**HAND OUT**

**INSTRUCTIONS LABORATORY
ORGANIC CHEMISTRY II**

****

**By :**

**Karim Th**

**DEPARTMENT OF CHEMISTRY EDUCATION**

FACULTY OF MATHEMATICS AND SCIENCE
YOGYAKARTA STATE UNIVERSITY

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**MEETING TO I
Safety in the Laboratory**

|  |  |
| --- | --- |
| Faculty / Study Program  | : Faculty of Mathematics and Science/  Chemical Education  |
| Courses & Code | : Practical Organic Chemistry II |
| Number of credits | : 1 credits |
| Semester and Time | : 4 and 2 x 50 x minute |

**A. Competency Standards**

After completing this subject students master the material safety in the laboratory

**B. Duties Prior to Practicum**

1. Mention the steps of safe working in the lab?.

**C. Learning Materials**

Before conducting the experiment in the organic chemistry laboratory praktikan should do the following:

* 1. open all the windows to keep fresh air circulating well
	2. turn on the blower if there is
	3. wearing lab coats
	4. should not move the chemicals from a place that has been provided

Once completed practicum then praktikan must do the following:

1. turn off all the electricity
2. turn off the faucet
3. close window
4. clean lab table
5. not dispose of solid waste in the water bath
6. cleaning the tools used in lab

7. restore the chemicals in the space provided

Work in the laboratory of organic chemistry has a huge risk to security, therefore praktikan required to understand and master some of the important things in working in the laboratory. Every experiment in the laboratory using chemicals and fittings as follows :

1. chemicals have the properties of explosive, flammable, corrosive and toxic

2. glassware is fragile and can be on a limb

3. reagents are highly reactive inorganic

* 1. power tools such as electric stoves, ovens, heating lamps, UV lamps, etc.
	2. water used for cooling a moment to stop the flow or leakage

ultra-violet radiation

The use of hazardous chemicals should be avoided, but if it should be done then the use of hazardous chemicals is made as small as possible or should be designed first experiment to be conducted. For example the use of materials, equipment, experimental conditions that are safe in doing the work. Experiments that use hazardous chemicals must be done in the cupboard acid.

To keep it in case of fire in the laboratory should be available fire extinguishers. But its use must be considered from any source of fire took place. If the fire comes from the chemicals can not be extinguished with water but must use a dry chemical or carbon dioxide.

Things that need attention to maintain the safety of laboratory work in organic chemistry is:

1. work carefully and really should not make jokes

2. should not eat and drink

3. keep your distance from the work of heating

4. chemical substances should not be heated using a tube directed to friends and or to the location of chemical

**D.** **Duties After Practice**

1. What needs to be done by praktikan to maintain the security work in laboratirium?.

**E. Bibliography**

1. Bansal Rajak, 1989, *Laboratory Manual in Organic Chemistry*, 18-20, Willey Estern Limited : New York

2. Johnson EL dan Stevenson R, 1977, *Basic Liquid Chromatography*, 17-19, Arian Associates Inc : California.

3. Michael P Doyle, William S Mungal, 1980, *Experimental Organic Chemistry*, John Wiley and Sons Inc : NewYork

**MEETING TO II
Know Your Ingredients Hazardous Chemicals**

|  |  |
| --- | --- |
| Faculty / Study Program  | : Faculty of Mathematics and Science /  Chemical Education |
| Courses & Code | : Practical Organic Chemistry II |
| Number of credits | : 1 credits |
| Semester and Time | : 4 and 2 x 50 x minute |

**A. Competency Standards**

After completing this subject students master the materialchemicals are dangerous in laboraorium organic chemistry.

**B. Duties Prior to Practicum**

1. Name five examples of flammablechemicals

2. What are two examples of chemicals that are toxic

**C. Learning Materials**

In the organic chemistry laboratory available hazardous chemicals,the use of hazardous chemicals should be avoided. But if in the experiment used the material so the process must be done in thecupboard acid. From the observation of each practicum waspraktikan less attention to the risks arising from use of thesechemicals. This is due to a lack of knowledge and understanding of the nature and types of chemicals that exist in the laboratory.

The nature and types of chemicals can be known from the legislationon  packaging and of hazardous materials, which determine the hazardous chemicals (hazardous chemicals), for example:

1. Corrosive: products of these chemicals affect the body's tissues and eye damage

2.Explosive: This product chemicals canexplode by heat andsparks., Shock or friction.

3. Oxidizing: the product of these chemicals can cause a fire, thesematerials generate heat when in contact with other organicmaterials.

4. Flammable: products of these chemicals have a "flash point" loweasily react with water or air to produce flammable gases, such asthe hydrogen from metal hydride

5. Harmful: products of these chemicals are irritants can cause burns to the skin and disrupt the respiratory system.

**D.** **Duties After Practice**

1.  Name five types of chemicals are flammable

2. What are the three chemicals that is explosive

**E. Bibliography**

1. Bansal Rajak, 1989, *Laboratory Manual in Organic Chemistry*, 18-20, Willey Estern Limited : New York

2. Johnson EL dan Stevenson R, 1977, *Basic Liquid Chromatography*, 17-19, Arian Associates Inc : California.

3. Michael P Doyle, William S Mungal, 1980, *Experimental Organic Chemistry*, John Wiley and Sons Inc : NewYork

**MEETING TO III
PRE TEST GENERAL**

|  |  |
| --- | --- |
| Faculty / Study Program  | : Faculty of Mathematics and Natural Sciences /  Chemical |
| Courses & Code | : Practical Organic Chemistry II |
| Number of credits | : 1 credits |
| Semester and Time | : 4 and 2 x 50 x minute |

A. **Competence**
Practicum can do with good organic chemistry II

B. **Student task**
Working on the pre test questions
C. **Material**
Laboratorium  materials are available on hand outs from an experiment I exsperiment VI.

**D. Bibliography**

1. Bansal Rajak, 1989, *Laboratory Manual in Organic Chemistry*, 18-20, Willey Estern Limited : New York

2. Johnson EL dan Stevenson R, 1977, *Basic Liquid Chromatography*, 17-19, Arian Associates Inc : California.

3. Michael P Doyle, William S Mungal, 1980, *Experimental Organic Chemistry*, John Wiley and Sons Inc : NewYork

**MEETING TO IV
EXPERIMENT I
IDENTIFICATION OF ORGANIC COMPOUNDS**

**A. Competence**

1.Identify alkane compounds, alkyl halides, alcohols, phenols,aldehydes and ketones..

2. Having the ability to identify some organic compounds.

**B. Duties Prior to Practicum**

1. Studying the physical and chemical properties of chemicals used in the reaction identifikaksi.

2. Studying the reactions of molecules and mechanisms of reactions that occur in the identification of organic compounds.

**C. Theory**

The structure of an organic compound can be identified using a chemical reaction that occurs in organic compounds. Organic reactions that occur depend on the functional group of organic compounds. Because of the changes that occur due to the ongoing reaction that determined by type of group function. Therefore, identification of organic compounds to test each compound has a particular characteristic. This means any organic compound can be identified using specific chemical reagents.
Some examples of reactions of organic compounds identified were:
1. Identification of Compounds alkenes
a). Reagent bromine in carbon tetra chloride.
Reagent bromine in carbon tetra chloride Br2/CCl4 red brown, his reaction to alkenes are addition reactions. Bromine heterolisis split into Br + and Br-alkene π electrons then strike Br + ions form a cyclic bromonium. Ion is attacked by nukleoofil Br-form colorless reaction products are compounds 1,2 - dibromo alkene.
Molecular reaction is as follows:



Mechanism reaction :







b). Potassium Permanganate reagent in alkaline
Oxidation reactions of alkenes using KMnO4 in alkaline oxidant that berwrna identification because purple is the reaction after the reaction, occurring divalent alcohol products, as a colorless solution.
Molecular reaction is as



 2. Identification of Alkyl halide compounds
Compounds alkyl halide or halogen senywa has the formula RX with R = alkyl or aryl and X is halogen (F, Cl, Br and I). If R = alkyl is an alkyl halide alifatis and if R = aryl / phenyl is an alkyl halide aromatic. Alkyl halide compound functional group is - X (- F, - Cl, - Br, - I). Functional groups using reagents can diidenntifikasi perag nitrate. Idetifikasi reaction is for example are:



If the halogen X is Cl in the compounds R - X will happen a white precipitate, if X = Br will happen yellowish white precipitate, and if X = I will place a yellow precipitate.

3. Identification of Compounds Alcohol
Alcohol has the formula R-OH when R = alkyl alcohol is alifatis and if R = aryl is an aromatic alcohol. Alcohol alifatis can be identified using chromic acid reagent. Primary alcohols with chromic acid will occur aldehydes and ketones secondary alcohol will occur. . While the aromatic alcohol can be identified using 5% FeCl3 reagent.
4. Identification of Compounds Aldehydes
Aldehyde has the formula R - COH can be identified using Fehling reagent, Benedict's reagent and reagent Tollen.s. Fehling and Benedict's reagent with aldehyde compounds at the end of the reaction will occur Cu2O precipitate the red brick. While reagents Tollen.s at the end of the silver mirror reaction will occur. Fehling reagent Fehling differentiated into A which consists of a solution of CuSO4 and. NaOH, while Fehling B solution consisting of sodium potassium tartrate together with Benedict's reagent Fehling reagent Benedict used the difference in citric acid instead of potassium sodium tartrate solution. Aldehyde reaction with Fehling reagent as follows:







Reagents Tollen, s is a solution of silver diamine which can be made from a solution of AgNO3 added a solution of NH4OH. Aldehydes with the reagent Tollen, s will form a silver mirror: The reaction is as follows:



5. Identification of Compounds Ketones
Ketone has the formula R - CO - R can be identified using the reagent 2,4 - dinitro fenilhidrazin. The reaction was positive if a yellow precipitate of compound terbenntuk osazon



6. Identification of Phenol Compounds
Phenol compounds are aromatic alcohol compounds, can be identified using FeCl3 Ragen 5% will occur komnplek compounds that bermerah, blue green or purple. For the phenol class of compounds of natural materials usually memnerikan violet color.



**D. Tools and Materials.**
tool:
1. Test tube rack
2. 10 test tubes
3. beaker
4. pipette
5. water bath
6. Bunsen heater

material:
1. samples
2. carbon tetrachloride
3. 2% bromine
4. silver nitrate in ethanol (saturated)
5. acetone
6. chromic acid (5 g CrO3 15 ml of water)
7. sulfuric acid
8. ethanol 95%
9. 2,4-dinitrophenylhydrazine
10. aquades
11. feriklorida 5%

**E. Learning Experience**
1. Compounds alkenes: Into a test tube 0.4 g input or 4 drops of sample in 1-2 ml of CCl4 add two drops of bromine solution of 2%. Later shaken if the color of bromine disappeared continue the addition of bromine solution to obtain a permanent color.
2. Alkyl halide compounds: Into a test tube mix 0, 4 g or ml of sample with 4 drops of silver nitrate solution in alcohol. Then the solution was shaken and left for 2 minutes, then observed whether the formed precipitate. If no precipitate, the solution was heated using a water bath until boiling, observe what happens sediment.
3. Alcohol: Into a test tube dissolve 1 drop of sample entries in 1 mL acetone and add 1 drop of chromic acid, then added 5 ml of concentrated sulfuric acid. Observe the orange color of chromic acid will be changed to bluish green. If the sample is used primary and secondary alcohols will form a precipitate.
4. Compounds Aldehydes: Into a test tube input 2 mL 95% ethanol into a test tube and add 2 drops of sample. Add reagent Fehling A and B then heated it happens akaan brick red precipitate.

5. Compounds Ketones: Into a test tube input 2 mL 95% ethanol into a test tube and add 2 drops of sample. Add the reagent 2,4-dintitrofenilhidrazin, kemdian whipped yellow precipitate will occur.
7. Phenol compounds: Put 1 drop of sample into a test tube and add 1 mL aquades (if it does not dissolve the aqudes replaced ethanol). Then add 1 drop of reagent feriklorida 5% and dogojok. Test is positif if the reaction terjadi color.

**F. Duties After Practice**
1. Give five molecular structures for each alkene compounds, alkyl halides, alcohols, aldehydes, ketones and aromatic alcohol.
2. Write the reaction of molecules that are involved in all these experiments.

**G. Bibliography**

1. Bansal Rajak, 1989, *Laboratory Manual in Organic Chemistry*, 18-20, Willey Estern Limited : New York

2. Johnson EL dan Stevenson R, 1977, *Basic Liquid Chromatography*, 17-19, Arian Associates Inc : California.

3. Michael P Doyle, William S Mungal, 1980, *Experimental Organic Chemistry*, John Wiley and Sons Inc : NewYork

 MEETING TO V

 EXPERIMENT II
 THIN LAYER CHROMATOGRAPHY

**A. Competence**
1. Making solvent / eluent suitable for compounds that are identified
2. Determining the price of Rf compound diidentifikaksi
3. Have the ability and proficiency in the separation and purification using thin-layer chromatography.

**B. Duties Prior to Practicum**
1. Preparing samples of natural materials
2. Studying the creation of natural extracts specified bhan
3. Mempelarai properties of some organic solvents
4. Study the usefulness of TLC

**C. Theory**
Analysis of thin-layer chromatography (TLC) is based on the distribution of liquid-solid phase where the solid phase or the absorbent in the form of a thin layer made of silica gel or alumina coated on a sheet of glass or plastic., As the liquid phase is the eluent used for the analyzed compounds merambatkan move through the solid phase. Properties of the absorber is the size of the particles and the homogeneity. Large particles commonly used are 1-25 microns. Particles of coarse grains will not provide a good separation of fine particles while providing a good separation as the eluent flow faster. Absorbers are widely used in TLC analysis are silica (SiO2) x and alumina (Al2O3) x. Silica is used given the binder (binder) which allows the calcium sulfate layer to be strong and adding to its adhesion to the glass backer. Silica like this in the trade were coded silica gel G. Alumina is widely used in TLC and in the trade of a layer of alumina can be either acidic or alkaline. Therefore, alumina for the separation of aromatic hydrocarbon compounds.
For the mobile phase usually digun will have a mixture of organic eluent polarity as low as possible so that the absorption components of the mixture can be reduced, eluent and sample propagates through the stationary phase and mobile phase interacts with the absorber. The main factor that causes the interaction is the polarity of the mobile phase and polarity of the compounds analyzed.. Eluent of high polarity can be derived by mixing the eluent is low polarity. The polarity of the eluent can be varied depending on the absorber and the samples analyzed, to create variations eluan note elution strength parameter (εo) in order to have a mixture of eluent having a polarity in accordance with keiinginan, elution strength of the eluent can be seen sepertri below.

|  |  |  |
| --- | --- | --- |
| NO | ELUEN | εo |
| 01. | n – pentana | 0,00 |
| 02. | Isooktana | 0,01 |
| 03. | Sikloheksana | 0,04 |
| 04. | carbontetrachlorida | 0,18 |
| 05. | chsilena | 0,28 |
| 06. | Toluen | 0,29 |
| 07. | Benzena | 0,32 |
| 08. | Ethyleter | 0,38 |
| 09. | chloroform | 0,40 |
| 10. | Ethylasetat | 0,58 |
| 11. | Ethanol | 0,88 |
| 12. | Methanol | 0,95 |

Movement (how quickly) the sample travels influenced by several factors: 1). chemical structure of the compounds analyzed, 2). Eluent, eluent used must have high purity, if the eluent used a mixture of some of the comparisons the eluent mixture must be precise. 3). Absorbent, absorbent activity can be achieved by eliminating water molecules that occupy the center of uptake of the absorber. Absorber will give you the price difference Rf (retordation factor) are different despite using the same eluent.



Rf eg price calculation is:



Rf senyawa A = 1,5 Cm / 6 Cm = 0,25

Rf senyawa B = 4,7 Cm/6 Cm = 0,78

**D. Tools and Materials**
tool:
1. Chamber
2. TLC plate
3. propipet
4. measuring pipette 1 mL
5. measuring pipette 5 mL
6. UV Lmpu
7. test tube
8. mortar
9. erlenmenyer 25 mL
10. 25 mL glass beaker
11. capillary pipe
12. spirit lamp
13. filter paper
14. glass funnel
material:
1. samples
2. methanol
3. dichloromethane
4. hexane
5. Chloroform

**E. Learning Experience**
1. Creating a micro pipette from the capillary tube, the way the capillary tube is held both ends and then trap the spirit of fire as he pulled. After breaking the capillary tube into two and then cut the ends to make a hole.
2. Preparing samples, natural ingredients such as green leaves, turmeric, flowers and so on. mashed with a mortar and then dissolved in a little methanol. Soak briefly and then filtered to separate the filtrate.
3. Prepare the TLC plate measuring 1 x 8 cm, with a pencil line the top and bottom with a distance of 1 cm from the edges.

 

4. Take samples with a micro pipette and totolksn TLC plate right below the middle line, then allowed to stand for 1-2 minutes to allow the solvent to evaporate.



5. Make mixture eluent with dichloromethane: hexane or hexane: chloroform with some comparative volume of 5 mL.
1. Enter one kind of solvent into the chamber and make saturated by entering paper then strain into a solution of tutp chambernya then propagate for 1-2 minutes

1. Put the TLC plate into the chamber and carefully, underlining that there should not be submerged eluent sample. Wait until there eluent bergarak up and henntikan after the eluent reaches the top.
2. Take a TLC plate and set aside to dry and then observe the stain on a TLC plate using UV light and calculate the price of its Rf.

3. Repeat the above procedure using another eluent.

**F. Duties After Practice**
1. How can you make a good eluent in the separation?.
2. Why are stains that form on the TLC plate as electronegative as berbentu tail round?.

**G. Bibliography**

1. Bansal Rajak, 1989, *Laboratory Manual in Organic Chemistry*, 18-20, Willey Estern Limited : New York

2. Johnson EL dan Stevenson R, 1977, *Basic Liquid Chromatography*, 17-19, Arian Associates Inc : California.

3. Michael P Doyle, William S Mungal, 1980, *Experimental Organic Chemistry*, John Wiley and Sons Inc : NewYork

**MEETING TO VI
EXPERIMENT III
EKTRAAKSI Soxhlet
ISOLATION OF SEEDS TRIMIRISTIN PALA**

**A. Competence**
1. Have the ability to separate organic compounds from natural materials
2. Calculate levels of nutmeg trimiristin
3. Have the competence to take the heat extraction and solvent extraction
**B. Duties Prior to Practicum**
1. Provides that have been mashed nutmeg
2. Menenttukan physical properties and molecular structure of compound trimiristin
3. Drawing a set of extraction tools sochlet
**C. theory**
A common way used for the separation of a mixture of organic compounds obtained from the reaction is the extraction of the liquid with liquid. The way the separation is based on comparison of the distribution of substances (samples) are dissolved in two solvents are not mutually dissolved. Comparison of solute in two solvents in the process of partitioning is called the distribution constant K, the balance at a specific temperature and state of the substance dissolved in a solution of the same substance and the distribution of solute A in solution 1 and solution 2 is formulated as follows:

 

If the weight of the solute = CE extracted using a solution whose volume is equal to the volume palarut and C0 is the weight of the substance before it is extracted, then:

CE = C0 ( K/K + 1 )

So for example, 2 parts of the solute can be dissolved in solvent ether and water and if the partition coefficient = 2 or 67% can be extracted using ether solvent then:

K = C eter /C H2O = 2/1 = 2

C eter = 100 ( 2 + 2 + 1 ) = 67 %

In the second extraction (extraction residue) extracted suppose there is 22% and the third extraction (extraction residue is extracted a second) there are 4% of the solute which can be seen in the ether layer then:

C2 eter = ( 100 – 67 )(2/3 ) = 22 %(

C3 eter = ( 100 – 67 – 22 ) (2/3 ) = 7 %

C H2O sesudah ekstraksi = ( 100 – 67 – 22 – 7 ) = 4 %

Extraction can be divided into cold extraction (solvent extraction) and heat extraction (Soxhlet extraction). Cold extraction performed unntuk liquid - liquid substances which are not mutually mixed and used in separating funnel. In this case must be considered agitation should be homogeneous. Heat extraction done to attract organic compounds contained in natural materials such as roots, stems, leaves, flowers and seeds, is using Soxhlet extraction.

Organic compounds found in natural materials such as trimiristin found in nutmeg. Trimiristin a substantial triglyceride esters contained in nutmeg (miristica fragrans). Trimiristin molecular formulas is C45H86O6. If there will be acid hydrolyzed trimiristin miristat and glycerol. Hydrolysis reaction can take place in a sam or alkali.

For Soxhlet extraction trimiristin used tools, nutmeg mashed and then wrapped using fat-free filter paper. Then inserted into a Soxhlet device. Tools associated with the Soxhlet flask filled with a solvent as much as 2 / 3 volume of the flask and then heated. At the time of heating the solvent will boil and steam will rise to the cooler and then steam mmengembun and will fall into the sample and Soxhlet meet trimiristin will dissolve. After the solvent memenuhia solvent will descend into the Soxhlet flask .. One round is called a circulating solvent. In general, extraction done as much, 8, 10, 12, 14 or 16 circulating solvent.

**D. Tools and Materials**
tool:
1. Soxhlet extraction of a set of tools
2. heater
3. glassware
4. Buchner filter
5. Buchi evaporator
6. means of determining the melting point

material
1. Finely powdered nutmeg
2. boiling stones
3. hexane
4. acetone
E. Learning Experience
1. Counting and sampling mmenimbang nutmeg as needed
2. Connecting the round flask which was given a stone pedestal boiling with Soxhlet extractor and input into the heater.
3. Enter the nutmeg seed samples that had been wrapped in soft fat-free filter paper into a soxhlet extractor.
4. Pour the solvent hexane in Soxhlet flask as much as 2 / 3 volume of the flask, then soxhlet associated with upright cooler.
5. Heat the mixture and do the extraction with 10 times the circulation.
6. After completion the mixture was filtered to reflux defecate, then evaporated.
7. The remaining hexane was dissolved in acetone, chill the mixture in ice to obtain ari-crystal. Pisahlan crystals using a Buchner filter and then dikerinkan.
8. Kkristal Weigh it and determine the melting point and purity.

**F. Duties After Practice**
1. Mention solvents other than hexane can be used for the isolation trimirisin
2. Soxhlet extraction for the separation of organic compounds from natural materials, whether daasr determination of the solvent.
3. why in the Soxhlet extraction is used coolant ball

**G. Bibliography**

1. Bansal Rajak, 1989, *Laboratory Manual in Organic Chemistry*, 18-20, Willey Estern Limited : New York

2. Johnson EL dan Stevenson R, 1977, *Basic Liquid Chromatography*, 17-19, Arian Associates Inc : California.

3. Michael P Doyle, William S Mungal, 1980, *Experimental Organic Chemistry*, John Wiley and Sons Inc : NewYork

MEETING TO VII
EXPERIMENT IV
ALDOL CONDENSATION CROSS

**A. competence**
1. Identify the physical properties and results raeksi between asetofenon benzaldehid
2. Calculate the yield-Claisen reaction between asetofenon and benzaldehid Scmidth
3. Have the ability to synthesize compounds kalkon
**B. Duties Prior to Practicum**
1. Determine the physical properties and function of materials used in the lab
2. Write down the chemical reactions that occur between asetofenon and benzaldehid
3. Determine the amount of each ingredient to be used if the desired results as much as 6.30 grams.
4. Determine the yield of the reaction.
**C. theory**
Condensation is a reaction in which two small molecules combine to form one large molecule with or without the loss of a small molecule. Misaalnya Claisen condensation of ethyl acetate which occurs in the presence of sodium etojsida in ethanol. Ά carbon atom of the ethyl acetate condenses with the carbonyl carbon of the ethyl acetate molecules to form carbon-carbon bonds are new.



Aldol condensation is a nucleophilic addition reaction of enolate ion to the carbonyl group with the reaction product β-hydroxy ketones or β-hydroxy aldehydes, enolate and a carbonyl group is attacked is the same two compounds. The reaction is called aldol condensation of aldehydes and because dituurunkan alcohol. Examples of aldol condensation reaction is the reaction between asetaldehid with an aqueous solution of sodium hydroxide to form enolate ions in low concentrations. Goes a reversible reaction, namely when the enolate ion reacts it will form another new enolate ion. Enolate ion reacts with another aldehyde molecule by mengadisi on the carbonyl carbon to form the alkoxide ion which then reacts with protons from water to produce the aldol product. Aldehydes which can condense with aldehydes aldol is Hά atom of the aldehyde carbonyl group that can form enolate ions in alkaline.



Condensation reaction between aldehydes or ketones with aldehyde or ketone carbonyl of the other so-called cross aldol condensation (cross aldol condensation). This reaction of aldehydes with no Hά atoms can not form enolate ions and so can not berdimerisasi in an aldol condensation. But if the aldehyde iitu mixed with another aldehyde or ketone having atomic Hά there will be a condensation of molecules of the two. \

Cross-aldol condensation reaction involving the use of aromatic aldehyde compounds and ketone compounds or alkyl aryl ketone as reactant is known as the Claisen-Schmidt reaction. This reaction involves the enolate ion of the ketone compounds that act as nukleofilyang will attack the carbonyl carbon of aromatic aldehyde compounds to produce β-hydroxy ketone compounds which further dehydrated compound ά, β-unsaturated ketones.

**D. Tools and Materials**
tool:
1. a set of tools reflux with three neck flask
2. magnetic stirrer
3. glassware
4. Buchner filter
5. digital scales
6. means of determining the melting point

**material**
1. NaOH
2. aquades
3. ethanol
4. asetofenon
5. Benzaldehid

**E. Learning Experience**
1. Mmenuliskan molecular reactions that occur
2. Calculating the amount of material needed if the desired results as much as 6.3 grams
3. Prepare and weigh in accordance with step number two
4. Preparing NaOH and ethanol in the mixture aquades into a three neck flask equipped with magnetic stirrer. The mixture was cooled with ice then asetofenon added into the mixture by stirring bertetes-drops. Stirring was continued for 2 hours at room temperature. After 2 hours the reaction was stopped and observed the results. If you see a precipitate immediately filtered using a Buchner filter. If camputan reactions that occur there is no visible precipitate then cooled in the refrigerator for 12 hours, then filtered and then dried.
5. Observe the color and weigh the precipitate that occurred then specify the melting point.

**F. Duties After Practice**
1. Why needed cooling the ball while the reaction does not require refrigeration
2. Why asetofenon added first beforebenzaldehid
3. Explain why the reaction does not require heating

**G. Bibliography**

1. Bansal Rajak, 1989, *Laboratory Manual in Organic Chemistry*, 18-20, Willey Estern Limited : New York

2. Johnson EL dan Stevenson R, 1977, *Basic Liquid Chromatography*, 17-19, Arian Associates Inc : California.

3. Michael P Doyle, William S Mungal, 1980, *Experimental Organic Chemistry*, John Wiley and Sons Inc : NewYork

4. William H. Brown, 1982, *Introduction to Organic Chemistry Third Edition*, Willard Grant Press : Boston Massachusetts

MEETING TO VIII
EXPERIMENT V
SYNTHESIS SALICYLIC ACID

**A. competence**

1. Identify the physical properties of the results raeksi salicylicacid synthesis

2. Calculate the yield of the reaction

3. Have the ability to synthesize organic compounds

**B. Duties Prior to Practicum**

1. Determine the physical properties and function of materialsused in the lab

2.Calculating the amount of material needed if you want tosynthesize salicylic acid 5 grams

3 . Draw a series of tools used

**C. theory**

R-COOR1 esters can be synthesized through the esterification reaction of carboxylic acids with alcohols using a catalyst of concentrated sulfuric acid. Example is the reaction between acetic acid with 1-propanol, propyl acetate will produce.



Esterification reaction is a reversible reaction and therefore formed ester can be hydrolyzed to form carboxylic acid and alcohol again. Ester hydrolysis reaction of carboxylic acid and alcohol into a reaction called saponification. Ester hydrolysis reactions can occur due to the influence of acid or alkaline, acid hydrolysis of esters under the influence of the reaction can be through several mechanisms tergazntung ester structure.

Ester methyl salicylate are contained in gondopuro oil, can be converted into salicylic acid in the presence of alkaline catalysts. . For example the reaction of propyl acetate hydrolysis as follows:



Methyl salicylate is a major component in gondopuro oil which is about 96-99%., The other component is a paraffin, alcohols, aldehydes and ketones. Hydrolysis reaction of methyl salicylate is as follows:



Salicylic acid has a relative molecular 138.12 as a crystalline compound with lelih point 159 ° C, the heating will decompose into phenol and carbon dioxide.

**D. Tools and Materials**
tool:
1. a set of tools reflux
2. measuring cup
3. a set of tools recrystallization
4. 250-ml glass beaker
5. Buchner filter
6. means of determining the melting point
7. filter paper
material
8. 5 M NaOH
9. 5 M sulfuric acid
10. gondopuro oil

**E. Learning Experience**
1. Write the reaction mechanism of methyl acetate ester hydrolysis
2. Calculating the amount of material needed if you want to synthesize salicylic acid 5 grams
3. A number of Noh has been calculated on the weight, put into 250 ml of Labo, which had been equipped with air through then add the oil in bertetes gondopuro-drops. Mixture refluxed for 30 minutes or until all solids dissolved. Then cooled to room temperature is reached the next 5 M sulfuric acid plus bertetes-drops while stirring. The addition of sulfuric acid was stopped after the deposition. The precipitate that happens is filtered using a Buchner filter then dried.
4. The precipitate which has been dried and then weighed and observed color, odor, determine the melting point and calculate rendemennya.
**F. Duties After Practice**
Write down the hydrolysis reaction of methyl salicylate and salicylic acid yield count that occurred

**G. Bibliography**

1. Bansal Rajak, 1989, *Laboratory Manual in Organic Chemistry*, 18-20, Willey Estern Limited : New York

2. Johnson EL dan Stevenson R, 1977, *Basic Liquid Chromatography*, 17-19, Arian Associates Inc : California.

3. Michael P Doyle, William S Mungal, 1980, *Experimental Organic Chemistry*, John Wiley and Sons Inc : NewYork

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MEETING TO IX
EXPERIMENT VI

STEREOCHEMICAL
COMPOUNDS WITH ONE ATOM C CHIRAL

**A. Competence**

1. Fischer projections of the compound with one chiral C atom
2. Identify the configuration R, S of the compound with one chiral C atom
3. Have the ability to create models of molecules

**B. Duties Prior to Practicum**
1. Make a model of the tetrahedral C atom has four different atoms of different materials everyday.
2. Studying the sequence of atoms or groups priorotas
3. Studying the writing of Fischer projection
4. Making models of Fischer projection

**C. theory**
Organic compounds that have carbon atoms bind to four different groups of atoms called C asimetrrs or chiral C atom. Such compounds would form two molecules that are mirror shadows that are not close to each other. Molecular compounds which are not close to each other are called enantiomers. Enantiomer derived from the Greek (Greek) of the enantio word (Opposite) and meros (part) Example: lactic acid and 2-methyl-1-butanol has a chiral C atom.
Write down the hydrolysis reaction of methyl salicylate and salicylic acid yield count that occurred

 

Fischer projection of Aton senyawaa which has one chiral C can be described as follows:



1. enclature (nomenclature) R-S is determined by the rules of Cham-Prelog-Ingold as follows:

2. ermine priority atom / group attached to the chiral C atom. A high priority - - the lowest priority D .1 - 2 - 3 - 4

2. The molecule viewed from the direction contrary to the atom / group has the lowest priority

3. When the group ABC has a clockwise sequence makaa compounds or chiral C atom has an R configuration (rekter = right and if BC has a sequence of counter-clockwise then the configuration S (sinester = left).

The order of priority clusters:

a). priority of fungsional group : F > Cl > SH> F > OH > NH2 > CH3 > H

b). ( CH3 )2 – CH – > CH3 – CH2 – CH2 –

c). CH3 – CH2 – > CH3 –

d). CH3O - > - OH

e). – OH > – CO2H > - CH3 > - H

Exsample :



Then the configuration of R is:



a). Latic acid



b). Gliseraldehid



**D. Tools and Materials**

1. two black balls

2. two red balls

3. two blue balls

4. two balls of gray

5. stem eight valence

6. eight ball with four colors

**E. Learning Experience**

1. Write priority atoms / clusters that have been determined

2. Taking one black ball (C sp3) and input four-bar into each holevalence.

3. Install the ball at each end of the valence as follows::

a). Red: for the priority to 1

b). Blue: for the priority to 2

c). Gray: for priority to 3

d). Without the ball: for priority to 4

The order of priority is determined by its atomic number isdecreased from the atom directly attached to the chiral C atom.When atoms are bound to the same chiral C atom, then thepriority is determined by the next atom.

1. Determine which compounds are the same and where the pair of enantiomers with the aid of an atomic model of the above as follows:







1. Determine the configuration R - S from each of thesestructures.

**F. Duties After Practice**

1. Draw Fischer R-S configuration of the glycine

2**.** Draw Fischer RS configuration of 2-methyl-1-butanol

**G. Bibliography**

1. Bansal Rajak, 1989, *Laboratory Manual in Organic Chemistry*, 18-20, Willey Estern Limited : New York

2. Johnson EL dan Stevenson R, 1977, *Basic Liquid Chromatography*, 17-19, Arian Associates Inc : California.

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