

Immobilized Palladium Nanoparticles within polymers as active catalysts for Suzuki-Miyaura reaction

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General Methods and Reagents

All reagents were purchased from Aladdin Reagent Company, Sigma-Aldrich Company and Alfa-Aesar Company without further purification. ¹H-NMR spectra were measured with a Bruker AVANCE 400D spectrometer in CDCl₃ using tetramethylsilane (TMS) as internal reference. TEM images were obtained using a JEOL JEM-2010 (200 kV) TEM instrument and SEM images were performed on a HITACHI S-4800 field-emission scanning electron microscope. Thermogravimetry (TG) analysis was carried out with a STA409 instrument under dry air at a heating rate of 10 °C/min. X-ray diffraction (XRD) patterns were collected on the Bruker D8 Advance powder diffractometer using Ni-filtered Cu K α radiation source at 40 kV and 20 mA, from 5° to 80° with a scan rate of 0.5° / min. The BET surface areas were measured at -77 °C with liquid nitrogen using a Micromeritics ASAP2010 analyzer. The samples were degassed at 150 °C under vacuum of 10⁻³ Torr before analysis. Fourier transform infrared (FT-IR) spectra were recorded on a Nicolet 360 FT-IR instrument (KBr discs) in the 4000-400 cm⁻¹ region. The amount of Pd was measured using a Jarrell-Ash 1100 ICP-AES spectrometer (Inductively Coupled Plasma-Atomic Emission Spectrometry).

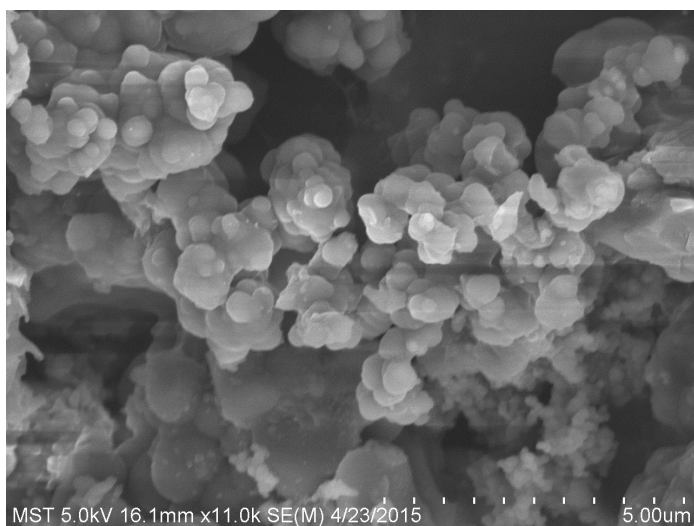


Fig. S1 Scanning electron micrograph (SEM) images of Pd@PNP catalyst

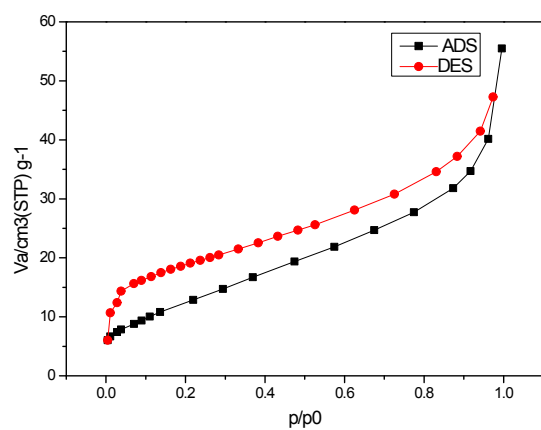


Fig. S2 Nitrogen adsorption-desorption isotherm of the catalyst Pd@PNP

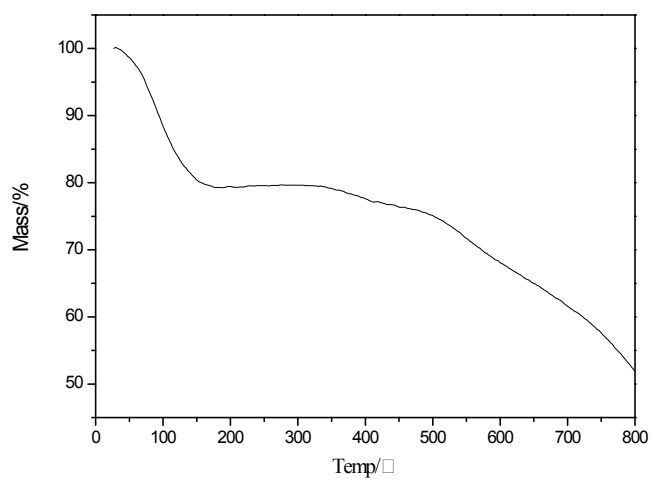


Fig. S3 TG curve of Pd@PNP catalyst

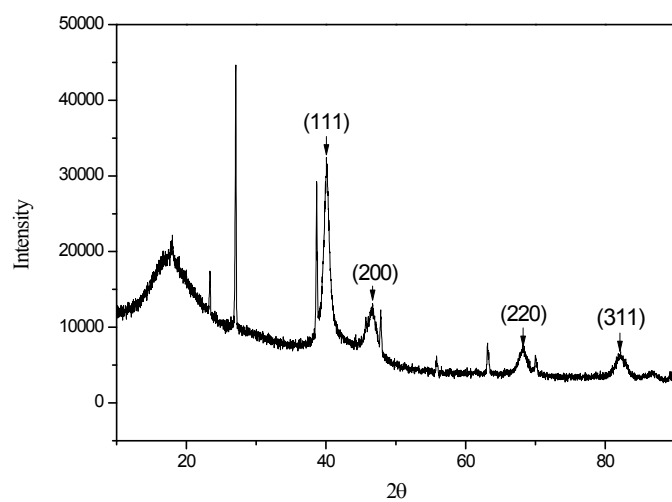


Fig.S4 XRD of Pd@PNP catalyst

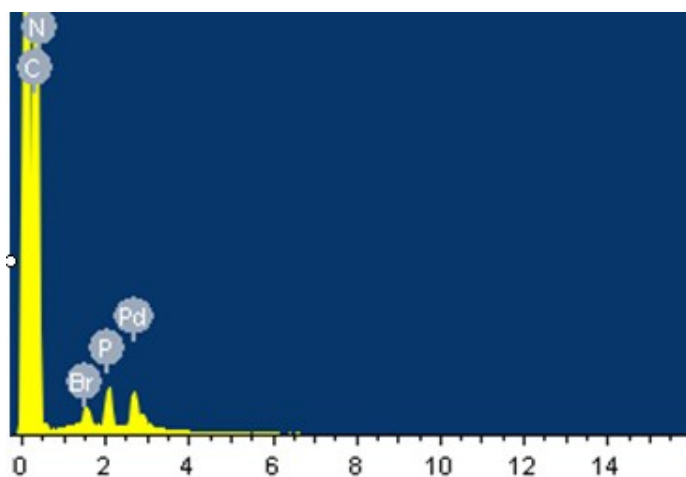


Fig. S5 EDX elemental analysis of Pd@PNP catalyst

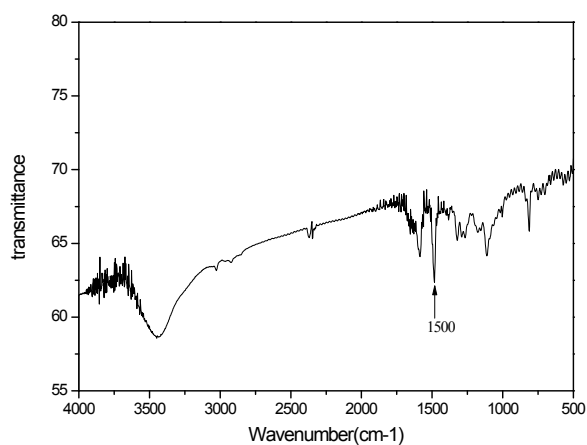


Fig. S6 The FT-IR spectra of Pd@PNP catalyst

The product data.

4-Nitro-biphenyl. ^1H NMR (500 MHz, CDCl_3): δ 7.48-7.56 (m, 3H), 7.68 (d, $J=7.5$ Hz, 2H), 7.79 (d, $J=9.0$ Hz, 2H), 8.35 (d, $J=9.0$ Hz, 2H).

2,6-Dimethyl-biphenyl. ^1H NMR (500 MHz, CDCl_3): δ 2.14 (s, 6H), 7.21-7.31 (m, 5H), 7.41-7.45 (m, 1H), 7.52 (t, $J=7.5$ Hz, 2H).

4-Acetylbiphenyl. ^1H NMR (500 MHz, CDCl_3): δ 2.69 (s, 3H), 7.45 (t, $J = 7.0$ Hz, 1H), 7.52 (t, $J = 7.5$ Hz, 2H), 7.68 (d, $J = 7.5$ Hz, 2H), 7.74 (d, $J = 8.5$ Hz, 2H), 8.08 (d, $J = 8.0$ Hz, 2H).

4-Phenylbenzaldehyde. ^1H NMR (500 MHz, CDCl_3): δ 7.45-7.55 (m, 3H), 7.69 (d, $J=7.0$ Hz, 2H), 7.81 (d, $J=8.0$ Hz, 2H), 8.00 (d, $J=8.0$ Hz, 2H), 10.11 (s, 1H).

4-Methyl-biphenyl. ^1H NMR (500 MHz, CDCl_3): δ 2.45 (s, 3H), 7.31 (t, $J = 4.5$ Hz, 2H), 7.38 (t, $J = 7.5$ Hz, 1H), 7.48 (t, $J = 8.0$ Hz, 2H). 7.55 (d, $J = 8.0$ Hz, 2H). 7.62-7.64 (m, 2H).

4-Phenylbenzotrile. ^1H NMR (500 MHz, CDCl_3): δ 7.46-7.55 (m, 3H), 7.64 (d, $J = 7.5$ Hz, 2H), 7.73-7.79 (m, 4H).

4-Tert-butylbiphenyl. ^1H NMR (500 MHz, CDCl_3): δ 1.44 (s, 9H), 7.39 (t, $J = 7.0$ Hz, 1H), 7.48-7.55 (m, 4H), 7.61 (d, $J=8.4$ Hz, 2H), 7.66 (d, $J=7.5$ Hz, 2H).

4-Methoxy-biphenyl. ^1H NMR (500 MHz, CDCl_3): δ 3.90 (s, 3H), 7.03 (d, $J = 9.0$ Hz, 2H), 7.35 (t, $J = 7.0$ Hz, 1H), 7.46 (t, $J = 7.5$ Hz, 2H), 7.57-7.61 (m, 4H).

2-Methoxy-biphenyl. ^1H NMR (500 MHz, CDCl_3): δ 3.90 (s, 3H), 7.07-7.15 (m, 2H), 7.42 (t, $J=7.5$ Hz, 3H), 7.51 (t, $J=7.5$ Hz, 2H), 7.63-7.65 (m, 2H).

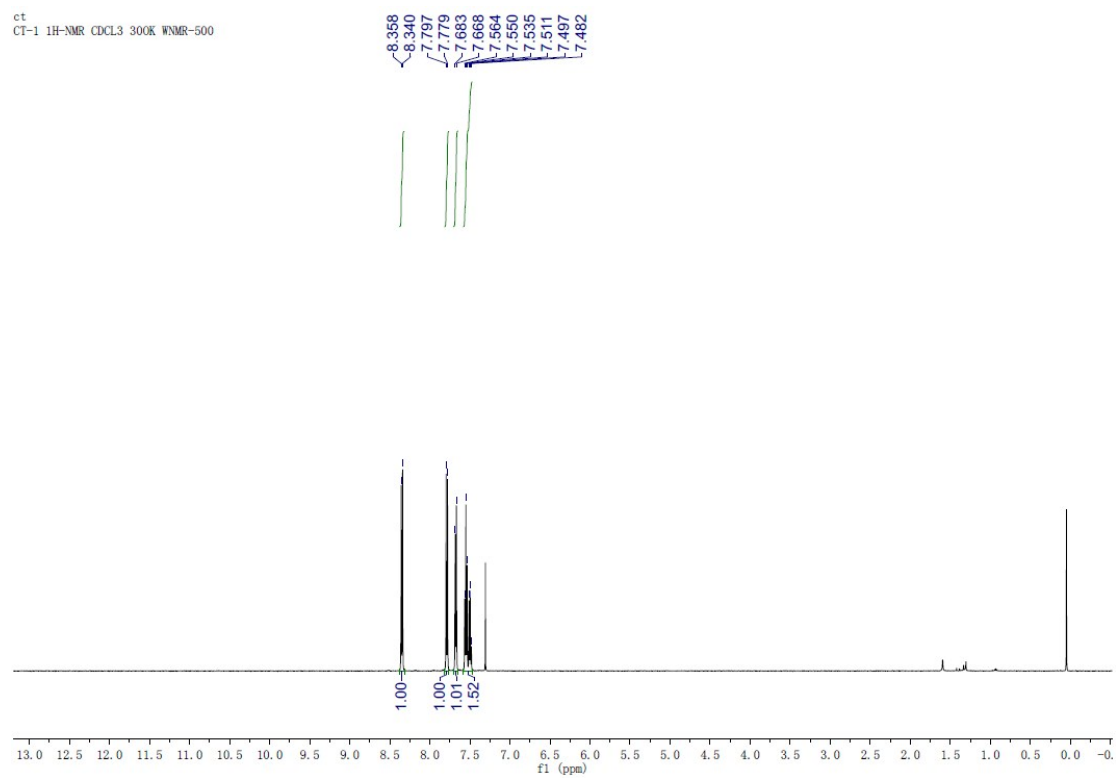
2-Nitro-biphenyl. ^1H NMR (500 MHz, CDCl_3): δ 7.36-7.40 (m, 2H), 7.44-7.55 (m, 5H), 7.65-7.68 (m, 1H), 7.89-7.91 (m, 1H).

Biphenyl-2-carbonitrile. ^1H NMR (500 MHz, CDCl_3): δ 7.48-7.62 (m, 7H), 7.70(t, $J = 7.5$ Hz, 1H), 7.82 (d, $J = 7.5$ Hz, 1H).

2-Methyl-biphenyl. ^1H NMR (500 MHz, CDCl_3): δ 2.38 (s, 3H), 7.31-7.36 (m, 4H), 7.43 (t, $J = 9.0$ Hz, 3H), 7.49-7.55 (m, 2H).

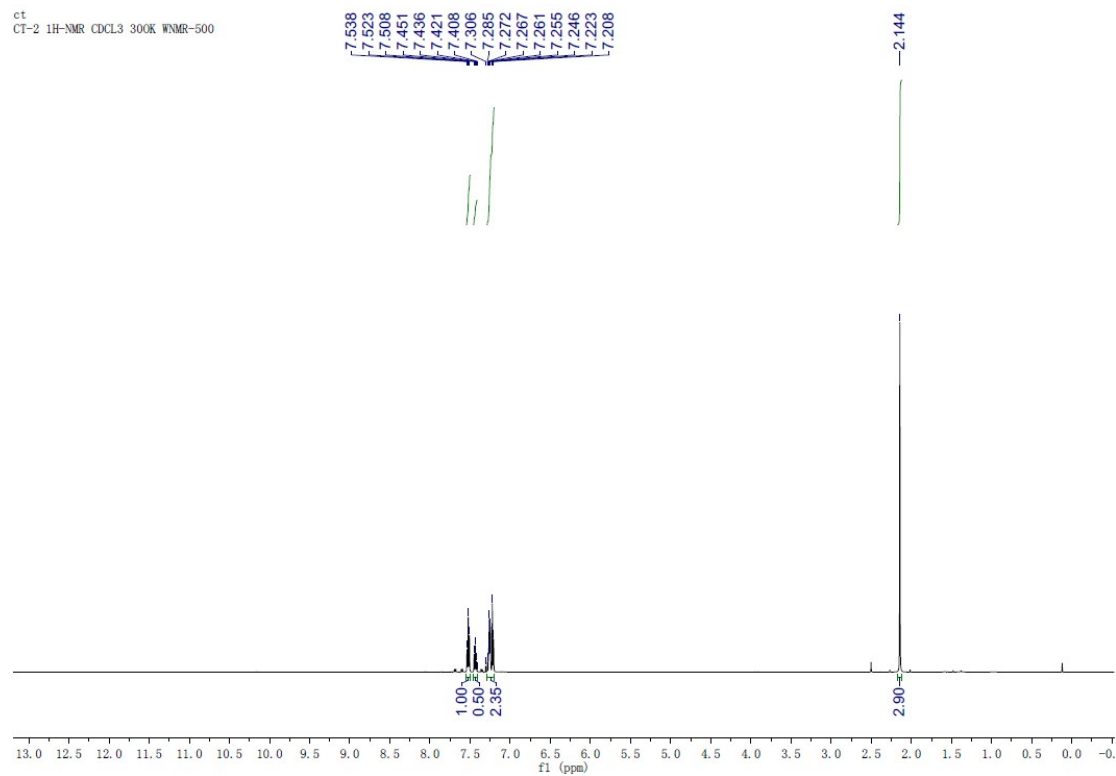
4-Nitro-biphenyl

ct
CT-1 1H-NMR CDCl3 300K WNMN-500



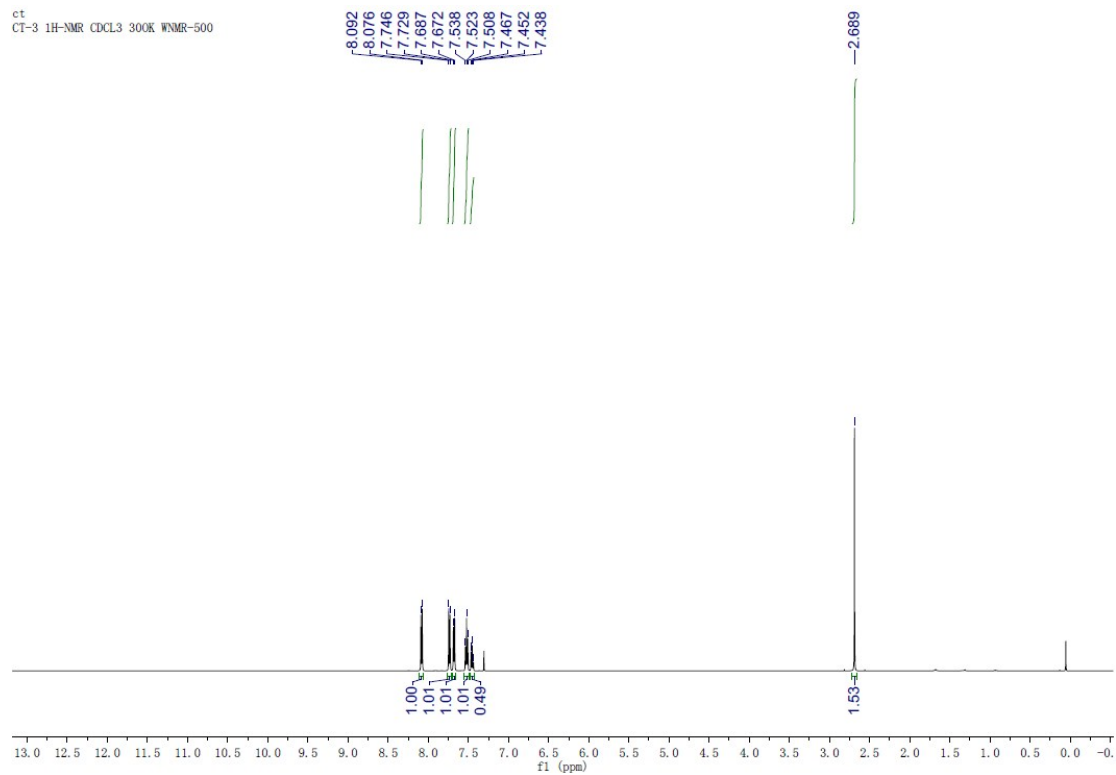
2,6-Dimethyl-biphenyl

ct
CT-2 1H-NMR CDCl3 300K WNMN-500



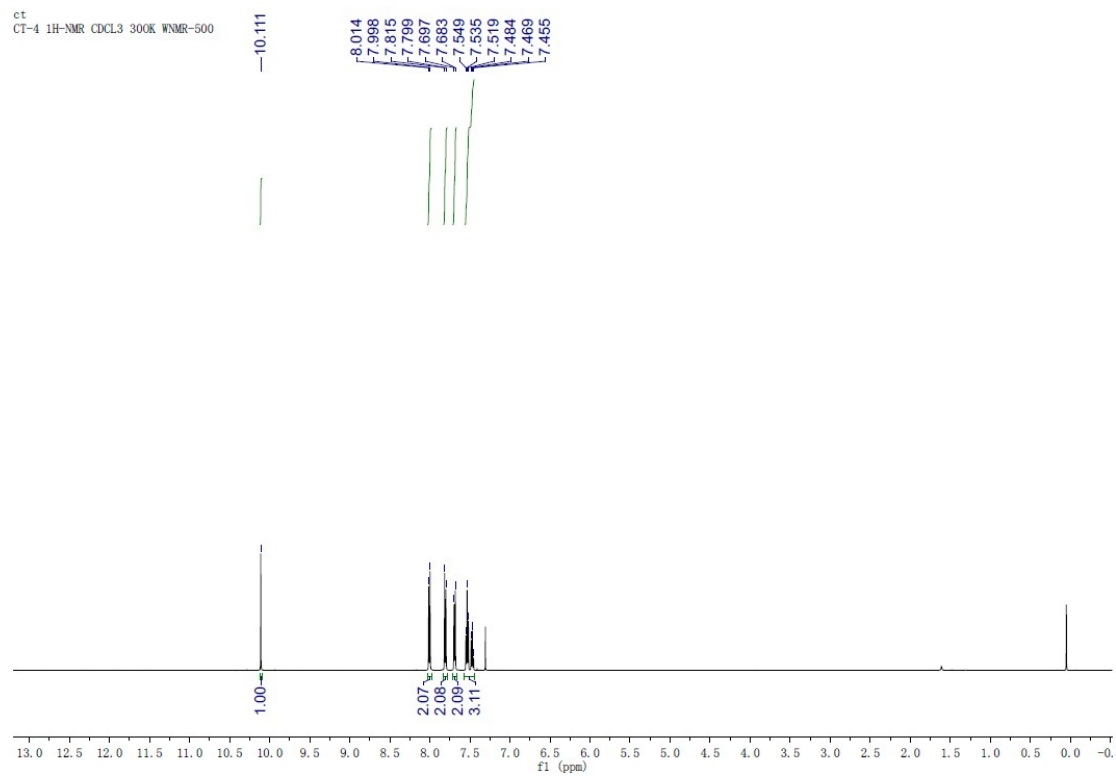
4-Acetylbiphenyl

ct
CT-3 1H-NMR CDCL3 300K WNM-500



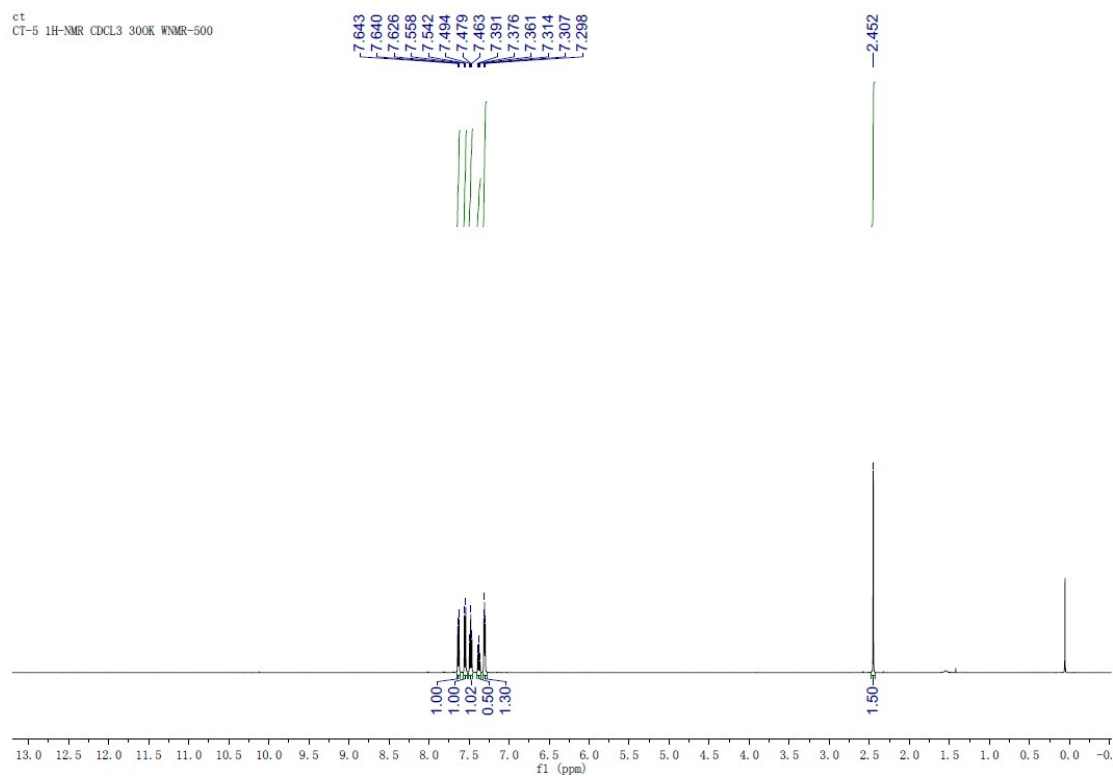
4-Phenylbenzaldehyde

ct
CT-4 1H-NMR CDCL3 300K WNM-500



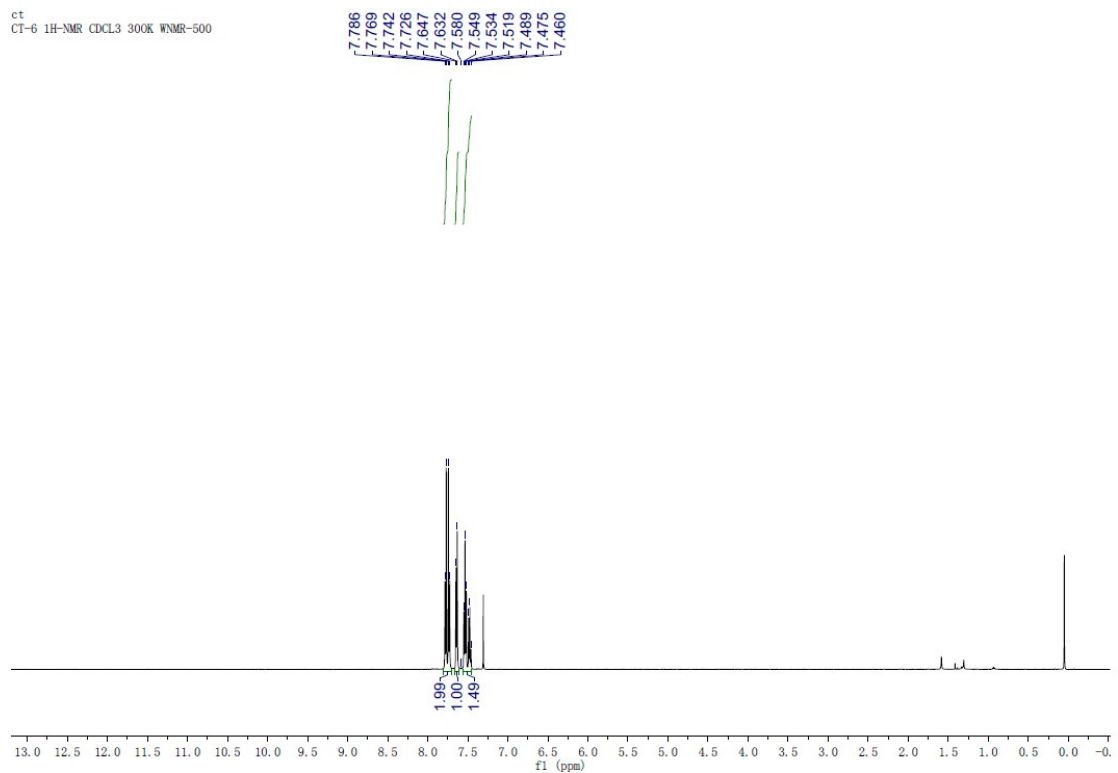
4-Methyl-biphenyl

ct
CT-5 1H-NMR CDCL3 300K WNM-500



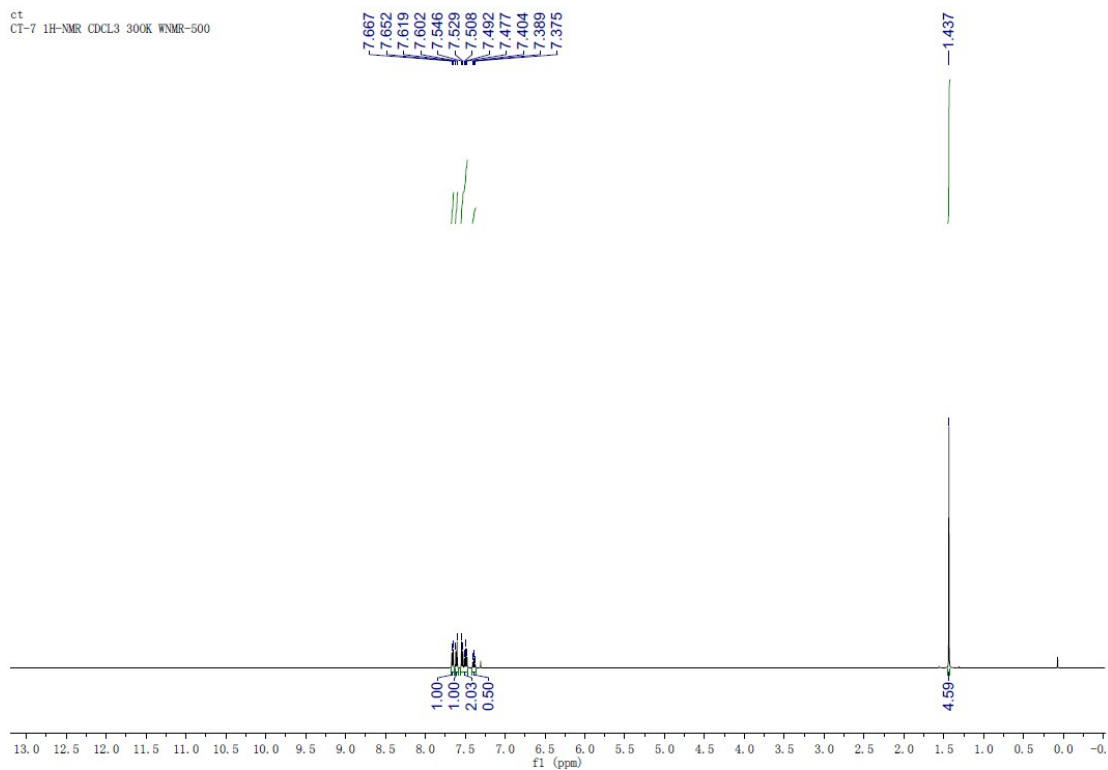
4-Phenylbenzotrile

ct
CT-6 1H-NMR CDCL3 300K WNM-500



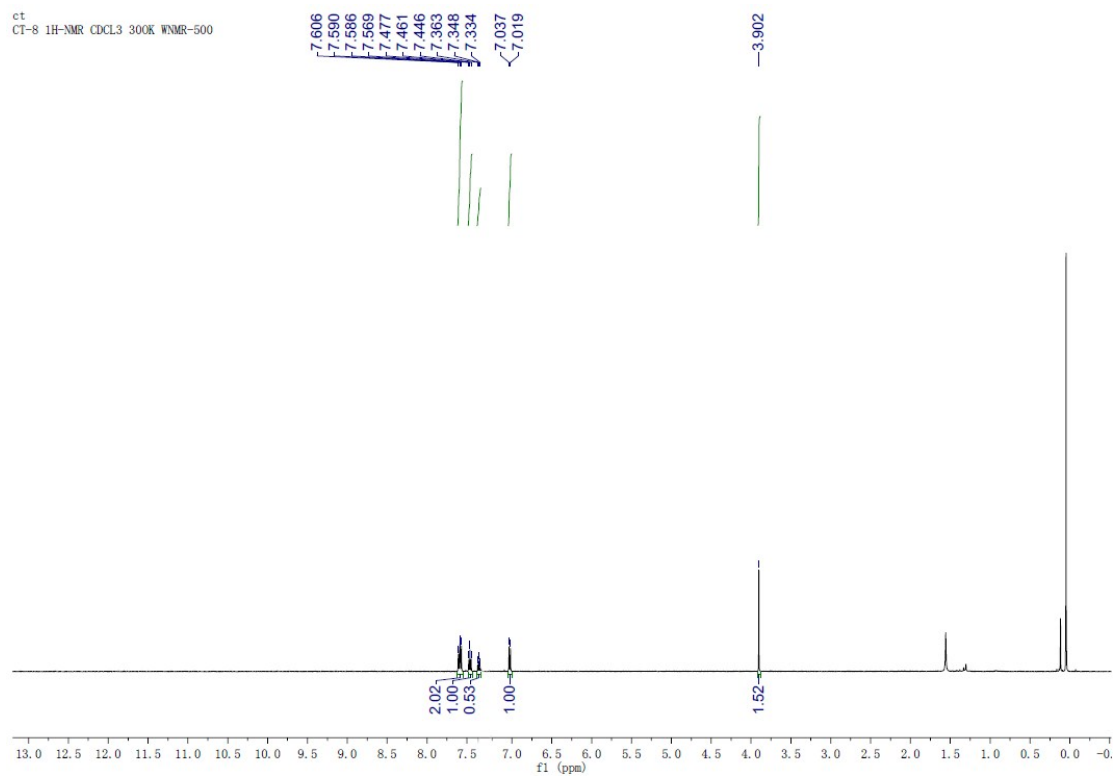
4-Tert-butylbiphenyl

ct
CT-7 1H-NMR CDCL3 300K WNM-500

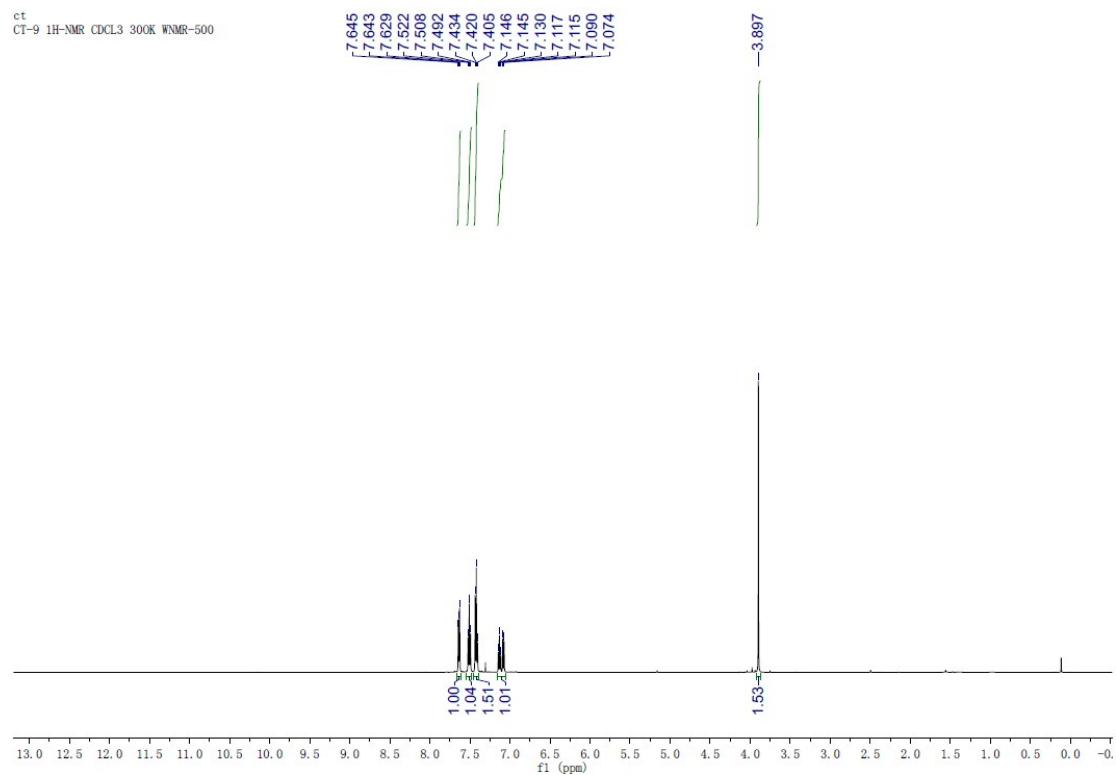


4-Methoxy-biphenyl

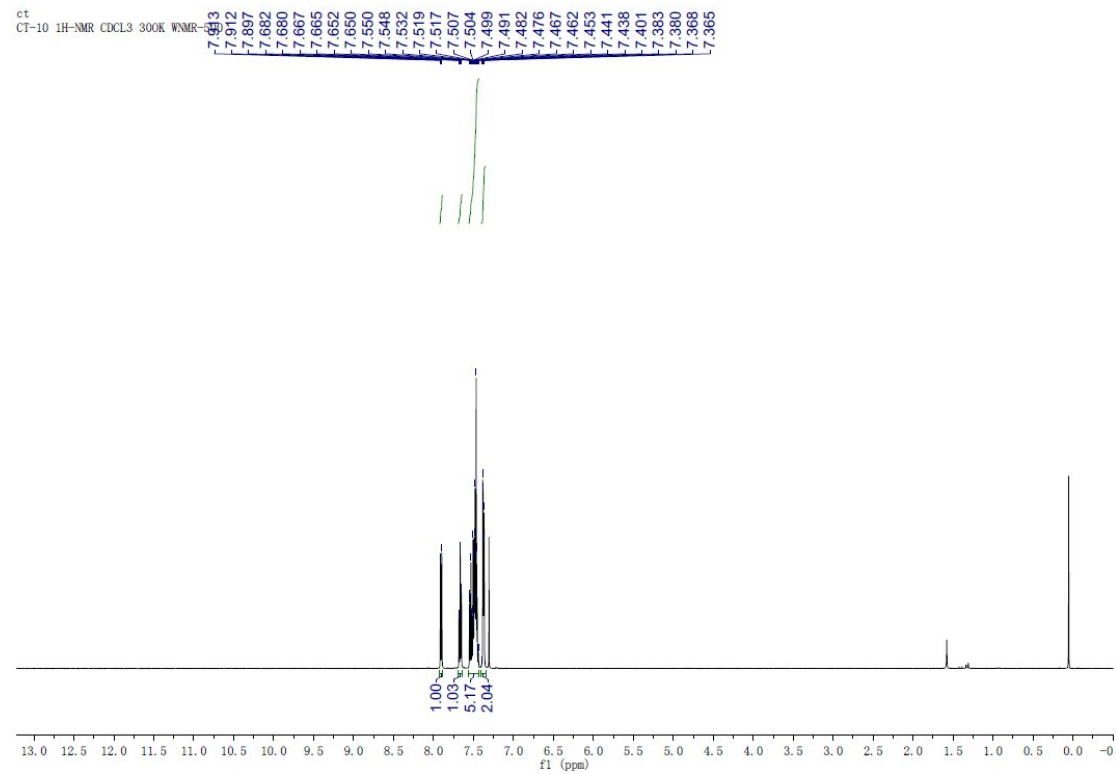
ct
CT-8 1H-NMR CDCL3 300K WNM-500



2-Methoxy-biphenyl

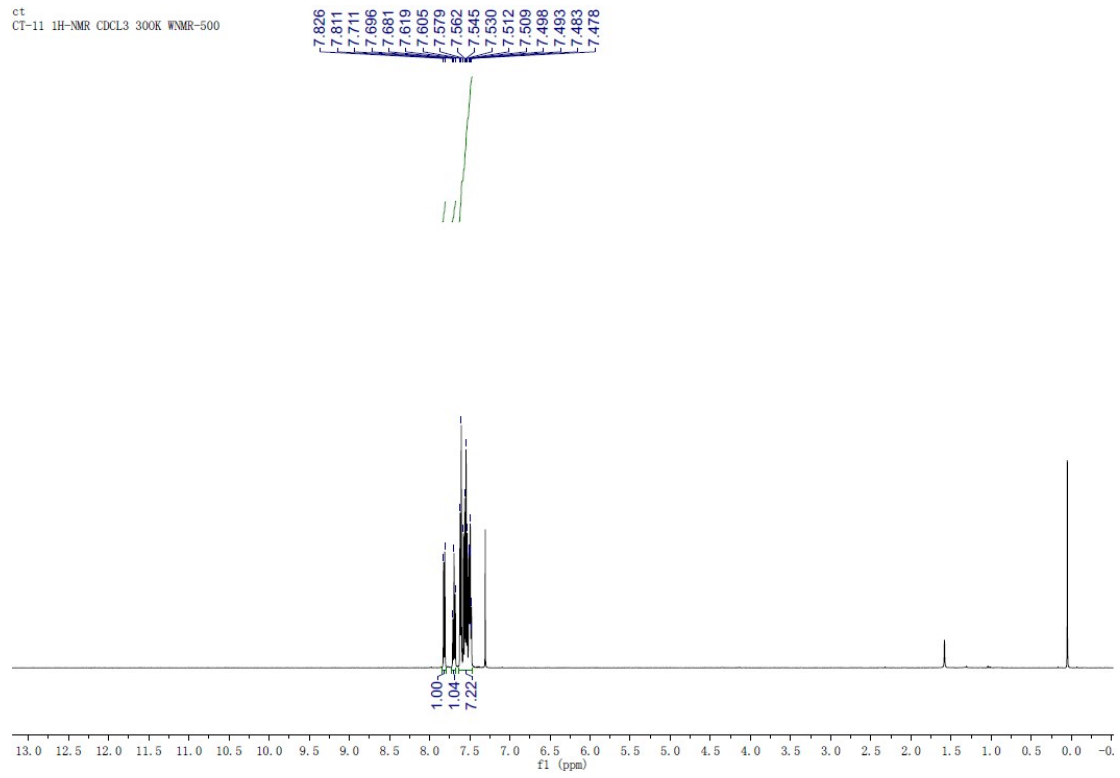


2-Nitro-biphenyl



Biphenyl-2-carbonitrile

ct
CT-11 1H-NMR CDCL3 300K WNM-500



2-Methyl-biphenyl

ct
CT-12 1H-NMR CDCL3 300K WNM-500

