Calibration of Residual Solvents (Ethanol)
7-8-15

In this document the formula for calibration of ethanol as a residual solvent will be explained. First with pure Ethanol, then with a smaller concentration. This formula can be used with most other residual solvents as well.

1µL of 100% pure (200 proof) Ethanol will be put into a 40 mL vial.

Ethanol has a density of 789.00 kg/m^3

M^3 = 1000L

789kg/1000L; divided by 1000 = 789g/1000mL

789g/1000mL; divided by 1000 = 789mg/1000µL

789mg/1000µL; divided by 1000 = 789µg/µL

789µg/µL = 789,000ng/µL

The amount being put into the vial, when using 1µL of pure Ethanol, is 789,000ng. The vial has a volume of 40mL; so the amount in each mL, after the Ethanol has had time to equilibrate into the headspace of the vial, is 19725ng/mL.
(789,000 ÷ 40 = 19725)

This vial will be the calibration standard for Ethanol. When calibrating, 1mL will be pulled from the headspace of the vial. As per the calculations, 1mL of the headspace will have 19725ng of Ethanol. This number (19725) will be put into the “Injected” box of the calibration screen.

Most often the results are requested to be in parts per million (ppm). To convert the amount injected into ppm, the standard and sample weight, in the “Integration” screen, needs to be changed. To find out what the ppm of the amount injected, and what the Standard weight should be, the following formulae can be used.

The sample weight will be 100 (representing the 100mg put into the vial for actual samples).

$$\frac{\text{Total weight of Ethanol in the vial}}{\text{(Sample Weight)}} \rightarrow \frac{789{,}000\text{ng}}{100\text{mg (convert into ng; 100,000,000ng)}}$$

$$\frac{789{,}000\text{ng}}{100{,}000{,}000\text{ng}} = \frac{7890\text{ng}}{1{,}000{,}000\text{ng}} = 7890\text{ppm}$$

To find out what the standard weight needs to be, use this formula.

$$\text{Standard Weight} = \frac{\text{ppm x Sample Weight}}{\text{amount of Ethanol in each mL (19725)}}$$

$$\text{Standard Weight} = \frac{7890 \times 100}{19725} \quad \frac{789{,}000}{19725} = 40 \qquad \text{Standard weight} = 40$$

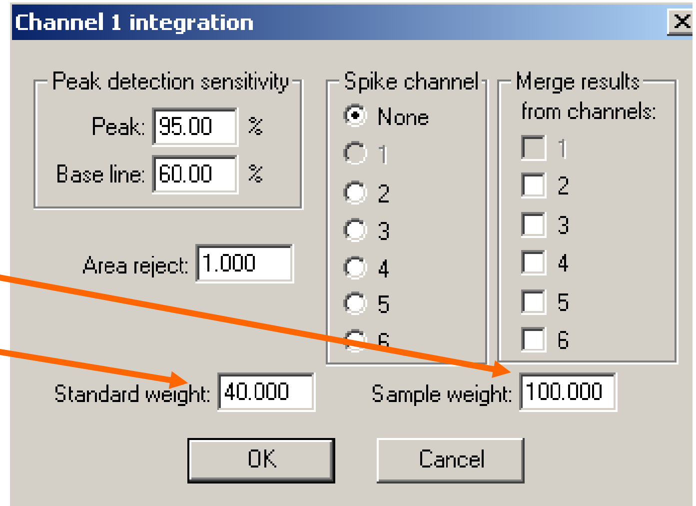
These results will be what is entered into the integration/calibration screens in Peaksimple. This is shown on the next page.

Calibration of Residual Solvents (Ethanol)

Standard Weight = 40

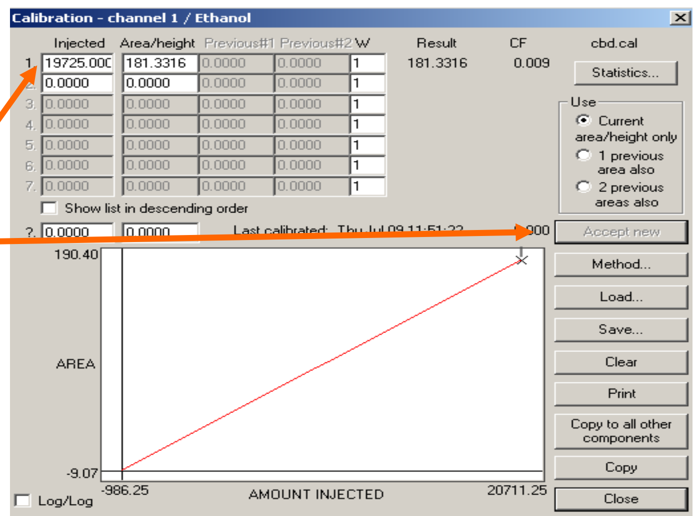
Sample weight = 100

Navigate into the integration screen for the channel being used for residual solvents, then enter the Standard weight (40) and Sample weight (100).

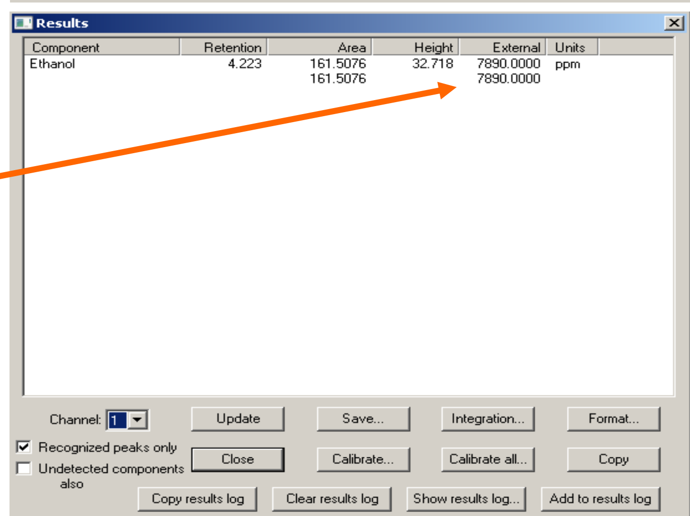


Amount of Ethanol in each mL (19725ng)

Navigate to the calibration screen for ethanol. Then enter the amount of Ethanol in each mL, and hit accept new.



To check that everything was done correctly, open the results screen. The ppm shown should be the same as the ppm reached through the formulae (7890 ppm)



On the next page, another example of the formulae will be shown using a different concentration of Ethanol.

Calibration of Residual Solvents (Ethanol)

Amount of Ethanol in standard = 2000ppm

1μL of 2000ppm Ethanol will be put into a 40 mL vial

2000ppm of Ethanol in 1μL = $\frac{789{,}000\text{ng}}{500}$ = 1578ng

The total amount of Ethanol in the 40 mL vial is 1578ng

The amount of Ethanol in each mL = $\frac{1578}{40}$ = 39.45

Total Ethanol in vial = 1578ng

Amount of Ethanol in each mL = 39.45ng

Sample weight = 100 (because of the 100mg of concentrate being put into the actual sample vial)

ppm = $\frac{\text{Total weight of Ethanol in the vial}}{\text{(Sample Weight)}}$ → $\frac{1578\text{ng}}{\text{100mg (convert into ng; 100,000,000ng)}}$

$\frac{1578\text{ng}}{100{,}000{,}000\text{ng}} = \frac{15.78\text{ng}}{1{,}000{,}000\text{ng}}$ = 15.78ppm

Standard weight = $\frac{\text{ppm x Sample Weight}}{\text{amount of Ethanol in each mL}}$ → $\frac{15.78 \text{ x } 100}{39.45}$ = 40

Standard weight = 40

For a standard with an Ethanol concentration of 2000ppm: the Standard weight is 40, the Sample weight is 100, and the amount of Ethanol in each mL is 15.78ng.

In the Integration screen, put 40 into the Standard weight, 100 into the Sample weight; in the calibration screen put 15.78 into the Injected column and click on Accept New.

After entering those parameters, the results screen should read 15.78ppm for Ethanol.

Calibration of Residual Solvents (Ethanol) Butane (revision 3-28-16)

The same method can be done for other residual solvents, although, for gases using molar mass is easier than density. In this next example, butane will be used.

1mL of butane will be injected into a 40mL vial

1 mol of butane = 58.12g/mol

A mol of gas, at room temperature (~20c) and not under pressure, takes up ~24,000mL of space.

$$\frac{58.12\text{g}}{24{,}000\text{mL}} \rightarrow \frac{58{,}120\text{mg}}{24{,}000\text{mL}} \rightarrow \frac{2.42\text{mg}}{\text{mL}}$$

In the 1mL injected into the 40mL vial, there is 2.42mg of butane

$$\frac{2.42\text{mg}}{40\text{mL}} \rightarrow \frac{2{,}420\text{ug}}{40\text{mL}} \rightarrow \frac{60.5\text{ug}}{\text{mL}} \rightarrow \frac{60{,}500\text{ng}}{\text{mL}}$$

When 1mL is pulled from the 40mL vial and injected into the GC, 60,500ng of actual butane will be put into the system. This 60,500 is the number that will be put into the “amount injected” cell of the peaksimple calibration. See page 2 of this document for the calibration screen.

To convert the answer into ppm, do the following. The sample weight for this will be 100mg. The sample weight can be edited in the integration screen (shown on page 2 of this document), and peaksimple will automatically adjust the result.

$$\frac{\text{Total weight of butane in the vial}}{\text{(Sample Weight)}} \rightarrow \frac{2{,}420\text{ug}}{100\text{mg}} \rightarrow \frac{2{,}420{,}000\text{ng}}{100{,}000{,}000\text{ng}} \rightarrow \frac{24{,}200\text{ng}}{1{,}000{,}000\text{ng}} \rightarrow 24{,}200\text{ppm}$$

With a 100mg sample, in a 40mL vial, 1mol of butane is equal to 24,200ppm

To find the appropriate standard weight to enter into the integration screen (see page 2 of document), do the following.

$$\frac{\text{ppm x Sample Weight}}{\text{amount of butane in each mL (60,500)}} \rightarrow \frac{24{,}200 \times 100}{60{,}500} \rightarrow \frac{2{,}420{,}000}{60{,}500} \rightarrow 40$$

The standard weight is 40

The majority of butane standards are not 100% pure butane. On the next page, this same process will be done for a more realistic standard at 1000ppm butane.

Calibration of Residual Solvents (Ethanol) Butane (revision 3-28-16)

For this example a butane standard, with a concentration of 1000ppm, will be used.

1mL of 1000ppm butane will be injected into a 40mL syringe

1 mol of butane = 58.12g/mol

A mol of gas, at room temperature (~20c) and not under pressure, takes up ~24,000mL of space.

$$\frac{58.12g}{24{,}000mL} \rightarrow \frac{58{,}120mg}{24{,}000mL} \rightarrow \frac{2.42mg}{mL}$$

This standard is not 100% pure, but is 1000ppm, so $\frac{2.42mg}{mL} \rightarrow \frac{2{,}420ug}{mL} \times 0.001 \rightarrow \frac{2.42ug}{mL}$

Injecting 1mL, at a concentration of 1000ppm, will put 2.42ug of butane into the 40mL vial.

Now find the amount in each mL of the vial

$$\frac{2.42ug}{40mL} \rightarrow \frac{2{,}420ng}{40mL} \rightarrow \frac{60.5ng}{mL}$$

When 1mL is pulled from the 40mL vial and injected into the GC, 60.5ng of actual butane will be put into the system. This 60.5 is the number that will be put into the "amount injected" cell of the peaksimple calibration. See page 2 of this document for the calibration screen.

$$\frac{\text{Total weight of butane in the vial}}{\text{(Sample Weight)}} \rightarrow \frac{2.42ug}{100mg} \rightarrow \frac{2{,}420ng}{100{,}000{,}000ng} \rightarrow \frac{2{,}420ng}{1{,}000{,}000ng} \rightarrow 24.2ppm$$

With a 100mg sample, a 1000ppm butane standard is equal to 24.2ppm

Now do the same formula from page 4 to find the standard weight

$$\frac{\text{ppm x Sample Weight}}{\text{amount of butane in each mL (60.5)}} \rightarrow \frac{2{,}42 \times 100}{60.5} \rightarrow \frac{2{,}420}{60.5} \rightarrow 40$$

The standard weight entered into the integration screen (see page 2 of this document), is 40.