and SAM.POINT.
  NUM.FETS SAM.POINT * / 0.5 - POINTS :=             \ Calculates points per cycle
  INTEGER
  DIM[ POINTS , SAM.POINT , NUM.FETS ] UNNAMED.ARRAY BECOMES> BUFFER.A
  DIM[ POINTS , SAM.POINT , NUM.FETS ] UNNAMED.ARRAY BECOMES> BUFFER.B
  DIM[ POINTS , NUM.FETS ] UNNAMED.ARRAY BECOMES> TEMP
  0 BUFFER.A :=                                      \ Zero all tokens
  0 BUFFER.B :=
  0 TEMP :=
  0 CYCLE# :=
  BUFFER.A EQUIV> PRESENT.ARRAY                      \ Sets PRESENT.ARRAY pointer.
;

: SET.A/D.TEMPLATE                                   \ Set up template for multiple FET acquisition
  TASK.CHNLS                                         \ Choose template, and set gain
  GAIN CASE
     1 OF 0 A/D.GAIN ENDOF
     2 OF 1 A/D.GAIN ENDOF
     4 OF 2 A/D.GAIN ENDOF
     8 OF 3 A/D.GAIN ENDOF
    ENDCASE
  0 NUM.FETS 1 - RESET.A/D.CHNLS                     \ Set the number of A/D channels
  SAM.POINT TEMPLATE.REPEAT                          \ Number of points averaged on the fly
  BUFFER.A BUFFER.B CYCLIC DOUBLE.TEMPLATE.BUFFERS
  0.1 CONVERSION.DELAY                               \ 1 - Line 2 was inserted to maintain reference
  SET.REF0 SET.REF1                                  \ 2   potentials. For unknown reason, when lines
  A/D.INIT                                           \ 3   1 and 3 execute without 2 the D/A is also
;                                                    \     initialized, resetting potentials to zero.

: SET.TASK.TABLE                                     \ Set up background multitasking parameters
  CLEAR.TASKS
  TASK.CHNLS 1 TASK A/D.IN>ARRAY                     \ Set task template, number, & function
  TASK.PERIOD.MS TASK.PERIOD                         \ 50 mS between task interrupts (max=54)
  ACQ.PERIOD TASK.PERIOD.MS / 1000 *                 \ Calculate and set number of task
  1 TASK.MODULO                                      \ interrupts between task executions
  PRIME.TASKS
;

: CREATE.TEMP.DAT                                    \ Set up file for on the fly hard data storage.
  RANDOM.FILE.CREATE \JEFF\DATA\TEMP.DAT             \ Same file name is reused -
  RANDOM.OPEN TEMP.DAT                               \ data is erased on next run.
  POINTS NUM.FETS CATENATE                           \ Store POINTS and NUM.FETS at beginning
  RANDOM.PUT                                         \ of file for recovery purposes. Other
  RANDOM.CLOSE                                       \ parameters must be recovered by hand.
;

: STORE.TEMP.DAT                                     \ Store data from TEMP token on the fly.
  RANDOM.OPEN TEMP.DAT
  ?RANDOM.FILE.SIZE DROP                             \ Move to end of file.
  TEMP RANDOM.PUT                                    \ Write TEMP to disk
  RANDOM.CLOSE
;

: UPDATE.FET1.TOKEN                                  \ These words extract the massaged
  TEMP XSECT[ ! , 1 ]                                \ data from TEMP and store the
  FET1.TOKEN SUB[ INDEX , POINTS ] :=                \ data in the appropiate FET#.-
;                                                    \ TOKEN one dimensional array.

: UPDATE.FET2.TOKEN
  TEMP XSECT[ ! , 2 ]
  FET2.TOKEN SUB[ INDEX , POINTS ] :=
;
```

```
: UPDATE.FET3.TOKEN
  TEMP XSECT[ ! , 3 ]
  FET3.TOKEN SUB[ INDEX , POINTS ] : =
;

: UPDATE.FET4.TOKEN
  TEMP XSECT[ ! , 4 ]
  FET4.TOKEN SUB[ INDEX , POINTS ] : =
;

: UPDATE.FET5.TOKEN
  TEMP XSECT[ ! , 5 ]
  FET5.TOKEN SUB[ INDEX , POINTS ] : =
;

: UPDATE.FET6.TOKEN
  TEMP XSECT[ ! , 6 ]
  FET6.TOKEN SUB[ INDEX , POINTS ] : =
;

: UPDATE.FET7.TOKEN
  TEMP XSECT[ ! , 7 ]
  FET7.TOKEN SUB[ INDEX , POINTS ] : =
;

: UPDATE.FET8.TOKEN
  TEMP XSECT[ ! , 8 ]
  FET8.TOKEN SUB[ INDEX , POINTS ] : =
;

: UPDATE.FET9.TOKEN
  TEMP XSECT[ ! , 9 ]
  FET9.TOKEN SUB[ INDEX , POINTS ] : =
;

: UPDATE.FET10.TOKEN
  TEMP XSECT[ ! , 10 ]
  FET10.TOKEN SUB[ INDEX , POINTS ] : =
;

: UPDATE.FET11.TOKEN
  TEMP XSECT[ ! , 11 ]
  FET11.TOKEN SUB[ INDEX , POINTS ] : =
;

: UPDATE.FET12.TOKEN
  TEMP XSECT[ ! , 12 ]
  FET12.TOKEN SUB[ INDEX , POINTS ] : =
;

: AVERAGE.AND.UPDATE                  \ Massages the raw data in the buffer indic-
                                      \ ated by PRESENT.ARRAY then calls the
                                      \ necessary UPDATE.FET#.TOKEN words.
  POINTS 1 +  1
  DO NUM.FETS 1 +  1                  \ Find average value for each point J of
    DO PRESENT.ARRAY                  \ FET I. Number of samples was determined
      XSECT[ J , ! , I ]              \ by SAM.POINT.
      MEAN
      TEMP [ J , I ] : =              \ Store values in TEMP token
    LOOP
  LOOP
  CYCLE# POINTS * 1 + INDEX : =       \ Calculate position to insert data
                                      \ into FET#.TOKEN
  NUM.FETS
  CASE                                \ Update the necessary FET#.TOKENs
    12 OF UPDATE.FET1.TOKEN           \ based on NUM.FETS.
```

```
      UPDATE.FET2.TOKEN          \ (note: Although this method gives
      UPDATE.FET3.TOKEN          \ bulky code, it is much faster
      UPDATE.FET4.TOKEN          \ than nested if's or loops.)
      UPDATE.FET5.TOKEN
      UPDATE.FET6.TOKEN
      UPDATE.FET7.TOKEN
      UPDATE.FET8.TOKEN
      UPDATE.FET9.TOKEN
      UPDATE.FET10.TOKEN
      UPDATE.FET11.TOKEN
      UPDATE.FET12.TOKEN
   ENDOF
11 OF UPDATE.FET1.TOKEN
      UPDATE.FET2.TOKEN
      UPDATE.FET3.TOKEN
      UPDATE.FET4.TOKEN
      UPDATE.FET5.TOKEN
      UPDATE.FET6.TOKEN
      UPDATE.FET7.TOKEN
      UPDATE.FET8.TOKEN
      UPDATE.FET9.TOKEN
      UPDATE.FET10.TOKEN
      UPDATE.FET11.TOKEN
   ENDOF
10 OF UPDATE.FET1.TOKEN
      UPDATE.FET2.TOKEN
      UPDATE.FET3.TOKEN
      UPDATE.FET4.TOKEN
      UPDATE.FET5.TOKEN
      UPDATE.FET6.TOKEN
      UPDATE.FET7.TOKEN
      UPDATE.FET8.TOKEN
      UPDATE.FET9.TOKEN
      UPDATE.FET10.TOKEN
   ENDOF
9 OF UPDATE.FET1.TOKEN
      UPDATE.FET2.TOKEN
      UPDATE.FET3.TOKEN
      UPDATE.FET4.TOKEN
      UPDATE.FET5.TOKEN
      UPDATE.FET6.TOKEN
      UPDATE.FET7.TOKEN
      UPDATE.FET8.TOKEN
      UPDATE.FET9.TOKEN
   ENDOF
8 OF UPDATE.FET1.TOKEN
      UPDATE.FET2.TOKEN
      UPDATE.FET3.TOKEN
      UPDATE.FET4.TOKEN
      UPDATE.FET5.TOKEN
      UPDATE.FET6.TOKEN
      UPDATE.FET7.TOKEN
      UPDATE.FET8.TOKEN
   ENDOF
7 OF UPDATE.FET1.TOKEN
      UPDATE.FET2.TOKEN
      UPDATE.FET3.TOKEN
      UPDATE.FET4.TOKEN
      UPDATE.FET5.TOKEN
      UPDATE.FET6.TOKEN
      UPDATE.FET7.TOKEN
   ENDOF
6 OF UPDATE.FET1.TOKEN
      UPDATE.FET2.TOKEN
      UPDATE.FET3.TOKEN
      UPDATE.FET4.TOKEN
      UPDATE.FET5.TOKEN
```

```
        UPDATE.FET6.TOKEN
      ENDOF
    5 OF UPDATE.FET1.TOKEN
        UPDATE.FET2.TOKEN
        UPDATE.FET3.TOKEN
        UPDATE.FET4.TOKEN
        UPDATE.FET5.TOKEN
      ENDOF
    4 OF UPDATE.FET1.TOKEN
        UPDATE.FET2.TOKEN
        UPDATE.FET3.TOKEN
        UPDATE.FET4.TOKEN
      ENDOF
    3 OF UPDATE.FET1.TOKEN
        UPDATE.FET2.TOKEN
        UPDATE.FET3.TOKEN
      ENDOF
    2 OF UPDATE.FET1.TOKEN
        UPDATE.FET2.TOKEN
      ENDOF
    1 OF UPDATE.FET1.TOKEN
      ENDOF
  ENDCASE
  CYCLE# 1 + CYCLE# :=                  \ Increment CYCLE#
;

: CHANGE.BUFFER
  0 PRESENT.ARRAY :=                            \ Zero old buffer token
  ?BUFFER.A/B                                   \ Set PRESENT.ARRAY pointer to
  IF BUFFER.B EQUIV > PRESENT.ARRAY             \ the buffer presently being
  ELSE BUFFER.A EQUIV > PRESENT.ARRAY           \ fulled.
  THEN
;

: CLEAN.UP                              \ Restore system upon termination of
  STOP.TASKS                            \ multiple FET acquisition.
  DECIMAL
  CR CR CR CR ." Cleaning up."
  CYCLE# POINTS * POINTS +              \ Test if FET#.TOKEN would have enough
  MAX.BUFF.SIZE 2 /                     \ room for last data set,
  < IF                                  \ then store data if possible.
    AVERAGE.AND.UPDATE
    STORE.TEMP.DAT
  THEN
  CR CYCLE# POINTS * . ." points acquired for each of" NUM.FETS . ." FETs."
  0S BECOMES> PRESENT.ARRAY
  DEFAULT.PROMPT
;

: ZERO.FET.TOKENS
  NUM.FETS 1 + 1
  DO 0
    " FET" I "." "CAT " .TOKEN" "CAT 32 "COMPRESS "EXEC
    :=
  LOOP
;
```

APPENDIX A

Multiple ISFET data acquisition program
File: Words4 Page 1 of 4

```
\ Graphics words for multiple FET acquisition display.
\ (needs DECLARE, WORDS3)

: SET.VUPORTS                          \ Setup graphics display with text
  INSTALL NOP IN PROMPT.XEQ            \ window
  GRAPHICS.DISPLAY
  {GRAPHICS.PROMPT} SCREEN.CLEAR {BORDER}
  VUPORT.MAIN VUPORT.CLEAR
  0 0 AXIS.ORIG                        \ Graph parameters
  1 1 AXIS.SIZE
;

: SET.MULTIPLE.GRAPHS.SCREEN           \ Setup individual graph windows for
  NORMAL.COORDS                        \ each FET
  0 0 AXIS.POINT
  1 4 AXIS.DIVISIONS
  0 0 TICK.JUST
  0 .02 TICK.SIZE
  HORIZONTAL
    AXIS.FIT.OFF
    0 POINTS WORLD.SET
    NO.LABELS
    AXIS.OFF
  VERTICAL
    AXIS.FIT.OFF
    0 4095 WORLD.SET
    NO.LABELS
  NUM.FETS 1 + 1
  DO " VUPORT.FET" I "." "CAT 32 "COMPRESS "EXEC          \ Sets vuport.
    SOLID OUTLINE
    NORMAL.COORDS
    -1 2 FIX.FORMAT
    0.05 0.1 POSITION -10.0 GAIN / "." LABEL          \ Label graphs
    0.05 0.9 POSITION +10.0 GAIN / "." LABEL
    0.85 0.9 POSITION I "." LABEL
    0.85 1  POSITION
    0.85 0.8 DRAW.TO
    1  0.8 DRAW.TO
    XY.AXIS.PLOT                                        \ Draw axes
    " PIX.BUFF." I "." "CAT 32 "COMPRESS                \ These two lines enable pixel
      "EXEC LINE.BUFFER.ON                              \ buffering for fast erasing.
    DOTTED
  LOOP
;

: REFRESH.FET1.PLOT                    \ These words erase the previous
  VUPORT.FET1                          \ plot, then replot old and
  PRESENT.ARRAY XSECT[ ! , 1 , 1 ]     \ new data for each individual
  ERASE.LINES                          \ vuport (graph).
  Y.DATA.PLOT
;
: REFRESH.FET2.PLOT
  VUPORT.FET2
  PRESENT.ARRAY XSECT[ ! , 1 , 2 ]
  ERASE.LINES
  Y.DATA.PLOT
;
: REFRESH.FET3.PLOT
  VUPORT.FET3
  PRESENT.ARRAY XSECT[ ! , 1 , 3 ]
  ERASE.LINES
  Y.DATA.PLOT
;
: REFRESH.FET4.PLOT
  VUPORT.FET4
  PRESENT.ARRAY XSECT[ ! , 1 , 4 ]
  ERASE.LINES
```

```
 Y.DATA.PLOT
;
: REFRESH.FET5.PLOT
 VUPORT.FET5
 PRESENT.ARRAY XSECT[ ! , 1 , 5 ]
 ERASE.LINES
 Y.DATA.PLOT
;
: REFRESH.FET6.PLOT
 VUPORT.FET6
 PRESENT.ARRAY XSECT[ ! , 1 , 6 ]
 ERASE.LINES
 Y.DATA.PLOT
;
: REFRESH.FET7.PLOT
 VUPORT.FET7
 PRESENT.ARRAY XSECT[ ! , 1 , 7 ]
 ERASE.LINES
 Y.DATA.PLOT
;
: REFRESH.FET8.PLOT
 VUPORT.FET8
 PRESENT.ARRAY XSECT[ ! , 1 , 8 ]
 ERASE.LINES
 Y.DATA.PLOT
;
: REFRESH.FET9.PLOT
 VUPORT.FET9
 PRESENT.ARRAY XSECT[ ! , 1 , 9 ]
 ERASE.LINES
 Y.DATA.PLOT
;
: REFRESH.FET10.PLOT
 VUPORT.FET10
 PRESENT.ARRAY XSECT[ ! , 1 , 10 ]
 ERASE.LINES
 Y.DATA.PLOT
;
: REFRESH.FET11.PLOT
 VUPORT.FET11
 PRESENT.ARRAY XSECT[ ! , 1 , 11 ]
 ERASE.LINES
 Y.DATA.PLOT
;
: REFRESH.FET12.PLOT
 VUPORT.FET12
 PRESENT.ARRAY XSECT[ ! , 1 , 12 ]
 ERASE.LINES
 Y.DATA.PLOT
;

: REFRESH.MULTIPLE.PLOTS                  \ Updates the necessary graphs based
 NUM.FETS                                 \ on NUM.FETS.
 CASE
   12 OF REFRESH.FET1.PLOT
        REFRESH.FET2.PLOT
        REFRESH.FET3.PLOT
        REFRESH.FET4.PLOT
        REFRESH.FET5.PLOT
        REFRESH.FET6.PLOT
        REFRESH.FET7.PLOT
        REFRESH.FET8.PLOT
        REFRESH.FET9.PLOT
        REFRESH.FET10.PLOT
        REFRESH.FET11.PLOT
        REFRESH.FET12.PLOT
     ENDOF
```

Multiple ISFET data acquisition program
File: Words4 Page 3 of 4

```
11 OF REFRESH.FET1.PLOT
    REFRESH.FET2.PLOT
    REFRESH.FET3.PLOT
    REFRESH.FET4.PLOT
    REFRESH.FET5.PLOT
    REFRESH.FET6.PLOT
    REFRESH.FET7.PLOT
    REFRESH.FET8.PLOT
    REFRESH.FET9.PLOT
    REFRESH.FET10.PLOT
    REFRESH.FET11.PLOT
  ENDOF
10 OF REFRESH.FET1.PLOT
    REFRESH.FET2.PLOT
    REFRESH.FET3.PLOT
    REFRESH.FET4.PLOT
    REFRESH.FET5.PLOT
    REFRESH.FET6.PLOT
    REFRESH.FET7.PLOT
    REFRESH.FET8.PLOT
    REFRESH.FET9.PLOT
    REFRESH.FET10.PLOT
  ENDOF
9  OF REFRESH.FET1.PLOT
    REFRESH.FET2.PLOT
    REFRESH.FET3.PLOT
    REFRESH.FET4.PLOT
    REFRESH.FET5.PLOT
    REFRESH.FET6.PLOT
    REFRESH.FET7.PLOT
    REFRESH.FET8.PLOT
    REFRESH.FET9.PLOT
  ENDOF
8  OF REFRESH.FET1.PLOT
    REFRESH.FET2.PLOT
    REFRESH.FET3.PLOT
    REFRESH.FET4.PLOT
    REFRESH.FET5.PLOT
    REFRESH.FET6.PLOT
    REFRESH.FET7.PLOT
    REFRESH.FET8.PLOT
  ENDOF
7  OF REFRESH.FET1.PLOT
    REFRESH.FET2.PLOT
    REFRESH.FET3.PLOT
    REFRESH.FET4.PLOT
    REFRESH.FET5.PLOT
    REFRESH.FET6.PLOT
    REFRESH.FET7.PLOT
  ENDOF
6  OF REFRESH.FET1.PLOT
    REFRESH.FET2.PLOT
    REFRESH.FET3.PLOT
    REFRESH.FET4.PLOT
    REFRESH.FET5.PLOT
    REFRESH.FET6.PLOT
  ENDOF
5  OF REFRESH.FET1.PLOT
    REFRESH.FET2.PLOT
    REFRESH.FET3.PLOT
    REFRESH.FET4.PLOT
    REFRESH.FET5.PLOT
  ENDOF
4  OF REFRESH.FET1.PLOT
    REFRESH.FET2.PLOT
    REFRESH.FET3.PLOT
    REFRESH.FET4.PLOT
```

```
   ENDOF
 3 OF REFRESH.FET1.PLOT
     REFRESH.FET2.PLOT
     REFRESH.FET3.PLOT
   ENDOF
 2 OF REFRESH.FET1.PLOT
     REFRESH.FET2.PLOT
   ENDOF
 1 OF REFRESH.FET1.PLOT
   ENDOF
ENDCASE
;
```

Multiple ISFET data acquisition program
File: Words5 Page 1 of 2

```
\ Permanent data storage and retrieval for multiple FET data acquisition.
\ (needs DECLARE; NUM.FETS CYCLE# and POINTS must be properly set)

: ?YES                                  \ [ - ] ( - "t/f")
  "INPUT 1 "LEFT                        \ Returns true to symbol stack if CAPITAL Y
  " Y" "=                               \ is inputted
;
: ?FILE.EXISTS                          \ [ - ] ( - "t/f")
  "DATA.FILE DEFER > FILE.SIZES         \ Returns true to symbol stack if a file
  0 < >                                 \ exists with filename.
  IF TRUE
  ELSE FALSE
  THEN
  DROP                                  \ DROP extra arguement returned by FILE.SIZES
;
: MULTIPLE.FET.FILE.TEMPLATE            \ Sets up file template of minimum size
                                        \ for the data set available.
  ?FILE.OPEN                            \ Check for unclosed file.
  IF FILE.CLOSE
  THEN
  FILE.TEMPLATE
    6 COMMENTS
    INTEGER DIM[ 5 ] SUBFILE            \ NUM.FETS, CYCLE#, POINTS, SAM.POINT, GAIN
    REAL DIM[ 3 ]  SUBFILE              \ ACQ.PERIOD, CONVERSION.DELAY, TASK.PERIOD
    REAL DIM[ 36 ]  SUBFILE             \ VZDS.VALUE's
    INTEGER DIM[ CYCLE# POINTS * ] SUBFILE          \ FET#.TOKEN data
    NUM.FETS TIMES
  END
;
: MULTIPLE.FET.STORE
  "DATA.FILE DEFER > FILE.OPEN
  "DATE "  " "CAT "TIME "CAT  1 >COMMENT
  CR CR ." Enter file comments. ( total of 5)"
  CR ." First comment should include title, book#, and page#."
  7 2 DO
    CR I 1 - . ." - " "INPUT
    I >COMMENT
  LOOP
  CR CR ." Data being saved in " "DATA.FILE "TYPE
  NUM.FETS CYCLE# CATENATE POINTS CATENATE SAM.POINT CATENATE GAIN CATENATE
  1 SUBFILE ARRAY >FILE
  ACQ.PERIOD ?CONVERSION.DELAY CATENATE TASK.PERIOD.MS CATENATE
  2 SUBFILE ARRAY >FILE
  VZDS.VALUE
  3 SUBFILE ARRAY >FILE
  NUM.FETS 1 + 1
  DO " FET" I "." "CAT " .TOKEN" "CAT 32 "COMPRESS "EXEC
    SUB[ 1 , POINTS CYCLE# * ]
    I 3 +  SUBFILE ARRAY >FILE
  LOOP
  FILE.CLOSE
;
: MULTIPLE.FET.FILE.CREATE                      \ [ - ]
  BEGIN
    CR ." Enter filename: "                     \ Prompt user for filename.
    "INPUT DEFER > DATA.FILE
    ?FILE.EXISTS                                \ Check if filename is already in use.
    IF  CR ." File already exists. Type 'Y' to erase old file - "
      ?YES
      IF  TRUE                                  \ Returns true to symbol stack if filename
      ELSE FALSE                                \ does not exist or if operator wishes to
      THEN                                      \ delete old file.
    ELSE TRUE
    THEN
  UNTIL
  "DATA.FILE DEFER > FILE.CREATE                \ Create file then doublecheck with user
  CR CR ." File will be saved as - " "DATA.FILE "TYPE
```

```
  CR ." Type 'Y' if correct - "
  ?YES
  IF MULTIPLE.FET.STORE
    CR ." File saved."
  ELSE
    "DATA.FILE DEFER> DELETE          \ Erase file and start over if filename
    MYSELF                            \ is wrong.
  THEN
;

: SAVE.MULTIPLE.DATA                  \ [ - ] Creates a file according to
  SCREEN.CLEAR                        \ user input and data specifications.
  MULTIPLE.FET.FILE.TEMPLATE
  MULTIPLE.FET.FILE.CREATE
  ONERR:
    ?ERROR# 552 =                     \ Error 552 - attempted to create sub-
    IF                                \ file with dimension = 0.
      CR ." Appears to be no data to save."
      CR ." Please check if NUM.FETS, CYCLE#, and POINTS are correct."
    ELSE ?ERROR# ERROR
    THEN
  CR CR
;

: RETRIEVE.MULTIPLE.DATA              \ [ - ] Transfer data from a disk file to
                                      \ existing FET#.TOKENs.
  ?FILE.OPEN                          \ Check for unclosed file.
  IF FILE.CLOSE
  THEN
  SCREEN.CLEAR
  BEGIN
    CR CR ." Enter filename for retrieval: "    \ Prompt user for filename.
    "INPUT DEFER> DATA.FILE
    ?FILE.EXISTS                      \ Check if file exists.
    IF TRUE
    ELSE CR "DATA.FILE "TYPE ." -- File cannot be found."
      FALSE
    THEN
  UNTIL
  CR CR ." Comments from " "DATA.FILE "TYPE
  "DATA.FILE DEFER> ?COMMENTS                  \ Opens file and prints out comments
  ." Retrieving data from " "DATA.FILE "TYPE
  1 SUBFILE
    FILE>UNNAMED.ARRAY                         \ Retrieve and reset integer parameters
    DUP DUP DUP DUP
    [ 1 ] NUM.FETS :=
    [ 2 ] CYCLE# :=
    [ 3 ] POINTS :=
    [ 4 ] SAM.POINT :=
    [ 5 ] GAIN :=
  2 SUBFILE
    FILE>UNNAMED.ARRAY                         \ Retrieve and reset real parameters
    DUP DUP
    [ 1 ] ACQ.PERIOD :=
    [ 2 ] CONVERSION.DELAY                     \ (note: This is a word, not a variable)
    [ 3 ] TASK.PERIOD.MS :=
  3 SUBFILE
    VZDS.VALUE FILE>ARRAY
  NUM.FETS 1 + 1
  DO " FET" I "." "CAT " .TOKEN" "CAT 32 "COMPRESS "EXEC
    SUB[ 1 , POINTS CYCLE# * ]
    I 3 + SUBFILE FILE>ARRAY
  LOOP
  FILE.CLOSE
  CR ." Data retrieval finished."
  CR CR
;
```

APPENDIX A

Multiple ISFET data acquisition program
File: Program1

```
\ High level words which run FET Controller experiments.
\ (needs DECLARE, WORDS0, WORDS1, WORDS2, WORDS3, WORDS4, WORDS5)


: TURN.ON                          \ Turns on FET Controller with zero settings
  SCREEN.CLEAR
  INITIAL.SET
  SET.DAC.RANGES
  CR CR ." FETs should be disconnected from FET Controller."
  CR ." Turn on FET Controller, then press ENTER."
  "INPUT "DROP
  ZERO.DACS
  CR CR
  ." All applied potentials are now set to 0 V." CR
  ." Please connect FET chips."
;

: SETUP.FETS
  NORMAL.DISPLAY SCREEN.CLEAR
  SET.DAC.RANGES CR
  SET.REFERENCES
  CR CR ." Enter number of FETs to be monitored {12} : "
    BEGIN #INPUT
      IF NUM.FETS : =
        NUM.FETS 1 <
        NUM.FETS 12 > OR
      ELSE FALSE
      THEN
    WHILE CR ." Invalid entry. Try again : "
       12 NUM.FETS : =
    REPEAT
  CR CR ." Enter number of samples to be averaged per point {8} : "
    BEGIN #INPUT
      IF SAM.POINT : =
        SAM.POINT 1 <
        SAM.POINT 100 > OR
      ELSE FALSE
      THEN
    WHILE CR ." Invalid entry. Try again : "
       8 SAM.POINT : =
    REPEAT
  CR CR ." Enter acquisition period in seconds {1} : "
    BEGIN #INPUT
      IF ACQ.PERIOD : =
        ACQ.PERIOD 0.20 <
        ACQ.PERIOD 60.0 > OR
      ELSE FALSE
      THEN
    WHILE CR ." Invalid entry. Try again : "
       1 ACQ.PERIOD : =
    REPEAT
  CR CR ." Enter gain {1} : "
    BEGIN #INPUT
      IF GAIN : =
        GAIN 1 < >
        GAIN 2 < > AND
        GAIN 4 < > AND
        GAIN 8 < > AND
      ELSE FALSE
      THEN
    WHILE CR ." Invalid entry. Try again : "
       1 GAIN : =
    REPEAT
  SET.VOLTAGES
;

: RUN.FETS
```

Multiple ISFET data acquisition program
File: Program1 Page 2 of 2

```
  SET.VUPORTS
  CR ." Setting up"
  ZERO.FET.TOKENS
  DECLARE.BUFFER.TOKENS
  CREATE.TEMP.DAT
  SET.MULTIPLE.GRAPHS.SCREEN
  SET.A/D.TEMPLATE
  SET.TASK.TABLE
  TRIGGER.TASKS
  CR CR CR CR ." Acquiring data.  Press SHIFT F1 to stop"
  BEGIN
    ?BUFFER.SWITCH
    IF  CR CR CR CR ." Massaging data"
      AVERAGE.AND.UPDATE
      CR ." Storing data"
      STORE.TEMP.DAT
      CHANGE.BUFFER
      CR CR CR ." Acquiring data.  Press SHIFT F1 to stop"
      CR ." Last point stored:" CYCLE# POINTS @ .
        ." (Max =" MAX.BUFF.SIZE 2 / . ." )"
    ELSE
      REFRESH.MULTIPLE.PLOTS
    THEN
    ?KEY IF
      PCKEY 84 = AND                  \ Gives TRUE when SHIFT F1 is pressed
    ELSE FALSE
    THEN
  UNTIL
  CLEAN.UP
  ONERR:
    CLEAN.UP
    ?ERROR# 169 =
    IF  CR ." Data acquisition terminated after tokens were full."
    ELSE
      NORMAL.DISPLAY
      DEF.VUPORT
      ?ERROR# ERROR
    THEN
;

: GO
  SETUP.FETS
  RUN.FETS
;
```

APPENDIX A

Multiple ISFET analysis program
File: Declare3

```
INTEGER DIM[ 36 ] ARRAY VZDS.COUNT        \ Integer value output to FET Control-
    128 VZDS.COUNT : =                    \ ler ( 0 to 255)

REAL DIM[ 36 ] ARRAY VZDS.INPUT           \ User input
    0 VZDS.INPUT : =
  DIM[ 36 ] ARRAY VZDS.VALUE              \ Actual voltage applied by FET Con-
    0 VZDS.VALUE : =                      \ troller

  DIM[ 36 ] ARRAY VZDS.MINIMUM            \ Experimentally determined parameters
  DIM[ 36 ] ARRAY VZDS.STEP

FILE.OPEN \JEFF\DATA\DACCONST.DAT         \ Fill minimum and step arrays with
3 SUBFILE VZDS.MINIMUM FILE>ARRAY         \ with default values for VZ = 10
4 SUBFILE VZDS.STEP FILE>ARRAY            \ and VD/VS = 5
FILE.CLOSE

INTEGER SCALAR NUM          0 NUM : =             \ Multipurpose input variable
    SCALAR GAIN         1 GAIN : =                \ Hardware A/D gain
    SCALAR VZ.RANGE       10 VZ.RANGE : =         \ Zeroing voltage range
    SCALAR VD/VS.RANGE      5 VD/VS.RANGE : =     \ Drain and source volt-
                                                  \ age range ( 5 or 10)
    SCALAR MAX.BUFF.SIZE 16384 MAX.BUFF.SIZE : =     \ Should be a power of 2
    SCALAR NUM.FETS        12 NUM.FETS : =        \ Number of FETs
    SCALAR SAM.POINT        8 SAM.POINT : =       \ Number of samples ave-
                                                  \ raged per point
    SCALAR POINTS        85 POINTS : =            \ Number of points taken
                                                  \ per FET per buffer
                                                  \ cycle
    SCALAR CYCLE#         0 CYCLE# : =            \ Count of number of
                                                  \ data buffer cycles
    SCALAR INDEX         0 INDEX : =              \ Used to access pro-
                                                  \ per FET#.TOKEN position

REAL   SCALAR CONV.FACT       0 CONV.FACT : =     \ General use conversion
                                                  \ factor
    SCALAR REF0          0 REF0 : =               \ Reference 0 voltage
    SCALAR REF1          0 REF1 : =               \ Reference 1 voltage
    SCALAR ACQ.PERIOD       1 ACQ.PERIOD : =      \ Acquisition period in
                                                  \ seconds (0.20 to 60)
    SCALAR TASK.PERIOD.MS   50 TASK.PERIOD.MS : =        \ Period in mS between
                                                         \ task interrupts ( < 55)
    SCALAR ?CONVERSION.DELAY 0.1 ?CONVERSION.DELAY : =

: CONVERSION.DELAY
  ?CONVERSION.DELAY : =
;

1 17 1 63  WINDOW {ARRAY.HORIZ.LABEL}     \ Custom array editor windows
1 14 14 15 WINDOW {ARRAY.VERTI.LABEL}
20  1 23 78 WINDOW {GRAPHICS.PROMPT}      \ Multiple FET acquisition text
                                          \ window
186 205 201 200 188 187 BORDER.CHARS      \ Text window border

\ The following FET#.TOKENS will each occupy 16K page in expanded memory.
\ With default MAX.BUFF.SIZE = 16K these tokens will hold 8K of data points.
TOKEN FET1.TOKEN   EXP.MEM> FET1.TOKEN
  INTEGER DIM[ MAX.BUFF.SIZE 2 / ] UNNAMED.ARRAY BECOMES> FET1.TOKEN
  0 FET1.TOKEN : =
TOKEN FET2.TOKEN   EXP.MEM> FET2.TOKEN
  INTEGER DIM[ MAX.BUFF.SIZE 2 / ] UNNAMED.ARRAY BECOMES> FET2.TOKEN
  0 FET2.TOKEN : =
TOKEN FET3.TOKEN   EXP.MEM> FET3.TOKEN
  INTEGER DIM[ MAX.BUFF.SIZE 2 / ] UNNAMED.ARRAY BECOMES> FET3.TOKEN
  0 FET3.TOKEN : =
TOKEN FET4.TOKEN   EXP.MEM> FET4.TOKEN
  INTEGER DIM[ MAX.BUFF.SIZE 2 / ] UNNAMED.ARRAY BECOMES> FET4.TOKEN
  0 FET4.TOKEN : =
```

```
TOKEN FET5.TOKEN   EXP.MEM > FET5.TOKEN
  INTEGER DIM[ MAX.BUFF.SIZE 2 / ] UNNAMED.ARRAY BECOMES> FET5.TOKEN
  0 FET5.TOKEN :=
TOKEN FET6.TOKEN   EXP.MEM > FET6.TOKEN
  INTEGER DIM[ MAX.BUFF.SIZE 2 / ] UNNAMED.ARRAY BECOMES> FET6.TOKEN
  0 FET6.TOKEN :=
TOKEN FET7.TOKEN   EXP.MEM > FET7.TOKEN
  INTEGER DIM[ MAX.BUFF.SIZE 2 / ] UNNAMED.ARRAY BECOMES> FET7.TOKEN
  0 FET7.TOKEN :=
TOKEN FET8.TOKEN   EXP.MEM > FET8.TOKEN
  INTEGER DIM[ MAX.BUFF.SIZE 2 / ] UNNAMED.ARRAY BECOMES> FET8.TOKEN
  0 FET8.TOKEN :=
TOKEN FET9.TOKEN   EXP.MEM > FET9.TOKEN
  INTEGER DIM[ MAX.BUFF.SIZE 2 / ] UNNAMED.ARRAY BECOMES> FET9.TOKEN
  0 FET9.TOKEN :=
TOKEN FET10.TOKEN  EXP.MEM > FET10.TOKEN
  INTEGER DIM[ MAX.BUFF.SIZE 2 / ] UNNAMED.ARRAY BECOMES> FET10.TOKEN
  0 FET10.TOKEN :=
TOKEN FET11.TOKEN  EXP.MEM > FET11.TOKEN
  INTEGER DIM[ MAX.BUFF.SIZE 2 / ] UNNAMED.ARRAY BECOMES> FET11.TOKEN
  0 FET11.TOKEN :=
TOKEN FET12.TOKEN  EXP.MEM > FET12.TOKEN
  INTEGER DIM[ MAX.BUFF.SIZE 2 / ] UNNAMED.ARRAY BECOMES> FET12.TOKEN
  0 FET12.TOKEN :=

TOKEN PLOT.ARRAY                  \ Token to be EQUIV > to data to be plotted

VUPORT VUPORT.MAIN                \ Single graphics window to be used with the
0.0 0.25 VUPORT.ORIG              \ {GRAPHICS.PROMPT} text window
1.0 0.75 VUPORT.SIZE
DEF.VUPORT                        \ Reset to default vuport.
```

APPENDIX A

Multiple ISFET analysis program
File: Words5 (2 pages)

Please see previous listing of Words5 in the Multiple ISFET data acquisition program.

APPENDIX A

Multiple ISFET analysis program
File: Words8 Page 1 of 2

```
\ Words for plotting and analysis of multiple FET data.
\ (needs DECLARE3, and data)
\ Also needs overlay WAVEOPS.SOV (permenantly loaded in ANALYZE)

: SET.ANALYSIS.GRAPH                    \ Setup graphics display
  INSTALL NOP IN PROMPT.XEQ
  GRAPHICS.DISPLAY
  {GRAPHICS.PROMPT} SCREEN.CLEAR {BORDER}
  VUPORT.MAIN VUPORT.CLEAR
  OUTLINE
  AXIS.DEFAULTS
  0.1 0.1 AXIS.ORIG
  0.85 0.85 AXIS.SIZE
  NORMAL.COORDS
  0.1 0.1 AXIS.POINT
  4 4 AXIS.DIVISIONS
  0.02 0.01 TICK.SIZE
  HORIZONTAL                            \ Fit X and Y scales to data
    AXIS.FIT.OFF
    0 CYCLE# POINTS * WORLD.SET
    NO.LABELS
    GRID.OFF
  VERTICAL
    AXIS.FIT.OFF
    PLOT.ARRAY []MIN/MAX
    CONV.FACT / 0.051 +
    CONV.FACT * SWAP
    CONV.FACT / 0.051 -
    CONV.FACT *
    SWAP WORLD.SET
    NO.LABELS
    GRID.OFF
  XY.AXIS.PLOT
;

: LABEL.GRAPH
  NORMAL.COORDS
  5 2 FIX.FORMAT
  0.03 0.95  POSITION AYMAX CONV.FACT / -10.0 GAIN / +  "." LABEL
  0.03 0.1  POSITION AYMIN CONV.FACT / -10.0 GAIN / +  "." LABEL
  4 0 FIX.FORMAT
  0.5  0.05  POSITION AXMAX 2 / 60 / ACQ.PERIOD *  "." LABEL
  0.9  0.05  POSITION AXMAX 60 / ACQ.PERIOD *  "." LABEL
  0.9  0.15  POSITION " Minutes" LABEL
  270 LABEL.DIR
  0.026 0.65 POSITION " Volts" LABEL
  0 LABEL.DIR
  -1 4 FIX.FORMAT
  WORLD.COORDS
;

: PLOT              \ Requires a FET#.TOKEN on stack
  4095 20.0 GAIN / /  CONV.FACT : =
  SUB[ 1 , CYCLE# POINTS * ] EQUIV > PLOT.ARRAY
  0.05 SET.CUTOFF.FREQ
  PLOT.ARRAY 50S + SMOOTH               \ Produces an smoothed array, offset from original
  SET.ANALYSIS.GRAPH
  LABEL.GRAPH
  PLOT.ARRAY Y.DATA.PLOT                \ Plot data
  Y.DATA.PLOT                           \ Plot smoothed data
  DEFAULT.PROMPT
;

: SET.ANALYSIS.GRAPH.HP                 \ Exactly the same as above except that the data
  HP7470                                \ is plotted on HP plotter
  PLOTTER.DEFAULTS
  7.8 10.15 PLOTTER.SIZE
```

```
  0.02 0.06 CHAR.SIZE
  10 SLANT
  AXIS.DEFAULTS
  0.1 0.1 AXIS.ORIG
  0.85 0.85 AXIS.SIZE
  NORMAL.COORDS
  0.1 0.1 AXIS.POINT
  4 4 AXIS.DIVISIONS
  0.02 0.01 TICK.SIZE
  HORIZONTAL
    AXIS.FIT.OFF
    0 CYCLE# POINTS * WORLD.SET
    NO.LABELS
    GRID.OFF
  VERTICAL
    AXIS.FIT.OFF
    PLOT.ARRAY []MIN/MAX
    CONV.FACT / 0.051 +
    CONV.FACT * SWAP
    CONV.FACT / 0.051 -
    CONV.FACT *
    SWAP WORLD.SET
    NO.LABELS
    GRID.OFF
  " VS 0;" GRAPH.COMMAND
  XY.AXIS.PLOT
  " VS;"  GRAPH.COMMAND
;

: LABEL.GRAPH.HP
  NORMAL.COORDS
  4 1 FIX.FORMAT
  " VS 0;" GRAPH.COMMAND
  0.018 0.94  POSITION AYMAX CONV.FACT / -10.0 GAIN / +  "." LABEL
  0.018 0.1  POSITION AYMIN CONV.FACT / -10.0 GAIN / +  "." LABEL
  4 0 FIX.FORMAT
  0.5  0.05  POSITION AXMAX 2 / 60 / ACQ.PERIOD *  "." LABEL
  0.9  0.05  POSITION AXMAX 60 / ACQ.PERIOD *  "." LABEL
  0.85  0.15  POSITION " Minutes" LABEL
  90 LABEL.DIR
  0.03 0.45 POSITION " Volts" LABEL
  " VS;"  GRAPH.COMMAND
  0 LABEL.DIR
  -1 4 FIX.FORMAT
  WORLD.COORDS
;

: HARD.PLOT              \ Requires a FET#.TOKEN on stack
  4095 20.0 GAIN / /  CONV.FACT :=
  SUB[ 1 , CYCLE# POINTS * ] EQUIV> PLOT.ARRAY
  0.05 SET.CUTOFF.FREQ
  PLOT.ARRAY 50S + SMOOTH
  SET.ANALYSIS.GRAPH.HP
  LABEL.GRAPH.HP
  2 COLOR
  PLOT.ARRAY Y.DATA.PLOT
  1 COLOR
  Y.DATA.PLOT
  ." Enter graph title : " "INPUT
  " VS 0;" GRAPH.COMMAND
  NORMAL.COORDS
  0.5 0.94 POSITION CENTERED.LABEL
  " VS;"  GRAPH.COMMAND
  DEFAULT.PROMPT
;
```

APPENDIX A

Test FET acquisition program
File: Declare2 Page 1 of 1

```
DT2800                                          \ Data Translation board DT2801

1 1 D/A.TEMPLATE DAC1                           \ DAC1 to be used for reference
                                                \ electrode control

4 DIGITAL.TEMPLATE PORT0                        \ Digital templates will be used for con-
5 DIGITAL.TEMPLATE PORT1                        \ trol of FET Controller operatation.
6 DIGITAL.TEMPLATE WORD
12 12 A/D.TEMPLATE TEST.CHNL                    \ Used for test circuit data acquisition

0.1 CONVERSION.DELAY                            \ DT2801 parameter ( 0.073 to 163.8 mS)

DAS.INIT


INTEGER SCALAR NUM          0 NUM :=                 \ Multipurpose input variable
   SCALAR VZ.RANGE      10 VZ.RANGE :=               \ Zeroing voltage range
   SCALAR VD/VS.RANGE     5 VD/VS.RANGE :=           \ Drain and source volt-
                                                     \ age range ( 5 or 10)
   SCALAR ACQ.PERIOD    100 ACQ.PERIOD :=            \ Acquisition period in mS
                                                     \ Minimum value tested is 100
   SCALAR LIMIT        6 LIMIT :=                    \ Acquisition time limit-Sec
   SCALAR POINTS        60 POINTS :=                 \ Number of points per VD-VG
                                                     \ setting
   SCALAR GAIN         2 GAIN :=                     \ Hardware A/D voltage gain
   SCALAR VD#          7 VD# :=                      \ Number of VD values to test
   SCALAR VG#          4 VG# :=                      \ Number of VG values to test

TOKEN DR0.COUNT
TOKEN REF1.COUNT
TOKEN DR0.VALUE
TOKEN REF1.VALUE

INTEGER DIM[ VD# ] UNNAMED.ARRAY BECOMES> DR0.COUNT
    DIM[ VG# ] UNNAMED.ARRAY BECOMES> REF1.COUNT
REAL  DIM[ VD# ] UNNAMED.ARRAY BECOMES> DR0.VALUE
    DIM[ VG# ] UNNAMED.ARRAY BECOMES> REF1.VALUE

DR0.COUNT @[ 1 ]                                     \ Vd voltage counts and actual values
  ENTER[ 128 , 141 , 153 , 179 , 204 , 229 , 255 ]
DR0.VALUE @[ 1 ]
  ENTER[ 0.004 , 0.516 , 0.989 , 2.014 , 3.000 , 3.985 , 5.010 ]

REF1.COUNT @[ 1 ]                                    \ Vg voltage counts and actual values
  ENTER[ 2048 , 2253 , 2458 , 2662 ]
REF1.VALUE @[ 1 ]
  ENTER[ 0.003 , 1.006 , 2.008 , 3.005 ]
STACK.CLEAR

186 205 201 200 188 187 BORDER.CHARS                 \ Text window border
20 1 23 78 WINDOW {GRAPHICS.PROMPT}                  \ Multiple FET acquisition text
                                                     \ window

TOKEN TEST.DATA    EXP.MEM > TEST.DATA
TOKEN RAW.TEST.DATA EXP.MEM > RAW.TEST.DATA

VUPORT VUPORT.MAIN                              \ Single graphics window to be used with the
0.0 0.25 VUPORT.ORIG                            \ {GRAPHICS.PROMPT} text window
1.0 0.75 VUPORT.SIZE

REAL DIM[ 2048 ] ARRAY PIX.BUFF                 \ Graphic pixel buffer for fast
                                                \ erasing of data.
```

<u>*APPENDIX A*</u>

<u>Test FET acquisition program</u>
<u>File: Words0 (5 pages)</u>

Please see previous listing of Words0 in the Multiple ISFET data acquisition program.

APPENDIX A

Test FET acquisition program
File: Words6 Page 1 of 2

```
\ Words to acquire chip test data.
\ (needs DECLARE2, WORDS0)

: INITIAL.SET
  WORD
  65408 DIGITAL.OUT
  DAC1
  2048 D/A.OUT
;

: SET.DAC.RANGES
  PORT1
  64. DIGITAL.MASK
  1. WRITE.BITS
  128. DIGITAL.MASK
  1. WRITE.BITS
;

BINARY
: LATCH.VD0
  PORT1
  11. DIGITAL.MASK
  00. WRITE.BITS
  110000. DIGITAL.MASK
  PULSE.BITS
;
DECIMAL

: DECLARE.TOKENS
  INTEGER DIM[ VD# , VG# , POINTS ] UNNAMED.ARRAY
  BECOMES> RAW.TEST.DATA
  0 RAW.TEST.DATA :=
  INTEGER DIM[ VD# , VG# ] UNNAMED.ARRAY
  BECOMES> TEST.DATA
  0 TEST.DATA :=
;

: SET.TEST.GRAPH
  INSTALL NOP IN PROMPT.XEQ
  GRAPHICS.DISPLAY
  {GRAPHICS.PROMPT} SCREEN.CLEAR {BORDER}
  VUPORT.MAIN VUPORT.CLEAR
  OUTLINE
  AXIS.DEFAULTS
  0.1 0.05 AXIS.ORIG
  0.9 0.9 AXIS.SIZE
  0.02 0.01 TICK.SIZE
  NORMAL.COORDS
  0.1 0.725 AXIS.POINT
  6 4    AXIS.DIVISIONS
  HORIZONTAL
    AXIS.FIT.OFF
    0 POINTS WORLD.SET
    GRID.OFF
    NO.LABELS
  VERTICAL
    AXIS.FIT.OFF
    819 2457 WORLD.SET
    GRID.OFF
    NO.LABELS
  NORMAL.COORDS
  4 1 FIX.FORMAT
  0.03 0.95 POSITION  10.0 GAIN / 5 / 0 SWAP - "." LABEL
  0.03 0.05 POSITION  10.0 GAIN / 5 / 3 * 0 + "." LABEL
  -1 1 FIX.FORMAT
  0.23 0.775 POSITION  LIMIT 6.0 / "." LABEL
  0.53 0.775 POSITION  LIMIT 2.0 / "." LABEL
```

```
  0.83 0.775 POSITION  LIMIT 6.0 / 5 * "." LABEL
  0.5 0.95  POSITION  " Seconds" CENTERED.LABEL
  270 LABEL.DIR
  0.025 0.75 POSITION  " Milliamps" LABEL
  0 LABEL.DIR
  XY.AXIS.PLOT
  PIX.BUFF LINE.BUFFER.ON
;



: TEST.FET
  LIMIT ACQ.PERIOD 1000.0 / / POINTS :=
  DECLARE.TOKENS
  ACQ.PERIOD SYNC.PERIOD
  TEST.CHNL GAIN A/D.GAIN
  A/D.INIT
  SET.TEST.GRAPH
  SYNC.ERROR.OFF
  0 TEST.DATA :=
  VD# 1 + 1
  DO
    DR0.COUNT [ I ] PORT0 DIGITAL.OUT
    LATCH.VD0
    VG# 1 + 1
    DO
      REF1.COUNT [ I ] DAC1 D/A.OUT
      ERASE.LINES
      SCREEN.CLEAR
      CR ." VD = " DR0.VALUE [ J ] . ." V "
      ."  VG = " REF1.VALUE [ I ] . ." V"
      CR ." SHIFT-F1 to end this voltage test."
      0 A/D.IN POSITION
      POINTS 1 + 1
      DO
        SYNCHRONIZE
        A/D.IN DUP
        RAW.TEST.DATA [ K , J , I ] :=
        I SWAP DRAW.TO
        ?KEY IF
          PCKEY 84 = AND
          IF LEAVE
          THEN
        THEN
      LOOP
      A/D.IN TEST.DATA [ J , I ] :=
    LOOP
  LOOP
  DEFAULT.PROMPT
;

: TURN.ON
  SCREEN.CLEAR
  INITIAL.SET
  SET.DAC.RANGES
  CR CR ." FETs should be disconnected from FET Controller."
  CR ." Turn on FET Controller, then press ENTER."
  "INPUT "DROP
  ZERO.DACS
  CR CR ." All applied potentials are now set to 0 V."
  CR ." Please connect FET chip to be tested."
;
```

APPENDIX A

Test FET acquisition program
File: Words7 Page 1 of 3

```
\ Permanent data storage, retrieval, and printout for FET test routine.
\ (needs DECLARE2 or DECLARE4)

: ?YES                                  \ [-]( - "t/f")
  "INPUT 1 "LEFT                        \ Returns true to symbol stack if CAPITAL Y
  " Y" "=                               \ is inputted
;

: ?FILE.EXISTS                          \ [-]( - "t/f")
  "DATA.FILE DEFER > FILE.SIZES         \ Returns true to symbol stack if a file
  0 < >                                 \ exists with filename.
  IF TRUE
  ELSE FALSE
  THEN
  DROP                                  \ DROP extra arguement returned by FILE.SIZES
;

: TEST.FET.FILE.TEMPLATE                \ Sets up file template of minimum size
                                        \ for the data set available.
  ?FILE.OPEN                            \ Check for unclosed file.
  IF FILE.CLOSE
  THEN
  FILE.TEMPLATE
    6 COMMENTS
    INTEGER DIM[ 5 ] SUBFILE                    \ ACQ.PERIOD, LIMIT, GAIN, VD#, VG#
    REAL DIM[ VD# ] SUBFILE                     \ Drain voltages applied
    REAL DIM[ VG# ] SUBFILE                     \ Gate (reference) voltages applied
    INTEGER DIM[ VD# , VG# , POINTS ] SUBFILE   \ Raw test data
    INTEGER DIM[ VD# , VG# ] SUBFILE            \ Extracted test data
  END
;

: TEST.FET.STORE
  "DATA.FILE DEFER > FILE.OPEN
  "DATE " " "CAT "TIME "CAT 1 >COMMENT
  CR CR ." Enter file comments. ( total of 5)"
  CR ." First comment should include title, book#, and page#."
  CR ." Second comment should include chip and gate ID."
  7 2 DO
    CR I 1 - . ." - " "INPUT
    I >COMMENT
  LOOP
  CR CR ." Data being saved in " "DATA.FILE "TYPE
  ACQ.PERIOD LIMIT CATENATE GAIN CATENATE VD# CATENATE VG# CATENATE
   1 SUBFILE ARRAY >FILE
  DR0.VALUE
   2 SUBFILE ARRAY >FILE
  REF1.VALUE
   3 SUBFILE ARRAY >FILE
  RAW.TEST.DATA
   4 SUBFILE ARRAY >FILE
  TEST.DATA
   5 SUBFILE ARRAY >FILE
  FILE.CLOSE
;

: TEST.FET.FILE.CREATE                  \ [-]
  BEGIN
    CR ." Enter filename: "             \ Prompt user for filename.
    "INPUT DEFER > DATA.FILE
    ?FILE.EXISTS                        \ Check if filename is already in use.
    IF CR ." File already exists. Type 'Y' to erase old file - "
      ?YES
      IF TRUE                           \ Returns true to symbol stack if filename
      ELSE FALSE                        \ does not exist or if operator wishes to
      THEN                              \ delete old file.
    ELSE TRUE
```

```
    THEN
  UNTIL
  "DATA.FILE DEFER > FILE.CREATE          \ Create file then doublecheck with user
  CR CR ." File will be saved as - " "DATA.FILE "TYPE
  CR ." Type 'Y' if correct - "
  ?YES
  IF TEST.FET.STORE
    CR ." File saved."
  ELSE
    "DATA.FILE DEFER > DELETE             \ Erase file and start over if filename
    MYSELF                                \ is wrong.
  THEN
;

: SAVE.TEST.DATA                          \ [ - ] Creates a file according to
  SCREEN.CLEAR
  TEST.FET.FILE.TEMPLATE                  \ user input and data specifications.
  TEST.FET.FILE.CREATE
  ONERR:
    ?ERROR# 552 =                         \ Error 552 - attempted to create sub-
    IF                                    \ file with dimension = 0.
      CR ." Appears to be no data to save."
      CR ." Please check if VD#, VG#, and POINTS are correct."
    ELSE ?ERROR# ERROR
    THEN
  CR CR
;

: RETRIEVE.TEST.DATA                      \ [ - ] Transfer data from a disk file to
                                          \ existing RAW.TEST.DATA and TEST.DATA files.
  ?FILE.OPEN                              \ Check for unclosed file.
  IF FILE.CLOSE
  THEN
  SCREEN.CLEAR
  BEGIN
    CR CR ." Enter filename for retrieval: "          \ Prompt user for filename.
    "INPUT DEFER > DATA.FILE
    ?FILE.EXISTS                                      \ Check if file exists.
    IF TRUE
    ELSE CR "DATA.FILE "TYPE ." -- File cannot be found."
      FALSE
    THEN
  UNTIL
  CR CR ." Comments from " "DATA.FILE "TYPE
  "DATA.FILE DEFER > ?COMMENTS                        \ Opens file and prints out comments
  ." Retrieving data from " "DATA.FILE "TYPE
  1 SUBFILE
    FILE > UNNAMED.ARRAY                              \ Retrieve and reset integer parameters
    DUP DUP DUP DUP
    [ 1 ] ACQ.PERIOD :=
    [ 2 ] LIMIT :=
    [ 3 ] GAIN :=
    [ 4 ] VD#   :=
    [ 5 ] VG#   :=
  VD# REAL RAMP BECOMES> DR0.VALUE                    \ Roundabout method of creating a real,
  0 DR0.VALUE :=                                      \ initialized, unnamed array. ( can't
  VG# REAL RAMP BECOMES> REF1.VALUE                   \ use BECOMES> with uninit. real array)
  0 REF1.VALUE :=
  2 SUBFILE
    DR0.VALUE FILE >ARRAY
  3 SUBFILE
    REF1.VALUE FILE >ARRAY
  LIMIT ACQ.PERIOD 1000.0 / / POINTS :=
  DECLARE.TOKENS
  4 SUBFILE
    RAW.TEST.DATA FILE >ARRAY
  5 SUBFILE
```

```
   TEST.DATA FILE>ARRAY
  FILE.CLOSE
  CR ." Data retrieval finished."
  CR CR
;

: CALCULATE.TEST.RESULTS
  10 GAIN /
  TEST.DATA
  20.0 GAIN / 4095 /
  * -
;

: PRINT.TEST.RESULTS
  CALCULATE.TEST.RESULTS
  7 2 FIX.FORMAT
  OUT>PRINTER
  CR CR ." VG              VD VOLTAGES"
  CR  ." VOLTAGES "
  VD# 1 + 1 DO
    DR0.VALUE [ I ] .
  LOOP
  CR
  VG# 1 + 1 DO
    CR REF1.VALUE [ I ] .
    7 3 FIX.FORMAT
    ."  "
    VD# 1 + 1 DO
      DUP [ I , J ] .
    LOOP
    7 2 FIX.FORMAT
  LOOP
  CR CR .DATE ."   " .TIME
  CR ." FET ID: "
  "INPUT "DROP
  DROP
  -1 4 FIX.FORMAT
  CONSOLE
;
```

APPENDIX A


```
\ For use in ANALYZE to examine TEST.FET results.
\ Use WORDS7 to retrieve data.


INTEGER SCALAR NUM          0 NUM :=              \ Multipurpose input variable
    SCALAR VZ.RANGE      10 VZ.RANGE :=           \ Zeroing voltage range
    SCALAR VD/VS.RANGE    5 VD/VS.RANGE :=        \ Drain and source volt-
                                                  \  age range ( 5 or 10)
    SCALAR ACQ.PERIOD    100 ACQ.PERIOD :=        \ Acquisition period in mS
                                                  \ Minimum value tested is 100
    SCALAR LIMIT         6 LIMIT :=               \ Acquisition time limit-Sec
    SCALAR POINTS         60 POINTS :=            \ Number of points per VD-VG
                                                  \  setting
    SCALAR GAIN          2 GAIN :=                \ Hardware A/D voltage gain
    SCALAR VD#           7 VD# :=                 \ Number of VD values to test
    SCALAR VG#           4 VG# :=                 \ Number of VG values to test

TOKEN DR0.VALUE
TOKEN REF1.VALUE

STACK.CLEAR

TOKEN TEST.DATA   EXP.MEM > TEST.DATA          \ Data is stored in expanded memory
TOKEN RAW.TEST.DATA EXP.MEM > RAW.TEST.DATA         \ Frees up main memory
TOKEN MEANS.TEST.DATA  EXP.MEM > MEANS.TEST.DATA

VUPORT VUPORT.MAIN                        \ Single graphics window to be used with the
0.0 0.0 VUPORT.ORIG                       \  {GRAPHICS.PROMPT} text window
1.0 1.0 VUPORT.SIZE

: DECLARE.TOKENS                          \ This word is required by WORDS7 to read in data
  INTEGER DIM[ VD# , VG# , POINTS ] UNNAMED.ARRAY
  BECOMES> RAW.TEST.DATA
  0 RAW.TEST.DATA :=
  INTEGER DIM[ VD# , VG# ] UNNAMED.ARRAY
  BECOMES> TEST.DATA
  0 TEST.DATA :=
  TEST.DATA BECOMES> MEANS.TEST.DATA
;

REAL DIM[ 2 ] ARRAY XSCALE                \ Scaling settings for ouput graphs
REAL DIM[ 2 ] ARRAY YSCALE
XSCALE @[ 1 ] ENTER[ 0 , 5 ]
YSCALE @[ 1 ] ENTER[ 0 , 1.5 ]
STACK.CLEAR
```

Test FET analysis program
File: Words7 (3 pages)

Please see previous listing of Words7 in the Test FET acquisition program.

APPENDIX A

Test FET analysis program
File: Words10 Page 1 of 1

```
\ Used to plot Vd vs. Current for varying Vg's graph.
\ Needs DECLARE4, and WORDS7 to retrieve data.


: CALC.MEANS                             \ Calculate mean current for each VD, VG setting
  TEST.DATA BECOMES> MEANS.TEST.DATA          \ Resize and initialize MEANS.TEST.DATA
  0 MEANS.TEST.DATA :=
  VD# 1 + 1 DO                           \ Calculate means and save in MEANS.TEST.DATA
    VG# 1 + 1 DO
      RAW.TEST.DATA XSECT[ J , I , ! ]
      MEAN
      MEANS.TEST.DATA [ J , I ] :=
    LOOP
  LOOP
;

: GO
  OUT>PRINTER                            \ Automatic printing of screen output
  RETRIEVE.TEST.DATA
  CALC.MEANS
  MEANS.TEST.DATA BECOMES> TEST.DATA
  PRINT.TEST.RESULTS                     \ Print mean currents for all VD, VG settings
  CALCULATE.TEST.RESULTS BECOMES> MEANS.TEST.DATA   \ Change digital data to voltages
  GRAPHICS.DISPLAY                       \ Setup graphics display
  VUPORT.MAIN VUPORT.CLEAR
  OUTLINE
  DOTTED
  XSCALE YSCALE XY.AUTO.PLOT             \ Plot axes
  SOLID
  VG# 1 + 1 DO                           \ Plot line for each VG
    DR0.VALUE MEANS.TEST.DATA XSECT[ ! , I ] XY.DATA.PLOT
  LOOP
  SCREEN.PRINT
  NORMAL.DISPLAY                         \ Reset to default settings
  CLEAR.TOKENS
  DEF.VUPORT
;
```

APPENDIX B

X-Y PLOTTING FROM THE OSCILLOSCOPE

The Houston Instrument *X-Y* recorder and the Tektronix oscilloscope used in Chapter 6 (Section 6.4.3) were not compatible. The oscilloscope's analog X and Y outputs could be directly hooked up to the recorder's inputs, but the pen lift was a problem. The oscilloscope uses a digital +5V output while the recorder requires a +12V, 500 mA analog input. The circuit in Figure B-1 was designed to use a transistor switch, controlled by the oscilloscope, and a relay to use the recorder's own +12V output to control the pen lift mechanism. This circuit was constructed on a small piece of breadboard and then glued onto the base of the recorder and hardwired to the recorder terminals. The cable from the auxiliary connector on the oscilloscope was then plugged into the breadboard.

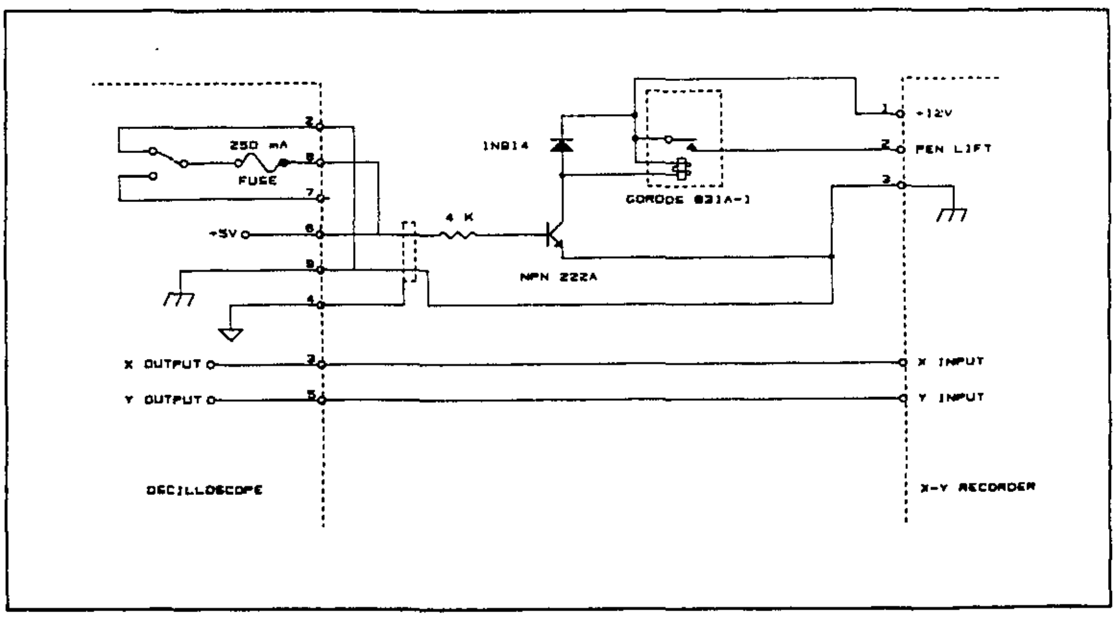

Figure B-1. Interface circuit between *X-Y* recorder and oscilloscope