
# DOSY automatic setup

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DOSY Discovery Day 2022

# Self-Diffusion and molecular weight (SEGWE)

- Approximation for the correlation between molecular weight and self-diffusion coefficient
- More advanced than previous estimations such as $r_H \propto \sqrt[3]{MW}$
- Takes into account the molecular interactions of solvent and solutes
- **Calculator available, input: temperature, solvent and MW or D**

$$D = \frac{k_B T \left( \frac{3\sqrt[3]{\frac{MW_S}{MW}}}{2} + \frac{1}{1+\sqrt[3]{\frac{MW_S}{MW}}} \right)}{6\pi\eta\sqrt[3]{\frac{3MW}{4\pi\rho_{eff}N_A}}}$$

$k_B$ : Boltzmann constant

$T$: temperature

$\eta$: viscosity

$\rho_{eff}$: effective density of the molecule (packing effects, geometry, solvation and flexibility)

$MW$ : molecular weight of the molecule

$MW_S$: molecular weight of the solvent

$N_A$: Avogadro number

**R. Evans et al, *Angew. Chem. Int. Ed.* (2013), 52: 3199–3202**
**R. Evans et al, *Anal. Chem.* (2018), *90* (6), 3987–3994**
**R. Evans, *Prog Nucl Magn Reson Spectrosc* (2020), 117, 33-69**
**https://www.nmr.chemistry.manchester.ac.uk/?q=node/432**

# Calculating gradient length (Stejskal-Tanner equation)

$$S = S_0 \, e^{-D\gamma^2\delta^2G^2\Delta'}$$

$$\delta = \sqrt{-\frac{ln\,{}^{S}/_{S_0}}{D\gamma^2G^2\Delta'}}$$

$S$ : signal amplitude [0.1 $S_0$]

$S_0$: signal amplitude without diffusion [$S_0$]

$D$ : diffusion coefficient [SEGWE]

$\gamma$ : gyromagnetic ratio (dependent on *x_domain*) [$\gamma_H$]

$\delta$ : gradient pulse width (***delta*** *or* ***smallDelta***)

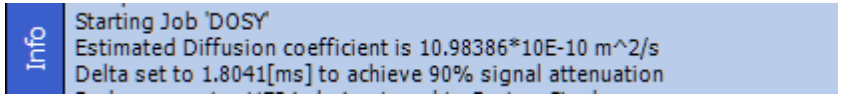
$G$ : gradient amplitude (***g***) [maximum usable gradient strength]

$\Delta'$ : corrected diffusion time

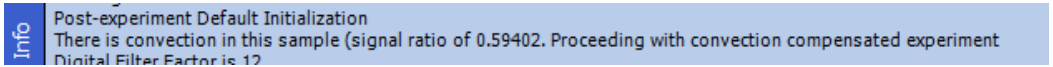
$\Delta$ : diffusion time (***diffusion_time***) [0.1 s]

# DOSY steps (automatic_setup)

1. Automatic setting of chosen sample parameters (temperature, temperature delay, shimming, tuning*,…)
2. Automatic estimation of diffusion coefficient using SEGWE with the **solute MW**
3. Automatic determination of gradient length to achieve 90% signal attenuation (0.1 s diffusion time and maximum usable gradient strength)




4. Automatic determination of 90 degrees pulse and setup (**optional**)
5. Automatic determination of sample convection (**optional**), by evaluating if a delay imbalance of 30ms leads to a signal loss of more than 20%.





6. Automatic acquisition of DOSY with/out convection compensation
7. Automatic DOSY plot automatically generation

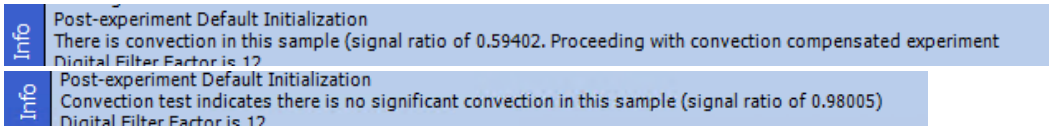



User input in bold

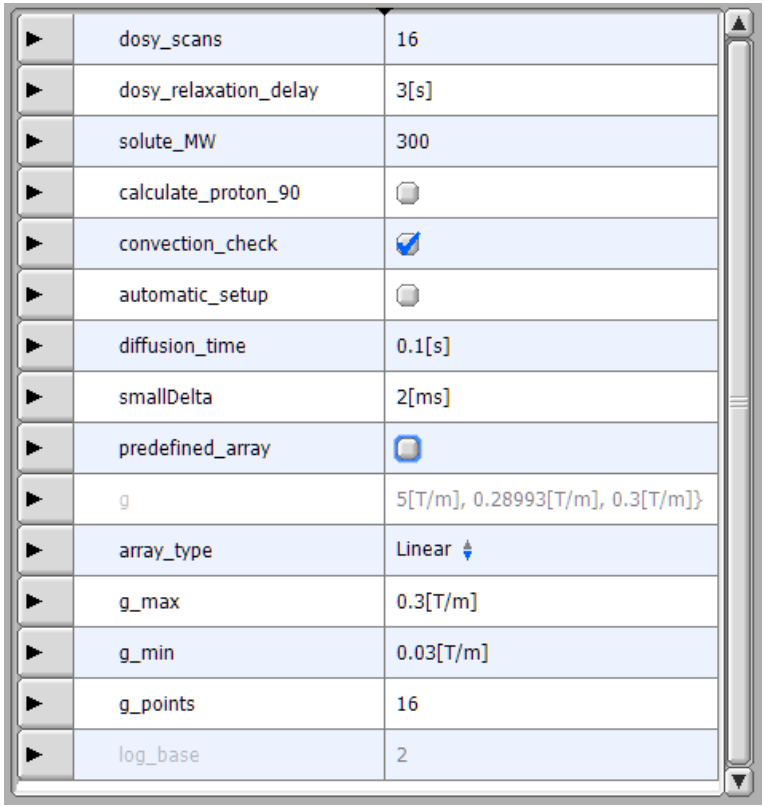
# DOSY steps (semi-automatic setup)

1. Automatic setting of chosen sample parameters (temperature, temperature delay, shimming, tuning*,…)
2. Choose **diffusion time** and **gradient length**
3. If predefined_array, type in/copy-paste **gradient strengths**

   If not predefined, setup array as linear / exponential / logarithmic with the weakest PFG strength, strongest PFG strength, (logarithm base if logarithmic array) and number of PFG values
3. Automatic determination of 90 degrees pulse and setup (**optional**)
4. Automatic determination of sample convection (**optional**), by evaluating if a delay imbalance of 30ms leads to a signal loss of more than 20%.



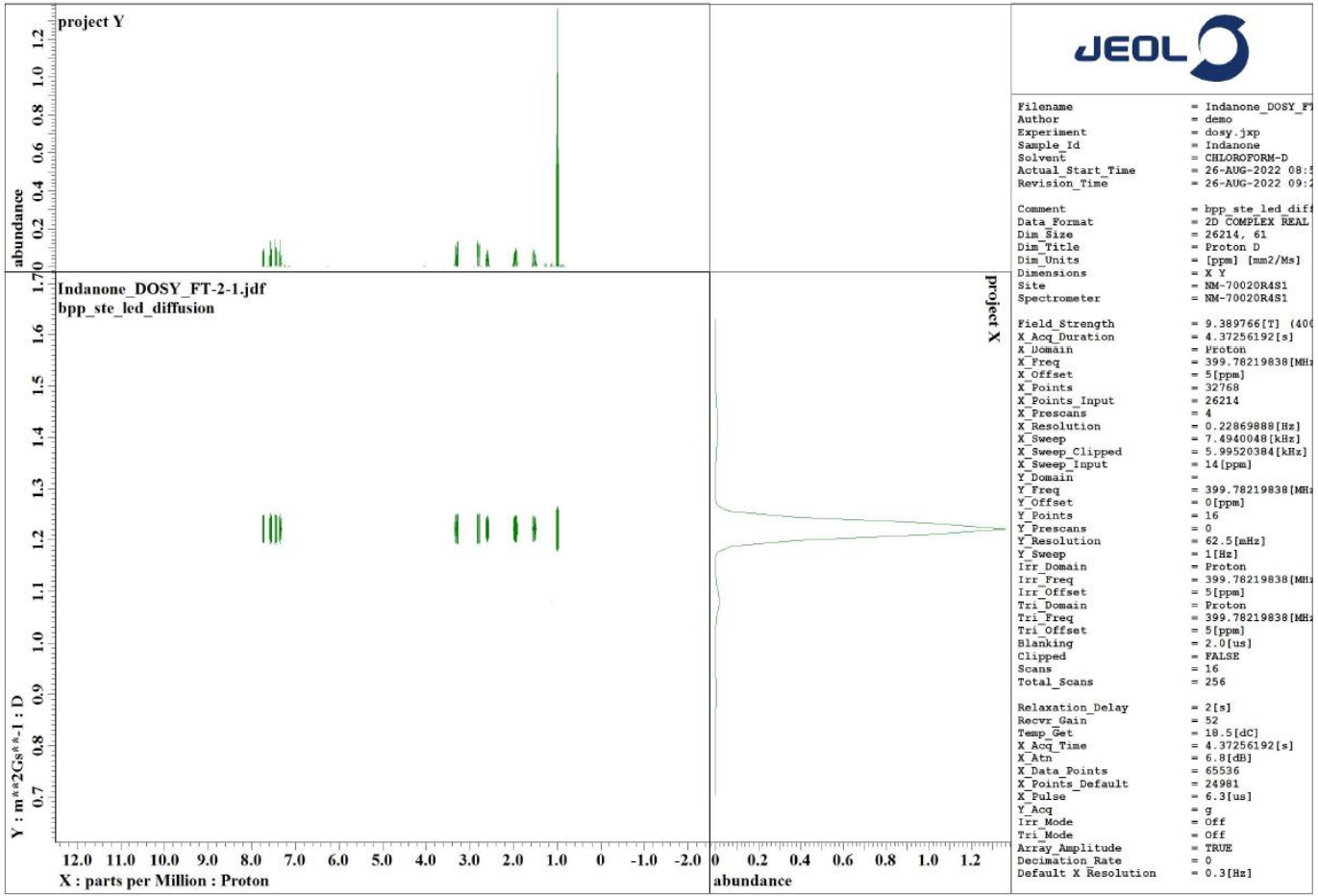

5. Automatic acquisition of DOSY with/out convection compensation
6. Automatic DOSY plot automatically generation






User input in bold

# Results from automatic_setup (Ethyl-indanone)




JEOL

| | |
|---|---|
| Filename | = Indanone_DOSY_FT |
| Author | = demo |
| Experiment | = dosy.jxp |
| Sample_Id | = Indanone |
| Solvent | = CHLOROFORM-D |
| Actual_Start_Time | = 26-AUG-2022 08:5 |
| Revision_Time | = 26-AUG-2022 09:2 |
| Comment | = bpp_ste_led_diff |
| Data_Format | = 2D COMPLEX REAL |
| Dim_Size | = 26214, 61 |
| Dim_Title | = Proton D |
| Dim_Units | = [ppm] [mm2/Ms] |
| Dimensions | = X Y |
| Site | = NM-70020R4S1 |
| Spectrometer | = NM-70020R4S1 |
| Field_Strength | = 9.389766[T] (40 |
| X_Acq_Duration | = 4.37256192[s] |
| X_Domain | = Proton |
| X_Freq | = 399.78219838[MHz |
| X_Offset | = 5[ppm] |
| X_Points | = 32768 |
| X_Points_Input | = 26214 |
| X_Prescans | = 4 |
| X_Resolution | = 0.22869888[Hz] |
| X_Sweep | = 7.4940048[kHz] |
| X_Sweep_Clipped | = 5.99520384[kHz] |
| X_Sweep_Input | = 14[ppm] |
| Y_Domain | = |
| Y_Freq | = 399.78219838[MHz |
| Y_Offset | = 0[ppm] |
| Y_Points | = 16 |
| Y_Prescans | = 0 |
| Y_Resolution | = 62.5[mHz] |
| Y_Sweep | = 1[Hz] |
| Irr_Domain | = Proton |
| Irr_Freq | = 399.78219838[MHz |
| Irr_Offset | = 5[ppm] |
| Tri_Domain | = Proton |
| Tri_Freq | = 399.78219838[MHz |
| Tri_Offset | = 5[ppm] |
| Blanking | = 2.0[us] |
| Clipped | = FALSE |
| Scans | = 16 |
| Total_Scans | = 256 |
| Relaxation_Delay | = 2[s] |
| Recvr_Gain | = 52 |
| Temp_Get | = 18.5[dC] |
| X_Acq_Time | = 4.37256192[s] |
| X_Atn | = 6.8[dB] |
| X_Data_Points | = 65536 |
| X_Points_Default | = 24981 |
| X_Pulse | = 6.3[us] |
| Y_Acq | = g |
| Irr_Mode | = Off |
| Tri_Mode | = Off |
| Array_Amplitude | = TRUE |
| Decimation_Rate | = 0 |
| Default X Resolution | = 0.3[Hz] |

PDF automatically generated after clicking the Submit Job button with the DOSY automation method

Note PDF printout is generated using multivariate analysis, results can be misleading

**Further analysis with other methods is recommended**, particularly for more complex samples

# Results from automatic_setup (Quinine + Geraniol + Camphene)

MW set to 324

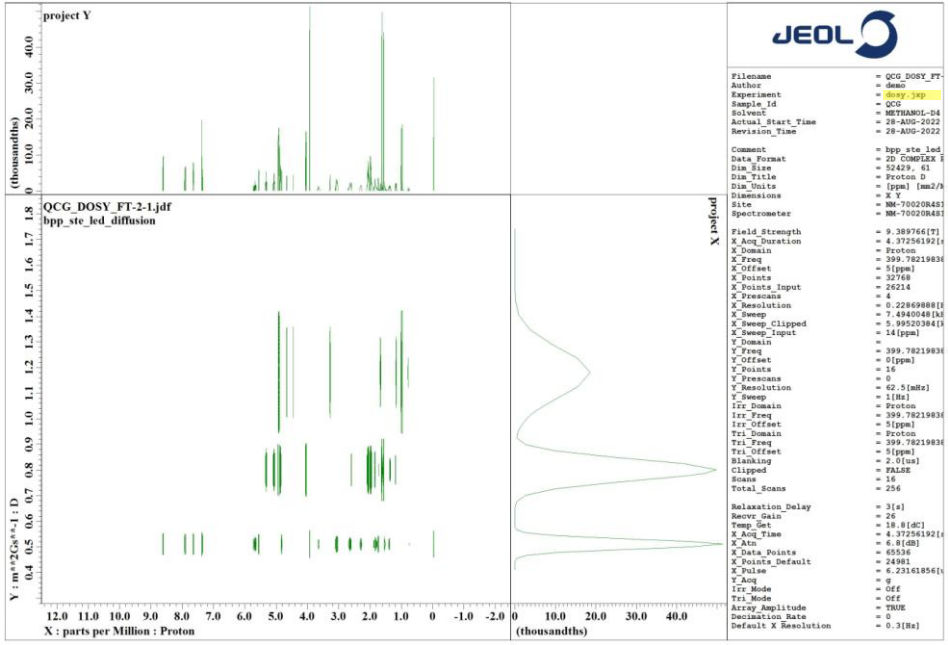
Info

Reached Lock State 'AUTOLOCK'
Estimated Diffusion coefficient is 7.78834*10E-10 m^2/s
Delta set to 2.14247[ms] to achieve 90% signal attenuation

1H 90 degree pulse width for QCG estimated as 6.12549[us]

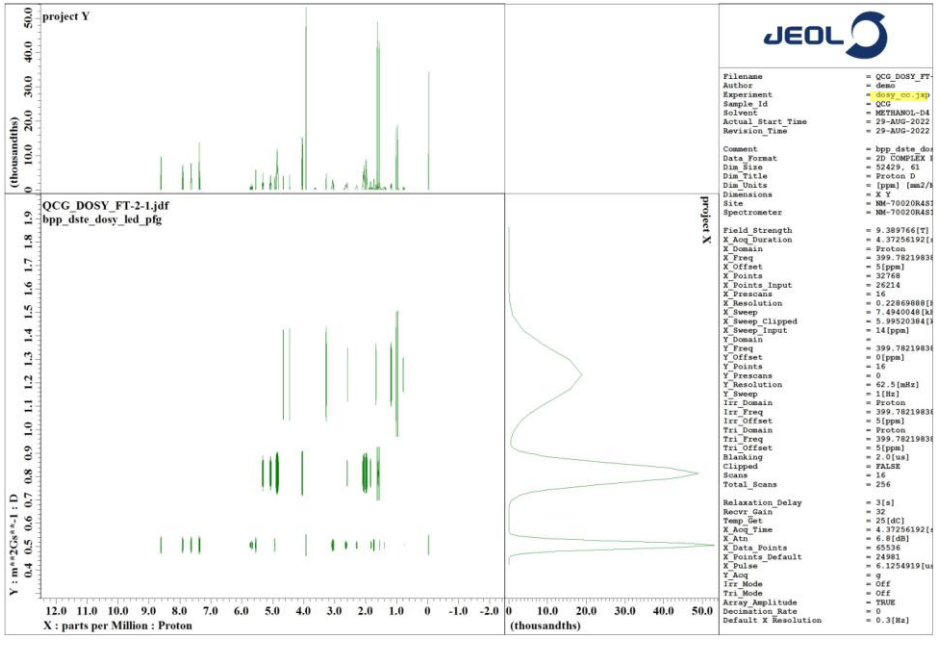
## Room temperature

Convection test indicates there is no significant convection in this sample (signal ratio of 0.9784)




## 25C

There is convection in this sample (signal ratio of 0.35024. Proceeding with convection compensated experiment

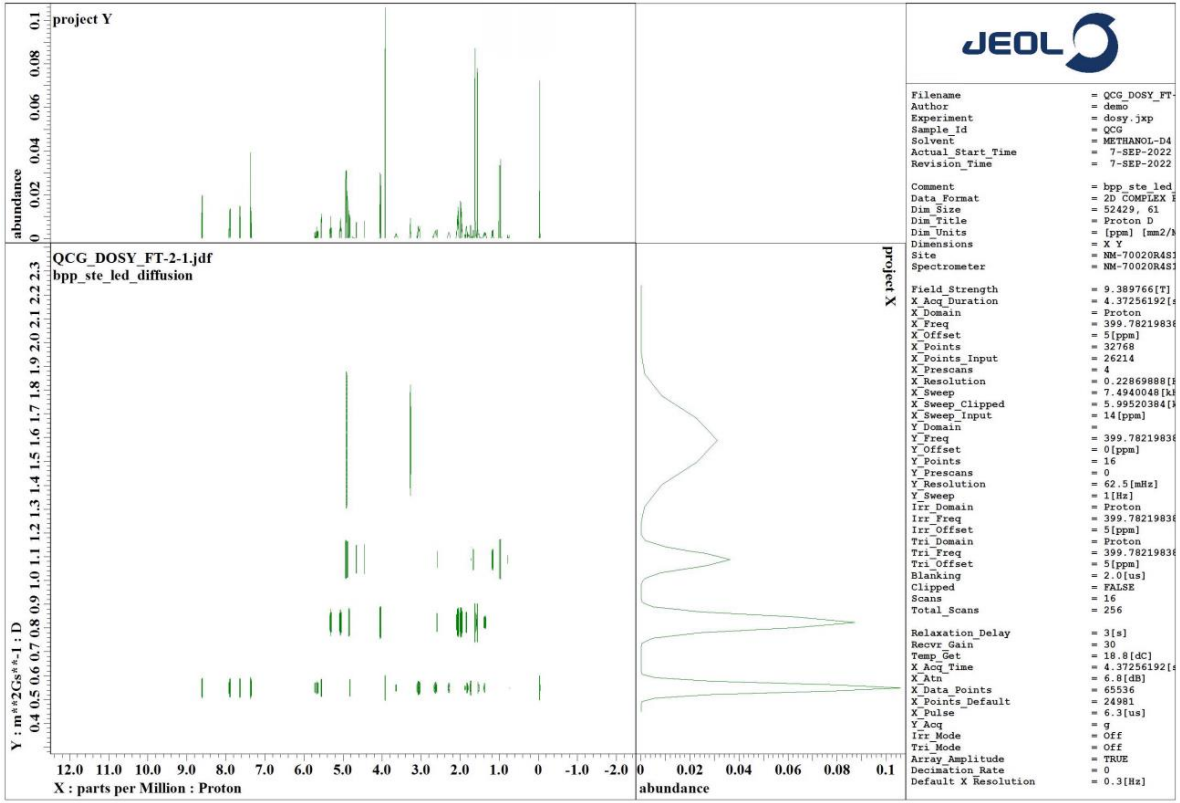


# Results from automatic_setup (Quinine + Geraniol + Camphene)

MW set to 136

Info

Reached Lock State 'AUTOLOCK'
Estimated Diffusion coefficient is 11.71103*10E-10 m^2/s
Delta set to 1.74719[ms] to achieve 90% signal attenuation




| | |
|---|---|
| Filename | = QCG_DOSY_FT- |
| Author | = demo |
| Experiment | = dosy_jxp |
| Sample_Id | = QCG |
| Solvent | = METHANOL-D4 |
| Actual_Start_Time | = 7-SEP-2022 |
| Revision_Time | = 7-SEP-2022 |
| Comment | = bpp_ste_led |
| Data_Format | = 2D COMPLEX |
| Dim_Size | = 52429, 61 |
| Dim_Title | = Proton D |
| Dim_Units | = [ppm] [mm2/ |
| Dimensions | = X Y |
| Site | = NM-70020R4S |
| Spectrometer | = NM-70020R4S |
| Field_Strength | = 9.389766[T] |
| X_Acq_Duration | = 4.37256192[ |
| X_Domain | = Proton |
| X_Freq | = 399.7821983 |
| X_Offset | = 5[ppm] |
| X_Points | = 32768 |
| X_Points_Input | = 26214 |
| X_Prescans | = 4 |
| X_Resolution | = 0.22869888[ |
| X_Sweep | = 7.4940048[k |
| X_Sweep_Clipped | = 5.99520384[ |
| X_Sweep_Input | = 14[ppm] |
| Y_Domain | = |
| Y_Freq | = 399.7821983 |
| Y_Offset | = 0[ppm] |
| Y_Points | = 16 |
| Y_Prescans | = 0 |
| Y_Resolution | = 62.5[mHz] |
| Y_Sweep | = 1[Hz] |
| Irr_Domain | = Proton |
| Irr_Freq | = 399.7821983 |
| Irr_Offset | = 5[ppm] |
| Tri_Domain | = Proton |
| Tri_Freq | = 399.7821983 |
| Tri_Offset | = 5[ppm] |
| Blanking | = 2.0[us] |
| Clipped | = FALSE |
| Scans | = 16 |
| Total_Scans | = 256 |
| Relaxation_Delay | = 3[s] |
| Recvr_Gain | = 30 |
| Temp_Get | = 18.8[dC] |
| X_Acq_Time | = 4.37256192[ |
| X_Atn | = 6.8[dB] |
| X_Data_Points | = 65536 |
| X_Points_Default | = 24981 |
| X_Pulse | = 6.3[us] |
| Y_Acq | = g |
| Irr_Mode | = Off |
| Tri_Mode | = Off |
| Array_Amplitude | = TRUE |
| Decimation_Rate | = 0 |
| Default X Resolution | = 0.3[Hz] |