

H&D Fitzgerald Ltd

DENSITY GRADIENT COLUMN FILLER

OPERATING MANUAL

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To be used with firmware version 2.01

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1 Introduction

This manual describes the operation of the H&D Fitzgerald density gradient column filler.

- It provides easy filling of a density gradient column with a mixture of 2 liquids.
- It can use any two miscible liquids to give the lighter and heavier densities you need.
- There is no need to pre-adjust the densities of your two liquids to near to those at the top and bottom of the finished column.
For example, it can produce a column covering the range from 900 to 950 kg/m³ using pure water with a density of 997 kg/m³ and isopropyl alcohol of density 783 kg/m³.
- Column volume, and upper and lower densities are fully programmable.
- It can be programmed to add variable buffer volumes at the top and bottom of the column.
- Details of the previous filling routine are automatically stored for re-use in the next fill.
- It can drive an optional column sweeping mechanism at one of eight pre-programmed speeds.
- Once the column has been calibrated with a set of floats, the density and height of each float can be entered into the filler. The software will then automatically calculate the density of any other object in the column from its position.

2 Warnings and safety precautions

This filler has been made in accordance with the European and International Regulations for the operation of electrical equipment, electromagnetic compatibility and safety requirements. Improper use or handling could result in damage and/or injury.

Read these operating instructions thoroughly before using your filler, to prevent damage to the equipment. Keep these instructions in a safe place.

Follow the instructions below to ensure safe and trouble free operation of your filler:

- Do not use this filler in a hazardous area
- The instrument needs 230 V \pm 20%
- The power can be switched off completely by disconnecting the mains lead from the power supply.
- The filler housing is designed to protect against liquid splashes, but it is not liquid tight.
- When cleaning the filler make sure that no liquid enters the housing. Use a slightly moistened cloth to clean the outside of the filler.
- Before connecting or disconnecting any of the tubes which will contain liquid, disconnect the instrument from the mains supply.
- Connect only the H&D accessories that are designed to be used with the filler.
- Do not open the filler housing. If the seal is broken, you will lose all claims under the manufacturer's warranty.
- If you have any problems with your filler, contact the manufacturer. The address is given on the front page of this manual.

3 Equipment supplied

The system includes the following components:-

- Filler unit with display and control unit.
- Mains lead.
- 2m of 4 mm i.d. x 6 mm o.d. Viton tubing to join the two liquid bottles to the filler.
- 4 m of 2 mm i.d. x 4 mm o.d. Viton tubing to used as an outlet tube into the column.
- Blue polyurethane tubes to join Viton tubes to push-fit connectors.
- Outlet tube weighted end.
- Optional extra - column sweeper.

4 Preparing the column filler for first start up

4.1 Operating conditions

The filler is designed to provide reliable column filling under normal ambient conditions.

When choosing the place to set up the filler:-

- Place it on a solid even surface.
- Do not place the filler near to a heater or air conditioning unit.
- The filler should ideally be situated within 1metre of the column.
- There should be room for the bottles of light and heavy liquid, and the waste bottle, to be placed safely near the filler.

4.2 Choosing the liquids

To program the filler you will need to know:-

- The density that you want at the top of your column
- The density that you want at the bottom of your column
- The volume of the column in litres
- The volume of the layer (if any) of light liquid at the top of the column. This is used in some situations to reduce column changes due to evaporation
- The volume of the layer (if any) of heavy liquid at the bottom of the column. This is sometimes used where there is a particularly deep sweeping basket for collecting samples.
- The density of your heavy liquid. It must have a density the same as or greater than the density that you want at the bottom of your column.
- The density of your light liquid. It must have a density which is the same as or less than the density that you want at the top of your column.
The column filler can be used with almost any liquids, but you will need to know their densities to $\pm 0.001 \text{ g/ml}$ (1 kg/m^3).
- Make sure that these two liquids are completely miscible.

4.3 Connecting the tubing

- The filler is supplied with two sizes of tubing – 4 mm and 6 mm.
- Use the 6 mm tube for the inlet line from the two bottles of liquids to the filler, and the 4 mm tube for the outlet line from the filler to your column.
- Measure the distance from the two liquid bottles to the back of the column filler. The tubing needs to be long enough to sit on the bottom of the bottles.
- Cut two pieces of 6 mm tube to this length.
- Push one end of one of the 6 mm tubes into the quick-connector labelled <LIGHT> on the back of the filler.
- Push the other end down to the bottom of the bottle containing the light liquid. Clip it to the top of the bottle.
- Push one end of the other 6mm tube into the quick-connector labelled <HEAVY> on the back of the filler.
- Push the other end down to the bottom of the bottle containing the heavy liquid. Clip it to the top of the bottle.
- Measure the distance that the tubing will need to travel from the back of the filler to the top of the gradient column and then down inside to the bottom of the column.
- Cut a piece of the 4 mm tube to this length.
- Push one end of the 4 mm tube into the quick-connector labelled <OUTLET> on the back of the filler.
- Push the outlet tube weighted end onto the other end of this tube, and place into the waste bottle.

4.4 Switching the filler on

- Use only the original mains lead that is supplied with the instrument.
- Push the IEC plug into the socket on the back of the instrument.
- Insert the mains plug into the 230V AC supply.
- Switch on the mains power.
- Press the green switch on the front panel - it will light up.
- The screen will briefly show <initialisation>, and will then display the *main screen menu* as shown below.

MAIN SCREEN MENU

Density Gradient column filler firmware v2.01	COLUMN FILLING
	COLUMN CALIBRATION
	DENSITY MEASUREMENT
	SWEEPER

4.5 Operating the filler

The filler can carry out three different types of operation: filling, sweeping, and calibration. These are all started via the main menu.

You can access the different menus using the arrow keys on the left or right hand side of the display. You can go through the menus as many times as you like, and will always have the option to save a change or to cancel it.

Filling the column

Select <column filling> from the main menu, and the following screen appears.

COLUMN FILLING MENU

PARAMETERS		START FILLING
top	950.0kg/m ³	PARAMETERS
bottom	995.0kg/m ³	
light	780.2kg/m ³	
heavy	998.2kg/m ³	
on top	0.0ml	
on bot	0.0ml	MAIN SCREEN
column	1.235 l	

When the instrument is brand new, the parameters will be those programmed in by the manufacturer. If you have previously used the instrument to fill a column, the parameters will be those used for the last fill, and if you are happy with these, and you are ready to start filling, press <start filling>. Otherwise, select <Parameters>, to enter the details for the next column you want to fill.

The parameters menu takes three screens, the first two cover column characteristics, whilst the third covers filler characteristics.

PARAMETERS PAGE 1 MENU

DENSITY AT THE TOP 950.0 kg/m ³	START FILLING
DENSITY AT THE BOTTOM 995.0 kg/m ³	
LIGHT LIQUID DENSITY 780.2 kg/m ³	NEXT PAGE
HEAVY LIQUID DENSITY 998.2 kg/m ³	MAIN SCREEN

PARAMETERS PAGE 2 MENU

COLUMN VOLUME 1.235 l	START FILLING
LIGHT LIQUID ON TOP 0.0 ml	
HEAVY LIQUID ON BOTTOM 0.0 ml	NEXT PAGE
UNITS kg/m ³	MAIN SCREEN

PARAMETERS PAGE 3 MENU

VOLUME OF LIGHT PUMP 39.5 µl	START FILLING
VOLUME OF HEAVY PUMP 43.3 µl	
INLET TUBING VOLUME 1.2 ml	NEXT PAGE
OUTLET TUBING VOLUME 3.7 ml	MAIN SCREEN

To change any of these, press the arrow next to the item to select it, and type in the new number. If you make a mistake, use the arrows to choose either <cancel> or <backspace>, or press the arrow next to <OK> to accept.

The <Volume of light pump> and <Volume of heavy pump> are the volumes in microlitres delivered per stroke. These have been calibrated during manufacture, and should not normally need changing. A label showing the calibrated volumes is stuck on the rear face of the filler.

The next step is to enter the amount of liquid that will be needed to fill the two tubes from the filling bottles to the filler, and for the tube which delivers liquid from the filler to the base of the column.

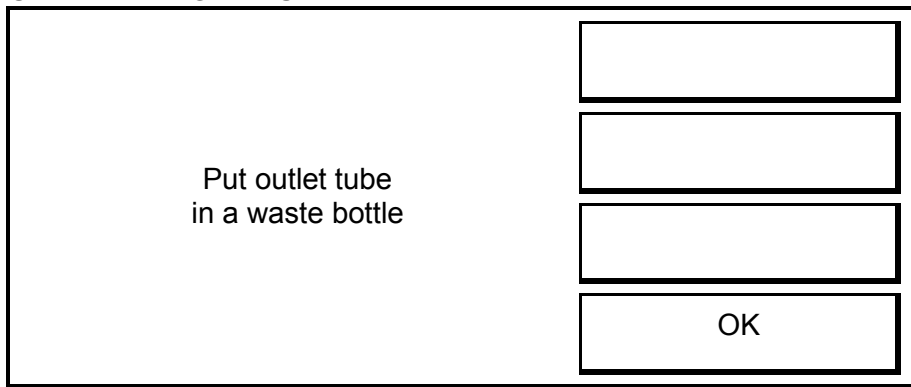
These values are used to ensure that the tubes are flushed with the liquids and are free of air bubbles.

4 mm tubing has a volume of 5 ml per metre of tube, and 6 mm tube volume is 13 ml per meter. These are minimum values, and unless you are short of liquids, we would suggest multiplying them by a factor of 3 to ensure complete flushing and de-airing.

Once all three screens are complete, select <start filling>

The filler software will check the data that has been entered for consistency. If there are no errors, the start filling menu will appear.

START FILLING MENU



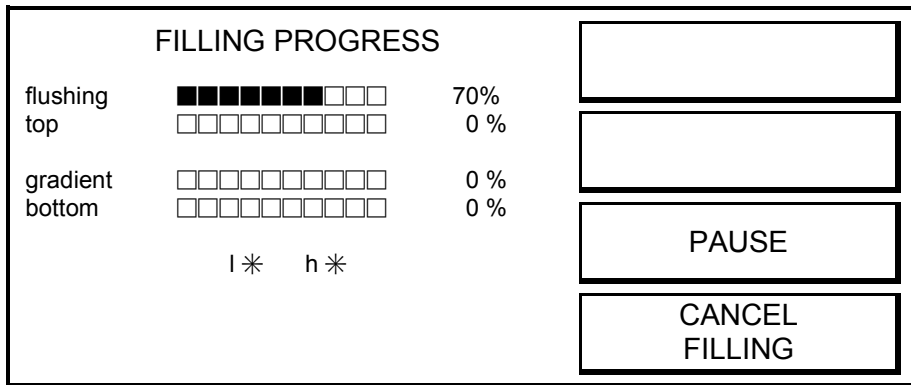
Put the outlet tube into a waste bottle, and press <OK>.

Progress menu 1 will appear, and the pumps will start to click.

The *flushing* bar graph will start turning black, and the percentage complete figure at the right hand end of the bar graph will increment.

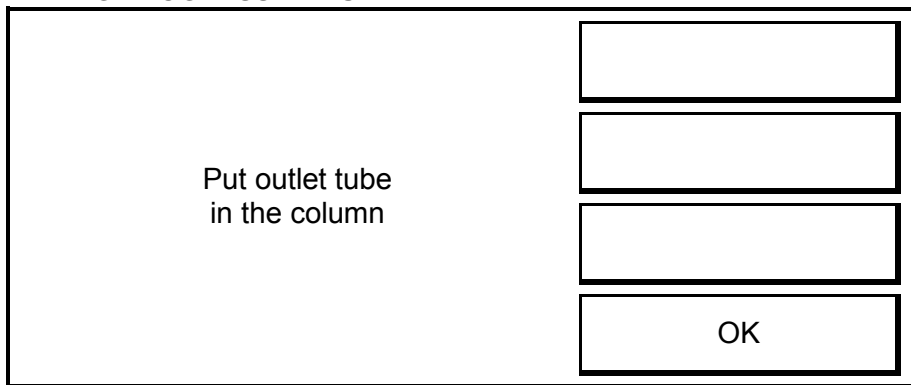
There are two small indicators on the screen, marked <l> and <h>. and these flash at each pump stroke.

FILLING PROGRESS MENU 1



Once flushing is complete, progress menu 2 appears on screen

FILLING PROGRESS MENU 2



Lower the outlet tube onto the base of the column, and press <OK>.

Column filling then starts, and the bar graph show the rate of progress.

FILLING PROGRESS MENU 3

FILLING PROGRESS		
flushing	■■■■■■■■■■■■■■■■	100%
top	■■■■■■■■■■■■■■■■	100 %
gradient	■■■■■□□□□□□□□	50 %
bottom	□□□□□□□□□□□□	0 %
l* h*		
[]		
[]		
[PAUSE]		
[CANCEL FILLING]		

You can choose to <pause> the filling at any stage, and if you do, the <pause> button will turn into a <continue> button to allow filling to proceed.

It is also possible to stop a filling by pressing the<cancel filling> button. Since this results in the loss of the liquids already pumped into the column, you are asked to confirm that you really do want to cancel before the system stops.

CANCEL FILLING MENU

Cancel filling – Are you sure?	[NO]
	[]
	[]
	[YES]

Once the column has been filled the screen will display <filling finished>. Press <OK> to return to the main menu.

5 Column calibration and density measurement

5.1 Column calibration

The column filler can hold a calibration table containing the heights and densities of up to ten floats. These are stored in a memory which does not lose its data when the device is switched off. If the height of a sample is then entered, the software uses linear interpolation to calculate its density.

Gently drop a set of calibrated floats into the filled column, and allow them to reach equilibrium.

Measure the height of each float above your column datum in mm.

Select <column calibration>, and the following screen appears.

COLUMN CALIBRATION MENU

	HEIGHT	DENSITY	
UP	165 mm	955.0 kg/m ³	SELECT
	> 272 mm	950.0 kg/m ³	
DOWN	379 mm	944.6 kg/m ³	NEW
	467 mm	939.9 kg/m ³	
	561 mm	935.0 kg/m ³	DELETE
	655 mm	929.9 kg/m ³	
	747 mm	924.8 kg/m ³	MAIN SCREEN
	840 mm	919.6 kg/m ³	
	926 mm	914.7 kg/m ³	
	1000 mm	910.5 kg/m ³	

If the float densities are correct, use the <up> and <down> keys to choose a float, and press <select>, otherwise select each float in turn and delete it.

SELECT MENU

Height : 165 mm	HEIGHT
Density : 955.0 kg/m ³	DENSITY
	OK
	MAIN SCREEN

Press the <height> key, and enter the current height of the float above the column datum. (The software does not allow a float to have a negative height)

HEIGHT MENU

FLOAT HEIGHT mm	BACKSPACE
—	
	CANCEL
	OK

If it is necessary to change the stored density of a float, press the <density> key, and enter the float density from its calibration certificate.

DENSITY MENU

FLOAT DENSITY	BACKSPACE
—	
	CANCEL
	OK

Once the calibration table has been entered into the filler, the density of samples can be determined by entering their heights above column datum. The software then uses linear interpolation to calculate the density of the sample.

5.2 Density measurement

Press <density measurement> on the *main menu*, and the *density measurement menu* appears.

DENSITY MEASUREMENT MENU

	HEIGHT
Height : 22 mm	
Density : 961.7 kg/m ³	
	MAIN SCREEN

Press the <height> key, and then enter sample's height above column datum. Once the <OK> is pressed, the display will change back to the *density measurement menu*, and the sample density will be displayed.

ENTER HEIGHT MENU

	BACKSPACE
—	
	CANCEL
	OK

6 Using the sweeper

The column filler has the capability to drive column sweeper. This is available as an optional extra. The sweeper is accessed by pressing the <sweeper> key on the *main menu*. The *sweeper menu* then appears.

Using this menu, it is possible to drive the sweeper up or down the column at any one of 8 speeds. Speed is selected using the <faster> and <slower> keys, and a small arrow will indicate which speed has been selected.

The distance to be travelled must then be entered on the distance menu, and the <go up> or <go down> key pressed to start the sweeper moving. It will automatically stop once it has travelled the programmed distance. If it is necessary to stop it before then, press the <stop> key.

SWEEPER MENU

DISTANCE	60.0 cm total 0.0 cm to go	GO UP
FASTER	speed 4395 mm/min	GO DOWN
SLOWER	675 mm/min	STOP
	450 mm/min	
	225 mm/min	
	> 123 mm/min	
	64 mm/min	
	26 mm/min	MAIN SCREEN
	13 mm/min	

DISTANCE MENU

DISTANCE, cm	BACKSPACE
—	
	CANCEL
	OK

7 Care & maintenance

Cleaning

To clean the filler

- Unplug the mains lead from the power supply.
- Do not use any aggressive cleaning agents.
- Carefully remove any liquid splashes with a cloth, and clean the filler using a piece of cloth which has been slightly moistened with either water, or water containing a small amount of detergent.
- After cleaning, wipe down the filler with a soft dry cloth.

To clean the tubing

- Disconnect the filler from the mains supply.
- Disconnect the tubing from the filler.
- Flush with a suitable solvent, and blow dry.

Repairs

With the exception of fitting new lengths of tubing, repair work must be carried out by trained service technicians.

Any attempts by untrained persons to perform repairs may damage the instrument.

The filler should not be used if:

- there is visible damage to the instrument or its mains lead
- it no longer functions properly

Any unauthorised removal / opening of the housing will invalidate the warranty.

8 Specification

Filling speed 2 litres in 3½ to 4 hours, depending on the selected density gradient.

Density gradient Any 2 miscible liquids covering the required density range can be used.

Selectable density units kg/m³ or g/ml

AC power supply 230 V

Frequency 50 – 60 Hz

Power consumption 5 W when filling, 15 W when driving sweeper.

Operating temperature normal ambient conditions

Dimensions 110 mm high, 300 mm wide, 270 mm deep (excluding mains lead)

Weight 2.3 kg

Filling example using isopropyl alcohol and water:

	kg/m ³	Top of column kg/m ³	Bottom of column kg/m ³
Light liquid (IPA) density	783		
Heavy liquid (H ₂ O) density	997		
Target density		905	975
Density achieved by filler		901	974

This was done using a shrinkage graph, to account for the very high shrinkage when water and IPA are mixed together.

A set of 13 calibrated floats was dropped into the column. Using a quadratic equation to fit the densities to the heights of the floats gave a standard error of estimate of 0.16 kg/m³. The uncertainty in the calibration of the floats was ± 0.10 kg/m³.

Filling examples using water and sodium bromide solutions:

Two columns were filled, using water and different concentrations of sodium bromide solution. The target densities were 1248.5 kg/m³ at the top, and 1282.5 kg/m³ at the bottom.

	kg/m ³	Top of column kg/m ³	Bottom of column kg/m ³
Water density	997.5		
Sodium bromide (1) density	1310.4		
Target density		1248.5	1282.5
Density achieved by filler		1248.2	1282.2

	kg/m ³	Top of column kg/m ³	Bottom of column kg/m ³
Water density	997.5		
Sodium bromide (2) density	1334.7		
Target density		1248.5	1282.5
Density achieved by filler		1249.7	1282.6

Using a quadratic equation to fit the densities to the heights of the floats gave a standard error of estimate of 0.13 kg/m^3 for one column, and 0.14 kg/m^3 for the second. The uncertainty in the calibration of the floats was $\pm 0.10 \text{ kg/m}^3$.