A Low-Power MOS-Only Potentiostatic ΔΣ ADC Architecture for Electrochemical Sensors

J. Pallarès[1], S. Sutula[1], J. Gonzalo-Ruiz[2], F. X. Muñoz-Pascual[2], L. Terés[1,3] and F. Serra-Graells[1,3]
paco.serra@imb-cnm.csic.es

[1]Institut de Microelectrònica de Barcelona, IMB-CNM(CSIC)

[2]MATGAS A.I.E.

[3]Dept. of Microelectronics and Electronic Systems
Universitat Autònoma de Barcelona

Nov 2014

1 Introduction

2 Electrochemical ΔΣ Architecture

3 Low-Power MOS-Only Circuits

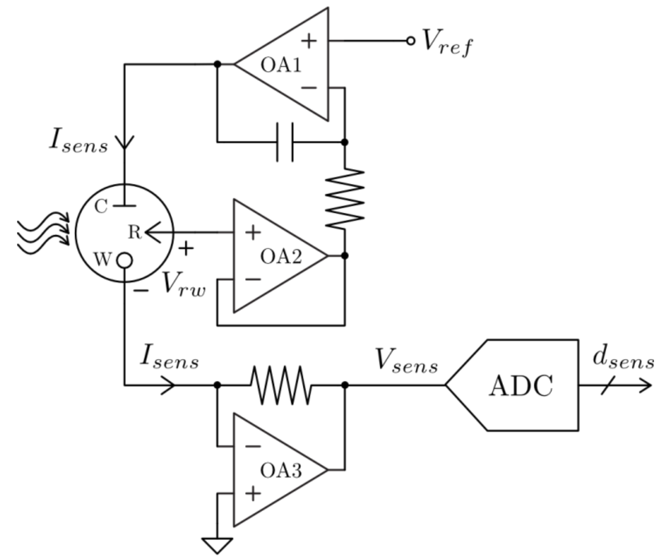
4 Low-Cost Monolithic Smart Sensor

5 Experimental Results

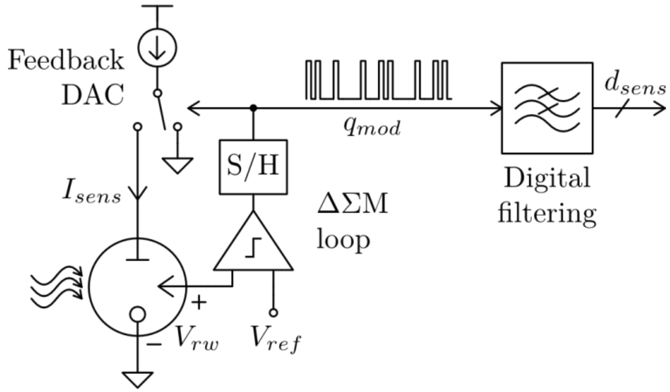
6 Conclusions

Motivation and Proposal

- **Integrated chemical** sensors
  - Interaction with microorganisms
  - Selectivity by functionalization
  - Speed limitation
  - Reduced life time
  - Packaging costs
- **Electrochemical** family
  - CMOS compatible
  - **Potentiostatic** biasing
  - **Amperometric** read-out



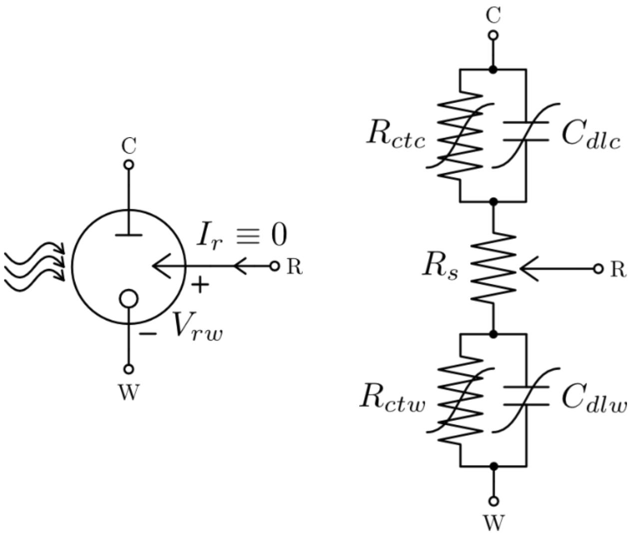
- **Classic** circuit interface require multiple OpAmps + resistors + ADC
- **Low-power MOS-only** circuit proposal based on mixed electronic and chemical domain potentiostatic ΔΣ modulator

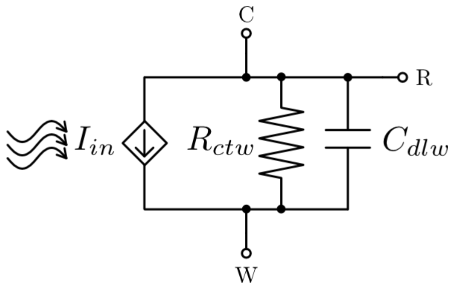


Sensor Modeling

- Reuse of **sensor dynamics** for circuit design needs accurate device modeling
- **<u>R</u>**eference, **<u>W</u>**orking and **<u>C</u>**ounter planar microelectrodes
- Non-linear electrical impedance model under **potentiostatic** operation:
  - R_s = electrolyte solution resistance
  - R_{ctx} = charge-transfer resistance
  - C_{dlx} = double-layer capacitance

- **Solution resistance** smaller than electrode-solution counterparts (10^2kΩ)
- Similar impedance results with internal (micro) or external (macro) **counter microelectrodes**
- Equivalent linear **dynamic model**





1 Introduction

2 Electrochemical ΔΣ Architecture

3 Low-Power MOS-Only Circuits

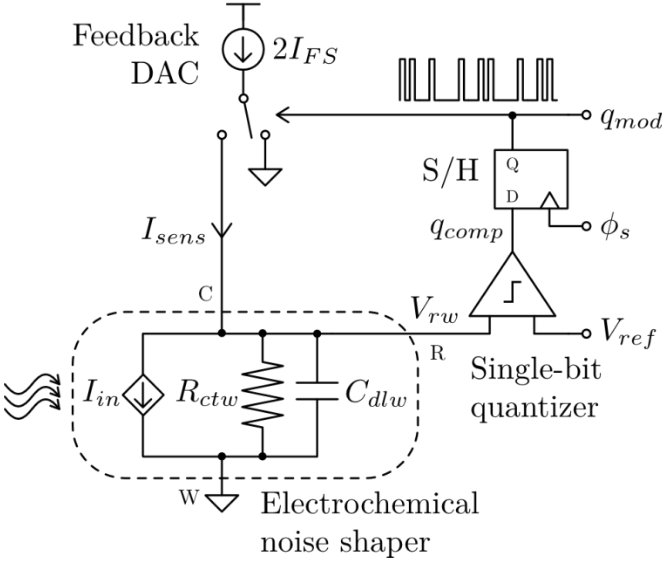
4 Low-Cost Monolithic Smart Sensor

5 Experimental Results

6 Conclusions

ΔΣ Modulator Architecture

- Behavior similar to **low-pass first-order single-bit CT** ΔΣ A/D modulator
- Error current is converted into voltage and shaped in frequency by the **electrochemical sensor** itself
- Quantization, S/H and DAC feedback in **electronic domain**
- **Amperometric** read-out through ΔΣ modulation of output bit stream q_{mod} by chemical input I_{in}
- Overall negative feedback ensures **potentiostatic** operation by keeping V_{rw} biased close to V_{ref} potential
- High oversampling ratios (**OSR>100**) can be easily obtained with kHz-range clock frequencies f_s



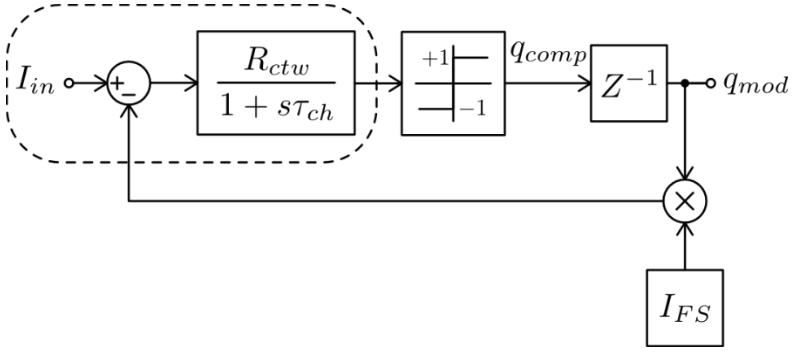
Class-A **full scale**: $I_{FS} = \frac{V_{ref}}{R_{ctw}}$

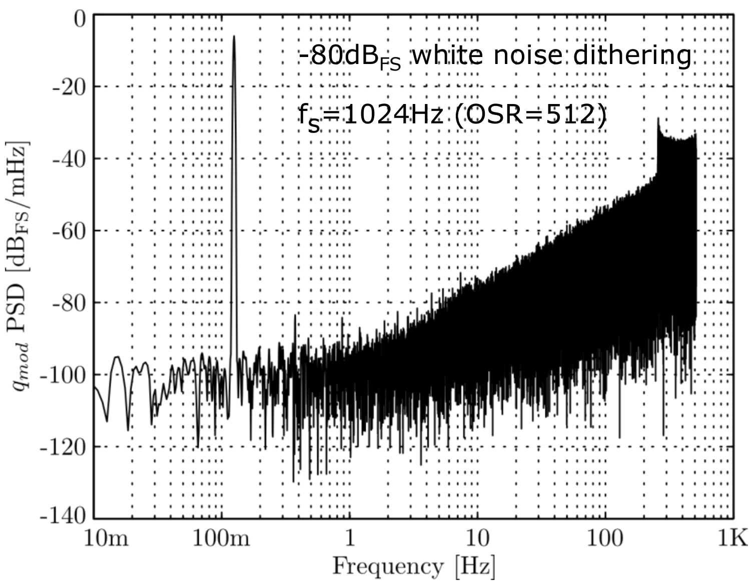
Electrochemical **time constant**:

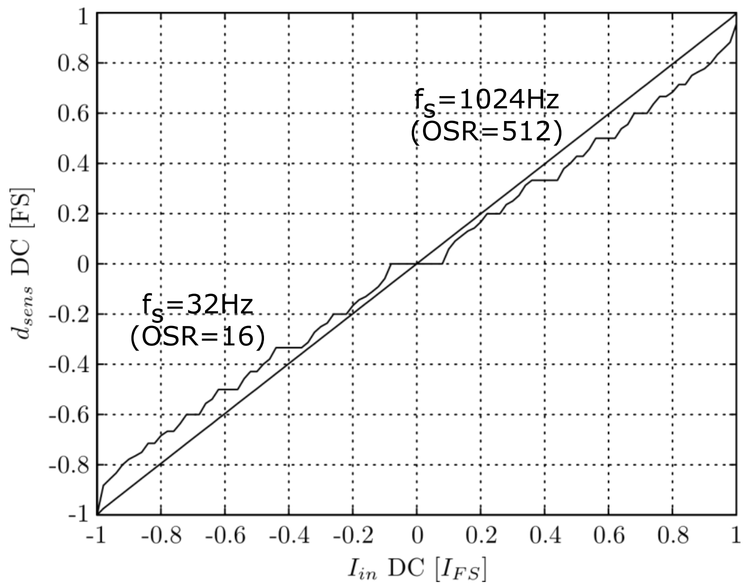
$$\tau_{ch} = R_{ctw} C_{dlw} \sim 10^{-1} s$$

ΔΣ Modulator Optimization

- Typical **tonal components** of first-order ΔΣ modulation are attenuated through **thermal noise dithering** at DAC
- Fractal **staircase** DC transfer function due to DT losses of CT electrochemical integrator is improved by **increasing OSR**







E.g. ΔΣM behavioral simulation for R_{ctw}=500kΩ, τ_{ch}=0.16s, V_{ref}=1V and I_{FS}=2μA

1 Introduction

2 Electrochemical ΔΣ Architecture

3 Low-Power MOS-Only Circuits

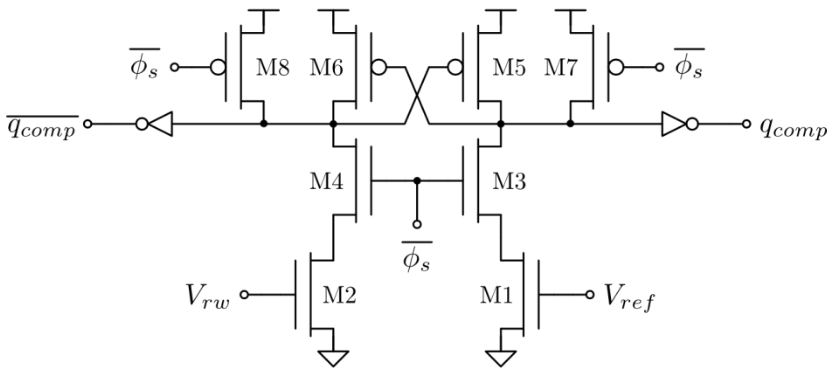
4 Low-Cost Monolithic Smart Sensor

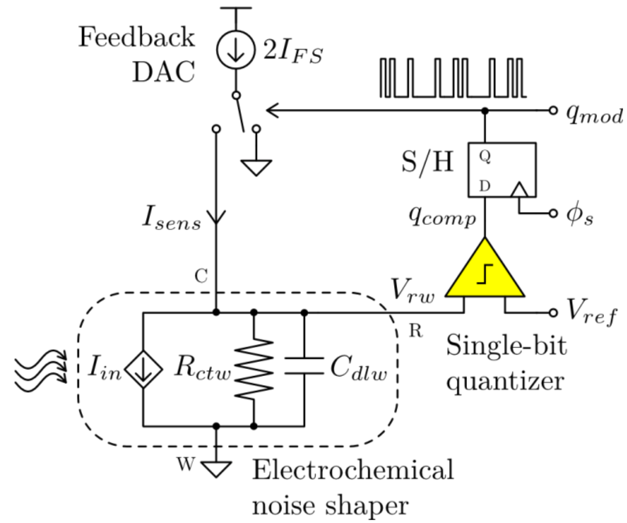
5 Experimental Results

6 Conclusions

Low-Power MOS-Only Circuits

- Two **analog blocks** only
- **Latched comparator** for 1-bit quantization

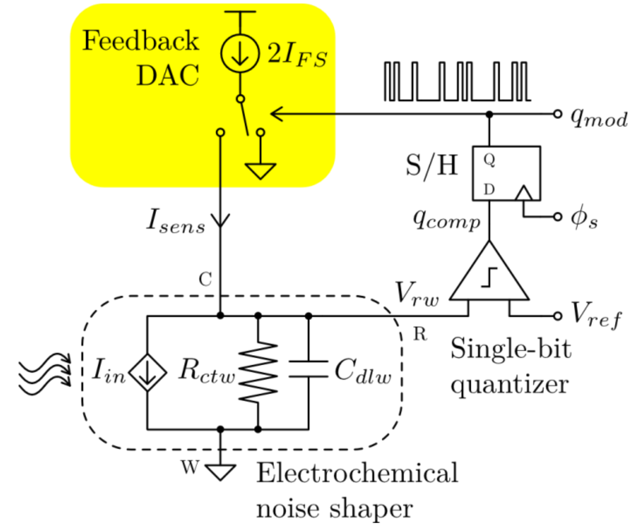


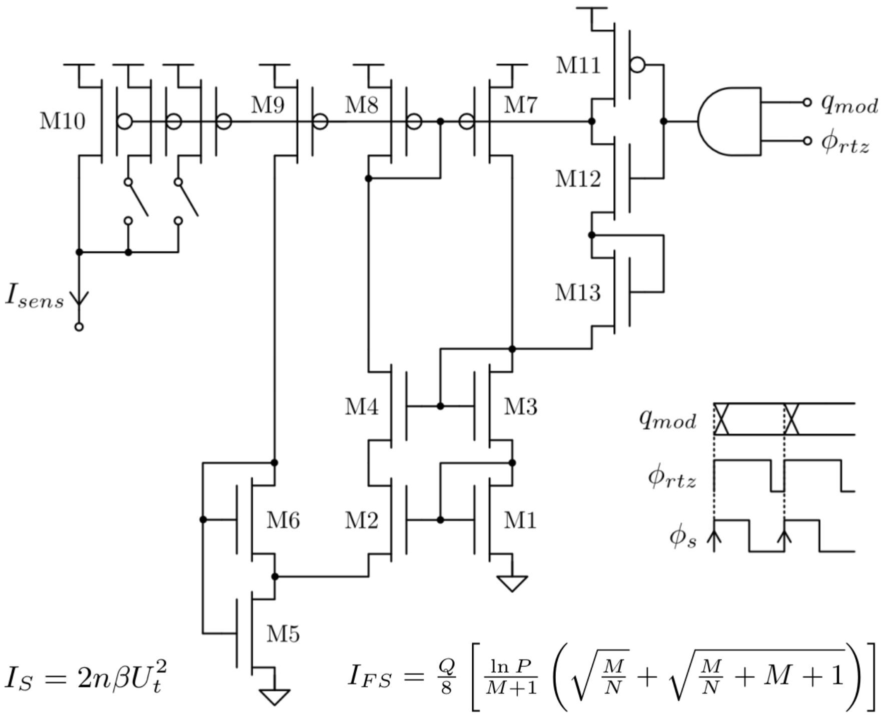


- **Technology mismatching** does not cause distortion but DC offset at V_{rw}
- If V_{ref} is chosen higher than redox potential, electrochemical signals can tolerate comparator **offsets** as large as ±10mV

Low-Power MOS-Only Circuits

- Two **analog blocks** only
  - **Latched comparator** for 1-bit quantization
  - **Compact reference** generator for current DAC





$$I_S = 2n\beta U_t^2 \qquad I_{FS} = \frac{Q}{8}\left[\frac{\ln P}{M+1}\left(\sqrt{\frac{M}{N}} + \sqrt{\frac{M}{N} + M + 1}\right)\right]^2 \left(\frac{W}{L}\right)_5 I_S$$

$$Q = \frac{(W/L)_{10}}{(W/L)_8} \quad M = \frac{(W/L)_9}{(W/L)_8} \quad N = \frac{(W/L)_6}{(W/L)_5} \quad P = \frac{(W/L)_2}{(W/L)_1}\frac{(W/L)_7}{(W/L)_8}$$

- **Circuit core** based on previous work from these authors **[6]**
- **Programmable full scale** for different integrated sensor designs
- **Power on/off** instead of current steering operation to reset all MOSFETs and reduce **flicker noise**
- **RTZ** signaling in order to avoid typical waveform asymmetries issues of CT ΔΣMs

1 Introduction

2 Electrochemical ΔΣ Architecture

3 Low-Power MOS-Only Circuits

4 Low-Cost Monolithic Smart Sensor

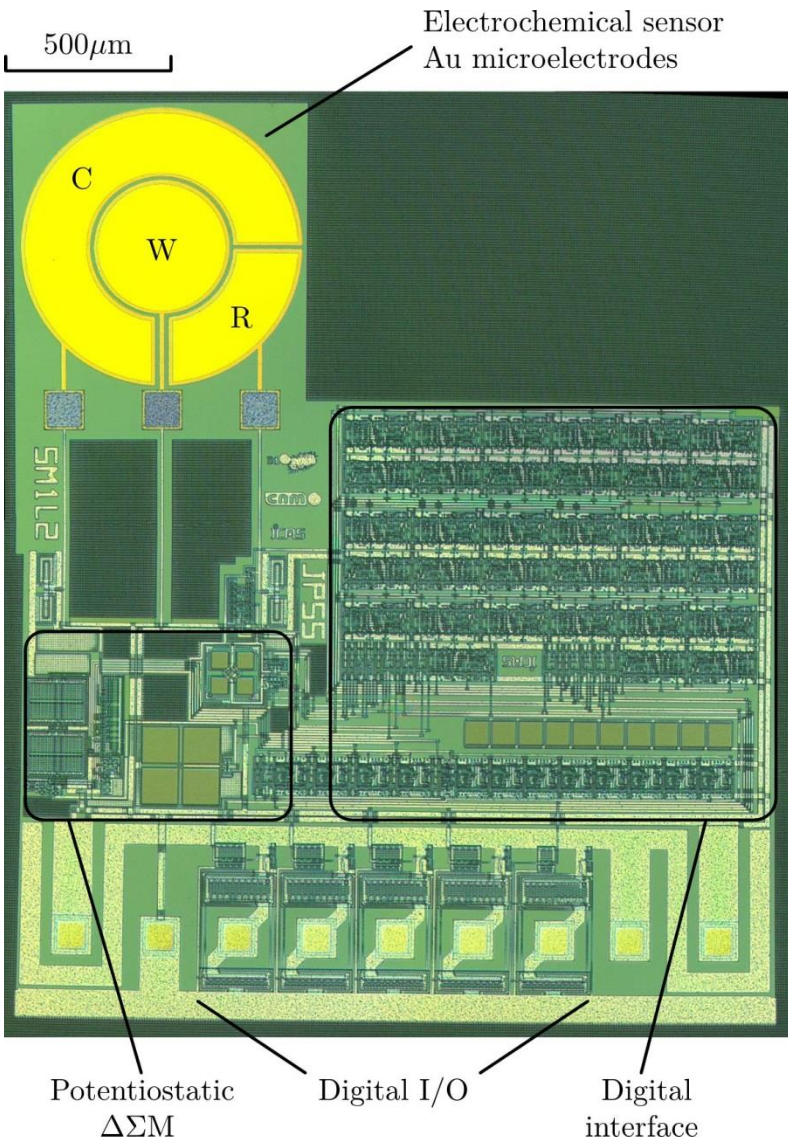
5 Experimental Results

6 Conclusions

Monolithic CMOS Integration

- Inexpensive 2.5μm 1M CMOS technology (**CNM25**)
- In-house **sensor post-processing** at wafer level consisting on sputtering of Ti(15nm)+Au(150nm) thin films and lithographic patterning by lift-off
- **Sensor** layout design: D_{in}=390μm, D_{out}=830μm and S=30μm
- **Sensor** electrical model: R_{ctw}=500kΩ and τ_{ch}=0.16s
- **ΔΣM** design parameters: V_{ref}=1V, I_{FS}=2μA and f_s=1024Hz (OSR=512)

- **Low area** overhead of proposed ΔΣM
- **Digital only interface** for low-pass filtering + V_{ref} and I_{FS} programming



2.3mm x 2.8mm (6.4mm²)

1 Introduction

2 Electrochemical ΔΣ Architecture

3 Low-Power MOS-Only Circuits

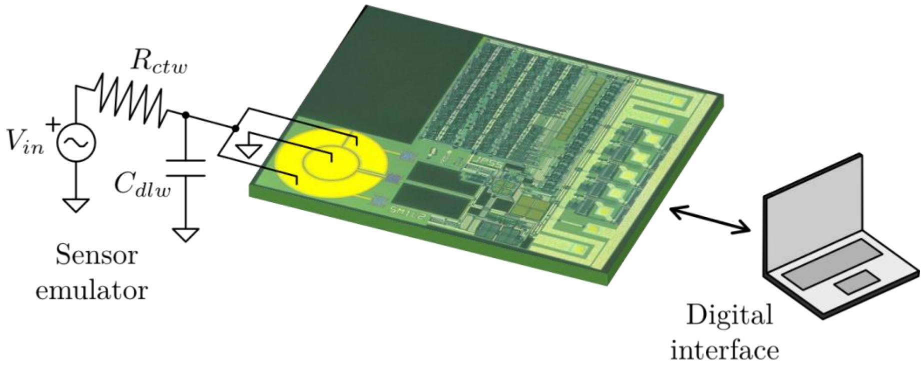
4 Low-Cost Monolithic Smart Sensor

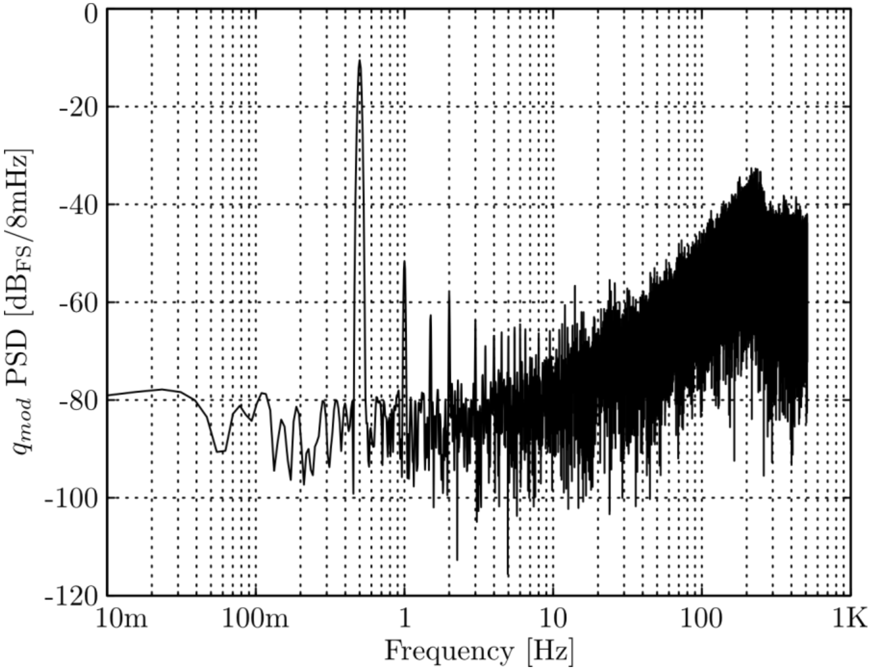
5 Experimental Results

6 Conclusions

Electrical Tests

- **Sensor emulation** with external network (R_{ctw}=510kΩ, C_{dlw}=330nF) and SRS DS360 generator in equivalent Thévenin configuration
- Although dithering noise at DAC should be increased, PSD returns good **robustness** against tones

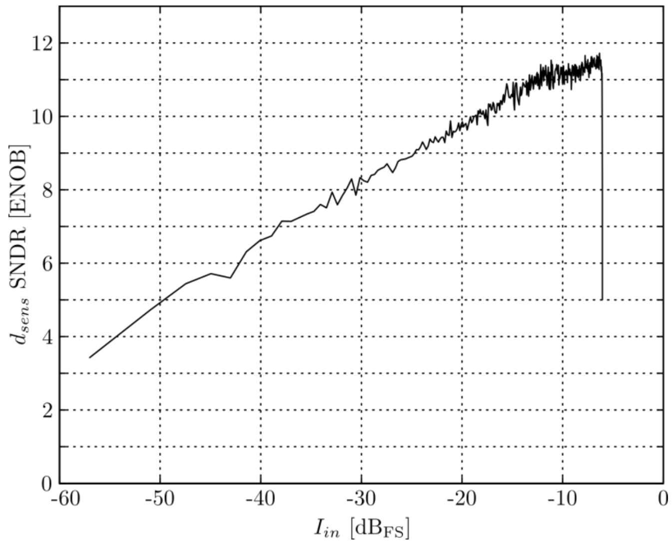


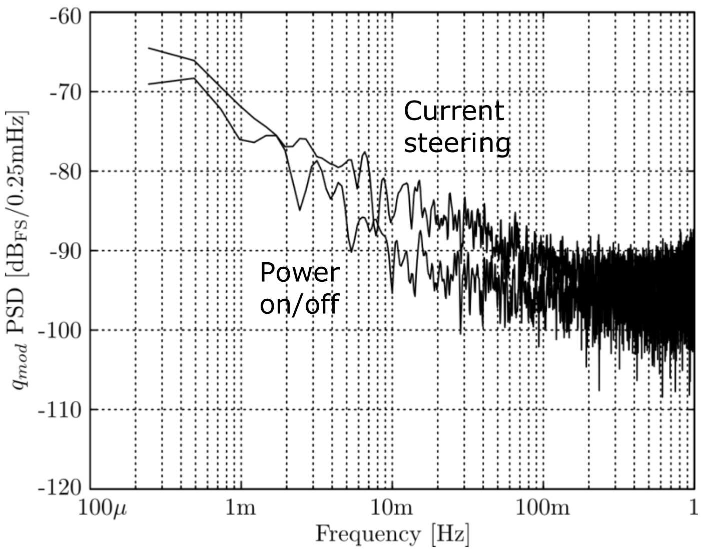


Electrical Tests

- **Sensor emulation** with external network (R_{ctw}=510kΩ, C_{dlw}=330nF) and SRS DS360 generator in equivalent Thévenin configuration
- Although dithering noise at DAC should be increased, PSD returns good **robustness** against tones
- Quasi-static response shows high enough **SNDR** to not limit electrochemical sensor resolution
- **Statistical analysis** on 9 samples returns DR deviations below ±0.5bit
- Experimental comparison between **power-on/off** and current steering DAC operation points to 3dB flicker noise reduction

2Hz noise bandwidth (OSR=256)



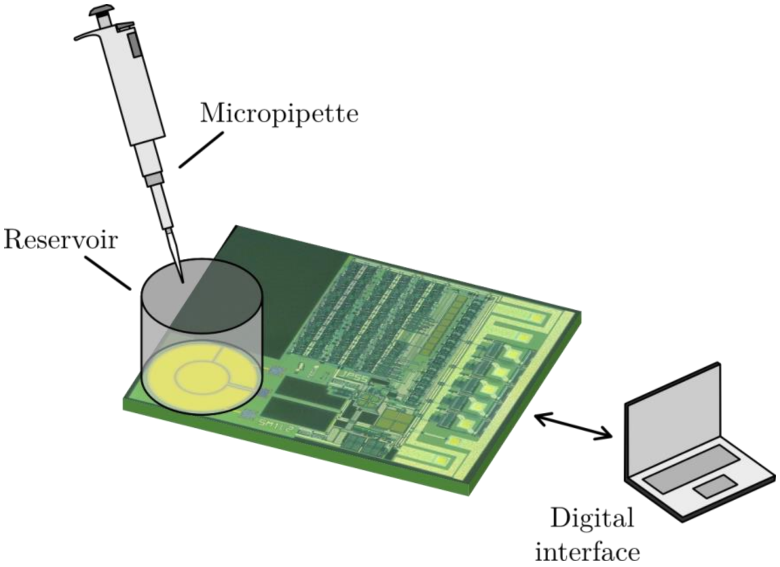


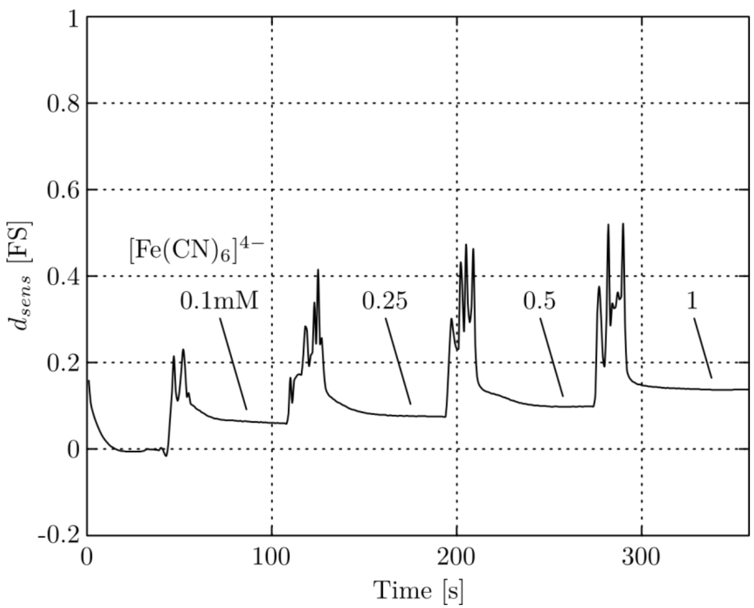
Electrochemical Tests

- **Standard experiment** based on Ferrocyanide ion oxidation into Ferricyanide

$$[\mathrm{Fe(CN)_6}]^{4-} \rightarrow [\mathrm{Fe(CN)_6}]^{3-} + 1\mathrm{e}^{-}$$

- **10μL reservoir** with Ferrocyanide dissolved in Phosphate buffer solution (PBS) at pH=7
- **Ion concentration** swept from 0.1mM to 1mM and **potentiostatic** V_{ref}=0.7V
- Electrochemical **time constant** as expected



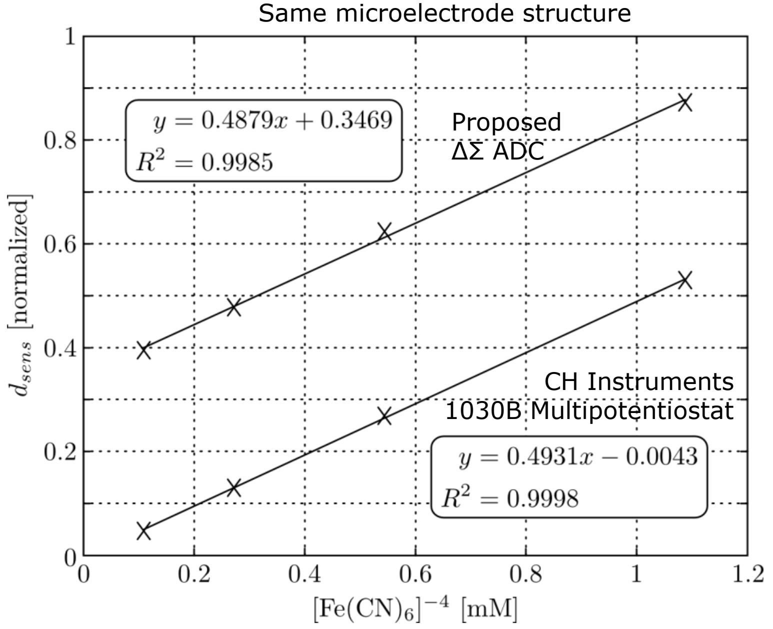


Electrochemical Tests

- **Standard experiment** based on Ferrocyanide ion oxidation into Ferricyanide

$$[\mathrm{Fe(CN)_6}]^{4-} \rightarrow [\mathrm{Fe(CN)_6}]^{3-} + 1\mathrm{e}^-$$

- **10μL reservoir** with Ferrocyanide dissolved in Phosphate buffer solution (PBS) at pH=7
- **Ion concentration** swept from 0.1mM to 1mM and **potentiostatic** V_{ref}=0.7V
- Electrochemical **time constant** as expected
- Remarkable **linearity** below 1mM
- Comparable to lab **desktop equipment**
- Good **performance** for sensing applications
- Very **low-power** operation compared to sensor consumption itself (can improve with low-voltage CMOS tech)



Parameter	Value	Units
Full scale range	2 to 32	μA
Potential range	0 to 5	V
Sampling frequency	1	kHz
Oversampling ratio	$\geq$256	
Electrical dynamic range	>10	ENOB
Residual standard deviation (n=6)	<15	%
Coefficient of determination (R^2)	0.9985	
Supply voltage	5	V
Power consumption at $2\mu A_{FS}$	25	μW
Die size	2.3×2.8	mm^2

1 Introduction

2 Electrochemical ΔΣ Architecture

3 Low-Power MOS-Only Circuits

4 Low-Cost Monolithic Smart Sensor

5 Experimental Results

6 Conclusions

Conclusions

- **ΔΣ ADC** architecture for potentiostatic biasing and amperometric reading of integrated **electrochemical sensors**
- ▲ Mixed **electro-chemical CT ΔΣ modulator** exploits dynamic behavior of sensors for analog minimalist implementation
- ▼ Accurate sensor **modeling** is needed
- ▲ **Low-power MOS-only** circuits for electronic part of ΔΣ ADC
- ▲ **25µW** complete smart sensor in **low-cost** 1M CMOS technology
- ▲ Electrical dynamic range exceeding **10bit** and electrochemical linearity close to **R^2=0.999** and **RSD<15%**
- ▲ Comparable in **performance** to commercial desktop equipment

Thanks for your attention!