Swern-Moffatt oxidation in a continuous flow microreactor

Analysis method setup

The methods below describe the analysis methods as used by *Future*Chemistry and act as a starting point or reference when setting up an analysis method on location.

GC method for benzyl alcohol and 1-phenyl ethanol

GC analysis was performed on a Shimadzu GC 2010 GC-FID equipped with a Quadrex 007 1701 column (length: 10 m, internal diameter: 0.1 mm, film thickness: 0.1 mm).

Table 1: GC program

Parameter	*Value*	*Parameter*	*Value*
Temperature program		**Split temperature**	300°C
0.0 – 0.85 min	98°C	**Injection volume**	1.0 µL
0.85 – 1.76 min	150°C/min	**Split**	200
1.76 – 1.86 min	235°C	**Column flow**	1.3 mL/min

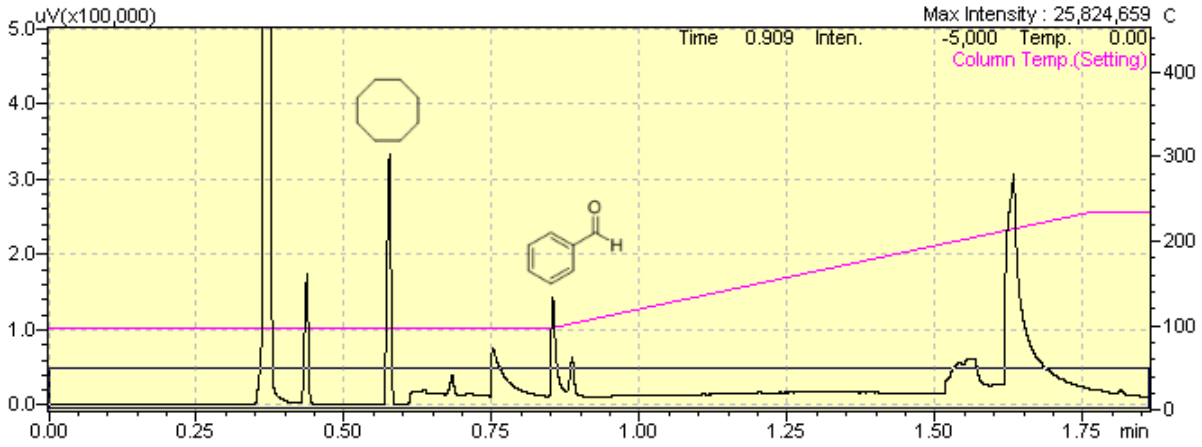

Figure 4: Example GC chromatogram (benzyl alcohol)

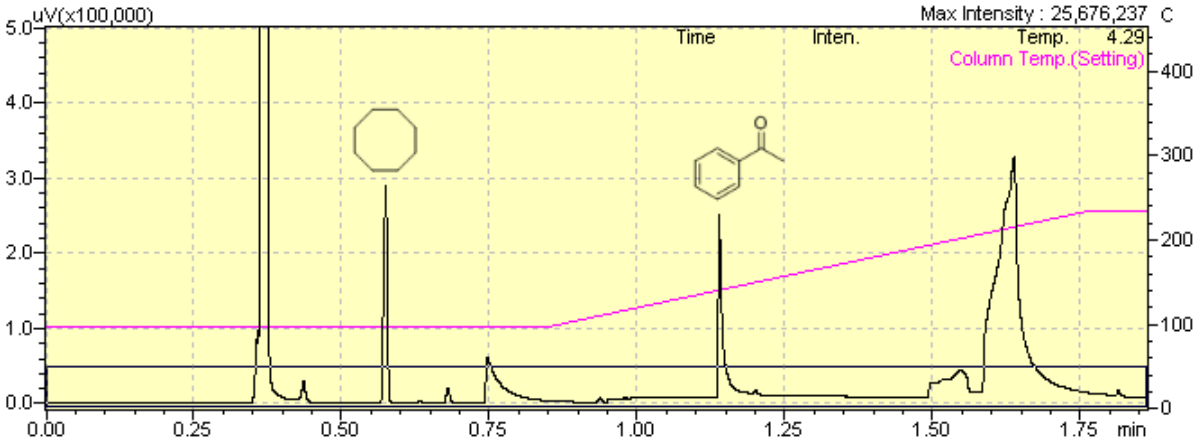

Figure 5: Example GC chromatogram (1-phenyl ethanol)

Table 2: Retention times of internal standard and products

Compound	Function	Retention time [min]
Cyclooctane	internal standard	0.58
Benzaldehyde	product	0.85
Acetophenone	product	1.15

Procedure:

- To set up a valid GC analysis method, first try to analyse a mixture of all components (i.e. a reaction mixture from your Basic Experiment) until all peaks are properly separated. A reference GC program is stated above, which acts as a starting point for your analysis method.
- Find the product and internal standard peaks by injecting these compounds (in DCM) onto the GC.
- How to solve?
    - If peaks overlap, decrease the temperature gradient (or use isocratic temperature).
    - If analysis takes too long, increase column starting temperature (and vice versa).

GC method for cinnamyl alcohol

GC analysis was performed on a Shimadzu GC 2010 GC-FID equipped with a Quadrex 007 1701 column (length: 10 m, internal diameter: 0.1 mm, film thickness: 0.1 mm).

Table 3: GC program

Parameter	Value	Parameter	Value
Temperature program		**Split temperature**	300°C
0.0 – 0.85 min	150°C	**Injection volume**	1.0 µL
0.85 – 1.70 min	100°C/min	**Split**	200
1.70 – 1.80 min	235°C	**Column flow**	1.25 mL/min

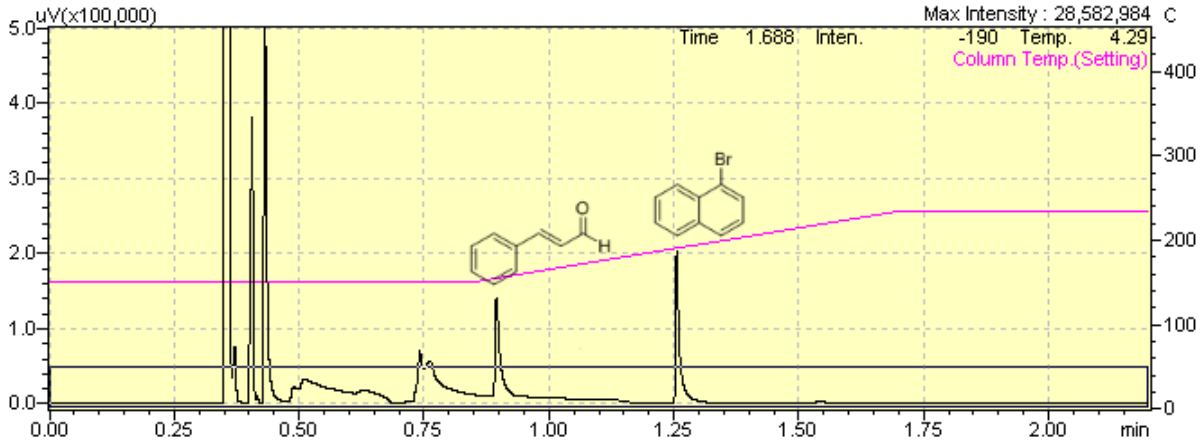

Figure 6: Example GC chromatogram (cinnamyl alcohol)

Table 4: Retention times of internal standard and products

Compound	Function	Retention time [min]
Cinnamaldehyde	product	0.90
1-Bromonaphthalene	internal standard	1.25

Procedure:

- To set up a valid GC analysis method, first try to analyse a mixture of all components (i.e. a reaction mixture from your Basic Experiment) until all peaks are properly separated. A reference GC program is stated above, which acts as a starting point for your analysis method.
- Find the product and internal standard peaks by injecting these compounds (in DCM) onto the GC.
- How to solve?
    - If peaks overlap, decrease the temperature gradient (or use isocratic temperature).
    - If analysis takes too long, increase column starting temperature (and vice versa).

Calibration

To measure percentage conversion of the formed product, a calibration is set up of the diketone substrate against an internal standard as in Table 2, using the concentrations from Table 3.

Table 5: Internal standard/substrate combination

Substrate	***Internal standard***	***Product***
Benzyl alcohol	Cyclooctane	Benzaldehyde
Cinnamyl alcohol	1-Bromonaphthalene	Cinnamaldehyde
1-Phenyl ethanol	Cyclooctane	Acetophenone

Table 6: Calibration samples

Sample	***Int.standard***	***Substrate***	***Corresponding yield***
1	20 mM	20 mM	100%
2	20 mM	15 mM	75%
3	20 mM	10 mM	50%
4	20 mM	5 mM	25%

Procedure:

- Prepare stock solutions of the internal standard (2-bromotoluene) and the products (benzaldehyde/acetophenone/cinnamaldehyde) in DCM. Use concentrations that can be diluted to the required sample concentrations using the pipettes available in your laboratory. (*The minimum amount you can accurately dispense from a pipette depends on the type and volume.*)
- Prepare four samples as in **Error! Reference source not found.**.
- Make sure you can analyse your samples with the GC analysis setup. If not, modify the analysis setup until valid chromatograms are obtained.
- From the obtained chromatograms, obtain peak areas.
- Set up a calibration method according to the literature.