

Guide to Dispersion Measurement

Purpose

Obtain a reliable measurement of instrument bandwidth.

Materials

Calibrated flow meter

ZDV union (Upchurch p/n U-411).

Conditions

Mobile Phase: 40:60, water:methanol

Sample: 1.0% acetone (v/v) in mobile phase

Flow: 100 μ L/min. (0.1 mL/min.)

Data Sampling Rate: \geq 10 Hz

Filter Response Time: \leq 0.1 sec.

Inj. Vol.: 0.5 μ L

Method

1. Connect ZDV union in place/position of column
2. Start flow; allow ample time for flow to stabilize and flow meter to obtain stable reading
3. Inject sample

Alternate Method: (To be used if column inlet and outlet tubing is not long enough to connect to the ZDV union)

1. Connect the ZDV unions (52807-U) to the column inlet and outlet tubing.
2. Install the High Performance Double ended fitting (53682-U) to the ZDV unions connected in Step 1. The High Performance Double ended fitting should now be connected in place of a column.
4. Start flow; allowing ample time for flow to stabilize and flow meter to obtain stable reading.
5. Inject sample.

Record

a) retention time (r.t.)

b) efficiency by peak width-at-half-height method (N)

Be sure baseline drawn is flat to insure an accurate measurement of peak height; (may need to adjust integration parameters to achieve this).

Results

$$\sigma = (\text{r.t.} * \text{flow}) / \sqrt{N} \quad [\text{make sure flow is in units of } \mu\text{L/min.}]$$

$$\text{Instrument Bandwidth (IBW)} = 4 * \sigma$$

Discussion

If $\text{IBW} \leq 25 \mu\text{L}$, well retained peaks ($k \geq 2$) can display efficiencies $\geq 90\%$ of specified column performance for 10 or 15 cm columns of 4.6 mm I.D.; if the length is 5 cm, peak efficiencies may be somewhat lower.

If $\text{IBW} \geq 25 \mu\text{L}$, well retained peaks ($k \geq 2$) can display efficiencies of 80-90% of specified column performance only for 10 or 15 cm columns of 4.6 mm I.D. If smaller columns are desired, system volume must be reduced by use of smaller flow cells and smaller I.D. tubing.

Re-ordering Information:

52806-U

HPLC Dispersion Measurement Kit

53682-U

High Performance Double ended fitting

52807-U

Nano Tight Union with 0.007 in. Thru-hole, pk. of 1