

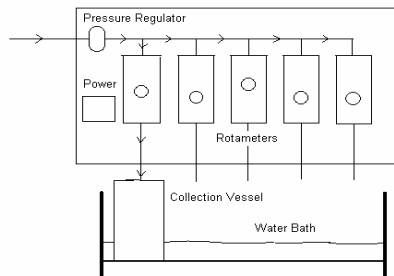
Introduction

- The use of CO₂ in SFC has already proven its utility in replacing a liquid component to be evaporated.
- With SFC, only the co-solvent (typically an alcohol) that is collected has to be removed for the acquisition of the desired material.
- Naturally, the increase in % co-solvent adds to the time for the recovery of dry sample.

Proposal

We propose to eliminate any further need for post-collection solvent removal by way of simultaneous N₂ introduction into the collection vessels. The unit utilizes a timing device, a pressure regulator and 5 manual rotameters. Additionally, a water bath and a programmable timer have been incorporated.

Assembly

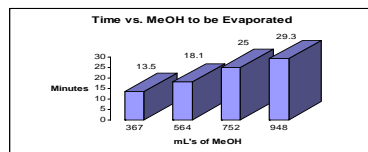


“House Nitrogen” (~45 psi) used to operate the SFC unit was teed off into the pressure regulator (oval) of the N₂ assembly. Nitrogen will only pass through the regulator if the power (square) is turned on. The gas is then diverted to five rotameters (circles) that functions to adjust the flow rate from 0-60 cfu. The stream is then directed through a hole in the cap of the collection vessel to aid in the evaporation of the co-solvent.

Cost Savings

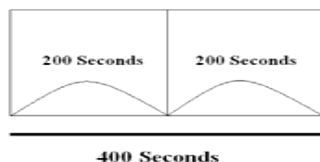
In its present state, it has been estimated that the cost of drydown using SFC is roughly 7 percent of the cost doing similar work by HPLC (Terry Berger – CoSMoS 2005). The cost for general solvent disposal in R&D is \$3.10 per gallon.

Rotovap Results – 2L Flask, 50 torr, 40C 125 rpm

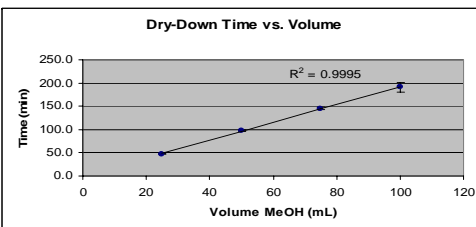


Methodology

- Between 10-25% MeOH (total flow = 90g/min) is typical for a SFC separation and has been utilized for this study.
- For the following data, 10 injections were made with 2 traps open for 200 seconds apiece (33 minutes).
- Thus, at the following percentages of MeOH (10,15,20 and 25), 367, 564, 752, and 940mL's, respectively, should be expected in each vessel.
- These experiments were set up in such a manner as to create a worst case scenario, i.e., peak widths of 3.3 minutes and baseline resolution.

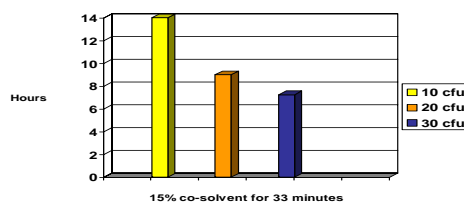


Times for Different Static Amounts of Solvent to be Evaporated using the Nitrogen Assembly without heat.

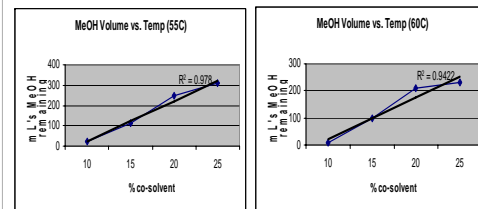


Flow Rate of N₂ = 30 cfu

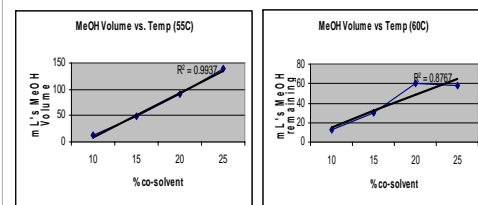
Time to evaporate 564mL (15% Co-Solvent), using the Nitrogen Assembly without heat at Different Nitrogen Flow Rates..



Remaining MeOH at 30 cfu using Various Water Bath Temperatures and Percentages of Co-solvent (Water 1 inch from bottom)



Remaining MeOH at 30 cfu using Various Water Bath Temperatures and Percentages of Co-solvent (Water 1 inch from top)



Safety Measures

- The safety personnel at GSK have approved the device.
- A stainless steel shelf (diffuser pan) in the water bath protects samples from the hot metal bath bottom.
- Water bath will shut off automatically if the sensor detects a temperature 5°C higher than the setpoint.
- A timer with independent control of both the water bath and the N₂ assembly is programmed to shut down the water bath near the completion of the last injection.

Conclusions

•The use of the Nitrogen assembly without any heat did not afford a great advantage. However, by using a nearly filled water bath at 60 degrees, a reduction of > 90% of MeOH was observed at all flow rates.

- Under the experimental conditions, we realize that some solvent still remains when the run is finished, however the Nitrogen continues to flow to allow for a completely dry product.
- The temperature inside the collection vessel is a moderate 35C, up to a set point of 60C for the water bath.
- Besides employee time savings, other advantages would be quicker turn-around time, accommodation for more sample throughput, cost and a reduced atmosphere.