

NATIONAL UNIVERSITY OF SINGAPORE
FACULTY OF SCIENCE

Experiment-Based Risk Assessment Form

Name of Department	<u>Chemistry</u>	Location of Lab	<u>S7-04</u>
Name of Laboratory	<u>Advanced Chemistry Teaching Lab</u>	Name of PI (lecturer-in-charge)	<u>Dr. Chng Yong Sheng</u>
Name of Lab Officer	<u>Leng Zhi Jin, Wong Ling Rong</u>	Name of Activity/Experiment	<u>CM5176 GIST (Sem 1) Synthesis and Separation of Nitrophenols</u>

Hazard Identification				Risk Evaluation & Control						
No	Description / Details of Steps in Activity	Hazard(s)	Possible Accident(s) or ill Health, and Persons-at-Risk	Existing Risk Control (Mitigation)	Severity	Likelihood (probability)	Risk Level	Additional Risk Control	Person Responsible	By (Date)
1	4.55 mL 30% nitric acid (25 mmol, 2.0 eq) in a three neck round bottom flask with a thermometer and stirring bar was cooled to 0 °C in an ice bath.	nitric acid: volatile, corrosive	Burns to eyes and skin or burns to the respiratory tract.	No eating or drinking in lab, use in fume hood. Wear gloves, safety glasses, long pants, covered shoes and lab coat. Use in the fumehood. Glass thermometers, not metal thermocouples should be used	2	1	2			
		stirrer: electrical hazard	electrocution	Ensure there are no exposed wires present	2	1	2			
		broken glassware	Cuts from broken glassware	Check glassware before use	2	1	2			
2	1.17 g phenol (12.5 mmol, 1.0 eq) was added to the addition funnel and dissolved in 10 mL of distilled water (white emulsion). The phenol solution was added to the mixture slowly under stirring so that the temperature was always kept between 5-10 °C. The mixture was stirred for further 30 min at room temperature. Additional 20 mL distilled water were added, and the reaction mixture was cooled to 0 °C again.	phenol: toxic	Harmful if swallowed or inhaled or absorbed through skin	No eating or drinking in lab, use in fume hood	2	1	2			
		phenol: corrosive	Burns to eyes and skin	Wear gloves, safety glasses, long pants, covered shoes and lab coat.	2	1	2			
		broken glassware	Cuts from broken glassware	Check glassware before use	2	1	2			
3	The remaining product mixture was dissolved in 50 mL diethyl ether and transferred to a separatory funnel. 50 mL water was added, the mixture was shaken thoroughly with venting. The layers were allowed to separate and the aqueous layer was drained. Sodium sulfate was added to the organic layer. The drying agent was filtered off and washed with diethyl ether. The solvent of the filtrate was evaporated under reduced pressure on a rotary evaporator.	sodium sulfate: irritant	Irritating to eyes and skin	Wear gloves, safety glasses, long pants, covered shoes and lab coat	2	1	2			
		diethyl ether: toxic	Harmful if swallowed or inhaled	No eating or drinking in lab, use in fume hood	2	1	2			
		diethyl ether: irritant	Irritating to eyes and skin	Wear gloves, safety glasses, long pants, covered shoes and lab coat	2	1	2			
		diethyl ether: flammable	Causes fire	Keep away from open flames/heat sources	2	1	2			
		rotary evaporator: electrical hazard	electrocution	Ensure there are no exposed wires present	2	1	2			
		rotary evaporator: evacuated system	Potential implosion risk: cuts from broken glassware.	Inspect all glassware visually, do not use any with cracks	2	1	2			
		broken glassware	Cuts from broken glassware	Check glassware before use	2	1	2			
4	0.5 - 0.6 g crude product was dissolved in 2 mL ethyl acetate for column chromatography. For thin layer chromatography a diluted sample was prepared (ca. 2 mg crude product in 1 mL ethyl acetate).	ethyl acetate: irritant	Irritating to eyes and skin. Harmful if swallowed or inhaled	No eating or drinking in lab, use in fume hood. Wear gloves, safety glasses, long pants, covered shoes and lab coat	2	1	2			

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		ethyl acetate: flammable	May cause fire if heated	Keep away from open flames/heat sources	2	1	2			
5	A silica-coated TLC plate was labeled with a pencil so that it has three lanes. On the left lane, phenol is spotted (pinch of phenol dissolved in 2 mL ethyl acetate) and in between a mixture of phenol and the crude product. The TLC plate was developed in the developing chamber (development solvent: hexane/ethyl acetate = 7/3).	phenol: toxic	Harmful if swallowed or inhaled	No eating or drinking in lab, use in fume hood	2	1	2			
		phenol: corrosive	Burns to eyes and skin	Wear gloves, safety glasses, long pants, covered shoes and lab coat.	2	1	2			
		ethyl acetate: irritant	Irritating to eyes and skin	No eating or drinking in lab, use in fume hood. Wear gloves, safety glasses, long pants, covered shoes and lab coat	2	1	2			
		ethyl acetate: flammable	May cause fire if heated	Keep away from open flames/heat sources	2	1	2			
		hexane: toxic	Harmful if swallowed or inhaled	No eating or drinking in lab, use in fume hood	2	1	2			
		hexane: irritant	Irritating to eyes and skin	Wear gloves, safety glasses, long pants, covered shoes and lab coat	2	1	2			
		hexane: flammable	May cause fire if heated	Keep away from open flames/heat sources	2	1	2			
		Broken spotting glass tube	Cuts from broken glassware	Check spotter carefully before use do not bend spotting tube	2	1	2			
6	The column was filled silica gel (50-100g, 0.063-0.200 mm) to a height of 20-25 cm. The column was filled with the eluting solvent (hexane/ethyl acetate = 7/3) - silica gel mixture, total volume 300 mL.	silica gel: toxic	Harmful if swallowed or inhaled	No eating or drinking in lab, use in fume hood	2	1	2			
		silica gel: irritant	Irritating to eyes, skins and lungs	Wear gloves, safety glasses, long pants, covered shoes and lab coat	2	1	2			
		ethyl acetate: toxic	Harmful if swallowed or inhaled	No eating or drinking in lab, use in fume hood	2	1	2			
		ethyl acetate: irritant	Irritating to eyes and skin	Wear gloves, safety glasses, long pants, covered shoes and lab coat	2	1	2			
		ethyl acetate: flammable	May cause fire if heated	Keep away from open flames/heat sources	2	1	2			
		hexane: toxic	Harmful if swallowed, inhaled or absorbed through skin	No eating or drinking in lab, use in fume hood. Wear gloves, safety glasses, long pants, covered shoes and lab coat	2	1	2			
		hexane: irritant	Irritating to eyes and skin	Wear gloves, safety glasses, long pants, covered shoes and lab coat	2	1	2			
		hexane: flammable	May cause fire if heated	Keep away from open flames/heat sources	2	1	2			
		Broken glassware	Cuts from broken glassware	Check glassware before use. Do not use switch off the stopcock when using the air pump	2	1	2			
7	The fractions were collected in test tubes (10-15 mL each. The solvent (hexane/ethylacetate mixture) was removed under reduced pressure on a rotary evaporator.	rotary evaporator: electrical hazard	electrocution	Ensure there are no exposed wires present	2	1	2			
		rotary evaporator: evacuated system	Potential implosion risk: cuts from broken glassware.	Inspect all glassware visually, do not use any with cracks	2	1	2			
		Broken glassware	Cuts from broken glassware	Check glassware before use	2	1	2			

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8	The dry product is added to a NMR test tube and 0.4 mL of deuterated solvent (CDCl ₃).	CDCl ₃ : volatile, toxic, possible carcinogen	Harmful if swallowed, inhaled or absorbed through skin	No eating or drinking in lab, use in fume hood. Wear gloves, safety glasses, long	2	1	2			
		CDCl ₃ : irritant	Irritating to eyes and skin	Wear gloves, safety glasses, long pants, covered shoes and lab coat	2	1	2			
		Broken glassware	Cuts from broken glassware	Check glassware before use	2	1	2			

Conducted By


Name Dr. Chng Yong Sheng

Signature 

Date 1/6/2022

Approved By

Name Assoc Prof Yeo Boon Siang

Signature 

Approval date 1-Jun-22 Next Revision date 31-May-25

(Maximum 3 years)