Application Note 27. Using regression scan for electrochemical 'treatment-during-measurement' experiments

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***Abstract*—This application note (AN27) describes the use of a specific analytical tool – the regression scan – to post-process electrochemical, thermochemical, and thermodynamic data collected in treatment-during-measurement (TdM) experiments. This approach allows identifying significant events in dynamics with low signal-to-noise ratio and exploring their temporal and functional properties. TdM experiments using electrochemical impedance spectrometers (EIS) are performed as a series of repeated or single-run attempts in automated or manual modes and complement the available instrumentation for precise EIS measurements.**

firmware version: > 1189.48; client version: > 1.3.40
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I. Introduction

A. What are 'treatment-during-measurement' and 'measurement-after-treatment' experiments?

'Treatment-during-measurement' (TdM) is a specific approach for measuring electrochemical or thermochemical signals with low or ultra low signal-to-noise-ratio. Typically, such measurements are required for monitoring electrochemical reactions with nonlinear kinetics (e.g. dissolving low concentration of gasses from the atmosphere, reversible reactions), measuring the reactivity of aqueous solutions, non-chemical water treatment or similar applications, which are further referred as experimental factors. Fluidic samples in TdM experiments are simultaneously treated by experimental factors and measured with *electrochemical impedance spectroscopy*. The measurements run continuously and start hours/days before the treatment. Advantages of TdM approach are: 1) long-term background measurements and accumulation of statistics; 2) better exploration of experimental factors and their interdependencies (e.g. the temperature, electromagnetic signals, mechanical impacts); 3) minimal systematic and random errors/distortions created by handling water samples (CO_2 dissolving, heating of fluids, small temperature changes and similar).

'Measurement-after-treatment' (MaT) approach is another way of performing EIS measurements, where samples are first treated by experimental factors, then handled (e.g. collected, transported) and finally measured. MaT approach is better suitable for cases where the influence of treatment on samples is much stronger than the distortions caused by handling. For instance, chemical changes are typically measured using the MaT approach.

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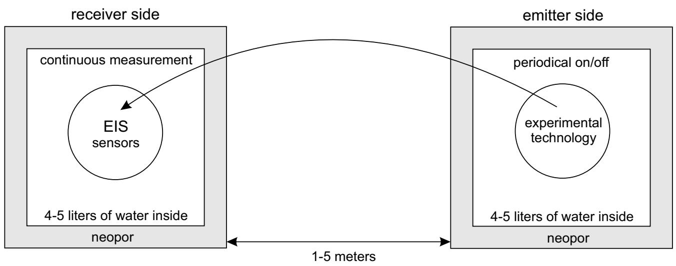
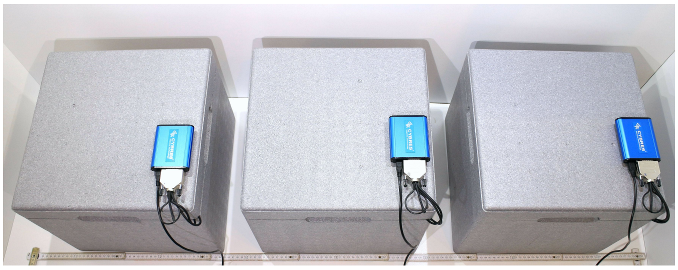
The significant event is a perturbation of EIS dynamics, whose origin can be linked to the experimental factors. Identification of *the significant event* requires a strong temporal correlation between application of such factors and response of the EIS dynamics.

B. What are 'repetitive attempts in automated way'?

In order to collect statistics and analyze the influence of environmental fluctuations, the experiments should be repeated at least 30 times. It is important that the conditions of these experiments are similar and that the involvement of laboratory personnel in the conduct and analysis of these experiments is avoided wherever possible.



(a)



(b)

Figure 1. **(a)** Setup with 'receiver side' and 'emitter side' in thick-wall neopor containers with 4-5 liters of water inside for compensating thermal changes; **(b)** Example of the setup with 3 EIS devices, one of them takes the role of 'emitter side' based on the DA module [1].

One possible way to organize such automated attempts is shown in Fig. 1(a). It consists of the 'receiver side' and 'emitter side' (in telecommunication terminology) in thick-wall neopor containers with 4-5 liters of water inside for compensating thermal changes. The EIS sensors on the receiver side are continuously recording data in TdM way. The experimental technology on the emitter side is automatically turned on and off with specified time intervals. For switching on/off, the relays with programmable timers or DA module of EIS devices can be used, see Sec. V-A. Fig. 1(b) shows the setup with 3 EIS devices, one of them has the role of 'emitter side' based on the DA module (see the user manual [1]). The distance between containers is typical for local experiments (1-5 meters taking into account EM interferences).

On/off timing depends on the experimental technology, thereby two effects should be taken into account. Firstly, the single event on the emitter side can generate two or three responses on the receiver side, see Fig. 7. The interval before the next attempt should provide enough time for manifestation of such effects. Secondly, environmental fluctuations related to daily or monthly cycles should be also considered. They can introduce essential perturbations into dynamics. Long intervals between attempts can include such fluctuations, which are sometimes not distinguishable from experimental influences and require special statistical approaches for their analysis, see the AN26 [2].

II. Thermochemical and thermodynamic analysis

A. *Relationship between temperature and electrochemical reactions in EIS measurements*

The electrical conductivity of water is directly proportional (and the impedance is inverse proportional) to temperature

$$EC_t = EC_{25}[1 + a(t_{25})], \quad (1)$$

where a varies between 0.0191 and 0.025, EC_t is the conductivity at t, EC_{25} is the conductivity at 25C [3]. Fig. 2 shows the experimental data on the relationship between electrical conductivity and temperature in EIS measurements for 20 hours. Performing the linear regression in Fig. 2(b) it can be seen that the overall decreasing temperature dynamics contains a small periodic variation in the temperature of the day cycle. This accordingly affects the electrical conductivity (impedance) and generally follows (1) (with an inverse relationship for impedance) with a coefficient of the order of $10^{-2} - 10^{-3}$). In other words, small temperature changes are sufficient to explain the nonlinearity of the EIS trend in accordance with (1).

Performing experiments, several anomalies can be detected, which are not explained by the dependency (1):

- a delay between changes of temperature and conductivity (impedance) when using thermally insulated containers (caused by a slow propagation of temperature inside the container and a large thermal inertia inside the container). In TdM experiments a simultaneous change of temperature and impedance in such setups can be observed, see Fig. 3.
- a simultaneous change of temperature and impedance is in many cases directly proportional to each other, whereas the delayed reaction is strongly inverse proportional and thus follows (1), see Fig. 3(b).
- dynamics of impedance (conductivity) does not follow the temperature dynamics. Fig. 3(c) shows the experiment, the temperature dynamics exactly indicates begin/end of the experiment, which however is not appeared in the impedance data.

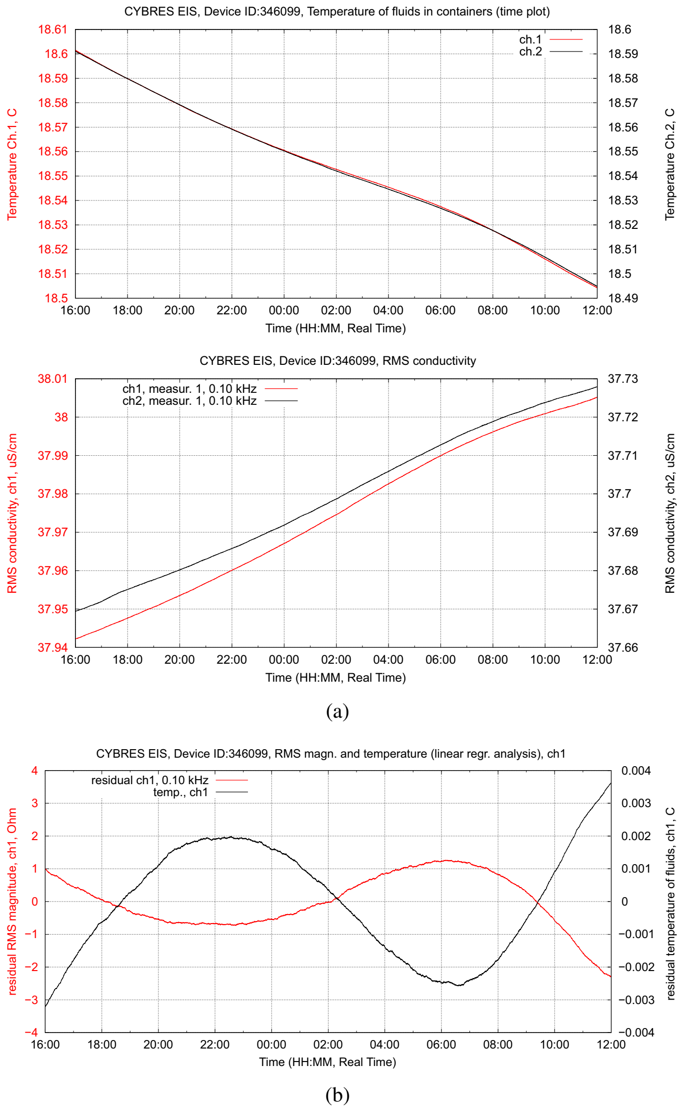


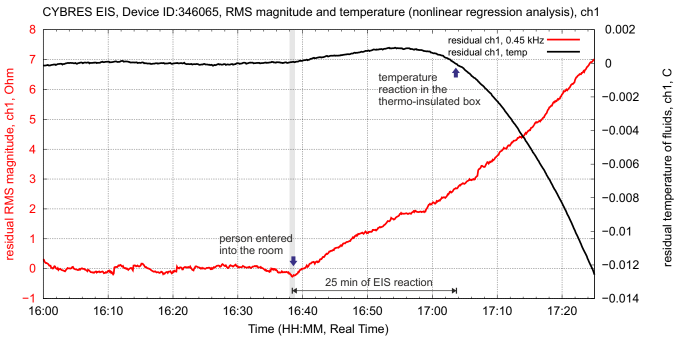
Figure 2. Experimental relationship between electrical conductivity and temperature in EIS measurements for 20 hours. **(a)** Temperature and conductivity dynamics; **(c)** Linear regression of impedance and temperature in channel 1.

- the dependency (1) does not explain the sudden changes (spikes) in temperature, which after 1-1.5 minutes begins to affect the impedance, see Fig. 4.
- changes (1)-(4) can appear only in one channel of the EIS system, whereas we expect that temperature will affect both channels in a similar way.

There are several hypotheses for explaining such a behavior of EIS and temperature data. First of all, due to ongoing electrochemical reactions that generate additional ions – the electrical conductivity of water is constantly increasing during the measurements – the experiments can affect exo-/endothermic electrochemical reactions, for example, with the participation of dissolved oxygen and carbon dioxide (see [4], [5], [6] for thermal effects related to water isomers). There are several candidates for the role of such reactions. First, the main suppliers of ions are water dissociation reactions

$$H_2O \rightleftharpoons H^+ + OH^- \quad (2)$$

(a)



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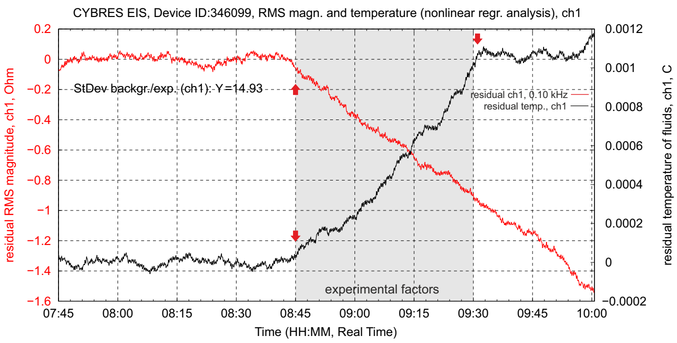


(c)

Figure 3. **(a)** EIS system in a container with a wall thickness of 5 cm and 5 kg of water inside (to create a temperature inertia). Due to the thermal inertia inside the container, the effect of temperature is delayed for several minutes and the EIS reaction to the non-temperature factor is visible; **(b)** Fast response of the EIS channel to the operator entering the room and almost 25-minute delay in the temperature response to changes in the external temperature; **(c)** Application of experimental factors on the channel 1. Shown is the EIS dynamics and temperature of the experimental channel. The session started at 8.45 and ended at 9.30, the change of temperature trend almost exactly correlates with the time of experiment.

and self-ionization

$$2H_2O \rightleftharpoons H_3O^+ + OH^- \quad (3)$$

that are endothermic[1], and the dissolution process of CO_2 from air to water

$$CO_2 + H_2O \rightleftharpoons H_2CO_3 \rightarrow H_2CO_3 \rightleftharpoons H^+ + HCO_3^- \quad (4)$$

is, on the contrary, exothermic. A change in the ionic composition observed in the form of a change in the conductivity trend also means a change in the amount of heat absorbed or released by these reactions.

The second candidate is electrochemical reactions with dissolved oxygen [8] along the four-electron path

$$O_2 + H_2O + 4e^- \rightarrow 4OH^-; E^0 = 0.401V \quad (5)$$

or along the two-electron path

$$O_2 + H_2O + 2e^- \rightarrow HO_2^- + OH^-; E^0 = 1.229V \quad (6)$$

and further

$$HO_2^- + H_2O + 2e^- \rightarrow 3OH^-; E^0 = 0.867V \quad (7)$$

which in turn are exothermic. Oxygen dissolved in water is present in the form of hydrated O_2 molecules obtained from the atmosphere or due to electrolysis (electrolysis at a high excitation frequency practically does not occur, but its intensity will increase with decreasing frequency)

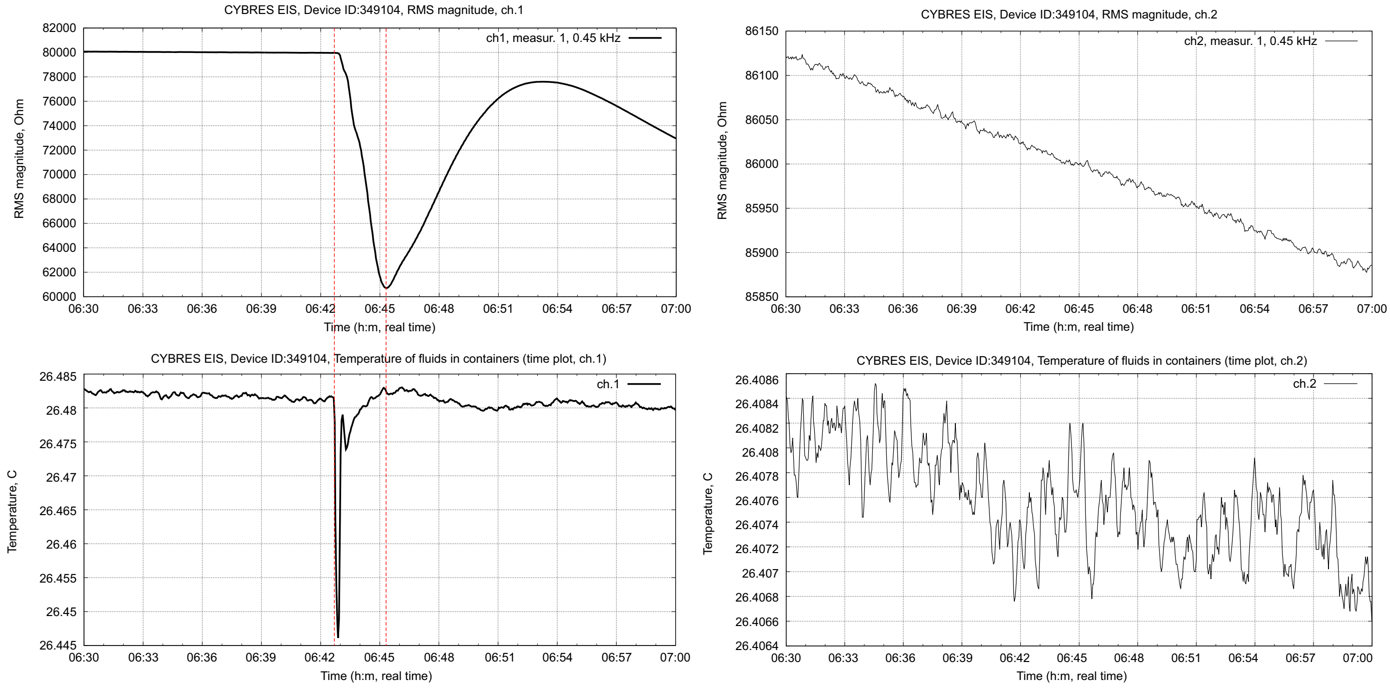
$$2H_2O = 2H_2 \uparrow + O_2 \uparrow, \quad (8)$$

whose reaction is endothermic. Here, a change in the ionic composition will also be recorded by a change in both conductivity and temperature.

The third candidate is the hydrated metal ions of the electrodes Cu^{2+}, Fe^{3+}, etc. (in the form of complex ions $[Cu(H_2O)_6]^{2+}$, $[Fe(H_2O)_6]^{3+}$, for example, with copper electrodes $Cu - 2e = Cu^{2+}$) dissolved in water. Various impurities that participate in further exo-/endothermic electrochemical reactions also fall into the same category.

Finally, it is worth mentioning the mechanisms of ion diffusion and proton transfer in the so-called hydrogen-bonded networks [9], [10], the behavior of which has not yet been sufficiently studied (which are characterized by abnormal changes in electrical conductivity). Similar effects are quite common in measurements, an example is shown in Fig. 4. Here, in channel 1, there is a sudden temperature change, which after 1-1.5 minutes begins to affect the impedance. The maximum impedance change is reached after 3 minutes, when the temperature has already stabilized at the previous level. Furthermore, after 10 minutes, a change in the EIS trend is observed, while the temperature trend is stable.

[1] Although the reactions of water dissociation and self-ionization are quite rare under normal conditions, the imposition of an external electric field during measurements can cause fluctuations, similar to those described in [7], which enhances the role of these reactions in the formation of additional ions.



(a) (b)

Figure 4. An example of an abrupt change in the EIS and temperature dynamics, as an illustration of the hypothesis of the mechanisms of ion diffusion and proton transfer in networks with hydrogen bonds and abnormal changes in electrical conductivity: **(a)** channel 1; **(b)** channel 2. In channel 1 there is a sudden change in temperature, which after 1-1.5 minutes begins to affect the impedance. The maximum impedance change is reached after 3 minutes, when the temperature has already stabilized at the previous level. Further, after 10 minutes, a change in the EIS trend is observed, while the temperature trend is stable. Channel 2 does not show such changes.

B. Thermochemical and thermodynamic analysis in EIS Client

Parameters and settings for thermochemical and thermodynamic analysis are marked by red colours in different menus of the EIS Client, see Fig. 5. They include impedance-temperature dependencies, statistical analysis and several other analytic instruments.

III. Methodology and Settings

A. When and how to use the regression scan?

The regression scan is useful for post-processing all electrochemical, thermochemical and thermodynamic data, obtained in TdM experiments.

I. Typical example of using the regression scan for enhancing regression analysis is shown in Fig. 6. It is applied as post-processing of recorded data from Fig. 6(a). At each step of the regression scan, the system performs a linear/nonlinear regression and computes the Ψ value – the relationship between standard deviations of mean in experimental σ_E and background σ_B areas, see [2]:

$$\Psi = k\frac{\sigma_E}{\sigma_B}, \tag{9}$$

where k defines the sign of Ψ based on the EIS dynamics in experimental region. These Ψ values are plotted as a histogram over time, see Fig. 6(b). The maximal points correspond to changes of trend, i.e. to the external event that changes the EIS/thermochemical/thermodynamic behaviour. Low-amplitude values represent the noise that can be removed by filtering, see Fig. 6(c). The example in Fig. 6 demonstrates analysis of automated periodical activation of 'experimental technology', discussed in Sec. I-B.

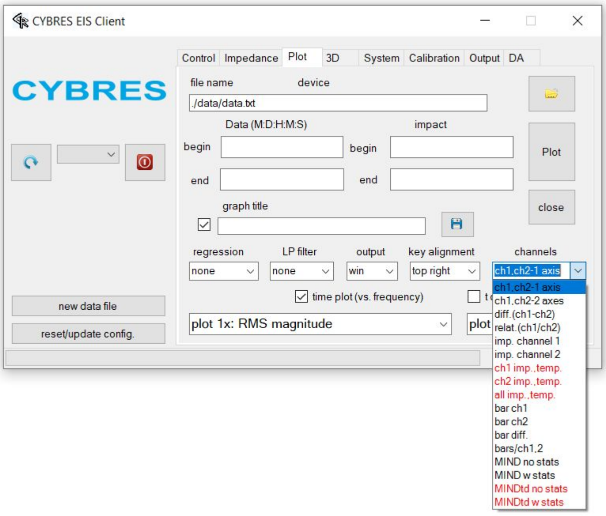
II. Regression scan can be used for exploring the nature of significant events. For instance, the single influence produced by experimental technology can generate double or even triple response of EIS sensors, see Fig. 7. The analysis allows determining whether this is an artefact of regression or true response of EIS sensors.

III. Regression scan can be used for exploring the long-term dependency between different values of EIS analysis: variation of impedance/temprature between channels, dependency of impedance from temperature, or temperature of fluids from external temperature, see Fig. 8.

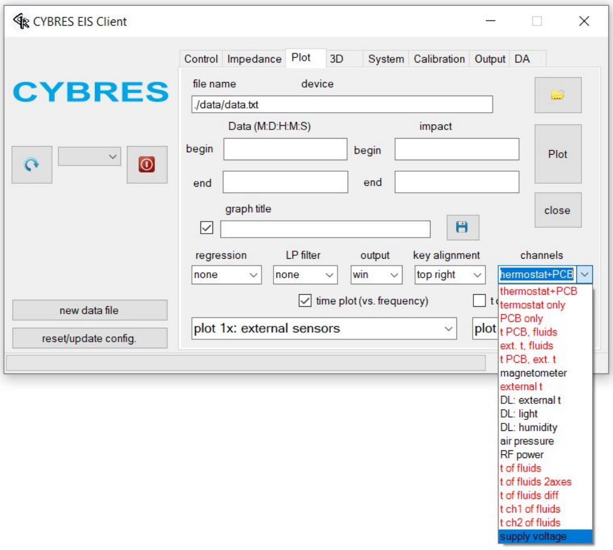
IV. In several cases not only separate values (like impedance or temperature) but also their combination (e.g. averaged mean) provide a significant response. In these cases the EIS device can be considered as multi-channel and multi-parametric device. Since all data is analysed by the same nonlinear regression (or regression scan) algorithm, one output value can be synthesized from 2, 4 or 6 sensors of EIS device by using a combined value Ψ_i^{comb}

$$\Psi_i^{comb} = 1/N \sum_N \Psi_{i,j} \tag{10}$$

where the index j passes through all available sensors at the step i. Fig. 8 shows the multiparametric analysis (MPA) based on data from six sensors (2x electrochemical, 2x thermochemical, 2x external temperature). The regression scan allow investigat-

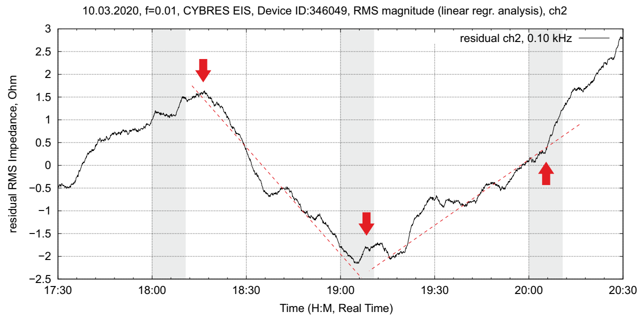


(a)



(b)

Figure 5. Parameters and settings for thermochemical and thermodynamic analysis are marked by red colours.



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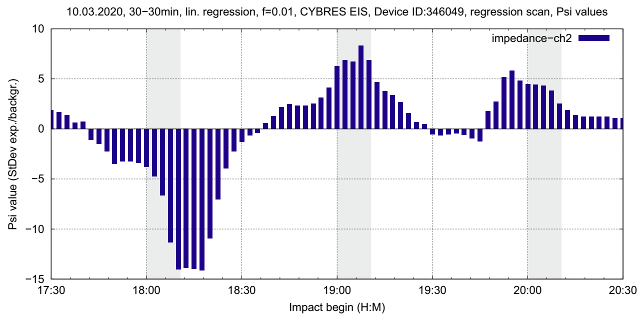

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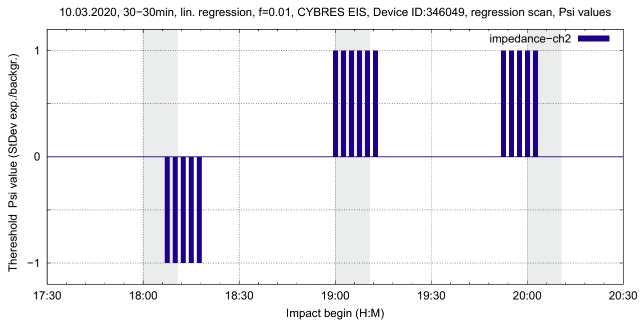


(c)

Figure 6. Example of the regression scan, experiments with the automated periodical activation of experimental factors, the gray bar shows the activation time (10 minutes of every hour). **(a)** Impedance of ch. 2 with linear regression; **(b)** Regression scan with 30-30 min of background and experiment areas; **(c)** Applying a burst filter normalized to ±1 for removing the low-amplitude noise.

ing which combinations of EIS parameters are correlated to the significant events and to analyse their properties.

The regression scan uses the instrument 'sequencer' and is performed in three following steps.

1) **Measurement step:** a) Run the measurements in continuous mode for a long time (days or weeks); b) Perform experiments without changing any measurement settings; c) After the experiment is finished, keep the EIS system measuring further.
2) **Regression scan step:** a) Copy *.dat file with measurement data, the EIS system is operating further in continuous mode; b) Start a second instance of the EIS Client and run the regression scan with the selected parameters. Note, the regression scan can take a few minutes/hours for processing. It produces the 'regressionScan_hhmm.dat' files as output.
3) **Plotting step:** Open the produced file 'regressionScan_hhmm.dat' with the plot option 'regression scan'. Select different parameters for this plot and for corresponding filters.

B. *Options for the regression scan*

The regression scan is available for 'plot 1x: RMS magnitude' (all options excluding 'bar' and 'MIND') and for 'plot 1x: external sensors' (all options excluding magnetometer/accelerometer, light, pressure, power and humidity). The following description is related to the numbers in the Fig. 9.

1. Select the regression time, see the numbers 1, 2 and 3. Select the earliest time for the impact time that you would like to analyse (the number 2). Set up the background time (the number 1) and the experiment time (the number 3). The timing

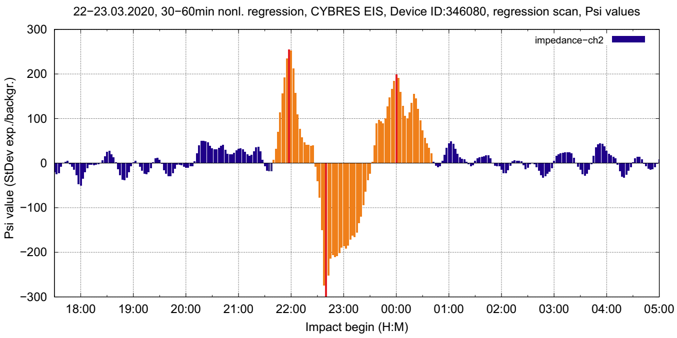


Figure 7. Example of the regression scan: three responses of EIS sensors on the single event on emitter side.

background-experiment is denoted as *the regression timing*, the example in Fig. 9 has 30-30 min. timing, see further discussion in Sec IV-B.

2. Select the regression type (linear or nonlinear, the number 4), see Sec. for further discussion.

3. Select the low-pass filter type (linear or nonlinear, the number 5), see Sec. for further discussion.

4. Select the type of output values for regression scan, the number 6 and 7. For instance, 'RMS magnitude ch1, ch2' will use the impedance channels 1, 2 for regression scan, the 'external sensors, external t' – the external temperature for regression scan.

5. Select the time step for increasing background and experiment, the number 8 and 9. Each regression scan step will be increased by these values (in sec). For regression scan use the same values for background and experiment.

6. Set up the number of steps for regression scan, the number 10. The system will perform N step.

7. Set up the delay between steps, the number 11. The regression scan requires a high computational power. This option introduces a delay between steps of regression scan. The value depends on computational power of PC and is typically between 5 and 10 sec.

8. Enable write log file, the number 14. This checkbox enables observing the results of regression scan in the form of histograms and writing output data into the file. If the checkbox is disabled, only regression curves are shown, the data will be not written into the file.

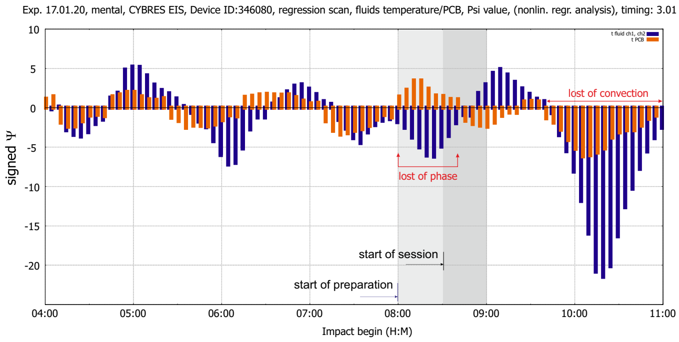
9. Start/stop regression, the numbers 12 and 13. The regression scan can be started or stopped by these buttons. The progress can be monitored in the field 'sequencing time'.

C. *Plot of the regression scan data*

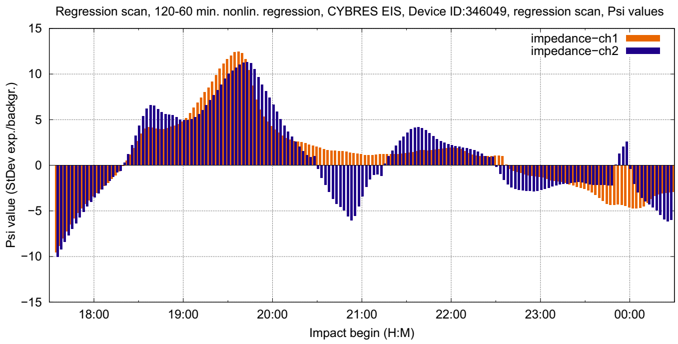
The regression scan can process 6 values: impedance of ch1/ch2, fluid temperature of ch1/ch2 and temperature of PCB/external temperature. The selection of these values for regression scan is defined on the step 4 in Sec. III-B. Note that only that output values, which have been precessed by the regression scan, can be also plotted.

For plot, open the data file 'regressionScan_hhmm.dat', where 'hhmm' denotes the GMT time, when the regression scan started. The following numbers are related to Fig. 10(a).

1) Select the plot 'regression scan'.



(a)



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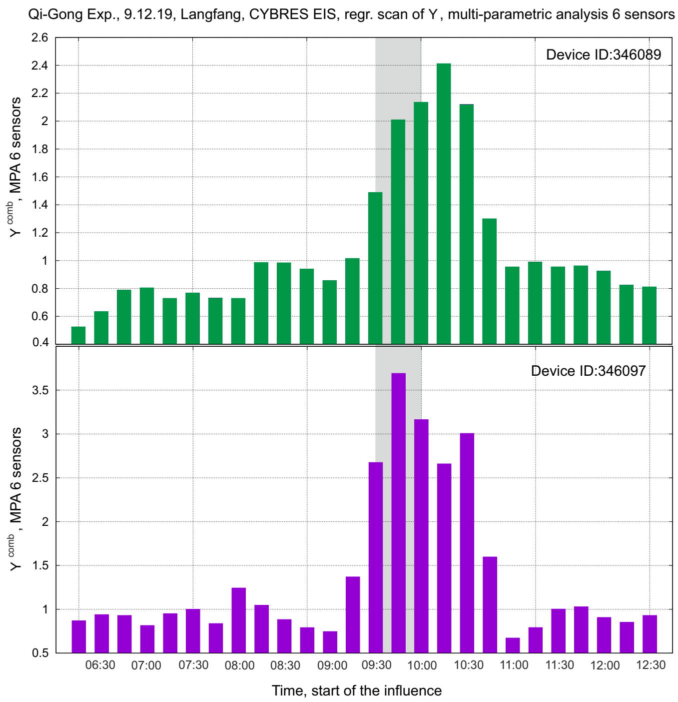
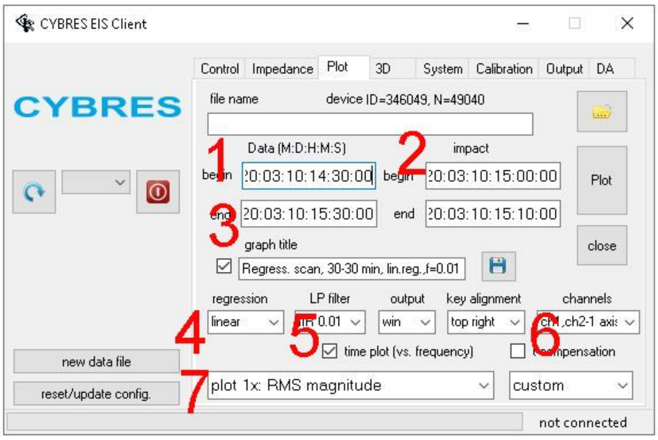
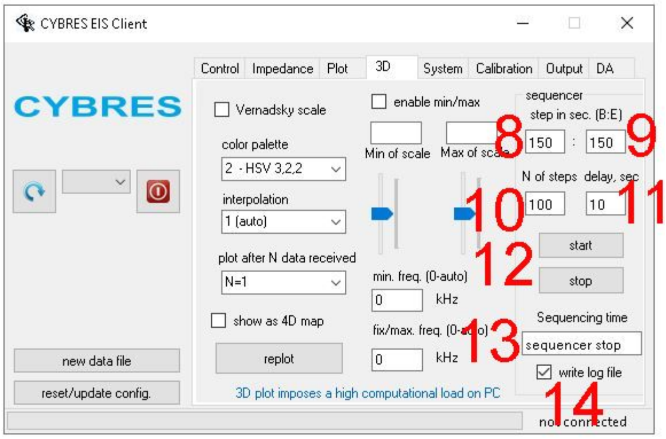


(c)

Figure 8. Examples of the regression scan for exploring dependency between different parameters of EIS analysis. **(a)** Dependency between temperature of fluids and external temperature; **(b)** Dependency between impedance channels; **(c)** Examples of the multiparameter analysis (MPA) within the regression scan, based on data from six sensors from each EIS device, Ψ_i^{comb} is calculated as the average of all Ψ_i from each sensor according to (10). The best signal-to-noise ratio is achieved by using data from all six sensors.



(a)



(b)

Figure 9. Setting the regressions scan, see description in text.

2) Specify start and end time of the dynamics of interest, the numbers 1 and 2.

3) Specify averaging filter.

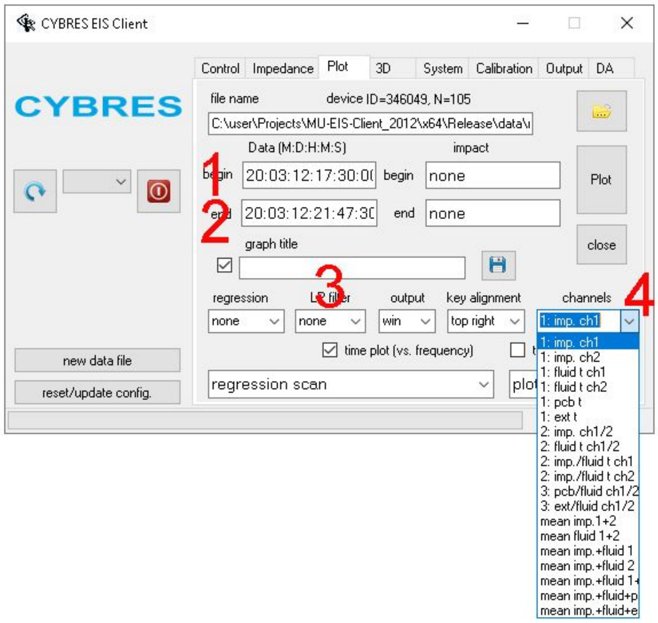
4) Select the output values for plotting (either separate value or their combinations). Note that these parameters are still in development and improvements and thus can differ in versions of the Client program.

D. Applying analytic filters

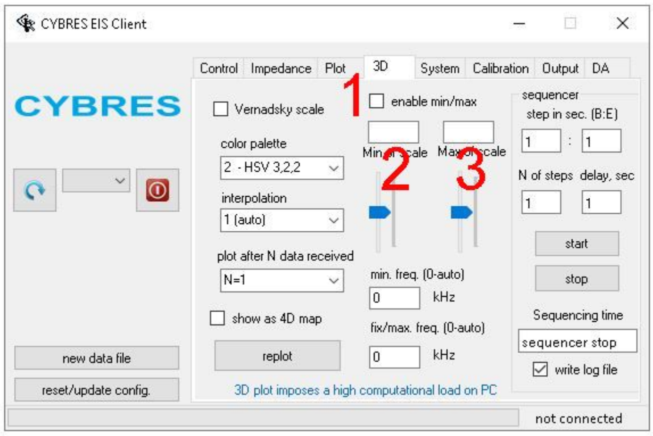
The obtained histograms can be processed by different filters with the goal of further analysis (these filters differ from low-pass filters used in the stages of regression scan and plot). The analytic filters are enabled by the checkbox 'enable min/max', see Fig. 13. The fields 'min' and 'max', the numbers 2 and 3, allow specifying parameters. Note that filters are still in development and improvements, see specifications in the script file '..\scripts\regressionScan.dat'.

```
#--------------------------------
#--periodical filter definition--
#--------------------------------

#timeSelect="%H"; # use hours
```



(a)



(b)

Figure 10. **(a)** Setting parameters of the plot for regressions scan, see description in text; **(b)** Setting filter parameters for further analysis, see description in text.

```
timeSelect="%M";  # use minutes
periodTimeMin=0;
periodTimeMax=70;

#--------------------------------
#--threshold filter definition--
#--------------------------------
#selectorMaxValues=1; # use Max values
selectorMaxValues=0;  # use +1;-1
```

1. Threshold filter. This is the basic filter that is applied to all regression scan plots. This filter imposes a 'min'/'max' threshold for removing low-amplitude noise. Values that passed the filter are represented by ±1 values at *selectorMaxValues=0* or by maximal values at *selectorMaxValues=1*.

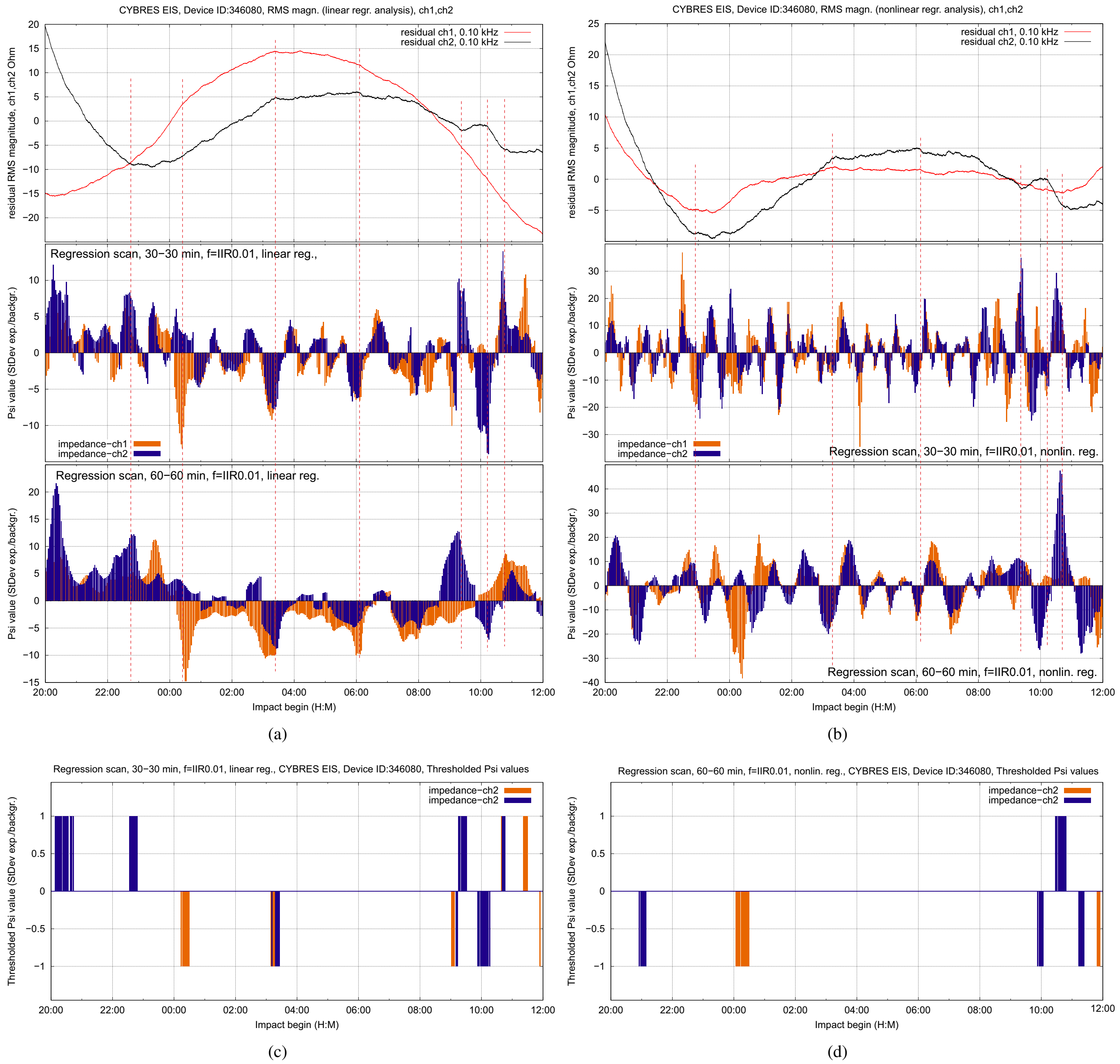


Figure 11. Application of linear **(a)** and nonlinear **(b)** regression for the regression scan. Cases with 30-30min and 60-60min timing are shown. **(c)** Application of the threshold filter to linear 30-30 regression scan. **(d)** Application of the threshold filter to nonlinear 60-60 regression scan, see discussion in Sec. IV-A.

2. Periodical filter. This filter applies the mask for temporal selection of periodical time windows where the values are plotted (for instance 10 minutes of every hour, time slot 8:00-10:00 of every day and so on). It can be combined with any other filter. For disabling the filter set the parameters outside the working area, e.g. *periodTimeMin=0* and *periodTimeMax=70* for minutes.

IV. Discussions

A. Discussion: linear vs. nonlinear regression

The regression analysis is an important tool for EIS systems and allows, among others, expressing weak dynamical changes in the well recognizable numerical form of Ψ values. Regression analysis uses linear and nonlinear regressions, which follow different goals and possess different advantages and disadvantages.

The nonlinear regression enables approximation of background region in 'more flat' form and thus better suitable for measurements with long background area. The disadvantage is a nonlinear sensitivity to different forms of dynamics: dynamics which can be well approximated by 5th order polynomials generate a disproportionately better Ψ scoring than other forms of dynamics.

Linear regression provides more linear results in relation to

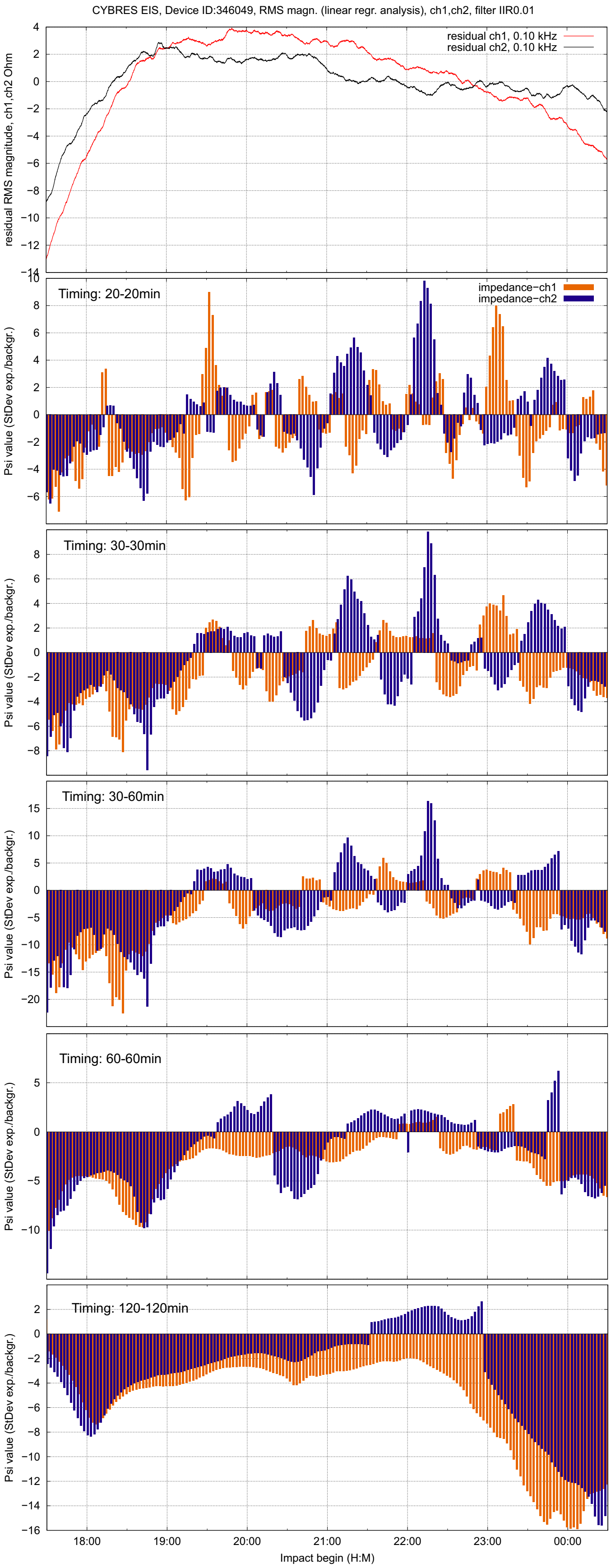


Figure 12. Examples of regression scan with different timing parameters.

different forms of EIS dynamics, however it is not suitable for background areas with strong nonlinear curves, which typically appear in long background measurements.

Fig.11 provides one example of EIS dynamics analysed by linear/nonlinear regression and regression scans with 30-30min and 60-60min timing. Finally, the results are processed by the threshold filter. We can see that the linear regression with short timing (30-30min) provides better results that reflect all significant events of EIS dynamics. Nonlinear regression missed several such events and produced a few imaginary responses caused by nonlinear nature of data.

B. *Discussion: regression timing*

The regression timing, see the step 1 in Sec. III-B, defines the background and experimental areas for regression. For example, '30-30min' means 30 min for background and 30 min for experimental areas. The Ψ values and the form of regression scan data are influenced by selection of timing parameters.

Fig. 12 shows examples of the regression scan with different timing parameters, from 20-20min, up to 120-120min. The most important is the first value that specifies the background region. Large values lead to averaging the dynamics and removing characteristic peaks (changes of trend). The best results are obtained with short timing intervals such as 30 or 40 min for the background region. The second timing value of experimental area defines the amplitude of the signal. It does not essentially change the form of regression scan curves and can be set equal to the background value.

C. *Discussion: influence of the low-pass filter*

LP filters during the regression scan are necessary because they remove high-frequency noise and average the trend. However, the LP filters can essentially change the maximal value and shift it along X axes (time). Fig. 13 demonstrates application of IIR (infinite impulse response) filter with the coefficient from 1 up to 0.005. The low coefficient values (0.005, 0.01) produce high Ψ score, however shift temporal dynamics. The high coefficient values (1, 0.5) produce noisy low-score Ψ output. For the regression analysis we recommend first to test whether the selected LP filter is suitable for a proper representation of data and them to use this value at all steps of analysis. Generally, the middle-range coefficient values for IIR filter (0.05, 0.01) appeared as best-choice-values for a wide range of dynamics.

V. THE 'EMITTER SIDE'

A. *Setting up the 'emitter side' with EIS devices*

EIS devices can support or even perform the role of 'emitter side'. This can be done by DA module, see [1]. Structure of detector-actuator (DA) module is shown in Fig. 14. It executes in-hardware and in-software real time signal processing by means of embedded numerical processors and real-time detectors. Examples of numerical processors are the mean, standard deviation or z score calculations. Examples of detectors are the time interval detector, the peak detector, the cyclical change

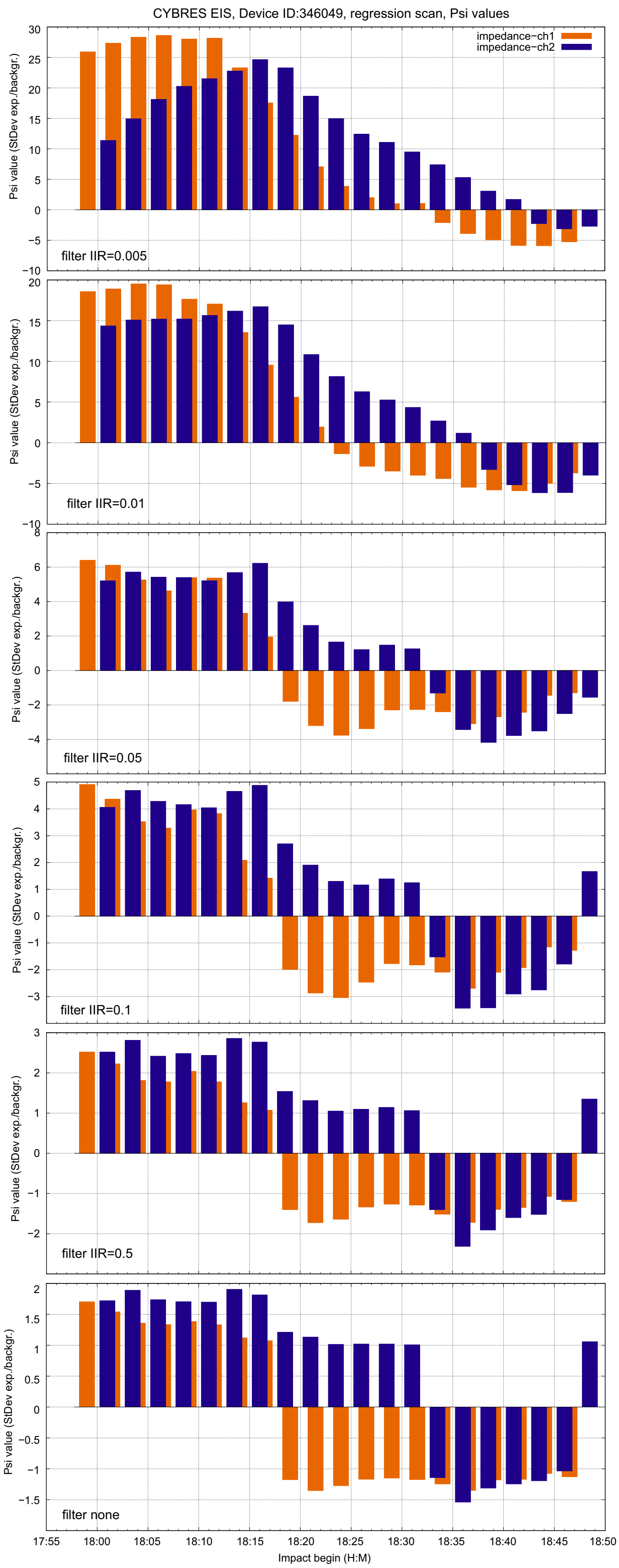


Figure 13. Example of regression scan with different values of low-pass IIR filter.

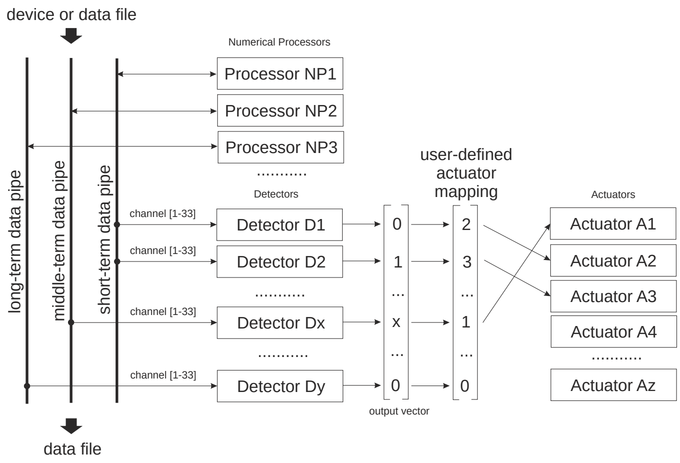


Figure 14. Schematic representation of the detector-actuator (DA) module.

detector, the noise levels detector, the gradient change detector, time detector and others.

The DA module detects changes in input data stream, in real time, and executes corresponding actuators. Programming is performed by means of DA scripts.

DA module can be used on the emitter side in two ways. First, it can specify time intervals for periodical on/off of the experimental technology. The following script executes 10min ON timing during 60 minutes (i.e. 10 min ON, 50 min OFF). As actuator, the internal blue LED (D101=41; D102=42;) or external relay EG-PM2 (D101=191; D102=912;) for any 110/220V power supply can be used, see Fig. 14. For instance, this scrip generates the timing of the experiment shown in Fig. 6.

```
-- define timer for 10-50min ON/OFF
P101=01:01:01:00:00:00 -2 -1;
P102=01:01:01:00:10:00 -2 -1;

-- define activity for timers
D101=41; use 191 for relay
D102=42; use 192 for relay

--actuator: embedded BLUE Light
A41=wk001*; turn blue LED on
A42=wk000*; turn blue LED off

--actuator: relay EG-PMS2 USB
A191=pm -on -Device1 -Socket1;
A192=pm -off -Device1 -Socket1;
```

The second way is based on EIS analysis of water samples, installed on the emitter side. It allows organizing complex feedback loops, example of this approach is shown in [11].

VI. Conclusion

The AN27 describes usage of regression scan for TdM experiments, where the EIS technology can be used for both 'receiver side' and 'emitter side'. This approach provides most accurate results for a single experiment as well as for a series of repetitive experiments performed in automated way. We recommend to use a linear regression with a short timing (e.g. 30-30min) and IIR0.01-IIR0.05 low-pass filter for the initial parameter set, which can be then adapted for other user cases. Obtained regression scan data can be further processed by analytical filters, the simplest one is the threshold-based filter with ± 1 output.

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