

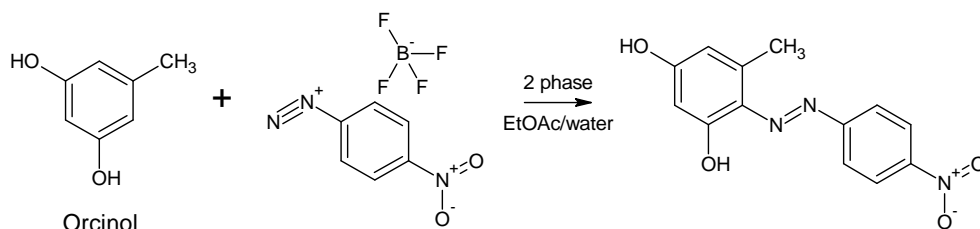
## AFRICA Application Note

Note Number : 12

### Biphasic Reactions

#### 1 Summary

A biphasic reaction involving electrophilic substitution of orcinol with a diazonium salt<sup>1</sup> has been examined in the AFRICA flow system. The two-phase product solution was separated in the FLLEX module to give the azo product in the organic stream and an aqueous waste stream.

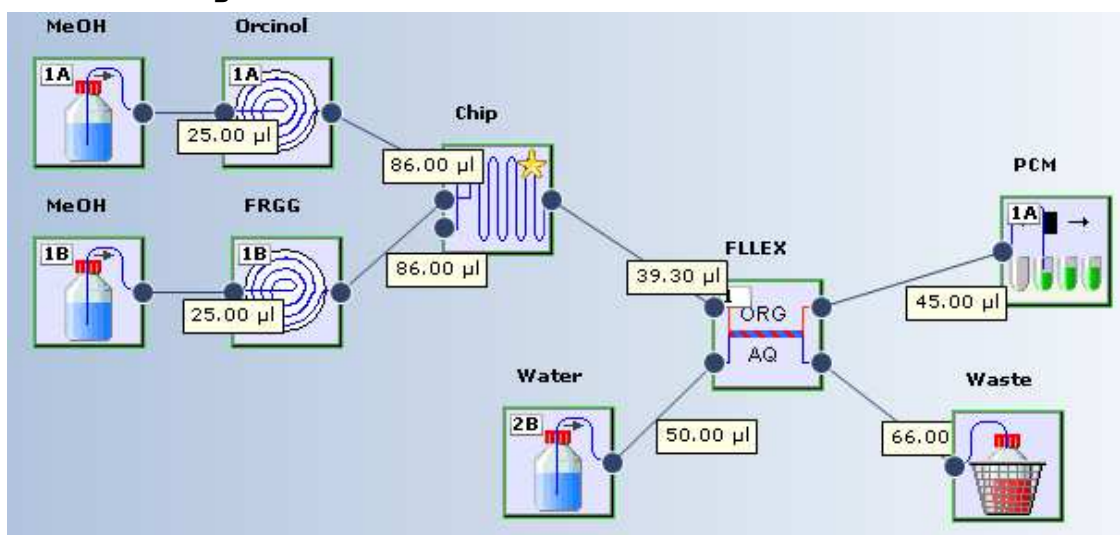


#### 2 Chemistry

Orcinol ( $10^{-3}$  molar in ethyl acetate) and 4-nitrobenzenediazonium tetrafluoroborate ( $10^{-4}$  molar in water) were pumped at equal rates into a 250 $\mu$ l reaction chip held at 20 $^{\circ}$ C with a reaction time of 1 minute. The resulting two-phase bright orange solution was separated in the FLLEX to yield the product azo compound in ethyl acetate and an aqueous waste stream.

The product azo solution was analysed by HPLC off-line and showed complete conversion of the starting diazonium salt.

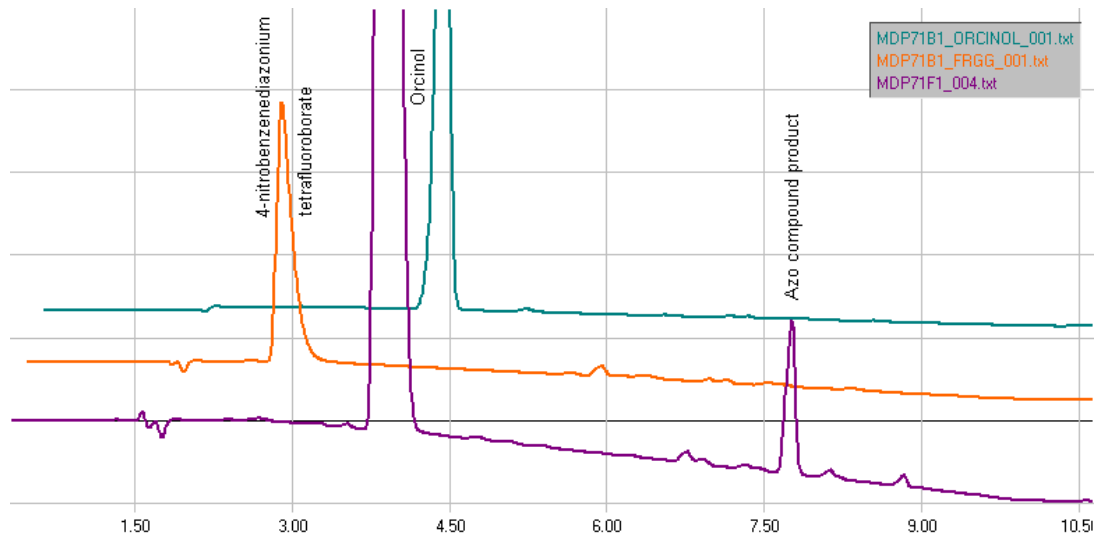
#### 3 Fluidics Layout



The fluidics layout for the reaction is shown above. The AFRICA system including FLLEX was controlled using Reaction Manager software.

<sup>1</sup> Hisamoto et al, Chem. Commun., 2001, 2662-2663

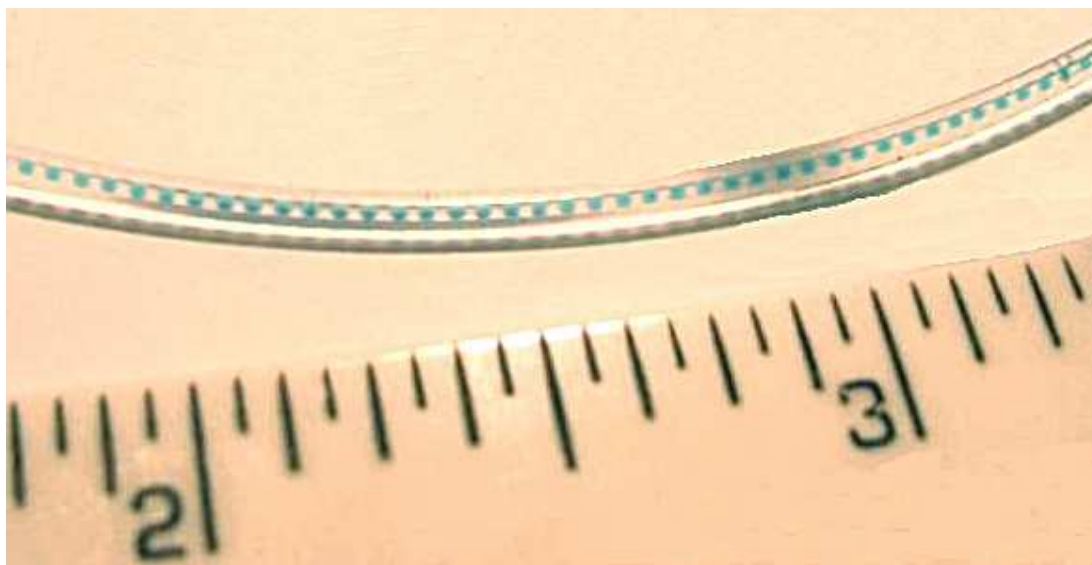
## 4 HPLC Analysis



Reverse phase HPLC was used to analyse starting materials and products. The traces above are both starting materials and the final separated organic phase, showing 100% consumption of the diazonium salt.

## 5 Biphasic flow

In addition to the normal glass chips, an FEP tube reactor system is available in for use with AFRICA with internal reaction volumes of 4 or 16mls.



This photograph shows a typical example of biphasic flow in 0.4mm inside diameter FEP tube. In this example, the aqueous phase has been dyed blue to ease visibility. Droplet integrity is maintained for a number of meters under these conditions, giving a large interfacial contact area for reactions involving phase transfers or for extractions.

## 6 Conclusion

The AFRICA flow system can be used for biphasic reactions. The biphasic reaction stream can be collected and separated off-line or passed to a FLLEX continuous extraction module for automatic phase separation.

Micro-flow reactors provide improved reaction conditions control over traditional batch reactors for bi-phasic reactions by improved control of interfacial area resulting in improved mass flow.