SI 1280 Technical Manual
Contents

Chapter

1 Introduction

Introduces the facilities of the SI1280 Electrochemical Measurement Unit, summarises the content of the manual, and lists the associated documentation.

2 Using the SI1280

Guides you through some simple uses of the SI1280 and signposts the way to more advanced uses.

3 ECI Command Functions

Provides a logical breakdown of the ECI facilities and lists the commands that are used to drive them.

4 FRA Command Functions

Provides a logical breakdown of the FRA facilities and lists the commands that are used to drive them.

5 GPIB (IEEE488.2) Interface

Describes the GPIB facilities of the SI1280

Appendix

Chapter 1

Introduction

This manual covers all models of the 1280 EMU. Unless otherwise stated the information contained in the manual relates both to the 1280A and the 1280B. Where performance details vary, this is pointed out.

1. ELECTROCHEMISTRY AND THE SI 1280

Electrochemical measurements rely on the electrical aspect of chemical processes to provide readable data. This is based on Faraday's law which relates the change in mass per unit area of a substance to the magnitude of the current flowing through it. Measurements are made of the voltage and current acting in an electrochemical "cell", whose basic form is a pair of metal electrodes immersed in an electrolyte.

The wide range of studies to which electrochemical measurements can be usefully applied includes: corrosion, effectiveness of protective coatings, batteries, and biological processes. For these measurements the S11280 Electrochemical Measurement Unit (EMU) can provide accurate d.c. polarization to establish the rate of ionization in the cell and frequency response analysis to study the cell impedance characteristics. The full spectrum of electrochemical measurement techniques can thus be employed to establish or study the mechanisms of various electrochemical phenomena.

2. FEATURES OF THE SI 1280

The S11280 EMU provides the combined facilities of an electrochemical interface (ECI) and a frequency response analyzer (FRA).

The ECI can be used either as a potentiostat or galvanostat, with selectable control loop bandwidth to ensure stable operation for various types of cell. Full compensation and correction facilities are provided to enable you to extract the most useful information from your experimental data, with the utmost precision.

The FRA provides a precision signal generator and an analyzer for measuring cell impedance data. A.c. measurements (20kHz max.) are possible up to the eighth harmonic of the generator frequency. Signal interference is rejected by integrating each measurement over a whole number of cycles of the fundamental (generator) frequency: the period of integration is user-defined, which allows an optimum compromise to be obtained between the degree of interference rejection and the measurement rate.

Control of the S11280 EMU is managed by simple commands that are applied from a controller, such as a personal computer, via the GPIB. Data from the S11280 EMU may be output in the form most suitable for your analysis requirements. ASCII and binary coded data is available, as well as ASCII coded status messages.

2.1. SI 1280 ACCESSORIES

Some useful accessories are provided with the SI1280 EMU, whilst others are available as options.

The accessories provided are:

- A 12861 Test Module. This simulates the characteristics of a simple electrochemical cell and can be used to verify the operation of your S11280 during familiarization. (See Chapter 2.)
- This technical manual.
- A power cord and spare a.c. supply fuses.
- Four leads, one metre long, for connecting an electrochemical cell to the SI1280.

The optional accessories are: a maintenance manual (12806001), a rack mounting kit (12801A) and a carrying case (12802A).

3. THE MANUAL

This manual aims to present the powerful facilities of the SI 1280 to you in the simplest and most visible way. You are thus made aware of what the facilities are and, by means of simple examples, are encouraged to start using them.

CHAPTER 2 starts with the measurement background and tells you what you should know and do before attempting to use the SI 1280. It then continues with some simple examples of use. To enable you to get some predictable results the examples assume that you are measuring the test module. However, it is a useful exercise to repeat the examples with an actual cell, remembering, of course, to modify the polarization parameters in accordance with the type of cell measured. The chapter concludes with a guide to other possible uses.

CHAPTER 3 gives full details of the ECI facilities and the commands used to control them.

CHAPTER 4 gives full details of the FRA facilities and the commands used to control them.

CHAPTER 5 gives full details of the GPIB interface in the SI 1280 and describes the format of the ECI and FRA output data.

APPENDIX A gives full details of how to install the SI 1280.

APPENDIX B lists the error codes, and explains the meaning of each one.

APPENDIX C contains the full specification of the SI 1280.

4. FURTHER READING

Understanding Electrochemical Cells, by A.M. Kauffman. (Technical Report 017/85) This book gives a simple non-mathematical treatment of what happens in an electrochemical cell during a corrosion process. It is intended for technicians who wish to obtain an intuitive grasp of the phenomena involved.

Identification of Electrochemical Processes by Frequency Response Analysis, by Claude Gabrielli. (Technical Report 004/83) This book gives a broad introduction to the many different techniques that can be used in electrochemistry. Although written by an electrochemist for electrochemists the book does not aim to be a deep study, but it does contain an extensive bibliography. You are thus directed to a number of classical treatises and scientific papers on many subjects of interest.

Use and Applications of Electrochemical Impedance Techniques, by Claude Gabrielli. (Technical Report No. 24) This book complements the author's other book (above), but is more application oriented. Again, reference is made to many scientific papers.

Use and Analysis of EIS Data for Metals and Alloys, by Florian Mansfeld. (Technical Report No. 26) This is a collection of reports, previously published by Solartron Instruments, which describes in some detail the different analytical processes required for data acquired by electrochemical impedance spectroscopy. The models proposed for the simulation and fitting of EIS data are discussed and each is illustrated by the interpretation of actual experimental data.

Chapter 2

Using the SI 1280

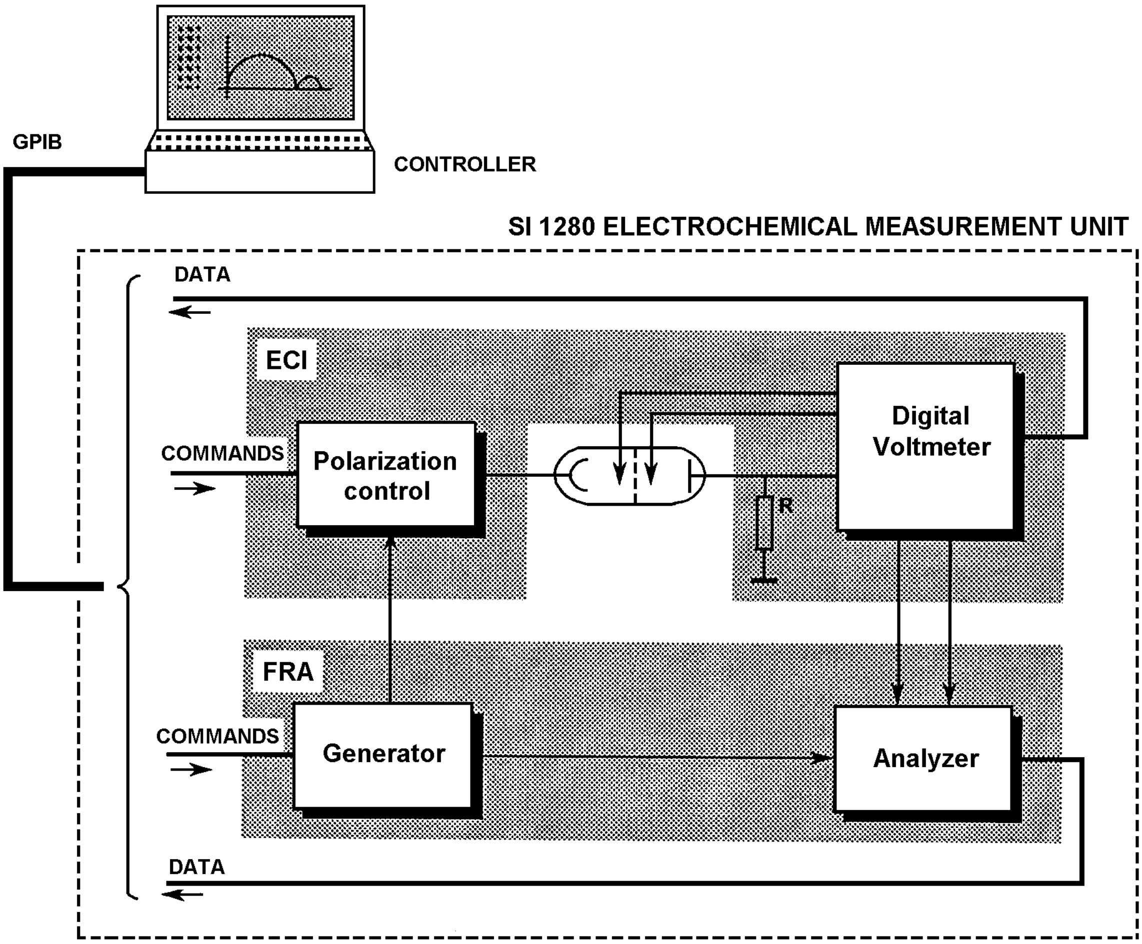


Fig 2.1 System schematic.

1. MEASUREMENT BACKGROUND

A simple schematic of the SI 1280 Electrochemical Measurement Unit is shown in Figure 2.1. This highlights the basic division of the unit into the Electrochemical Interface (ECI) and the Frequency Response Analyzer (FRA).

In accordance with commands received from the controller, the ECI maintains the d.c. polarization of an electrochemical cell in a user-defined state and measures the resulting cell behaviour.

When required, the FRA can be commanded to generate an a.c. perturbating signal for the cell, this signal being added to the d.c. polarization provided by the ECI. The a.c. component of cell polarization is then analyzed to provide cell impedance data.

The ECI and the FRA receive their commands from the controller, and return ASCII measurement and status data to the controller, via the GPIB. Measurement data is also available in binary form.

1.1. THE CONTROLLER

The controller can be any device that is able to run a control program in keeping with your electrochemical analysis requirements, and that has a GPIB (IEEE 488) interface. Where the SI 1280 is to be used in the field, e.g. for corrosion analysis, then a laptop PC, with the same capability, is recommended.

1.2. INSTALLATION

Before any attempt is made to use the SI 1280 it should be properly installed, as described in Appendix A. This is most important, since it involves the safety of the operator and the continuing serviceability of the SI 1280 and the electrochemical cell.

The installation procedure covers the following important items:

- Safety precautions.
- A.c. supply voltage selection and fuses.
- Inputs and outputs, GPIB setups (including device address).
- GPIB setups (including device address).
- Environmental details (EMC, temperature, humidity, etc.).
- Rack mounting.

1.3. APPROACH TO PROGRAMMING

It is assumed that you have sufficient expertise to program the controller for GPIB operation: if you do not, it is recommended, that you study the relevant information in the controller operating manual. The information that you will need regarding the GPIB interfaces in the SI 1280 is contained in Chapter 5 of this manual.

Full details of the ECI and FRA commands and their functions are given in Chapters 3 and 4 of this manual. Also, a simple introduction to the application of these commands is given in the examples in the present chapter. This information, combined with a knowledge of computer programming and electrochemistry, should enable you to write a comprehensive electrochemical control and analysis program.

Should you wish to widen your knowledge of electrochemical measurement techniques, several technical monographs are available, free of charge, from Solartron. See the list at the end of Chapter 1.

1.4. COMMAND TYPES

All commands for the SI 1280 consist of two alphabetic characters, followed either by an integer or a real number.

The integer commands are used either to choose a control setting or to command a direct action. For example, in digital voltmeter control in the ECI the command **RG**2 selects the 200mV input range, whilst the command **RU**1 switches the digital voltmeter on.

Real number commands are used for the precise definition of a parametric value. A typical command of this type is

PV0.025

which defines a polarization voltage of + 25mV for the ECI potentiostat.

Real numbers are stored in the SI 1280 memory in floating-point format, and are output as such. They can, however, be entered in any convenient form. For example, a polarization voltage of +25mV could be entered as **PV**0.025 or **PV**25E-3: for either of these, however, the polarization value would be stored as + 2.5000E-02.

Control values, thus entered, can be queried, usually by preceding the two alphabetic characters with a question mark. The response to the **?PV** command in the example above would be + 2.5000E-02.

The response to a control setup query always consists of two digits. For example, "03", obtained in response to a **?IL** command, indicates that the current limit presently selected is 2mA.

1.5. CONTROL STATEMENTS

At the controller the commands for the SI 1280 will be enclosed in program statements, whose form depends on the high level language used by the controller. Therefore, it is not intended to use actual program statements in the examples of use in this chapter. Instead, the command that should be sent by the controller is stated, together with the SI 1280 response and any data that the controller should be prepared to read back.

1.6. DEFAULT SETUP

When the SI 1280 is first switched on the ECI and FRA are automatically initialized. The default setup thus obtained provides a convenient point from which setting up may begin.

You can also select the default setup by command. The default setup for the ECI is obtained with the commands **BK**3 (reset) and **BK**4 (initialize). reset selects the default setup, but leaves any measurement data in the history file intact; initialize selects the default setup and clears the history file. The default setup for the FRA is obtained with the commands TT1 (initialize) and TT2 (reset): initialize stops the generator, returns all control setups to the default state, clears the history file, and sets the elapsed time clock to zero; reset does exactly the same, except that the content of the history file is retained.

The default setting of each control parameter is indicated in Chapters 3 and 4, by underlining the associated command.

2. EXAMPLES OF ECI AND FRA CONTROL

This section gives some simple examples of how to use the SI 1280 commands to set up the ECI and FRA.

It is assumed that the SI 1280 has been installed in accordance with the information given in Appendix A of this manual, and that the GPIB address has been set as described in Chapter 5. It is also assumed that the controller is properly installed and running, and has been programmed so that you can send the SI 1280 commands individually and check the response.

The first three examples show you how to set up the ECI to make single V,I measurements, and how to use a stepped sweep and a ramp sweep. These examples illustrate the three ways in which the data for polarization curves may he obtained.

The last example shows you how to set up the ECI and the FRA for a frequency sweep, to obtain cell impedance data.

2.1. PRELIMINARY ACTIONS

1. Connect a 12861 Test Module to the cell terminals at the rear of the SI 1280. The test module circuit is shown in Figure 2.2.

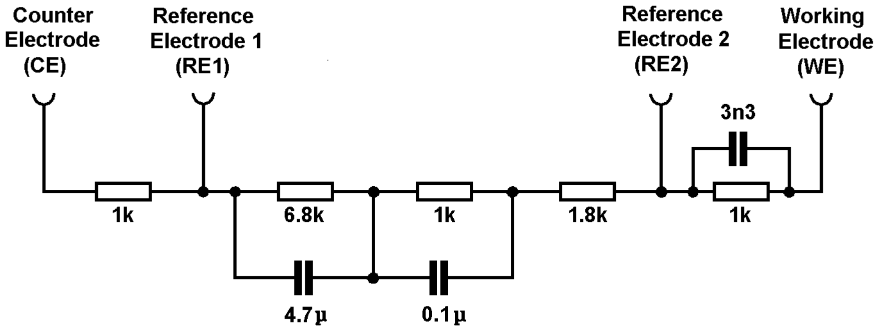


Fig 2.2 12861 test Module circuit.

2. Switch on the SI 1280, when the ECI default setup should be:

- Potentiostat.
- Full standby.
- Ready for PolV mode.
- Polarization bandwidth limit ON, at bandwidth "C" (> 1MHz).
- Auto-range for current measurement.
- Cell current limit=2A.
- Current off limit action. return to standby.
- IR compensation OFF.
- Real part correction OFF.
- Output conditioning OFF.
- Sweep OFF.
- Digital voltmeter OFF.
- Output data (when measurements taken): Para 1=ΔRE; Para 2=I.
- History file CLOSED.
- GPIB output OFF.

Each of the default settings can be checked with a query command. For example, the query command **?ON** should get the response "00", which indicates that the Pol V/I ON mode is selected.

2.2. MAKING SINGLE V,I MEASUREMENTS

This example illustrates the setup of a polarization voltage for the potentiostat, the reading of the resulting ΔRE and I values, a current off-limit action resulting from a polarization update, the assignment of a new current limit, and a further polarization setup and result reading.

Write ECI:

"**PW**1;	Switch polarization ON. Polarization ON sequence takes approximately 1 second and goes from full standby to polarization voltage applied. By default, the initial polarization is 0V.
PV 1E-02;	Set polarization voltage to 10mV (=potential across RE1 and RE2, i.e. ΔRE).
GP1;	Select ASCII output, with the elapsed time.
TR0;	Set the DVM for single measurement trigger.
RU1;"	Command DVM to run. DVM makes a single measurement of ΔRE and I. (These are the output parameters selected by default).

Read ECI:	Read ΔRE, I, and the elapsed time. (45 bytes)

Write ECI:

"**PV**0.2;	Set polarization voltage to 200mV.
IL2;	Select current limit of 200µA.
RU1;	Command DVM to run. (Makes single measurement.)
Read ECI:"	Read updated values of ΔRE, I, and the elapsed time. (45 bytes)

By setting in a succession of increasing (or decreasing) values of polarization voltage, and measuring the result for each one, the data for a polarization curve can be obtained. Alternatively you can use the ECI sweep facility, as described in Sections 2.3 and 2.4.

2.3. USING THE STEPPED SWEEP FACILITY

This example illustrates how to set up a stepped polarization sweep, how to set up the history file to store the results, and how to read the results from the history file.

Write ECI:

“**BK**4;	Initialize the ECI.
PB9;	Select control loop bandwidth type J (= 8Hz).
RR4;	Select the 2mA current range. (standard resistor = 100Ω.)
IL3:	Select full-scale current limit of 2mA.
DL5;	Set sweep delay to 5 seconds.
SM4;	Define the number of sweep segments as 4.
SA0.4;	Define sweep voltage level A as 0.4V.
SB1.2;	Define sweep voltage level B as 1.2V.
SC-0.6;	Define sweep voltage level C as -0.6V.
SD1.2;	Define sweep voltage level D as 1.2V.
TE2;	Define the time per sweep step as 2 seconds.
VS0.1;	Define the volts per sweep step as 0.1V.
FS60;	Define the file size, i.e. the maximum number of results that the file is to hold. For the present example this is 60 results.
FL1;	Open the history file.
TR3;	Set the DVM for sweep synchronized measurements. (In step sweep this gives one reading for each step.)
DG3;	Select 3 X 9 digits for the DVM.
RG2;	Select 2V input range for the DVM.
PW1;	Select polarization ON.
SW2;”	Start a stepped sweep.

The present sweep will take about 1 minute and 44 seconds. Progress of the sweep can be checked by querying the sweep status with the **?ST** command. For sweeps of long duration the ECI can be programmed to issue a request for service (SRQ) at the end of a sweep.

Write ECI:

"**GP**1;	Set the GPIB for compressed ASCII output, with time.
VF2;”	List file, i.e. send contents of the history file to the GPIB.

Read ECI: Read 53 results from the history file.

2.4. USING THE RAMP SWEEP FACILITY

This example illustrates how to set up a ramp polarization sweep, how to set up the history file to store the results, and how to read the results from the history file.

Write ECl:

“**BK**4;	Initialize the ECI.
PB9;	Select control loop bandwidth type J (= 8Hz).
RR4;	Select the 2mA current range. (standard resistor = 100Ω.)
IL3;	Select full-scale current limit of 2mA.
DL5;	Set sweep delay to 5 seconds.
SM4;	Define the number of sweep segments as 4.
VA0.4;	Define sweep voltage level A as + 0.4V.
TA6;	Define time of segment A as 6 seconds.
VB1.8;	Define sweep voltage level B as + 1.8V.
TB2;	Define time of segment B as 2 seconds.
VC-2;	Define sweep voltage level C as -2V.
TC6;	Define time of segment C as 6 seconds.
VD-1.2;	Define sweep voltage level D as -1.2V.
TD 4;	Define time of segment D as 4 seconds.
FS50;	Define the file size, i.e. the maximum number of results that the file is to hold. For the present example this is 50 results.
FL1;	Open the history file.
TR3;	Set the DVM for sweep synchronized measurements. (In ramp sweep this gives continuous readings during the sweep only.)
DG3;	Select 3 X 9 digits for the DVM.
RG2;	Select 2V input range for the DVM.
PW1;	Select polarization ON.
SW1;"	Start a ramp sweep.

The present sweep will take about 20 seconds. Progress of the sweep can be checked by querying the sweep status with the **?ST** command. For sweeps of long duration the ECI can be programmed to issue a request for service (SRQ) at the end of a sweep.

Write ECI:

“**GP**1;	Set the GPIB for compressed ASCII output, with time.
VF2;	List file, i.e. send contents of the history file to the GPIB.
Read ECI:	Read the results from the history file.

2.5. OBTAINING CELL IMPEDANCE DATA (FREQUENCY SWEEP)

This example illustrates how to use a frequency sweep to get cell impedance data.

Write ECI:

“**BK**4;	Initialize the ECI.
PW1;	Switch polarization ON.
RR4;	Select the 2mA current range. (standard resistor=100Ω.)
IL3;	Select current limit of 2mA.
PV1.5;	Define the d.c. polarization voltage as 1.5V.
PI0;	Select a gain of X 1 for the a.c. input from the FRA.
BR1;"	Select bias reject ON. *DRE (= 1.5V) is measured, and subtracted from the output to the FRA.*

Write FRA:

“**TT**1	Initialize the FRA, when the default setup should be:

GENERATOR

- 100Hz Sinewave.
- Zero amplitude and bias.
- Frequency sweep OFF.
- Stop mode =freeze.

ANALYZER

- Auto-range input.
- Integration time = 0.1s.
- Analysis of fundamental frequency.
- Result source = Chan 2 ÷ Chan 1 = ΔRE ÷ I = cell impedance.
- Scaling OFF.
- History file ON (always).
- Result coordinates = *r, q*.
- GPIB output OFF.

AM1.2;	Set the amplitude of the FRA generator output to 1.2V.
MA2000;	Set the maximum sweep frequency to 2kHz.
MI1000;	Set the minimum sweep frequency to 1Hz.
GS20;	Define the number of frequency points in the sweep as 20.
SE1;	Set the sweep direction to UP.
IS1;	Set the analyzer integration time to 1 second.
RE;"	Set the analyzer to run continuously.

The frequency sweep in this example will now run for about 21 seconds, after which the FRA history file should contain 20 cell impedance results. *See overleaf.*

Write FRA:	
"**OP**2,1;	Switch on the FRA compressed ASCII output to the GPIB.
?FP0;	Query the FRA file population.
Read FRA:	File population should be 20 (results), as stated above.
Write FRA:	
FO;"	Command FRA file to output all results.
Read FRA:	Read the 20 impedance results from the FRA history file. (See Fig 2.3 for the locus of the values you should obtain.)

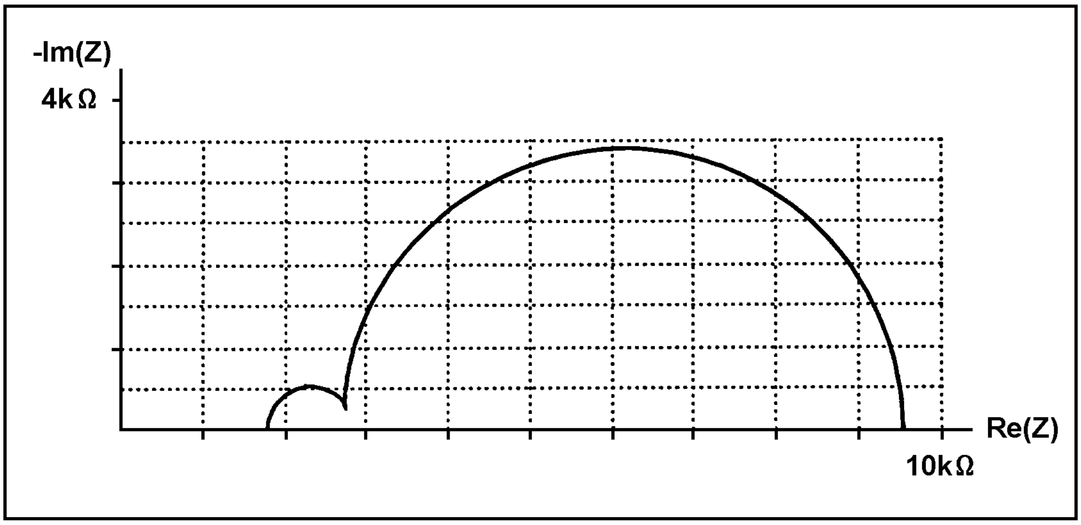


Fig 2.3 12861 Test Module Z plot.

3. GUIDE TO THE SI 1280 MEASUREMENT FACILITIES

Fig 2.4 gives a broad guide to the measurement facilities provided by the SI 1280. The block diagram indicates the general data flow, whilst the block numbers point (a) to the function summaries at either side of the diagram and (b) to a detailed description in the relevant chapter and section of the manual, e.g. "3,2" refers to Chapter 3, Section 2.

Using the SI 1280

ECI FACILITIES

3,2 D.C. polarization by potentiostat or galvanostat, with selectable Standby and ON modes.

3,3 Selectable control loop bandwidth, to ensure polarization stability with various types of cell.

3,4 Fixed range or autorange current sensing, with user defined current limit and off-limit action.

3,5 IR compensation, feedback or sampled. Compensates for the effect of parasitic resistance in three-terminal cells. (Can be used only with potentiostat.

3,6 Real part correction.Compensates for the effect of parasitc resistance in three-terminal cells. (Can be used with potentiostat or galvanostat).

3,7 Selectable conditioning for the ECI output, consisting of voltage and current bias rejection, low-pass filtering, and voltage and current amplification.

3,8 Ramped sweep cycle or stepped sweep cycle, for obtaining polarization curve data. Ramp and sweep yields polarization resistance.

3,9 Digital voltmeter control, with selectable scale length, input range, measurement trigger, averaging and nulling.

3,10 Control of output parameters, history file and GPIB output.

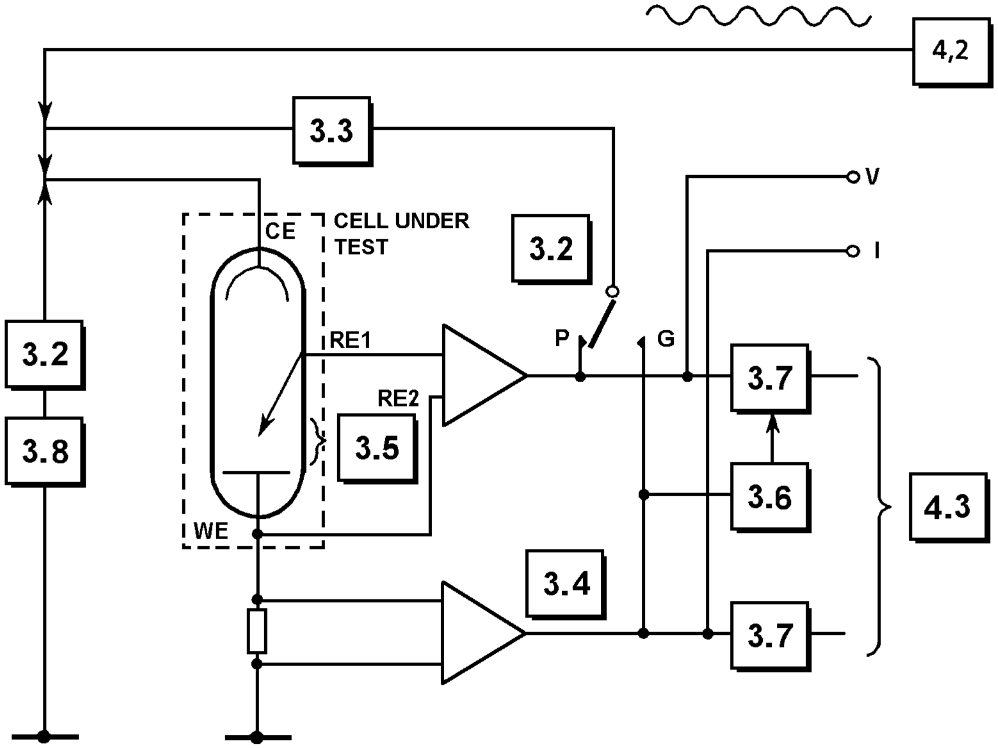


Fig 2.4 *SI 1280 Measurement facilities*

FRA FACILITIES

4,2 A.C. generator, with the following facilities:-

- Sine, square, or triangular waveform.
- User-defined frequency, 1mHz to 20kHz
- User-defined amplitude, up to 10V rms. (5.11Vrms max. for triangular waveform.)
- User-defined bias, +/-10V.
- Frequency sweep

4,3 Analyser, with the following facilities:-

- User-defined integration time, for variable degree of noise and harmonic rejection.
- Fixed range or autorange input
- A.c. measurements up to eighth harmonic of the generator frequency.

4,4 Data output control, with the following facilities:-

- Selection of result source, scaling and coordinates.
- History file control
- GPIB output control

Note that the numbers in the boxes refer to the relevant sections in chapters 3 and 4. For example, “3,2” refers to Chapter 3 Section 2

TYPICAL APPLICATION OF THE SI 1280

Figures 2.5 and 2.6 show the results of a study of the anodic behaviour of pure iron in a sulphuric acid medium. This study, which is frequently used to obtain practical results related to the dielectric properties of the metal oxide, electrochemical machining, etc, is described by Claude Gabrielli in Technical Report No. 004/83t. The procedure for obtaining results such as this, with the SI 1280, is outlined below.

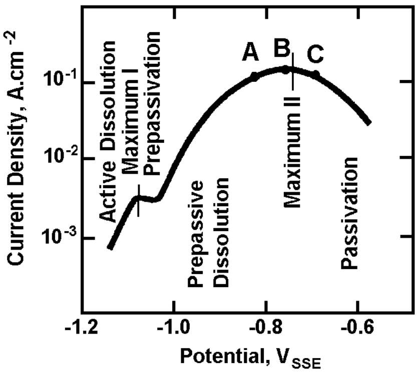


Fig 2.5 *Steady-state current-voltage curve for pure iron in sulphuric acid aqueous medium (pH = 5); disc electrode (A = 0.2cm^2) rotating at 1600 rpm.*

Since the current-voltage curve is shown to have both a positive and a negative slope (Figure 2.5), which depends on the applied polarization, it follows that the ECI should operate as a potentiostat. And, since anodic behaviour is being studied, presumably with a three-terminal cell, then IR compensation can be applied. To obtain the current-voltage curve the polarization voltage V_{SSE}* is stepped from -1.15V to -0.58V in user-defined increments and the resulting I,V values are stored in the ECI history file.

The three impedance spectra are obtained by setting the FRA to make a swept frequency analysis, between 0.1Hz and 10kHz, at points A, B, and C on the current-voltage curve. At these points a small-amplitude swept-frequency signal from the FRA generator is superimposed on the d.c. polarization applied by the ECI. Signal conditioning to remove the d.c. bias is applied at the ECI output and the resulting pure a.c. signal is analysed by the FRA to obtain the impedance data. For each frequency sweep the results are stored in the FRA history file.

When all measurements have been made the results are read from the history files by the controller, processed, and output to a plotter. The impedance spectra obtained may be something like those shown in Figure 2.6.

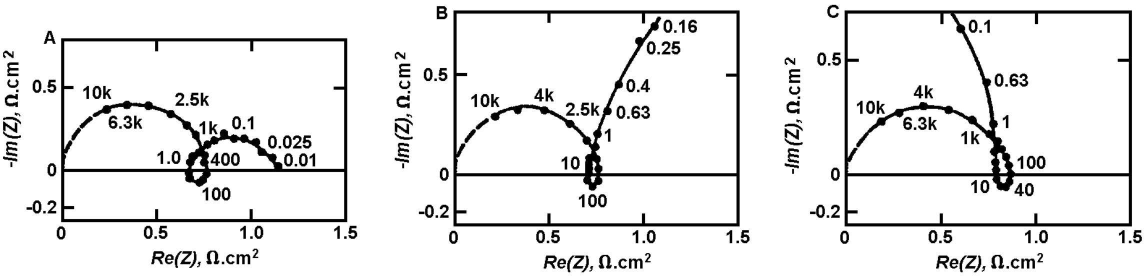


Fig 2.6 Impedance spectra for pure iron in sulphuric acid aqueous medium (pH = 5); disc electrode (A=0.2cm^2) rotating at 1600 rpm. Dc. polarization as shown in Figure 2.5.

tObtainable from Solartron. *Saturated sulphate electrode.

Chapter 3

ECI Command Functions

1. ECI STRUCTURE

The Electro-Chemical Interface (ECI) provides for the d.c. polarization of an electrochemical cell, and allows an a.c. perturbation signal to be applied to the cell from the FRA.

To give a broad idea of its operation, the ECI is represented, in Fig 3.1, as a series of functional blocks. A brief description of each block and its relationship with other blocks is given below. Reference is given, throughout, to the ECI control information in the remainder of the chapter: in the text this is done by means of superscripted numbers which point to the appropriate section (e.g. the "^{2}" which follows **POLARIZATION** ^{2} means *"for details of polarization control, see Section 2).*

To provide a stable polarization source for the cell the ECI is operated either as a **potentiostat** ^{2} or as a **galvanostat** ^{2}. By virtue of negative feedback the potentiostat holds the applied potential constant, whilst the galvanostat holds the cell current constant. The control loop for this consists of **POLARIZATION** ^{2}, **BANDWIDTH** ^{3} and the cell, together with ∆RE for the potentiostat or **CURRENT SENSING** ^{4} for the galvanostat.

Since the polarization feedback loop includes the cell, instability may be caused by the reactive components (mainly capacitive) of the cell impedance: these can produce positive feedback at the higher frequencies and thus cause the system to oscillate. To compensate, a reduced ***polarization bandwidth*** ^{3} can be selected by the user.

To obtain the optimum resolution in the measurement of small or large currents, eight current ranges are provided. ***Current range selection*** ^{4} can be made automatically or any fixed range can be selected. To ensure that the cell current remains at a safe level, a ***current limit*** ^{4} and an ***off-limit action*** ^{4} can be selected by the user.

When the ECI is used as a potentiostat the effect of parasitic resistance in a three- terminal cell (or the effect of the electrolyte resistance in a two-terminal cell) can be compensated for by **IR COMPENSATION** ^{5}. This ensures accurate definition of the double-layer polarization voltage. When the ECI is used as a galvanostat, however, the effect of parasitic resistance can still be corrected, but, in this case, with **REAL PART CORRECTION** ^{6}. This adjusts the measured cell voltage to offset the effect of the parasitic resistance and thus enables the FRA input sensitivity to be optimized in cell impedance measurement. (Real part correction can also be used when the ECI is acting as a potentiostat.)

Any dc offsets at the ECI output to the FRA can be corrected for by **VOLTAGE CONDITIONING** ^{7} and **CURRENT CONDITIONING** ^{7}. This, again, allows the FRA to be used at optimum input sensitivity, which minimizes the effect of noise.

In addition to being set to one precisely defined value, cell polarization can be ***ramped*** or ***stepped*** with the **SWEEP** ^{8} function. This facilitates the generation of potentiodynamic or potentiostatic current-voltage curves. Note that a ramp sweep provides for direct measurement of the cell polarization resistance.

A precision **DIGITAL VOLTMETER** ^{5} allows the various parameters of cell d.c. polarization to be measured, for output to the GPIB. Any two parameters, at any one time, can he measured, in accordance with the setting of **PARAMETER SELECT** ^{10}. Measurement results can be output in compressed ASCII or binary format, with optional time information. To save time in data transmission, up to 450 results can be stored in the ECI **HISTORY FILE** ^{10} and output to the GPIB consecutively.

Remote commands to the ECI are interpreted by the **MICROPROCESSOR** and routed to the appropriate functional block. These commands are presented, throughout the chapter, in tabular form. (See Chapter 2, Section 1.4 for the real number format.) The default settings are indicated by underlining the associated command. Parametric values default to zero, except where otherwise stated.

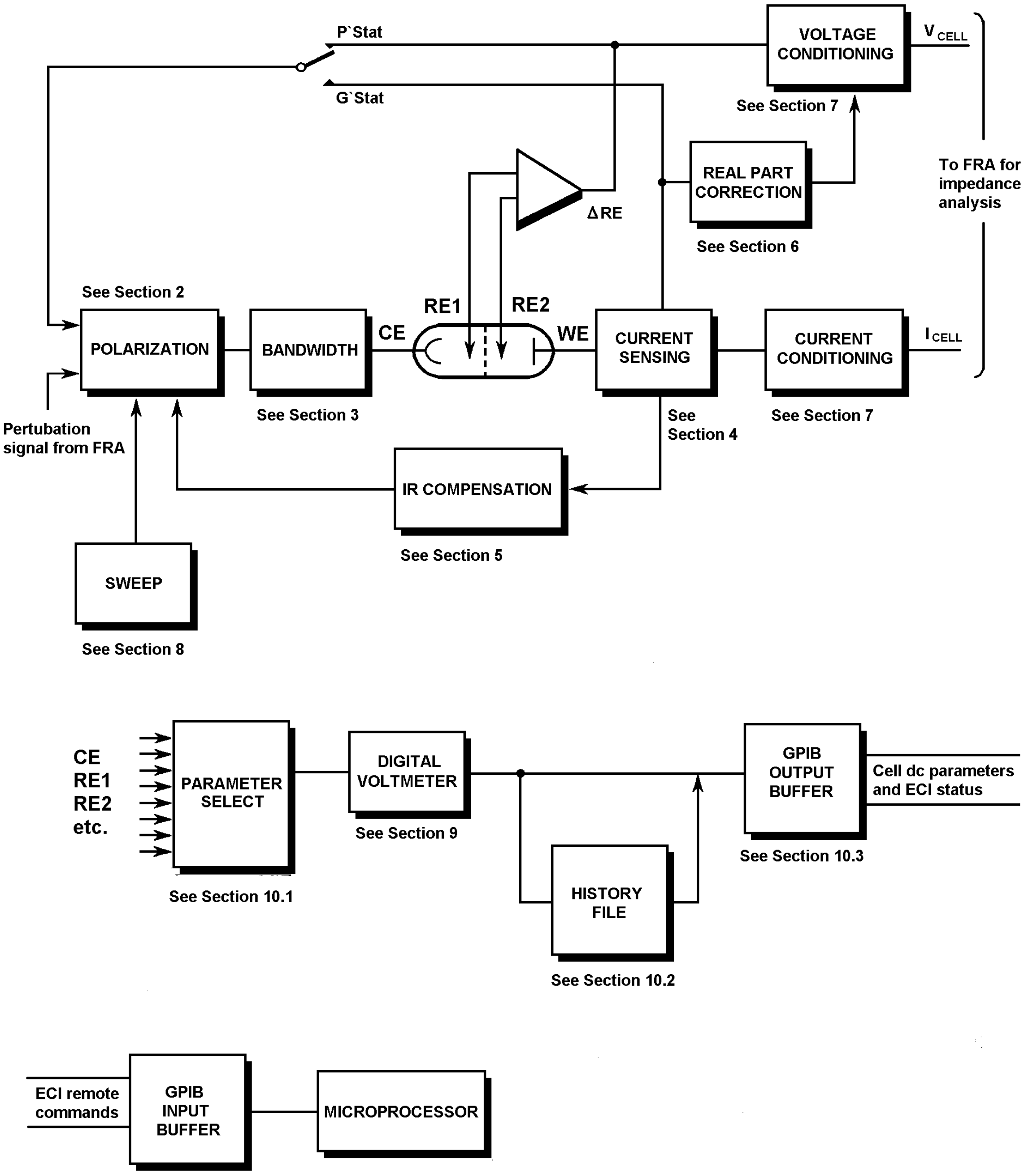


Fig 3.1 ***ECI functional blocks*.**

2. CELL POLARIZATION

The ECI may be used either as a potentiostat or as a galvanostat, to provide d.c. polarization for an electrochemical cell. In either mode, the dc polarization level may have an ac perturbation signal from the FRA added to it. The commands used to select the type of polarization, and to define the magnitude, are listed in Table 3.1.

Table 3.1 Cell Polarization

Command	Polarization
<u>PO</u><u>0</u> **PO**1	Sets ECI to operate as a potentiostat. Sets ECI to operate as a galvanostat.
PV*F* **PC***F*	Defines the d.c. potential that is to be applied across RE1 and RE2 for the potentiostat. (*F*=-14.5V to + 14.5V)* Defines the direct current that is to flow through the cell for the galvanostat. (*F*=-2A to +2A)*
PI0 **<u>PI</u>**<u>1</u>	An a.c. signal from the FRA is added to the polarization selected by **PV***F* or **PC** *F*, at a gain of X 1. An a.c. signal from the FRA is added to the polarization selected by **PV***F* or **PC** *F*, at a gain of X 0.01 (-40dB).
PW1 **<u>PW</u>**<u>0</u>	Selects polarization ON. (ECI assumes ON mode.) Selects polarization OFF. (ECI assumes standby mode.)

F= real number (See Chapter 2, Section 1.4)

* Note that the potentiostat and galvanostat ranges of the 1280A are -12.8V to + 12.8V and -1A to + 1A.

Cell polarization may thus be set to one precisely defined level, or varied in precisely defined steps to obtain an accurate polarization curve. At any point on this curve the cell impedance may be determined by applying an a.c. perturbation signal from the FRA and analysing the result. (See Chapter 4.) Note that polarization curves may also be conveniently obtained by using the sweep facility. (See Section 8.)

A further command (**PW***I*) switches the polarization on and off. To avoid the cell being subjected to high voltages or currents, in the short period before polarization stabilizes, the ECI goes through a controlled polarization sequence. This sequence starts from a selectable *standby state* and progresses to a selectable *on mode*. See Sections 2.1 through 2.3 below.

2.1. STANDBY STATE

Before the cell is polarized - and after, when polarization is switched off - the cell connections within the ECI are set to "standby". Either of two standby states may be selected: “full standby", in which electrodes CE, RE1 and RE2 are isolated, and "half standby”, in which only CE is isolated. Full standby is the default state. Should you require half standby, this must be selected prior to polarizing the cell. The commands for selecting either standby state are listed in Table 3.2, together with the valid measurements that may be made. (Other measurements may be made, but are invalid.)

Table 3.2 Standby State

Command	Standby State	Valid Measurements
<u>BY</u><u>0</u>	Full stand-by.	POL, ΣPOL, CE.
BY1	Half stand-by.	RE1, RE2, ΔRE, ΔRE-Bi, POL, ΣPOL, CE, I, I-Bi.

2.2. POLARIZATION ON MODE

On completion of the "polarization on" sequence (see Section 2.3) the cell may be polarized either "at rest" or at a previously defined level. This is known as the polarization "on mode" and it is selected by the command ON*n*. See Table 3.3.

Table 3.3 Polarization ON Mode

Command	On Mode
<u>ON</u><u>0</u>	Prepares the ECI for the **Pol V/I** mode. When polarization ON is selected the cell is polarized at its previously defined level. By default, this is zero volts for the potentiostat and zero current for the galvanostat; otherwise, it is the level defined by the last **PV***F* or **PC***F* command (see Table 3.1).
ON1	Prepares the ECI for the **Rest V/I** mode. When polarization ON is selected the cell is held at its rest potential (potentiostat) or at zero current (galvanostat) until the polarization is defined by a **PV***F* or **PC***F* command.

2.3. POLARIZATION ON SEQUENCE

When it receives a polarization ON command (**PW**1) the ECI goes through a controlled polarization sequence. Depending on the standby state and ON mode selected some steps may be missed out, as shown in Table 3.4.

Table 3.4 Mode Polarization ON Sequence

Sequence Followed				Full Sequence
Pol V/I & Full Standby	Pol V/I & Half Standby	Rest V/I & Full Standby	Rest V/I & Half Standby	
1	-	1	-	1. Full standby. (Initial state.)
2	2	2	2	2. Half standby. (RE1 and RE2 connected.)
3	-	3	-	3. One second pause. (RE1 and RE2 settle.)
4	4	4	4	4. ΔRE Measured. (Cell's rest potential.)
5	5	5	5	5. POL V set to rest potential and bandwidth Type J* selected.
6	6	6	6	6. All electrodes connected.
7	7	7	7	7. 40ms pause (for polarization to stabilize).
8	8	8	8	8. Selected bandwidth is set.
9	9	-	-	9. Selected polarization applied to cell.

*For the G STAT mode POL I is set to zero and bandwidth Type C is selected.

3. CONTROL LOOP BANDWIDTH

Various bandwidths may be selected for the cell polarization control loop. This is to ensure the stable operation of the ECI, as a potentiostat or galvanostat, in the measurement of various cells. The bandwidth commands and the bandwidths selectable are listed in Table 3.5.

Note (from footnotes of Table 3.5) that some bandwidths are reduced when "limit" or “cutout" is selected for the overload type. (See Section 4.2.) Note also that the bandwidths quoted are approximate and are based on the unity gain cell configuration shown in Fig 3.2.

Table 3.5 Control Loop Bandwidth

Command	**Bandwidth**	
SY0	Bandwidth limit ON.	
SY1	Bandwidth limit OFF.	
	Galvanostat:	
GB0	Type A	150kHz**
GB1	Type B	100kHz**
GB2	Type C	>7.5kHz***
	Potentiostat:	
PB0	Type A	600kHz*
PB1	Type B	360kHz*
PB2	Type C	>1MHz*
PB3	Type D	> 600kHz*
PB4	Type E	24kHz*
PB5	Type F	8kHz
PB6	Type G	2.4kHz
PB7	Type H	800Hz
PB8	Type I	80Hz
PB9	Type J	8Hz

*12kHz when limit or cutout selected for O/L type.
**25kHz when limit or cutout selected for O/L type. Stable with capacitive cells when standard resistor ≤100Ω.
*** 5kHz when limit or cutout selected for O/L type.

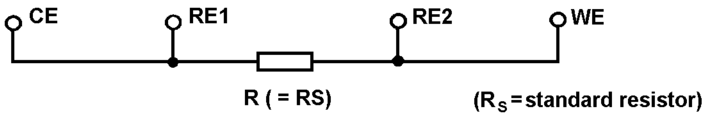


Fig 3.2 ***Unity gain “cell".***

When in doubt as to the stability of the cell polarization, start with a low bandwidth, e.g. type J for the potentiostat, and increase the bandwidth for successive measurements until an unstable point (if any) is reached. Then reduce the bandwidth sufficiently to achieve stable operation. For particularly unstable systems, of course, you may have to reduce the bandwidth well below the stable operating point and start again.

4. CURRENT SENSING

The value of the current flowing through an electrochemical cell is measured by sensing the voltage developed across a standard resistor connected in series with the cell, and dividing this voltage by the resistance value. (Fig 3.3) A *positive* current is said to be flowing when it passes into the counter electrode (CE) and out of the working electrode (WE).

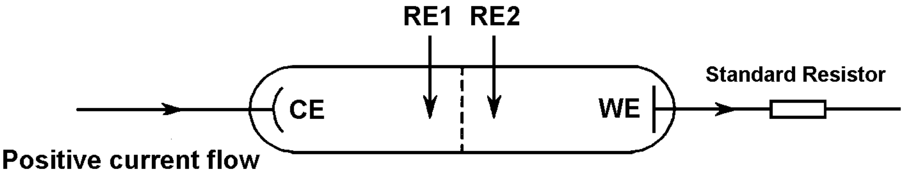


Fig 3.3 ***Four-terminal electrochemical cell***

4.1. CURRENT RANGE

The current range of the ECI is determined by selecting a standard resistor which develops 200mV at full scale current. See Table 3.6. This selection may be fixed, or made automatically.

Table 3.6 Standard Resistor Selection

Command	**Standard Res. Value**	**Full-Scale Current** (At 200mV)
RR0	Auto-range	
RR1	0.1Ω	2A
RR2	1Ω	200mA
RR3	10Ω	20mA
RR4	100Ω	2mA
RR5	1kΩ	200µA
RR6	10kΩ	20µA
RR7	100kΩ	2µA
RR8*	1MΩ	200nA

*The 200nA range is not available on the 1280A.

On auto-range (RRO) an appropriate standard resistor is switched in automatically, to suit the current presently being measured. However, slight discontinuities occur in the cell drive whenever the range changes: so, with sensitive cells, it is advisable to use a fixed range. The following limitations also apply:

- Auto-range is not suitable for a.c. work, i.e. with the FRA.
- Auto-range is not allowed with the following facilities in action: current bias rejection, IR compensation or real part correction.
- Auto-range operates by sensing the cell current and connecting the appropriate standard resistor just before a measurement is made. Therefore it is possible for a rapidly rising current to be measured on too low a range, resulting in an off-limit condition.

4.1.1. Current Limit

The current limit defines the highest current range to be used in autorange mode. Should the measured current exceed the full scale value of this range then an off-limit action is triggered. Any one of three off-limit actions may be selected, as described in Section 4.2.

The commands and associated values for the current limit are listed in Table 3.7

Table 3.7 Current Limit Selection

Command	**Full-Scale Current** (At 200mV)
IL0	2µA
IL1	20µA
IL2	200µA
IL3	2mA
IL4	20mA
IL5	200mA
IL6	2A

4.2. CURRENT OFF-LIMIT ACTION

An off-limit action is taken when the cell current exceeds:

(a) the full-scale value of a fixed range (by more than 25% approx.) or

(b) the current limit value in auto-range.

The action to be taken is selected with an **OL**n command. (See Table 3.8.) Note that an error code is generated also. (See Appendix B.)

Table 3.8 Current Off-limit Action

Command	**Overload Action**
OL0	Cut-out. The ECI goes to the "stand-by" state and the cell current is cut off completely.
OL1	Limit. The cell current limits at the full-scale value of a fixed range (or the I LIMIT value).
OL2	No limit. The cell current is uncontrolled and may rise to >±2A*. **Caution:** Some cells may be damaged by an excessive current flow.

* In the 1280A the cell current may rise to > ± 1A.

5. IR COMPENSATION

IR compensation may be used only with the ECI in potentiostat mode. It compensates for the voltage drop across the cell parasitic resistance (Rp) and allows the voltage across the cell double-layer impedance (Z_D) to approach the user-defined polarization voltage. (Rp is the resistance of the electrolyte between the reference and working electrodes. See Fig 3.4.)

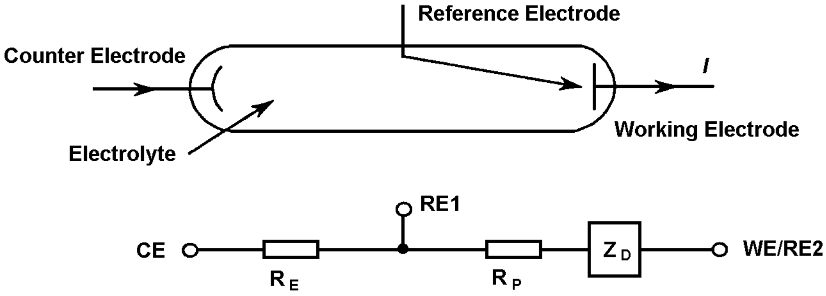


Fig 3.4 *Three-terminal electrochemical cell and equivalent circuit*.

There are two types of IR compensation, "feedback" and "sampled". (See Sections 5.1 and 5.2 below.) The commands for selecting the compensation type and compensation ON/OFF are listed in Table 3.9.

Table 3.9 IR Compensation Type and ON/OFF

Command	Function
CT0 **CT**1	Selects feedback IR compensation. Selects sampled IR compensation.
CC0 **CC**1	IR compensation OFF. IR compensation ON.

5.1. FEEDBACK IR COMPENSATION

Feedback IR compensation requires the user to specify an estimated value for the parasitic resistance (Rp). To compensate for the voltage drop across Rp the ECI measures the cell current (I) and increases the polarization voltage by the value I X Rp. If the value of Rp has been estimated correctly this results in the double layer voltage stabilizing at a value close to the user-defined polarization voltage. The command for entering the estimated value of Rp is shown in Table 3.10.

Table 3.10 Feedback IR Compensation

Command	Function
IC*F*	Defines the value of the parasitic resistance Rp (0Ω to 1MΩ).

F= real number (See Chapter 2, Section 1.4)

The measured value of ΔRE with I X Rp subtracted is available as ΔRE-Bi. Using this, the double layer impedance can be determined from:

$$Z_D = (\Delta \text{RE-Bi}) \div \text{I}$$

Note that, when bias reject is selected, Bi is the sum of I X Rp and the d.c. voltage measured at the ECI output. This means that feedback IR compensation and bias reject can be used together for optimum measurement accuracy. (Bias reject allows a more sensitive range to be used on the FRA - see Section 7.1.)

A simple way to obtain an initial value in the estimation of Rp is to measure the cell impedance with the FRA, using a high frequency signal. For such a frequency Z_D is minimal. Therefore, the potential ΔRE is developed across Rp alone and the impedance measurement ΔRE ÷ I is a close approximation to Rp.

5.2. SAMPLED IR COMPENSATION

Sampled IR compensation periodically interrupts the cell current. During the current off time the IRp drop disappears immediately and allows the double-layer impedance voltage (which decays relatively slowly) to be sampled and held. The ECI feedback circuit monitors this voltage and maintains it at the user-defined polarization value. The commands for feedback IR compensation are listed in Table 3.11.

Table 3.11 Sampled IR Compensation Command

Command	Function
IN*F*	Defines the cell current *off* time (26.6μs to 1.36ms).
IP*I*	Defines the cell current *off*. *on* ratio (1:*I*) where *I* = integer 1 to 255.
<u>RO</u><u>0</u>	ΔRE and FRA measure actual waveforms.
RO1	ΔRE and FRA measure sampled levels.

F= real number (See Chapter 2, Section 1.4)

Fig 3.5 shows an example of the cell voltage and current waveforms. Either the actual cell waveforms or the sampled and held ones may be selected for ΔRE measurement and output to the FRA.

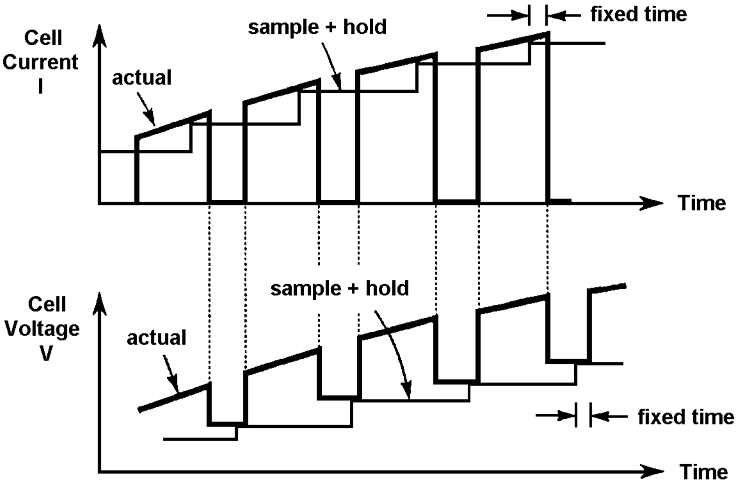


Fig 3.5 *Example waveforms for sampled IR compensation.*

The cell impedance, Z_D, can he determined as follows:

1. Select sampled level measurements with command RO1 and measure the zero- current value of ΔRE.
2. Select actual waveform measurements with command ROO and measure the average current.

   Alternatively, estimate the average current by using sampled level measurements and a correction factor. The correction factor can be calculated from the off-on ratio of the cell waveform, as follows:

Correction factor = 1 / (off-on ratio + 1)

3. Divide the value of ∆RE measured in step 1 by the average current value measured in step 2.

NOTE: With ac impedance measurements the frequency of the a.c. perturbation signal is limited to about 10% of the interrupt frequency.

6. REAL PART CORRECTION

Real part correction is an alternative to IR compensation and it is applicable both to potentiostatic and galvanostatic measurements. Whereas IR compensation is applied to the cell itself (via the polarization control loop), real part correction simply adjusts the measured cell voltage to offset the effect of parasitic resistance (Rp). Real part correction is thus a post-measurement function and does not effect polarization stability.

The commands for real part correction are listed in Table 3.12.

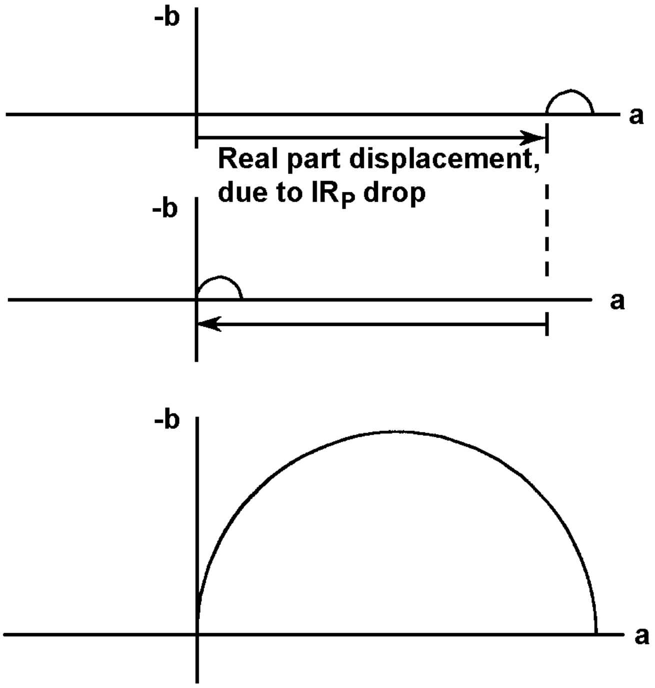
Table 3.12 Real Part Correction

Command	Function
CC0 **CC**2	Real part correction OFF. Real part correction ON.
RP*F*	Defines the value of the parasitic resistance Rp (0Ω to 1MΩ).

F= real number (See Chapter 2, Section 1.4)

To set up real part correction you are required to enter an estimated value for Rp, with the command RP*F*. For three terminal measurements this value is multiplied by the measured value of cell current to obtain the IRp drop and the result subtracted from the measured cell voltage. For two-terminal measurements the same approach is used to eliminate the voltage drop across the electrolyte resistance R_E.

Real part correction is useful when measuring a.c. impedance in cases where the voltage across the parasitic resistance is significant compared to the double-layer voltage being measured. As shown in Fig 3.6, the IRp drop displaces the cell impedance plot along the positive real axis, which lowers the measurement accuracy. Applying real part correction shifts the origin towards the imaginary axis, thus allowing a more sensitive FRA range to be selected and the measurement accuracy to be improved.



1. Displacement of impedance plot, due to IRP drop, necessitates a low sensitivity FRA scale
2. Real part correction shifts origin of impedance plot
3. More sensitive FRA scale selected.

Fig 3.6 *Effect of real part correction.*

7. ECI OUTPUT CONDITIONING

The measured cell voltage and current outputs of the ECI can be conditioned so that useful information is emphasised in relation to noise and signal offsets. The facilities available for this are listed in Table 3.13.

Table 3.13 ECI Output Conditioning Facilities

Command	Function
VT0	Voltage bias reject, auto.
VT1	Voltage bias reject, fixed.
VR*F*	Fixed rejection voltage. (F = 0V to ± 14.5A)**
IT0	Current bias reject, auto.
IT1	Current bias reject, fixed.
IR*F*	Fixed rejection current. (F = 0A to ± 2A)**
BR0	Bias reject OFF.
BR1	Bias reject ON. For auto bias reject the signal bias is measured, stored, and subtracted from all subsequent measurements. For fixed bias reject a user-defined value is subtracted.
FI0	Filter OFF.
FI1	Filter ON. A 10Hz low-pass filter is switched into the measured voltage and current outputs.*
VX0	Voltage amplification off.
VX1	X10 voltage amplification.*
IX0	Current amplification off.
IX1	X10 current amplification.*

F= real number (See Chapter 2, Section 1.4)

*Affects FRA measurements only.
**In the 1280A the range of fixed rejection voltage is 0V to ± 12.8V and the range of fixed rejection current is 0A to ± 1A.

7.1. VOLTAGE AND CURRENT BIAS REJECTION

Bias rejection may be used to cancel out the dc component of the measured voltage and current outputs of the ECI. This allows a more sensitive FRA range to be used and thus improves the measurement accuracy.

The level of bias rejection may be entered automatically or as a fixed value. Note the difference in action of the **BR**1 command. With automatic bias rejection this command starts a single measurement: the dc bias is detected, entered as the bias reject value, and subtracted from all subsequent measurements. Should the signal bias be prone to drift then auto reject should be actioned periodically. With fixed bias rejection **BR**1 simply applies the previously defined bias value.

7.2. LOW PASS FILTER A

A 10Hz low-pass filter may be switched into the measured voltage and current outputs. This reduces wideband noise in low frequency and dc measurements and allows the more sensitive FRA ranges to be used for improved measurement accuracy. Do not filter a signal of frequency greater than 5Hz, however, otherwise it will be attenuated.

7.3. VOLTAGE AND CURRENT AMPLIFICATION

Measured voltage and current signals of very low amplitude may be amplified by a factor of ten.

8. SWEEP

Sweep allows the d.c. polarization of a cell to be swept between defined limits, either as a smooth ramp or as a series of steps. A cycle of four sweep "segments" is defined by the user and this cycle is repeated in accordance with a user-defined number of segments (1 to 99,999). The common sweep commands are listed in Table 3.14.

Table 3.14 Sweep Definition

Command	Function
DL*F*	Delay in seconds (*F* = 0 to 100,000s)
SM*F*	Number of segments (*F* = 1 to 99,999; default = 2)
<u>**OF**0</u> **OF**1	Off mode = standby Off mode = freeze
<u>**SW**0</u> **SW**1 **SW**2	Stop sweep. Start ramp sweep. Start stepped sweep.
?ST	Returns the sweep status. (see Section 8.4.)

F= real number (See Chapter 2, Section 1.4)

Swept polarization may be used with either the potentiostat or the galvanostat. Voltage and current levels may both be stored, but only one set is used at a time, p'stat or g'stat.

The off mode, selected by command **OF***I*, determines whether the cell polarization is maintained (freeze) or disconnected (standby) when a sweep ends or is stopped by the user. (The off mode is not selectable on the 1280A.)

8.1. RAMP SWEEP CYCLE

A ramp sweep provides for dynamic polarization of a cell and the elements of the sweep cycle are shown in Fig 3.7. The ramp definition commands are listed in Table 3.15. These are used in conjunction with the sweep definition commands listed in Table 3.14.

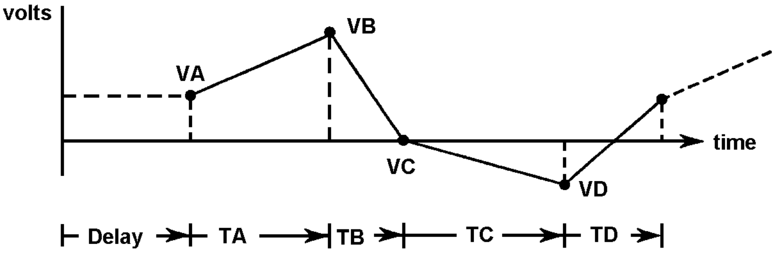


Fig 3.7 *Ramp sweep cycle (potentiodynamic polarization).*

Table 3.15 Ramp Sweep: Segment Definition

Command	Function	
VA*F*	Voltage level V_A	(*F*= 0 to ± 14.5V)*
JA*F*	Current level I_A	(*F*= 0 to ± 2A)*
TA*F*	Segment time T_A	(*F*= 10ms to 100,000s)
VB*F*	Voltage level V_B	(*F* = 0 to ± 14.5V)*
JB*F*	Current level I_B	(*F*= 0 to ± 2A)*
TB*F*	Segment time T_B	(*F*= 10ms to 100,000s)
VC*F*	Voltage level V_C	(*F* = 0 to ± 14.5V)*
JC*F*	Current level I_C	(*F*= 0 to ± 2A)*
TC*F*	Segment time T_C	(*F* = 10ms to 100,000s)
VD*F*	Voltage level V_D	(*F* = 0 to ± 14.5V)*
JD*F*	Current level I_D	(*F*= 0 to ± 2A)*
TD*F*	Segment time T_D	(*F* = 10ms to 100,000s)

F= real number (See Chapter 2, Section 1.4)

*In the 1280A the voltage level range is 0V to ± 12.8V and the current level range is 0A to ± 1A.

A ramp sweep is started with the **SW**1 command, and the sweep sequence is:

1. The delay allows the cell to settle at the starting polarization level.
2. Cell polarization ramps linearly to the second polarization level over time TA (first segment), to the third polarization level over time TB (second segment), and so on, until the return to the first polarization level over time TD (fourth segment). Note that the segment times are shared by the voltage and current sweeps. With sync. trigger selected, polarization parameters are measured continuously.
3. The ramp sweep cycle is repeated, and continues to be repeated until the last segment is completed. Then, depending on the off mode selected, the ECI is either switched to the standby state, or it maintains cell polarization at the final level. The final level can be any of the polarization levels specified and depends on the number of segments specified (i.e. the sweep can stop part-way through a cycle).

Note: A sweep may be stopped anywhere before the end, with the command **SW**0.

(In the 1280A the off mode is not selectable. At the end of a sweep the ECI is set to standby. The polarization value within the ECI remains at the final level.)

CAUTION: To avoid damage to sensitive cells at the start of a polarization sweep, set the ON mode to “rest V/I". (See Section 2.2.)

8.2. STEPPED SWEEP CYCLE

The elements of a stepped sweep cycle are shown in Fig 3.8. The commands for defining the stepped sweep segments are listed in Table 3.16. These are used in conjunction with the sweep commands listed in Table 3.14.

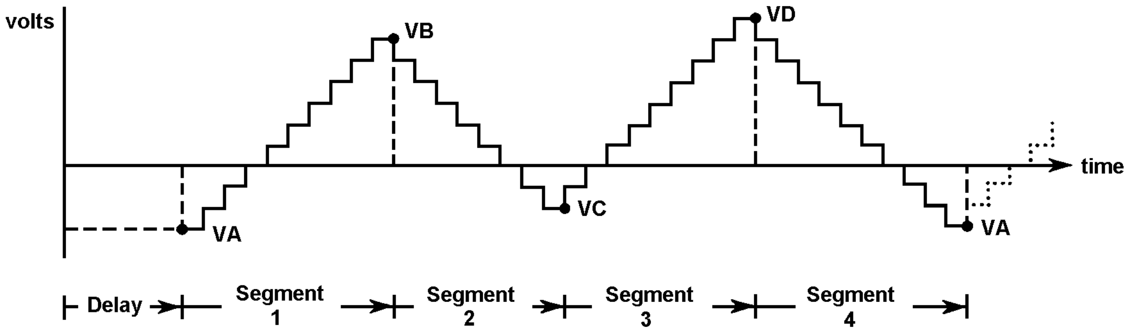


Fig 3.8 *Stepped sweep cycle (potentiostatic polarization).*

Table 3.16 Stepped Sweep: Segment Definition

Command	Function	
SA*F*	Voltage level V_A	(*F*= 0 to ± 14.5V)*
KA*F*	Current level I_A	(*F*= 0 to ± 2A)*
SB*F*	Voltage level V_B	(*F*= 0 to ± 14.5V)*
KB*F*	Current level I_B	(*F*= 0 to ± 2A)*
SC*F*	Voltage level V_C	(*F*= 0 to ± 14.5V)*
KC*F*	Current level I_C	(*F*= 0 to ± 2A)*
SD*F*	Voltage level V_D	(*F*= 0 to ± 14.5V)*
KD*F*	Current level I_D	(*F*= 0 to ± 2A)*
TE*F*	Time per step	(*F* = 10ms to 100,000s)
VS*F*	Volts per step	(*F* = 5µV to 29.0V)*
IS*F*	Current per step	(*F* = 5pA to 4A)*

F= real number (See Chapter 2, Section 1.4)

*In the 1280A the voltage level range is 0V to ± 12.8V and the current level range is 0A to ± 1A. The other ranges for stepped sweep are: time per step = 10ms to 100,000s, volts per step = 100µV to 25.6V, and current per step = 1nA to 2A.

A stepped sweep is started with the **SW**2 command, and the sweep sequence is:

1. The delay allows the cell to settle at the starting polarization level.
2. Cell polarization is incremented or decremented in user-defined steps, and progresses in accordance with the user-defined voltage levels. Note that the time/step value is shared by stepped voltage and stepped current sweeps. With sync. trigger selected, polarization parameters are measured at each step.
3. The stepped sweep cycle is repeated, and continues to be repeated until the last segment is completed. Then, depending on the off mode selected, the ECI is either switched to the standby state, or it maintains cell polarization at the final level. The final level can be any of the polarization levels specified and depends on the number of segments specified (i.e. the sweep can stop part-way through a cycle). **Note:** A sweep may be stopped anywhere before the end, with the command **SW**0.

(In the 1280A the off mode is not selectable. At the end of a sweep the ECI is set to standby. The polarization value within the ECI remains at the final level.)

CAUTION: To avoid damage to sensitive cells at the start of a polarization sweep, set the ON mode to “rest V/I”. (See Section 2.2.)

8.2.1. Step Size Limits

Limits are imposed as to the minimum values which may he entered for step definition. Minimum volts/step and current/step values (Table 3.17) depend on the maximum excursion from the first polarization level, whilst the minimum time/step values (Table 3.18) depend on the measurement resolution.

Table 3.17 Minimum Volts/Step Values

Maximum Excursion (from first polarization level)		**Minimum Volts/Step** (or I_{STEP} X Rs)
$V1 \leq 20mV$	(or $I1 \times R_S \leq 20mV$)	5µV
$20mV < V1 \leq 200mV$	(or $20mV < I1 \times R_S \leq 20mV$)	50µV
$200mV < V1$	(or $200mV < I1 \times R_{S)}$	100µV

Should an attempt be made to enter a polarization step value that is too small, two error codes are made available: "28", which calls for a minimum step of 100µV, and “29”, which calls for a minimum step of 50µV.

Table 3.18 gives the minimum permitted values for time per step with the DVM trigger set to "sync". Error code 52 is made available if an attempt is made to enter shorter times.

Table 3.18 Minimum Time/Step Values

Number of Digits	**Minimum Time/Step***
5 X 9	2.2s
4 X 9	800ms
3 X 9	500ms
Fast** 3 X 9	100ms

*In the 1280A the minimum Time/Step values are: 2.15s, 750ms, 425ms, and 65ms.

**Fast measurements rely on setting the DVMs for their optimum measurement rate, as follows:

- Use a fixed DVM input range.
- Use a fixed standard resistor.
- Set DVM drift correct OFF.

8.3. COMMANDS NOT ALLOWED DURING SWEEP

The following commands are not allowed whilst a sweep is in progress. Error code 51 is made available in each case.

I Measure: Resistor and I Limit (galvanostat), **DVM**: Averaging, **Sweep**: all settings.

Action: P'stat/G'stat, Bias Reject.

8.4. SWEEP STATUS

The status of a sweep may be queried with the command **?ST**. The possible responses to this command are as follows:

0 = Sweep not running. 1 =Sweep initializing. 2 = Sweep delay running.
3 = Segment 1 running. 4 =Segment 2 running. 5 = Segment 3 running.
6 = Segment 4 running.

9. DIGITAL VOLTMETER CONTROL

The ECI contains two digital voltmeters (DVMs) which measure the various parameters of cell dc polarization. One DVM is dedicated to voltage measurement and the other to current measurement. (Selection of the parameters to be measured is made with the data output commands **PX/** and **PY/**. see Section 10.1.) The commands for the various DVM control functions are listed in Table 3.19.

Table 3.19 DVM Control Functions Command

Command	Function	
DG0	Number of Digits	5 X 9
DG1		4 X 9 (50Hz ac supply)
DG2		4 X 9 (60Hz ac supply)
DG3		3 X 9
RG0	Input Range *	Autorange
RG1		200mV
RG2		2V
RG3		20V
RG4		200V
TR0	Measurement Trigger	Single measurement.
TR1		Continuous measurements.
TR3		Sweep synchronized measurements.
DC0	Drift correction on.	
DC1	Drift correction off.	
AV0	Averaging off.	
AV1	Averaging on.	
NU0	Null off.	
NU1	Null on.	
NU2	Null evaluate.	
RU0	Digital voltmeter HALT.	
RU1	Digital voltmeter RUN.	

*For voltage measurement only.

9.1. MEASUREMENT RESOLUTION AND READING RATE

The resolution of the DVM measurement results is determined by the number of digits selected, three, four, or five. It should be noted, however, that higher resolution is obtained at the expense of the reading rate, i.e. higher resolution measurements take longer.

9.2. INPUT RANGE

The voltage DVM may either be set for autoranging or any one of four fixed input ranges may be selected. (See Table 3.19) Autoranging allows an input range in keeping with the measured voltage to be selected automatically.

The current DVM has an input voltage range of 200mV. Various ranges of cell current are provided for by a choice of standard resistor values. (See Section 4.)

9.3. MEASUREMENT TRIGGER

Measurements may be triggered in any one of three ways, single, recycled, or sweep synchronized, as described below. Two cell parameters (as selected by the parameter commands **PX/** and **PY/**:see Section 10.1) are measured simultaneously.

9.3.1. Single Measurements

Single measurement triggering is selected with the command **TR**0. A single set of measurements is made (within approximately 30ms) each time a DVM run command (**RU**1) is given. Single measurement triggering is always selected on initialization, e.g. on power up.

9.3.2. Recycled Measurements

Recycled measurement triggering is selected with the command **TR**1. On receiving a run command (**RU**1) the DVMs proceed to make measurements continuously. The measurement rate is determined by the measurement resolution and the speed of the listening device(s) on the GPIB. Recycled measurements can be halted with the **RU**0 command.

9.3.3. Synchronized Measurements

Synchronized measurement triggering is selected with the command **TR**3. The DVMs measure only during the actual sweep segments, not during sweep initialization or delay, etc.

During a ramp sweep the DVMs measure continuously, at a rate determined by the measurement resolution.

During a stepped sweep a measurement is made at each voltage level within the sweep. Each measurement is made as late as possible, to give the cell the maximum settling time. Any drift correction (see Section 9.4) is made just after the measurement and is applied before the result is output.

Should the requested sweep step time be too short for the DVMs to complete a measurement within each step, then an error code is made available for output and the original step time is retained. The remedy is either to select parameters that will increase the measurement rate, or to increase the step time. See Section 8.2.1 for details of minimum step time versus the measurement resolution.

9.4. DRIFT CORRECTION

With drift correct ON selected (command **DC**0) correction is applied every ten seconds to the DVM(s) presently engaged in measuring. For the voltage measuring DVM, correction is applied only to the particular range(s) in use. With drift correct OFF selected (command **DC**1) correction is not applied.

9.5. AVERAGING

With averaging ON selected (command **AV**1) the DVM results are output as a cumulative arithmetic mean. The mean value develops over the first ten measurements and thereafter is output, once per measurement cycle, as a running mean of the last ten results. (This is sometimes known as "walking window" averaging.)

9.6. NULLING

Nulling compensates for stray voltages induced in the cell leads. The three commands involved are:

Evaluate (**NU**2)	This starts the nulling sequence in which the stray voltages are measured and stored and then applied as null values. Note: Before an **NU**2 command is given, the terminations at the cell end of the leads should be connected as follows: **RE**1 and **RE**2 grounded and **WE** and **CE** open-circuited. The nulling sequence takes approximately 75 seconds. On completion, nulling is applied. This can be verified with the query command **?NU**, which should, in this case, give the response "01".
Null ON (**NU**1)	Re-applies previously evaluated null values, after Null OFF has been selected.
Null OFF (**NU**0)	Null values no longer applied, but retained for further use (when they are re-applied in accordance with the Null ON command). These values may be overwritten by another evaluate sequence.

10. DATA OUTPUT

Controlling the data output of the ECI involves selecting the cell polarization parameters to be output and setting up the history file and the GPIB interface. The setups for each of these are described below, in Sections 10.1 through 10.3.

10.1. PARAMETERS TO BE OUTPUT

The results to be output (and filed, if the history file is open) are selected as parameters 1 and 2. The time at which each result is measured is always filed and is always made available for output. The commands used to select parameters 1 and 2 are listed in Table 3.20.

Table 3.20 Output Parameter Selection

Command			
Par1	**Par2**	**Parameter**	
PX0	**PY**0	CE	Counter electrode.
PX1	**PY**1	RE1	Reference electrode 1.
PX2	**PY**2	RE2	Reference electrode 2.
RX3	**PY**3	ΔRE	V_{RE1} - V_{RE2}. (FRA Channel 2)
PX4	**PY**4	ΔRE - Bi	ΔRE - V_{BIAS}.
PX5	**PY**5	I	Cell current. (FRA Channel1)
PX6	**PY**6	I - Bi	Cell current - bias current.
PX7	**PY**7	$\delta\Delta RE \div \delta I$	Polarization resistance.*
PX8	**PY**8	$\delta I \div \delta\Delta RE$	Polarization conductance.*
PX9	**PY**9	POL	Dc polarization from ECI.
PX10	**PY**10	ΣPOL	POL + dc bias from FRA generator.
PX11	**PY**11	DVM	Zero (For nulling).

*Available only with Sweep.

10.2. HISTORY FILE

Measurement results may be stored in the ECI history file, for subsequent output to an external device. Each result consists of a user-defined pair of polarization parameters and the time at which they were measured. The commands for operating the file, and for querying the file status, are listed in Table 3.21.

Table 3.21 History File Functions

Command	**Function**
FL0	Closes the file.
FL1	Opens the file.
FS*l*	Defines the file size (number of results). *l* = 1 to ~ 450.
UF*l*	Defines the location from which the file is to be updated.
VF1	Clears the file, i.e. erases the file contents.
VF2	Outputs the contents of the file to the GPIB.
?FP0	Returns the number of results filed. (0 to ~ 450)
?FP1	Returns the file pointer (= location of last result filed).
?NR	Returns the number of readings taken. (0 to 99999).

The recommended sequence for operating the history file is as follows:

1. Select the results to be stored, i.e. polarization parameters 1 and 2, as described in Section 10.1 above.
2. Define the file size.
3. Open the file.

Once the file is opened a result is filed every time a measurement is completed. When the file is full the earliest results are overwritten by the latest.

10.3. GPIB

GPIB setups are used to select the data output format, "long on", "short on", "long dump" or "short dump", and to define the standard GPIB functions such as Serial Poll and Parallel Poll. The commands for these setups are listed in Table 3.22.

A full description of the GPIB functions is given in Chapter 5 of the manual.

Table 3.22 GPIB Functions

Command	Function
GP0	Output off.
GP1	Long on: Compressed ASCII format, with time.
GP2	Short on: Compressed ASCII format, without time.
GP3	Long dump: Binary format, with time.
GP4	Short dump: Binary format, without time.
	Note: for a full description of GPIB output formats, see Chapter5, Section 6.
OS0	Output separator = comma (,)
OS1	Output separator same as terminator.
OT0	Output terminator = cr lf
OT1	Output terminator = cr lf and EOI
OT2	Output terminator = cr
OT3	Output terminator = cr and EOI
SV*I*	Specifies which serial poll bit(s) may initiate a service request. ***I*** = 1, 2, 4, 8, etc, to define a specific bit, or several such integers may be added together to define several bits, e.g. 10 defines bits 2 and 8. (See Chapter 5, Section 7.)
PP*I*	Specifies which GPIB data line the ECI is to assert, to identify itself in a parallel poll. ***I*** = 0 to 8. (See Chapter 5, Section 7.)
PS0	Parallel poll sense true: "1" (Low on bus) = service request.
PS1	Parallel poll sense false: "0" (High on bus) = service request.

11. BREAK / SELF-TEST

Commands are available to halt the present operation of the ECI and to make it perform various self-tests. These commands are listed in Table 3.23.

Table 3.23 Break and Self-test

Command	Function	
BK0	Break	Halts the present operation of the ECI and returns it to the standby state, as defined by the BY command (see Section 2.1 in this chapter).
BK1 or **BK**2	Check	Starts a self-test of the ECI digital circuitry. On completion of this test the ECI is set to the standby state; the DVM and sweep are switched off. Any failure causes the test routine to abort and a fail code is generated.
BK3	Reset	Returns all control setups to the default state, but retains the content of the history file.
BK4	Initialize	Returns all control setups to the default state and clears the history file.

Note: To give the ECI time to implement a **BK/** command a one second pause must be allowed between this command and any other command which follows it.

The overall result of a self-test may be obtained with the query command **?TS**0: a "0" indicates a pass, whilst a number other than "0" indicates failure. The cause of failure can be ascertained with the specific query commands shown in Table 3.24.

Table 3.24 Self-test Results

Command	Function
?TS	Overall result of self-test.
?TS1	RAM, 1 or 2.
?TS2	ROM, 1 or 2.
?TS3	Timer, 1, 2 or 3.
?TS4	GPIB

12. ERROR QUERY

The last error detected by the ECI can be queried with the **?ER** command. The response is a two-digit number between "00" and "99". The meaning of each error code is given in Appendix B. Note that "00" signifies no error.

The code representing the last error can be cleared with the **CE** command. This can be helpful when debugging control programs.

13. ECI SOFTWARE VERSION

The status and issue of the ECI software can be ascertained with the **?VN** command. The response is a four digit number, "5102", followed by a two-letter code. The first letter represents the software status and the second the issue.

14. TIME

A starting value for the ECI elapsed time can be entered with the command **TM***I,I*, where *I,I* represent a pair of two-digit numbers which define hours and minutes. This facility could be used to reinstate the elapsed time, should it be necessary to initialize the ECI in the middle of an experiment.

Chapter 4

FRA Command Functions

1. FRA STRUCTURE

Basically, the FRA consists of a signal generator which provides a.c. perturbation for the cell, and an analyzer to measure the cell's response. See Fig 4.1.

The **generator** facilities include:

- Sine, square, or triangular waveform.
- User-defined frequency. (1mHz to 20kHz)
- User-defined amplitude. (0V to 7V rms)
- User-defined bias. (-10V to + 10V)
- Logarithmic frequency sweep between 1mHz (minimum) and 20kHz (maximum).
- Selectable stop point on waveform.

The **analyzer** facilities include:

- User-defined integration time, to provide just enough interference rejection for the signal being measured.
- Automatic input ranging, or selectable fixed input range.
- Single or repetitive measurements.
- Result scaling.
- History file (400 results approx.)
- Selectable result coordinates: *a +jb*; *r,q*; or r(dB), *q*.
- Compressed ASCII, or binary, output to the GPIB.

Each of these facilities is described in the following pages.

The remote commands are interpreted by the FRA microprocessor and routed to the appropriate functional block. These commands are presented, throughout the chapter, in tabular form. Each command consists of a two-letter code, followed either by an integer that species a functional setting or a real number that defines a parametric value. (See Chapter 2, Section 1.4 for real number format.) The default settings are indicated by underlining the associated command. Parametric values default to zero, except where otherwise stated.

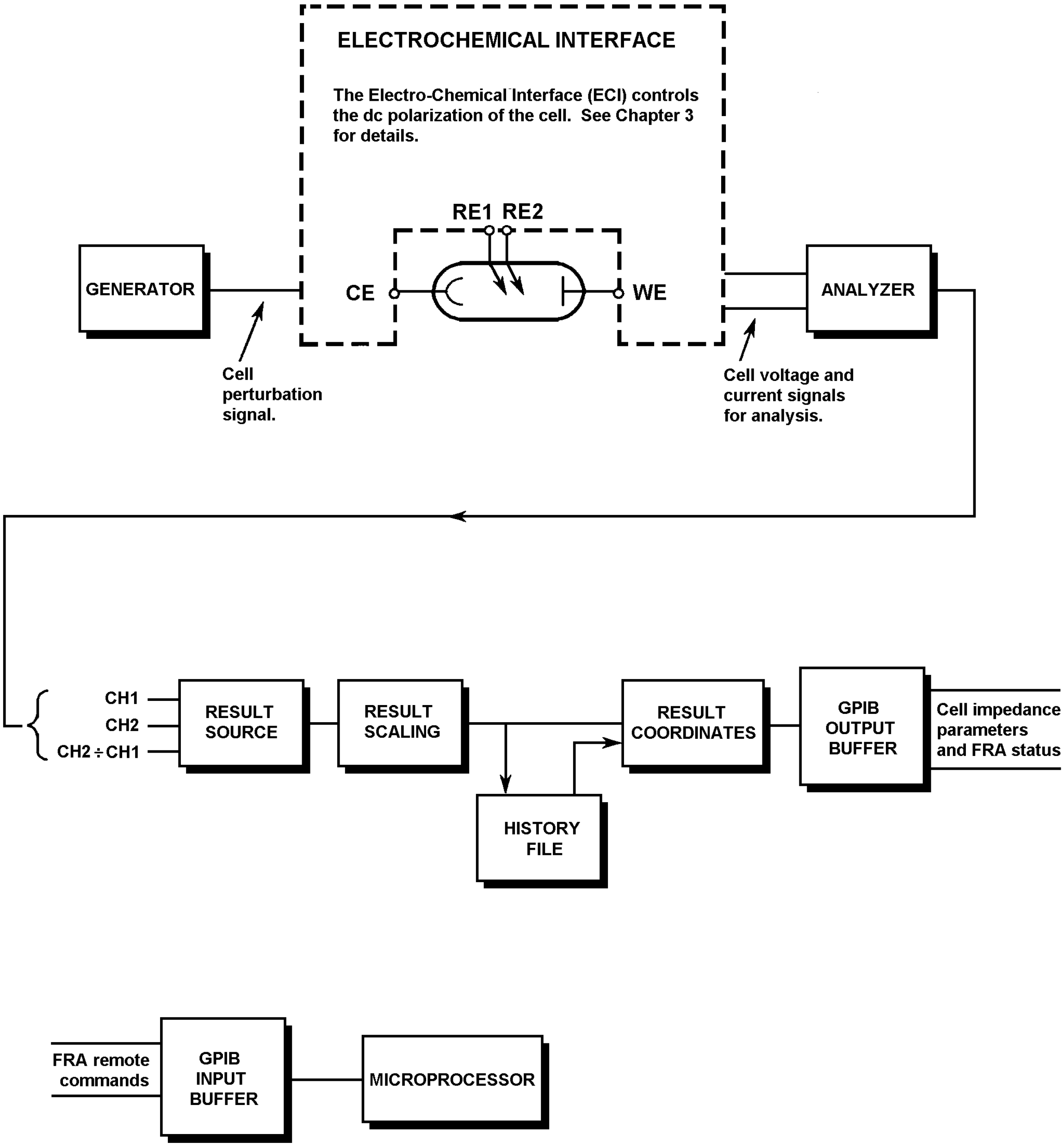


Fig 4.1 *FRA functional blocks.*

2. GENERATOR

The generator provides a very stable and accurate signal for cell perturbation, with selectable waveform, frequency, amplitude and bias. These signal parameters and the commands to set them up are described below.

Table 4.1 Generator Signal Definition

Command	Function	
<u>**WV**0</u> **WV**1 **WV**2	Waveform	Sinewave Squarewave Triangular wave
FR*F*	Frequency	*F* = 1mHz to 20kHz (default value = 100Hz)
AM*F*	Amplitude	*F* = 0V to 7V
BI*F*	Bias	*F* = -10V to +10V

F= real number (See Chapter 2, Section 1.4)

2.1. WAVEFORM

Any one of three waveforms may be selected for the generator output. A sinewave (**WV**0) is the most usual choice, and is the default setting. However, the other two waveforms can be useful should you need a high-performance function generator. The triangular output (**WV**2) can be set up as a slow ramp, which is useful for linearity measurements, and the squarewave (**WV**1) can be used for loop transient response testing.

2.2. FREQUENCY

The frequency of the generator output may be specified with the **FR***F* command. The digital technique used by the FRA generates a discrete set of frequencies, which means that the frequency selected may not be exactly the one requested. However, the selected value is always within the requested value ± 0.25%, and is normally much closer.

When the cell response over a range of frequencies is required the generator output frequency may be incremented (or decremented) either by issuing successive **FR***F* *commands or by setting up a frequency sweep (see Section 2.5).*

2.3. AMPLITUDE

The amplitude of the generator output is set with the **AM***F* command, where *F* is the rms value (*not* the peak value) of the amplitude and can be up to 7V (5.11V for triangular waveform) with a resolution of 0.01V. The peak value is calculated by multiplying the rms value by the following crest factors:

√2 (≈ 1.414)	for the sinusoidal output,
√3, (≈ 1.732)	for the triangular output,
1.000	for the square wave output.

Note that the generator amplitude (peak value)+ bias can not exceed ±10V. Any command that would produce this situation is ignored and Error Code 22 is evoked.

2.4. BIAS

Additional d.c. bias may be applied to the cell from the generator output. (Fig 4.2) Bias is set with the **BI***F* command, to a maximum of ± 10V, and appears immediately at the generator output. The bias resolution is 0.02V. Note that the generator peak voltage +bias must not exceed ± 10V; Error Code 22 is generated, should this happen.

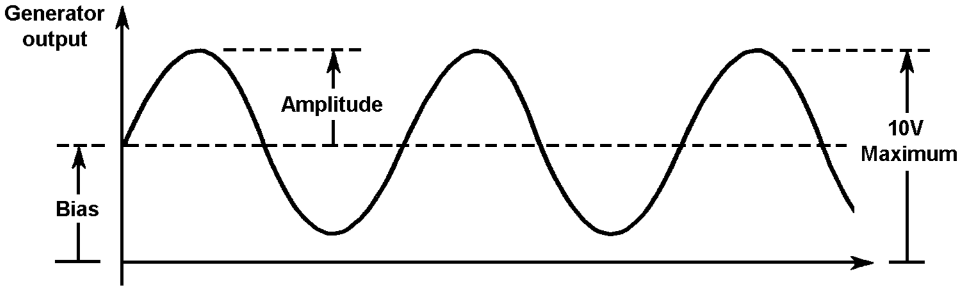


Fig 4.2 *Generator amplitude and bias.*

2.5. FREQUENCYSWEEP

The commands for setting up a frequency sweep are listed in Table 4.2. The procedure is as follows:

1. Define the frequency range, by entering the maximum and minimum frequencies. (**MA***F* and **MI***F* commands)
2. Define the number of frequency points within that range. (**GS***I* command)
3. Define the sweep direction, sweep up (increasing frequency) or sweep down (decreasing frequency). (**SE***I* command)

Table 4.2 Sweep Setup

Command	Function	
MA*F*	Max. frequency	*F* = 1mHz to 20kHz (default = 20kHz)
MI*F*	Min. frequency	*F* = 1mHz to 20kHz (default = 100Hz)
GS*I*	Log sweep	I = 2 to 9999 points (default = 100 points)
<u>**SE**0</u>	Sweep off	
SE1	Sweep up	
SE2	Sweep down	

F= real number (See Chapter 2, Section 1.4)

On the last command (**SE***I*) the FRA calculates the successive values for each frequency point, so that they are equally spaced on a logarithmic scale. This means that the ratio between successive frequencies is the (n-1)th root of $F_{max} \div F_{min}$.

For example, if F_{max} is 10kHz and F_{min} is 100Hz, and the number of frequency points is 100, the successive frequencies are in the ratio

$_{99}\surd$ (10,000 ÷ 100) = $_{99}\surd$ 100 = 1.0476

The frequency points are thus: 100Hz, 104.76Hz, 109.74Hz, 114.97Hz... etc. up to 10kHz. (The sweep thus embraces 100 frequency points which it covers in 99 steps.)

Once the sweep parameters are set the analysis is started, using either the **SI** (SIngle) or **RE** (REcycle) commands. Note that a **BK** (BreaK) command stops a sweep and sets the **SE** parameter back to 0, i.e. the sweep is switched off.

The time taken to complete a sweep depends on the frequency range chosen and on the integration time.

The time to complete a sweep of *n* steps is approximately:

n X (*measurement delay* + *integration time*),

where the measurement delay (which allows the cell to settle after each change of frequency) is 100ms, and the integration time is that set by the **IS***F* command. For a CH2/CH1 measurement the integration time is doubled.

This calculation is approximate, because the sweep time is affected by other factors, typically software delays, and the drift correction delays which take place every minute.

2.6. GENERATOR START AND STOP CONTROL

Since no impedance measurements can be made without the cell being perturbed, you will notice that some of the generator commands act also on the analyzer and, similarly, some of the analyzer commands act also on the generator. To avoid any confusion, the commands which act primarily on the generator are listed in Table 4.3, whilst those which act primarily on the analyzer are listed in Table 4.5. In each case the secondary action of a command is indicated. All actions taken by the generator, whether as the result of a generator command or an analyzer command, are covered in the text below.

Table 4.3 Generator Start and Stop Commands

Command	Function
RG	Run Generator Allows the generator to be started without starting the analyzer.
SG	Stop Generator Stops the generator and, if running, the analyzer. The final state of the generator output depends on the stop mode.
SM0	Freeze output Selects stop mode-freeze. When the SG command is given the generator output is held at its present level.
SM1	Kill output Selects stop mode-kill. Immediately the SG command is given the generator output is set to zero volts.
SQ0	Stop on Quadrant Holds the generator output at vector angle 0°
SQ1	″ ″ ″ ″ 90°
SQ2	″ ″ ″ ″ 180°
SQ3	″ ″ ″ ″ 270°

NOTE: Generator output bias is independent of the generator start and stop control and is applied immediately the bias value is entered. (See Section 2.4.)

2.6.1. Starting the Generator

The generator may be started, without starting the analyzer, with an **RG** command. When a SIngle, or REcycled, measurement command is given to the analyzer, however, the generator is started automatically, one second before measurement begins.

The generator waveform, frequency or amplitude may be altered while the generator is running, but not whilst a measurement is in progress. To make a series of measurements with progressive signal settings, use single measurements.

2.6.2. Stopping the Generator

The generator may be stopped in several ways:

(a) With an **SG** (Stop Generator) command. The generator output is "frozen" "killed", as previously defined by the **SM** (Stop Mode) command. See Fig 4.3.

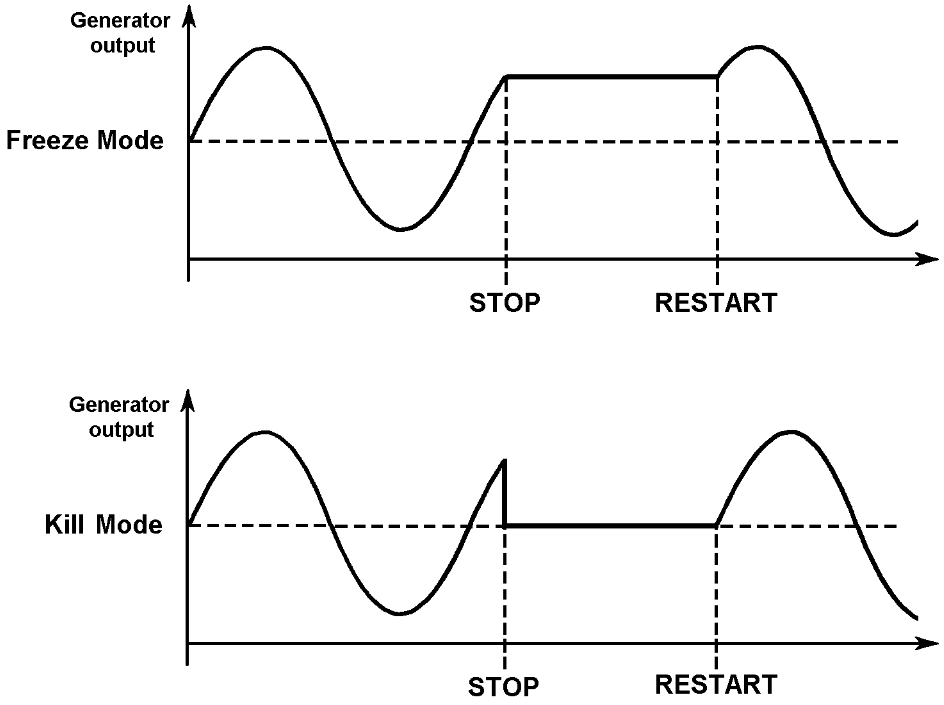


Fig 4.3 *Effect of SG and RG commands in Freeze and Kill modes.*

(b) With an **SQ** (Stop on Quadrant) command. The generator output is held at a previously defined vector angle. See Fig 4.4. Note that, with a low frequency signal, the generator output may continue for some time before the defined stop point is reached.

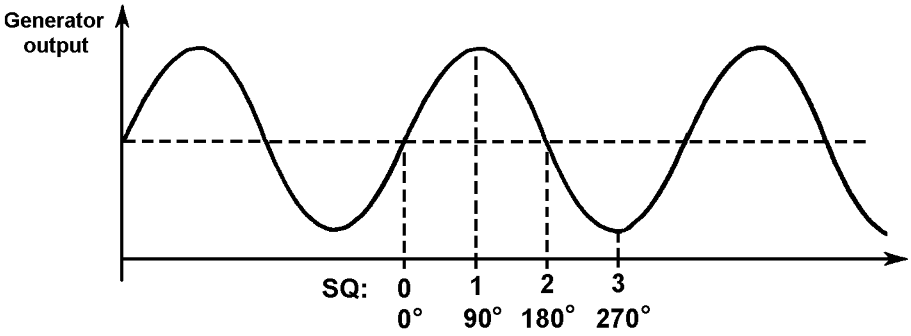


Fig4.4 *Stopping points for SQ (Stop on Quadrant)command.*

(c) With a TT1 (initialize) or TT2 (reset) command. This has the same effect as killing the generator output, but necessitates re-entering the FRA setup.

The generator may be restarted with an **RG**, **SI** or **RE** command. In cases (a) and (b) the waveform continues from the stop point, as indicated in Fig 4.3: if a sweep was in progress then measurement continues from the sweep stop point.

3. ANALYZER

The analyser receives two inputs, the cell voltage (ΔRE) and the cell current (I). Each input is correlated with the generator output and the magnitude and phase (in relation to the generator signal) is calculated. (ΔRE and I may thus be accurately measured at each generator signal frequency. Either parameter may be output directly, or the cell impedance may be derived from them (see Section 4.1, "Result Source").

The analyser settings to be made before analysis can begin are the integration time and the input range. These are described below, in Sections 3.1 and 3.2. The analyzer setup commands are listed in Table 4.4.

Table 4.4 Analyzer Setup

Command	Function
RA1,0 (**RA**2,0)	Channel 1 (Channel2) Input range: Auto
RA1,1 (**RA**2,1)	30mV
RA1,2 (**RA**2,2)	300mV
RA1,3 (**RA**2,3)	3V
RA1,4 (**RA**2,4)	30V
RA1,5 (**RA**2,5)	300V
HA1 to **HA**8	Defines the harmonic to be measured. "1" = fundamental, "2" = second harmonic, and so on.
IS*F*	Integration time F = 0.1s to 10,000s (default = 0.1s)

F = real number (See Chapter 2, Section 1.4)

3.1. INTEGRATION TIME

The accuracy of a correlation measurement can be degraded by noise and harmonic signals contributed by the cell. However, noise and harmonic rejection can be dramatically improved by increasing the integration time to cover an integral number of generator cycles. A demonstration of this is given in Fig 4.4.

The curve set annotated "N = 1" in Fig 4.4 defines the rejection obtained for one cycle of integration. (An integration cycle is the process of integrating the products of correlation over one generator cycle.) From this it can be seen that good rejection is obtained, at harmonics of the fundamental, but frequencies between the harmonics, or noise, can easily corrupt the result.

The curve set annotated "N = 10" shows the improvement obtained by using ten cycles of integration. Here, the lowest frequency that can be rejected by the correlation process is one tenth of the fundamental frequency (f_G). Better rejection is thus obtained, at frequency increments of one tenth f_G, and the general level of rejection is improved also.

Further improvement is obtained by using 100 cycles of integration (N=100), which gives excellent rejection of noise. However, remember that increasing the integration time also decreases the measurement rate. So, choose an integration time that gives just sufficient rejection for your purpose. To check the amount of interference in your analysis results make several measurements in succession in the recycle mode and note the "scatter": then increase the integration time and repeat the process. The integration time is about right when a further increase gives no appreciable reduction in scatter.

The integration time (0.1s to 10000s) is set with the **IS***F* command and is automatically rounded, on entry, to cover an integral number of integration cycles. The minimum integration time is 0.1s or the period corresponding to one integration cycle, whichever is the longer; this is also the default setting.

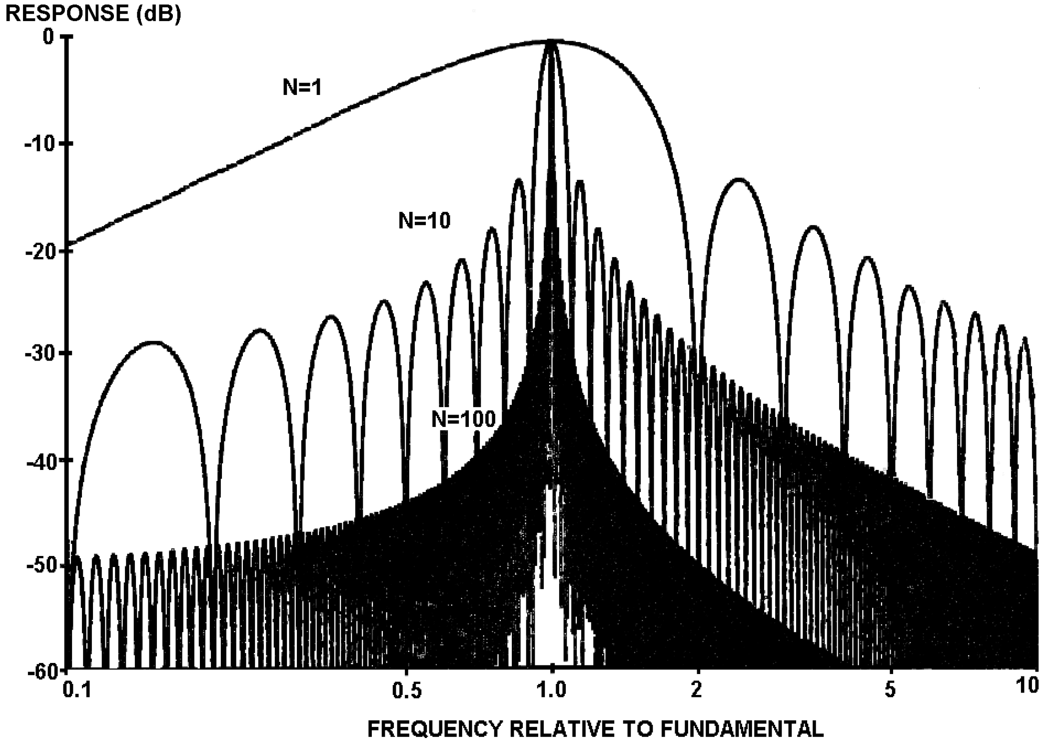


Fig4.4 *Harmonic rejection curves for N cycles of integration.*

3.2. INPUT RANGE

Autorange or a fixed input range may be selected for each of the two inputs of the analyzer. Autorange is normally the best choice, as the range is adjusted automatically to suit the level of input signal. This is done for each measurement frequency, which is particularly useful during sweeps.

During autoranging the analyzer may have to make several measurements to select the correct range and this takes time, particularly at very low frequencies. Therefore, time may be saved by using a fixed range that covers all input levels. However, this may impair the accuracy of some measurements, so a compromise must sometimes be made between measurement speed and accuracy.

3.3. HARMONIC MEASUREMENT

The use of harmonic measurements can reveal details of the corrosion mechanism. When a large amplitude sinewave is imposed about the d.c. polarization of the cell, measurement of the direct polarization current and of the first second and third harmonics allows the corrosion current and the Tafel coefficients to be evaluated.

(See *Use and Applications of Electrochemical Impedance Techniques* (Technical Report No. 24) by Claude Gabrielli: Chapter 2, Section 2.2. This book is freely available, on demand, from Solartron.)

The analyzer is able to measure the response of the cell at any harmonic of the generator frequency, up to the eighth. Note, however, that the frequency of the harmonic to be measured must not exceed 20kHz. By default, the analyser measures the first harmonic, i.e. the cell response at the fundamental frequency.

Rejection of harmonics other than the one being measured applies in a similar way to that shown in Fig 4.4. The signal is still analyzed over N cycles of the fundamental, but the "lobe" of the harmonic being measured takes the place occupied by the fundamental in Fig 4.4: the other harmonics are aligned with the maximum rejection points, as before.

3.3.1. Harmonic Measurement Procedure

The simple procedure for measuring the harmonic data is as follows:

1. Set up the generator to provide the fundamental frequency (or fundamental *frequencies* in the case of a sweep).
2. Select the harmonic to be measured.
3. Make the measurement.
4. For other harmonics, repeat steps 2 and 3.

3.4. STARTING AND STOPPING THE ANALYZER

Impedance measurements involve both the generator and the analyzer. Therefore, some of the analyzer commands act also on the generator and, similarly, some of the generator commands act also on the analyzer. To avoid confusion the commands which act primarily on the analyzer are listed in Table 4.5, whilst those which primarily on the generator are listed in Table 4.3. Secondary actions are described in each case. All analyzer actions, due either to an analyzer command or to a generator command, are described below.

Table 4.6 Analyzer Start and Stop Commands

Command	Function	
SI	SIngle	Starts the generator, if stopped, and, after a one second delay*, starts a single measurement.
RE	REcycle	Starts the generator, if stopped, and, after a one second delay*, starts continuous measurements.
SA	Stop Analyzer	Stops the analyzer, without completing the 1 measurement cycle.

*Delay applicable only if generator was not running.

Once all the relevant control parameters have been set, measurement can begin. SI (*single*) starts the generator if it is stopped, and makes a single measurement. SI starts a measurement only if the analysers are not busy: therefore a rapid succession of SI commands does not result in a series of results, unless each measurement is allowed to finish before the next SI command is given.

RE (*recycle*) also starts the generator if it is stopped, and the analyzer then makes repeated measurements. These continue until an SI (SIngle), SA (Stop Analyzer) or SG (Stop Generator) command is given. If sweep is enabled, the measurements are at successive values of generator frequency, and stop once the end frequency (maximum or minimum) has been reached.

Both SI and RE take effect immediately, i.e. the analysers do not wait until the output reaches the 0° point of the cycle. SA stops the analyzer, and does not complete the measurement or produce a result.

To restart measurements after they have been stopped, use RE or SI. If sweep is on, and was stopped before reaching the end, measurements continue from the point of interruption, using the frequency selected when the stop command was given. To restart at the minimum frequency, simply re-set one of the sweep parameters: MI, MA, GS or SE. (See Section 2.5.)

4. DATA OUTPUT

Figure 4.5 shows the functions to be set up for the FRA data output. Details are given in Sections 4.1 through 4.5.

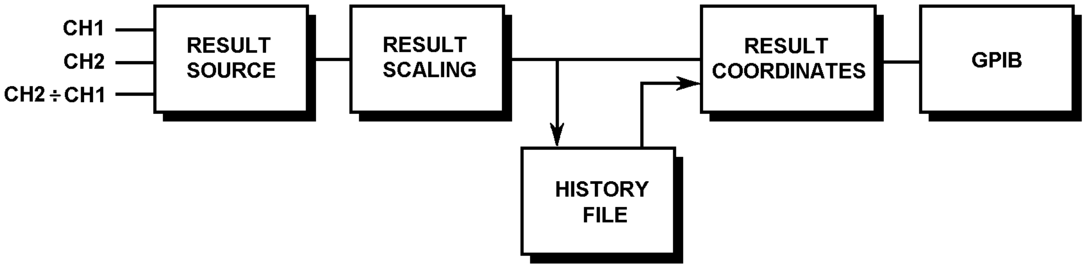


Fig 4.5 *Data output functions of the FRA.*

Results are made available for output to the GPIB as they occur and, at the same time, are stored in the history file. Therefore, results may be output singly or in sets. Stored results may be output in all three coordinate forms: *a +j b*, *r*,*q* and *r*(dB),*q*.

4.1. RESULT SOURCE

The result source may be selected from Analyzer Channel 2 (cell voltage), Analyzer Channel 1 (cell current), or the ratio of the two. The latter source is the default setting and is the one to use for impedance measurements. Table 4.6 lists the result source commands.

Table 4.6 Result Source

Command	Function
SO0100	Result source is Channel 1. (Channel 2 is not measured.)
SO0200	Result source is Channel 2. (Channel 1 is not measured.)
SO0201	Result source is Channel 2 ÷ Channel 1.

4.2. RESULT SCALING

Result scaling allows results to be divided by a user-defined vector. Table 4.7 lists the result scaling commands. The entry of a scaling vector affects all subsequent measurements, until deselected (by **FN**0).

Table 4.7 Result Scaling

Command	Function
FN0	Sets scaling to divide by unity, i.e. scaling off.
FN1	Sets scaling to divide by *r*,*q*. (*r* and *q* are defined by **RF***F* and **TF***F*)
FN2	Sets scaling to divide by last result.
FN3	Sets scaling to divide by last magnitude.
RF*F*	Defines *r* value to be used by **FN**1 command. *F* = -10^9 to $+ 10^9$
TF*F*	Defines *q* value to be used by **FN**1 command. F= -180° to + 180°

F = real number (See Chapter2, Section 1.4)

4.3. HISTORY FILE

All measurement results are automatically stored in the FRA history file, in *a+jb format, and each includes the frequency at which it was measured. The results, thus stored, are used for subsequent output to an external device.*

The content of the history file is determined by the result source and scaling setups. Once a result is stored the source and scaling setups can have no further effect on it, e.g. you can't rescale a stored result. It is possible, however, to output the same stored result in any, or all, of the three coordinate forms. (See Section 4.4.)

The commands used to control the history file are listed in Table 4.8.

Table 4.8 History File

Command	Function
?NR	Function Returns number of results written to file. (This is not necessarily the same as the file population, as earlier results may have been overwritten.)
?FP0	Returns the file population, e.g. the number of results actually in the file.
?FP1	Returns the location number of the result presently being accessed.
?FS	Returns the overall file size (> 500). (This is constant for any issue of firmware, but may vary between issues.)
UF*I*	Outputs result at file location *I* *I* = 1 to ~ 400
FO	Output all results in file.

The history file can contain a maximum of approximately 400 results. Should the file become full. however, the earliest results are overwritten by the latest. Query command **?FP**0 returns the number of results stored in the file, i.e. the file population. Query command **?NR** returns the number of results written to the file since it was last cleared: if this number is greater than the file population then some results have been overwritten.

Results are output from the history file with the command **UF***I* (single result) or **FO** (all stored results).

4.4. RESULT COORDINATES

Results can be output in Cartesian, polar or log polar form, as selected by the commands listed in Table 4.9.

Results stored in the history file in *a* + *jb* form and have the selected coordinates assigned during output. Therefore the same results may, if required, be output in more than one form.

Table 4.9 Result Coordinates

Command	Function
CO0	Selects coordinates *a* + *j b*.
CO1	Selects coordinates *r*, *q*.
CO2	Selects coordinates *r*(dB), *q*.

F = real number (See Chapter 2, Section 1.4)

4.5. GPIB

GPIB setups are used to select the data output format, ASCII or binary, and to define the standard GPIB functions such as Serial Poll and Parallel Poll. The commands for these setups are listed in Table 4.10.

Table 4.10 GPIB Functions

Command	Function
OP2,0	Output off.
OP2,1	ASCII: Compressed ASCII format.
OP2,2	Binary: Binary format. Fast "dump" output, for subsequent data processing. **Note**: for a full description of GPIB output formats, see Chapter 5, Section 6.
OS0	Output separator = comma (,)
OS1	Output separator same as terminator.
OT0	Output terminator = cr lf
OT1	Output terminator = cr lf and EOI
OT2	Output terminator = cr
OT3	Output terminator = cr and EOI
SV*I*	Specifies which serial poll bit(s) may initiate a service request. *I* = 1, 2, 4, 8, etc, to define a specific bit, or several such integers may be added together to define several bits, e.g. 10 defines bits 2 and 8. (See Chapter 5, Section 7.)
PP*I*	Specifies which GPIB data line the ECI is to assert, to identify itself in a parallel poll. *I* = 0 to 8. (See Chapter 5, Section 7.)
PS0	Parallel poll sense true: "1" (Low on bus) = service request.
PS1	Parallel poll sense false: "0" (High on bus) = service request.

5. BREAK / SELF-TEST

Commands are available to halt the present operation of the FRA and to make it perform various self-tests. These commands are listed in Table 4.11.

Table 4.11 Break and Self-test

Command	Function	
BK	Break	Halts the present operation of the FRA and returns it to the ready state. Control setups unaffected. History file unaffected.
TT0	Drift Correct	Measures and nullifies any offsets due to drift that may be present in the analyzer input amplifiers. Actioned at next measurement. Also actioned automatically every minute or once per integration period, whichever is the longer.
TT1	Initialize	The generator is stopped, all control setups are returned to the default state the history file is cleared, and the elapsed time clock is set to zero.
TT2	Reset	Exactly the same effect as initialize (**TT**1) except that the content of the history file is retained.
TT3	Check	Starts a simple self-test of the FRA digital circuitry. Any failure causes the test routine to abort and a fail code is generated.
TT4	Test	Starts a self-test of the entire FRA circuitry, both analog and digital. Any failure causes the test routine to abort and a fail code is generated. Test should be used in preference to check.

Note: To give the FRA time to implement a **BK** command a one second pause must be allowed between this command and any other command which follows it.

The overall result of a self-test may be obtained with the query command **?TS**0: a "0" indicates a pass, whilst a number other than "0" indicates failure. The cause of failure can be ascertained with the specific query commands shown in Table 4.12.

Table 4.12 Self-test Results

Command	Function
?TS0	Overall result of self-test.
?TS1	RAM test result.
?TS2	ROM test result.
?TS3	Timer test result.
?TS4	*Not used.*
?TS5	*Not used.*
?TS6	Analyzer test result.

6. LAST ERROR QUERY

The last error detected by the FRA can be queried with the **?ER** command. The response is a two-digit number between "00" and "99". The meaning of each error code is given in Appendix B. Note that "00" signifies no error.

The code representing the last error detected can be cleared with the **CE** command. This can be helpful when debugging control programs.

7. FRA SOFTWARE VERSION

The status and issue of the FRA software can be ascertained with the **?VN** command. The response is a four-digit number, "5101", and a two-letter code. The first letter represents the software status and the second the issue.

8. TIME

A starting value for the FRA elapsed time can be entered with the command **TM***I,I*, where *I,I* represent a pair of two-digit numbers which define hours and minutes. This facility could be used to reinstate the elapsed time, should it be necessary to initialize the FRA in the middle of an experiment.

Chapter 5

GPIB Interface

1. INTRODUCTION

The GPIB is an extremely flexible and easy way of combining programmable instruments and computers into a system. First introduced in 1975, the GPIB has since found world wide acceptance and there is now a wide range of GPIB compatible equipment from which the system designer may choose.

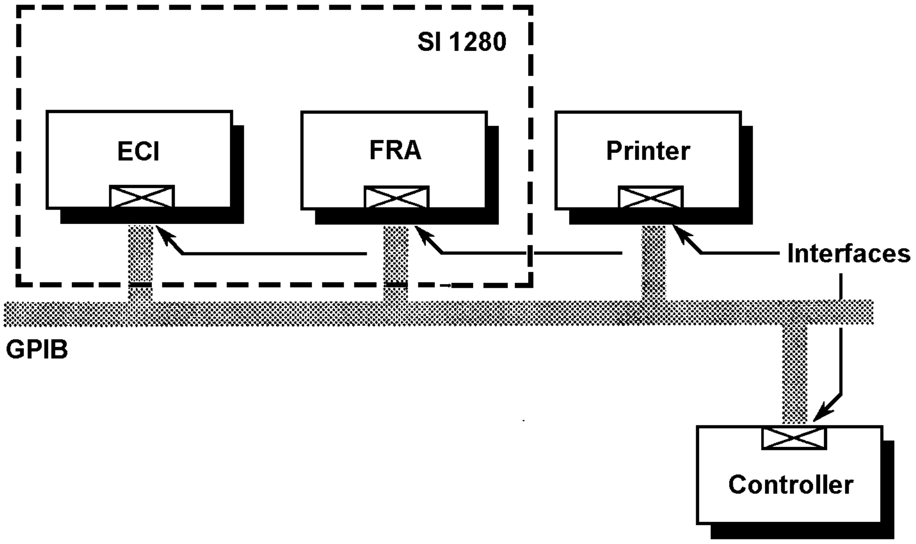


Fig 5.1 *Typical SI 1280 GPIB system*

GPIB stands for "General Purpose Interface Bus", which is the popular name for standard digital interfaces that allow devices of different type and manufacture to be connected together, so that they may operate as a system.

For those unfamiliar with the GPIB, here are the meanings of the more commonly used terms.

- A "Device" is any instrument compatible with GPIB operation.
- The term "Interface", with regard to a GPIB device, means the logic circuitry and bus drivers that deal with the signals sent on the bus, in accordance with the GPIB protocol.
- A "Talker" is a device that can send information via the GPIB, whilst a "Listener" is a device that can receive this information.
- A "Controller" is a device that is able to assign Talker and Listener roles to itself and to other devices on the GPIB. This allows the controller to send control messages to devices on the GPIB and to receive measurement and control status information from them. The overall purpose of a controller is to coordinate the operation of all devices on the GPIB, in accordance with a control program.

The documents which fully define the GPIB are the IEEE Standard 488-1978, the IEC 625-1 and the ANSI Standard MC1.1 (from the first standard, the GPIB is also widely known as the IEEE 488 Interface).

2. GPIB CAPABILITY CODES

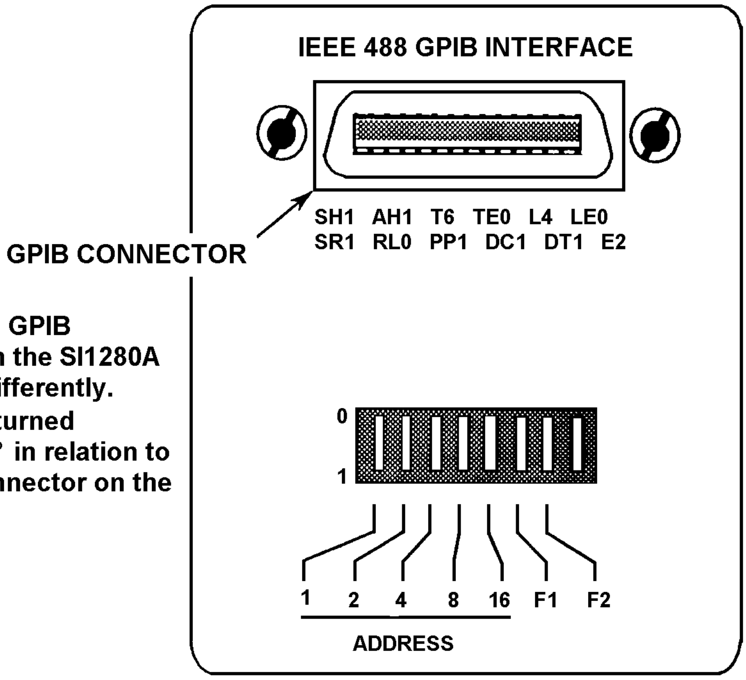
The capability of a device interface can be identified by the series of alphanumeric codes, which normally appear against the bus connector. The alphabetic part of the codes represent a basic interface function, such as a talker or listener, whilst the numeric part represents the capability of that function.

The GPIB Interface in the instrument conforms to the following sub-functions within the standard, as listed on the rear panel:

SH1	Source handshake.
AH1	Acceptor handshake.
T6	Basic talker, serial poll, no talk only mode, unaddressed if MLA (My Listener Address).
TE0	No extended talker capability.
L4	Basic listener, no listen only mode, unaddressed if MTA (My Talker Address).
LE0	No extended listener capability.
SR1	Complete service request capability.
RL0	Remote control capability only.
PP1	Parallel poll with remote configuration.
DC1	Complete device clear capability, including selective device clear.
C0	No controller capability.
DT0	No device trigger capability.
E2	Tri-state drivers.

3. GPIB CONNECTOR

Connection to the GPIB is made via the 24-way connector on the IEEE 488/GPIB interface. See Figure 5.2. The pin connections confirm to the IEEE 488, 1978 standard.



Note that the GPIB connector on the SI1280A is oriented differently. That is, it is turned through 180° in relation to the GPIB connector on the SI1280B.

Fig 5.2 *GPIB interface on the SI1280B*

4. GPIB SWITCHES

Some interface functions are set by miniature toggle switches on the rear panel of the instrument. These functions are described in the following sections.

Once the GPIB switches have been set the instrument must be made to read them, so that their settings can he acted on. The switches are read automatically at power-on, or when an INITIALIZE or RESET instruction is given.

The GPIB switch functions are as follows:

4.1. INPUT TERMINATOR

Switches F1 and F2 select the terminating character for the GPIB input commands to the SI 1280 as follows:

Switch F1	Switch F2	Terminator Selected
0	0	LF (Line Feed).
1	0	CR (Carriage return).
0	1	; (Semicolon).
1	1	EOI (End or Identify).

An example of input terminator selection on switches F1 and F2 is shown in Fig 5.3.

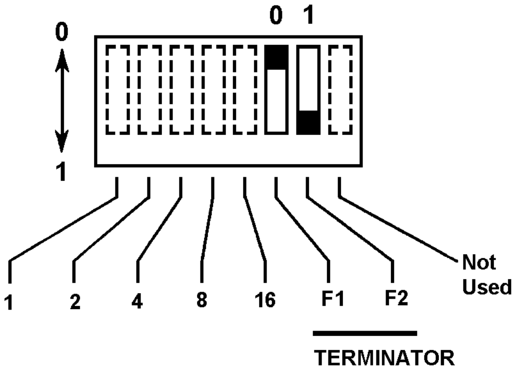


Fig 5.3 *Example: setting a semicolon terminator.*

The choice of command terminator is usually determined by the type of controller used and should be defined in the relevant controller handbook. Any command terminator other than the one selected is ignored by the interface.

4.2. DEVICE ADDRESS

The ADDRESS switches allow you to select a unique address (between 0 and 26) for each device on the GPIB. This number is used to distinguish a device from others on the bus.

For the SI 1280 the address must be an oven integer and is selected by the user. Note that if an odd address is selected on the switches the SI 1280 responds to the preceding even address.

The SI 1280 uses one GPIB interface but assigns four addresses, two for each device (i.e. the ECI and FRA). The first address is selected via the toggle switches: the other three addresses are automatically assigned, and follow on from this. See figure 5.4. **Note**: To avoid system malfunction, no other device on the GPIB can be set to any of the four addresses used by the SI 1280.

The assignment of GPIB addresses to the ECI and FRA works as follows:

The **ECI Major address** is selected via the switches. This is used for ASCII commands and data.

The **ECI Minor address** is the ECI Major address + 1. This is used for high speed binary dump outputs.

The **FRA Major address** is the ECI Major address + 2. This is used for ASCII commands and data.

The **FRA Minor address** is the ECI Major address + 3. This is used for high speed binary dump outputs.

An example of an address switch setup is shown in Fig 5.4.

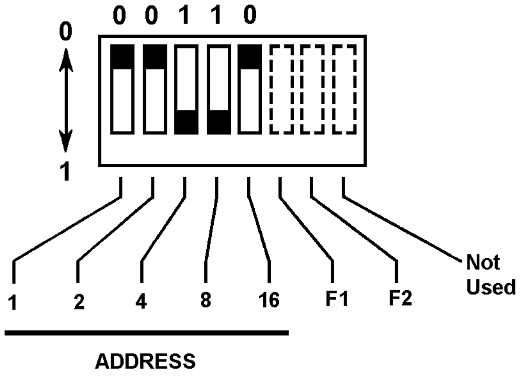


Fig 5.4 *Example:setting an ECI Major address to "12".*

In this example the ECI major address is 12. Therefore the ECI minor address is 13, the FRA major address is 14, and the FRA minor address is 15.

5. GPIB OUTPUT FORMATS

The output of measurement results to the GPIB is controlled by the GPIB Data Output setting. The results can be output as ASCII characters or as Binary data. The ASCII output can be interpreted by a controller or a printer whereas the binary can only be interpreted by a controller. These outputs have the following formats.

5.1. NORMAL ASCII OUTPUT FORMAT

When the instrument is set for talker-listener operation data is output to the GPIB in a compressed form, suitable for interpretation by a GPIB controller.

The ECI uses the following commands to obtain compressed ASCII output for the ECI :

GP1 - Long on (ASCII with time)
GP2 - Short on (ASCII without time)

see the command function chapters for further details.

The ECI gives each parameter a constant field width of 12 ASCII characters (bytes), comprising of a six-digit fixed point part and a two-digit exponent.

With 'long on' selected, each set of readings is output with the format:

± p.ppppp E ± pp /	*± q. qqqqq E ± qq /*	*ee/ ee/*	*hh/mm/ss/ss//*
Parameter 1	Parameter 2	Error codes 1 and 2	Time

where:

error codes 1 and 2 represent the error states of parameters 1 and 2,

" */* " represents the output separators,

and " *//* " represents the output terminator.

Select the required separator and terminator via the Data Output - GPIB commands.

With 'short on' selected, the time data is omitted and the output terminator is sent after the second error code.

The error code is represented by a single digit character which is ASCII '1' if there is an overload error or ASCII '0' otherwise.

The **FRA** uses the following command to obtain compressed ASCII output format:

OP2,1 - ASCII output

See the command function chapters for further details.

The FRA automatically allocates to each parameter a constant field width of 11 characters, comprising of a five-digit fixed point part and a two-digit exponent.

A complete reading takes the form:

± *f.ffff* E ± *ff*, ± *a.aaaa* E ± *aa*, ± *b.bbbb* E ± *bb*, *e cr*

frequency	a	b	error code
frequency	r	θ	error code
frequency	r(dB)	θ	error code

where *e* represents single digit error code. Select the required separator and terminator via the Data Output - GPIB commands.

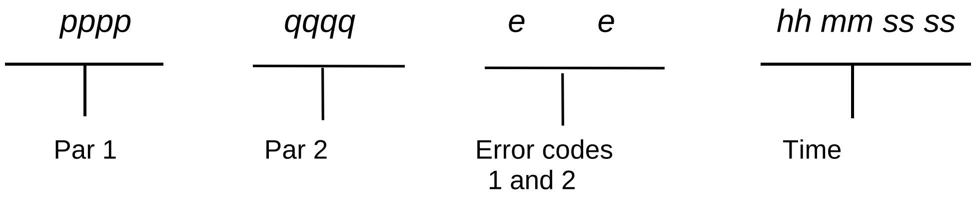
5.2. BINARY 'DUMP MODE' OUTPUT

Dump Mode format, in which results are compressed into a binary form to occupy fewer bytes, is the fastest way to output data from the SI 1280.

The ECI uses the following commands for binary output:

GP3 -	Long dump (binary with time)
GP4 -	Short dump (binary without time)

With 'long dump' selected, each set of readings is output with the format:



Each parameter consists of a four-byte floating-point number, compared with the twelve bytes needed for ASCII. Each error code is carried by a single byte which takes the value "0" for no error, "1" for current DVM overload, and "2" for voltage DVM overload.

The time reading consists of eight binary-coded decimal digits, each pair of digits being carried by a byte. The time reading thus consists of tens and units of hours, tens and units of minutes, tens and units of seconds, and tenths and hundredths of seconds.

The following section 5.2.1 explains how to decode the four parameter bytes from binary to decimal notation.

Output separators and terminators are not sent in dump mode format. However, EOI (End or Identify) is automatically asserted simultaneously with the last byte if either [cr+EOI] or [crlf+EOI] has been selected via the Data Output - GPIB commands.

With 'short on' selected, the time data is omitted but the output format is otherwise identical.

The FRA uses the following command for binary output:

OP2,2 - Binary dump

The output format is:

aaaabbbbffff

which represents 3 X 4 byte floating point number (see section 5.2.1), where:

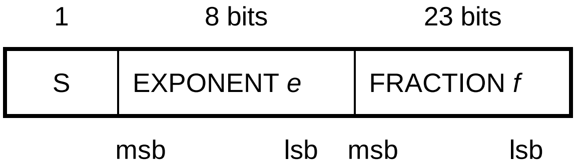
aaaa = in phase data
bbbb = quadrature data
ffff = frequency data

The following section 5.2.1 explains how to decode the four parameter bytes from binary to decimal notation.

Output separators and terminators are not sent in dump mode format. However, EOI (End or Identify) is automatically asserted simultaneously with the last byte if either [cr + EOI} or [crlf+ EOI] has been selected via the Data Output - GPIB commands.

5.2.1. 4 - Byte Floating Point Format

The floating point format conforms to the ANSI / IEEE Standard 754. It consists of a 4-byte (32 bit) floating point number:



S = Sign bit
msb = most significant bit
lsb = least significant bit

Provided that the exponent value "e" is greater than 0 and less than 255, the value that the floating point number represents is obtained from the expression:

$(-1)^s\ 2^{e-127}\ (1.f)$

in which,

$(-1)^s$ represents the sign, [S = 0 gives a positive number, since $(-1)^0 = 1$,and
S = 1 gives a negative number, since $(-1)^1 = -1$]

2^{e-127} represents the base (2) raised to the power of an exponent *e* -127,

and (1.f) represents the fixed point part.

Note that the “1" before the decimal point in the fixed point part is implied; it is not included in the floating point number. Special cases, where e = 0 or 255, are:

1. If e = 0 and f = 0 then the number is zero.
2. If e = 255 and f = 0 then the number is ± ∞.
3. If e = 0 and f ≠ 0 then the number is a denormalized number $(-1)^s\ 2^{e-127}$ (0.f).
4. If e = 255 and f ≠ 0 then the number is a not a number, regardless of s.

EXAMPLE: Converting a 4-byte floating point number to decimal.

Byte 1 contains 01000001_2 (most significant byte)
Byte 2 contains 11000000_2
Byte 3 contains 00000000_2
Byte 4 contains 00000000_2 (least significant byte)

Arranged with the most significant byte on the left and the least significant byte on the right, these bytes form the following binary number:

		EXPONENTe	*FRACTION f*
Sign bit →	0	10000011	10000000000000000000000

From this:

The sign bit value of '0'indicates that the number is positive

The exponent value of $10000011_2 = 131_{10}$

represents an exponent of

$2^{131-127}$

$= 2^{4}$

$= 16_{10}$

The fraction part $= 1.f_{2}$

$= 1.10000000000000000000000_{2}$

$= 1.5_{10}$

Therefore the decimal equivalent of the floating point number is

1.5 x 16 = + 24

6. SERIAL POLL/PARALLEL POLL

The instrument can be configured to request service from a GPIB controller when a particular event has occurred, e.g. on *end of operation*, or *measurement ready*. The controller may then poll the devices on the GPIB to find the source of the request. Two types of polling can be used, *serial* or *parallel*.

6.1. SERIAL POLL

In serial poll the controller addresses each device separately (serially) and obtain a "Status Byte" from each device. This will reveal which of the instruments was causing the SRQ and the reason. Due to the serial action, the process tends to be slow, but yields unique information.

6.1.1. ECI/FRA Status Byte

The ECI and the FRA each have a status byte, but the bit significances are the same in each case. Therefore the following description covers both devices.

A serial poll interrogates each device in turn, to examine its *status byte*. The status byte holds the status of all events for which it is possible to request service:

128 Data Ready	64 RQS	32 Not Used	16 End of File	8 Not used	4 End of Sweep	2 End of Measure	1 Error

When an event occurs the corresponding bit is set to '1'. The events represented are:

Bit	**Event**	**Comment**
128	Data Ready	Set when data is available for ASCII or binary output to the GPIB.
64	Request for service.	Set to '1' when the instrument requests service. This bit allows the controller to identify the source of the request. All 8 bits of the status byte appear on the DIO lines when the instrument is serial polled.
32	Not Used.	
16	End of File.	Final result written to, or read from, history file.
8	Not used.	
4	End of Sweep.	Instrument has finished a sweep.
2	End of Measure.	Instrument has finished a measurement.
1	Error.	Set when an error affecting the other bits in the STB is detected. It does not register all possible SI 1280 errors.

The values of all the bits, except bit 1 and bit 64, continuously follow status changes. If, for example, the instrument is measuring repetitively (RECYCLE) bit 2 is set to '1' as each measurement is completed, then reset to '0' as a new measurement starts.

6.1.2. Setting up a Service Request

The instrument can be enabled to request service (and set the RQS bit) for one or more specified events. Alternatively, if several of the status bits must he monitored simultaneously, the controller can be programmed to serial poll at regular intervals without using a SRQ at all. Remember that the SI 1280 is actually two devices, so there are two status bytes to be set up.

To enable a service request, send the remote command.

SV*n*

where *n* is an integer, in the range 1 to 255, which represents an event, or combination of events, for which service is to be requested.

For example, *SV*8 (STB = 00001000) results in a service request at the next *end of plot whilst SV*25 (STB = 00010101) results in a service request at whichever event occurs first out of *end of file, end of sweep*, or *error*.

Once the instrument has requested service, it must be reconfigured before it can request service again. The query commands ?SV read the status request mask.

Code SV0 unconfigures an existing interrupt, and also clears bit 1 ('error').

6.2. PARALLEL POLL

For parallel poll operation previously assigned devices are allocated a specific data line (out of 8 available) such that, upon a parallel poll instruction, only those devices allocated to specific lines are able to energise them. In this way the controller may separate up to 8 instruments in a unique fashion; or by sharing the 8 lines amongst two or more devices provided unlimited polling facilities.

In practice the user program would use a parallel poll to establish which device is requesting service, and then a serial poll to find out the reason.

6.2.1. Setting up a Parallel Poll

The instrument can he configured to give a parallel poll *true/false* response on a selected GPIB data line, to indicate whether or not the instrument is requesting service. However, the instrument must first be configured for serial poll.

To set up a parallel poll configuration send the remote command:

PP*n*

where n is an integer from 1 to 8, defining which GPIB data line is to carry the response.

Sending PP0 unconfigures parallel poll.

To select the sense of the parallel poll line send the remote command SE*n*, where *n* = 1 signifies *true* and *n* = 0 signifies *false*.

The parallel poll response is also cleared by any change to the serial poll value, and by power-off.

Unlike serial poll, parallel poll need not he reconfigured after each service request.

7. SI 1280 COMMAND SUMMARY

7.1. ECI COMMANDS

Note that the underlined commands relate to the default settings.

7.1.1. Cell Polarization

Command	Polarization
PO0 **PO**1	Sets ECI to operate as a potentiostat. Sets ECI to operate as a galvanostat.
PV*F* **PC***F*	Defines the d.c. potential that is to be applied across RE1 and RE2 for the potentiostat. (*F* = -14.5V to + 14.5V)* Defines the direct current that is to flow through the cell for the galvanostat. (*F* = -2A to + 2A)*
PI0 **PI**1	An a.c. signal from the FRA is added to the polarization selected by **PV***F* or **PC***F*, at a gain of X 1. An a.c. signal from the FRA is added to the polarization selected by **PV***F* or **PC***F*, at a gain of X 0.01 (-40dB).
PW1 **PW**0	Selects polarization ON. (ECI assumes ON mode.) Selects polarization OFF. (ECI assumes standby mode.)

F = floating point number (± *n.nnnn*E ± *nn*).

* Note that the polarization potential and current ranges of the 1280A are -12.8V to + 12.8V and -1A to + 1A.

7.1.2. Standby State

Command	Standby State	Valid Measurements
BY0	Full stand-by.	DVM I/P, POL, ΣPOLL, CE.
BY1	Half stand-by.	RE1, RE2, ΔRE, ΔRE-Bi, POL, ΣPOL, CE, I, I-Bi.

7.1.3. Polarization On Mode

Command	On Mode
ON0	Prepares the ECI for the **Pol V/I** mode.
ON1	Prepares the ECI for the **Rest V/I** mode.

7.1.4. Control Loop Bandwidth

Command	**Bandwidth**	
SY0	Bandwidth limit ON.	
SY1	Bandwidth limit OFF.	
	Galvanostat:	
GB0	Type A	150kHz*
GB1	Type B	100kHz*
GB2	Type C	> 7.5kHz**
	Potentiostat:	
PB0	Type A	600kHz***
PB1	Type B	360kHz***
PB2	Type C	> 1MHz***
PB3	Type D	> 600kHz***
PB4	Type E	24kHz***
PB5	Type F	8kHz
PB6	Type G	2.4kHz
PB7	Type H	800Hz
PB8	Type I	80Hz
PB9	Type J	8Hz

*** 12kHz when limit or cutout selected for O/L type.

** 5kHz when limit or cutout selected for O/L type.

* 25kHz when limit or cutout selected for O/L type. Stable with capacitive cells when standard resistor 100Ω.

7.1.5. Standard Resistor Selection

Command	**Standard Res. Value**	**Full-Scale Current** (At 200mV)
RR0	Auto-range	
RR1	0.1Ω	2A
RR2	1Ω	200mA
RR3	10Ω	20mA
RR4	100Ω	2mA
RR5	1kΩ	200µA
RR6	10kΩ	20µA
RR7	100kΩ	2µA
RR8 ****	1MΩ	200nA

****The 200nA range is not available on the 1280A.

7.1.6. Current Limit Selection

Command	**Full-Scale Current** (At 200mV)
IL0	2µA
IL1	20µA
IL2	200µA
IL3	2mA
IL4	20mA
IL5	200mA
IL6	2A

7.1.7. Current Off-Limit Action

Command	**Overload Action**
OL0	Cut-out. The ECI goes to the "stand-by" state and the cell current is cut off completely.
OL1	Limit. The cell current limits at the full-scale value of a fixed range (or the I LIMIT value).
OL2	No limit. The cell current is allowed to rise to 2A.

7.1.8. IR Compensation Type and On/Off

Command	**Function**
CT0 **CT**1	Selects feedback IR compensation. Selects sampled IR compensation.
CC0 **CC**1	IR compensation OFF. IR compensation ON.

7.1.9. Feedback IR Compensation

Command	**Function**
IC*F*	Defines the value of the parasitic resistance Rp (0Ω to 1MΩ)

F = floating point number (± *n.nnnn*E ± *nn*).

7.1.10. Sampled IR Compensation

Command	**Function**
IN*F* **IP***I*	Defines the cell current *off* time (26.6µs to 1.36ms). Defines the cell current *off:on* ratio (1:*I*) where I = integer 1 to 255.
RO0 **RO**1	Selects actual sampled waveforms for output to FRA. Selects sampled and held waveforms for output to FRA.

F = floating point number (± *n.nnnn*E ± *nn*).

7.1.11. Real Part Correction

Command	**Function**
CC0 **CC**2	Real part correction OFF. Real part correction ON.
RP*F*	Defines the value of the parasitic resistance Rp (0Ω to 1MΩ).

F = floating point number (± *n.nnnn*E ± *nn*).

7.1.12. ECI Output Conditioning Facilities

Command	Function
VT0 **VT**1	Voltage bias reject, auto. Voltage bias reject, fixed.
VR*F*	Fixed rejection voltage. (*F* = 0V to ± 14.5V)*
IT0 **IT**1	Current bias reject, auto. Current bias reject, fixed.
IR*F*	Fixed rejection current. (*F* = 0A to ± 2A)*
BR0 **BR**1	Bias reject OFF. Bias reject ON.
FI0 **FI**1	Filter OFF. Filter ON. A 10Hz low-pass filter is switched into the measured voltage and current outputs.
VX0 **VX**1	Voltage amplification off. X 10 voltage amplification.
IX0 **IX**1	Current amplification off. X 10 current amplification.

F = floating point number (± *n.nnnn*E ± *nn*).

On the 1280A the range of fixed rejection voltage is 0V to ± 12.8V and the range of fixed rejection current is 0A to ± 1A.

7.1.13. Sweep Definition

Command	Function
DL*F*	Delay in seconds (*F* = 0 to 100,000s)
SM*F*	Number of segments (*F* = 1 to 99,999; default = 2)
OF0 **OF**1	Off mode = standby Off mode = freeze*
SW0 **SW**1 **SW**2	Stop sweep. Start ramp sweep. Start stepped sweep.
?ST	Returns the sweep status.

F = floating point number (± *n.nnnn*E ± *nn*).

*On the 1280A the 'freeze' off mode is not available: the OF*I* command has no effect.

7.1.14. Ramp Sweep: Segment Definition

Command	Function	
VA*F*	Voltage level V_A	(*F* = 0 to ± 14.5V)*
JA*F*	Current level I_A	(*F*= 0 to ±2A)*
TA*F*	Segment time T_A	(*F* = 10ms to 100,000s)*
VB*F*	Voltage level V_B	(*F* = 0 to ± 14.5V)*
JB*F*	Current level I_B	(*F* = 0 to ±2A)*
TB*F*	Segment time T_B	(*F* = 10ms to 100,000s)*
VC*F*	Voltage level V_C	(*F* = 0 to ± 14.5V)*
JC*F*	Current level I_C	(*F*= 0 to ±2A)*
TC*F*	Segment time T_C	(*F* = 10ms to 100,000s)*
VD*F*	Voltage level V_D	(*F* = 0 to ± 14.5V)*
JD*F*	Current level I_D	(*F*= 0 to ±2A)*
TD*F*	Segment time T_D	(*F* = 10ms to 100,000s)*

F = floating point number (± *n.nnnn*E ± *nn*).

*On the 1280A the voltage level range is 0 to ± 12.8V, and the current level range is 0 to ± 1A.

7.1.15. Stepped Sweep: Segment Definition

Command	Function	
SA*F*	Voltage level V_A	(*F* = 0 to ± 14.5V)*
KA*F*	Current level I_A	(*F*= 0 to ±2A)*
SB*F*	Voltage level V_B	(*F* = 0 to ± 14.5V)*
KB*F*	Current level I_B	(*F* = 0 to ±2A)*
SC*F*	Voltage level V_C	(*F* = 0 to ± 14.5V)*
KC*F*	Current level I_C	(*F*= 0 to ±2A)*
SD*F*	Voltage level V_D	(*F* = 0 to ± 14.5V)*
KD*F*	Current level I_D	(*F*= 0 to ±2A)*
TE*F*	Time per step	(*F* = 10ms to 100,000s)
VS*F*	Volts per step	(*F* = 5µV to ± 29.0V)*
IS*F*	Current per step	(*F* = 5pA to 4A)*

F = floating point number (± *n.nnnn*E ± *nn*).

*On the 1280A the voltage level range is 0 to ± 12.8V, and the current level range is 0 to ± 1A. The other ranges for stepped sweep are: time per step = 10ms to 100,000s, volts per step = 100µV to 25.6V, and current per step = 1nA to 2A.

7.1.16. DVM Control Functions

Command	Function	
DG0	Number of Digits	5 X 9
DG1		4 X 9 (50Hz ac supply)
DG2		4 X 9 (60Hz ac supply)
DG3		3 X 9
RG0	Input Range*	Autorange
RG1		200mV
RG2		2V
RG3		20V
RG4		200V
TR0	Measurement Trigger	Single measurement.
TR1		Continuous measurements.
TR3		Sweep synchronized measurements.
DC0	Drift correction on.	
DC1	Drift correction off.	
AV0	Averaging off.	
AV1	Averaging on.	
NU0	Null Off.	
NU1	Null on.	
NU2	Null evaluate.	
RU0	Digital voltmeter OFF.	
RU1	Digital voltmeter ON.	

. *For voltage measurement only.

7.1.17. Output Parameter Section

Command			
Par 1	**Par 2**	**Parameter**	
PX0	**PY**0	CE	Counter electrode.
PX1	**PY**1	RE1	Reference electrode 1.
PX2	**PY**2	RE2	Reference electrode 2.
PX3	**PY**3	ΔRE	$V_{RE\ 1}$ -V_{RE2}. (FRA Channel 2)
PX4	**PY**4	ΔRE - Bi	ΔRE - $V_{BIAS.}$
PX5	**PY**5	I	Cell current. (FRA Channel 1)
PX6	**PY**6	I -Bi	Cell current - bias current.
PX7	**PY**7	δΔRE ÷ δI	Polarization resistance.*
PX8	**PY**8	δI ÷ δΔRE	Polarization conductance.*
PX9	**PY**9	POL	Dc polarization from ECI.
PX10	**PY**10	ΣPOL	POL + dc bias from FRA generator.
PX11	**PY**11	DVM	Zero (for nulling).

*Available only with Sweep.

7.1.18. History File Function

Command	Function
FL0	Closes the file.
FL1	Opens the file.
FS*l*	Defines the file size (number of results). *l* =1 to ~ 450.
UF*l*	Outputs result at file location *l*. *l* =1 to ~ 450.
VF1	Clears the file, i.e. erases the file contents.
VF2	Outputs the contents of the file to the GPIB.
?FP0	Returns the number of results filed. (0 to ~ 450)
?FP1	Returns the file pointer (= location of last result filed).
?NR	Returns the number of readings taken. (0 to 99999)

7.1.19. GPIB Functions

Command	Function
GP0	Output off.
GP1	Long on: Compressed ASCII format, with time.
GP2	Short on: Compressed ASCII format, without time.
GP3	Long dump: Binary format, with time.
GP4	Short dump: Binary format, without time.
OS0	Output separator = comma (,)
OS1	Output separator same as terminator
OT0	Output terminator = cr lf
OT1	Output terminator = cr lf and EOI
OT2	Output terminator = cr
OT3	Output terminator = cr and EOI
SV*l*	Specifies which serial poll bit(s) may initiate a service request. *l* = 1, 2, 4, 8, etc, to define a specific bit, or several such integers may be added together to define several bits, e.g. 10 defines bits 2 and 8.
PP*l*	Specifies which GPIB data line the ECI is to assert, to identify itself in a parallel poll. *l* = 0 to 8.
PS0	Parallel poll sense true: "1" (Low on bus) = service request.
PS1	Parallel poll sense false: "0" (High on bus) = service request.

7.1.20. Break and Self-test Functions

Command	Function	
BK0	Break	Halts the present operation of the ECI and returns it to the standby state.
BK1 or **BK**2	Check	Starts a self-test of the ECI digital circuitry. On completion of this test the ECI is set to the standby state; the DVM and sweep are switched off.
BK3	Reset	Returns all control setups to the default state, but retains the content of the history file.
BK4	Initialize	Returns all control setups to the default state and clears the history file.

7.1.21. Self-test Results

Command	Function
?TS	Overall result of self-test.
?TS1	RAM, 1 or 2.
?TS2	ROM, 1 or 2.
?TS3	Timer, 1, 2 or 3.
?TS4	GPIB

7.1.22. Last Error Query, Clear Error, ECI Software Version, Time

Command	Function
?ER	Returns the code of the last error detected by the ECI.
CE	Clears the "last error" code and the two error codes on the ECI status page.
?VN	Returns the status and issue of the ECI software.
TM*I,I*	Sets a starting value for the elapsed time. (*I,I* = *hh,mm*)

7.2. FRA COMMANDS

7.2.1. Generator Signal Definition

Command	Function
WV0 **WV**1 **WV**2	Waveform Sinewave Squarewave Triangular wave
FR*F*	Frequency *F* = 1mHz to 20kHz (default value = 100Hz)
AM*F*	Amplitude *F* = 0V to 7V sine, 5.1V triangle, 7.9999V square
BI*F*	Bias *F* = -10V to + 10V

F = floating point number (± *n.nnnnE* ± *nn*).

7.2.2. Sweep Setup

Command	Function
MA*F* **MI***F*	Max. frequency *F* = 1mHz to 20kHz (default = 20kHz) Min. frequency *F* = 1mHz to 20kHz (default value = 100Hz)
GS*I*	Log sweep *I* = 2 to 9999 points (default = 100 points)
SE*0* **SE**1 **SE**2	Sweep off Sweep up Sweep down

F = floating point number (± *n.nnnnE* ± *nn*).

7.2.3. Generator Start and Stop Commands

Command	Function
RG	Run Generator Allows the generator to be started without starting the analyzer.
SG	Stop Generator Stops the generator and, if running, the analyzer. The final state of the generator output depends on the stop mode.
SM0 **SM**1	Freeze output Selects stop mode-freeze. Kill output Selects stop mode-kill.
SQ*0* **SQ**1 **SQ**2 **SQ3**	Stop on Quadrant Holds the generator output at vector angle 0° ″ ″ ″ ″ 90° ″ ″ ″ ″ 180° ″ ″ ″ ″ 270°

7.2.4. Analyzer Setup

Command	Function
RA1,0 (**RA**2,0)	Channel 1 (Channel2) Input range: Auto
RA1,1 (**RA**2,1)	30mV
RA1,2 (**RA**2,2)	300mV
RA1,3 (**RA**2,3)	3V
RA1,4 (**RA**2,4)	30V
RA1,5 (**RA**2,5)	300V
HA1 to **HA**8	Defines the harmonic to be measured. "1" = fundamental, "2" = second harmonic, and so on.
IS*F*	Integration time *F* = 0.1s to 10,000s (default = 0.1s)

F = floating point number (± *n.nnnnE* ± *nn*).

7.2.5. Analyzer Start and Stop Commands

Command	Function
SI	SIngle Starts the generator, if stopped, and, after a one second delay*, starts a single measurement.
RE	REcycle Starts the generator, if stopped, and, after a one second delay*, starts continuous measurements.
SA	Stop Analyzer Stops the analyzer, without completing the 1 measurement cycle.

*Delay applicable only if generator was not running.

7.2.6. Result Source

Command	Function
SO0100	Result source is Channel 1. (Channel 2 is not measured.)
SO0200	Result source is Channel 2. (Channel 1 is not measured.)
SO0201	Result source is Channel 2 ÷ Channel 1.

7.2.7. Result Scaling

Command	Function
FN0	Sets scaling to divide by unity, i.e. scaling off.
FN1	Sets scaling to divide by *r*,*q*. (*r* and *q* are defined by **RF***F* and **TF***F*)
FN2	Sets scaling to divide by last result.
FN3	Sets scaling to divide by last magnitude.
RF*F*	Defines *r* value to be used by **FN**1 command. *F* = -10^9 to + 10^9
TF*F*	Defines *q* value to be used by **FN**1 command. F= -180° to + 180°

F = floating point number (± *n.nnnnE* ± *nn*).

7.2.8. History File

Command	Function
?NR	Function Returns number of results written to file. (This is not necessarily the same as the file population, as earlier results may have been overwritten.)
?FP0	Returns the file population, e.g. the number of results actually in the file.
?FP1	Returns the location number of the result presently being accessed.
?FS	Returns the overall file size (> 500). (This is constant for any issue of firmware, but may vary between issues.)
UF*I*	Outputs result at file location *I* *I* = 1 to ~ 400
FO	Output all results in file.

7.2.9. Result Coordinates

Command	Function
CO0	Selects coordinates *a* + *j* *b*.
CO1	Selects coordinates *r*, *q*.
CO2	Selects coordinates *r*(dB), *q*.

F = floating point number (± *n.nnnnE* ± *nn*).

7.2.10. GPIB Functions

Command	Function
OP2,0	Output off.
OP2,1	ASII: Compressed ASCII format.
OP2,2	Binary: Binary format. Fast "dump" output, for subsequent data processing.
OS0	Output separator = comma (,)
OS1	Output separator same as terminator.
OT0	Output terminator = cr lf
OT1	Output terminator = cr lf and EOI
OT2	Output terminator = cr
OT3	Output terminator = cr and EOI
SV*I*	Specifies which serial poll bit(s) may initiate a service request. *I* = 1, 2, 4, 8, etc, to define a specific bit, or several such integers may be added together to define several bits, e.g. 10 defines bits 2 and 8.
PP*I*	Specifies which GPIB data line the ECI is to assert, to identify itself in a parallel poll. *I* = 0 to 8.
PS0	Parallel poll sense true: "1" (Low on bus) = service request.
PS1	Parallel poll sense false: "0" (High on bus) = service request.

7.2.11. BREAK / SELF-TEST

Command	Function	
BK	Break	Halts the present operation of the FRA and returns it to the ready state. Control setups unaffected. History file unaffected.
TT0	Drift Correct	Measures and nullifies any offsets due to drift that may be present in the analyzer input amplifiers. Actioned at next measurement. Also actioned automatically every minute or once per integration period, whichever is the longer.
TT1	Initialize	The generator is stopped, all control setups are returned to the default state the history file is cleared, and the elapsed time clock is set to zero.
TT2	Reset	Exactly the same effect as initialize (**TT**1) except that the content of the history file is retained.
TT3	Check	Starts a simple self-test of the FRA digital circuitry. Any failure causes the test routine to abort and a fail code is generated.
TT4	Test	Starts a self-test of the FRA digital circuitry, both analog and digital. Any failure causes the test routine to abort and a fail code is generated. Test should be used in preference to check.

7.2.12. Self-test Results

Command	Function
?TS0	Overall result of self-test.
?TS1	RAM test result.
?TS2	ROM test result.
?TS3	Timer test result.
?TS4	*Not used.*
?TS5	*Not used.*
?TS6	Analyzer test result.

7.2.13. Last Error Query, Clear Error, FRA Software Version, Time

Command	Function
?ER	Returns the code of the last error detected by the FRA
CE	Clears the “last error” code and the two error codes on the FRA status page.
?TS4	Returns the status and issue of the FRA software.
TM *I,I*	Sets a starting value for the elapsed time. (*I,I* = *hh,mm*)

Appendix A
Installation

Section *Page*

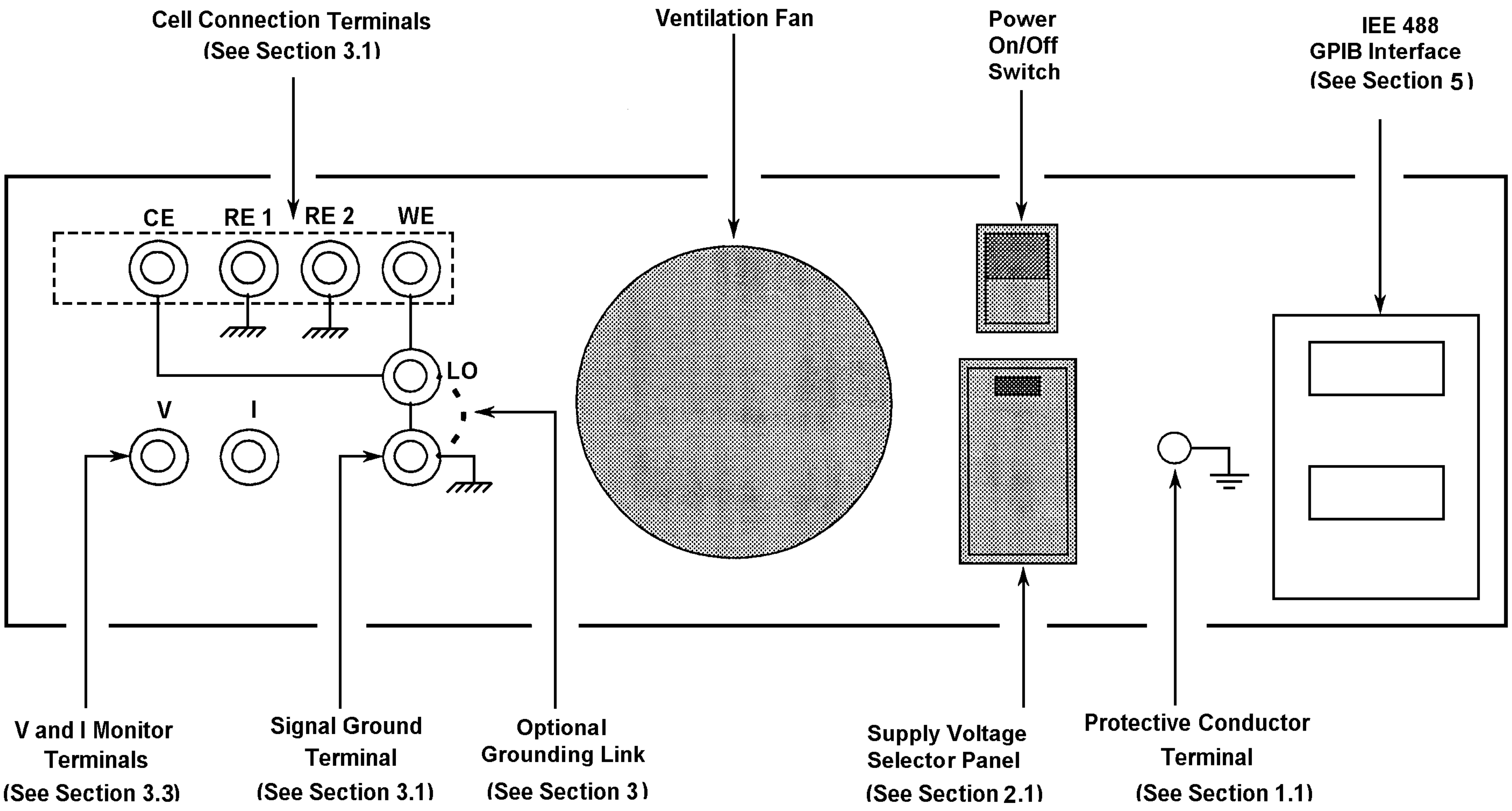


Fig A.1 Back panel layout of the SI1280B

(Note that this differs from the back panel layout of the SI1280A. The main differences are that the SI1280A has the power on/off switch on the front panel, has no V and I monitor terminals, and the layout of the GPIB Interface is different.)

1. SAFETY PRECAUTIONS

The equipment described in this manual has been designed in accordance with EN61010-1 Safety Requirements for Electronic Measuring Apparatus, and has been supplied in a safe condition. To avoid injury to an operator or service technician the safety precautions given below, and throughout the manual, must be strictly adhered to, whenever the equipment is operated, serviced or repaired.

The equipment is designed solely for electronic measurement and should be used for no other purpose. Solartron accept no responsibility for accidents or damage resulting from any failure to comply with these precautions.

1.1. GROUNDING

To minimize the hazard of electrical shock it is essential that the equipment is connected to a protective ground whenever the power supply, measurement or control circuits are connected, even if the equipment is switched off.

A ***PROTECTIVE GROUND*** is connected via the ac supply cord. The cord must be plugged into an ac line outlet with a protective ground contact. When an extension lead is used, this must also contain a ground conductor. Always connect the ac supply cord to the supply outlet before connecting the control and signal cables; and, conversely, always disconnect control and signal cables before disconnecting the ac supply cord. The ac ground connection must have a continuous current rating of 25A.

A high quality ***SIGNAL GROUND***, for measurement purposes, is provided through a terminal on the rear panel. This is linked internally to *PROTECTIVE GROUND*.

A *PROTECTIVE CONDUCTOR* terminal, linked directly to the *PROTECTIVE GROUND*, is available on the rear panel of the instrument. This can be used when it is required to bond the instrument to the metalwork of a rack or cabinet. Normally the instrument is grounded through the ground conductor of the ac supply cord: where both protective grounds are used it must be ensured that these grounds are, and will remain, at the same potential.

1.2. AC SUPPLY VOLTAGE

Before switching on the equipment ensure that the ac voltage selector is set to correspond with the local ac supply voltage.

Never operate the equipment from a line voltage or frequency in excess of that specified for the voltage selector setting used. Otherwise, the insulation of internal components may break down and cause excessive leakage currents.

1.3. FUSES

Before switching on the equipment check that the fuses accessible from the exterior of the equipment are of the correct rating. The rating of the ac line fuse must be in accordance with the voltage of the ac supply.

Should any fuse continually blow, do not insert a fuse of a higher rating. Switch the equipment off, clearly label it "unserviceable" and inform a service technician.

1.4. EXPLOSIVE ATMOSPHERES

NEVER OPERATE the equipment, or any sensors connected to the equipment, in a potentially explosive atmosphere. It is NOT intrinsically safe and could possibly cause an explosion.

1.5. SAFETY SYMBOLS

For the guidance and protection of the user, the following safety symbols appear on the equipment:

SYMBOL	MEANING
	**Refer to the technical manual for detailed instructions of use.**
	**Ground point. Internally connected to the protective conductor terminal and intended as a Ov reference point for measurements.**
	**Protective conductor terminal.**

1.6. AVOID UNSAFE EQUIPMENT

The equipment may be unsafe if any of the following statements apply:

- The equipment shows visible damage.
- The equipment has failed to perform an intended operation.
- The equipment has been subjected to prolonged storage under unfavorable conditions.
- The equipment has been subjected to severe physical stress.

If in any doubt as to the serviceability of the equipment, don't use it. Get it properly checked out by a qualified service technician.

1.7. LIVE CONDUCTORS

When the equipment is connected to its measurement inputs or supply, the opening of covers or removal of parts could expose live conductors. The equipment must be disconnected from all power and signal sources before it is opened for any adjustment, replacement, maintenance or repair. Adjustments, maintenance or repair, must be done only by qualified personnel, who should refer to the Maintenance Manual.

1.8. EQUIPMENT MODIFICATION

To avoid introducing safety hazards, never install non-standard parts in the equipment, or make any unauthorized modification. To maintain safety, always return the equipment to Solartron for service and repair.

2. A.C. SUPPLY

2.1. VOLTAGE SELECTOR

Check that the setting of the line voltage selector on the rear panel corresponds with the nominal line voltage of the local a.c. supply. (The setting in use is displayed in a small window in the selector flap.)

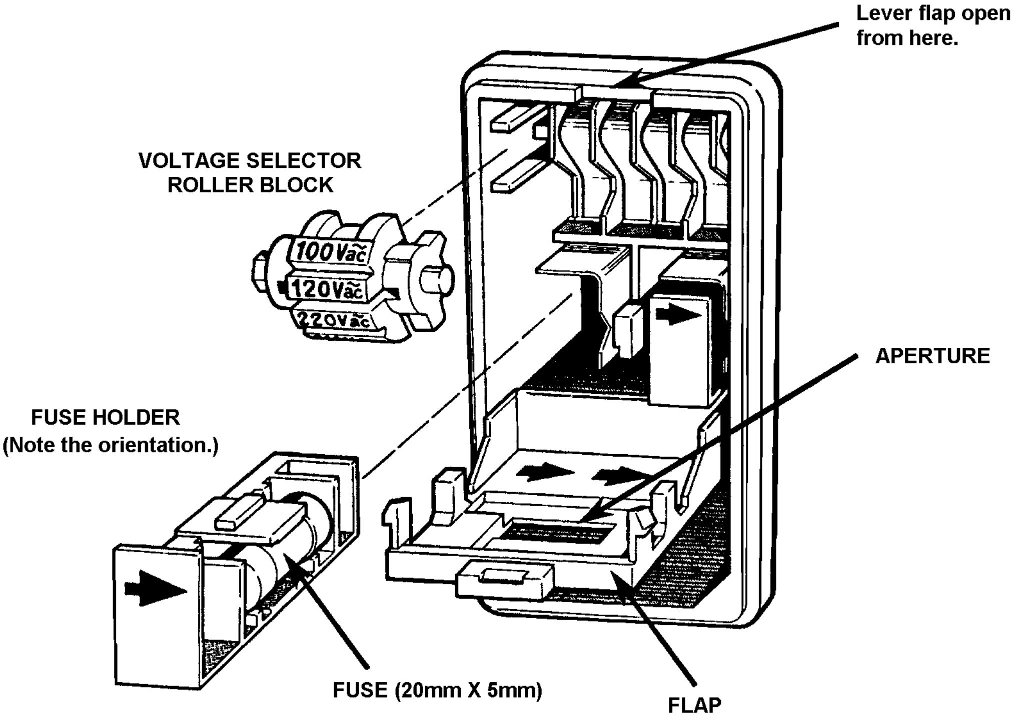


Fig. A.2 *Line voltage selector.*

Table A.1 lists the settings available and the corresponding line voltage ranges.

Table A.1 Line Voltage Selector

Selector Setting	Line Voltage Range
100V	90 - 110V
120V	108 - 132V
220V	198 - 242V
240V	216 - 264V

In some localities the nominal line voltage may not correspond exactly with any selector setting. In such a case, choose the selector setting whose line voltage range includes the nominal line voltage. Should the nominal line voltage he covered by two ranges, choose the lower of the two: this ensures that the d.c. supplies of the SI 1280 are fully attained.

For example: (a) in the case of a 110V supply choose the "100V" setting or (b) in the case of a 230V supply choose the "220V" setting.

Should you need to change the line voltage setting it is a simple matter to lift the selector flap with a thin-bladed screwdriver and turn the roller block so that the correct voltage appears in the window when the flap is closed. Fig A.2 shows the line voltage selector flap open to reveal the roller block inside.

Changing the line voltage selector setting sometimes necessitates inserting a.c. supply fuses of a different value. These fuses are located below the roller block, as shown in Fig A.1. The fuse values are:

1 A, SLO-BLO (T1,0A) for 220V or 240V operation
2A, SLO-BLO (T2,0A) for 100V or 120V operation

These fuses must be 20mm x 5mm cartridge type.

2.2. POWER SWITCH

The power switch of the SI1280B is situated on the rear panel, adjacent to the supply voltage selector panel. The power switch of the SI1280A is situated on the rear panel.

2.3. POWER CABLE

Connect the SI 1280 to the a.c. supply with the power cord provided. At one end the power cord has an IEC socket, which fits into a mating connector on the SI 1280, and at the other end is a power plug appropriate to the country of destination.

Some countries have no fixed preference of power plug and for these the power cord is provided only with an IEC socket. The wires at the other end of the cord should be connected to the user's power source, via a suitable connector, according to the following colour code:

BROWN = LIVE
BLUE = NEUTRAL
GREEN/YELLOW = GROUND

An IEC socket and power cord other than the one supplied may be used, but it must be correctly wired as shown in Fig A.3.

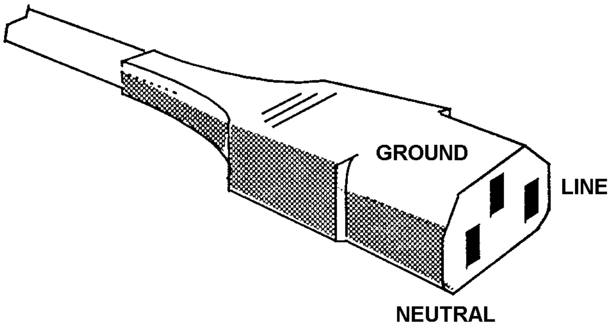


Fig.A.3 *IEC power socket connections.*

The ground lead should be capable of carrying 25A.

3. SI1280 INPUTS AND OUTPUTS

All input and output connections to the SI1280 are made at terminals situated on the rear panel. Figures A.4 through A.6 show how to connect a four-, three-, or two- terminal electrochemical cell to these terminals. The function of each terminal is explained in Sections 3.1 through 3.3.

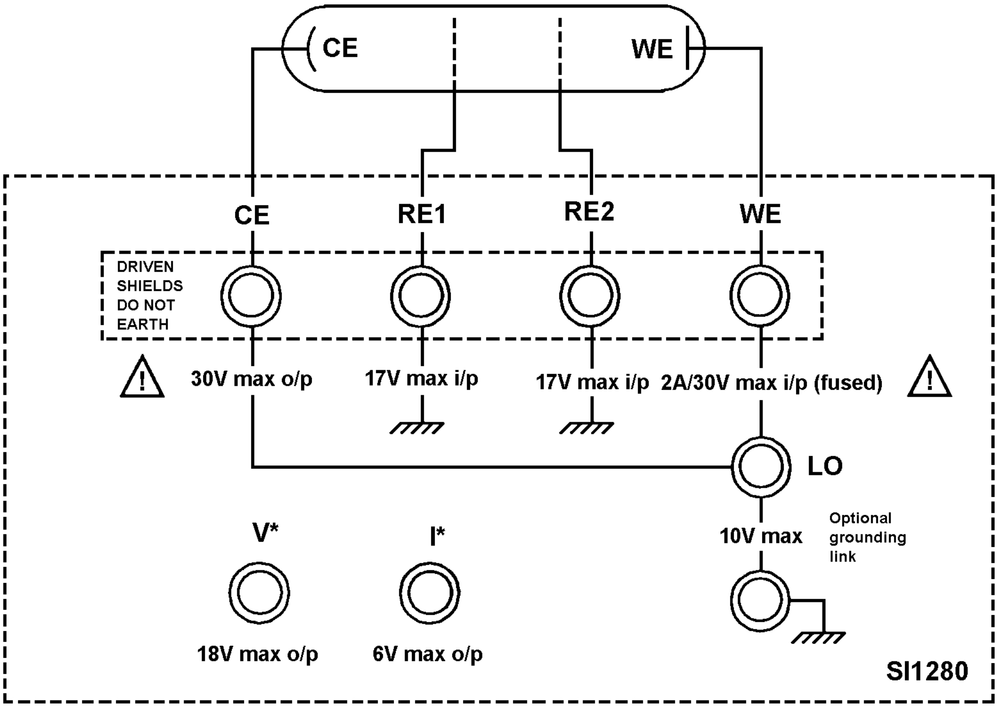


Fig A.4 *Four-terminal cell connections. (*The V and I terminals are not available on the SI1280A.)*

3.1. CE, WE, LO AND GROUND

The polarization voltage or current is applied to the cell through the counter electrode terminal CE and the working electrode terminal WE. The current into the WE terminal is the measured cell current.

The screen connections (outers) of the BNC connectors for CE, RE1, and RE2 are connected internally to screen driver amplifiers. This enables the outers to closely follow the inners. The same is true for the WE connector, but only on ranges RR1 to RR4: on ranges RR5 to RR8 the WE screen is connected to an active ground. Therefore the screens of these connectors must not be connected to ground, to any other voltage, or to each other. The polarization circuit has a floating supply referenced to the LO terminal.

With in-the-field applications, such as corrosion monitoring, one of the cell electrodes, usually the working electrode, is often grounded inherently; a typical example is a steel joist, or pipe, embedded in the ground. To ensure that the polarization circuit is grounded at one point only, you are given the option of allowing the internal common return of the cell connections to float, i.e. the LO terminal is left unconnected. (This avoids polarization instability being caused by a ground loop.)

However, it is important that the cell is connected to ground somewhere. So, if the cell has no inherent connection to ground, the LO terminal and ground terminal should he connected with the link provided. See Fig A.4.

Where the cell container is metal and is grounded it is usually best to ground CE. Note, however, that this reduces the frequency response of the polarization loop. Alternatively, WE may be grounded. On ranges RR1 to RR5 this will cause the accuracy of impedance measurements to be degraded above 10kHz. On range RR6 some noise may be evident. On ranges RR7 and RR8 grounding WE is not advised.

3.2. RE1 AND RE2

The cell voltage is measured at the cell reference electrodes, which are connected to the RE1 and RE2 inputs. These inputs have a high impedance that is maintained over the frequency range by internally driven guard screens. As with the CE and WE connections, the BNC outers must NOT be connected to ground or to any other voltage. The connections to RE1 and RE2 for four, three, and two-terminal cells are shown in Figs A.4 through A.6.

CAUTION: Reversing the connections to RE1 and RE2 gives positive feedback, which results in uncontrolled cell voltage or current.

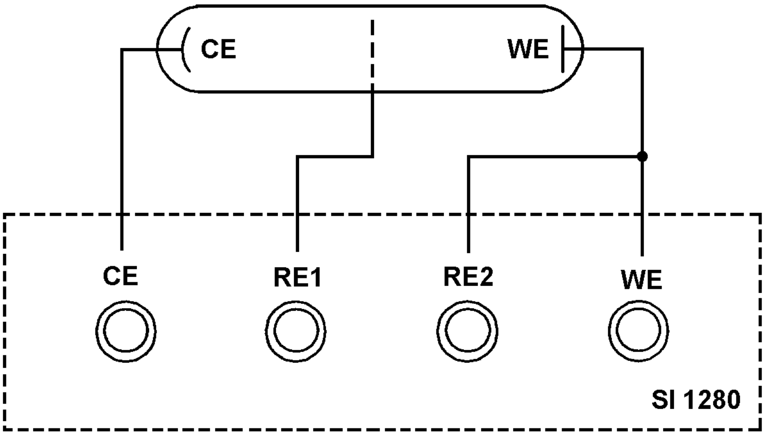


Fig A.5 *Three-terminal cell connections.*

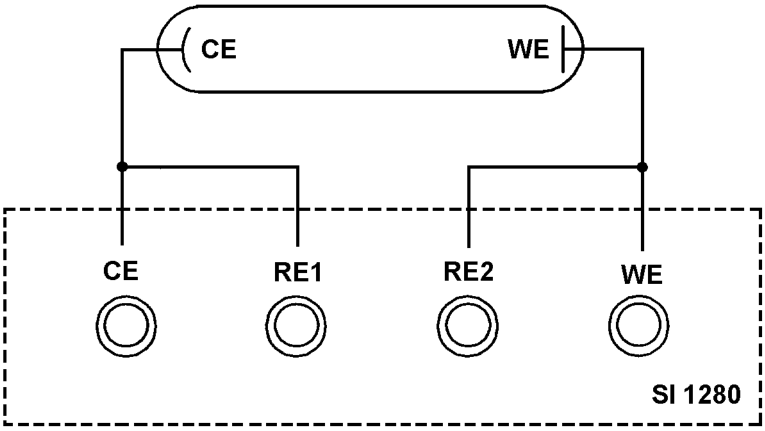


Fig A.6 *Two-terminal cell connections.*

3.3. V AND I OUTPUTS

The V and I outputs are connected to the conditioned voltages that represent the measured cell voltage and current. These outputs allow cell waveforms to be monitored on an oscilloscope. (The V and I outputs are connected internally to the FRA inputs.)

The V and I outputs are ground referenced, through the BNC outers. External voltages must NOT be connected to these outputs.

Note that the V and I outputs are not available on the SI1280A.

4. ACCESSORIES

Table 2.1 lists the accessories which should be supplied with the instrument. If any accessories are missing contact your local supplier.

Table 2.1 Accessories

Item	Use	Qty
12861 Test Module.	Gives predictable test results.	1
Technical manual.	Describes all SI 1280 functions.	1
Leads, one metre long, BNC to 4mm (color-tagged: 1 plain, 1 red, 1 green, 1 blue).	Measurement leads, for connecting cell to SI 1280.	4
Crocodile clips	To fit to each measurement lead.	4
Power cord.	Connection of a.c. supply to SI1280.	1
Fuses: 1A, 2A; glass, antisurge, 20 X 5mm.	1A for 198-264V; 2A for 90-132V.	2 of each
Fuses: 7A; glass, fast blow, 6.25 X 1.87mm.	Fitted internally to: WE, CE, and the d.c. power supplies.	4

5. GPIB CONNECTOR

Connection to the GPIB is made via the 24-way connector on the IEEE 488/GPIB interface. (See Fig A.7.) The pin connections conform to the IEEE 488, 1978 standard. Refer to Chapter 5 for more details on the GPIB and Chapter 5, Section 4, for details of the address switches. (Note that the orientation of the GPIB Interface on the SI 1280A is different to that shown in Fig A.7: the connector is rotated through 180° and the connector and address switches are aligned horizontally.)

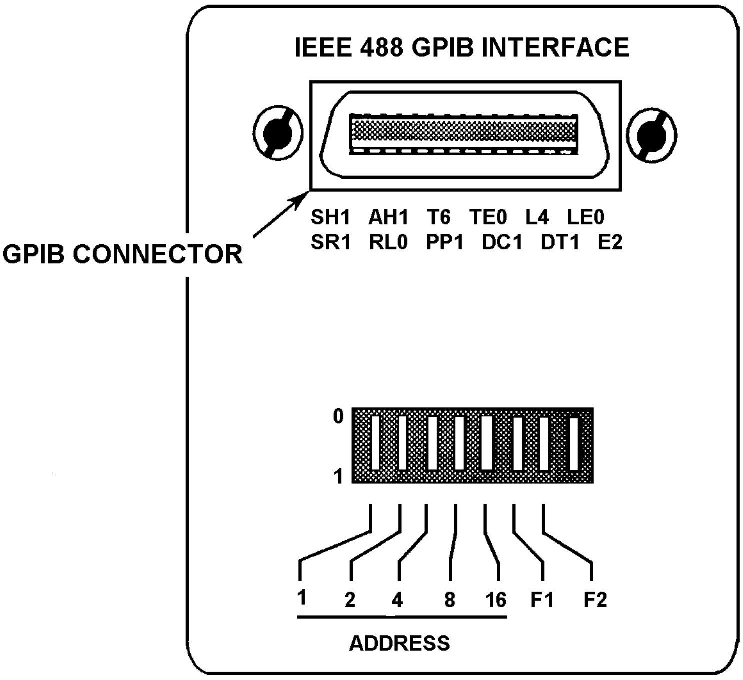


Fig A.7 GPIB Interface.

6. ENVIRONMENTAL INFORMATION

6.1. EMC

The SI1280B meets the requirement of EN50081-1 for emission and EN50082-1 for immunity. Note, however, that high levels of radiated or conducted radio frequency interference, as defined in EN50082-1, may reduce the accuracy of low level measurements. In such cases, which are unusual in practice, the interference can be mitigated by removing the source, or by screening.

6.2. VENTILATION

The instrument has fan-assisted ventilation. Air is drawn in through slots in the front panel and exhausted through a vent in the rear.

To ensure that the instrument always runs cool, keep the air inlet and outlet clear. Leave a gap of at least 4 inches (100mm) between the rear panel and any vertical obstruction such as a wall.

When the instrument is rack-mounted the rack itself should be well ventilated.

A thermal sensor monitors the internal temperature of the instrument. If this temperature approaches the working limit then error code 34 is generated. You should then check the ventilation path. If the temperature does not fall to a safe value within one minute then the instrument goes into the STANDBY state.

6.3. SITING THE INSTRUMENT

To ensure that the instrument remains in a safe and serviceable state it is important that its operating, and storage, environment conforms with the limitations stated in the SI 1280 Specification. Although the instrument is contained in a ruggedized case and conforms to a high standard of safety, it should be afforded all the precautions normally taken with electrical equipment.

Note that the instrument is not water- or damp-proof. Therefore it should not be operated in the field, without adequate protection, in rain, drizzle, mist, or in any other conditions in which moisture is liable to find ingress. Neither should it be operated in a hostile industrial environment, in the presence of sprays, corrosive atmospheres, etc.

Should the SI 1280 be required to operate in a hostile environment then it should be mounted in a protective enclosure. The instrument should always be operated within the limits of temperature, humidity and vibration stated in the SI 1280 Specification. See Appendix C.

7. RACKMOUNTING

The instrument is intended to be tray-mounted, on mounts that support the instrument from the underside of the case.

A pair of rack mounting cars, supplied in the optional Rack Mounting Kit 12801A, are substituted for the instrument finisher trims. Screws inserted through the ears and into the rack keep the instrument in place.

CAUTION: The rack mounting cars must be used only to prevent the instrument sliding out of the rack. They are not designed to support the whole weight of the instrument.

Appendix B

Error Codes

Section **Page**

1. ECI ERROR/WARNING CODE SUMMARY

1.1. GROUP 0: Command Structure Errors

01 Unknown command.

02 Argument mismatch.

03 Argument out of range.

04 Floating point format error

05 Illegal request for value.

1.2. GROUP 2: Parametric Interaction Errors

21 IRC not allowed in galvanostatic (G STAT) mode.

22 Polarisation current (POL I) and/or current bias rejection (I REJECT) inconsistent with standard resistor selected.

23 Necessary parameter in sweep not entered.

26 IRCΩ or RPCΩ inconsistent with standard resistor selected.

28 Ramp sweep: ramp rate>100V/s

Step sweep: V/STP<100µV when maximum excursion from RMP V1>200mV; I/STP X standard resistor<100µV when maximum excursion (X standard resistor) from STP I1>200ms.

29 Ramp sweep: ramp rate<100V/s

Step sweep: V/STP < 50µV when maximum excursion from RMP V1   200mV and >20mV;

I/STP X standard resistor < 50µV when maximum excursion from STP I1   200mV and >20mV.

1.3. GROUP 3: Overloads and Warnings

31 Current DVM overload.

32 Voltage DVM overload.

33 Both DVMs overload.

34 Thermal cut-out. The temperature of the output stage is monitored every 80s, and the cut-out operates if it exceeds 95°C, switching the ECI to standby. Cut-out is cancelled when the monitored temperature drops to 80°C. Excessive ambient temperatures, cooling fan failure, or high output stage power dissipation (e.g. when discharging a battery) may cause Error 34.

35 RE1 overload.

36 RE1-RE2 overload.

37 Current overload.

38 Current limit.

39 ECI standby.

1.4. GROUP 4: File Errors

44 File empty.

45 Illegal file access. Not allowed while the analyzer is running.

46 Illegal file size entry

47 File position out of bounds.

1.5. GROUP 5: Run-Time Errors

51 Commands not allowed during sweep.

52 Measurement rate not achievable in sync. sweep.

53 Null evaluation not performed or completed.

1.6. GROUP 9: Calibration Errors

91 Commands not allowed if not in calibration mode.

92 Offsets or multipliers out of range.

93 Calibration constants in error; recalibration recommended.

94 Calibration constants in error; recalibration necessary.

95 Calibration constants in error; Unit now totally uncalibrated.

2. FRA ERROR/WARNING CODE SUMMARY

2.1. GROUP 0: Command Structure Errors

01 Unknown Command.

02 Argument mismatch. The wrong type, or number of arguments was used.

03 Argument out of range.

04 Floating point format error. A floating point number was incorrectly entered e.g. 1.2.5E2 instead of 1.25E2.

05 Illegal request for value. Some parameter modes cannot be interrogated e.g. It is meaningless to send ?SG "What is the value of 'Stop Generator' "

2.2. GROUP 2: Combined Parameter

22 The combination of AMPL, BIAS and MODULATION (if used) exceeds the maximum output voltage of 15V peak.

26 *WARNING:* The selected number of points/sweep, is greater than the available file size. The sweep is still allowed to run, but some results will not be filed.

27 FRMAX < FRMIN. If SWEEP ENABLE is on it will be switched off.

2.3. GROUP 3: Generator

30 Generator stopped.

2.4. GROUP 4: HISTORY FILE

44 History file empty.

45 Illegal file access. It is illegal to display or list the History File whilst the Analyzer is running.

47 File pointer out of bounds. The user has attempted to read a History File entry that is outside the entries allocated to the file.

2.5. GROUP 7: System

70	Out of memory. No further memory is available for the operation attempted. Delete unwanted programs, or reduce program to make more room.
73	Calibration constants in error; 1 out of 3. One out of the three internal copies of calibration constants is corrupted. The FRA is still usable and measurements are valid, but contact a Solartron service facility.
74	Calibration constants in error; 3 out of 3. All of the three internal copies of calibration constants are corrupted. The FRA is still usable but measurement accuracy is not guaranteed. Contact Solartron Instruments immediately.
75	Commands not allowed if not in 'calibration mode'. The FRA must be set internally before calibration commands can be used.
76	Offsets or multipliers out of range. Attempted calibration has failed.

2.6. GROUP 8: Measurement Validity

81	Overload-common mode > 50V is not possible.

Appendix C

SI1280 Specifications

1. SI1280 ELECTROCHEMICAL MEASUREMENT UNIT SPECIFICATIONS

This specification covers both the SI1280A and the S11280B. Where the performance differs the parameters relevant to the SI1280A are shown in brackets.

1.1. MEASUREMENT CONFIGURATION

Cell connections	2, 3 or 4 Terminal, all floating	
Common mode voltage, LO to GND	10V	
Working electrode		
current measurement resistor (R_m) range	0.1Ω to 1MΩ	(1280A: 0.1Ω to 100kΩ)
full scale current ranges	200nA to 2A	(1280A: 2µ A to 2A)
maximum resolution	1.0pA	(1280A: 100pA)
limit of error	0.1%±0.05% of range	
Counter electrode		
output voltage, wrt LO	>±20V	
current, subject to thermal protection limits	2A	(1280A: 1A)
slew rate, potentiostatic control	>10V/µs	
output short-circuit protected		
Reference electrodes		
input impedance	>10GΩ	
capacitance	50pF	
current	<1nA	
maximum resolution	1µV	
limit of error	0.1%±100µV	
maximum voltage wrt GND	±10V peak	
rejection, f<20kHz	70dB	
Integration time	0.1 to 10^5s	
Harmonics measured	first 8, up to maximum 20kHz	
limits of error, relative to fundamental		
> 0.01 full scale	± 0.2dB	
> 0.001 full scale	± 1dB	

1.2. DC POLARISATION

DC polarisation		
voltage range	± 14.5V	(1280A: ±12.8V)
limits of error V<3.2V	0.2%±200µV	
V>3.2V	0.2%±2mV	
maximum resolution	100µV	
current range	±2A	(1280A: ±1A)
limit error	0.2%±0.1% of range	
maximum resolution	100pA	(1280A: ±1nA)
DC sweep: analogue ramp		
ramp rate	10µV/s to 100V/s	(1280A: 100µV to 100V/s)
minimum segment duration	10ms	
minimum increment	5mV	
DC sweep: stepped ramp		
minimum step height	5µV	
minimum step duration	10ms	
maximum step duration	10^5s	
Bandwidth, 100Ω resistive load, unity gain	>80kHz	

1.3. AC POLARISATION

Waveform	sine, square, triangular
Frequency	
range	1mHz to 20kHz
maximum resolution	1 part in 4000
Amplitude	
ranges	0 to 10V peak, 10mV resolution
	0 to 100mV peak, 100µV resolution
Distortion	<1%

1.4. IMPEDANCE MEASUREMENT

Limits of error (for a 25mV simulation of a unity gain cell with $R_{cell}= R_m$) and no error due to reference electrode bandwidth. RE1 and RE2 capacitance must be corrected to obtain accuracy at high impedance.

R_m	**Bandwidth**			
	f<1kHz	f<3kHz	f<10kHz	f<20kHz
0.1Ω to 10kΩ	0.5%, 0.5°	1.5%, 1.5°	3%, 3°	3%, 3°
100kΩ	0.5%, 0.5°	1.5%, 1.5°	3%, 3°	3%, 6°
1MΩ*	0.5%, 0.5°	not applicable	not applicable	not applicable

*Applicable to 1280B only.

1.5. IR COMPENSATION AND REAL PART CORRECTION

Current interruption	
interruption time	26.6µs to 1.36ms
off: on range	1:1 to 1:255
Feedback compensation and real part correction	
range	0 to 1000%R_m
resolution	1%R_m
limit of error	
f < 1kHz	0.2%±1% R_m,0.2°
f > 1kHz	2%±1% R_m,2°

1.6. BIAS REJECTION

Voltage	
range	±14.5V (1280A: ±12.8V)
limit of error	0.2% ± 10mV
resolution	5mV

Current	
ranges	200nA to 2A (1280A: 2µA to 1A)
limit of error	0.2%±1% of range
resolution	1% of range

1.7. INTERFACE

Parallel port	IEEE 488 (1978)

1.8. GENERAL

Power supply, switch selectable	90-100V, 108-132V, 198-242V, 216-264V 48 to 65Hz
Consumption	210VA
Temperature	
operating	0 to 50°C (32 to 122°F)
storage	-30 to 70°C (-22 to 158°F)
specification limits	10 to 30°C (50 to 86°F)
Humidity, non condensing	95% at 40°C
Vibration, tested to DEF STD 66/31	
operating	5-16Hz ±0.1mm 16-30Hz $0.98ms^{-2}$
storage	5-13Hz ±1.5mm 13-150Hz $9.8ms^{-2}$

Safety	
complies with	IEC 1010-1 (EN61010-1)
Electromagnetic Compatibility*	
complies with	EN50081-1 and EN50082-1

* Applicable to 1280B only.

Note: High levels of radiated or conducted radio frequency interference, as defined in EN50082-1, may reduce the accuracy of low level measurements. (See Appendix A, Section 6. 1, in this manual.)

Dimensions	
height	140mm (5.5ins)
width	437mm (17.2ins)
depth	457mm (18ins)
weight	11.35kg (25lbs)

1.9. ORDERING INFORMATION

1280B Electrochemical Measurement Unit

Accessories included:
- test module
- hardware operating manual
- power cord
- spare fuses
- 4 leads, 1m long, BNC to 4mm

Options:

maintenance manual	(12806001)
rack mounting kit	(12801A)
carry case	(12802A)