A new approach: Dynamic QTGA

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Overview

1. Introduction
2. Standard approach for the QTGA
3. New approach for the QTGA
4. Possible Pros/Cons of the new approach
5. Summary

Some terminology recapping (ECSS-Q-TM-70-52A)

Quartz Crystal Microbalance(QCM) Thermogravimetric analysis (TGA): Measures weight/mass change(loss/gain) and the rate of weight change as a function of temperature, time and atmosphere.

TML	: Total Mass Loss
RML	: Recovered mass loss
CVCM	: Collected volatile condensable material
KC	: Knudsen Cell
LN2	: Liquid Nitrogen
DOK	: Dynamic outgassing Knudsen
Standard Test	: TGA performed at the end of the test
EOT	: End of Test

Introduction-1

Outgassing is the release of molecules into vacuum that were trapped on or in a material.

Types of mechanisms:

- Desorption
- Evaporation
- Diffusion
- Transportation
- Sticking/Condensation
- *Re-emission*
- *Reflection*

Relevant standard ECSS-Q-TM-70-52A Kinetic outgassing of materials for space

Introduction-2

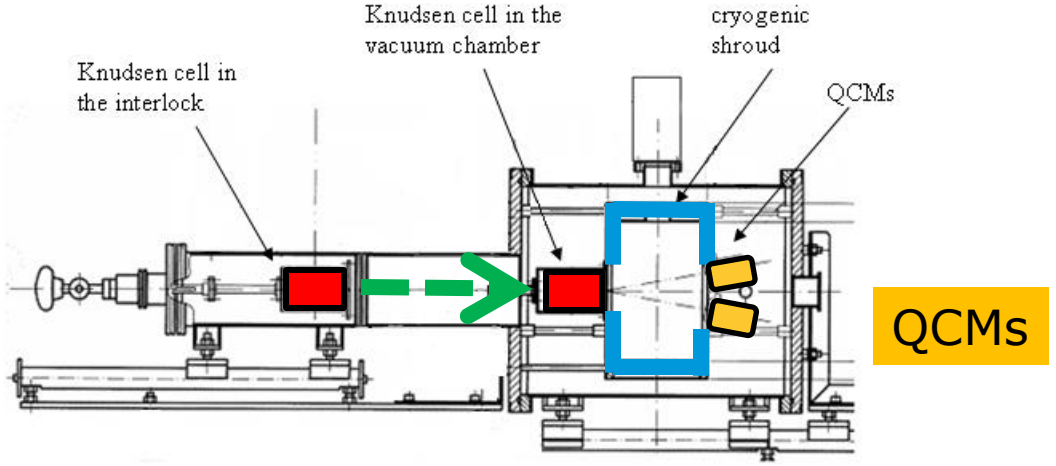
A QCM can mimic a critical surface and through a controlled increase in temperature provide re-emission characteristics of condensed molecular mass.

- Increase our understanding on the kinetics of the condensed molecular mass (e.g. temperature of evaporation, rate of evaporation)
- Increase our understanding on the nature of the condensed molecular mass (e.g. creep, crystallize, react)
- Support contamination simulation tools with more accurate input parameters

Relevant standard ECSS-Q-TM-70-52A Kinetic outgassing of materials for space

Standard QTGA approach and the DOK schematic

Sample temperature is increased from 25°C to X°C by steps of 25°C every 24h. The number of steps and the duration at the max. test temperature is adjusted based on the real application temperature and the max. permitted test temperature of the sample.



$T_{max} \cong 450°C$

$\bar{T} \cong -175°C$

$P < 5 \times 10^{-6} mbar$

$T_{QCM,1} \cong -170°C$

$T_{QCM,2} \cong -25°C$

$T_{QCM,3} \cong -75°C$

$T_{QCM,4} \cong -50°C$

QCM Models and Set temperatures

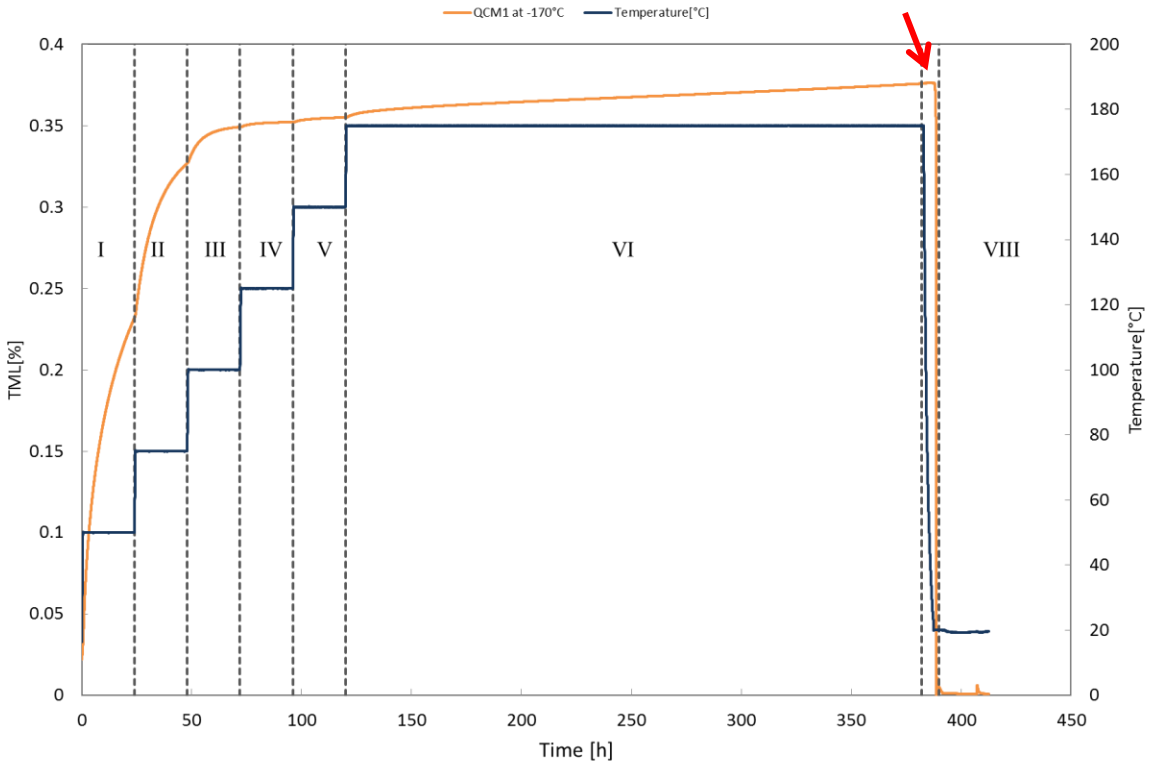
QCM	Model	Temperature Range [°C]*	Set Temperature [°C]	QTGA
QCM1	CQCM	-199 – 100	-170	EOT
QCM2	TQCM	-80 – 100	-25	EOT
QCM3	TQCM	-80 – 100	-75	EOT
QCM4	TQCM	-80 – 100	-50	EOT

EOT: End of Test

*Temperature range values are taken from the manufacturers website.

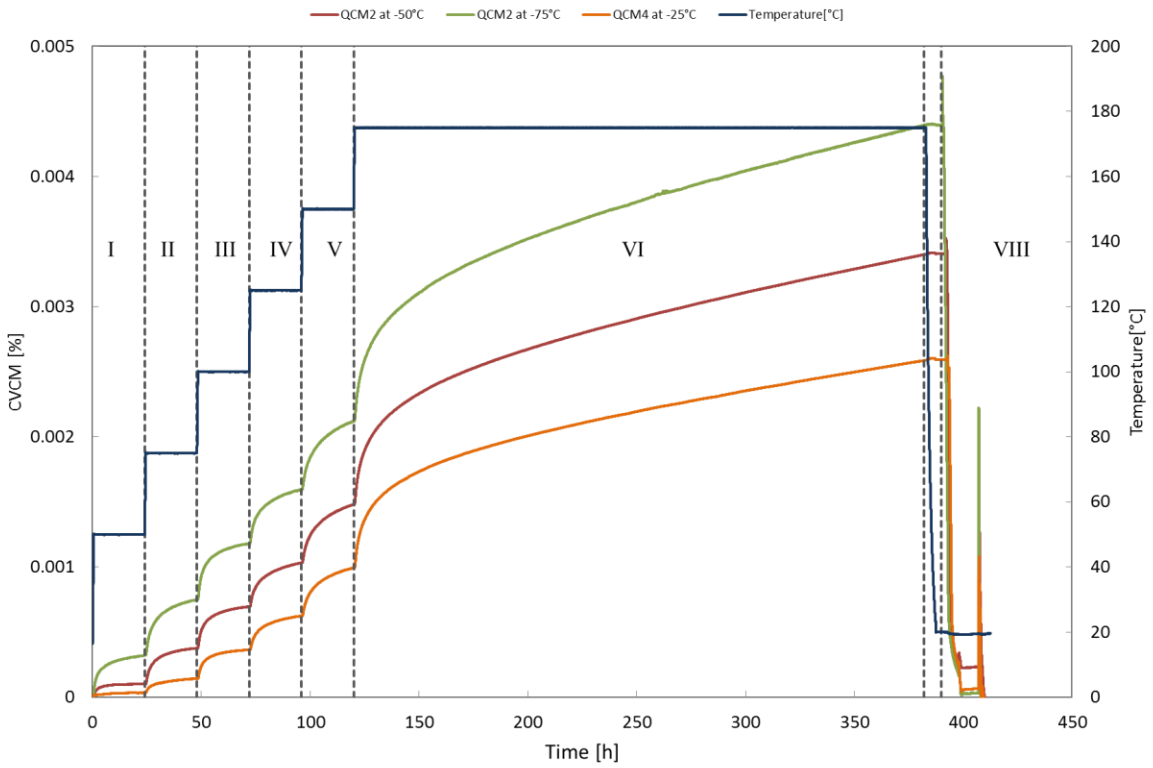
Standard Test – (in-situ) TML (%) of Sample_X

Max. permitted mass loss value by the mathematical model for this sample



Test Steps	Test Duration [h]	Sample Temperature [°C]
I	24	50
II	24	75
III	24	100
IV	24	125
V	24	150
VI	169	175
EOT		
VII	~10	20
VIII	QTGA	

Standard Test – (in-situ) CVCM(%) of Sample_X



Test Steps	Test Duration [h]	Sample Temperature [°C]
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III	24	100
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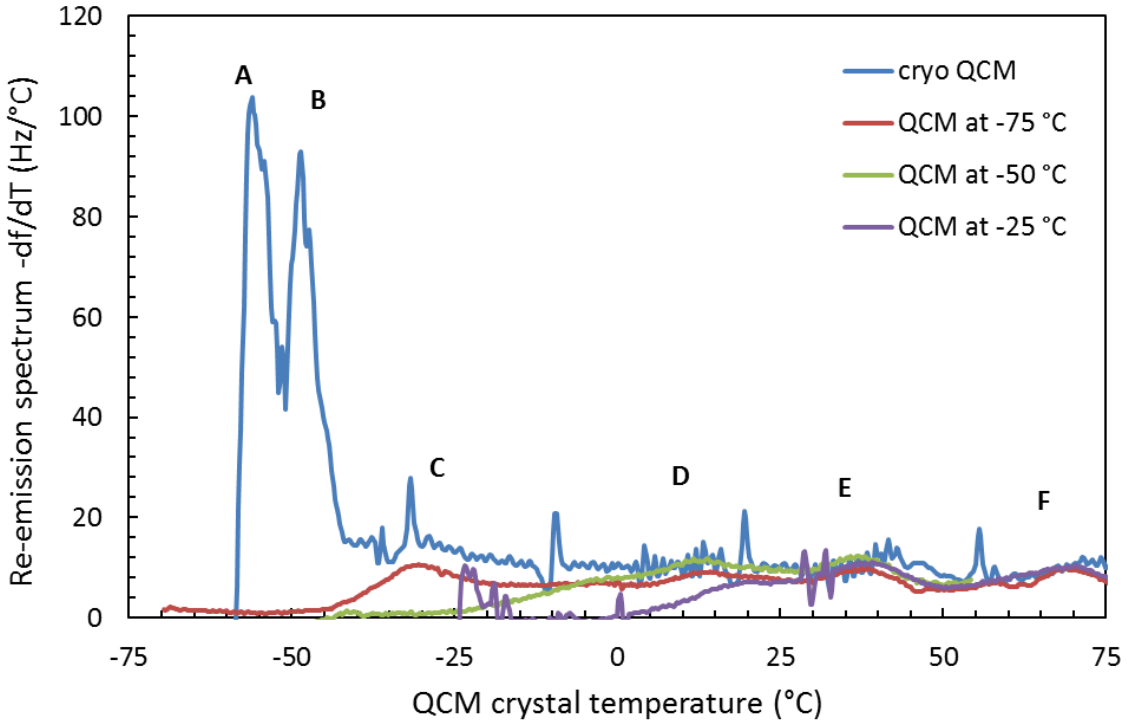
EOT		
VII	~10	20
VIII	QTGA	

Standard Test – QTGA



Test Steps	Test Duration [h]	Sample Temperature [°C]
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V	24	150
VI	169	175
EOT		
VII	~10	20
VIII	**QTGA**	

Output parameters generated by current approach

1) Mathematical species

Time constant $\tau_{0,i}$ (h)	Initial mass $W_{0,i}$ (%)
0.5	0.0058
4.482096	0
40.17836	0.13660
360.1665	0.14052
3228.602	0.04488
28941.81	0.00067
259439.9	0.00312
2325669	0.00356
20847740	0.01035

2) A table that contains activation energies and temperature-time constant

Terms	Temperature (°C)	Acceleration Factor $K_{i\rightarrow i+1}$	Apparent Activation Energy $E_{i\rightarrow i+1}$ (kJ·mol^{-1})	Residence time-temperature dependency coefficient k_e
I→II	75	4.12	52.9	0.0834
II→III	100	3.65	52.9	
III→IV	125	5.66	56.0	
IV→V	150	20.68	85.7	
V→VI	175	23.18	169.7	

3) TML, RML and CVCM of the sample
4) A clear description of the sample and the test methodology
5) TGA analysis
6) re-emission parameters are calculated by the assumption of $T_{ref} + 50K$

Dynamic QTGA approach and the DOK schematic

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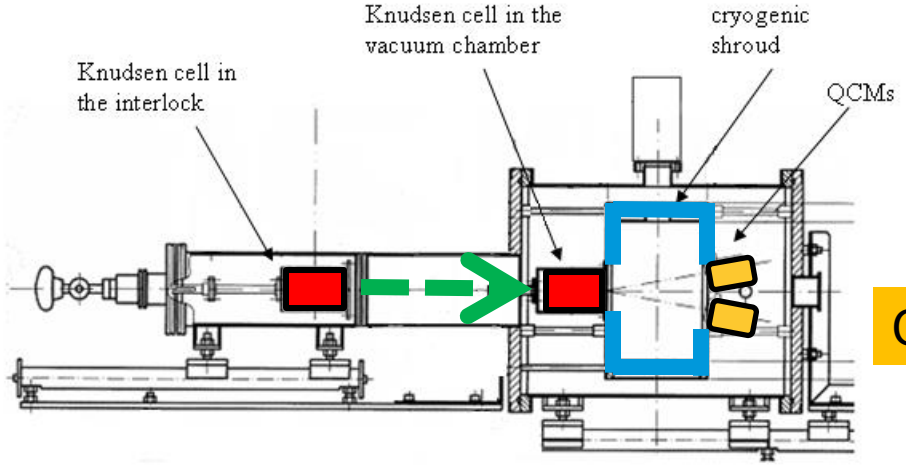

QCMs

$P < 5 \times 10^{-6} mbar$

$T_{QCM,1} \cong -170°C$

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$T_{QCM,3} \cong -75°C$

$T_{QCM,4} \cong -75°C$

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QCMs Models and temperatures

QCM	Model	Temperature Range [°C]*	Set Temperature [°C]	QTGA
QCM1	CQCM	-199 – 100	-170	EOT
QCM2	TQCM	-80 – 100	-25	EOT
QCM3	TQCM	-80 – 100	-75	16h after each Temperature step change
QCM4	TQCM	-80 – 100	-75	EOT

EOT: End of test

*Temperature range values are taken from the manufacturers website.

Example Test with the new QTGA approach:

Sample_X2 :

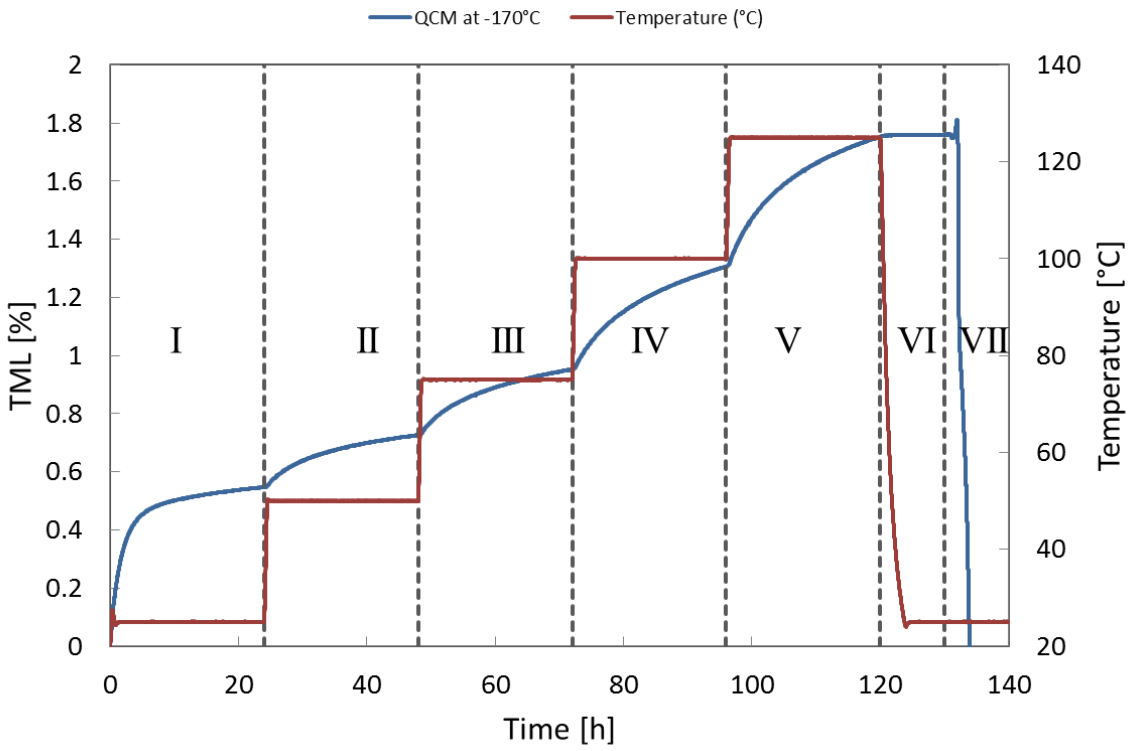
Chemical composition: Two-part silicone rubber

TML(%) : 1.95

RML(%) : 1.93

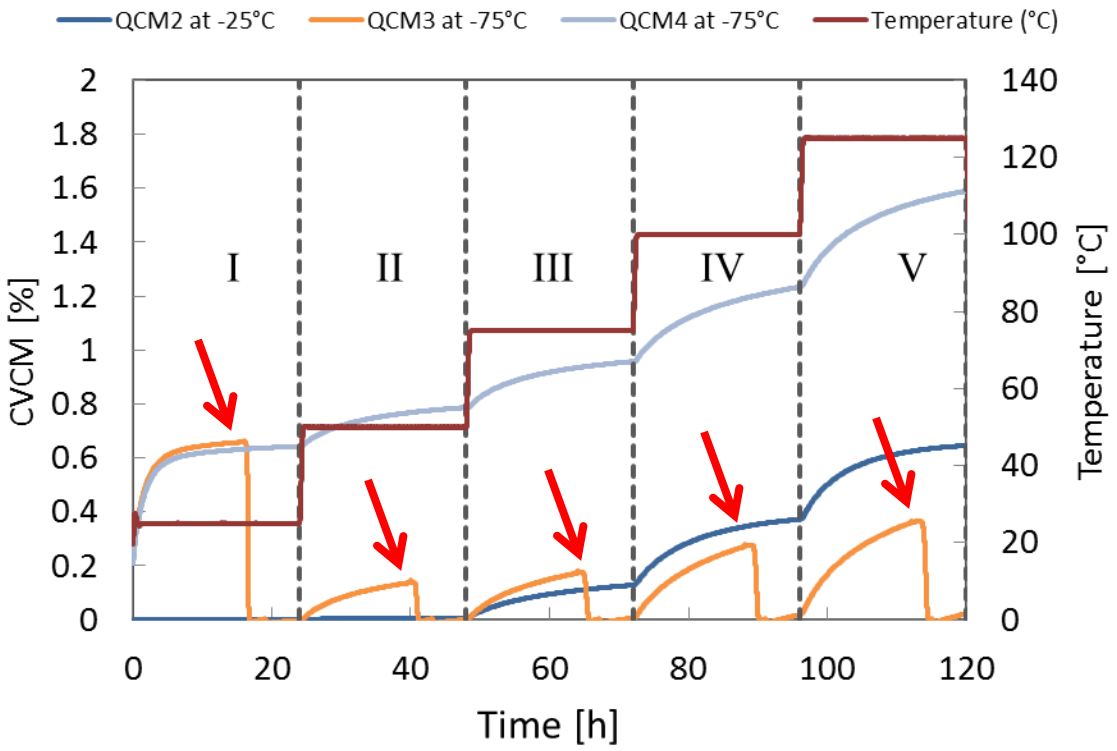
CVCM(%) at -75°C : 1.59

Test #2 – (in-situ) TML of Sample_X2



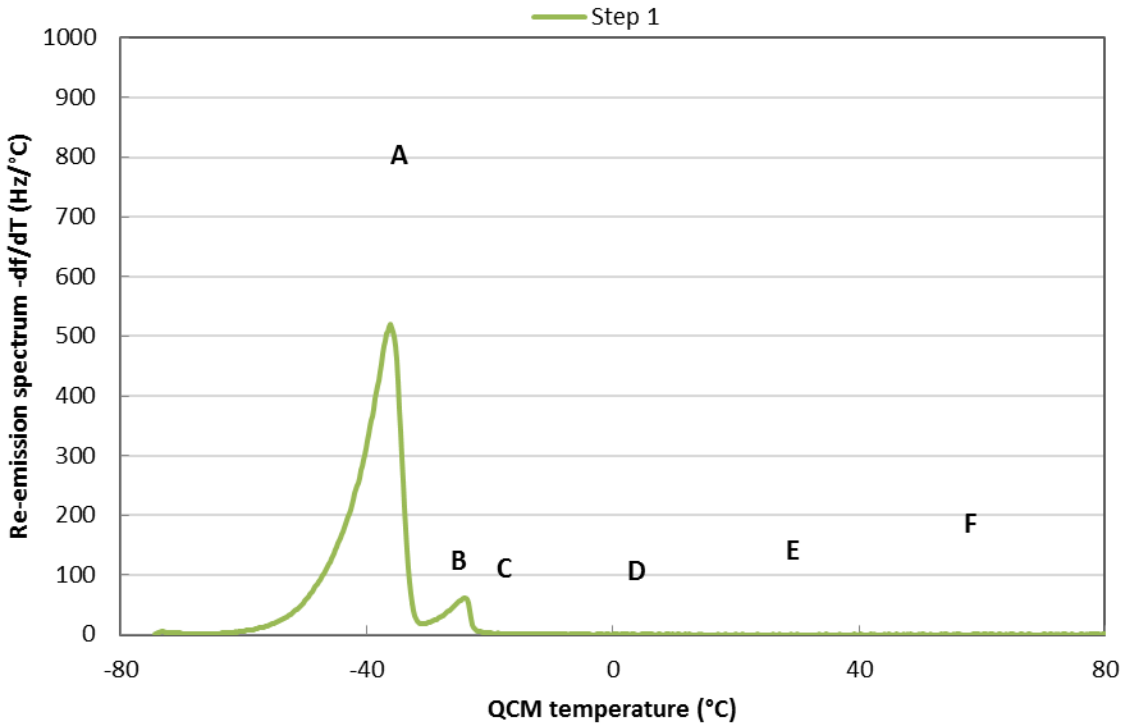
Test Steps	Test Duration [h]			Sample Temperature [°C]
		QTGA QCM,3		
I	16	4	4	25
II	16	4	4	50
III	16	4	4	75
IV	16	4	4	100
V	16	4	4	125
EOT				
VI	~10			20
VII	QTGA			

Test #2 – (in-situ) CVCM of Sample_X2



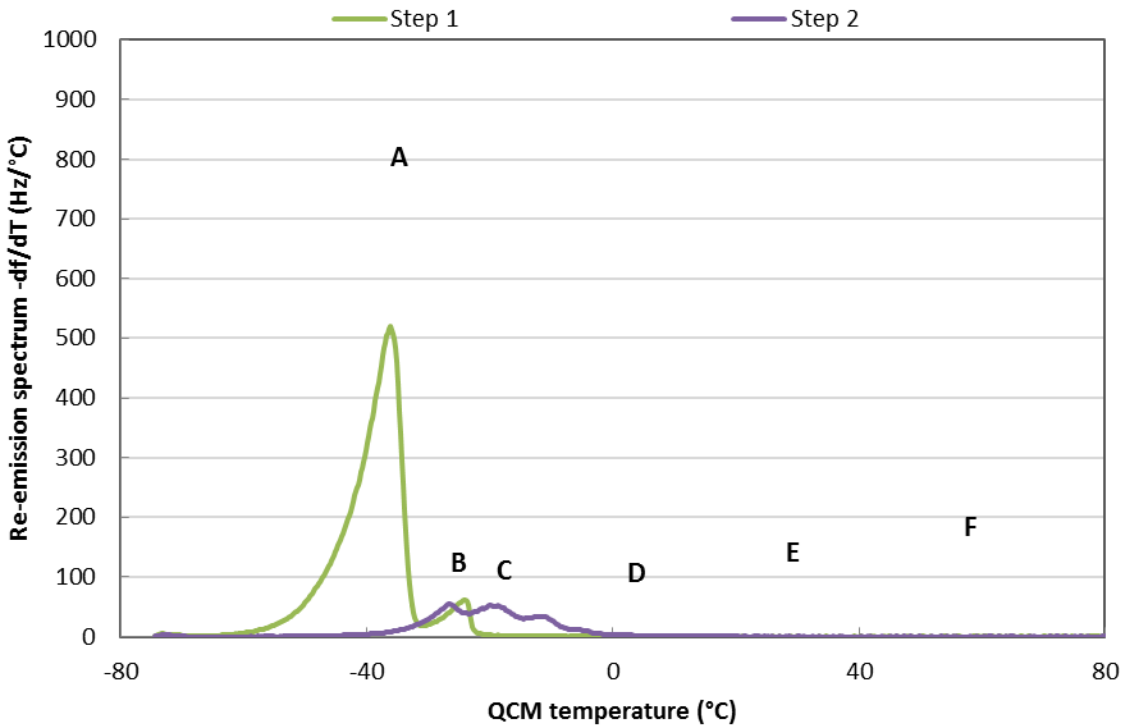
Test Steps	Test Duration [h]			Sample Temperature [°C]
		QTGA QCM,3		
I	16	4	4	25
II	16	4	4	50
III	16	4	4	75
IV	16	4	4	100
V	16	4	4	125
EOT				
VI	~10			20
VII	QTGA			

QCM3 QTGA – Test Step I of Sample_X2



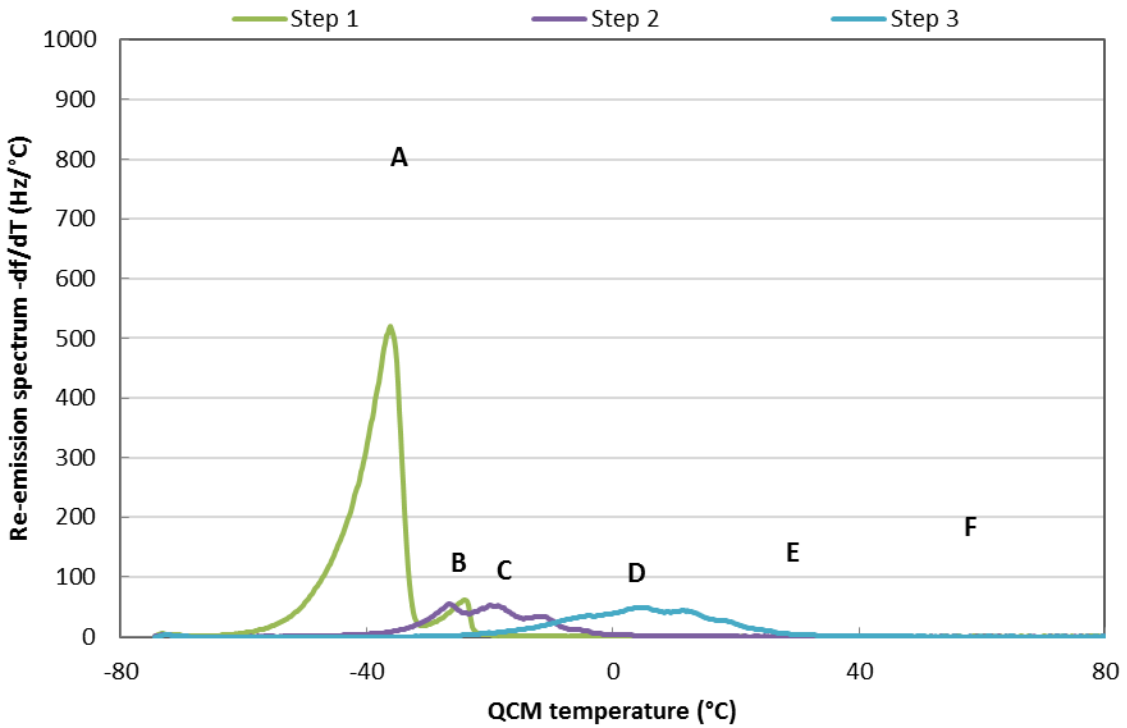
Test Steps	Test Duration [h]			Sample Temperature [°C]
		QTGA QCM,3		
I	**16**	**4**	**4**	**25**
II	16	4	4	50
III	16	4	4	75
IV	16	4	4	100
V	16	4	4	125
EOT				
VI	~10			20
VII	QTGA			

QCM3 QTGA – Test Step I-II of Sample_X2



Test Steps	Test Duration [h]			Sample Temperature [°C]
		QTGA QCM,3		
I	16	4	4	25
II	16	4	4	50
III	16	4	4	75
IV	16	4	4	100
V	16	4	4	125
EOT				
VI	~10			20
VII	QTGA			

QCM3 QTGA – Test Step I-III of Sample_X2



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I	16	4	4	25
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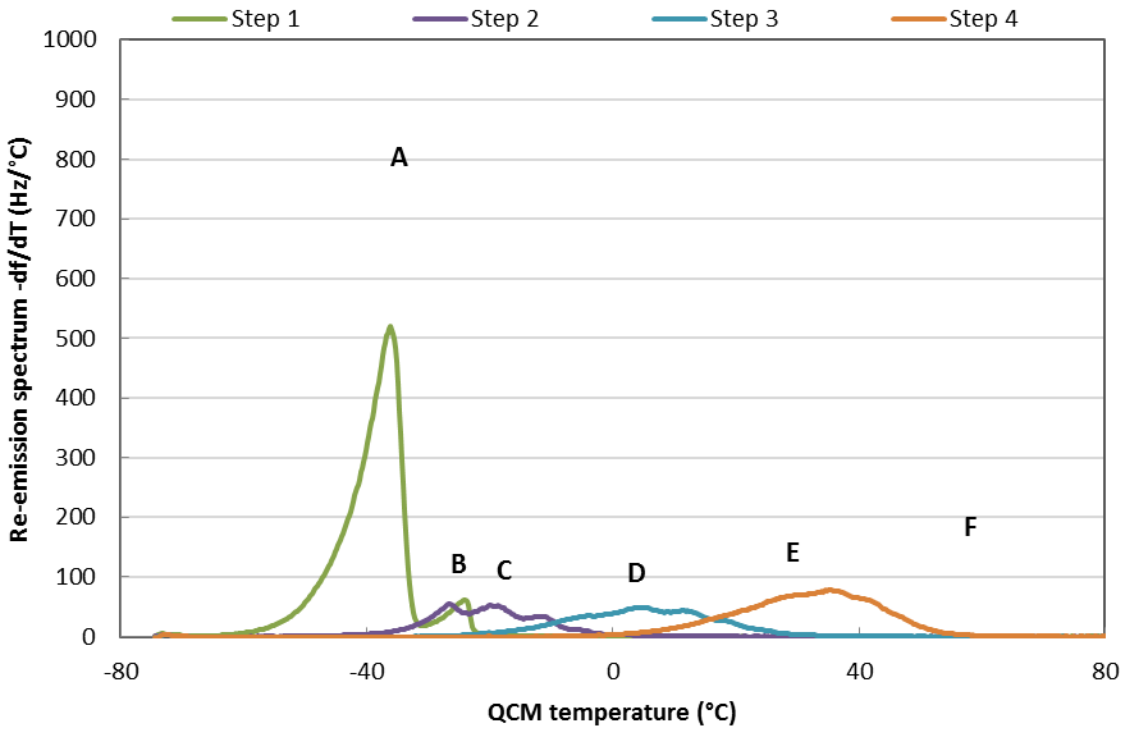
V	16	4	4	125
EOT				
VI	~10			20
VII	QTGA			

QCM3 QTGA – Test Step I-IV of Sample_X2



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EOT				
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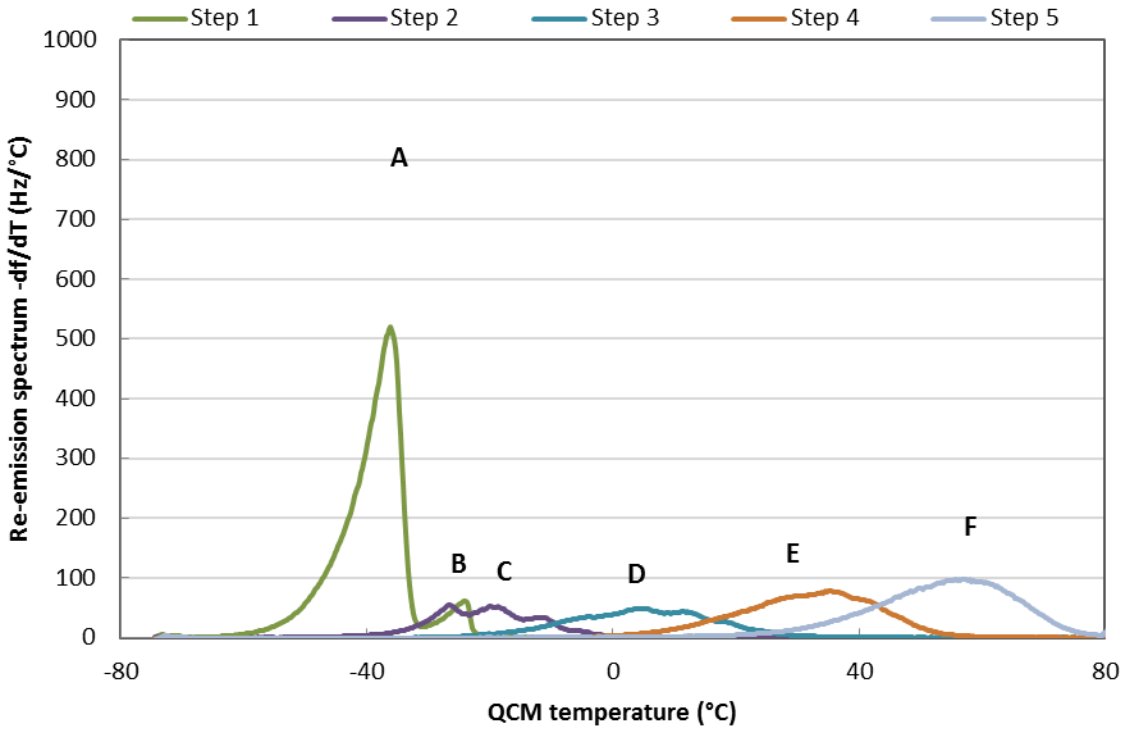
VII	QTGA			

QCM3 QTGA – Test Step I-V of Sample_X2



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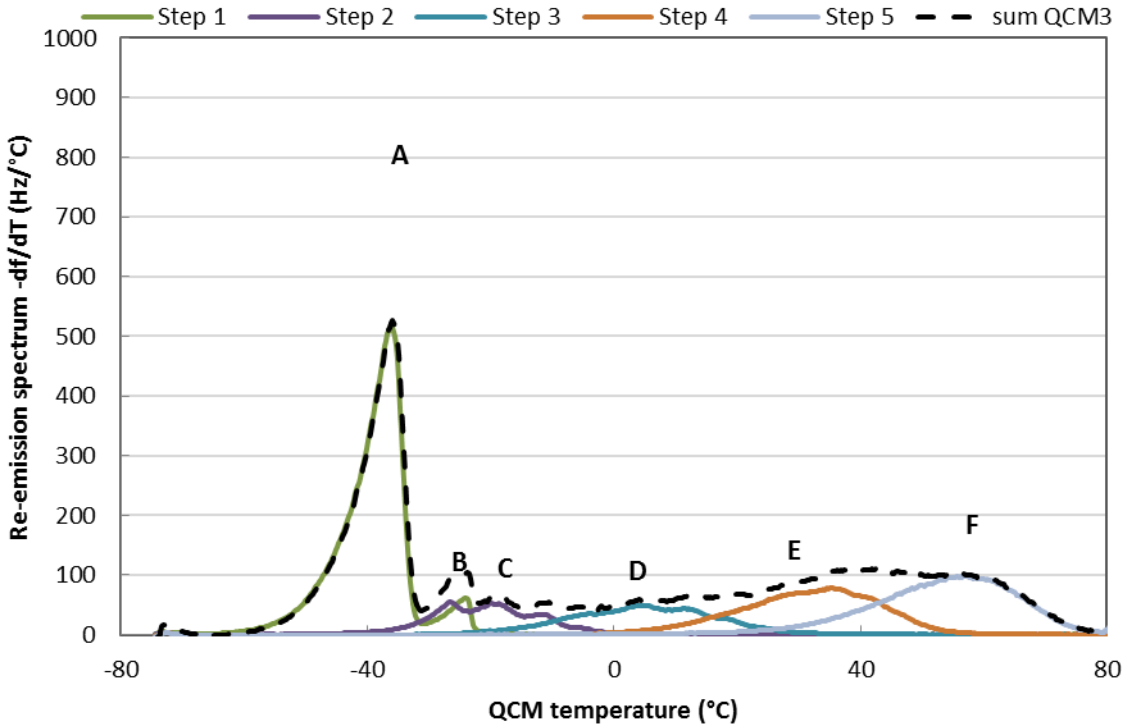
QCM3 QTGA – Test Step I-V of Sample_X2



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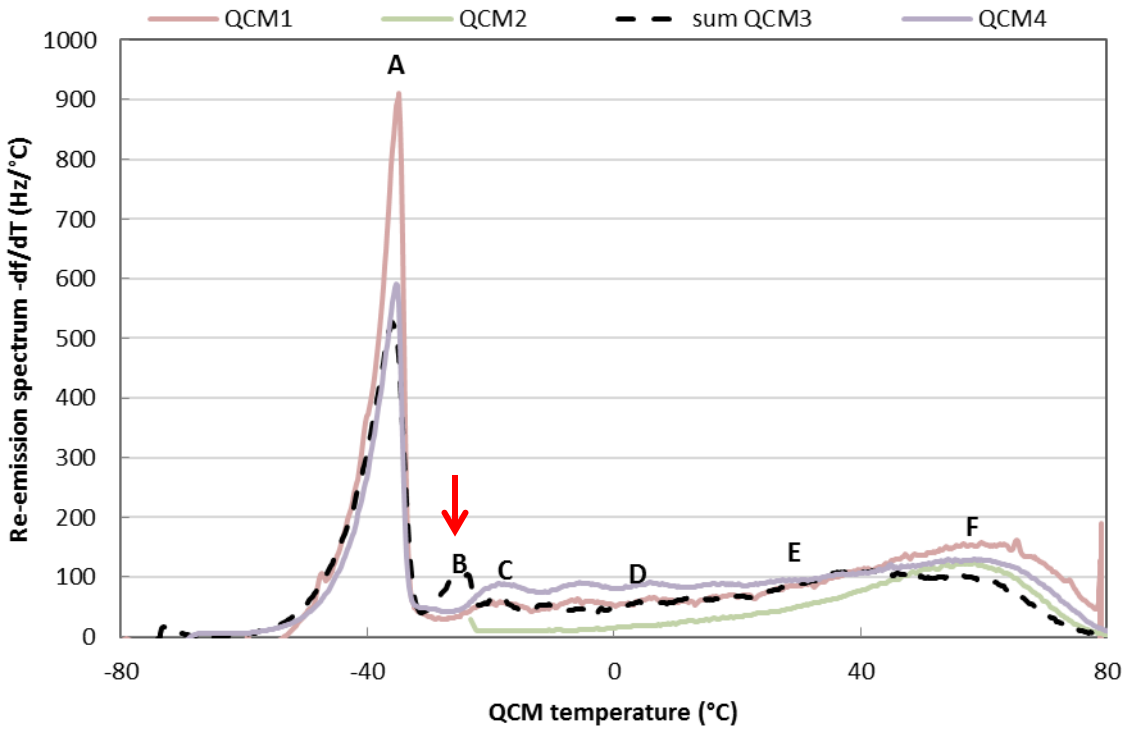
QCM3 QTGA – Test Step I-V of Sample_X2



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2) A table that contains activation energies and temperature-time constant

Reaction	Temperature (°C)	Acceleration Factor $K_{i\rightarrow i+1}$	Apparent Activation Energy $E_{i\rightarrow i+1}$ (kJ·mol^{-1})	Residence time-temperature dependency coefficient k_e
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3) TML, RML and CVCM of the sample
4) A clear description of the sample and the test methodology
5) TGA analysis that contains further information which may improve contamination modelling
6) re-emission parameters are calculated by the assumption of $T_{ref} + 50K$

Summary

1. Increased understanding of the temperature evaluation of condensed molecules with a little more effort.
2. Possibilities to further characterize the kinetics of re-emitted molecules with the use of additional techniques (e.g. RGA, GC/MS, FTIR)
3. Support contamination simulation tools with more accurate input data.

THANK YOU