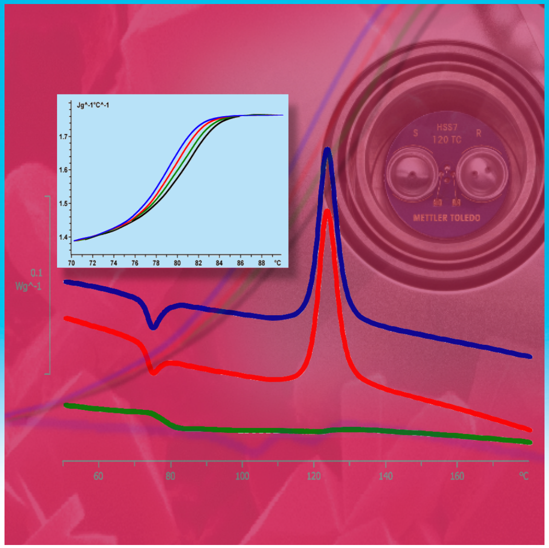
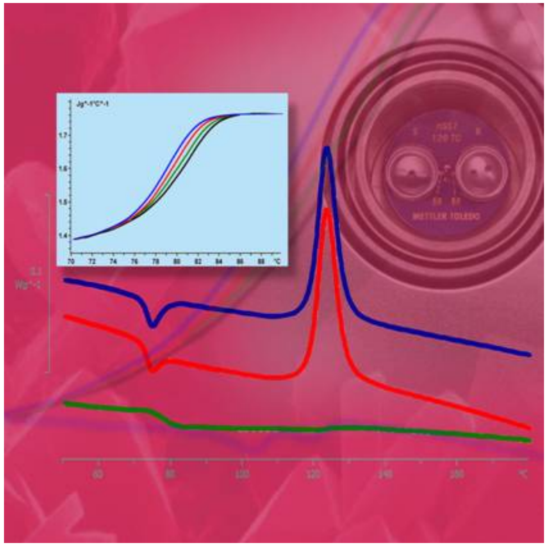
TOPEM® - Measuring program and evaluation parameters



J.E.K. Schawe

METTLER TOLEDO



Introduction

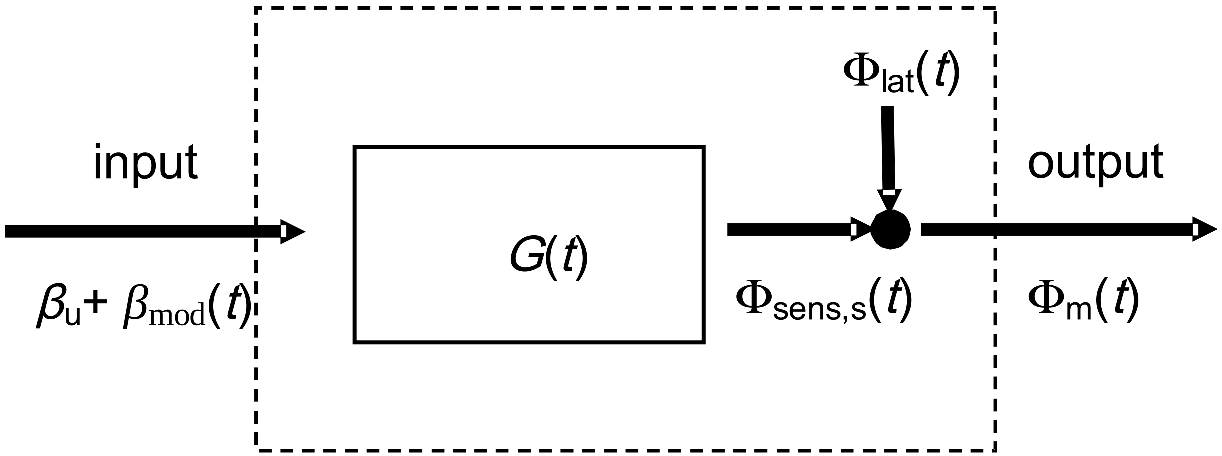
Temperature program

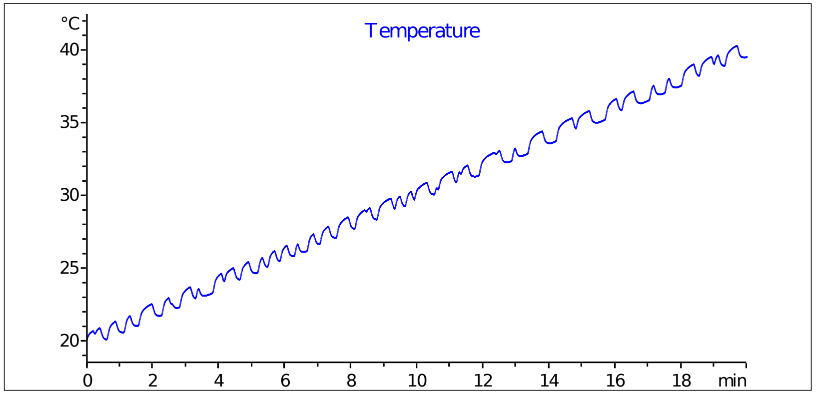
Evaluation parameters

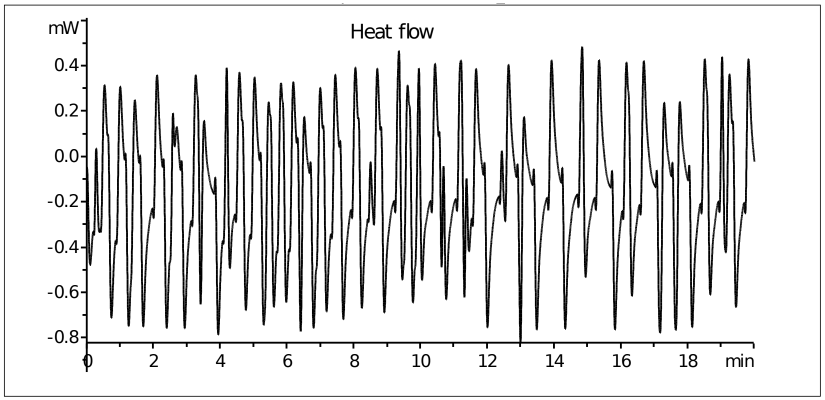
Conclusion

METTLER TOLEDO

TOPEM® Principle







characterization of the system by a test-signal with a wide frequency range

test-signal: **stochastic temperature** perturbation $\delta T(t)$

METTLER TOLEDO

TOPEM® Principle

TOPEM®

Step 1 — Measured curves, T(t) and ϕ (t)

↓

Step 2 — TOPEM evaluation (system charcterization)

↓

Step 3 — Quasi static curves

c_{p0}, ϕ_{total}, $\phi_{reversing}$, $\phi_{non\text{-}reversing}$

↓

Step 4 — Frequencies →

↓

Frequency dependent curves

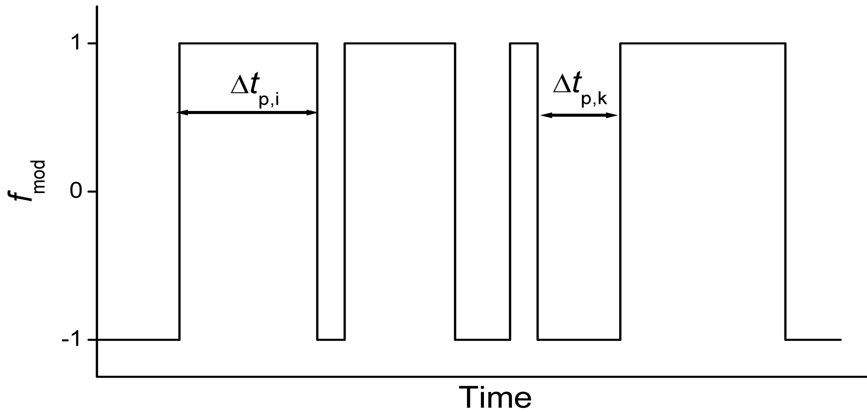
$c_p^*(\omega)$ (c_p', c_p'', φ)

METTLER TOLEDO

TOPEM®: measuring program

TOPEM®

Modulation function (switching times)



FRS5: $\Delta\ t_{min}$=15 s; $\Delta\ t_{max}$=30 s (default)

HSS7: $\Delta\ t_{min}$=30 s; $\Delta\ t_{max}$=60 s

low frequency measurements: larger $\Delta\ t_{max}$ ($\Delta\ t_{max}$=500 s); heating rate

minimum switching time: high frequencies -> instrument behavior

maximum switching time: low frequencies -> sample behavior

$\Delta\ t_{min}$ to small --> less sample information (noisy results)

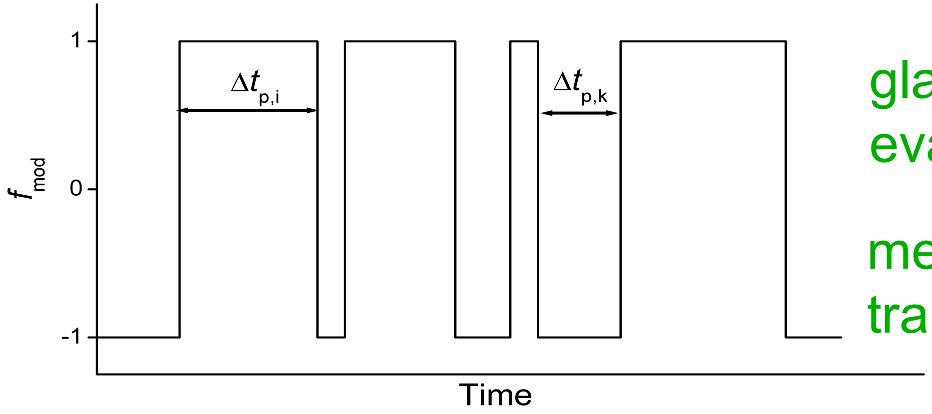
$\Delta\ t_{max}$ to long --> no switch in an evaluation window (noisy results)

METTLER TOLEDO

TOPEM®: measuring program

TOPEM®

Modulation function (pulse height)



glass transition, crystallization, evaporation, etc: ± 0.5 K (default)

melting, 1st or 2nd order phase transition: ± 0.05 < ± 0.001 K

The puls hight is determined by the thermal behavior of the sample (linearity)

puls height to low --> noisy results

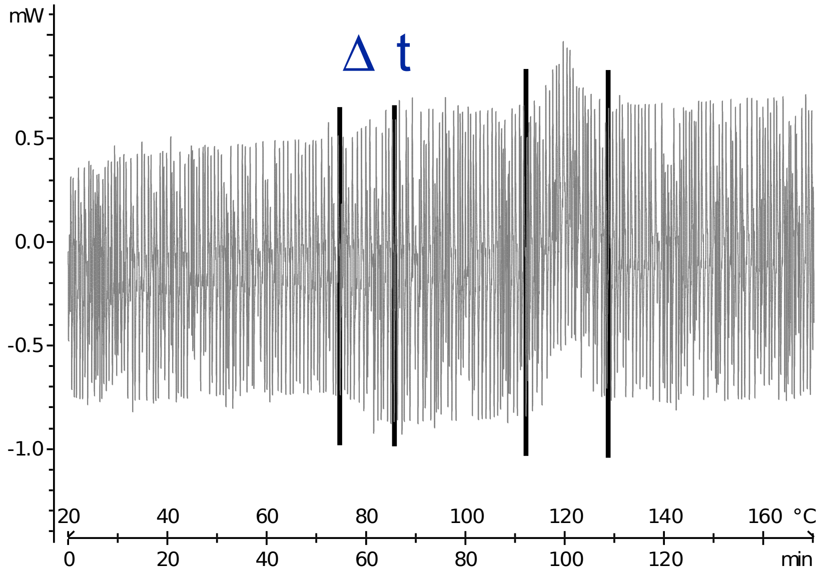
puls height to large --> non linear conditions (possibly wrong results)

METTLER TOLEDO

TOPEM®: measuring program

TOPEM®

The maximum heating rate depends on the minimum width of the calculation window



Windows width ≤ Δ t / 3

Example:

calculation window: 120 s

⇓

minimum duration of the thermal event: 6 min

wdith of the thermal event: 10 K

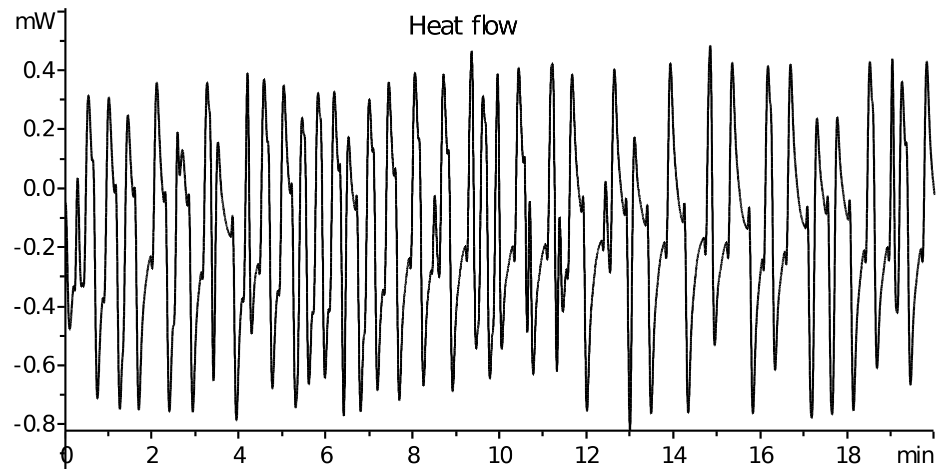
⇓

maximum heating rate:
10 K / 6 min = 1.7 K/min

METTLER TOLEDO

TOPEM®: data evaluation

Important parameters of the TOPEM evaluation:



Width of calculation window Δt_{calc}

Shift of calculation window Δt_s

Width of smoothing window Δt_{smooth}

Sample response parameter n_a

Instrument response parameter n_b

TOPEM®: data evaluation

TOPEM®

Sample response parameter n_a

Instrument response parameter n_b

Impuls response function

$n_b \approx t_m / 0.1$ s (sampling interval)

$n_a \geq 2$

n_a characterize the relaxation of the impulse response function.

Roule of thumb:

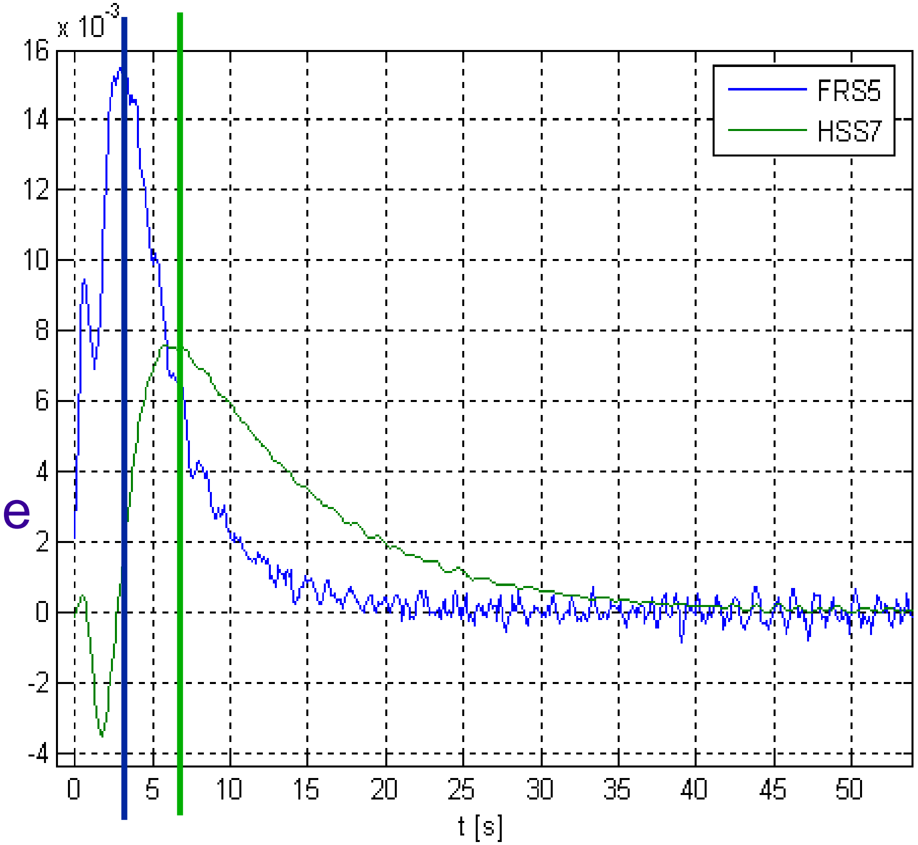
large n_a --> large frequency range

default: $n_a = 20$, $n_b = 45$

(FRS5, Al-crucible)

HSS7(Al-crucible):

$n_b = 60$



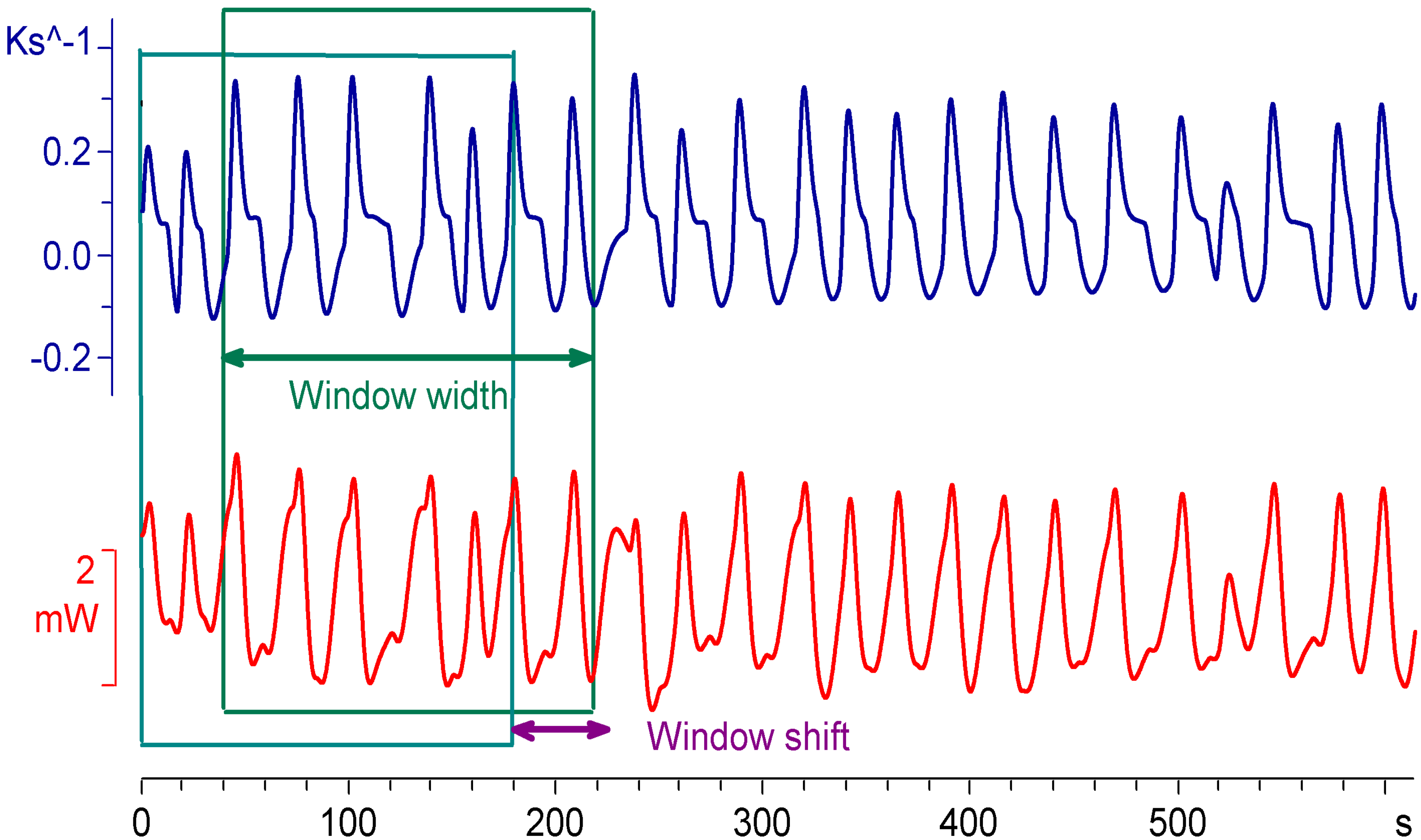
t_m = 4.5 s 6.0 s

METTLER TOLEDO

TOPEM®: data evaluation

TOPEM®

calculation window



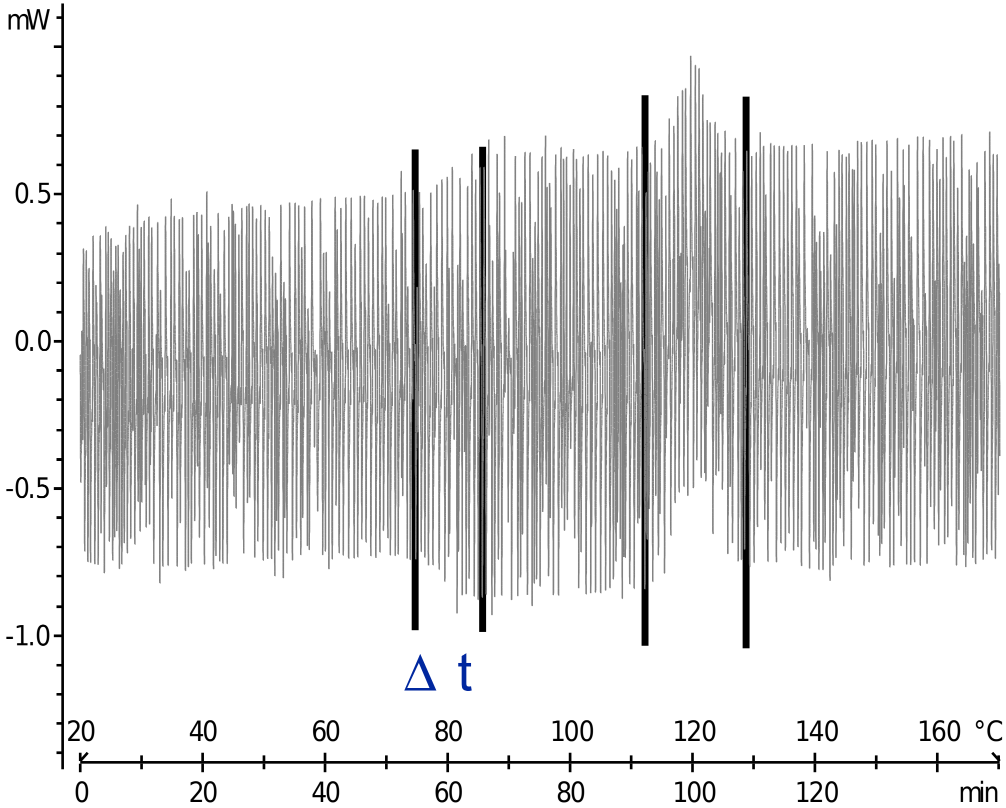
METTLER TOLEDO

TOPEM®: data evaluation

TOPEM®

calculation window

windows width $t_{cw} \leq \Delta t / 3$



METTLER TOLEDO

TOPEM®: data evaluation

TOPEM®

calculation window

windows width $t_{cw} \leq \Delta t / 3$

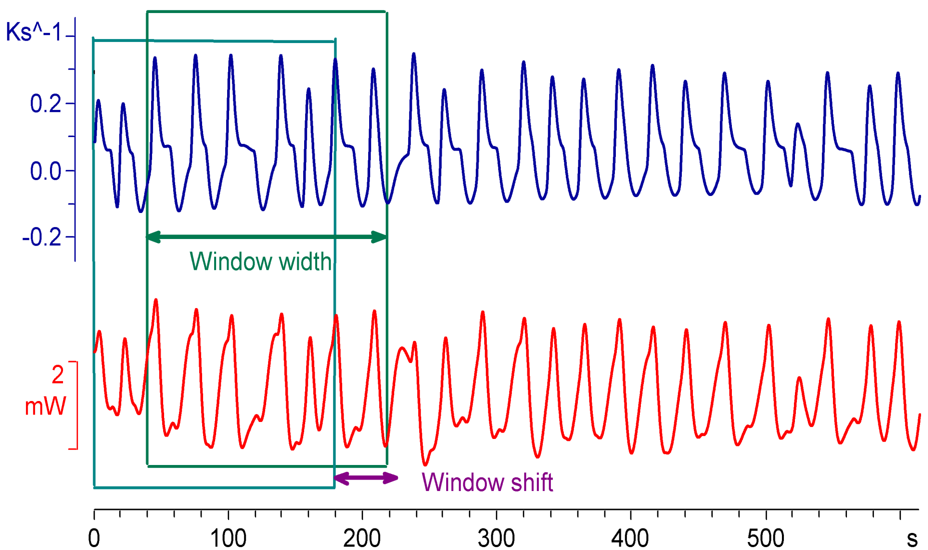
large t_{cw} --> better signals

$t_{cw}/si \approx 10 (n_a+n_b)$
lower limit

$t_{cw}/si \approx 20 (n_a+n_b)$
default

$t_{cw}/si \approx 50 (n_a+n_b)$
optimum

$t_{cw}/si >> 50 (n_a+n_b)$
very low frequencies



si: sampling interval
default: si=0.1 s

METTLER TOLEDO

TOPEM®:conclusion

FRS5 sensor and Al- crucibles

	method			evaluation				
	β /K/min	Puls hight /K	Puls width /s	calculation window /s	Window shift /s	na	nb	remarcs
High precision with frequency evaluation	≤0.5	1 melting ≤0.1..0.002	15..30 to very low frequencies 15..100 ..500	≥120 ≥1000	≥10	20..50 200	45	Calculation window: Three in a thermal event. Best results: (na+nb)50*sampling interval normal: (na+nb)20*sampling interval
Fast processes	≤0.5	≤0.1	15..30	120	≥10	50	45	
normal measurements	0.5..2	1…0.002	15..30	120	1.. 10	2..20	45	
Fast measurements	5..15	0.5..1 melting ≤0.1	5..15	15..30	1	2..5	45	not recommented