Ketyl Radical Test Solution – Thought Process, Preparation, and Use

NOTES:
This is a qualitative test for the presence of H_2O or O_2 in hydrocarbon solvents.
This test is strictly qualitative. This is not a quantitative determination of O_2 / H_2O.
Use DRY THF for preparation! If unsure, stir THF over sodium for 24 h and distill.

THF FW = 72.11 d = 0.899
1 mL = 0.0123 mol = 7.424×10^{21} particles = 7.424×10^{15} millions

Desire < 10 parts H_2O per million parts THF
=> 7.424×10^{16} particles H_2O in 1 mL THF

Estimate 1 drop from 9" pipette is 175 µL (± 50 µL)
Propose that 4x ketyl radical is sufficiently sensitive

Benzophenone ($Ph_2C{=}O$) FW = 182.22
Ketyl Radical ($Ph_2C\text{-}O^{\cdot}Na^{+}$) FW = 205.21
Conc. of one drop should be: 5×10^{-7} mol / 175 µL = 1.026×10^{-4}g / 0.175 mL = 0.6 mg/mL

This requires 12 mg of ketyl radical in 20 mL THF, or 10.7 mg benzophenone in 20 mL THF

Dissolved 17.0 mg benzophenone in 20 mL THF with ~12 mg Na^0. Stirred for 48 h. Solution became dark inky purple. Tested box solvents by adding one drop of ketyl radical solution to 1 mL of solvent – Petroleum ether, diethyl ether, toluene, and benzene remain purple. THF (when dry) may appear blue due to dilution. The expected order of color change is usually: purple, blue, green, yellow, clear. Halogenated solvents cannot be tested by this method as they undergo radical chemistry, as will proteo-solvents, acetone, and acetonitrile.

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