

eksigent

application note

**a rugged, automated sampling system for
real-time reaction monitoring**

The ExpressRT™-100 system incorporates a novel sampling probe designed to prevent sample line blockage in heterogeneous mixtures.



expressRT•100

a rugged, automated sampling system for real-time reaction monitoring

introduction

Chemical process development and optimization require quality analytical data taken over the course of a reaction. With integrated reaction sampling and chromatography, the ExpressRT-100 system allows for continuous unattended monitoring of your chemical process.

Not all reactions are performed using homogenous, particle-free solutions. Obviously, automated sampling and the subsequent analysis of reaction mixtures containing small particles present a challenge to the accuracy and precision of online reaction monitoring. The ExpressRT-100 system incorporates a novel sampling probe designed to prevent these potential problems from occurring. A filter is located at the end of the sampling probe where it can trap particulate matter, preventing it from traveling through the sampling line where it would interfere with quenching, dilution and analysis of the reaction samples (figures 1 & 2). Filter materials include 316 stainless steel, PTFE, Nylon, cellulose acetate and paper with pore sizes ranging from 0.2 –0.5 μm .

An experiment was performed to test the effectiveness and capacity of the filter. Carbon black was added to a solution of methanol containing two analytes. This solution was repetitively sampled and each sample chromatographically analyzed. Samples were taken over a period of four days and the chromatographic results compared to determine to what extent, if any, the quantitative precision was affected by the accumulation of carbon black particles in the system.

Figure 1. ExpressRT-100 sampling probe design.

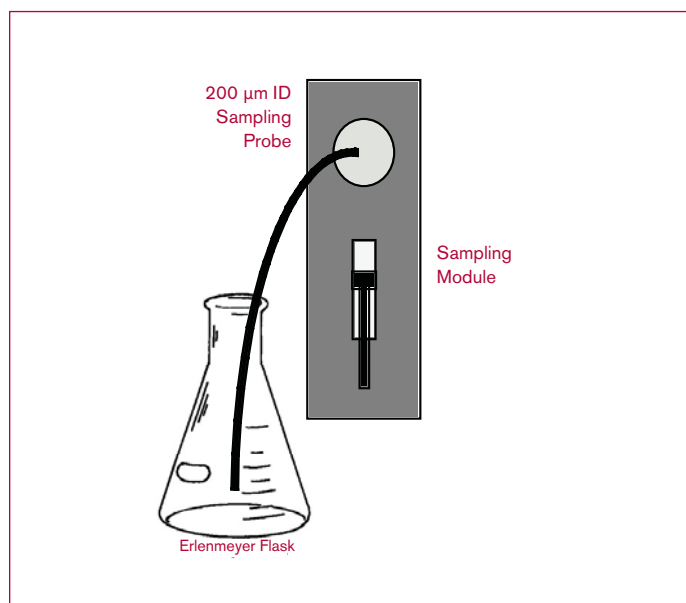
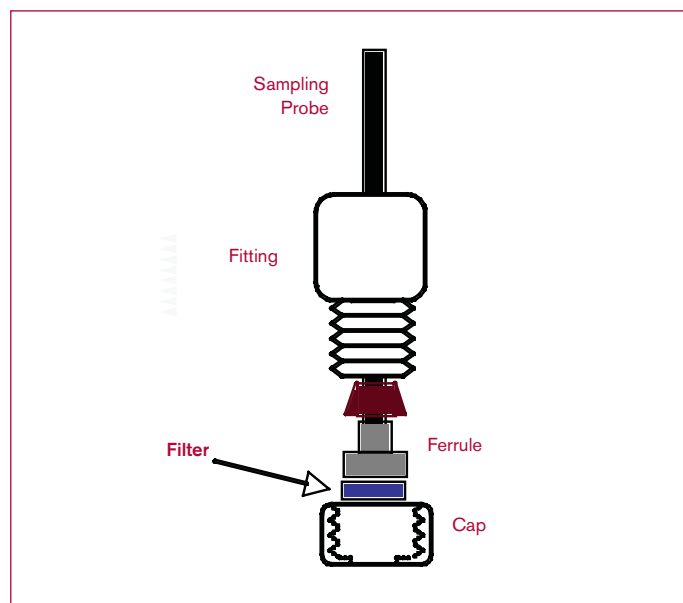


Figure 2. Enlargement of sample probe filter.



method

To begin, 0.2 g each of salicylic acid and methyl salicylate were dissolved in 200 mL of methanol. Then, 0.5 g of carbon black was added and the mixture stirred in a 500 mL Erlenmeyer flask. The “reaction” was monitored over four days for a total of 600 samples. Then 18 μ L of sample were drawn directly from the reaction vessel and diluted 100-fold with methanol. This dilution was delivered to the chromatography module’s sample loop where 40 nL was then injected. Chromatography was performed on a 50 x 0.3mm Eksigent chromXP 3C18-EP-120 column with an isocratic mobile phase composition of 40% 0.1% TFA in H₂O and 60% MeOH. Salicylic acid and methyl salicylate were detected @ 214 nm. Retention times for salicylic acid and methyl salicylate were 1.9 and 2.4 minutes respectively (figure 3).

results

Data for the last 400 samples collected during the experiment was processed and analyzed. Chromatograms were integrated to determine the peak areas for each component. The peak areas were then exported to a spreadsheet and graphed as a function of sample number (figure 4). Any change in peak area between samples would indicate a failure in filtration. However, as seen in figure 4, there was no indication that the filter failed to effectively perform its function of preventing particulate matter from entering the system. Also, the capacity of the filter is clearly high enough to be used reliably without affecting quantitation over an extended period of time.

Figure 3. Typical separation of salicylic acid and methyl salicylate sampled from carbon black-containing slurry.

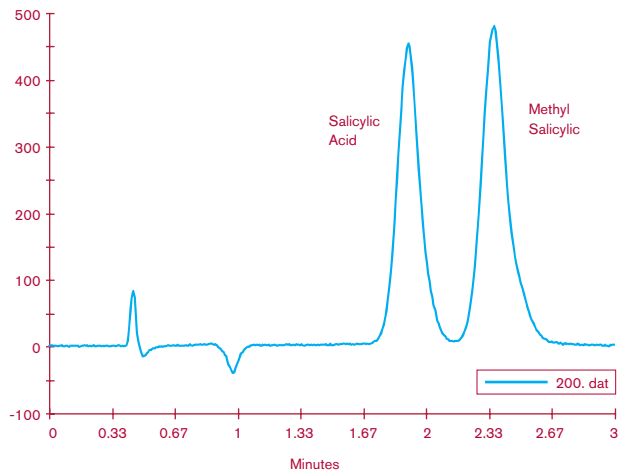
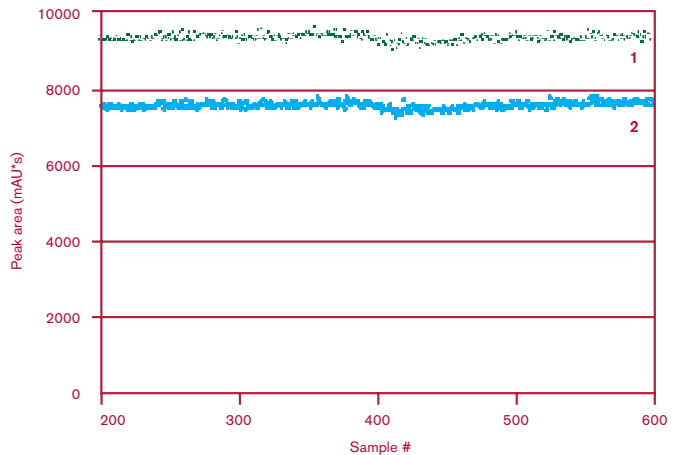


Figure 4. Peak areas for (1) methyl salicylate and (2) salicylic acid plotted as a function of sample number.





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