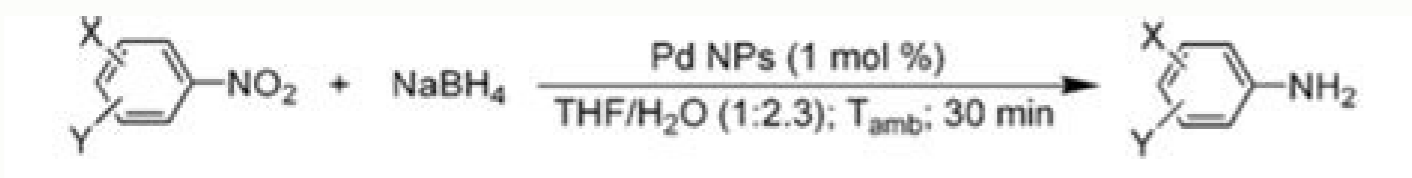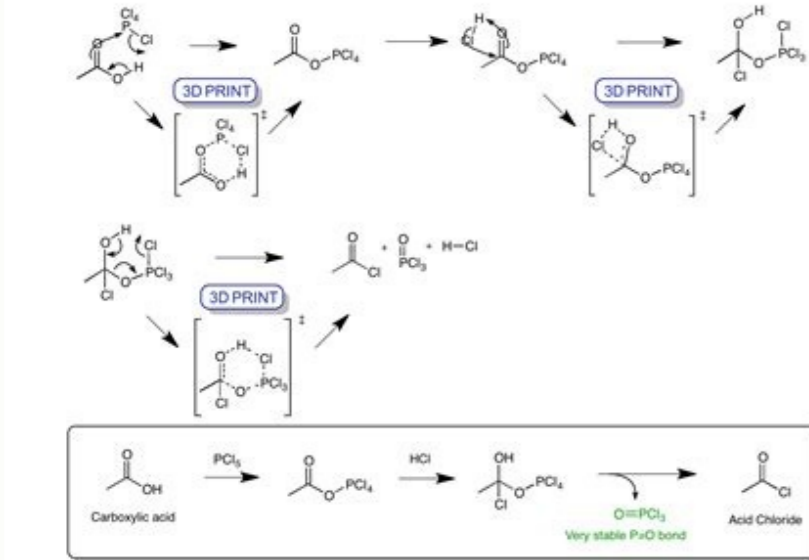I'm not robot!

product	catalyst (mol.%)	yield (%, isol.)	ee (%)	product	catalyst (mol.%)	yield (%, isol.)	ee (%)
(structure OH/CN, OMe)	0.25	96	95	(furan structure OH/CN)	0.25	83	96
(structure OH/CN, F)	0.5	95	91	(structure OH/NO₂, Br)	0.5	54	91
(structure OH/CN, NC)	0.25	97	86	(structure OMe OH/NO₂)	0.5	93	83
				(structure Ph OH/Cl)	0.25	93	91

In-4 (0.02 mol%)

HCOOH (4 equiv.)

water, 80 °C

- High efficiency; ▲ Full conversion;
- Purification by extraction;
- Wide scope; ▲ Excellent yields;
- Excellent functionality tolerance.

X—NO₂ + NaBH₄ → (Pd NPs (1 mol %), THF/H₂O (1:2.3); T_amb; 30 min) → X—NH₂

Substituents X,Y	Pd NPs 1 Selectivity[b] / %	Pd NPs 2 Selectivity[b] / %
2-F (Y=H)	100	100
4-Br (Y=H)	69	54
2-Cl-5-OH	100	100
3-Cl-4-NH₂	100	100
3-Br-4-OH	97	100
3-Cl-4-OH	100	100
2-I (Y=H)		
3-I (Y=H)	—[b]	—[b]
4-I (Y=H)		

[a]All the reactions were carried out using (0.05 mmol) of the aromatic nitro compound, 1 mol% of Pd NPs, 11 equivalents of NaBH₄ and 9.9 mL of THF/H₂O (1:2.3); [b]for iodo-nitrobenzene, the selectivity values are not shown because a complex mixture products was obtained.
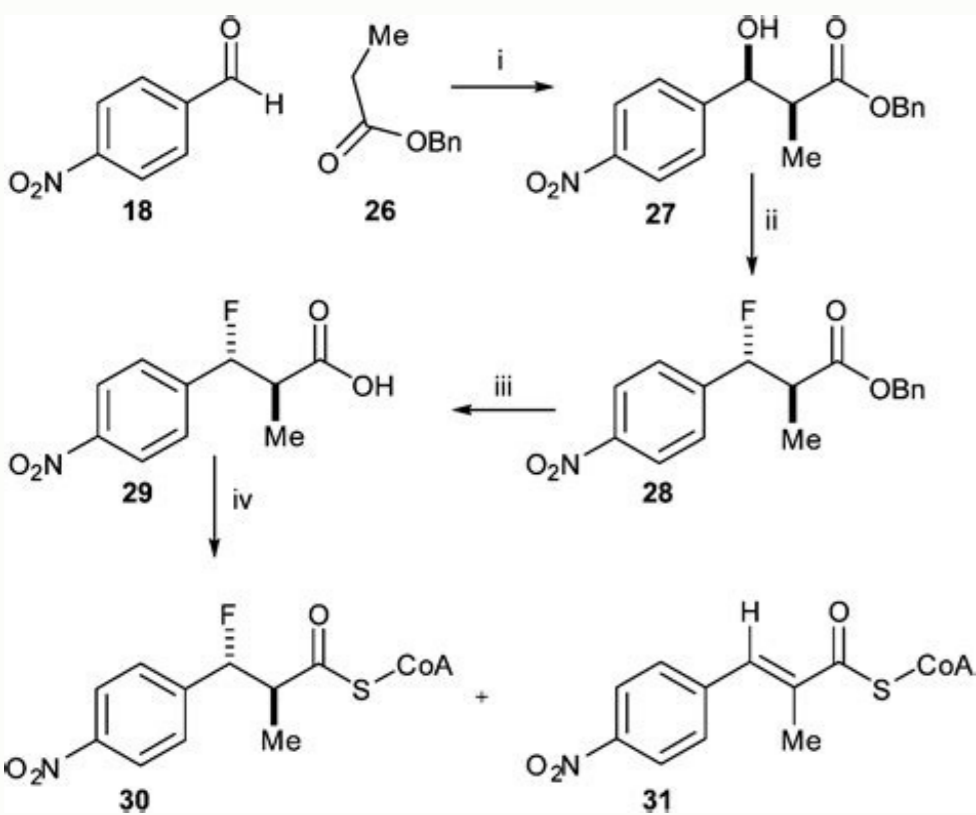
Nitro reduction zinc acetic acid. Nitro reduction to amine mechanism.

N.A. Cortesse, R.F. Heck: J. Org. Chem.,22, 3491 (1977);ibid. N.A. Cortesse, R.F. Heck: J. Org. Chem.,43 3985 (1978). Google Scholar C. Jimenez, J.M. Marinas, R. Pèrez-Ossorioy, J.V. Sinisterra: An. Quim.,73, II 64 (1977). Google Scholar Comunicación Presentada al VII Simposio Iberoamericano de Catalisis. La Plata (Argentina) Julio, 1980.E.E. Gonzo, M. Boudart: J. Catal.,52, 462 (1978). Google Scholar A. Bar Ilan, J. Manassen: Proceedings Vth Int. Congress of Catalysis, Vol 2. p. II 49. Elsevier, Amsterdam, 1973. Google Scholar Page 2 Citation counts are provided from Web of Science and CrossRef. The counts may vary by service, and are reliant on the availability of their data. Counts

will update daily once available. N-doped metal materials with enhanced stability and abundant porosity have attracted tremendous attention in catalytic reactions. Herein, a simple solvothermal approach was demonstrated to significantly enlarge the pore dimension of conventional microporous zeolitic imidazolate framework (ZIF) incorporated with two kinds of central metals (Co, Zn), while maintaining the original ZIF crystal morphology. Upon further pyrolysis, the resulting mesoporous Co-Zn/N-C material could possess the highly dispersed metal particle on the N-doped carbon, with satisfactory pore volume and surface area. The partial vaporization of Zn and the stabilizing effect of N, illustrated by XRD, HRTEM, HAADF-STEM with mapping, SEM, Raman Spectrum, BET, and TGA, were able to remarkably increase the accessibility of substrate toward active sites and prevent the aggregation of metal particles, respectively. Under mild reaction conditions, the N-stabilized Co-Zn/N-C exhibited good activity and selectivity in transfer hydrogenation of various nitro compounds to corresponding amines, where a synergistic role among Co, Zn, and N was responsible for its superior performance to other tested catalysts. In addition, the N-doped non-noble metal/carbon heterogeneous catalyst was fairly stable and could be reused several times without obvious deactivation.Keywords: mesoporous bimetallic material, N-doped carbon, transfer hydrogenation, nitrogen-containing compounds, heterogeneous catalystWith the progress of modern society, the increasing requirement of fine chemicals and other important materials such as dyes, pigments, food additives, pharmaceuticals, and herbicides have brought a series of problems, demanding prompt solution from scientists around the world (Li et al., 2018; Sudarsanam et al., 2019). Especially, aromatic or aliphatic amines are among the most significant fundamental molecules in these chemical industries (Zhou et al., 2017; Li et al., 2019). Generally, amines are produced by catalytic hydrogenation or transfer hydrogenation of nitro compounds in the presence of heterogeneous noble metal catalysts, which have drawbacks in price and reserve with complex preparation methods (Sudarsanam et al., 2018; Yuan et al., 2018). In addition, the utilization of gaseous H2 will give rise to a number of problems such as safety and transportation problems (Zeynizadeh et al., 2016; Formenti et al., 2019). Therefore, there are two urgent issues need to be solved: (1) seeking alternatives to gaseous hydrogen sources. (2) A simple and low-cost approach to prepare the catalyst featured with high efficiency and high stability for transfer hydrogenation of nitro compounds.Formic acid (FA), derivable from biomass as well as CO2 reduction, is recyclable and sustainable, and has attracted increasing concerns in the hydrogenation of nitro compounds. The utilization of formic acid as hydrogen source seemed to be a good solution to solve the above-mentioned problems caused by H2 (Zhou and Zhang, 2017; Zhou et al., 2017; Du et al., 2018; Yuan et al., 2018), while it should be pointed out that the used catalyst must be stable enough to resist the acidic formic acid (Li et al., 2016). In this regard, it is highly desirable to design an efficient while robust catalyst to meet the above requirements.Zeolitic imidazolate frameworks (ZIFs) are a branch of metal-organic frameworks (MOFs) materials in possession of carbon, nitrogen and transition metals with a highly porous structure (Salunkhe et al., 2016). The metal ions and imidazolate strongly coordinate with each other to keep the structural integrity of ZIFs, even being used in solvents. Moreover, the preparation process of this type of materials is very simple and inexpensive (Chen et al., 2015; Yang et al., 2015; Wang et al., 2016; Park et al., 2019). It was reported that the introduction of secondary metal nodes into the ZIF framework can further improve the catalyst performance in various fields (Yang et al., 2015; Yan et al., 2017; Bai et al., 2018; Wang et al., 2018), due to the formation of more defects in the catalyst framework or surface (Chen et al., 2018). Also, close contact between two metals can produce excellent synergistic effects and thus improve their inherent properties. However, it is rarely reported the use of bimetallic catalysts for the selective reduction of nitro compounds using formic acid as a hydrogen source.Herein, a simple solvothermal method was utilized to prepare the Co-Zn-ZIF material, followed by high-temperature pyrolysis in N2 to get the mesoporous Co-Zn/N-C catalyst, which was proved to be highly efficient for hydrogenation of nitrobenzene to aniline using formic acid as a hydrogen donor. This developed solid catalyst was widely applicable to a wide range of nitro compounds while remained good stability and recyclability.All the used nitro and amino compounds, methanol (MeOH, 99%), ethanol (EtOH, 99%), tetrahydrofuran (THF, 99%), acetonitrile (CH3CN, 99%), and 1,2-dichloroethane (CH2ClCH2Cl, 99%), cobalt nitrate (98%), zinc nitrate (98%), copper nitrate (98%), nickel nitrate (98%), and 2-methylimidazole (98%) were purchased from Beijing Innochem Technology Co., Ltd. Acetonitrile (MeCN, 99%), dichloromethane (DCM, 99%), and xylene (99%) were purchased from Sigma-Aldrich Co. LLC. N,N'-Dimethylformamide (DMF, 99%) and dimethyl sulfoxide (DMSO, 99%) were purchased from Tianjin Kermel Co., Ltd.The ZIF analogous materials (Co-Zn/N-C-T, T denotes pyrolysis temperature of 600, 700, and 800°C) were prepared by using a solvothermal and pyrolysis method. With a general procedure, 0.5 mmol cobalt nitrate and 0.5 mmol zinc nitrate were dissolved into 40 mL ethanol in a 100 mL beaker, and the resulting mixture was labeled as solution A (magenta solution). Then, 8 mmol 2-methylimidazole with equivalent triethylamine was dissolved into 20 mL ethanol contained in a 150 mL round-bottom flask, marked as solution B (transparent solution). Solution A was dropwise added into solution B and stirred at 25°C for 0.5 h, and the purple precipitate was observed as A was added into B. Co-Zn-ZIF was obtained after centrifugation, washing with ethanol to neutral to ensure that triethylamine is moved away, then drying at 80°C overnight. Then, the resulting solid was subjected to pyrolysis at the specific temperature (800, 700, or 600°C) for 3 h with the heating ramp of 5°C/min in a nitrogen atmosphere. Upon cooling down to room temperature, the magnetic black powder Co-Zn/N-C-T was collected. For comparison, Co/N-C-T and Zn/N-C-T were also prepared via the same synthetic procedures by only changing the bimetal to single metal nitrate (1 mmol cobalt nitrate or zinc nitrate).The employed nitrogen tube muffle furnace (OTF-1200X-50) was bought from Hefei Kejing materials Technology Co., Ltd. TG analysis manifests that the pyrolysis temperature of ≥ 600°C is enough for carbonization of Co-Zn-ZIF under pyrolysis conditions (Figure S1).The Micromeritics ASAP 2010 instrument (Tristar II 3020, Norcross, GA) was used to test BET surface areas of the as-prepared porous catalysts by nitrogen physisorption measurements at 77 K. An aberration-corrected FEI Tecnai G2 F20 S-TWIN (S) TEM (Hillsboro, OR) operating at 300 kV with the energy dispersive X-ray (EDX) spectra were used to get STEM-HAADF imaging. The Physical Electronics Quantum 2000 Scanning ESCA Microprobe (Physical Electronics Inc., PHI, MN) equipped with a monochromatic Al Kα anode was used to measure the XPS (X-ray photoelectron spectroscopy) of the as-prepared catalysts. D/max-TTR III X-ray powder diffractometer (Rigaku International Corp., Tokyo) using Cu Kα radiation source was utilized to test the XRD (X-ray diffraction) patterns of different catalysts. The Renishaw RM2000 was employed to collect Raman spectra at room temperature from 100 to 3,000 cm−1 with 532 nm argon ion laser. Inductively coupled plasma optical emission spectroscopy (ICP-OES) analysis was conducted using a PerkinElmer Optima 8000 instrument. The thermal gravimetric (TG) measurements were conducted by Shimadzu DTG-60AH differential thermal analyzer.All the catalytic transfer hydrogenation reactions were carried out in 15 mL max schlenk tube. Firstly, 0.2 mmol nitro compound, 20 mg Co-Zn/N-C (5 mol% Co), 2.0 mL THF, and 6 equiv. HCOOH were added into the tube and sealed. Then, the tube was transferred into an oil bath preheated to the desired reaction temperature and magnetically stirred at 600 rpm for specific reaction time. The time was recorded as soon as the tube was placed into the oil bath. When the reaction time was up to the required time, the tube was taken out and cooled down to ambient temperature using flow tap-water. Then, 3 mL THF and 10 mg naphthalene as internal standard were added into the reaction mixture.Upon the completion of the reaction, the filter membrane was used to remove solid particles from the liquid solution, followed by quantitative analysis with gas chromatography (GC Agilent 7890B with an HP-5 column (30 m × 0.320 mm × 0.25 μm) and FID). The quantification of the obtained products was conducted by referring to the standard curves (R2 > 0.99) made from commercial samples. The product structures were identified by gas chromatography-mass spectrometry (GC-MS Agilent 6890N GC/5973 MS). Then, nitrobenzene conversion (C, %) and aniline yield (Y, %) for the model reaction were calculated according to the below equations:C =

[1−mole concentration of nitrobenzene in productmole concentration of initial nitrobenzene] ×100%   Y =[mole concentration of anilinemole concentration of initial nitrobenzene] ×100%The nitrogen adsorption-desorption isotherms and pore distribution of Co-N-C-800 and CoZn-N-C-800 are shown in Figure 1. It can be clearly found that all the catalysts exhibit mesoporous structures in consideration of type-IV curve and H4-type hysteresis loop at the relative pressure P/P0 of 0.4–1.0. Among them, the Co-Zn/N-C-800 sample reveals relatively large volume (average pore diameter 12.7 nm), and the pore distribution also shows that the pore width of Co-Zn/N-C-800 is mainly located in the range of 0–1.5 nm and 2–50 nm (Figure S2). Meanwhile, the BET surface area of Co-Zn/N-C-800 is 411.3 m2/g, which is larger than that of Co/N-C-800 (257.1 m2/g). It is expected that cobalt can be reduced while zinc is partially evaporated to generate additional holes in the pyrolysis process at 800°C and N2 flow, thus increasing the surface area.To more intuitively understand the catalyst morphology, SEM, TEM, and STEM-HAADF were utilized to characterize Co/N-C-800 and Co-Zn/N-C-800 (Figures 2a–f). Both materials have the tubular graphene structure and the metal particles are fully dispersed on the carbon carrier, which is consistent with a previous report. The metal nanocatalysts would be generated from the reduction of the metal ions/clusters with reducing gases (e.g., CO) in situ generated from the pyrolysis of organic species, and these metal nanocatalysts were capable of further catalyzing the organic units to form the graphene structures and N-doped carbon nanotubes (Chen et al., 2008; Xia et al., 2016; Gong et al., 2019). The formed graphene structure can effectively enhance the resistance of the catalyst to acid, thus possibly improving its catalytic activity. The lattice fringe images of the catalyst show the formation of graphitic carbon C (002) plane with 0.36 nm and Co (111) plane with 0.21 nm (Figure 2g). Also, the elemental mapping of Co-Zn/N-C-800 clearly states the well-proportioned distribution of Co, N, and C at the nanoscale (Figures 2h-l) (Co: yellow, C: red, Zn: blue, N: orange).SEM images of Co-Zn/N-C-800 (a,b), TEM images of for Co/N-C-800 (c), and Co-Zn/N-C-800 (d,e), HRTEM images of Co particle and C graphic of Co-Zn/N-C-800 (f,g), STEM-HAADF image (h), and Co: yellow (i), C: red (j), Zn: blue (k), and N: orange (l) mappings of Co-Zn/N-C-800.The Co/N-C-800 and Co-Zn/N-C-800 catalysts were further characterized by XPS, and the obtained high-resolution XPS spectra of Co2p of Co/N-C-800, fresh Co-Zn/N-C-800, and reused Co-Zn/N-C-800 are shown in Figure 3. Metallic Co (green line), Co-O (magenta line) and Co-Nx (yellow line) were detected, and located at 778.4, 781.7, and 780.0 eV, respectively (Park et al., 2019). Compare to that of Co/N-C-800, a negative CO2p peak shift in the fresh and reused Co-Zn/N-C-800 catalysts could be observed, possibly due to the incorporation of Zn species (Wu et al., 2018). Based on the integral area of Co in different valence states, the percentage composition of the metal Co content is also listed in this figure. No significant difference in the metallic Co content between Co/N-C-800 (34.8%) and Co-Zn/N-C-800 (32.4%). These results demonstrate the slight interaction between the metal species of Co and the nitrogen species, including pyridine N 398.5 eV (deep green line) pyrrolic N 400.1 eV and graphic N 401.1 eV (yellow line) that probably combined with metallic Co and fixed in the catalyst structure (Figures 3D–F), which is well supported by the XPS spectra of Co2p (Figures 3A-C). The presence of graphic N can find a clue from the formed graphene structure, as verified by TEM (Figure 2). Also, the XPS spectra of C1s (shown in Figure S3) show three peaks at 284.6, 285.8, and 288.6 eV, illustrating the existence of C = C/C-C, C-N, and N = C-N bonds in the materials, respectively (Shen et al., 2017; Zhao et al., 2017). In addition, the existence of the Zn element in fresh Co-Zn/N-C-800 and even the one reused for five times was affirmed by the XPS spectra of Zn2p peak (Figure 4), which is consistent with ICP analysis (the atomic ratio of Zn2p, can 20% Zn left after pyrolysis).XRD patterns (Figure 5) show a significant peak at 25.3 degree (C 002) in those three materials (fresh Co-Zn/N-C-800, reused Co-Zn/N-C-800 and Co/N-C-800), which is assigned to the graphitized carbon, while the peaks at 44.3° and 51.4° belong to Co (111) and Co (002) respectively, which is strongly agreeing with the characterization results of TEM and XPS.Raman spectra of those three samples are provided in Figure 6, and the D and G peaks are located at 1,345 cm−1 and 1,570cm−1, respectively. The relative peak intensity of D and G (ID/IG) represents the degree of the graphitization of carbon materials (Wang et al., 2017). It can be seen that the ID/IG ratios of reused Co-Zn/N-C-800, fresh Co-Zn/N-C-800, and Co/N-C-800 are 1.149, 1.12 and 1.08, respectively, suggesting that Co-Zn/N-C-800 without the inclusion of Zn has a higher degree of graphitization than both fresh and reused Co-Zn/N-C-800 catalysts. That is to say, the fresh Co-Zn/N-C-800 has more defects compared to the Co/N-C-800. In addition, the reused Co-Zn/N-C-800 has a slightly increased value of ID/IG ratios, as compared with the fresh counterpart. It is indicated that the structural defects of the catalyst increase after recycle.The influence of different catalysts on transfer hydrogenation of nitrobenzene is shown in Table 1. It was found that Co/N-C-800 gave 71.0% nitrobenzene conversion and 57.6% aniline yield. When Co-Zn/N-C-800 was used instead of Co/N-C-800, relatively high nitrobenzene conversion of 83.9% and 75.6% aniline yield could be achieved. This result shows that the introduction of Zn into the catalyst may enhance its productivity and selectivity. Meanwhile, the residue of Zn might contribute to the enhanced productivity, due to the synergistic effect of Zn and Co species in the hydrogen transfer process (Kong et al., 2017). In contrast, 3.2% nitrobenzene conversion and

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