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**Adosy\_ste\_setup\_BBFO** Topspin 3.6.5 600MHz.

Usage: Optimize critical parameters for **DOSY** experiment for solution without solvent suppression at BBFO probe.

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Parameters to be optimized: **p30**, **d20**, **rg** and **ns**.

For standard D2O/Cu test sample, **sine.100** gradient used, **p30** 1.0ms, **d20** 0.06s; **d1** 7s; **ns** 1

\*\*\*\*\*WARNING:

DONOT increase **p30** more than 2.5ms.

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General Instruction:

1. Create experiment **#1**: set stable temperature (type **edte**), tune and shim well.
  - Run 1H NMR (**rpar Ah1**): obtain **o1p** and **sw**
  - Remark: For 100% water, detune the probe by 10MHz, and calibrate 90.
2. Calibrate proton 90 degree pulse at **#1**. Obtain **p1** and **p11** value for your sample.
  - Automation: type **pulsecal**
  - Manually: **rpar Ah1\_90\_water**
3. Create experiment **#2**:
  - **rpar Adosy\_ste\_setup\_BBFO**
  - **getprosol**
4. For staff only:
  - Check the record for gradient calibration (**gradpar cf** with **edhead** gradient history).
5. Update **o1p**, **sw** and **p1** (from step 1-2), **ns** 1, **d1** 7s to **#2**
  - Set **d20** (default 60ms, typical 50-150ms)
  - Check to use the following default values as a starter {**gpz6** 2; **gpz7** -17.13 (fixed); gradient pulse shape: **sine.100**}
6. Optimize: **p30** from 1ms (typical 1-2ms sample dependent)

- Set a value of **p30** between 1.3-1.5ms for water,
  - Demo sample (H<sub>2</sub>O/D<sub>2</sub>O with Cu<sup>+2</sup>). uses 1.0ms.
  - **rga** and **zg** to collect and phase the first spectrum properly.
7. Do three measurements vary parameter **gpz6**:
- At #2, type **i** or **iexpno** to create #3
  - Change **gpz6** to **95** at #3
  - At #2, type **multizg**, then type **2**
  - At #2 and #3, type **efp;apk**
  - Compare peak intensities for #2 and #3 using multi-display; signals should decrease approx. **50** ratio.
8. Adjust **p30** values:
- If signal decay too rapidly, return to #2 and #3, decrease the **p30** value by 0.1ms; repeat **multizg** at step 7.
  - If signal decay too slowly, return to #2 and #3, increase the **p30** value by 0.1ms; repeat **multizg** at step 7.
  - Remark: **p30** should not exceed 2.5ms. If **p30** is out of its range, go to the next step.
9. If step 8 fails, adjust **d20** by 0.1ms and repeat steps 7 & 8, then step 9 as needed.
- **d20** (default 60ms, typical 50-150ms)
  - The larger the **p30** or/and **d20**, the faster the decay in intensities.
10. Optimize **ns** for your sample:
- Set **gpz6** set to 95% with **ns** = 8
  - **zg** to collect a spectrum; check the sufficient intensities of the signal of interests by increasing **ns** = 8