A Quick Study - Comparison of Chromatography Normal Phase vs. Flash on the Gilson PLC 2020

Application Note

Keywords

Purification, Normal Phase Chromatography, Flash Chromatography, PLC 2020, Preparative Chromatography

Introduction

This study was performed by Matthew Smith with Gilson UK in the United Kingdom.

This application note provides a quick comparison of three pharmaceutical compounds using high pressure normal phase and low pressure flash chromatography on the Gilson PLC 2020 Personal Purification System. Comparison of peak shape, peak retention time, chromatography run time, fraction volume, and solvent consumption were all studied to determine the most efficient and optimal chromatography method.

The touch screen control on the PLC 2020 allows for simple and quick modifications to method gradients. The built-in solvent selection valve offers efficient mobile phase choices to be made for faster compound elution and/or increased compound resolution during semi-preparative and preparative chromatography runs. Additional studies on the comparison of high pressure normal phase and low pressure flash chromatography will provide further details on purification percent recovery, peak width, and peak resolution.

Figure 1. Gilson PLC 2020 Personal Purification System

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Materials & Methods

Materials & Methods for the PLC 2020 System

All solvents used were HPLC grade or higher. All reagents were ACS grade or better.

Compounds: ~ 100 mg each in Hexane

Benzocaine – anesthetic compound
Caffeine – natural
Dipyridamole – cardiovascular drug

General Conditions:

Injection: 1 mL
Flow Rate: 25 mL/min

Normal Phase Preparative System:

Injection (mL): 1
Column: Macherey-Nagel; NUCLEODUR® VarioPrep 21 x 50 mm; 5 µ

Flash Preparative System:

Injection (mL): 1
Column: Macherey-Nagel; CHROMABOND® 25g SiOH; 21.2 x 16.5 mm

Results & Summary

Table 1. Summary - Normal Phase (NP) vs. Flash Chromatography Injections on the Gilson PLC 2020

Peak Name	NP/Flash Peak Retention Time (min)	NP/Flash Fraction Collection Volume (mL)	NP/Flash Total Run Time (min)	NP/Flash Total Solvent Consumption per Injection (mL)
Caffeine	4.8/7.3	15/40	6/10	150/250
Benzocaine	2.9/4.5	11/19		
Dipyridamole	0.7/1.4	7/11		

Figure 2. High Pressure Normal Phase Chromatography Injection on the Gilson PLC 2020

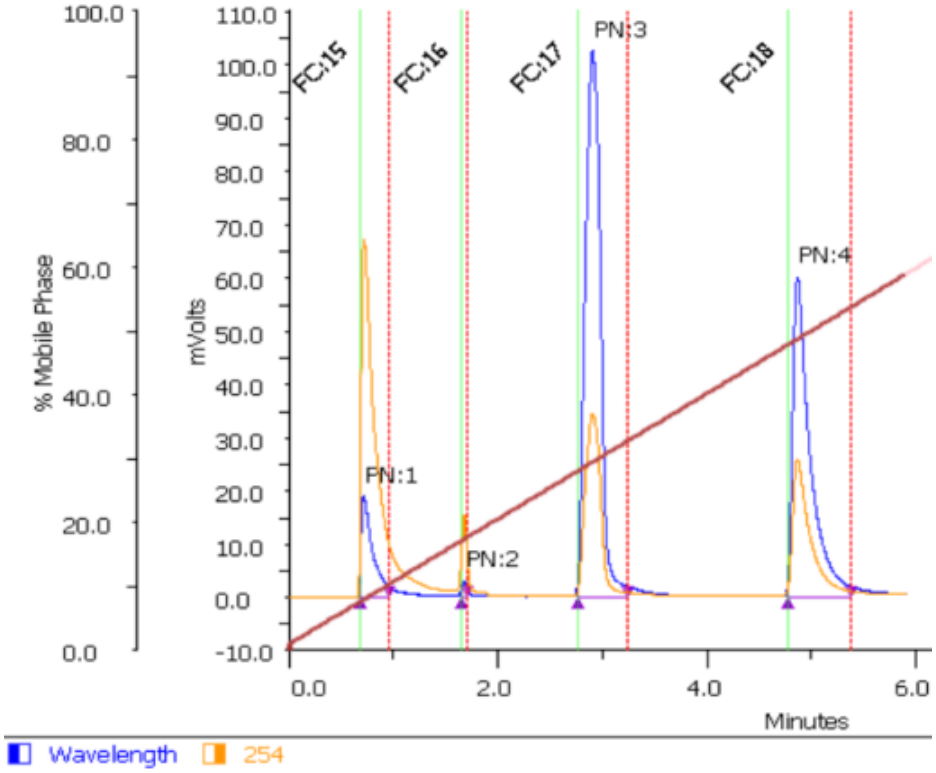
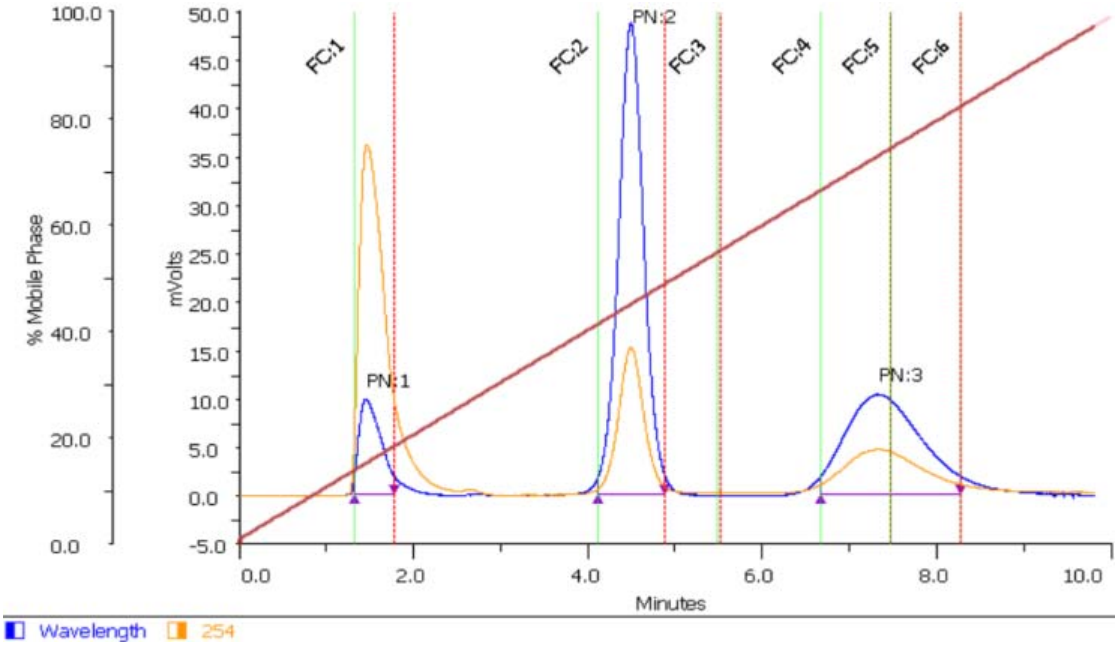


Figure 3. Low Pressure Flash Chromatography Injection on the Gilson PLC 2020



Visual comparison of peak shape immediate shows a comparison of compound peak response. Peak height values for each of the three compounds injected on the normal phase column are almost double the peak height values for the same compounds injected on the flash column. Flash chromatography shows much wider peaks, with the caffeine peak requiring three fraction tubes to collect the full peak in 40 mL versus a single fraction tube to collect the same compound from the normal phase chromatography injection.

Run time using a high pressure, normal phase column is reduced by 40% over using a low pressure flash column, and this directly correlates to savings in mobile phase required per injection. The amount of mobile phase used per injection depends on the flow rate used, and in this quick study, one liter of mobile phase solvent would be saved for every ten injections made if the normal phase column were used instead of the flash column.

Future studies will provide additional supporting information for this quick study that clearly shows the benefits of using high pressure normal phase instead of low pressure flash chromatography for peak shape, peak retention time, chromatography run time, fraction volume, and solvent consumption.