FRA probe

ZAHNER
SCIENTIFIC INSTRUMENTS

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1 Introduction

The Zahner's Zennium potentiostats have limited voltage and current ranges (i.e., Zennium X: Voltage range *±15 V*, Current range: *±4 A*). Even with an extension of a Zahner's electronic load (EL 1000), the maximum working voltage and sink current are *100 V* and *200 A* respectively. In many applications, very high voltages or currents are needed. Here, a high power electronic device may carry out the simple DC measurements but AC measurements are usually not possible. For the AC measurements (i.e., EIS) the (external) high power device can be connected with the Zahner's Zennium potentiostats via a frequency response analyser (FRA) probe. Besides the impedance measuring capabilities of the Zahner's electrochemical potentiostats themselves, the potentiostats offer the feature to acquire the frequency response of an external device (EXD) by its FRA probe. The FRA probe is fully supported by the Thales software. Only the gain factors have to be set for each EXD individually.

1.1 Requirement of the EXD

The EXD must provide an analog control input and analog signal output so that it can be connected to the FRA probe.

When an external device is used with the Zennium potentiostat via a FRA probe then the Zennium potentiostat works as a controller and the specifications of the potentiostat are irrelevant. Only the specifications of the EXD are relevant, hence the user must carefully select an EXD for the measurements. The user must pay attention to the "*Rise Time*" of the EXD. The *Rise Time* entails that how fast the EXD will apply the applied current/voltage.

The EXD specification range should be close to the desired current/voltage range of the system under investigation. An EXD with a very high current/voltage range will usually have a low resolution when it will be used for measurements at smaller current/voltage scales.

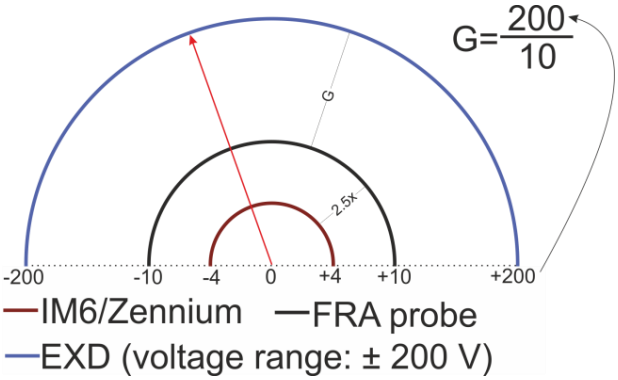
The EXD must be able to operate in the "constant current" (galvanostatic mode) and "constant voltage" (potentiostatic mode) settings. With this, the electrochemical impedance spectroscopy (EIS) measurements can be made in galvanostatic mode (constant current) or potentiostatic mode (constant voltage). For low ohmic objects, the constant current mode is mostly used.

The FRA can be configured to control a source, a sink, or a 4-quadrant electronic device.

The analog control input of the EXD should be in the range of ≤ ± 10 V.

The analog signal output of the EXD should be in the range of ≤ ± 10 V.

1.2 FRA –EXD correlation



The potential ranges of all Zennium series potentiostats for FRA application are ±4 V.

The FRA is an interface which connects the Zennium series potentiostats with the EXD. The FRA works as an amplifier/divider which modifies the output/input of the potentiostats by a factor of 2.5 respectively. Now, this ±10 V of the FRA probe can be correlated with the voltage/current range of the EXD. This way the Zahner's potentiostats can control the EXD. In the image on the right, the correlation factor (G) – for voltage signal – between the FRA and EXD is 20. Here, if one wants to apply a voltage of +100 V via the EXD then in Zennium potentiostats a voltage of +2 V will be applied.

Using the same controls, the Zennium series potentiostats can easily control EXD to produce AC signals, making the EXD capable of EIS measurements.

2 Installation of the FRA interface (hardware)

The FRA probe consists of two interface boxes that are connected to the *Probe E* and *Probe I* connectors of the Zennium series potentiostat. Make sure that nothing is connected to the 4-BNC inputs of the potentiostat at the same time. Connect the FRA using the following steps

1. Connect the *Probe E* cable of the FRA to the *Probe E* input of the Zennium.
2. Connect the *Probe I* cable of the FRA to the *Probe I* input of the Zennium.
3. Connect the *Signal Out* of the FRA to the analog control input of the EXD.
4. Connect the *Current In* of the FRA to the analog current output of the EXD.
5. Connect the *Voltage In* of the FRA to the analog voltage output of the EXD.

Different EXDs have different Analog I/O connectors, hence the user must custom-build a wire to connect the FRA probe to the Analogue I/O of the EXD.

3 Setting up the FRA gain factors (software)

Before setting up the gain factors, the EXD must be turned off or the output from the EXD must be turned. By turning off the output of the EXD, you disconnect the cell from the Zennium + FRA and avoid possible damage to the connected cell.

To set the gain factors of the EXD in the Thales main menu, please proceed as follows:

1. Click on the FRA icon on the Thales software and then click on the "*EIS: Spectra vs. Frequency*".

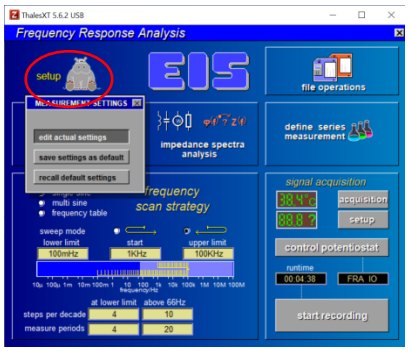
2. The software displays a warning that you must disconnect your cells. Disconnect the connected cell before proceeding further (Turn off your EXD).

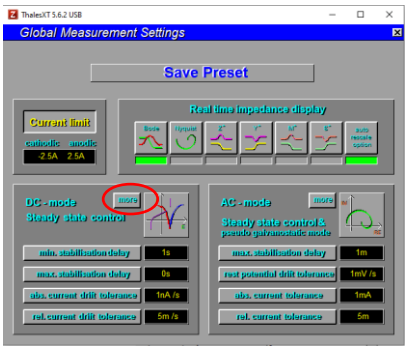
3. Click *"Yes"* to continue.
   NOTE: After clicking "Yes", the Zennium series potentiostat is always ON. This is indicated by the orange "POT" LED above the BNC and Lemosa connectors.

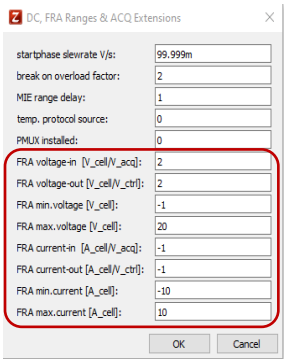
4. In the FRA menu, click on the "Hippo-pictogram" and then click on "*edit actual settings"* to enter the Global Measurement Settings window.

5. In the "*DC-mode*" section, click on **more** to enter the FRA settings.

6. Enter the current and voltage gains (see section 3.1).







7. Enter current and voltage ranges of the EXD.

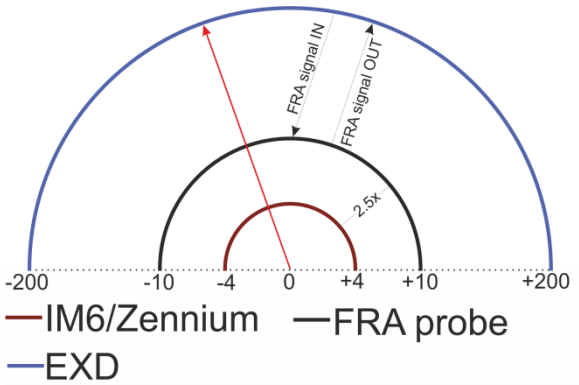
8. Click OK.

9. Turn on the EXD or EXD device output for measurement.

10. Activate the external control from the EXD (if option is available in EXD).

3.1 Determining the current and voltage gain

As explained in section 1.1, the FRA works as an interface between the low voltage/current potentiostat (Zennium series potentiostats) and the high voltage/current source or sink (EXD), allowing Zennium potentiostats to control the EXD provided that the correct gains are set. The current and voltage gains for EXD are the same as that of the correlation factor (G) explained in section 1.1.



To completely use the ±10 V control output of a FRA probe, the voltage and current gains are calculated by using the formulas provided below

$$Voltage\ gain = \frac{Maximum\ voltage\ limit\ of\ the\ EXD}{10}$$

$$Current\ gain = \frac{Maximum\ current\ limit\ of\ the\ EXD}{10}$$

NOTE: If an EXD has a different positive and negative voltage (or current) range (for example: -200 V – +10 V) then the user must calculate the gain using the higher limit (here 200).

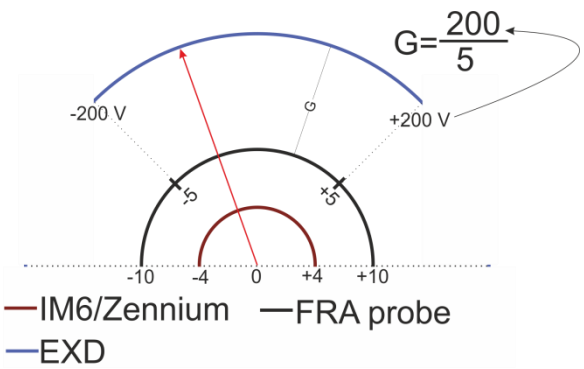
In FRA settings, 2 gains for the signal out (control signal) and signal in (acquisition signal) are provided. These gains are defined below

- FRA voltage in [V_cell/V_acq] → cell voltage / measured voltage signal
- FRA voltage out [V_cell/V_ctrl] → cell voltage / controlled voltage signal
- FRA current in [A_cell/V_acq] → cell current / measured voltage signal
- FRA current out [A_cell/V_ctrl] → cell current / controlled voltage signal

3.2 Limited signal input range of an EXD

Usually, the EXDs have an up to ±10 V range for the signal control and current/voltage input. However, there are some EXDs which offer a low input range. Here, we will calculate the gain factors for an EXD device with analog input/output of up to ±5 V. The output voltage/current range of the EXD has to be correlated with the ±5 V of the FRA. The gain factor will be calculated similarly to the equation shown in the image.

$$Current\ gain = \frac{Maximum\ current\ limit\ of\ the\ EXD}{Allowed\ EXD\ input\backslash output\ range}$$



3.3 FRA settings

In this section, FRA settings are defined for an EXD (a load). In addition, further explanations on different FRA settings are also provided.

EXD specifications: Voltage range: -200 V – 0 V; Current range: 0 A – 100 A.

Entry	Value	Meaning
FRA voltage in [V_cell/V_acq]	20	The cell voltage [V_cell] is the measured voltage [V_acq], acquired by the Zennium, multiplied by the gain factor given here (20), i. e. V_cell = Vacq * gain.
FRA voltage out [V_cell/V_ctrl]	20	The cell voltage [V_cell] is the settled voltage [V_ctrl], set by the Zennium, multiplied by the gain factor given here (20), i.e. V_cell = V_ctrl * gain.
FRA min voltage [V_cell]	-200	Minimum voltage, the EXD can supply.
FRA max voltage [V_cell]	10[(1)]	Maximum voltage, the EXD can supply.
FRA current in [A_cell/V_acq]	-10	The current through the cell [A_cell] is the product from the measured voltage [V_acq] - acquired by the Zennium and the gain factor given here (-10), i.e. A_cell = V_acq * gain
FRA current out [A_cell/V_ctrl]	-10	The settled current through the cell [A_cell] is the product from the control- voltage [V_ctrl] (set by the Zennium) and the gain factor given here (-10), i.e. A_cell = V_ctrl * value
FRA min current [A_cell]	0	Minimum current, the EXD can supply
FRA max current [A_cell]	100	Maximum current, the EXD can supply

[(1)] The Thales software handles the minimum and maximum limits of the EXD very strictly. For safety purposes, the Thales software will stop a FRA experiment immediately, provided one of these limits is violated solely for a single data point of the measurement. Therefore it is useful to select a slightly ‘higher’ value for the extremes to avoid interrupting the experiment.

For instance with a load as an EXD, if you perform an experiment where the current decreases to 0 A then the measured voltage may become slightly positive due to internal offsets (in the mV or µV range), setting a current of 0 A. Therefore the maximum value for V_cell is set to ‘(+)10’ whereas practically, the EXD can supply only negative values.

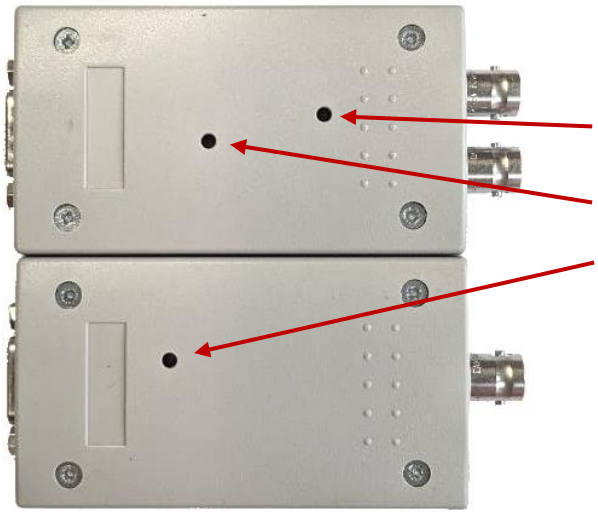
FRA Technical specifications

Control output	±10 V (DC + AC)
Current input	±10 V
Voltage input	±10 V
Frequency response	0 – 1 MHz
Inputs & outputs	differential in/out
Gain factors	by software

The EXDs with higher specifications (high current/voltage range) will usually have longer rise times. Hence the user should not use an unnecessarily big EXD with the Zennium potentiostat and FRA for the EIS measurements.

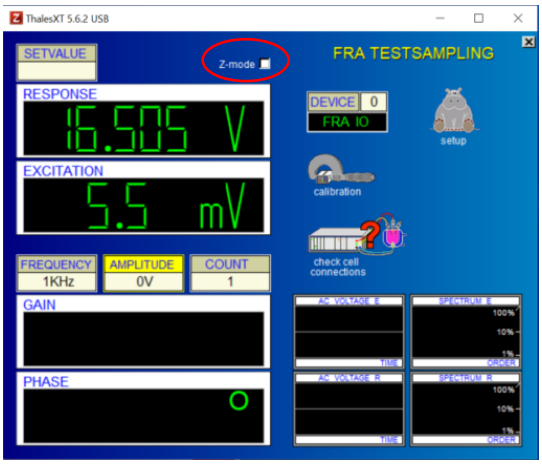
3.4 FRA offset adjustment

The offset voltages of all three signals can be adjusted. For this a slotted screwdriver (size 0.4 x 2.5 mm) is needed. The potentiometer adjustment range is 10-13 turns, depending on the component used. The end stop can be detected by a soft click.



4 FRA test sampling window

Once the FRA probe is properly installed, the user can set up the DC voltage/current and the AC amplitude in the FRA test sampling window. In the image below, the *RESPONSE* and *EXCITATION* show the voltage and current of the cell respectively. If the *Z-mode* is activated, the *RESPONSE* and *EXCITATION* are replaced with the *DC VOLTAGE* and *DC CURRENT.* The units are also modified as that of the EXD. Also, the user can choose between the potentiostatic (constant voltage) and galvanostatic (constant current) mode. This Z-mode-enabled FRA test sampling window is similar to the EIS test sampling window. The user can directly set the desired voltage/current value of the EXD in the Thales software.



Note: Before switching between the CURRENT and the VOLTAGE mode, the user must manually stop the output from the EXD. In addition, before turning on the output, the user must make sure that the correct corresponding CC or CV mode in the EXD is set for CURRENT or VOLTAGE mode measurement.

4.1 Maximum AC voltage/current

The maximum allowed AC voltage/current amplitude for EIS measurement depends upon the main device (Zennium, Zennium pro/X etc).

Zennium Pro/Zennium X:

The Zennium pro and Zennium X can provide a maximum of ±2 V AC voltage (50% of ±4 V). Hence for AC measurement using FRA, the maximum AC signal can be 50% of the EXD voltage/current range. For example, if an EXD has a 100 A current range then a maximum AC amplitude of 50 A is possible.

Zennium:

A Zennium can provide a maximum of ±1 V AC voltage (25 % of ±4 V). Hence for AC measurement using FRA, the maximum AC signal can be 25 % of the EXD voltage/current range. For example, if an EXD has a 100 A current range then a maximum AC amplitude of 25 A is possible.

5 Turning off the Zennium potentiostat

As previously explained in section 3, the Zennium potentiostat is always on during the FRA usage. Hence after the FRA measurements, the potentiostat must be manually turned off by clicking on the EIS icon on Thales main window. A green “POT” LED at the front panel of the Zennium series potentiostat indicates that the potentiostat is turned off.

6 Setting up the FRA in remote

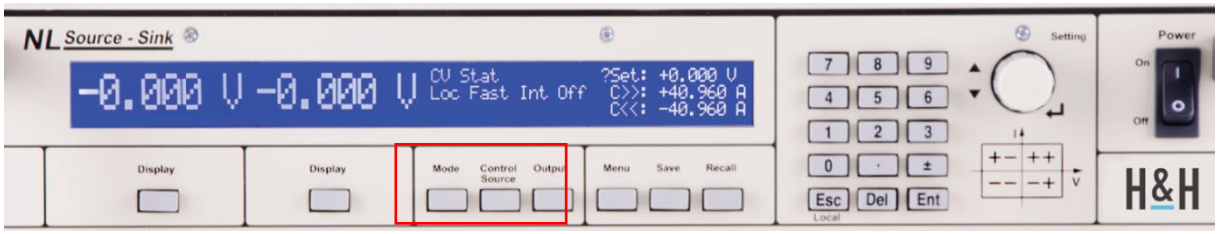
Like Zahner’s potentiostats, the FRA probe can also be controlled remotely using remote control software (i.e., LabVIEW, C++, and Python). The information about FRA remote settings are provided in the Zahner’s Remote-2 manual.

7 Setting up a Höcherl & Hackl source/load

The image below shows a Zennium pro connected with a Höcherl & Hackl (H&H) source/sink (NL1V20C10) via a FRA probe. The H&H device is connected to a test object (battery) using the current-carrying cables and the sense cables.

7.1 The H&H EXD

The image below shows the front panel of the H&H power supply/load. The main buttons of interest are *Mode*, *Control Source*, and *Output*.



Mode:

Switch between different operations modes

- Constant current (CC)
- Constant voltage (CV)

Control Source:

Switch between EXD's

- Internal control
- External control (control via Zennium potentiostat via FRA probe)

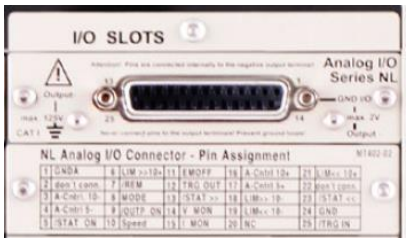
Output:

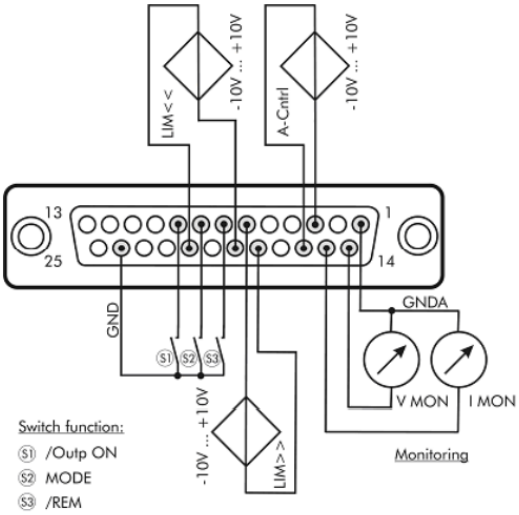
Turn output on or off.

7.2 Custom-build wire for the H&H EXD

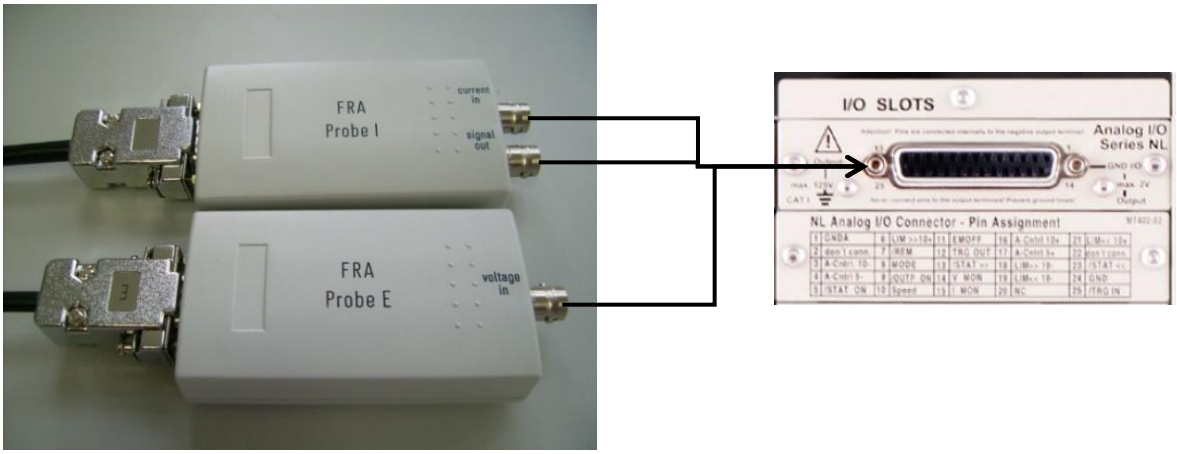
Each company has its Analog I/O ports hence a single kind of cable cannot be manufactured which connects the FRA probe with different EXDs. Therefore the user must build his/her cable which suits the EXD.

The image on right shows the Analog I/O port of the H&H EXD. This analog I/O port can be found at the back panel of the H&H EXD. The information about the port pins is available in the H&H user manual. The schematic of the Analog I/O port from the H&H manual is provided below. This information is necessary for building a wire.





This custom build wire will on one side connect with the *current in*, *signal out,* and *voltage in* BNC connectors of the FRA probe and on the other side with the Analog I/O port of EXD.



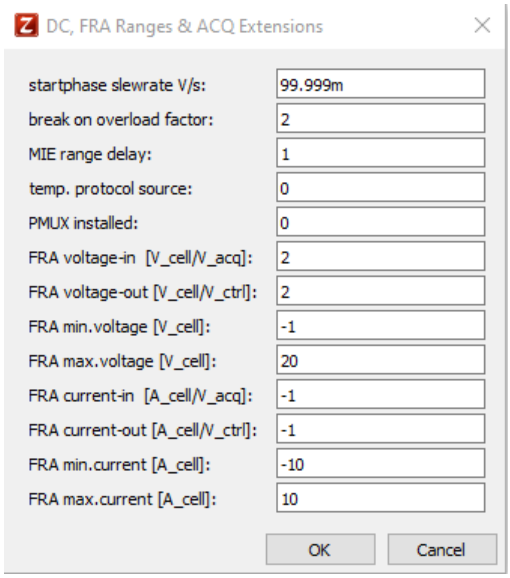
7.3 EIS measurement with the H&H EXD

The H&H EXD (NL1V20C10) has a voltage range of +1/-20 V and a current range of ± 10 A. Since the voltage range is asymmetric, the larger value of 20 is used to calculate the voltage gain. The calculated gains and the current/voltage range are provided in the FRA setting menu.

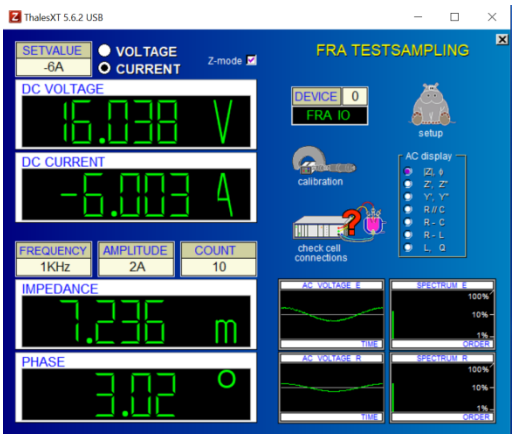
$$Voltage\ gain = \frac{Maximum\ voltage\ limit\ of\ the\ EXD}{10} = \frac{20}{10} = 2$$

$$Current\ gain = \frac{Maximum\ current\ limit\ of\ the\ EXD}{10} = \frac{10}{10} = 1$$

NOTE: Current gain is written with a negative sign.



After appropriate gains are provided and the FRA connections are made then the user can carry out the EIS measurement. In the image (right), a 6 A DC discharging current and 2 A AC amplitude is set in the FRA test sampling window. Now the user can provide the frequency range and can carry out the EIS measurement.



Note: Before switching between the CURRENT and the VOLTAGE mode, the user must manually stop the Output from the EXD. In addition, before turning on the output, the user must make sure that the correct corresponding CC or CV mode in the EXD is set for CURRENT or VOLTAGE mode measurement.