ACCENDO CORPORATION

SEGMENT INSIGHTS

Insight #5:
Pump Calibration—Conjure

January 26, 2011

PUMP CALIBRATION

BEFORE PROCEEDING WITH THIS CALIBRATION PROCEDURE, CONTACT ACCENDO CORPORATION. THEY WILL MODIFY THE EXISTING CALIBRATION FILES TO FIT THE FORMAT FOR THIS CALIBRATION PROCEDURE. THIS MODIFICATION IS ONLY REQUIRED FOR INITIAL FIELD CALIBRATION.

CALIBRATION OF FLOW RATES

This Segment Insight explains how to calibrate the Feed Module Pump by following steps:

- Access pump calibration data
- Program a series of segment with differing incubation rates based upon current (percent) values in the active calibration file;
- Load and run the calibration segments;
- Using the segment data file, calculate the actual flow rates;
- Modify the pump calibration file;
- Verify calibration.

IMPORTANT:

- Microsoft Office Access is required to read and modify the Conjure Calibration File
- Microsoft Office Excel is required to read and modify the Propel Calibration File

Note:

This Segment Insight covers both the Conjure™ and Propel™ Feed Module, but the pump calibration file types for each are different and therefore have slight differences in how they are accessed and modified. In addition, the calibration segment run will take several hours to complete and therefore appropriate system time should be scheduled.

STEP 1: ACCESSING PUMP CALIBRATION FILES

Conjure (Requires Microsoft Office Access)

Locate and open the Conjure Pump Calibration file

- Use the link provided to access the calibration file, C:\Program Files\Accendo\Conjure_FeedCalibration.mdb
- Depending upon the version of Microsoft Office Access, one or more security prompts will appear in order to open the database file.
- Once the Microsoft Office Access file is open, double-click to open the **tblCalibrateData** table (Figure 1), once opened the calibration data should appear (Figure 2). The actual PumpSpeed (%) and ActualRate (µL/minute) values maybe differ.

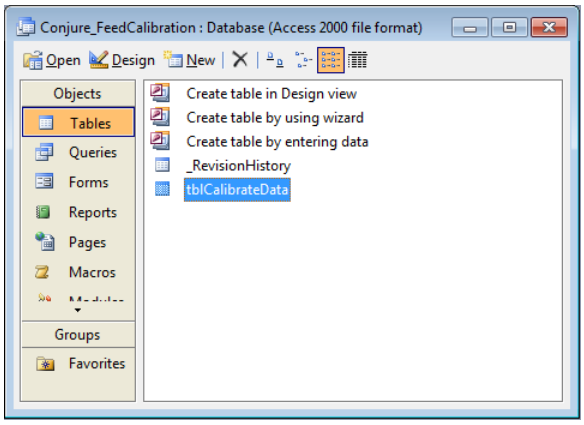

Figure 1

tblCalibrateData : Table

TimeStamp	SolventName	Pressure	PumpSpeed	ActualRate	Temperature
4/19/2007 9:25:00 AM	Solvent	0	0	0	25
4/19/2007 9:25:00 AM	Solvent	0	1	40	25
4/19/2007 9:25:00 AM	Solvent	0	2	70	25
4/19/2007 9:25:00 AM	Solvent	0	4	150	25
4/19/2007 9:25:00 AM	Solvent	0	7	220	25
4/19/2007 9:25:00 AM	Solvent	0	10	260	25
4/19/2007 9:25:00 AM	Solvent	0	15	490	25
4/19/2007 9:25:00 AM	Solvent	0	20	660	25
4/19/2007 9:25:00 AM	Solvent	0	30	950	25
4/19/2007 9:25:00 AM	Solvent	0	50	1700	25
4/19/2007 9:25:00 AM	Solvent	0	100	3425	25
		0	0	0	0

Figure 2

Propel (Requires Microsoft Office Excel)

Locate and open the Excel Pump Calibration file

- Use the link provided to access the calibration file, C:\Program Files\Accendo\Propel\PropelFeedCalibration.csv
- Depending upon the version of Microsoft Office Excel, one or more security prompts will appear in order to open the database file.
- The calibration table (Figure 3), has Pump Speed (%) Colum A and represents Actual Flow Rate (µL/minute) Column B, specific system values maybe different.

	A	B
1	100	3425
2	50	1700
3	30	950
4	20	660
5	10	260
6	7	220
7	4	150
8	2	70
9	1	40

Figure 3

Step 2: Program Calibration Segment Run

Conjure

- Use the link below to download and save the Conjure_Calibration_Files.zip folder on to the Conjure Desktop Computer (internet access will be required).

  http://www.accendocorporation.com/Pump_Calibration/Pump_Calibration_Files.zip
- Open the Pump_Calibration_File.zip folder and launch **Pump_Calibration_Worksheet.xls** (Figure 5)
- With both the **Conjure_FeedCalibration.mdb** (Figure 4) and **Pump_Calibration_Worksheet.xls** (Figure 5) files open; enter into the Pump_Calibration_Worksheet.xls; the Reactor Volume (Cell A2) and the ActualRates from the Conjure_FeedCalibration.mdb to Column C of the Pump_Calibration_Worksheet.xls;
- Use the calculated Programmed Incubation Rates (Column D) from the Pump_Calibration_Worksheet.xls for Step 3.

tblCalibrateData : Table

TimeStamp	SolventName	Pressure	PumpSpeed	ActualRate	Temperature
4/19/2007 9:25:00 AM	Solvent	0	0	0	25
4/19/2007 9:25:00 AM	Solvent	0	1	40	25
4/19/2007 9:25:00 AM	Solvent	0	2	70	25
4/19/2007 9:25:00 AM	Solvent	0	4	150	25
4/19/2007 9:25:00 AM	Solvent	0	7	220	25
4/19/2007 9:25:00 AM	Solvent	0	10	260	25
4/19/2007 9:25:00 AM	Solvent	0	15	490	25
4/19/2007 9:25:00 AM	Solvent	0	20	660	25
4/19/2007 9:25:00 AM	Solvent	0	30	950	25
4/19/2007 9:25:00 AM	Solvent	0	50	1700	25
4/19/2007 9:25:00 AM	Solvent	0	100	3425	25
		0	0	0	0

Figure 4

	A	B	C	D	E	F
1	Reactor Volume (µL)	Pump Speed (%)	Calibration Speed (µL/minute)	Programmed Incubation Rate (Minutes)	Actual Incubation Rate (Minutes)	Actual Pump Speed (µL/Minute)
2	2100	100	3425.00	Do not use 100% flow rate		
3	Enter actual reactor volume in the cell above	50	1700.00	1.24	1.00	2100.00
4		30	950.00	2.21	2.00	1050.00
5		20	660.00	3.18	3.00	700.00
6		10	260.00	8.08	8.00	262.50
7		7	220.00	9.55	9.00	233.33
8		4	150.00	14.00	14.50	144.83
9		2	70.00	30.00	31.00	67.74
10		1	40.00	52.50	50.00	42.00
11		Enter calibration file values		Do not modify Incubation Rate Cells	Enter values of actual incubation rate from Segment File	Do not modify Incubation Rate Cells

Figure 5

Propel

- Use the link below to download and save the Conjure_Calibration_Files.zip folder on to the Conjure Desktop Computer (internet access will be required).

  http://www.accendocorporation.com/Pump_Calibration/Pump_Calibration_Files.zip
- Open the Pump_Calibration_File.zip folder and launch **Pump_Calibration_Worksheet.xls** (Figure 5)
- With both the **PropelFeedCalibration.csv** (Figure 6) and **Pump_Calibration_Worksheet.xls** (Figure 7) files open; enter into the Pump_Calibration_Worksheet.xls; the Reactor Volume (Cell A2) and the Actual Rates from the PropelFeedCalibration.csv to Column B of the Pump_Calibration_Worksheet.xls;
- Use the calculated Programmed Incubation Rates (Column D) from the Pump_Calibration_Worksheet.xls for Step 3.

	A	B
1	100	3425
2	50	1700
3	30	950
4	20	660
5	10	260
6	7	220
7	4	150
8	2	70
9	1	40

Figure 6

	A	B	C	D	E	F
1	Reactor Volume (µL)	Pump Speed (%)	Calibration Speed (µL/minute)	Programmed Incubation Rate (Minutes)	Actual Incubation Rate (Minutes)	Actual Pump Speed (µL/Minute)
2	2100	100	3425.00	Do not use 100% flow rate		
3	Enter actual reactor volume in the cell above	50	1700.00	1.24	1.00	2100.00
4		30	950.00	2.21	2.00	1050.00
5		20	660.00	3.18	3.00	700.00
6		10	260.00	8.08	8.00	262.50
7		7	220.00	9.55	9.00	233.33
8		4	150.00	14.00	14.50	144.83
9		2	70.00	30.00	31.00	67.74
10		1	40.00	52.50	50.00	42.00
11		Enter calibration file values		Do not modify Incubation Rate Cells	Enter values of actual incubation rate from Segment File	Do not modify Incubation Rate Cells

Figure 7

STEP 3: PROGRAM CALIBRATION RUNS

Conjure

- From within the Conjure Wizard, open the **Pump_Calib_Step_1.cnj** located in the downloaded the Pump_Calibration_File.zip folder;
- When the Conjure File Options dialog box opens, select "Jump to Results Screen" and click the **Okay** button (Figure 8);
- In the Wizard Results Screen, manually enter the Residence Time (min.) for each of the Programmed Incubations from the Pump_Calibration_Worksheet.xls determined in Step 2. Create one segment for each (Figure 9) a total of 9 segments will be programmed;
- Save the new Wizard File.

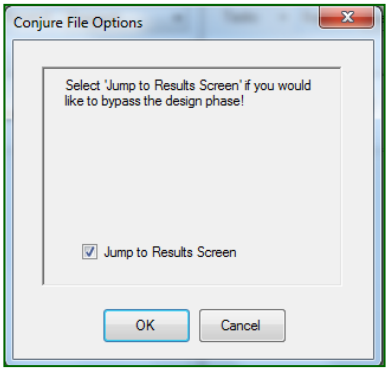

Figure 8

Reactor Temp [°C]	Resid. Time [min]
25.0	1.18
25.0	1.81
25.0	2.87
25.0	3.86
25.0	5.58
25.0	7.73
25.0	12.62
25.0	21.50
25.0	32.33

Figure 9

Propel

- Launch Propel User Interface and log in under the Screening Mode.
  - From the Propel User Interface, programmed one segment for each of the Programmed Incubation rate values (Figure 10) from the Pump_Calibration_Worksheet.xls.
  - Save the new Propel file.

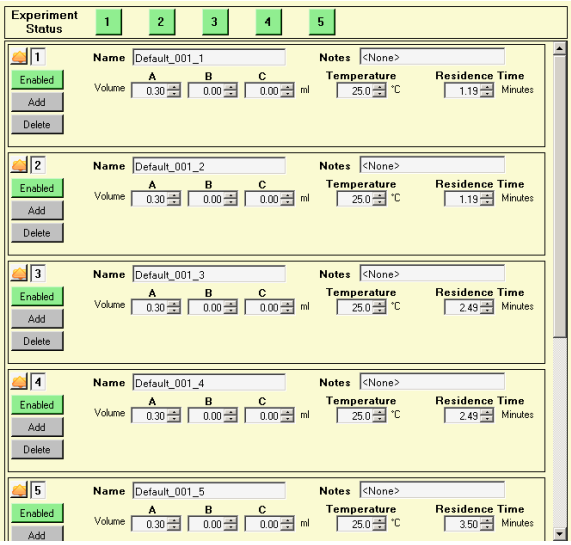

Figure 10

Step 4: Run the calibration

Conjure and Propel

- Load Ethanol/Methanol as the Transport Solvent;
- Start the pump to prime the reactor with Transport Solvent;
- If required, perform a Fluid Prime to prime the syringes;
- Verify that the system pressure;
- Load Toluene into Bottle A;
- Prime Bottle A;
- Reset the UV signal detection values based upon Ethanol and Toluene
- Load the recipes and start the calibration.

Step 5: Analyze and adjust the calibration file

Conjure

- Open the Conjure_FeedCalibration.mdb file as per Step 1;
- Open the Pump_Calibration_Worksheet.xls as per Step 2;
- Open the Segment Data File, which was created after the end of the Conjure calibration file. The file will be located in C:Conjure\Data the file will have a file name starting with SegmentData, followed by the date (YYYYMMDD) and have a file extension of .csv, for example: SegmentData_20110107_145230.csv;
- In the SegmentData file, under Column H, the actual incubation rate will be listed for each segment (Figure 11).

	A	B	C	D	E	F	G	H
1	Segment Data			Analytical Data				2nd Stage
2	Segment Name	Exp #	DOEstd #	DItn Ratic	Resp 1	Resp 2	Resp 3	2nd Rxtr min
3	pump calibration_A1_1101111517_001	1	0	-1	Missing	Missing	Missing	25.81415
4	pump calibration_A1_1101111517_002	2	0	-1	Missing	Missing	Missing	25.81601
5	pump calibration_A1_1101111517_003	3	0	-1	Missing	Missing	Missing	14.82954
6	pump calibration_A1_1101111517_004	4	0	-1	Missing	Missing	Missing	14.68895
7	pump calibration_A1_1101111517_005	5	0	-1	Missing	Missing	Missing	9.119936
8	pump calibration_A1_1101111517_006	6	0	-1	Missing	Missing	Missing	9.065058
9	pump calibration_A1_1101111517_007	7	0	-1	Missing	Missing	Missing	6.7758
10	pump calibration_A1_1101111517_008	8	0	-1	Missing	Missing	Missing	6.771347
11	pump calibration_A1_1101111517_009	9	0	-1	Missing	Missing	Missing	5.355584
12	pump calibration_A1_1101111517_010	10	0	-1	Missing	Missing	Missing	5.419808
13	pump calibration_A1_1101111517_011	11	0	-1	Missing	Missing	Missing	3.555832
14	pump calibration_A1_1101111517_012	12	0	-1	Missing	Missing	Missing	3.545458
15	pump calibration_A1_1101111517_013	13	0	-1	Missing	Missing	Missing	1.78837
16	pump calibration_A1_1101111517_014	14	0	-1	Missing	Missing	Missing	1.797725

Figure 11

- Enter the actual incubation rate into the Pump_Calibration_Worksheet.xls (Column E) to calculate the actual flow rate (µL/Minute) as shown in Figure 12.
- The Pump_Calibration_Worksheet.xls calculates the Actual Pump Speeds (Column F) for each of the incubation rates;

	A	B	C	D	E	F
1	Reactor Volume (µL)	Pump Speed (%)	Calibration Speed (µL/minute)	Programmed Incubation Rate (Minutes)	Actual Incubation Rate (Minutes)	Actual Pump Speed (µL/Minute)
2	2100	100	3425.00	Do not use 100% flow rate		
3	Enter actual reactor volume in the cell above	50	1700.00	1.24	1.00	2100.00
4		30	950.00	2.21	2.00	1050.00
5		20	660.00	3.18	3.00	700.00
6		10	260.00	8.08	8.00	262.50
7		7	220.00	9.55	9.00	233.33
8		4	150.00	14.00	14.50	144.83
9		2	70.00	30.00	31.00	67.74
10		1	40.00	52.50	50.00	42.00
11		Enter calibration file values		Do not modify Incubation Rate Cells	Enter values of actual incubation rate from Segment File	Do not modify Incubation Rate Cells

Figure 12

- In the Conjure_FeedCalibration.mdb, enter the ActualRates values that correspond to the Pump Speed (%) (Figure 13).

tblCalibrateData : Table

TimeStamp	SolventName	Pressure	PumpSpeed	ActualRate	Temperature
4/19/2007 9:25:00 AM	Solvent	0	0	0	25
4/19/2007 9:25:00 AM	Solvent	0	1	40	25
4/19/2007 9:25:00 AM	Solvent	0	2	70	25
4/19/2007 9:25:00 AM	Solvent	0	4	150	25
4/19/2007 9:25:00 AM	Solvent	0	7	220	25
4/19/2007 9:25:00 AM	Solvent	0	10	260	25
4/19/2007 9:25:00 AM	Solvent	0	15	490	25
4/19/2007 9:25:00 AM	Solvent	0	20	660	25
4/19/2007 9:25:00 AM	Solvent	0	30	950	25
4/19/2007 9:25:00 AM	Solvent	0	50	1700	25
4/19/2007 9:25:00 AM	Solvent	0	100	3425	25
		0	0	0	0

Figure 13

Propel

- Open the PropelFeedCalibration.csv file as per Step 1;
- Open the Pump_Calibration_Worksheet.xls as per Step 2;
- Open the Segment Data File, which was created after the end of the Propel calibration file. The file will be located in C:Accendo\Propel*Username*\Data the file will have a name such as Data_########_######.csv, for example: Data_20110211_132206.csv;
- In the Segment Data file, under Column K, the actual incubation rate will be listed for each segment (Figure 14).
- Enter the actual incubation rate into the Pump_Calibration_Worksheet.xls (Column E) to calculate the actual flow rate (µL/Minute) as shown in Figure 15.
- The Pump_Calibration_Worksheet.xls calculates the Actual Pump Speeds (Column F) for each of the incubation rates;

K
System Dat
Rxtr Time
2.55
2.9
4.67
8.97
8.48
12.7
12.62

Figure 14

	A	B	C	D	E	F
1	Reactor Volume (µL)	Pump Speed (%)	Calibration Speed (µL/minute)	Programmed Incubation Rate (Minutes)	Actual Incubation Rate (Minutes)	Actual Pump Speed (µL/Minute)
2	2100	100	3425.00	Do not use 100% flow rate		
3	Enter actual reactor volume in the cell above	50	1700.00	1.24	1.00	2100.00
4		30	950.00	2.21	2.00	1050.00
5		20	660.00	3.18	3.00	700.00
6		10	260.00	8.08	8.00	262.50
7		7	220.00	9.55	9.00	233.33
8		4	150.00	14.00	14.50	144.83
9		2	70.00	30.00	31.00	67.74
10		1	40.00	52.50	50.00	42.00
11		Enter calibration file values		Do not modify Incubation Rate Cells	Enter values of actual incubation rate from Segment File	Do not modify Incubation Rate Cells

Figure 15

- In the PropelFeedCalibration.csv, enter the Actual Rates flow rate value for each Pump Speed (%) (Figure 16).

	A	B
1	100	3425
2	50	1700
3	30	950
4	20	660
5	10	260
6	7	220
7	4	150
8	2	70
9	1	40

Figure 16

- Close all the files and if desired run a verification run of a single segment for each Pump Speed (%) based upon the Incubation Rate.

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