

Experimental Protocol: Resonant Two-Photon Synthesis of 1D Furan Nanothreads (FNT) from Hemp-Derived Precursors

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1 Objective

To verify the hypothesis that **Two-Photon Absorption (TPA)** at $\lambda = 450$ nm can initiate the polymerization of hemp-derived furan into ordered crystalline nanothreads at a reduced static pressure of **5 GPa**. This protocol seeks to identify the $[4 + 4]$ cycloaddition "seed" formation through non-linear photonic excitation.

2 Materials & Precursor Preparation

- **Precursor:** Bio-derived Furan (C_4H_4O), extracted via decarbonylation of hemp-hurd furfural.
- **Purity:** $> 99.9\%$ (verified via GC-MS), anhydrous.
- **Pressure Standard:** Ruby fluorescence microspheres ($Cr^{3+} : Al_2O_3$) for *in-situ* ruby manometry.

3 Apparatus Configuration

3.1 High-Pressure Cell

- **Primary:** Piston-Cylinder Apparatus (for scalability) or Symmetric Diamond Anvil Cell (DAC) with Type IIa diamonds.
- **Gasket:** Rhenium or T301 Steel, pre-indented to $35\ \mu\text{m}$ with a $150\ \mu\text{m}$ sample chamber.

3.2 Photonic Initiation System

- **Laser Source:** Femtosecond Ti:Sapphire pulsed laser (doubled).
- **Excitation Wavelength:** $\lambda_{ex} = 450$ nm.
- **Pulse Duration:** < 100 fs.
- **Repetition Rate:** 80 MHz.
- **Target Energy:** $E_{sum} \approx 5.5$ eV (Resonant S_1 transition via TPA).

4 Experimental Procedure

1. **Loading:** Under an inert *Ar* atmosphere, load liquid furan and ruby spheres into the sample chamber.
2. **Compression Phase:** Increase static pressure at a rate of 0.5 GPa/h until the **Phase III** transition (trans-planar ordering) is observed ($\approx 4.8 - 5.2$ GPa).
3. **Photonic Initiation:** Align the 450 nm laser focal point to the center of the sample volume. Apply pulses at a flux of $\Phi \approx 1.5 \text{ J/cm}^2$.
4. **Observation:** Monitor *in-situ* Raman spectra for the emergence of sp^3 CH stretching modes ($\sim 2900 - 3000 \text{ cm}^{-1}$) and the decline of sp^2 aromatic peaks.
5. **Decompression:** Slowly release pressure (0.2 GPa/h) to ambient to ensure structural relaxation without "unzipping."

5 Safety & Hazard Control

- **Class 4 Laser Protocol:** All personnel must wear OD7+ eyewear rated for 450 nm.
- **Pressure Safety:** Utilize a secondary Lexan shield during the 5 GPa hold to mitigate potential anvil failure or gasket blowout.

6 Expected Analytical Signatures

Metric	Pre-Synthesis (Monomer)	Post-Synthesis (FNT)
Raman Shift ($C = C$)	$\sim 1500 \text{ cm}^{-1}$ (Strong)	Disappeared/Weakened
Raman Shift (sp^3)	Negligible	$\sim 1330 \text{ cm}^{-1}$ (Broad)
XRD d -spacing	N/A	$\sim 4.8 \text{ \AA}$ (Hexagonal)
Appearance	Clear Liquid	Translucent Solid Fiber

Table 1: Comparative markers for 5 GPa furan-to-nanothread transformation.