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Report Title

Final Report: Acquisition of a Nanosecond Laser Flash Photolysis Spectrometer to Enhance Understanding of Photochemistry and Reaction Kinetics in Undergraduate Research/Curriculum

ABSTRACT

The acquisition and installation of the nanosecond flash photolysis spectrometer have been completed. The instrument is used to enhance both research and teaching at Spelman College. This acquisition includes: (1) LP980 laser flash photolysis instrument (Edinburgh Instruments), (2) Nd:YAG laser, (3) cuvette holder (-40 – 110 °C), and (4) laser table. The laser system generates 532, 355, and 266 nm wavelengths. The current setup also includes: (1) an iCCD (intensified charge-coupled device) camera, 180-850 nm, (2) Hamamatsu R928 photomultiplier (200-870 nm), and (3) A Si-photodiode detector (200-1100 nm). Training to use the instrument was completed in the Fall 2015. The instrument has been used to support research on transient absorption studies of nitroaromatic compounds. The system facilitated the study of laser induced transient absorption and emission decay photochemistry. Currently, the instrument is used to study the interaction of light with nitroaromatic compounds with a focus on 1-nitropyrene and 1,4-dinitrobenzene. The preliminary data was generated using 1-nitropyrene. The results were consistent with published data. The development of new classroom teaching module based on the 1-nitropyrene results is currently in progress. The module will be integrated into the physical chemistry laboratory course in the 2016 – 2017 academic year.

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Technology Transfer

Final Report

PROPOSAL TITLE: Acquisition of a Nanosecond Laser Flash Photolysis Spectrometer to Enhance Understanding of Photochemistry and Reactions Kinetics in Undergraduate Research/Curriculum

PI: Dr. Yassin A. Jeilani

Instrument: Nanosecond laser flash photolysis spectrometer including a pulsed Nd:YAG laser

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Table 2. Components of the Instrument

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Table 4. Selected solvents and reagents

Abstract

The acquisition and installation of the nanosecond flash photolysis spectrometer have been completed. The instrument is used to enhance both research and teaching at Spelman College. This acquisition includes: (1) LP980 laser flash photolysis instrument (Edinburgh Instruments), (2) Nd:YAG laser, (3) cuvette holder (-40 – 110 °C), and (4) laser table. The laser system generates 532, 355, and 266 nm wavelengths. The current setup also includes: (1) an iCCD (intensified charge-coupled device) camera, 180-850 nm, (2) Hamamatsu R928 photomultiplier (200-870 nm), and (3) A Si-photodiode detector (200-1100 nm). Training to use the instrument was completed in the Fall 2015. The instrument has been used to support research on transient absorption studies of nitroaromatic compounds. The system facilitated the study of laser induced transient absorption and emission decay photochemistry. Currently, the instrument is used to study the interaction of light with nitroaromatic compounds with a focus on 1-nitropyrene and 1,4-dinitrobenzene. The preliminary data was generated using 1-nitropyrene. The results were consistent with published data. The development of new classroom teaching module based on the 1-nitropyrene results is currently in progress. The module will be integrated into the physical chemistry laboratory course in the 2016 – 2017 academic year.

1. Description of the instrument

We purchased a flash photolysis system (Table 1) with capabilities for transient absorption/emission studies of nitroaromatics including explosives byproducts. For studies focusing on munitions waste byproducts, an iCCD camera in the ultraviolet region was purchased from Andor Technology. This camera is used for the direct spectral measurement of excited-state absorptions. The software allows control of the delay time from the initial laser pulse. The software (Edinburgh Instruments) provides an automated parameter interface for routine measurement of spectra over the course of the measurement. This allows a spectral resolution of approximately 2 nm across the range of approximately 250-850 nm. The wavelengths for the kinetics studies is selected based on the transient absorption spectra recorded with the iCCD camera. The kinetics studies are performed with the photomultiplier-tube (PMT) detector. This PMT detector is capable of measuring transient decay kinetics at individual wavelengths. The iCCD camera permits measurements at multiple wavelengths; therefore, the PMT measurements can be set to those specific wavelengths to measure the transient absorption lifetime. Also, a Si-photodiode detector is included to extend wavelengths to the near infrared region. A sample holder with temperature control (temperature range of -40 °C – 110 °C) capabilities allows the study of the temperature effect on reaction kinetics.

Table 1. Components and features of the LP980 Instrument

#	Description
1	Edinburgh Instruments LP980-K Laser Flash Photolysis Spectrometer. The sensitivity of the LP980-K (minimum detectable optical density) is $\Delta OD = 0.002$ (single shot, fast detector option) with an overall response function of <7 ns (laser permitted, fast detector option)
2	150 W CW Xenon lamp with power supply
3	Sample Chamber: Large sample chamber
4	TMS302-A monochromator for one kinetic detector and one ICCD
5	Standard sample holder of the LP980. The laser beam and probe beam intersect perpendicular at the sample position.
6	Detector 1: Analyzing photomultiplier in standard LP980 housing, featuring Hamamatsu R928 side window photomultiplier.
7	Detector 2: Si Pin Photodiode, Spectral range: 200 nm-1100 nm
8	Digital Storage Oscilloscope (Tektronix MDO3032) for transient recording
9	Computer and monitor
10	Grating 600 grooves/mm, 1000 nm blaze
11	Diffuse Reflectance Option Sample holder for solid, non-transparent samples
12	Mount for iCCD detector

In addition to the main instrument, the complete system requires the purchase of the complete list of items described in Table 2. The two major components in Table 2 are (1) the iCCD camera (Andor), and (2) the laser system (Spectra Physics).

Table 2. Components of the Instrument

#	Description
1	Supplier: <i>Edinburgh Instruments</i> , Nanosecond laser flash photolysis spectrometer
2	Supplier: <i>Andor</i> , Scientific camera, USB iStar ICCD, 1024x256 pixels CCD, 26x26 μm pixel size, 15.8 fps, 332 sps FVB, 1:1 fibre optic coupled image intensifier, Ø25mm Gen 2 W-AGT photocathode, 16% peak QE typ., P43 phosphor, 7ns gate width, 500 kHz photocathode repetition rate, Intelligate, integrated 3x output ultra-low jitter digital delay generator, cooling to -35 °C, USB 2.0 interface, And SOLIS (T) Software for Time-resolved solutions
3	Supplier: <i>Spectra-Physics</i> , Quanta-Ray INDI series laser, Model # INDI-40-10, Includes: (1) Pair of mounted 532 nm, 355 nm, and 266 nm dichroic beam splitters, (2) INDI-INT CONTROL allows for both internal control or Computer Control for INDI laser system, and (2) Beam dump for internal INDI use
4	Supplier: <i>Quantum Northwest</i> , Temperature-Controlled Cuvette Holder for use in the Edinburgh Instruments FLS980 Photoluminescence Spectrometer
5	Supplier: <i>Coherent</i> , FieldMaxII-TO Laser Power Meter (RoHS)
6	Supplier: <i>Newport</i> , Laser Table: Custom IG Breadboard: 3.0' x 8.0' x 2.3" and Custom INT Nonisolated Integrity Stand
7	Supplier: <i>Applied Photophysics</i> , Stopped-flow accessory (Model RX2000)

2. Instrument installation and setup

The instrument installation and software training was completed in fall 2015. Currently, the instrument is used to study the kinetics of the interaction of light with nitroaromatic compounds. The software supplied by the Edinburgh Instrument allows (1) operation of the instrument, (2) automated data processing, and (3) control of the laser. Figure 1 shows the instrument currently installed in the laboratory. The PMT detector (Figure 1) can be replaced with the Si Pin Photodiode (Spectral range: 200 nm-1100 nm).

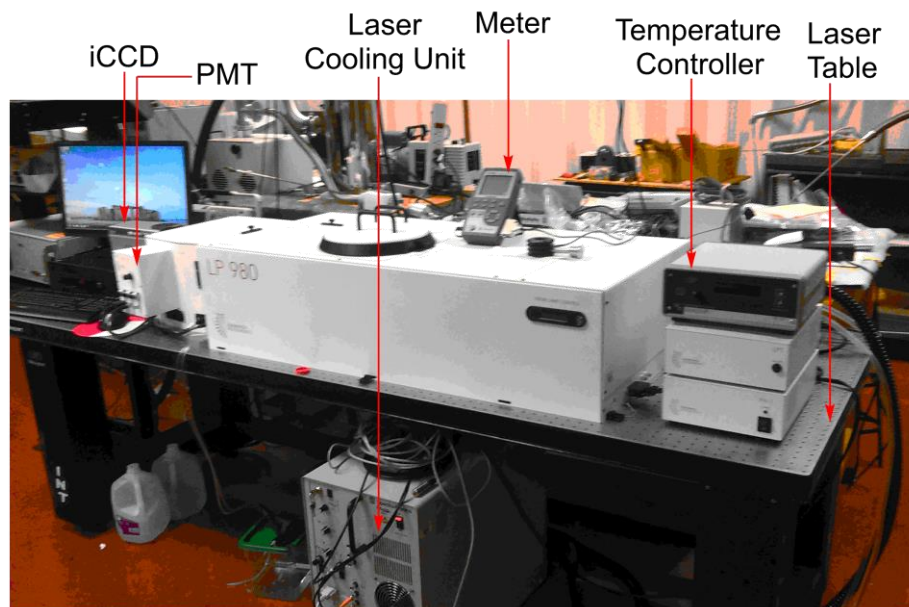


Figure 1. LP 980 Flash photolysis instrument

The laser system was purchased from Spectra Physics (Figure 2). The stopped flow accessory (Model RX2000) was purchased from Applied Photophysics Ltd (Figure 3). The accessory has a dead time of 8 ms and requires just 120 μL of each reagent per determination. It also includes an in-built temperature probe with digital display of thermostat temperature.



Figure 2. Laser (from Spectra Physics) and laser table (Newport) setup in the laboratory



Figure 3. Stopped-flow accessory

3. Statement of the problem studied

The plan is to use the flash photolysis system to study the transient absorption kinetics of nitroaromatics including explosives byproducts and other nitroaromatics. These studies will contribute in understanding current environmental problems associated with the fate of these pollutants.

4. Summary of the most important results to demonstrate the new instrument capabilities

The quality of the data generated by the instrument was tested using chemical standards. To test the performance, 1-nitropyrene (NPY) was selected to generate preliminary data because published data of NPY is available for comparison. The spectra were generated using the third (355 nm, maximum energy of 150 mJ) harmonic output from the Nd:YAG laser (Spectra Physics) as the excitation source.

The transient absorption spectrum (Figure 4) of NPY was obtained using the iCCD detector. The spectrum is consistent with the previously reported transient absorption spectrum of NPY.¹ The negative absorbance between ~325 and ~430 nm is associated with the bleaching of the ground state of NPY.

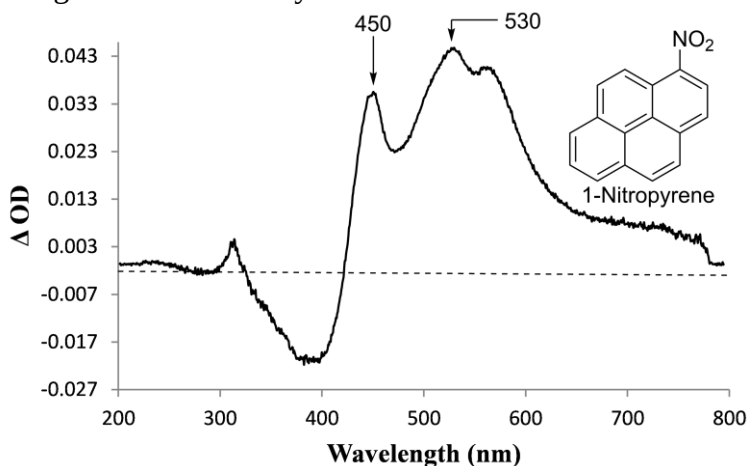


Figure 4. Transient absorption spectrum of NPY (6.5×10^{-5} M) in acetonitrile

The transient absorption spectrum of NPy in acetonitrile shows two peak maxima at 450 nm and 530 nm. The decay rates of the absorbance at these two peaks can now be targeted using the PMT detector. The kinetics data for NPy in acetonitrile was recorded using the PMT detector (Figure 5-a). The fitting to exponential decay (Figure 5-b) is performed using Edinburgh Instruments software. The decay rate of the absorbance at 530 nm was 235 ns ($\chi^2 = 1.562$).

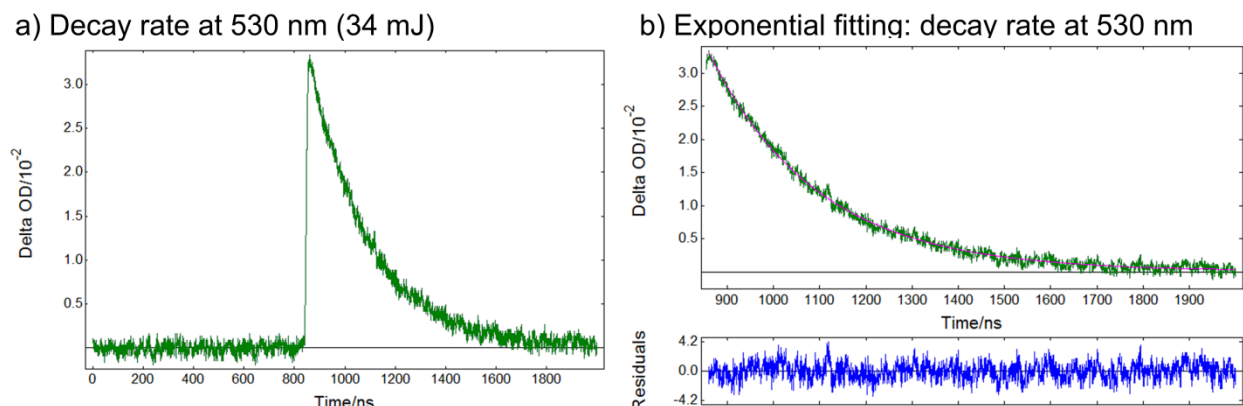


Figure 5. Decay kinetics at 530 nm

Similarly, Figure 6 shows the decay kinetics at 450 nm and excitation energy of 34mJ. The decay rate was 200 ns ($\chi^2 = 1.296$). We find that the decay rate for the absorptions at 420 nm is faster than the decay rate at 530 nm. This observation is consistent with the previous study.¹

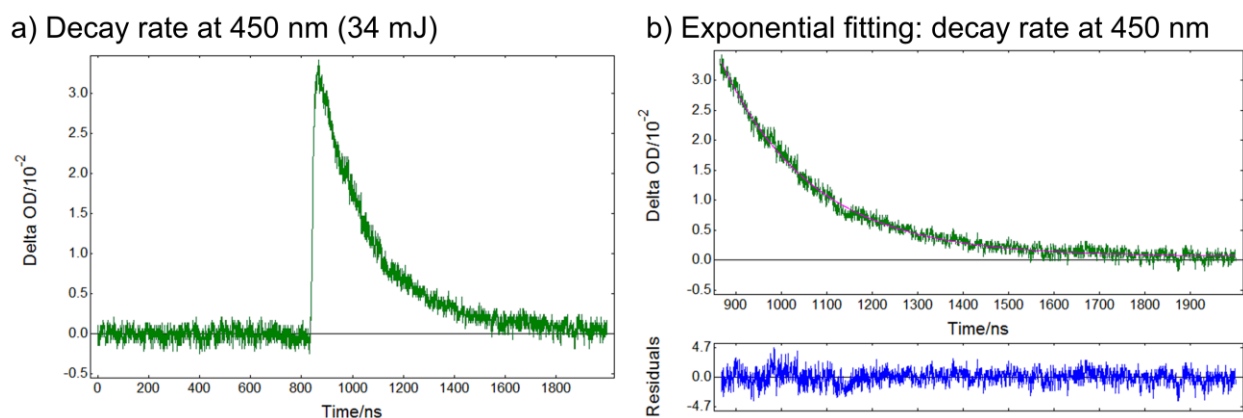


Figure 6. Decay kinetics at 450 nm (34 mJ)

Spectra were recorded in acetonitrile at long observations after the laser pulse and laser energy of 34 mJ (Figure 7). Figure 7 shows that the transient absorptions persist for 400 ns after the laser pulse. The data shown in Figure 4 and 6 are the data routinely recorded by the instrument. These techniques can also be extended to measure the decay rates for quenching reactions. These capabilities can be used to study comprehensively: (a) transient absorption spectra and decay kinetics, (b) kinetics of electron transfer reactions, and (c) solvent and temperature effects on these reactions. In all these cases, the types of data collected are similar to those in Figures 4-6.

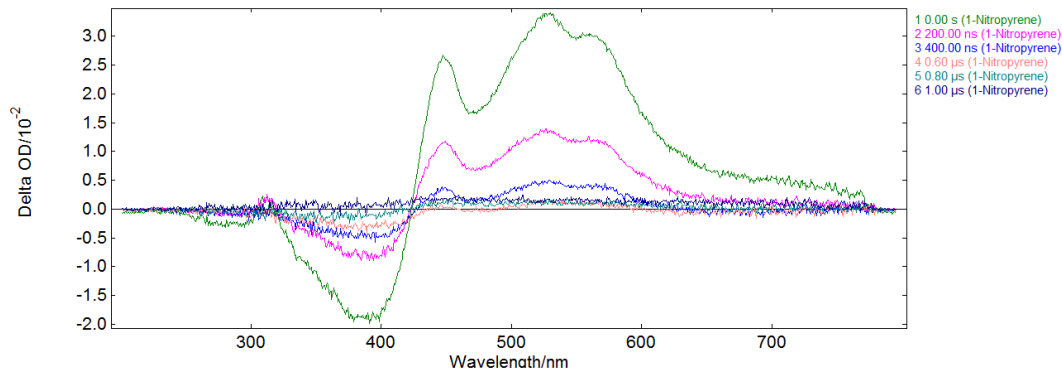


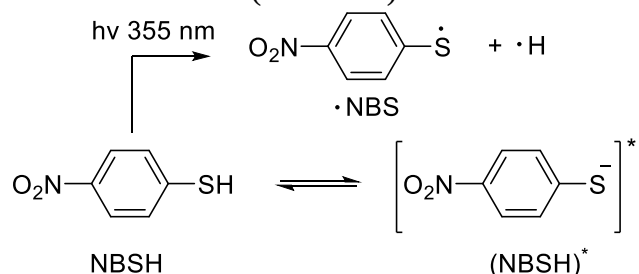
Figure 7. Transient absorption spectra collected at 200 ns, 400 ns, 600 ns, 800 ns, and 1 μ after the laser pulse

5. Impact of this acquisition of research and education capabilities

The new instrument makes it possible to train students in chemical reaction kinetics. The advantages of these capabilities will make it possible to seek funding to support the training of undergraduate students. The acquisition also had a major impact on faculty development opportunities. Spelman College received the 2016 Woodrow Wilson National Fellowship - Career Enhancement Fellowship to support the PI (Dr. Jeilani). This fellowship will support flash photolysis studies of dinitropyrenes. These studies will contribute to understanding the environmental fate of the dinitropyrenes. Also, the PI established a new collaboration with Prof. Michael C. Heaven at Emory University. This collaboration will help Spelman College in further advancing the new initiatives in transient absorption spectroscopy.

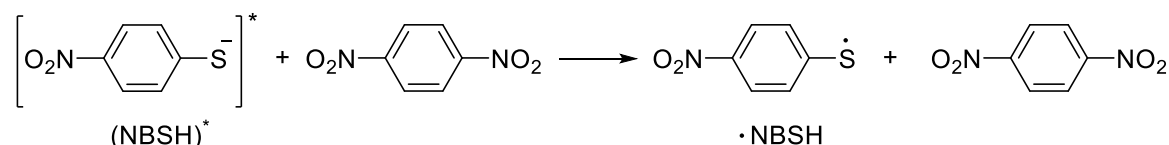
6. Plans for flash photolysis studies of dinitrobenzenes

The next step is to target dinitrobenzenes using a 266 nm wavelength for the excitation source by the techniques described for the Npy. Dinitrobenzenes have often been used as electron acceptors in electron transfer processes. Most often these reactions are reported from the perspective of the electron donors; there is a gap in understanding of these reactions from the perspective of the electron acceptors (dinitrobenzenes). Therefore, the first project will focus on electron transfer processes of dinitrobenzenes. Alam and Ito reported the electron transfer reaction between 1,4-dinitrobenzene (1,4-DNB) and 1,4-nitrobenzenethiol (NBSH).² In this study, transient absorption spectra in the visible and near-infrared (IR) region were reported using 355 nm laser photolysis of NBSH (electron donor) in the presence of 1,4-DNB (electron acceptor) in acetonitrile. The transient spectra of $^3(\text{NBT})^*$ showed the fast decay of part of $^3(\text{NBT})^*$ within few hundred nanoseconds at 400 nm and a slow decay absorption partly attributed to $\bullet\text{NBS}$ (Scheme 1).



Scheme 1

The radical anion of 1,4-DNB showed peaks in the near IR region at 870 and 910 nm attributed to the radical anion of 1,4-DNB (1,4-DNB^{•-}). The first order rate constants obtained from the decay profiles of ³(NBT)^{-*} were consistent with those observed in the rise time profiles of 1,4-DNB^{•-}. This observation was used as evidence that an electron transfer process was taking place between NBSH and 1,4-DNB (Scheme 2).



Scheme 2

Therefore, the strategy in this project is to identify an electron transfer process using (1) the absorptions at 870 and 910 nm to study reactions between 1,4-DNB and electron donors, and (2) kinetics decay profiles of transients characteristic for electron donors. Also, these reactions will be studied at different temperatures. To accomplish these objectives, the flash photolysis instrument includes: (1) Si Pin Photodiode (spectral range of 200 nm-1100 nm) that will allow the detection of 870 and 910 nm peaks, (2) cuvette holder with capabilities to obtain spectra at a range of temperatures (-40 °C – 110 °C), and (3) iCCD detector to study these reactions comprehensively. Also, future studies will include transient Raman spectra to understand better the structure of the transients. This project will focus on both 1,4-DNB and 1,2-dinitrobenzene. To accomplish these projects, the compounds (Table 3) to start these studies were ordered and received in the PI's research laboratory.

Table 3. List of compounds to start the first project in laser flash photolysis

#	Compound	#	Compound
1	1,4-Dinitrobenzene	9	Azulene
2	1,2-Dinitrobenzene	10	4-Cyanophenol
3	Ferrocene	11	2-Nitrotoluene
4	4-Phenylphenol	12	1,8-Dihydroxyanthraquino
5	2,4-Dinitroaniline	13	<i>cis</i> -Stilbene
6	3-Nitrotoluene	14	<i>trans</i> -Stilbene
7	1,3-Cyclohexadiene	15	Cyclohexane
8	1,4-Cyclohexadiene		

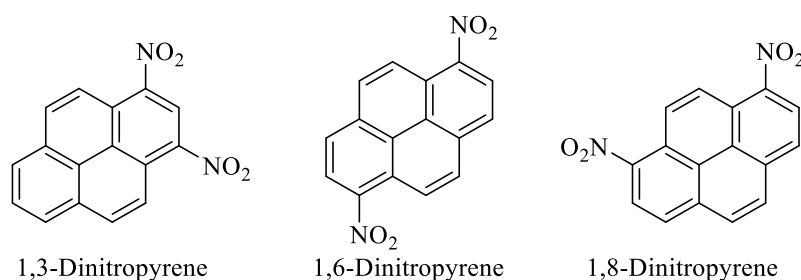
Also, solvent effect on these reactions will be studied. Table 4 shows the selected solvents and electron donors that will be considered for these reactions. Students participating in these projects will develop a better understanding of the interaction of light with explosives byproducts. The features in the flash photolysis system were specifically will enhance capabilities at Spelman College to train students in research areas of interest to the United States Department of Defense (DoD).

Table 4. Selected solvents and reagents

Triplet Quencher	Arylphenol Quenchers	Solvents	Electron Donors
Perylene	1-Naphthol	Methanol	Triethylamine
Azulene	2,6-Dimethoxyphenol	Acetonitrile	1,4-Dimethoxybenzene
Anthracene	4-Hydroxy-3-methoxybenzoic acid	Cyclohexane	Dithieno[3,2-b:2',3'-d]thiophene
Ferrocene	Dimthylaniline	Benzene	1,4- Diazabicyclo[2.2.2]octane (DABCO)

7. Plans for flash photolysis studies of dinitropyrenes

Dinitropyrenes (DNPs) are products of incomplete combustion of petroleum products such as diesel. DNPs (Figure 8) are nitropolycyclic hydrocarbons and have been identified in the environmental particulate matter. Photochemical transformation of DNPs leads to toxic aromatic compounds. The proposed project focuses on the kinetics of the phototransformation of DNPs using nanosecond laser flash photolysis. Photochemistry of DNPs is important because of the potential transformation of DNPs into biologically active compounds. This study contributes to understanding factors and reaction mechanisms that determine photoreactivity of DNPs relevant to the environmental fate of DNPs.

**Figure 8:** Structures of the selected dinitropyrenes

8. Integrating kinetics modules in physical chemistry laboratory course

The data generated for the preliminary data using NPy will be used to teach transient absorption spectroscopy in the physical chemistry laboratory course (SCHE 346 L). Now that the experiments have been tested in our laboratories, this laboratory module will be integrated in the spring 2017 semester because this course is not offered during the fall semester. Also, we identified two experiments based on stopped flow kinetics. The first experiment is the reduction of potassium ferricyanide by l-ascorbic acid. This experiment was selected because ascorbic acid is used instead of using concentrated strong acids. The second experiment is based on the reaction of dye with a bleaching agent is monitored over time. The plan is to evaluate these experiments during summer 2016. The goal is to adapt these experiments in the physical chemistry laboratory during spring 2017. These modules will enhance student understanding of the kinetics of chemical reactions.

9. Plans for student involvement in research of DoD interest

One of the primary goals of this grant is to train undergraduate students in activities of interest to Department of Defense (DoD) with a focus on chemical reactions and dynamics. To

achieve this goal, new activities in understanding the light-induced transformations of nitroaromatic compounds have been adapted to train students. Also, the PI will dedicate the summer and the fall of 2016 to train students in transient absorption kinetics using the techniques described for the preliminary data. These activities are supported by the recent fellowship awarded to the PI. About five students are expected to participate in these activities by the end of 2016. The number of students trained in projects of DoD interest is expected to increase over time.

10. Conclusions

The infrastructure at Spelman College to train students in research of DoD interest has been significantly enhanced by the acquisition of the flash photolysis system. The preliminary data were in agreement with published data. Also, the software supplied by the vendor allows an easy operation of the instrument and data processing that facilitates training undergraduate students.

11. References

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- (3) Measuring Rapid Kinetics Data with a Commercial Spectrophotometer Using a Stopped-Flow Accessory, Hi-Tech Scientific Application Note, Publication Number AN.014.S20.
- (4) Comerford, J. Monitoring fast reactions using Stopped Flow Kinetics on the Cary 50/60 UV-Vis, Agilent Application Note 2011, Publication Number SI-A-1177.