

Combining Lyophilization and Centrifugal Evaporation

Creating a fast drying process that results in soft, unattached & rapidly re-suspendable dry sample

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THE PROBLEM

Centrifugal Evaporation

- Method of choice for solvent removal with large numbers of parallel samples < 50 mls
- Fast, good for automation

However:

- Final dry sample often a “pellet” so slow to re-suspend
- Dry sample often attached to vessel

Lyophilisation

- “Fluffy” appearance
- Easy to re-suspend
- Sample not attached to vessel

However:

- Slow
- Pre-freezing required

EQUIPMENT USED

Evaporator

- Genevac HT4 for initial trials. Capacity for 4 x 5 samples
- Genevac HT12 for in experimentation with higher volumes. Capacity for 12 x 5 samples



HT4



HT12



Autoswitching condenser

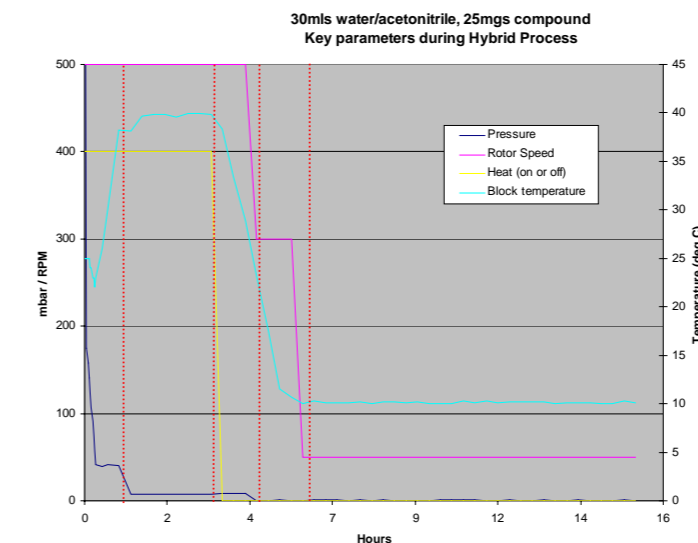
ORIGINAL PROCESS

Vessel : 40ml scintillation vials
Volume : 30mls
Solvents: acetonitrile/water 30/70
Drying : **48 hrs lyophilization**
Other : manual pre-freezing

HYBRID PROCESS

Vessel : 40ml scintillation vials
Volume : 30mls
Solvents: acetonitrile/water 30/70
Drying : **16hrs total runtime**
Other : No pre-freezing required

HYBRID PROCESS IN ACTION



- Stage 1 : Remove acetonitrile
- Stage 2 : Remove significant quantity of water
- Stage 3 : Cool water
- Stage 4 : Freeze water
- Stage 5 : Lyophilize remaining water

THE CHALLENGE

Create Hybrid Process

- Speed of centrifugal evaporation
- Final finish of Lyophilization
- Remove need for pre-freezing
- Make compatible with automated workflow

SPECIAL FEATURES

Control

- In run monitoring of sample temperature
- Ability to slow rotor considerably under software control, after solution is frozen
- Multi-step program, different processes can be combined into one automated run

DEVELOPMENT

Essentially iterative,

- Varied material of tube holders (polyprop/aly)
- Varied pressure, temperature, time profiles
- Used reference compound in each trial to ensure comparability of results

END RESULT

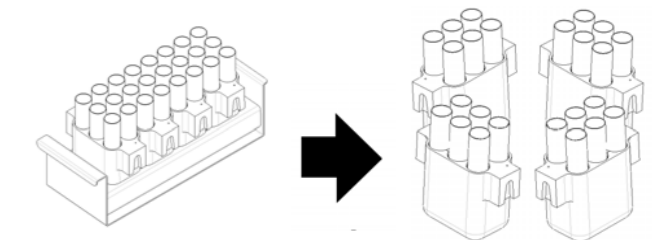
Fast & unmanned

- Tubes taken straight from fraction collector to evaporator
- 16 hr drying time – overnight
- Single instrument handling all sample throughput

RELATED WORK

Avoiding Tube Handling

- Segmented rack concept eliminates individual tube handling without necessitating huge evaporator



Replica of industry standard fraction collector rack, breaks apart into segments that can be placed straight into evaporator for drying.

- Racks have been designed to fit Gilson, Waters, & Shimadzu fraction collectors, and some liquid handlers.

FUTURE WORK

Easier method set-up

- Further research into ideal parameters
- Auto-programming of hybrid run based on details of application