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Research Article

SYNTHESIS AND ANTI-OXIDANT ACTIVITY OF DIBENZALKETONES

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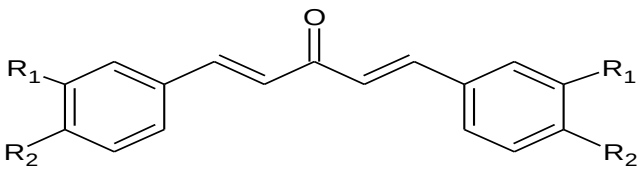
ABSTRACT

Antioxidant activity which plays key role for detecting the compounds whether they have affinity towards various types of cancer cells and other related chronic diseases. Hence we felt worthwhile to study in detail about the antioxidant activity of some synthesized compounds. As chalcones have numerous pharmacological actions towards various disease we focused on the α, β-unsaturated keto function in chalcones, which gives impetus to synthesize different Dibenzalketone derivatives and to test the compounds for antioxidant activity.

1. INTRODUCTION

Chalcone is an aromatic ketone that forms the central core for a variety of important biological compounds. Chalcone can be prepared by an aldol condensation between a aldehyde and an ketone in the presence of a catalyst. Aldol condensation is also known as Claisen-Schmidt rection[1].The aldol reaction is one of the most powerful methods available for forming a carbon-carbon bond[2]. In this reaction, the conjugate base of an aldehyde or ketone adds to the carbonyl group of another aldehyde or ketone to give a β-hydroxyaldehyde or β-hydroxyketone product.

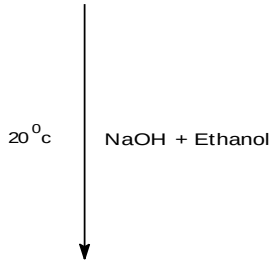
There are several methods available for the synthesis of chalcones. The most widely used is the base-catalyzed such as sodium hydroxide (NaOH)[3], potassium hydroxide (KOH)[4], barium hydroxide $Ba(OH)_2$[5], and lithium hydroxide ($LiOH.H_2O$)[6]. The acid-catalyzed that had been used to synthesize chalcones includes aluminum trichloride ($AlCl_3$)[7], dry HCl[8], boron trifluoride-etherate (BF_3-Et_2O)[9], titanium tetrachloride ($TiCl_4$)[10] and ruthenium trichloride ($RuCl_3$)[11]. Chalcones show antimicrobial[12], antimalarial[13], anticancer[14], antioxidant[15] and anti-inflammatory[16,] and antitubercular[17] properties.The presence of a reactive α, β-unsaturated keto function in chalcones was found to be responsible for their antimicrobial activity. Recently, more attention has been paid to the synthesis of α, α'-bis(substituted benzylidene) cycloalkanones[18,19] are known as the dibenzalketone derivatives. These derivatives can be prepared by the crossed aldol condensation of cycloalkanones with aldehydes and ketones. Different complexes of metal ions such as Mn(II), Fe(II), Co(II), Ni(II),cu(II) and Zn(II) with different ligands have been used for aldol condensation[20]. There are several methods available for the synthesis of dibenzalketones. Some of them are: Bis(p-methoxy-phenyl)telluroxide and KF-Al_2O_3 have been used for crossed-aldol condensation of cycloalkanones with aromatic aldehydes under microwave irradiation[21,22]. Anhydrous RuCl, and $TiCl_3(SO_3CF_3)$ have also been used for this purpose under solvent free conditions[23,24]. Dibenzalacetone has a conjugated system and is expected to be easilyoxidized[25].The more the double bond, the easier it will be oxidized. Therefore, it is assumed that Dibenzalacetone and its derivatives will show antimicrobial and antioxidant activity.

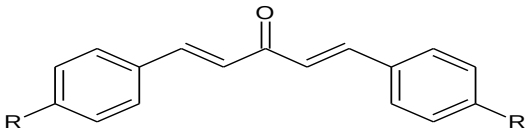


General Structure of Dibenzalketone

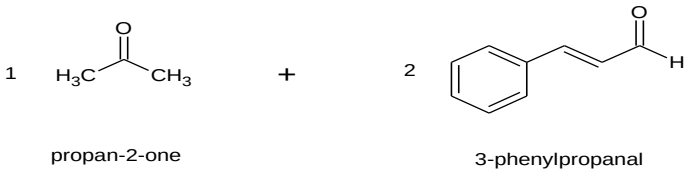
2. EXPERIMENT POSTULATION

propan-2-one benzaldehyde

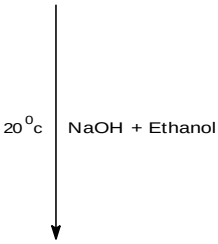


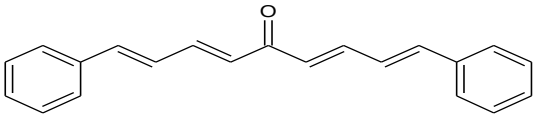


R = CHO-Benzaldehyde
R = Cl - 4- Chloro enzaldehyde
R = NO_2 - 4-Nitro benzaldehyde
R = MeO- 4-Methoxy benzaldehyde

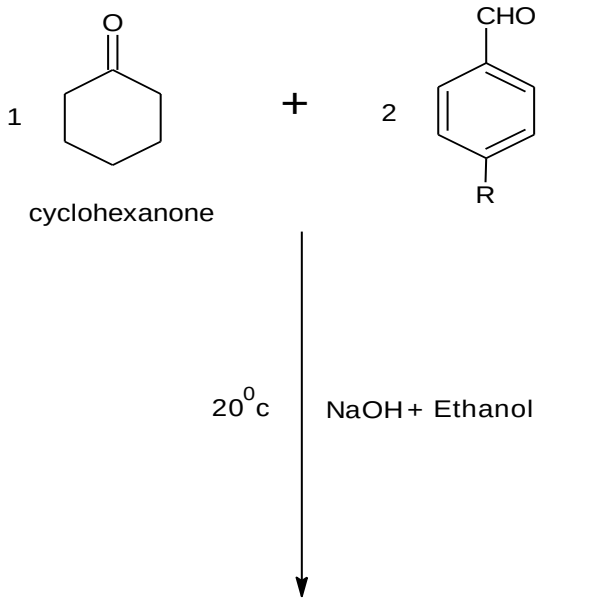


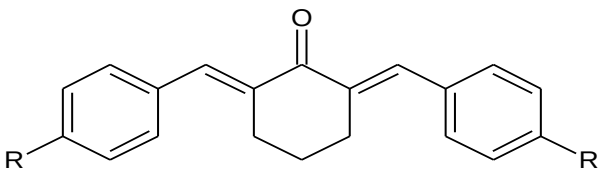
propan-2-one 3-phenylpropanal



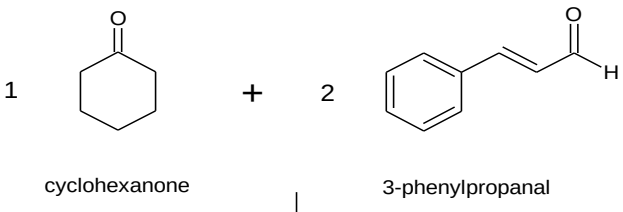


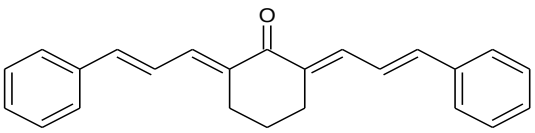
(1*E*,3*E*,6*E*,8*E*)-1,9-diphenylnona-1,3,6,8-tetraen-5-one





R = CHO-Benzaldehyde
R = Cl - 4-Chloro benzaldehyde
R = NO_2- 4-Nitro benzaldehyde
R = MeO- 4-Methoxy benzaldehyde



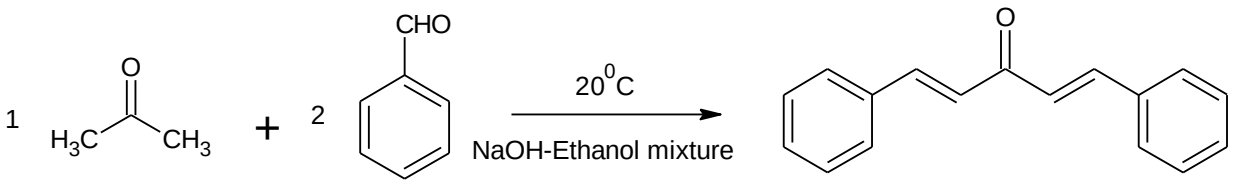


(2*E*,6*E*)-2,6-bis[(2*E*)-3-phenylprop-2-en-1-ylidene]cyclohexanone

3. EXPERIMENTAL SECTION

Preparation of 1,5-diphenylpenta1,4-dien-3-one

Reaction:



propan-2-one
M. F - C_3H_6O
M.W - 58

benzaldehyde
M.F -C_7H_6O
M.W – 106

(1*E*,4*E*)-1,5-diphenylpenta-1,4-dien-3-one
M.F - $C_{17}H_{14}O$
M.W - 234

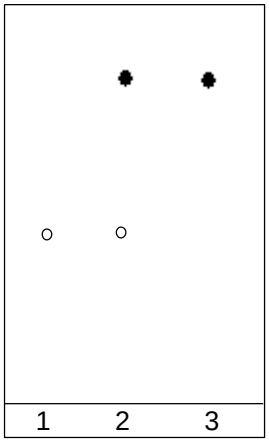
Procedure

Prepare an ice-water bath in a 250ml beaker, place 15ml of ethanol and 20ml of aqueous 10% NaOHinto a 100 ml beaker & add a stirbar. Place the 100ml beaker into the ice-water bath. Set then the entire assembly on to a magnetic stirrer. While stirring, cool the solution to 20°c. After the solution reaches to 20°c, remove the ice-water bath, continue to stir the solution.

Prepare the mixture of 2.1 ml (2 m mol) of fresh Benzaldehyde and 758 µl (1 m mol) of Acetone in a test tube over a period of 5-10 mins, add the Benzaldehyde-acetone mixture to the ethanol-NaOH solution in a small portions, then stir the reaction for another 30 mins. Cool the mixture using the ice water bath for one hour. collect the crystals by vaccum filtration. Wash the crystals by suspending them in 50ml of distil water. Again collect the crystals by vaccum filtration. Finally recrystalised with ethyl acetate.

Check the filtrate by testing the last few drops of water using red-litmus paper. If the litmus changes to blue, wash the crystals again until red-litmus does not changes colour. Keep a small sample aside to dry for a crude melting point measurement[56].

TLC studies



1: starting material (1)

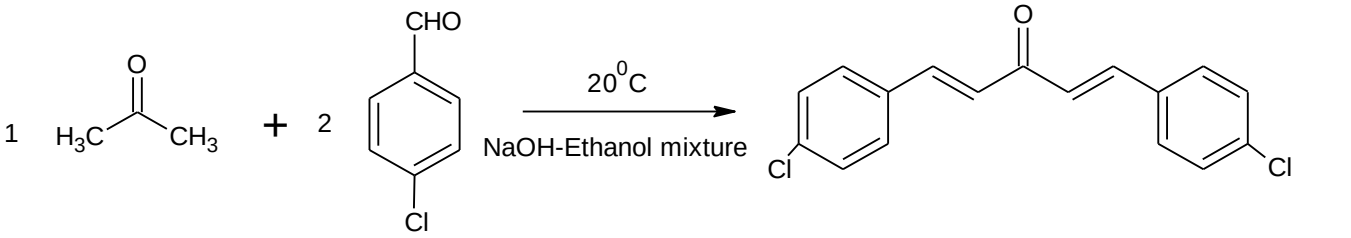
2: co-spot (1& 1a)

3: product (1a)

R_f values

Mobile phase :Benzene:Methanol (9:1)
R_f value of the product : 0.87
$R_f$value of the product : 0.91
Percentage yield : 82%

Preparation of 1,5-bis(4-chlorophenyl)penta-1,4-dien-3-one
Reaction



propan-2-one 4-chlorobenzaldehyde (1*E*,4*E*)-1,5-bis(4-chlorophenyl)penta-1,4-dien-3-one

M. F - C_3H_6O M.W - 58

M.F -C_5H_3ClO M.W -140

M.F - $C_{17}H_{12}Cl_2O$ M.W - 303

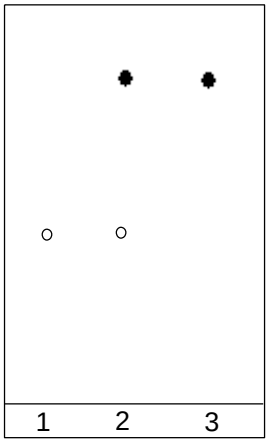
Procedure

Prepare an ice-water bath in a 250ml beaker, place 15ml of ethanol and 20ml of aqueous 10% NaOHinto a 100 ml beaker & add a stirbar. Place the 100ml beaker into the ice-water bath. Set then the entire assembly on to a magnetic stirrer. While stirring, cool the solution to 20°c. After the solution reaches to 20°c, remove the ice-water bath, continue to stir the solution.

Prepare the mixture of 2.8ml (2m mol) of fresh 4-Chloro benzaldehyde and 758µl (1 m mol) of Acetone in a test tube over a period of 5-10 mins, add the 4-Chloro benzaldehyde-acetone mixture to the ethanol-NaOH solution in a small portions, then stir the reaction for another 30 mins. Cool the mixture using the ice water bath for one hour. Collect the crystals by vaccum filtration. Wash the crystals by suspending them in 50ml of distil water. Again collect the crystals by vaccum filtration. Finally recrystalised with ethyl acetate.

Check the filtrate by testing the last few drops of water using red-litmus paper. If the litmus changes to blue, wash the crystals again until red-litmus does not changes colour. Keep a small sample aside to dry for a crude melting point measurement[56].

TLC studies



1: starting material (1)

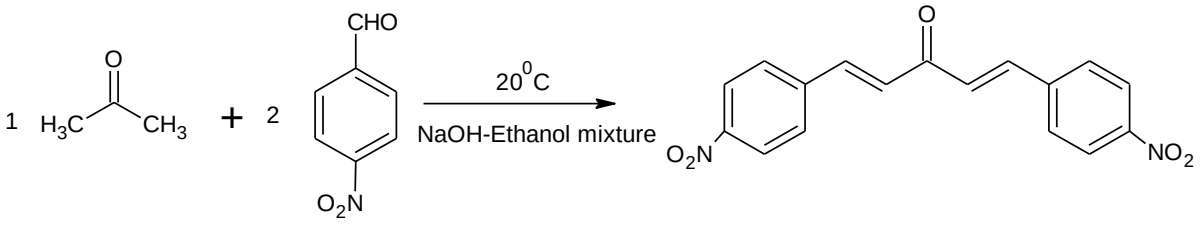
2: co-spot (1& 1b)

3: product (1b)

R_f values

Mobile phase : Benzene : Methanol (9:1)
$R_f$value of the starting material : 0.89
$R_f$value of the product : 0.92
Percentage yield : 99%

Preparation of (1*e*, 4*e*)-1,5-bis(4-nitrophenyl)penta-1,4-dien-3-one:
Reaction



propan-2-one 4-nitrobenzaldehyde (1*E*,4*E*)-1,5-bis(4-nitrophenyl)penta-1,4-dien-3-one

M. F - C_3H_6O M.F - $C_6H_4NO_2$ M.F - $C_{17}H_{12}N_2O_5$
M.W – 58 M.W -151 M.W - 324.

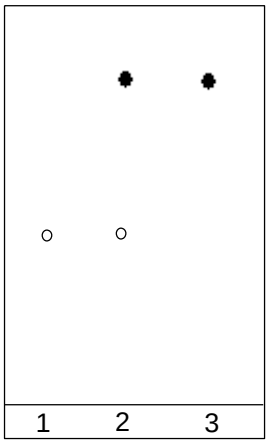
Procedure

Prepare an ice-water bath in a 250ml beaker, place 15ml of ethanol and 20ml of aqueous 10% NaOHinto a 100 ml beaker & add a stirbar. Place the 100ml beaker into the ice-water bath. Set then the entire assembly on to a magnetic stirrer. While stirring, cool the solution to 20°c. After the solution reaches to 20°c, remove the ice-water bath, continue to stir the solution.

Prepare the mixture of 2.1ml (2 m mol) of fresh 4-Nitro benzaldehyde and 758µl (1m mol) of Acetone in a test tube over a period of 5-10 mins, add the 4-Nitro benzaldehyde-acetone mixture to the ethanol-NaOHsolution in a small portions, then stir the reaction for another 30 mins. Cool the mixture using the ice water bath over night, collect the crystals by vaccum filtration. Wash the crystals by suspending them in 50ml of distil water. Again collect the crystals by vaccum filtration. Finally recrystalised with ethyl acetate.

Check the filtrate by testing the last few drops of water using red-litmus paper. If the litmus changes to blue, wash the crystals again until red-litmus does not changes colour. Keep a small sample aside to dry for a crude melting point measurement[56].

TLC studies



1: starting material (1)

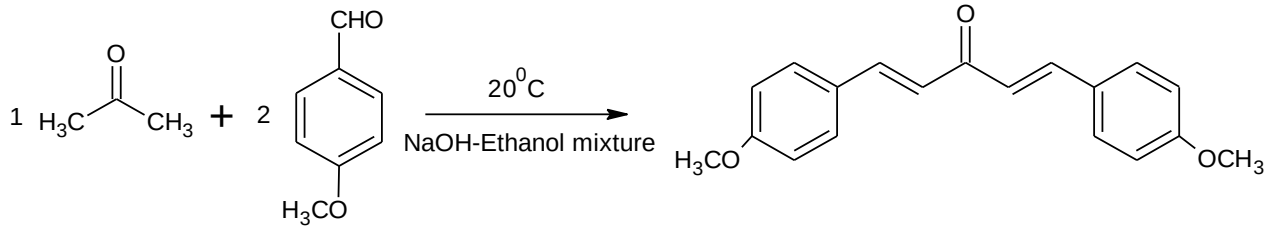
2: co-spot (1& 1c)

3: product (1c)

R_f values

Mobile phase: Benzene : Methanol (9:1)
R_f value of the starting material : 0.91
$R_f$value of the product : 0.94
Percentage yield : 66%

Preparation of (1*e*, 4*e*)-1, 5-bis(4-methoxyphenyl)penta-1,4-dien-3-one:
Reaction



propan-2-one 4-methoxybenzaldehyde

M. F - C_3H_6O M.F - $C_8H_8O_2$
M.W -58 M.W - 136

(1*E*,4*E*)-1,5-bis(4-methoxyphenyl)penta-1,4-dien-3-one

M.F - $C_{19}H_{18}O_3$
M.W - 294

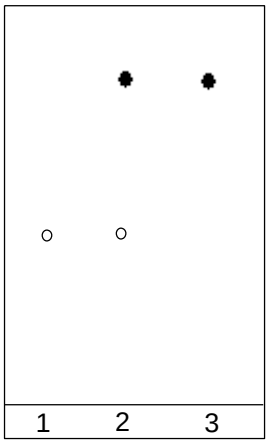
Procedure

Prepare an ice - water bath in a 250ml beaker, place 15ml of ethanol and 20ml of aqueous 10% NaOHinto a 100 ml beaker & add a stirbar. Place the 100ml beaker into the ice-water bath. Set then the entire assembly on to a magnetic stirrer. While stirring, cool the solution to 20°c. After the solution reaches to 20°c, remove the ice-water bath, continue to stir the solution.

Prepare the mixture of 2.8ml (2 m mol) of fresh 4-Methoxy benzaldehyde and 758µl(1 m mol) of Acetone in a test tube over a period of 5-10 mins, add the 4-Methoxy benzaldehyde-acetone mixture to the ethanol-NaOH solution in a small portions, then stir the reaction for another 30 mins. Cool the mixture using the ice water bath overnight. Collect the crystals by vaccum filtration. Wash the crystals by suspending them in 50ml of distil water. Again collect the crystals by vaccum filtration. Finally recrystalised with ethyl acetate.

Check the filtrate by testing the last few drops of water using red-litmus paper. If the litmus changes to blue, wash the crystals again until red-litmus does not changes colour. Keep a small sample aside to dry for a crude melting point measurement[56].

TLC studies



1: starting material (1)

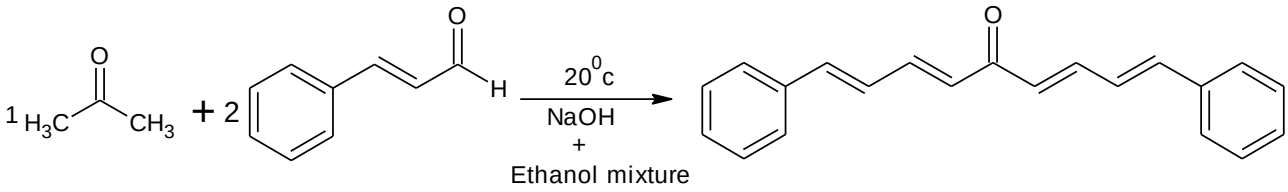
2: co-spot (1& 1d)

3: product (1d)

R_f values

Mobile phase : Benzene : Methanol (9:1)
R_f value of the starting material : 0.89
$R_f$value of the product : 0.92
Percentage yield : 59%

Preparation of (1e,3e,6e,8e)-1,9-diphenylnona-1,3,6,8-tetraen-5-one
Reaction



propan-2-one — M. F - C_3H_6O, M.W -58

3-phenylpropanal — M.F - $C_9H_{10}O$, M.W - 132

(1*E*,3*E*,6*E*,8*E*)-1,9-diphenylnona-1,3,6,8-tetraen-5-one — M.F - $C_{21}H_{18}O$, M.W - 286

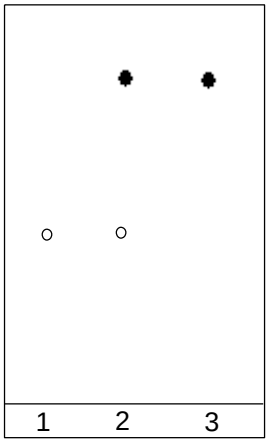
Procedure

Prepare an ice - water bath in a 250ml beaker, place 15ml of ethanol and 20ml of aqueous 10% NaOHinto a 100 ml beaker & add a stirbar. Place the 100ml beaker into the ice-water bath. Set then the entire assembly on to a magnetic stirrer. While stirring, cool the solution to 20°c. After the solution reaches to 20°c, remove the ice-water bath, continue to stir the solution.

Prepare the mixture of 1.5ml (2 mmol) of fresh Cinnamaldehyde and 758 µl (1 m mol) of Acetone in a test tube over a period of 5-10mins, add the Cinnamaldehyde –acetone mixture to the ethanol-NaOHsolution in a small portions, then stir the reaction for another 30 mins. Cool the mixture using the ice water bath for one hour. Collect the crystals by vaccum filtration. Wash the crystals by suspending them in 50ml of distil water. Again collect the crystals by vaccum filtration. Finally recrystalised with ethyl acetate.

Check the filtrate by testing the last few drops of water using red-litmus paper. If the litmus changes to blue, wash the crystals again until red-litmus does not changes colour. Keep a small sample aside to dry for a crude melting point measurement[56].

TLC studies



1: starting material (1)

2: co-spot (1& 1e)

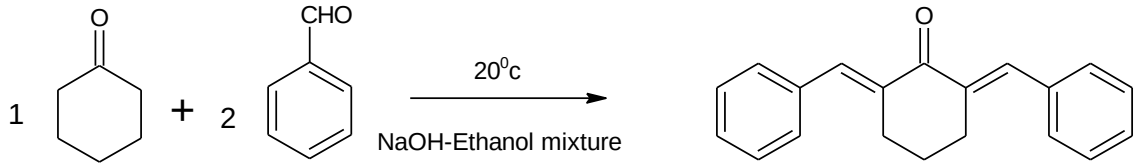
3: product (1e)

R_f values

Mobile phase : Benzene : Methanol (9:1)
R_f value of the starting material : 0.91
$R_f$value of the product : 0.93
Percentage yield : 60.7%

Preparation of (2*e*, 6*e*)-2,6-dibenzylidenecyclohexanone

Reaction



cyclohexanone
M. F - $C_6H_{10}O$
M.W - 98

benzaldehyde
M.F - C_7H_6O
M.W - 106.

(2*E*,6*E*)-2,6-dibenzylidenecyclohexanone
M.F - $C_{20}H_{18}O$
M.W - 274

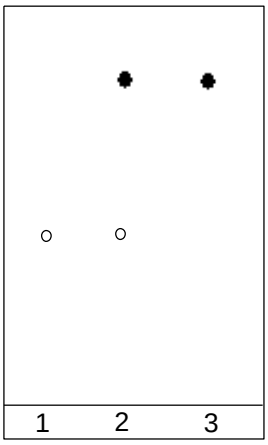
Procedure

Prepare an ice-water bath in a 250ml beaker, place 15ml of ethanol and 20ml of aqueous 10% NaOHinto a 100 ml beaker & add a stirbar. Place the 100ml beaker into the ice-water bath. Set then the entire assembly on to a magnetic stirrer. While stirring, cool the solution to 20°c. After the solution reaches to 20°c, remove the ice-water bath, continue to stir the solute.

Prepare the mixture of 2.1 ml (2 m mol) of fresh Benzaldehyde and 1263.3µl (1 m mol) of Cyclohexanone in a test tube over a period of 5-10 mins, add the Benzaldehyde-Cyclohexanone mixture to the ethanol-NaOH solution in a small portions, then stir the reaction for another 30 mins. Cool the mixture using the ice water bath for one hour. Collect the crystals by vaccum filtration. Wash the crystals by suspending them in 50ml of distil water. Again collect the crystals by vaccum filtration. Finally recrystalised with ethyl acetate.

Check the filtrate by testing the last few drops of water using red-litmus paper. If the litmus changes to blue, wash the crystals again until red-litmus does not changes colour. Keep a small sample aside to dry for a crude melting point measurement[56].

TLC studies



1: starting material (2)

2: co-spot (2& 2a)

3: product (2a)

R_f values

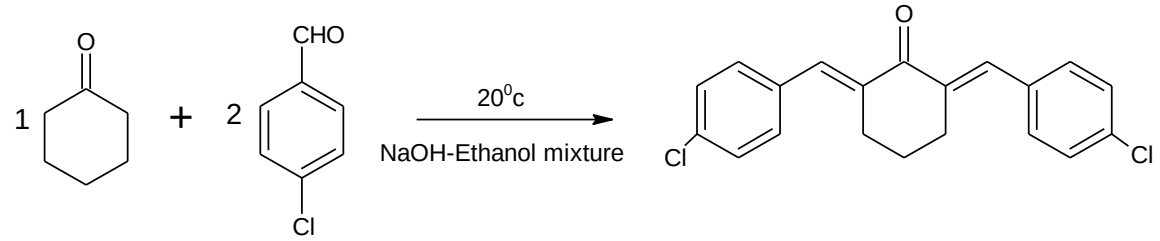
Mobile phase: Benzene : Methanol (8:2)

R_f value of the starting material : 0.87

R_f value of the product : 0.92

Percentage yield : 70%

Preparation of (2e,6e)-2,6(4-chlorobenzylidene)cyclohexanone
Reaction



cyclohexanone 4-chlorobenzaldehyde (2*E*,6*E*)-2,6-bis(4-chlorobenzylidene)cyclohexanone

M. F - $C_6H_{10}O$ M.F - C_5H_3ClO M.F - $C_{20}H_{16}Cl_2O$
M.W - 98 M.W - 140 M.W - 343

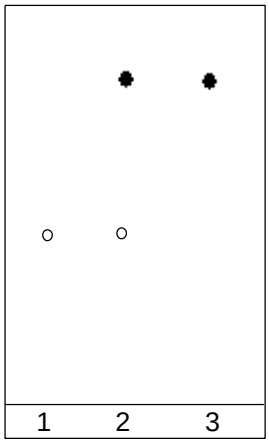
Procedure

Prepare an ice-water bath in a 250ml beaker, place 15ml of ethanol and 20ml of aqueous 10% NaOHinto a 100 ml beaker & add a stirbar. Place the 100ml beaker into the ice-water bath. Set then the entire assembly on to a magnetic stirrer. While stirring, cool the solution to 20°c. After the solution reaches to 20°c, remove the ice-water bath, continue to stir the solution.

Prepare the mixture of 2.8 ml (2 m mol) of fresh 4-Chloro benzaldehyde and 1263.3µl (1 m mol) of Cyclohexanone in a test tube over a period of 5-10 mins, add the 4-Chloro benzaldehyde-Cyclohexanone mixture to the ethanol-NaOH solution in a small portions, then stir the reaction for another 30 mins. Cool the mixture using the ice water bath for one hour. Collect the crystals by vaccum filtration. Wash the crystals by suspending them in 50ml of distil water. Again collect the crystals by vaccum filtration. Finally recrystalised with ethyl acetate.

Check the filtrate by testing the last few drops of water using red-litmus paper. If the litmus changes to blue, wash the crystals again until red-litmus does not changes colour. Keep a small sample aside to dry for a crude melting point measurement[56].

TLC plates



1: starting material (2)

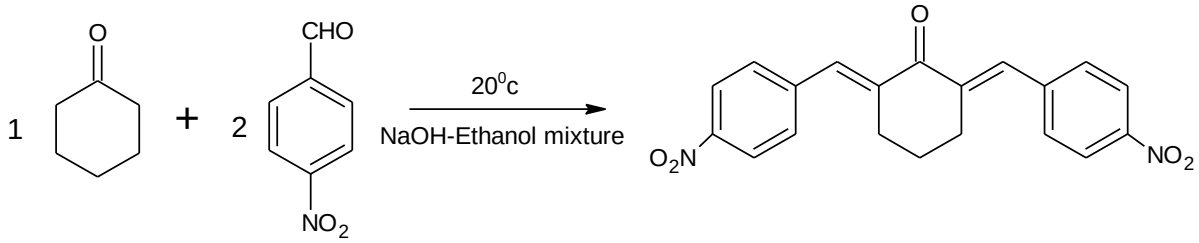
2: co-spot (2& 2b)

3: product (2b)

R_f values

Mobile phase : Benzene : Methanol (8:2)
$R_f$value of the starting material : 0.89
R_f value of the product : 0.94
Percentage yield : 70.1%

Preparation of (2e, 6e)-, 2, 6 bis (4-nitrobenzylidene)cyclohexanone:
Reaction



cyclohexanone 4-nitrobenzaldehyde (2*E*,6*E*)-2,6-bis(4-nitrobenzylidene)cyclohexanone

M. F - $C_6H_{10}O$ M.F - $C_6H_4NO_2$ M.F - $C_{20}H_{16}N_2O_5$
M.W - 98 M.W - 151 M.W - 364.

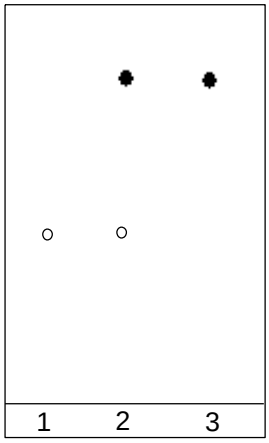
Procedure

Prepare an ice-water bath in a 250ml beaker, place 15ml of ethanol and 20ml of aqueous 10% NaOHinto a 100 ml beaker & add a stirbar. Place the 100ml beaker into the ice-water bath. Set then the entire assembly on to a magnetic stirrer. While stirring, cool the solution to 20°c. After the solution reaches to 20°c, remove the ice-water bath, continue to stir the solution.

Prepare the mixture of 2.1 ml (2 mmol) of fresh 4-Nitro benzaldehyde and 1263.3µl (1 mmol) of Cyclohexanone in a test tube over a period of 5-10 mins, add the 4-Nitro benzaldehyde-Cyclohexanone mixture to the ethanol-NaOH solution in a small portions, then stir the reaction for another 30 mins. Cool the mixture using the ice water bath over night, collect the crystals by vaccum filtration. Wash the crystals by suspending them in 50ml of distil water. Again collect the crystals by vaccum filtration. Finally recrystalised with ethyl acetate.

Check the filtrate by testing the last few drops of water using red-litmus paper. If the litmus changes to blue, wash the crystals again until red-litmus does not changes colour. Keep a small sample aside to dry for a crude melting point measurement[56].

TLC plates



1: starting material (2)

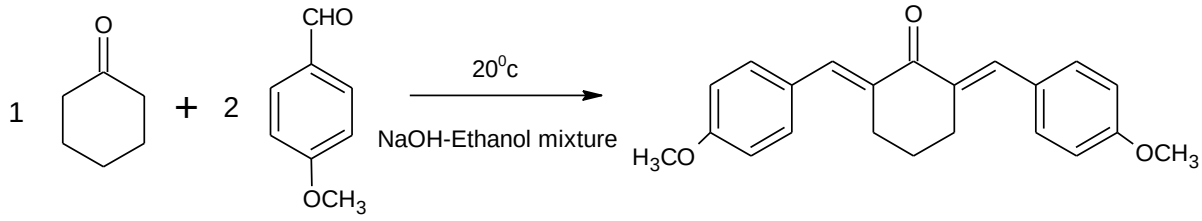
2: co-spot (2& 2c)

3: product (2c)

R_f values

Mobile phase : Benzene : Methanol (8:2)
$R_f$value of the starting material : 0.91
R_f value of the product : 0.96
Percentage yield : 72%

Preparation of(2*e*, 6*e*)-2, 6-bis(4methoxybenzylidene)cyclohexanone
Reaction



cyclohexanone — M. F - $C_6H_{10}O$, M.W - 98

4-methoxybenzaldehyde — M.F - $C_8H_8O_2$, M.W - 136

(2*E*,6*E*)-2,6-bis(4-methoxybenzylidene)cyclohexanone — M.F - $C_{22}H_{22}O_3$, M.W - 334

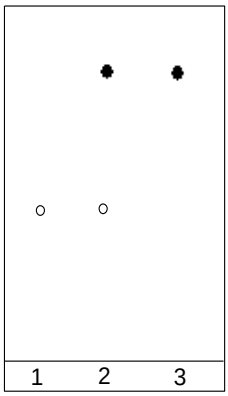
Procedure

Prepare an ice-water bath in a 250ml beaker, place 15ml of ethanol and 20ml of aqueous 10% NaOH into a 100 ml beaker & add a stirbar. Place the 100ml beaker into the ice-water bath. Set then the entire assembly on to a magnetic stirrer. While stirring, cool the solution to 20°c. After the solution reaches to 20°c, remove the ice-water bath, continue to stir the solution.

Prepare the mixture of 2.8 ml (2 m mol) of fresh 4-Methoxy benzaldehyde and 1263.3µl (1 m mol) of Cyclohexanone in a test tube over a period of 5-10 mins, add the 4-Methoxy benzaldehyde-Cyclohexanone mixture to the ethanol-NaOH solution in a small portions, then stir the reaction for another 30 mins. Cool the mixture using the ice water bath over night. Collect the crystals by vacuum filtration. Wash the crystals by suspending them in 50ml of distil water. Again collect the crystals by vacuum filtration. Finally recrystalised with ethyl acetate.

Check the filtrate by testing the last few drops of water using red-litmus paper. If the litmus changes to blue, wash the crystals again until red-litmus does not changes color. Keep a small sample aside to dry for a crude melting point measurement[56].

TLC plates



1: starting material (2)

2: co-spot (2& 2d)

3: product (2d)

R_f values

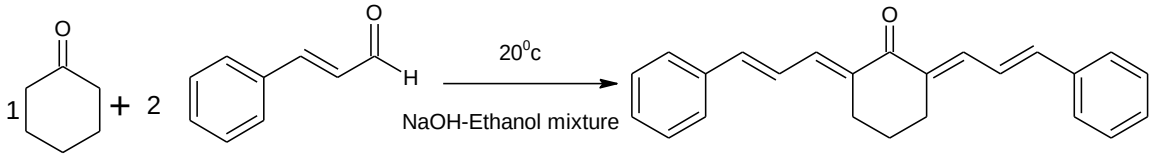
Mobile phase : Benzene : Methanol (8:2)

R_f value of the starting material : 0.89

R_f value of the product : 0.93

Percentage yield : 60%

Preparation of 1, 1'-[cyclohexane-1, 3-diylidenedi(1*e*,3*e*)prop-1-en-1-yl-3-ylidene]dibenzene : Reaction



cyclohexanone 3-phenylpropanal (2*E*,6*E*)-2,6-bis[(2*E*)-3-phenylprop-2-en-1-ylidene]cyclohexanone

M.F - $C_6H_{10}O$ M.F - $C_9H_{10}O$ M.F - $C_{24}H_{22}O$
M.W- 98 M.W -121 M.W - 326

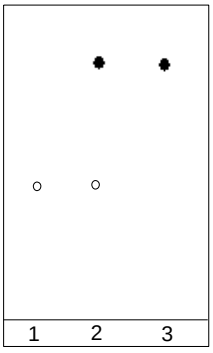
Procedure

Prepare an ice-water bath in a 250ml beaker, place 15ml of ethanol and 20ml of aqueous 10% NaOHinto a 100 ml beaker & add a stirbar. Place the 100ml beaker into the ice-water bath. Set then the entire assembly on to a magnetic stirrer. while stirring, cool the solution to 20°c. After the solution reaches to 20°c, remove the ice-water bath, continue to stir the solution.

Prepare the mixture of 2.1 ml (2 m mol) of fresh Cinnamaldehyde and 1263.3µl (1 m mol) of Cyclohexanone in a test tube over a period of 5-10 mins, add the cinnamaldehyde-Cyclohexanone mixture to the ethanol-NaOH solution in a small portions, then stir the reaction for another 30 mins. Cool the mixture using the ice water bath for one hour. Collect the crystals by vaccum filtration. Wash the crystals by suspending them in 50ml of distil water. Again collect the crystals by vaccum filtration. Finally recrystalised with ethyl acetate.

Check the filtrate by testing the last few drops of water using red-litmus paper. If the litmus changes to blue, wash the crystals again until red-litmus does not changes colour. Keep a small sample aside to dry for a crude melting point measurement[56].

TLC plates



1: starting material (2)

2: co-spot (2& 2e)

3: product (2e)

R_f values

Mobile phase : Benzene : Methanol (8:2)
$R_f$value of the starting material: 0.91
R_f value of the product : 0.95
Percentage yield :64.1%

Antimicrobial Assay

Apparatus and Microorganism

The list of apparatus which were used in the antibacterial assay, were as follows: incubation bottle (500 mL), sample bottles, tips and micropipette (10 µL and 500 µL), Whatman paper disc (6 mm), disposable petri dishes (8 mm), disposable micro-titer well U shape, needles, glass rod, cotton and test tubes (13 mm diameter,140 mm length). The apparatus were sterilized by autoclave for 15 minutes at 121°C. Two bacteria namely Escherichia coli, Staphylococcus aureus and aeruginosa were used in the antibacterial assay.

Preparation of Nutrient Agar and Broth

Antimicrobial assay was performed using nutrient agar and nutrient broth for bacteria. Nutrient agar (28g) and nutrient broth (30g) each was suspended in one liter of distilled water. All solutions were sterilized by autoclave for 15 minutes at 121^0c.each compound (1mg) was dissololved in DMSO (1ml).

Culturing Microbe

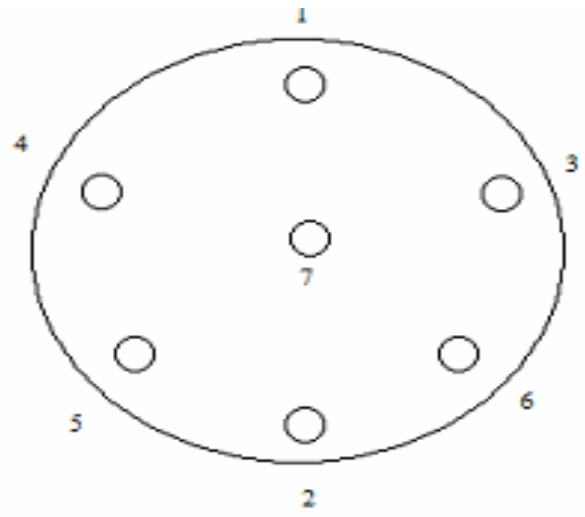
Each of the selected microbes was impregnated in nutrient broth (20 ml) insterilized conical flask (250 ml). The flasks were sealed and kept in an incubator for24 hours at 37 ± 1^0c.

Preparation of Agar Plate

Sterilized agar (17 mL) was pipetted into petri dishes immediately. The agar was let to cool before the dishes were kept in a refrigerator. The petri dishes were kept upside down in the refrigerator.

Disc Diffusion Method

The disc diffusion method was carried out on the cultures of microbes. The discs were prepared by impregnating them in DMSO solution of each sample. The paper disc containing 1 mg of compound was placed on the agar surface previously inoculated with suspension of each microbe to be tested. All determinations were made in duplicate. Streptomycin sulphate (30 µg/disc) was used as the positive control. Inhibition diameter was determined after incubation at 37oC ± 1 for 24hours. The antimicrobial activity was indicated by the presence of clear inhibition zones around each disc[53].



4. TOTAL ANTIOXIDANT ACTIVITY

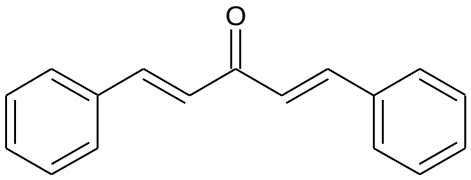
The total antioxidant activity was eluted using the method described by Prieto et al (1999).Ascorbic acid was used as the standard antioxidant drug. 3ml of the extract/standard drug (0.1, 0.3,1 and 3 mg/ml) was placed in a test tube. 0.3 ml of reagent solution (0.6M sulphuricAcid, 28mM Sodium Phosphate, 4Mm Ammonium molybdate) was then added and the resulting mixture was incubated at 95^0C for 90 min. After the mixture has cooled to room temperature, the absorbance of the each solution was measured using UV-Visible spectrophotometer at 695nm against blank.

The total antioxidant capacity was expressed as ascorbic acid equivalent using the Graph pad for Window version 4.02 (Graph pad software, aSanDiego, CA,USA).

5. RESULTS

Physical results

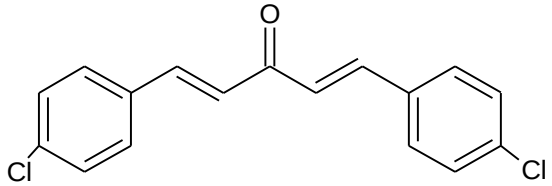
Table 1:
1, 5-diphenylpenta-1,4-diyn -3-one



(1*E*, 4*E*)-1,5-diphenylpenta-1,4-dien-3-one
(1a)

S.No	Compound 1(a) Results	
1	Molecular formula	$C_{17}H_{14}O$
2	Molecular weight	234
3	Percentage yield	82%
4	R_f value	0.91
5	Melting point	110^0c
6	Reported melting point	110^0c

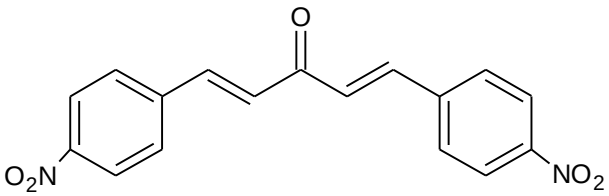
Table 2:
(1E,4E)-1,5-bis(4-chlorophenyl)penta-1,4-dien-3-one



(1*E*,4*E*)-1,5-bis(4-chlorophenyl)penta-1,4-dien-3-one
1(b)

S.No	Compound 1(b) Results	
1	Molecular formula	$C_{17}H_{12}Cl_2O$
2	Molecular weight	303
3	Percentage yield	99%
4	R_f value	0.92
5	Melting point	130-131^{0}c
6	Reported melting point	131-132^{0}c

Table 3:
(1*E*, 4*E*)-1,5-bis(4-nitrophenyl)penta-1,4-dien-3-one

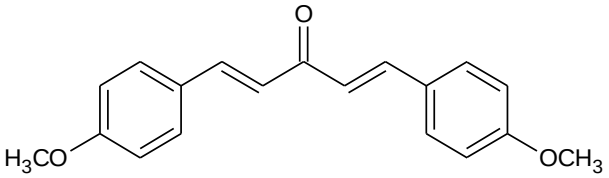


(1*E*, 4*E*)-1, 5-BIS (4-NITROPHENYL)PENTA-1,4-DIEN-3-ONE

1(c)

S.No	Compound 1(c) Results	
1	Molecular formula	$C_{17}H_{12}N_2O_5$
2	Molecular weight	324
3	Percentage yield	66%
4	R_f value	0.94
5	Melting point	147-148^{0}c
6	Reported melting point	147^{0}c

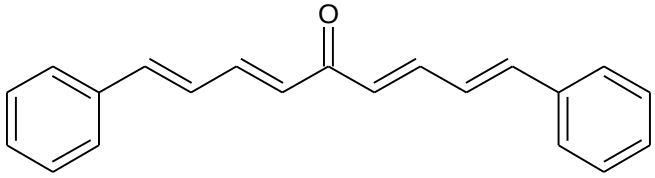
Table 4:
(1*E*, 4*E*)-1,5-bis(4-methoxyphenyl)penta-1,4-dien-3-one



(1*E*, 4*E*)-1,5-BIS(4-METHOXYPHENYL)PENTA-1,4-DIEN-3-ONE :
1(d)

S.No	Compound 1(d) Results	
1	Molecular formula	$C_{19}H_{18}O_3$
2	Molecular weight	294
3	Percentage yield	59%
4	R_f value	0.92
5	Melting point	188-189^{0}c
6	Reported melting point	187-188^{0}c

Table 5:
(1*E*,3*E*,6*E*,8*E*)-1,9-diphenylnona-1,3,6,8-tetraen-5-one :

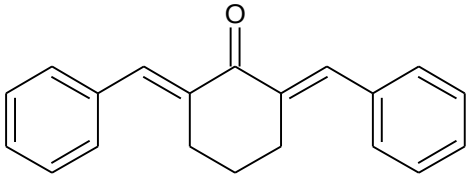


(1*E*,3*E*,6*E*,8*E*)-1,9-diphenylnona-1,3,6,8-tetraen-5-one

1(e)

S.No	Compound 1(e) Results	
1	Molecular formula	$C_{21}H_{18}O$
2	Molecular weight	286
3	Percentage yield	60.7%
4	R_f value	0.93
5	Melting point	169-170^{0}c
6	Reported melting point	172^{0}c

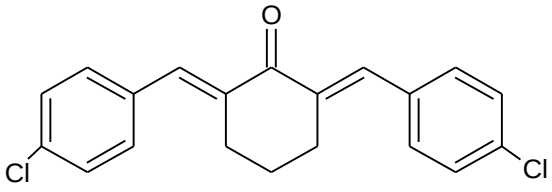
Table 6:
(2*E*,6*E*)-2,6-DIBENZYLIDENECYCLOHEXANONE



(2*E*,6*E*)-2,6-dibenzylidenecyclohexanone
2(a)

S.No	Compound 2(a) Results	
1	Molecular formula	$C_{20}H_{18}O$
2	Molecular weight	274
3	Percentage yield	70%
4	R_f value	0.92
5	Melting point	115-118^{0}c
6	Reported melting point	117^{0}c

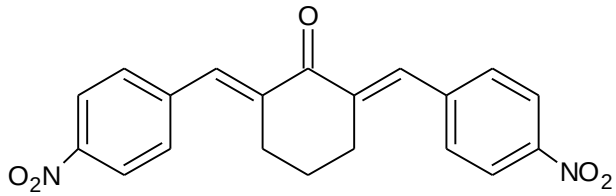
Table 7:
(2E, 6E)-, 2, 6-bis(4-chlorobenzylidene)cyclohexanone



2(b)

S.No	Compound 2(b) Results	
1	Molecular formula	$C_{20}H_{16}Cl_2O$
2	Molecular weight	343
3	Percentage yield	70.1%
4	R_f value	0.94
5	Melting point	146^{0}c
6	Reported melting point	147-148^{0}c

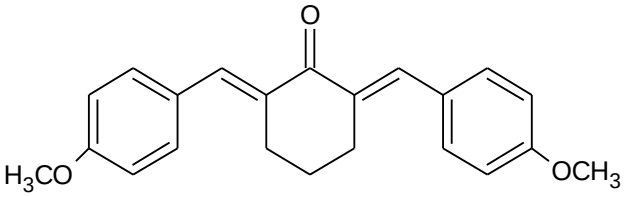
Table 8:
(2*E*,6*E*)-2,6-BIS(4-NITROBENZYLIDENE)CYCLOHEXANONE



(2*E*,6*E*)-2,6-bis(4-nitrobenzylidene)cyclohexanone
2(c)

S.No	Compound 2(c) Results	
1	Molecular formula	$C_{20}H_{16}N_2O_5$
2	Molecular weight	364
3	Percentage yield	72%
4	R_f value	0.96
5	Melting point	160-161^{0}c
6	Reported melting point	159^{0}c

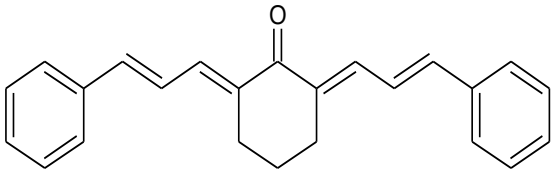
Table 9:
(2*E*,6*E*)-2,6-BIS(4-METHOXYBENZYLIDENE)CYCLOHEXANONE



(2*E*,6*E*)-2,6-bis(4-methoxybenzylidene)cyclohexanone
2(d)

S.No	Compound 2(d) Results	
1	Molecular formula	$C_{22}H_{22}O_3$
2	Molecular weight	334
3	Percentage yield	60%
4	R_f value	0.93
5	Melting point	202-204^{0}c
6	Reported melting point	203-204^{0}c

Table 10:
(2*E*,6*E*)-2,6-BIS[(2*E*)-3-PHENYLPROP-2-EN-1-YLIDENE]CYCLOHEXANONE



(2*E*,6*E*)-2,6-bis[(2*E*)-3-phenylprop-2-en-1-ylidene]
Cyclohexanone 2(e)

S.No	Compound 2(e) Results	
1	Molecular formula	$C_{24}H_{22}O$
2	Molecular weight	326
3	Percentage yield	64.1%
4	R_f value	0.95
5	Melting point	182-184^{0}c
6	Reported melting point	180^{0}c

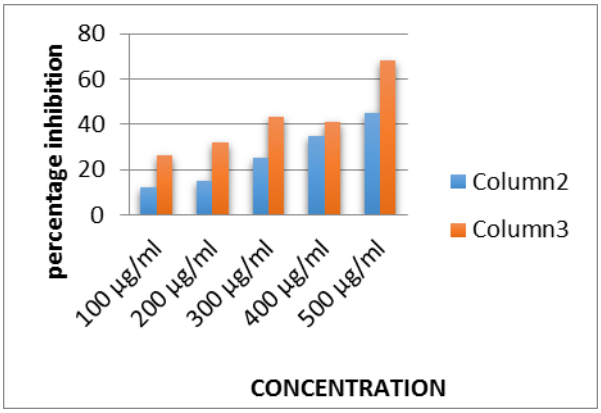
ACTIVITY RESULTS

S.NO	sample	Mean zone of inhibition(mm/mg)							
		Staphylococcus Aureus				Echerichia coli			
		50 µg	100 µg	150 µg	200 µg	50 µg	100 µg	150 µg	200 µg
1	Control(DMF)	-	-	-	-	-	-	-	-
2	Benzyl penicillin	19	23	27	32	-	-	-	-
3	streptomycin	-	-	-	-	20	24	29	34
4	1a	-	-	-	-	-	-	-	-
5	1b	-	-	-	-	-	-	-	-
6	1c	-	-	-	-	-	-	-	-
7	1d	-	-	-	-	-	-	-	-
8	1e	-	-	-	-	-	-	-	-
9	2a	-	-	-	-	-	-	-	-
1o	2b	-	-	-	-	-	-	-	-
11	2c	-	-	-	-	-	-	-	-
12	2d	-	-	-	-	-	-	-	-
13	2e	-	-	-	-	-	-	-	-

ANTI-OXIDANT ACTIVITY RESULTS
COMPOUND (1a)
Table 1:

CONCENTRATIONS	ABSORBANCE OF COMPOUND (1a)	ABSORBANCE OF ASCORBIC ACID(POSITIVE CONTROL)
100 µgm/ml	0.025333±0.004509	0.258233±0.001305
200 µgm/ml	0.046±0.0004583	0.3042±0.000755
300 µgm/ml	0.09167±0.007638	0.3324±0.001015
400 µgm/ml	0.145331±0.005033	0.433567±0.00095
500 µgm/ml	0.257667±0.003512	0.7322±0.0007552

Solubility: Methanol

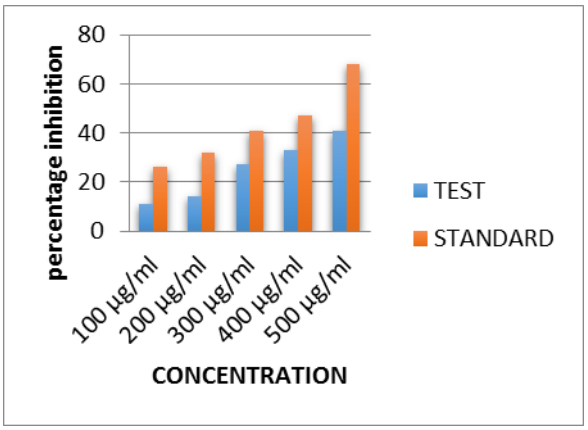


Graph.1:

COMPOUND (2a)
Table 1:

CONCENTRATIONS	ABSORBANCE OF COMPOUND (1a)	ABSORBANCE OF ASCORBIC ACID(POSITIVE CONTROL)
100 µgm/ml	0.01±0.000954	0.258233±0.001305
200 µgm/ml	0.012433±0.000551	0.3042±0.000755
300 µgm/ml	0.036433±0.000603	0.3324±0.001015
400 µgm/ml	0.059733±0.000643	0.433567±0.00095
500 µgm/ml	0.0985±0.0005	0.7322±0.0007552

Solubility: Methanol



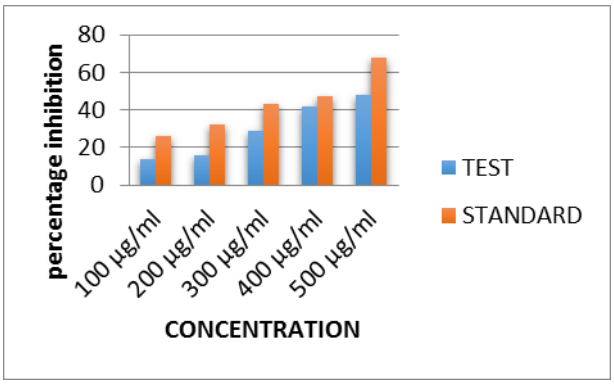
Graph. 2:

COMPOUND (2d)

Table 1:

CONCENTRATIONS	ABSORBANCE OF COMPOUND (1a)	ABSORBANCE OF ASCORBIC ACID(POSITIVE CONTROL)
100 µgm/ml	0.008433±0.000551	0.258233±0.001305
200 µgm/ml	0.010333±0.001172	0.3042±0.000755
300 µgm/ml	0.023733±0.000404	0.3324±0.001015
400 µgm/ml	0.32433±0.000666	0.433567±0.00095
500 µgm/ml	0.02633 ±0.000471	0.7322±0.0007552

Solubility: Methanol



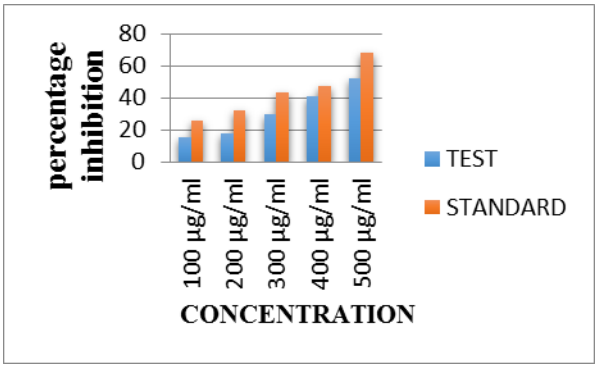
Graph.3:

COMPOUND (2e)

Table 1:

CONCENTRATIONS	ABSORBANCE OF COMPOUND (1a)	ABSORBANCE OF ASCORBIC ACID(POSITIVE CONTROL)
100 µgm/ml	0.013267±0.000702	0.258233±0.001305
200 µgm/ml	0.029833±0.001021	0.3042±0.000755
300 µgm/ml	0.045267±0.003591	0.3324±0.001015
400 µgm/ml	0.118433±0.001504	0.433567±0.00095
500 µgm/ml	0.21467±0.005686	0.7322±0.0007552

Solubility: Methanol



Graph. 4:

6. SUMMARY AND CONCLUSION

1. Ketones (Acetone, Cyclohexanone) were treated with different aldehydes(4-Chloro benzaldehyde, 4-nitrobenzaldehyde, 4-methoxybenzaldehyde, cinnamaldehyde) by aldol condensation to give the corresponding ‘‘dibenzal ketone derivatives’’ (1a-1e, 2a-2e),title compounds in good yield.
2. All the compounds synthesized were characterized by physical (R_f values, M.P., Molecular weight, molecular formula).
3. The title compounds were screened for antibacterial and anti-oxidant activity. According to the results obtained, it can be concluded that none of the synthesized compounds have antibacterial activity.
4. As for the ant-oxidant activity, (2a), (2e),compounds showed better activity.
5. Further lead optimization should be carried out for the better expected anti-oxidant activity.

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