LABORATÓRIO DE INSTRUMENTAÇÃO
E FÍSICA EXPERIMENTAL DE PARTÍCULAS

1 2 9 0 FACULDADE DE CIÊNCIAS E TECNOLOGIA UNIVERSIDADE D COIMBRA

Particle Tracks Reconstruction Using Passive Dosimeters

A Path Towards Cell Level Dosimetry

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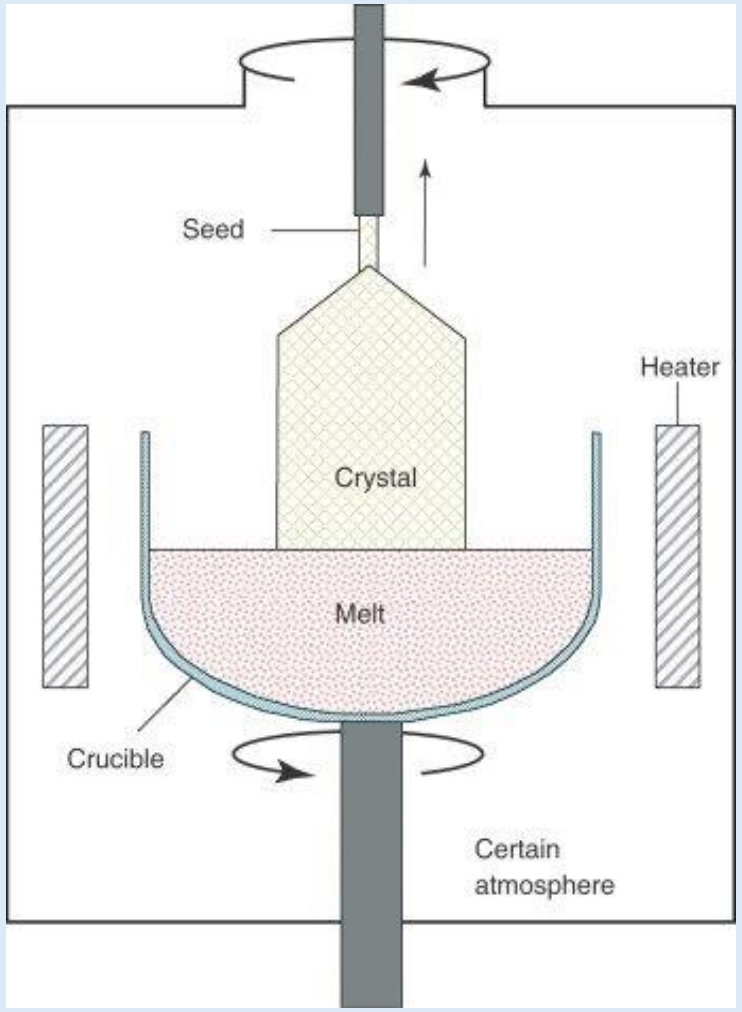
Czochralski Method



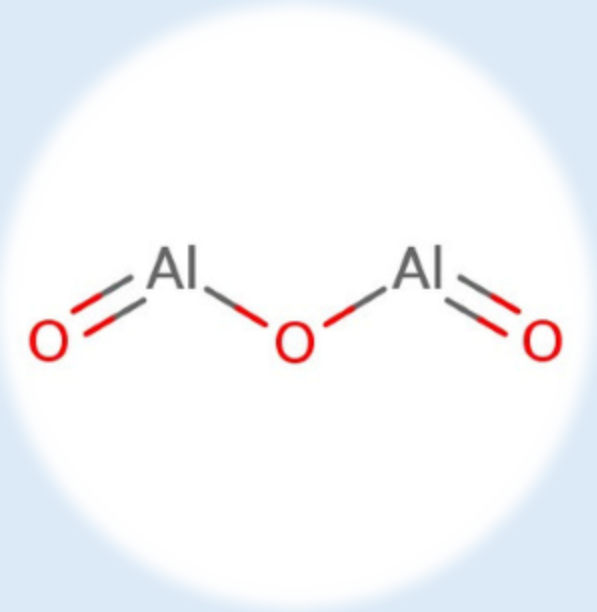
Advantages

- Alumina powder melted;
- Growth of large monolithic crystal: 20 cm of length and 5 cm of width;
- Later cut in 8x4x0.5 mm (custom size for dosimetry).

Disavantages

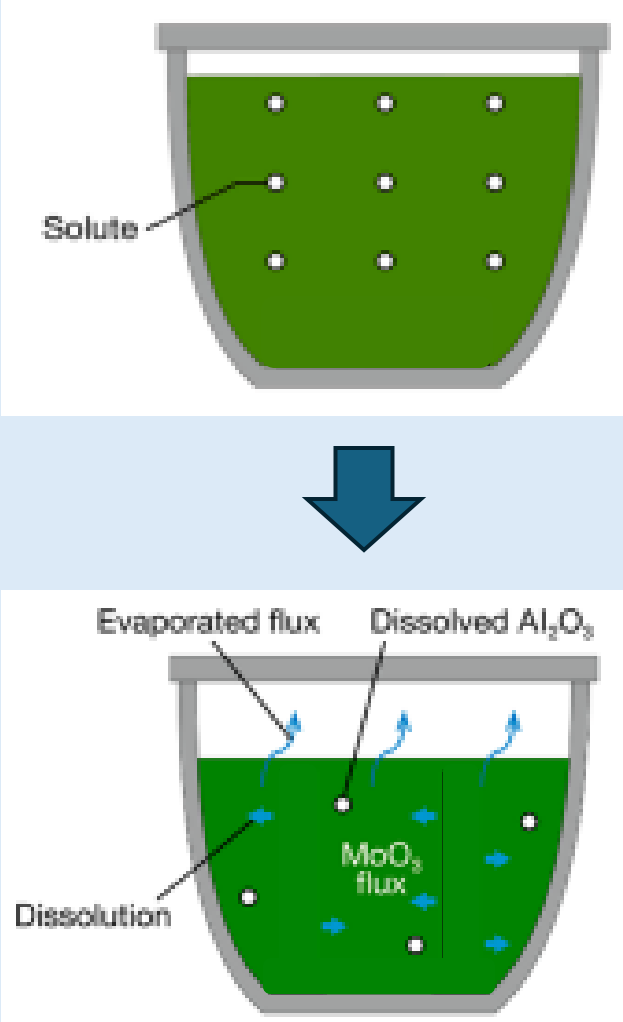
- Complex and expensive production condition.

Furnace Temperature > 2072 ºC



Aluminum Oxide – Al2O3
Melting Point = 2072 ºC

Flux Method



Operation: MoO3, a reagent capable of dissolving alumina at a lower temperature, is added.

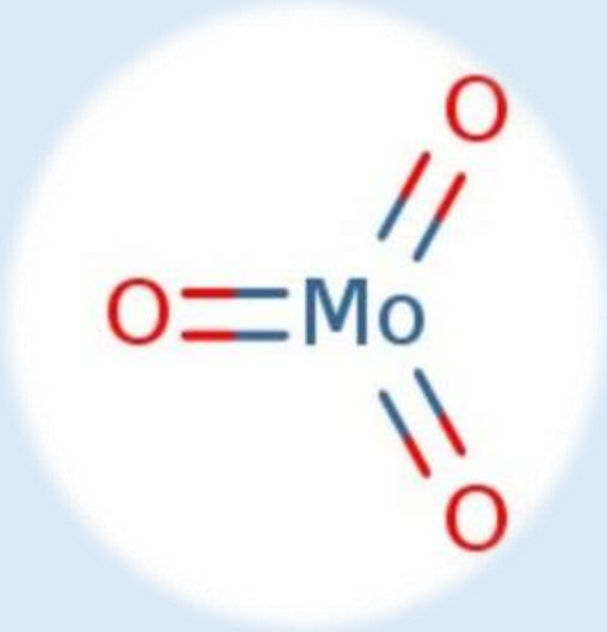
Advantages

- More than 10x cheaper.

Disavantages

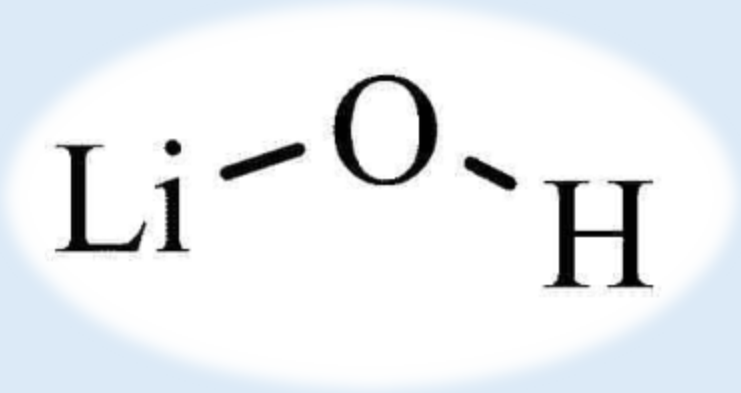
- Small size crystals;
- Impurities contamination;
- FNTD characteristics need to be evaluated.

Furnace Temperature:
around 1100 ºC and 1200 ºC

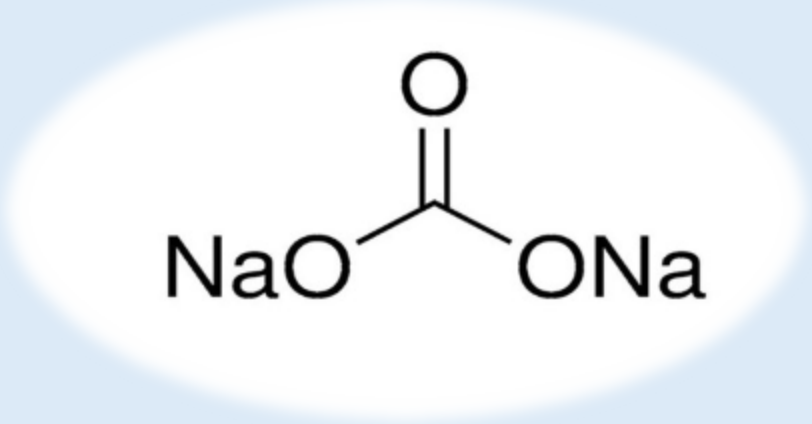


Molybdenum Trioxide – MoO3
Melting Point = 795 ºC
Boiling Point = 1155 ºC

Inhibitors



Lithium Hydroxide - LiOH



Sodium Carbonate - Na2CO3

2 LiOH ⇨ Li2O + H2O

Na2CO3 ⇨ Na2O + CO2

Operation: It delays the evaporation of molybdenum.

Note: the percentages of the inhibitors are adjusted for their Li2O and Na2O forms.

Methodology

Muffle Furnace

1 hour, 150 ºC

Scale

precision of 0.1 mg

Mixing

Pressing

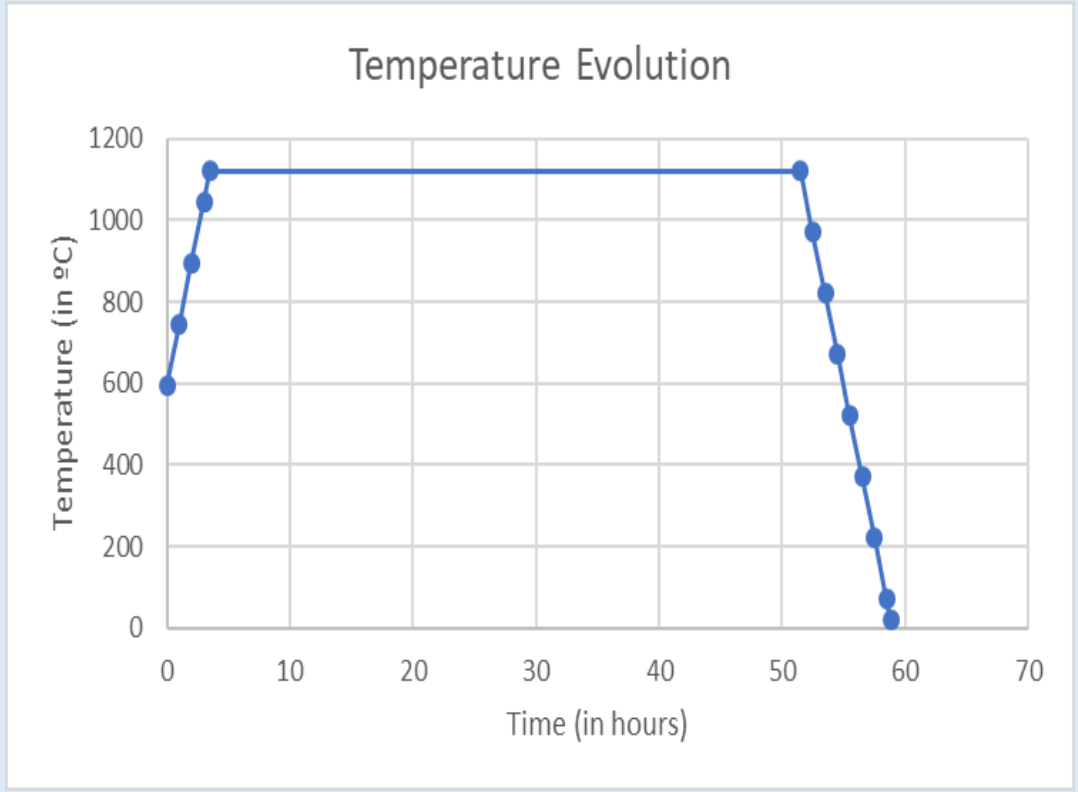
LiOH / Na2CO3 Al2O3 MoO3

The Furnaces

Holding Temperature: **1120 ºC**

Heating
150 ºC/h

Starting Point
595 ºC

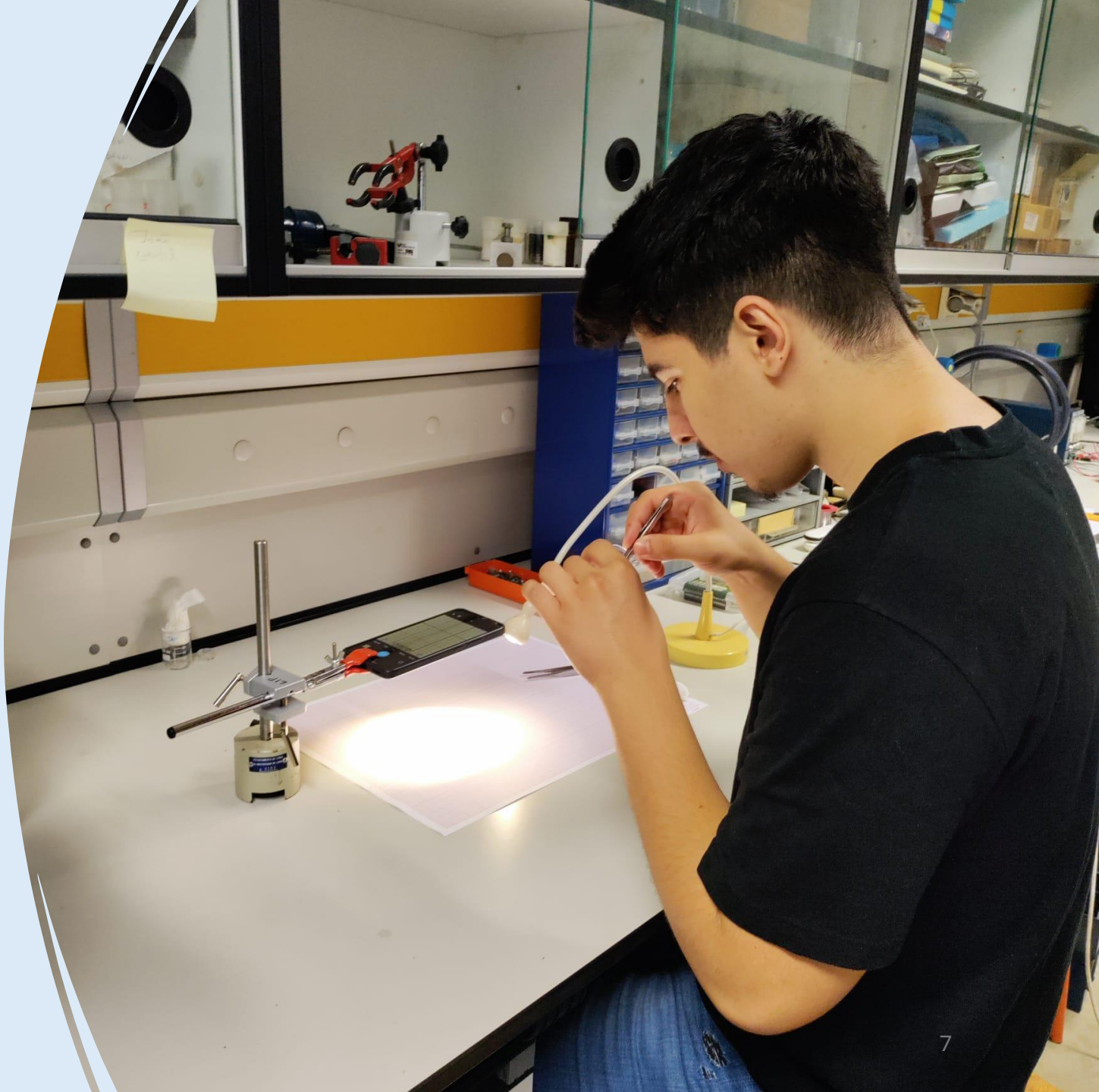


Cooling
150 ºC/h

Ending Point
20 ºC

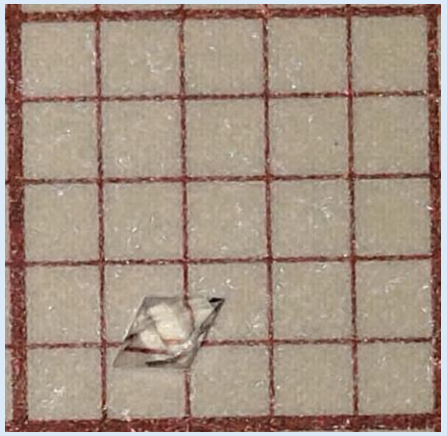
Characterization

- Counting the crystals;
- Compare their dimensions;
- Observe whether the crystals were faceted or irregular;
- Observe whether the crystals were nucleated or transparent;
- Observe any other anomaly.



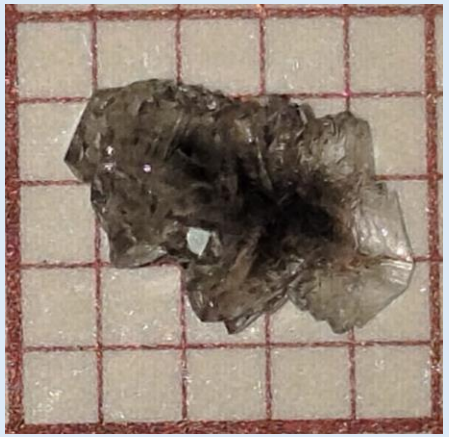
Fiji Software

Characteristics of Crystals



- Small
- Regular
- Transparent
- Faceted
- Anucleated

- Medium
- Irregular
- Transparent
- Faceted
- Anucleated



- Large
- Irregular
- Opaque
- Domed
- Nucleated

- Medium
- Regular
- Opaque
- Faceted
- Nucleated

Crystal Growth Conditions

Run	Inhibitor	Name	Molar % of inhibitor	Molar % of Solute	Holding Time	Crystal Production
1	LiOH	L1	15 %	6 %	48 hours	No
2	Na2CO3	N1	15 %	6 %	48 hours	No
3	LiOH	L2	10 %	8 %	48 hours	Yes
4	Na2CO3	N2	10 %	8 %	48 hours	Yes
5	LiOH	L3	5 %	8 %	48 hours	Yes
6	Na2CO3	N3	5 %	8 %	48 hours	Yes
7	LiOH	L4	15 %	8 %	96 hours	No
8	Na2CO3	N4	10 %	8 %	72 hours	Yes

Effect of Inhibitor Percentage

8% of solute, 48 hours of holding time, using LiOH

Run	% Inhibitor	% Flux	Length (Average ± S.D.)	Width (Average ± S.D.)	Nº of crystals
L3	5 %	68.3 %	1.84 ± 0.97 mm	1.40 ± 0.71 mm	72
L2	10 %	34.9 %	1.58 ± 0.86 mm	1.21 ± 0.70 mm	50

L3
5,18 mm

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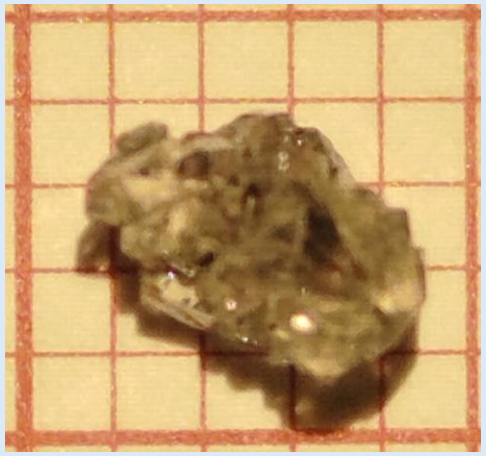


L2
3,91 mm

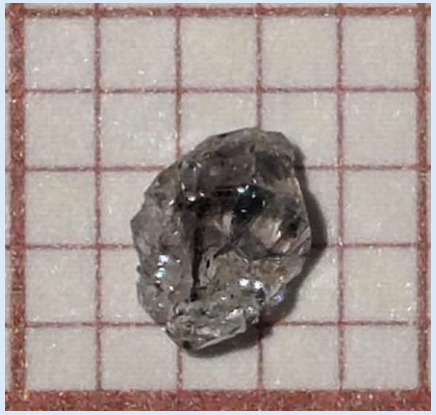
- L3 has a higher flux than L2;
- L3 has larger crystals than L2;
- L3 has much more regular (bipyramidal) crystals than L2;
- Both are nucleated (half of them) and equal in number.

Effect of Inhibitor Percentage

8% of solute, 48 hours of holding time, using Na2CO3

Run	% Inhibitor	% Flux	Length (Average ± S.D.)	Width (Average ± S.D.)	Nº of crystals
N3	5 %	75.9 %	1.29 ± 0.57 mm	1.01 ± 0.47 mm	158
N2	10 %	50.1 %	0.96 ± 0.29 mm	0.77 ± 0.26 mm	161

N3
3,05 mm



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N2
2,04 mm

- N3 has a higher flux than N2;
- N3 has larger crystals than N2;
- In N3 almost all are nucleated, while in N2 all are transparent;
- Both are irregular and equal in number.

Effect of Holding Time

10% of inhibitor, 8% of solute, using Na2CO3

Run	Holding Time	% Flux	Length (Average ± S.D.)	Width (Average ± S.D.)	Nº of crystals
N2	48 hours	50.1 %	0.96 ± 0.29 mm	0.77 ± 0.26 mm	161
N4	72 hours	52.3 %	1.03 ± 0.34 mm	0.85 ± 0.31 mm	111

N2
2,04 mm

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N4
2,18 mm

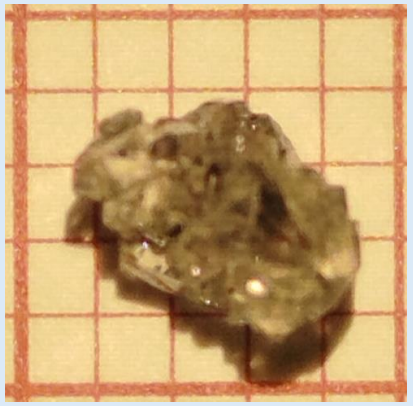
- N4 is (almost insignificantly) larger in flux and crystal size than N2;
- N4 produced fewer crystals than N2 (they probably fused together);
- Both the crystals in N2 and N4 were transparent and irregular.

Effect of Type of Inhibitor

10% of inhibitor, 8% of solute, 48 hours of holding time

Run	Inhibitor	% Flux	Length (Average ± S.D.)	Width (Average ± S.D.)	Nº of crystals
L2	LiOH	34.9 %	1.58 ± 0.86 mm	1.21 ± 0.70 mm	50
N2	Na2CO3	50.1 %	0.96 ± 0.29 mm	0.77 ± 0.26 mm	161

L2
3,91 mm



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N2
2,04 mm

- The sample with Na2CO3 has a higher flux than the LiOH sample;
- The sample with LiOH has larger crystals than the Na2CO3 sample;
- The sample with Na2CO3 has three times as many crystals as the LiOH sample;
- While the N2 crystals are all transparent and irregular, the L2 crystal were almost all of them irregular but were nucleated.

Effect of Type of Inhibitor

5% of inhibitor, 8% of solute, 48 hours of holding time

Run	Inhibitor	% Flux	Length (Average ± S.D.)	Width (Average ± S.D.)	Nº of crystals
L3	LiOH	68.3 %	1.84 ± 0.97 mm	1.40 ± 0.71 mm	72
N3	Na2CO3	75.9 %	1.29 ± 0.57 mm	1.01 ± 0.47 mm	158

L3
5,18 mm

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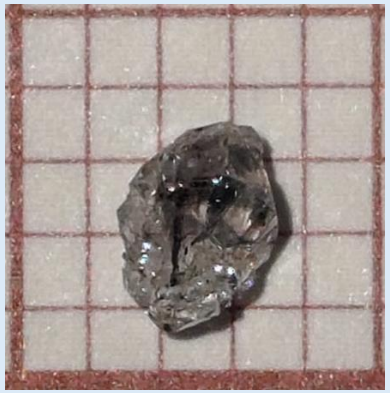
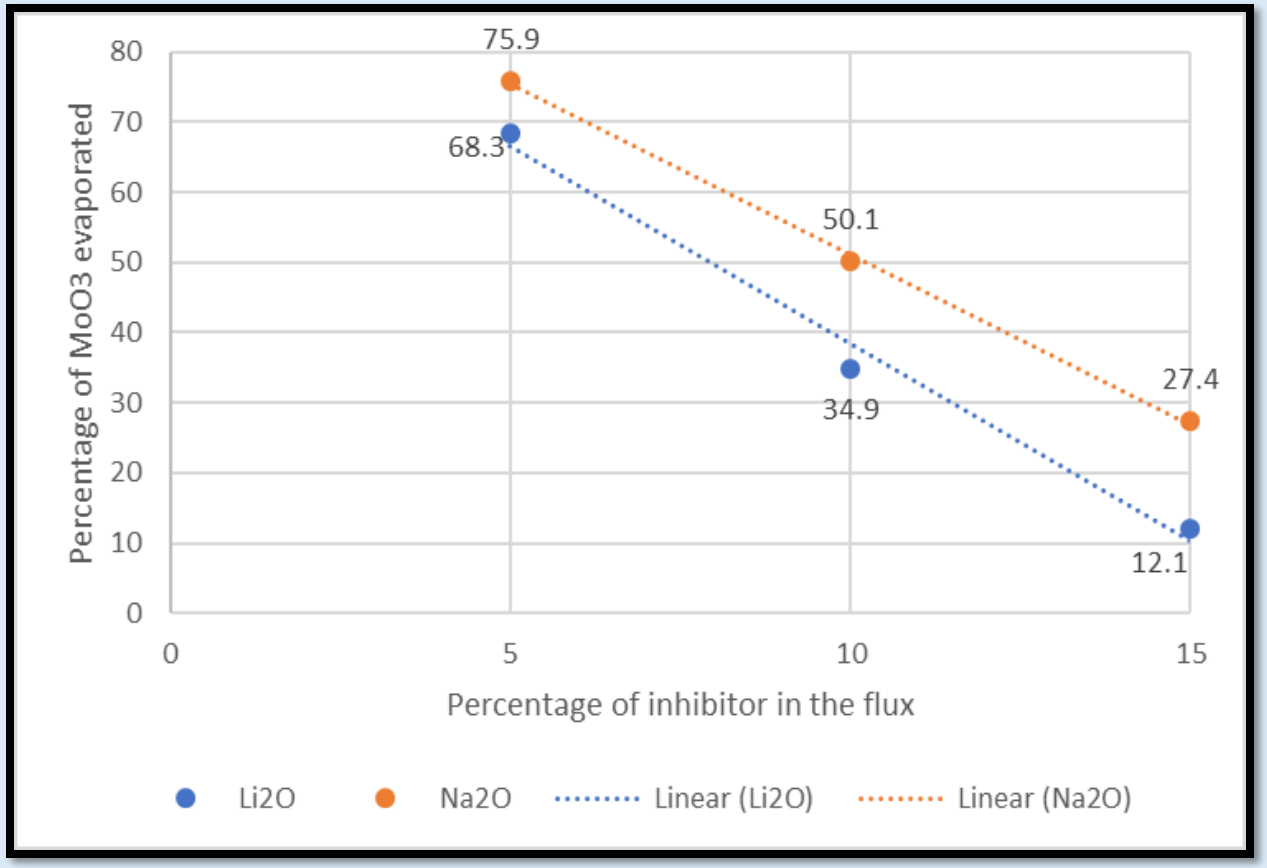


N3
3,05 mm

- The sample with Na2CO3 has a higher flux than the LiOH sample;
- The sample with LiOH has larger crystals than the Na2CO3 sample;
- The sample with Na2CO3 has three times as many crystals as the LiOH sample;
- Both samples have nucleated crystals, but while N3 crystals were all irregular, half of L3 crystals were regular (especially the smaller ones).

Conclusion

Percentage of MoO3 Evaporated after 48 hours of holding time for each Percentage of Inhibitor in the Flux



LiOH:

- Larger crystals;
- Fewer crystals;
- The larger crystals were irregular, but the smaller ones were regular;
- Always nucleated except for the smaller ones.

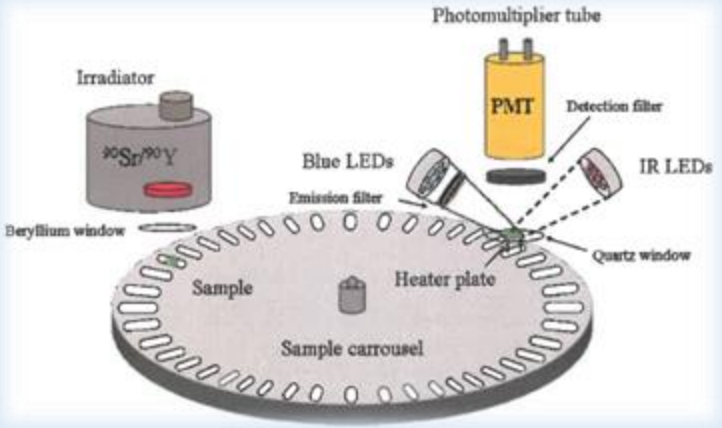
Na2CO3:

- Smaller crystals;
- More crystals;
- Always irregular;
- Always transparent if we use enough inhibitor.

Note: with 15% of inhibitor we use 6% of solute, but with 10% and 5% of inhibitor we use 8% of solute.

Future Work

Riso TL/OSL Reader



Next Parameters to test:

- Try increasing the holding temperature;
- For LiOH, try with less than 5 % of inhibitor;
- For Na2CO3, try between 5 % and 10 % of inhibitor.

Confocal Fluorescence Microscopy

X-Ray Diffraction - XRD