

# DeltaChrom™ SCS 200 Sample Clean-up System

## User manual



Rev. A, 11/2000

P/N WI-DR00112301



349 N Science Park Rd., State College PA 16803  
Phone: 800-345-5557 / 814-234-7317 Fax: 814-238-6072  
[www.laballiance.com](http://www.laballiance.com)

## CONTENTS

<b>CONTENTS.....</b>	<b>2</b>
<b>INTRODUCTION.....</b>	<b>3</b>
<b>CONTROL MODULE SCS 200 .....</b>	<b>4</b>
CONNECTIONS.....	4
OPERATIONS .....	4
<b>250 x 16 MM STEEL COLUMN PAH PREP 10 µm.....</b>	<b>7</b>
DESCRIPTION .....	7
INSTALLATION AND CALIBRATION .....	7
SUITABLE MOBILE PHASES .....	7
SAMPLE ANALYSIS .....	8
STORAGE AND MAINTENANCE .....	8
SPECIFICATIONS: .....	8
<b>SCS 200 INSTALATION .....</b>	<b>10</b>
SITE PREPARATION.....	10
SYSTEM INSTALLATION.....	10
<i>Mobile phase inlet .....</i>	<i>10</i>
<i>Power.....</i>	<i>10</i>
<i>Active piston backflush.....</i>	<i>10</i>
<i>Pump priming.....</i>	<i>10</i>
<i>Waste .....</i>	<i>10</i>
<i>Column flushing .....</i>	<i>11</i>
<b>SCS 200 OPERATION .....</b>	<b>12</b>
<b>TECNICAL SUPPORT .....</b>	<b>13</b>

## INTRODUCTION

This User manual contains all information necessary for installation and operation of DeltaChrom™ SCS 200 Sample Clean-up System.

SCS 200 isolates one fraction from injected sample by gel permeation chromatography (GPC). The system consists of a Series I isocratic pump, sample injector RH-7010, GPC column, two-position selection switch, backpressure relief valve, and control module.



**Picture 1: Sample Clean-up System DeltaChrom™ SCS 200**

Sample (fat or vegetable oil containing pollutants) is injected through the injector on the column. During the elution the sample components are separated and the fraction, containing desired pollutants, is collected in predetermined time to a prepared vessel. Flow rate, total analysis time, start and duration of fraction collection are set up on the control module.

## CONTROL MODULE SCS 200

### Connections

On the back of control module there are six connectors:

A	B	C
D	E	F

Connector A controls the pump (connected to a RS232 port of the Series I or II pump).

Connector C is for optional PC control

Connector D is for the starting switch of the sample injector

Connector E is for the selection valve

Connector F is for the power supply.

### Operations

After connecting to a power source, the initial display appears:

DeltaChrom SCS 200
1.00

By pressing MODE a programming mode is entered and the parameters can be keyed in. By pressing RUN the pump is started and the system is in ready state awaiting injection.

MODE switches between programming and active mode. After entering the parameters in program mode the system is set to ready mode by pressing MODE again.

The display appearance is shown below for both modes, the display can be scrolled up and down by the UP and DOWN arrows. On first line is a parameter name, on second is editable numerical value.

Programming mode:

xxx is last entered value

---

FLOW RATEx.xx [mL/min]

---

---

RUN TIMExxx.x [min]

---

---

COLLECT STARTxxx.x [min]

---

---

COLLECT TIMExx.x [min]

---

---

Active mode

---

READY (RUN) (xxx.x)WASTE (COLLECT)

---

The text (alternatives in parenthesis) displays the system status. In Ready mode it is possible to stop the pump by pressing CL (the initial display is shown)

By injecting a sample (or pressing RUN) the analysis is started, the run time and selection valve position are displayed. After the preset run time elapses, the selection valve is for short period of time switched to collect position to flush the collection capillary with fresh solvent not to contaminate the next collected fraction. Then is the system ready for next analysis.

In Run mode the keys CL, RUN and MODE are inactive. Pressing DOWN arrow can interrupt the analysis, when following display appears:

---

CL stop    0    manual

---

By pressing CL the system is in Ready State and by pressing CL again the pump is stopped.

By pressing 0 a Manual mode is selected and the system functions can be controlled manually according to displayed instructions.

## 250 x 16 MM STEEL COLUMN PAH PREP 10 µm

### Description

The column is designed for isolation of pollutants (PCB's, PAH's, pesticides) from oils and fats by gel permeation chromatography. The column is packed by a rigid, macroporous PS-DVB sorbent with a mean pore diameter of 50 nm and particle diameter of 10 µm. With proper solvent selection, the selectivity can be optimized for different types of pollutants

### Installation and calibration

The column is connected to a system and flushed by at least 50 mL of solvent at flow rate of 2-3 mL/min. Before use, the column should be calibrated as follows:

a) Elution volume of fat: 200 mg of refined vegetable oil or other water free fat in mobile phase (max 10% w/w) is injected on column and eluted at 3 mL/min. The elution is monitored by suitable detector (RI or UV) or gravimetrically after evaporation of collected fractions. The fat should be fully eluted within first 30-35 mL.

b) Elution of pollutants: The pollutant dissolved in mobile phase at concentration corresponding to expected content in fat and the sensitivity of used analytical method is injected on column and eluted. Starting from 25 mL, 5 mL fractions are collected, evaporated and analyzed for injected pollutant. The pooled fractions should contain at least 90% of injected pollutant.

If the separation of fat and pollutant is not satisfactory, it can be improved by changing the mobile phase composition or by decreasing the injected sample quantity. The concentration of sample should not exceed 10 % and the sample solvent should be close to the mobile phase composition, otherwise a elution zone deformations can occur

The used solvent purity is critical for this application, blank runs should be performed to exclude contamination.

### Suitable mobile phases

By selecting suitable mobile phase, the elution of different pollutants can be optimized. The column is compatible with all commonly used GPC solvents. Recommended are chloroform, dichloromethane, and mixtures of cyclohexane with ethylacetate, dichloromethane or acetone in different ratios. With chloroform and dichloromethane, the separation is governed mainly by size exclusion effects, by using cyclohexane mixtures with the above

mentioned solvents, considerable adsorption effects take place (please note that the elution of PAH can be quite lengthy in such case).

## Sample analysis

Sample (corresponding to 200-300 mg of fat per injected volume) is dissolved or extracted in suitable solvent. The solution should be dried (by anhydrous Na<sub>2</sub>SO<sub>4</sub>) and filtered (0,45 µm membrane filter). The mobile phase composition should be matched if possible (E.g. if cyclohexane/ethylacetate is used as mobile phase and extraction is performed by hexane, the sample should be diluted with appropriate amount of ethylacetate before injection). After injection, a fraction containing the pollutants is collected in predetermined time. The collected fraction is concentrated by evaporation, the residue is dissolved in suitable solvent and analyzed by appropriate method (PCBs by GC-ECD or GC-MS, PAH's by HPLC with fluorimetric detection). Residual traces of fat can be removed by mixing the sample in isooctane with a few drops of concentrated sulfuric acid (only for stable analytes).

## Storage and Maintenance

Chlorinated solvents release chlorine on oxidation, which is harmful to both column material and sorbent. For long term storage, the column should be flushed with noncorrosive or at least stabilized solvent. The column should be protected before particulates by a suitable filter. Samples containing water can lead to irreproducible results. The column must be stored tightly capped, it should not dry out.

## Specifications:

Catalogue Number:	WI-02077
Column Dimensions	250 mm x 16 mm ID
Sorbent	Styren-divinylbenzene copolymer, mean pore diameter 50 nm
Solvent in column	cyclohexane-ethylacetate 50:50
Particle diameter	10 µm
Recommended flow rate	1.0 –2,0 mL/min
Max. operating pressure	7 MPa
Max. temperature	50 °C



Efficiency (toluene, 20 µl injection)	> 20 000 tp/m
Asymmetry (toluene)	0.6 - 1.5

## SCS 200 INSTALATION

### Site preparation

System SCS 200 is designed for standard laboratory conditions. It should be placed on flat surface in well ventilated area.

Recommended temperature is  $10 \div 30$  °C, non-condensing, relative humidity  $20 \% \div 90 \%$ .

### System installation

#### ***Mobile phase inlet***

Connect the inlet capillary to the pump, install the inlet filter on the other end and place the capillary to the mobile phase container

#### ***Power***

Connect the control module to the pump, sample injector, selection valve and power supply according to the description on page 5. Connect the pump power cord to the pump and power socket and place the Control module power supply to another power socket. Switch on the pump.

#### ***Active piston backflush***

For SCS 200 applications the backflush is usually not used.

#### ***Pump priming***

Disconnect the column and connect to the capillary by suitable union 20 mL syringe. Fill the inlet capillary by suction with mobile phase and flush the pump by prime. Do not forget to flush also the sample loop (sample injector should be in inject position). Flush the pump with at least 20 mL of mobile phase at  $3 - 5$  mL/min.

#### ***Waste***

The three waste capillaries (leading from selection valve in waste position, sample injector waste and backpressure regulator should be securely placed in suitable container. The outlet from backpressure regulator must be controlled regularly, if liquid

appears there, the system pressure limit is exceeded due to some blockage at column inlet or sample injector..

### ***Column flushing***

Before first use, the column should be flushed with at least 50 mL of fresh mobile phase at 3 mL/min and calibrated according to the procedure described on page 8.

## SCS 200 OPERATION

After installation described in the previous chapter, set up the desired parameters on control module and put the system in Ready mode. Put a suitable collection vessel to a holder and place the collection capillary (left on the selection valve in the front view).

Set the sample injector to the Load position and fill the loop with sample by a suitable LUER syringe. At least 4 mL should be injected to ensure proper loop filling. Inject the sample and start a run by switching the sample injector into Inject position

After the preset analysis time elapses, change the collection vessel and inject next sample.

The function of system should be controlled regularly by injecting and analyzing known samples and checking the recovery. The volume of collected fractions also serves as a check for correct flow rate.

## TECNICAL SUPPORT

<b>Phone</b>	800-345-5557 814-234-7317
<b>Fax</b>	814-238-6072
<b>E-mail</b>	techsupport@laballiance.com
<b>Address</b>	349 N Science Park Rd. State College, PA 16803

Before contacting technical support please have ready following information:

<b>User</b>	
<b>Product</b>	DeltaChrom™ SCS 200
<b>Serial number</b>	for example. A04037
<b>Program version</b>	for example. 1.01