



AFRL-OSR-VA-TR-2014-0014

(HBCU) - Incorporation of FT-NMR into Research
Infrastructure and Chemistry Curriculum at Bowie State
University

ALAN ANDERSON

BOWIE STATE UNIVERSITY

01/09/2014
Final Report

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FINAL AFOSR TECHNICAL REPORT

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Air Force of Scientific Research
Award# FA9550-12-1-0448

(HBCU) Incorporation of FT-NMR into Research Infrastructure and Chemistry Curriculum at Bowie State University

Start Date: September 1, 2012
End Date: September 30, 2013

Amount: \$154,000

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14. ABSTRACT The grant award was used to purchase and install an Anasazi 90-MHz Fourier Transform NMR to bring NMR capability, a cornerstone technique for organic chemists, to the Department of Natural Science which previously did not have NMR capability. The Eft FT NMR software system consist of two programs: WinPNMR, a data acquisition program and NUTS (Acorn NMR Inc.) A NMR Data Processing program for the processing of 1D and 2D experiments including COSY, DEPT, HETCOR, T1, T2 and kinetics. In addition to H1 probes, other Nuclei accessories were purchased allowing for the acquisition of C13, F19, and P31 to Si29 data. The award has broadly impacted research, research productivity and capabilities, and education in the chemical sciences at Bowie State University, a Historically Black College and University and Primarily Undergraduate Institution.												
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Contents

1. Introduction	2
2. Equipment Purchased	3
3. Installation and Warranty	6
4. Accomplishments	7
4.1. Research.....	7
4.2. Undergraduate Education	7
5. Examples of NMR spectra using N-propyl benzoate as sample.	8
6. HONORS and AWARDS	11
7. Publications (9/01/12- 9/30/13).....	11

1. Introduction

The grant award was used to purchase and install an Anasazi 90-MHz Fourier Transform NMR to bring NMR capability, a cornerstone technique for organic chemists, to the Department of Natural Science which previously did not have NMR capability. The Anasazi instrument was selected after an examination of several NMR manufacturers including Varian Inc. and Bruker Corporation. The Anasazi 90-MHz NMR was chosen as ideal for the following reasons: 1) The system's resolution, sensitivity and capability are adequate for the current related research underway by the PI, 2) The use of permanent magnets makes the system easy to maintain and does not require costly cryogenic liquids and 3) The system has a user-friendly software interface well-suited for undergraduate research education. The Eft FT NMR software system consist of two programs: WinPNMR, a data acquisition program and NUTS (Acorn NMR Inc.) A NMR Data Processing program for the processing of 1D and 2D experiments including COSY, DEPT, HETCOR, T1, T2 and kinetics. In addition to H1 probes, other Nuclei accessories were purchased allowing for the acquisition of C13, F19, and P31 to Si29 data.

The system was ordered in December 2012 and arrived in late August 2013. Anasazi personnel set up the system and console on-site with the assistance of an electrician contracted by the University. Several students were also extensively trained by Anasazi personnel along with the PI in the use and maintenance of the equipment. The NMR was in routine use by the PI and student users within a couple of weeks.

The Anasazi 90-MHz Fourier Transform NMR has served to increase the research productivity, capabilities, and chemical sciences education of Bowie State University, a Historically Black College and University and Primarily Undergraduate Institution. The NMR will be integrated into the upper-level chemistry CHEM 202: Organic Chemistry II class in the near future.

2. Equipment Purchased

- I. **Eft-GENII** : The basic proton only EFT90 Fourier Transform NMR includes ¹H observation at 90 MHz. It uses an Anasazi 90MHz NMR magnet system, probe and shims. The basic EFT90 package includes:
- a. System Computer- Dual Core Pentium 4 TM 2.4 GHz PC including: Windows 7, 2GB RAM, CD-RW, dual 40GB Hard Disk, Disk, Mouse, Keyboard, network card, 15" SVGA LCD Color Monitor, and standard output ports; parallel, serial, and USB.
 - b. Analog Digital Converter- Dual channel 12Bit 100 kHz ADC with filtering, programmable (400Hz to 102.4KHz) Butterworth filter.
 - c. Software- The Eft FT NMR software operating systems consist of two NMR programs:
 - i. WinPNMR (Anasazi Instruments Inc.) - A NMR data acquisition program that includes those instrument control and display functions commonly used during instrument setup and acquisition of 1D and 2D data. The library of easily used 1D and 2D experiments includes, COSY, DEPT, HETCOR, T1, T2, and kinetics.
 - ii. NUTS (Acorn NMR Inc.) - A NMR data processing program for processing of the acquired 1D and 2D data. Included is a library of Anasazi processing Macros for 1D and 2D experiments including: COSY, DEPT HETCOR, T1, T2, and kinetics.
 - d. Pulse Programmer- The Enhanced State-Machine pulse programmer has 100ns time resolution, 32K words of program memory, 4096 program steps, 15 programmable outputs, and a 32-bit delay counter.
 - e. Rf System- Direct Digital Synthesized Rf heterodyne system with quadrature phase detection. The spectrometer frequency, transmitter output, four digital transmitter/receiver

phases, and receiver gain are under computer control.

- f. Field Control- The field offset and shim currents are controlled by 12bit digital to analog converters in the spectrometer console. Y, X, and Z field gradients can be pulsed under control of the pulse programmer.

- II. **AII H-1 Probe:** Anasazi Instruments Single Channel proton probe.
- III. **AII 30 element Shim set:** Anasazi Instruments Shim Set for use with the AII Probe.
- IV. **Hard Copy Device:** An inkjet printer is included for hardcopy printout.
- V. **Computer-control Shim System and Power Supply:** The Eft automated computer control shim system replaces the EM manual shim controls.
- VI. **Computer Style Table:** For mounting computer monitor, keyboard, mouse, and printer.
- VII. **Tesla AII90 Magnet system:** Includes magnet temperature controller and sample spin system for use with the Eft only
- VIII. **Carbon Accessory:** The carbon accessory converts the basic single channel (H1)Eft system to dual channel H1 and C13 operation.

The C13 accessory includes:

- a. Fixed frequency carbon RF observe module
- b. C/H 5mm ambient temperature probe with H1 decoupling.
- c. Broadband H1 decoupler.

- d. H1/C13 switching hardware, computer controlled.
- e. C13 experiment MACROS with default operating parameters.

IX. Other Nuclei Accessory: The other nuclei accessory converts the carbon/proton (C/H) Eft system and probe to a two-channel broad band system and probe which covers the range of 90MHz – 50MHz (H1 and F19) and 25MHz – 10MHz (P31 to S29).

The Other Nuclei Accessory includes:

- a. Broadband RF interface and probe tuning hardware.
- b. MACROS with default operating parameters for some of the more commonly observe nuclei within the range of the probe.
- c. DDS RF Frequency Synthesizer

X. Air Source for Eft90 Spectrometer: Includes diaphragm air pump, air dryer, and all necessary plumbing packaged in a quiet box for trouble-free air source.

3. Installation and Warranty

The equipment was installed on August 28, 2013 by Anasazi Instruments and a Electrical Contractor secured by the University.



Figure 1: Anasazi personnel installing the 90-MHz FT NMR System

Anasazi Instruments Inc. warrants its RF hardware and NMR software against defects in materials and workmanship for a period of five years after installation or sixty-three months after delivery, whichever comes first. In the event of a failure at any time within the Warranty period, Anasazi Instruments Inc., at its option will repair or replace the defective product or part.



Figure 2: Photographs of the Anasazi 90-MHz FT NMR Console System and Magnet

4. Accomplishments

The award will have broader impacts on research and the education in the chemical sciences at Bowie State University, a Historically Black College and University and Primarily Undergraduate Institution.

4.1. Research

The NMR instrumentation has further enabled PI and student researchers to conduct research on the novel synthesis of functionalized aryl β -enaminones. The enaminone targets are potentially capable of forming self-assembled, polarized layers that confer unique functionality on organic field-effect transistors (OFETs); the key devices used for organic circuits in electronic devices intended for conformal, transparent, and printable electronics. AFSOR has stated interest in chemical sensing and the PI and a collaborator will submit future proposals to support this area.

4.2. Undergraduate Education

The instrument will also advance research-related education by its incorporation into an upper-level research laboratory organic course. By introducing more hands-on activities, like FT-NMR and linking laboratory and classroom experiences, students will gain a much better conceptual understanding of chemistry which would aid them in employing the broader applications of chemistry to their future academic or professional interest.



Figure 3: Senior undergraduate researcher Edward Ollie using the NMR system. Edward has applied to top Chemistry programs and is interested in a career in chemistry/technology.

5. Examples of NMR spectra using N-propyl benzoate as sample.

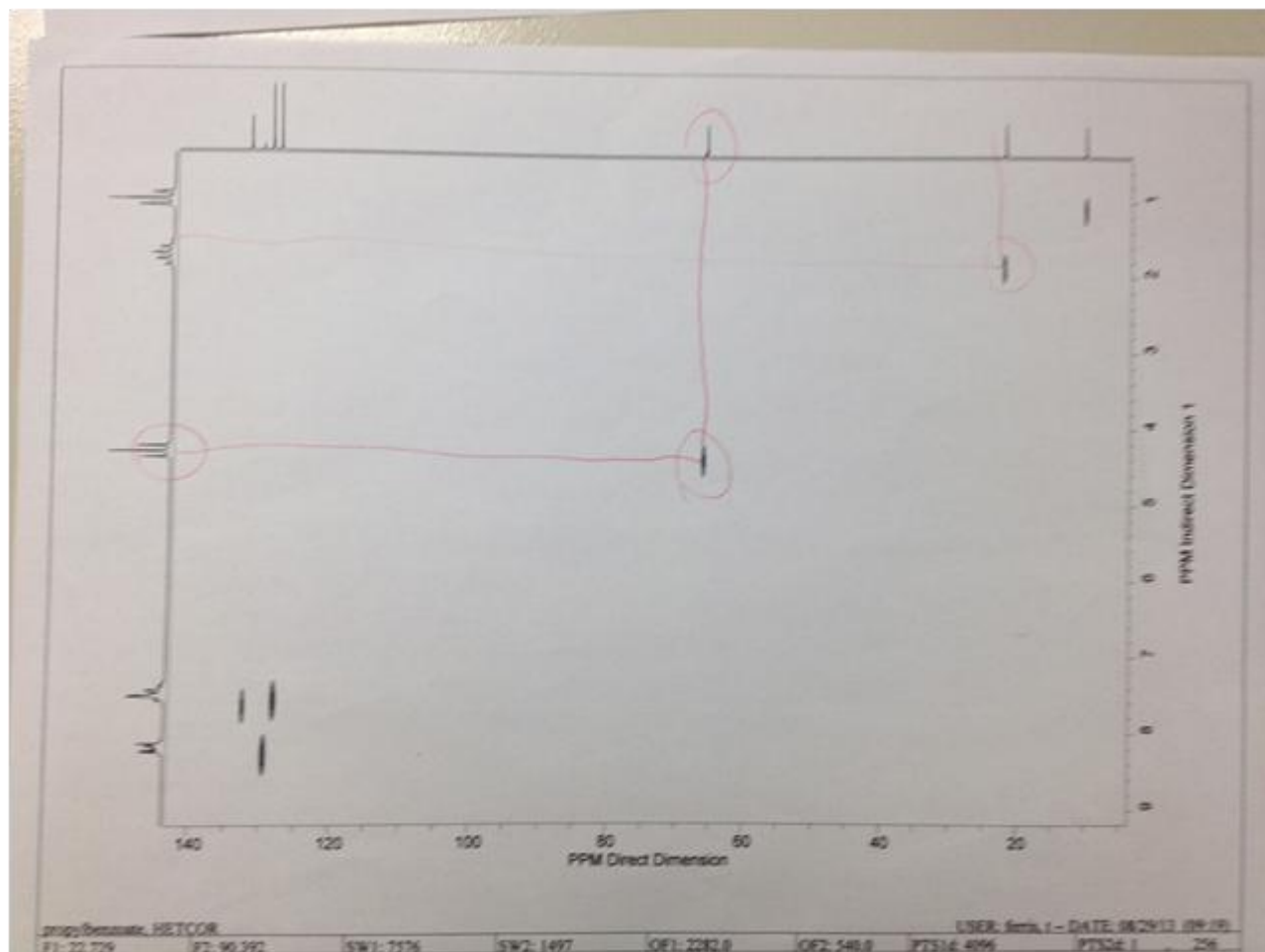


Figure 4: HETCOR of N-propyl benzoate

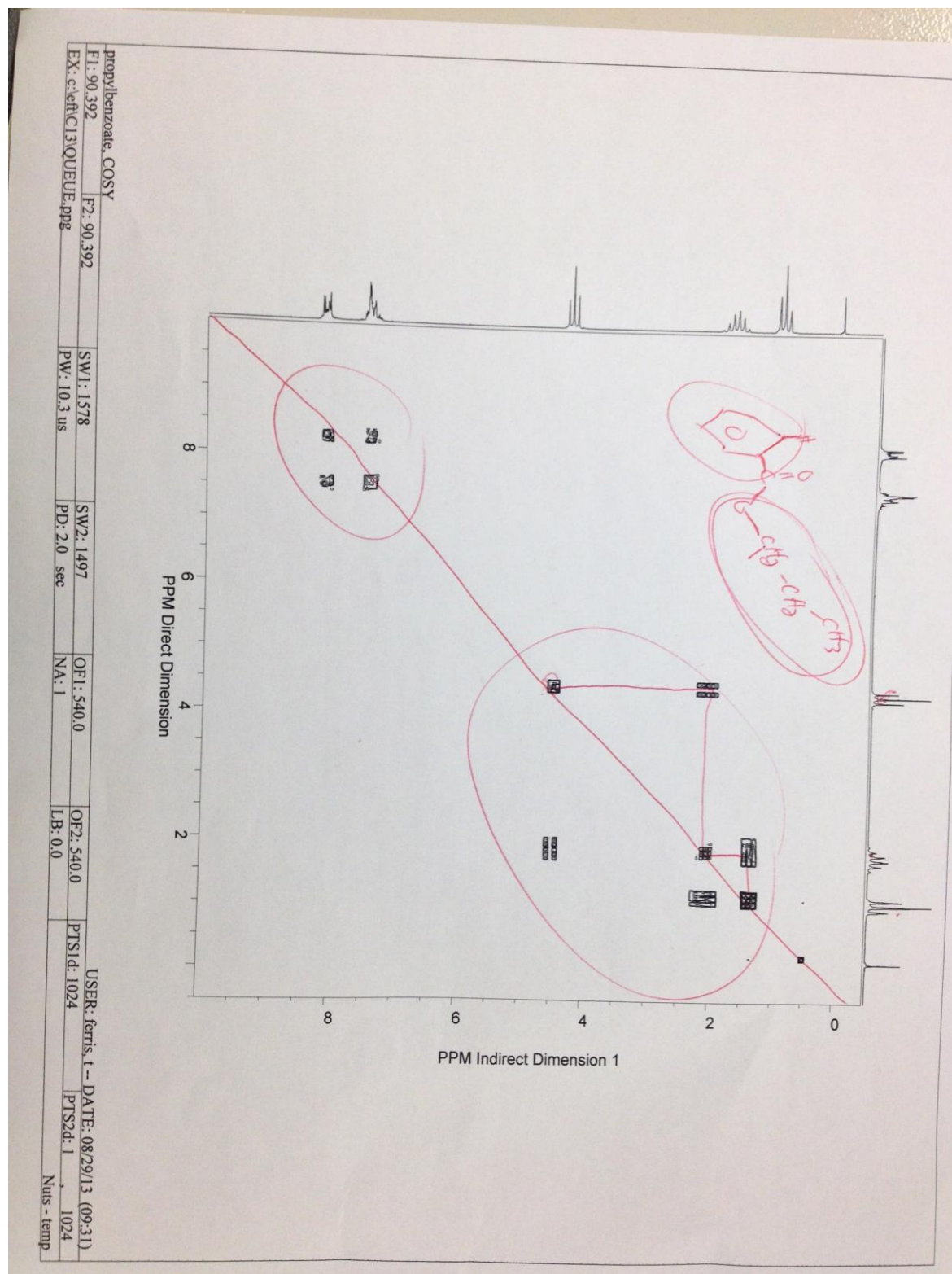


Figure 5: COSY of N-propyl benzoate

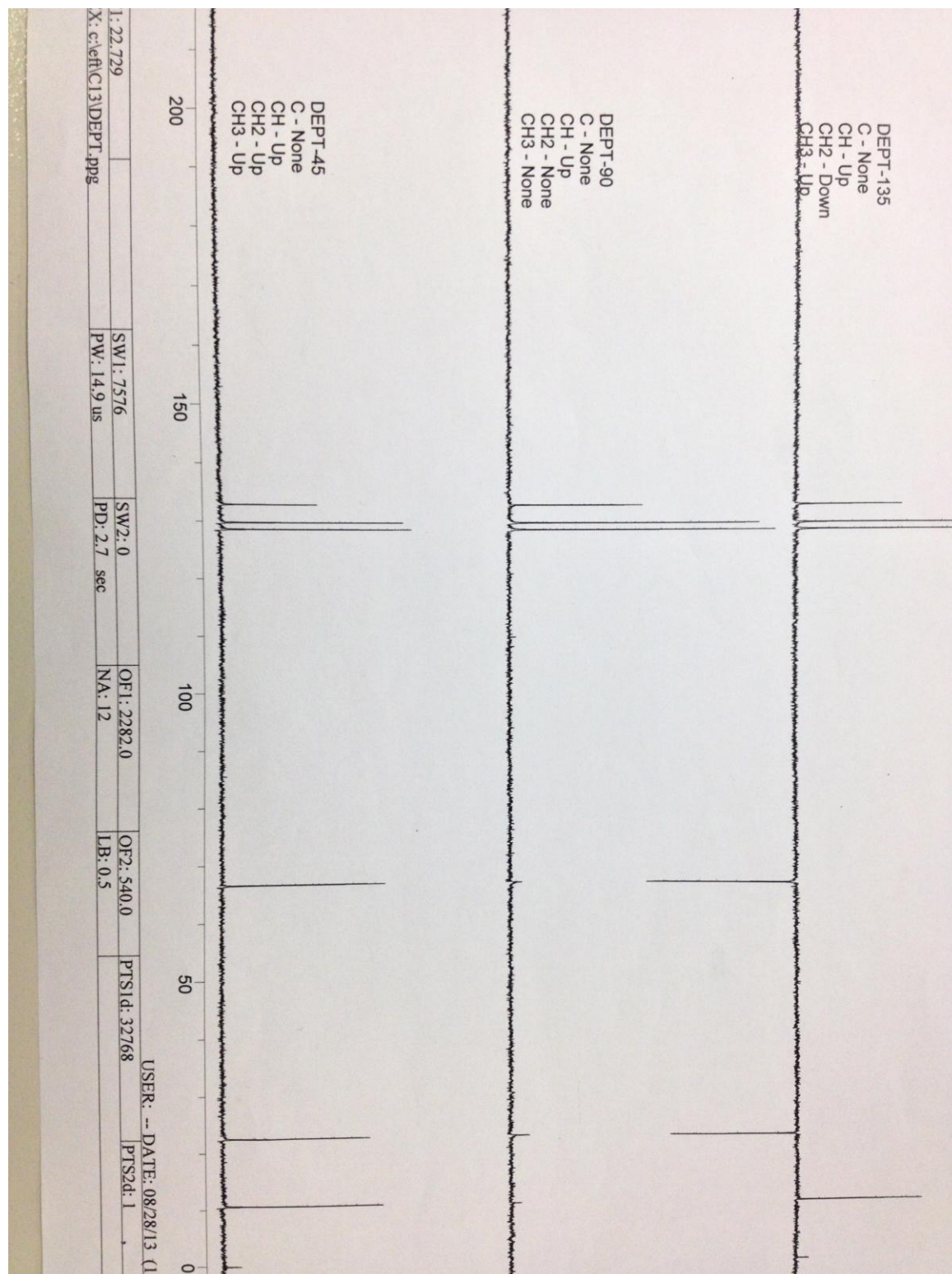


Figure 6: C13/DEPT of N-propyl benzoate

6. HONORS and AWARDS

NASA MIRS Fellow 2013

Inducted, Sigma Xi, Full Member, 2012

Elected Secretary, Chemical Society of Washington, 2011-present

Ad Hoc Reviewer, Research Corporation for Science Advancement, 2011

2011 Peer Review Panel Member, NSF-TUES Program, 2011

Most Outstanding Young Faculty, College of Arts and Sciences, Bowie State University, 2010

NIH Postdoctoral Researcher Training Grant Award, Harvard University, 2003-2006

Howard University Chemistry Graduate Student Association, President, 2000-2001

Most Outstanding Organic Graduate Teaching Assistant Award, 2001

7. Publications (9/01/12- 9/30/13)

1. “2-Chloro-3-dibutylamino-1,4-naphthoquinone”: Oladapo Bakare, Ray J. Butcher, Solomon Berhe, and Alan J. Anderson *Acta Cryst. E* (*Acta Cryst.* (2013). E69, o1230)

2. Synthesis and Characterization of Novel Unsymmetrical and Symmetrical 3-Halo- or 3-Methoxy-substituted 2-Dibenzoylamino-1,4-naphthoquinone Derivatives. Yakini Brandy, Nailah Brandy, Emmanuel Akinboye, Malik Lewis, Claudia Mouamba, Seshat Mack, Ray J. Butcher, Alan J. Anderson and Oladapo Bakare *Molecules* 2013, 18(2), 1973-1984