Technical note

Delta-8 Multichannel Microtensiometer: High Throughput Surface Tension of Ink Samples

Pekka Suomalainen

October 2, 2006

Overview

In the development of ink formulations the properties such as optimal wetting and stable drop formation have to be ensured by adjusting the surface tension of the base product. In addition to surfactants the formulated systems usually include components as dyes or pigments, dispersants, humectants and solvents, many of which themselves affect the surface characteristics of the final product. Monitoring such large environment spaces has conventionally required vast amounts of sample material. Additionally, conventional surface tension instruments are limited by their slow throughput for this kind of tasks.

The purpose of this technical note is to introduce a high throughput screening tool, Kibron Delta-8, to solve the above bottleneck. Taking advantage of 96-well microtiter plates and a novel microbalance, surface tension based assays can be analyzed fast, reliably, using low volume samples.

Experimental

Surface tension measurements were carried out on a Kibron Delta-8 multichannel microtensiometer in standard configuration. The instrument utilizes eight parallel microbalances and probes fixed to meet the positions of the wells in a 96-well format. The resolution of the balances is 0.02 mN/m. The surface tension measurement is based on the *Du Nouy* -method, *i.e.* the maximum force exerted by the surface tension is recorded as the thin needles (*probes*) are withdrawn from the solutions. The probes have a diameter of 0.5 mm and the measurement solution is completely wetting their surface. The instrument features automatic cleaning of the probes by heating. A possibility to allocate a special cleaning solution in the well(s) and row(s) of the 96 well plate also exists. This might be a desired step prior to heating in the case of viscous samples or as a single cleaning procedure in the case of UV-curable samples, which may prevent the use of electric cleaning unit. The software which controls the Delta-8 (*ScriptWorks*) provides programmable scripting language(s) for designing the experiments. The standard measurement loop, i.e. the cleaning of the probes and the measurement of the 96-well plate, takes approx. 10 minutes.

Set of three different ink-samples were used in a study: *eco* -solvent based ink, *pigment* ink, and *dye* ink, all provided by a well established ink-manufacture in Europe. Ink dispensing to a 96-well plate was done with *Biohit* 8-channel pipette. Fifty (50) microliters of each ink was allocated as displayed in Table 1. Additionally, Peg-200 was used to make sure probe cleaning has been efficient between the different ink samples. The cleaning step was carried out between every column measured. Prior any measurments the instrument was calibrated with DMSO to a value of 43.0mN/m.

In order to check the accuracy of the Delta-8, surface tension determinations were also carried out using the *Wilhelmy* technique (Kibron *DeltaPi*).

Results and discussion

Precision

Table 1 summarizes the measured surface tension values with the Delta-8 and provides the statistics on the reproducibility of 8 microbalances (channels) within the instrument. Standard deviations between parallel channels fell between one percent of the average values indicating channel independent operation. Small standard deviations observed, (Tables 1 & 2) for 4 different compounds studied, further demonstrates the efficacy of the cleaning procedure. A slight increase in the values can be seen, however (compare columns 11 and 5, 9 and 3 etc). The shift is likely due to changes in the composition and properties of the inks, which may vary over time due to changes in humidity and temperature. Also, the increase may be associated to negligible deviations from both the original radius and circularity of the cross-section of the probes, caused by the cleaning sequence. In the longer run, these can be avoided by re-calibration and eventually, by changing the probes.

Accuracy

Surface tension values measured using the *Wilhelmy* -technique gave 33.2, 32.6, and 29.2 (mN/m) for the *pigment*-, *dye*-, and *eco* -solvent based inks respectively. This is in line with the results obtained by the Delta-8 (Table 2). Importantly, the results more generally indicate that there is no compromise in accuracy and precision with an added speed or measuring from low sample volumes.

Concluding remarks

The results demonstrate the suitability of the Kibron Delta-8 for accurate, repeatable and fast determination of surface tension. Increased productivity of surface tension based research can be achieved by running experiments in parallel, and making use of automation and miniaturized assays.

Table 1: Surface tension values (mN/m) for ink-samples and statistics for individual microbalances (A-H).

Sample	**Eco**	**PEG**	**Pig**	**PEG**	**Dye**	**PEG**	**Eco**	**PEG**	**Pig**	**PEG**	**Dye**	**PEG**
Row/Column	**1**	**2**	**3**	**4**	**5**	**6**	**7**	**8**	**9**	**10**	**11**	**12**
A	28.9	45.4	32.2	45.6	33.1	45.4	28.8	45.4	32.2	45.3	32.9	45.4
B	29.0	45.7	32.4	46.0	33.1	45.6	28.9	45.6	32.4	45.5	32.9	45.5
C	28.8	45.4	32.2	45.6	32.9	45.2	28.7	45.3	32.1	45.2	32.7	45.3
D	29.3	46.0	32.7	46.4	33.5	45.9	29.2	45.9	32.6	45.7	33.2	45.6
E	29.4	46.0	32.8	46.7	33.5	46.0	29.3	46.0	32.7	45.8	33.3	45.8
F	29.0	45.6	32.5	46.2	33.2	45.7	28.9	45.9	32.5	45.5	33.0	45.5
G	29.3	45.9	32.7	46.1	33.3	45.6	29.1	46.0	32.5	45.4	33.0	45.4
H	29.3	46.4	32.7	46.1	33.4	45.7	29.2	45.9	32.4	45.5	33.0	45.5
Average	**29.1**	**45.8**	**32.5**	**46.1**	**33.3**	**45.6**	**29.0**	**45.8**	**32.4**	**45.5**	**33.0**	**45.5**
St.dev.	**0.23**	**0.34**	**0.24**	**0.37**	**0.21**	**0.26**	**0.22**	**0.28**	**0.20**	**0.20**	**0.19**	**0.15**
CV %	**0.77**	**0.75**	**0.73**	**0.81**	**0.64**	**0.56**	**0.75**	**0.61**	**0.61**	**0.43**	**0.56**	**0.33**

Table 2: Overall statistical analysis for measured samples (Number of measured samples is shown in parentheses).

Stats/Sample	**Eco** (16)	**Pig** (16)	**Dye** (16)	**PEG** (48)
Average	29.1	32.5	33.1	45.7
St.dev.	0.2	0.2	0.2	0.3
CV %	0.7	0.6	0.7	0.7