Supporting Information for

Efficient Subnanometric Gold-Catalyzed Hydrogen Generation via Formic Acid Decomposition under Ambient Conditions

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1. Chemicals and materials

Formic acid (HCOOH, FA, 99 wt%), triethylamine (NEt_3, 99.5 wt%), triethanolamine ($N(CH_2CH_2OH)_3$, 99 wt%), diethanolamine ($NH(CH_2CH_2OH)_2$, 98.5 wt%), ethanolamine ($NH_2CH_2CH_2OH$, 98.5 wt%), N,N-dimethylethanolamine (($CH_3)_2NCH_2CH_2OH$, 99.5 wt%), N,N,N',N'-tetramethylethylenediamine (($CH_3)_4(NCH_2)_2$, 99.5 wt%), N,N-dimethyl-*n*-butylamine (($CH_3)_2NCH_2CH_2CH_2CH_3$, 98 wt%), ethylene glycol ($HOCH_2CH_2OH$, 99 wt%), polyvinyl alcohol (($CH_2CHOH)_n$, PVA, 99 wt%), polyvinylpyrrolidone (PVP, K30), zirconium oxychloride octahydrate ($ZrOCl_2\cdot8H_2O$, 98 wt%), sodium formate (HCOONa, 99.5 wt%), potassium formate (HCOOK, 99 wt%), ammonium formate ($HCOONH_4$, 98 wt%), palladium chloride ($PdCl_2$), rhodium chloride hydrate ($RhCl_3\cdot xH_2O$), chloroplatonic acid hexahydrate ($H_2PtCl_6\cdot6H_2O$) and chloroauric acid tetrahydrate ($HAuCl_4\cdot4H_2O$), were supplied by Alfa Aesar and used without further purification.

HCOOD (98% OD, < 5% D_2O), DCOOH (> 99.5%, 98% D), DCOOD (98% D, < 5% D_2O) were obtained from Cambridge Isotope Laboratories and used with further purification by anhydrous $CuSO_4$.

5.0 wt% Pd/C (stock # 38300), 1.0 wt% Ir/C (stock # 38330) and 5.0 wt% Ru/C (stock # 11748) were purchased from Alfa Aesar.

2. Catalyst characterization

2.1 Elemental analysis: The Au loading of the catalysts was measured by inductively coupled plasma atomic emission spectroscopy (ICP-AES) using a Thermo Electron IRIS Intrepid II XSP spectrometer.

2.2 BET analysis: The BET specific surface areas of the prepared catalysts were determined by adsorption–desorption of nitrogen at liquid nitrogen temperature, using a Micromeritics TriStar 3000 equipment. Sample degassing was carried out at 300 ^{o}C prior to acquiring the adsorption isotherm.

2.3 X-ray diffraction (XRD) analysis: The crystal structures of ZrO_2 and Au-containing zirconia samples (Au/ZrO_2 or Au/ZrO_2-NCs) were characterized with powder X-ray diffraction (XRD) on a Bruker D8 Advance X-ray diffractometer using the Ni-filtered Cu Kα radiation source at 40 kV and 40 mA. The percentage of monoclinic phase (M%) in the oxide "support" was measured according to the equation:[S1] M% = $I_{M(\bar{1}11)}/1.6I_{M(\bar{1}11)}+I_{T(111)}$, where $I_{M(\bar{1}11)}$ and $I_{T(111)}$ are the integrated intensities of the monoclinic ($\bar{1}11$) (2θ = 28.5^o) and tetragonal (111) (2θ = 30.4^o) planes, respectively.

2.4 X-ray photoelectron spectroscopy (XPS): XPS data were recorded with a Perkin Elmer PHI 5000C system

equipped with a hemispherical electron energy analyzer. The spectrometer was operated at 15 kV and 20 mA, and a magnesium anode (Mg Kα, hν = 1253.6 eV) was used. The C 1s line (284.6 eV) was used as the reference to calibrate the binding energies (BE).

2.5 Transmission electron microscopy (TEM): A JEOL 2011 microscope operating at 200 kV equipped with an EDX unit (Si(Li) detector) was used for the TEM investigations. The samples for electron microscopy were prepared by grinding and subsequent dispersing the powder in ethanol and applying a drop of very dilute suspension on carbon-coated grids.

2.6 Quantitative analysis of trace CO in the H_2-containing stream: CO concentration with very low levels can be reliably measured by using a gas chromatograph (Agilent 6890 GC) analysis system[S2] equipped with a methanizer and a flame ionization detector (FID, detection limit ~ 1.0 ppmv). Prior to the analysis, the catalyst in methanizer was reduced *in situ* by 5 vol% H_2/Ar at 400 ^{o}C for 1 h. The evolved gas was first cleaned by CarboTex (BET: 1200 m^2 g^{-1}) for organic amine-trapping and concentrated NaOH solution for CO_2-trapping and $MgSO_4$ powder for moisture-trapping, then flowing the methanizer at 300 ^{o}C under atmospheric pressure. The effluent from methanizer was again cleaned by $MgSO_4$ powder and later analyzed by the FID. For calibration purpose, the methanation data of a series of premixed reference gases containing different concentrations CO in H_2 were also collected using the similar method (see Figure S4).

2.7 Diffuse Reflectance (DR) UV–Vis measurements: DR UV-Vis spectra of the solids diluted in $BaSO_4$ were recorded at room temperature on a Shimadzu UV 2450 Spectrometer equipped with an integrating sphere and using $BaSO_4$ as reference.

2.8 Diffuse Reflectance Infrared Fourier Transform (DRIFT) measurements: The CO adsorption experiments were carried out on a Bruker Vector 22 FTIR spectrometer equipped with a MCT detector and Harrick diffuse reflectance accessory. Spectra were obtained on the apparatus loaded with 50 mg of catalyst. Prior to the saturated adsorption of 0.5 vol% CO/He at room temperature, sample was subjected to the pretreatment with Helium flow at 200 ^{o}C for removing any other gases and moisture. Each spectrum was obtained after the 10 min evacuation treatment with Helium flow at room temperature and by subtracting the background (base spectrum) of the unloaded sample.

2.9 Extended X-ray absorption fine structure (EXAFS): The X-ray absorption data at the Ir L_3-edge of the samples were recorded at room temperature in transmission mode using ion chambers or in the fluorescent mode

with silicon drift fluorescence detector at beam line BL14W1 of the Shanghai Synchrotron Radiation Facility (SSRF), China. The station was operated with a Si (311) double crystal monochromator. During the measurement, the synchrotron was operated at energy of 3.5 GeV and a current between 150–210 mA. The photon energy was calibrated with standard Pt metal foil. Data processing was performed using the program ATHENA. All fits to the EXAFS data were performed using the program ARTEMIS.

2.10 CO chemisorption measurements: CO chemisorption measurements were performed at −116 $^\circ$C.[S3] A home-made equipment was used for pulse chemisorption. In principle the system was made of a U-shaped quartz reactor equipped with an oven controlled by a PID temperature programmer, mass flow meters, sampling valve, a quadrupole mass detector. Before chemisorption the following standard pretreatment procedure was applied: the sample (200 mg) was reduced in H_2 flow (40 mL min^{-1}) at 250 $^\circ$C for 30 min, cooled in H_2 to ambient temperature, purged in He flow and finally hydrated at ambient temperature. The hydration treatment was performed by contacting the sample with a He flow (10 mL min^{-1}) saturated with water. The sample was then cooled in He flow to −116 $^\circ$C (attained and maintained by an ethanol-liquid nitrogen cryogenic mixture in a Dewar flask) for CO chemisorption. For standard measurements the following operating conditions are recommended: CO content in pulses (5 vol%), helium flow rate (30 mL min^{-1}), pulse size (0.5 mL), time interval between pulses (4 min).

3. Catalyst preparation

3.1 Preparation of 0.5 wt% Au/TiO$_2$ catalyst: 0.5 wt% Au/TiO$_2$ catalyst was prepared by a modified DP method as introduced by He et al.[S4] Briefly, an appropriate amount of TiO$_2$ (Evonik, P25, specific surface area: 45 m^2 g^{-1}, 70% anatase and 30% rutile) was added to the aqueous solution of HAuCl$_4$ (0.5 mM) at a fixed pH (8.0) adjusted with NaOH (0.2 M). The mixture was aged for 2 h at 80 $^\circ$C under stirring, after which the suspension was cooled to 25 $^\circ$C. Extensive washing with deionized water was followed until it was free of chloride ions. The sample was dried under vacuum at room temperature for 12 h before reduction in 5 vol% H_2/Ar at 300 $^\circ$C for 2 h.

3.2 Preparation of the Au/TiO$_2$-NCs catalyst: In analogy to the case with Au/ZrO$_2$-NCs, the Au/TiO$_2$-NCs was prepared by decreasing the initial concentration of the HAuCl$_4$ stock solution, decreasing the overall Au content in the final catalyst and employing a milder thermal activation conditions. Briefly, a suitable amount of TiO$_2$ (P25) was added to the aqueous solution of HAuCl$_4$ (0.125 mM) at a fixed pH (8.0) adjusted with NaOH (0.1 M). The mixture was allowed to age for 6–8 h at room temperature under stirring, after which the suspension was washed extensively with deionized water until it was free of chloride ions. The sample was dried under vacuum at 25 $^\circ$C for 12 h before reduction in 5 vol% H_2/Ar at 250 $^\circ$C for 2 h. The final gold loading was about 0.3 wt% as

determined by ICP-AES. As disclosed by TEM (Figure S12e), the Au/TiO$_2$-NCs sample contains very fine gold NPs whose mean particle size was ca. 1.1 nm.

3.3 Preparation of 1.0 wt% Au/SiO$_2$ catalyst: 1.0 wt% Au/SiO$_2$ catalyst was prepared by following the modified DP procedure as described above. Briefly, an appropriate amount of SiO$_2$ (Evonik, Aerosil 380, specific surface area: 380 m^2 g^{-1}) was introduced into an aqueous solution of HAuCl$_4$. After stirring at 75 $^{\circ}$C for 1 h the solution was cooled to 25 $^{\circ}$C and the solution pH was brought to 8.5 by adding 0.25 M NH$_4$OH under stirring. After 6 h stirring at 25 $^{\circ}$C the catalyst was washed extensively with deionized water until it was free of chloride ions. The sample was dried at 110 $^{\circ}$C in air for 1 h and reduced in flowing 5 vol% H$_2$/Ar at 350 $^{\circ}$C for 2 h.

3.4 Preparation of 0.6 wt% Au/Al$_2$O$_3$ catalyst: 0.6 wt% Au/Al$_2$O$_3$ catalyst was prepared by the DP procedure as described by Iglesia et al.[S5] To obtain the Au/Al$_2$O$_3$ catalyst, the pH of the aqueous solution of HAuCl$_4$ was brought to 7.0 by adding NaOH (0.5 M), an appropriate amount of the support (Aluminum oxide C, Evonik) was added to the neutralized solution, and the resulting suspension was stirred at 80 $^{\circ}$C for 1 h. The catalyst precursor was separated from the mother liquor, washed with deionized water, dried in air for 1 day, and calcined in 25 vol% O$_2$/Ar at 300 $^{\circ}$C for 2 h.

3.5 Preparation of 1.0 wt% Au/C catalyst: Au/C catalyst was prepared by the procedure as described elsewhere.[S6] Typically, the PVA was added (Au/PVA = 1.5:1, wt/wt) to a aqueous solution containing 0.5 mM HAuCl$_4$ at 25 $^{\circ}$C under vigorous stirring. The obtained solution was then left under stirring for 10 min. A following rapid injection of an aqueous solution of 0.1 M NaBH$_4$ (Au/NaBH$_4$ = 1:5, mol/mol), led to formation of a dark orange-brown solution, indicating the formation of the gold sol. Activated carbon (refluxed with 2.5 wt % HNO$_3$ solution at 93 $^{\circ}$C for 6 h) was then added to the colloidal gold solution under stirring and kept in contact until total adsorption (1 wt% of gold on the support) occurred. After 2 h stirring, the slurry was filtered and the resulting sample was dried at 100 $^{\circ}$C in air for 4 h.

3.6 Preparation of 2.0 wt% Pd/ZrO$_2$, 1.0 wt% Pt/ZrO$_2$, and 1.0 wt% Rh/ZrO$_2$ catalysts: All these catalysts were prepared using the routine incipient wetness technique. Appropriate amount of ZrO$_2$ (1.0 g as prepared ZrO$_2$ powdered sample) was added to the aqueous solution of PdCl$_2$ or H$_2$PtCl$_6$·6H$_2$O or RhCl$_3$·xH$_2$O. After a perfect mixing of the corresponding slurries and static standing for 12 h, the samples were dried at 100 $^{\circ}$C for 12 h, followed by reduction in 5 vol% H$_2$/Ar at 400 $^{\circ}$C for 2 h.

3.7 Preparation of Ag@Pd/C catalyst: The Ag@Pd/C (10 wt% metal, n_{Ag}:n_{Pd} = 1:1) catalyst was prepared with

the method as introduced by Tedsree et al.[S7] Ag@Pd (n_{Ag}:n_{Pd} = 1:1) NPs was prepared by sequential reductions with ethylene glycol. Ag NPs were initially prepared by the above method, and Pd salts were then reduced in presence of Ag seed nanoparticles in the sequential reduction with ethylene glycol under stirring. Activated carbon (0.2 g) was first dispersed in 100 mL of ethanol under sonication for 1 h. A sample containing PVP-stabilized Ag@Pd particles, with a metal loading of 10 wt%, was added dropwise onto the dispersed carbon in ethanol under sonication. The resulting mixture was stirred overnight and then filtered. The obtained nanoparticles were washed extensively with deionized water and ethanol to remove any residual chloride ions and PVP and dried at 80 ^{o}C under a N_2 atmosphere.

3.8 Preparation of Pd-Au/C catalyst: The Pd-Au/C (20 wt% Pd, n_{Pd}:n_{Au} = 3:1) catalyst was prepared following the method as developed by Zhou et al.[S8] Activated carbon was dispersed into 300 mL ethanol and water (1:1, V/V). Then the calculated amount of Pd ($PdCl_2$) was added into the suspension. 2 h later, specific atom proportion of Au ($HAuCl_4 \cdot 4H_2O$) was added. After 2 h stirring, the suspension was reduced by 0.4 g $NaBH_4$ diluted with 100 mL water. The result production was filtered, washed and dried at 80 ^{o}C, after 4 h stirring. At last, the catalyst was triturated to be ready to use.

Table S1. Activity of Au/ZrO$_2$ catalyst for dehydrogenation of formic acid[a]

Entry	Base	Temp. (°C)	V$_{gas}$ (mL)	TOF (h^{-1})[b]	TON[c]
1	Base-free	80	1212	550	402 (818)
2	Base-free	40	40	15	11 (27)
3	HCOONa	40	226	77	68 (153)
4	HCOOK	40	190	63	56 (129)
5	HCOONH$_4$	40	118	39	33 (80)

[a] Reaction conditions: 5.0 mL scale of solution, 53.0 mmol FA, 30.0 µmol Au, inorganic bases were 5.9 mmol in entries 3–5, reaction time 3 h. [b] Initial TOF after 20 min. [c] TON for 1 h, numbers in parenthesis refer to TON for 3 h.
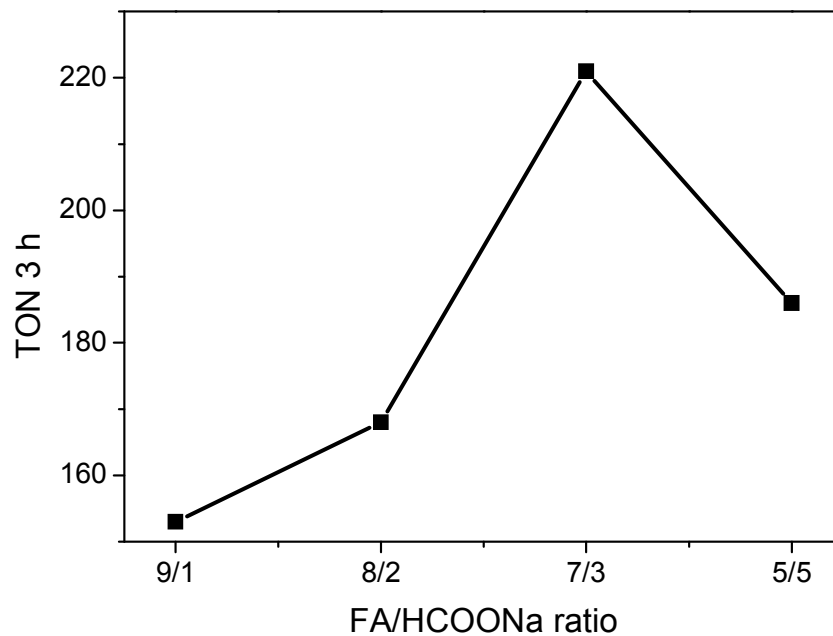
Figure S1. TON as a function of the ratio of FA/HCOONa (11.8 M) using the Au/ZrO$_2$ catalyst. Reaction conditions: 5.0 mL scale of initial solution, 30.0 µmol Au, 40 °C, reaction time 3 h.
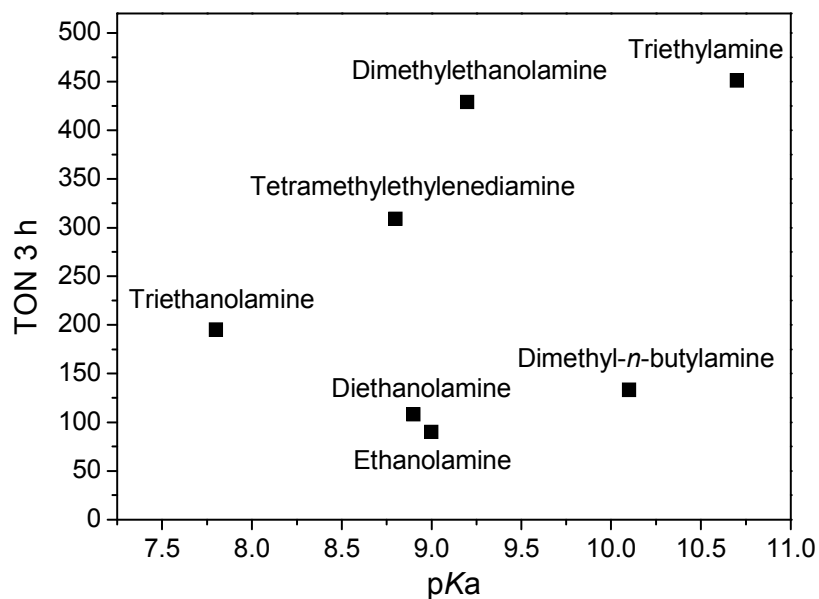
Figure S2. The influence of different N-containing compounds on gas evolution from a 5:2 FA-amine system using the Au/ZrO$_2$ catalyst. Reaction conditions: 5.0 mL scale of adducts, n(FA)/n(Au) = 1750, 40 ^{o}C, reaction time 3 h. Note that there is only a weak relationship between the reaction rate and the pKa value of the amines, indicating that amine basicity is not the key factor determining the catalytic performance of the present Au-catalyzed formic acid decomposition process.
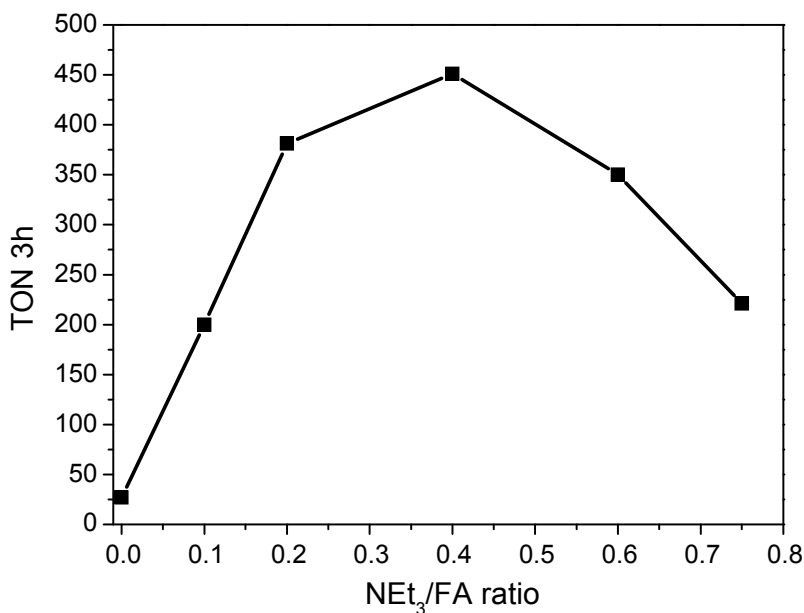
Figure S3. TON as a function of the ratio of FA/NEt$_3$ adducts using the Au/ZrO$_2$ catalyst. Reaction conditions: 5.0 mL scale of adducts, n(FA)/n(Au) = 1750, 40 ^{o}C, reaction time 3 h.
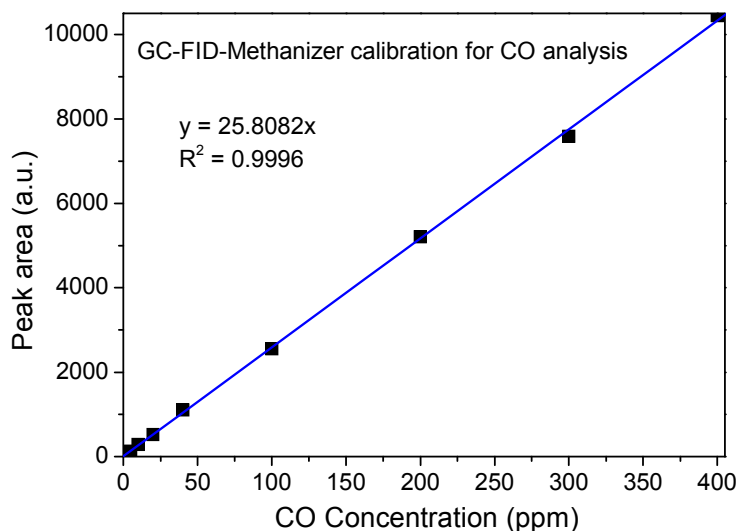
Figure S4. Calibration curve for CO quantitative analysis using the GC-FID-Methanizer.

Table S2. Activity of various catalysts for dehydrogenation of formic acid[a]

Entry	Catalyst	D_{NP} (nm)	V_{gas} (mL)	CO (ppm)[b]	TOF (h^{-1})[c]	TON[d]
1	ZrO_2	—	0	—	—	—
2	Pd/ZrO_2	2.8	0	—	—	—
3	Pt/ZrO_2	2.4	0	—	—	—
4	Rh/ZrO_2	2.5	0	—	—	—
5	Ru/C	2.4	0	—	—	—
6	Ir/C	2.6	0	—	—	—
7	Pd/C	2.5	10	—	20	7 (7)
8	Au/ZrO_2-NCs	0.8	2380	—	912	741 (1607)
9[e]	Au/ZrO_2-NCs	0.8	2374	—	908	733 (1602)
10[f]	Au/ZrO_2-NCs	0.8	553	4	1593	1318 (2990)

[a] Reaction conditions: 5.0 mL scale of adducts (53.0 mmol FA, 21.2 mmol NEt_3), 30.0 μmol metal, 40 °C, reaction time 3 h. [b] The CO concentration detected in gas stream after CO_2 removal. Apart from the case in entry 10, in all experiments no detectable CO was found in the evolved gaseous mixture. [c] Initial TOF after 20 min. [d] TON for 1 h, numbers in parenthesis refer to TON for 3 h. [e] Under inert atmosphere of N_2. [f] 3.75 μmol Au, reaction at 50 °C. Note that only 4 ppm of CO was detected in this CO_2-removed gas stream, showing great potential for fuel cell applications.
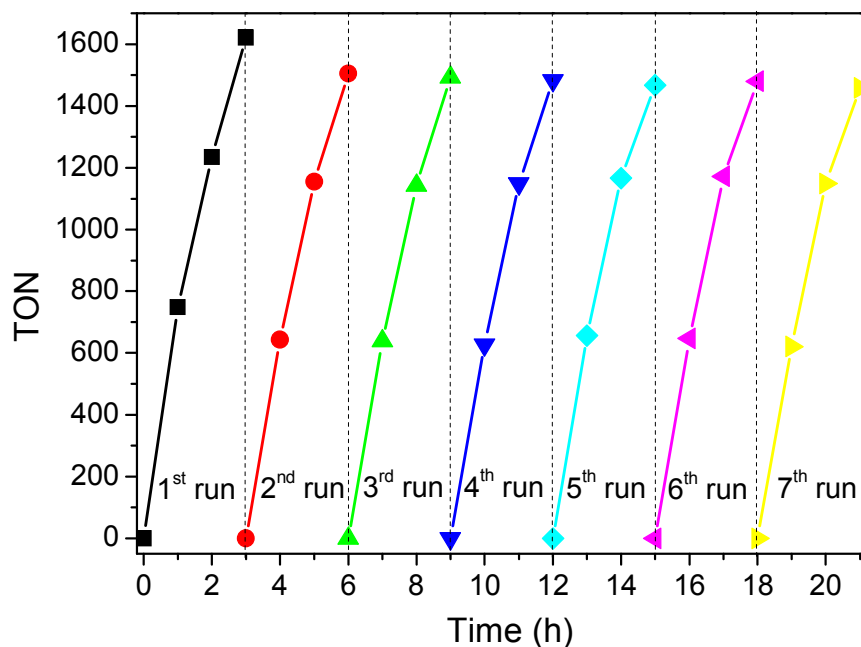
Figure S5. Reuse of the Au/ZrO$_2$-NCs catalyst for hydrogen generation via decomposition of formic acid. Reaction conditions: 5.0 mL scale of adducts (53.0 mmol FA, 21.2 mmol NEt$_3$), 3.75 μmol Au, 40 °C. Note that the gas production rate of the 2nd run was only marginally lower than the fresh catalyst in the 1st run and kept a constant value in the later runs.
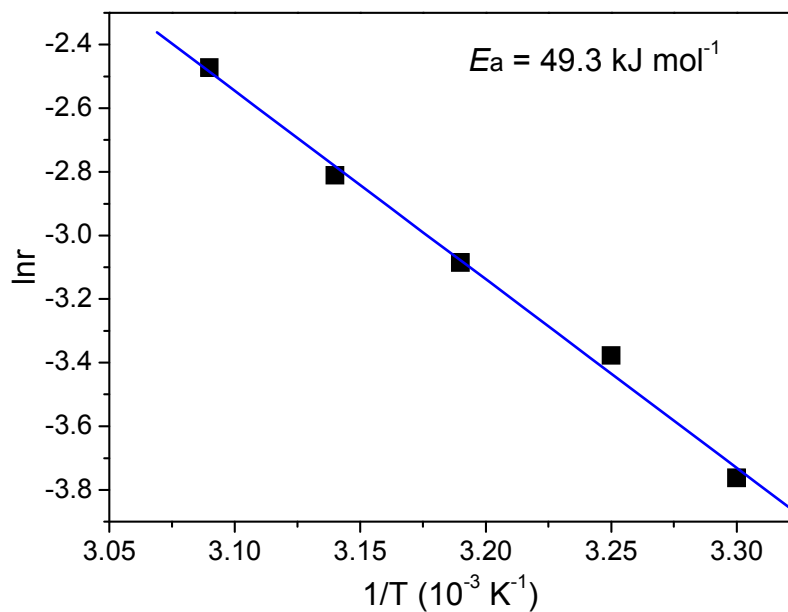

Figure S6. Arrhenius plot for Au/ZrO$_2$-NCs catalyzed hydrogen evolution from the TEAF system. Reaction conditions: 5.0 mL scale of adducts (53.0 mmol FA, 21.2 mmol NEt$_3$), 3.75 μmol Au.
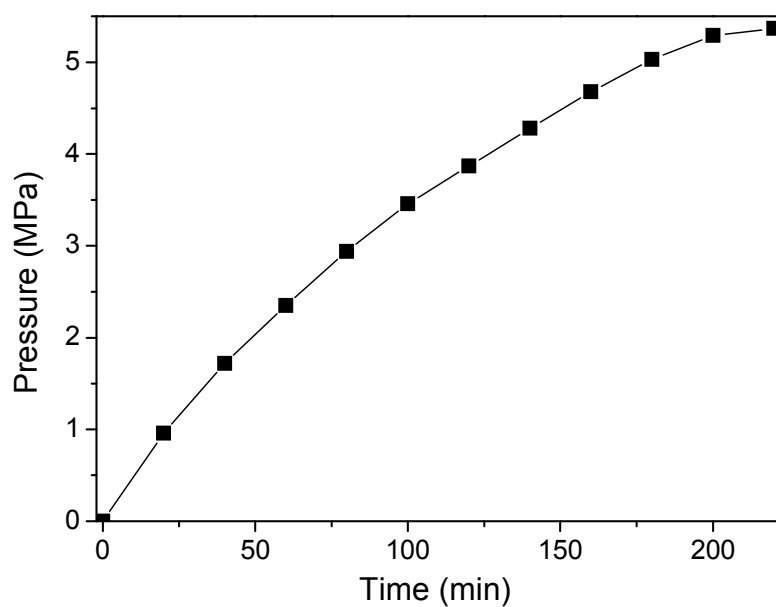
Figure S7. Pressure-time profile for FA decomposition using Au/ZrO$_2$-NCs catalyst in a closed system. Reaction conditions: 5.0 mL scale of adducts (53.0 mmol FA, 21.2 mmol NEt$_3$), 30.0 μmol Au, 40 ^{o}C.
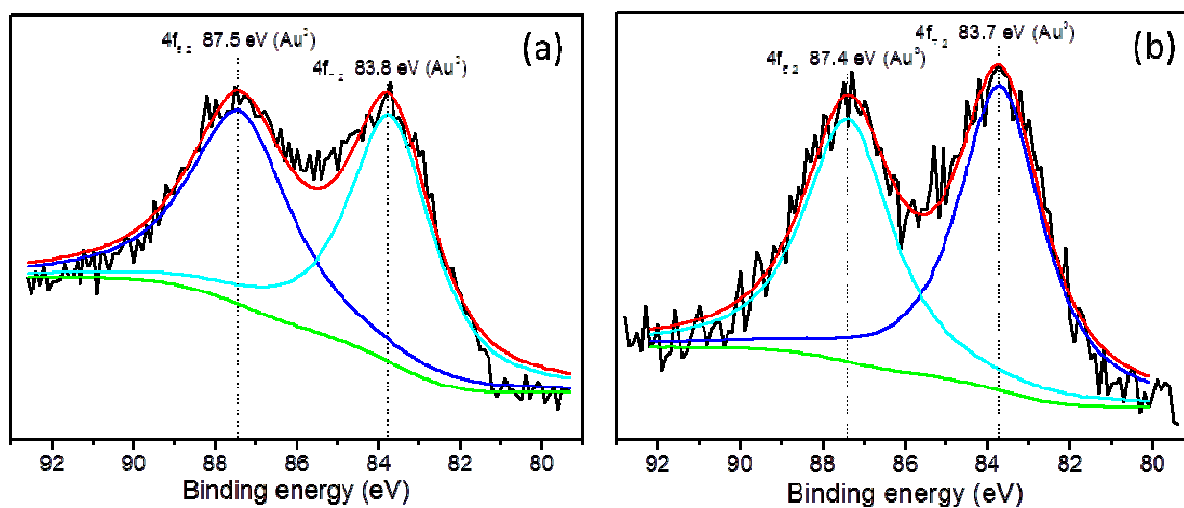
Figure S8. The binding energy of Au^0 for (a) fresh and (b) used Au/ZrO$_2$-NCs catalysts.

Table S3. Changes of surface atomic ratio of Au/Zr according to the XPS peaks (Au $4f_{7/2}$; Zr $3d_{5/2}$) of Au/ZrO$_2$ and Au/ZrO$_2$-NCs catalysts.

Catalyst	Bulk n_{Au}/n_{Zr} [a]	Surface n_{Au}/n_{Zr} [b]
Au/ZrO$_2$	0.005	0.013
Fresh Au/ZrO$_2$-NCs	0.005	0.026
Used Au/ZrO$_2$-NCs	0.005	0.025

[a] The bulk Au/Zr atomic ratio was determined from ICP-AES. [b] Surface $n_{Au}/n_{Zr} = (A_{Au}/S_{Au})/(A_{Zr}/S_{Zr})$, A is peak area, atomic sensitivity factor $S_{Au} = 2.8$ and $S_{Zr} = 1.3$.
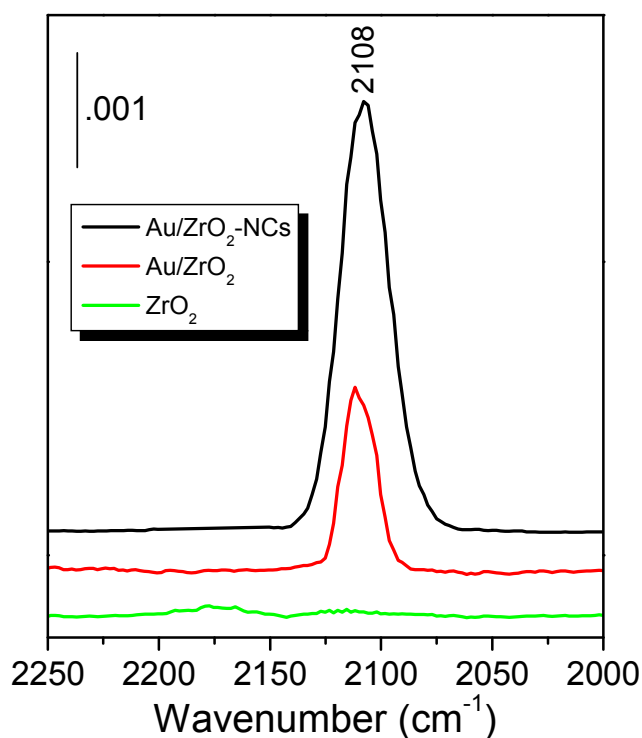
Figure S9. DRIFT spectra of CO adsorbed at room temperature for Au/ZrO$_2$-NCs, Au/ZrO$_2$, and ZrO$_2$. Noted that there is only single peak of CO adsorption on gold catalysts at 2108 cm^{-1} attributed to the vibration of CO adsorbed on Au^0 species.[S9]
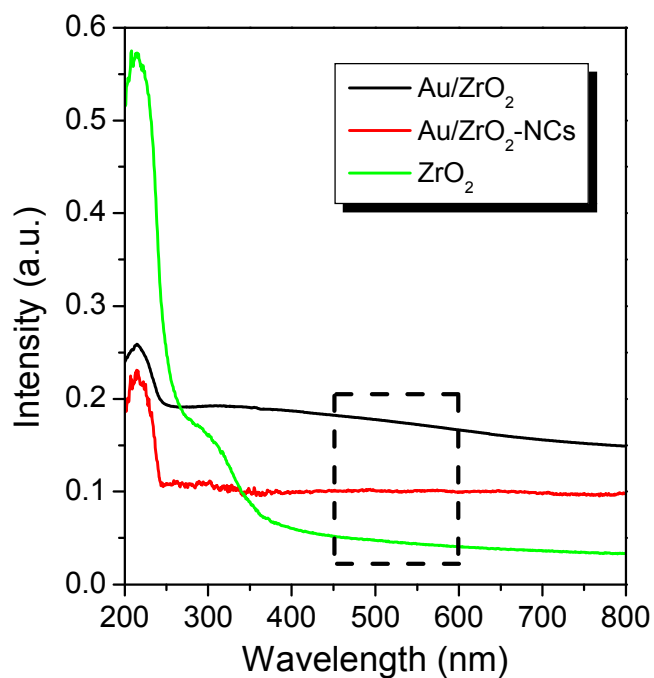
**Figure S10.** Diffuse reflectance UV–Vis spectra of ZrO_2 support, Au/ZrO_2 and Au/ZrO_2-NCs catalysts.
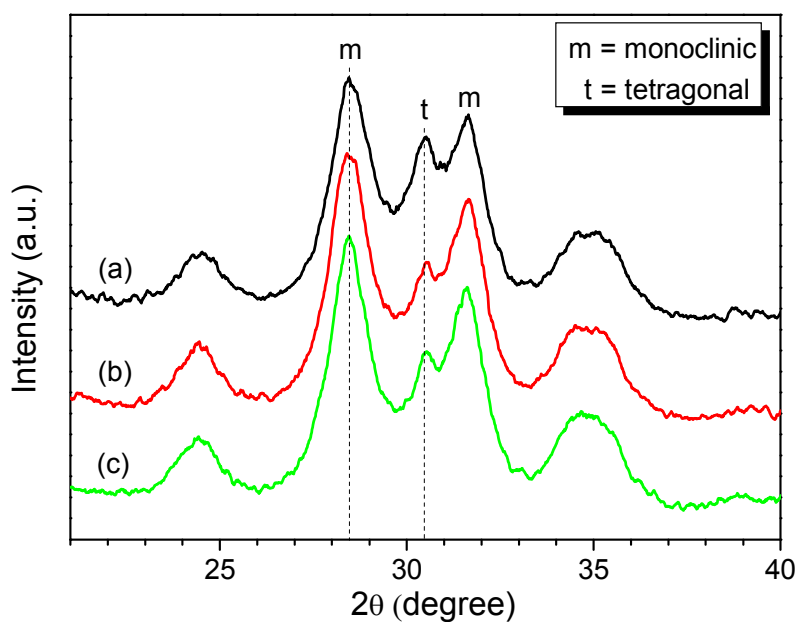
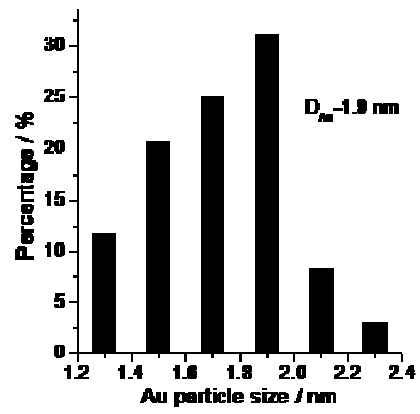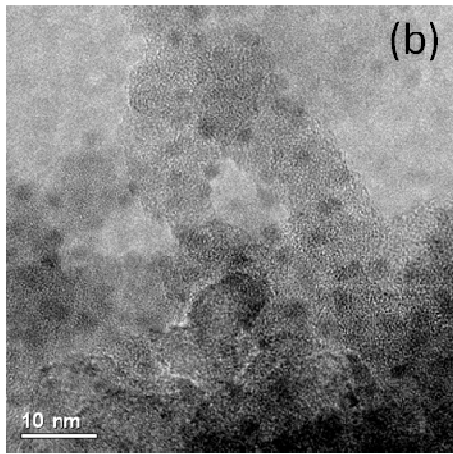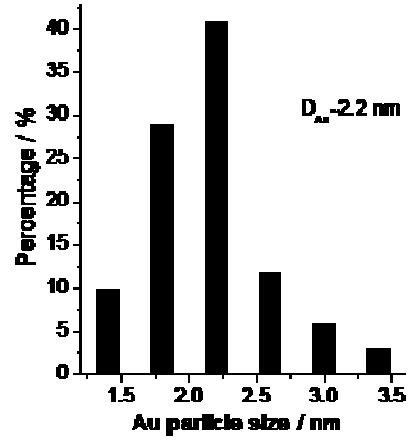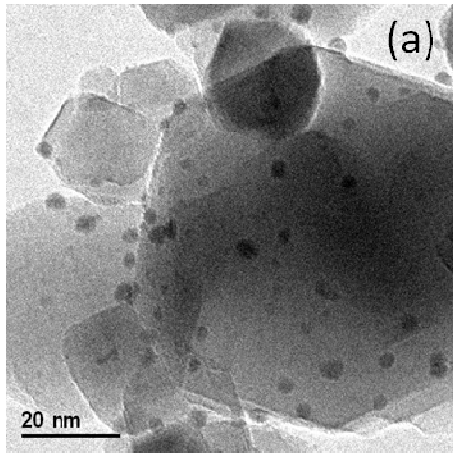**Figure S11.** XRD patterns of (a) ZrO_2, (b) fresh and (c) used Au/ZrO_2-NCs. Note that the deposition of gold resulted in a slight phase transformation of ZrO_2 from tetragonal to monoclinic phase in the Au/ZrO_2-NCs catalyst and there was no significant change between fresh and used Au/ZrO_2-NCs sample.
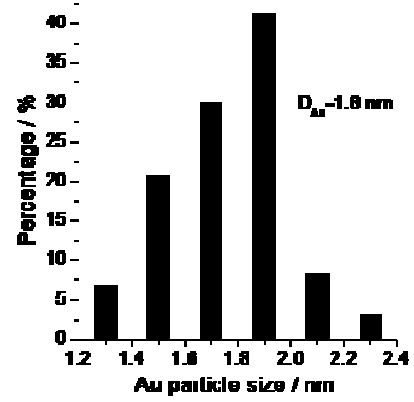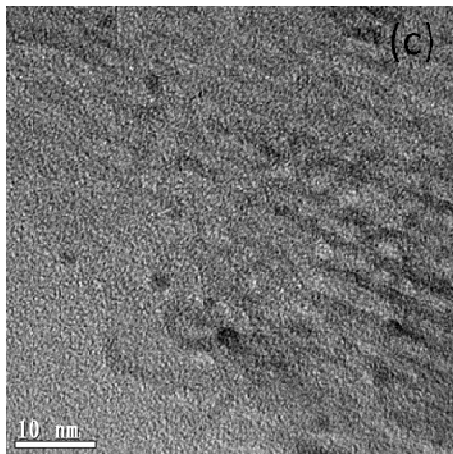
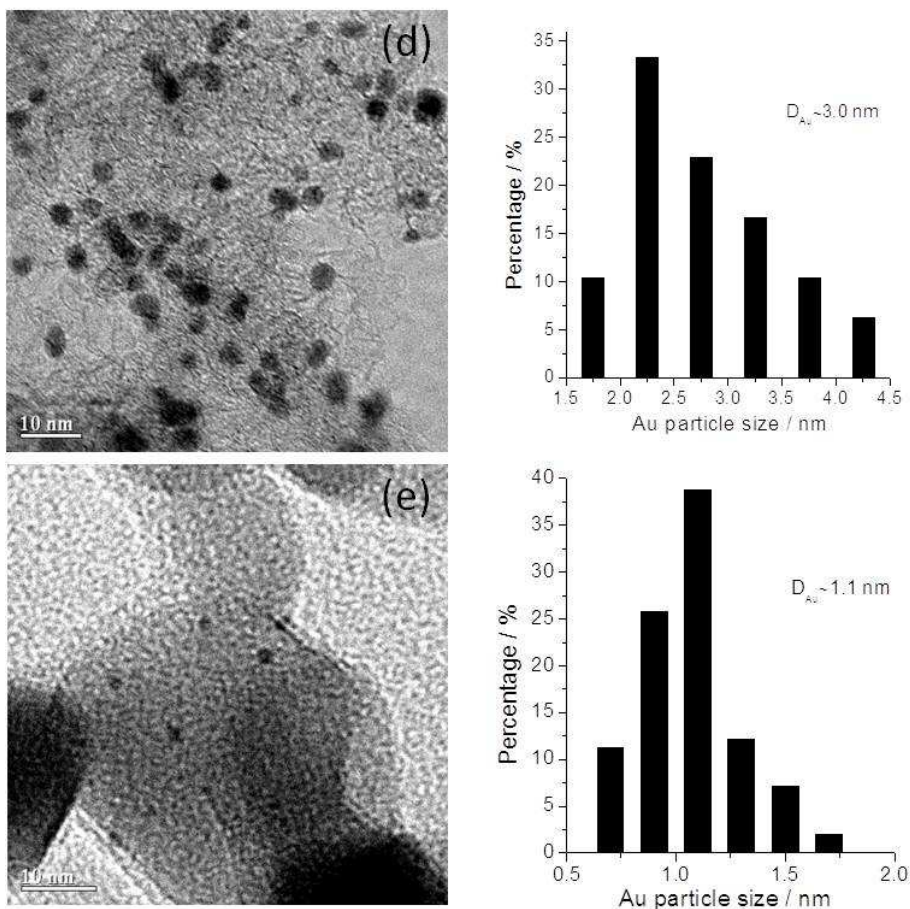**Figure S12.** TEM images of (a) Au/TiO$_2$, (b) Au/SiO$_2$, (c) Au/Al$_2$O$_3$, (d) Au/C, and (e) Au/TiO$_2$-NCs.

Table S4. CO chemisorption data at −116 $^{\circ}$C[a]

Catalyst	mL$_{CO(STP)}$/g$_{Au}$[b]	mol$_{CO}$/mol$_{Au}$[b]	Dispersion (%)[c]	Size (nm)[c]
Au/ZrO$_2$	25.5	0.224	64	1.8
Fresh Au/ZrO$_2$-NCs	38.0	0.333	95	0.8
Used Au/ZrO$_2$-NCs	37.4	0.329	94	0.8

[a] In general, an error of ±10% was established for the titration results. [b] Based on total Au. [c] Dispersion calculated according to the known parameters of Au/ZrO$_2$ catalyst. Assuming an equal Au/CO chemisorption stoichiometry for gold NPs or subnano-clusters dispersed on ZrO$_2$ support, the particle size was calculated based on an assumption of a quasi-hemispherical model of gold particle (see below).[S1,S10]
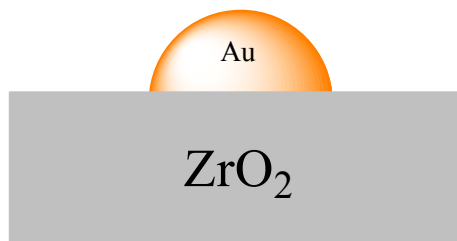
For Au/ZrO$_2$ catalyst, the Au particle size was 1.8 nm from TEM data.

The total surface area, and the number of exposed atoms, at a given metal amount, is in inverse proportion with particle diameter (see Ref. S10).

The number of gold particles $N_1 = \dfrac{m_{Au}}{\dfrac{2}{3}\pi(\dfrac{d_{Au}}{2})^3 \rho_{Au}}$

The gold particle diameter (d_{Au}) of Au/ZrO$_2$ catalyst is 1.8 nm

$\rho_{Au} = 19.3 \times 10^3$ kg m^{-3}

The overall surface area of gold particles $S = 2\pi(\dfrac{d_{Au}}{2})^2 N_1$

N_S = total number of surface gold atoms

Dispersion = $\dfrac{N_S}{N_T} = \dfrac{Sk}{n_{Au}N_A}$

N_T = total number of gold atoms

The gold atom density (k) is 1.15×10^{19} m^{-2}

Assuming the case of Au/ZrO$_2$, Dispersion (Au) = 64.2 %

$N_A = 6.02 \times 10^{23}$ mol^{-1} $\qquad n_{Au} = m_{Au}/M_{Au}$

The Au particle size of Au/ZrO$_2$-NCs catalyst was calculated inversely according to the corresponding dispersion which can be obtained from CO titration.

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