

Batch Measurements Using the BAS-Zahner IM6 and IM6e Impedance Analyzers

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This article discusses the various functions of the IM6 and IM6e impedance analyzers that are used to set up series of experiments, either on a single cell or on multiple cells.

The ability to program a series of experiments on a given instrument can considerably increase the efficiency of that instrument, since it allows operation of the instrument by inexperienced users, or, in some case, no users at all. Batch measurements can be set up on the BAS-Zahner IM6 and IM6e analyzers (referred to as IM6/e) on a number of different levels.

- Multiple impedance measurements on a single cell as a function of time, current, potential, or an external variable (**automatic series measurements**)

- Multiple impedance measurements on multiple cells in series as a function of time, current, potential, or an external variable (**multiple cell measurements**)
- Combinations of the different electrochemical techniques available on the IM6/e (e.g., impedance and cyclic voltammetry) (**Script**)

Automatic Series Measurement

Batch measurements of impedance spectra are set up from within the

impedance (EIS) software using the menu shown in **F1**. Impedance spectra are run as a function of a control parameter (**Variable parameter**) such as the applied DC potential or time, or an external parameter such as temperature (this requires an additional module that allows the IM6/e to measure this parameter).

The sequence of the measurements (i.e., how the control parameter is varied) is determined by the **Mode** selection. For **Linear scan**, the spectra are run at defined regular intervals (e.g., at 100 mV intervals of the DC potential or 10 minute intervals). When **Mode = Setpoint list**, each one of the series of values for the control parameter is entered by the user (i.e., the interval of the control parameter between experiments does not have to be constant). When **Mode = Manually**, the value is determined by the user before each experiment (i.e., the series is no longer a batch measurement). The advantage of using the series measurement in the manual mode rather than running each experiment individually is that the data files can be analyzed as a series (e.g., automatic fitting of each member of the series to the same equivalent circuit).

One limitation of the automatic series measurement is that the values

F1

Menu for automatic series measurements.

The screenshot shows the 'IM6 Impedance measurement' software interface. At the top, it says 'IM6 Impedance measurement' and 'Series measurement'. The main title is 'Define Series measurement'. Below this, there are three buttons for 'Mode': 'Linear scan', 'Setpoint list', and 'Manually'. To the right of these is a 'Series file name' input field. On the far right, there is a 'Start series measurement' button. Below the mode selection, there is a 'Variable parameters' section with several controls: 'Time' (with a clock icon), 'Potential' (with a graph icon), 'Current' (with a graph icon), 'Analog control channel', 'Analog input channel', and 'Interface control channel'. Each parameter has a corresponding input field or checkbox.

of the AC parameters (i.e., the AC amplitude, frequency range, and number of measured cycles) are the same for each experiment. If different values of these parameters are required for different experiments, then the Script function should be used (*vide infra*).

Multiple cell measurements

The IM6/e can be used to run impedance spectra of multiple cells in sequence (note that simultaneous measurements are not possible). These measurements require additional hardware; either a multiplexer or external potentiostats with an EPC40 interface card.

The RMUX multiplexer card plugs into the expansion slot of the IM6/e, and provides up to 16 channels (i.e., up to 16 cells can be connected). The applied DC potential can be set independently for each channel. Once the required number of channels has been specified, the IM6/e will cycle sequentially through the channels recording an impedance spectrum for each channel. One limitation of this approach is that when a channel is not active (i.e., the IM6/e is running an impedance spectrum on a different channel), no potential is applied to the cell, and the cell returns to its open circuit potential. Therefore, the RMUX multiplexer cannot be used if a constant potential must be applied to a cell at all times. An alternative approach that provides constant polarization for a channel is to use an external potential connected to the IM6/e using an EPC40 interface card. If a Zahner external potentiostat is used, the applied potential can be controlled from the IM6/e software.

The number of channels available for external potentiostats depends on the number of available expansion slots, as an expansion slot is required for an EPC40 card. Since there is only one expansion slot on the IM6e, only four channels are available, as an EPC40 card can interface four external potentiostats. In

contrast, there are four expansion slots on the IM6, so up to 16 channels are available.

The multiple cell measurement capability of the IM6/e can be combined with a series measurement as a function of time to allow the instrument to cycle continuously through the channels. The only limitation inherent in this capability is that the number of data files that can be stored in one series is 100.

Script

So far in this article, we have discussed setting up batch measurements of impedance spectra. However, impedance spectroscopy is often used in combination with DC electrochemical techniques (e.g., linear polarization), and so Script was developed to allow batch measurements of combinations of impedance spectra, cyclic voltammograms, and linear polarization curves. The Script function can also be used to automate other functions of the IM6/e software (e.g., fitting multiple impedance spectra to the same equivalent circuit).

There are various levels of complexity in Script.

- When the data from an experiment are saved, the experimental parameters are also saved. Therefore, a series of experiments can be set up by loading previously stored experiments, and then storing the data from the new experiment. An example of such a script is shown below (comments are in italics).

SCRIPT1

```
MEAS_OPEN_EIS(2,"examples","test1")
  Load impedance data file
MEAS_EIS
  Record impedance spectrum
MEAS_SAVE_EIS(2,"examples","test2")
  Save impedance spectrum
MEAS_OPEN_IE(2,"examples","ie1")
  Load IE data file
```

```
MEAS_IE
  Record IE curve
MEAS_OPEN_EIS(2,"examples","test1")
  Reload impedance data file
MEAS_EIS
  Record second impedance spectrum
MEAS_SAVE_EIS(2,"examples","test3")
  Save second impedance spectrum
SCRIPT_END
```

The above script loads an EIS experiment ("test1" from the "examples" subdirectory on the hard drive of the IM6/e), runs the experiment, and stores the new data. A linear polarization experiment is then loaded and run. Finally, a second impedance spectrum is run and the data are saved.

- The next level of complexity is to change some of the experimental parameters for an EIS experiment once an EIS experiment has been loaded using the **SENDPARAM** and **SETUPIM6** commands.

SCRIPT1

```
MEAS_OPEN_EIS(2,"examples","test1")
  Load impedance data file
gosubSENDPARAM
  Send control parameters to Script
Ampl=10
  AC amplitude = 10 mV
Pset=1
  DC potential = 1 V
Pot=-1
  Potentiostat on
SETUPIM6
  Return parameters to IM6 program
MEAS_EIS
  Record impedance spectrum
MEAS_SAVE_EIS(2,"examples","test2")
  Save impedance spectrum
SCRIPT_END
```

In this script, a data file is first loaded, then the AC amplitude and the DC potential are changed, and the potentiostat is switched on. The impedance spectrum is then recorded and the data are saved.

- If more detailed control of the experiment is required,

there are routines that can be written by the user that incorporate the basic Script commands and ANDI-Basic functions. One example of such a routine is given below.

```

SCRIPT1
SRC_PATH$="examples"
DES_PATH$="examples"

DIR_EIS$(2,SRC_PATH$,"pser*")
LOOPEND%=Filecount-1

CLRSIM
ANA_OPEN_MOD(2,"examples",
"scr_demo")
forFILE%= 0toLOOPEND%
ANA_OPEN_EIS(2,SRC_PATH$,
Name$(FILE%))
FITEIS(0.1,100000,20)
ANA_SAVE_MOD(2,DES_PATH$,
"@"+ Name$(FILE%))
nextFILE%

CLRSIM

forFILE%=0toLOOPEND%
ANA_OPEN_MOD(2,DES_PATH$,
Name$(FILE%))
nextFILE%

ANA_PLOT_MOD(LOOPEND%+1)

SCRIPT_END

```

- In the first two lines, two directory paths are specified.
- In the next line, EIS measurement data files starting with "pser" in the specified directory are found using the **DIR** command, and the number of data files (**Filecount-1**) is assigned to the **LOOPEND%** variable.
- The impedance data analysis program **SIM** is initialized (**CLRSIM**), and a model (equivalent circuit) data file ("scr_demo") is loaded (**ANA_OPEN_MOD**). This will be used to fit all the measurement data files found by the **DIR** command.

- Since each of the detected EIS files is to be fitted, the fitting is most efficiently done using a loop, which is defined using ANDI-Basic commands. One cycle of this loop entails loading an EIS measurement data file (**ANA_OPEN_EIS**), fitting to the loaded model file (**FITEIS**), and then saving the optimized model file (**ANA_SAVE_MOD**). **Name\$ (FILE%)** defines the file name, based from the information returned to Script by the **DIR** command.
- Before reloading all of the model files, **SIM** is reinitialized (to prevent possible data overflow). The model files are the reloaded (**ANA_OPEN_MOD**)

and plotted on the PC screen (**ANA_PLOT_MOD**).

This article has provided a brief description of the methods for setting up automated measurements on the IM6/e. These require differing levels on the part of the user. The automatic series and multiple cell measurements are simple to set up, and should lie within the capabilities of both experienced and inexperienced users. A simple series of experiments can be readily programmed using simple Script commands, although the more complicated scripts require greater expertise. However, once the scripts have been written, they can easily be loaded and used by all levels of IM6/e operators.