ELSD Tuning Examples

ELSD Performance

The SofTA ELSD performs best when the conditions are optimized to provide the best signal to noise ratio for the separation. In many applications the detection limit requirements are high enough that the universal operating conditions, Spray Chamber 30C Drift Tube Temperature 60 and Filter BLT 5, provide acceptable results. There are applications where optimization of the conditions will improve the results considerable.

1. The mobile phase may be difficult to evaporate, high aqueous or volatile buffer content or high flow rates

2. The required detection limit is low, 100ng or lower

3. The analyte is semi-volatile.

ELSD Conditions to Optimize

The drift tube temperature and the Thermo-Split spray chamber temperature are selected to provide the maximum detector response with minimum baseline noise. The temperatures are selected based on the solvent volatility and mobile phase flow rate. Some experimentation will be required to optimize the ELSD.

When setting the ELSD temperatures for a new method, select 30°C for spray chamber temperature and 60°C for drift tube temperature. These temperatures should then be adjusted for the best signal to noise ratio during method optimization. For the best performance, a mobile phase that is highly organic and volatile requires an ambient or elevated spray chamber temperature and moderately high drift tube temperature. With highly aqueous or high boiling point organic mobile phases, the best performance will at sub-ambient spray chamber temperatures and moderate drift tube temperatures.

Thermo-Split Spray Chamber Temperature

The Thermo-Split Spray Chamber can operate from 10°C to 70°C. The Spray Chamber temperature controls the vapor phase split ratio. For an easily evaporated mobile phase, the split ratio can be set low. To achieve this, the Thermo-Split chamber is heated. As the aerosol traverses the chamber, it partially evaporates, shifting the particle size distribution low enough for essentially all the particles to negotiate the bend. So, when highly organic mobile phases are used, the Thermo-Split chamber is used at ambient or elevated temperatures. Under these conditions a majority of the aerosol particles pass through the chamber and are carried into the evaporative zone.

For difficult to evaporate mobile phases, or high flow rates, the split ratio needs be high so the Thermo-Split chamber is cooled. When the aerosol exiting the nebulizer encounters a cooled environment, it partially condenses into larger particles whose momentum carries them into the wall and down the drain. By making the walls suitably cold, 99+% of an aqueous stream can be diverted away from the evaporative zone.

Drift Tube Temperature

The drift tube temperature can be set from ambient to 120°C. The drift tube temperature is set at a temperature high enough to evaporate the mobile phase and not vaporize the analyte. A higher drift tube temperature may give result in a quieter baseline but smaller peak. The drift tube temperature should always be higher than the spray chamber temperature but only as high as needed to achieve a quite baseline.

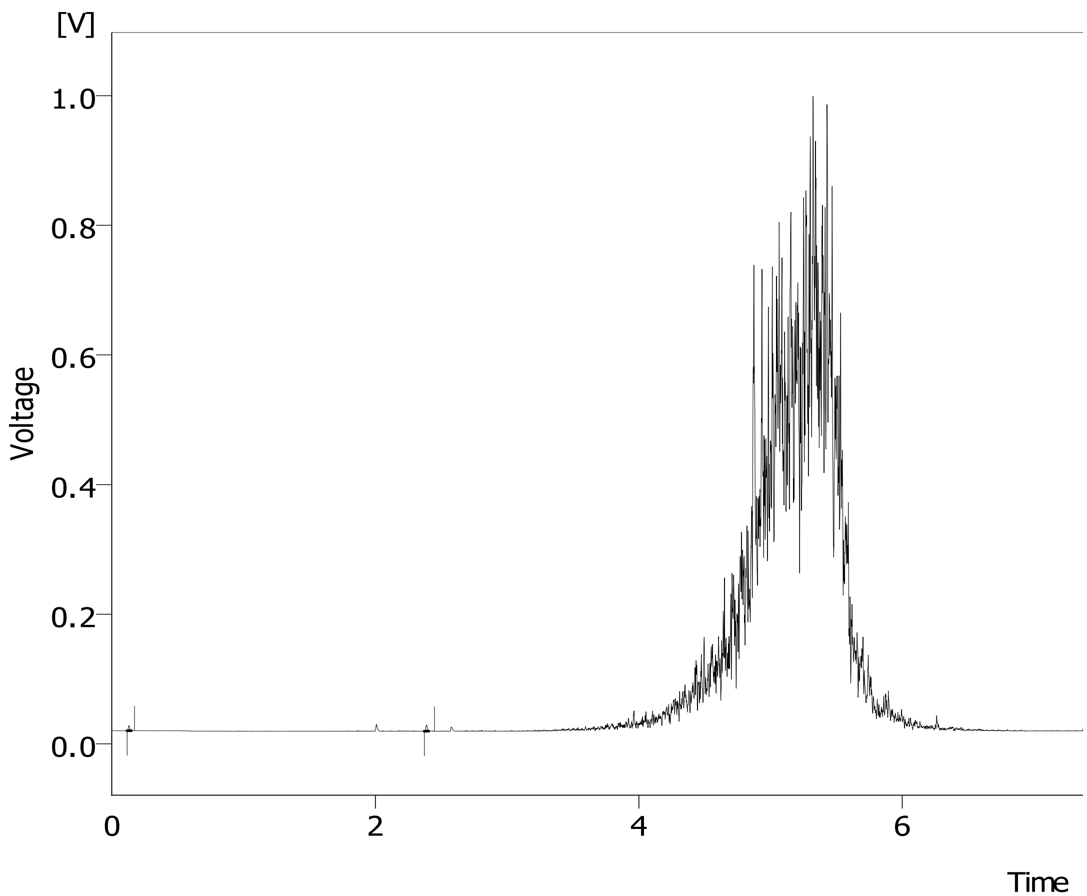
Filter type and weight

There are two filter types to choose from RC (FLT) and Baseline (BFT.) Filter weight is the level of baseline noise filtration. OFF indicates no filtration. 10 is maximum filtration. In most cases, select BFT for baseline filtering. The FLT setting applies a RC filter to the entire signal. For high-speed chromatography, less than a 5 sec peak width, select BFT and turn the weight OFF. If the peak widths are 5 to 30 seconds use the BFT filter with a weight of 1 to 10. For peak widths greater than 30 seconds, select the FLT setting with a weight of 1 to 10.

The following examples highlight the affects of Spray chamber temperature, Drift tube temperature and Filter.

Drift Tube Temperature Optimization

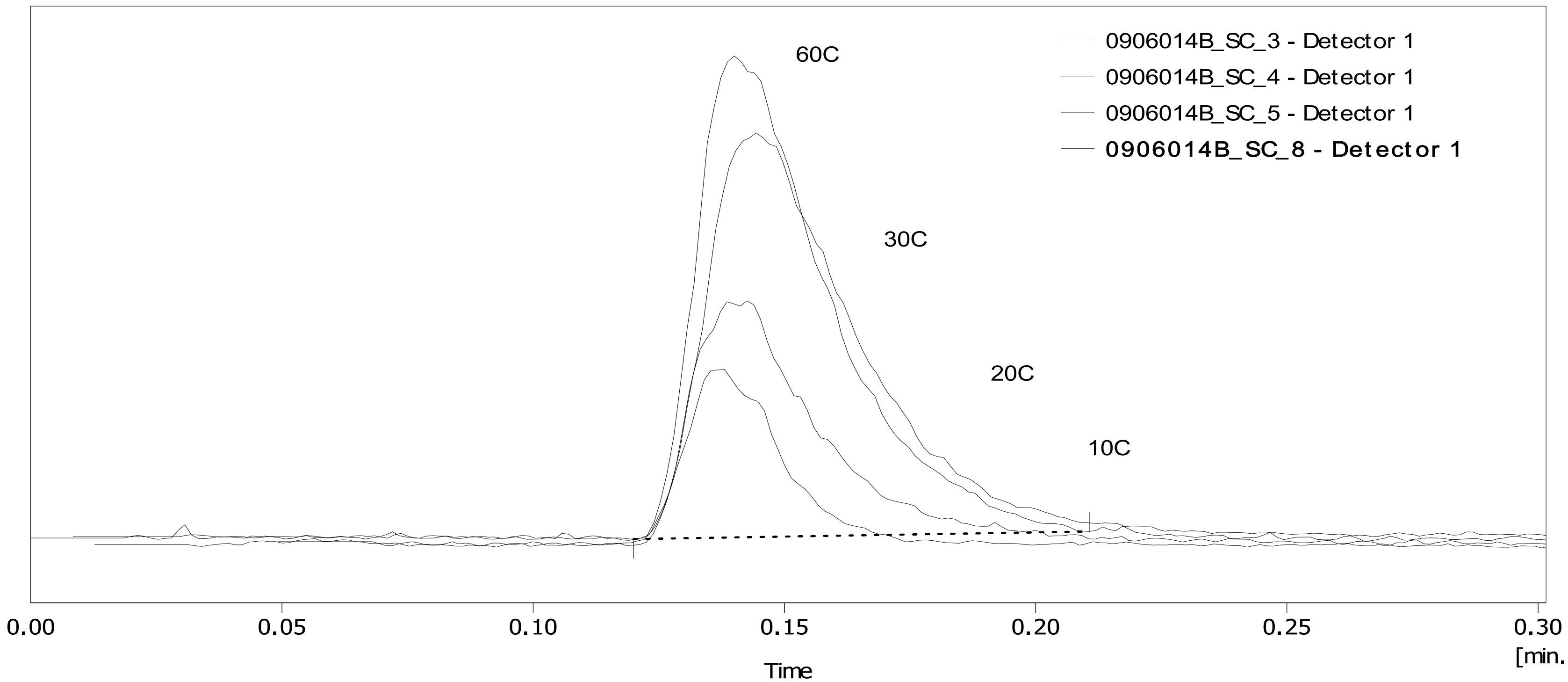
In this example a difficult to evaporate mobile phase was selected, 1ml/min Water. The Spray Chamber was set at 30 and the drift tube was varied. At time 0 the drift tube was 60C. The drift tube cooled to 50C by time 5 and returned to 60 after 6 minutes.



This example illustrated how a 5C temperature change dramatically increases or decreases the baseline noise.

Spray Chamber Temperature Optimization

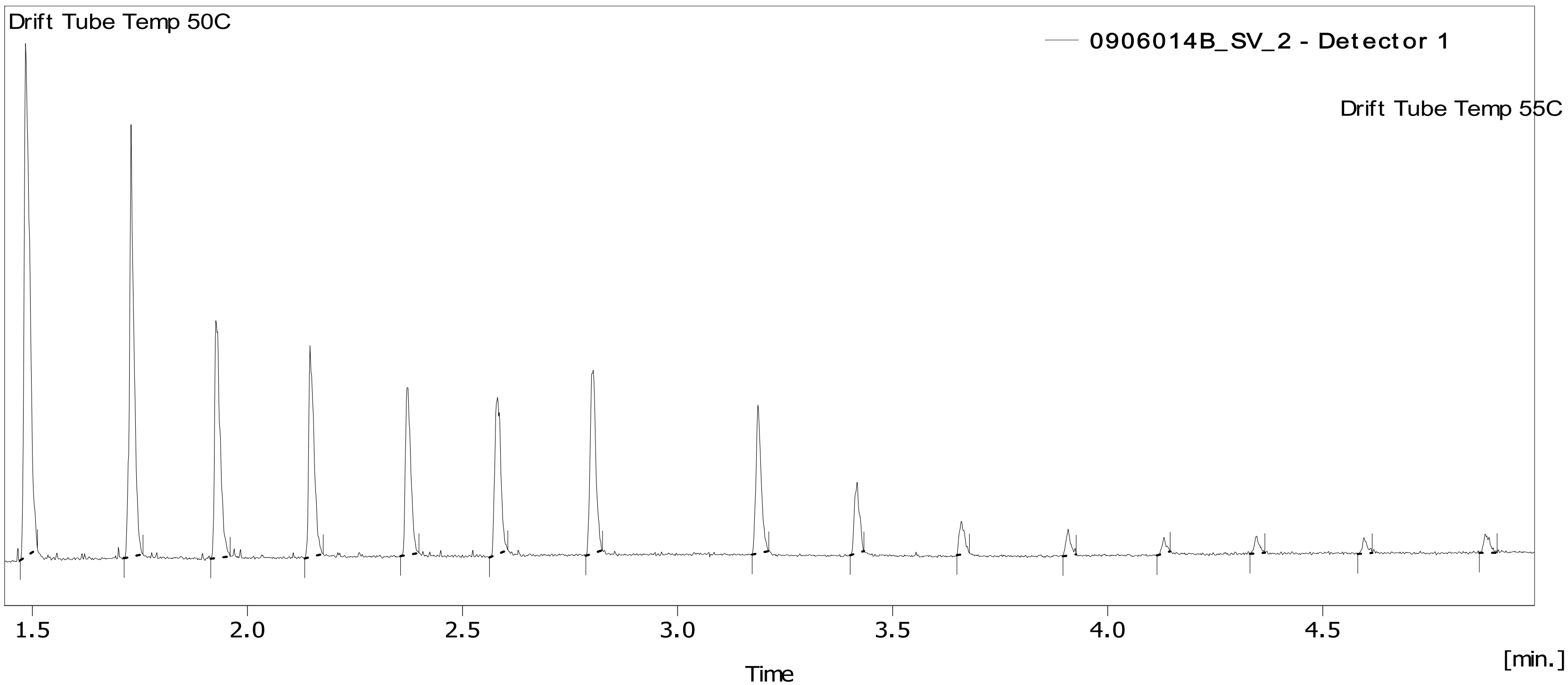
In this example an easy to evaporate mobile phase was selected, 0.5ml/min Methanol. The Drift Tube temperature was held constant at 40C. The Spray chamber temperature was increased from 10C to 60C. You can see the effects of reducing the spray chamber temperature in the image below.



Semi-Volatile Analyte Detection

It might be assumed from the first two examples that the best conditions would be a hot spray chamber and a hot drift tube. Under these conditions all of the vapor would enter the mobile phase and all of the mobile phase will be evaporated. This example shows why these conditions are not optimal, especially when the analyte is semi-volatile.

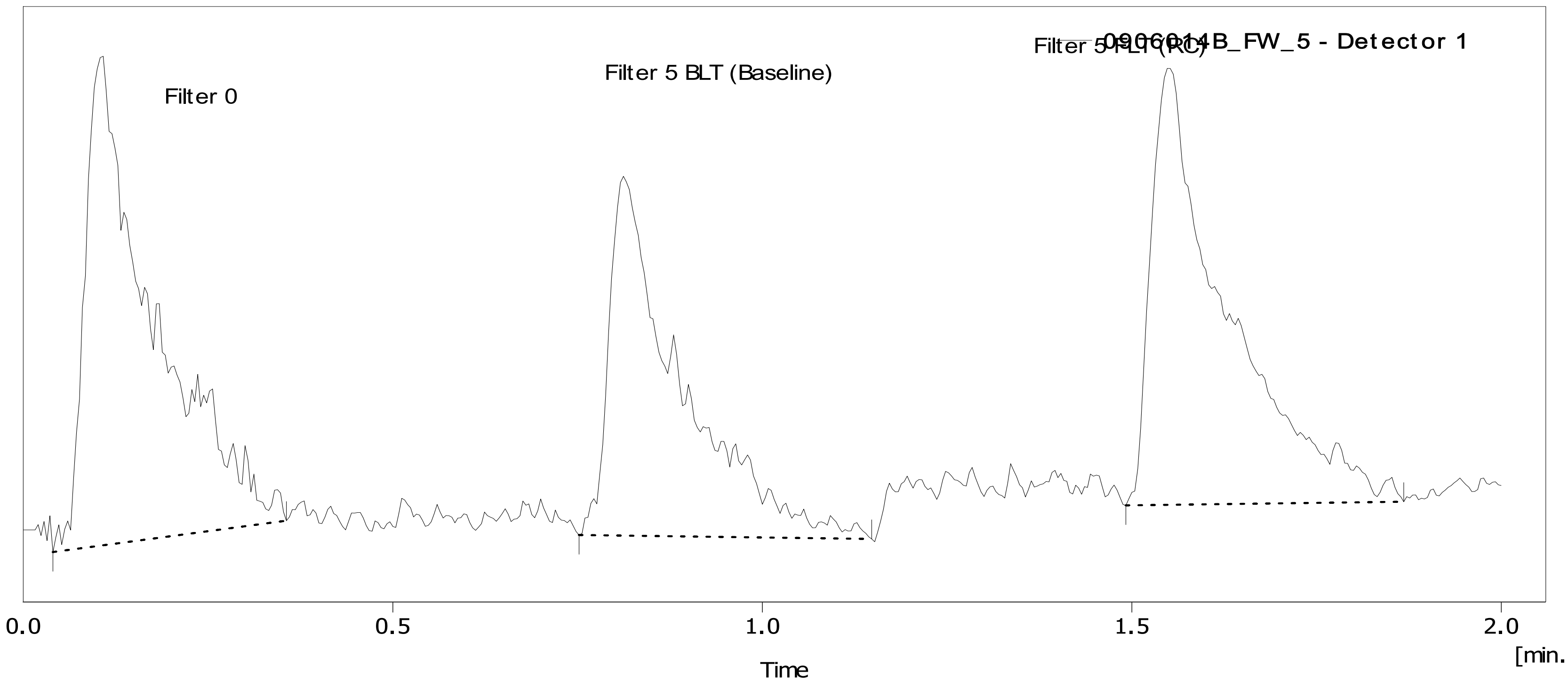
In this example the mobile phase was 0.5mL/min of 50:50 Methanol:water. The Spray chamber was selected at a moderate 30C. The drift tube temperature was heated from 50 to 55C. The analyte, semi-volatile Methyl Paraben, was injected periodically.



This example show that a small increase in drift tube temperature will decrease the signal of the analyte significantly.

Filter Type

In this example both filter types, Baseline and RC, are compared to no filtering. The mobile phase was 1ml/min water. A length of wide ID tubing was inserted between the injection valve and the detector to simulate broad peaks. The Spray Chamber and Drift Tube temperatures were selected to provide significant noise for the example, SC 30, DT 56.

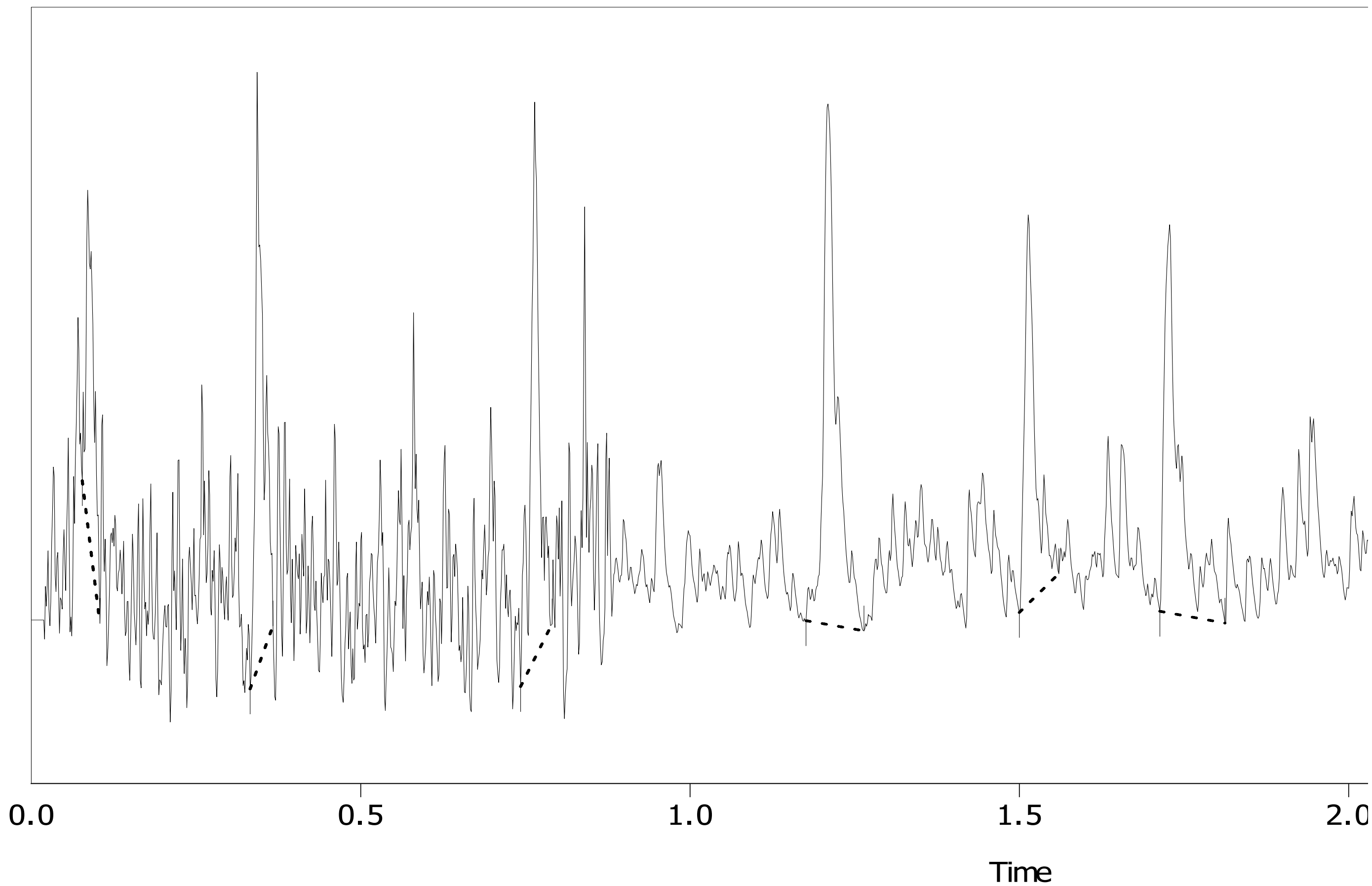


The baseline filter weight 5 slightly improves the baseline noise translated to the peaks, The RC filter 5 significantly improves the baseline and peak noise.

Filter Weight

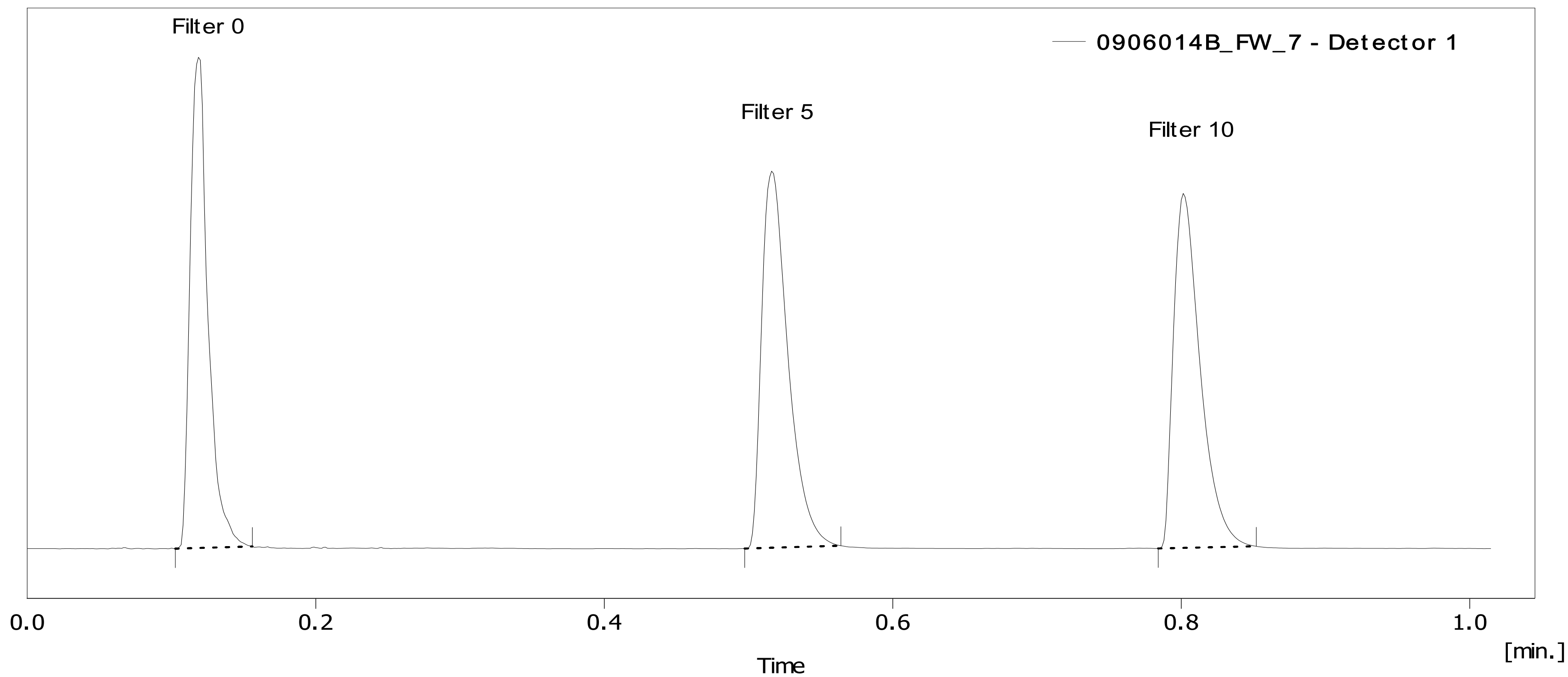
In these example the affect of the filter weight for both types is shown. Again, the Spray Chamber and Drift Tube temperatures, SC 30, DT 53, were deliberately selected to provide significant noise for the example. The mobile phase was 1ml/min water. The analyte was 100ng NaBenzoate.

This example shows the Baseline filter at filter 0 and 5



The baseline is considerable improved with filter 5.

The next example shows the affect of filter weight on the RC or FLT Filter type. The conditions are the same but the concentration is 1000ng instead of 100ng.



This example shows that while a larger filter filter weight with RC filter type decrease the noise it also broadens and decreases the peak heights.

Please note that the filter should not be used to compensate for poor evaporation of the mobile phase. The Spray Chamber and Drift Tube temperatures should be optimized for the best signal to noise before a filter is applied. In these examples, raising the Drift Tube temperature from 53 to 60C would eliminate all of the noise.