# WORK PERMIT

**Department of Chemical and Biological Engineering**

化學及生物工程學系

Project Title : A New Class of Electrocatalysts for Hydrogen Evolution Reaction

Researcher(s) : Tiehuai LI

Supervisor(s) : Prof. Minhua SHAO

Work Plan No. : 17026

Date of Approval : 12/7

Date of Revalidation : N/A

Signature of Approval :

Prof. Marshal LIU
Acting DSO

HONG KONG UNIVERSITY OF SCIENCE AND TECHNOLOGY
DEPARTMENT OF CHEMICAL AND BIOLOGICAL ENGINEERING

# A New Class of Electrocatalysts for Hydrogen Evolution Reaction

## Work Plan #17026

Researcher: Tiehuai LI

Supervisor: Dr. Minhua SHAO

04/25/2017

## 1. General Information

| | |
|---|---|
| Name of Researcher: | Tiehuai LI |
| Name of Project Supervisors: | Dr. Minhua SHAO |
| Project Title: | A New Class of Electrocatalysts for Hydrogen Evolution Reaction |
| Research Area: | Chemical Engineering |
| Location: | Lab 7102 |
| Proposed Start Date: | May. 2017 |

## 2. Experiment/Project Description

### Objective:

The aims and the objectives of this project are:

a.) to develop $MoS_2$, non-precious metal materials and Pd-based materials as catalysts for hydrogen evolution reaction in water splitting;

b.) to optimize the preparation conditions to tune and control the structures of the catalysts;

c.) to study the activity of the developed catalysts;

d.) to improve the efficiency of hydrogen evolution reaction with use of the developed catalysts .

### Experiment Description:

To synthesis $MoS_2$ materials.

To make surface modification of Pd.

To synthesize different alloy nanoparticles supported on carbon black, and characterize the samples by XRD, TEM, SEM, etc.

To further evaluate their catalytic activities in a three-electrode cell.

## 3. Equipment List

| Equipment | Location |
|---|---|
| Electronic balance | 7102 |
| Pipette (80µl, 1.5ml, 0.5ml) | 7102 |
| Stirring heater | 7102 |
| 50 ml, 100 ml, 200 ml Volumetric Flask | 7102 |
| Beakers (of various volumes), test-tubes, sample holder bottles | 7102 |
| pH meter | 7102 |
| Beakers (of various volumes), test-tubes, sample holder bottles | 7102 |
| X-ray diffraction (XRD) equipment | 2150 |
| Transmission Electron Microscope (TEM) | 2213, 2218 |
| Scanning Electron Microscopy (SEM) | 1125 |
| Induced Coupled Plasma (ICP) | 7101 |
| Raman spectroscopy | ~~7101~~&2153 7106 |
| Oven | 7102 SC |
| Ultrasonic instrument | 7102 |
| Centrifuge | 7101&6114 SC |
| Fume hood | 7102 |
| Potentiostat | 7102 |
| Furnace | 7102 |
| Home-made electrochemical cell | 7102 |

# 4. Experimental Procedures

**4.1 Synthesis of $MoS_2$ nanoparticles**

(4.1.1) The growth of monolayer $MoS_2$ on (5)Au foils is performed by CVD inside a tubular furnace equipped with a 2-in.-diameter quartz tube. Substrates are cleaned in (1)acetone for 30 min and then (2)isopropanol for 15 min.

(4.1.2) The quartz boat containing 100 mg (3)sulfur powder is placed on the upstream region. Place another boat containing of 15 mg (4)$MoO_3$ powder located downstream and is 18 cm away from the boat of sulfur. (5)Au foils are placed downside of the (4)$MoO_3$.

(4.1.3) The CVD growth occurs at atmospheric pressure while ultra-high-purity (30)argon is flowing. The growth recipe is: sit 4 h at 105℃ with 500 sccm, ramp to 700℃ at 15 ℃/min with 10 sccm, sit 5 min at 700 ℃, cool to 570 ℃ without feedback (20 min) with 10 sccm, open furnace and flow 500 sccm for rapid cooling.

(4.1.4) Replace the substrate with other metal foils such as (6)Cu, (7)Ni, (8)Co, (9)Pd, (10)Ag, (11)Mo, (12)Fe, (13)Ru, (14)Re, (15)Rh, etc. Repeat the processes (4.1.1) – (4.1.3) to obtain $MoS_2$ monolayer on different substrates.

**4.2 Synthesis of non-precious metal materials**

(4.2.1) Add 1 g (16)carbon black to a 100 ml beaker. Then add appropriate ultrapure water in the beaker. Stir and sonicate the solution to make carbon well dispersed. Dissolve appropriate amount of (17)$Ni(NO_3)_2 \cdot 6H_2O$ and (18)$Cu(NO_3)_2 \cdot 3H_2O$ to achieve 1:1 ratio Ni:Cu in ultrapure water.

(4.2.2) Add the metal precursor solutions to the carbon dispersion. Stir and ultrasonically blend the solution. Then the wet catalysts are dried in air or in a vacuum oven at 110℃ overnight and annealed at 500℃ in a furnace for 5 h.

(4.2.3) Replace the precursors with other metal salts including (19)$AgNO_3$, (20)$MoCl_3$, (21)$ReCl_3$, (22)$RuCl_3$, (23)$AuCl_3$, (24)$CoCl_2$, (25)$IrCl_3$, etc. Repeat the processes (4.2.1)-(4.2.2) to get alloys combined by different metals.

**4.3 Surface modification of Pd**

**4.4 The electrochemical tests**

(4.4.1)The electrochemical measurements are performed in a three-electrode system with an electrochemical workstation. Typically, 4 mg of sample and 30 μL (26)Nafion solution are dispersed in 1 ml

water-(27)ethanol solution with volume ratio of 3:1 by sonicating for 1 h to form a homogeneous ink. Then 5 μl of the dispersion is loaded onto a glassy carbon electrode as the working electrode. Use Ag/AgCl electrode as the reference electrode, a Pt mesh as a counter electrode. All the potentials are calibrated to a reversible hydrogen electrode (RHE).

(4.4.2)Two kinds of electrolytes, the 1 M (28)KOH solution, and the 0.5 M (29)$H_2SO_4$ electrolyte solution are used for electrocatalytic activity tests;

(4.4.3) 25 ml electrolyte is added to the cell. Prior to experiments, (30)Ar gas is purged into the solution for 45 min to remove other gases and a slow flow of (30)Ar is maintained above the solution during measurements.

(4.4.4) Linear sweep voltammetric (LSV) and cyclic voltammetric (CV) with scan rate of 50 mV/s are conducted in the range of +0.3 V and -0.35 V vs RHE.

**4.5 Characterization of the prepared catalysts**

The prepared samples will be characterized by the following tests.

*Transmission Electron Microscope(TEM)*

The TEM images of a typical sample are obtained on a JEOL 2010F microscope;

*Scanning Electron Microscopy (SEM)*

The SEM images of a typical sample are obtained on a JSM-7100F (JEOL) microscope;

*X-ray diffraction (XRD) equipment*

The crystal structure and material types are obtained on a PW101290 1030 (Philips) equipment;

*Induced Couple Plasma (ICP)*

The amount of metal loaded can be analyzed induced coupled plasma (ICP model: Perkin Elmer Optima 3000 XL). The weighed catalyst is dissolved in high concentration nitric acid (65%) for 24 hours. The mixture is then filtered to remove any residue. The solution is then diluted until the concentration of the metal falls within 1 – 10 ppm. Finally, the measurement of the concentration will be performed and the metal loading can be obtained.

*BET*

The specific surface area of the NPs/C will be investigated by $N_2$ adsorption and desorption and calculated by BET method.

## 5. PROCEDURE TEMPLATE

| Experimental Procedure No. | Experimental Procedure Description | Scale (Mass/Volume) | Location (Fumehood,benctop,etc) | Method New or Existing |
|---|---|---|---|---|
| 4.1.1 | Clean the substrate in (1)acetone,(2) isopropanol. | (1)Acetone: adequate<br>(2)Isopropanol: adequate | Fumehood -7102 | Existing |
| 4.1.2 | Put the (3)sulfur powder upstream and the (4)$MoO_3$ powder, (5)Au foil on the downstream region successively of the quartz tube. | (3)Sulfur powder: 100mg<br>(4)$MoO_3$ power: 15mg<br>(5)Au foil | Furnace -7102 | Existing |
| 4.1.3 | Heat the sample under 105℃ for 4 h with 500 sccm (30)argon gas and get $MoS_2$ monolayer on (5)Au under 700℃ with 10 sccm argon. | | Furnace -7102 | Existing |
| 4.1.4 | Replace the substrate with other metal foils such as (6)Cu, (7)Ni, (8)Co, (9)Pd, (10)Ag, (11)Mo, (12)Fe, etc. Repeat the process (4.1.1) – (4.1.3). | Metal foils | Fumehood -7102&Furnace-7102 | Existing |
| 4.2.1 | Disperse carbon black in ultrapure water. Dissolve (17)$Ni(NO_3)_2 \cdot 6H_2O$ and (18)$Cu(NO_3)_2 \cdot 3H_2O$ to achieve 1:1 ratio. | Carbon black: 1 g<br>(17)$Ni(NO_3)_2 \cdot 6H_2O$: 1.3g<br>(18)$Cu(NO_3)_2 \cdot 3H_2O$: 1 g | Fumehood -7102 | Existing |
| 4.2.2 | Add the metal precursor solutions to the C dispersion. Dry the solution at 110℃ in air overnight and annealed at 500℃ in a furnace for 5 h. | | Fumehood -7102&Furnace-7102 | Existing |
| 4.2.3 | Replace the precursors with other metal solutions including (19)$AgNO_3$, (20)$MoCl_3$, (21)$ReCl_3$, (22)$RuCl_3$, (23)$AuCl_3$, (24)$CoCl_2$, (25)$IrCl_3$, etc. Repeat the processes (4.2.1)-(4.2.2). | metal precursors | Fumehood -7102&Furnace-7102 | Existing |

| | | | | |
|---|---|---|---|---|
| 4.3.1 | Sample and (26)Nafion solution are dispersed in water-(27)ethanol solution to form a homogeneous ink and loaded onto a glassy carbon electrode as work electrode. Ag/AgCl electrode is used as the reference electrode, a Pt foil as a counter electrode. | (26)Nafion: 30 µL<br>(27)Ethanol: 0.25 ml | Bench-7102 | Existing |
| 4.3.2 | Two kinds of electrolytes, the 1 M (28)KOH solution, and the 0.5 M (29)$H_2SO_4$ electrolyte solution are used for electrocatalytic activity tests; | 1M (28)KOH: 25 ml<br>0.5M (29)$H_2SO_4$: 25 ml | Bench-7102 | Existing |
| 4.3.3 | Electrolyte is added to the cell. (30)Ar gas is purged into the solution. | | Bench-7102 | Existing |
| 4.3.4 | Linear sweep voltammetric (LSV) and cyclic voltammetric (CV) with scan rate of 50 mV/s are conducted in the range of +0.3 V and -0.35 V vs RHE. | | Bench-7102 | Existing |
| 4.4 | **Sample Characterization** | | 7102/2150/2213/2218/1125 | Existing |

# 6. HAZOP Template

## Hazard and Operability Analysis

**4.1.1**

| NO | HAZARD | HAZARD EFFECT | SEVERITY | PROBABILITY | RISK | MINIMISE RISK BY | RESIDUAL RISK |
|---|---|---|---|---|---|---|---|
| 1 | Contact with chemicals | Causes severe skin and eye burns. | H | M | H | Wear protective gloves, face shield and lab coats; Conduct experiments in the fume hood | L |
| 2 | Volatile chemicals inhalation | Harmful by inhalation. | H | M | H | Take the measurement within the fume hood | L |
| 3 | Flammable chemicals | Fire, explosion | H | L | M | Keep it away from heated surface | L |
| FINAL ASSESSMENT: | | | | | | OVERALL RISK: | L |

Remark: Severity - Low:1 (Minor injuries, first aid); Medium:2 (Hospitalization, medical leave); High:3 (Serious injuries, fatality)
Probability - Unlikely:1; Possible:2; Very Likely: 3.

## HAZOP Template
## Hazard and Operability Analysis

**4.1.2**

| NO | HAZARD | HAZARD EFFECT | SEVERITY | PROBABILITY | RISK | MINIMISE RISK BY | RESIDUAL RISK |
|---|---|---|---|---|---|---|---|

| | | | | | | | |
|---|---|---|---|---|---|---|---|
| 1 | Contact with chemicals | Causes severe skin and eye burns. | H | M | H | Wear protective gloves, face shield and lab coats; Conduct experiments in the fume hood | L |
| 2 | Volatile chemicals inhalation | Harmful by inhalation. | H | M | H | Take the measurement within the fume hood | L |
| 3 | High temperature of the furnace | Skin burn if touched | M | L | M | Set a warning sign and operate with protective gloves. | L |
| FINAL ASSESSMENT: | | | | | | OVERALL RISK: | L |

Remark: Severity - Low:1 (Minor injuries, first aid); Medium:2 (Hospitalization, medical leave); High:3 (Serious injuries, fatality)
Probability - Unlikely:1; Possible:2; Very Likely: 3.

# HAZOP Template
# Hazard and Operability Analysis

**4.1.3**

| NO | HAZARD | HAZARD EFFECT | SEVERITY | PROBABILITY | RISK | MINIMISE RISK BY | RESIDUAL RISK |
|---|---|---|---|---|---|---|---|
| 1 | Contact with chemicals | Causes severe skin and eye burns. | H | M | H | Wear protective gloves, face shield and lab coats; Conduct experiments in the fume hood | L |
| 2 | Volatile chemicals inhalation | Harmful by inhalation. | H | M | H | Take the measurement within the fume hood | L |
| 3 | High temperature of the furnace | Skin burn if touched | M | L | M | Set a warning sign and operate with protective gloves. | L |
| FINAL ASSESSMENT: | | | | | | OVERALL RISK: | L |

Remark: Severity - Low:1 (Minor injuries, first aid); Medium:2 (Hospitalization, medical leave); High:3 (Serious injuries, fatality)
Probability - Unlikely:1; Possible:2; Very Likely: 3.

# HAZOP Template
# Hazard and Operability Analysis

**4.1.4**

| NO | HAZARD | HAZARD EFFECT | SEVERITY | PROBABILITY | RISK | MINIMISE RISK BY | RESIDUAL RISK |
|---|---|---|---|---|---|---|---|
| 1 | Contact with chemicals | Causes severe skin and eye burns. | H | M | H | Wear protective gloves, face shield and lab coats; Conduct experiments in the fume hood | L |
| 2 | Volatile chemicals inhalation | Harmful by inhalation. | H | M | H | Take the measurement within the fume hood | L |
| 3 | High temperature of the furnace | Skin burn if touched | M | L | M | Set a warning sign and operate with protective gloves. | L |
| 4 | Flammable chemicals | Fire, explosion | H | L | M | Keep it away from heated surface | L |
| FINAL ASSESSMENT: | | | | | | OVERALL RISK: | L |

Remark: Severity - Low:1 (Minor injuries, first aid); Medium:2 (Hospitalization, medical leave); High:3 (Serious injuries, fatality)
Probability - Unlikely:1; Possible:2; Very Likely: 3.

# HAZOP Template
# Hazard and Operability Analysis

**4.2.1**

| NO | HAZARD | HAZARD EFFECT | SEVERITY | PROBABILITY | RISK | MINIMISE RISK BY | RESIDUAL RISK |
|---|---|---|---|---|---|---|---|

| | | | | | | | |
|---|---|---|---|---|---|---|---|
| 1 | Contact with chemicals | Causes severe skin and eye burns. | H | M | H | Wear protective gloves, face shield and lab coats; Conduct experiments in the fume hood | L |
| 2 | Volatile chemicals inhalation | Harmful by inhalation. | H | M | H | Take the measurement within the fume hood | L |
| 3 | Hot surface of the stirring heater and solution | Skin burn if touched | M | L | M | Set a warning sign and operate with protective gloves. | L |
| FINAL ASSESSMENT: | | | | | | OVERALL RISK: | L |

Remark: Severity - Low:1 (Minor injuries, first aid); Medium:2 (Hospitalization, medical leave); High:3 (Serious injuries, fatality)
Probability - Unlikely:1; Possible:2; Very Likely: 3.

# HAZOP Template
# Hazard and Operability Analysis

**4.2.2**

| NO | HAZARD | HAZARD EFFECT | SEVERITY | PROBABILITY | RISK | MINIMISE RISK BY | RESIDUAL RISK |
|---|---|---|---|---|---|---|---|
| 1 | Contact with chemicals | Causes severe skin and eye burns. | H | M | H | Wear protective gloves, face shield and lab coats; Conduct experiments in the fume hood | L |
| 2 | Volatile chemicals inhalation | Harmful by inhalation. | H | M | H | Take the measurement within the fume hood | L |
| 3 | Hot surface of the stirring heater, furnace and solution | Skin burn if touched | M | L | M | Set a warning sign and operate with protective gloves. | L |
| FINAL ASSESSMENT: | | | | | | OVERALL RISK: | L |

Remark: Severity - Low:1 (Minor injuries, first aid); Medium:2 (Hospitalization, medical leave); High:3 (Serious injuries, fatality)
Probability - Unlikely:1; Possible:2; Very Likely: 3.

# HAZOP Template
# Hazard and Operability Analysis

**4.2.3**

| NO | HAZARD | HAZARD EFFECT | SEVERITY | PROBABILITY | RISK | MINIMISE RISK BY | RESIDUAL RISK |
|---|---|---|---|---|---|---|---|
| 1 | Contact with chemicals | Causes severe skin and eye burns. | H | M | H | Wear protective gloves, face shield and lab coats; Conduct experiments in the fume hood | L |
| 2 | Volatile chemicals inhalation | Harmful by inhalation. | H | M | H | Take the measurement within the fume hood | L |
| 3 | Hot surface of the stirring heater, furnace and solution | Skin burn if touched | M | L | M | Set a warning sign and operate with protective gloves. | L |
| FINAL ASSESSMENT: | | | | | | OVERALL RISK: | L |

Remark: Severity - Low:1 (Minor injuries, first aid); Medium:2 (Hospitalization, medical leave); High:3 (Serious injuries, fatality)
Probability - Unlikely:1; Possible:2; Very Likely: 3.

# HAZOP Template
# Hazard and Operability Analysis

**4.3.1**

| NO | HAZARD | HAZARD EFFECT | SEVERITY | PROBABILITY | RISK | MINIMISE RISK BY | RESIDUAL RISK |
|---|---|---|---|---|---|---|---|

| | | | | | | | |
|---|---|---|---|---|---|---|---|
| 1 | Contact with chemicals | Causes skin and eye burns. | M | M | M | Wear protective gloves, face shield and lab coats; Conduct experiments in the fume hood | L |
| 2 | Volatile chemicals inhalation | Harmful by inhalation. | H | M | H | Take the measurement within the fume hood | L |
| 3 | Flammable chemicals | Fire, explosion | H | L | M | Set up the muffle furnace carefully and check the temperature within 2h; keep it away from heated surface | L |
| FINAL ASSESSMENT: | | | | | | OVERALL RISK: | L |

Remark: **Severity** - Low:1 (Minor injuries, first aid); Medium:2 (Hospitalization, medical leave); High:3 (Serious injuries, fatality)
**Probability** - Unlikely:1; Possible:2; Very Likely: 3.

# HAZOP Template
# Hazard and Operability Analysis

**4.3.2**

| NO | HAZARD | HAZARD EFFECT | SEVERITY | PROBABILITY | RISK | MINIMISE RISK BY | RESIDUAL RISK |
|---|---|---|---|---|---|---|---|
| 1 | Contact with chemicals | Causes skin and eye burns. | M | M | M | Wear protective gloves, face shield and lab coats; | L |
| FINAL ASSESSMENT: | | | | | | OVERALL RISK: | L |

Remark: **Severity** - Low:1 (Minor injuries, first aid); Medium:2 (Hospitalization, medical leave); High:3 (Serious injuries, fatality)
**Probability** - Unlikely:1; Possible:2; Very Likely: 3.

# HAZOP Template

## Hazard and Operability Analysis

**4.3.3**

| NO | HAZARD | HAZARD EFFECT | SEVERITY | PROBABILITY | RISK | MINIMISE RISK BY | RESIDUAL RISK |
|---|---|---|---|---|---|---|---|
| 1 | Contact with chemicals | Causes severe skin and eye burns. | M | M | M | Wear protective gloves, face shield and lab coats; | L |
| FINAL ASSESSMENT: | | | | | | OVERALL RISK: | L |

Remark: Severity – Low:1 (Minor injuries, first aid); Medium:2 (Hospitalization, medical leave); High:3 (Serious injuries, fatality)
Probability – Unlikely:1; Possible:2; Very Likely: 3.


## HAZOP Template
## Hazard and Operability Analysis


**4.3.4**

| NO | HAZARD | HAZARD EFFECT | SEVERITY | PROBABILITY | RISK | MINIMISE RISK BY | RESIDUAL RISK |
|---|---|---|---|---|---|---|---|
| 1 | Contact with chemicals | Causes severe skin and eye burns. | M | M | M | Wear protective gloves, face shield and lab coats; | L |
| FINAL ASSESSMENT: | | | | | | OVERALL RISK: | L |


Remark: Severity – Low:1 (Minor injuries, first aid); Medium:2 (Hospitalization, medical leave); High:3 (Serious injuries, fatality)
Probability – Unlikely:1; Possible:2; Very Likely: 3.



## HAZOP Template
## Hazard and Operability Analysis


**4.4 Sample Characterization**

| NO | HAZARD | HAZARD EFFECT | SEVERITY | PROBABILITY | RISK | MINIMISE RISK BY | RESIDUAL RISK |
|---|---|---|---|---|---|---|---|
| 1 | Wrong operation | Personal injury; Property loss | H | M | H | Being well trained and operating according to the instruction of the manual | L |
| FINAL ASSESSMENT: | | | | | | OVERALL RISK: | L |

## 7. Operating Conditions

**Synthesis of $MoS_2$ monolayer on substrate**

- Pressure : (30)Ar atmospheric
- Temperature : 105℃-700℃
- Flow rates : batch operations therefore not applicable

**Synthesis of alloy nanoparticles**

- Pressure : atmospheric
- Temperature :100℃-500℃
- Flow rates : batch operations therefore not applicable

**Electrochemical Tests**

- Pressure : (30)Ar atmospheric
- Temperature : room temperature
- Flow rates : batch operations therefore not applicable

## 8. Services List

Electricity (AC 220V, 50Hz)

Double de-ionized water

Tap water

Compressed (30)Argon

## 9. Chemicals List

| Number | Chemical | Purity | Quantity per Experiment |
|---|---|---|---|
| (1) | Acetone ($CH_3COCH_3$) | >99.9% | 50ml |
| (2) | Isopropanol($C_3H_8O$) | >99.5% | 50ml |
| (3) | Sulfur powders | >99.5% | 100mg |
| (4) | Molybdenum oxide($MoO_3$) | 99.9% | 15mg |
| (5) | Gold foils | 99.99%, | 50mg |

| | | | |
|---|---|---|---|
| | | thichness~25μm | |
| (6) | Copper foils | 99.99%, thichness~25μm | 50mg |
| (7) | Nickle foils | 99.99%, thichness~25μm | 50mg |
| (8) | Cobalt foils | 99.99%, thichness~25μm | 50mg |
| (9) | Palladium foils | 99.99%, thichness~25μm | 50mg |
| (10) | Silver foils | 99.99%, thichness~25μm | 50mg |
| (11) | Molybdenum foils | 99.99%, thichness~25μm | 50mg |
| (12) | Iron foils | 99.99%, thichness~25μm | 50mg |
| (13) | Ruthenium foils | 99.99%, thichness~25μm | 50mg |
| (14) | Rhenium foils | 99.99%, thichness~25μm | 50mg |
| (15) | Rhodium foils | 99.99%, thichness~25μm | 50mg |
| (16) | Carbon black | 99.95% | 1g |
| (17) | Nickel(II) nitrate hexahydrate ($Ni(NO_3)_2 \cdot 6H_2O$) | >98.5% | 1.3g |
| (18) | Copper(II) nitrate trihydrate ($Cu(NO_3)_2 \cdot 3H_2O$) | >99% | 1g |
| (19) | Silver nitrate ($AgNO_3$) | >99% | 0.4g |
| (20) | Molybdenum(III) chloride ($MoCl_3$) | >99% | 0.5g |
| (21) | Rhenium(III) chloride ($ReCl_3$) | >99% | 0.4g |
| (22) | Ruthenium(III) chloride ($RuCl_3$) | >99% | 0.5g |
| (23) | Gold(III) chloride ($AuCl_3$) | >99% | 0.4g |
| (24) | Cobalt(II) chloride ($CoCl_2$) | >99% | 0.6g |
| (25) | Iridium(III) chloride ($IrCl_3$) | >99% | 0.4g |
| (26) | Nafion solution | ~5% | 30μL |
| (27) | Ethanol ($CH_3CH_2OH$) | >99.8% | 10ml |

| | | | |
|---|---|---|---|
| (28) | Potassium hydroxide (KOH) | >99.99% | 25ml |
| (29) | Sulfuric acid ($H_2SO_4$) | 99.999% | 25ml |
| (30) | Argon | >99.999% | |

## 10. Biological Agents List

N/A

## 11. Summary of Relevant Hazards and Incompatibilities

| Number | Material | Summary of Hazards | Incompatibilities |
|---|---|---|---|
| (1) | Acetone | Irritant, highly volatile and flammable | Flame, hot surface, static electricity, spark, strong oxidizing agents, strong reducing agents |
| (2) | Isopropanol | Irritant and high flammable | heat, hot surfaces, sparks, open flames and other ignition sources. |
| (3) | Sulfur powder | Irritant | Strong oxidizing agents, amines, bases |
| (4) | Molybdenum oxide | Irritant | Strong oxidizing agents, Strong acids |
| (5) | Gold foils | Not a hazardous substance | Halogens, Hydrogen peroxide, Ammonia |
| (6) | Copper foils | Not a hazardous substance | Strong acids, Strong oxidizing agents, Acid chlorides, Halogens |
| (7) | Nickle foils | Toxic | acids, Oxidizing agents, Oxygen, Methanol, organic solvents, etc. |
| (8) | Cobalt foils | Toxic | Oxidizing agents, Mineral acids,Acetylene, etc. |
| (9) | Palladium foils | Not a hazardous substance | Strong acids, Halogens |
| (10) | Silver foils | Not a hazardous substance | Oxygen, Strong acids and strong bases |
| (11) | Molybdenum foils | High flammable solid | Heat, flames and sparks. Extremes of temperature and direct sunlight |
| (12) | Iron foils | Not a hazardous substance | acids, Oxygen, Strong oxidizing agents, Halogens, Phosphorus |
| (14) | Rhenium foils | High flammable solid | Heat, flames and sparks. Extremes of temperature and direct sunlight |
| (15) | Rhodium foils | Flammable solid | Heat, flames and sparks. Extremes of temperature and direct sunlight |
| (19) | Silver nitrate | Corrosive and toxic | Strong reducing agents, Alcohols, Ammonia, Magnesium, Strong bases |
| (18) | Copper(II) nitrate trihydrate | Oxidizing, irritant and toxic | Reducing agents, Organic materials, Powdered metals |
| (17) | Nickel(II) nitrate hexahydrate | Oxidizing, irritant and toxic | Organic materials, Powdered metals, Strong reducing agents, acids |
| (20) | Molybdenum(III) | Irritant | Avoid moisture.Strong oxidizing agents |

| | | | |
|---|---|---|---|
| | chloride | | |
| (21) | Rhenium(III) chloride | Irritant | Avoid moisture.Strong oxidizing agents |
| (22) | Ruthenium(III) chloride | Corrosive | Zinc |
| (23) | Gold(III) chloride | Irritant | Avoid moisture, Light and Ammonia, Amines. |
| (24) | Cobalt(II) chloride | Toxic | Avoid moisture and Oxidizing agents, Alkali metals |
| (25) | Iridium(III) chloride | Irritant | Strong oxidizing agents acids, Bases, etc. |
| (26) | Nafion solution | Irritant corrosive and flammable | Strong oxidizing agents, strong reducing agents,flame, hot surface, static electricity, spark |
| (29) | Sulfuric acid | Corrosive and irritant | Strong oxidizing agents, Reducing agents, Bases, Metals |
| (28) | Potassium hydroxide | Corrosive and toxic | Nitro compounds, Organic materials,etc. |
| (27) | Ethanol | High flammable | Heat, flames and sparks. Extremes of temperature and direct sunlight |
| (30) | Argon | Asphyxiation | Titanium is the only element that will burn in nitrogen; Lithium reacts slowly with nitrogen at ambient temperatures. |

## 12. Waste List

(1) The metal ions contained solution will be collected in the metalic waste container (located at 7/F service corridor).
(2) Mixture of residual (1)acetone, (2)isopropanol, (27)ethanol, etc. will be disposed in the Non-Halogenated waste solvent container (located at 7/F service corridor).
(3) Diluted (29)$H_2SO_4$ solutions will be disposed in the inorganic acids waste container (located at 7/F service corridor).

## 13. Assessment of Significant Risks

**Synthesis of $MoS_2$ monolayer on substrate**

- Pressure : (30)Ar atmospheric
- Temperature :105℃-700℃
- Flow rates : batch operations therefore not applicable

**Synthesis of alloy nanoparticles**

- Pressure : atmospheric
- Temperature :100℃-500℃
- Flow rates : batch operations therefore not applicable

## 14. Safety Precautions

*Safety Training Required*

☐ The researcher must attend regular courses (and in some courses obtain a passing result) offered by the Safety and Environmental Office of the University (HKUST) and understand thoroughly the safety concepts of these courses and be able to apply them before being allowed to conduct any experiments or use any of the equipment associated.

☐ The researcher must understand and be aware of all the procedures for handling, and in some cases dealing, with all possible emergency situations and scenarios.

☐ The researcher must undergo training in handling equipment and conducting experiments competently before performing it on his/her own.

☐ Equipment training offered by technical staff for nitrogen adsorption (Omnisorp 100CX), ICP (Perkin Elmer Optima), Elemental Analyser (Vario ELII Eliminator), TGA (Shimadzu TGA-50) are required before conducting the tests.

☐ XRD, XRF, XPS, TEM, and FTIR will be conducted by the technical staff of the MCPF.

*General Laboratory Safety Rules*

It is HKUST policy to ensure that employees and students who are engaged in potentially hazardous operations receive both general and job-specific safety training prior to conducting these operations. Each department will establish a management scheme to implement this policy. Each supervisor will evaluate the nature of the work and will determine what safety training is needed for employees and students under his/her supervision.

General safety courses listed below cover a wide variety of safety issues. In addition to assigning employees and students to attend these courses, supervisors shall also provide job-specific safety training to ensure the safe performance of potentially hazardous operations.

Mandatory Courses (Chemical Safety II / Hazardous Waste Management, MC03; Pressure Safety, MC05; Chemical Safety I / Chemical Safety for Laboratory Users, MC07) and Discretional Courses (Fire Safety and Fire-fighting Equipment, DC03) should be taken in terms of this project.

*Personal Protective Equipment*

☐ Laboratory coat, safety goggles, and nitrile/polyvinyl alcohol gloves because of the presence of solvents such as toluene (from Instant Glove + CPC Database) as the outer layer with rubber gloves as the inner layer must be worn at all times when performing the experiments, transporting, handling and cleaning chemicals and equipment and clearing chemical spills.

☐ Dust mask should be worn when collecting and weighing solid particles.

☐ Heat resistant gloves will be worn when handling hot items..

*Handling Organic Compounds*

Personal Protective Equipment should be used when handling organic compounds. When disposing of organic waste, organic solvents with halogens (e.g. trichloromethane, trichloroethylene, and dichloromethane) should be collected in "Halogenated Solvents" container and other liquid organic compounds with halogens. "Non-halogenated Solvents" container is for organic solvents and other organic compounds without halogens (e.g. acetone, hexane, and petroleum ethers).

*Handling Flammable Liquid*

Carefully read the manufacturer's label on the container of any flammable liquid before storing or using it. Practice good housekeeping in flammable liquid storage areas. Clean up spills immediately then place the cleanup rags in a closed, bottom ventilated, metal container. Only use approved metal safety containers or the original manufacturer's container to store flammable liquids. Keep the containers closed when not in use; stored away from exits or passageways.

*Handling Heat Process*

Be aware of heat process and mind high temperature equipment, especially the temperature controller. Don't touch the high temperature unit directly.

## 15. Action in Case of Abnormal or Emergency Situations

**(A) In case of loss of containment or accidental spillage**

(1) Minor spillage (<100mL)
Alert co-workers
If safe to do so, confine the spill with appropriate material and/or turn off remotely all heat/ignition sources if flammable vapour is involved.
Ask for assistance is necessary.
Press the emergency ventilation button (do not activate this button in case of fire).
Inform the security office at x8999 or 23588999 with mobile phone when it is safe to do so.
Evacuate everyone in the affected area. Leave contaminated clothing and close the door.
Activate local warning system to prevent others from entering the room.
If possible, maintain a safe distance from the scene, keep the entrance or access routes in sight and help to prevent entry to the affected room.
If conditions allow, remain to assist the emergency response team.

(2) Solid chemicals
Alert co-workers
If safe to do so, confine the spill with appropriate material and/or turn off remotely all heat/ignition sources if flammable vapour is involved.
Ask for assistance is necessary.
If confident, clean the spillage properly, being protected by Personal Protective Equipment.

(3) Major spillage (>1L)
Alert co-workers
If safe to do so, confine the spill with appropriate material and/or turn off remotely all heat/ignition sources if flammable vapour is involved.
Ask for assistance is necessary.
Press the emergency ventilation button (do not activate this button in case of fire).
Inform the security office at x8999 or 23588999 with mobile phone when it is safe to do so.
Evacuate everyone in the affected area. Leave contaminated clothing and close the door.
Activate local warning system to prevent others from entering the room.
If possible, maintain a safe distance from the scene, keep the entrance or access routes in sight and help to prevent entry to the affected room.
If conditions allow, remain to assist the emergency response team.

**(B) Fire-fighting measures**

**When the fire alarm is heard**

- Check if there is any sign of fire in the vicinity.
- If there is fire or smoke, or there is an announcement to evacuate, then evacuate to the assembly point as far as practicable and report to the fire & safety officer.
- If there is no sign of a fire, stay alert and pay attention to announcement until the fire alarm is silenced.
- Evacuate if the alarm has sounded for more than two minutes.
- If the buzzer sound which indicates fire alarm is activated in an adjacent fire zone is heard, stay alert and pay attention to announcement.
- If both the buzzer and the fire alarm are heard, treat as if the fire alarm is heard.

**If a fire is discovered**

- Perform emergency shut downproceduresif possible.
- Activate the fire alarm by pressing the breakglass fire alarm button.
- Report to Security Control Centre by dialling 8999.
- Alert other people. If safe to do so, try to put out the fire by fire fighting equipment.
- Do not take any personal risk. If the fire gets beyond control, evacuate immediately as listed above.
- Close the door of the room on fire.

**Fire fighting equipment**

- Water from the hose reels is good for wood and paper fire, structural fire, but not for oil, electrical or metal fire.
- The most common fire extinguishers on campus is the carbon dioxide type (black container) which are good for general purposes, including oil and electrical fire.
- Some laboratories have dry powder fire extinguishers (blue container), which are good for chemical fire, including metal fire.
- Sand can be used to contain flammable liquid as well as put out a fire, including metal fire.
- Fire blanket can be used when someone's clothing catches fire.

**Evacuation procedures**

- Remain calm. Walk; do not run, especially when travelling on staircases.
- Immediately leave the building and go to the assembly point using the nearest exit.
- Try to help those who may have difficulties traveling such as disabled and pregnant persons.
- Donotusethelifts.
- Report to your Fire & Safety Officer at the assembly point as far as practicable.
- Do not return to the building until permission is given by the Fire Services Department Officer in charge of the scene.

**When clothing is on fire**

- Do not run
- Drop to the floor and roll to extinguish the fire.
- If fire blanket is available, wrap around body to help smother the fire.