Analysis of the 12th ECG-COMON Round-Robin on dummy cells

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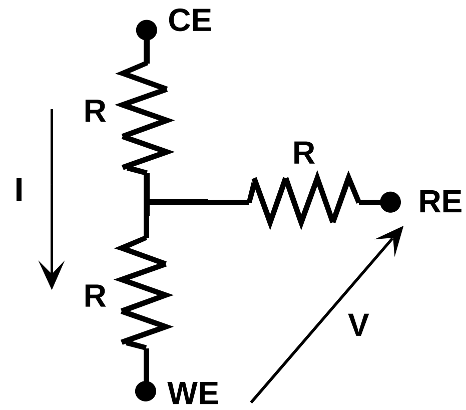
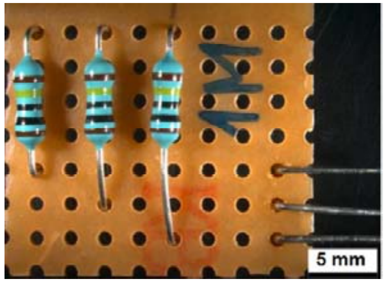
Round-Robin on Electrochemical Noise #12



- 3 identical resistors in star arrangement (ZRA mode)
- R = 1 MΩ and R = 100 MΩ
- thermal noise : low but known (Ψ_V = 6kTR, Ψ_I = 2kT/R)

- 20 participants: missing PalmSens
- 24 data sets received (4 for CANMET, 2 for VTT, CNRS)



→ total: 149 files, 298 time records, 298 PSDs

Analysis performed in frequency domain (FFT as in last RR test):
- only way to validate EN measurements
- measurements have to be performed at different sampling frequencies (2 to 4 for all participants)

Aim: compare the new results with those measured in 2014 and 2016

Hardware and software used in RR #12

Organisation	Contact person	Device and software
AREVA (Germany)	Matthias Herbst	Gamry Ref600, ESA410
Bio-logic (France)	Nicolas Murer	Bio-Logic SP-300, EC-Lab Express 5.56
Canmet Materials (Canada)	Jennifer Collier	Solartron 1287A , CorrWare (2 instruments)
Canmet Materials (Canada)	Jean-Philippe Gravel	Solartron 1287A, CorrWare + PAR Parstat, Ver.Stud. 2.5
CEA Saclay (France)	Benoit Gwinner	Bio-Logic SP-200, EC-Lab V10.40
CETIM Mulhouse (France)	Xavier Ledoux	Gamry Ref600+, ESA410
CNRS (France)	Kieu ngo	Maxion + Gamry Ref600, ESA410
Curtin (Australia)	Yang Hou	Gamry Ref600, ESA410 v6.25
Gamry (USA)	Dominik Moosbauer	Gamry Ref600+, ESA410 v7.04
HRL Laboratories (USA)	John Vajo	Solartron 1287 , CorrWare
INSA Lyon (France)	José Bolivar	Gamry Ref600+, ESA410 v7.03
IPS (Germany)	Peter Schrems	IPS PGU 10V-1A-IMP-S
IRSN, Cadarache (France)	Walter John Chitty	Gamry Ref600+, ESA410
NNL (England)	Jordan Knapp	Gamry Ref600, ESA410
PSI (Switzterland)	Stefan Ritter	IPS EcmNoise
Rolls-Royce (England)	Tony Horner	ACM, ACM Instruments v5
SIKA (Switzterland)	Bakalli Mirdash	Gamry Ref600, ESA410 v7.03
VTT (Finland)	Konsta Sipilä	Gamry Ref600, EN120 and ESA410
WITg (Switzterland)	Arnulf Hörtnagl	IPS PGU 10V-1A-IMP-S, EcmWin v 2.6.1
ZAG (Slovenia)	Bojan Zajec	IPS HRU/ZRA FG-B-2M

3 manufacturers

11 Gamry, 4 Solartron, 2 Bio-logic, 4 IPS, 1 Maxion, 1 PAR (Ametek), 1 ACM

Algorithm for PSD calculation with FFT

in this work: each time record divided in N sections of M = 512 data points

N = int(number of data in the file / 512): from 8 to 350

loop N times

{

acquisition of M data points of $x(t)$

linear detrending

remove the mean value of x (not informative since corresponds to frequency 0)

multiply by the Hann window

FFT

PSD calculation:

$$\Psi_x(m\Delta f) = \frac{2}{T}\left|X_T(m\Delta f)\right|^2 = \frac{2}{M}\Delta t\left|\sum_{n=0}^{M-1} x(n\Delta t)\, e^{-2i\pi mn/M}\right|^2$$

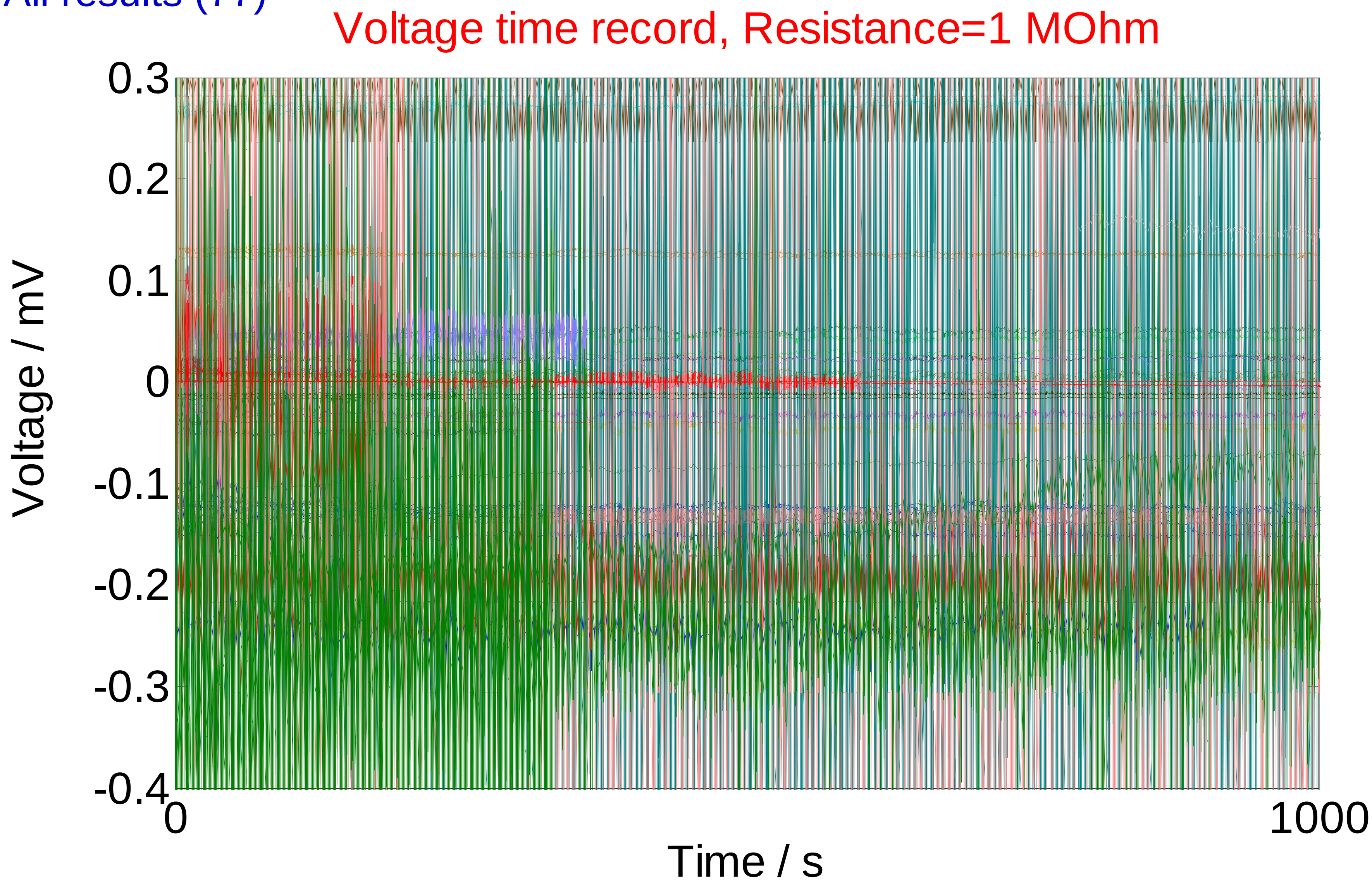
}

average the N PSDs

if Hann: multiply the result by 8/3.

Part 1 – RR12: analysis of the voltage time records (R = 1 MΩ)

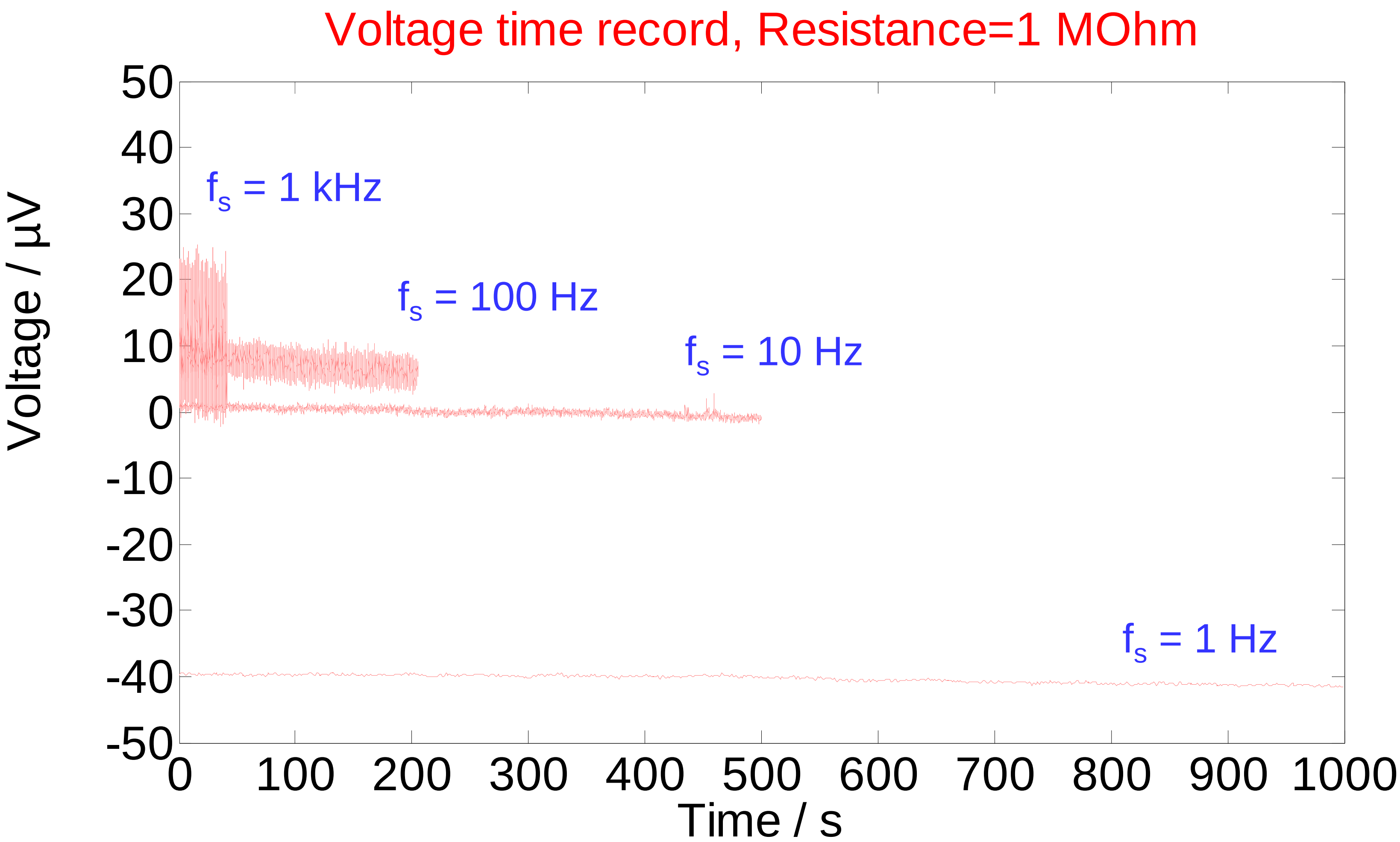
All results (77)



quite impossible to see all time records in the same plot

Part 1: analysis of the voltage time records (R = 1 MΩ)

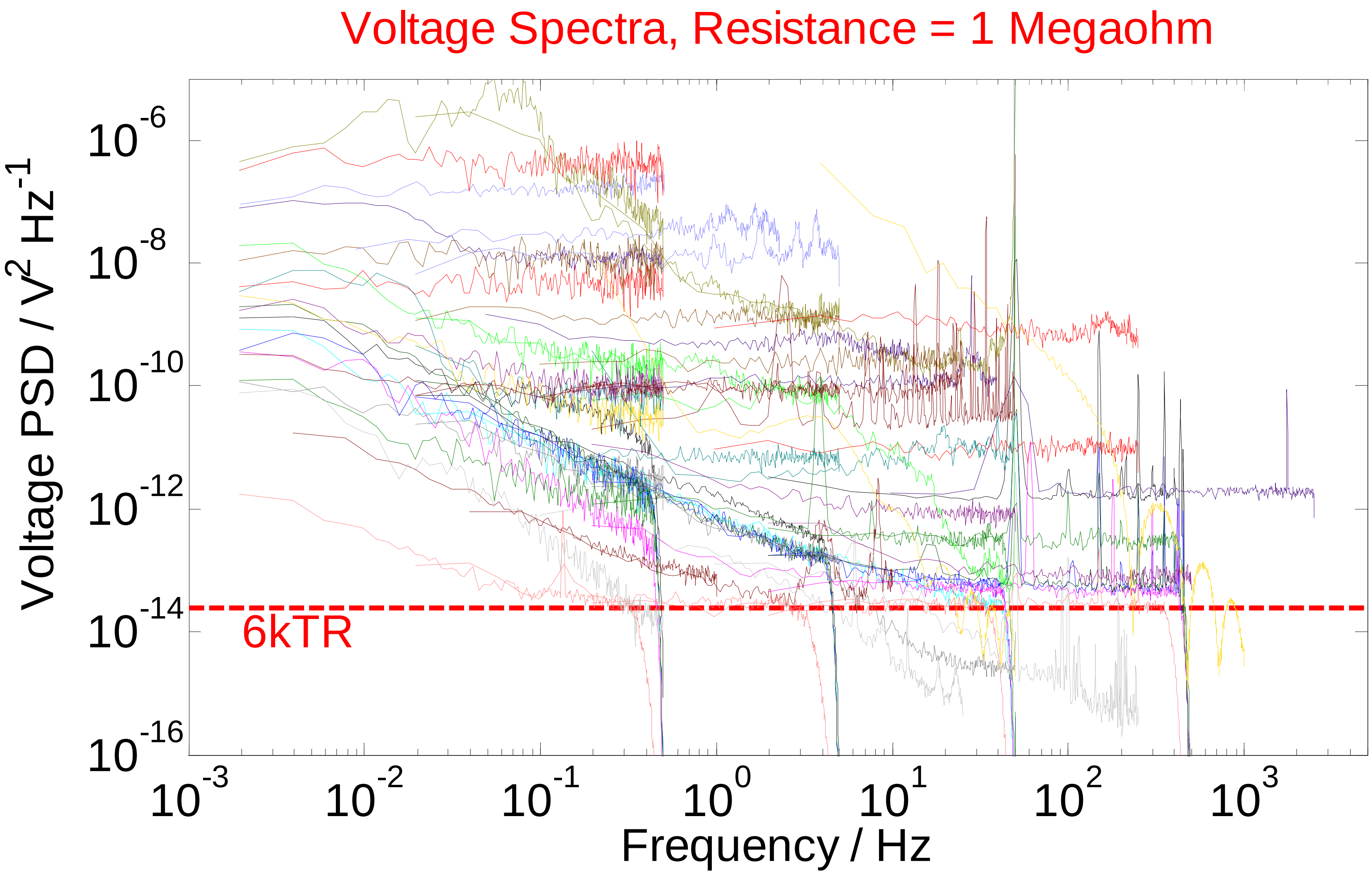
Maxion (4)



difficult to analyze a white noise in the time domain ➡ frequency domain

Part 1 – RR8 – 2014: analysis of the voltage PSDs (R = 1 MΩ)

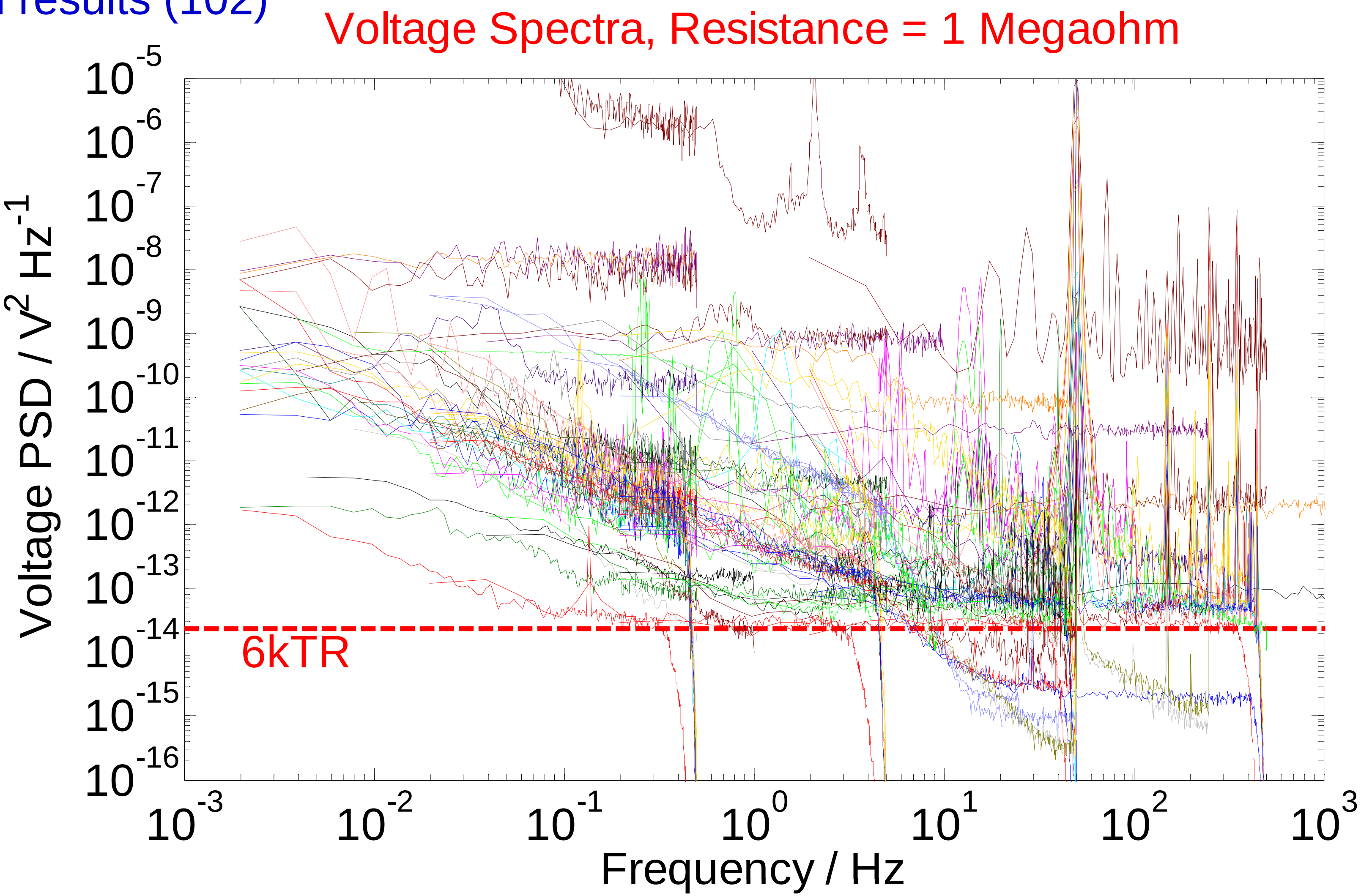
All results but 4 (65)



large scatter: not better than PSD measured before 2008 (round-robin paper)

Part 1 – RR11 – 2016: analysis of the voltage PSDs (R = 1 MΩ)

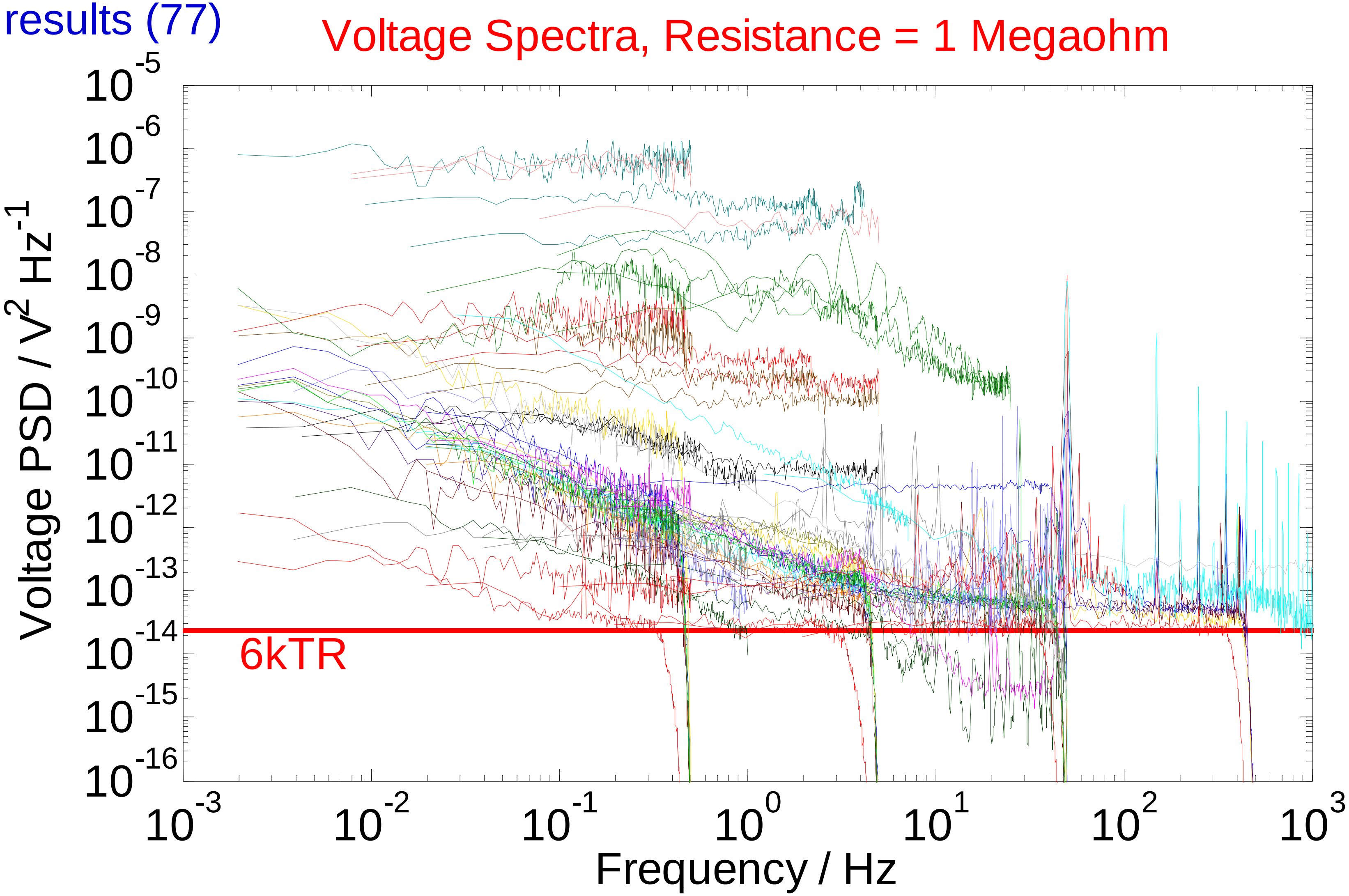
All results (102)



large scatter: not better than PSD measured in 2014

Part 1 – RR12 – 2017: analysis of the voltage PSDs (R = 1 MΩ)

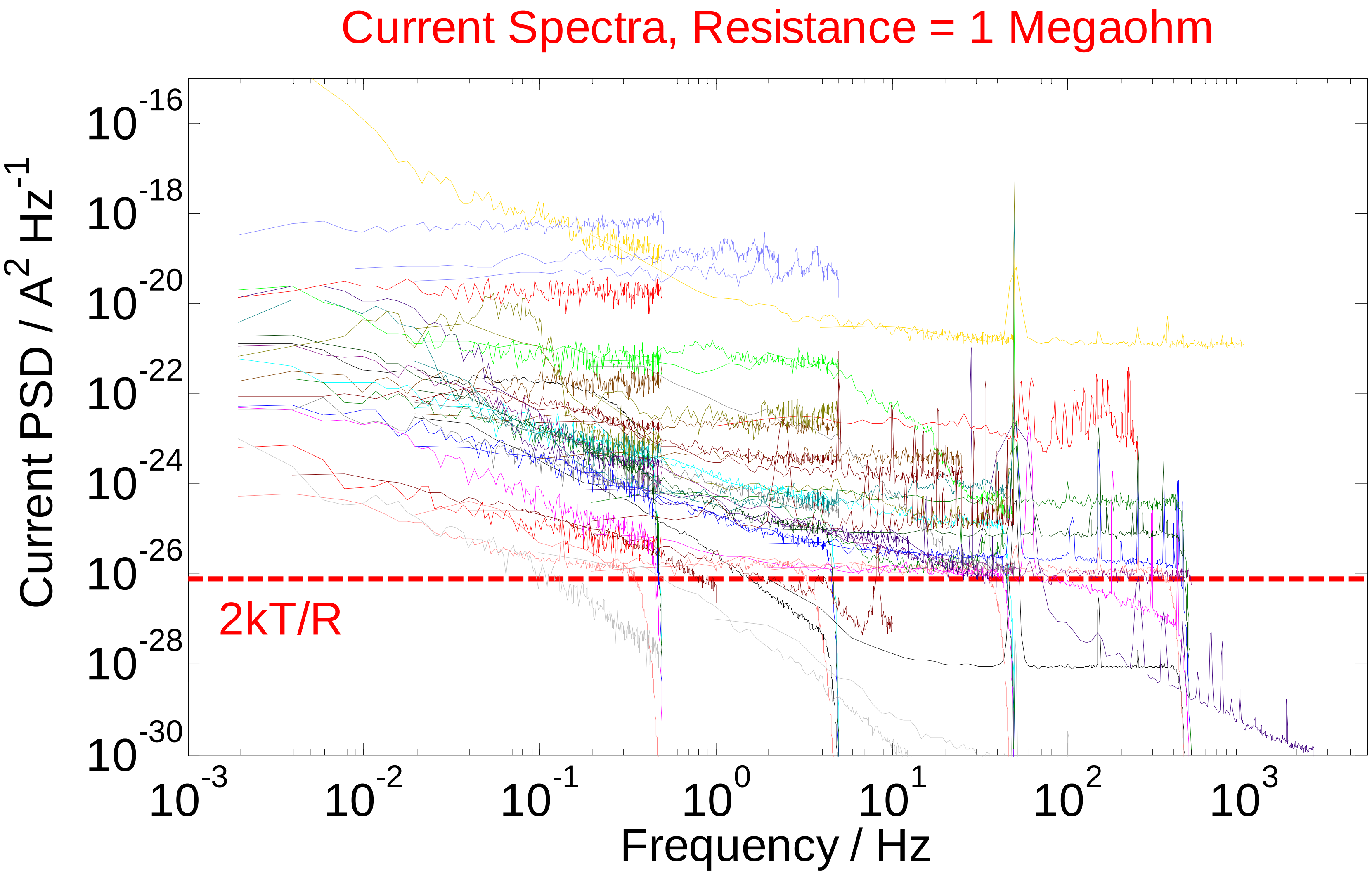
All results (77)



large scatter: not better than PSD measured in 2016

Part 2 – RR8 – 2014: analysis of the current PSDs (R = 1 MΩ)

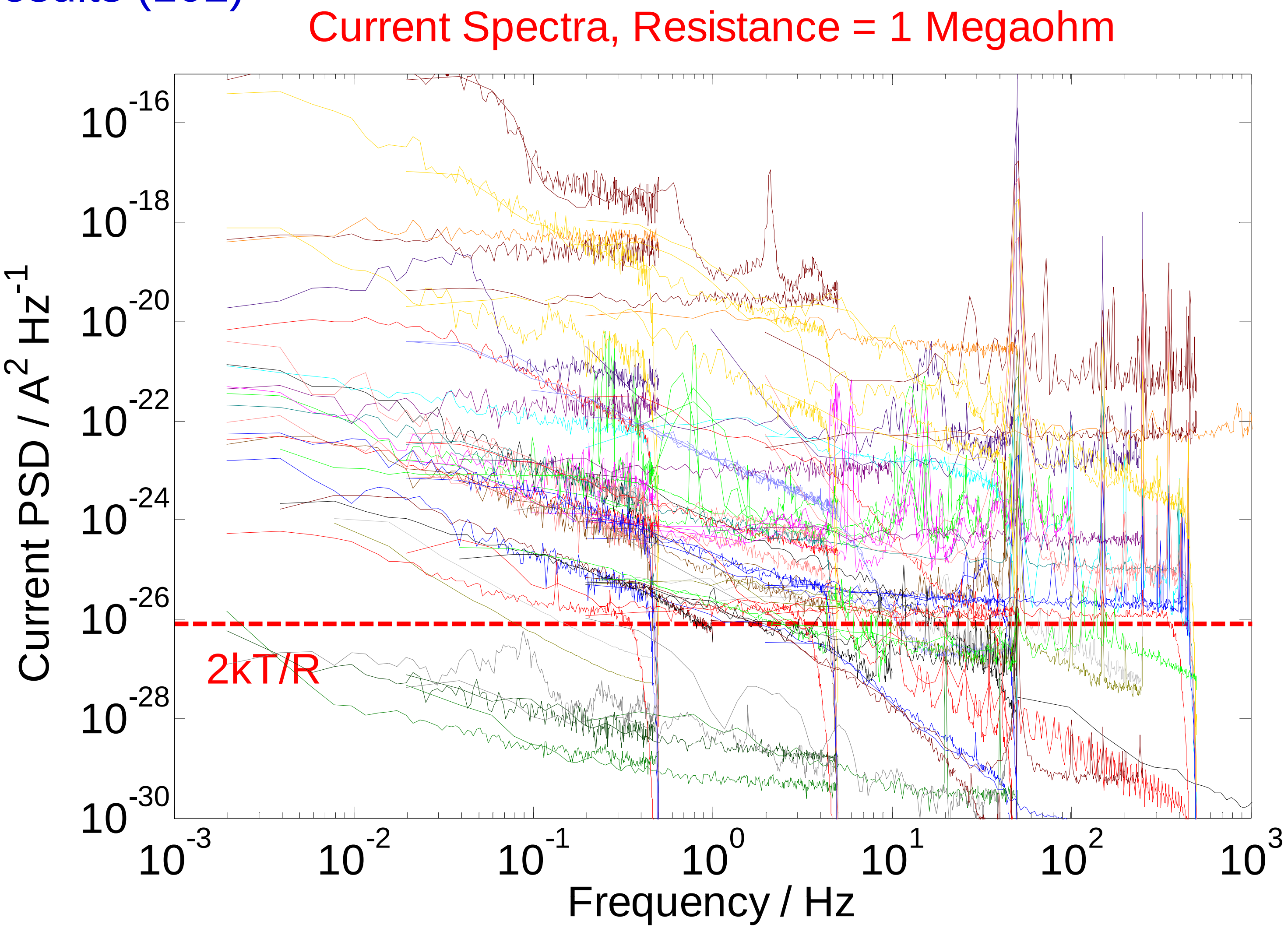
All results but 6 (63)



large scatter: not better than PSD measured before 2008 (round-robin paper)

Part 2 – RR11 – 2016: analysis of the current PSDs (R = 1 MΩ)

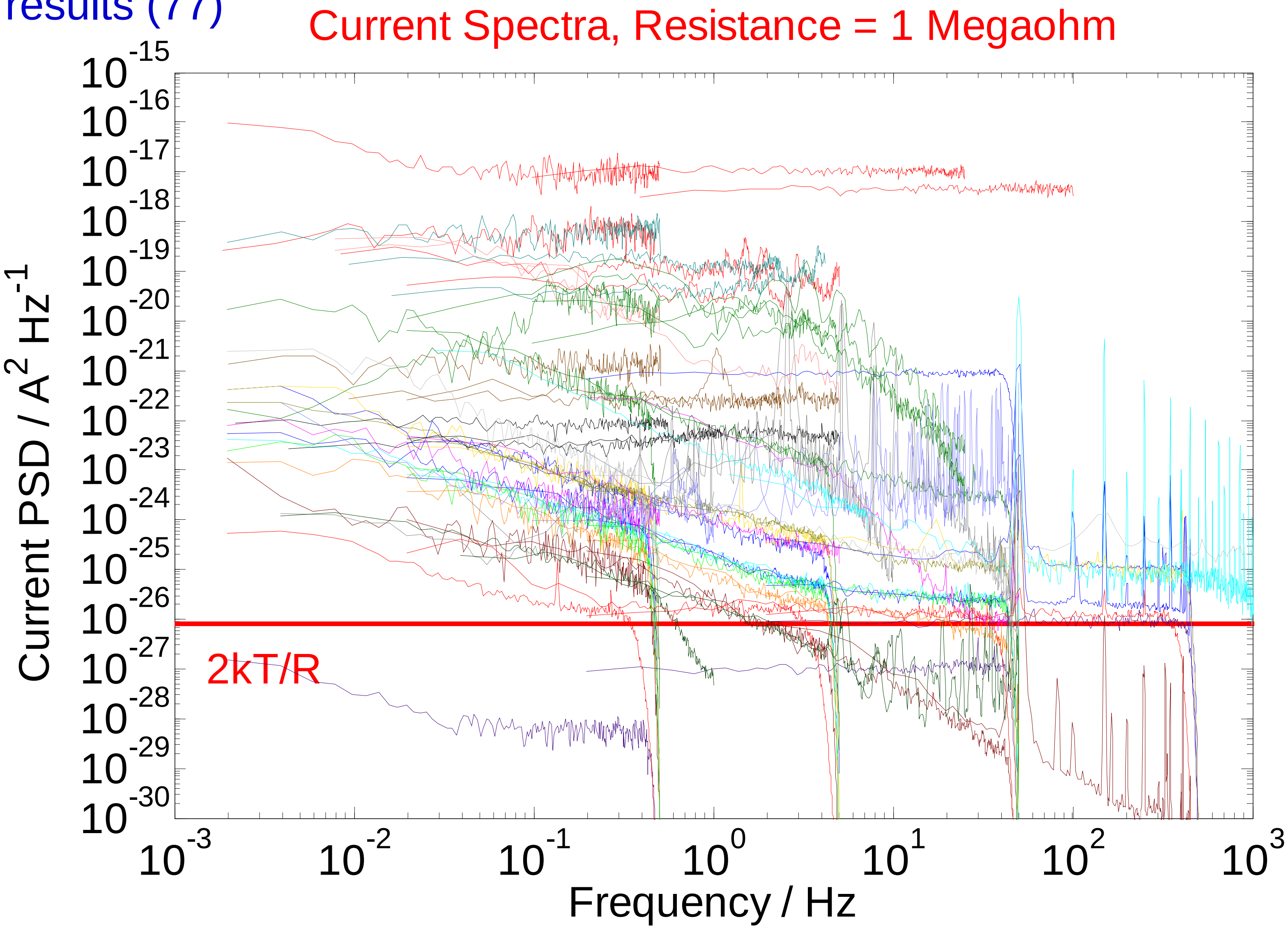
All results (102)



large scatter: not better than PSD measured in 2014

Part 2 – RR12 – 2017: analysis of the current PSDs (R = 1 MΩ)

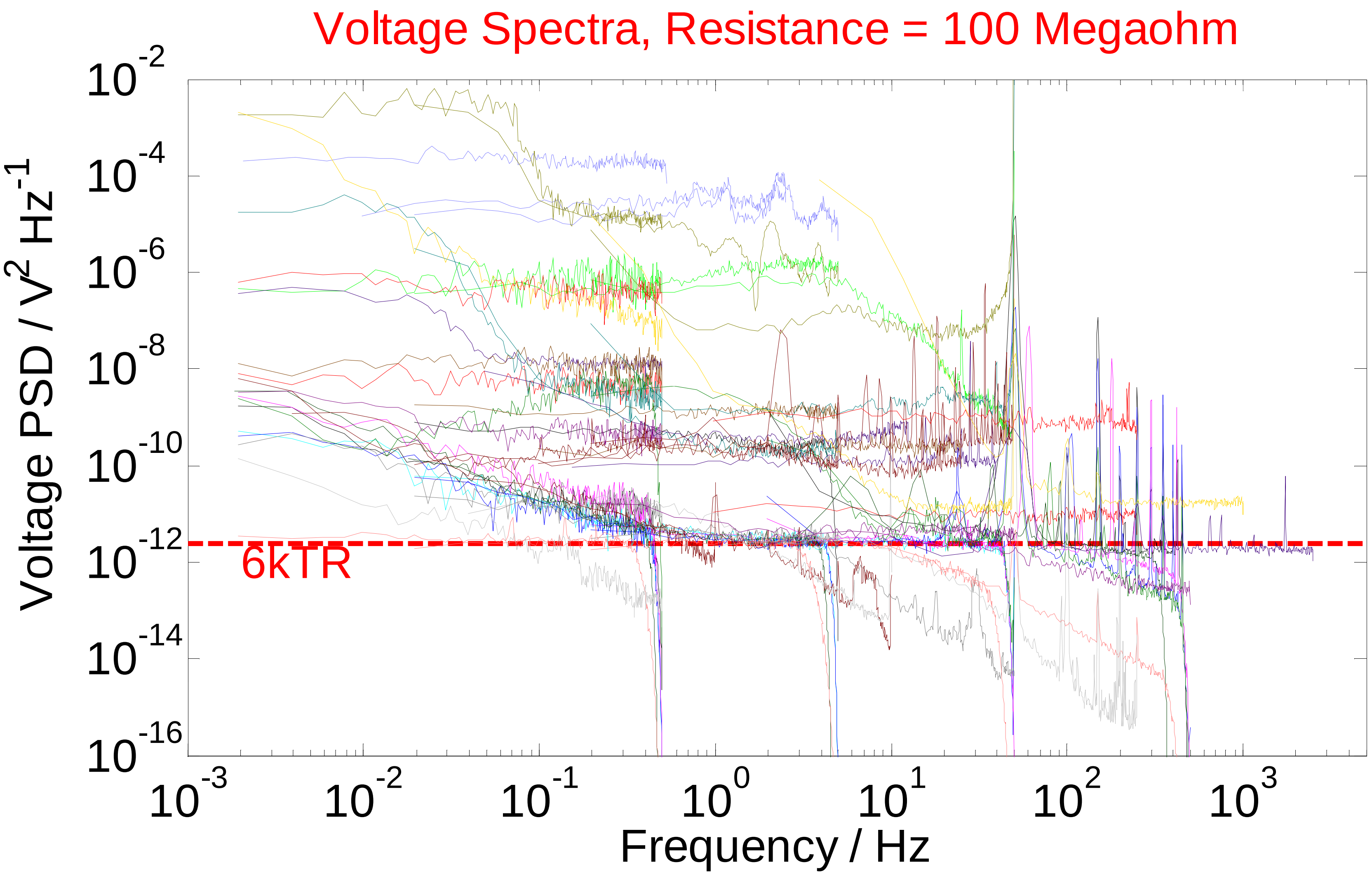
All results (77)



large scatter: not better than PSD measured in 2016

Part 3 – RR8 – 2014: analysis of the voltage PSDs (R = 100 MΩ)

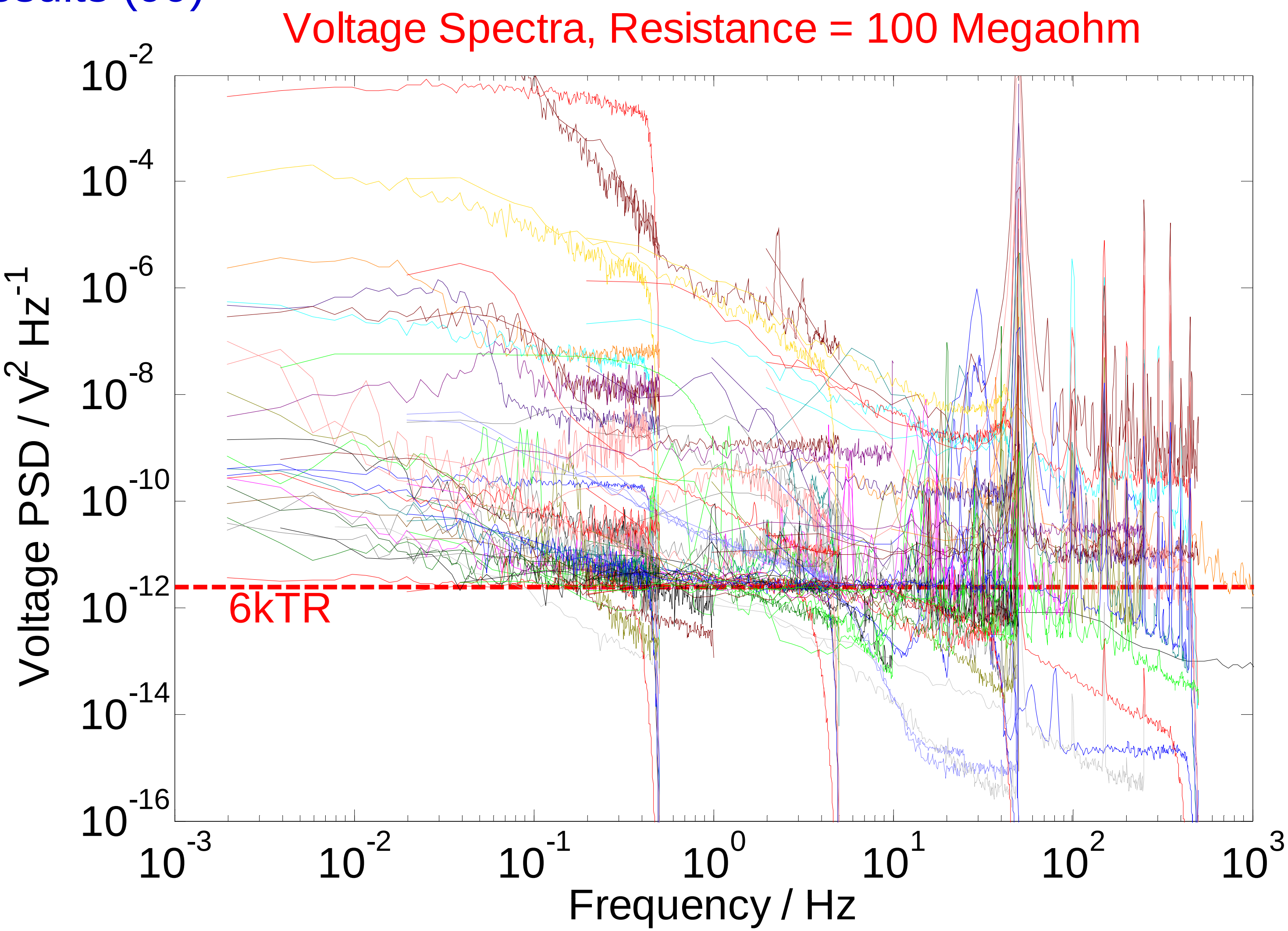
All results but 5 (64)



still large scatter while the voltage noise PSD is 100 times higher

Part 3 – RR11 – 2016: analysis of the voltage PSDs (R = 100 MΩ)

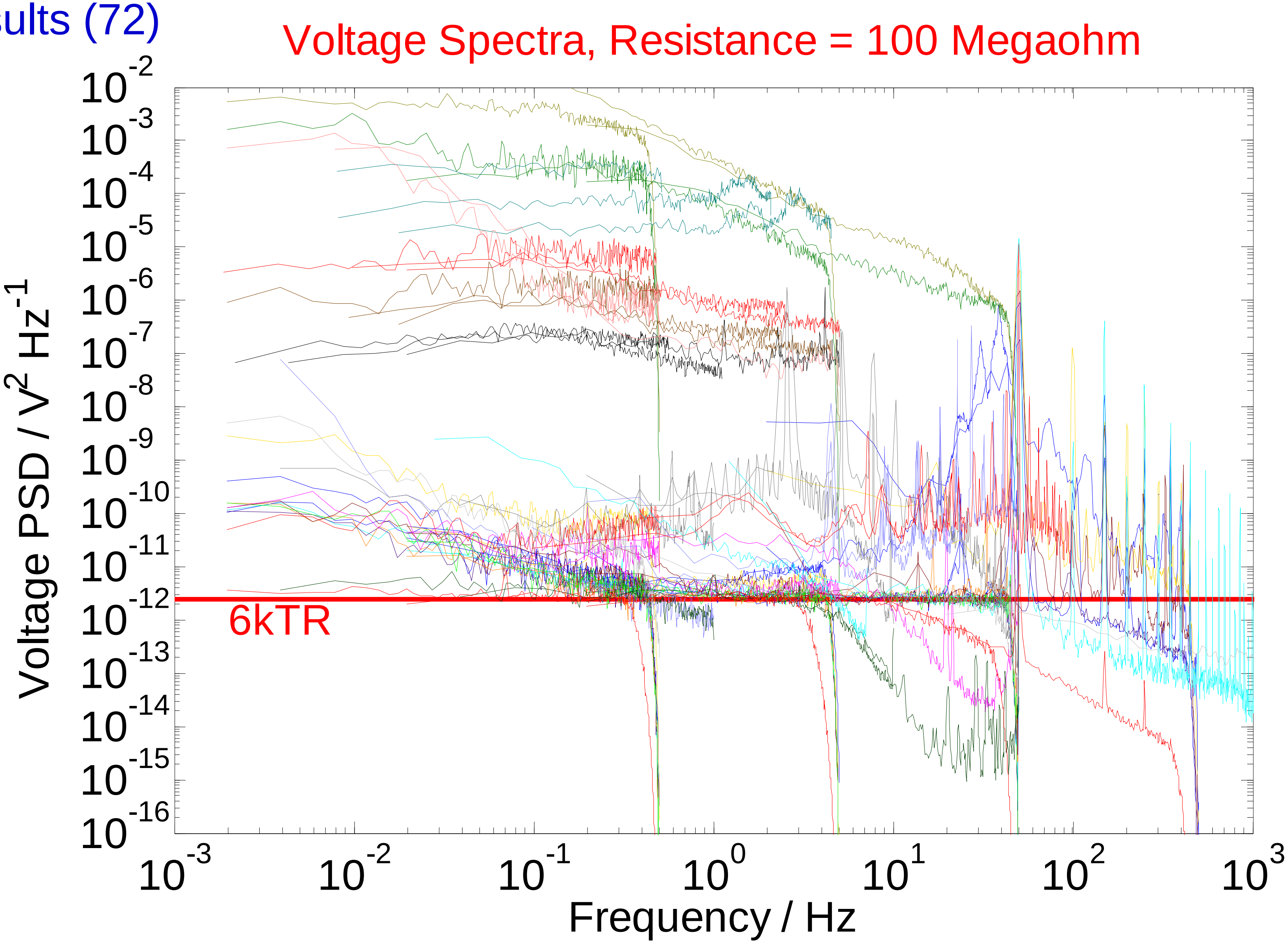
All results (99)



large scatter: not better than PSD measured in 2014

Part 3 – RR12 – 2017: analysis of the voltage PSDs (R = 100 MΩ)

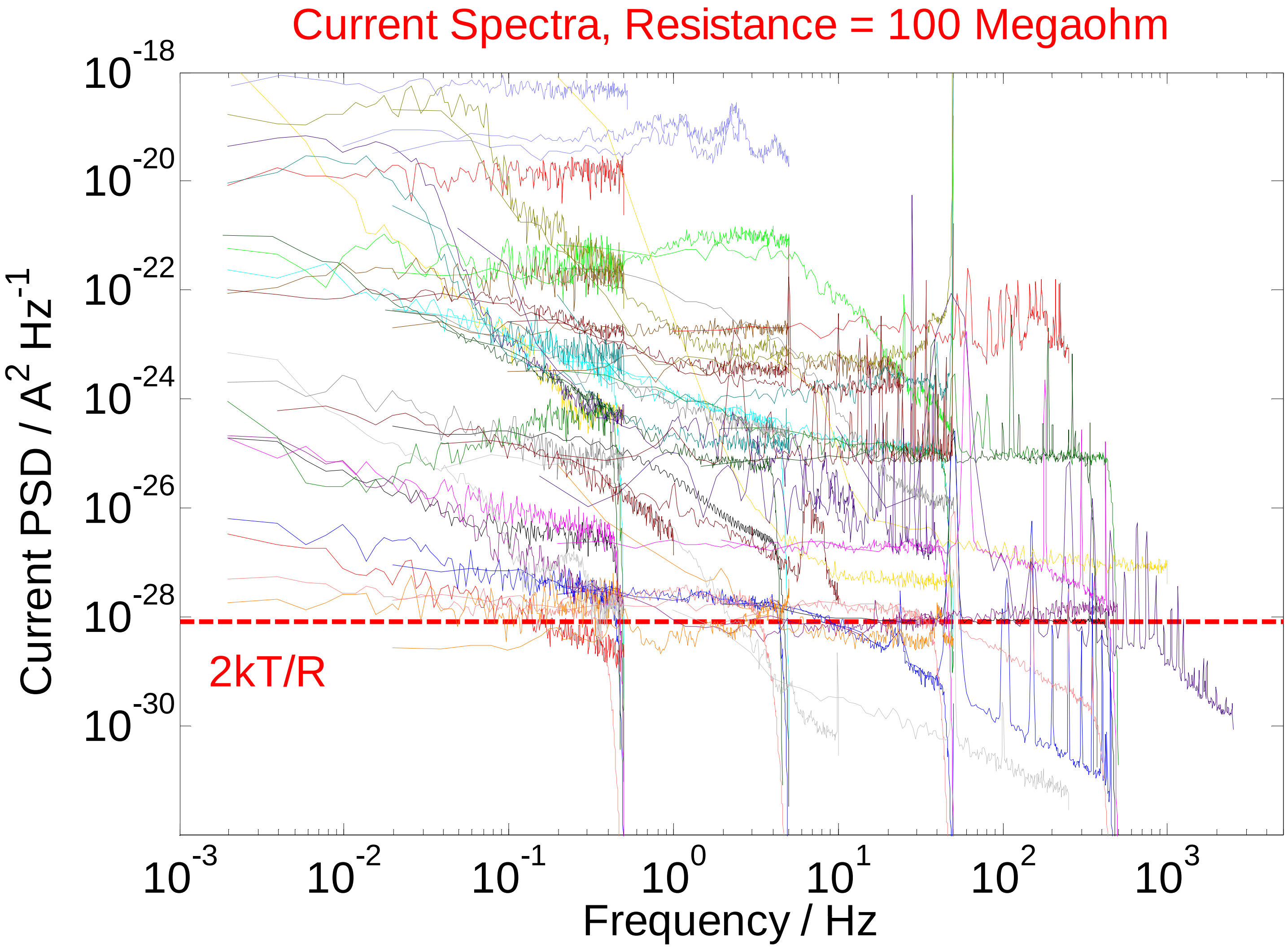
All results (72)



large scatter: not better than PSD measured in 2016

Part 4 – RR8 – 2014: analysis of the current PSDs (R = 100 MΩ)

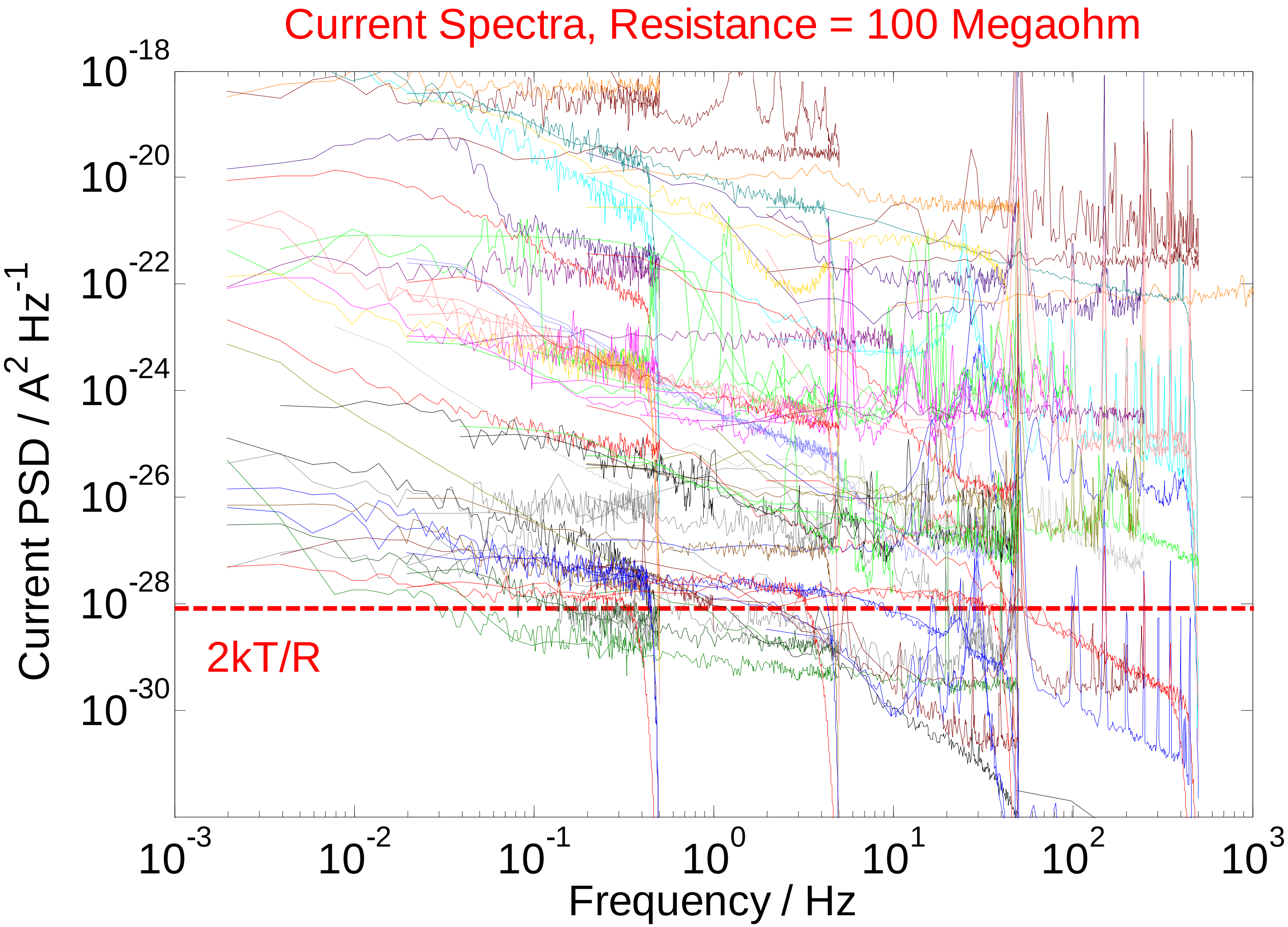
All results but 3 (66)



large scatter but the current noise PSD is low

Part 4 – RR11 – 2016: analysis of the current PSDs (R = 100 MΩ)

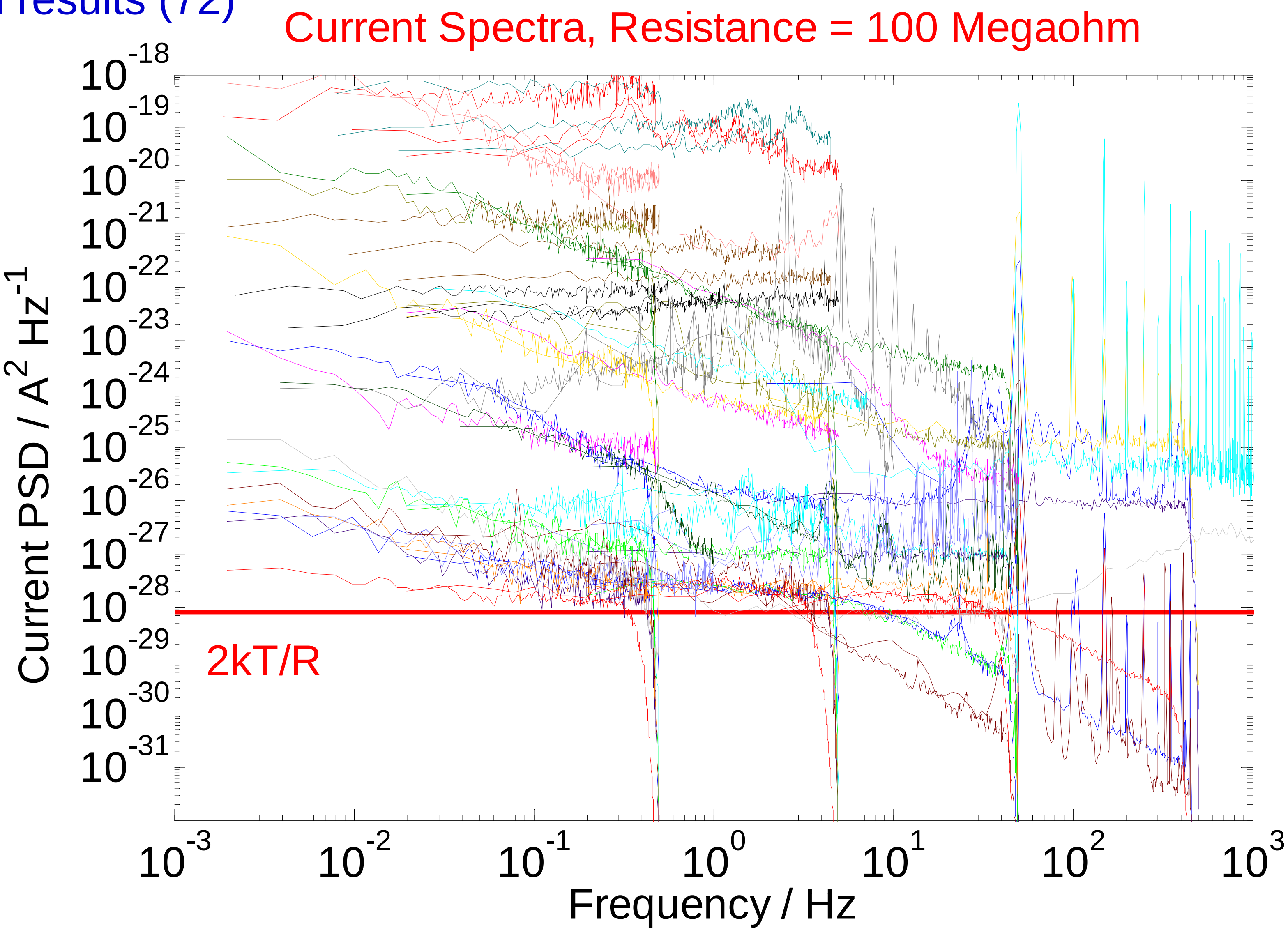
All results (99)



large scatter: not better than PSD measured in 2014

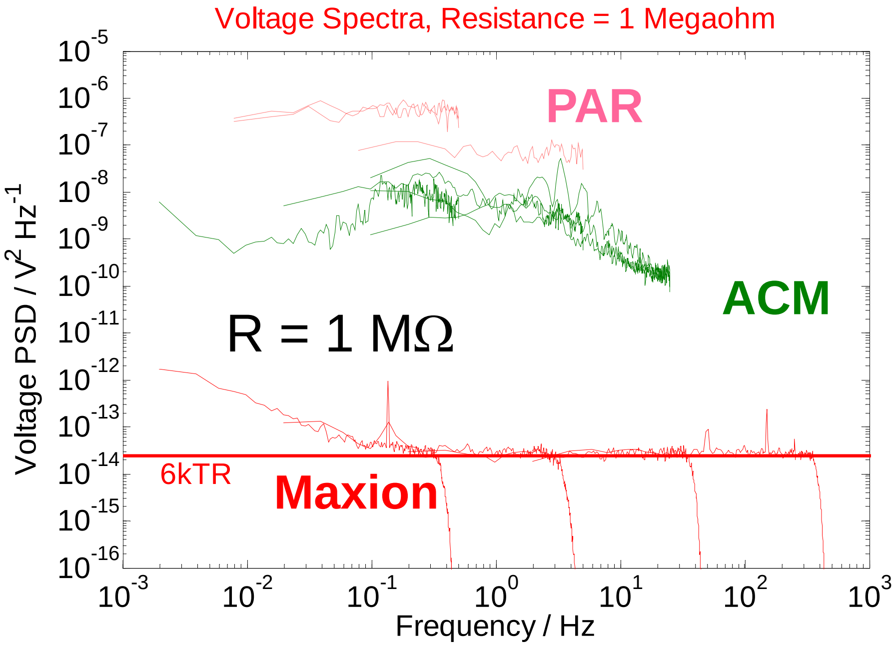
Part 4 – RR12 – 2017: analysis of the current PSDs (R = 100 MΩ)

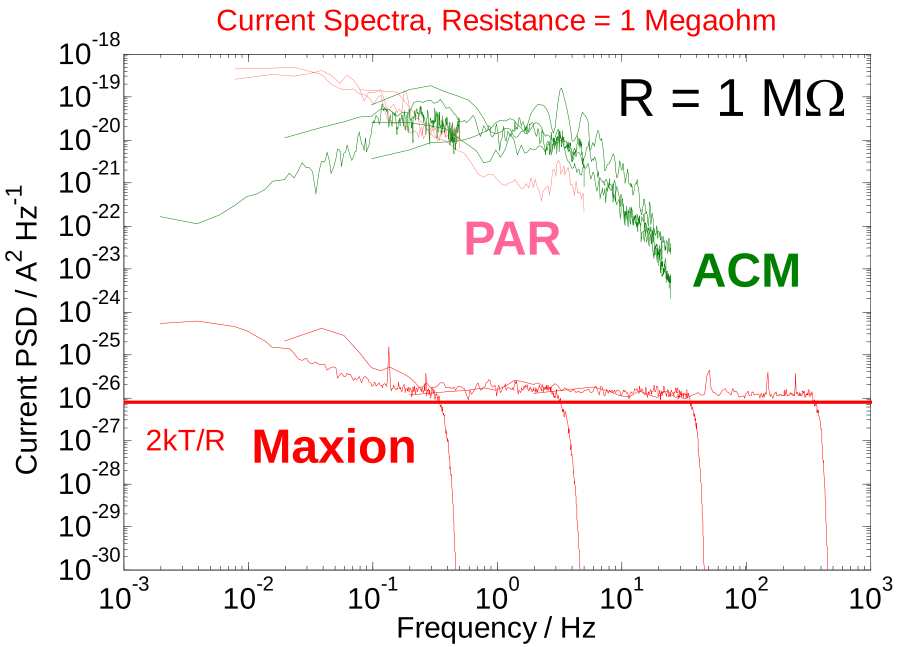
All results (72)

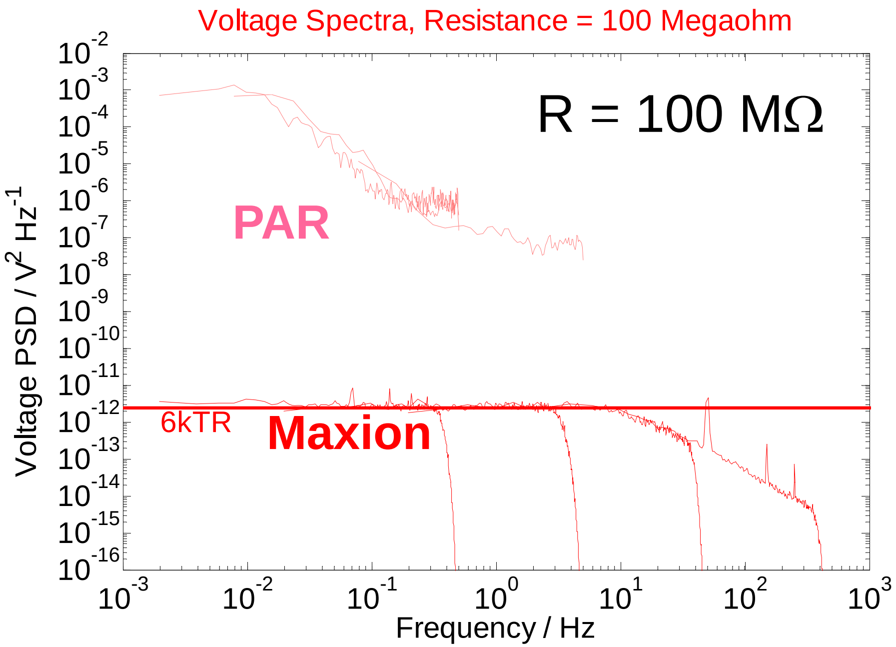


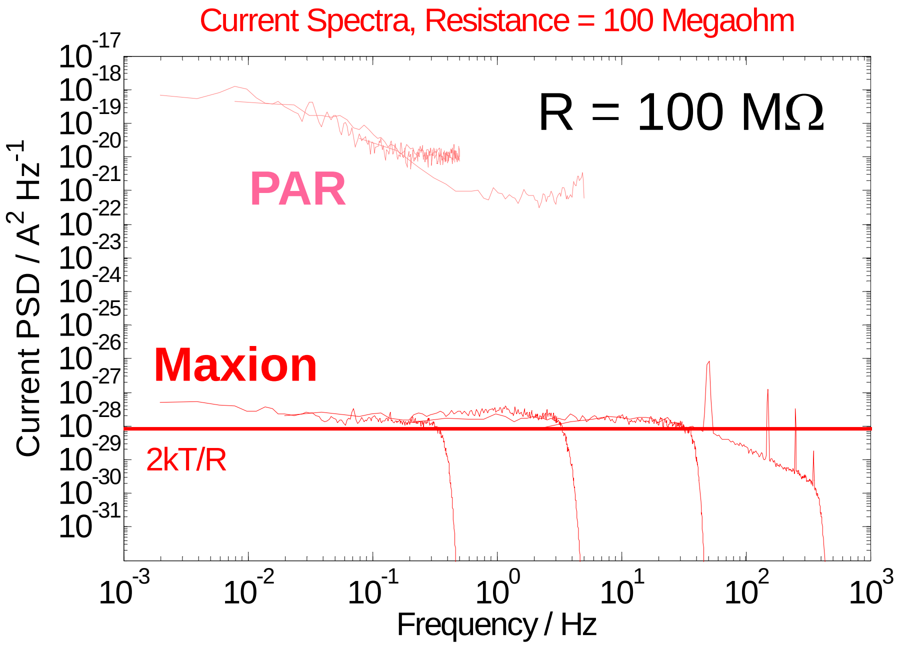
large scatter: not better than PSD measured in 2016

PAR, ACM, Maxion results for R = 1 MΩ and 100 MΩ (RR12 – 2017)

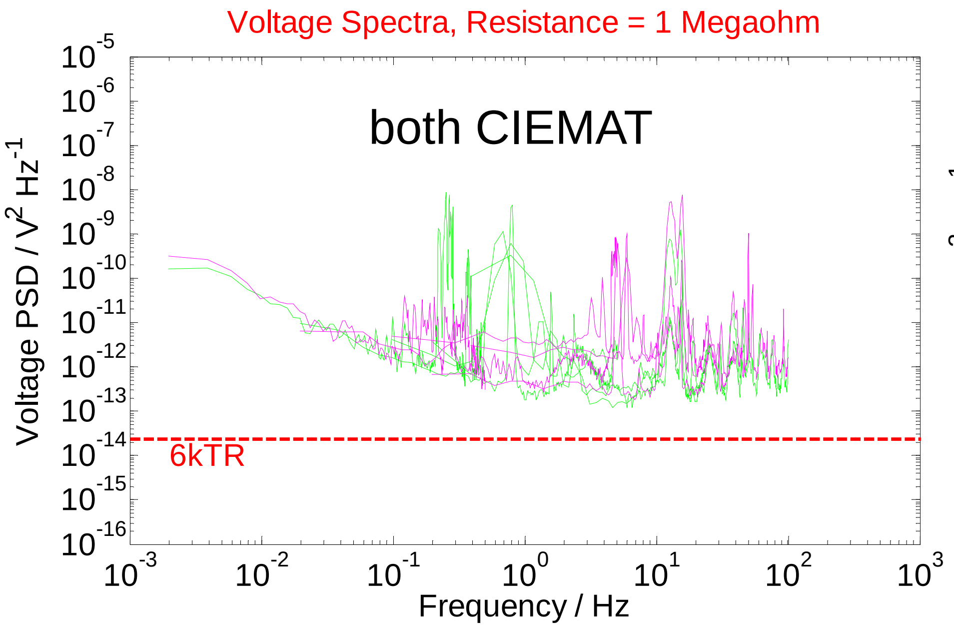


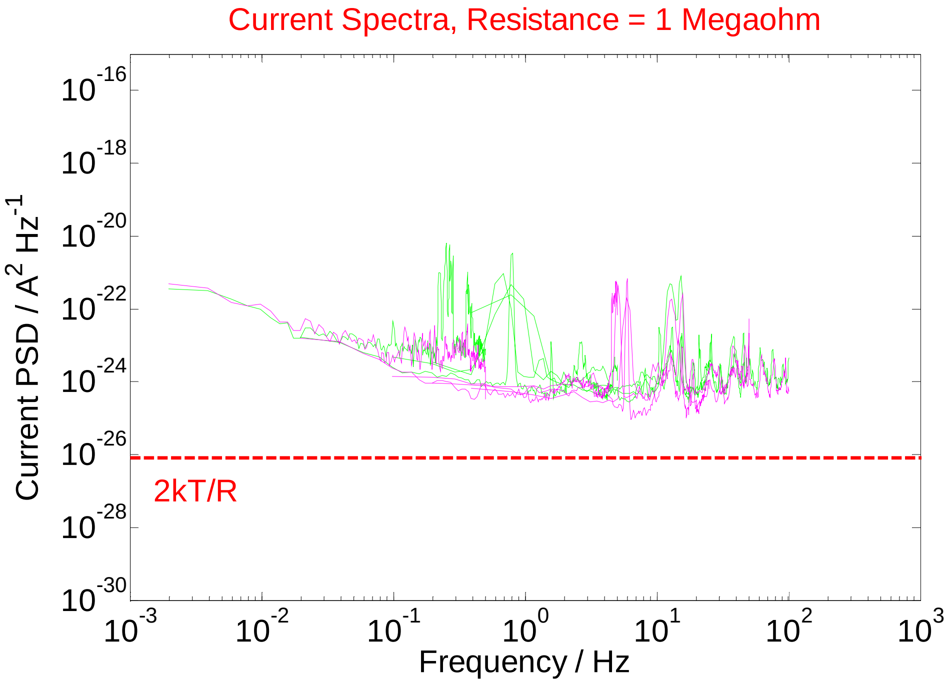






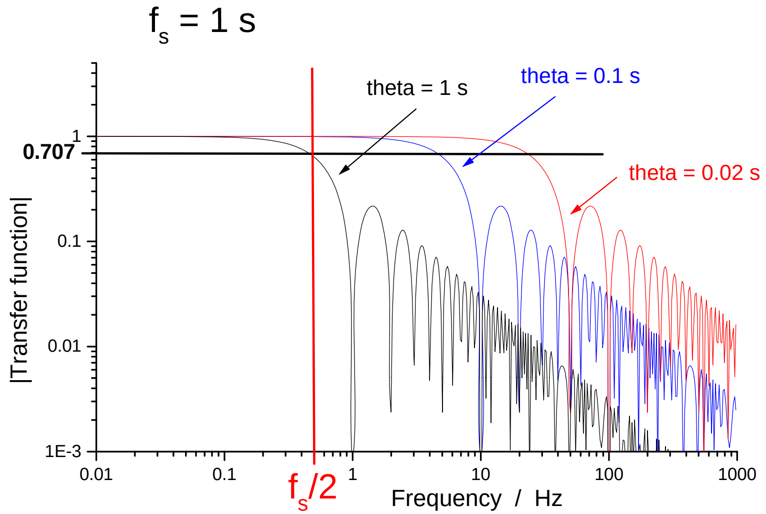
Solartron results for R = 1 MΩ (RR11 – 2016)



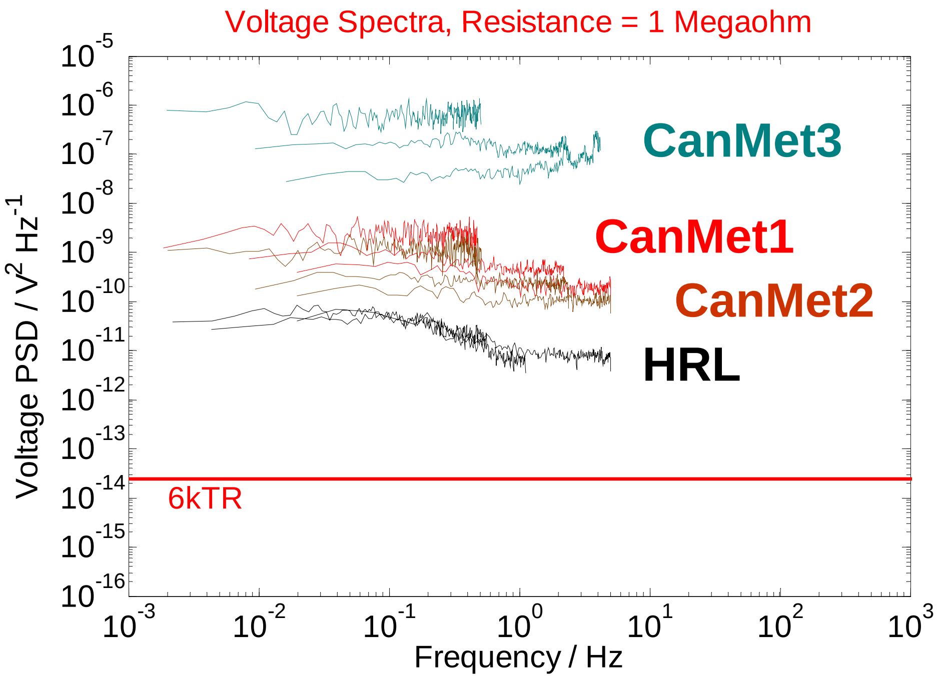


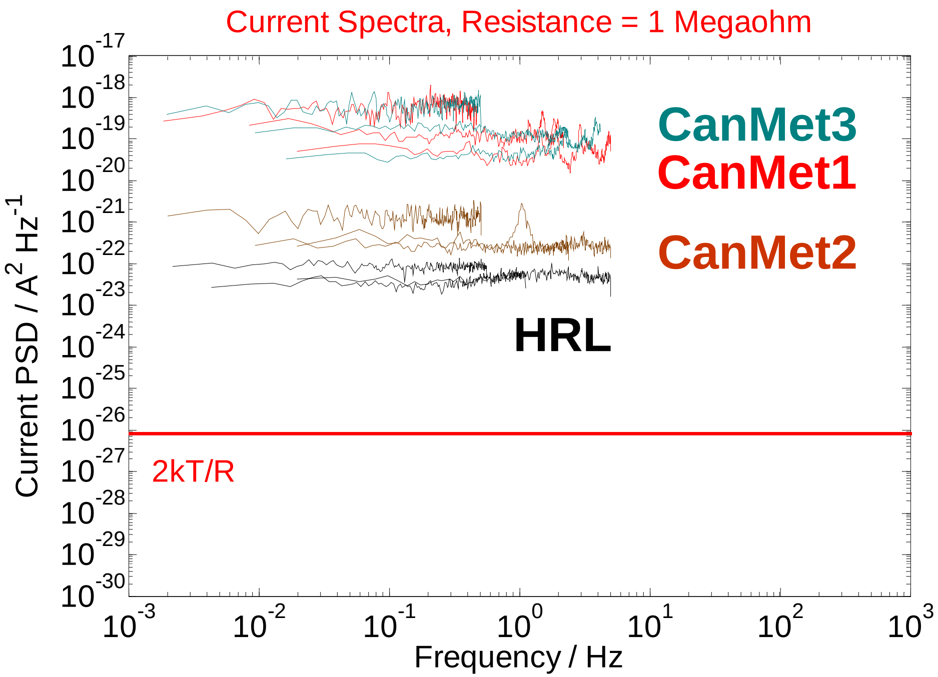
Anti-aliasing filtering by integration of the analog signal during time 1 / f_s like in digital voltmeters

➡ not efficient for eliminating aliasing of 50 Hz and harmonics



Solartron results for R = 1 MΩ (RR12 – 2017)





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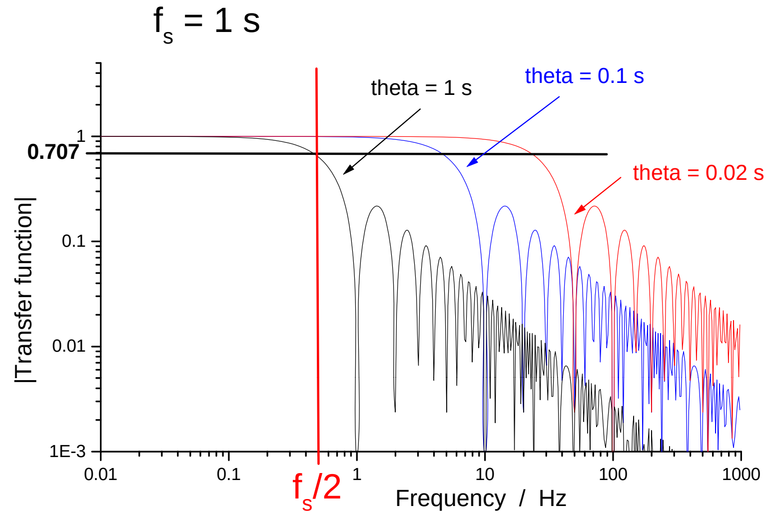
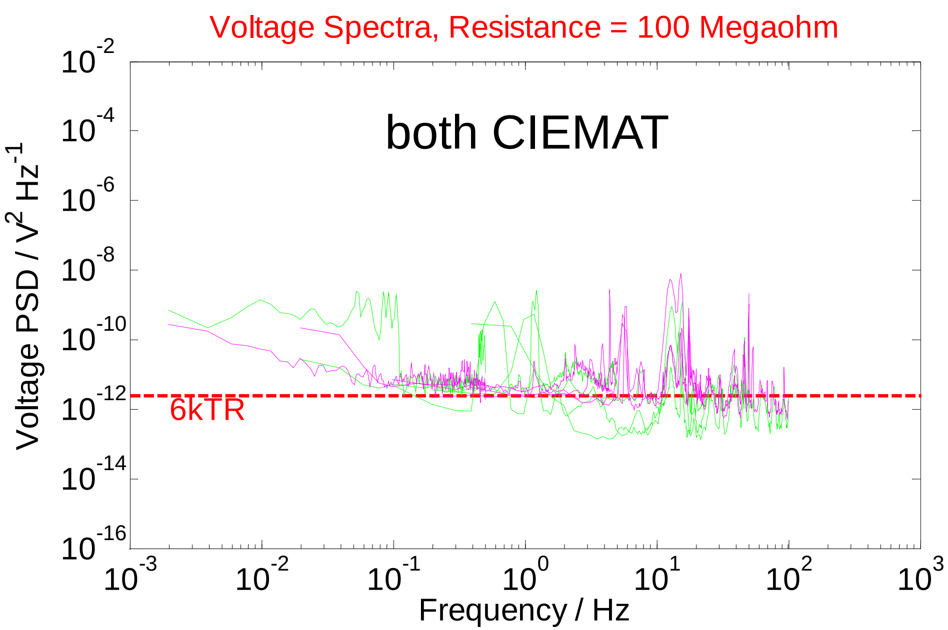
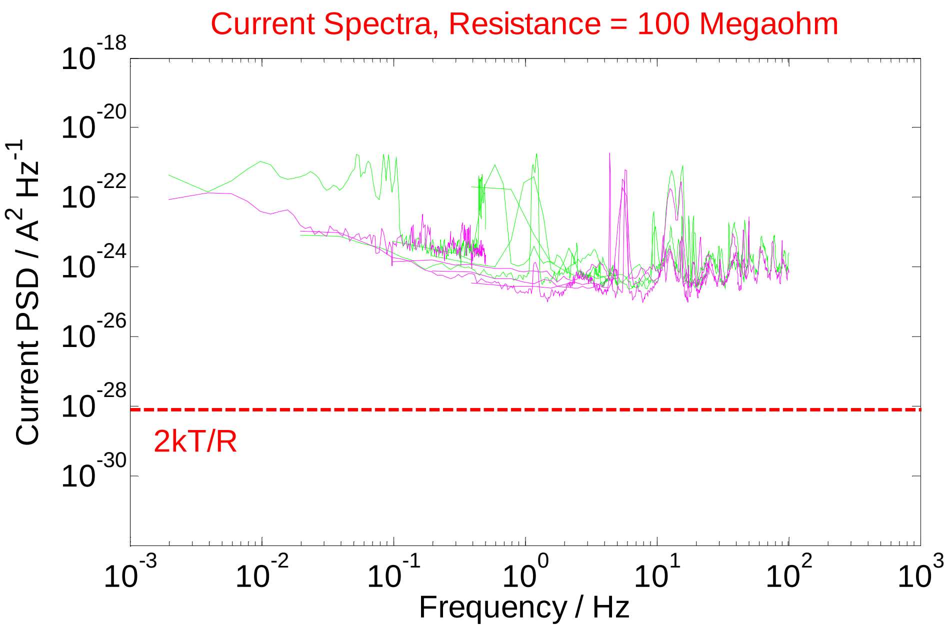


Solartron results for R = 100 MΩ (RR11 – 2016)





Anti-aliasing filtering by integration of the analog signal during time 1 / f_s like in digital voltmeters

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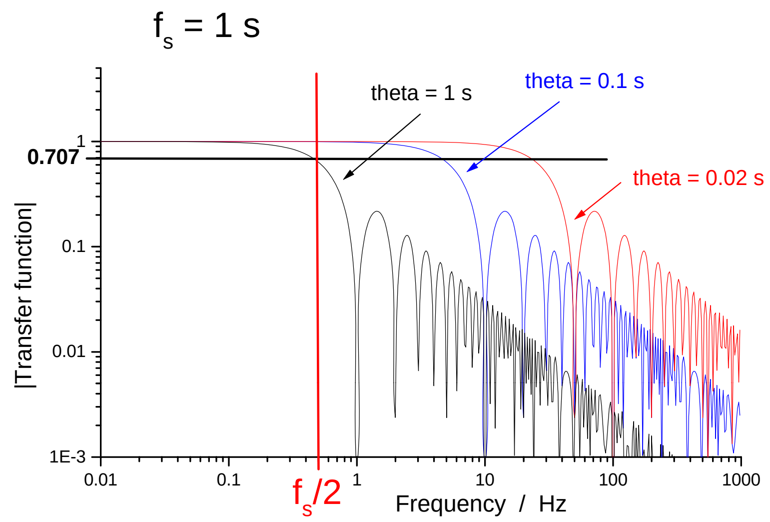
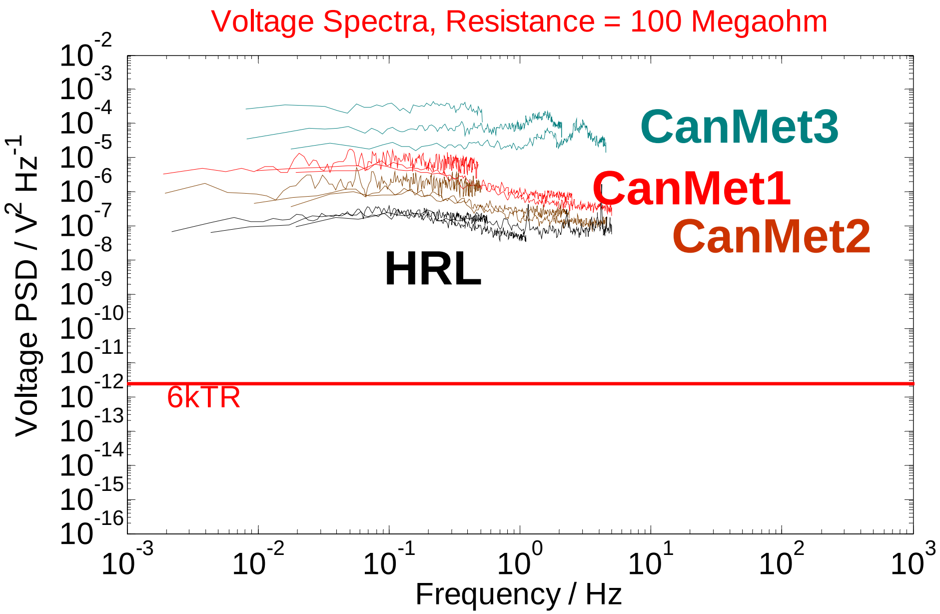
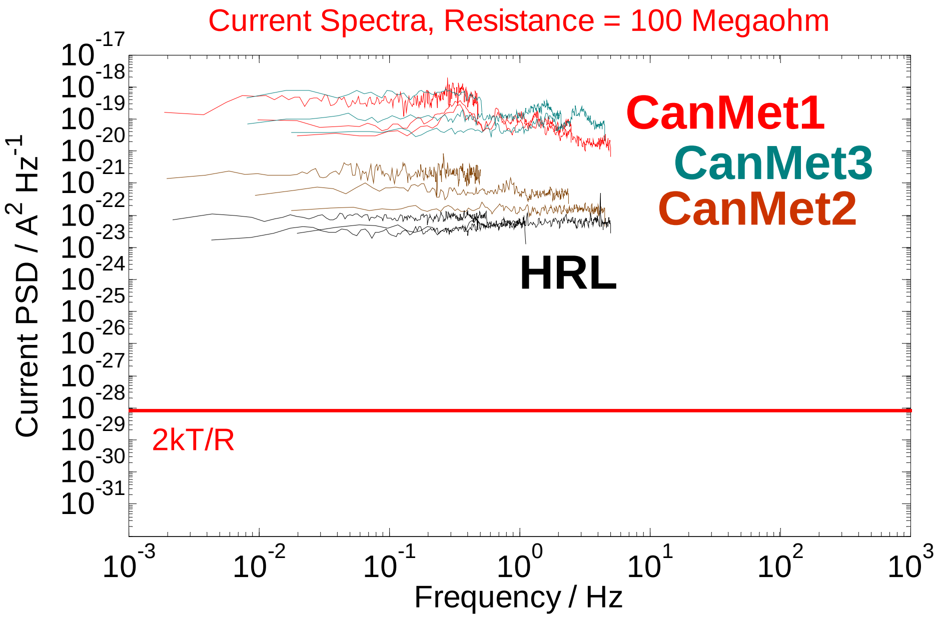


Solartron results for R = 100 MΩ (RR12 – 2017)

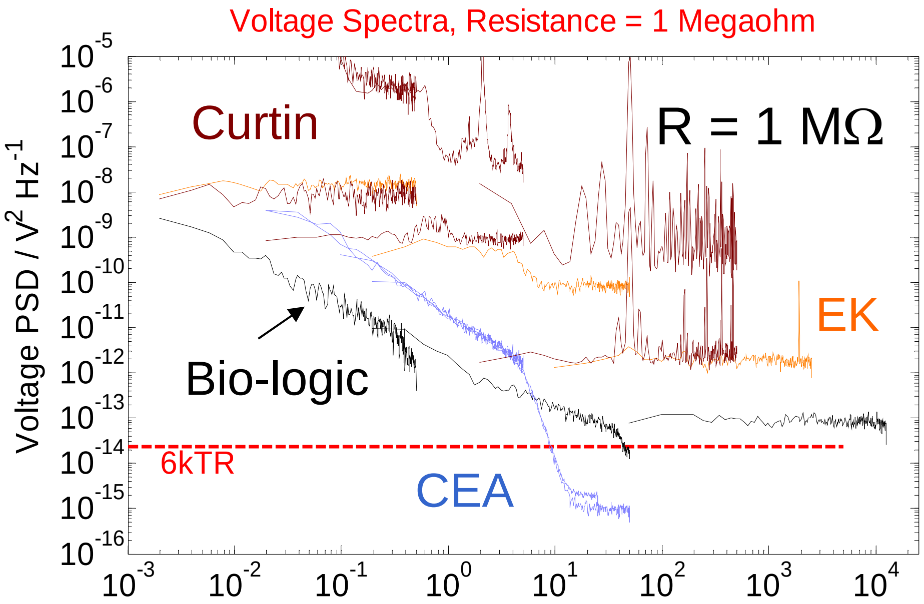


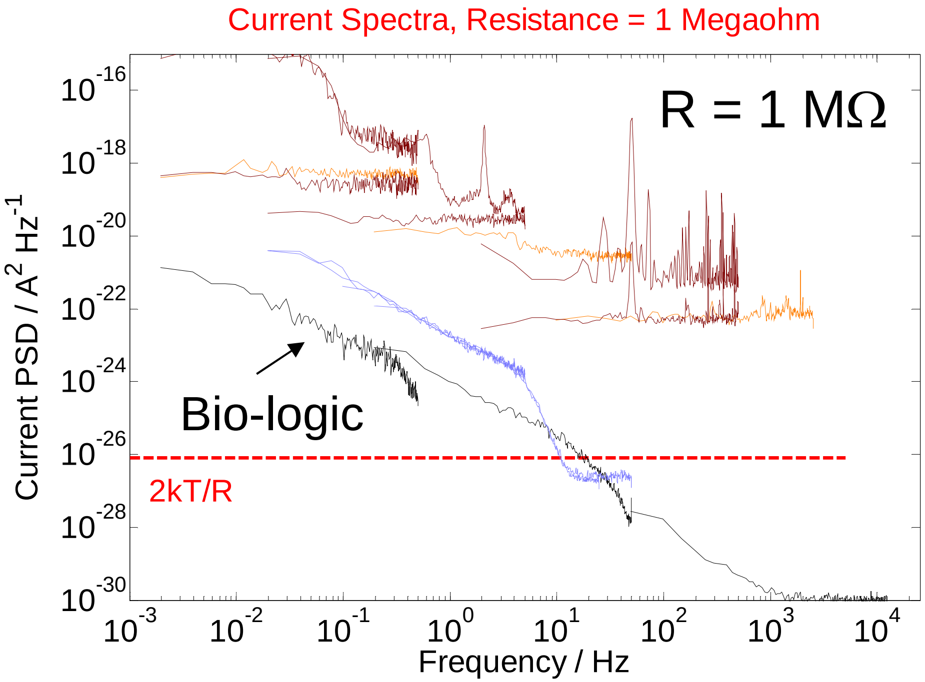


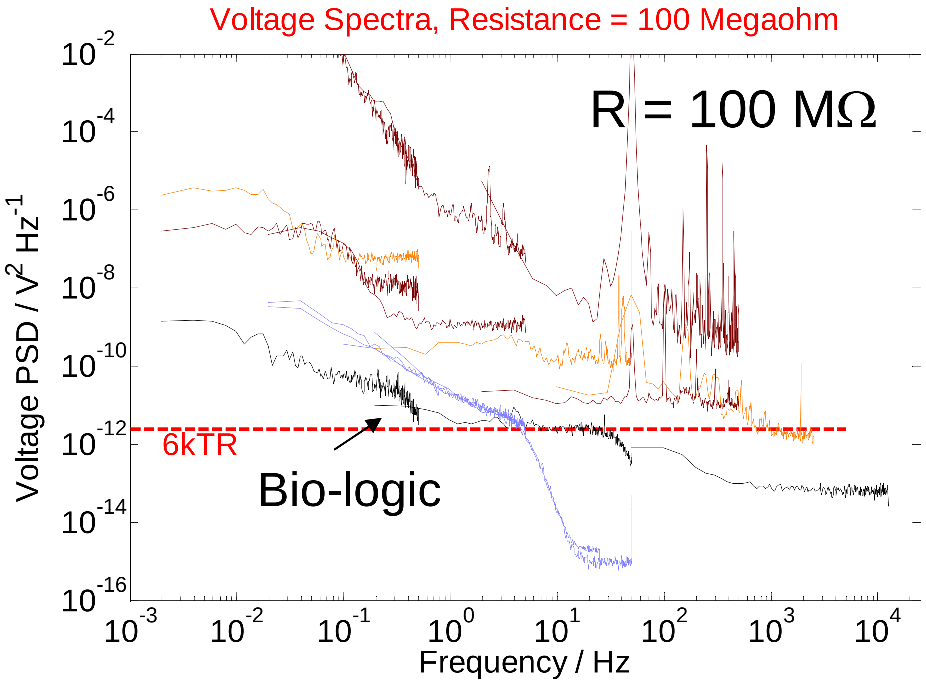
for Solarton 1287:

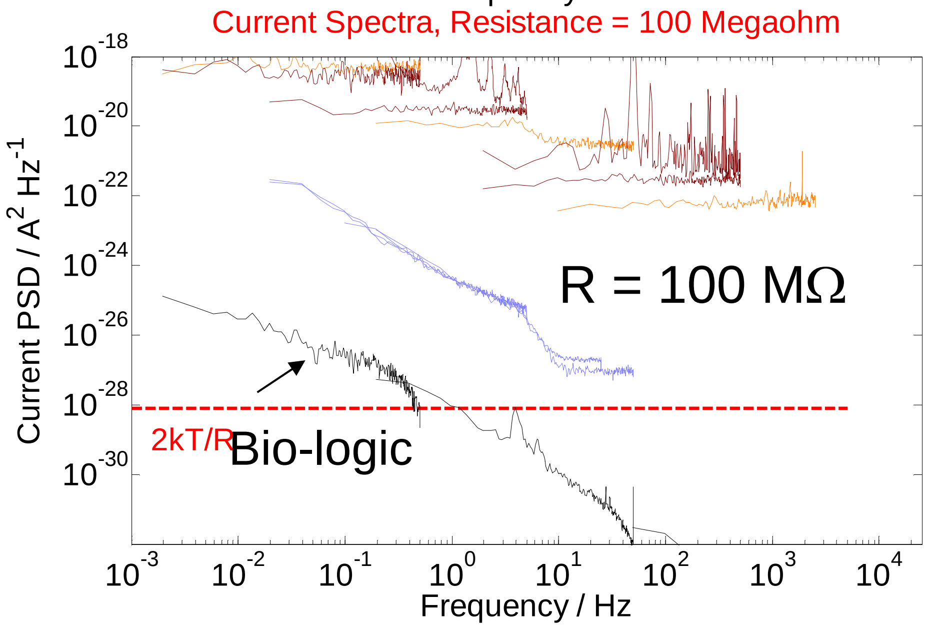
- Anti-aliasing filtering exists (integration of the analog signal during time 1 / f_s)
- not efficient for eliminating aliasing of 50 Hz and harmonics
- the user has to be trained to use it correctly

Bio-logic results for R = 1 MΩ and 100 MΩ (RR11 – 2016)

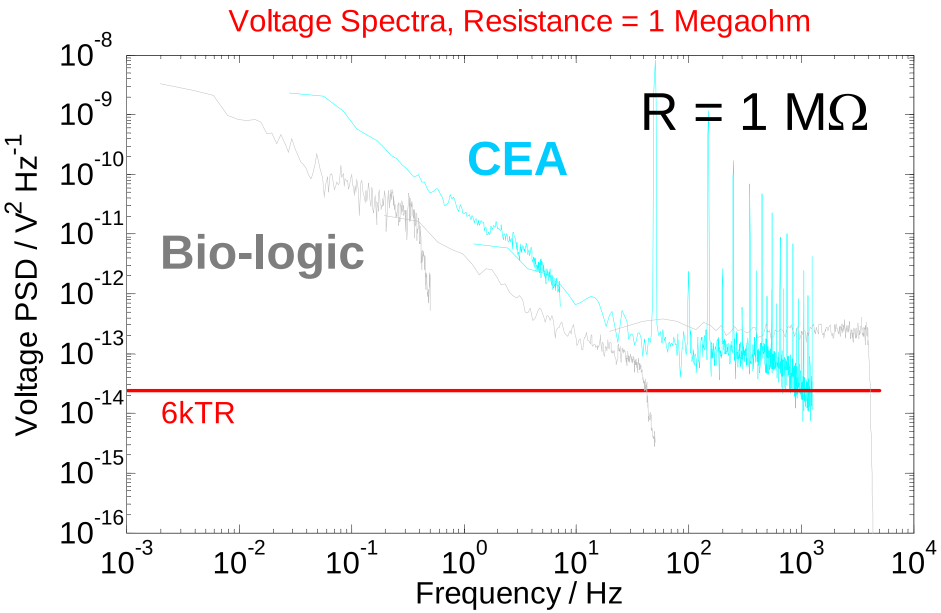


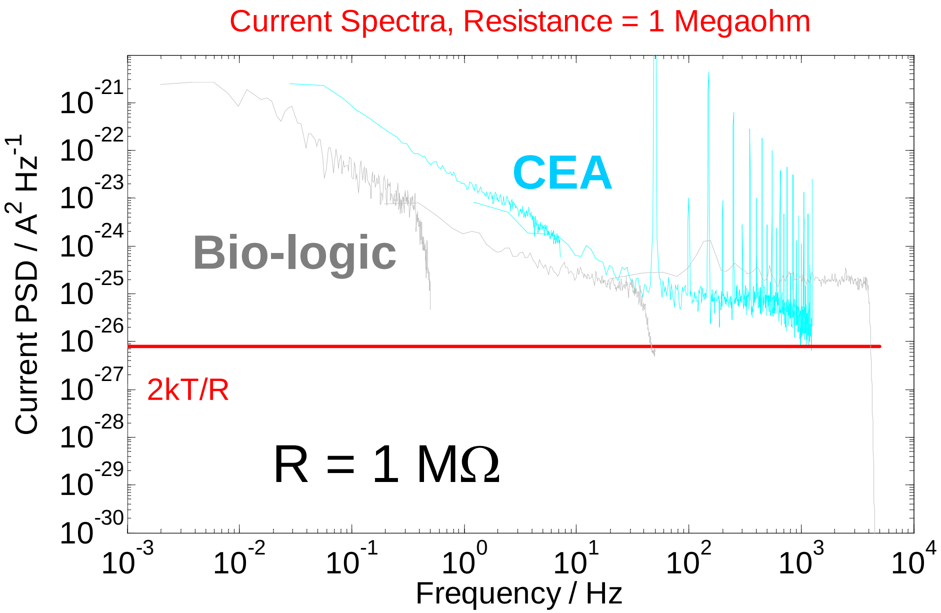


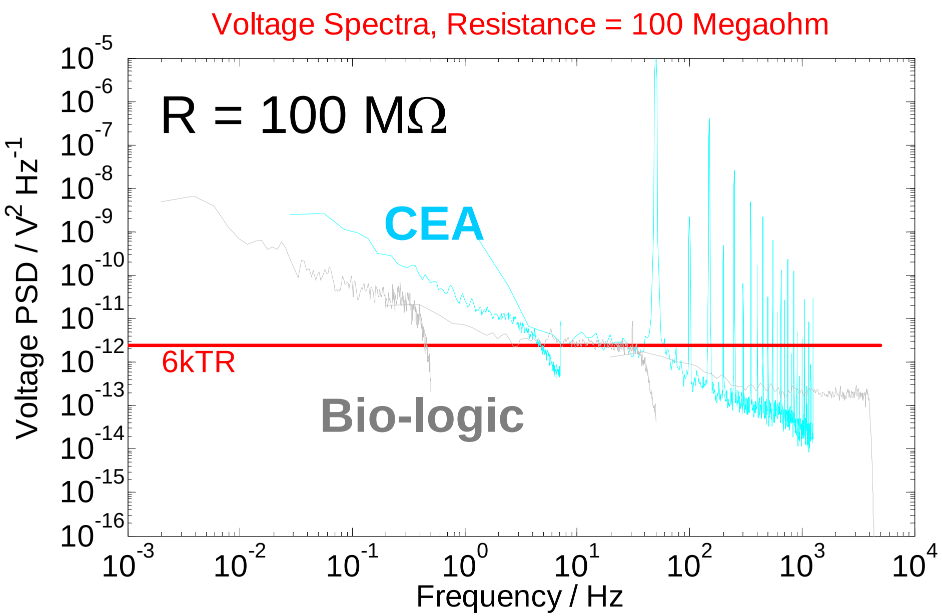


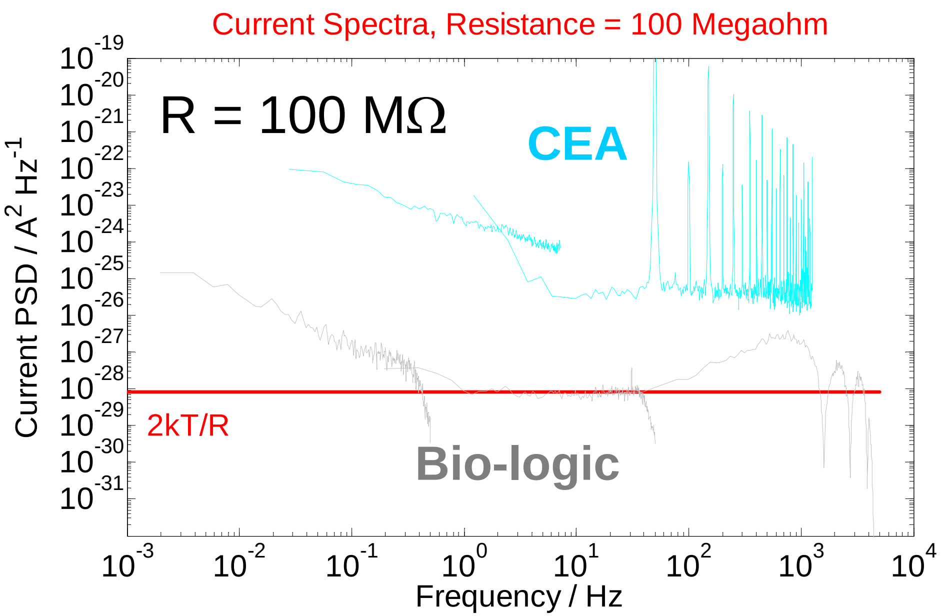


Bio-logic results for R = 1 MΩ and 100 MΩ (RR12 – 2017)

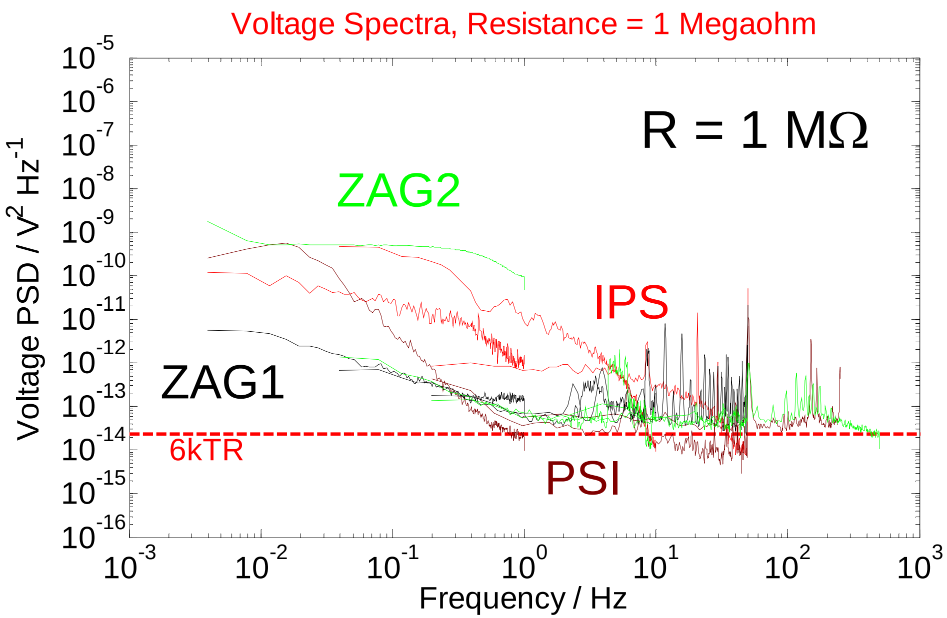


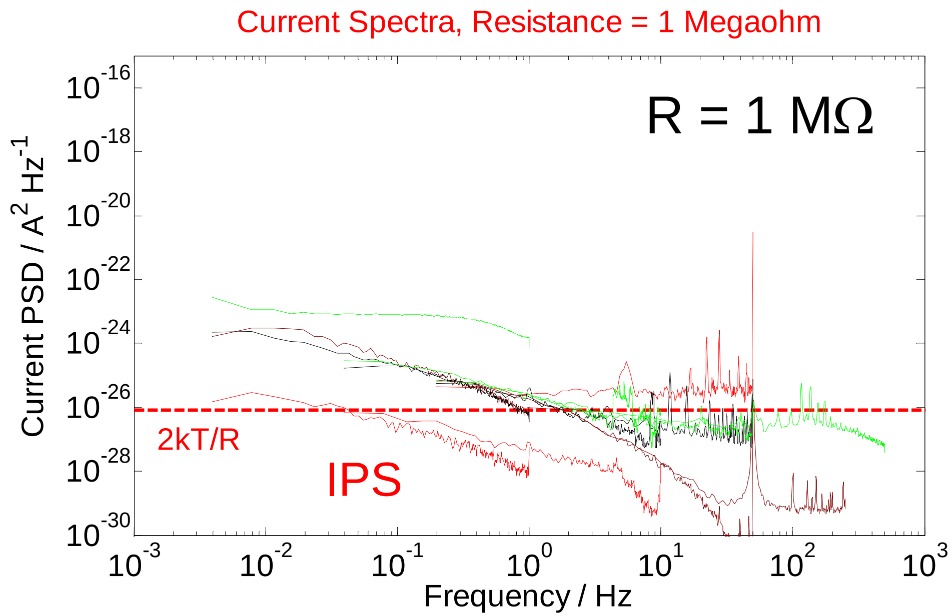


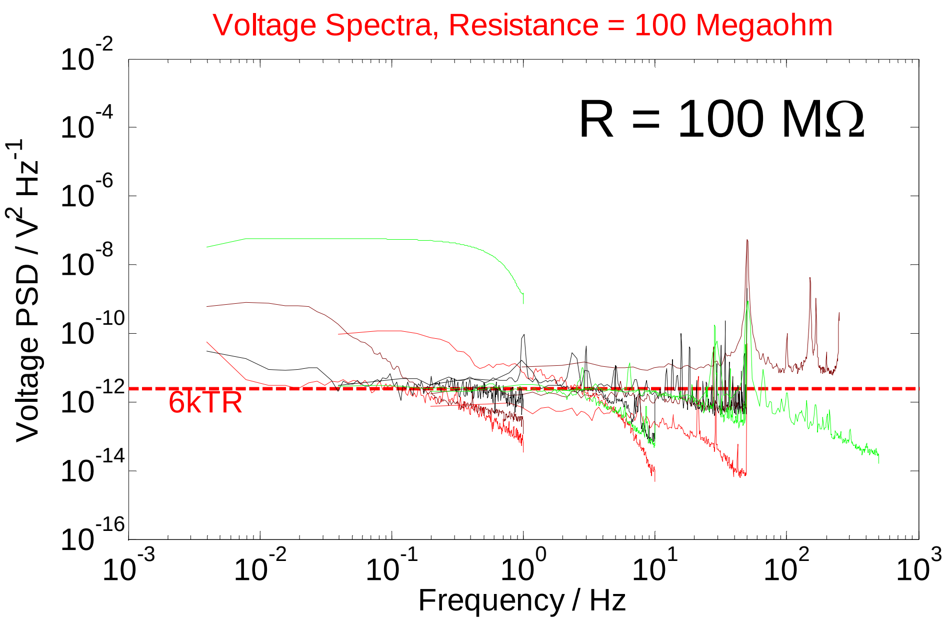


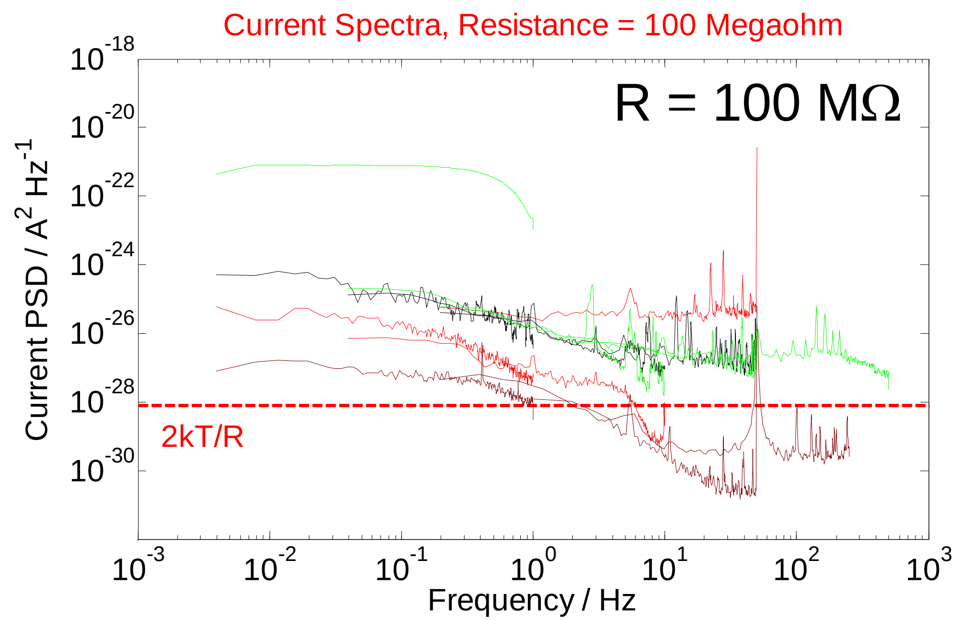


IPS results for R = 1 MΩ and 100 MΩ (RR11 – 2016)



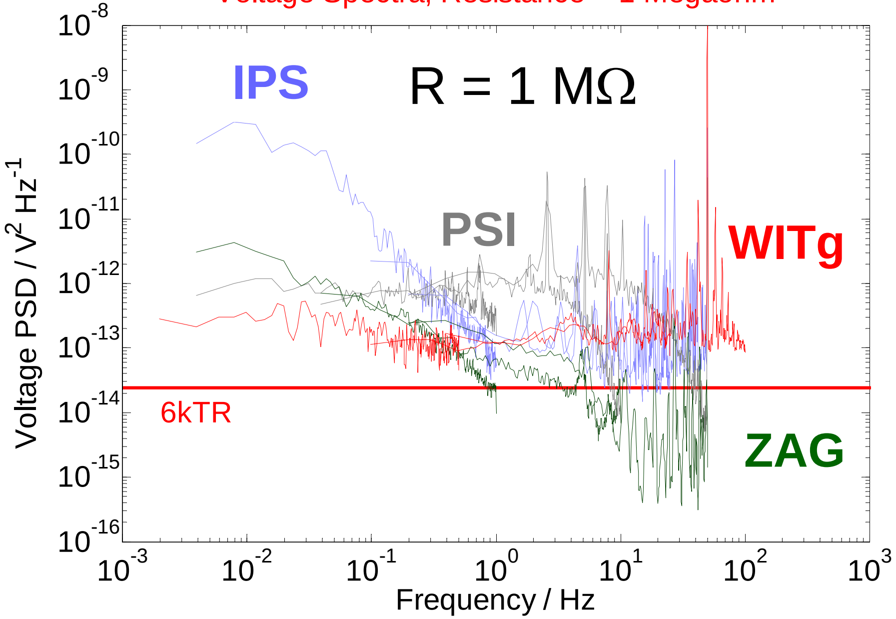




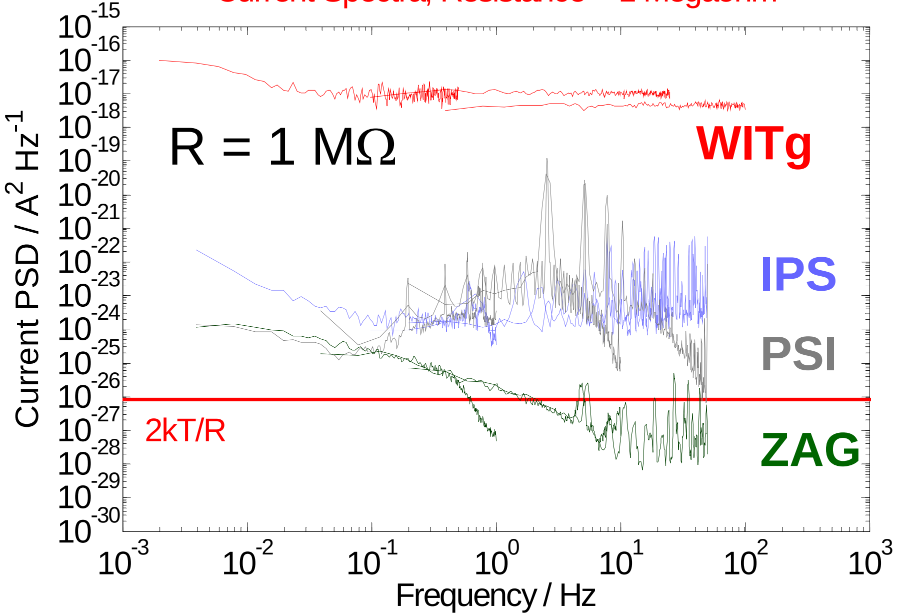


IPS results for R = 1 MΩ and 100 MΩ (RR12 – 2017)

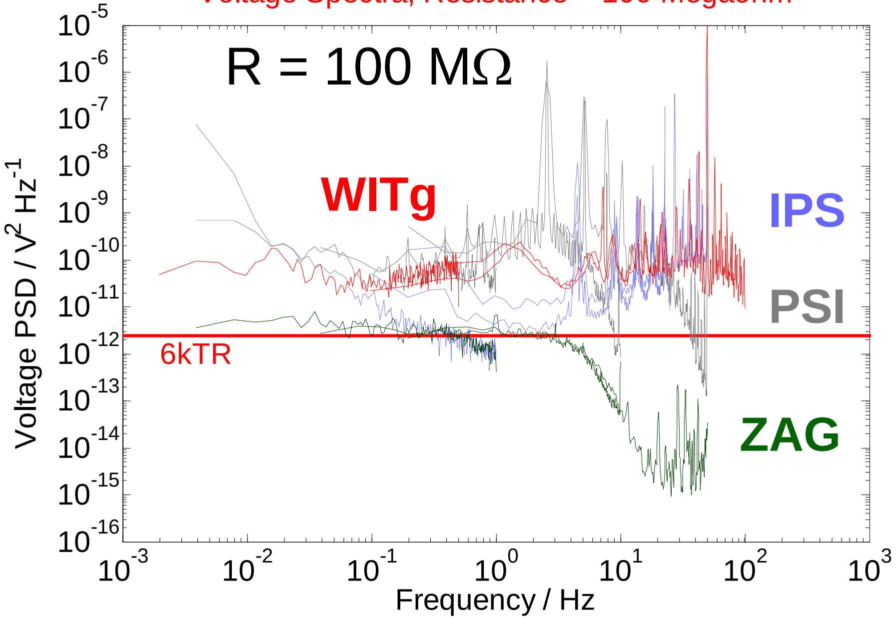
Voltage Spectra, Resistance = 1 Megaohm



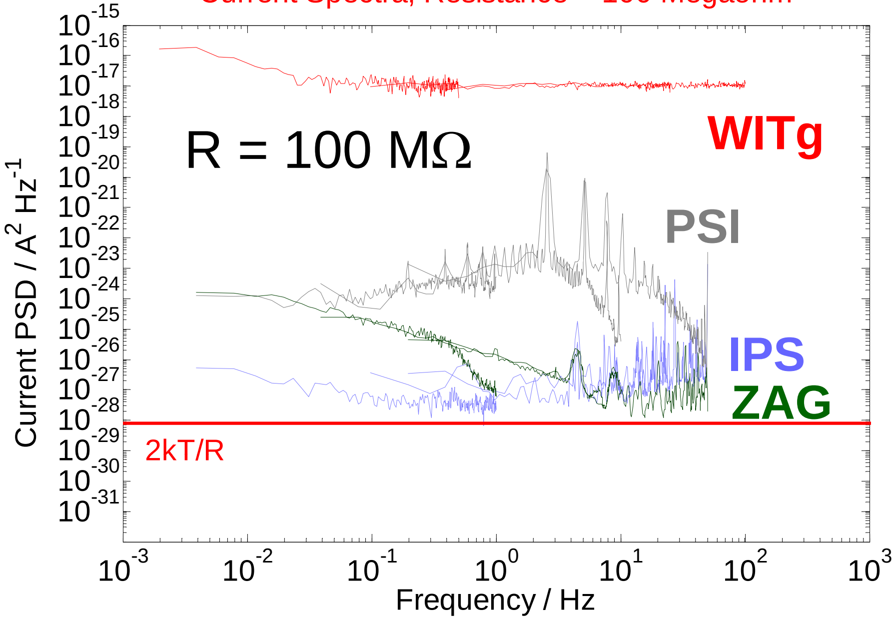
Current Spectra, Resistance = 1 Megaohm



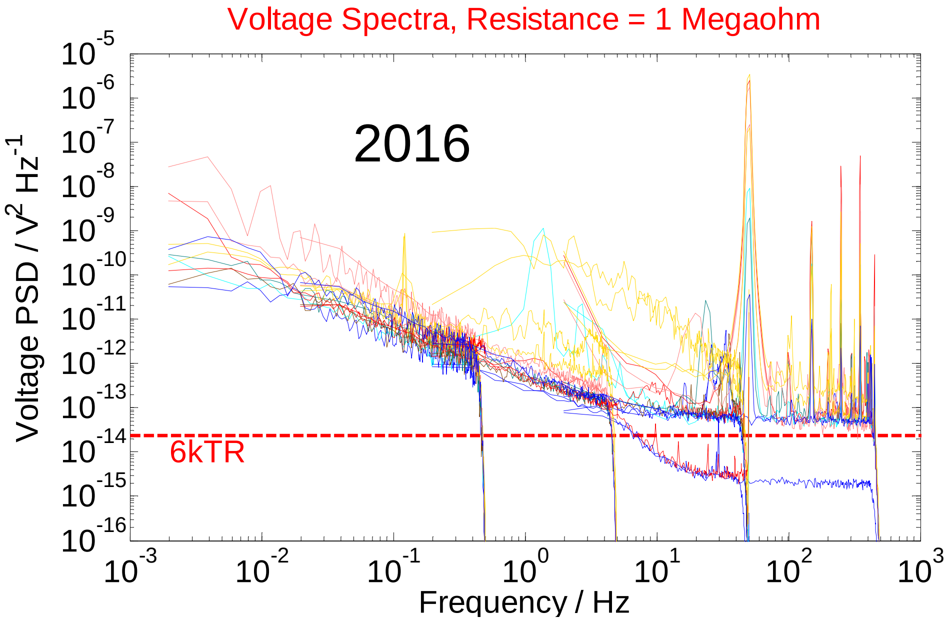
Voltage Spectra, Resistance = 100 Megaohm

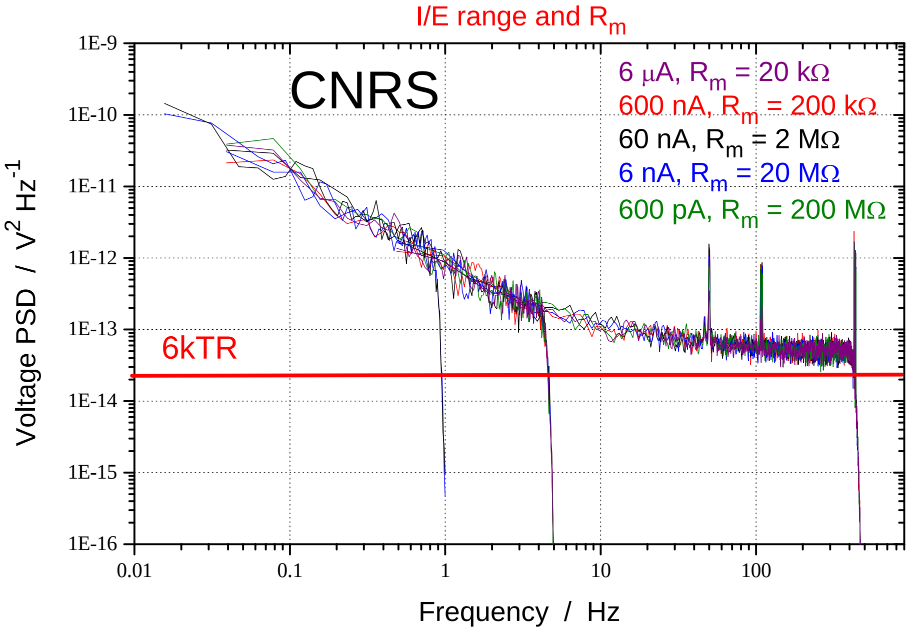


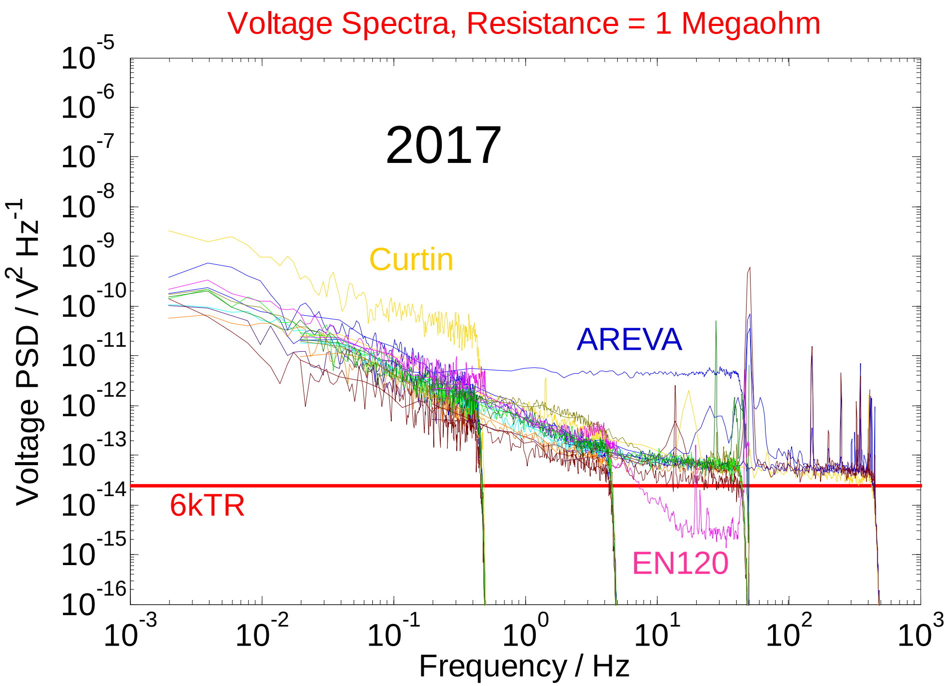
Current Spectra, Resistance = 100 Megaohm



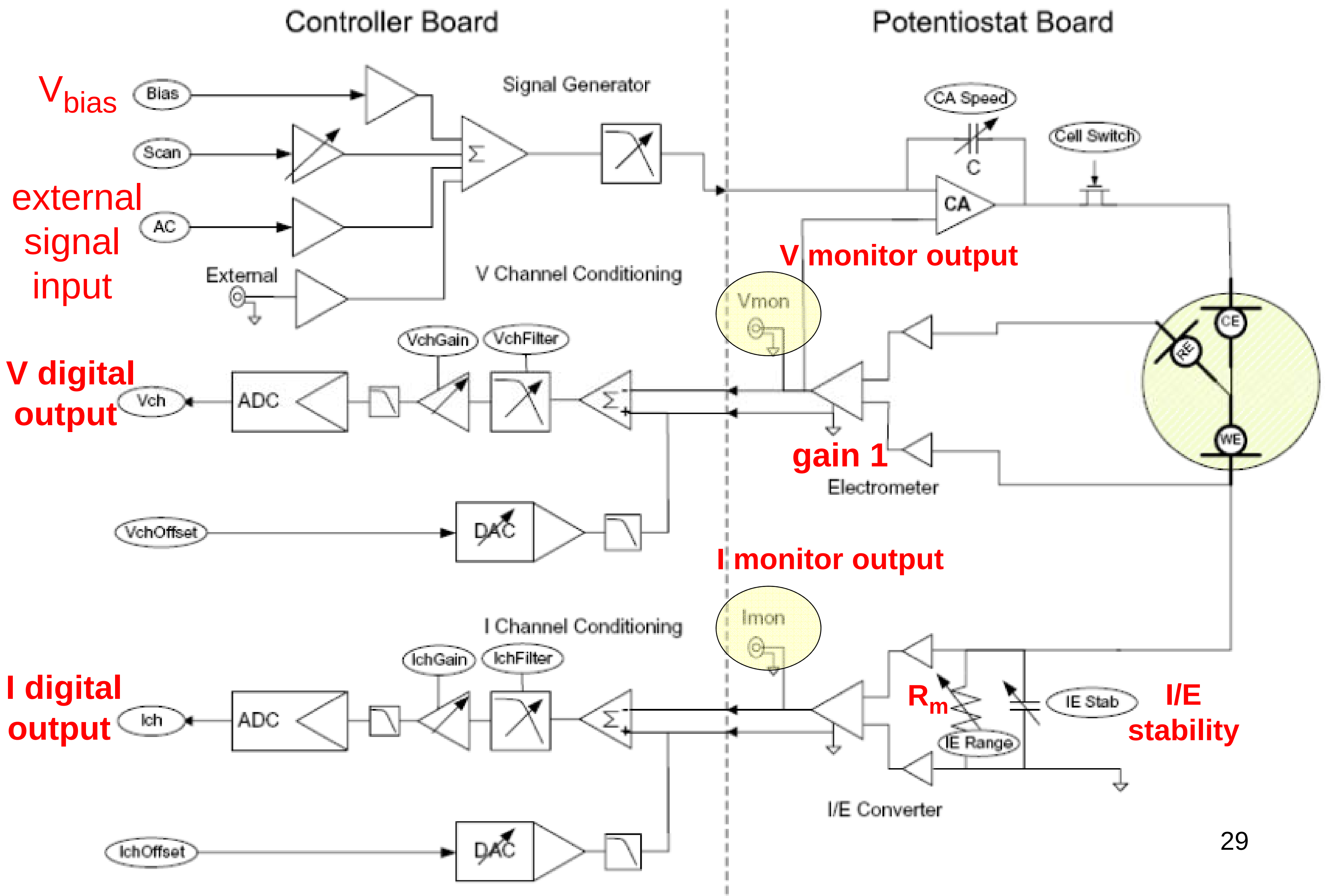
Gamry results for R = 1 MΩ: voltage



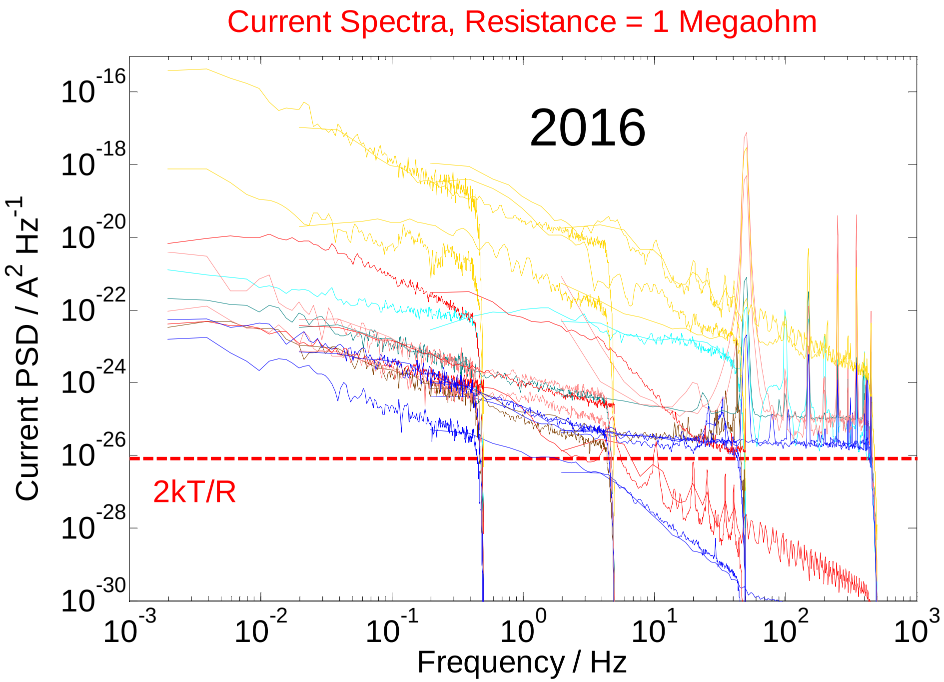


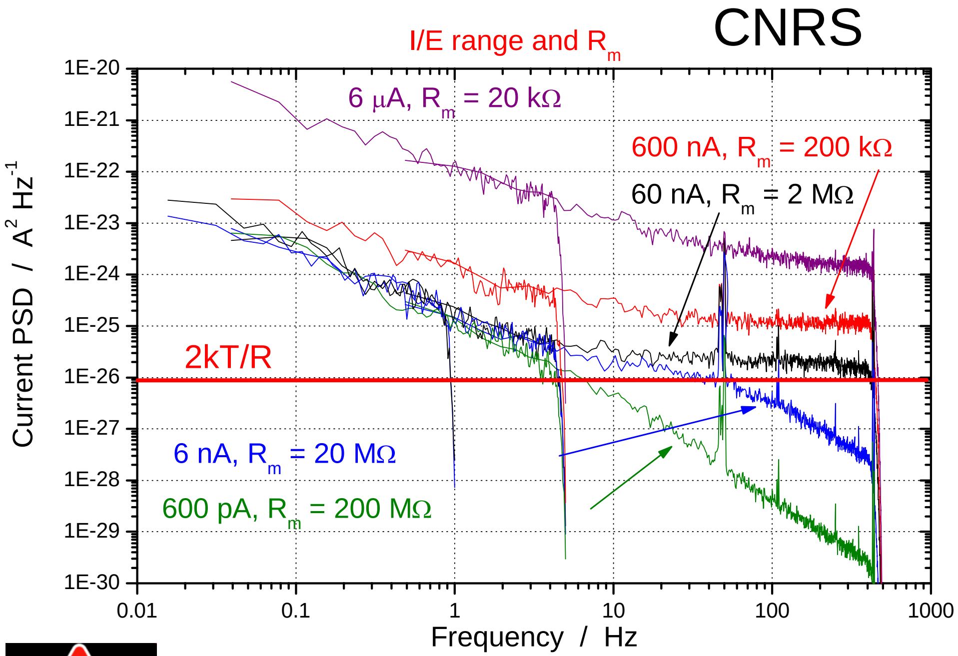


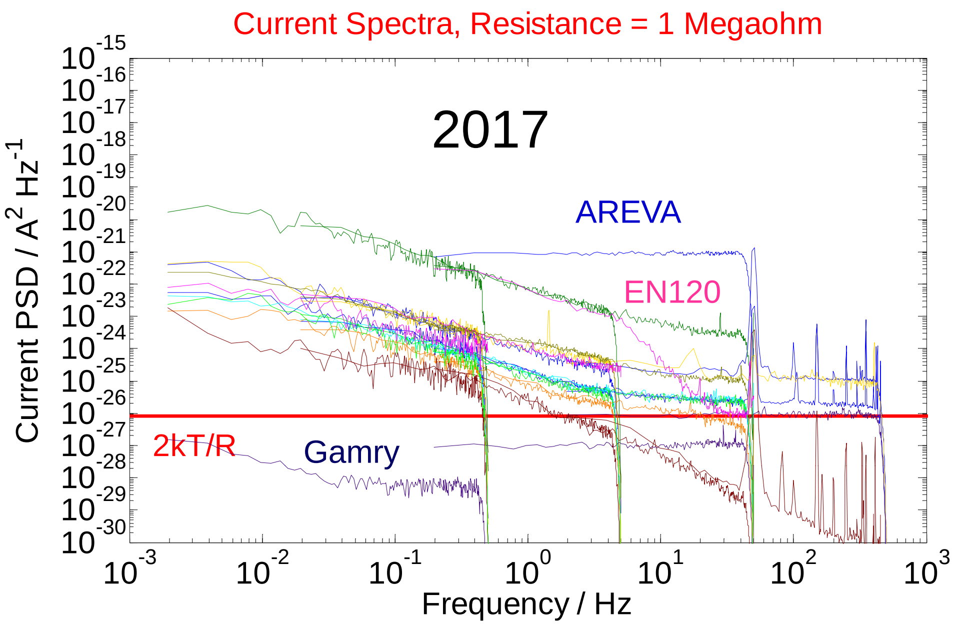
Ref 600 analog circuit



Gamry results for R = 1 MΩ : current







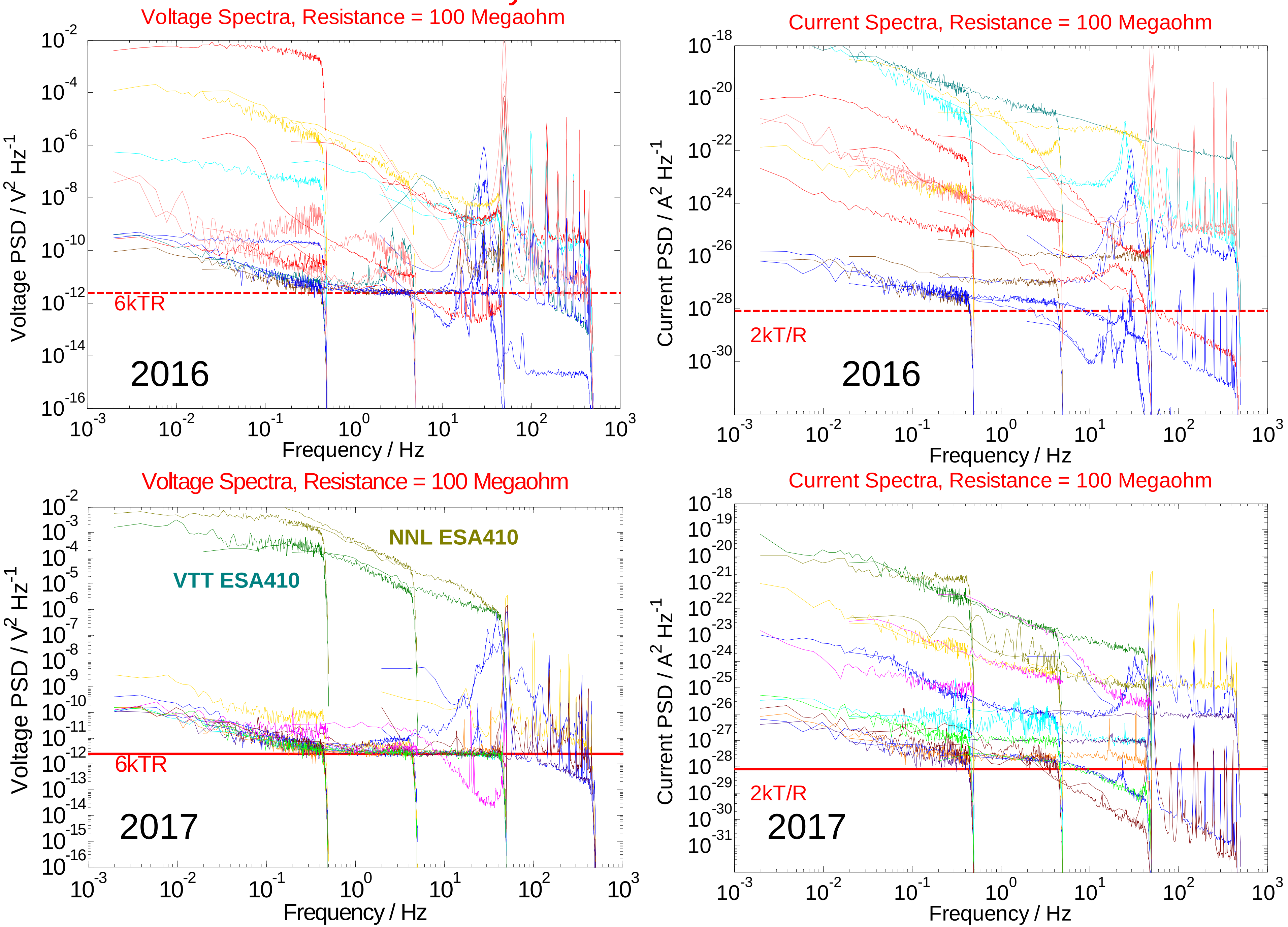
increasing the sensitivity (I/E range)

→ decrease the frequency bandwidth

For I/E stability fast: C = 150 pF

I/E range	R_m	f_c
6 µA	20 kΩ	50 kHz
600 nA	200 kΩ	5 kHz
60 nA	2 MΩ	500 Hz
6 nA	20 MΩ	50 Hz
600 pA	200 MΩ	5 Hz

Gamry results for R = 100 MΩ



Conclusions on RR #12

• new EN RR test on dummy cells with 24 data sets including 3 from manufacturers

• still large scatter in the PSDs, especially the current PSDs

• good PSD measurements (= good PSD overlaps) for only a few commercial equipments (Gamry with ESA-410, Bio-logic?, IPS?)

• even with good equipments, data provided by common users are often wrong: they do not set the right parameters in the setup for ENM

➡ what can be done? organisation of training course ?

• the manufacturers should provide:
- improved software (no access to filters, no conflicting choice of f_s and I/E range limiting the frequency bandwidth…)
- a tutorial for ENM with dummy cells

Procedure of EN measurement (Gamry Ref600 and ESA410)

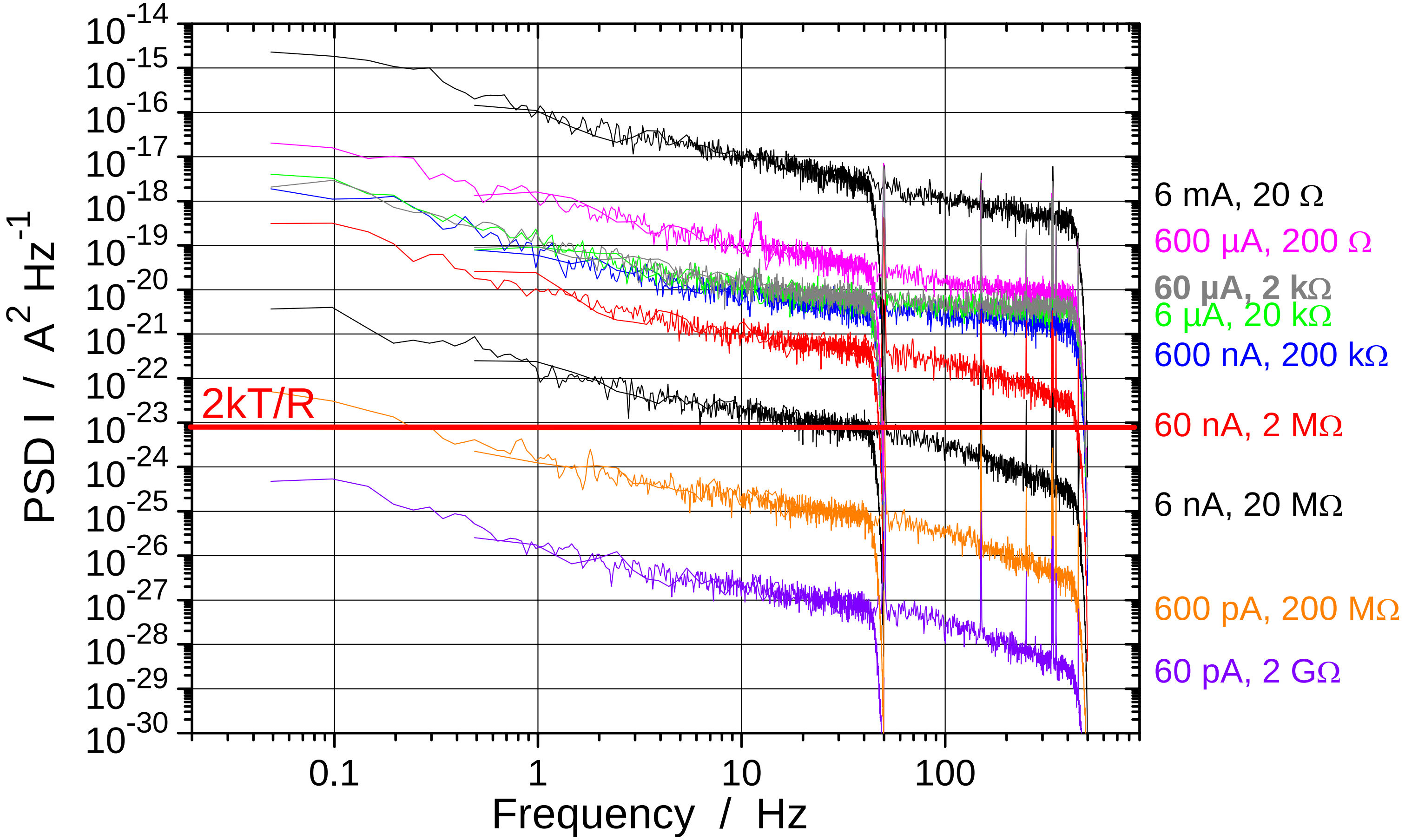
1. Connect all electrodes. Input file name, choose test mode, sampling frequency, test time.
2. Toggle cell on.
3. Set all IE range, Current channel, Voltage channel first to **auto mode**, let the software choose a measure range without overload. Overload may disappear after 10 to 20 seconds.
4. Set all IE range, Current channel, Voltage channel to **manual mode**. Try to decrease IE range down to get a minimum setting without overload. Once the overload indicator is triggered for more than 20 s, meaning a too-low current range for the measured value, the user should set the range one order up.
5. Decrease voltage channel and current channel manually down to get a minimum range without overload.
6. For the voltage fluctuations, evaluate the peak-to-peak amplitude on the screen during the measurements, and check this value is lower that the Vch range.
7. For the current fluctuations, evaluate the peak-to-peak amplitude on the screen during the measurements, multiply it with the gain in V/A of the I-E given below and check this value is lower that the Ich range.

I/E range	60 pA	600 pA	6 nA	60 nA	600 nA	6 µA	60 µA	600 µA	6 mA	60 mA	600 mA
R_m in Ω	2 G	200 M	20 M	2 M	200 k	20 k	2 k	200	20	2	0.2
G in V/A	5×10^{10}	5×10^{9}	5×10^{8}	5×10^{7}	5×10^{6}	5×10^{5}	5×10^{4}	5×10^{3}	500	50	5

8. If the EN is expected to increase during the experiment, it may be necessary to use higher I/E , voltage, and current ranges.
9. Start to record EN data.
10. Observe the overload indicators. Once the overload warning, the data may be unreliable.
11. When finish test, toggle cell off.

Dummy cell : R = 1 kΩ

ESA410, v6.04, mode Fast, Vch = 30 mV, Ich = 30 mV or 300 mV



How the lowest PSDs can be explained?

Thanks for your attention