

## Star-shaped Thiourea Derivative as Single Molecule Conductive Thin Film

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### Authors' contributions

This work was carried out in collaboration between all authors. The corresponding author WMK proposed the overall idea, concept and designed the molecular framework of the ideal molecule for this study. Author CWY carried out the synthesis, analyses, managed the literature searches and performed the statistical analysis. As co-researcher, author HS verified the performance and behavior of the conductive film. Lastly, author RR refined the spectroscopic and analytical data who prior involved in the product isolation as well as carried out further task on data analysis. All authors read and approved the final manuscript.

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### ABSTRACT

The electronic delocalization in extended  $\pi$ -orbital system of conjugated organic molecules brings significant contribution and has lead for further investigation to exploit this system to be used as potential conductive thin film. In this work, a star-shaped thiourea,  $N^1, N^3, N^5$ -tris(4-(nonyloxy)phenyl)- $N$ -(benzene-1,3,5 tricarboxyl) thiourea (**STU**) based on Donor(D)- $\pi$ -Acceptor(A) system was successfully synthesized from continuous reactions of intermediate compound 4-nonyloxy aniline hydrochloride with benzene-1,3,5-tricarboxylic acid chloride prior to form active substrate in the form of film. These compounds were characterized via several spectroscopic and analytical methods namely IR, UV-Vis, CHNS elemental analysis,  $^1\text{H}$  and  $^{13}\text{C}$  NMR as well TGA

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analysis. The investigation of its potential as dopant system in conductive film was fabricated on ITO substrate prior to the evaluation of its conductivity properties which was carried out by Four Point Probe. The findings from the conductivity analysis revealed that the prepared film performed better and exhibited increasing conductivity up to  $0.1066 \text{Scm}^{-1}$  under maximum light intensity of  $50 \text{Wm}^{-2}$ . Thus, this proposed molecular structure of A-C(O)NHC=SNHAr-D has given ideal indication to act as conductive film and has opened wide potential for various applications particularly in molecular electronic devices.

**Keywords:** Thiourea; conductive film; dopant system.

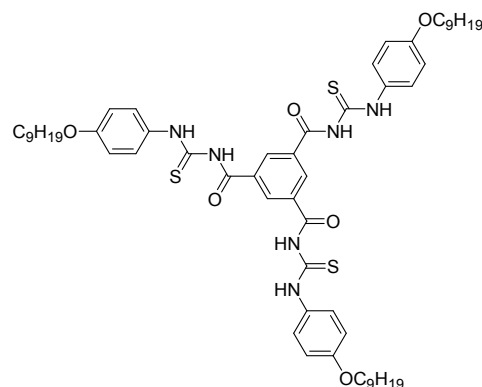
## 1. INTRODUCTION

In recent years, development of thin film technology has attracted a great deal of attention from researchers in the field of molecular electronic [1-3]. In these respects, variety of techniques have been developed for thin film deposition process via Chemical Bath Deposition (CBD), successive ionic layer absorption and reaction (SILAR), sol-gel process and spray pyrolysis to improve device performances [4-6]. Among such techniques, spin coating becomes the premier choice for thin film fabrication since this technique gives many advantages such as having low cost to produce, simple fabrication technique and showing capability of low-temperature processes [7-9]. The preparation of molecule-based conductors in a practical usable form of thin film is a challenging subject to be applied in any electronic devices [10]. Conductive film based on single organic molecule as active elements has become potentially promising alternatives over readily available conventional developed systems [11-13]. This is due to the fact that this type of compound offers unique properties mainly due to it contains fair number of alternating single and double or triple bonds which the electron can easily flow from one reservoir to another [14-16]. However, the quest to design the ideal molecular systems that possess high conjugated system to move along the molecular system is rather challenging and vital in order for these molecules to act as active materials in molecular wire applications [17,18]. Thus, the choice of ideal materials to perform the function at an optimum level is very important.

Recently, the versatility of conjugated thiourea system has attracted great attention as a potential molecular framework to be applied as conducting film due to its unique coordination electrons lone pairs of N, S and O atoms which involve in the resonance that lead to electrical conductivity properties [19-22]. Thus, conjugated thiourea derivative was used in this study as focalpoint compound in the respect of thiourea

moiety in general have proven to show many advantageous properties such as ease synthetic work-out, unique photophysical and coordination to name a few.

In previous occasions, we have introduced mono-substituted linear [12,13] and 'V'-shaped systems [21,22] of thiourea derivatives for potential molecular wire candidates with indicated promising conductivity in high intensity of light which worked at an utmost performance including in a dark condition. However, the report on organic materials and investigation on suitable fabrication methods are rather limited. Thus, in the light of these previous works, the performance of unique molecular framework based on Donor (D)- $\pi$ -Acceptor (A) system featuring star-shaped thiourea was carried out prior to form conductive film as shown in Fig.1. This present work involved the design, characterization as well as electrical properties of the targeted molecule, **STU**. Additionally, the characterization tasks were extensively studied and modified with the goal of making **STU** potentially useful as active layer in conductive film.



**Fig. 1. Molecular structure of  $N^1, N^3, N^5$ -tris(4-(nonyloxy)phenyl)- $N^2$ -(benzene-1,3,5-tricarbonyl) thiourea (STU)**

## 2. EXPERIMENTAL DETAILS

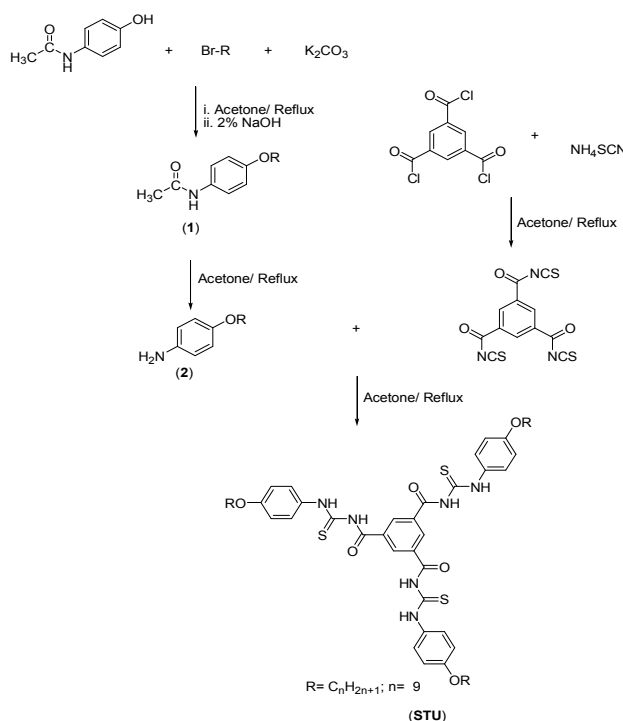
### 2.1 Material and Instrumentation

All reactions were carried out under an ambient atmosphere and no special procedures were taken to exclude air or moisture during work-up. The chemicals and reagents were purchased commercially from Merck, Fisher Scientific and R & M Chemicals. They were used as received without further purification at any time during experimental work. The infrared (IR) spectrum was recorded on a Fourier Transform-Infrared Spectrometer, Perkin Elmer Spectrum 100 in the range of 4000-400  $\text{cm}^{-1}$  by using potassium bromide (KBr) pellets. For UV-Vis analysis, all compounds including the precursor were recorded using UV-Vis Spectrophotometer Shimadzu UV-1601 PC using quartz cuvette cell and methanol was chosen as solvent. The NMR spectra of  $^1\text{H}$  (400.11MHz) and  $^{13}\text{C}$  (100.61MHz) NMR were recorded using Bruker Avance III 400 spectrometer with deuterated chloroform ( $\text{CDCl}_3$ ) as solvent and chemical shift values ( $\delta_{\text{H}}/\delta_{\text{C}}$ ) were given in parts per million (ppm) relative to solvent resonance as internal standard at room temperature respectively. Besides that, thermogravimetric analysis was performed using Perkin-Elmer TGA Analyzer from 30°C to 700°C at a heating rate of 10°/min under nitrogen

atmosphere. The experimental percentage of C, H, N and S elements of the synthesized compounds were determined by using CHNS Analyzer Flash EA 1112 series. Afterwards, **STU**/ITO thin film was deposited via spin coating technique by using Spin Coater Model WS-400B-6NPP-LITE. The electrical conductivity of the film was measured under various intensity of light by using Four Point Probe.

### 2.2 General Overview of Synthetic Work

For synthetic work, the synthesis of **STU** involved several continuous reactions which began with formation of the precursor, *N*-(4-(nonyloxy)phenyl) acetamide (**1**) and from this, was continued with the formation of 4-nonyloxy aniline (**2**) as an intermediate. Then **2** was reacted with benzene-1,3,5-tricarboxylic acid chloride to obtain the targeted compound (**STU**). However, synthetic work-up and characterization of **1** and **2** have been reported in previous occasions [12,13,23,24]. Therefore, some modifications in synthetic work and further characterization on the spectroscopic and analytical tasks were carried out for the interest of this report and are discussed further. The synthetic approach of **STU** featuring thiourea moiety is summarized in Scheme 1.



Scheme 1. General overview of synthetic work

### 2.2.1 Synthesis of *N*-(4-(nonyloxy)phenyl)acetamide (**1**)

The precursor, **1** was prepared from an equimolar reaction between *N*-(4-hydroxyphenyl)acetamide (10.0 g, 66.2 mmol), nonyl bromide (12.6 ml, 66.2 mmol) and potassium carbonate (9.2 g, 66.2 mmol) via Williamson ether synthesis. The reaction was carried out at reflux temperature with continuous stirring for ca. 48 hours to give light brown solution and white precipitate. When adjudged completion by Thin Layer Chromatography (TLC) using eluent system of (hexane:ethyl acetate) (3:2), the solution was taken to dryness to afford cream-like solid. The solid obtained was stirred with 2% of sodium hydroxide solution for ca. 1 hour. Then, the mixture was filtered and rinsed with little amount of diethyl ether to afford white solid of **1**. IR (KBr):  $\nu(\text{N-H})$  3292 $\text{cm}^{-1}$ ,  $\nu(\text{C-H})$  2851 $\text{cm}^{-1}$ ,  $\nu(\text{C=O})$  1660 $\text{cm}^{-1}$ ,  $\nu(\text{C-N})$  1237 $\text{cm}^{-1}$ ,  $\nu(\text{C-O})$  1174 $\text{cm}^{-1}$ .

### 2.2.2 Synthesis of 4-nonyloxy aniline (**2**)

White solid of **1** (6.4 g, 66.2 mol) was put at reflux in a mixture of concentrated hydrochloric acid (50 ml) and ethanol (50 ml) for ca. 2 hours with continuous stirring to afford brown solution of 4-nonyloxy aniline hydrochloride. This solution was cooled at room temperature prior to extraction of the organic layer with 150 ml of water and 150 ml of dichloromethane. The organic layer was separated and dried over anhydrous  $\text{MgSO}_4$  to afford off-white colour of **2** after the solvent was removed *in vacuo*. IR (KBr):  $\nu(\text{N-H})$  3430 $\text{cm}^{-1}$ ,  $\nu(\text{C-H})$  2919 $\text{cm}^{-1}$ ,  $\nu(\text{C-N})$  1251 $\text{cm}^{-1}$ ,  $\nu(\text{C-O})$  1169 $\text{cm}^{-1}$ .

### 2.2.3 Synthesis of *N*<sup>1</sup>,*N*<sup>3</sup>,*N*<sup>5</sup>-tris(4-(nonyloxy)phenyl)-*N*<sup>4</sup>-(benzene-1,3,5-tricarbonyl) thiourea (STU)

A suspension of benzene-1,3,5-tricarboxylic acid chloride (0.1 ml, 85 mmol) in 50 ml acetone was added with ammonium thiocyanate (0.2 g, 2.5 mmol) in 50 ml acetone to give white solution. The solution was stirred at room temperature for ca. 2 hours before **2** (0.5 g, 2.5 mmol) was added. After stirring for another ca. 2 hours, the colour of the solution turned from white to pale yellow. After adjudged completion by TLC (hexane:ethyl acetate) (3:2), the reaction mixture was filtered and the filtrate was left to cool at room temperature. The yellow filtrate was added with 3 ice cubes and filtered to obtain yellow precipitate. The yellow precipitate was then

recrystallized from acetonitrile to afford the title compound **STU** (0.8 g, 61% yield).  $\text{C}_{57}\text{H}_{78}\text{N}_6\text{O}_6\text{S}_3$  requires: C, 65.32; H, 8.68; N, 8.46; S, 10.28. Found: C, 65.86; H, 7.57; N, 8.09; S, 9.23%. IR (KBr):  $\nu(\text{N-H})$  3435  $\text{cm}^{-1}$ ,  $\nu(\text{C-H})$  2922  $\text{cm}^{-1}$ ,  $\nu(\text{C=O})$  1646 $\text{cm}^{-1}$ ,  $\nu(\text{C-N})$  1245 $\text{cm}^{-1}$ ,  $\nu(\text{C-O})$  1173  $\text{cm}^{-1}$ ,  $\nu(\text{C=S})$  718  $\text{cm}^{-1}$ .  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 400.11 MHz):  $\delta$  0.86 (t,  $^3J_{\text{HH}} = 7\text{Hz}$ , 9H,  $3\times\text{CH}_3$ ); 1.27-1.74 (m, 42H,  $21\times\text{CH}_2$ ); 3.97 (s, 6H,  $\text{OCH}_2$ ); 6.93-6.98 (pseudo-d,  $^3J_{\text{HH}} = 9\text{Hz}$ , 6H,  $3\times\text{C}_6\text{H}_4$ ); 7.57-7.73 (pseudo-d,  $^3J_{\text{HH}} = 9\text{Hz}$ , 6H,  $3\times\text{C}_6\text{H}_4$ ); 8.51-8.73 (m, 3xH, Ar- $\text{C}_6\text{H}_3$ ); 10.32, 11.89 (2 x s, 1H, NH).  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 100.61 MHz):  $\delta$  14.41 ( $\text{CH}_3$ ); 22.55, 25.99, 29.11, 29.19, 29.25, 29.43, 31.73 (6 x  $\text{CH}_2$ ); 68.29 ( $\text{CH}_2\text{O}$ ); 114.86, 122.42, 122.52, 126.48, 131.13, 132.36, 135.26 (Ar- $\text{C}_6\text{H}_4$ ); 169.10 (C=O); 178.58 (C=S).

### 2.3 Fabrication of Thiourea Thin Film on ITO Substrate

Next, **STU** was deposited on the ITO substrate by using a spin coating technique via Spin Coater Model WS-400B-6NPP-LITE. The process undergone 6 stages of spin (500 rpm for 30 seconds, 1000 rpm for 15 seconds, 1500 rpm for 20 seconds, 2000 rpm for 30 seconds, 4000 rpm for 15 seconds and 5000 rpm for 15 seconds) to complete one cycle. Then, the solution was deposited again onto the substrate and was spin again. The process was repeated the same way to finish the 6 cycles for each sample. Fig. 2 shows the general illustration of layer of arrangement in the film prepared by using the spin coating method.

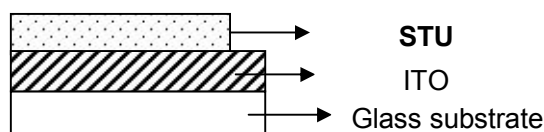


Fig. 2. The general illustration of arrangement layer thin film

### 2.4 Electrical Conductivity of Thin Film Under Various Intensity of Light

For electrical conductivity characterization, the sheet resistivity in produced films was measured by using four point probing system consists of the Jandel Universal Probe combined with a Jandel RM3 Test Unit according to the method proposed by Smits [25]. The electrical conductivity of thin film was measured in the dark and light condition with light intensity of  $10\text{ Wm}^{-2}$

to  $50 \text{ Wm}^{-2}$  by using Four Point Probe and LI-200 Pyranometer Sensor with LI-1400 Data Logger. The conductivity of the film can be calculated using the following equation [26] as shown below:

Sheet resistance for films;

$$R_s = 4.532 \times V / I \quad \text{Equation 1}$$

Where,  $R_s$  is the sheet resistance (resistivity) which relies on a geometric factor  $k$  (4.532),  $V$  is the voltage measured and  $I$  is the current applied from the test unit. Thus, electrical conductivity can be determined which it is the reciprocal (inverse) of the electrical resistivity,  $\sigma$  as shown in Equation 2.

$$\sigma = 1/R_s \quad \text{Equation 2}$$

Where,  $\sigma$  = electrical conductivity and  $R_s$  = sheet resistance (resistivity)

### 3. RESULTS AND DISCUSSION

#### 3.1 Spectroscopic Studies

The infrared spectrum of **STU** revealed six absorption bands of interest namely  $\nu(\text{N-H})$ ,  $\nu(\text{C-H})$ ,  $\nu(\text{C=O})$ ,  $\nu(\text{C-N})$ ,  $\nu(\text{C-O})$  and  $\nu(\text{C=S})$ . The band at  $3435 \text{ cm}^{-1}$  represents a stretching vibration for secondary thioamide (N-H) which believed to be the existence of intramolecular hydrogen bonding [27-29]. Strong absorption band of  $\nu(\text{C=O})$  was assigned at  $1671 \text{ cm}^{-1}$  that related to the resonance effect with the fused-aromatic and phenyl ring [30,31]. Meanwhile, the presence of  $\nu(\text{C=S})$  stretching vibration at low frequency around  $718 \text{ cm}^{-1}$  as reported previously was due to the effect of less double bond character of the sulphur atom and existence of intramolecular hydrogen bonding with N-H in the molecular framework [32,33].

The electronic transitions analysis of **STU** showed the existence of two main strong chromophores of carbonyl (C=O) and thiocarbonyl (C=S) which can be observed at  $\lambda_{\text{max}}$  228 nm and 279 nm. The absorption band of C=O chromophore can be identified at  $\lambda_{\text{max}}$  228 nm due to the effect of  $\pi$ -conjugation of carbonyl with the phenyl group, thus the transition of  $\pi \rightarrow \pi^*$  shifted to longer wavelength. Strong absorption

band centered at  $\lambda_{\text{max}}$  279 nm can be attributed to  $\pi \rightarrow \pi^*$  and  $n \rightarrow \pi^*$  transitions of C=S and phenyl in thiourea moiety. The broad absorption band observed in the region was due to  $\pi$ -conjugation of this compound with the phenyl rings ( $\pi - \pi^*$  transition) and orbitals overlapping between C=O and C=S.

$^1\text{H}$  NMR spectrum of **STU** showed methyl resonance at  $\delta_{\text{H}}$  0.86 ppm while protons for alkyl group were detected in the range of  $\delta_{\text{H}}$  1.27-1.74 ppm. For R-O-CH<sub>2</sub>, the signals of proton were identified at  $\delta_{\text{H}}$  3.97 ppm because the protons on carbon attached to the oxygen was deshielded due to high electronegativity of the oxygen [12,13,21]. At higher chemical shift, aromatic protons of the molecule can be found in between  $\delta_{\text{H}}$  6.93-8.73 ppm which showed pseudo-doublet resonances of the system and they were strongly affected by *para*-substitution of the aromatic rings [22,34]. Protons for N-H were observed as two singlet resonances in two different chemical environment due presence of two N-H groups in this compound which can be observed at  $\delta_{\text{H}}$  10.32 ppm and  $\delta_{\text{H}}$  11.89 ppm respectively.

As expected,  $^{13}\text{C}$  NMR spectral analysis showed methyl group resonance which can be clearly observed at  $\delta_{\text{C}}$  14.41 ppm and carbon resonances for alkyl group were detected in the range of  $\delta_{\text{C}}$  22.56-31.73 ppm. While the chemical shift for R-CH<sub>2</sub>-O- can be found at  $\delta_{\text{C}}$  68.16 ppm due to deshielding effect in presence of oxygen atom that withdrew certain amount of electrons from the alkyl chain [12,27]. Meanwhile, the resonance for both aromatic ring carbons were assigned in the range of  $\delta_{\text{C}}$  114.86-135.26 ppm where corresponded to four phenyl rings in the compounds. The C=O and C=S resonances which were slightly deshielded which can be observed at  $\delta_{\text{C}}$  164.73 ppm and  $\delta_{\text{C}}$  179.42 ppm respectively. The formation of intra-molecular hydrogen bonding in the compound and the increasing electronegativity of oxygen and sulphur have caused the deshielding effect to higher chemical shifts [35,36].

#### 3.2 Thermal Analysis

Thermal stability of **STU** was evaluated by using thermogravimetric analysis (TGA) at heating rate  $10^\circ\text{C}/\text{min}$  under nitrogen atmosphere with temperature range of  $30\text{--}700^\circ\text{C}$ . The thermogravimetric curves of **STU** is depicted in Fig. 3.

The degradation process of **STU** showed three-stage degradation pattern of major mass loss. Initial degradation took place at around 200°C ( $T_{onset}$ ) and ended at around 250°C ( $T_{offset}$ ) while second stage started to degrade at 280°C ( $T_{onset}$ ) and ended at 320°C ( $T_{offset}$ ). During the decomposition of these stages, the total mass loss was about 65%. Afterwards, it followed by other mass loss of 35% in third stage which it began to degrade at around 365°C ( $T_{onset}$ ) and ended at 420°C ( $T_{offset}$ ). The thermal stability of **STU** increased as the temperature of degradation increased and it was due to the number of aliphatic carbon chains and overlapping orbitals between C=S and C=O. Therefore, it can be summarized that **STU** exhibited thermal stability at high temperature and it gave good indication for the potential of fabrication of thin film by showing remarkable performance under prolonged thermal stress.

### 3.3 Electrical Conductivity of Thin Film under Various Intensity of Light

In order to investigate its conductivity in various conditions, as shown in Fig. 4, the electrical conductivity (EC) study of **STU** in thin film form coated on the ITO glass was measured under various intensity of light conditions ( $Wm^{-2}$ ) by using Four-Point Probe. It showed the electrical conductivity increased by the increasing amount of light intensities. The highest conductivity was observed for **STU** under maximum light intensity ( $50 Wm^{-2}$ ) with the conductivity value,  $0.1066 Scm^{-1}$  that could be explained by its molecular weight and the conjugation length. In this case, proliferative performance of conductivity caused by the introduction of star-shaped thiourea was due its unique properties, in which the system possesses an extensive conjugated double bonds in its molecular framework.

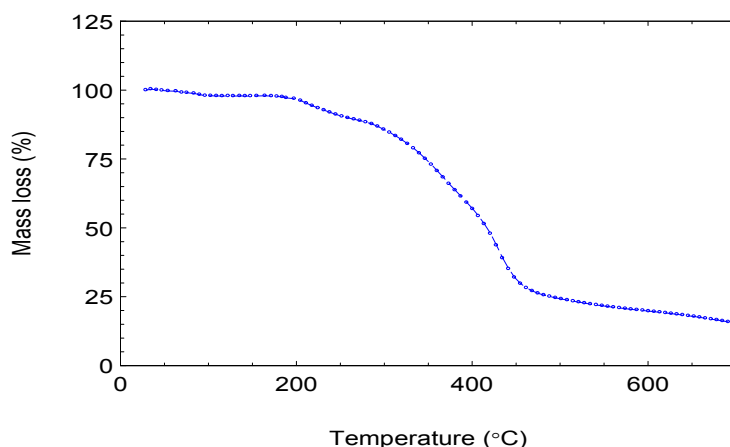


Fig. 3. TGA thermogram of STU

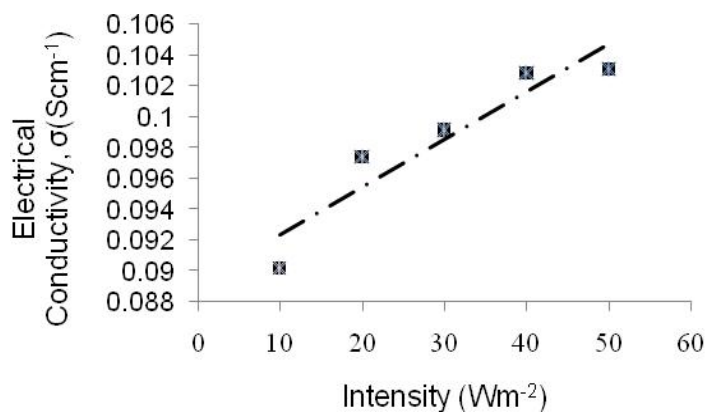


Fig. 4. Electrical conductivity values under various intensity of light

As a conclusion, the electrical conductivity analysis showed positive results under various intensity of light which revealed the film was successfully coated on the ITO substrate. Thus, according to the conductivity result, **STU** or perhaps similar type of molecule featuring thiourea system can be applied as an active layer in conductive materials for the interest of advanced material applications.

#### 4. CONCLUSION

The performance of single molecule compound of star-shaped thiourea derivative of  $N^1, N^3, N^5$ -tris(4-(nonyloxy)phenyl)- $N^2$ -(benzene-1,3,5-tricarbonyl) thiourea (**STU**) has been successfully synthesized and characterized as dopant system in conductive film. All synthesized compounds including precursors were spectroscopically and analytically characterized by typical selected  $^1\text{H}$  and  $^{13}\text{C}$  NMR, FT-IR, UV-Vis spectroscopic analysis. From the electrical study, the layer of **STU**/ITO thin film exhibited higher conductivity under maximum light intensity up to  $0.1066 \text{ Scm}^{-1}$  under maximum light intensity of  $50 \text{ Wm}^{-2}$ . Therefore, these findings have shown great and possible opportunities for any related future research in this area particularly in microelectronic applications.

#### COMPETING INTERESTS

Authors have declared that no competing interests exist.

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