(A*STAR, n.d.) (Asia Research News, n.d.)

Internship at A*STAR Institute of Chemical and Engineering Science

Done by Alyssa Png Kai Wen (S10192905)

(A*STAR, n.d.)

- Singapore's lead public sector R&D agency
- Nurturing talent and leaders
  - Their own research Institutes
  - The wider research community
  - Industry
- Aims to bridge the gap between academia and industry in terms of research and development

(Asia Research News, n.d.)

Institute of Chemical and Engineering Sciences

A*STAR

Carries out world-class research programs in chemistry and chemical engineering sciences

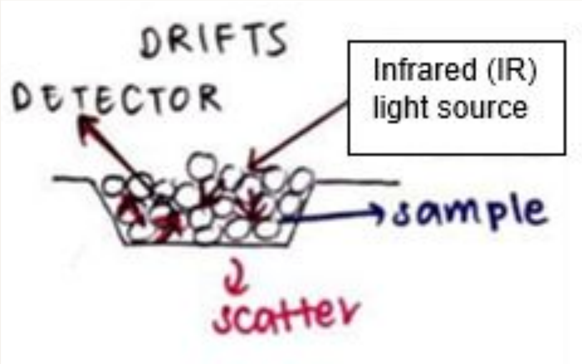
Department:

Process and Catalysis Research

02
DRIFTS

Diffuse Reflectance Infrared
Fourier-Transform Spectroscopy

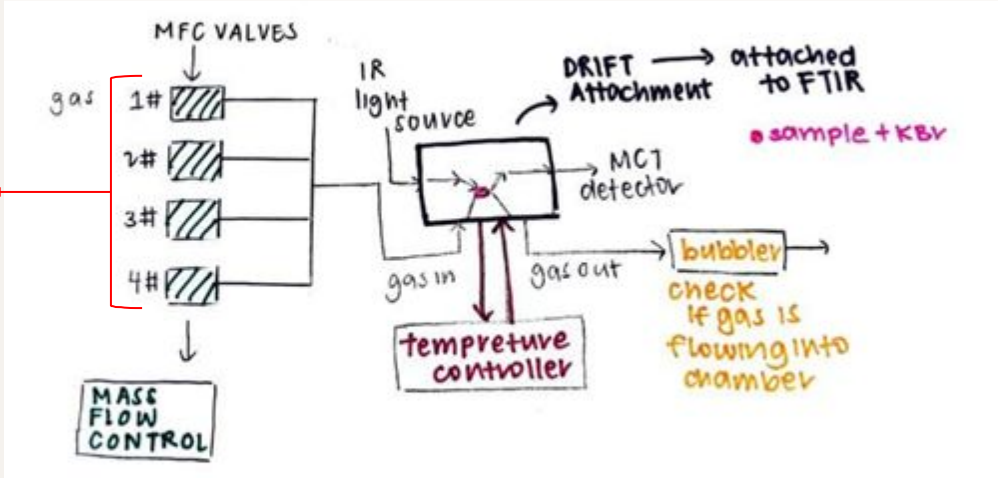
DRIFTS



- IR ray
  - Interacts with the particles
  - Reflect off their surface
- Measure the infrared spectrum
  - Observe the transformation of reactions occuring on the surface of the catalysts

(Geminibv, n.d.)

Manually control the flow rate of the gas reactants

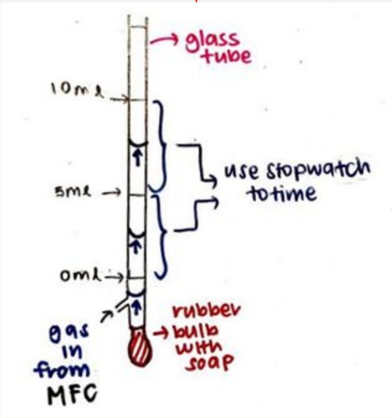


(Geminibv, n.d.)

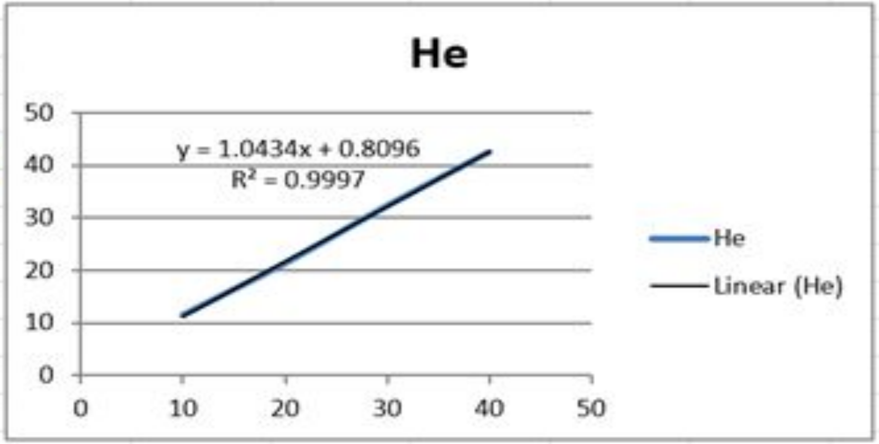
Testing accuracy of Mass Flow Controller (MFC)

Equation of the graph is utilised to find the actual flow rate of each set flow rate

- Used to minimise instrumental error
- Ensure the credibility of the experimental data

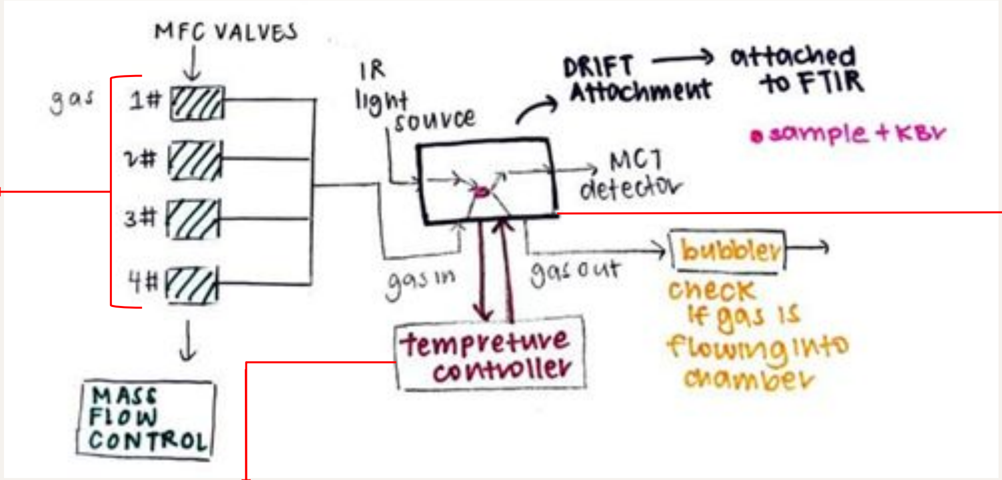


y=mx+c	
m	c
1.0434	0.8096
Set	Actual
30	32.1116



(Geminibv, n.d.)

Manually control the flow rate of the gas reactants



(Perkin Elmer, n.d.)

FTIR spectrometer

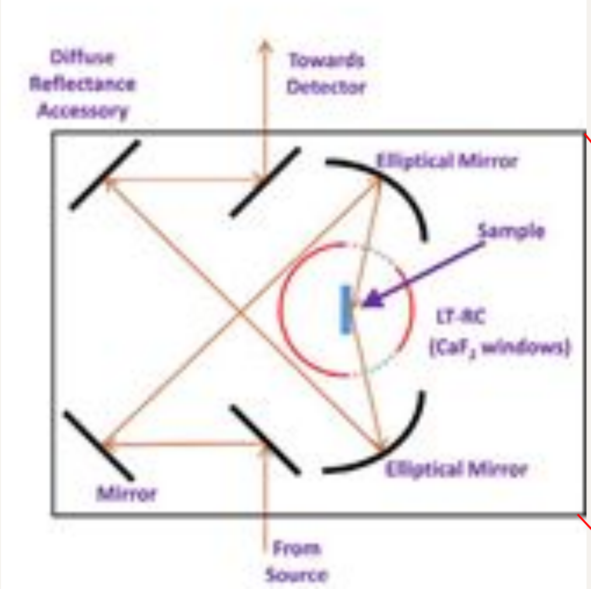
Maintain the temperature of the DRIFTS chamber at optimum

(Harrick Scientific Products, Inc., 2016)

DRIFTS Attachment

6 strategically placed mirrors

1. Direct IR light onto sample:
   a. Diffuse reflectance to occur
2. Reflect light from sample to detector

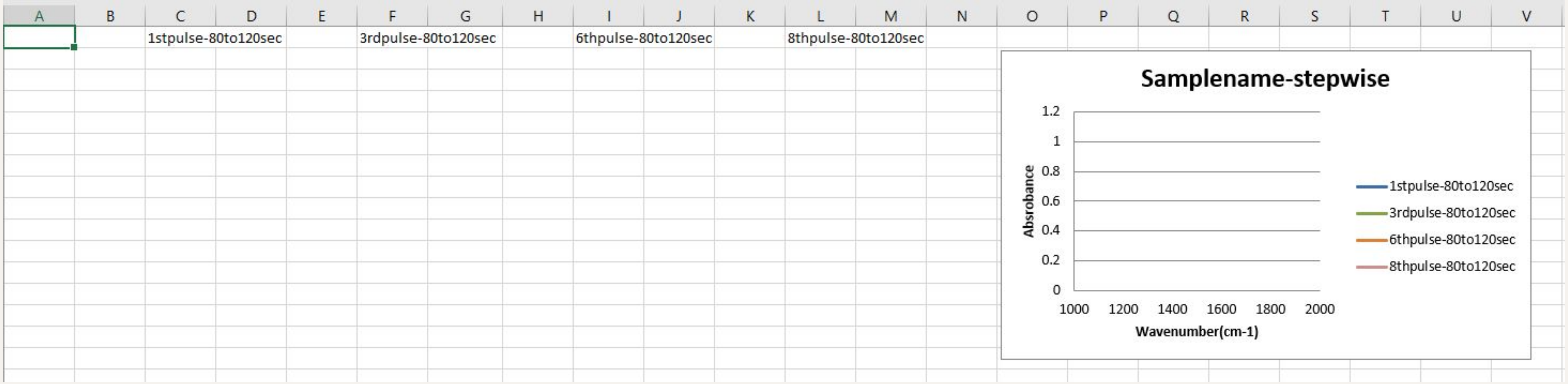


(Bu, et al., 2017)

(Perkin Elmer, n.d.)

(Thermo Fisher Scientific Logo, n.d.)

Plotting of graphs



To ease plotting of Graphs:

- Set up templates
- Copy data over and graph will be plotted automatically

Stepwise Pulsing

01 — 02 — 03 — 04

01 Pretreatment

1. Reduction of sample
2. Background spectrum

02 Absorption

1. Gas reactant A
2. One Spectrum taken after 30mins

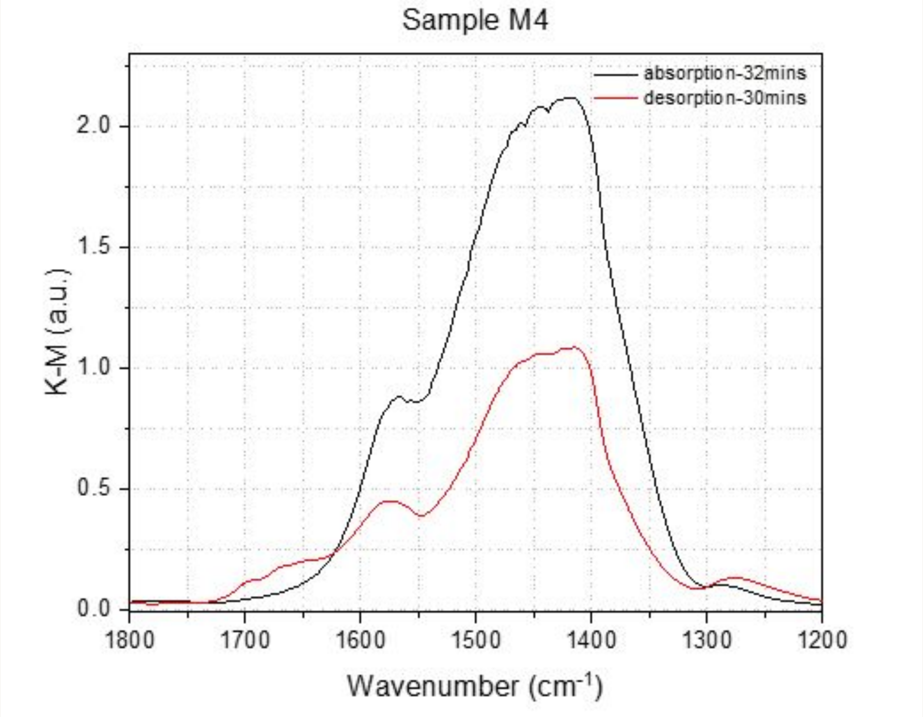
03 Desorption

1. Helium, inert gas
   a. Physically absorbed
2. Spectrum taken every 5 mins for 30 mins

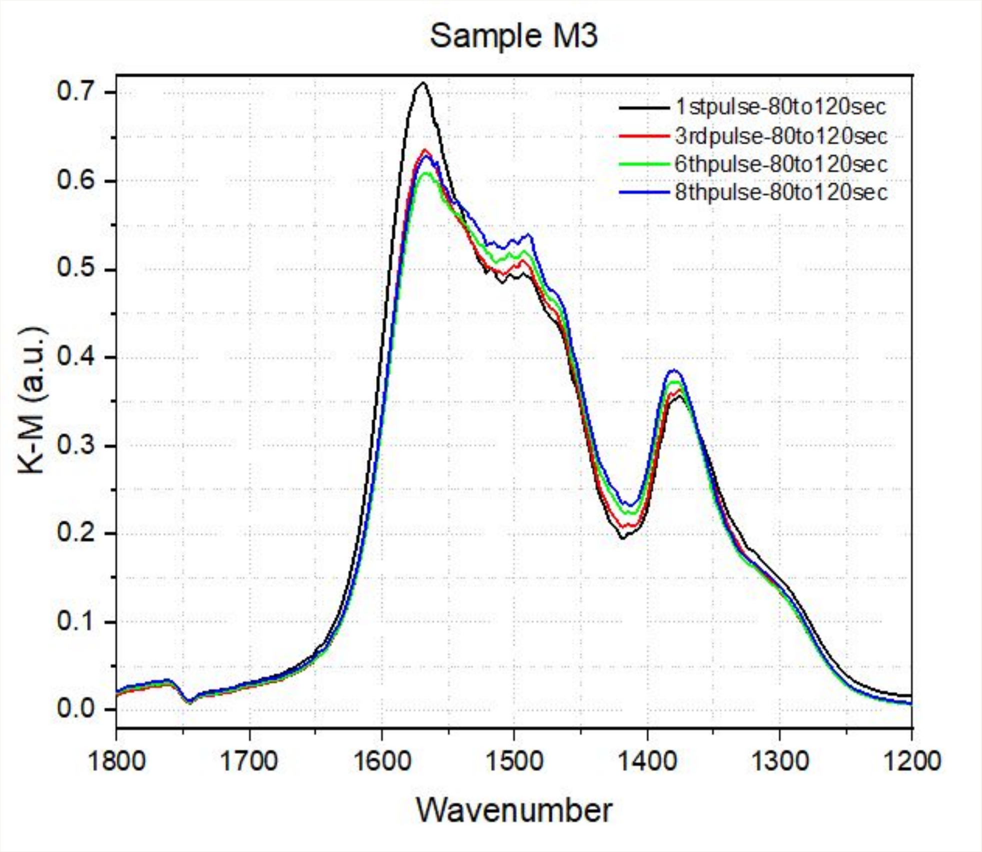
04 Pulsing

1. Gas Reactant B (10sec)
2. Desorption for 2 mins:
   a. 20,40,80,120 sec
3. 8th pulses

Stepwise: Absorption and Desorption peak



Stepwise: Pulsing



Stepwise Continuous Flow

01 —— 02 —— 03 —— 04

Pretreatment

1. Reduction of sample
2. Background spectrum

Absorption 1

1. Gas reactant A
2. One Spectrum taken after 30mins

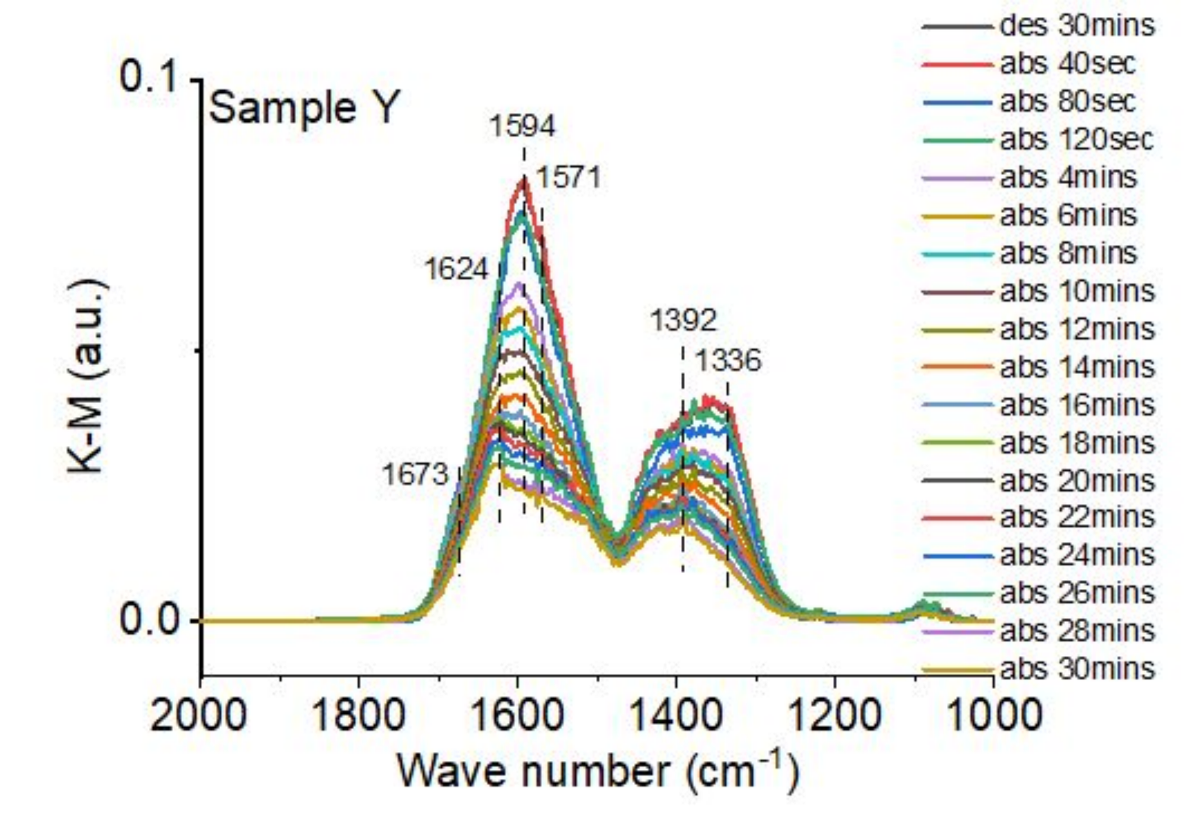
Desorption

1. Helium, inert gas
   a. Physically absorbed
2. Spectrum taken every 5 mins for 30 mins

Absorption 2

1. Gas Reactant B
2. First 2 mins:
   a. 40,80,120 sec
3. Every 2 mins for 30 mins

Stepwise: Continuous Flow



Mixed Gas

Pretreatment

01

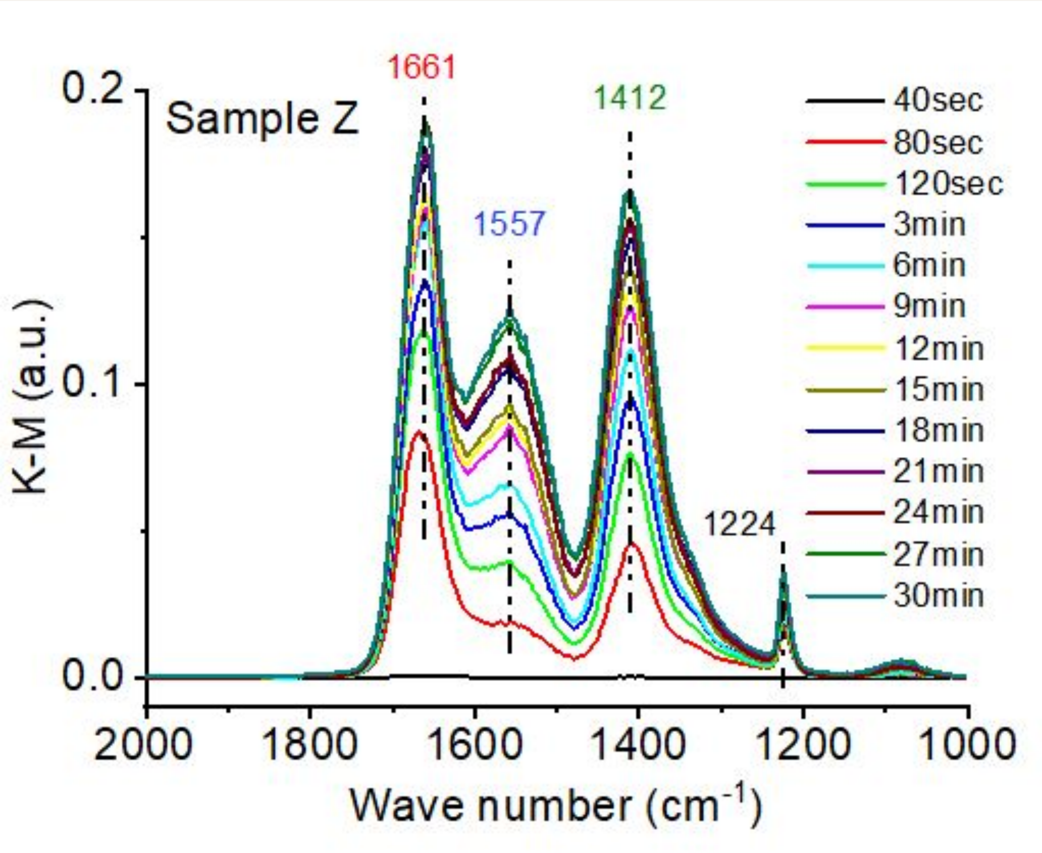
1. Reduction of sample
2. Background spectrum

Absorption

02

1. Mixed Gas:
   a. Same Flow rate for both Gas reactants
2. First 2 mins:
   a. 40, 80,120 sec
3. Every 3 mins for 30 mins

Mixed Gas

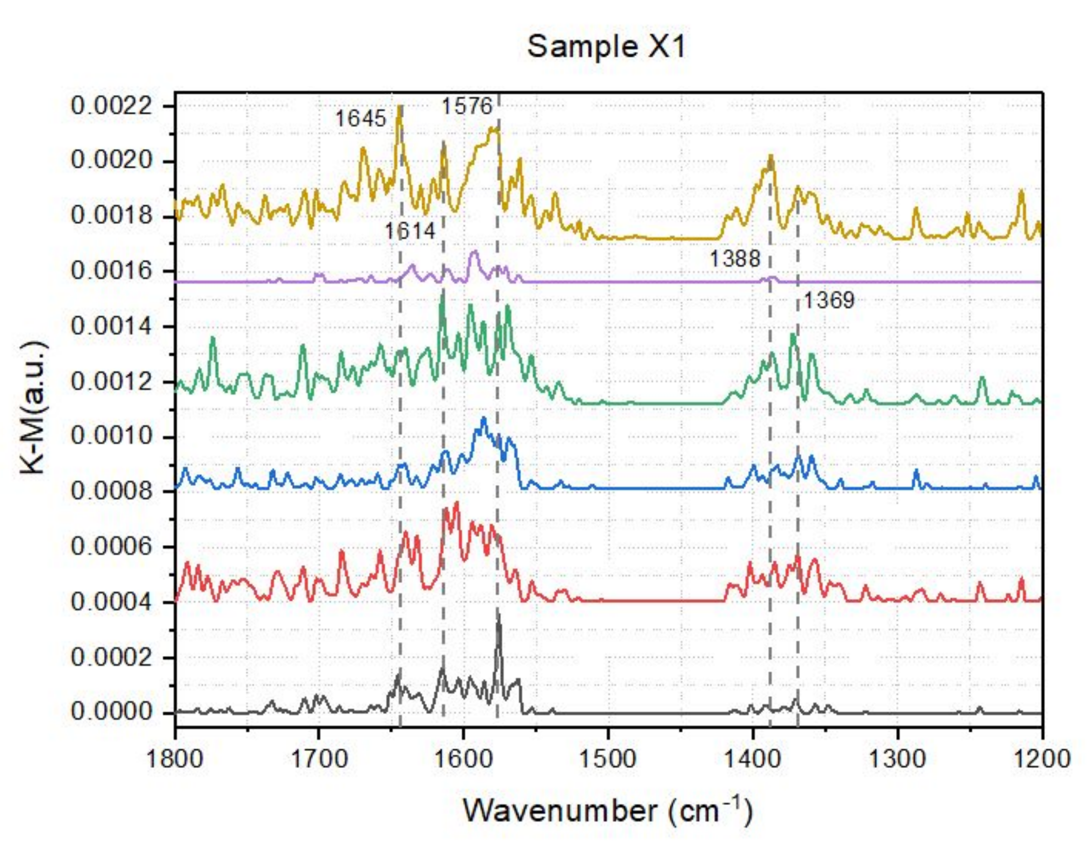


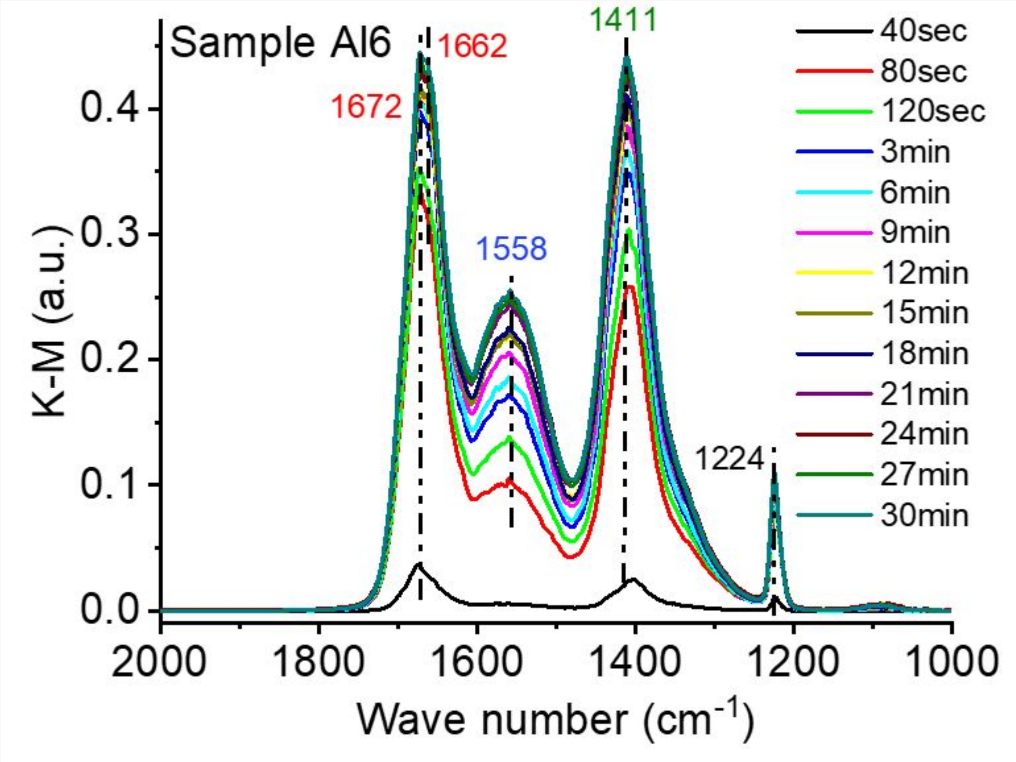
Sample Z
1661
1557
1412
1224
40sec
80sec
120sec
3min
6min
9min
12min
15min
18min
21min
24min
27min
30min
0.2
0.1
0.0
K-M (a.u.)
2000
1800
1600
1400
1200
1000
Wave number (cm-1)

DATA ANALYSIS

01: Low Intensity peaks

- Height <0.01
- Dark samples
  - Low diffuse reflectance occurrence
- Chemical Composition, Preparation method and KBr





02: Label peaks

Each peak represents respective species of intermediate or product

- Reference tables:
  - Matches the obtained peaks and reference based on wavelength
- Sample = product

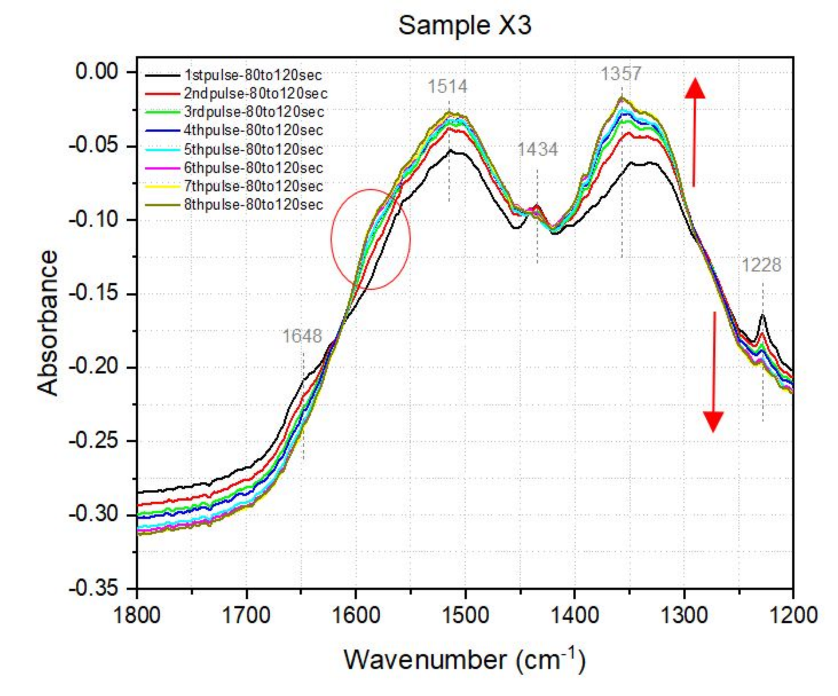
IR Absorptions of Common Functional Groups

Functional Group	*Absorption Location (cm^{-1})*	*Absorption Intensity*
Alkane (C–H)	2,850–2,975	Medium to strong
Alcohol (O–H)	3,400–3,700	Strong, broad
Alkene (C=C) (C=C–H)	1,640–1,680 3,020–3,100	Weak to medium Medium
Alkyne (C≡C) (C≡C–H)	 2,100–2,250 3,300	 Medium Strong
Nitrile (C≡N)	2,200–2,250	Medium
Aromatics	1,650–2,000	Weak
Amines (N–H)	3,300–3,350	Medium
Carbonyls (C=O) Aldehyde (CHO) Ketone (RCOR) Ester (RCOOR) Acid (RCOOH)	 1,720–1,740 1,715 1,735–1,750 1,700–1,725	Strong

(Winter, n.d.)

03: Understanding peaks

- Decrease in peaks:
  - Intermediate species converted to a product
- Increase in peaks:
  - May result from the decrease of another peak
- Shift in peaks:
  - Addition or deletion a new species
- Shoulders(circled):
  - Formation of a new product
- Overall shape of the spectra:
  - Samples with similar composition and structure
  - Determine the reliability and credibility of experiment data



03
CATALYST

Catalysts are foreign substances responsible for speeding up chemical reactions.

Calculations (Excel sheet)

Calculation a lot easier:

- Minimise calculation errors

Log in all previous calculations to refer to in the future

A	B	C	D	E	F	G	H
Chemical	MW	Volume			Elements	MW	
NaOH		250			H	1.00797	
Na2CO3		100			He	4.0026	
Urea					Li	6.941	
Ni(NO3)2					Be	9.01218	
Co(NO3)2					B	10.81	
Fe(NO3)3					C	12.011	
Mg(NO3)					N	14.0067	
Zn(NO3)2					O	15.9994	
Al(NO3)3					F	18.9984	
CeN3O9.					Ne	20.179	
CH3NO3					Na	22.9898	

"DATA" sheet stores:

- All the chemical and elements names and MW
- Volume of the various volumetric flasks used

Calculations (Excel sheet)

```
C3    =IFERROR(VLOOKUP(B3,DATA!$A$1:$B$201,MATCH("MW",DATA!$A$1:$B$1,FALSE),FALSE),"")
```

Solutions In Volumetric Flask

Solution names	Chemical	MolarWeight	Volume(ml)	Concentration	Mole	Mass(g)
		40	250	1	0.25	10
		105.99	100	0.15	0.015	1.58985
		375.13	100	0.5	0.05	18.7565
		403.9972	100	0.024	0.0024	0.96959
		290.8	100	0.16	0.016	4.6528
		195.1545	100	0.8277	0.08277	16.1529
		375.13	100	0.39	0.039	14.6301
		375.13	100	0.5	0.05	18.7565
		261.4799	100	0.2	0.02	5.2296
		291.03	100	0.12	0.012	3.49236

Catalyst (Solid Reactant)

Catalyst Name	Chemical	MolarWeight	Mole	Mass
		403.9972	0.00036	0.1474

Catalyst (Liquid Reactant)

Catalyst Name	Chemical	Concentration (M)	Mole	Volume (mL)
		0.009738	0.000025	2.56726227
		0.023	0.00000966	0.42

- Simple basic calculations
- Data validation feature
  - Selection of chemical names
- “VLOOKUP”
  - Find the chemical name in the given cell (J3) in the table given (A1:B201)
  - Print the corresponding MW (“MATCH”)

Calculations (Excel sheet)

Name of sample	Reactant A	Fixed mass of A	% of other reactant(s)	Total Mass of other reactant(s)
		2	2	0.040816327
		2	2	0.040816327

M2 =IFERROR(((NUMBERVALUE(LEFT(L2, FIND(":",L2)-1))*G2)/((NUMBERVALUE(LEFT(L2, FIND(":",L2)-1))*G2)+(NUMBERVALUE(RIGHT(L2,LEN(L2)-FIND(":",L2)))*J2)))*

	F	G	H	I	J	K	L	M	N	O	P
1	Reactant B	Molarweight	Conc of B	Reactant C	Molarweight	Conc of C	B:C	Mass of B	Vol. of B (ml)	Mass of C	Vol. of C (ml)
2		195.09	0.0095		58.9332	0.02	1:1	0.031347	16.91363397	0.009469	8.033976135
3		195.09	0.0095		58.9332	0.02	10:1	0.039619	21.37717411	0.001197	1.01541577

Calculations (Excel sheet)

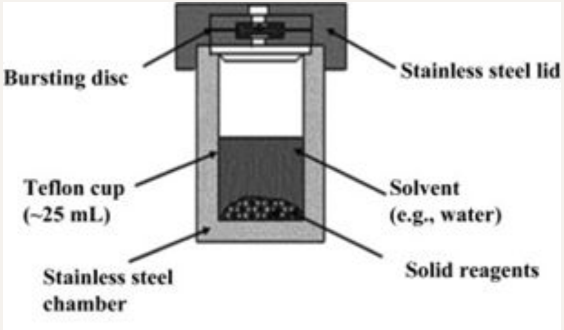
FIND()

- Determine the index of the “:” in the inputted string
- Ratio of B: “LEFT(L2, FIND(":",L2)-1)”
- Ratio of C: “RIGHT(L2, LEN(L2)-FIND(":",L2))

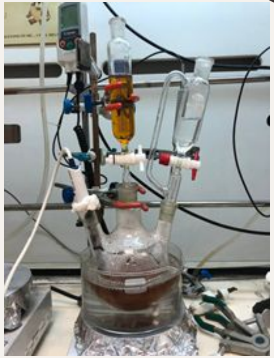
M2 =IFERROR(((NUMBERVALUE(LEFT(L2, FIND(":",L2)-1))*G2)/((NUMBERVALUE(LEFT(L2, FIND(":",L2)-1))*G2)+(NUMBERVALUE(RIGHT(L2,LEN(L2)-FIND(":",L2)))*J2)))*

	F	G	H	I	J	K	L	M	N	O	P
1	Reactant B	Molarweight	Conc of B	Reactant C	Molarweight	Conc of C	B:C	Mass of B	Vol. of B (ml)	Mass of C	Vol. of C (ml)
2		195.09	0.0095		58.9332	0.02	1:1	0.031347	16.91363397	0.009469	8.033976135
3		195.09	0.0095		58.9332	0.02	10:1	0.039619	21.37717411	0.001197	1.01541577
4											

Preparation Methods



(Kaflé, 2019)



Impregnation

Solid sample to fully absorb a liquid substance

Hydrothermal

Obtain solid crystals

- high-temperature aqueous solutions
- high vapour pressure

Precipitation

Insoluble solid

- two or more aqueous solutions

Collection Methods

(ProfiLab24.com, n.d.)

Centrifugation

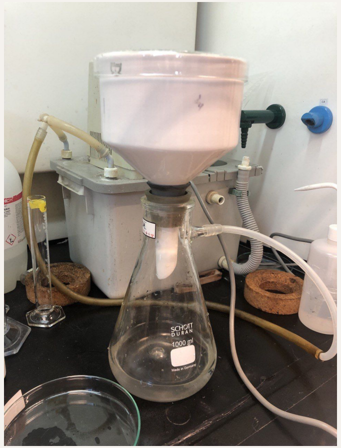
- Balance tube = Sample tube
- Rotor spins
  - Centrifugal force applied onto each particle

Rotary Evaporator

Efficient removal of the solvent via evaporation

- Lower boiling point
- Rotation

(Profilab24, n.d.)



Filtration

- Vacuum pump
  - Draw air within the conical flask
- Solvent is suction through the filter paper

Testing Catalytic Performance

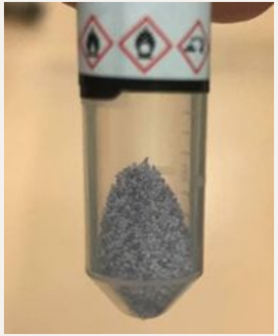
(Mineral Innovative Technologies, n.d.)

(Science First, 2011)

01

Particle size

- Hydraulic Press
  - Compress the powder
    - Increase particle size
- Crushed large flakes
- Strainer
  - Different Grid size

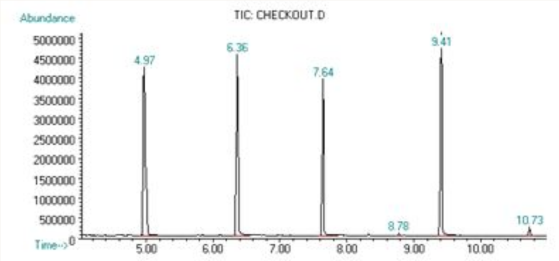


02

Glass Tube

- Mark center of reactor
- Create a fixed bed to support sample
  - Fine wool

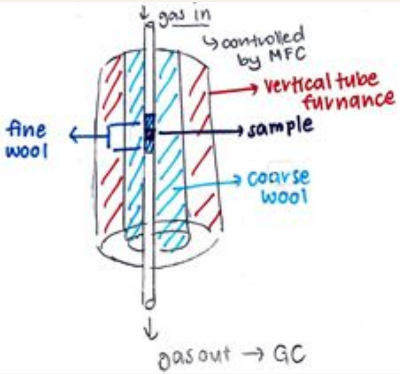
Testing Catalytic Performance



(George Mason University , 1998)

04 Gas Chromatography

- Chromatograph
  - Identification of peaks
  - Area of each peak:
    - Concentration
- Compared between
  - different temperature
  - With and without
    - Higher yield of desire products

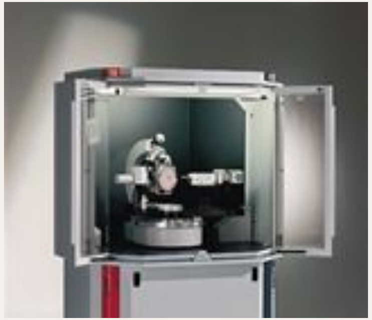


03 Reactor

- Gas reactant was introduced
  - Interact with catalyst
- Vertical tube furnace
  - Reaction occurs at different temperature

Chemical Composition

(Geochemical Instrumentation and Analysis, n.d.)



X-ray diffraction analysis (XRD)

(Bruker, n.d.)

X-ray Fluorescence analysis (XRF)

(Technology Networks, 2020)

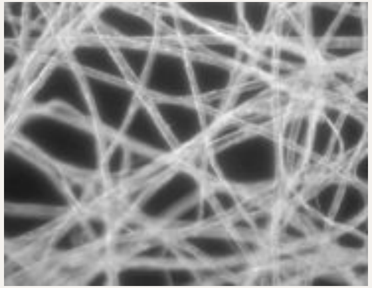
Inductively coupled plasma-optical emission (ICP-OES)

Nuclear Magnetic Resonance (NMR) spectrometer

(Bruker, n.d.)

Scanning Electron Microscope (SEM)

(Earth Observatory of Singapore, n.d.)



Transmission Electron Microscope (TEM)

(Gaston & Le, 2020)

04

Conclusion & Reflection

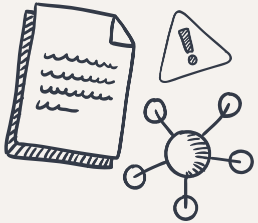
What it takes to be in Research?

Creative and innovative

- Think outside of the box
- Finding the best solution
    - Trials and errors
- Problem-solvers
    - Improve lives

Learning from Failures

- Encounter more failure than success
- Reading other scientist research papers
- Lead to new discoveries:
    - No guarantee outcome



Data analysis

- Essential for the improvement of the project
- Bring about credibility to the research
    - Clear diagrams
    - Convey the message of the research

Lesson Learnt

1. **Contributions depends on me**
    a. Take initiative
        i. Ask questions
        ii. Help out in any way
    b. Take responsibility
        i. Improve knowledge on experiments
        ii. Provide quality work by the end of the date line
2. **Independence**
    a. Conducting multiple experiments
        i. Time-management
    b. Troubleshoot
        i. Think on my toes
        ii. Make the most out of the resources i had
3. **Teamwork**
    a. Mutual respect
        i. Open minded
    b. Effective communication
        i. Tackling problems

Thank you for your kind attention

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