Flow cells with GC working electrode

QC procedure

Reference	User manuals (100.0010, 102.0010 and 110.0010)
Instrumentation	Flow Injection Analysis (FIA) system in optimum condition consisting of:

- LC 110 pump, analytical (193.0035) /LC110S pump (193.0035S)
- OR 100 organizer rack (184.0035) with pulse damper and degasser
- 2 DECADE II™ detectors with 4 channels (171.00xx)
- Manual injector with 2 mL loop
- C18 column, 53 x 4.0 mm ID, particle size 5 µm (730.0004)
- 3, 5 and 9-port manifolds (low-dead volume)
- Back-pressure regulator of 40 psi (leakage test)

Pre-conditioned column must be electrochemically clean. If not, the background current will be unacceptably high. For reversed phase columns flushing with 80% methanol (in water) for 3 days at a low flow rate is recommended. Integration software: Dialogue™ Elite.

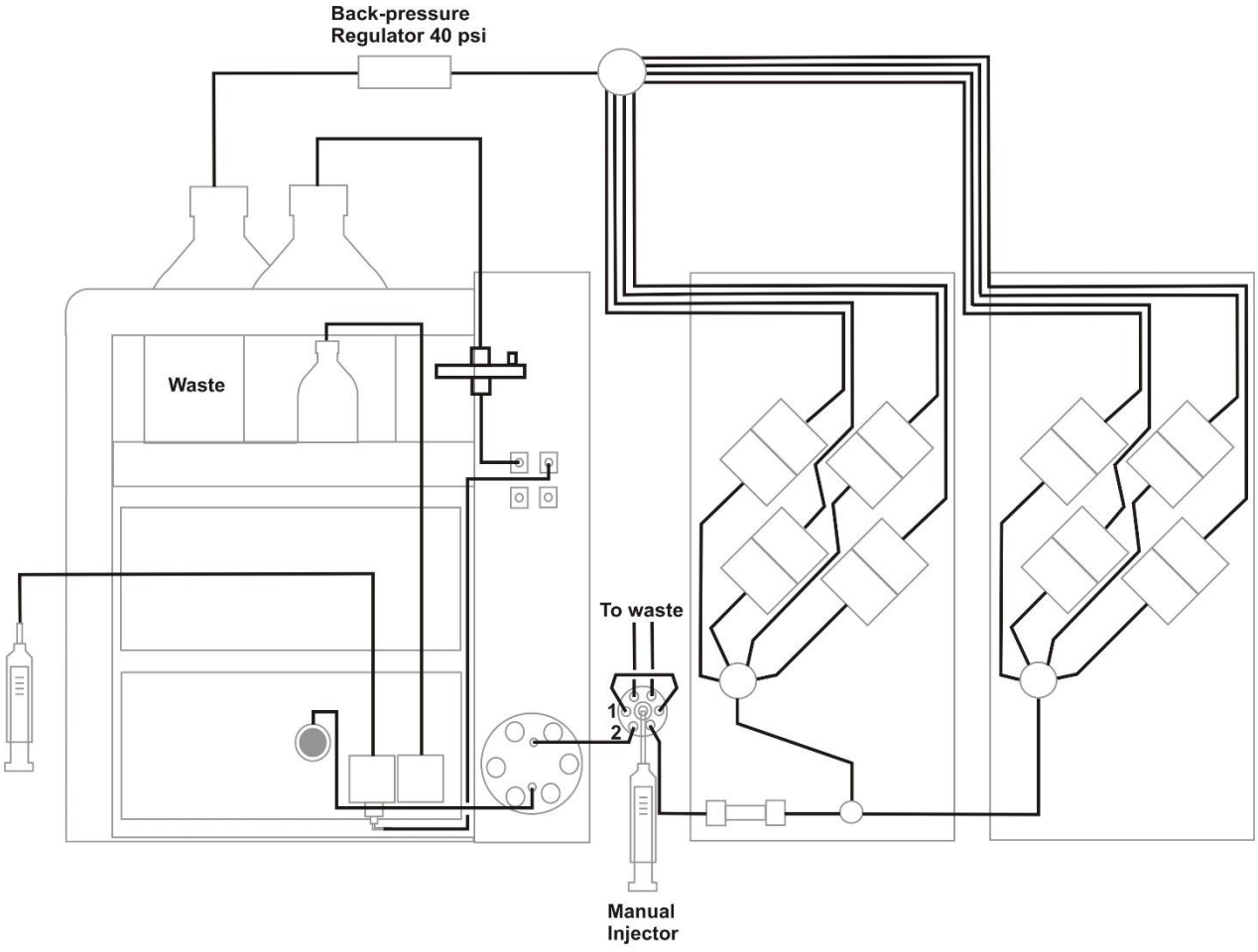

Figure 1. Schematic representation of the QC set-up with 8 VT-03 flow cells.
Note: the manual injector is replaced by a Genie syringe pump.

QC procedure

The analytical performance of flow cells with glassy carbon (GC) WE is checked by measuring the S/N ratio. The *signal* is measured by analysing a sample in an optimised *pseudo* flow injection analysis system, as schematically depicted in figure 1 on the previous page. All chemicals, including water, should be of the highest purity available. In the test set-up 8 flow cells are tested simultaneously.

1. Flow cells must be installed as described in the installation guide in the flow cell user manual. For system configuration see Fig. 1. Prepare the system with the conditions as given in Table 1.
2. New or refurbished flow cells are allowed to stabilize for at least 24 hours at a flow rate of 300µL/min before starting a QC test.
3. The cells must be run for at least 4 hours at a flow rate of 2 mL/min and visually checked for leakage. At 0.5 mL/min the 40 psi regulators generate a back pressure of approximately 2.80-3.0 bar under the specified LC conditions.
4. Switch on the HPLC pump at a flow rate of 1.0 mL/min. After ½ hour of stabilisation the system is ready for a noise measurement. Measure the baseline noise for 15 minutes with a detector setting of 1 nA/V, a data rate of 5 Hz and a filter setting of 0.5 Hz. The peak-to-peak noise is measured as the average of 30 segments of 30 seconds. The peak-to-peak noise for each segment is determined by an Excel routine and calculated as: n = highest value – lowest value. Each segment has 150 data points, the 5 highest and 5 lowest values are rejected.
5. Switch on the HPLC pump at a flow rate of 2 mL/min and let the system stabilize for 10 minutes. Inject 3 times 2mL of 0.1 µmol/l Noradrenaline dissolved in mobile phase. Range: 50 nA/V, data rate 5 Hz and filter setting of 0.5 Hz. Run time: 15 minutes.

Table 1. HPLC-EC conditions for performance check.

Detector	DECADE II, 2 detectors in parallel each with 4 channels
Output	Digital RS232 output of the DECADE II
Column	Reversed Phase C18, 53 x 4.0 mm ID, 5 µm, pre-conditioned for ECD
Mobile phase	PQ buffer (50mM Acetic acid; 0.1 mM EDTA; 2 mM KCl; pH 4.5) with 5% methanol; the buffer must be properly degassed, and filtered at 0.2 µm
Sample	2 mL injection of 0.1 µmol/l noradrenaline (dissolved in mobile phase)
Flow rate	Stabilisation: 300 µL/min, Noise test 1.0 mL/min, signal measurement: 2.0 ml/min. Flow rate and sample are splitted over 8 cells.
Flow cell	VT-03 with 0.7 mm GC WE (71xx), 2 mm GC WE (28xxx) , 3 mm GC WE (49xxx), FLEXCELL™ (20Axxx) or Reactor™ cell (102xxx) with GC WE disk
E-cell	+770mV (*vs.* Ag/AgCl in saturated KCl), +620 mV (vs. ISAAC™) +420 mV (vs. HyREF™)
Integrator	DECADE II dialogue data-acquisition software, 5 Hz data rate

QC criteria

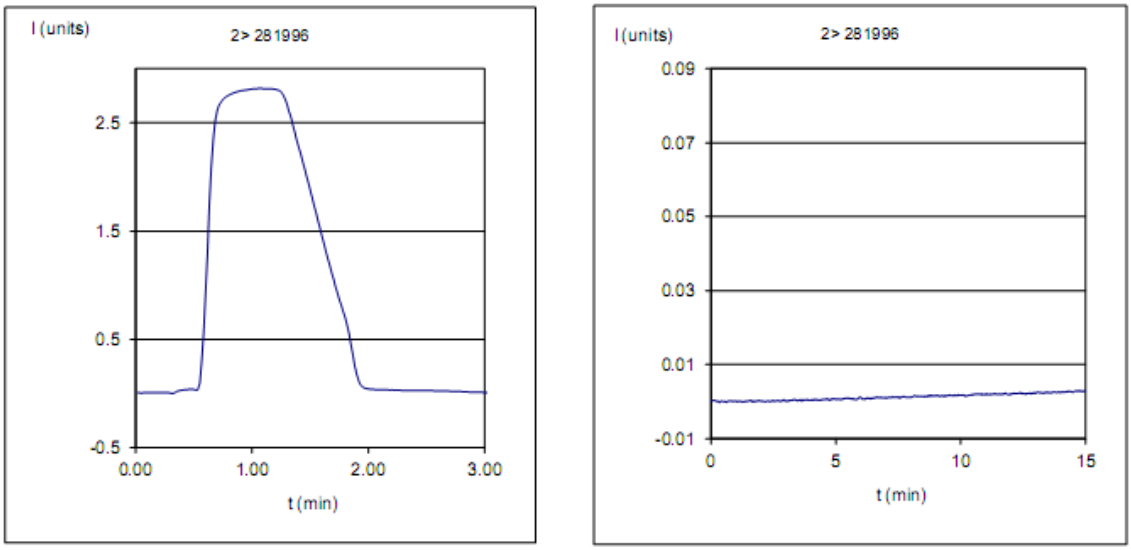

Fig. 2. Flow injection analysis of 0.1 µmol/L noradrenaline at 50 nA/V (left). Noise measurement at 1 nA/V (right).

QC criteria VT-03 flow cells with 2 - 3 mm GC WE

VT03 Cell	**Specified**	
	3 mm ID WE	2 mm ID WE
Signal	> 3 nA	> 1.5 nA
Noise (p-p)	< 4.6 pA	< 3.1 pA
Signal-to-noise	> 1600	> 1600

QC criteria FLEXCELL and Reactor cell

FlexCell/Reactor	**Specified**
Signal	> 6 nA
Noise (p-p)	< 19 pA
Signal-to-noise	> 500

QC criteria SenCell

SenCell	**Specified**
Signal	> 4 nA
Noise (p-p)	< 3.1 pA
Signal-to-noise	> 1600