UNIVERSITI PUTRA MALAYSIA

PREPARATION OF NANO METAL SULPHIDES BY THERMAL DECOMPOSITION OF SYNTHESIZED METAL DITHIOCARBAMATE COMPLEXES

NURUL HIDAYAH BINTI ABDULLAH

FS 2015 25

PREPARATION OF NANO METAL SULPHIDES BY THERMAL DECOMPOSITION OF SYNTHESIZED METAL DITHIOCARBAMATE COMPLEXES

By

NURUL HIDAYAH BINTI ABDULLAH

Thesis Submitted to the School of Graduate Studies, Universiti Putra Malaysia, in Fulfilment of the Requirements for the Degree of Master of Science

March 2015

Abstract of thesis presented to the Senate of Universiti Putra Malaysia in fulfilment of the requirement for the degree of Master of Science

PREPARATION OF NANO METAL SULPHIDES BY THERMAL DECOMPOSITION OF SYNTHESIZED METAL DITHIOCARBAMATE COMPLEXES

By

NURUL HIDAYAH BINTI ABDULLAH

March 2015

Chairman : Zulkarnain bin Zainal, PhD
Faculty : Science

Metal sulphides are important semiconductor materials, which are useful in various applications such as solar cells, optical coatings, photoconductors, and transductors. In this study, several metal dithiocarbamates have been synthesized as single source precursors for metal sulphides production. The preparation of metal dithiocarbamate was done from the reaction between N-ethylcyclohexanamine with carbon disulphide and metal salt in alkaline media. Some difficulties related to high reaction temperatures, precursors instability, and difficult synthetic procedures have somewhat limited the synthesis of single precursor to certain metal complexes. These compounds were found to be effective precursors for nanomaterial fabrication by thermal decomposition of metal dithiocarbamates in a tube furnace. Considering the wide scope of the subject, current research is restricted to the dithiocarbamates with eight metals, namely, zinc(II), nickel(II), copper(II), cobalt(II), cadmium(II), indium(III), antimony(III), and bismuth(III). Besides, no surfactant was used in this study due to the toxicity although many researchers preferred to use surfactants to control the sizes and shapes of the final product. All metal complexes were characterized by Fourier transform infrared (FT-IR) spectroscopy, carbon, hydrogen, nitrogen and sulphur (CHNS) analyses, direct injection mass spectrometry (DIMS) analysis, thermal gravimetric and differential thermal gravimetric (TGA/DTG) analyses, differential scanning calorimetry (DSC), inductively coupled plasma (ICP) and single crystal X-ray diffraction (XRD) analysis. The physical and elemental analyses of Zn, Cd, Cu dan Ni dithiocarbamates (DTC) complexes were confirmed to have the molecular formula of $C_{18}H_{32}N_2S_4M$ (where M is for Zn, Cd, Cu and Ni metal). On the other hand, the molecular formula of $C_{27}H_{48}N_3S_6M$ was confirmed for Co, Sb, In, and Bi-DTC. The formation of Zn, Ni, Cu and Sb-DTC crystals were analyzed by single crystal X-ray diffraction analysis to determine the detailed molecular geometry and intermolecular interaction. Thermal study indicated the percentages of residues left were close to the theoretical values attributed to the decomposition of the organic species of the complex.

The synthesized metal dithiocarbamates were subjected to thermal treatment at different calcination durations for the preparation of nano metal sulphides. The properties of the metal sulphides were characterized by powder X-ray diffraction (XRD), field emission scanning electron microscopy (FESEM), energy dispersive X-ray (EDX), transmission electron microscopy (TEM) and ultraviolet-visible (UV-Vis) reflectance spectroscopy. From XRD spectra, metal sulphides were obtained after heat treatment of metal dithiocarbamates for 2, 4 and 6 h at temperature 400 °C. No impurities were observed from the spectra indicated the purity of the product. Different calcination durations did not affect the crystalline phase but only cause a slight change in the peak intensities. FESEM and TEM showed some of the metal sulphides were in the form of nanoparticles, nanowhiskers, and nanorods. However some of them were agglomerated. Energy Dispersive X-Ray (EDX) analysis showed the composition of metal sulphides were close to the theoretical values. The direct band gap observed for all metal sulphides except for cadmium sulphide indicated the blue shift if compared to the bulk sample. The band gap energies of the resultant metal sulphides could possess interesting optical properties and might have significance for future nanoscale device applications.

Abstrak tesis yang dikemukakan kepada Senat Universiti Putra Malaysia sebagai memenuhi keperluan untuk ijazah Sarjana Sains

PENYEDIAAN NANO LOGAM SULFIDA DARIPADA PENGURAIAN TERMA LOGAM DITIOKARBAMAT KOMPLEKS YANG DISINTESIS

Oleh

NURUL HIDAYAH BINTI ABDULLAH

Mac 2015

Pengerusi : Zulkarnain bin Zainal, PhD
Fakulti : Sains

Logam sulfida adalah bahan semikonduktor penting yang boleh digunakan dalam pelbagai aplikasi seperti sel solar, lapisan optik, fotokonduktor dan transduktor. Dalam kajian ini, beberapa logam ditiokarbamat telah disintesis sebagai prekursor sumber tunggal untuk penghasilan logam sulfida bersaiz nano. Penyediaan logam ditiokarbamat dilakukan daripada tindak balas antara N-etilsikloheksilamina dengan karbon disulfida dan garam logam di dalam medium beralkali. Beberapa masalah yang berkaitan dengan suhu tindak balas yang tinggi, ketidakstabilan prekursor, dan posedur sintetik yang sukar telah menyebabkan sedikit sebanyak limitasi sintesis prekursor tunggal kepada kompleks logam tertentu. Sebatian ini telah didapati boleh dijadikan prekursor yang efektif untuk fabrikasi bahan nano melalui penguraian terma logam ditiokarbarbamat di dalam relau tiub. Memandangkan skop subjek yang luas, kajian ini adalah terhad kepada ditiokarbamat dengan lapan logam, iaitu zink(II), nikel(II), kuprum(II), kobalt(II), kadmium(II), indium(III), antimoni (III), dan bismut (III). Selain itu, tiada surfaktan yang digunakan di dalam kajian ini kerana ketoksikan walaupun ramai penyelidik lebih suka untuk menggunakan surfaktan untuk mengawal saiz dan bentuk produk akhir. Semua logam kompleks dilakukan pencirian menggunakan analisis spektroskopi inframerah (FTIR), karbon, hidrogen, nitrogen, sulfur (CHNS) analisis, spektroskopi jisim suntikan secara terus (DIMS), termogravimetri analisis dan termogravimetri pembezaan (TGA/DTG), kalorimetri pengimbas pembezaan (DSC), induktif plasma pasangan (ICP), dan analisis kristal tunggal pembelauan sinar-X (XRD). Sifat fizikal dan analisis elemen bagi Zn, Cd, Cu dan Ni ditiokarbamat (DTC) kompleks mengesahkan formula molekul adalah $C_{18}H_{32}N_2S_4M$ di mana (M= logam Zn, Cd, Cu dan Ni). Manakala formula molekul $C_{27}H_{48}N_3S_6M$ telah dipastikan bagi Co, Sb, In, and Bi-DTC. Pembentukan kristal bagi Zn, Ni, Cu dan Sb-DTC telah dianalisis menggunakan kristal tunggal pembelauan sinar-X analisis untuk menentukan geometri molekul secara terperinci dan interaksi antara molekul. Kajian terma menunjukkan peratus baki yang tinggal adalah hampir sama dengan nilai teori yang terhasil daripada penguraian bahagian kompleks organik.

Logam ditiokarbamat yang disintesis telah dilakukan rawatan terma pada tempoh pengkalsinan yang berbeza untuk penghasilan nano logam sulfida. Sifat-sifat logam sulfida telah dicirikan menggunakan serbuk pembelauan sinar-X (XRD), mikroskopi pengimbas pancaran medan elektron (FESEM), penyerakan tenaga sinar-X (EDX), mikroskopi transmisi elektron (TEM) dan pantulan spektroskopi ultraungu cahaya nampak (UV-Vis). Daripada spektrum-spektrum XRD, logam sulfida telah terhasil selepas haba terma dikenakan ke atas logam ditiokarbamat selama 2, 4 dan 6 jam pada suhu 400 °C. Tiada bendasing yang dilihat di dalam spektrum-spektrum menunjukkan keaslian produk. Perubahan tempoh pengkalsinan tidak memberikan kesan terhadap fasa kristal tetapi menyebabkan sedikit perubahan berlaku pada intensiti puncak-puncak. FESEM dan TEM menunjukkan sebahagian daripada logam sulfida adalah di dalam bentuk nanopartikel, nanowiskes, dan nanorod. Walaubagaimanapun, sebahagian daripadanya adalah bergumpal. Analisis penyerakan tenaga sinar-X (EDX) menunjukkan komposisi logam sulfida adalah hampir sama dengan nilai teori. Jurang tenaga langsung yang terhasil untuk semua logam sulfida kecuali kadmium sulfida menunjukkan anjakan biru berbanding dengan bahan bersaiz besar. Nilai jurang tenaga bagi logam sulfida yang terhasil mempunyai ciri-ciri optik yang menarik dan mungkin mempunyai kepentingan dalam aplikasi peranti bersaiz nano pada masa akan datang.

ACKNOWLEDGEMENTS

In The Name of ALLAH S.W.T., the Most Merciful, Most Compassionate
For the Blessing and Strength

First and foremost, I would like to express my sincere and deepest appreciation to my supervisor, Prof. Dr. Zulkarnain bin Zainal, for his valuable suggestions, guidance, encouragement and inspiration throughout this study. My pleasure thanks also to my co-supervisors, Dr. Tan Kar Ban, Dr. Mohamed Ibrahim Mohamed Tahir and Dr. Sidik bin Silong for their supervision and invaluable advice and cooperation. Special thanks are extended to all the laboratory assistants in Chemistry Department, who contribute directly or indirectly in my research.

I would like to share my happiness at this moment with my friends (Saudah, Aqilah, Rebi, Atun, Alia, Kak Bem and Kak Ain) who helped me a lot and give useful advice and suggestions. Last but not least, I would like to express my deepest gratitude to my beloved family, Abdullah bin Long, Zainun binti Hassan, and all my siblings who has always believed in me, understand and give full support during difficult times. Without their unconditional and endless love, it would not have been possible for me to complete the study. This study supported by Universiti Putra Malaysia and Graduate Research Fellowship (GRF). Their financial support is gratefully acknowledged.

I certify that a Thesis Examination Committee has met on 19 March 2015 to conduct the final examination of Nurul Hidayah binti Abdullah on her thesis entitled "Preparation of Nano Metal Sulphides by Thermal Decomposition of Synthesized Metal Dithiocarbamate Complexes" in accordance with the Universities and University Colleges Act 1971 and the Constitution of the Universiti Putra Malaysia [P.U.(A) 106] 15 March 1998. The Committee recommends that the student be awarded the Master of Science.

Members of the Thesis Examination Committee were as follows:

Abdul Halim bin Abdullah, PhD
Associate Professor
Faculty of Science
Universiti Putra Malaysia
(Chairman)

Tan Yen Ping, PhD
Senior Lecturer
Faculty of Science
Universiti Putra Malaysia
(Internal Examiner)

Wan Azelee Wan Abu Bakar, PhD
Professor
Universiti Teknologi Malaysia
Malaysia
(External Examiner)

ZULKARNAIN ZAINAL, PhD
Professor and Deputy Dean
School of Graduate Studies
Universiti Putra Malaysia

Date: 13 May 2015

This thesis was submitted to the Senate of Universiti Putra Malaysia and has been accepted as fulfilment of the requirement for the degree of Master of Science. The members of Supervisory Committee were as follows:

Zulkarnain Zainal, PhD
Professor
Faculty of Science
Universiti Putra Malaysia
(Chairman)

Mohamed Ibrahim Mohamed Tahir, PhD
Senior Lecturer
Faculty of Science
Universiti Putra Malaysia
(Member)

Tan Kar Ban, PhD
Senior Lecturer
Faculty of Science
Universiti Putra Malaysia
(Member)

BUJANG KIM HUAT, PhD
Professor and Dean
School of Graduate Studies
Universiti Putra Malaysia

Date:

Declaration by graduate student

I hereby confirm that:

- this thesis is my original work;
- quotations, illustrations and citations have been duly referenced;
- this thesis has not been submitted previously or concurrently for any other degree at any other institutions;
- intellectual property from the thesis and copyright of thesis are fully-owned by Universiti Putra Malaysia, as according to the Universiti Putra Malaysia (Research) Rules 2012;
- written permission must be obtained from supervisor and the office of Deputy Vice-Chancellor (Research and Innovation) before thesis is published (in the form of written, printed or in electronic form) including books, journals, modules, proceedings, popular writings, seminar papers, manuscripts, posters, reports, lecture notes, learning modules or any other materials as stated in the Universiti Putra Malaysia (Research) Rules 2012;
- there is no plagiarism or data falsification/fabrication in the thesis, and scholarly integrity is upheld as according to the Universiti Putra Malaysia (Graduate Studies) Rules 2003 (Revision 2012-2013) and the Universiti Putra Malaysia (Research) Rules 2012. The thesis has undergone plagiarism detection software.

Signature: _______________________ Date: __________________

Name and Matric No.: Nurul Hidayah binti Abdullah (GS27920)

Declaration by Members of Supervisory Committee

This is to confirm that:

- the research conducted and the writing of this thesis was under our supervision;
- supervision responsibilities as stated in the Universiti Putra Malaysia (Graduate Studies) Rules 2003 (Revision 2012-2013) are adhered to.

Signature: ________________________
Name of Chairman of Supervisory Committee: **Zulkarnain bin Zainal**

Signature: ________________________
Name of Member of Supervisory Committee: **Tan Kar Ban**

Signature: ________________________
Name of Member of Supervisory Committee: **Mohamed Ibrahim bin Mohamed Tahir**

TABLE OF CONTENTS

Page

CHAPTER

1. INTRODUCTION

2. LITERATURE REVIEW

3. MATERIALS AND METHODOLOGY

4. RESULTS AND DISCUSSION

4.2 Characterization of Metal Sulphides

LIST OF TABLES

LIST OF FIGURES

Figure **Page**

LIST OF ABBREVIATIONS

CHNS	Carbon, Hydrogen, Nitrogen and Sulphur
ICP-AES	Inductively Coupled Plasma-Atomic Emission Spectroscopy
ICP-OES	Inductively Coupled Plasma-Optical Emission Spectroscopy
MS	Mass Spectrometry
DIMS	Direct Injection Mass Spectrometry
GCMS	Gas Chromatograph-Mass Spectrometer
FT-IR	Fourier Transform Infrared
TGA	Thermal Gravimetric Analysis
DSC	Differential Scanning Calorimetry
DTG	Differential Thermal Gravimetric
XRD	X-Ray Diffraction
FWHM	Full width at half maximum
FE-SEM	Field Emission Scanning Electron Microscopy
EDX	Energy Dispersive X-Ray
TEM	Transmission Electron Microscopy
UV-Vis-NIR	Ultraviolet-visible-near infrared
DTC	Dithiocarbamate
MDTC	Metal Dithiocarbamate
UATR	Universal Attenuated Total Reflection
JCPDS	Joint Committee of Powder Diffraction Standards
NCs	Nanocrystals
1D	One-dimensional
TOPO	Trioctyl phosphine oxide
TOP	Trioctyl phosphine
OA	Oleic acid
OM	Oleylamine
ODE	Octadecene
HDA	Hexadecylamine
CVTC	Chemical vapour transport-condensation
MOCVD	Metal organic chemical vapour deposition
PAA	Porous anodic alumina
DMF	Dimethyl formamide
AACVD	Aerosol-assisted chemical vapour deposition
VLS	Vapour–liquid–solid technique
M	Metal
RB	Rhodamine B
EDTA	Ethylenediaminetetraacetic acid
SDS	Sodium dodecylsulphate
PC	Photoconductivity

CHAPTER 1

INTRODUCTION

1.1 General Introduction of Dithiocarbamates

Dithiocarbamates are versatile chelating agents with generic anion formula of $-S_2CNR'_2$ (Figure 1.1) that are well known important class of metal-coordinating agents that bind strongly and selectively to many metal ions (Jayaraju *et al.*, 2012; Nabipour *et al.*, 2010; Tiekink, 2008). The resonance form (II), has significant contribution towards the stability of such complexes by ensuring that this anion is a very effective ligand for metals (Tiekink, 2008). Preparation of dithiocarbamates compound have been studied extensively due to their potential applications for treatment of bacterial and fungal infections, possible treatment of AIDS, anticancer agents and as synthetic precursors for the deposition of metal sulphide nanoparticles (Buac *et al.*, 2012, Jayaraju *et al.*, 2012; Nabipour *et al.*, 2010; Tiekink, 2008; Xie *et al.*, 2004; Faraglia *et al.*, 2001). Among the sulphur ligands, the dithiocarbamate species (RR-NCS_2−) with 3-electron donors are important family of classical anionic ligands that capable of stabilising metal centres in a variety of oxidation states (Dutta *et al.*, 2002).

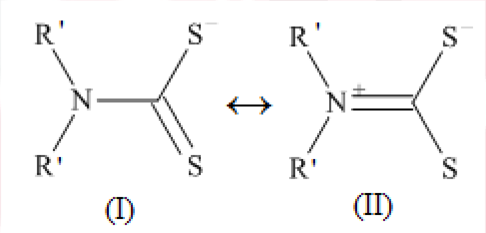


Figure 1.1: Generic structures for (I) the dithiocarbamate anion, (II) an important resonance structure for the dithiocarbamate anion (Tiekink, 2008)

1.2 Metal Dithiocarbamates as Single Source Precursors

In recent years, metal dithiocarbamates have attracted increasing attention on account of their potential as single source precursors for preparation of metal sulphide (Xie *et al.*, 2004; Romano and Alves, 2006). The utilization of single source precursors for preparation of metal sulphides offer several advantages owing to the synthesis process can be conducted under anaerobic condition which is important due to some of group II-VI and III-V are air sensitive. Furthermore, the use of volatile, sometimes toxic and/or pyrophoric precursors also can be avoided. Besides, the utilization of one volatile precursor can make the purification be handled easily than that of two or more volatile precursors that mostly contribute to the impurities into the nanoparticles. In addition, low temperature deposition routes are also possible for production of semiconductor nanoparticles. Hence, these factors can affect the optical properties of the compound relative to the predicting optical properties owing to the development of particles with various sizes and shapes (Pickett and Brien, 2001).

Many potential single-molecular precursors have been synthesized but some problems related to low volatility and lacks of stoichiometric control are still the main concern for researchers. In most cases, the formation of large particles possessing wide size distributions were attained owing to the solid state thermolysis method employed and the absence of a capping agent, both of which fail to separate the nucleation and growth processes. The agglomeration of the particles also occurs due to the incomplete coordination of surface atoms in nanoparticles which makes them highly reactive. Thus, this problem is overcomed by passivating (capping) the "bare" surface atoms with protecting groups which give benefit by protecting the particle from its surrounding environment, and provides electronic stabilization to the surface. Generally, the capping agent takes the form of a Lewis base compound covalently bound to surface metal atoms but other compound such as an organic polymer forming a sheaf around the particle, or an organic group bonded directly to the surface, have been employed (Pickett and Brien, 2001).

1.3 Synthetic procedures of metal dithiocarbamates

There are two different techniques have been employed for preparation of metal dithiocarbamates. The first technique involved the preparation of ligand, followed by the synthesis of metal complexes in two separate processes (Mthethwa *et al.*, 2009; Jayaraju *et al.*, 2012). Meanwhile, other techniques involved one pot synthesis of direct reaction between metal salt with carbon disulphide, and secondary amine in methanol solution (Dutta *et al.*, 2002; Sivagurunathan *et al.*, 2014). The preparation of metal dithiocarbamates has been reported extensively by facile reaction between metal salt with carbon disulphide and secondary amine in ethanol or methanol solution (Nomura *et al.*, 1987; Yin *et al.*, 2008; Awang *et al.*, 2011; Sivagurunathan *et al.*, 2014; Breviglieri *et al.*, 2000). Other solvents such as acetone, acetonitrile, benzene and chloroform have been used for synthesizing metal dithiocarbamates (Nomura *et al.*, 1987; Oliveira *et al.*, 1999). Most of the reaction was conducted in ice cold solution between 0 to 5 °C (Awang *et al.*, 2011; Sivagurunathan *et al.*, 2014). However, room temperature preparation of gallium (III), indium (III), copper (II), nickel (II) and manganese (II) dithiocarbamates also have been reported with high yield of products (Dutta *et al.*, 2002; Jayaraju *et al.*, 2012; Travnicek *et al.*, 2008). Potassium hydroxide and sodium hydroxide have been utilized to provide basicity of solution for preparation of metal dithiocarbamates (Wang *et al.*, 2009; Jayaraju *et al.*, 2012).

Up to now, several ligands have been used for preparation of single source precursors for metal sulphide production. For example diethyldithiocarbamate has been used for synthesis of PbS, Cu_2S, In_2S_3 and ZnS (Plante *et al.*, 2010; Acharya *et al.*, 2012; Zhai *et al.*, 2006). Meanwhile, the preparation of HDA capped ZnS, CdS and HgS nanoparticles have been reported by utilizing N-methyl-N-phenyl dithiocarbamate. Furthermore, Tris (N,N-ethylbutyl dithiocarbamate) was used to synthesize In_2S_3 thin films. Pyrrolidine dithiocarbamate also has been used for preparation of CdS nanocrystals (Nirmal *et al.*, 2011). Besides, N-methyl-N-phenyl dithiocarbamate has been used for preparation of ZnS, CdS and HgS nanoparticles (Onwudiwe and Ajibade, 2011a). In addition, piperidine dithiocarbamate (DTC), has been used for preparation of HDA capped CdS nanoparticles (Mththewa *et al.*, 2009). Other researchers also demonstrated the utilization of N-ethylbutyldithiocarbamate and 2-ethylpiperidinedithiocarbamate for preparation of ZnS thin film (Seo *et al.*, 2005). On the other hand, dialkyldithiocarbamate has been used for preparation of Bi_2S_3 nanoparticles and nanorods (Monteiro *et al.*, 2001; Wang *et al.*, 2009).

1.4 Problem Statement and Scope of Research

Some difficulties related to high reaction temperatures, precursors instability, and lengthy synthetic procedures have limited the single precursor approach to certain metal complexes. Therefore, in this study the synthesized metal dithiocarbamates will be investigated to determine the suitability of these precursors for metal sulphides production. Considering the wide scope of the subject, present research was restricted to the dithiocarbamates derived from nickel(II), copper(II), zinc(II), cobalt(II), cadmium(II), In(III), Sb(III), and Bi(III). N-ethyl cyclohexanamine was used as an amine source for preparation of metal dithiocarbamates. Although some of the synthesized metal dithiocarbamates have been reported before but the study only focused on the application in biological activities. In this study, the synthesized metal dithiocarbamates were used as precursors for metal sulphides production for application in the semiconductor field. Thermal decomposition of the synthesized metal dithiocarbamates at different calcination times was investigated to study the effects on the morphology, particle sizes and band gap energy of the product obtained. Metal dithiocarbamates were characterized by Fourier transform infrared (FT-IR) spectroscopy, carbon, hydrogen, nitrogen and sulphur (CHNS) analyses, direct injection mass spectrometry (DIMS) analysis, thermal gravimetric and differential thermal gravimetric (TGA/DTG) analyses, differential scanning calorimetry **(**DSC), inductively coupled plasma (ICP) and single crystal X-ray diffraction (XRD) analysis. On the other hand, the synthesized metal dithiocarbamates were subjected to thermal treatment at different calcination durations for the preparation of nano metal sulphides. The properties of the metal sulphides were characterized by powder X-ray diffraction (XRD), field emission scanning electron microscopy (FESEM), energy dispersive X-ray (EDX), transmission electron microscopy (TEM) and ultraviolet-visible (UV-Vis) reflectance spectroscopy.

1.5 Research Objectives

The objectives of this research are:

1. To prepare eight metal dithiocarbamate precursors derived from copper(II), nickel(II), cadmium(II), zinc(II), cobalt(II), indium(III), antimony(III) and bismuth(III) by *in-situ* chemical reaction between N-ethyl cyclohexanamine, carbon disulphide, potassium hydroxide and metal salt in ethanolic solution.
2. To characterize metal dithiocarbamates using various phsico-chemical, spectroscopic and where possible, single crystal X-Ray Diffraction analyses.
3. To prepare metal sulphides from thermal decomposition of metal dithiocarbamates at different calcination times of 2, 4 and 6 hours.
4. To characterize metal sulphides and determine effects of different calcination times on the phases, morphologies, particle sizes and band gap energy.

REFERENCES

Agilent. (2011). CrysAlis PRO Agilent Technologies UK Ltd, Yarnton, England.

Abadi, P.G.S., Niasari, M.S., and Davar, F. (2013). Hydrothermal synthesis, characterization and optical properties of 3D flower like indium sulfide nanostructures. *Superlattices and Microstructures* 53: 76–88.

Abbas, A., Reza, A., Borujeni, A., Najafi, M., and Bagheri, A. (2010). Ultrasonic/surfactant assisted of CdS nano hollow sphere synthesis and characterization. *Materials Characterization* 62(1): 94–98.

Acharya, S., Dutta, M., Sarkar, S., Basak, D., Chakraborty, S., and Pradhan, N. (2012). Synthesis of Micrometer Length Indium Sulfide Nanosheets and Study of Their Dopant Induced Photoresponse Properties. *Chemistry of Materials* 24: 1779−1785.

Afzaal, M., Malik, M.A., and O'Brien, P. (2004). Indium sulfide nanorods from single-source precursor. *Chemical Communications*: 334–335.

Ajibade, P.A., and Ejelonu, B.C. (2013). Group 12 dithiocarbamate complexes:Synthesis, spectral studies and their use as precursors for metal sulfides nanoparticles and nanocomposites. *Spectrochimica Acta Part A: Molecular and Biomolecular Spectroscopy* 113: 408–414.

Alemi, A., Hanifehpour, Y., and Joo, S.W. (2011). Synthesis and Characterization of Sb_2S_3 Nanorods via Complex Decomposition Approach. *Journal of Nanomaterials*: 1-5 doi:10.1155/2011/186528.

Almond, M.J., Redman, H., and Rice, D.A. (2000). Growth of thin layers of metal sulfides by chemical vapour deposition using dual source and single source precursors: routes to Cr_2S_3 , α-MnS and FeS. *Journal of Materials Chemistry* 10: 2842–2846.

Ariponnammal, S., and Srinivasan, T. (2013). Growth and Characterization of Cobalt Sulphide Nanorods. *Research Journal of Recent Sciences* 2: 102–105.

Aso, K., Kitaura, H., Hayashi, A., and Tatsumisago, M. (2011). Synthesis of nanosized nickel sulfide in high-boiling solvent for all-solid-state lithium secondary batteries. *Journal of Materials Chemistry* 21: 2987-2990.

Atay, F., Kose, S., Bilgin, V., and Akyuz, I. (2003). Electrical, Optical, Structural and Morphological Properties of NiS Films. *Turkish Journal of Phys*ics 27: 285–291.

Awang, N., Baba, I., and Yamin, B.M. (2011). Synthesis, Characterization and Crystal Structure of Triphenyltin (IV) N-alkyl-N-cyclohexyldithiocarbamate Compounds. *World Applied Sciences Journal* 12(5): 630–635.

Bao, H., Cui, X., Li, C.M., Gan, Y., Zhang, J., and Guo, J. (2007). Photoswitchable Semiconductor Bismuth Sulfide (Bi_2S_3) Nanowires and Their Self-Supported Nanowire Arrays. *Journal of Physical Chemistry C* 111: 12279–12283.

Bao, S., Li, C.M., Guo, C., and Qiao, Y. (2008). Biomolecule-assisted synthesis of cobalt sulfide nanowires for application in supercapacitors. *Journal of Power Sources* 180: 676–681.

Begum, A., Hussain, A., and Rahman, A. (2011). Preparation and Characterization of Bismuth Sulphide Nanocrystalline Thin Films by Chemical Bath Deposition Method in Acidic Aqueous Media. *Chalcogenides Letters 8*(4): 283–289.

Benedini, V.D., Antunes, P.A., Cavalheiro, É.T.G., and Chierice, G.O. (2006). Thermoanalytical and Solution Stability Studies of Hexamethylenedithiocarbamates. *Journal of the Brazilian Chemical Society* 17(4): 680–688.

Birol, H., Rambo, C. R., Guiotoku, M., and Hotza, D. (2013). Preparation of ceramic nanoparticles via cellulose- assisted glycine nitrate process: a review. *RSC Advances* 3: 2873–2884.

Biswal, J.B., and Garje, S.S. (2013). Preparation of Antimony Sulfide Nanostructures From Single-Source Antimony Thiosemicarbazone Precursors Jasmine. *Synthesis and Reactivity in Inorganic, Metal-Organic, and Nano-Metal Chemistry* 43: 461–465.

Biswal, J.B., Sawant, N.V, and Garje, S.S. (2010). Deposition of rod-shaped antimony sulfide thin films from single-source antimony thiosemicarbazone precursors. *Thin Solid Films* 518(12): 3164–3168.

Black, J., Conwbll, E.M., Electric, S., Seigle, L., and Spencer, C.W. (1957). Electrical and Optical Properties of Some $M_2^{V\text{-}B}N_3^{V1\text{-}B}$ Semiconductors. *Journal of Physics and Chemistry of Solids* 2: 240–251.

Boadi, N.O., Malik, A., Brien, O., Awudza, J.A.M., and Brien, P.O. (2012). Single source molecular precursor routes to lead chalcogenides. *Dalton Transactions* 41: 10497–10506.

Bonati, F., and Ugo, R. (1967). Organotin(iv) n,n-disubstituted dithiocarbamates. *Journal of Organometallic Chemistry* 10: 257–268.

Borah, J.P., Barman, J., and Sarma, K.C. (2008). Structural And Optical Properties Of ZnS Nanoparticles. *Chalcogenides Letters* 5(9): 201–208.

Breviglieri, S.T., Cavalheiro, E.T.G., and Chierice, G.O. (2000). Correlation between ionic radius and thermal decomposition of Fe(II), Co(II), Ni(II), Cu(II) and Zn(II) diethanoldithiocarbamates. *Thermochimica Acta* 356: 79–84.

Buac, D., Schmitt, S., Ventro, G., Kona, F.R. and Dou, Q.P. (2012). Dithiocarbamate-Based Coordination Compounds as Potent Proteasome Inhibitors in Human Cancer Cells. *Mini Reviews in Medicinal Chemistry* 12(12): 1193–1201.

Cansizoglu, M. F., Engelken, R., Seo, H., and Karabacak, T. (2010). High Optical Absorption of Indium Sulfide Nanorod Arrays Formed by Glancing Angle Deposition. *Journal of the American Chemical Society* 4(2): 733–740.

Cao, F., Liu, W., Zhou, L., Deng, R., Song, S., Wang, S., Su, S., and Zhang, H. (2011). Well-defined Sb_2S_3 microspheres : High-yield synthesis, characterization, their optical and electrochemical hydrogen storage properties. *Solid State Sciences* 13(6): 1226–1231.

Cassagneau, T., Hix, G.B., Jones, D.J., Maireles-torres, P., Rhomari, M., and Roziere, J. (1994). Nano / nanocomposite Systems : In situ Growth of Particles and Clusters of Semiconductor Metal Sulfides in Porous Silica-pillared Layered Phosphates. *Journal of Materials Chemistry* 4(2): 189–195.

Castro, J.R., Dale, P., Mahon, M.F., Molloy, K.C., Peter, L.M. (2007). Deposition of Antimony Sulfide Thin Films from Single-Source Antimony Thiolate Precursors. *Chemistry of Materials* 19: 3219–3226.

Castro, J.R., Molloy, K.C., Liu, Y., Lai, S., Dong, Z., White, J., and Tiekink, E.R.T. (2008). Formation of antimony sulfide powders and thin films from single-source antimony precursors. *Journal of Materials Chemistry* 18: 5399–5405.

Chadha, R., Arora, P., Saini, A., and Jain, D.S. (2010). Solvated Crystalline Forms of Nevirapine : Thermoanalytical and Spectroscopic Studies. *AAPS PharmSciTech 2*(3): 1328–1339.

Chadha, R., Kuhad, A., Arora, P., and Kishor, S. (2012). Characterisation and evaluation of pharmaceutical solvates of Atorvastatin calcium by thermoanalytical and spectroscopic studies. *Chemistry Central Journal* 1–15.

Chakraborty, I., and Moulik, S.P. (2005). Preparation of CoS_2 Nanoparticles in Aqueous Micellar Media of Anionic Surfactants: AOT and SDS. *Journal of Surface Science and Technology* 21(3-4): 195–204.

Chandran, A., Francis, N., Jose, T., and and George, K.C. (2010). Synthesis, Structural Characterization and Optical Band Gap Determination of ZnS Nanoparticles. SB *Academic Review* XVII(1): 17–21.

Chao, J., Liang, B., Hou, X., Liu, Z., Xie, Z., Liu, B., Song, W., Chen, G., Chen, D., Shen, G. (2013). Selective synthesis of Sb_2S_3 nanoneedles and nanoflowers for high performance rigid and flexible photodetectors. *Optical Society of America* 21(11): DOI:10.1364/OE.21.013639.

Che, G., Lakshmi, B.B., Martin, C.R., Fisher, E.R., and Ruoff, R.S. (1998). Chemical Vapor Deposition Based Synthesis of Carbon Nanotubes and Nanofibers Using a Template Method. *Chemistry of Materials* 10: 260–267.

Chen, R., So, M.H., Che, C., and Sun, H. (2005). Controlled synthesis of high crystalline bismuth sulfide nanorods: using bismuth citrate as a precursor. *Journal of Materials Chemistry* 15: 4540–4545.

Deepshikha, and Basu, T. (2011). A Review on Synthesis and Characterization of Nanostructured Conducting Polymers (NSCP) and Application in Biosensors. *Analytical Letters* 44: 37–41.

Deshmukh, L.P., and Mane, S.T. (2011). Liquid Phase Chemical Deposition of Cobalt Sulphide Thin Films: Growth and Properties. *Digest Journal of Nanomaterials and Biostructures*, 6(3): 931–936.

Dumbrava, A., Badea, C., Prodan, G., Popovici, I., and Ciupina, V. (2009). Zinc Sulfide Fine Particles Obtaine at Low Temperature. *Chalcogenide Letters* 6(9): 437–443.

Dunne, P.W., Starkey, C.L., Gimeno-fabra, M., and Lester, E. H. (2014). The rapid size- and shape-controlled continuous hydrothermal synthesis of metal sulphide nanomaterials. *Royal Society of Chemistry* 6: 2406–2418.

Dutta, D.P., Jain, V.K., Knoedler, A., and Kaim, W. (2002). Dithiocarbamates of gallium (III) and indium (III): syntheses, spectroscopy, and structures. *Polyhedron* 21: 239–246.

Ehsan, M.A., Peiris, T.A.N., Wijayantha, K.G.U., Olmstead, M.M., Arifin, Z., Mazhar, M., Lo, K.M., and McKee, V. (2013). Development of molecular precursors for deposition of indium sulphide thin film electrodes for photoelectrochemical applications. *Dalton Transactions* 42(30): 10919–10928.

Emadi, H., Salavati-niasari, M., and Davar, F. (2012). Synthesis and characterization of cobalt sulfide nanocrystals in the presence of thioglycolic acid via a simple hydrothermal method. *Polyhedron* 31(1): 438–442.

Fang, B. X., Bando, Y., Liao, M., Gautam, U. K., Zhi, C., Dierre, B., Liu, B., Tianyou, Z., Takashi, S., Yasuo, K., Golberg, D. (2009). Single-Crystalline ZnS Nanobelts as Ultraviolet-Light Sensors. *Advanced Materials* 21: 2034–2039.

Fang, Z., Liu, Y., Fan, Y., Ni, Y., Wei, X., Tang, K., Shen, J., Chen, Y. (2011). Epitaxial Growth of CdS Nanoparticle on Bi_2S_3 Nanowire and Photocatalytic Application of the Heterostructure. *Journal of Physical Chemistry C* 115: 13968–13976.

Faraglia, G., Fregona, D., Sitran, S., Giovagnini, L., Marzano, C., Baccichetti, F., Casellato, U., Graziani, R. (2001). Platinum(II) and palladium(II) complexes with dithiocarbamates and amines : synthesis , characterization and cell assay. *Journal of Inorganic Biochemistry* 83: 31–40.

Gaba, M., and Dhingra, N. (2011). Microwave Chemistry: General Features and Applications. *Indian Journal of Pharmaceutical Education and Research* 45(2)*: 175–183.*

Gedanken, A. (2004). Using sonochemistry for the fabrication of nanomaterials. *Ultrasonics Sonochemistry* 11: 47–55.

Ghosh, S., Mukherjee, A., Kim, H., and Lee, C. (2003). Influence of annealing on the structural properties of chemically synthesized CdS nano-crystallites. *Materials Chemistry and Physics* 78(3): 726–732.

Ghoshal, S., Kushwah, N.P., Pal, M.K., Jain, V.K., and Nethaji, M. (2008). Tris(dithiocarboxylato) indium(III): Thermal studies and crystal structure of [In(S_2Ctol)$_3$]. *Journal of Chemical Sciences* 120(3): 305–308.

Gong, Y., Hao, Z., Sun, J.L., Shi, H., Jiang, P., and Lin, J. (2013). Metal (II) complexes based on 1,4-bis-(3-pyridylaminomethyl)benzene: structures, photoluminescence and photocatalytic properties. *Dalton Transactions* 42: 13241–13250.

Govender, K., Smyth-Boyle, D., and O'Brien, P. (2002). Improved Routes towards Solution Deposition of Indium Sulfide Thin Films for Photovoltaic Applications: *Materials Research Society Symposium Proceedings* 692: 525–530.

Hassan, M.L., and Ali, A.F. (2008). Synthesis of nanostructured cadmium and zinc sulfides in aqueous solutions of hyperbranched polyethyleneimine. *Journal of Crystal Growth* 310: 5252–5258.

He, J.H., Zhang, Y.Y., Liu, J., Moore, D., Bao, G., and Wang, Z.L. (2007). ZnS/Silica Nanocable Field Effect Transistors as Biological and Chemical Nanosensors. *Journal of Physical Chemistry C.* 111(33): 12152–12156.

He, Y., Kriegseis, W., Bläsing, J., Polity, A., Krämer, T., Hasselkamp, D., Meyer, B. K., Hardt, M., and Krost, A. (2002). (001)-Textured Cu_2S Thin Films Deposited by RF Reactive Sputtering. *Japanese Journal of Applied Physics* 41: 4630–4634.

Honda, M., Komura, M., Kawasaki, Y., Tanaka T., and Okawara, R. (1968). Infra-red and PMR Spectra of some organo-Tin (IV) N,N-Dimethyldithiocarbamates. *Journal of Inorganic and Nuclear Chemistry* 30: 3231–3237.

Hu, B.Y., Chen, J., Chen, W., and Li, X. (2004). Synthesis of Nickel Sulfide Submicrometer-Sized Hollow Spheres Using a γ-Irradiation Route. *Advanced Functional Materials* 14(4): 383–386.

Hu, H., Mo, M., Yang, B., Zhang, X., Li, Q., Yu, W., and Qian, Y. (2003). Solvothermal synthesis of Sb_2S_3 nanowires on a large scale. *Journal of Crystal Growth* 258: 106–112.

Itoh, K., Kuzuya, T., and Sumiyama, K. (2006). Morphology and Composition-Controls of Cu_xS Nanocrystals Using Alkyl-Amine Ligands. *Materials Transactions* 47(8): 1953–1956.

Jayaraju, A., Ahamad, M. M., Rao, R.M., Sreeramulu, J., and Kumar, E.V.S. (2012). Synthesis, characterization and biological evaluation of novel dithiocarbamate metal complexes. *Journal of Chemical and Pharmaceutical Research* 4(3): 1601-1605.

Jeroh, M.D., and Okoli, D.N. (2012a). Optical and structural properties of amorphous antimony sulphide thin films : Effect of dip time. *Advances in Applied Science Research* 3(2): 793–800.

Jeroh, M.D., and Okoli, D.N. (2012b). The Influence of Dip Time on The Optical Properties of Antimony Tri-sulphide Thin Films. *International Journal of Research and Reviews in Applied Sciences* 12(3): 431–437.

Jing, D., and Guo, L. (2006). A Novel Method for the Preparation of a Highly Stable and Active CdS Photocatalyst with a Special Surface Nanostructure. *Journal of Physical Chemistry B.* 110(2): 11139–11145.

Jung, H., Park, C., and Sohn, H. (2011). Electrochimica Acta Bismuth sulfide and its carbon nanocomposite for rechargeable lithium-ion batteries. *Electrochimica Acta* 56(5): 2135–2139.

Jung, Y.K., Kim, J.Il, and Lee, J. (2010). Thermal Decomposition Mechanism of Single-Molecule Precursors Forming Metal Sulfide Nanoparticles. *Journal of the American Chemical Society* 132(1): 178–184.

Kamazani, M.M., and Niasari, M.S. (2013). Preparation of Stochiometric Cu_2S Nanoparticles by Ultrasonic Method. *Proceedings of The International Conferences Nanomaterials: Applications and Properties 2*(2): 02PCN02(3pp)

Kim, Y.H., Lee, H., Shin, D., Park, M., and Moon, S. (2010). Synthesis of shape-controlled β-In_2S_3 nanotubes through oriented attachment of nanoparticles. *Chemical Communications* 46: 2292–2294.

Kim, Y., and Walsh, D. (2010). Metal sulfide nanoparticles synthesized via enzyme treatment of biopolymer stabilized nanosuspensions. *Nanoscale* 2: 240–247.

Koh, Y.W., Lai, C.S., Du, A.Y., and Tiekink, E.R.T. (2003). Growth of Bismuth Sulfide Nanowire Using Bismuth Trisxanthate Single Source Precursors. *Chemistry of Materials* 15(24): 4544–4554.

Kumar, N., Raman, N., and Sundaresan, A. (2014). Synthesis and Properties of Cobalt Sulfide Phases : CoS_2 and Co_9S_8. *Zeitschrift fur anorganische und allgemeine Chemie* 640(6): 1069–1074.

Kuzuya, T., Itoh, K., and Sumiyama, K. (2008). Low polydispersed copper-sulfide nanocrystals derived from various Cu-alkyl amine complexes. *Colloid and Interface Science* 319: 565–571.

Lahewil, A.S.Z., Al-douri, Y., Hashim, U., and Ahmed, N.M. (2012). Structural and optical investigations of cadmium sulfide nanostructures for optoelectronic applications. *Solar Energy* 86: 3234–3240.

Lai, C., Huang, K., Cheng, J., Lee, C., Lee, W., and Huang, C. (2009). Oriented growth of large-scale nickel sulfide nanowire arrays via a general solution route for lithium-ion battery cathode applications. *Journal of Materials Chemistry* 19: 7277–7283.

Lai, C., Lu, M., and Chen, L. (2012). Metal sulfide nanostructures : synthesis, properties and applications in energy conversion and storage. *Journal of Materials Chemistry* 22: 19–30.

Lai, S.C., Huang, K., Cheng, J., Lee, C., Hwang, B., and Chen, L. (2010). Direct growth of high-rate capability and high capacity copper sulfide nanowire array cathodes for lithium-ion batteries. *Journal of Materials Chemistry* 20: 6638–6645.

Lee, S.S., Yoon, S.H., Seo, K.W., and Shim, I. (2005). Preparation of In_2S_3 Thin Films by MOCVD Using Single Source Precursors: Tris(N,N-ethylbutyldithiocarbamato) indium(III) and Tris(2-ethylpiperidinedithiocarbamato) indium(III). *Bulletin of the Korean Chemical Society* 26(9): 1453–1456.

Levi, S. A., Palchik, O., Palchik, V., Slifkin, M. A., Weiss, A.M., and Gedanken, A. (2001). Sonochemical Synthesis of Nanophase Indium Sulfide. *Chemistry of Materials* 13(19): 2195–2200.

Li, H., Zhu, Y., Chen, S., Palchik, O., Xiong, J., Koltypin, Y., Gofer, Y., and Gedanken, A. (2003). A novel ultrasound-assisted approach to the synthesis of CdSe and CdS nanoparticles. *Solid State Chemistry* 172: 102–110.

Lin, H., Huang, C.P., Li, W., Ni, C., Shah, S.I., and Tseng, Y. (2006). Size dependency of nanocrystalline TiO_2 on its optical property and photocatalytic reactivity exemplified by 2-chlorophenol. *Applied Catalysis B: Environmental*, 68(1): 1–11.

Liu, H., Hu, L., Watanabe, K., Hu, X., Dierre, B., and Kim, B. (2013). Cathodoluminescence Modulation of ZnS Nanostructures by Morphology, Doping, and Temperature. *Advanced Functional Materials*: DOI: 10.1002/adfm.201203711.

Liu, X. (2005). Hydrothermal synthesis and characterization of nickel and cobalt sulfides nanocrystallines. *Materials Science and Engineering B* 119: 19–24.

Liu, Y., Zhang, M., Gao, Y., Zhang, R., and Qian, Y. (2007). Synthesis and optical properties of cubic In_2S_3 hollow nanospheres. *Materials Chemistry and Physics* 101: 362–366.

Lou, W., Chen, M., Wang, X., and Liu, W. (2007). Novel Single-Source Precursors Approach to Prepare Highly Uniform Bi_2S_3 and Sb_2S_3 Nanorods via a Solvothermal Treatment. *Chemistry of Materials* 19: 872–878.

Mamba, S.M., Mishra, A.K., Mamba, B.B., Njobeh, P.B., Dutton, M.F., and Fosso-Kankeu, E. (2010). Spectral, thermal and in vitro antimicrobial studies of cyclohexylamine-N-dithiocarbamate transition metal complexes. *Spectrochimica Acta. Part A, Molecular and Biomolecular Spectroscopy* 77(3): 579–87.

Maneeprakorn, W., Malik, M.A., and Brien, P.O. (2010). The preparation of cobalt phosphide and cobalt chalcogenide (CoX, X $_=$ S,Se) nanoparticles from single source precursors. *Journal of Materials Chemistry* 20: 2329–2335.

Martinez, R.R., Huicochea, R.M., Alvarez, J.A.G., Höpfl, H., and Tlahuext, H. (2008). Synthesis, heteronuclear NMR and X-ray crystallographic studies of two dinuclear diorganotin (IV) dithiocarbamate macrocycles. *Arkivoc(Archive for Organic Chemistry) v*: 19–30.

Mehlape, M.A. (2013). Computational Modeling Studies of Cobalt Pentlandite (Co_9S_8), PHD thesis, University of Limpopo.

Menezes, D.C., de Lima, G.M., Porto, A.O., Donnici, C.L., and Ardisson, J.D. (2004). Synthesis, characterisation and thermal decomposition of tin (IV) dithiocarbamate derivatives – single source precursors for tin sulfide powders. *Polyhedron* 23: 2103–2109.

Messina, S., Nair, M.T.S., and Nair, P.K. (2007). Antimony sulfide thin films in chemically deposited thin film photovoltaic cells. *Thin Solid Films* 515: 5777–5782.

Mohammad, A., Varshney, C., and Nami, S. (2009). Synthesis, characterization and antifungal activities of 3d-transition metal complexes of 1-acetylpiperazinyldithiocarbamate, $M(acpdtc)_2$. *Spectrochimica Acta Part A, Molecular and Biomolecular Spectroscopy* 73(1): 20–24.

Mondal, G., Bera, P., Santra, A., Jana, S., Mandal, T. N., Mondal, A., Seok, S. I., and Bera, P. (2014). Precursor-driven selective synthesis of hexagonal chalcocite

(Cu_2S) nanocrystals: structural, optical, electrical and photocatalytic properties. *New Journal of Chemistry* 38: 4774–4782.

Monteiro, O.C., Nogueira, H.I.S., and Trindade, T. (2001). Use of Dialkyldithiocarbamato Complexes of Bismuth (III) for the Preparation of Nano- and Microsized Bi_2S_3 Particles and the X-ray Crystal Structures of $[Bi\{S_2CN(CH_3)(C_6H_{13})\}_3]$ and $[Bi\{S_2CN(CH_3)(C_6H_{13})\}_3(C_{12}H_8N_2)$. *Chemistry of Materials* 13: 2103–2111.

Moon, H., Nam, C., Kim, C., and Kim, B. (2006). Synthesis and photoluminescence of zinc sulfide nanowires by simple thermal chemical vapor deposition. *Materials Research Bulletin* 41: 2013–2017.

Moriarty, P. (2001). Nanostructured materials. *Reports on Progress in Physics* 64: 297–381.

Motevalizadeh, L., Heidary, Z., and Abrishami, M.E. (2014). Facile template-free hydrothermal synthesis and microstrain measurement of ZnO nanorods. *Bulletin of Materials Science 37*(3): 397–405.

Mthethwa, T., Pullabhotla, V.S.R.R., Mdluli, P.S., Wesley-smith, J., and Revaprasadu, N. (2009). Synthesis of hexadecylamine capped CdS nanoparticles using heterocyclic cadmium dithiocarbamates as single source precursors. *Polyhedron* 28: 2977–2982.

Mu, C., Yao, Q., Qu, X., Zhou, G., Li, M., and Fu, S. (2010). Colloids and Surfaces A : Physicochemical and Engineering Aspects Controlled synthesis of various hierarchical nanostructures of copper sulfide by a facile microwave irradiation method. *Colloids and Surfaces A: Physicochemical and Engineering Aspects* 371: 14–21.

Mustafa, I.A., and Taqa, A. A. (2001). Coordination Compounds of Tin and Bismuth. Complexes of Tin(Iv) and Bismuth(Iii) With N,N-Dialkyldithiocarbamates. *Synthesis and Reactivity in Inorganic and Metal-Organic Chemistry* 31(4): 517–526.

Nabipour, H., Ghammamy, S., Ashuri, S., and Aghbolagh, S. (2010). Synthesis of a New Dithiocarbamate Compound and Study of Its Biological Properties. *Organic Chemistry Journal* 2: 75–80.

Nagaveena, S., and Mahadevan, C.K. (2014). Preparation by a facile method and characterization of amorphous and crystalline nickel sulfide nanophases. *Journal of Alloys and Compounds* 582: 447–456.

Nakamura, M., and Fujimori, A. (1993). Metal-nonmetal transition in NiS induced by Fe and Co substitution: X-ray-absorption spectroscopic study. *Physical Review B* 48(23): 16942–16947.

Neugebauer, C.A., Miller, D.C., and Hall, J.W. (1968). Polycrystalline CdS Thin Film Field Effect Transistors: Fabrication, Stability, And Temperature Dependence. *Thin Solid Films* 2: 57–78.

Niasari, M.S., Behfard, Z., Amiri, O., Khosravifard, E., and Mashkani, S.M.S. (2013). Hydrothermal Synthesis of Bismuth Sulfide (Bi_2S_3) Nanorods: Bismuth (III) Monosalicylate Precursor in the Presence of Thioglycolic Acid. *Journal of Cluster Science* 24: 349–363.

Niasari, M.S., Davar, F., and Mazaheri, M. (2009). Synthesis , characterization and magnetic properties of NiS_{1+x} nanocrystals from [bis(salicylidene)nickel(II)] as new precursor. *Materials Research Bulletin* 44: 2246–2251.

Niasari, M.S., Esmaeili, E., and Sabet, M. (2013). Synthesis and Characterization of Cu_2S Nanostructures Via Hydrothermal Method by a Polymeric Precursor. *Journal of Cluster Science* 24: 799–809.

Nicolais, L.F., and Carotenuto, G. (2008). Synthesis of Polymer-Embedded Metal, Semimetal, or Sulfide Clusters by Thermolysis of Mercaptide Molecules Dissolved in Polymers. *Recent Patents on Materials Science* 1: 1–11.

Nirmal, R.M., Pandian, K., and Sivakumar, K. (2011). Applied Surface Science Cadmium (II) pyrrolidine dithiocarbamate complex as single source precursor for the preparation of CdS nanocrystals by microwave irradiation and conventional heating process. *Applied Surface Science* 257(7): 2745–2751.

Nomura, R., Takabe, A., and Matsuda, H. (1987). Facile Synthesis of Antimony Dithiocarbamate Complexes. *Polyhedron* 6(3): 411–416.

Okamura, H., Naitoh, J., Nanba, T., Matoba, M., Nishioka, M., and Anzai, S. (1998). Optical study of the metal-nonmetal transition in $Ni_{1-\delta}S$. *Condensed Mater* 2: 1–4.

Okoli, D.N., and Okoli, C.N. (2012). Optimal Growth and Characterization of Cobalt Sulphide Thin Films Fabricated Using the Chemical Bath Deposition Technique. *Jornal of Natural Sciences Research* 2(3): 5–9.

Oliveira, M.M., Pessoa, G.M., Carvalho, L.C., Peppe, C., Souza, A.G., and Airoldi, C. (1999). N,N’-Dialkyldithiocarbamate chelates of indium (III): alternative synthetic routes and thermodynamics characterization. *Thermochimica Acta* 328: 223–230.

Ondrusova, D., Jona, E., and Simon, P. (2002). Thermal Properties of N-ethyl-N-phenyl Dithiocarbamates and Their Influence on The Kinetics of Cure. *Journal of Thermal Analysis and Calorimetry* 67: 147–152.

Onwudiwe, D.C., and Ajibade, P.A. (2012). Thermal Studies of Zn(II), Cd(II) and Hg(II) Complexes of Some N-Alkyl-N-Phenyl-Dithiocarbamates. *International Journal of Molecular Sciences* 13(8): 9502–13.

Onwudiwe, D.C., and Ajibade, P.A. (2011a). Synthesis , Characterization and Thermal Studies of Zn (II), Cd (II) and Hg (II) Complexes of N-Methyl-N-Phenyldithiocarbamate : The Single Crystal Structure of $[(C_6H_5)(CH_3)NCS_2]_4Hg_2$. *International Journal of Molecular Sciences* 12: 1964–1978.

Onwudiwe, D.C., and Ajibade, P.A. (2011b). ZnS , CdS and HgS Nanoparticles via Alkyl-Phenyl Dithiocarbamate Complexes as Single Source Precursors. *International Journal of Molecular Sciences* 12: 5538–5551.

Panigrahi, P.K., and Pathak, A. (2013). The Growth of Bismuth Sulfide Nanorods from Spherical-Shaped Amorphous Precursor Particles under Hydrothermal Condition. *Journal of Nanoparticles:* 1–12.

Pearce, C.I., Pattrick, R.A.D., and Vaughan, D.J. (2006). Electrical and Magnetic Properties of Sulfides. *Reviews in Mineralogy and Geochemistry* 61: 127–180.

Pejova, B., and Bineva, I. (2013). Sonochemically Synthesized 3D Assemblies of Close-Packed In_2S_3 Quantum Dots: Structure, Size Dependent Optical and Electrical Properties. *Journal of Physical Chemistry* 117: 7303−7314.

Pickett, N.L., and Brien, P.O. (2001). Syntheses of Semiconductor Nanoparticles Using Single-Molecular Precursors. *The Chemical Record* 1: 467–479.

Plante, I.J., Zeid, T.W., Yang, P., and Mokari, T. (2010). Synthesis of metal sulfide nanomaterials via thermal decomposition of single-source precursors. *Journal of Materials Chemistry* 20: 6612–6617.

Qian, X.F., Zhang, X.M., Wang, C., Xie, Y., and Qian, Y.T. (1999). The Preparation and Phase Transformation of Nanocrystalline Cobalt Sulfides via a Toluene Thermal Process. *Inorganic Chemistry* 38(11): 2621–2623.

Qin, A., Fang, Y., Ou, H., and Liu, H. (2005). Formation of Various Morphologies of Covellite Copper Sulfide Submicron Crystals by a Hydrothermal Method without Surfactant. *Crystal Growth and Design* 5(3): 855–860.

Qin, Z., Sun, H., Jiang, Z., Jiao, X., Chen, D., and Online, V. A. (2013). Synthesis of metal sulfide nanoboxes based on Kirkendall effect and Pearson hardness. *CrystEngComm* 15: 897–902.

Ramya, K., Reddy, M.V, and Reddy, K.T.R. (2013). Characterization of Thermally Evaporated In_2S_3 Films for Solar Cell Application. *Conference Papers In Energy*: 1-4.

Ramya, M., and Ganesan, S. (2013). Influence of thickness and temperature on the properties of Cu_2S thin films. *Iranian Journal of Science and Technology* 37A3: 293–300.

Rasband, W.S., ImageJ, U. S. National Institutes of Health, Bethesda, Maryland, USA, http://imagej.nih.gov/ij/, 1997-2014.

Revaprasadu, N., and Mlondo, S.N. (2006). Use of metal complexes to synthesize semiconductor nanoparticles. *Pure and Applied Chemistry* 78(9): 1691–1702.

Revathi, N., Prathap, P., and Reddy, K.T.R. (2010). Synthesis and characterization of In_2S_3 nanocrystalline material for photovoltaic application. *Energy Procedia* 2: 195–198.

Reza, H., Norozi, A., Hossein, M., and Semnani, A. (2009). Nanoparticles of zinc sulfide doped with manganese , nickel and copper as nanophotocatalyst in the degradation of organic dyes. *Journal of Hazardous Materials* 162: 674–681.

Romano, R., and Alves, O.L. (2006). Semiconductor/porous silica glass nanocomposites via the single-source precursor approach. *Materials Research Bulletin* 41: 376–386.

Rui-zhou, Z., and Xian-zhou, Z. (2013). Molecular structure, NMR parameters, IR spectra of N-(1,3,4-thiadiazol-2-yl)-1-[1-(6-chloropyridin-3-yl)methyl]-5-methyl-1H-[1,2,3]triazol-4-carboxamide by density functional theory and ab-initio Hartree-Fock calculations. *Indian Journal of Pure and Applied Physics* 51: 164–173.

Sam, M., Bayati, M.R., Mojtahedi, M., and Janghorban, K. (2010). Applied Surface Science Growth of Cu_2S/CdS nano-layered photovoltaic junctions for solar cell applications. *Applied Surface Science* 257(5): 1449–1453.

Saraf, R. (2012). High Efficiency and cost effective Cu_2S/CdS thin-film solar cell. *Journal of Electronics Engineering 2*(4): 47–51.

Saravanan, N., Teh, G.B., Yap, S.Y.P., and Cheong, K.M. (2008). Simple synthesis of ZnS nanoparticles in alkaline medium. *Journal of Materials Science: Materials in Electronics* 19(12): 1206–1208.

Sarwar, M., Ahmad, S., Ahmad, S., Ali, S., and Awan, S.A. (2007). Copper(II) complexes of pyrrolidine dithiocarbamate. *Transition Metal Chemistry* 32: 199–203.

Senapati, U.S., Jha, D.K., and Sarkar, D. (2013). Green Synthesis and Characterization of ZnS nanoparticles. *Research Journal of Physical Sciences* 1(7): 1–6.

Seo, K.W., Yoon, S.H., Lee, S.S., and Shim, I. (2005). Preparation of ZnS Thin Film Using Zn (dithiocarbamate)$_2$ Precursors by MOCVD Method. *Bulletin of the Korean Chemical Society* 26(10): 1582–1584.

Shahid, M., Ruffer, T., Lang, H., Awan, S.A., and Ahmad, S. (2009). Synthesis and crystal structure of a dinuclear zinc(II)-dithiocarbamate complex, bis{[(μ^2-pyrrolidinedithiocarbamato-S,S′)(pyrrolidinedithiocarbamato-S,S')zinc(II)]}. *Journal of Coordination Chemistry* 62(3): 440-445.

Shen, S., Zhang, Y., Peng, L., Xu, B., Du, Y., Deng, M., Xu, H., and Wang, Q. (2011). Generalized synthesis of metal sulfide nanocrystals from single-source precursors: size, shape and chemical composition control and their properties. *CrystEngComm* 13: 4572–4579.

Shuai, X., and Shen, W. (2012). A facile chemical conversion synthesis of Sb_2S_3 nanotubes and the visible light-driven photocatalytic activities. *Nanoscale Research Letters* 7: 199.

Shukla, S.N., Gaur, P., and Rai, N. (2014). Complexes of tetraethylthiuram disulphide with group 12 metals: single-source precursor in metal sulphide nanoparticles' synthesis. *Applied Nanoscience*: DOI 10.1007/s13204-014-0351-0.

Sivagurunathan, G.S., Ramalingama, K., and Rizzoli, C. (2014). Continuous Shape Measure of electronic effect free steric distortions in tris(dithiocarbamato) indium(III): Synthesis, spectral, electrochemical, single crystal X-ray structural investigations and BVS calculations on tris(dithiocarbamato)indium(III) complexes. *Polyhedron* 72: 96–102.

Sobhani, A., Niasari, M.S., and Mashkani, S.M.H. (2012). Single-Source Molecular Precursor for Synthesis of Copper Sulfide Nanostructures. *Journal of Cluster Science* 23: 1143–1151.

Sonawane, M.S., and Patil, R.S. (2014). Characterizations of Chemically Deposited Nanocrystalline Cobalt Sulphide Thin Films. *International Journal of Advanced Scientific and Technical Research* 3(4): 57–64.

Spasevska, H., Kitts, C.C., Ancora, C., and Ruani, G. (2012). Optimised In_2S_3 Thin Films Deposited by Spray Pyrolysis. *International Journal of Photoenergy:* doi:10.1155/2012/637943.

Srikanth, S., Suriyanarayanan, N., Prabahar, S., Balasubramanian, V., and Kathirvel, D. (2011). Effect of concentration On the Structural properties of Synthesized Sb_2S_3 Bulk Powder. *Archives of Applied Science Research* 3(1): 168–174.

Suchanek, W. L., and Riman, R. E. (2006). Hydrothermal Synthesis of Advanced Ceramic Powders. *Advances in Science and Technology* 45: 184-193.

Sweeney, R.Y., Mao, C., Gao, X., Burt, J.L., Belcher, A.M., Georgiou, G., and Iverson, B.L. (2004). Bacterial Biosynthesis of Cadmium Sulfide Nanocrystals. *Chemistry and Biology* 11: 1553–1559.

Tadjarodi, A., and Khaledi, D. (2010). Preparation of CuS nanoparticles by microwave irradiation. *14th International Electronic Conference on Synthetic Organic Chemistry (ECSOC-14)*: 1–5.

Tao, F., Zhao, Y., Zhang, G., and Li, H. (2007). Electrochemical characterization on cobalt sulfide for electrochemical supercapacitors. *Electrochemistry Communications* 9: 1282–1287.

Tepantlan, C.S. (2008). Structural, optical and electrical properties of CdS thin films obtained by spray pyrolysis. *Revista Mexicana De Fisica* 54(2): 112–117.

Thammakan, N., and Somsook, E. (2006). Synthesis and thermal decomposition of cadmium dithiocarbamate complexes. *Materials Letters* 60: 1161–1165.

Thirumaran, S., and Ramalingam, K. (2000). Mixed ligand complexes involving amino acid dithiocarbamates, substituted phosphines and nickel (II). *Transition Metal Chemistry* 25: 60–62.

Tiekink, E.R.T. (2008). Tin dithiocarbamates: applications and structures. *Applied Organometallic Chemistry* 22: 533–550.

Travnicek, Z., Pastorek, R., and Slovak, V. (2008). Novel octahedral nickel(II) dithiocarbamates with bi- or tetradentate N-donor ligands: X-ray structures of $[Ni(Bzppzdtc)(phen)_2]ClO_4 \cdot CHCl_3$ and $[Ni(Bz_2dtc)_2(cyclam)]$. *Polyhedron* 27(1): 411–419.

Tsipis, C.A., and Manoussakis, G.E. (1976). Syntheses Complexes and Spectral Study of New Iodohis (dialkyldithiocarbamate) of Arsenic, Antimony and Bismuth. *Inorganica Chimica Acta* 18: 35–45.

Uzar, N., and Arikan, M. C. (2011). Synthesis and investigation of optical properties of ZnS nanostructures. *Bulletin of Materials Science* 1: 4–9.

Wan, H., Ji, X., Jiang, J., Yu, J., Miao, L., Zhang, L., Bie, S., Chen, H., Ruan, Y. (2013). Hydrothermal synthesis of cobalt sulfide nanotubes: The size control and its application in supercapacitors. *Journal of Power Sources* 243: 396–402.

Wang, C.Z., Yu, R., and Krakauer, H. (1996). Polarization dependence of Born effective charge and dielectric constant in $KNbO_3$. *Physical Review B* 54(16): 161–168.

Wang, D.Q., Chen, D.R., and Jiao, X.L. (2004). Synthesis of Nickel Sulfide Particles by Solvothermal Process. *Chinese Chemical Letters* 15(1): 79–82.

Wang, D., Zheng, W., Hao, C., Peng, Q., and Li, Y. (2009). A Synthetic Method for Transition-Metal Chalcogenide Nanocrystals. *Chemistry-A European Journal* 15: 1870–1875.

Wang, H., Lu, Y., Zhu, J., and Chen, H. (2003). Sonochemical Fabrication and Characterization of Stibnite Nanorods. *Inorganic Chemistry* 42(20): 6404–6411.

Wang, H., Zhu, J.J., and Chen, H. (2002). Sonochemical Method for the Preparation of Bismuth Sulfide Nanorods. *Journal of Physical Chemistry B* 106(15): 3848–3854.

Wang, J., Chou, S., Chew, S., Sun, J., and Forsyth, M. (2008). Nickel sulfide cathode in combination with an ionic liquid-based electrolyte for rechargeable lithium batteries. *Solid State Ionics* 179(40): 2379–2382.

Wang, Q.X., Gao, F., Xing, S., Weng, W., and Shui, Z. (2008). Preparation of cauliflower-like bismuth sulfide and its application in electrochemical sensor. *Chinese Chemical Letters* 19: 585–588.

Wang, W., Germanenko, I., and El-shall, M.S. (2006). Room-Temperature Synthesis and Characterization of Nanocrystalline CdS, ZnS, and $Cd_xZn_{1-x}S$. *Chemistry of Materials* 14(29): 3028–3033.

Wang, Y., Chen, J., Wang, P., Chen, L., Chen, Y., and Wu, L. (2009). Syntheses, Growth Mechanism, and Optical Properties of [001] Growing Bi_2S_3 Nanorods. *Journal of Physical Chemistry C* 113(36): 16009–16014.

Wang, Z., Pan, L., Hu, H., and Zhao, S. (2010). Co_9S_8 nanotubes synthesized on the basis of nanoscale Kirkendall effect and their magnetic and electrochemical properties. *CrystEngComm* 12: 1899–1904.

Waters, J., Crouch, D., Raftery, J., and Brien, P.O. (2004). Deposition of Bismuth Chalcogenide Thin Films Using Novel Single-Source Precursors by Metal-Organic Chemical Vapor Deposition. *Chemistry of Materials* 16(22): 3289–3298.

Wei, F., Zhang, J., Wang, L., and Zhang, Z. (2006). Solvothermal Growth of Single-Crystal Bismuth Sulfide Nanorods using Bismuth Particles as Source Material. *Crystal Growth and Design 6*(8): 1942–1944.

Wu, J., Qin, F., Cheng, G., Li, H., Zhang, J., Xie, Y., Yang. H.J., Lu, Z., Yu, X., Chen, R. (2011). Large-scale synthesis of bismuth sulfide nanorods by microwave irradiation. *Journal of Alloys and Compounds* 509: 2116–2126.

Wu, Y., Wadia, C., Ma, W., Sadtler, B., Alivisatos, A.P. (2008). Synthesis and Photovoltaic Application of Copper (I) Sulfide Nanocrystals. *Nano letters* 8(8): 2551-2555.

Xie, G., Qiao, Z.-P., Zeng, M.-H., Xiao-Ming Chen, and Gao, S.-L. (2004). A Single-Source Approach to Bi_2S_3 and Sb_2S_3 Nanorods via a Hydrothermal Treatment. *Crystal Growth and Design* 4(3): 513–516.

Xiong, Y., Xie, Y., Du, G., and Tian, X. (2002). A solvent-reduction and surface-modification technique to morphology control of tetragonal In2S3 nanocrystals. *Journal of Materials Chemistry* 12: 98–102.

Yang, J., Bao, C., Zhu, K., Yu, T., Li, F., Liu, J., Li, Z., and Zou, Z. (2014). High catalytic activity and stability of nickel sulfide and cobalt sulfide hierarchical nanospheres on the counter electrodes for dye-sensitized solar cells. *Chemical Communications* 50: 4824–4826.

Yin, H. (2008). Synthesis , characterizations and crystal structures of new antimony (III) complexes with dithiocarbamate ligands. *Polyhedron* 27: 663–670.

Yin, H.D., and Xue, S.C. (2006). Synthesis and characterization of organotin complexes with dithiocarbamates and crystal structures of (4-$NCC_6H_4CH_2)_2Sn(S_2CNEt_2)_2$ and (2-$ClC_6H_4CH_2)_2$ $Sn(Cl)S_2CNBz_2$. *Applied Organometallic Chemistry* 2: 283–289.

Yin, Y., Erdonmez, C.K., Cabot, A., Hughes, S., and Alivisatos, A.P. (2006). Colloidal Synthesis of Hollow Cobalt Sulfide Nanocrystals. *Lawrence Berkeley National Laboratory*, 1–35.

Yousfi, O. (2009). Transformations de phase des Sulfures de Nickel dans les verres trempés, Doctoral dissertation, Institut National Polytechnique de Grenoble-INPG.

Yu, Y., Jin, C.H., Wang, R.H., Chen, Q., and Peng, L. (2005). High-Quality Ultralong Bi_2S_3 Nanowires : Structure, Growth, and Properties. *Journal of Physical Chemistry B* 109: 18772–18776.

Zhai, T., Gu, Z., Ma, Y., Yang, W., Zhao, L., and Yao, J. (2006). Synthesis of ordered ZnS nanotubes by MOCVD-template method. *Materials Chemistry and Physics* 100: 281–284.

Zhang, H.T., Wu, G., and Chen, X.H. (2005). Synthesis and magnetic properties of NiS_{1+x} nanocrystallines. *Materials Letters* 59: 3728–3731.

Zhang, Z., Lim, W.P., Wong, C. T., Xu, H., Yin, F., and Chin, W. S. (2012). From Metal Thiobenzoates to Metal Sulfide Nanocrystals : An Experimental and Theoretical Investigation. *Nanomaterials* 2: 113–133.

Zhao, P., Zeng, Q., and Huang, K. (2009). Fabrication of β-NiS hollow sphere consisting of nano flakes via a hydrothermal process. *Materials Letters* 63: 313–315.

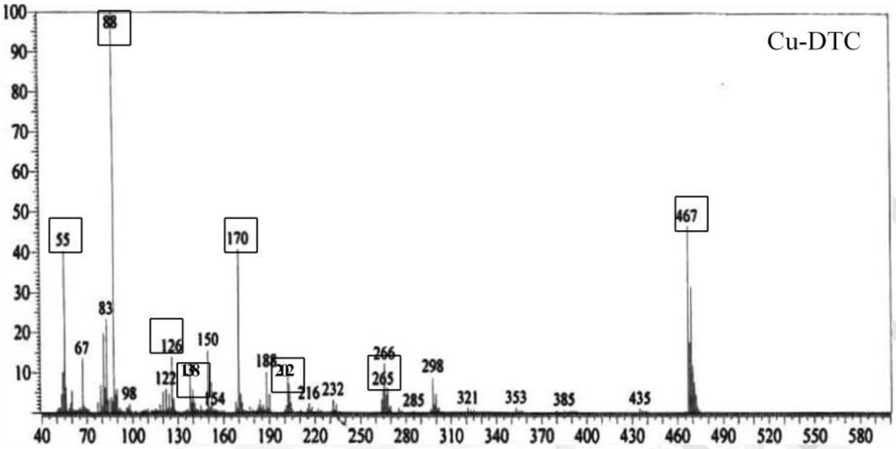
Zhao, Q., Xie, Y., Zhang, Z., and Bai, X. (2007). Size-selective Synthesis of Zinc Sulfide Hierarchical Structures and Their Photocatalytic Activity. *Crystal Growth and Design* 7(1): 153-158.

Zhou, J., Bian, G., Zhang, Y., Zhu, Q., Li, C., and Dai, J. (2007). One-Dimensional Indium Sulfides with Transition Metal Complexes of Polyamines. *Inorganic Chemistry* 46(16): 6347-6352.

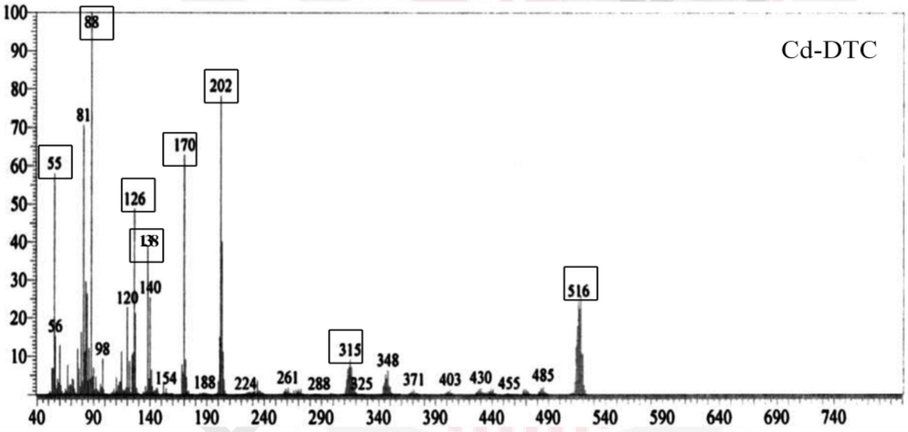
Zou, J., Zhang, J., Zhang, B., Zhao, P., and Huang, K. (2007). Low-temperature synthesis of copper sulfide nano-crystals of novel morphologies by hydrothermal process. *Materials Letters* 61: 5029–5032.

APPENDICES

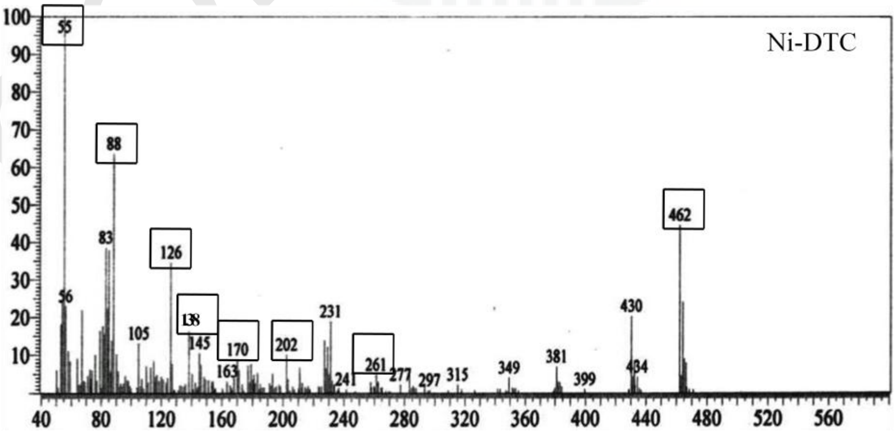
Appendix A: Mass Spectra of Metal Dithiocarbamates



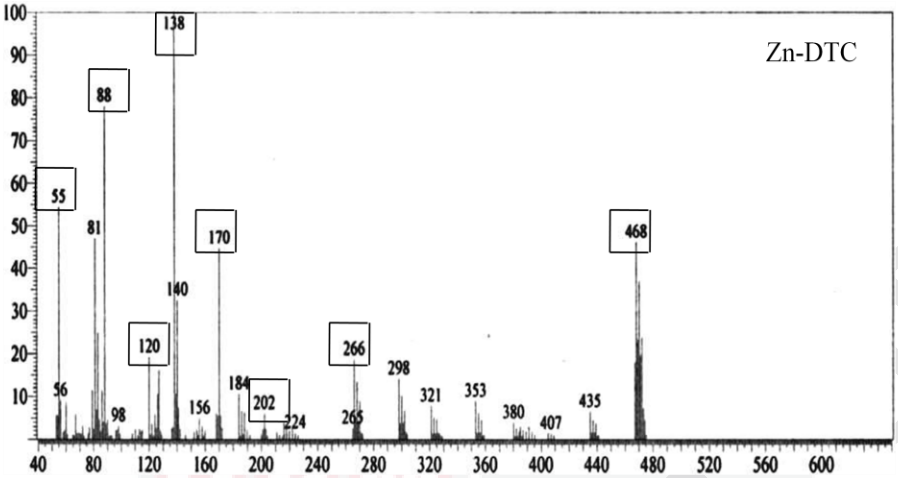
Mass Spectrum of Cu-DTC



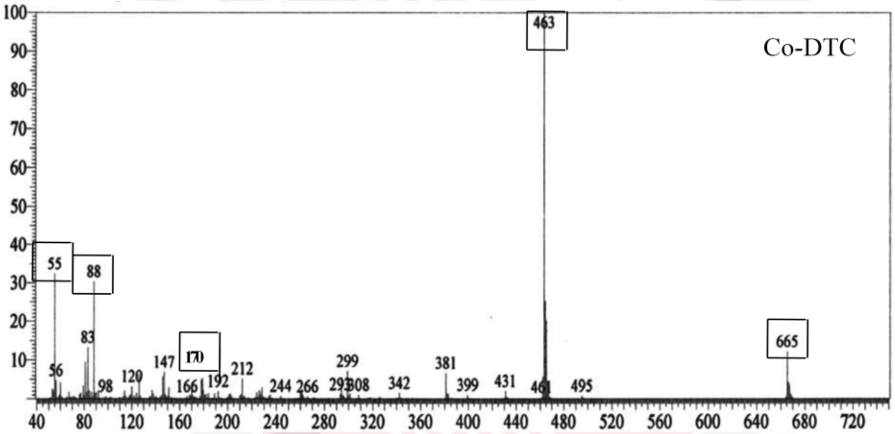
Mass Spectrum of Cd-DTC



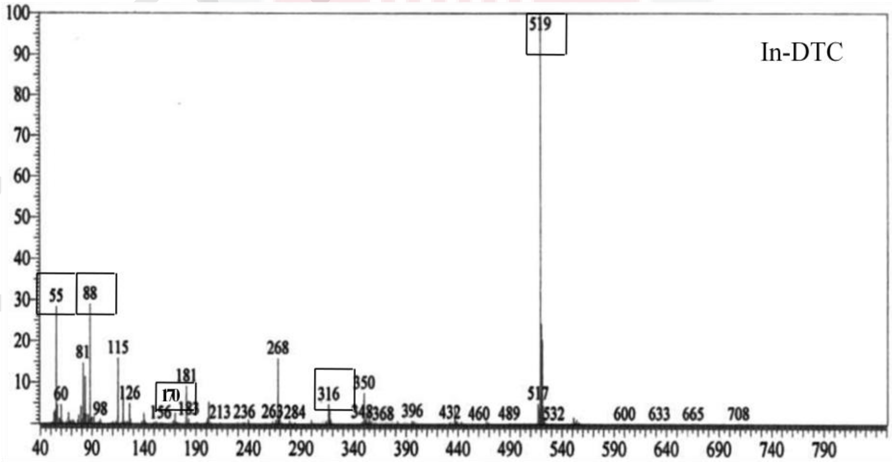
Mass Spectrum of Ni-DTC



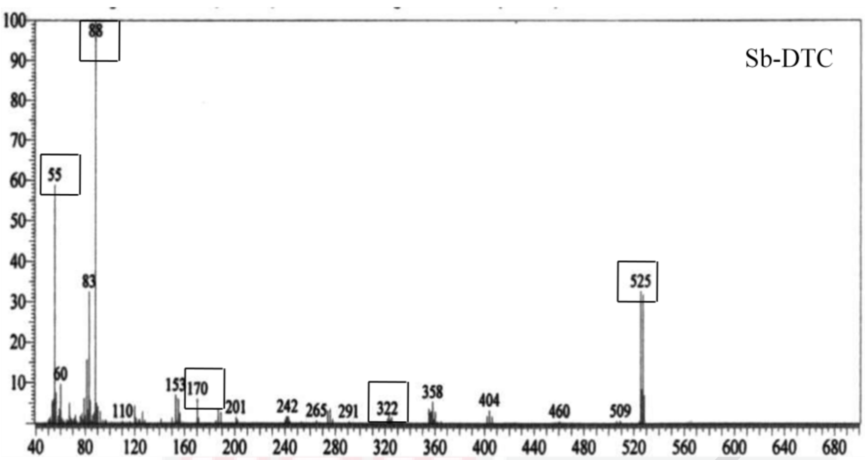
Mass Spectrum of Zn-DTC



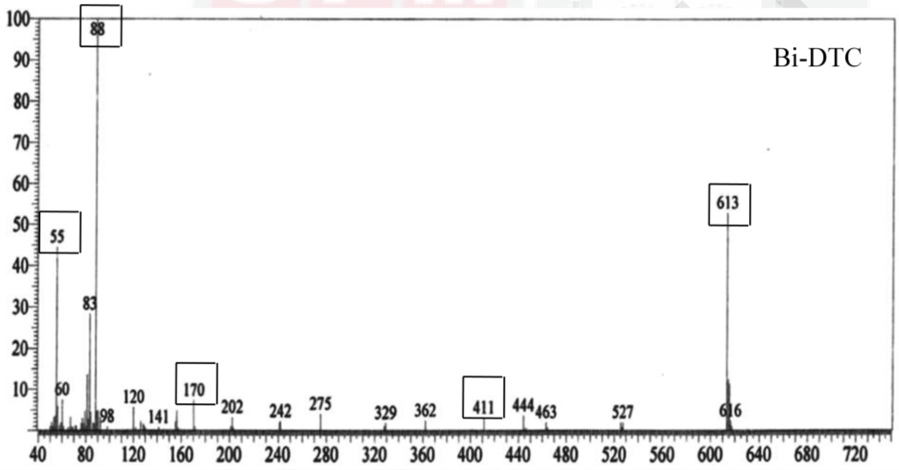
Mass Spectrum of Co-DTC



Mass Spectrum of In-DTC



Mass Spectrum of Sb-DTC



Mass Spectrum of Bi-DTC

Appendix B: Single Crystal XRD analysis

1. Crystal data for Cu-DTC

$C_{18}H_{32}CuN_2S_4$	?
$M_r = 468.28$	$D_x = 1.452$ Mg m^{-3}
Monoclinic, $P2_1/n$	Melting point: ? K
Hall symbol: ?	Mo $K\alpha$ radiation, $\lambda = 0.71070$ Å
$a = 10.4601$ (3) Å	Cell parameters from 5418 reflections
$b = 18.5191$ (8) Å	$\theta = 2–29°$
$c = 11.0847$ (5) Å	$\mu = 1.42$ mm^{-1}
$\beta = 94.192$ (3)°	$T = 100$ K
$V = 2141.49$ (15) Å^3	Block-like, dark brown
$Z = 4$	0.23 × 0.13 × 0.08 mm
$F(000) = 988$	

Data Collection

Oxford Diffraction Gemini diffractometer	4128 reflections with $I > 2.0\sigma(I)$
Radiation source: ?	$R_{int} = 0.035$
graphite	$\theta_{max} = 28.9°$, $\theta_{min} = 2.2°$
ω scans	$h = -13 \rightarrow 13$
Absorption correction: multi-scan CrysAlis, (Oxford Diffraction, 2002)	$k = -24 \rightarrow 22$
$T_{min} = 0.73$, $T_{max} = 0.89$	$l = -14 \rightarrow 15$
14203 measured reflections	Standard reflections: 0
4909 independent reflections	

Refinement

Refinement on F^2	Secondary atom site location: ?
Least-squares matrix: full	Hydrogen site location: difference Fourier map
$R[F^2 > 2\sigma(F^2)] = 0.031$	H-atom parameters constrained
$wR(F^2) = 0.073$	Method = Modified Sheldrick $w = 1/[\sigma^2(F^2) + (0.03P)^2 + 1.4P]$, where $P = (\max(F_o^2,0) + 2F_c^2)/3$
$S = 0.97$	$(\Delta/\sigma)_{max} = 0.002$
4893 reflections	$\Delta\rho_{max} = 0.66$ e Å^{-3}
226 parameters	$\Delta\rho_{min} = -0.49$ e Å^{-3}
0 restraints	Extinction correction: None
? constraints	Extinction coefficient: ?
Primary atom site location: structure-invariant direct methods	

Bond lengths (Å) for Cu-DTC

Cu1—S2i	2.9624 (6)	C13—H132	0.972
Cu1—S2	2.3266 (5)	C13—H133	0.953
Cu1—C3	2.7569 (19)	C13—H131	0.963
Cu1—S4	2.2929 (5)	S14—C15	1.7236 (18)
Cu1—S14	2.3170 (5)	C15—S16	1.7234 (19)
Cu1—C15	2.7567 (19)	C15—N17	1.329 (2)
Cu1—S16	2.3048 (5)	N17—C18	1.484 (2)
S2—C3	1.7333 (18)	N17—C24	1.476 (2)
C3—S4	1.7208 (19)	C18—C19	1.525 (3)
C3—N5	1.325 (2)	C18—C23	1.531 (3)
N5—C6	1.494 (2)	C18—H181	0.971
N5—C12	1.472 (2)	C19—C20	1.533 (3)
C6—C7	1.530 (3)	C19—H191	0.979
C6—C11	1.521 (3)	C19—H192	0.980
C6—H61	0.979	C20—C21	1.524 (3)
C7—C8	1.531 (3)	C20—H201	0.984
C7—H71	0.962	C20—H202	0.984
C7—H72	0.975	C21—C22	1.533 (3)
C8—C9	1.522 (3)	C21—H211	0.980
C8—H81	0.976	C21—H212	0.976
C8—H82	0.979	C22—C23	1.534 (3)
C9—C10	1.527 (3)	C22—H221	0.973
C9—H91	0.974	C22—H222	0.982
C9—H92	0.970	C23—H231	0.966
C10—C11	1.535 (3)	C23—H232	0.963
C10—H101	0.974	C24—C25	1.524 (3)
C10—H102	0.978	C24—H241	0.975
C11—H111	0.973	C24—H242	0.966
C11—H112	0.970	C25—H252	0.956
C12—C13	1.517 (3)	C25—H253	0.960
C12—H121	0.972	C25—H251	0.961
C12—H122	0.966		

Bond angle (°) for Cu-DTC

S2i—Cu1—S2	84.694 (17)	N5—C12—H121	109.2
S2i—Cu1—C3	91.06 (4)	C13—C12—H121	108.9
S2—Cu1—C3	38.72 (4)	N5—C12—H122	107.7
S2i—Cu1—S4	98.664 (18)	C13—C12—H122	108.8
S2—Cu1—S4	77.171 (18)	H121—C12—H122	108.5
C3—Cu1—S4	38.48 (4)	C12—C13—H132	108.7
S2i—Cu1—S14	97.807 (17)	C12—C13—H133	109.9
S2—Cu1—S14	176.85 (2)	H132—C13—H133	110.2
C3—Cu1—S14	138.98 (4)	C12—C13—H131	108.7
S4—Cu1—S14	100.496 (19)	H132—C13—H131	109.5
S2i—Cu1—C15	100.28 (4)	H133—C13—H131	109.7
S2—Cu1—C15	143.03 (4)	Cu1—S14—C15	84.68 (7)

C3—Cu1—C15	168.60 (5)	S14—C15—Cu1	56.81 (6)
S4—Cu1—C15	136.69 (4)	S14—C15—S16	113.21 (11)
S14—Cu1—C15	38.50 (4)	Cu1—C15—S16	56.41 (6)
S2i—Cu1—S16	98.760 (17)	S14—C15—N17	123.18 (14)
S2—Cu1—S16	104.548 (18)	Cu1—C15—N17	179.16 (14)
C3—Cu1—S16	141.06 (4)	S16—C15—N17	123.61 (13)
S4—Cu1—S16	162.58 (2)	C15—S16—Cu1	85.07 (6)
S14—Cu1—S16	77.028 (18)	C15—N17—C18	120.39 (15)
C15—Cu1—S16	38.53 (4)	C15—N17—C24	120.44 (15)
Cu1i—S2—Cu1	95.306 (17)	C18—N17—C24	118.95 (15)
Cu1i—S2—C3	98.83 (6)	N17—C18—C19	110.84 (15)
Cu1—S2—C3	84.19 (7)	N17—C18—C23	113.30 (15)
S2—C3—Cu1	57.10 (5)	C19—C18—C23	111.32 (16)
S2—C3—S4	113.06 (11)	N17—C18—H181	106.8
Cu1—C3—S4	56.01 (6)	C19—C18—H181	107.7
S2—C3—N5	123.31 (14)	C23—C18—H181	106.5
Cu1—C3—N5	177.12 (14)	C18—C19—C20	110.00 (16)
S4—C3—N5	123.63 (14)	C18—C19—H191	108.0
C3—S4—Cu1	85.51 (6)	C20—C19—H191	110.8
C3—N5—C6	119.11 (15)	C18—C19—H192	108.5
C3—N5—C12	120.22 (15)	C20—C19—H192	109.8
C6—N5—C12	120.66 (15)	H191—C19—H192	109.7
N5—C6—C7	111.71 (15)	C19—C20—C21	110.88 (17)
N5—C6—C11	113.53 (15)	C19—C20—H201	109.8
C7—C6—C11	111.29 (16)	C21—C20—H201	108.6
N5—C6—H61	106.1	C19—C20—H202	108.6
C7—C6—H61	105.9	C21—C20—H202	109.1
C11—C6—H61	107.8	H201—C20—H202	109.9
C6—C7—C8	109.50 (15)	C20—C21—C22	111.18 (17)
C6—C7—H71	108.4	C20—C21—H211	109.8
C8—C7—H71	110.0	C22—C21—H211	109.4
C6—C7—H72	109.3	C20—C21—H212	108.1
C8—C7—H72	110.0	C22—C21—H212	108.2
H71—C7—H72	109.6	H211—C21—H212	110.2
C7—C8—C9	111.39 (17)	C21—C22—C23	111.87 (16)
C7—C8—H81	109.0	C21—C22—H221	109.3
C9—C8—H81	107.9	C23—C22—H221	108.9
C7—C8—H82	109.4	C21—C22—H222	108.9
C9—C8—H82	109.6	C23—C22—H222	108.8
H81—C8—H82	109.4	H221—C22—H222	109.0
C8—C9—C10	111.01 (16)	C22—C23—C18	109.02 (16)
C8—C9—H91	108.9	C22—C23—H231	110.1
C10—C9—H91	109.0	C18—C23—H231	110.7
C8—C9—H92	110.9	C22—C23—H232	108.4
C10—C9—H92	107.9	C18—C23—H232	110.5
H91—C9—H92	109.0	H231—C23—H232	108.1
C9—C10—C11	111.47 (17)	N17—C24—C25	112.76 (16)
C9—C10—H101	108.6	N17—C24—H241	107.3

C11—C10—H101	110.4	C25—C24—H241	110.3
C9—C10—H102	108.3	N17—C24—H242	108.1
C11—C10—H102	108.5	C25—C24—H242	108.8
H101—C10—H102	109.6	H241—C24—H242	109.5
C10—C11—C6	109.16 (16)	C24—C25—H252	107.9
C10—C11—H111	110.0	C24—C25—H253	109.3
C6—C11—H111	109.5	H252—C25—H253	111.0
C10—C11—H112	108.7	C24—C25—H251	108.6
C6—C11—H112	109.5	H252—C25—H251	109.8
H111—C11—H112	110.0	H253—C25—H251	110.1
N5—C12—C13	113.64 (16)		

2. Crystal data for Ni-DTC

$C_9H_{16}NNi_{0.50}S_2$	?
M_r = 231.72	D_x = 1.431 Mg m^{-3}
Orthorhombic, *Pbca*	Melting point: ? K
Hall symbol: ?	Mo *Kα* radiation, λ = 0.71073 Å
a = 9.9135 (4) Å	Cell parameters from 3099 reflections
b = 12.4601 (5) Å	θ = 2–29°
c = 17.4087 (6) Å	μ = 1.30 mm^{-1}
V = 2150.38 (14) Å^3	*T* = 100 K
Z = 8	Prismatic, green
F(000) = 984	0.32 × 0.13 × 0.09 mm

Data Collection

Oxford Diffraction Gemini diffractometer	2114 reflections with $I > 2.0\sigma(I)$
Radiation source: ?	R_{int} = 0.031
graphite	θ_{max} = 28.7°, θ_{min} = 2.3°
ω scans	$h = -10 \rightarrow 13$
Absorption correction: multi-scan CrysAlis, (Oxford Diffraction, 2002)	$k = -15 \rightarrow 15$
T_{min} = 0.73, T_{max} = 0.89	$l = -22 \rightarrow 19$
9128 measured reflections	Standard reflections: 0
2518 independent reflections	

Refinement

Refinement on F^2	Secondary atom site location: ?
Least-squares matrix: full	Hydrogen site location: difference Fourier map
$R[F^2 > 2\sigma(F^2)]$ = 0.029	H-atom parameters constrained
$wR(F^2)$ = 0.065	Method = Modified Sheldrick $w = 1/[\sigma^2(F^2) + (0.02P)^2 + 2.33P]$, where $P = (\max(F_o^2,0) + 2F_c^2)/3$

S = 0.97	$(\Delta/\sigma)_{\max}$ = 0.001
2510 reflections	$\Delta\rho_{\max}$ = 0.61 e Å^{-3}
115 parameters	$\Delta\rho_{\min}$ = −0.51 e Å^{-3}
0 restraints	Extinction correction: None
? constraints	Extinction coefficient: ?
Primary atom site location: structure-invariant direct methods	

Bond lengths (Å) for Ni-DTC

Ni1—C3i	2.6934 (19)	C8—C9	1.529 (3)
Ni1—S4i	2.2102 (4)	C8—H81	0.967
Ni1—S2i	2.1990 (5)	C8—H82	0.971
Ni1—S2	2.1990 (5)	C9—C10	1.522 (3)
Ni1—C3	2.6934 (19)	C9—H91	0.973
Ni1—S4	2.2102 (4)	C9—H92	0.964
S2—C3	1.7240 (18)	C10—C11	1.531 (2)
C3—S4	1.7275 (19)	C10—H101	0.978
C3—N5	1.317 (2)	C10—H102	0.961
N5—C6	1.492 (2)	C11—H111	0.990
N5—C12	1.484 (2)	C11—H112	0.976
C6—C7	1.521 (3)	C12—C13	1.516 (3)
C6—C11	1.529 (3)	C12—H121	0.986
C6—H61	0.988	C12—H122	0.969
C7—C8	1.527 (3)	C13—H132	0.969
C7—H71	0.974	C13—H133	0.962
C7—H72	0.961	C13—H131	0.965

Bond angle (°) for Ni-DTC

C3^{i}—Ni1—S4^{i}	39.74 (4)	H71—C7—H72	108.6
C3^{i}—Ni1—S2^{i}	39.67 (4)	C7—C8—C9	111.55 (16)
S4^{i}—Ni1—S2^{i}	79.401 (17)	C7—C8—H81	108.7
C3^{i}—Ni1—S2	140.33 (4)	C9—C8—H81	109.7
S4^{i}—Ni1—S2	100.599 (17)	C7—C8—H82	109.0
S2^{i}—Ni1—S2	179.995	C9—C8—H82	108.4
C3^{i}—Ni1—C3	179.996	H81—C8—H82	109.4
S4^{i}—Ni1—C3	140.26 (4)	C8—C9—C10	110.26 (16)
S2^{i}—Ni1—C3	140.33 (4)	C8—C9—H91	109.2
S2—Ni1—C3	39.67 (4)	C10—C9—H91	111.2
C3^{i}—Ni1—S4	140.26 (4)	C8—C9—H92	108.5
S4^{i}—Ni1—S4	179.995	C10—C9—H92	108.8
S2^{i}—Ni1—S4	100.599 (17)	H91—C9—H92	108.9
S2—Ni1—S4	79.401 (17)	C9—C10—C11	111.20 (15)
C3—Ni1—S4	39.74 (4)	C9—C10—H101	110.1
Ni1—S2—C3	85.82 (6)	C11—C10—H101	109.3

S2—C3—Ni1	54.51 (5)	C9—C10—H102	108.7
S2—C3—S4	109.37 (10)	C11—C10—H102	108.6
Ni1—C3—S4	54.88 (5)	H101—C10—H102	108.9
S2—C3—N5	124.62 (14)	C10—C11—C6	109.98 (16)
Ni1—C3—N5	178.28 (13)	C10—C11—H111	108.3
S4—C3—N5	126.01 (14)	C6—C11—H111	109.6
C3—S4—Ni1	85.38 (6)	C10—C11—H112	109.1
C3—N5—C6	120.82 (15)	C6—C11—H112	109.0
C3—N5—C12	119.90 (15)	H111—C11—H112	110.8
C6—N5—C12	118.64 (15)	N5—C12—C13	113.40 (16)
N5—C6—C7	109.64 (14)	N5—C12—H121	109.1
N5—C6—C11	112.04 (15)	C13—C12—H121	108.6
C7—C6—C11	111.77 (15)	N5—C12—H122	107.2
N5—C6—H61	106.6	C13—C12—H122	109.6
C7—C6—H61	109.2	H121—C12—H122	109.0
C11—C6—H61	107.4	C12—C13—H132	107.5
C6—C7—C8	111.30 (15)	C12—C13—H133	108.9
C6—C7—H71	108.9	H132—C13—H133	110.2
C8—C7—H71	109.1	C12—C13—H131	110.3
C6—C7—H72	108.9	H132—C13—H131	110.5
C8—C7—H72	110.0	H133—C13—H131	109.5

3. Crystal data for Zn-DTC

$C_{18}H_{32}N_2S_4Zn$	?
M_r = 470.11	D_x = 1.405 Mg m^{-3}
Monoclinic, $P2_1/c$	Melting point: ? K
Hall symbol: ?	Mo $K\alpha$ radiation, λ = 0.71073 Å
a = 13.1079 (2) Å	Cell parameters from 42931 reflections
b = 14.3315 (2) Å	θ = 2–29°
c = 11.9614 (2) Å	μ = 1.49 mm^{-1}
β = 98.4533 (12)°	T = 100 K
V = 2222.61 (6) Å^3	Block-like, colourless
Z = 4	0.36 × 0.24 × 0.21 mm
F(000) = 992	

Data Collection

Oxford Diffraction Gemini diffractometer	5218 reflections with $I > 2.0\sigma(I)$
Radiation source: ?	R_{int} = 0.037
graphite	θ_{max} = 29.0°, θ_{min} = 2.2°
ω scans	$h = -17 \rightarrow 17$
Absorption correction: multi-scan	$k = -19 \rightarrow 19$

CrysAlis, (Oxford Diffraction, 2002)	
$T_{\min}$ = 0.63, $T_{\max}$ = 0.73	$l = -15 \rightarrow 15$
94463 measured reflections	Standard reflections: 0
5604 independent reflections	

Refinement

Refinement on F^2	Secondary atom site location: ?
Least-squares matrix: full	Hydrogen site location: difference Fourier map
$R[F^2 > 2\sigma(F^2)] = 0.019$	H-atom parameters constrained
$wR(F^2) = 0.044$	Method = Modified Sheldrick $w = 1/[\sigma^2(F^2) + (0.02P)^2 + 1.42P]$, where $P = (\max(F_o^2,0) + 2F_c^2)/3$
$S = 0.96$	$(\Delta/\sigma)_{\max} = 0.001$
5587 reflections	$\Delta\rho_{\max}$ = 0.40 e Å^{-3}
226 parameters	$\Delta\rho_{\min}$ = −0.25 e Å^{-3}
0 restraints	Extinction correction: None
? constraints	Extinction coefficient: ?
Primary atom site location: structure-invariant direct methods	

Bond lengths (Å) for Zn-DTC

Zn1—S4^{i}	2.3821 (3)	C13—H133	0.955
Zn1—S2	2.3498 (3)	C13—H131	0.971
Zn1—S4	2.7542 (3)	S14—C15	1.7380 (11)
Zn1—S14	2.3566 (3)	C15—S16	1.7204 (11)
Zn1—S16	2.4488 (3)	C15—N17	1.3329 (13)
S2—C3	1.7201 (11)	N17—C18	1.4871 (13)
C3—S4	1.7545 (11)	N17—C24	1.4797 (13)
C3—N5	1.3212 (13)	C18—C19	1.5288 (15)
N5—C6	1.4854 (13)	C18—C23	1.5252 (16)
N5—C12	1.4814 (14)	C18—H181	0.977
C6—C7	1.5291 (15)	C19—C20	1.5308 (16)
C6—C11	1.5290 (15)	C19—H191	0.973
C6—H61	0.976	C19—H192	0.985
C7—C8	1.5321 (18)	C20—C21	1.519 (2)
C7—H71	0.981	C20—H201	0.979
C7—H72	0.966	C20—H202	0.978
C8—C9	1.525 (2)	C21—C22	1.523 (2)
C8—H81	0.987	C21—H211	0.983
C8—H82	0.962	C21—H212	0.959
C9—C10	1.5229 (19)	C22—C23	1.5330 (17)
C9—H91	0.979	C22—H221	0.963
C9—H92	0.961	C22—H222	0.983
C10—C11	1.5274 (16)	C23—H231	0.977

C10—H101	0.969	C23—H232	0.981
C10—H102	0.980	C24—C25	1.5211 (17)
C11—H111	0.963	C24—H241	0.976
C11—H112	0.980	C24—H242	0.956
C12—C13	1.5230 (16)	C25—H252	0.960
C12—H121	0.969	C25—H253	0.971
C12—H122	0.959	C25—H251	0.956
C13—H132	0.964		

Bond angle (°) for Zn-DTC

S4^{i}—Zn1—S2	103.901 (10)	C12—C13—H132	110.1
S4^{i}—Zn1—S4	88.827 (9)	C12—C13—H133	110.0
S2—Zn1—S4	70.352 (9)	H132—C13—H133	108.0
S4^{i}—Zn1—S14	116.460 (10)	C12—C13—H131	110.7
S2—Zn1—S14	137.623 (11)	H132—C13—H131	109.0
S4—Zn1—S14	97.054 (9)	H133—C13—H131	109.0
S4^{i}—Zn1—S16	108.925 (10)	Zn1—S14—C15	84.72 (4)
S2—Zn1—S16	103.826 (10)	S14—C15—S16	117.13 (6)
S4—Zn1—S16	162.244 (10)	S14—C15—N17	121.58 (8)
S14—Zn1—S16	75.740 (9)	S16—C15—N17	121.29 (8)
Zn1—S2—C3	92.91 (4)	C15—S16—Zn1	82.25 (4)
S2—C3—S4	116.76 (6)	C15—N17—C18	121.68 (9)
S2—C3—N5	121.63 (8)	C15—N17—C24	120.37 (9)
S4—C3—N5	121.58 (8)	C18—N17—C24	117.52 (8)
Zn1—S4—C3	79.43 (3)	N17—C18—C19	111.52 (9)
Zn1—S4—Zn1^{i}	91.173 (9)	N17—C18—C23	111.24 (9)
C3—S4—Zn1^{i}	100.75 (3)	C19—C18—C23	112.41 (9)
C3—N5—C6	121.28 (9)	N17—C18—H181	106.3
C3—N5—C12	120.45 (9)	C19—C18—H181	106.9
C6—N5—C12	117.86 (8)	C23—C18—H181	108.2
N5—C6—C7	113.47 (9)	C18—C19—C20	110.04 (10)
N5—C6—C11	109.56 (9)	C18—C19—H191	109.6
C7—C6—C11	111.72 (9)	C20—C19—H191	109.3
N5—C6—H61	105.8	C18—C19—H192	109.3
C7—C6—H61	107.8	C20—C19—H192	110.5
C11—C6—H61	108.2	H191—C19—H192	108.2
C6—C7—C8	108.75 (10)	C19—C20—C21	110.98 (10)
C6—C7—H71	110.3	C19—C20—H201	110.0
C8—C7—H71	108.0	C21—C20—H201	110.2
C6—C7—H72	109.5	C19—C20—H202	109.1
C8—C7—H72	109.8	C21—C20—H202	108.1
H71—C7—H72	110.5	H201—C20—H202	108.4
C7—C8—C9	111.95 (10)	C20—C21—C22	110.48 (11)
C7—C8—H81	109.2	C20—C21—H211	109.7
C9—C8—H81	109.6	C22—C21—H211	109.3
C7—C8—H82	108.7	C20—C21—H212	109.4
C9—C8—H82	108.7	C22—C21—H212	109.5

H81—C8—H82	108.6	H211—C21—H212	108.5
C8—C9—C10	111.02 (10)	C21—C22—C23	111.03 (12)
C8—C9—H91	109.8	C21—C22—H221	108.8
C10—C9—H91	108.5	C23—C22—H221	109.4
C8—C9—H92	109.7	C21—C22—H222	108.9
C10—C9—H92	109.4	C23—C22—H222	108.8
H91—C9—H92	108.3	H221—C22—H222	110.0
C9—C10—C11	110.73 (11)	C22—C23—C18	111.11 (10)
C9—C10—H101	109.8	C22—C23—H231	109.2
C11—C10—H101	109.4	C18—C23—H231	108.8
C9—C10—H102	108.5	C22—C23—H232	109.6
C11—C10—H102	108.5	C18—C23—H232	109.1
H101—C10—H102	109.9	H231—C23—H232	108.9
C6—C11—C10	110.01 (10)	N17—C24—C25	114.77 (9)
C6—C11—H111	109.2	N17—C24—H241	106.4
C10—C11—H111	109.2	C25—C24—H241	110.3
C6—C11—H112	109.4	N17—C24—H242	108.0
C10—C11—H112	110.3	C25—C24—H242	109.1
H111—C11—H112	108.7	H241—C24—H242	108.0
N5—C12—C13	112.29 (9)	C24—C25—H252	108.8
N5—C12—H121	108.4	C24—C25—H253	110.8
C13—C12—H121	109.3	H252—C25—H253	108.3
N5—C12—H122	106.9	C24—C25—H251	110.7
C13—C12—H122	110.6	H252—C25—H251	109.4
H121—C12—H122	109.3	H253—C25—H251	108.7

4. Crystal data for Sb-DTC

$C_{27}H_{48}N_3S_6Sb$	$F(000) = 1512$
$M_r = 728.84$	?
Triclinic, $P\bar{1}$	$D_x = 1.457$ Mg m^{-3}
Hall symbol: ?	Melting point: ? K
$a = 9.7295$ (3) Å	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
$b = 17.1268$ (5) Å	Cell parameters from 28598 reflections
$c = 20.5133$ (6) Å	$\theta = 2$–$29°$
$\alpha = 99.878$ (3)°	$\mu = 1.23$ mm^{-1}
$\beta = 99.057$ (2)°	$T = 100$ K
$\gamma = 91.057$ (3)°	Block, pale yellow
$V = 3321.95$ (18) Å^3	0.25 × 0.22 × 0.09 mm
$Z = 4$	

Data Collection

Oxford Diffraction Gemini diffractometer	13143 reflections with $I > 2.0\sigma(I)$
Radiation source: ?	$R_{\mathrm{int}} = 0.035$
graphite	$\theta_{\mathrm{max}} = 28.8°$, $\theta_{\mathrm{min}} = 2.2°$
ω scans	$h = -13\rightarrow13$
Absorption correction: multi-scan CrysAlis, (Oxford Diffraction, 2002)	$k = -23\rightarrow22$
$T_{\mathrm{min}} = 0.75$, $T_{\mathrm{max}} = 0.90$	$l = -27\rightarrow27$
85445 measured reflections	Standard reflections: 0
15831 independent reflections	

Refinement

Refinement on F^2	Secondary atom site location: ?
Least-squares matrix: full	Hydrogen site location: difference Fourier map
$R[F^2 > 2\sigma(F^2)] = 0.038$	H-atom parameters constrained
$wR(F^2) = 0.094$	Method = Modified Sheldrick $w = 1/[\sigma^2(F^2) + (0.03P)^2 + 11.78P]$, where $P = (\max(F_o^2,0) + 2F_c^2)/3$
$S = 1.05$	$(\Delta/\sigma)_{\mathrm{max}} = 0.001$
15773 reflections	$\Delta\rho_{\mathrm{max}} = 1.71$ e Å^{-3}
667 parameters	$\Delta\rho_{\mathrm{min}} = -1.34$ e Å^{-3}
0 restraints	Extinction correction: None
? constraints	Extinction coefficient: ?
Primary atom site location: structure-invariant direct methods	

Bond lengths (Å) for Sb-DTC

Sb101—S102	2.5433 (8)	Sb201—S202	2.5422 (9)
Sb101—S104	2.8679 (9)	Sb201—S204	2.9394 (9)
Sb101—S114	2.5513 (8)	Sb201—S214	2.5456 (9)
Sb101—S116	2.8732 (9)	Sb201—S216	3.0086 (9)
Sb101—S126	2.5743 (9)	Sb201—S226	2.5364 (8)
Sb101—S128	3.0493 (8)	Sb201—S228	2.8453 (9)
S102—C103	1.753 (3)	S202—C203	1.747 (3)
C103—S104	1.700 (3)	C203—S204	1.706 (3)
C103—N105	1.337 (4)	C203—N205	1.333 (4)
N105—C106	1.482 (4)	N205—C206	1.481 (4)
N105—C112	1.481 (4)	N205—C208	1.488 (4)
C106—C107	1.531 (5)	C206—C207	1.522 (5)
C106—C111	1.534 (4)	C206—H2062	0.975
C106—H1061	0.990	C206—H2061	0.978
C107—C108	1.531 (5)	C207—H2071	0.963

C107—H1072	0.972	C207—H2073	0.963
C107—H1071	0.974	C207—H2072	0.955
C108—C109	1.530 (5)	C208—C209	1.528 (5)
C108—H1082	0.979	C208—C213	1.525 (5)
C108—H1081	0.968	C208—H2081	0.981
C109—C110	1.524 (5)	C209—C210	1.521 (5)
C109—H1091	0.977	C209—H2092	0.964
C109—H1092	0.977	C209—H2091	0.972
C110—C111	1.524 (5)	C210—C211	1.528 (5)
C110—H1102	0.978	C210—H2101	0.974
C110—H1101	0.972	C210—H2102	0.976
C111—H1112	0.970	C211—C212	1.521 (5)
C111—H1111	0.970	C211—H2112	0.970
C112—C113	1.521 (5)	C211—H2111	0.971
C112—H1121	0.979	C212—C213	1.533 (5)
C112—H1122	0.972	C212—H2122	0.968
C113—H1132	0.965	C212—H2121	0.974
C113—H1131	0.961	C213—H2131	0.967
C113—H1133	0.962	C213—H2132	0.965
S114—C115	1.755 (3)	S214—C215	1.761 (3)
C115—S116	1.711 (3)	C215—S216	1.702 (3)
C115—N117	1.326 (4)	C215—N217	1.329 (4)
N117—C118	1.487 (4)	N217—C218	1.491 (4)
N117—C124	1.482 (4)	N217—C224	1.479 (4)
C118—C119	1.532 (4)	C218—C219	1.528 (5)
C118—C123	1.529 (4)	C218—C223	1.534 (4)
C118—H1181	0.980	C218—H2181	0.984
C119—C120	1.536 (5)	C219—C220	1.532 (5)
C119—H1191	0.962	C219—H2191	0.969
C119—H1192	0.973	C219—H2192	0.973
C120—C121	1.521 (5)	C220—C221	1.523 (5)
C120—H1202	0.971	C220—H2202	0.980
C120—H1201	0.967	C220—H2201	0.972
C121—C122	1.528 (5)	C221—C222	1.520 (5)
C121—H1212	0.970	C221—H2212	0.968
C121—H1211	0.979	C221—H2211	0.973
C122—C123	1.530 (5)	C222—C223	1.531 (5)
C122—H1221	0.974	C222—H2221	0.975
C122—H1222	0.970	C222—H2222	0.970
C123—H1232	0.967	C223—H2232	0.977
C123—H1231	0.975	C223—H2231	0.970
C124—C125	1.520 (5)	C224—C225	1.521 (5)
C124—H1242	0.970	C224—H2242	0.974
C124—H1241	0.981	C224—H2241	0.974
C125—H1251	0.962	C225—H2251	0.966
C125—H1253	0.957	C225—H2253	0.959
C125—H1252	0.957	C225—H2252	0.960
S126—C127	1.758 (3)	S226—C227	1.756 (3)

C127—S128	1.694 (3)	C227—S228	1.706 (3)
C127—N129	1.344 (4)	C227—N229	1.327 (4)
N129—C130	1.475 (4)	N229—C230	1.490 (4)
N129—C132	1.481 (4)	N229—C236	1.482 (4)
C130—C131	1.522 (5)	C230—C231	1.537 (5)
C130—H1301	0.971	C230—C235	1.526 (5)
C130—H1302	0.976	C230—H2301	0.988
C131—H1312	0.965	C231—C232	1.526 (5)
C131—H1311	0.961	C231—H2312	0.969
C131—H1313	0.964	C231—H2311	0.971
C132—C133	1.528 (5)	C232—C233	1.523 (6)
C132—C137	1.533 (5)	C232—H2321	0.974
C132—H1321	0.979	C232—H2322	0.973
C133—C134	1.529 (5)	C233—C234	1.521 (6)
C133—H1331	0.971	C233—H2332	0.971
C133—H1332	0.967	C233—H2331	0.973
C134—C135	1.520 (5)	C234—C235	1.527 (5)
C134—H1342	0.970	C234—H2342	0.975
C134—H1341	0.968	C234—H2341	0.975
C135—C136	1.526 (6)	C235—H2351	0.976
C135—H1352	0.979	C235—H2352	0.966
C135—H1351	0.977	C236—C237	1.520 (6)
C136—C137	1.531 (5)	C236—H2362	0.972
C136—H1361	0.974	C236—H2361	0.975
C136—H1362	0.971	C237—H2373	0.959
C137—H1372	0.977	C237—H2372	0.964
C137—H1371	0.967	C237—H2371	0.958

Bond angle (°) for Sb-DTC

S102—Sb101—S104	66.09 (3)	S202—Sb201—S204	65.09 (3)
S102—Sb101—S114	88.64 (3)	S202—Sb201—S214	80.70 (3)
S104—Sb101—S114	153.47 (3)	S204—Sb201—S214	100.47 (3)
S102—Sb101—S116	90.56 (3)	S202—Sb201—S216	144.71 (3)
S104—Sb101—S116	104.38 (3)	S204—Sb201—S216	122.89 (2)
S114—Sb101—S116	66.16 (2)	S214—Sb201—S216	64.21 (2)
S102—Sb101—S126	84.58 (3)	S202—Sb201—S226	90.93 (3)
S104—Sb101—S126	98.23 (3)	S204—Sb201—S226	150.99 (3)
S114—Sb101—S126	86.74 (3)	S214—Sb201—S226	90.83 (3)
S116—Sb101—S126	152.60 (3)	S216—Sb201—S226	86.09 (3)
S102—Sb101—S128	147.94 (3)	S202—Sb201—S228	82.56 (3)
S104—Sb101—S128	116.12 (3)	S204—Sb201—S228	92.80 (3)
S114—Sb101—S128	89.47 (3)	S214—Sb201—S228	151.78 (3)
S116—Sb101—S128	117.69 (2)	S216—Sb201—S228	127.38 (3)
S126—Sb101—S128	63.36 (2)	S226—Sb201—S228	66.85 (3)
Sb101—S102—C103	92.40 (11)	Sb201—S202—C203	93.03 (11)
S102—C103—S104	118.22 (19)	S202—C203—S204	118.55 (18)
S102—C103—N105	120.5 (2)	S202—C203—N205	118.5 (2)

S104—C103—N105	121.3 (3)	S204—C203—N205	122.9 (2)
C103—S104—Sb101	82.93 (11)	C203—S204—Sb201	81.02 (11)
C103—N105—C106	123.6 (3)	C203—N205—C206	121.0 (3)
C103—N105—C112	119.3 (3)	C203—N205—C208	121.5 (3)
C106—N105—C112	117.0 (3)	C206—N205—C208	117.5 (3)
N105—C106—C107	110.8 (3)	N205—C206—C207	113.2 (3)
N105—C106—C111	110.5 (3)	N205—C206—H2062	108.3
C107—C106—C111	112.4 (3)	C207—C206—H2062	108.6
N105—C106—H1061	107.2	N205—C206—H2061	108.1
C107—C106—H1061	108.4	C207—C206—H2061	108.8
C111—C106—H1061	107.4	H2062—C206—H2061	110.0
C106—C107—C108	111.3 (3)	C206—C207—H2071	110.2
C106—C107—H1072	109.2	C206—C207—H2073	109.4
C108—C107—H1072	108.2	H2071—C207—H2073	109.0
C106—C107—H1071	109.6	C206—C207—H2072	110.6
C108—C107—H1071	110.1	H2071—C207—H2072	108.5
H1072—C107—H1071	108.4	H2073—C207—H2072	109.1
C107—C108—C109	110.7 (3)	N205—C208—C209	112.6 (3)
C107—C108—H1082	109.3	N205—C208—C213	110.9 (3)
C109—C108—H1082	110.6	C209—C208—C213	111.4 (3)
C107—C108—H1081	108.3	N205—C208—H2081	106.4
C109—C108—H1081	108.5	C209—C208—H2081	108.5
H1082—C108—H1081	109.4	C213—C208—H2081	106.6
C108—C109—C110	109.9 (3)	C208—C209—C210	109.9 (3)
C108—C109—H1091	109.2	C208—C209—H2092	109.9
C110—C109—H1091	109.6	C210—C209—H2092	110.0
C108—C109—H1092	109.0	C208—C209—H2091	108.1
C110—C109—H1092	109.5	C210—C209—H2091	109.5
H1091—C109—H1092	109.5	H2092—C209—H2091	109.4
C109—C110—C111	111.7 (3)	C209—C210—C211	111.2 (3)
C109—C110—H1102	108.6	C209—C210—H2101	108.9
C111—C110—H1102	108.4	C211—C210—H2101	109.7
C109—C110—H1101	108.9	C209—C210—H2102	108.5
C111—C110—H1101	109.7	C211—C210—H2102	108.7
H1102—C110—H1101	109.5	H2101—C210—H2102	109.8
C106—C111—C110	111.8 (3)	C210—C211—C212	111.5 (3)
C106—C111—H1112	108.2	C210—C211—H2112	108.9
C110—C111—H1112	108.1	C212—C211—H2112	108.1
C106—C111—H1111	110.1	C210—C211—H2111	108.9
C110—C111—H1111	110.2	C212—C211—H2111	110.2
H1112—C111—H1111	108.5	H2112—C211—H2111	109.2
N105—C112—C113	112.7 (3)	C211—C212—C213	110.6 (3)
N105—C112—H1121	108.8	C211—C212—H2122	109.8
C113—C112—H1121	109.2	C213—C212—H2122	108.7
N105—C112—H1122	107.9	C211—C212—H2121	109.3
C113—C112—H1122	108.3	C213—C212—H2121	109.0
H1121—C112—H1122	110.0	H2122—C212—H2121	109.4
C112—C113—H1132	109.3	C212—C213—C208	109.7 (3)

C112—C113—H1131	110.1	C212—C213—H2131	109.0
H1132—C113—H1131	108.9	C208—C213—H2131	109.5
C112—C113—H1133	110.8	C212—C213—H2132	110.3
H1132—C113—H1133	108.5	C208—C213—H2132	109.4
H1131—C113—H1133	109.1	H2131—C213—H2132	108.9
Sb101—S114—C115	91.45 (11)	Sb201—S214—C215	94.55 (11)
S114—C115—S116	118.15 (19)	S214—C215—S216	118.61 (19)
S114—C115—N117	119.7 (2)	S214—C215—N217	118.5 (2)
S116—C115—N117	122.2 (2)	S216—C215—N217	122.9 (3)
C115—S116—Sb101	82.00 (11)	C215—S216—Sb201	80.69 (11)
C115—N117—C118	121.5 (3)	C215—N217—C218	122.0 (3)
C115—N117—C124	120.3 (3)	C215—N217—C224	120.1 (3)
C118—N117—C124	117.7 (3)	C218—N217—C224	117.6 (3)
N117—C118—C119	112.0 (3)	N217—C218—C219	112.7 (3)
N117—C118—C123	111.1 (3)	N217—C218—C223	111.0 (3)
C119—C118—C123	111.8 (3)	C219—C218—C223	111.9 (3)
N117—C118—H1181	106.7	N217—C218—H2181	106.2
C119—C118—H1181	107.9	C219—C218—H2181	107.3
C123—C118—H1181	107.0	C223—C218—H2181	107.4
C118—C119—C120	110.0 (3)	C218—C219—C220	110.2 (3)
C118—C119—H1191	109.1	C218—C219—H2191	108.5
C120—C119—H1191	109.4	C220—C219—H2191	109.4
C118—C119—H1192	109.1	C218—C219—H2192	109.7
C120—C119—H1192	109.6	C220—C219—H2192	109.3
H1191—C119—H1192	109.6	H2191—C219—H2192	109.7
C119—C120—C121	111.5 (3)	C219—C220—C221	111.3 (3)
C119—C120—H1202	109.2	C219—C220—H2202	109.1
C121—C120—H1202	109.1	C221—C220—H2202	109.3
C119—C120—H1201	109.2	C219—C220—H2201	108.5
C121—C120—H1201	108.9	C221—C220—H2201	109.7
H1202—C120—H1201	108.9	H2202—C220—H2201	108.9
C120—C121—C122	110.7 (3)	C220—C221—C222	110.6 (3)
C120—C121—H1212	109.4	C220—C221—H2212	110.1
C122—C121—H1212	108.9	C222—C221—H2212	109.3
C120—C121—H1211	109.2	C220—C221—H2211	108.8
C122—C121—H1211	109.6	C222—C221—H2211	108.7
H1212—C121—H1211	109.0	H2212—C221—H2211	109.2
C121—C122—C123	110.9 (3)	C221—C222—C223	111.8 (3)
C121—C122—H1221	109.9	C221—C222—H2221	109.1
C123—C122—H1221	109.1	C223—C222—H2221	108.1
C121—C122—H1222	108.9	C221—C222—H2222	109.3
C123—C122—H1222	108.6	C223—C222—H2222	108.7
H1221—C122—H1222	109.5	H2221—C222—H2222	109.8
C122—C123—C118	109.6 (3)	C218—C223—C222	109.9 (3)
C122—C123—H1232	109.6	C218—C223—H2232	109.4
C118—C123—H1232	108.8	C222—C223—H2232	110.1
C122—C123—H1231	109.6	C218—C223—H2231	108.9
C118—C123—H1231	109.1	C222—C223—H2231	108.8

H1232—C123—H1231	110.1	H2232—C223—H2231	109.8
N117—C124—C125	113.7 (3)	N217—C224—C225	112.9 (3)
N117—C124—H1242	108.4	N217—C224—H2242	108.6
C125—C124—H1242	108.9	C225—C224—H2242	108.8
N117—C124—H1241	108.2	N217—C224—H2241	108.8
C125—C124—H1241	109.0	C225—C224—H2241	108.8
H1242—C124—H1241	108.5	H2242—C224—H2241	108.8
C124—C125—H1251	109.0	C224—C225—H2251	108.8
C124—C125—H1253	109.1	C224—C225—H2253	109.8
H1251—C125—H1253	109.8	H2251—C225—H2253	108.5
C124—C125—H1252	109.4	C224—C225—H2252	110.6
H1251—C125—H1252	109.8	H2251—C225—H2252	109.2
H1253—C125—H1252	109.7	H2253—C225—H2252	109.9
Sb101—S126—C127	95.49 (11)	Sb201—S226—C227	91.31 (12)
S126—C127—S128	119.43 (19)	S226—C227—S228	118.5 (2)
S126—C127—N129	117.7 (2)	S226—C227—N229	119.5 (3)
S128—C127—N129	122.9 (2)	S228—C227—N229	122.0 (3)
C127—S128—Sb101	81.19 (11)	C227—S228—Sb201	82.39 (12)
C127—N129—C130	120.4 (3)	C227—N229—C230	121.9 (3)
C127—N129—C132	121.2 (3)	C227—N229—C236	119.9 (3)
C130—N129—C132	118.0 (3)	C230—N229—C236	117.7 (3)
N129—C130—C131	113.8 (3)	N229—C230—C231	109.6 (3)
N129—C130—H1301	106.9	N229—C230—C235	112.6 (3)
C131—C130—H1301	108.4	C231—C230—C235	111.4 (3)
N129—C130—H1302	108.8	N229—C230—H2301	107.5
C131—C130—H1302	108.8	C231—C230—H2301	107.5
H1301—C130—H1302	110.0	C235—C230—H2301	108.1
C130—C131—H1312	108.4	C230—C231—C232	110.3 (3)
C130—C131—H1311	109.7	C230—C231—H2312	108.8
H1312—C131—H1311	109.8	C232—C231—H2312	108.6
C130—C131—H1313	108.6	C230—C231—H2311	109.3
H1312—C131—H1313	110.4	C232—C231—H2311	109.6
H1311—C131—H1313	109.9	H2312—C231—H2311	110.2
N129—C132—C133	111.9 (3)	C231—C232—C233	111.3 (3)
N129—C132—C137	111.8 (3)	C231—C232—H2321	108.8
C133—C132—C137	111.0 (3)	C233—C232—H2321	109.1
N129—C132—H1321	107.0	C231—C232—H2322	109.0
C133—C132—H1321	107.5	C233—C232—H2322	108.4
C137—C132—H1321	107.4	H2321—C232—H2322	110.2
C132—C133—C134	109.9 (3)	C232—C233—C234	110.8 (3)
C132—C133—H1331	109.3	C232—C233—H2332	109.0
C134—C133—H1331	109.7	C234—C233—H2332	109.0
C132—C133—H1332	108.9	C232—C233—H2331	108.8
C134—C133—H1332	108.7	C234—C233—H2331	109.6
H1331—C133—H1332	110.3	H2332—C233—H2331	109.6
C133—C134—C135	110.9 (3)	C233—C234—C235	110.8 (3)
C133—C134—H1342	109.4	C233—C234—H2342	109.5
C135—C134—H1342	109.4	C235—C234—H2342	108.9

C133—C134—H1341	109.1	C233—C234—H2341	109.4
C135—C134—H1341	109.1	C235—C234—H2341	108.8
H1342—C134—H1341	109.0	H2342—C234—H2341	109.5
C134—C135—C136	111.2 (3)	C234—C235—C230	110.3 (3)
C134—C135—H1352	109.8	C234—C235—H2351	108.9
C136—C135—H1352	108.3	C230—C235—H2351	109.6
C134—C135—H1351	109.0	C234—C235—H2352	110.4
C136—C135—H1351	109.1	C230—C235—H2352	109.2
H1352—C135—H1351	109.5	H2351—C235—H2352	108.4
C135—C136—C137	111.0 (3)	N229—C236—C237	112.1 (3)
C135—C136—H1361	108.7	N229—C236—H2362	108.0
C137—C136—H1361	108.7	C237—C236—H2362	109.8
C135—C136—H1362	109.3	N229—C236—H2361	107.8
C137—C136—H1362	109.7	C237—C236—H2361	109.5
H1361—C136—H1362	109.3	H2362—C236—H2361	109.6
C132—C137—C136	110.3 (3)	C236—C237—H2373	110.0
C132—C137—H1372	108.6	C236—C237—H2372	110.1
C136—C137—H1372	109.9	H2373—C237—H2372	108.7
C132—C137—H1371	109.2	C236—C237—H2371	110.1
C136—C137—H1371	110.0	H2373—C237—H2371	108.7
H1372—C137—H1371	108.8	H2372—C237—H2371	109.1

Appendix C: Reference JCPDS file, lattice parameter, main scattering angle and crystal planes of the calcined samples obtained at temperature 400 °C

Product	Lattice Parameter (Å)	JCPDS File No.	Main scattering angles, 2θ (°)	Crystal Planes [hkl]
CdS (2 h)	a = 4.136	06-0314 (CdS- hexagonal)	24.95	[100]
	b = 4.136		26.63	[002]
	c = 6.713		28.32	[101]
			43.85	[110]
			48.00	[103]
CdS (4 h)		06-0314	24.81	[100]
			26.50	[002]
			28.19	[101]
			43.73	[110]
			47.87	[103]
CdS (6 h)		06-0314	24.71	[100]
			26.40	[002]
			28.09	[101]
			43.61	[110]
			47.76	[103]
Cu$_2$S (2 h)	a = 11.82	09-0328 (Cu$_2$S- orthorhombic)	32.74	[362]
	b = 27.05		37.43	[382]
	c = 13.43		45.85	[600]
			48.39	[346]
			53.81	[337]
Cu$_2$S (4 h)		09-0328	32.83	[362]
			37.55	[382]
			45.96	[600]
			48.51	[346]
			53.97	[337]
Cu$_2$S (6 h)		09-0328	32.79	[362]
			37.45	[382]
			45.93	[600]
			48.43	[346]
			53.85	[337]
ZnS (2 h)	a = 3.820	005-0492 (ZnS- hexagonal)	27.12	[100]
	b = 3.820		28.54	[002]
	c = 6.260		30.60	[101]
			47.57	[110]

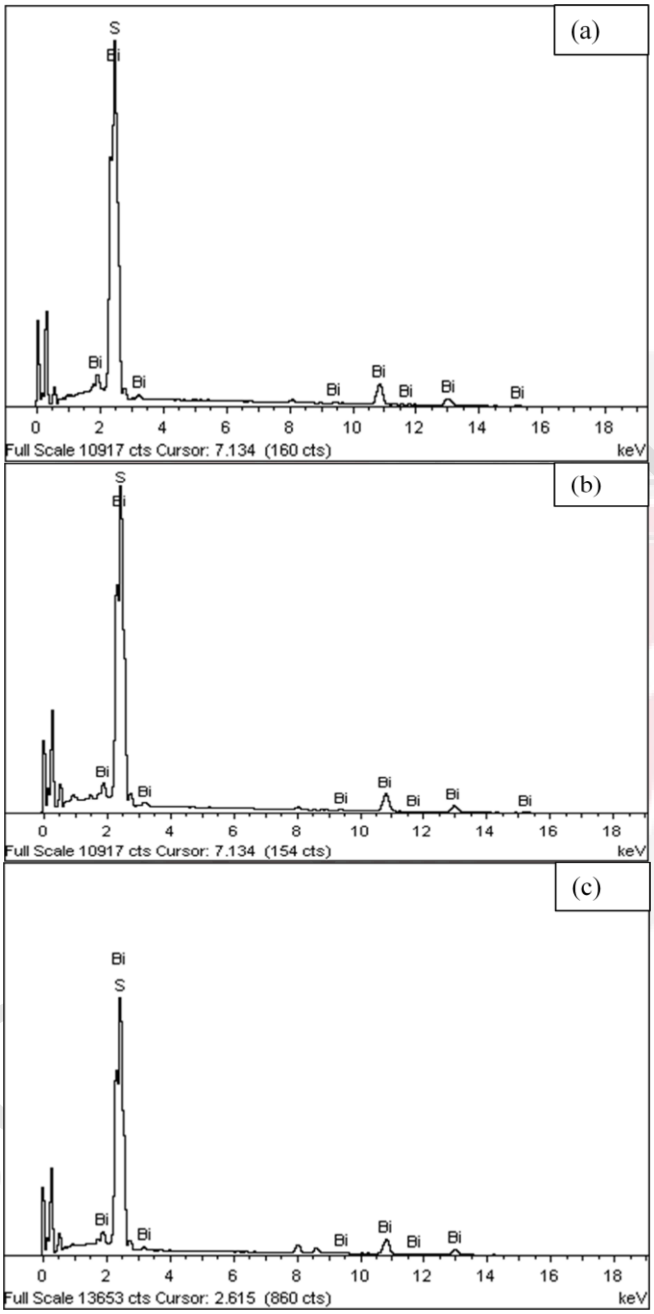
			51.86	[103]
ZnS (4 h)		005-0492	26.78	[100]
			28.25	[002]
			30.24	[101]
			47.30	[110]
			51.58	[103]
ZnS (6 h)		005-0492	26.98	[100]
			28.48	[002]
			30.42	[101]
			47.54	[110]
			51.76	[103]
Sb_2S_3 + Sb (2 h)	a = 11.2390 b = 11.3130 c = 3.8411	42-1393 (Sb_2S_3- orthorhombic)	24.87	[130]
			28.55	[320]
			29.14	[121]
			32.27	[221]
			39.95	[340]
			42.92	[421]
			46.70	[501]
			47.28	[151]
	a = 4.3084 b = 4.3084 c =11.2740	01-085-1322 (Sb- rhombohedral)	41.82	[110]
			51.47	[202]
			68.47	[122]
Sb_2S_3 + Sb (4 h)		42-1393	24.86	[130]
			28.55	[320]
			29.13	[121]
			32.25	[221]
			39.94	[340]
			42.91	[421]
			46.68	[501]
			47.21	[151]
		01-085-1322	41.81	[110]
			51.48	[202]
			68.47	[122]
Sb_2S_3 + Sb (6 h)		42-1393	24.99	[310]
			28.69	[320]
			29.28	[211]
			32.40	[221]
			40.10	[430]
			43.05	[250]
			46.83	[501]
			47.37	[151]

		01-085-1322	41.95	[110]
			51.60	[202]
			68.61	[122]
NiS (2 h)	a = 9.6190	065-3686 (NiS- rhombohedral)	32.17	[300]
	b = 9.6190		35.65	[021]
	c = 3.1499		40.41	[211]
			48.78	[131]
			52.58	[401]
	a = 3.4398	01-089-1957 (NiS- hexagonal)	34.41	[101]
	b = 3.4398		45.50	[102]
	c = 5.3482			
NiS (4 h)		065-3686	32.19	[300]
			35.66	[021]
			40.43	[211]
			48.80	[131]
			52.60	[401]
		01-089-1957	34.48	[101]
			45.61	[102]
			53.28	[110]
NiS (6 h)		065-3686	32.14	[300]
			35.62	[021]
			40.39	[211]
			48.77	[131]
			52.56	[401]
		01-089-1957	34.38	[101]
			45.49	[102]
			53.18	[110]
In_2S_3 (2 h)	a = 10.734	032-0456 (In_2S_3- cubic)	20.83	[211]
	b = 10.734		27.26	[311]
	c = 10.734		32.84	[400]
			38.59	[421]
			43.68	[511]
			47.91	[440]
In_2S_3 (4 h)		032-0456	20.81	[211]
			27.26	[311]
			33.04	[400]
			38.43	[421]
			43.58	[511]
			47.81	[440]
			55.94	[533]

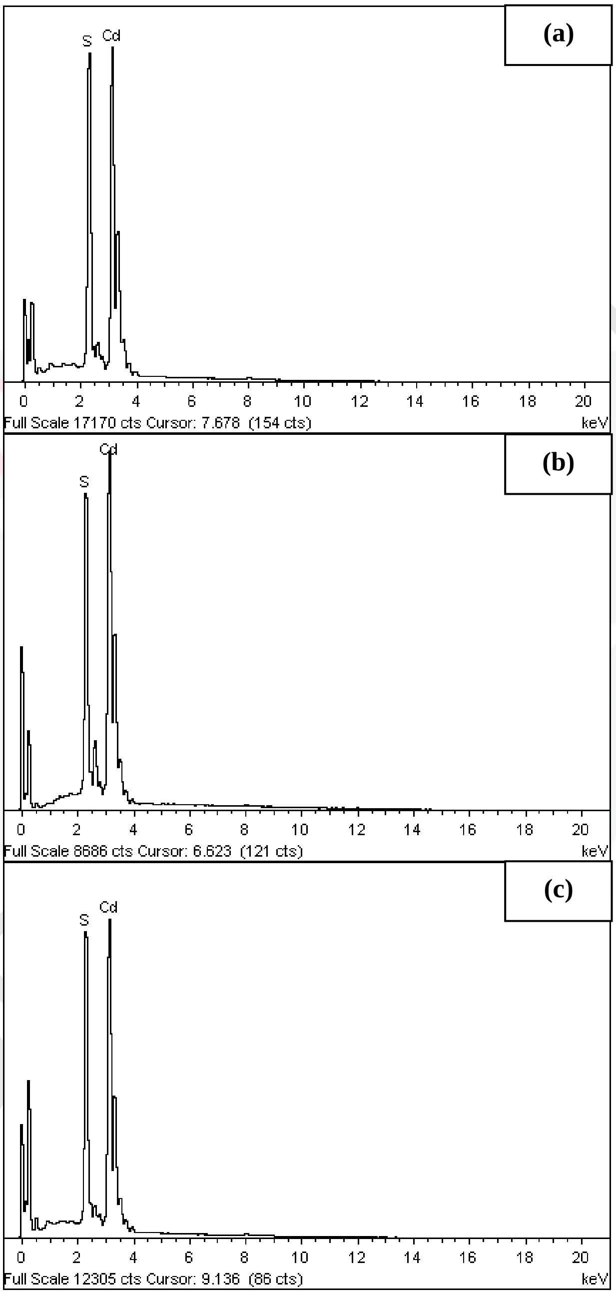
In_2S_3		032-0456	20.90	[211]
(6 h)			27.42	[311]
			32.68	[400]
			38.55	[421]
			43.86	[511]
			47.95	[440]
			55.74	[533]
Co_9S_8 +	a = 9.9273	065-1765	29.77	[311]
CoS +	b = 9.9273	(Co_9S_8	31.10	[222]
Co_3S_4	c = 9.9273	cubic)	39.54	[331]
(2 h)			47.51	[511]
			52.04	[440]
			73.20	[731]
			76.72	[800]
	a = 3.368	065-3418	35.30	[101]
	b = 3.368	(CoS hexagonal)	46.94	[102]
	c = 5.170		54.41	[110]
	a = 9.4020	19-03673	38.31	[400]
	b = 9.4020	(Co_3S_4cubic)	65.02	[553]
	c = 9.4020			
Co_9S_8 +		065-1765	29.54	[311]
CoS +			30.88	[222]
Co_3S_4			39.24	[331]
(4 h)			47.24	[511]
			51.80	[440]
			72.98	[731]
			76.49	[800]
		065-3418	35.06	[101]
			46.86	[102]
			54.32	[110]
		19-03673	38.35	[400]
			64.98	[533]
Co_9S_8 +		065-1765	29.61	[311]
CoS +			30.97	[222]
Co_3S_4			39.33	[331]
(6 h)			47.35	[511]
			51.87	[440]
			73.02	[731]
			76.64	[800]

		065-3418	35.07 46.82 54.28	[101] [102] [110]
		19-03673	38.34 64.97	[400] [533]
Bi_2S_3 (2 h)	a = 11.15 b = 11.3 c = 3.981	017-0320 (Bi_2S_3- Orthorhombic)	24.95 28.51 31.68 46.44 52.53	[130] [211] [221] [431] [351]
Bi_2S_3 (4 h)		017-0320	25.15 28.70 31.87 46.62 52.75	[130] [211] [221] [501] [312]
Bi_2S_3 (6 h)		017-0320	24.87 28.43 31.62 46.36 52.48	[130] [211] [221] [431] [351]

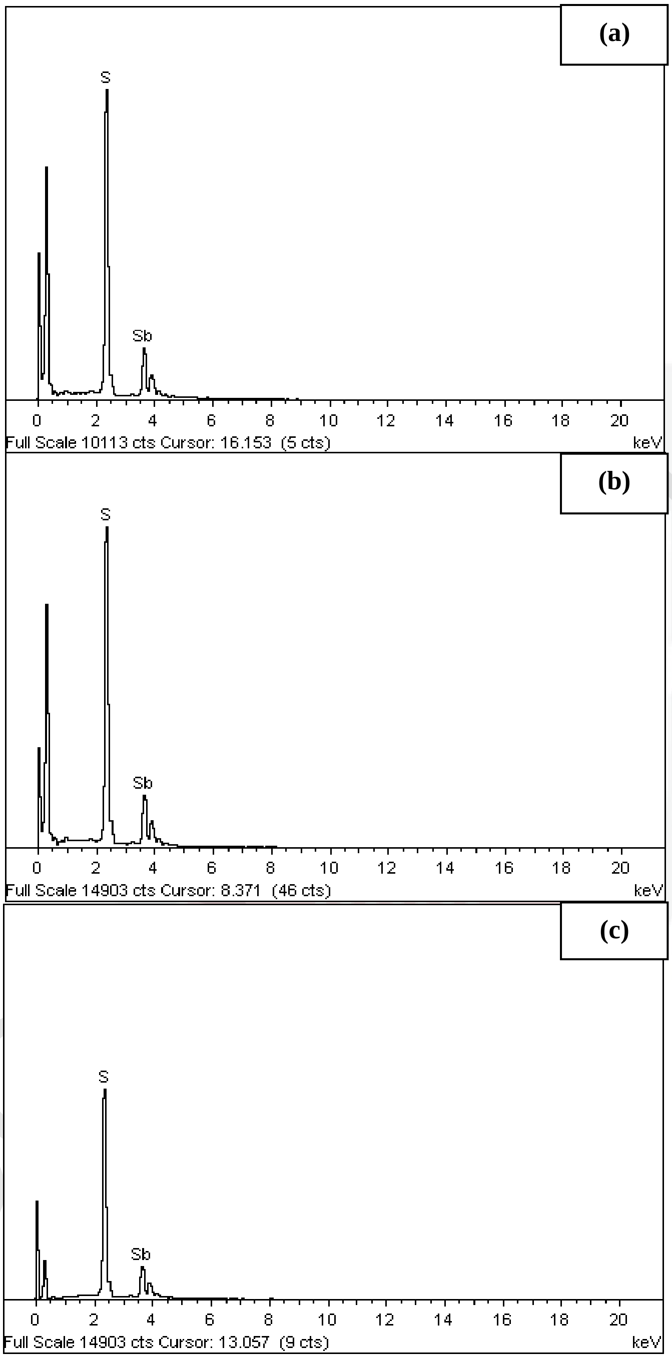
Appendix D: EDX spectra of metal sulphides



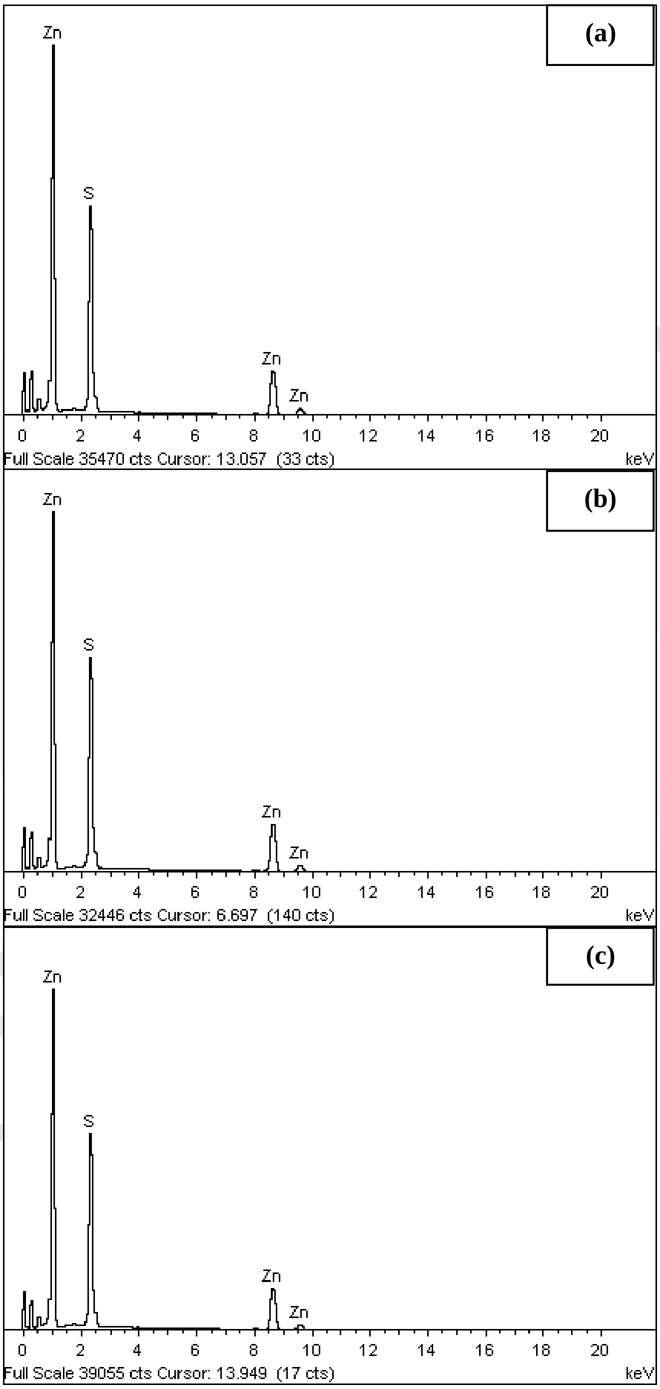
EDX Spectra of bismuth sulphide at calcination times of (a) 2 h (b) 4 h and (c) 6 h at temperature 400 °C



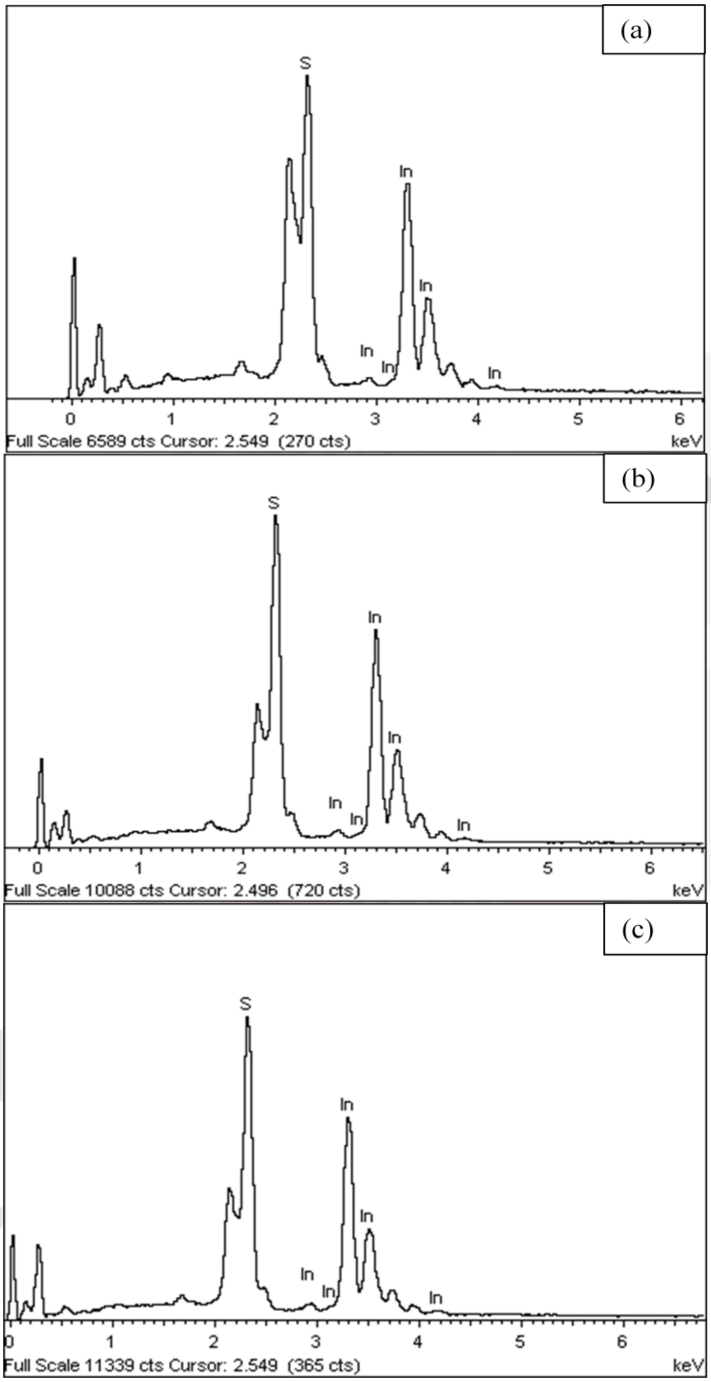
EDX Spectra of cadmium sulphide at calcination times of (a) 2 h (b) 4 h and (c) 6 h at temperature 400 °C



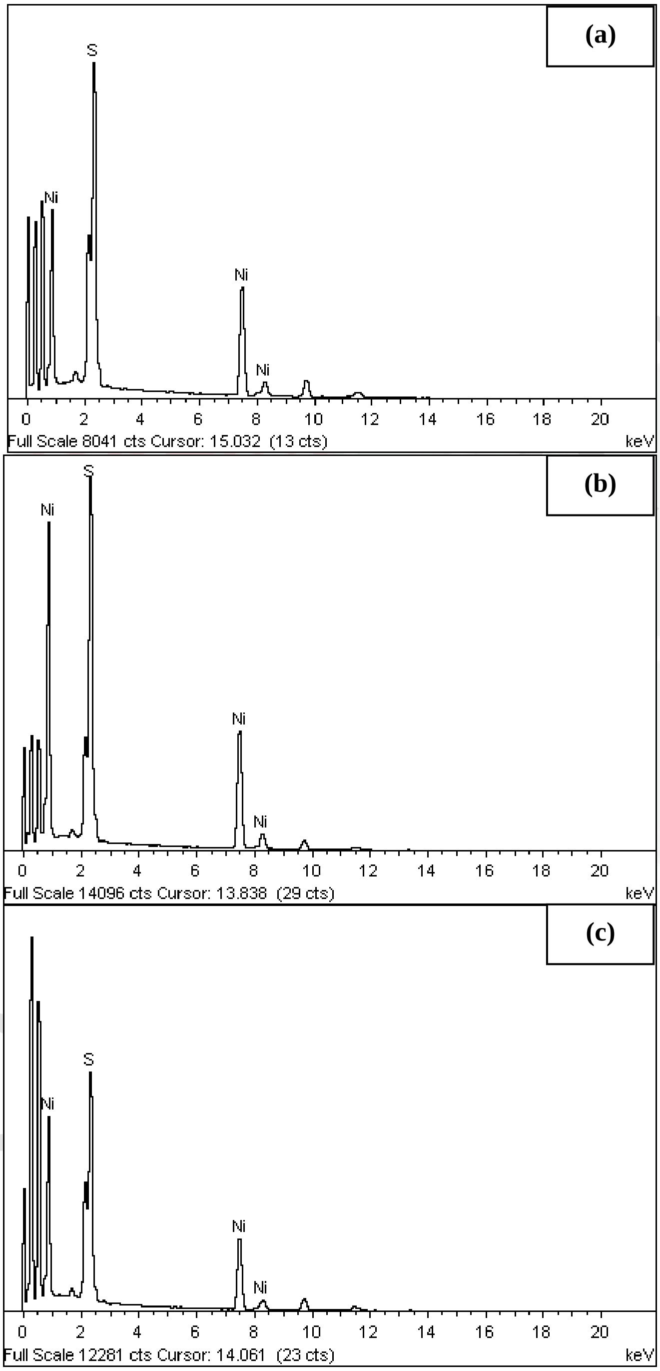
EDX Spectra of antimony sulphide at calcination times of (a) 2 h (b) 4 h and (c) 6 h at temperature 400 °C



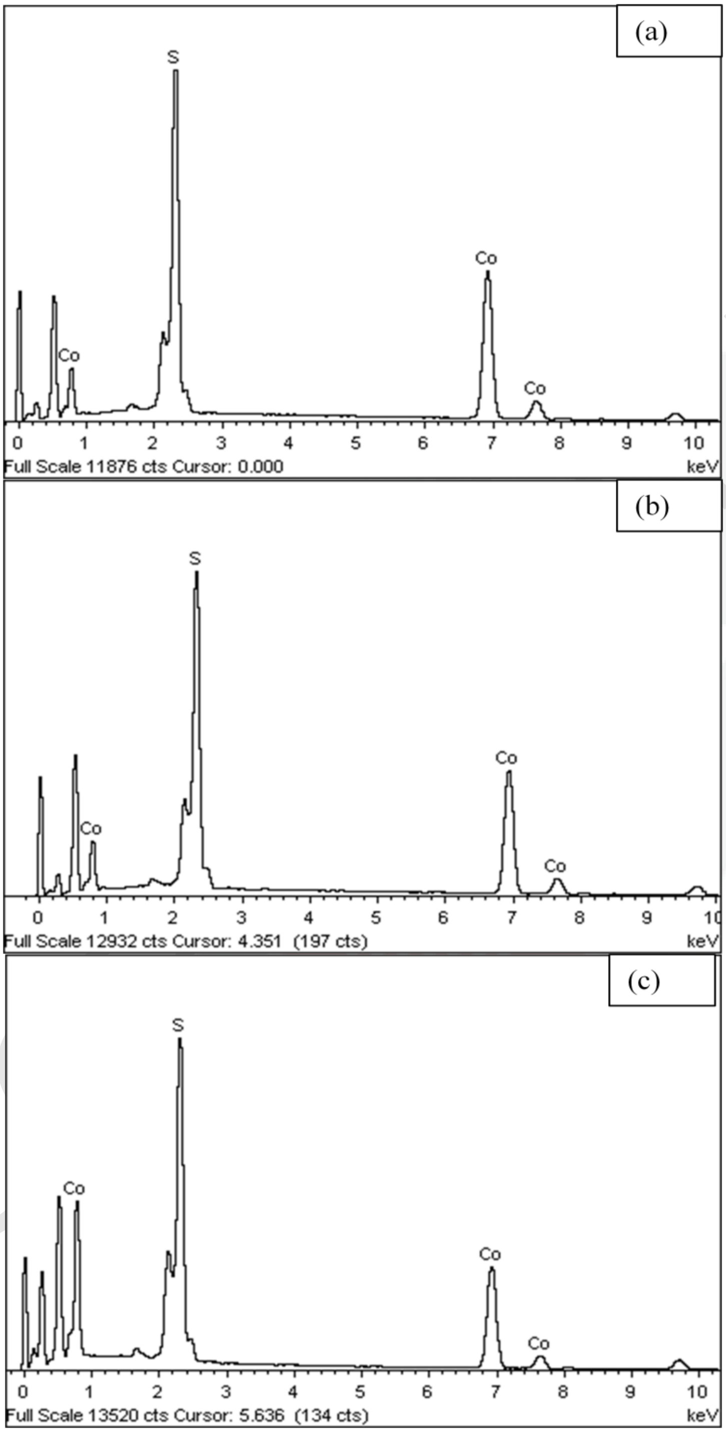
EDX Spectra of zinc sulphide at calcination times of (a) 2 h (b) 4 h and (c) 6 h at temperature 400 °C



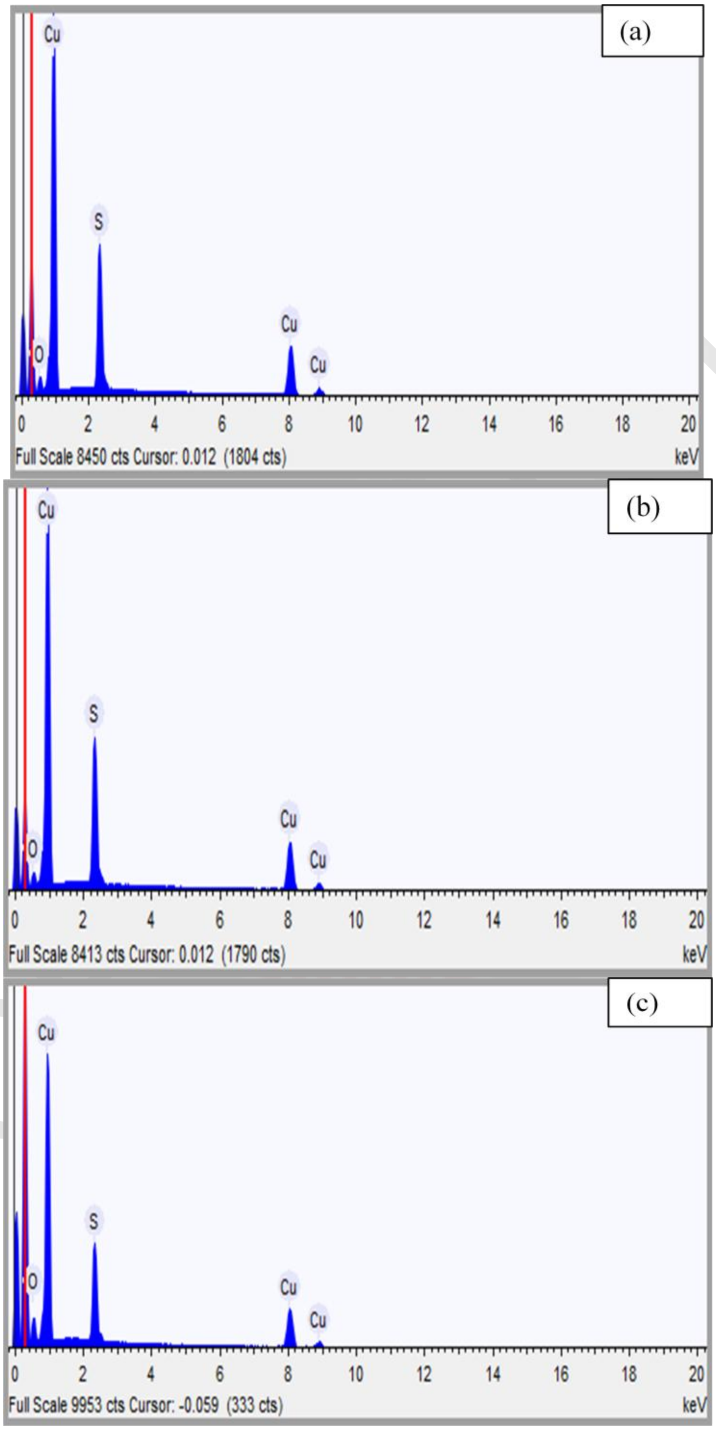
EDX Spectra of indium sulphide at calcination times of (a) 2 h (b) 4 h and (c) 6 h at temperature 400 °C



EDX Spectra of nickel sulphide at calcination times of (a) 2 h (b) 4 h and (c) 6 h at temperature 400 °C



EDX Spectra of cobalt sulphide at calcination times of (a) 2 h (b) 4 h and (c) 6 h at temperature 400 °C



EDX Spectra of copper sulphide at calcination times of (a) 2 h (b) 4 h and (c) 6 h at temperature 400 °C

BIODATA OF STUDENT

Name : Nurul Hidayah binti Abdullah
Date of Birth : 04th December 1988
Place of Birth : Dungun, Terengganu
E-mail : riahida04@yahoo.com

Education Background:

1995-2000	Sekolah Rendah Kebangsaan Balai Besar, Dungun, Terengganu.	UPSR
2001-2003	Sekolah Menengah Kebangsaan Tengku Intan Zaharah, Dungun, Terengganu.	PMR
2004-2005	Sekolah Menengah Kebangsaan Tengku Intan Zaharah, Dungun, Terengganu.	SPM
2006-2007	Kolej Matrikulasi Pahang.	MATRICULATION
2007-2010	Universiti Putra Malaysia, Serdang, Selangor.	BACHELOR OF SCIENCE (HONS.)-PETROLEUM CHEMISTRY

LIST OF PUBLICATIONS

1. N.H. Abdullah, Z. Zainal, S. Silong, M.I.M. Tahir, K.B. Tan and S.K. Chang (2016). Synthesis of zinc sulphide nanoparticles from thermal decomposition of zinc N-ethyl cyclohexyl dithiocarbamate complex. *Mater. Chem. Phys.*

List of seminars/conferences/workshops attended

1. Regional Fundamental Science Congress (FSC 2011)-Poster Presenter
2. The 24th Regional Symposium in Malaysian Analytical Sciences, 2011 (SKAM-24)-Poster Presenter
3. Fundamental Science Congress (FSC 2012)-Poster Presenter
4. 5th Fundamental Science Congress (FSC 2013)-Oral Presenter
5. Workshop on Electron Microscopy for Material Science Research, 2011-Participant
6. Workshop on Advanced Materials and Nanotechnology, 2013 (WAMN 2013)-Participant