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Dr Charles W. Spangler

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This program has, as its stated goal, the design of the first organic polymeric superconductor. In year one we have embarked on the design of several model compounds so as to ascertain the most appropriate synthetic methodology for the polymer systems, and to determine the existence of any potential problems in solubility and processibility parameters in the chosen systems. Upon synthesis the redox properties of the model compounds and their compatibility with various counter ions will be determined, as well as their packing efficiency via x-ray spectroscopy.

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Dr Charles Y-C. Lee	(202) 767-5021	NL

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Interim Technical Report

Prepared for: Air Force Office of Scientific Research

Title: "Design Strategies for the Preparation of Polymeric Organic Superconductors"

Grant Number: F49620-92-J-0533 (FY92 URI/RIP)

Grant Period: September 30, 1992 - September 29, 1995

Principal Investigator: Professor Charles W. Spangler

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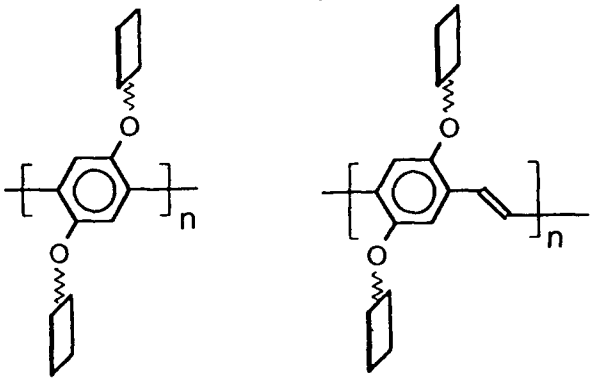
Introduction

This program has, as its stated goal, the design of the first organic polymeric superconductor. In year one we have embarked on the design of several model compounds so as to ascertain the most appropriate synthetic methodology for the polymer systems, and to determine the existence of any potential problems in solubility and processibility parameters in the chosen systems. Upon synthesis the redox properties of the model compounds and their compatibility with various counter ions will be determined, as well as their packing efficiency via x-ray spectroscopy.

Synthesis of Model Compounds

In collaboration with Professor John Reynolds of the University of Florida, we determined that the following model compounds would be synthesized at Northern Illinois University during the first year to mimic poly (p-phenylene) and poly (p-phenylene vinylene) with TTF and ET pendant groups:



Our intent is to synthesize the monomer, dimer and trimers with hydrogen end caps and variable length methylene spacers separating the TTF and ET moieties from the rigid rod polymer backbone: $-(CH_2)_n-$; n = 1,2,3 ---. The synthesis of the first ET molecule with pendant attachment group and a 3-carbon spacer has recently been accomplished and is outlined in schemes 1 and 2. We are currently hydrolyzing compound 7 to the free alcohol, and are attempting to couple it to the hydroquinene dianion. An alternative synthetic approach to this model compound utilizing an "inside-out" strategy is also being studied, and is in the final stage involving coupling of compound 10 to compound 4 by established reaction protocol via asymmetric coupling with $P(OMe)_3$. Both approaches can be carried out in good yield. When the final product has been purified, its' redox properties and electrocrystallization will be studied in collaboration with Professor Reynolds.

The synthesis of the first PPV model compound will also utilize the derivstized ET compound 10, except that it will be coupled to bromo-hydroquinene, and the subsequent product coupled with Bu_3Sn CH = CH $SnBu_3$ via Stille coupling. We hope to complete both the final synthesis and electrocrystallization of these materials during year 2.

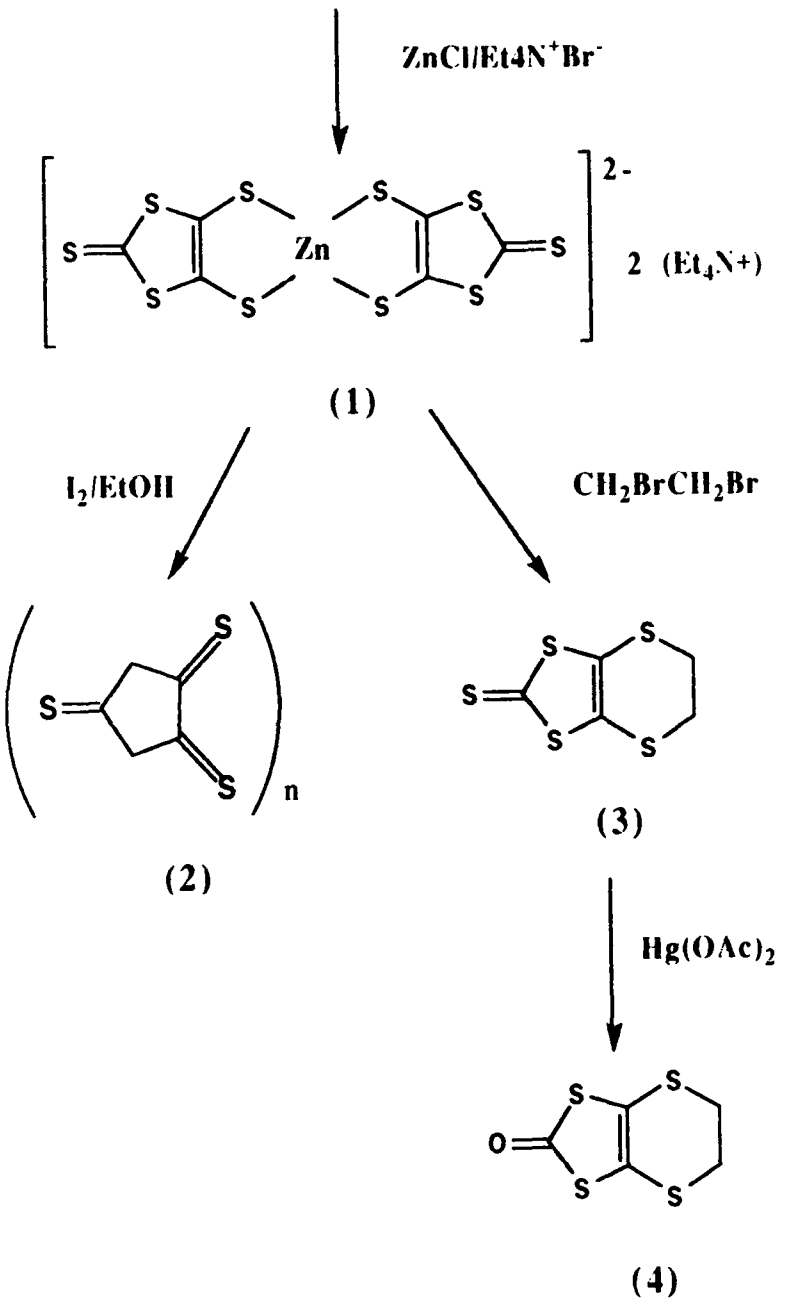
Personnel

The following postdoctoral students worked on this project during the year.

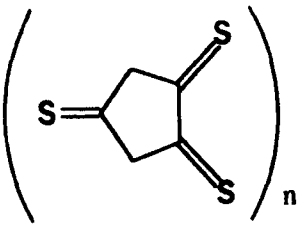
Dr. Eric Nickel
Dr. Pei-Kang Liu
Dr. Tom Hall
Dr. LinFang Zhu

Drs. Nickel and Liu only worked a few months each before resigning to accept permanent positions.

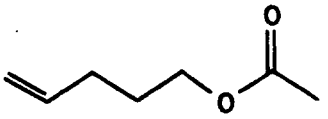
Scheme 1

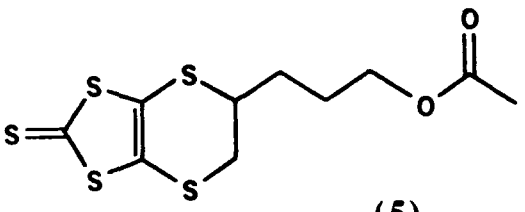

Na/CS2/DMF
ZnCl/Et4N+Br-
Zn
2-
2 (Et4N+)
(1)
I2/EtOH
CH2BrCH2Br
n
(2)
(3)
Hg(OAc)2
O
(4)

Scheme 2



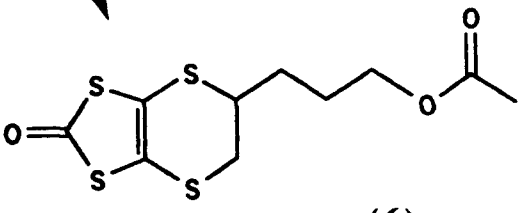
(2)





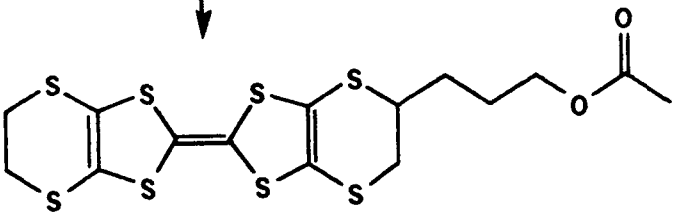
(5)

$Hg(OAc)_2$

(4) + 

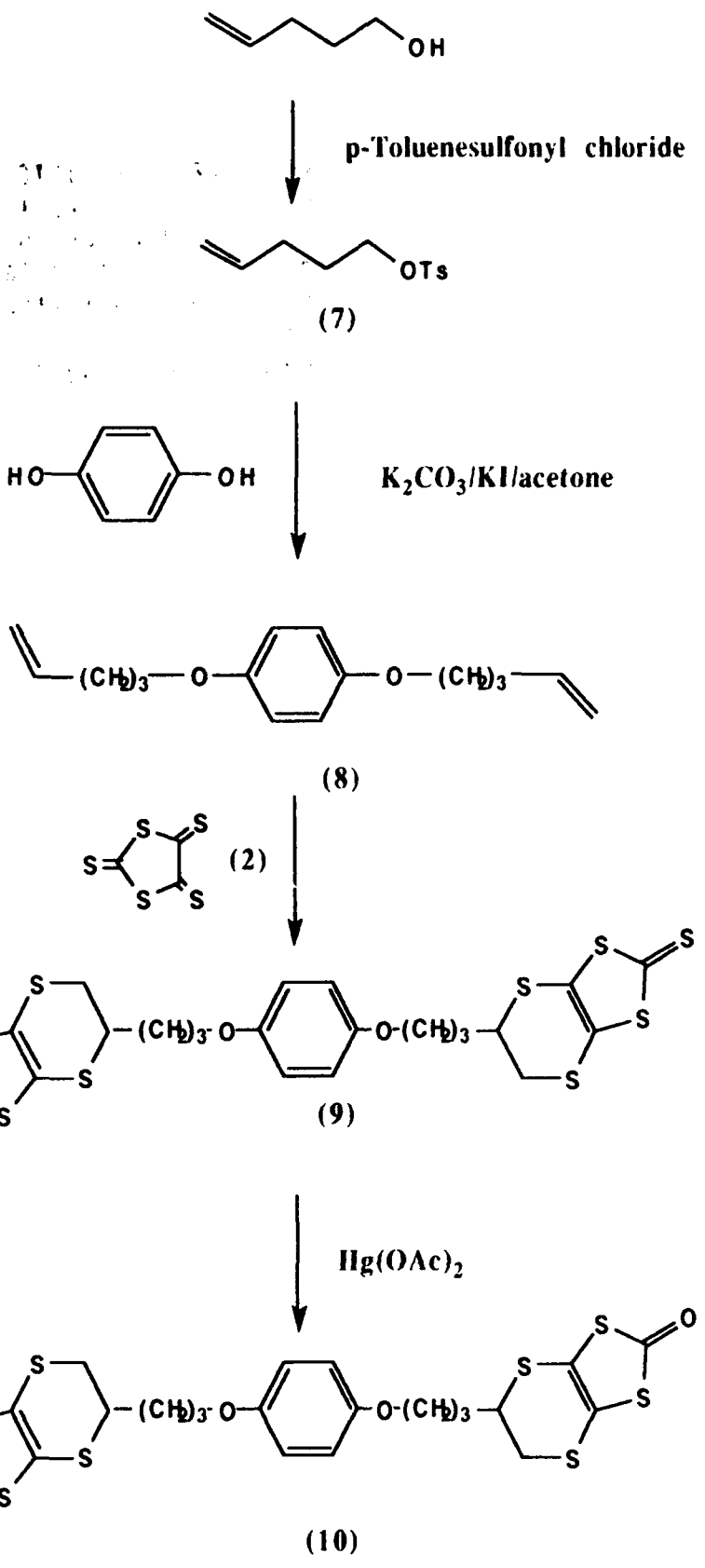
(6)

$P(OEt)_3$



(7)

Scheme 3


OH
p-Toluenesulfonyl chloride
OTs
(7)
HO
OH
K_2CO_3/KI/acetone
(8)
(2)
(9)
$Hg(OAc)_2$
(10)