

# In-situ Raman spectroscopy as a probe for the effect of power on microwave-promoted Suzuki coupling reactions

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## Apparatus description

Microwave reactions were conducted using a modified monomode microwave unit (CEM Discover<sup>®</sup>). The machine consists of a continuous  
10 Focused<sup>™</sup> microwave power delivery system with operator selectable power output from 0-300 W. Reactions were performed in glass  
vessels (capacity 10 mL) sealed with a septum. The pressure is controlled by a load cell connected to the vessel. The temperature of the  
contents of the vessel was monitored either using a calibrated infrared temperature control mounted under the reaction vessel using a  
calibrated fiber-optic probe inserted into the reaction vessel by means of a sapphire immersion well. All experiments were performed using a  
stirring option whereby the contents of the vessel are stirred by means of a rotating magnetic plate located below the floor of the microwave  
15 cavity and a Teflon-coated magnetic stir bar in the vessel.

The Raman system was provided by Enwave Optronics ([www.enwaveopt.com](http://www.enwaveopt.com)).

EXCITATION SOURCE:	NIR, frequency stabilized, narrow linewidth diode laser at 785 nm. 20 Laser power at sample ~200 mW. Linewidth < 2 cm <sup>-1</sup> . Fiber-coupled laser output (100 μm, 0.22 NA).
FIBER-OPTIC PROBE:	Permanently-aligned two single fiber combination 100 μm excitation fiber, 200 μm collection fiber. Working distance: 8 mm (standard). 25 Rayleigh rejection: O.D. > 7 at laser wavelength.
CCD DETECTOR:	High sensitivity linear CCD array. Temperature regulated (at 13 °C) operation for long integration time and stable dark reference subtraction. Pixel Size: 14 μm x 200 μm (2048 Pixels); 16 Bit digitization. 30
SPECTROGRAPH:	Symmetrical crossed Czerny-Turner design. Resolution: ~10 cm <sup>-1</sup> at 785 nm. Excitation spectral coverage: 300 cm <sup>-1</sup> to 2400 cm <sup>-1</sup> . Built-in software calibration.
SYSTEM SOFTWARE:	Data collection software, data files exported into .dat format, converted to .txt format and imported into 35 Microsoft Excel.

To interface the microwave unit and Raman spectrometer, a hole (0.8 cm i.d.) was drilled in the microwave cavity and an RF stub attached to  
the outer cavity wall (to prevent microwave leakage) and an extender (2.16 cm i.d.) attached to this, reaching through to the outer casing of  
the microwave unit. The fiber-optic probe was placed into the cavity and the laser focused through a quartz light tube. A distance between  
40 the reaction vessel and fiber-optic probe tip of 0.5 cm was optimal.