

SFC Fraction Collection: Value of Simultaneous Evaporation

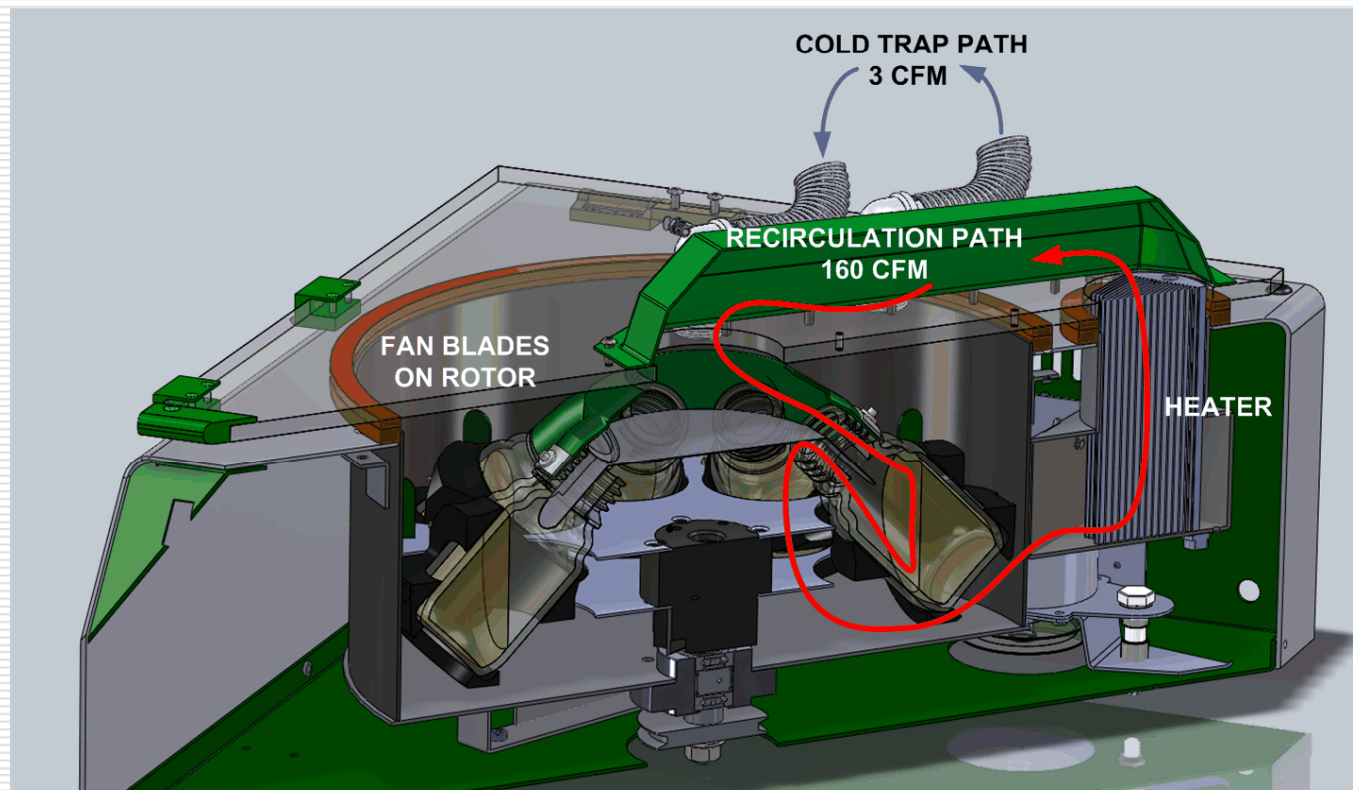
EAS 2009

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Herbert J. Hedberg



Centrifan RE & AF



No vacuum... Recirculating Evaporation... Like nitrogen blowdown except centrifugal force preserves sample mass/integrity



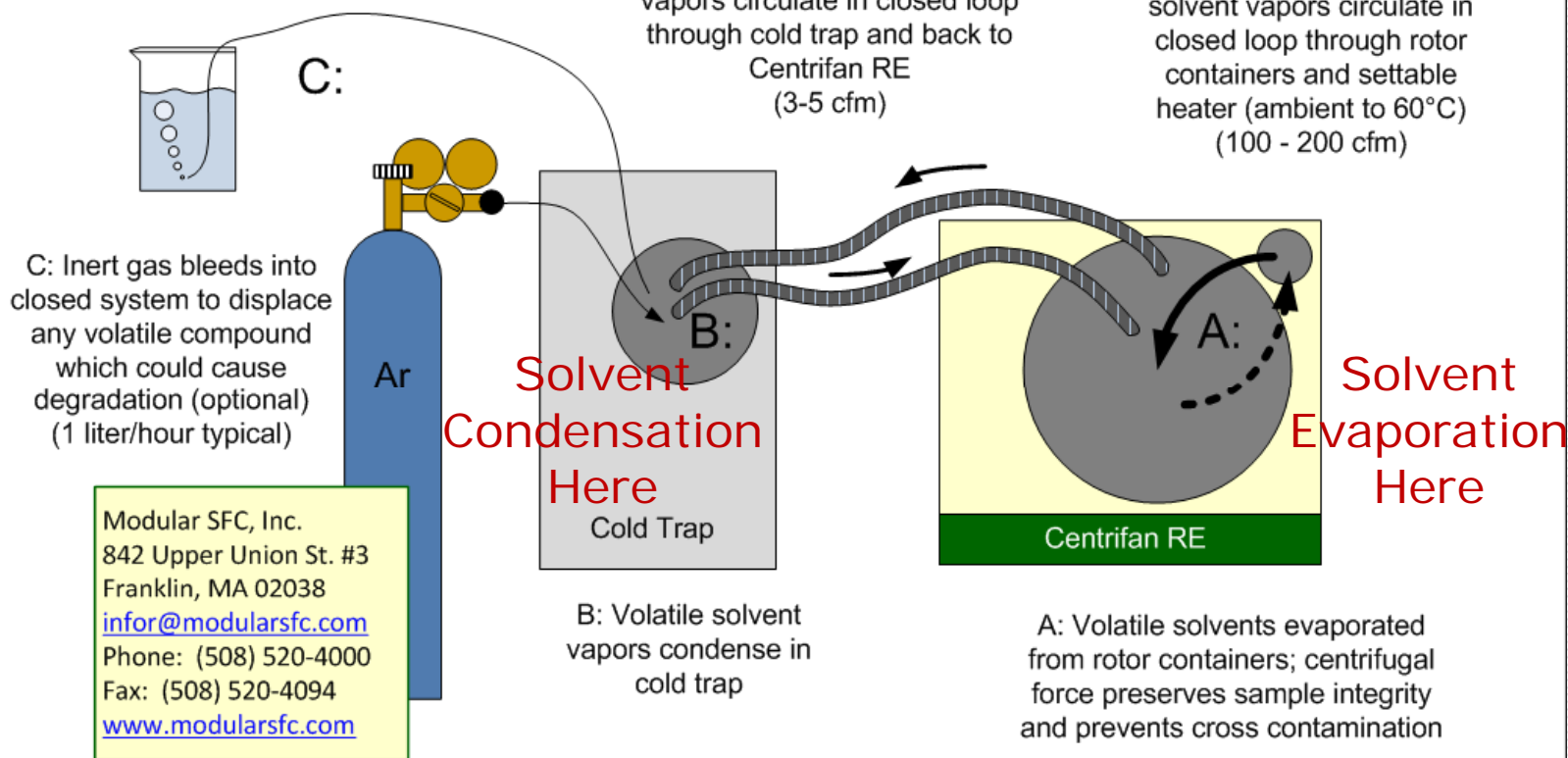
Centrifan™ RE with Cold Trap

Shows optional connection for inert gas bleed and vent

C: Inert gas excess vents to hood (recommended)

B: Inert gas and volatile solvent vapors circulate in closed loop through cold trap and back to Centrifan RE (3-5 cfm)

A: Inert gas and volatile solvent vapors circulate in closed loop through rotor containers and settable heater (ambient to 60°C) (100 - 200 cfm)

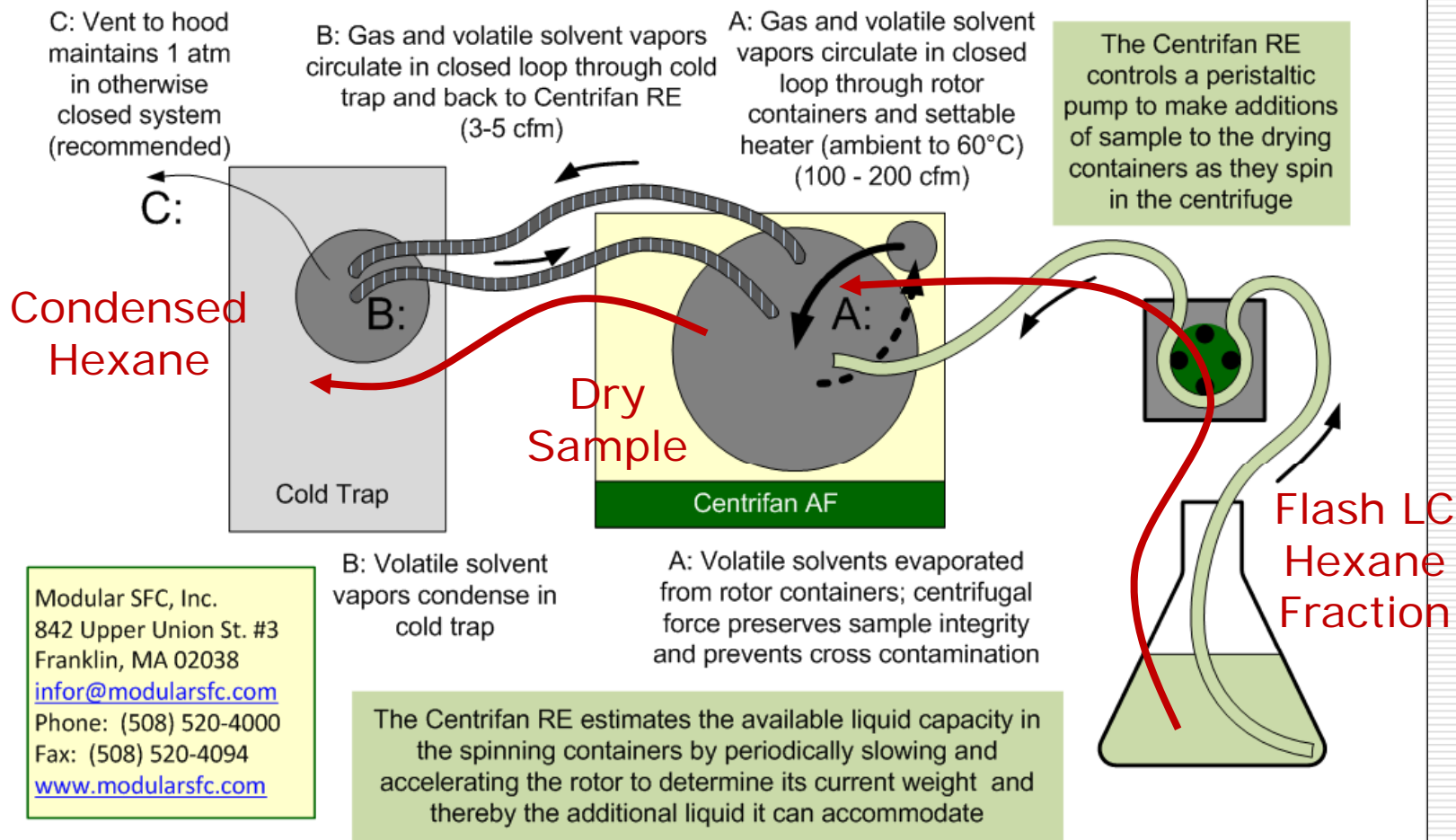


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Centrifan™ AF with Cold Trap

Shows optional connections for **Auto-Fill** feature



Centrifan RE @ 40°C

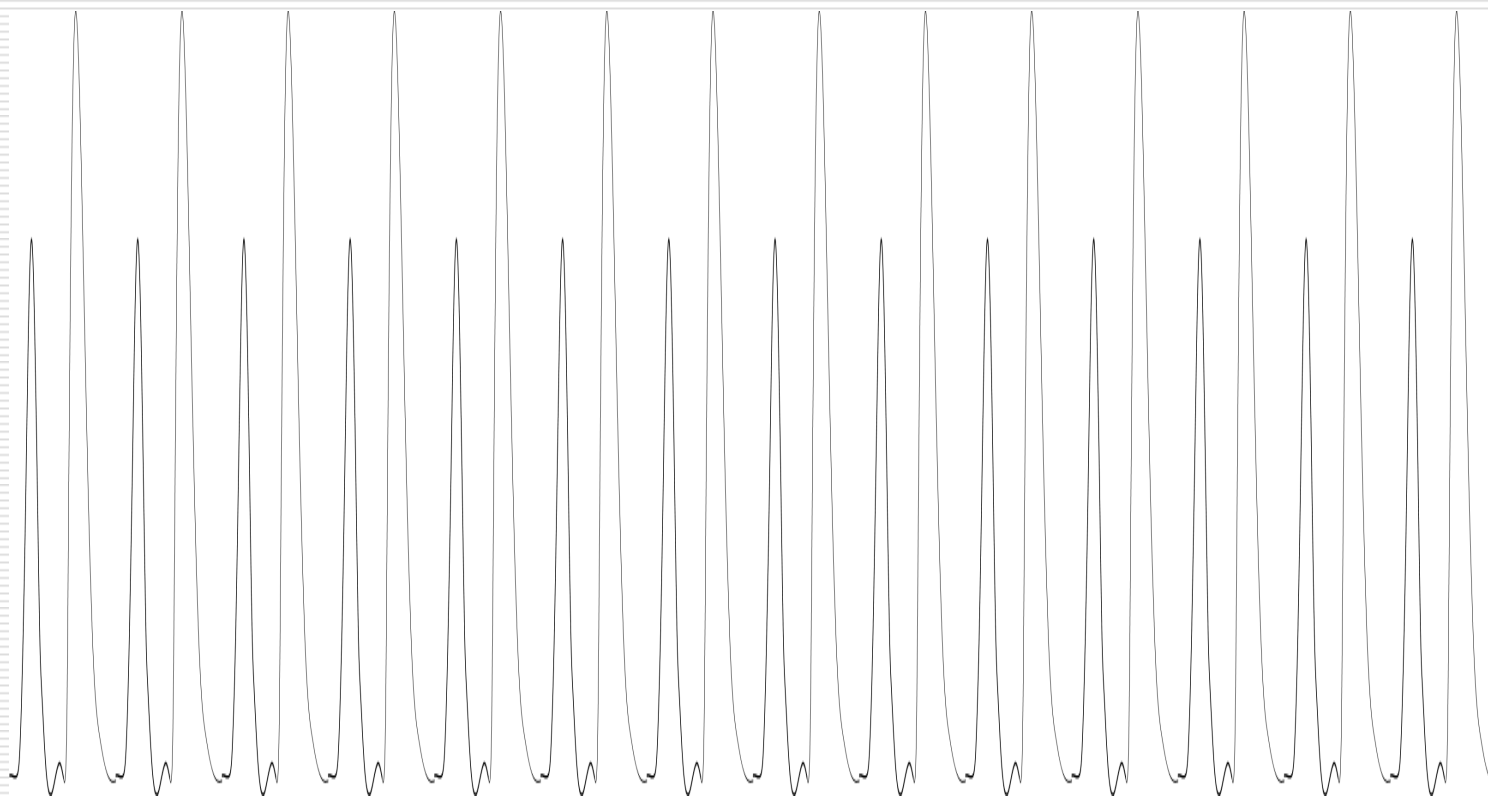
- 250mL Bottle (rate is per bottle)
 - MeCl 1.5 mL/min; 90 mL/hr (720 mL/hr rotor)
 - Water .15 mL/min; 9 mL/hr (72 mL/hr rotor)
 - MeOH 1.5 mL/min; 90 mL/hr (720 mL/hr rotor)

- 96-well plate (2.2mL/well)
 - Water .13 mL/min; 7.8 mL/hr (31.2 mL/hr rotor)

SFC w/ 4 fractions (4 waste)



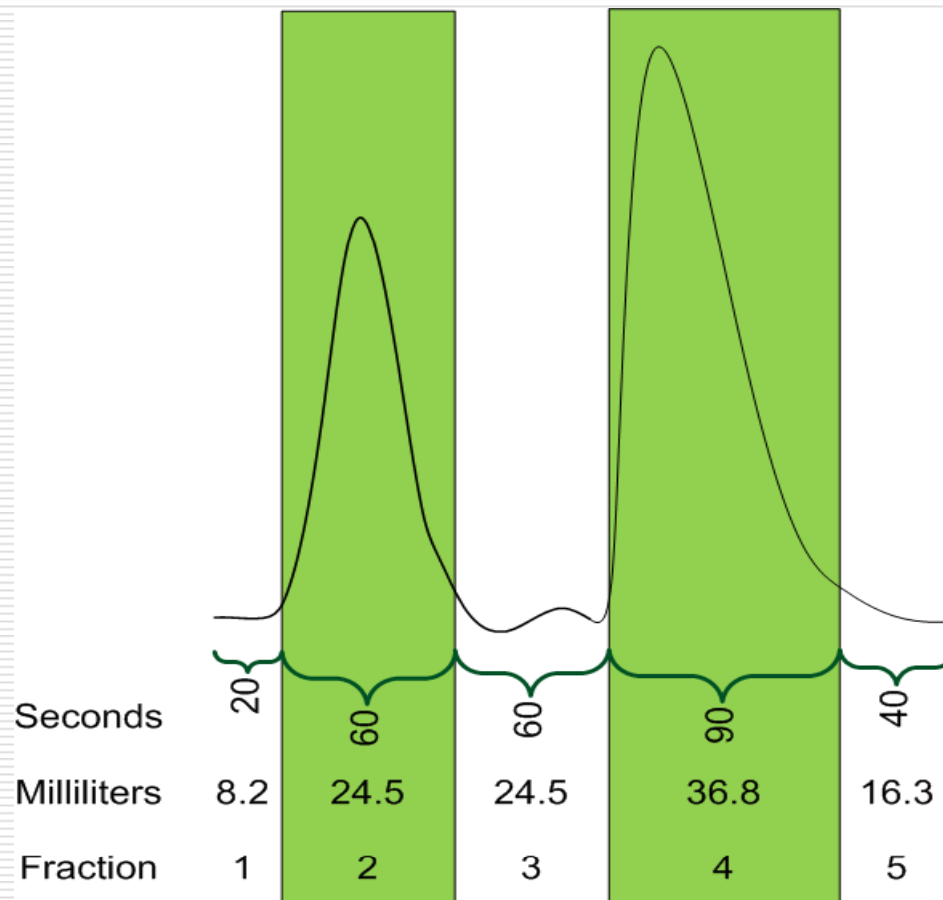
Stacked Injection Method



Some Particulars...

- 70 g/min mobile phase (CO₂+MeOH)
- 35% Modifier (isocratic)
- Injections were 200 milligrams
- 4.5 minutes between injections
- Volume of 2nd enantiomer 50% greater than that of the first

Single Injection Detail



Stacked Injection Method

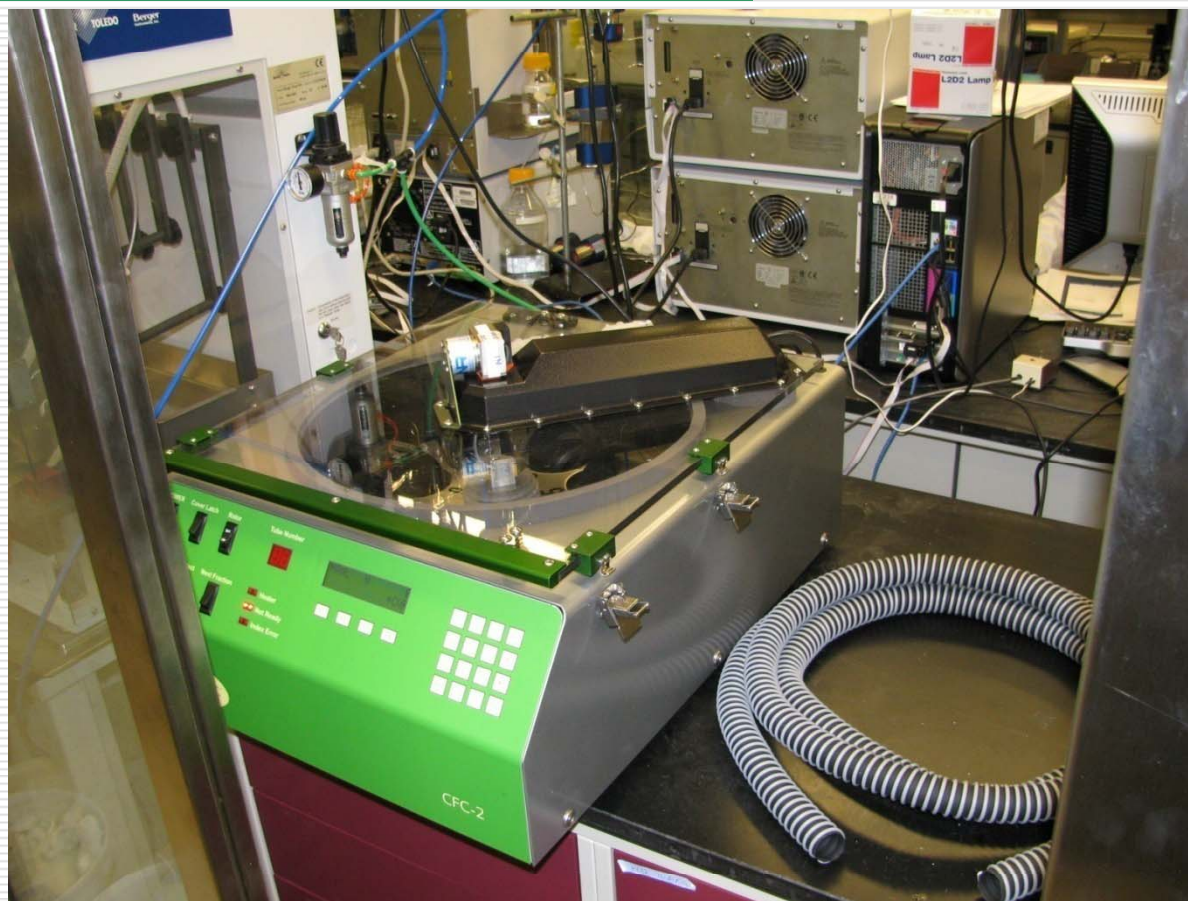
Sample per injection (mg) 200

Compound	Fraction	Time (sec)	Time (min)	Volume (mL)
Waste	1	20	0.33	8.2
Chiral 1	2	60	1.00	24.5
Waste	3	60	1.00	24.5
Chiral 2	4	90	1.50	36.8
Waste	5	40	0.67	16.3
N/A	6	N/A	N/A	N/A
N/A	7	N/A	N/A	N/A
N/A	8	N/A	N/A	N/A
		270	4.5	110.3

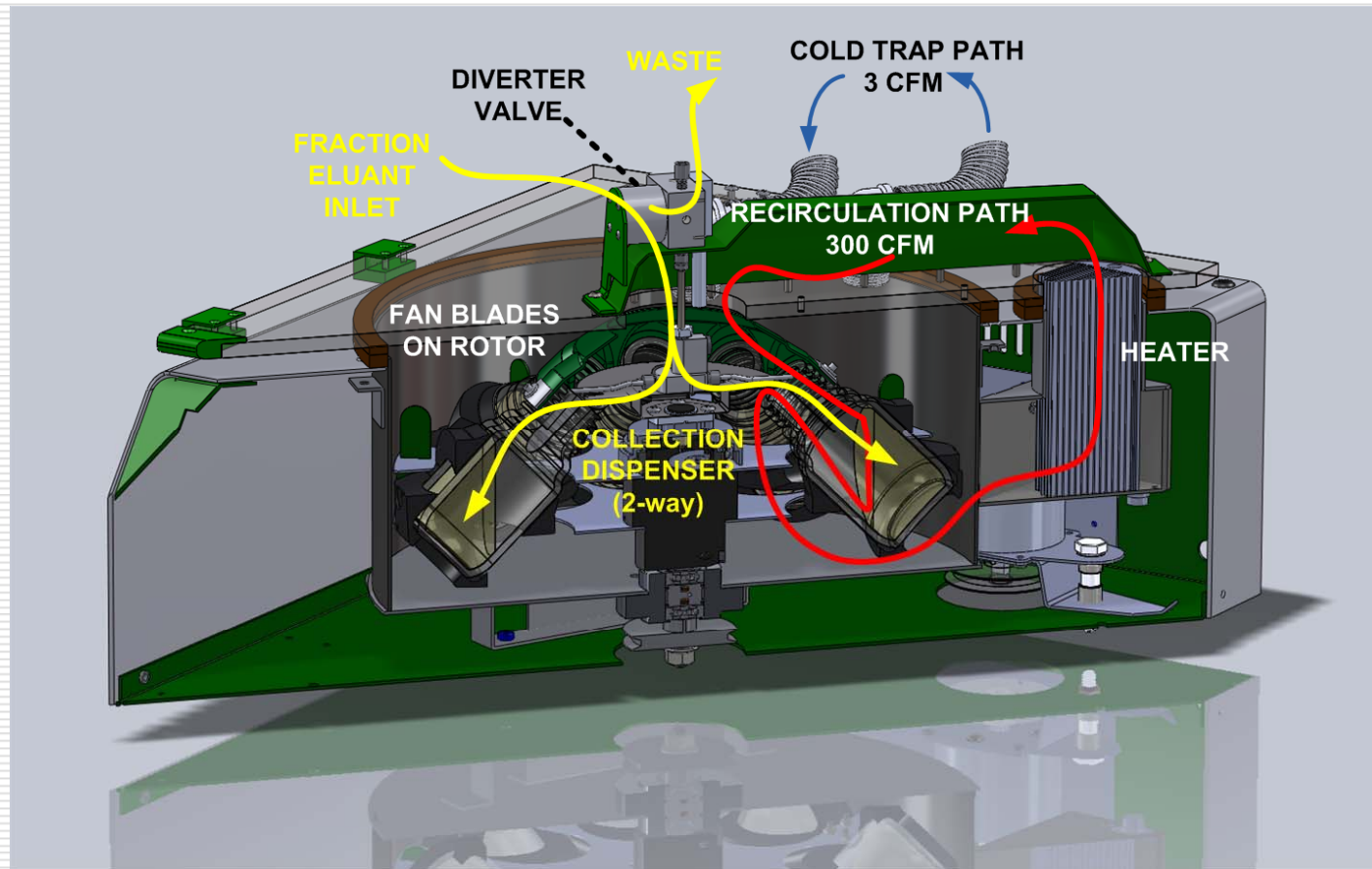
Reset



CFC-3 Connected to BPR Outlet



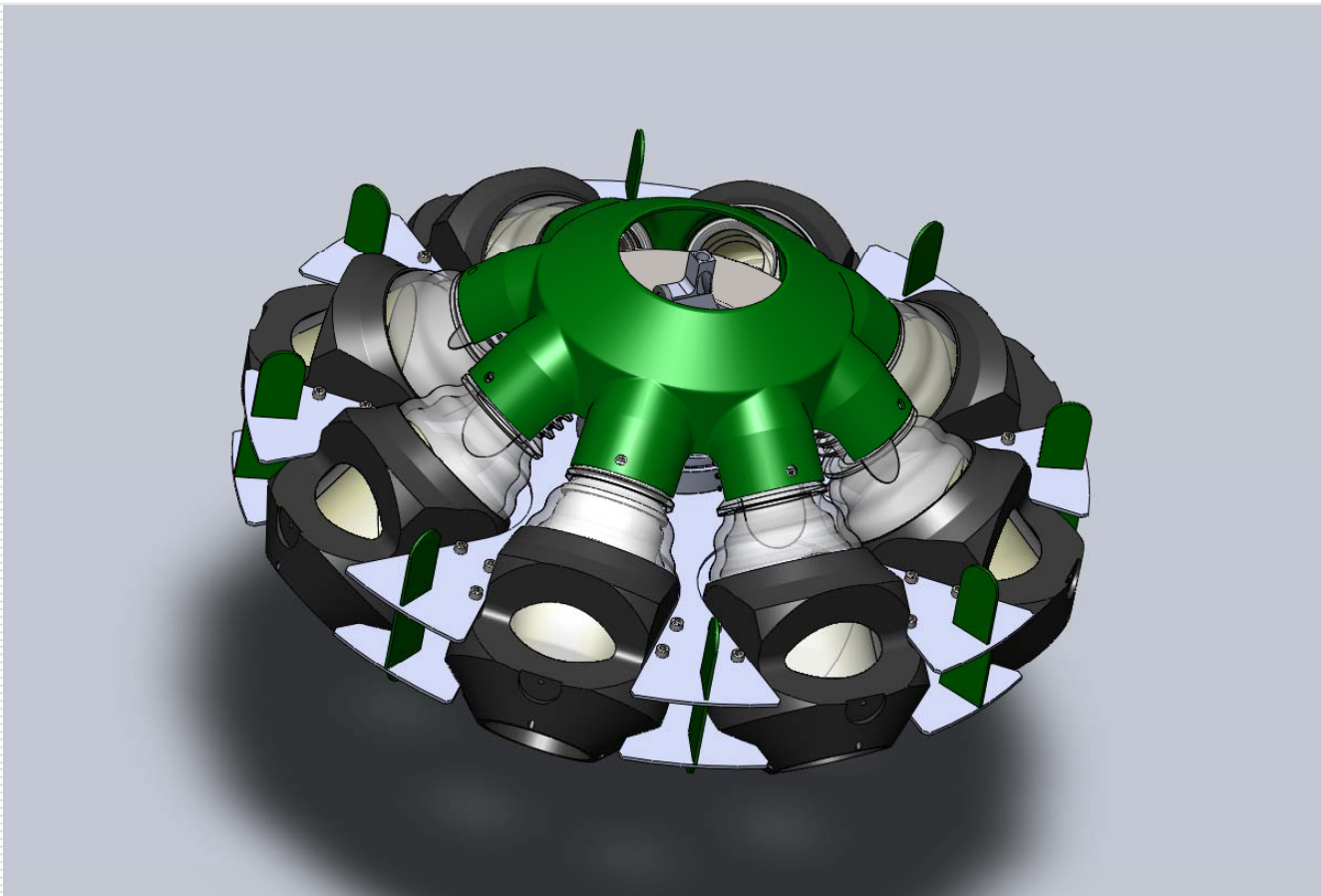
CFC-3: Fraction Collection & Recirculating Evaporation



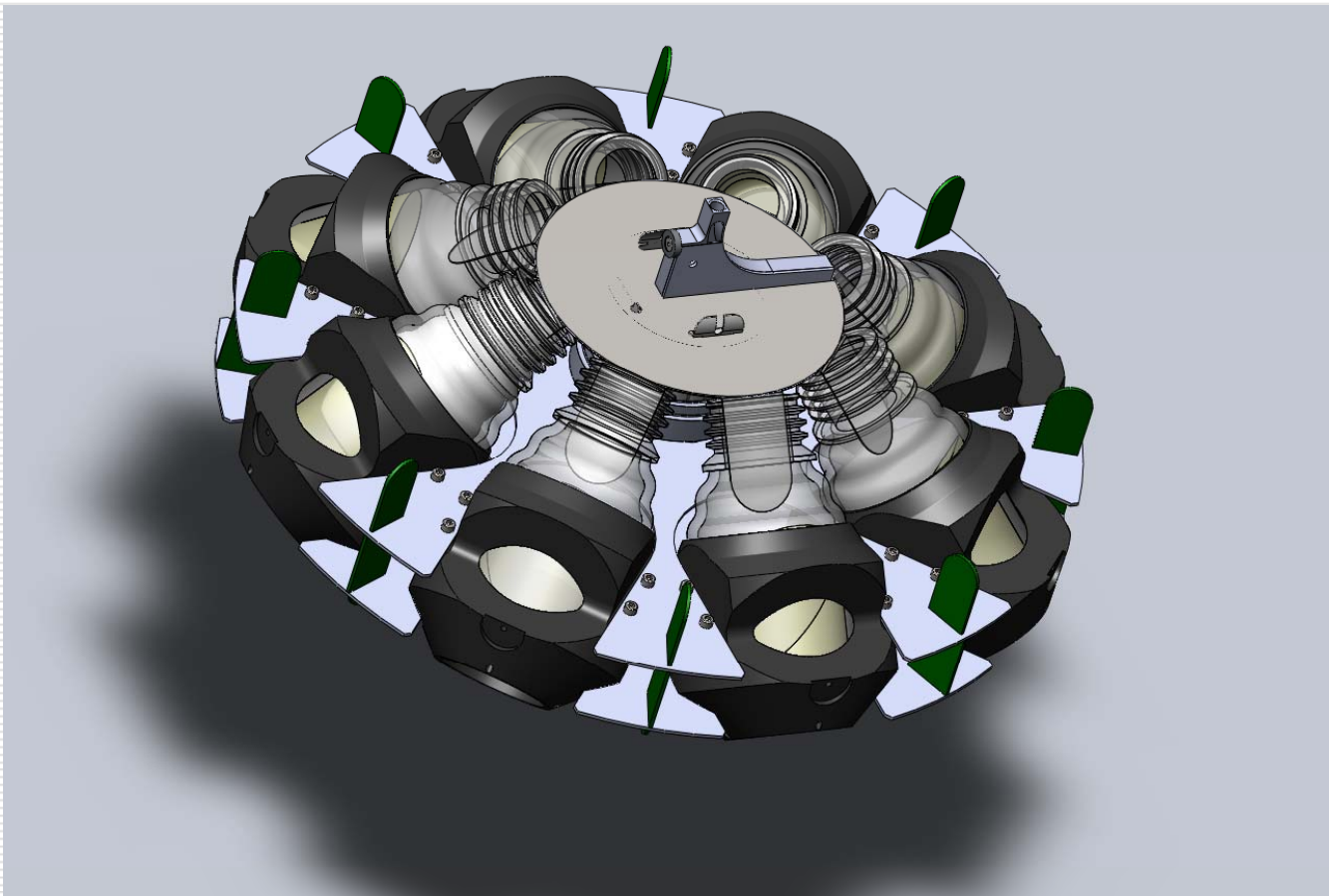
Single Dispensing Rotor

Dispensing Outlets	1
Containers in rotor	8
Container Volume (250 mL size)	200
Fraction Containers	8
Fraction Volume	200
System flow rate (g/min)	70
Modifier %	35%

8 x 250mL Rotor



Single Container Dispense



Fraction Accumulation Rate

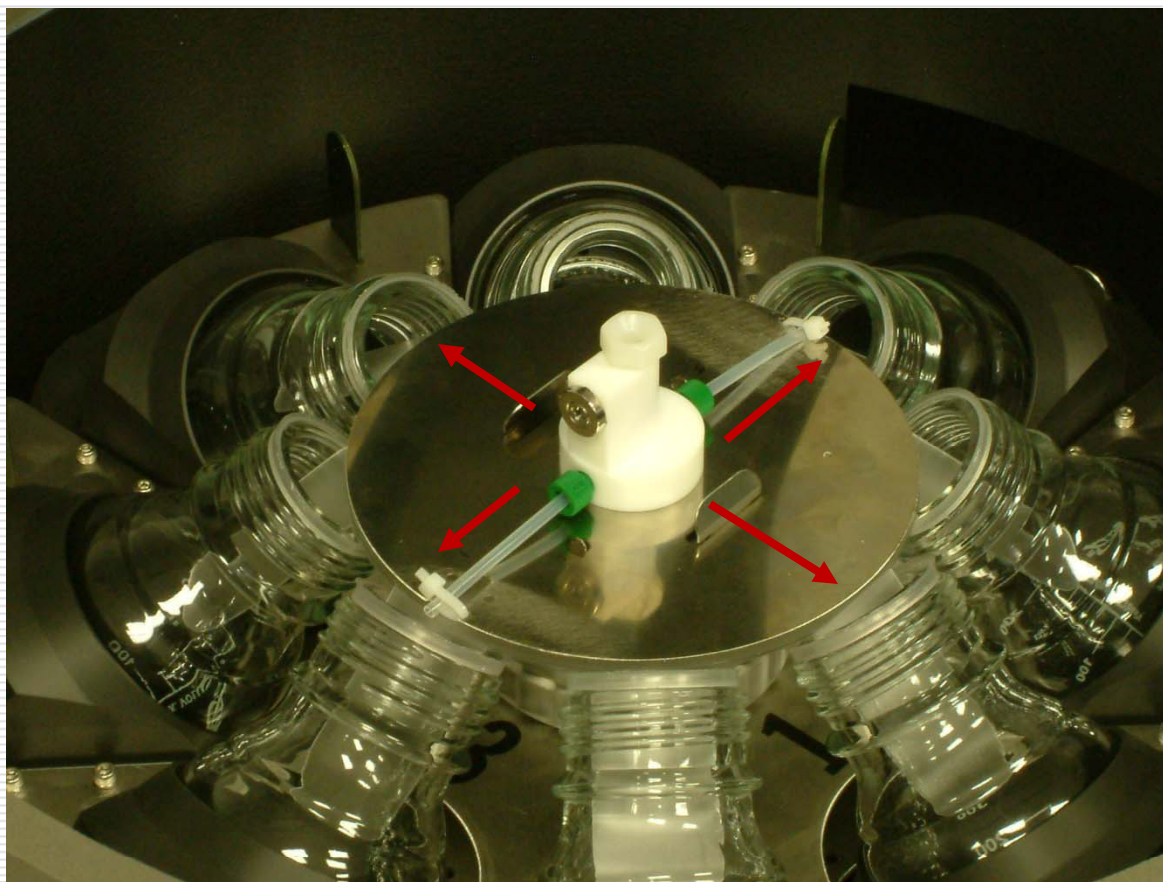
Stack run time (min)	4.5
Chiral 1 duration (min)	1.0
Chiral 2 duration (min)	1.5
Waste volume per inj (mL)	49.0
Chiral 1 volume per inj (mL)	24.5
Chiral 2 volume per inj (mL)	36.8
Chiral 1 effective flow rate (mL/min)	5.4
Chiral 2 effective flow rate (mL/min)	8.2

Collector Capacity

Evaporation rate per bottle (mL/min)	1.5
Evaporation rate per fraction (mL/min)	1.5
Chiral 1 net gain (mL/min)	3.9
Chiral 2 net gain (mL/min)	6.7
Run time for Chiral 1 = 200mL (min)	51
Run time for Chiral 2 = 200mL (min)	30
Number of injections	7
Rotor capacity, processed sample (g)	1.3

*Need much more capacity
before requiring container
replacement*

Install Dual/Quad Dispenser



Dispense to 2, 4 Containers

Dispensing Outlets	1	2	4
Containers in rotor	8	8	8
Container Volume (250 mL)	200	200	200
Fractions in rotor	8	4	2
Fraction Volume (mL)	200	400	800

Possible Good Value Proposition

Multi-Dispensing Increases Capacity



	<i>Single Dispenser</i>	<i>Dual Dispenser</i>	<i>Quad Dispenser</i>
Evap rate per bottle (mL/min)	1.5		
Evap rate per fraction (mL/min)	1.5	3.0	6.0
Chiral 1 net gain (mL/min)	3.9	2.4	dry
Chiral 2 net gain (mL/min)	6.7	5.2	2.2
Run time to Chiral 1 = full (min)	51	164	dry
Run time to Chiral 2 = full (min)	30	77	369
Number of injections	7	17	82
Rotor capacity, processed sample (g)	1.3	3.4	16.4

*Suggests significant
unattended operation*

Process Time Savings with 2, 4 Dispensing

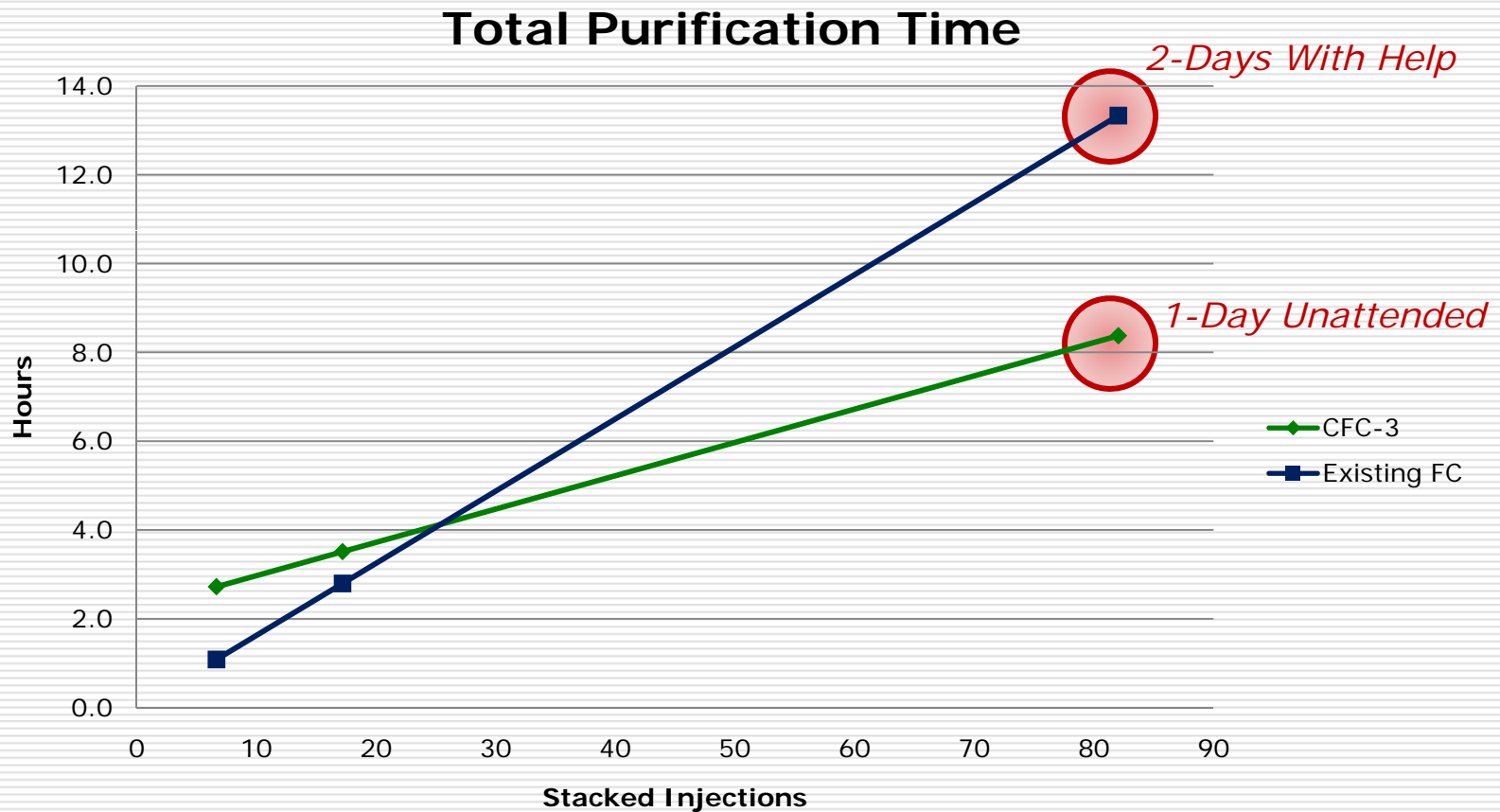


Number of injections	7	17	82
Rotor capacity, processed sample (g)	1.3	3.4	16.4
CFC-3			
Volume remaining to evaporate (mL)	200	400	800
Evap. rate (mL/min/container)	1.5		
Containers	1	2	4
Time to finish evaporation (hr)	2.2	2.2	2.2
Time to run stacks (hr)	0.5	1.3	6.2
Total purification time (hr)	2.7	3.5	8.4
Existing FC			
Chiral 1 volume - no evap (mL)	163	422	2010
Chiral 2 volume- no evap (mL)	245	632	3015
Rotorvap time at .7L/hr. (hr)	0.6	1.5	7.2
Time to run stacks (hr)	0.5	1.3	6.2
Total purification time (hr)	1.1	2.8	13.3
Rotorvap oper. at \$30/hr.	\$33	\$60	\$230

1-Day Unattended

2-Days With Help

Simultaneous Evaporation Reduces Purification Time



Summary

- ❑ Centrifan RE or AF offers safe, non-vacuum, high speed evaporation
- ❑ CFC-3 combines fraction collection & recirculating evaporation technology
- ❑ Simultaneous evaporation reduces processing time for all purification jobs
 - SFC (organic modifiers)
 - HPLC (normal and reverse phase solvents)
 - Flash LC (normal phase solvents)

Contact Information

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