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Centre for Innovative Manufacturing
in Continuous Manufacturing and Crystallisation



Integrated platforms for chemical synthesis and crystallisation

Victor Sans

Cronin Lab

<http://www.chem.gla.ac.uk/cronin/>

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Engineering and Physical Sciences
Research Council

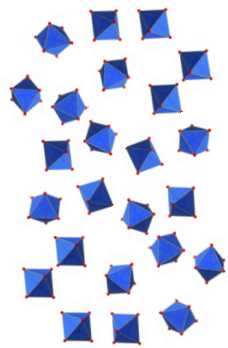


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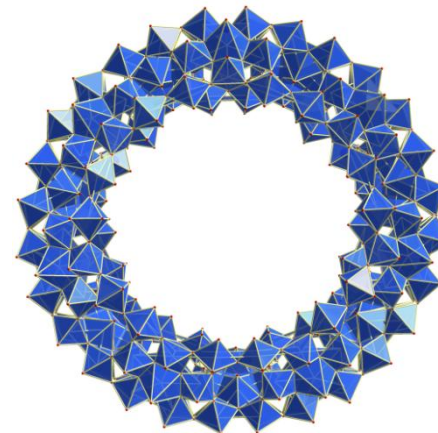
- Polyoxometalates are anionic oxide molecules derived from partially reduced early transition metals, usually Mo, W and V formed by self-assembly under acidic conditions
- Vast range of potential applications in biology, electronics, magnetism, catalysis
- Extremely dependent on reaction conditions



One-pot



- pH
- Temperature
- ionic strength
- reducing agent



2

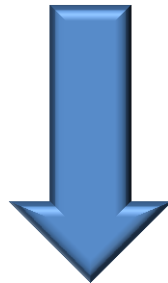


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- Difficult to synthesise/crystallise
- Low yields/poor reproducibility
- Labour intensive
- Harsh reaction conditions
- Discovery difficult and limited to specialised laboratories



Automated flow set-ups have the potential to overcome these limitations.

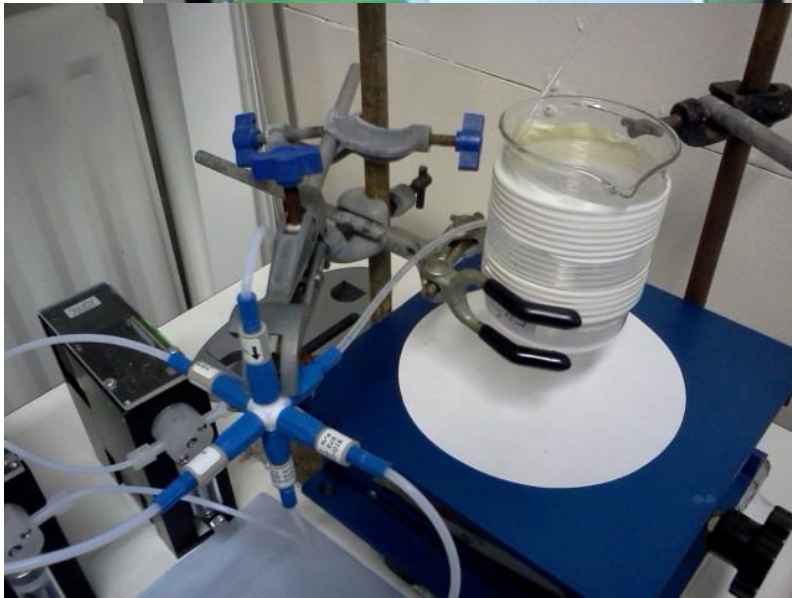


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CMAC

The 'CMAC' experiment



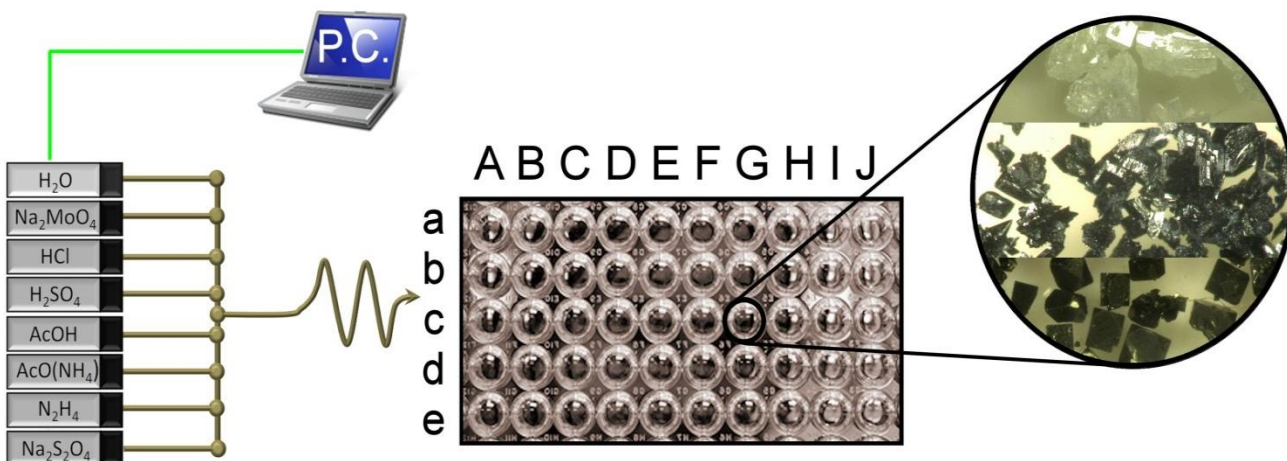


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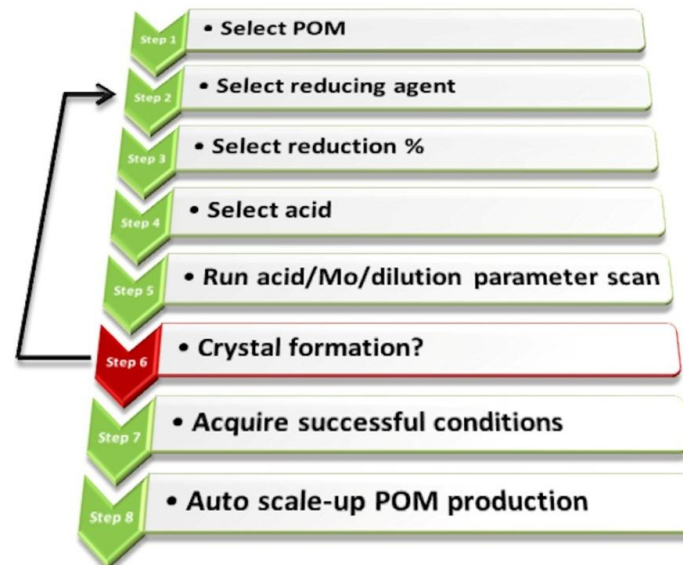
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Linear Flow System



	Flow rate ratios		Acid:Molybdenum ratio									
			0:10	1:9	2:8	3:7	4:6	5:5	6:4	7:3	8:2	9:1
	H ₂ O	Reagents	A	B	C	D	E	F	G	H	I	J
a	8	2										
b	6	4										
c	4	6										
d	2	8										
e	0	10										



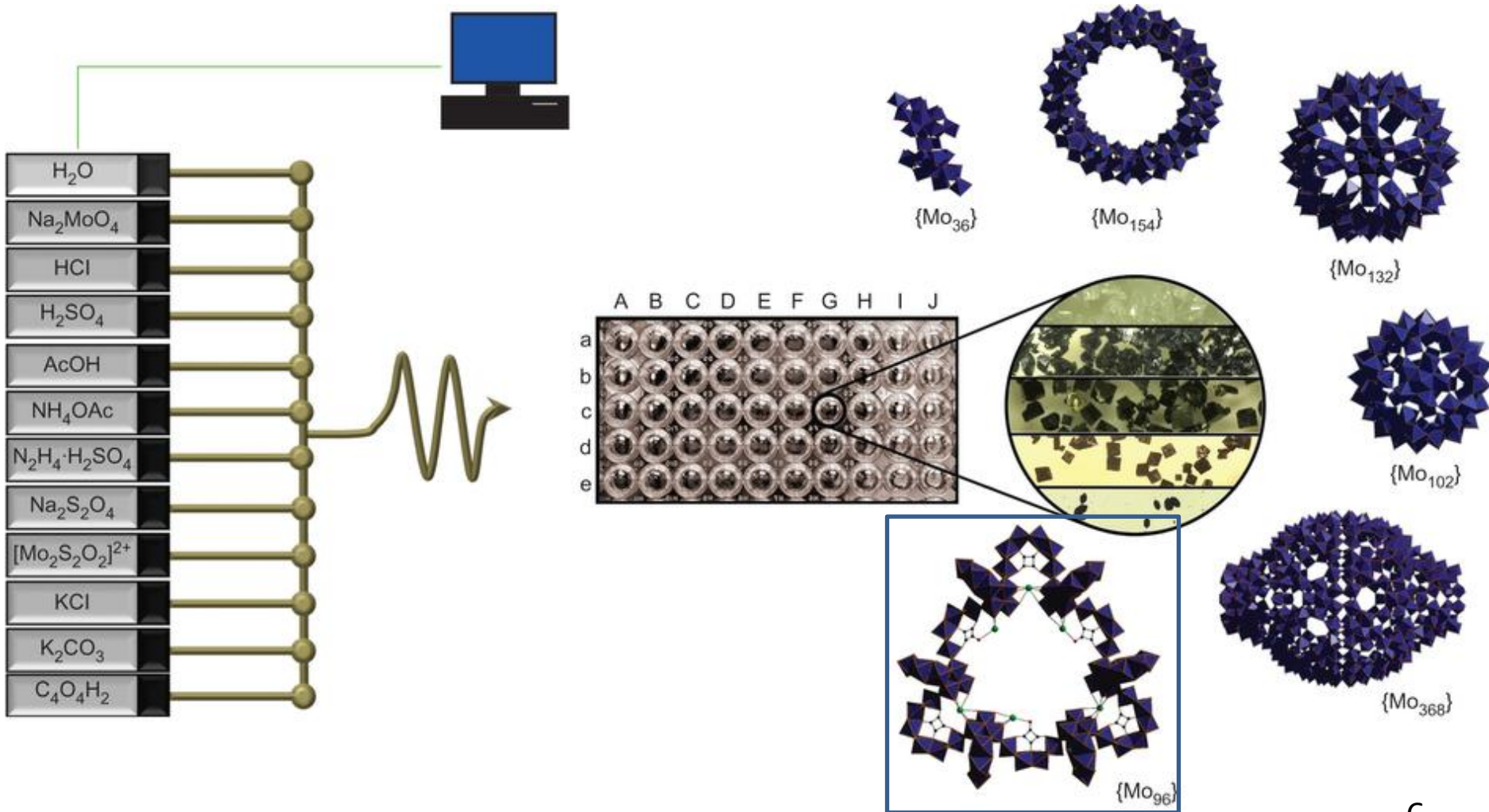


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Polyoxometalate discovery and scale-up



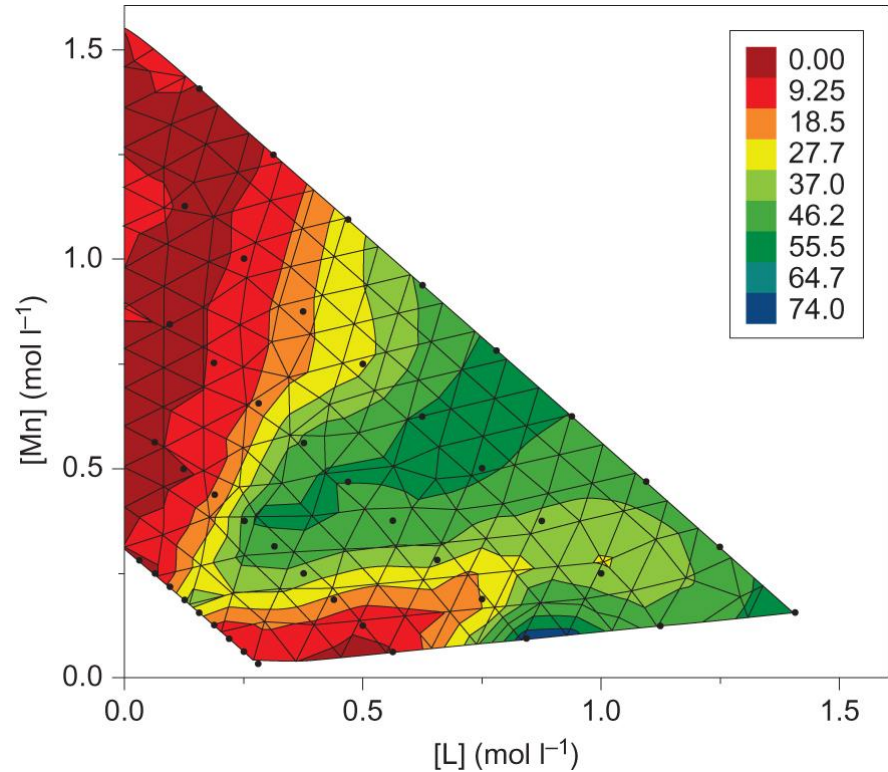
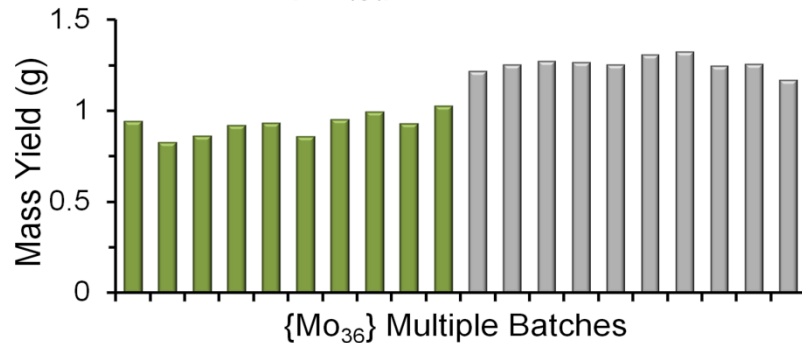
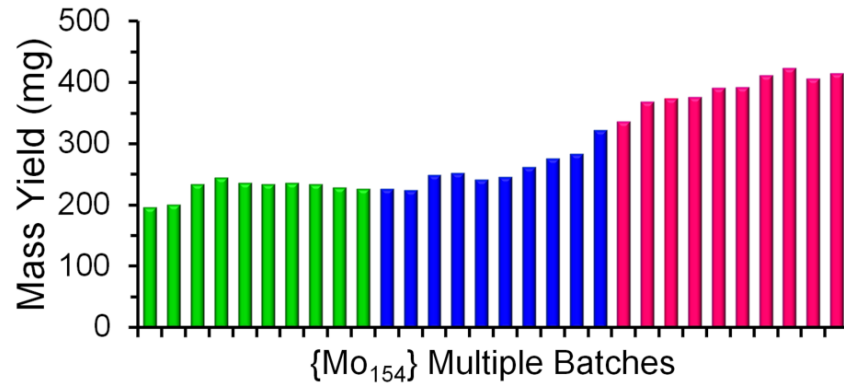


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Automated screening of reaction space



- Highly reproducible results
- Rapid optimisation of reaction conditions

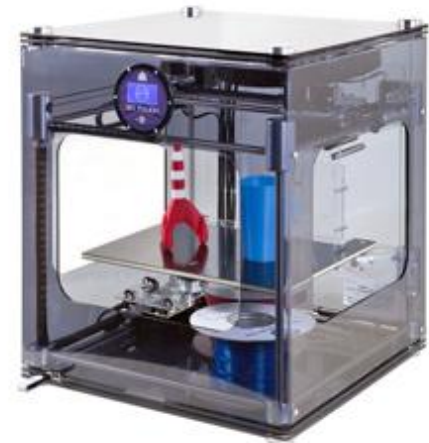
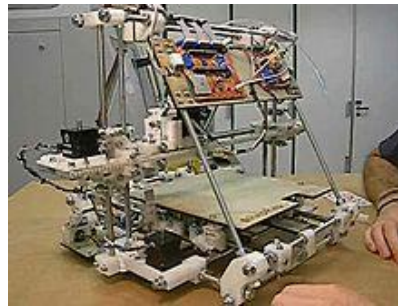
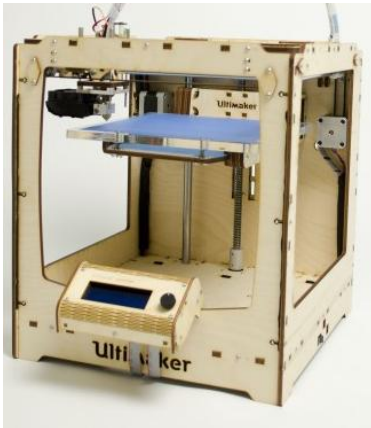
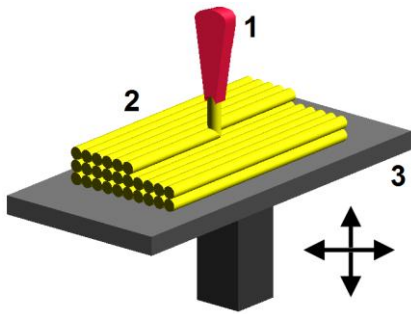


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3D-printing, design and fabricate at home



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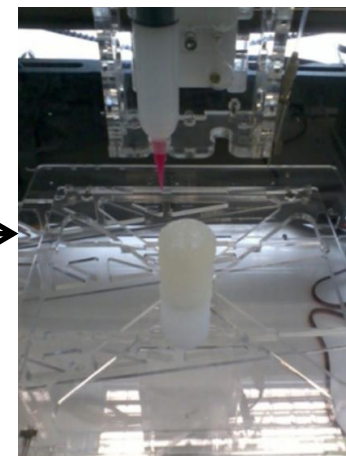
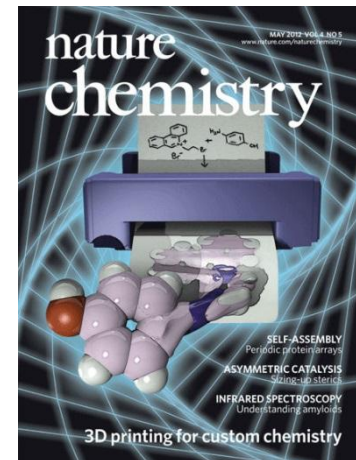
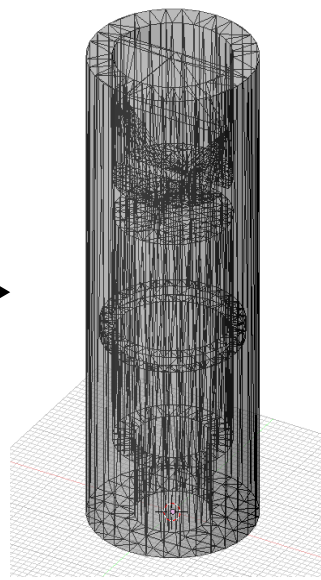
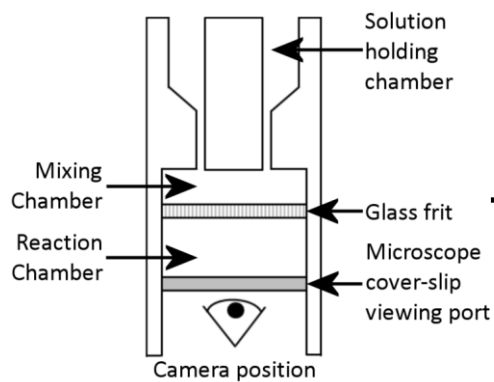
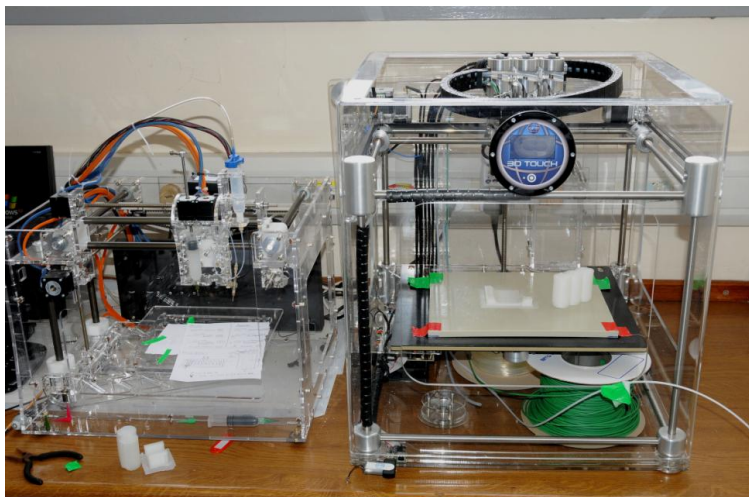


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3D-printing chemical 'reactionware'



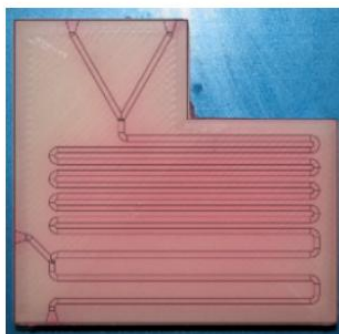
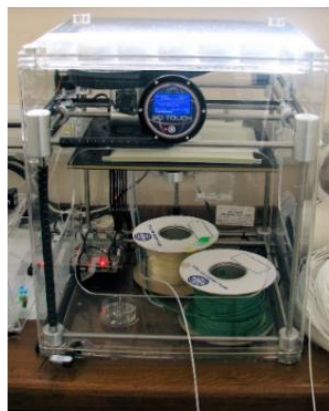
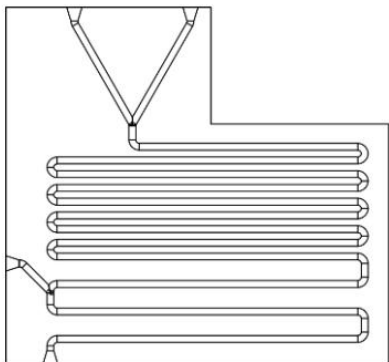


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3D-print your own reactors

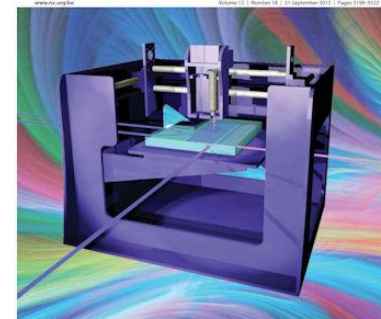


Lab on a Chip

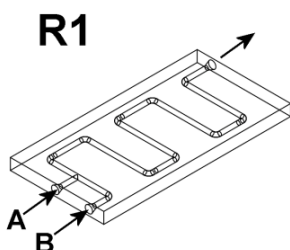
Miniaturation for chemistry, physics, biology, materials science and bioengineering

www.rsc.org/loc

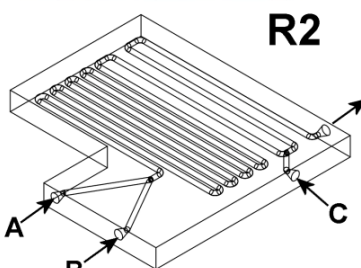
Volume 12 | Number 12 | 23 September 2012 | Pages 3267-3271



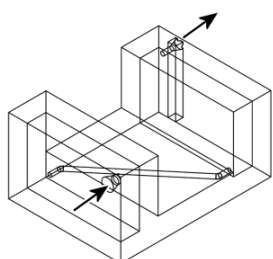
RSC Publishing



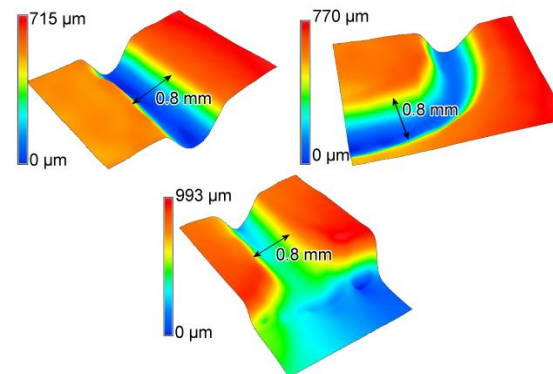
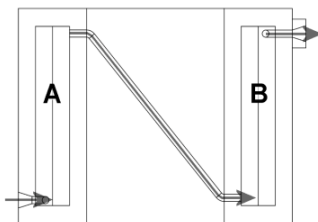
R1



R2



R3



	R1	R2	R3
Printing Time (h)	2	4	4
Reactor Weight (g)	3.9	8.8	13.4
Printing Materials Cost (US\$)	0.09	0.20	0.31
Dimensions (mm)	25x50x3	56x52x6	40x30x20

Lab Chip, 2012, 12, 3267-3271



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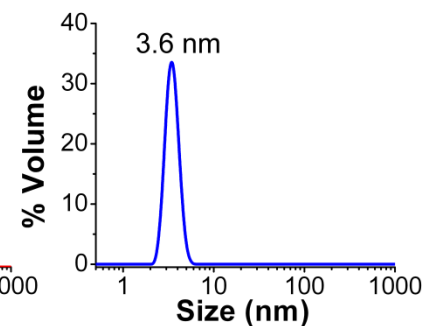
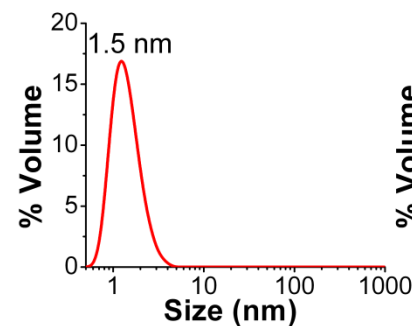
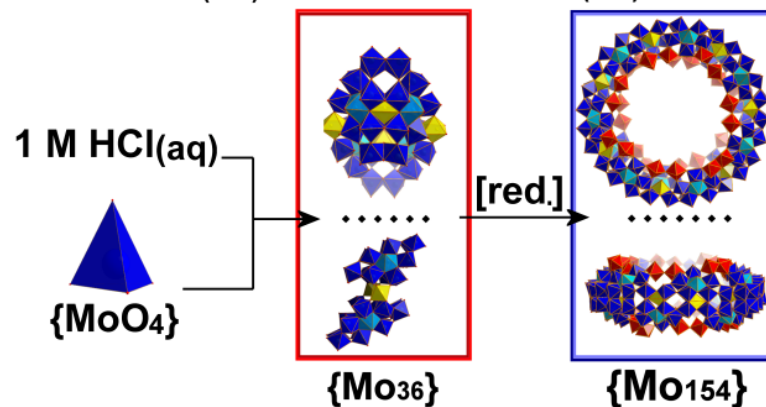
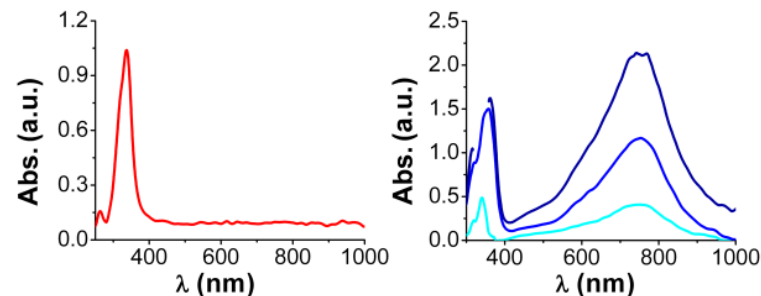
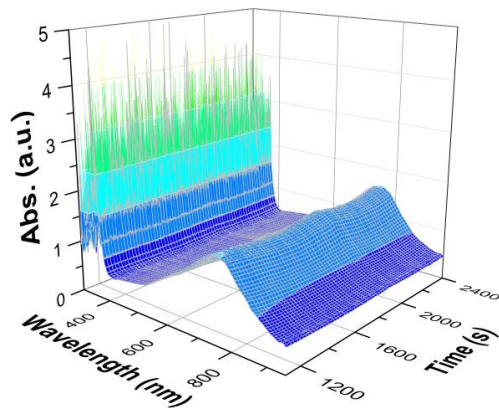
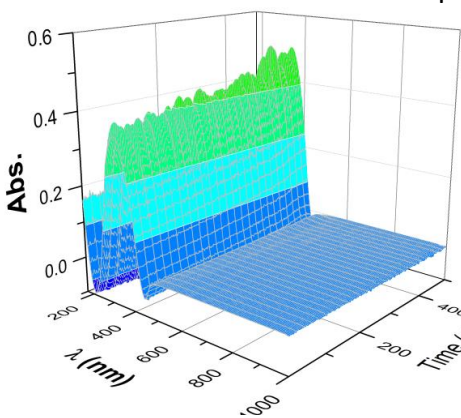
Inorganic chemistry in 3D-printed reactionware

$\{\text{Mo}_{36}\}$
 $\{\text{Mo}_{154}\}$



$\text{Na}_2\text{S}_2\text{O}_4$

$\{\text{MoO}_4\}$ HCl



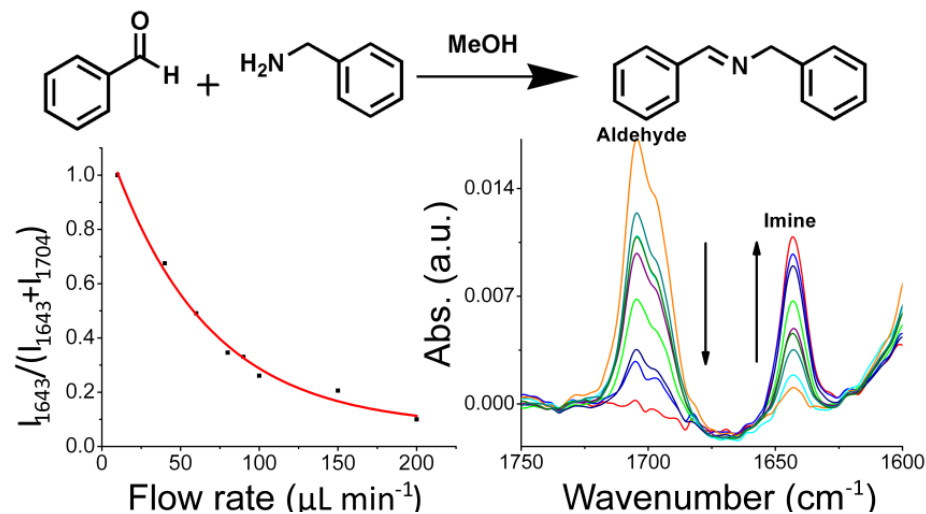
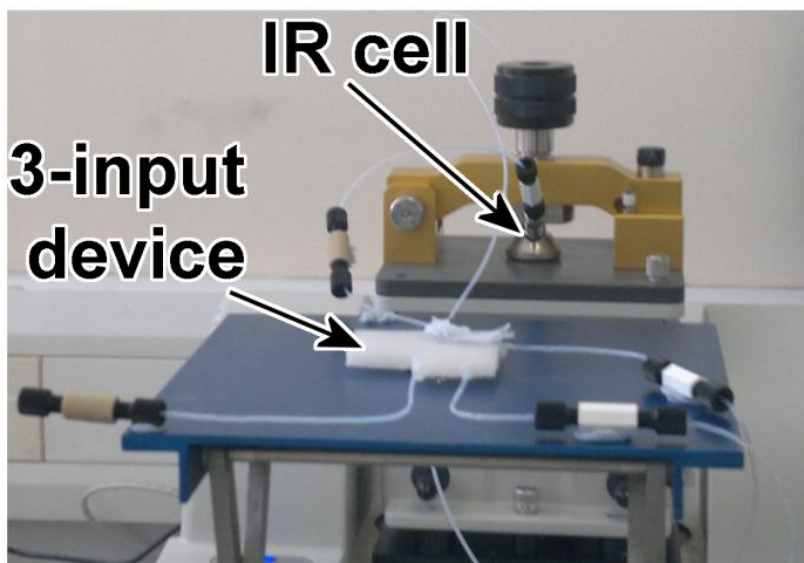


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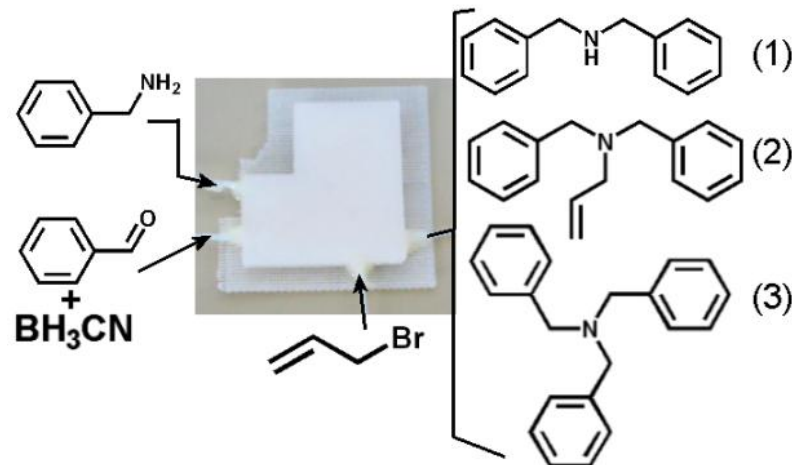
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Organic Chemistry in 3D-printed devices



- Continuous-flow synthesis of a simple imine.
- Rapid and facile kinetic study possible.
- Multi-step synthesis in one reactor.



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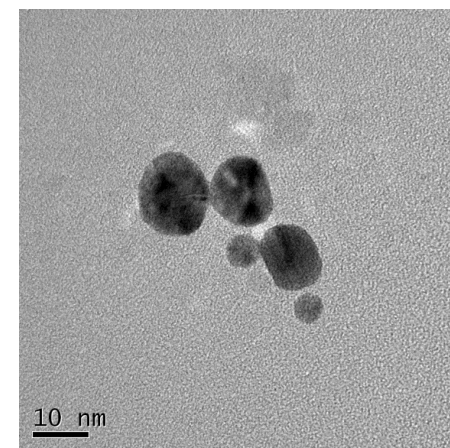
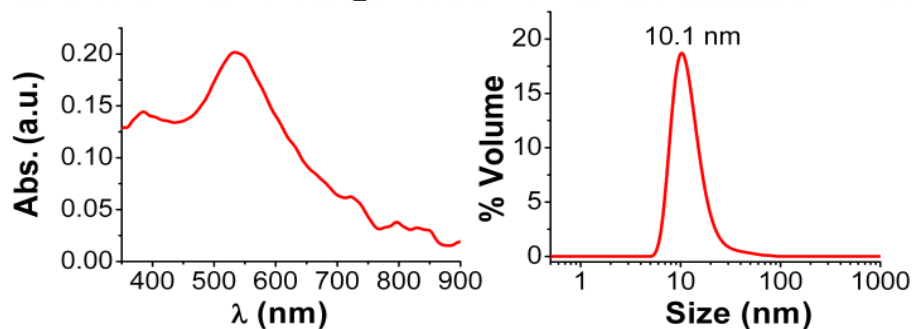
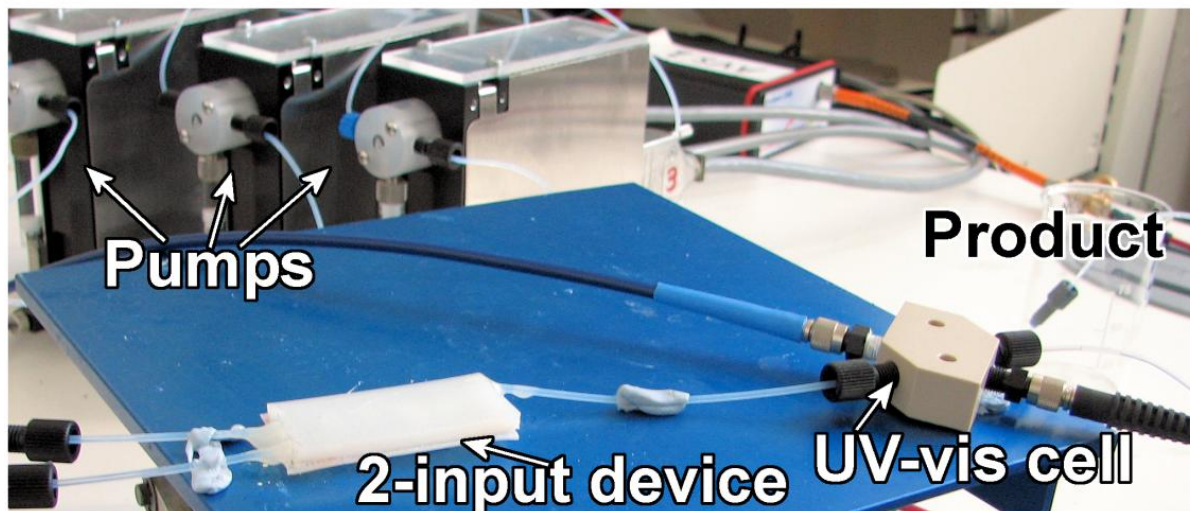


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Nanomaterials synthesis in 3D-printed reactionware

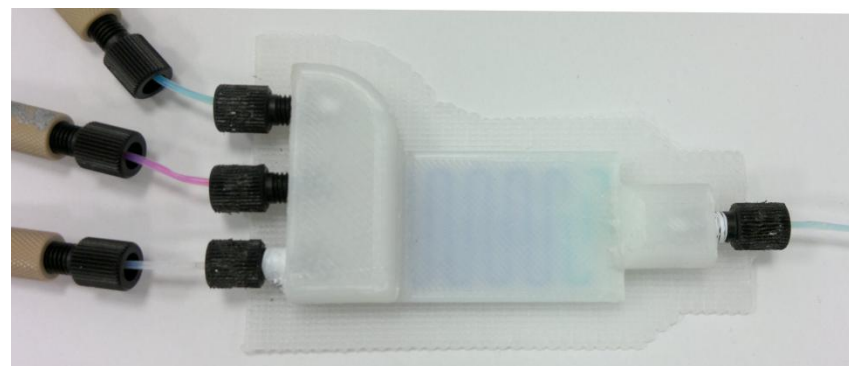
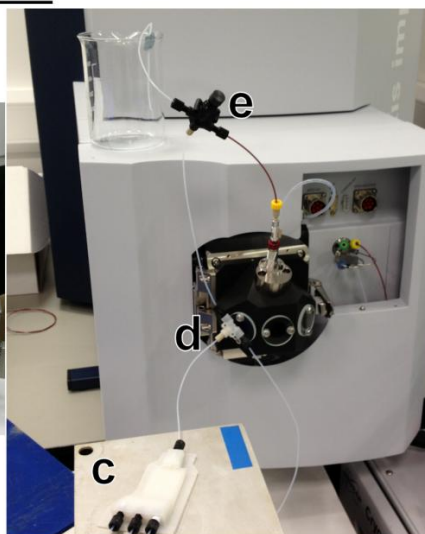
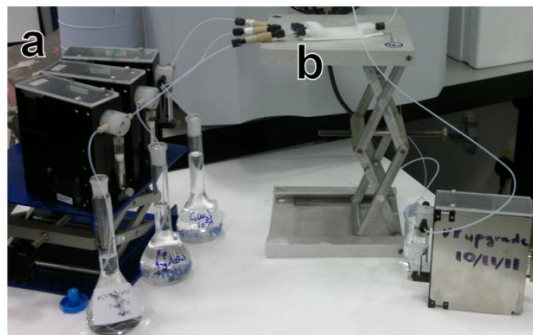
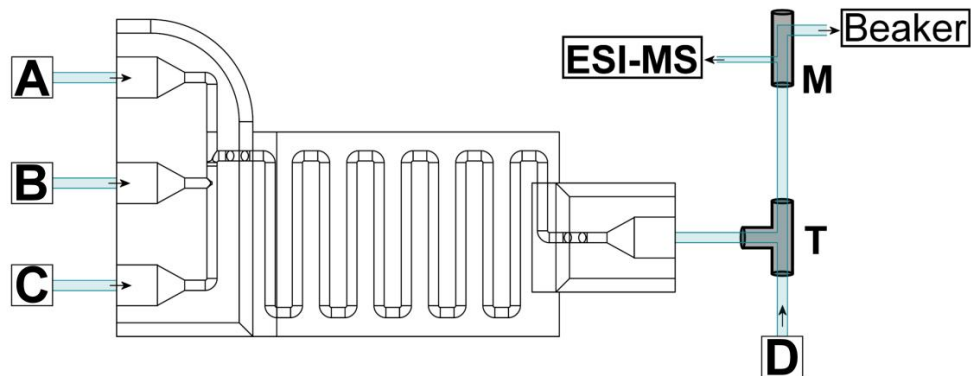


Synthesis of gold nanoparticles in flow mixing aqueous solutions of HAuCl_4 with NABH_4 -citrate.



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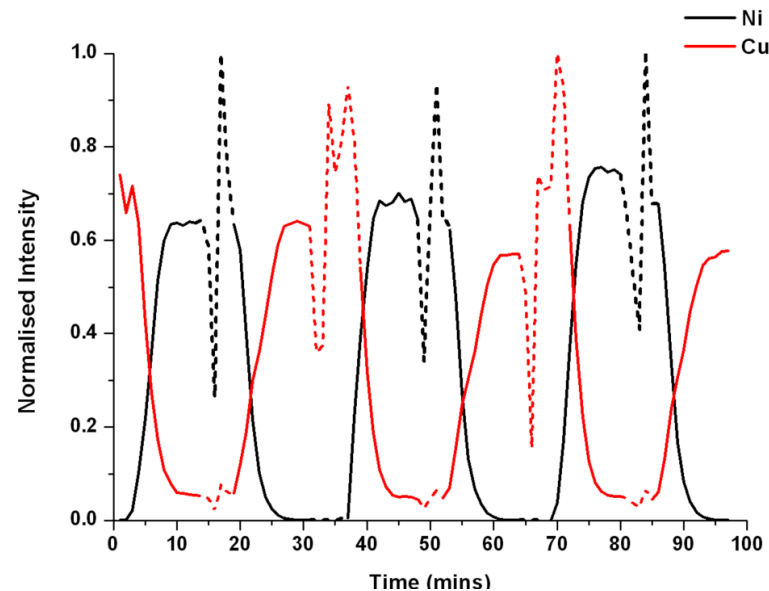
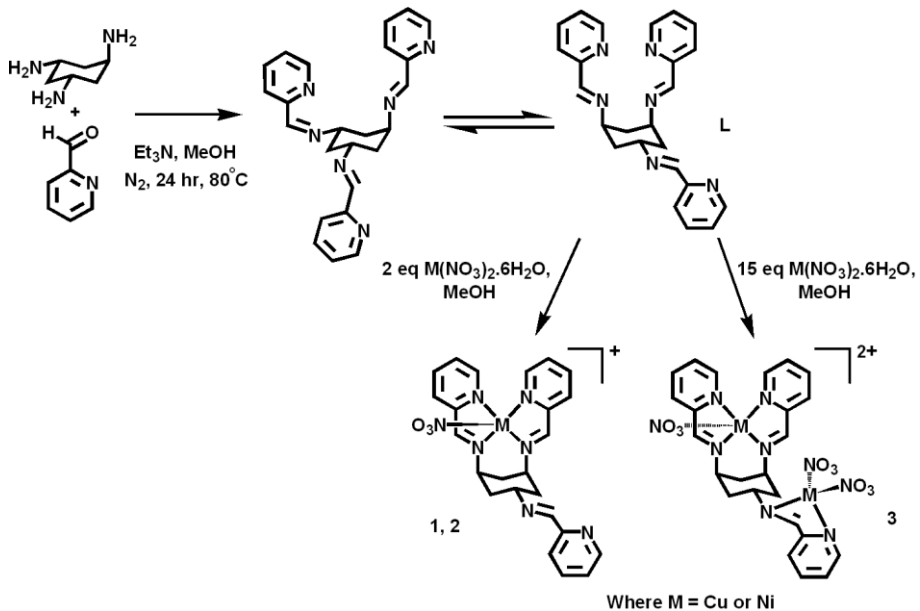
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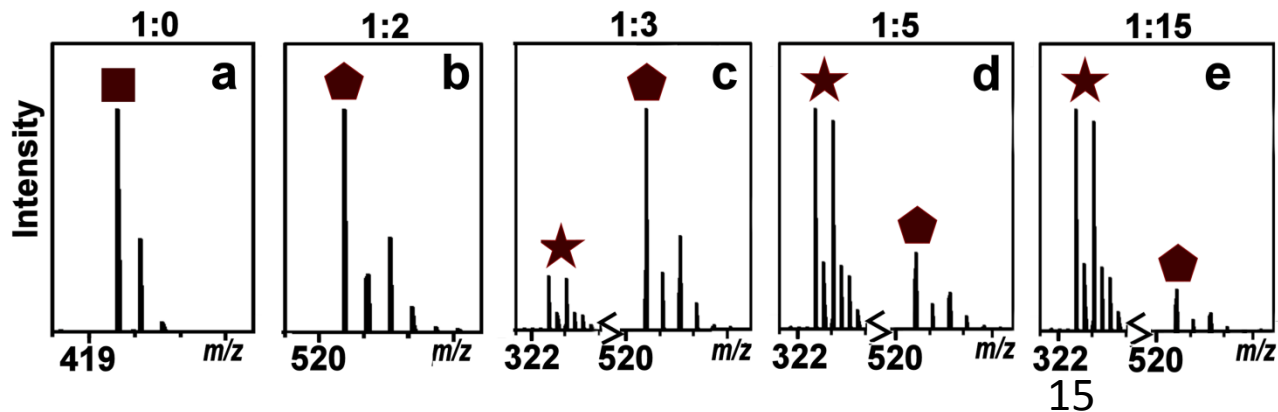


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Flow rate of $\text{Cu}(\text{NO}_3)_2$ →



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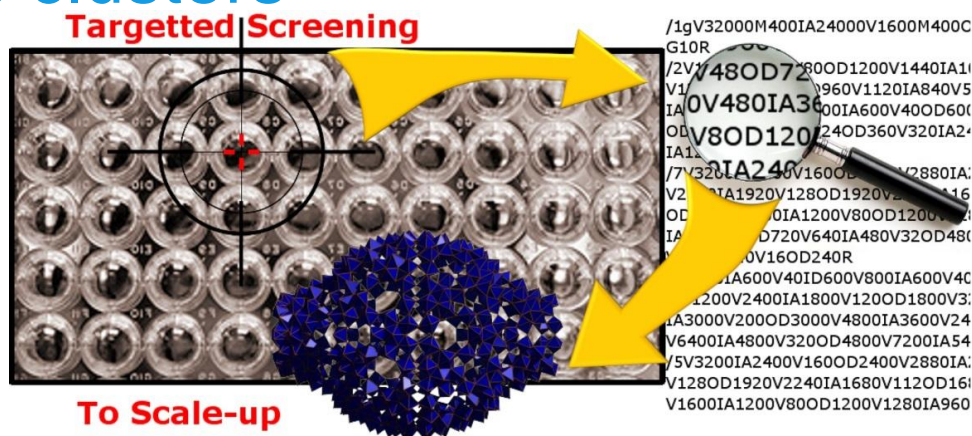


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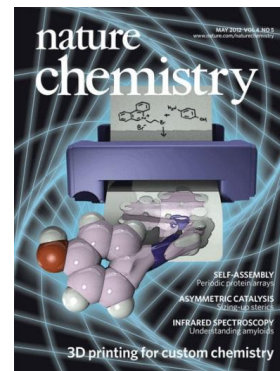
- Automated flow array for discovery and scale-up of inorganic clusters



- Flow chemistry in 3D-printed devices

<http://www.beilstein.tv/tvpost/3d-printed-millifluidic-reactionware-for-synthesis-and-analysis>

<http://www.beilstein.tv/tvpost/3d-printed-reactionware/>



Lab on a Chip





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Acknowledgements

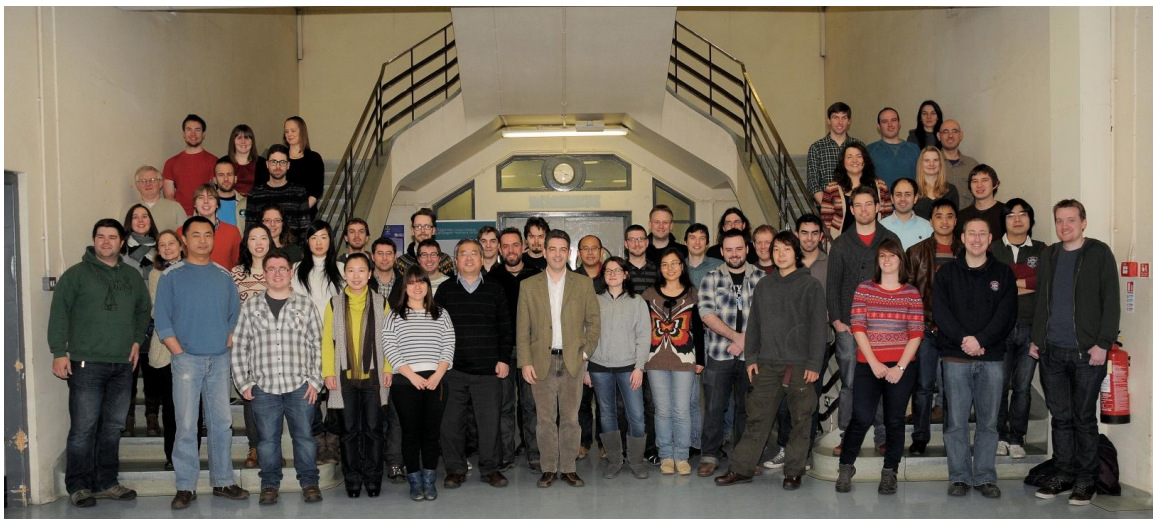
Prof. Lee Cronin

Dr. Craig J. Richmond Dr. Mali S. Rosnes

A. Ruiz de la Oliva Dr. Jennifer Boyle

V. Dragone Dr. De-Liang Long

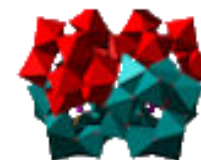
Dr. Phil Kitson Cronin group



The Cronin Group 2012/13



University
of Glasgow



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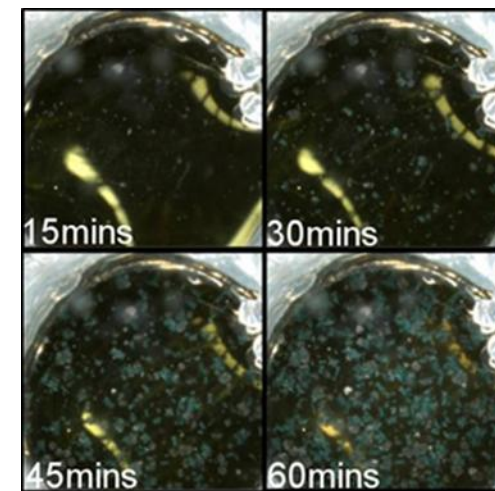
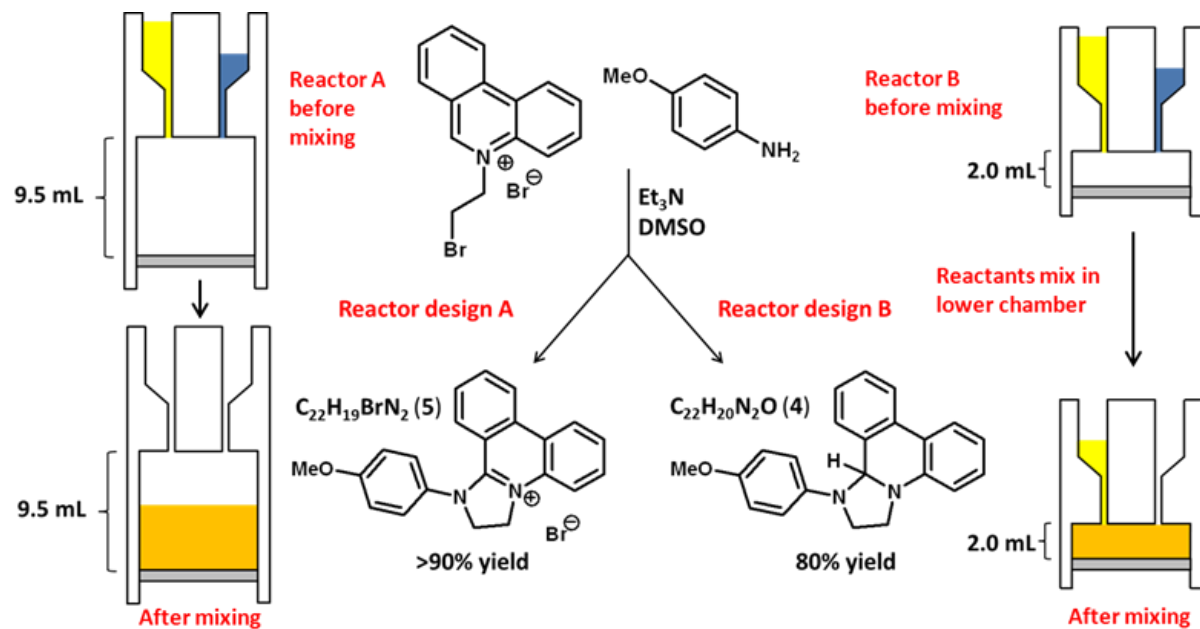
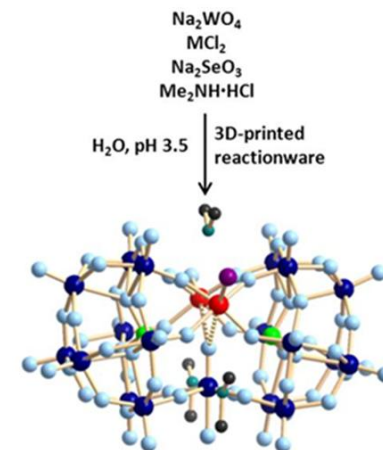
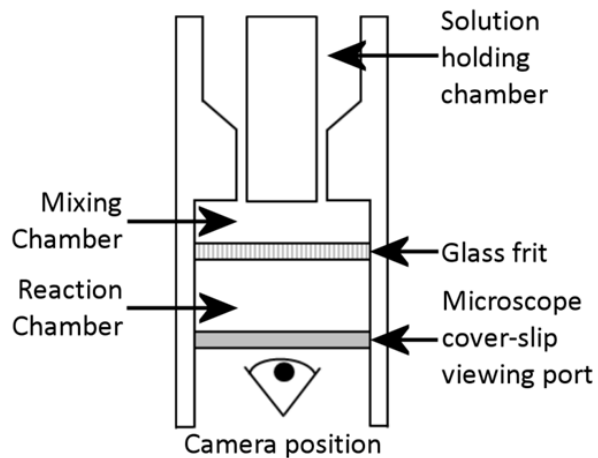
The Leverhulme Trust

Chemical synthesis in 3D printed reactionware

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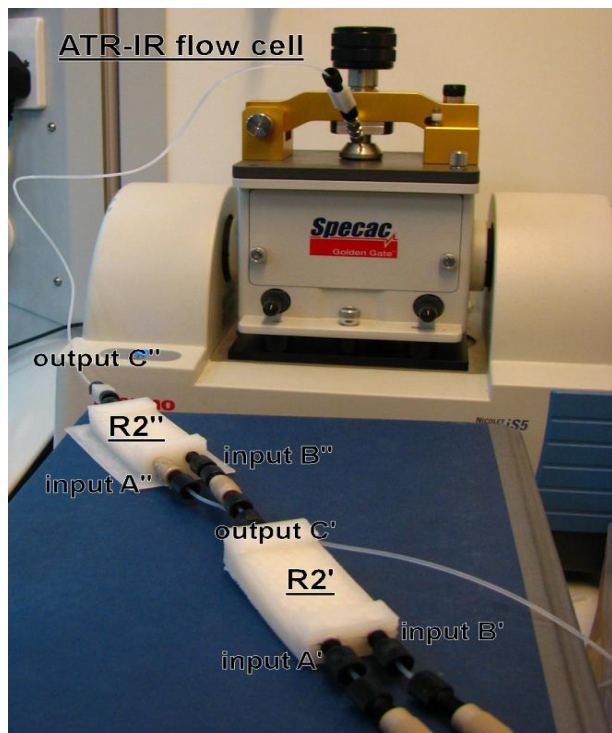
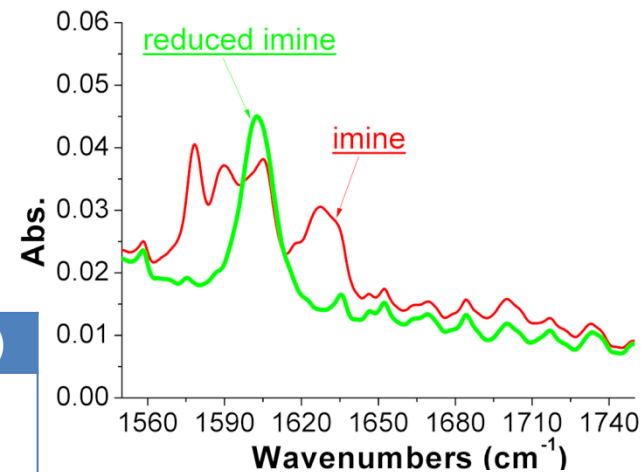
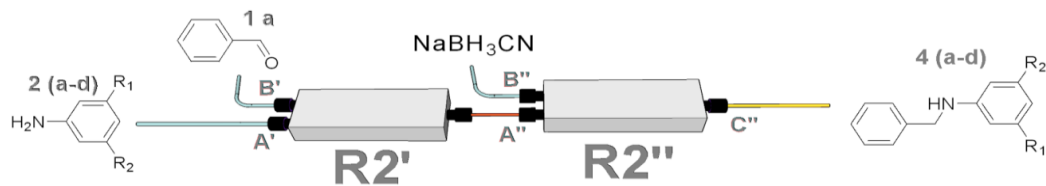
CMAC



Organic chemistry in 3D-printed reactionware

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Entry	Product	Yield (%)
1	 4a	78
2	 4b	99
3	 4c	96
4	 4d	97