

**Comprehensive two-dimensional  
supercritical fluid and  
gas chromatography  
(SFCxGC)**

**By**

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Submitted in partial fulfillment of the requirements for the degree  
**Doctor of Philosophy**

In the

**Faculty of natural and agricultural sciences  
University of Pretoria**

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## **Summary**

A novel chromatographic method was devised that makes use of the superb group separation power of normal phase supercritical fluid chromatography (SFC) combined with a fast second separation by a resistively heated gas chromatograph (GC). The SFC was operated isothermally with stopped flow to provide the time required for the GC analysis. The GC analysis had a typical cycle time of 1 minute. During this time the GC column was independently heated at a rate of 450°C/min to 250°C and actively cooled down again to -50°C before the next GC injection takes place. This was achieved with an in-house designed, resistively heated, temperature programmable gas chromatograph. Various temperature measurement circuits were also evaluated. An interface was developed that allows transfer between the SFC and the GC in such a way that the entire eluent from the first separation is analyzed by the second separator. Chromatographic resolution was not lost during the transfer process from the first to the second separation stages. The interface also allows for the exchange of the carrier gas used in the second gas chromatographic separation to provide for the maximum separation speed. In the first separation, a silica gel packed column and the novel application of a silica gel porous layer open tubular capillary column was used for SFC group separation. The SFCxGC<sub>ftp</sub> was applied to petrochemical samples and essential oils and the results were compared to that obtained with a commercially available GCxGC system.

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# List of LabVIEW Programs

(Requires LabVIEW 5.1 or higher)

## 1. Fast GC Phillips circuit

### a. Simple control.llb

- Run *simple control.vi*

### b. Temperature calibration.llb

- Run *temperature calibration.vi*

## 2. Fast GC Resistive heating circuit

### a. Simple control.llb

- Run *simple control.vi*

### b. Temperature calibration.llb

- Run *temperature calibration.vi*

## 3. Fast GC with thermocouple

### a. Fast GC.llb

- Run *fast GC.vi*

### b. Simple control.llb

- Run *simple control.vi*

## 4. SFCxGC

### a. SFCxGC.llb

- Run *SFCxGC1.vi*

## 5. Data compilation

- Run *columns-to-matrix.vi*
-

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