COMPLEXATION STUDY OF INDIUM(III) BROMIDE AND STRONTIUM(II) CHLORIDE WITH CHALCONE'S DERIVATIVES

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This report is submitted in partial fulfilment of the requirement for the degree of Bachelor of Science with Honours

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Thank you.
Complexation Study of Indium(III) Bromide and Strontium(II) Chloride with Chalcone's Derivatives

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ABSTRACT

The synthesis and spectroscopic characterization of indium(III) and strontium(II) complexes with chalcone's are reported in this thesis. Tautomerization occurred before the coordination of chalcone (1-phenyl-3(4-hydroxy-phenyl)-propenone) to strontium(II) chloride and indium(III) bromide. For 1(4-hydroxy-phenyl)-3-phenyl-propenone, the complexation occurred at the deprotonated oxygen atom from the hydroxyl group. The ligands appeared as a uninegative monodentate fashion for all the synthesized indium(III) and strontium(II) complexes. The Sr:ligand ratio is 1:1 for all Sr(II) complexes forming three-coordinated molecule in trigonal planar geometry. The In:ligand ratio is 1:3 in complexation with 1-phenyl-3(4-hydroxy-phenyl)-propenone forming a six-coordinated molecule in octahedron structure and 1:1 ratio in complexation of indium(III) with 1(4-hydroxy-phenyl)-3-phenyl-propenone. The reactions of chalcone's with indium(III) bromide and strontium(II) chloride gave a new coordination mode of In-O and Sr-O bonds, respectively.

Key words: Chalcone, indium(III) complex, strontium(II) complex

ABSTRAK


Kata kunci: Kalkon, kompleks indium(III), kompleks strontium(II)
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 CHAPTER 1 INTRODUCTION

This project is a study on the complexation of indium(III) bromide and strontium(II) chloride with chalcone as a ligand. Indium and strontium are two elements which are on the same period but from different groups in the Periodic table. Therefore, the main purpose of this study is to see the difference between strontium(II) and indium(III) complexes in terms of number of coordination as well as their coordination modes.

1.1 Indium

Indium, a very soft silvery-white element discovered by Ferdinand Reich and Hieronymus Richter in 1863 (Heiserman, 1992). Indium belongs to Group 13 in Periodic table with the electronic configuration of [Kr] 4d^{10}5s^{2}5p^{1}. The atomic number of indium is 49 and atomic mass of 114.82 (Daintith, 2000). Naturally, indium has two isotopes namely indium-113 (4.28%) and indium-115 (95.72%) (Rodgers, 2002). Group 13 elements commonly achieve an oxidation state of three by forming covalent compound via ns→np promotion (Massey, 2000). This is the reason why indium can be found in widely stable compounds like indium(I), indium(II), and indium(III) (Daintith, 2000). Indium is normally found in zinc blend and some iron ores and can also be obtained from zinc flue dust (Daintith, 2000).

Indium has coordination numbers of 4, 5 or 6. The coordination number depends on the number of added ligands (dioxane, halide, triethylphosphine or pyridine) (Massey, 2000). Indium compound can have octahedron geometry when coordinated with chalcone (Wong, 2004) and a tetrahedron geometry when coordinated with 2-hydroxychalcone (Tay, 1999).
Indium is a by-product in the production of zinc, lead and sulphuric acid (Massey, 2000). According to Wada et al., 1999, indium in the form of InAs, InSb and InP is widely applied in semi-conductor science and technologies producing outcomes such as optical and microwave communication system, indium phosphide-based lasers, photodiodes and LEDs. Indiums can also function as special-purpose electroplates and special fusible alloys (Daintith, 2000).

1.2 Strontium

Strontium, an alkaline-earth metal, was first discovered by Martin Klaproth and Thomas Hope in 1798 and was later isolated by Humphry Davis in 1808. Strontium is a soft yellowish metallic element which belongs to Group 2 in Periodic table (Daintith, 2000) and has the electronic configuration of \([\text{Kr}]\ 5s^2\). With the atomic mass of 87.62 and atomic number 38, strontium has four natural isotopes namely strontium-84 (0.56%), strontium-86 (9.86%), strontium-87 (7.02%) and strontium-88 (82.56%) (Rodgers, 2002). Besides, strontium can be found in the form of strontianite (\(\text{SrCO}_3\)) and celestine (\(\text{SrSO}_4\)) minerals (Daintith, 2000).

Strontium ion has a coordination number of eight (Addison et al., 1978) and the strontium salt has a cubic crystal structure (Rodgers, 2002). Strontium(II) complex can have bicapped trigonal prism geometry when coordinated with triethanolamine ligands (Kazak et al., 2003).

In industries, strontium can be produced through roasting of ore to give oxide, and later by the reduction with aluminium. This element is highly reactive is applied in certain
alloys and also functions as a vacuum getter (Daintith, 2000).

For Group II elements, generally, it plays an important role in making of fireworks. Strontium salts contribute as colorizers and the salts gives scarlet colour to the fireworks (Rodgers, 2002).

1.3 Chalcone

Benzylideneacetophenone or better known as chalcone is a compound prepared through crossed aldol condensation of acetophenone and benzaldehyde (Lehman, 1988). Chalcone is largely accumulated in many plants and vegetables and are biologically active molecules in human diet (Tomas-Barberan et al., 2000). According to Eddarir et al., 2003, chalcone acts as a key precursor in synthesis of various flavanoid as they can be easily transformed into flavones through cyclization process in acidic medium. Over the years, chalcones bearing non natural substituents has been synthesized and developed to produce drugs which are active as anti-cancer, anti-malaria, anti-tuberculosis and anti-cardiovascular diseases (Eddarir et al., 2003). The structure of chalcone (1, 3-diphenyl-2-propen-1-one) is as shown in Figure 1.3.1.

![Structure of chalcone](image)

Figure 1.3.1: Structure of chalcone

The synthesis of chalcone can be done through several reaction methods such as the Suzuki coupling reaction and the Claisen-Schmidt reaction. According to Eddarir et al., 2003,
the chalcone is usually synthesized through the Claisen-Schmidt reaction (Figure 1.3.2) in basic medium in ethanol. According to Lehman, 1988, this reaction is regarded as a reaction between an aromatic aldehyde and an aliphatic aldehyde or ketone which produces $\alpha,\beta$-unsaturated aldehyde or ketone under basic condition. The mechanism of the Claisen-Schmidt is as shown in Scheme 1.3.1. This method was discovered by Schmidt in 1880 and later revised by Claisen (Lehman, 1988).

\[
\begin{align*}
\text{Benzaldehyde} & \quad \text{Acetophenone} \quad \text{Benzylideneacetophenone} \\
\text{OH} & \quad \text{PhCH}=\text{CHCOCH}_3 \\
\text{Scheme 1.3.1: Claisen-Schmidt Condensation Reaction Mechanism} \quad \text{(Lehman, 1988).}
\end{align*}
\]
1.4 Objectives

There are two objectives in this study:

1. To synthesise of indium(III) bromide and strontium(II) chloride complexes with chalcone as ligand.

2. To characterise the complexes using FT-IR, $^1$H-NMR, and CHN.
CHAPTER 2 LITERATURE REVIEW

2.1 Chalcone

According to Eddarir et al., 2003, chalcone or benzylideneacetophenone especially chalcones bearing oxygenated function on the aromatic rings are key precursor of all flavanoids and exists as biologically active molecules in many various plant species. Arty et al., 2000, reported that various chalcone derivatives show properties of inflammatory, antibacterial, antiviral and gastric protectant activities. Sogawa et al., 1993, found that chalcone compound containing a 3,4-dihydroxycinnamoyl structure strongly inhibit lipid peroxidation. Eddarir et al., 2003, has reported that chalcones bearing non natural substituents have been synthesized over the years in the development of drugs which are active against cancer, malaria, leishmaniasis, tuberculosis, and cardiovascular diseases. Furthermore, chalcones are important for their properties in modulating the regulation of biochemical pathways such as NO or tyrosine kinase (Eddarir et al., 2003).

2.2 Synthesis of Chalcone

There are various methods that have been used over the years to synthesize chalcone compounds. Among the most commonly used is the Suzuki coupling reaction and the Claisen-Schmidt reaction.

In the Suzuki coupling reaction, there are pathways that are obviously possible for the synthesis of chalcones which is the coupling between activated cinnamic acid and phenylboronic acids (Scheme A) and the coupling between activated benzoic acids and phenylvinylboronic acids (Scheme B). Eddarir and co-workers, 2003, employed several
conditions for Scheme A. They used the protocol described by Bumagin et al., 1999, (solvent: acetone, water 3:1; catalyst PdCl₂ 3%, base: Na₂CO₃) which gave a moderate yield between 23-37%. The use of McCarthy’s conditions (solvent: anhydrous toluene; catalyst: tetrakis(triphenylphosphine)palladium (0); base: caesium carbonate) (Haddach and McCarthy, 1999) produced a yield of 41-51% chalcone. Meanwhile, the coupling done as in Scheme B under the McCarthy’s condition gave an isolated yield of 68-93% chalcone compounds (Eddarir et al., 2003).

Another recently found synthesizing method is carried out under the influence of catalysts like copper (II) 2,2'-bipyridyl or a complex of cobalt(II) acetate and a 4-vinylpyridine-styrene-divinylbenzene co-polymer. The reaction produces a high yield of chalcone and the catalysts may be recovered and reused (Trost, 1999).

A recently reported procedure was done by adding sodium hydroxide pellets to a rapidly stirring solution of the ketone and aldehyde in absolute ethanol at room temperature. The chalcones produced are in the crystalline form, isolated through simple filtration and the yield was reported to be 72-99% (Trost, 1999).

According to Eddarir et al., 2003, the synthesis of chalcones are usually carried out using the Claisen-Schmidt reaction in polar solvent in basic medium, the yield is purified through separation and a complex mixture is often produced at the end of the reaction. In this project, the synthesis will be carried out using this reaction mechanism which involves the acetophenone with benzaldehyde in basic conditions. Sogawa et al., 1993, reported that, the synthesis of chalcone done under basic conditions using Claisen-Schmidt condensation of
acetophenone with 3,4-dihydroxy-benzaldehyde obtained a yield below 10% compared to condensation of acetophenone with bis(tetrahydropyranyl) ether which yielded about 45%. However, the synthesis of a small series of the title compounds under acidic conditions described by Herdan et al., 1990, yielded 75-80%. Even though condensation reaction happening in an acidic medium gives a better yield, it is only proven on a small series of chalcone compound compared to condensation done in basic conditions which have been proved to produce various chalcone compounds successfully.

2.3 Etherification

There are various studies being carried out on the etherification reaction. Xinglong et al., 2002, conducted a study on the reductive etherification of alcohols with carbonyl compounds through different trialkylsilyl ether intermediates. They applied the BiBr₃/Et₃SiH catalytic system to conduct the research. 88-90% yield of ether was produced when benzyloxydiisopropylsilane was treated with aliphatic or aromatic aldehydes and ketone. Under the same condition, an optically pure alkoxy silane reacting with an aldehyde gave a good yield of ether compound with a complete retention of stereochemistry.

Wright et al., 1997, has done a study on the preparation of t-butyl esters and ethers from t-butanol. In the concern of ether synthesis, the method designed has proven to give good yield for a variety of aliphatic, benzylic, aromatic and protected amino alcohols. Secondary alcohol gave a lower yield compared to primary alcohol. It was reported that the substitution of cyclohexane as the solvent instead of dichloromethane improved the yield of secondary alcohol ethers by 10-20%.
Yeap et al., 2004, successfully synthesized cholestryl 4-decyclophenyl-4'-benzoates (100ABCh) from 4-n-Decyloxy-4'-biphenylcarboxylic acid. The carboxylic acid was synthesized through alkylation of 4-hydroxybiphenyl-4'-carboxylic acid. In this reaction, the alkylated carboxylic acid was converted into its acid chloride by using excess of thionyl chloride. The yield was then esterified with cholesterol under inert atmospheres in presence of pyridine as the base. The synthesis scheme is as shown in Figure 2.3.1.

![Synthesis Scheme](image)

**Figure 2.3.1: Synthesis of cholestryl 4-decyclophenyl-4'-benzoates (Yeap et al., 2004)**

From the IR spectrum, it was found that the terminal phenolic hydroxyl group attached to one of the biphenyl moiety end was substituted by OR (R=\(C_nH_{2n+1}\), \(n=10\)). A band within
2926-2852 cm\(^{-1}\) supported the presence of the alkyl groups. A strong absorption band at 1706 cm\(^{-1}\) and 1286 cm\(^{-1}\) was observed due to the carbonyl (C=O) group present in the ester and the stretching of C\(_{29}\)-C\(_{28}\)-O respectively. An additional band with weak intensities was found at 1256 cm\(^{-1}\) due to the stretching of C\(_{36}\)-O in ether bond.

The etherification method used in this study is similar to the esterification study done by Ng and her co-workers, 2004. In this study, structural modification of cholesteryl hydrogen phthalate was done to produce methyl-, ethyl- and isopropyl- derivatives by using dichloromethane as a solvent and is later treated with concentrated sulphuric acid. The yield obtained was 56%, 50% and 93% of methyl-, ethyl- and isopropyl- derivatives respectively.

2.4 Complexation of Indium

According to Massey, 2000, the organo derivatives of gallium, indium and thallium, although apparently weakly associated in the solid state, formation of dimers like the aluminium may not occur. This may be due to the adverse metal-metal steric interaction which would occur in the M-C bridge system.

Shen and co-workers, 2000, reported the reaction of gallium and indium alkyls R\(_3\)M (M= Ga, In; R= Me, Et) with the Schiff-base ligands of N-salicylidene 2-aminopyridine or N-salidylidene 2-methoxyaniline at room temperature, in 1:1 ratio proceeded smoothly affording the corresponding intramolecular coordinated complexes as shown in Scheme 2.4.1. The high yields are isolated as yellow solids and the entire product gave satisfactory elemental analysis results and was characterized by IR, \(^1\)H-NMR and mass spectroscopy respectively (Shen et al., 2000).
Wong (2004) reported that the IR spectrum of indium(III) complex with chalcone showed a new strong absorption band at 2987.0 cm$^{-1}$, which is due to the C-H stretching frequency. This proves that tautomerization occurred during the complexation of indium(III) with chalcone. A new absorption band occurred at 505.3 cm$^{-1}$ due to a new In-O bond between the indium and oxygen atom from the ligand. The study postulated that the complexation between indium(III) with chalcone compound gave a calculated ratio of In:ligand as 1:3.

2.5 Complexation of Strontium

Dietz and Jense, 2004, have conducted a complexation study of strontium ion absorbed from an aqueous nitric acid solution on an extraction chromatographic resin. Measurements they made using extended X-ray absorption fine-structure (EXAFS) to determine the coordination environment of strontium ions. It was found that the stoichiometric ratio of strontium:crown ether:nitrate is 1:1:2, which is consistent with the metal ions absorbed onto the resin. This report shows that the strontium ion is enveloped in the crown ether ring and is a bidentate ligand as both the nitrate anions are coordinated to the metal.
Kazak and his co-workers, 2003, studied on an eight-coordinate strontium complex with two tetradentate triethanolamine (tea) ligands. The reaction was carried out by adding tea drop by drop into a methanol solution containing SrCl₂.6H₂O and sodium saccharine (Nasac.2H₂O). The solution was stirred continuously for 1 hour at room temperature. The reaction yielded 80% of crystallized compound. The crystallization process occurred in an orthorhombic system and resulted in an eight-coordinated strontium complex with bicapped trigonal prism geometry. From the IR spectrum, the complex gave a strong and broad band in the range of 3200-3600 cm⁻¹ due to the vibration of the hydroxyl group of tea. The band is also probably due to the lattice water in Sr(sac)₂ which has strong hydrogen bond. At around 2900 cm⁻¹, weak bands occurred corresponding to C-H vibrations of the CH₂ from the tea ligand. Strong absorption bands at 1616 and 1625 cm⁻¹ and at 1578 and 1475 cm⁻¹ appeared due to carbonyl (C=O) and C-C vibrations. While at frequency range below 600 cm⁻¹, weak band of Sr-O stretching was observed.

Shen et al., 2004, synthesized and characterized 4,4,4-trifluoro-1-(2-thienyl)-1,3-butanedionato strontium complex. The complexes were stabilized by using oxygen and nitrogen containing ancillary ligands. The complexation reaction yielded approximately 91% based on strontium. Observation on the IR spectrum of the complex showed weak absorption bands at 574 and 466 cm⁻¹. These weak bands are corresponding to the Sr-O vibrations. It was also found that proton chemical shifts of ancillary ligand tetraglyme were shifted down-field from 3.30-3.52 ppm to 3.36-3.84 ppm as compared to the free tetraglyme ligand. The chemical shift was due to the coordination of oxygen atom to the metal ion.
CHAPTER 3 MATERIALS AND METHODS

3.1 Experimental

The chemicals involved in this study are:

i. Ethanol 99%

ii. Acetophenone

iii. Benzaldehyde

iv. 4-hydroxybenzaldehyde

v. 4-hydroxyacetophenone

vi. Potassium hydroxide

vii. Hydrochloric acid

viii. Concentrated sulfuric acid

ix. Anhydrous Magnesium Sulphate

x. Dichloromethane

xi. Methanol 99%

xii. Isopropanol 99%

xiii. Triethylamine

xiv. Strontium(II) chloride

xv. Indium(III) bromide
3.2 Synthesis of Chalcone

3.2.1 1-phenyl-3(4-hydroxy-phenyl)-propenone (R1)

Potassium hydroxide, KOH (1.00 g, 0.018 moles) was dissolved in 10 ml of ethanol 95% in a 250 ml conical flask. The solution was put at room temperature. Acetophenone (0.62 g, 0.004 moles) was added into the solution followed by 4-hydroxybenzaldehyde (0.50 g, 0.004 moles). The solution was stirred for 4 hours before being put at room temperature. At the end of the reaction, the solution was cooled in an ice bath and drop by drop of HCl 8 M was added in until an acidic condition is formed. Light yellow solid precipitates were obtained. The precipitates are filtered with Funnel filter before being air-dried. Yield, 0.57 g, 64%.

3.2.2 1(4-hydroxy-phenyl)-3-phenyl-propenone (R2)

The procedure to synthesis R2 is similar to those described in Section 3.2.1 except 4-hydroxybenzaldehyde was replaced with benzaldehyde (0.50 g, 0.004 moles). Yield, 1.03 g, 96%.

3.2 Etherification of Chalcone

3.3.1 Synthesis of MeO.R1 (R3)

Anhydrous magnesium sulphate, MgSO₄ (4.81 g, 40 mmoles) was dissolved in dichloromethane (40 ml) and was added concentrated sulfuric acid (0.55 ml, 10 mmoles) and the mixture was stirred for 15 minutes. R1 (5.34 g, 10 mmoles) followed by methanol (2.0 ml, 50 mmoles) were added into the mixture and the stirring continued for 48 hours continuously. The excess solvent was evaporated under open air condition and the product was collected. Yield 0.42 g, 18%.
3.3.2 Synthesis of EtO.R1 (R4)

The procedure of chalcone etherification to ethyl chalcone is similar to those described in Section 3.3.1 except methanol was replaced with ethanol (2.8 ml, 50 mmoles). Yield, 0.12 g, 5%.

3.3.3 Synthesis of C(CH₃)₂O.R1 (R5)

The procedure of chalcone etherification to isopropyl chalcone is same to those described in Section 3.3.1 except methanol was replaced with isopropyl alcohol (3.8 ml, 50 mmoles). Yield, 0.10 g, 4%.

3.3 Synthesis of indium complexes by reaction with chalcone

3.4.1 Synthesis of InBr₃.R1

R1 (0.672 g, 0.003 moles), triethylamine (0.327 g, 0.003 moles), 16 ml of ethanol 99.8%, and indium(III) bromide (1.00 g, 0.003 moles) were mixed into a 100 ml three necked round bottom flask. The mixture was stirred until a homogenous aqueous phase was formed. The solution formed was refluxed at 94 °C for 4 hours under nitrogen condition. During the cooling phase, the excessive solvent was evaporated using a rotor vapor and a deep yellow solid formed. The solid was filtered and dried in a desiccator. Yield, 2.37 g, 14%.

3.4.2 Synthesis of InBr₃.R2

The procedure of InBr₃.R2 synthesis is close to the synthesis of InBr₃.R1 as described in Section 3.4.1 except R1 was replaced with R2 (0.836 g, 0.003 moles). Yield, 3.6 g, 21%.
3.4 Synthesis of strontium complexes by reaction with chalcone

3.5.1 Synthesis of SrCl$_2$.R1

The procedure of SrCl$_2$.R1 synthesis is similar to those described in Section 3.4.1 except InBr$_3$ was changed to SrCl$_2$ (0.476 g, 0.003 moles). Yield, 5.75 g, 50%.

3.5.2 Synthesis of SrCl$_2$.R2

The procedure of SrCl$_2$.R2 synthesis is similar to those described in Section 3.5.1 except R1 was replaced by R2 (0.836 g, 0.003 moles). Yield, 8.99 g, 78%.

3.5 Characterization

The chalcone’s infrared spectra were recorded using the Shimazu 8001PC Fourier-Transform Infrared Spectrophotometer. The wavelength range used is between 4000 and 400 cm$^{-1}$. Samples in powder form were pressed into disc using pure potassium bromide (KBr). The C, H and N percentages were recorded using Perkin Elmer CHN Series II 2400. $^1$H-NMR spectra were recorded in CDCl$_3$ and DMSO-d$_6$ solution on Advance 400 MHz Bruker FT-NMR Spectrometer.
CHAPTER 4 RESULTS

The physical appearance and analytical data of R1-R2 and their complexes are presented in Table 4.1 and physical and analytical data of etherified R1 (R3-R5) are shown in Table 4.2. Table 4.3 and Table 4.4 show the IR data recorded whereas the $^1$H-NMR data are presented in Table 4.6.

Table 4.1: Physical and Analytical data

<table>
<thead>
<tr>
<th>Compound</th>
<th>Formula</th>
<th>Colour</th>
<th>M.P (°C)</th>
<th>Found (Calcd.) %</th>
</tr>
</thead>
<tbody>
<tr>
<td>R1</td>
<td>$C_{15}O_2H_{12}$</td>
<td>Pale brown</td>
<td>186 - 188</td>
<td>C 35.34 H 2.00 N -</td>
</tr>
<tr>
<td>SrCl$_2$.R1</td>
<td>$SrCl_2.C_{15}O_2H_{12}$</td>
<td>Brown</td>
<td>&gt;200</td>
<td>C (80.36) H (5.36) N -</td>
</tr>
<tr>
<td>InBr$_3$.R1</td>
<td>InBr$<em>3$. $(C</em>{15}O_2H_{12})_3$</td>
<td>Yellow</td>
<td>178 - 184</td>
<td>C 77.73 H 5.08 N -</td>
</tr>
<tr>
<td>InBr$_3$.R1</td>
<td>InBr$<em>3$. $(C</em>{15}O_2H_{12})_3$</td>
<td>Brown</td>
<td>&gt;200</td>
<td>C (47.04) H (3.14) N -</td>
</tr>
</tbody>
</table>

Table 4.2: Physical data of R3, R4 and R5

<table>
<thead>
<tr>
<th>Compound</th>
<th>Formula</th>
<th>Colour</th>
<th>M.P (°C)</th>
</tr>
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<tbody>
<tr>
<td>R3</td>
<td>CH$_3$O.R1</td>
<td>dark yellow</td>
<td>154-159</td>
</tr>
<tr>
<td>R4</td>
<td>C$_2$H$_5$O.R1</td>
<td>dark brown</td>
<td>178-182</td>
</tr>
<tr>
<td>R5</td>
<td>C(CH$_3$)$_2$O.R1</td>
<td>dark brown</td>
<td>105-110</td>
</tr>
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</table>