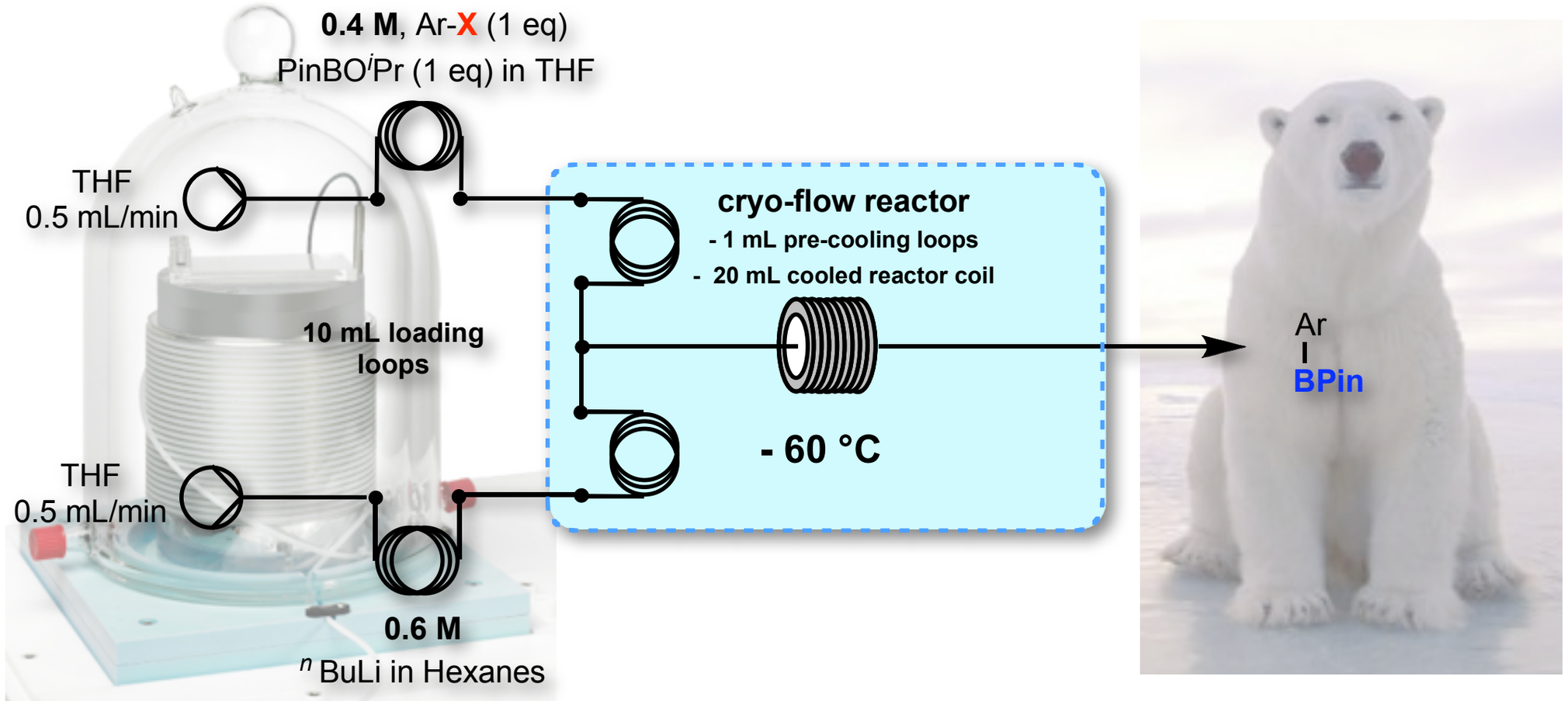
A New Enabling Technology for the Convenient Laboratory Scale Continuous Flow Processing at Low Temperatures



Duncan L. Browne

Innovative Technology Centre

CRD CAMBRIDGE REACTOR DESIGN

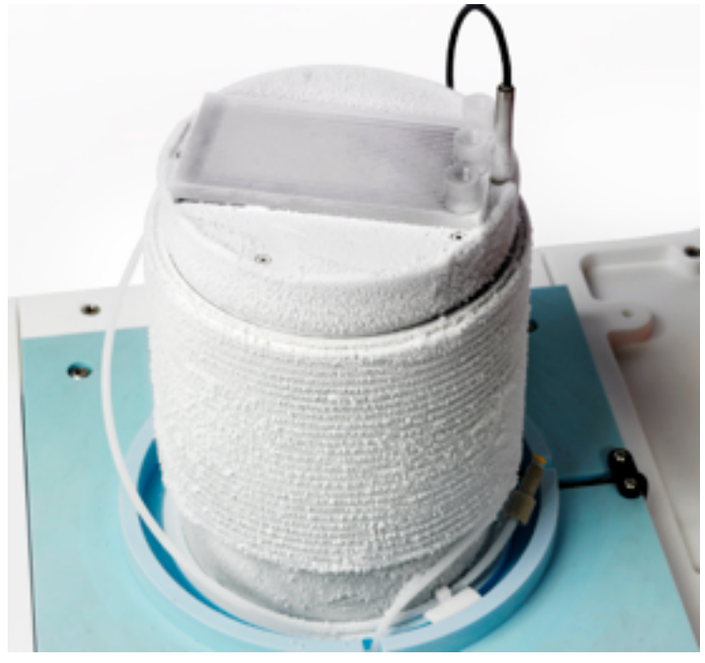
UNIVERSITY OF CAMBRIDGE
Department of Chemistry

The Polar Bear Reactor

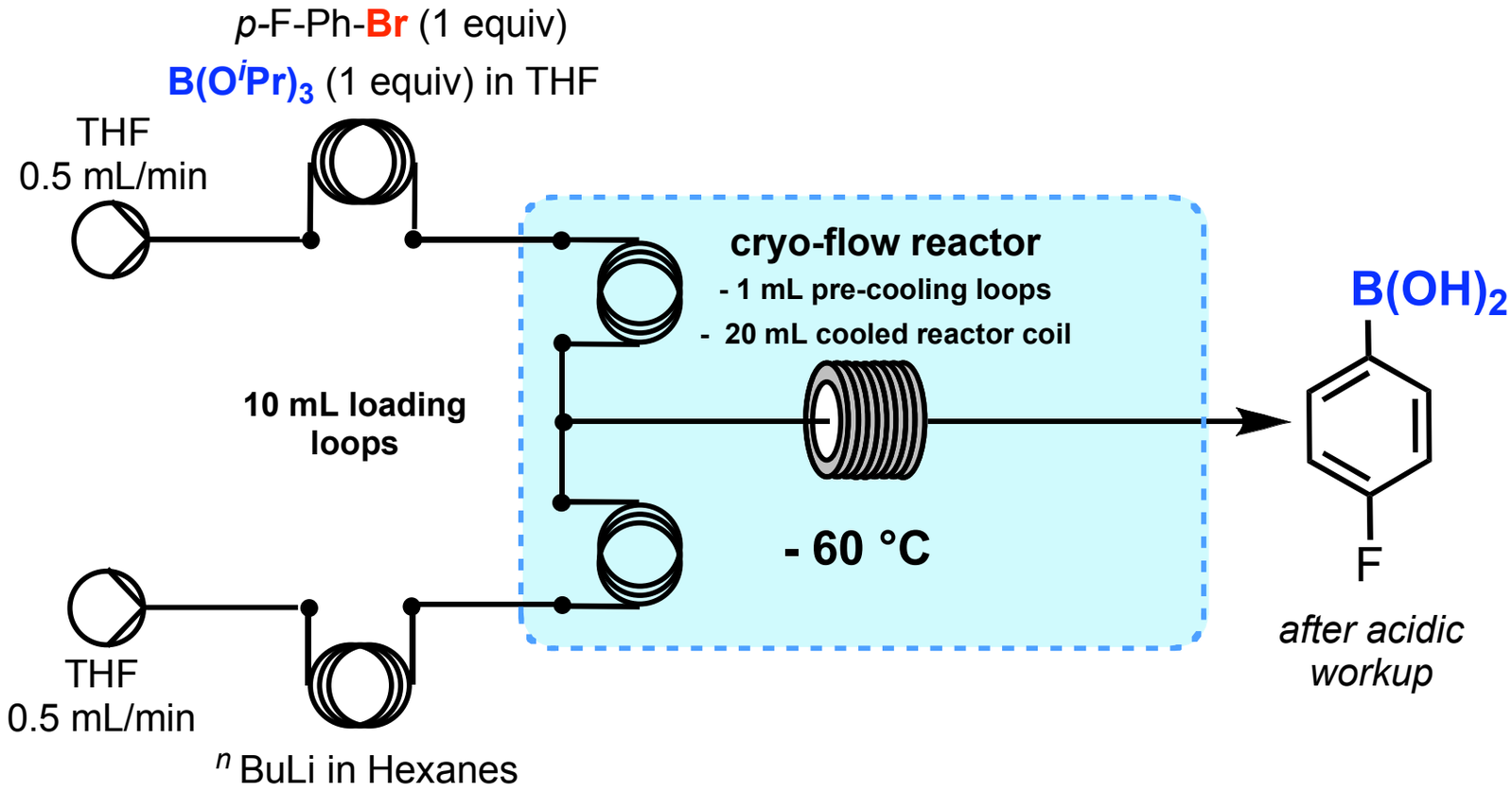
- The Cryo-flow reactor can get to any temperature between ambient and -89 °C.

- No use of cooling fluids or cryogenic materials like the current alternatives.

- Effectively it is the batch cryostat equivalent for flow, where the current alternatives are either ice baths equivalents and/or far less convenient to use.



Demonstrating the use of the Cryo-flow Reactor



Entry	[BuLi]	[Ar-Br]	Yield
1	0.15 M	0.10 M	68%
2	0.30 M	0.20 M	70%
3	0.45 M	0.30 M	75%
4	0.60 M	0.40 M	77% (430 mg)

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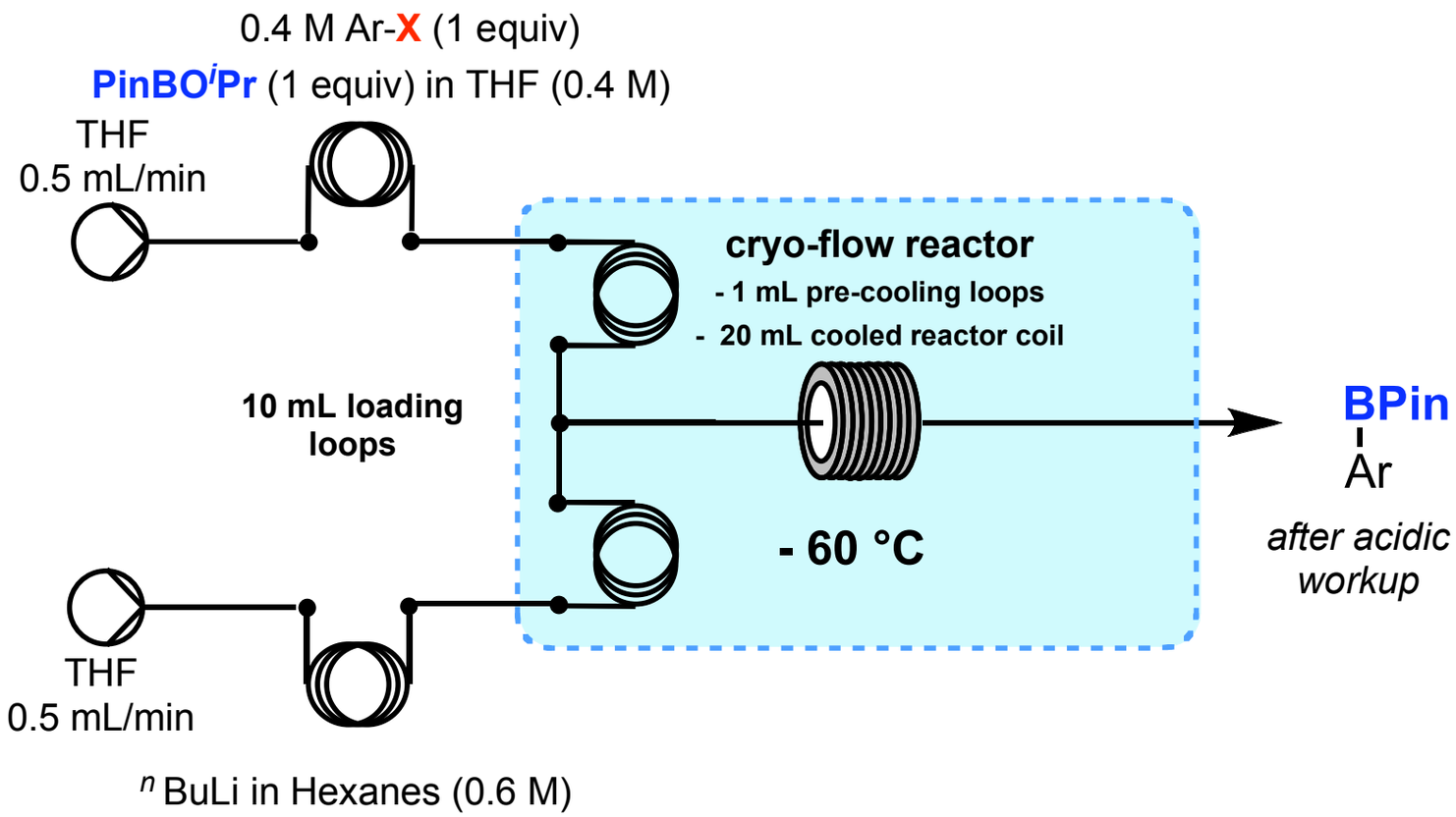
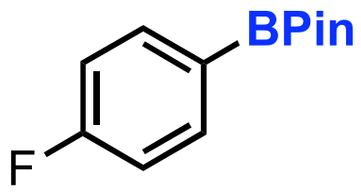
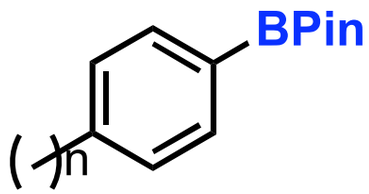
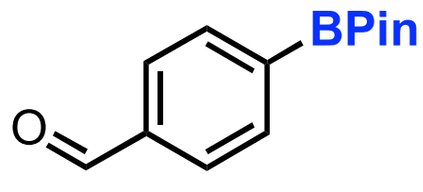




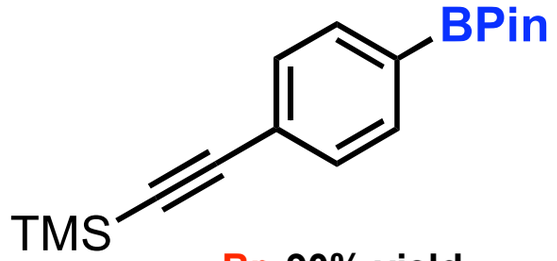
Br, **74% yield**
~ 650 mg



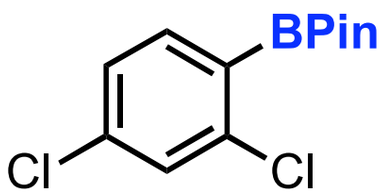
n = 1, **Br**, **88% yield** ~ 770 mg
n = 2, **Br**, **90% yield** ~ 840 mg



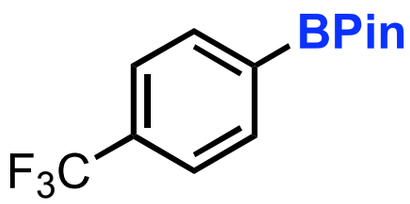
Br, **95% yield**
~ 900 mg
(from dimethylacetal)



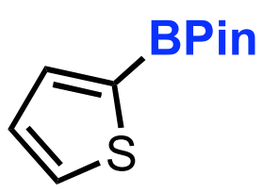
Br, **90% yield**
~ 540 mg
(0.2 M Ar-X)



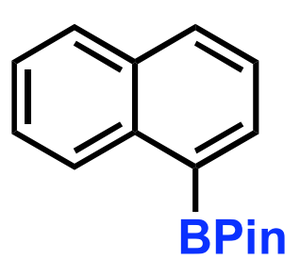
I, **93% yield**
~ 1010 mg



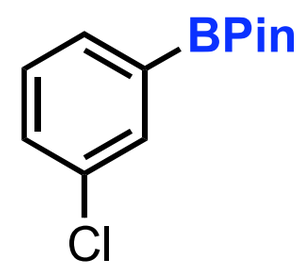
Br, **76% yield**
~ 825 mg



I, **78% yield**
~ 660 mg



Br, **93% yield**
~ 950 mg



I, **95% yield**
~ 900 mg

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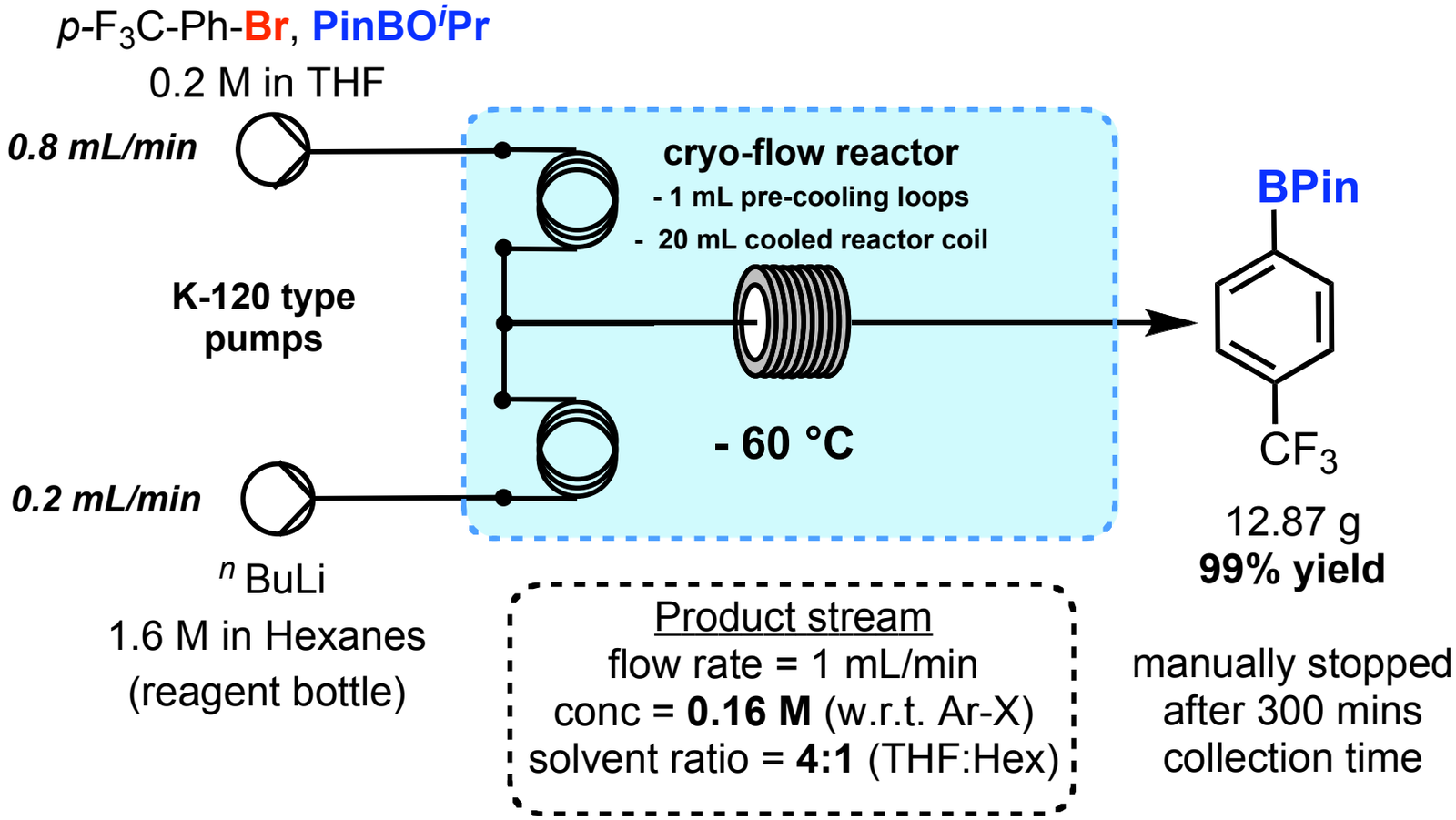
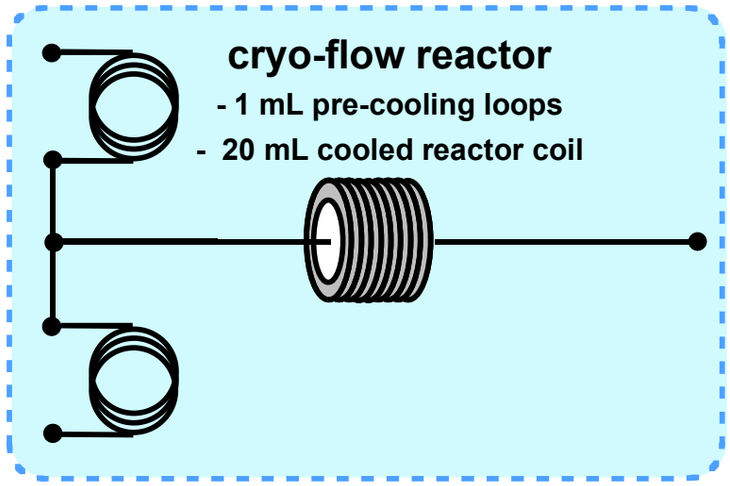


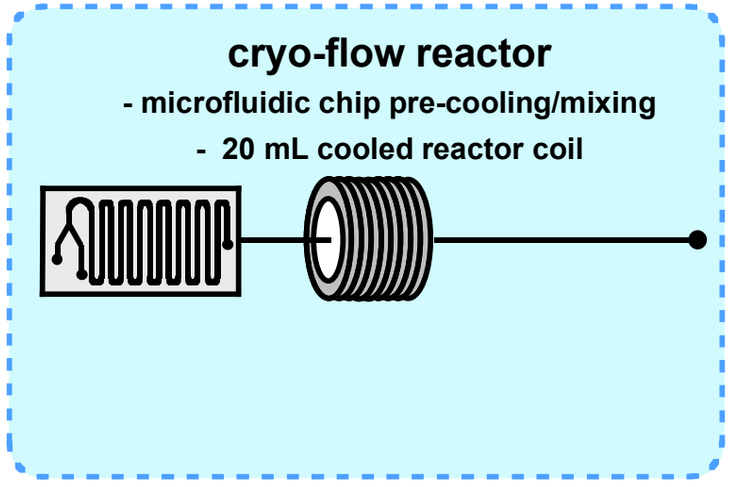
- 5 hours continual processing of *n*-BuLi through piston pumps

- 12 days continuous cooling to -60 °C with no notable frosting of the cooled parts!

Org. Lett. **2011**, accepted, awaiting proofs.

Further Advances and Developments





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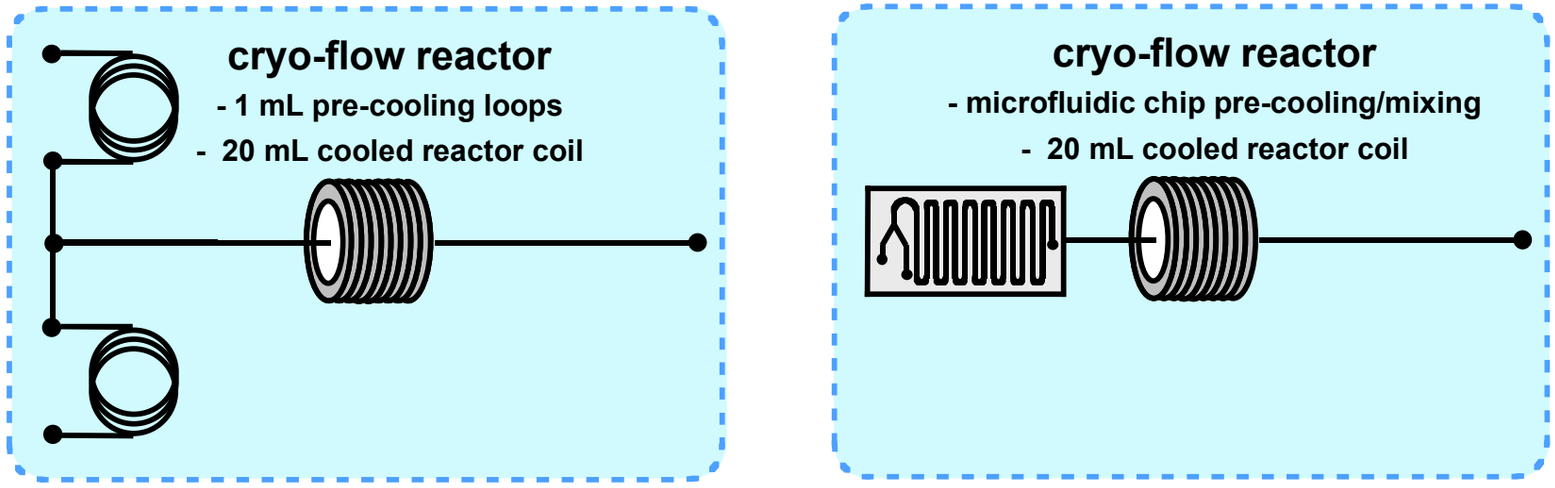

☑ Can you conduct low temperature chemistry in the Polar Bear?

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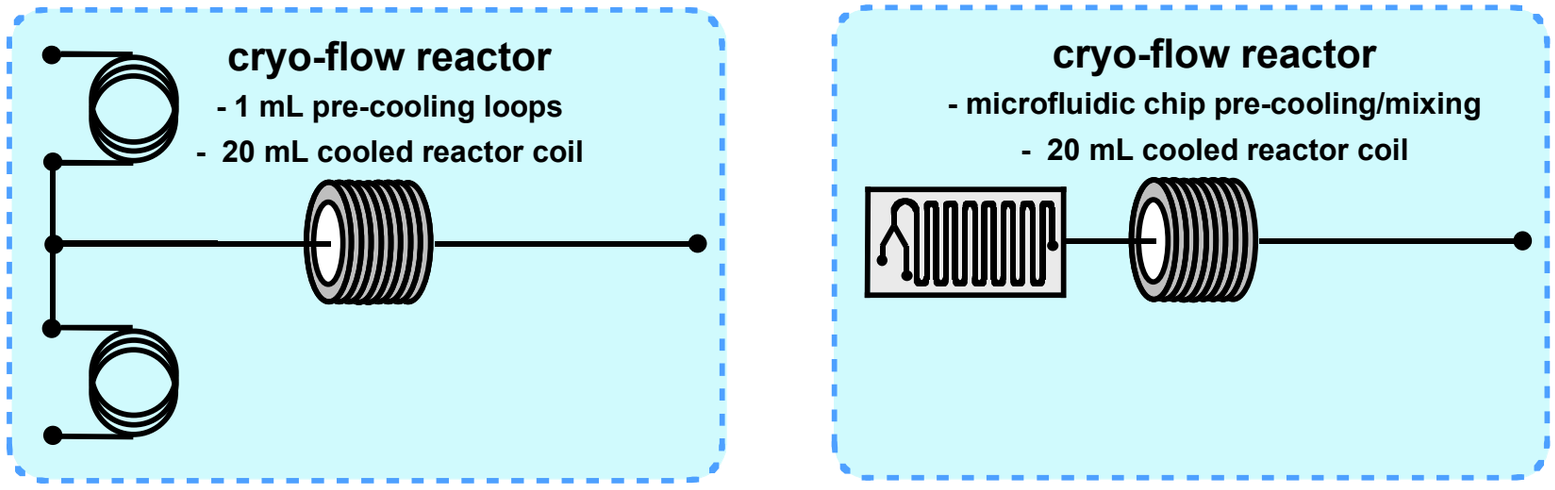

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- ☐ Explore the use of microfluidic chips.

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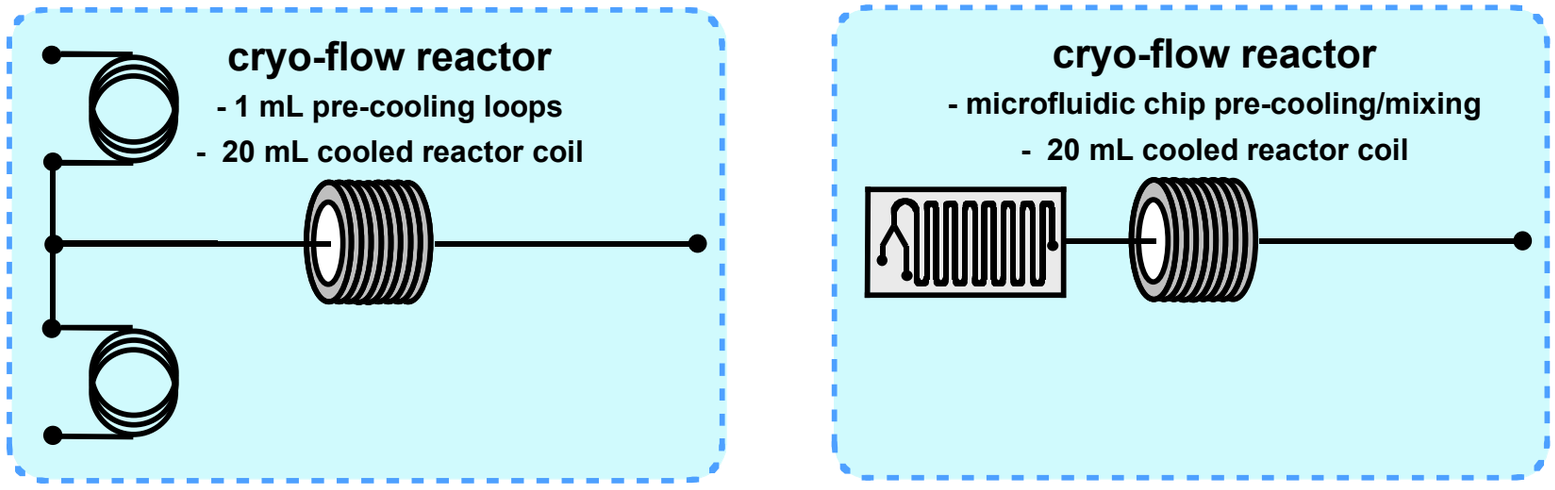

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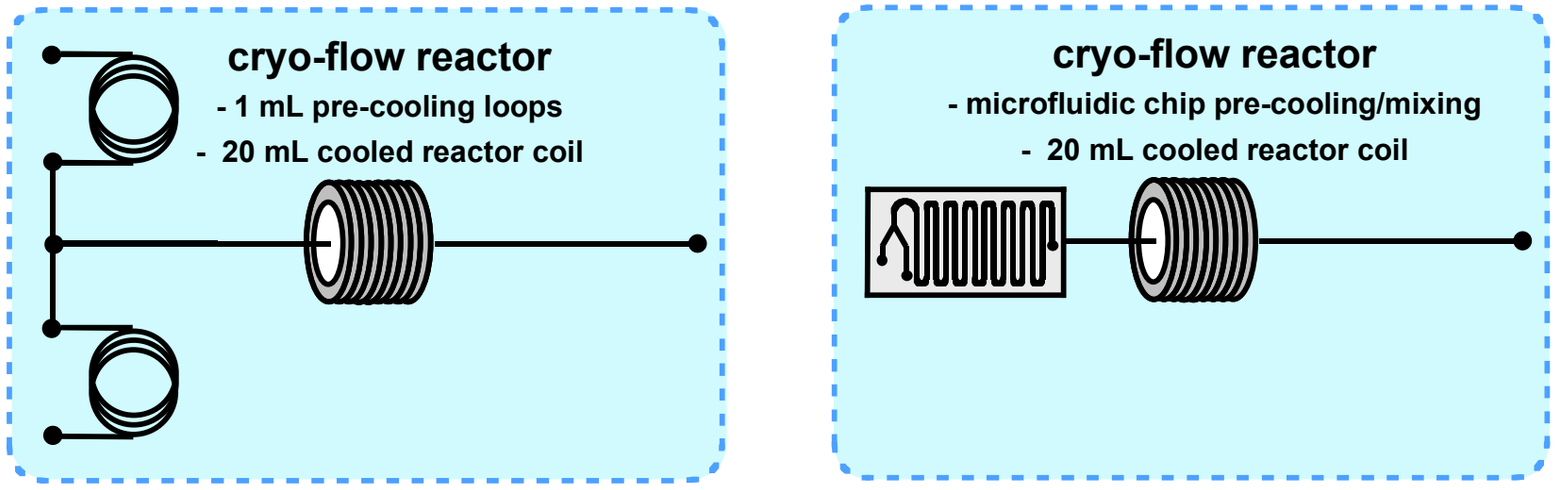

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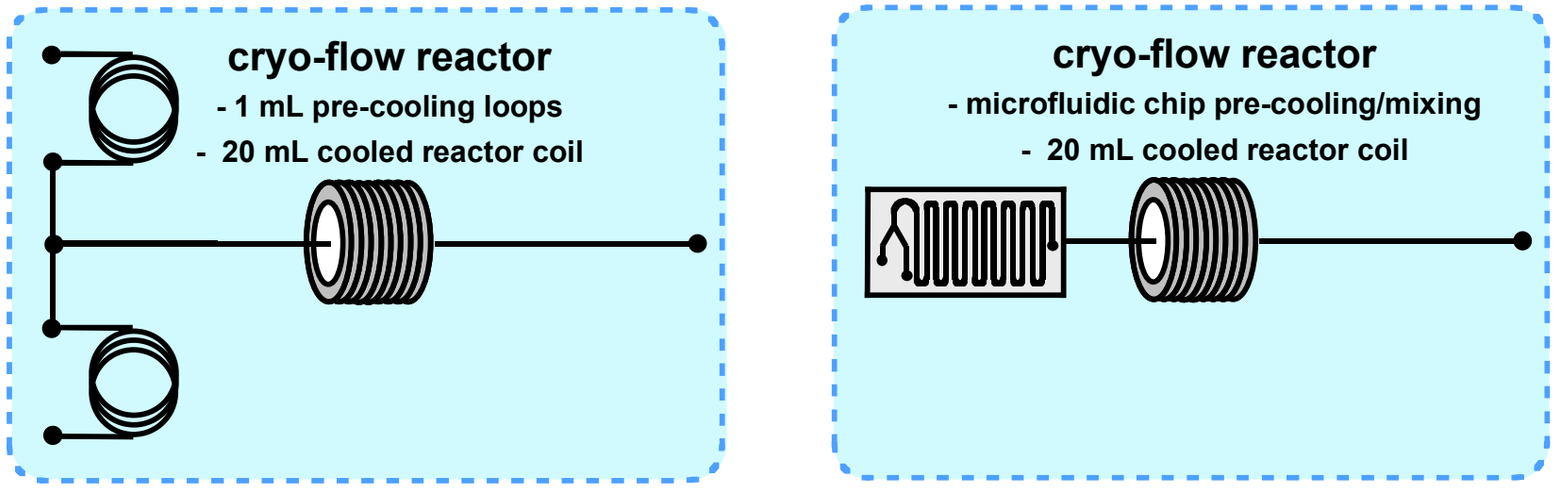

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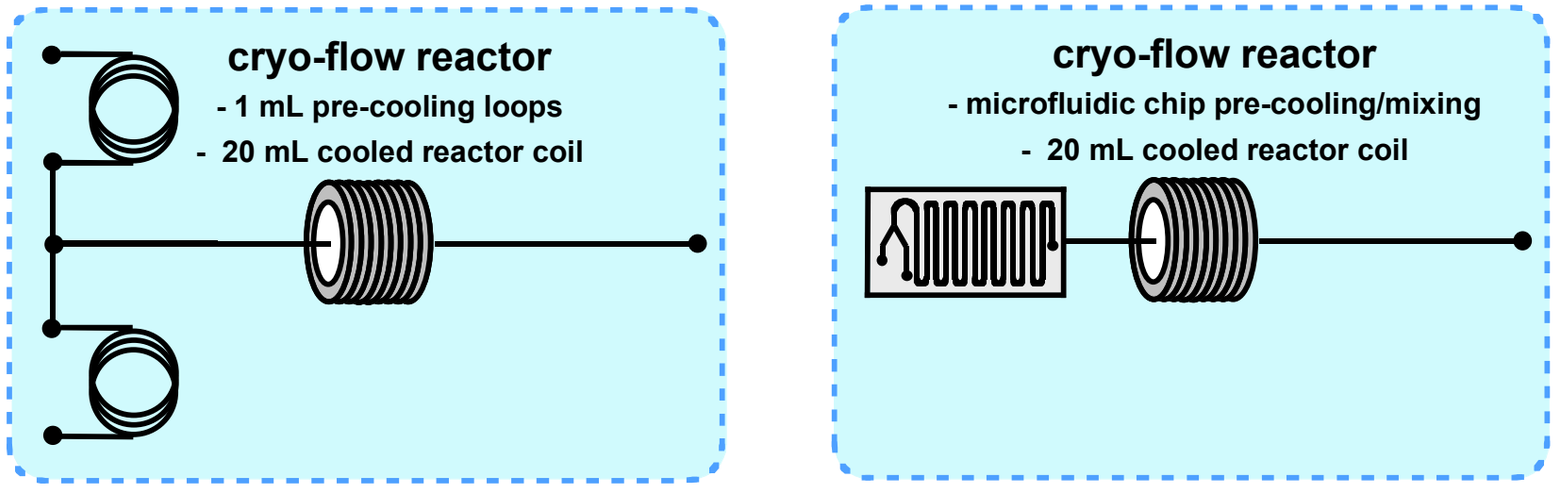

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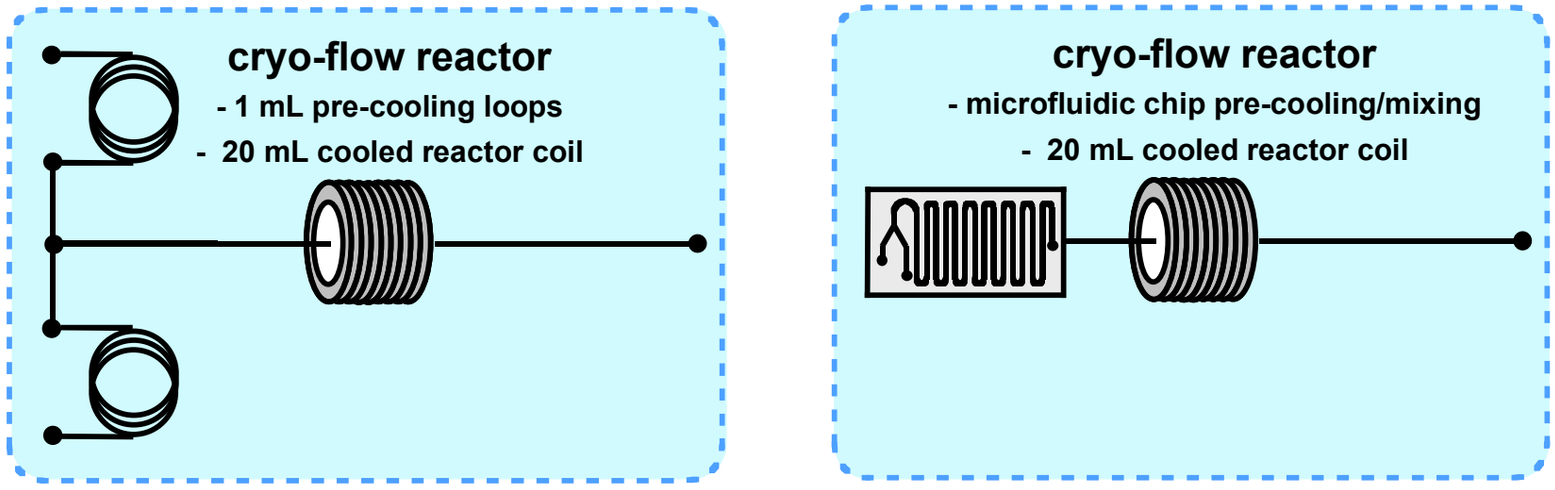

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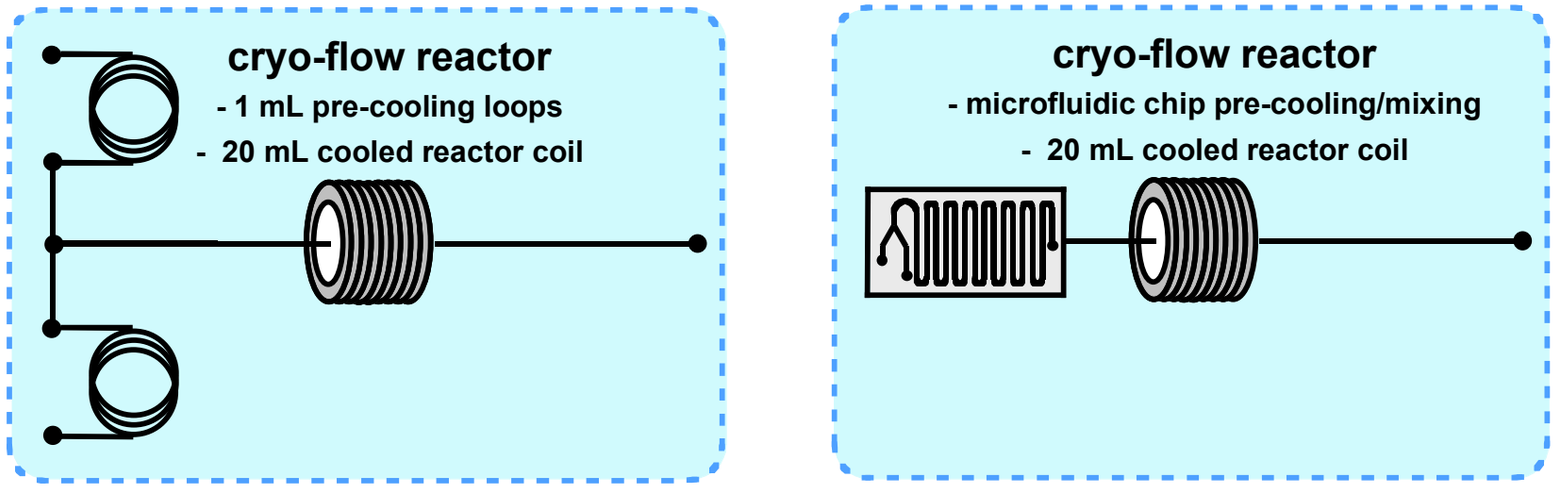

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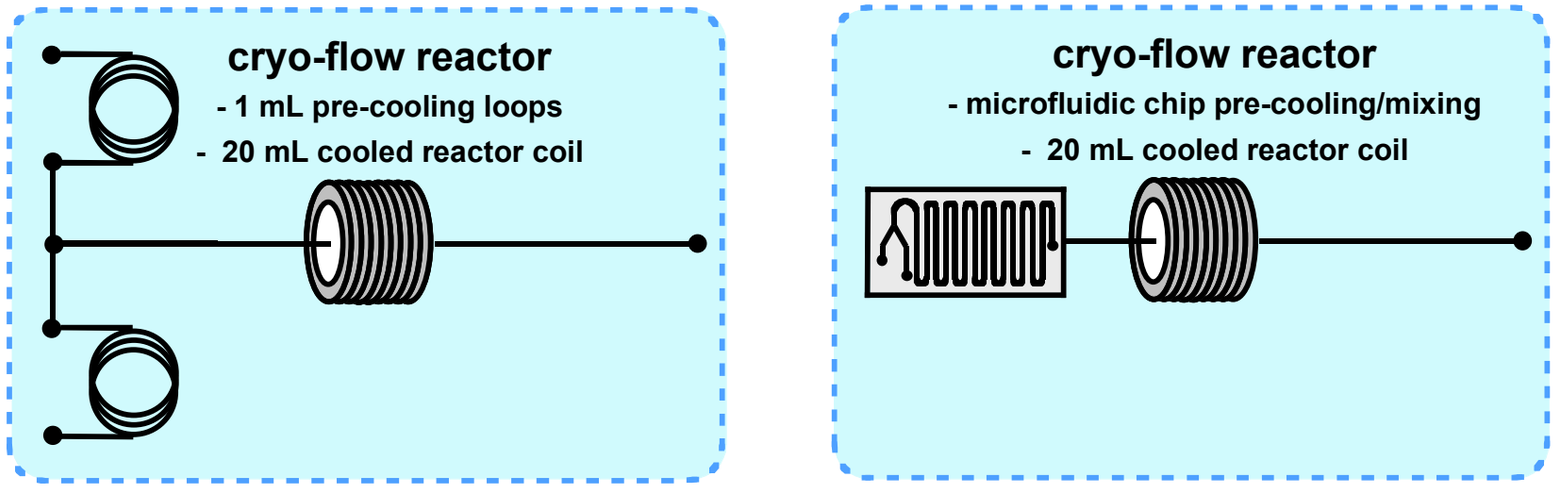

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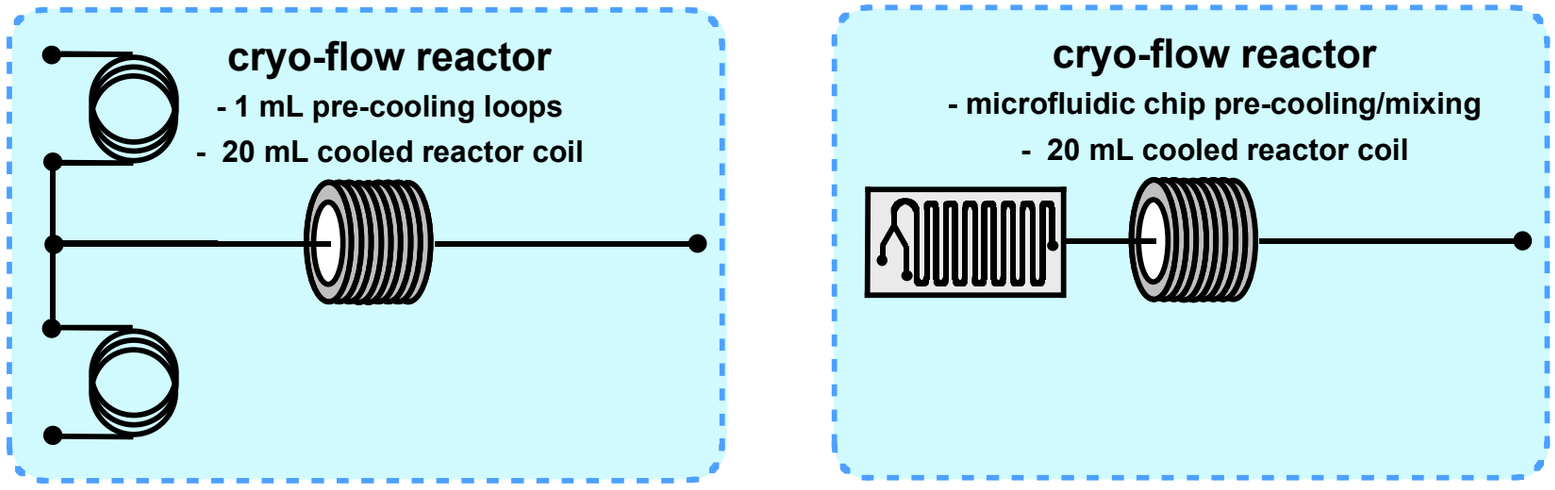

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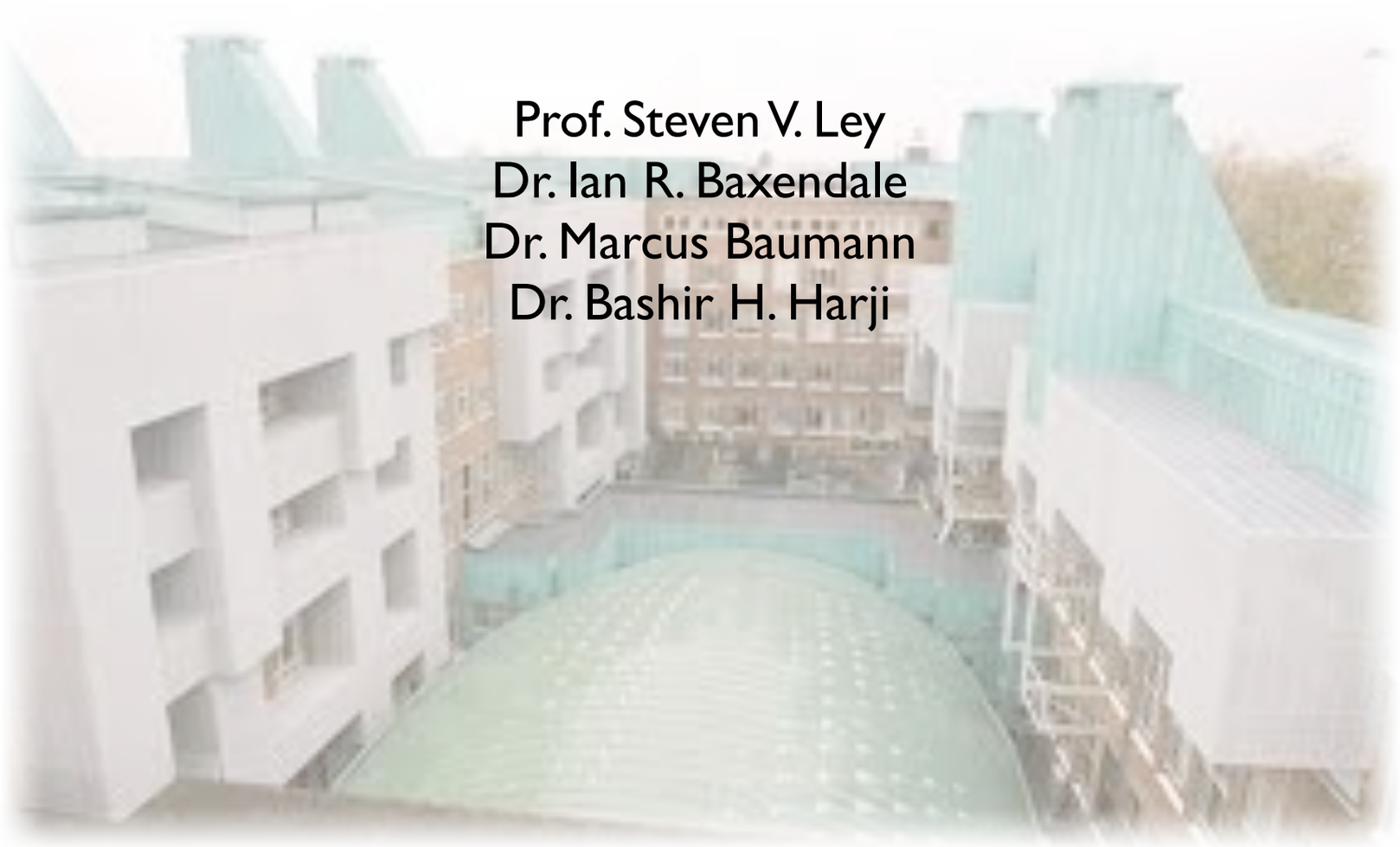
- ☐ Use the output as a feedstock for another flow device.

Acknowledgements



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