

Q. How can I synthesize hundreds of supported metal catalysts per week to rapidly discover and optimize new catalysts?

By using the **Freeslate** Metals Impregnation System.

While much effort has been focused on the development of parallel reactors, true acceleration of heterogeneous catalyst development cannot be fully realized without the ability to prepare catalysts rapidly. Freeslate's capabilities in this area are unmatched and enabled by our flexible and configurable Core Module 3 (CM3, **Fig. 1**) platform. This robotic platform integrates unit operations of solid support dispensing, precursor solution preparation, solution pH measurement, and incipient wetness or other impregnation techniques. The Freeslate Metals Impregnation System (MIPS, **Fig. 2**) lets you synthesize hundreds of supported catalysts (**Fig. 3**) per week, producing high quality materials for screening in either high throughput reactors or larger scale individual reactors and allowing you to rapidly discover and optimize new heterogeneous catalysts.

Key Features

- Support dosing: Automated, gravimetric dispensing of solid support particles
- Precursor solution preparation: Automated liquid dispensing and dilution to prepare single or multiple component solutions at the desired concentration
- pH measurement: Automated pH probe allows for measurement and adjustment of pH in precursor solutions
- Particle fluidization during impregnation: High intensity shaker (**Fig. 4**) provides good fluidization of solid particles during impregnation to produce highly uniform materials
- Facile experiment design: Reagent amounts are calculated automatically based on the desired scale and percent loading of each component
- Exact process specification: All aspects of the recipe including order of addition, solvent, temperature, mixing time, and speed are specified independently for each component in the array-based library design
- Parallel dispensing: Multi-dispense tip increases throughput for the impregnation step allowing for multiple synthetic runs per day
- Integrated informatics: Freeslate LEA (Lab Execution and Analysis) software provides a comprehensive solution for experiment design, execution, and data capture and analysis, making it easy to correlate screening results with ratios and compositions created using the MIPS

Applications

- Hydroprocessing and other refining catalysts
- Biomass and sugar conversion catalysts
- Hydrogenation catalysts
- Broad range of supports including inorganic oxides and carbons
- Powdered supports, shaped supports, and extrudates
- Sol-gel chemistry
- Aqueous and organic solutions

References

- Hernandez-Pichardo et al., [Study By High-Throughput Experimentation Of The Effect Of The Pretreatment And Precursors On The Catalytic Activity Of Tungstated Zirconia Catalysts](#), *Catalysis Communications*, **2009**, 10, 1828-1834.
- Turner et al., [High-Throughput Heterogeneous Catalyst Research](#), *Surface Science*, **2009**, 603, 1763-1769.

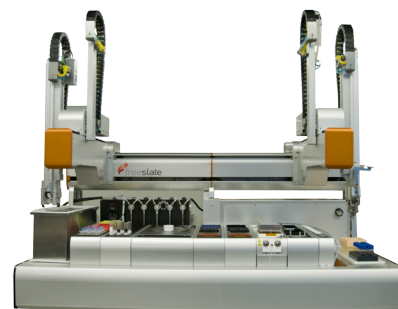


Fig. 1: Core Module 3 System.



Fig. 2: MIPS deck elements.

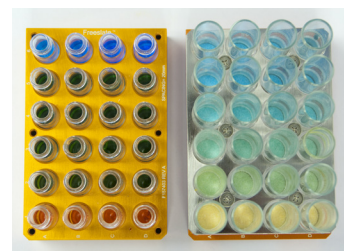


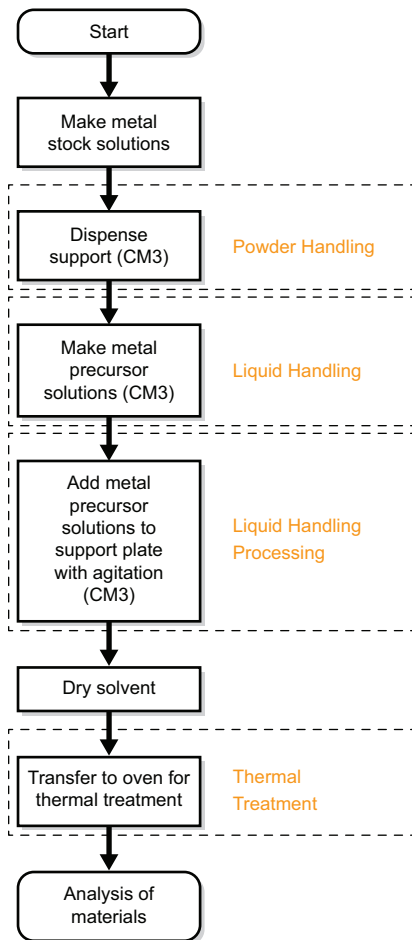
Fig. 3: Metal precursor solutions and finished catalysts.



Fig. 4: Vertical shaker.

Specifications

Representative Workflow



Functional Requirements

Vial/Plate Gripper

Vial handling:
Minimum vial diameter: 8 mm
Maximum vial diameter: 60 mm
Plate handling:
Maximum plate weight: 1000 g with grooves

4-Tip Liquid Dispenser

Dimensions (OD, ID, L): 2.03 mm x 0.76 mm x 130 mm
Variable fixed tip pitches: 9 mm, 13 mm, 30 mm
Extendible tip: 1
Syringe sizes: 50 mL to 10 mL

pH Probe

Range: pH 1-13
Resolution: 0.02 pH
Repeatability: ± 0.1 pH

Solid Dispenser

Learning algorithm allows system to remember optimum settings for materials
Flexible dosing system allows for large dynamic range of repeatable, automated dosing using:
Powdernium™ powder dispenser:
Traditional stirrer dispense mechanism
Hopper volume range: 10 mL to 100 mL
SV powder dispense:
Unique vibratory dispensing mechanism
Used for highly precise dispensing of small amounts of precious materials (as low as 0.5 mg tested)
Hopper maximum volume: 4 mL

*Contact Freeslate for a detailed list of all dispense conditions tested

Viscous Liquid Dispenser

Positive Displacement Tip (PDT) technology ideal for precise dispensing of organic solvents and highly viscous materials
Uses disposable tips from 10 mL to 1000 mL
Viscosity: 2 cP to 100 cP
Disposable tips: no washing required and no sample carryover
Accommodates widely available tips from Eppendorf and Rainin
*Contact Freeslate for a full list of supported PDT volumes

Balance

Weighing:
Maximum weight: 1200 g
Sensitivity: 0.1 mg
Readability: 0.1 mg full range
Repeatability:
At very high weight (measured >200 g): 0.25 mg
At low weight (measured up to 200 g): 0.15 mg
Response Time:
Medium accuracy setting: <8 s
High accuracy setting: <15 s
Camera:
Resolution: 1032 pixels (max wide) x 779 pixels (tall). Approx 9.2 pixels/mm

Functional Requirements

Heating/Cooling/Stirring Station

Heater Control, Zones 2 & 3:
Vial temperature range: Ambient to 180 °C
Vial temperature uniformity within each heated zone: 5 °C
Gradient between 2 zones: 20 °C max
Chiller, Zone 1:
Vial temperature range: -20 °C to 120 °C
Stirring
Maximum stirring speed: 750 rpm

Vertical Shaker

Parameters:
Amplitude adjustment: Manual
Frequency: 50/60 Hz (same as AC frequency)
Mass compatibility
Mass: Minimum mass 0.5 kg. Maximum mass 4 kg

Whisking

Whisk for break-up of aggregates and distribution of precursor

Capping Station

Vial capping/de-capping station with integrated cap storage for entire plates
Vial range: 2 mL to 250 mL
Height: 32 mm to 86 mm
*Contact Freeslate for a list of compatible vials

Facilities Specifications and Requirements

Envelope

Without enclosure:
Dimensions (W x L x H): 780 mm x 2060 mm x 1400 mm
Including enclosure:
Dimensions (W x L x H): 1016 (1500 with MMI) mm x 2430 mm x 2200 mm (includes integrated table)

Weight

Without enclosure:
~300 kg (depends on configuration, excludes the PC)
Including enclosure:
~450 kg (depends on configuration, excludes the PC)

Power

CM3:
US: 208 V +/- 10 %, 60 Hz, 20 A
EU: 220 V +/- 10 %, 50 Hz, 16 A
Computer:
US: 115 V +/- 10 %, 60 Hz
EU: 220 V +/- 10 %, 50 Hz

Gases

Requires: Clean Dry Air 0.5 MPa to 0.9 MPa (70 psi to 30 psi), 4 L/min