

## Real-Time Monitoring of Milk Powder Browning and Oxidation Products, Using SIFT-MS

Spray drying of milk solids is necessarily an exacting process. Under the conditions encountered in spray dryers, milk powder may undergo a series of exothermic reactions that lead to increased temperatures and potentially explosive combustion, which may cripple an entire facility.<sup>1</sup> The likelihood of explosive combustion is difficult to anticipate, as it depends on several factors, including the properties of the milk being dried. Combustion is currently detected via constant monitoring of carbon monoxide (CO) concentration in the dryer, as CO is a combustion product.<sup>2</sup> This method is not without its drawbacks, such as the possibility of interference from other combustion sources and the short time interval between detection of powder combustion and imminent explosion. A more suitable method would detect the presence of products unique to reactions occurring in the dryer, and be capable of monitoring the processes that lead to combustion to predict the event within a reasonable time frame.

This study represents the first stage in developing a Selected Ion Flow Tube Mass Spectrometry (SIFT-MS) method for the real-time detection of milk powder reaction products relevant to spray drying. The primary reactions occurring under these conditions are Maillard and lipid oxidation reactions. The VOCs observed using SIFT-MS were products of these reactions, and these offer great potential for providing an early warning of hazardous situations in milk powder spray dryers.

### Experimental

A new analytical technique, SIFT-MS, identifies and quantifies Volatile Organic Compounds (VOCs) directly from air in real-time.<sup>3,4</sup> Based on sound principles of chemical ionization mass spectrometry and precisely controlled ion reaction kinetics, SIFT-MS uses a sequence of three reagent ions to resolve interfering

species, differentiate isobaric compounds and produce intrinsically quantitative data without laborious calibration procedures.

Samples of 0.1 g of generic branded, New Zealand-made skim and whole milk powders were used in this study. The experimental setup is shown in Figure 1. Milk powder was placed inside a 0.5-inch o.d. stainless steel tube (the "sample tube"), which was loosely plugged at both ends with glass wool to position the powder at the center of the heating furnace during the experiment.

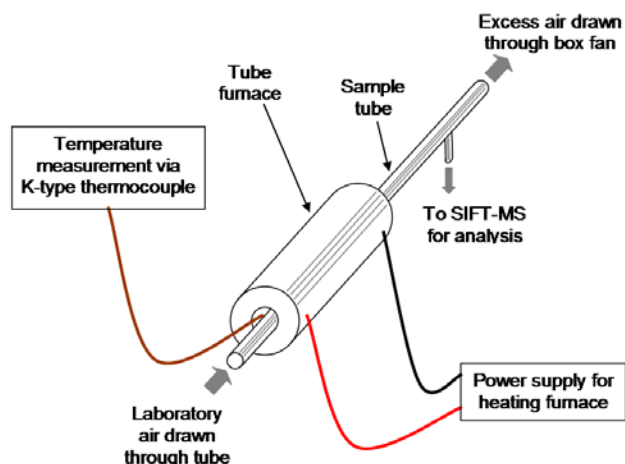


Figure 1: Experimental setup for heating and analysis of milk powder samples.

The tube was placed in a resistively-heated tube furnace and heated at constant voltage from room temperature to approximately 180 °C, which was measured using a K-type thermocouple placed in the furnace next to the sample tube. Laboratory air (i.e. containing 21% oxygen) was pulled through the sample tube and past the SIFT-MS inlet by a small box fan at a flow rate of approximately 250 standard cubic centimeters per minute (scm). The gas stream was tangentially sampled into the SIFT-MS at a flow rate of 30 scm.

## Results and Discussion

Figure 2 shows the scan-by-scan evolution of the  $\text{NO}^+$  reagent ion full mass scan as the temperature of 1 gram of skim milk powder rises from 80 °C to 150 °C. Significant changes occur over time and the peaks are readily identifiable as compounds that are known to occur in browning skim milk powder.<sup>5-7</sup>

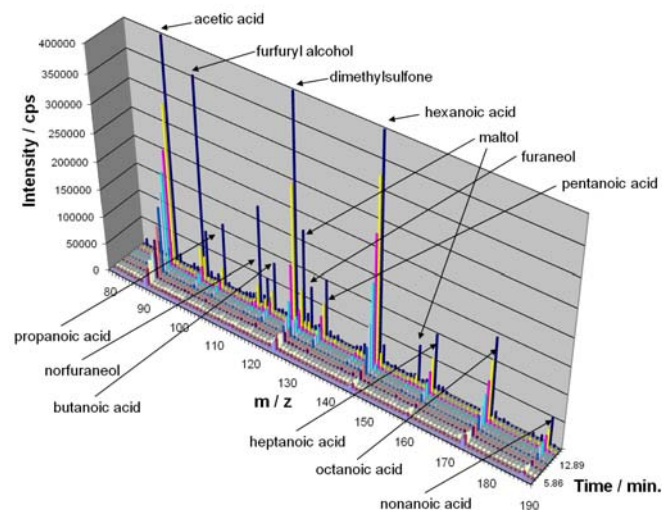


Figure 2: A three-dimensional representation of an expanded region of the mass scan data obtained using the  $\text{NO}^+$  reagent ion of SIFT-MS over the duration of an experiment in which 1 gram of skim milk powder was heated. The temperature increased from approximately 80 to 150 °C over the time shown.

Figures 3 to 5 present the results of selected ion mode (SIM) analyses for volatile fatty acids, heterocyclic compounds and miscellaneous compounds when 0.1 grams of milk powders were heated to approximately 180 °C. Experiments on samples of 0.1 to 1 gram of exhibited very similar behaviors. All compounds were analyzed in the same experiment with a cycle time of less than seven seconds.

It is evident from the data in Figures 3 to 5 that although there are some significant differences between skim and whole milk powders, the same compounds dominate the headspace of both powders over this temperature range. The heterocyclic compounds are well-known Maillard reaction products<sup>5,6</sup> characteristic of foods comprised of proteins and reducing sugars. Various volatile fatty acids have been reported previously for browning<sup>5,6</sup> and pyrolyzing<sup>7</sup> milk-based powders.

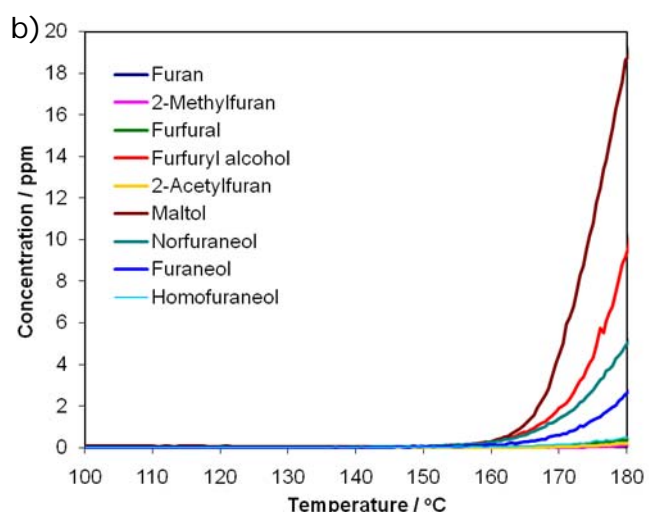
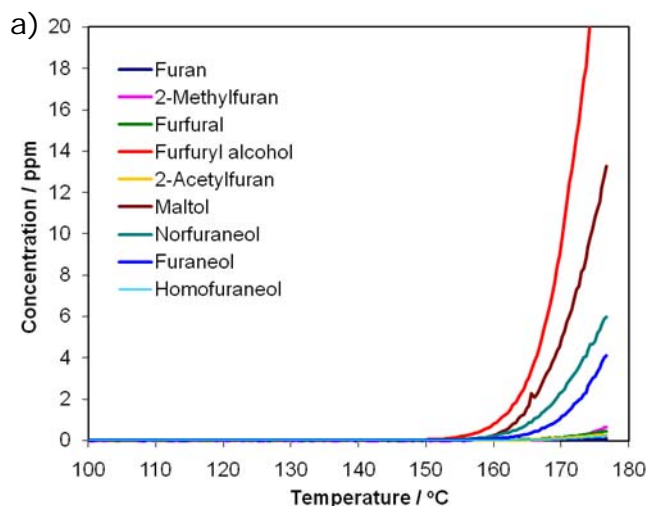


Figure 3: SIFT-MS SIM results for various volatile fatty acids produced when 0.1 g of (a) skim- and (b) whole-milk powders were heated in the tube furnace.

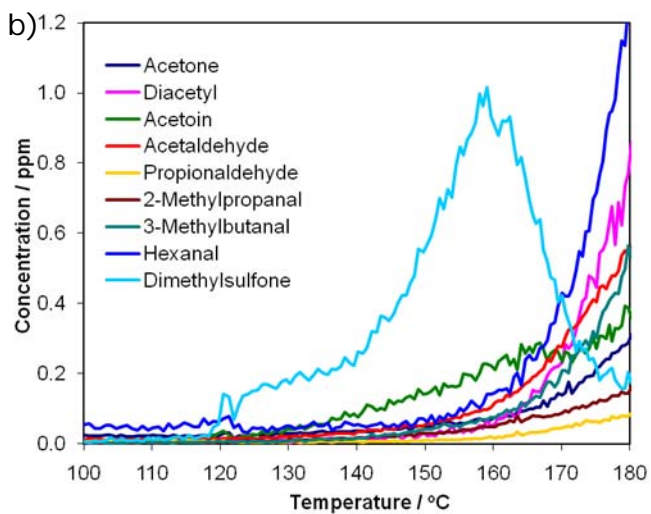
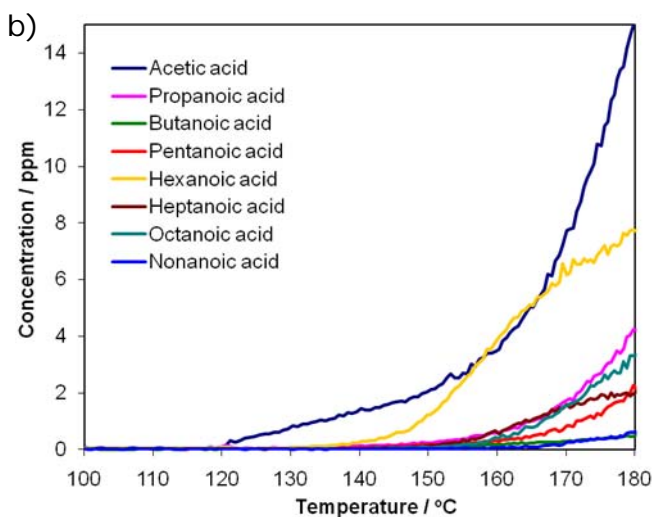
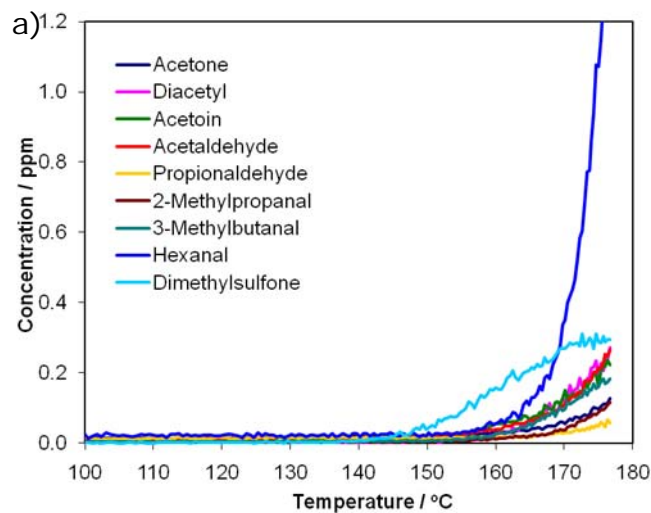
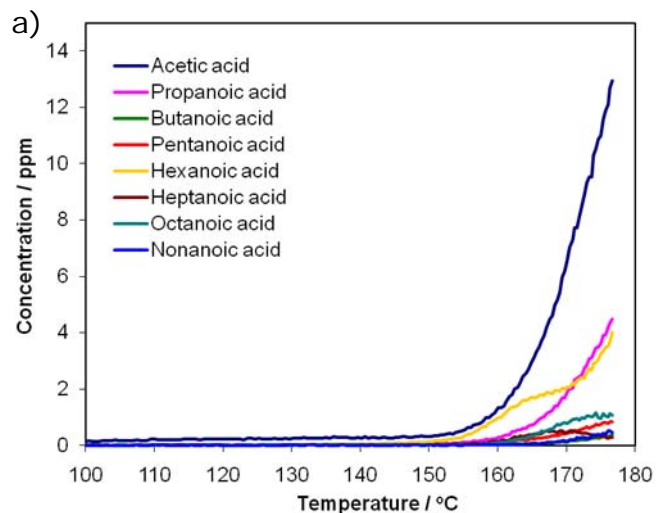


Figure 4: SIFT-MS SIM results for several heterocyclic compounds produced when 0.1 g of (a) skim- and (b) whole-milk powders were heated in the tube furnace.

Figure 5: SIFT-MS SIM results for miscellaneous compounds produced when 0.1 g of (a) skim- and (b) whole-milk powders were heated in the tube furnace.

## Conclusion

The results presented here demonstrate that the Syft Technologies Voice200<sup>®</sup> SIFT-MS instrument can sensitively detect and quantify compounds indicating the early stages of smoldering in both skim and whole milk powder. This offers the potential to provide much more specific information on the state of milk powder in drying towers than is possible via detection of CO alone, which is usually produced at more advanced stages in the smoldering process.<sup>2</sup> Quality control applications are also feasible.

Sensitive detection of Maillard reaction products in real time using SIFT-MS also provides the baking industry with a unique opportunity to monitor the browning process via important volatile flavor compounds.

For more information about this unique technology, please contact your nearest Syft Technologies office or visit [www.syft.com](http://www.syft.com).

## References

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International enquiries  
Syft Technologies Ltd.

3 Craft Place Middleton  
PO Box 28 149  
Christchurch, New Zealand  
Website [www.syft.com](http://www.syft.com)

Phone +64 3 338 6701  
Facsimile +64 3 338 6704  
Email [sales@syft.com](mailto:sales@syft.com)

North American enquiries  
Syft Technologies Inc.

1525 Park Manor Blvd, Suite 272  
Pittsburgh, PA15205 4805, USA  
Phone 888 200 5991  
Email [NAInfo@syft.com](mailto:NAInfo@syft.com)