Defense Threat Reduction Agency
8725 John J. Kingman Road, MS
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USSTRATCOM CENTER FOR COMBATING WMD

DTRA-TR-15-35

TECHNICAL REPORT

New Organic Scintillators for Neutron Detection

Distribution Statement A. Approved for public release; distribution is unlimited.

March 2016

HDTRA1-11-1-0045

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13. SUPPLEMENTARY NOTES

14. ABSTRACT

This project, funded by DTRA, proposes to develop novel boron containing organic single crystal detectors for neutron detection as an alternative for 3He based detectors. The goal of the proposed effort is to investigate novel organic scintillators that are capable of efficient thermal and fast neutron detection, and provide effective neutron/gamma discrimination based on pulse shape as well as pulse height analysis. The organic compounds we plan to investigate are derivatives of the azaborines.

15. SUBJECT TERMS

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UNIT CONVERSION TABLE

U.S. customary units to and from international units of measurement[*]

U.S. Customary Units	Multiply by → ← Divide by[†]	International Units
Length/Area/Volume		
inch (in)	2.54 × 10^{-2}	meter (m)
foot (ft)	3.048 × 10^{-1}	meter (m)
yard (yd)	9.144 × 10^{-1}	meter (m)
mile (mi, international)	1.609 344 × 10^{3}	meter (m)
mile (nmi, nautical, U.S.)	1.852 × 10^{3}	meter (m)
barn (b)	1 × 10^{-28}	square meter (m^2)
gallon (gal, U.S. liquid)	3.785 412 × 10^{-3}	cubic meter (m^3)
cubic foot (ft^3)	2.831 685 × 10^{-2}	cubic meter (m^3)
Mass/Density		
pound (lb)	4.535 924 × 10^{-1}	kilogram (kg)
unified atomic mass unit (amu)	1.660 539 × 10^{-27}	kilogram (kg)
pound-mass per cubic foot (lb ft^{-3})	1.601 846 × 10^{1}	kilogram per cubic meter (kg m^{-3})
pound-force (lbf avoirdupois)	4.448 222	newton (N)
Energy/Work/Power		
electron volt (eV)	1.602 177 × 10^{-19}	joule (J)
erg	1 × 10^{-7}	joule (J)
kiloton (kt) (TNT equivalent)	4.184 × 10^{12}	joule (J)
British thermal unit (Btu) (thermochemical)	1.054 350 × 10^{3}	joule (J)
foot-pound-force (ft lbf)	1.355 818	joule (J)
calorie (cal) (thermochemical)	4.184	joule (J)
Pressure		
atmosphere (atm)	1.013 250 × 10^{5}	pascal (Pa)
pound force per square inch (psi)	6.984 757 × 10^{3}	pascal (Pa)
Temperature		
degree Fahrenheit (°F)	[T(°F) − 32]/1.8	degree Celsius (°C)
degree Fahrenheit (°F)	[T(°F) + 459.67]/1.8	kelvin (K)
Radiation		
curie (Ci) [activity of radionuclides]	3.7 × 10^{10}	per second (s^{-1}) [becquerel (Bq)]
roentgen (R) [air exposure]	2.579 760 × 10^{-4}	coulomb per kilogram (C kg^{-1})
rad [absorbed dose]	1 × 10^{-2}	joule per kilogram (J kg^{-1}) [gray (Gy)]
rem [equivalent and effective dose]	1 × 10^{-2}	joule per kilogram (J kg^{-1}) [sievert (Sv)]

[*] Specific details regarding the implementation of SI units may be viewed at http://www.bipm.org/en/si/.

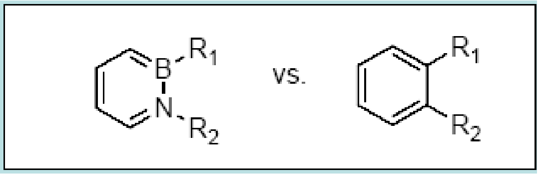
[†] Multiply the U.S. customary unit by the factor to get the international unit. Divide the international unit by the factor to get the U.S. customary unit.

2015-11-16

Table of Contents

Grant/Award #: HDTRA1-11-1-0045
PI Name: Edgar van Loef
Organization/Institution: Radiation Monitoring Devices, Inc.
Project Title: New Organic Scintillators for Neutron Detection

I. Abstract



In this project, Radiation Monitoring Devices (RMD) proposes to develop novel boron containing organic single crystal detectors for neutron detection as an alternative for ^{3}He based detectors that will fulfill the needs of the Defense Threat Reduction Agency as stated in the Broad Agency Announcement HDTRA1-08-10-BRCWMD-BAA. The goal of the proposed effort is to investigate novel organic scintillators that are capable of efficient thermal and fast neutron detection, and provide effective neutron/gamma discrimination based on pulse shape as well as pulse height analysis. The organic compounds we plan to investigate are derivatives of the azaborines. These compounds contain both boron and hydrogen that are necessary for the detection of thermal (with ^{10}B) as well as fast neutrons (with ^{1}H). Azaborines are organic compounds consisting of benzene rings where adjacent carbon atoms are replaced by nitrogen and boron atoms, see Figure 1.

Joining RMD in the project will be the research group of Professor Shih-Yuan Liu at the University of Oregon (UoO). The team at UoO provides cutting edge research and chemical synthesis of azaborines. A large part of this effort is executed by the team at UoO who will engage a dedicated post-doctoral student and several graduate students to work exclusively on this effort. Laboratory training to and the synthetic chemistry of the azaborines will be their main task. More specifically, the team at UoO will focus on the investigation of optimal pathways to the synthesis of the azaborines *via* Dewar, Liu and other synthetic routes. RMD will construct a crystallizer system that will be capable of growing organic scintillators based on the azaborines for neutron detection. Crystals of the azaborines will be grown at RMD. At the end of the program, RMD and UoO expect to have developed the azaborine research to a stage where it can aid the monitoring of special nuclear materials (SNMs) to counter weapons of mass destruction.

II. Objectives

The objective of the proposed effort is to research and develop novel boron containing organic single crystals as high performance neutrons detectors as alternatives for ^{3}He based detectors that will fulfill the needs of the Defense Threat Reduction Agency as stated in the Broad Agency Announcement HDTRA1-08-10-BRCWMD-BAA.

III. Relevance

The proliferation of the weapons of mass destruction such as nuclear weapons is a serious threat in the world today. One way to passively determine the presence of nuclear weapons is to detect and identify characteristic signatures of highly enriched uranium and weapons grade plutonium. Neutrons and gamma rays are two signatures of these materials. Gamma ray detection techniques are useful because the presence of gamma rays of specific energies can confirm the presence of a particular isotope. This technique however, has one significant drawback: In the presence of dense surrounding

Table I. *Thermal Neutron Absorption Reactions, Q values and Cross Sections.*

Isotope	Reaction	Q value (MeV)	σ_{th} (barns)
^{3}He	$^{3}_{2}He + ^{1}_{0}n \rightarrow ^{3}_{1}H + ^{1}_{1}p$	0.764	5330
^{6}Li	$^{6}_{3}Li + ^{1}_{0}n \rightarrow ^{3}_{1}H + ^{4}_{2}\alpha$	4.780	940
^{10}B	$^{10}_{5}B + ^{1}_{0}n \rightarrow ^{7}_{3}Li + ^{4}_{2}\alpha$	2.310 (94%) 2.792 (6%)	3840
^{157}Gd	$^{157}_{64}Gd + ^{1}_{0}n \rightarrow ^{158}_{64}Gd + \gamma$ conversion electrons (70-182 keV)	8.0	25900

material such as lead, gamma ray attenuation can be significant. This can mask the gamma signatures of these special nuclear materials (SNMs).

Neutrons, on the other hand, easily penetrate dense, high atomic number materials compared to gamma rays. For heterogeneous or dense materials such as samples of metals, oxides, and nuclear waste, gamma ray attenuation can be too high to permit accurate corrections of the measured signal. Under these circumstances, passive assay techniques based on neutron detection are preferable. When detected, neutrons are a direct indicator of the presence of spontaneously fissioning isotopes (plutonium and californium) and induced fissions (uranium). Thus, neutron detection is an important component of the overall detection techniques used in identifying SNMs.

Important requirements for neutron detection includes high detection efficiency, a high light yield, and efficient gamma – ray discrimination. Currently, thermal neutron detectors are based on either gaseous ^{3}He tubular designs or inorganic crystals containing ^{6}Li or ^{157}Gd isotopes. However, RMD believes that the azaborines will provide a strong alternative as both a thermal and fast-neutron scintillators.

Azaborines have many properties that makes them very interesting materials for thermal as well as fast-neutron detection. First, due to the presence of boron 1,2-azaborines can serve as potential thermal neutron detectors via the ^{10}B thermal neutron capture reaction (see **Table I**). Secondly, detection of fast-neutrons is accomplished by scattering of neutrons with the hydrogen atoms of the azaborines. Hydrogen is the preferred nuclide to use because a colliding neutron can transfer part or all of its kinetic energy to the proton (hydrogen atom) in one collision. This implies that the average recoil proton energy will be half the initial neutron energy. Consequently, the energy of the recoil proton is directly proportional to the energy of the incident neutron thus preserving information on the energy distribution of the neutron flux.

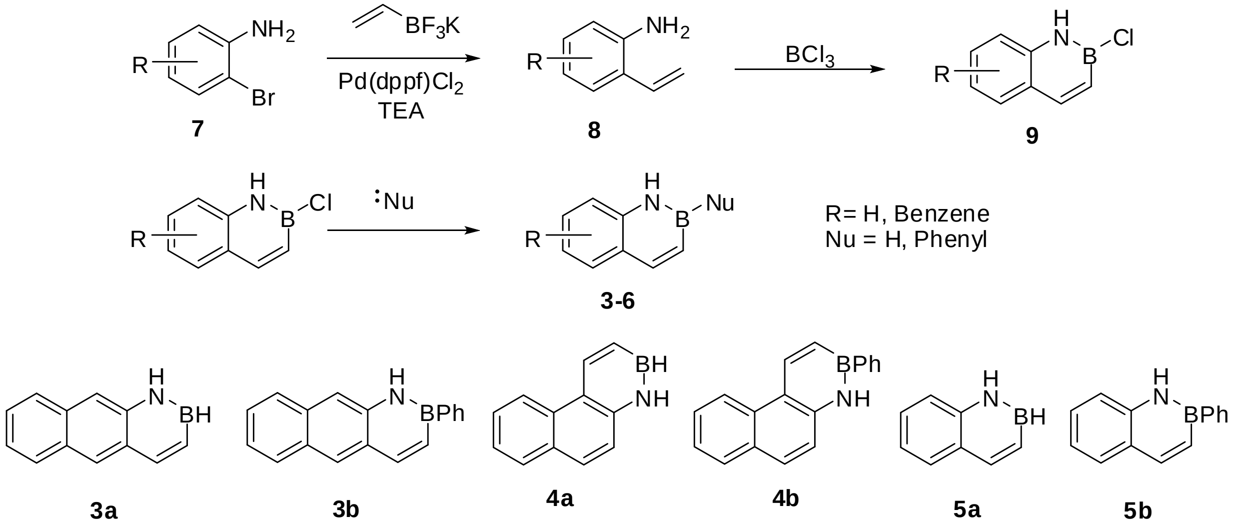
The advantage of the azaborines over ^{3}He based systems is that these organic materials can detect both thermal and fast-neutrons and in principle do not require neutron moderation towards thermal energies to efficiently detect neutrons. Moderation can lead to degradation of spectroscopic information, loss of information on the source of fissile materials, and inefficient detection of weapons of mass destruction. In contrast to ^{3}He based systems, it is expected that the signal from neutrons provided by the organic scintillators retains the necessary spectroscopic information.

IV. Status of the Effort

In the early 1960’s Dewar demonstrated that 1-aza-2-bora-nathpthalene (referred to as NH-BH naphthalene) could be readily synthesized from ortho amino styrene and BCl_3.[1] We envisioned being able to use this synthetic route to synthesize a wide variety of linear and bent acenes (Scheme 1).

Compounds **4a/4b** and **5a/**5b were synthesized from the commercially available starting materials Benzylidene-naphthalen-1-yl-amine and 2-bromoaniline, respectively, on a gram scale in good yield (Scheme 1, R=H) during the first year of the program. With compounds **4a, 4b, 5a,** and **5b** in hand, attention was focused on the synthesis of **3a** and **3b** during the second year of the program. After several attempts, compound **3b** was successfully synthesized on a gram scale. Work since then has been focused on obtaining the parent NH-BH Anthracene (compound **3a**) and scale-up of the synthesis of compound **3a and 3b**.

Subsequently, the synthesized compounds were characterized using NMR and UV-VIS spectroscopy to determine the purity and yield of the synthesis. For both measurements, the spectra clearly indicated the presence of the B-N moiety in the azaborine molecules.



Scheme 1. General synthetic route to BN acenes

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[1] Dewar, M. J. S.; Diet, R.; *J. Chem. Soc.*, **1959**, 2728-271

V. Accomplishments

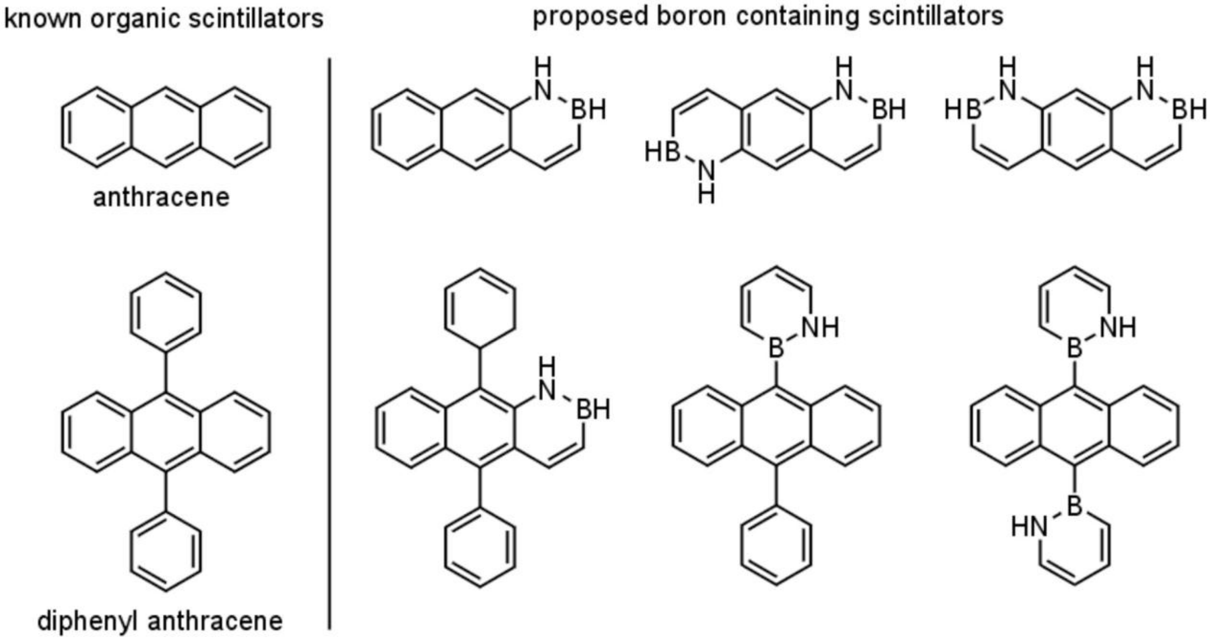
A. Technical Highlights

- Synthesis of novel azaborines
- Chemical characterization
- Crystal growth from solution
- Characterization of the scintillation properties

B. Technical Progress

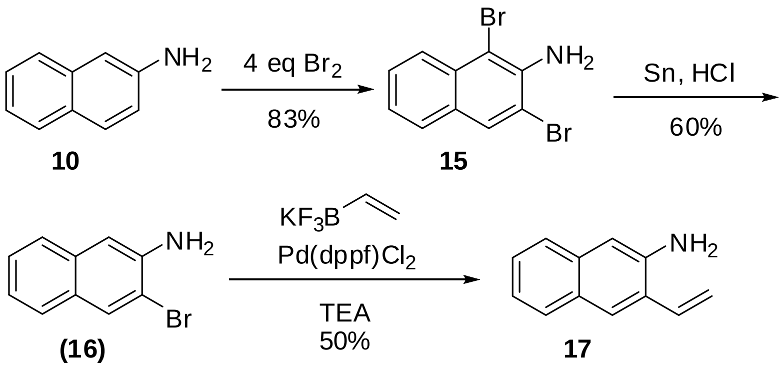
1. Synthesis

As part of the program to research and develop novel boron containing organic single crystals as high performance neutrons detectors, we plan to synthesize six new azaborines incorporating boron-nitrogen heterocycles at the University of Oregon. These molecules were chosen based on the favorable performance of known organic scintillators exhibited in terms of overall light yield, high α/β ratio, and good neutron/gamma discrimination. The six azaborines are shown in **Scheme 2**, as well as their all-carbon analogs.



Scheme 2. Six azaborines and their all-carbon analogs.

To synthesize the anthracene compounds (**3a** and **3b**, see Scheme 1), we initially envisioned a similar route to **3** as the route used to synthesize **4**. Literature precedence indicated that over bromination of **10** followed by a selective debromination with tin would yield compound **16**. Suzuki coupling of **16** with potassium vinyltrifluoroborate should yield **17** (**Scheme 3**).[2]



Scheme 3. Initial route to **17**

After obtaining proposed product **17**, repeated cyclization attempts in a wide variety of conditions with several boron sources yielded only a four coordinate boron species by ^{11}B NMR. The ability to synthesize compounds **4**, and **5**, indicated that the cyclization reaction should work on a variety of substrates. Calculations done by Gabriel J. Lovinger on the relative energies of the cyclization to get to **4a** and **3a** indicated that there was little difference in the reaction coordinate diagram and the ΔG was similar for both reactions (**Figure 2**). This information suggested that perhaps we had been synthesizing a different isomer of **17** then the expected product.

To confirm that we were indeed synthesizing the correct isomer of **17**, single crystal XRD of precursor **16** was performed. Surprisingly, XRD indicated that the desired product was not formed in this reaction, rather compound **18** was formed (**Figure 3**).

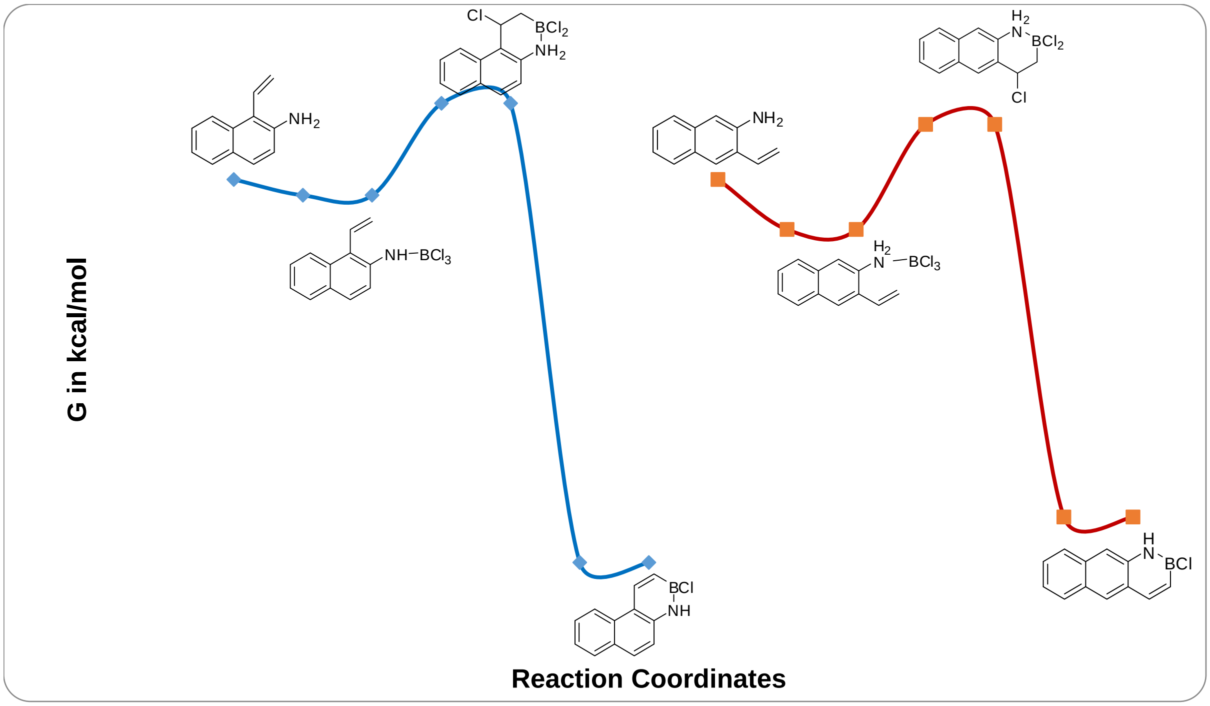


Figure 2. Reaction Coordinate diagram for anthracene and phenanthracene (calculated at DFT B3LYP 6-21G**).

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[2] Hathway; *J. Am. Chem. Soc.*, **1916,** 812-813

Once it was determined that the current route to **3** was inadequate, a new synthetic strategy was devised (**Scheme 4**).[3] An exhaustive literature search indicated that 2, 3 asymmetrically substituted naphthalene's were quite rare, however, both compounds **19** and **20** are commercially available. It is possible to synthesize **20** from the much cheaper **19**, however at the current time the Liu lab lacks the high pressure vessels needed for this reaction. From **20**, Fischer esterification yields **21** in good yield.[13] Reduction by LAH followed by protection of the nitrogen with a pivaloyl group affords the alcohol **23.**[13] Oxidation of the alcohol by MnO_2 yields the corresponding aldehyde **24**.[3] Deprotection of the amine followed by a Wittig reaction with **24** yields the ring closing precursor **17**. Ring closing with BCl_3 followed by reduction with LAH provides compound **3**. This reaction scheme did indeed furnish the title compound **3**. However, the 8 step synthesis is tedious and the oxidation with MnO_2 to compound **24** takes up to 7 days to run to completion. Because of these limitations, a new synthetic strategy was developed (**Scheme 5**).

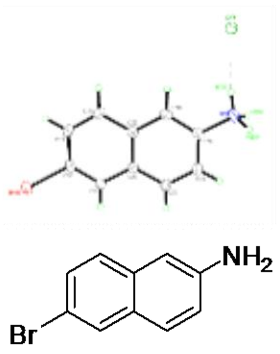
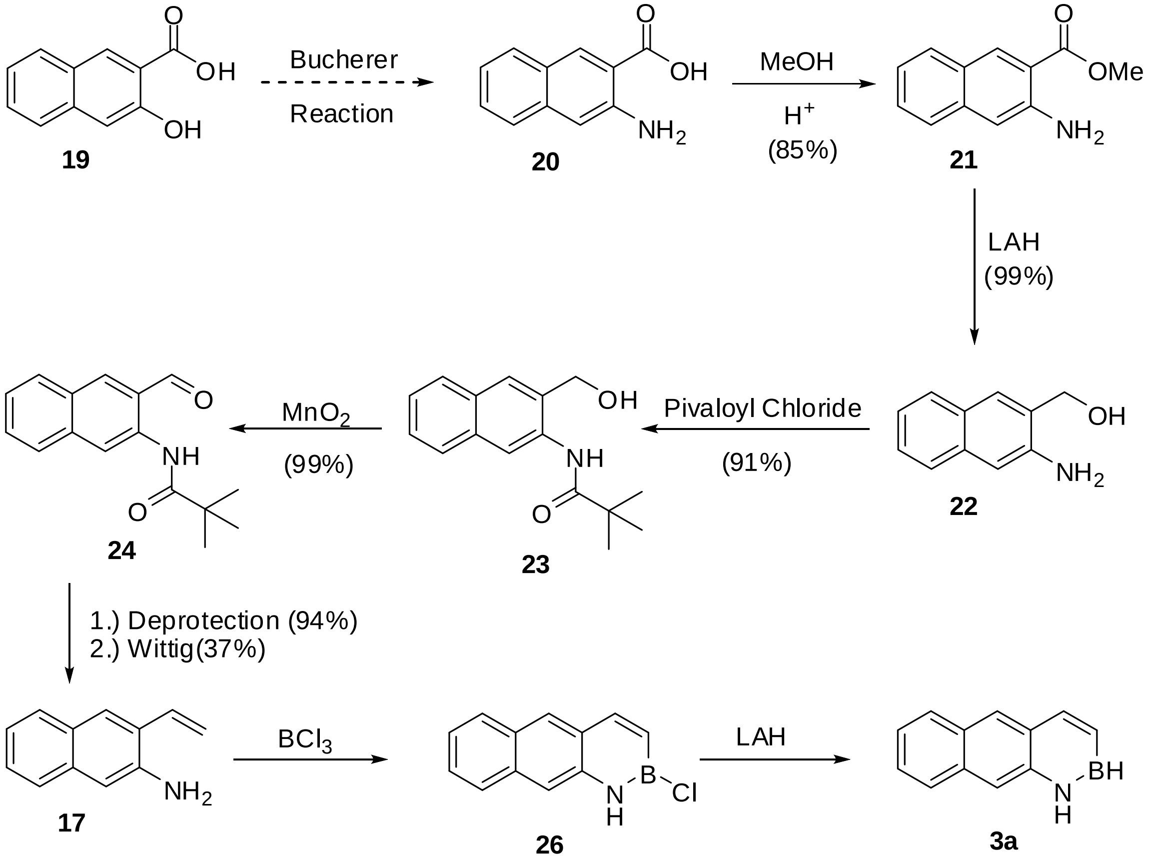


Figure 3. X-ray structure of **18**



Scheme 4. Revised synthesis to **3**

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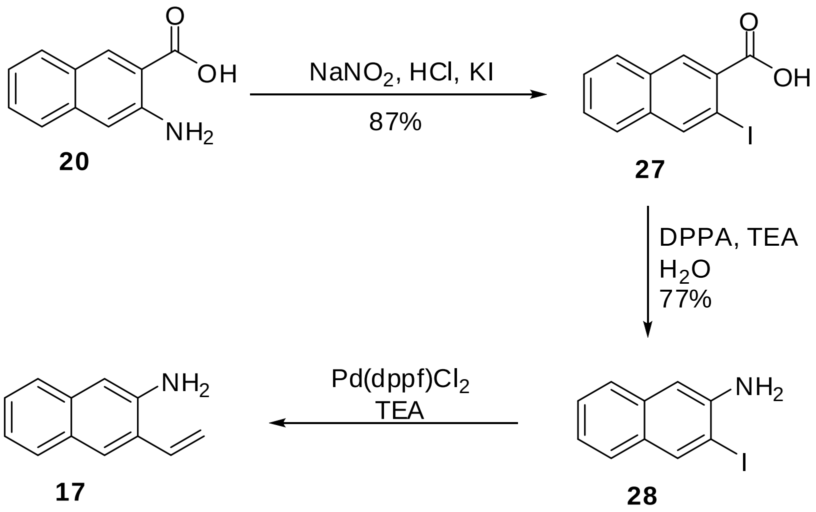
[3] Taffarel, E., Chirayil, S., Thummel, R. P.; *J. Org. Chem.* **1994,** *59*, 823-828



Scheme 5. New route BN anthracene

Starting material **20** is subjected to standard Sandmeyer conditions leading to compound **27** in excellent yield. A Curtius rearrangement using diphenylphosphoryl azide (DPPA) results in the formation of compound **28** in excellent yield. Suzuki coupling leads to compound **17**. From compound **17**, conditions previously mentioned lead to title compound **3a** and **3b**. The route discussed in **Scheme 5** leads to compound **17** in 3 steps, 36% overall yield in 2 days time.

2. Chemical Characterization

To characterize compounds **3a** and **3b**, ^{11}B, ^{1}H, ^{13}C NMR and UV/VIS spectroscopy measurements were performed. Compound **3a** shows a slight blue shift in absorbance compared to the all-carbon analogue. However, the fluorescence from **3a** matches the fluorescence from the all-carbon analogue very well, see **Figure 4**.

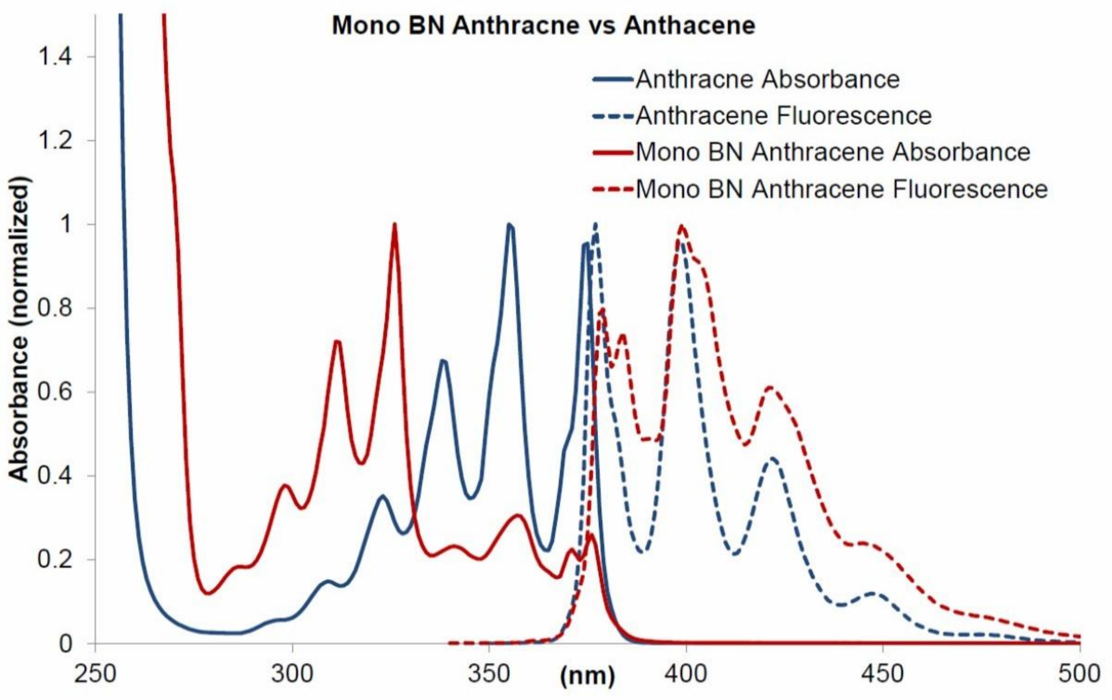


Figure 4. UV/VIS spectrum of azaboraanthracene (**3a**) vs. anthracene.

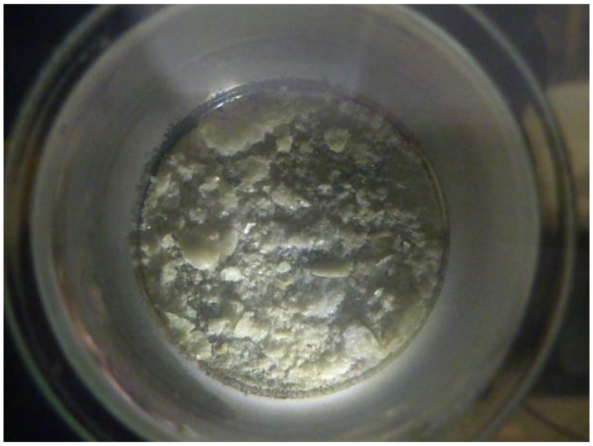


Figure 5. Azaboranaphthalene from acetonitrile.

Figure 6. Azaboranaphthalene in methanol.

3. Crystal Growth from Solution

Once the purity of the azaborine compounds were confirmed, the powders were send to RMD for solution growth experiments. At RMD, a special oxygen-free glovebox is available to perform the solution growth experiments. Hereto, a small amount of the azaborine (about 100 mg) was dissolved in 10 ml of an organic solvent such as benzene, toluene, chlorobenzene, diethylether, isopropylether, THF, methanol, ethanol, isopropanol, acetonitrile, and pyridine. Subsequently, the solvent was slowly evaporated and the progress of crystal growth recorded.

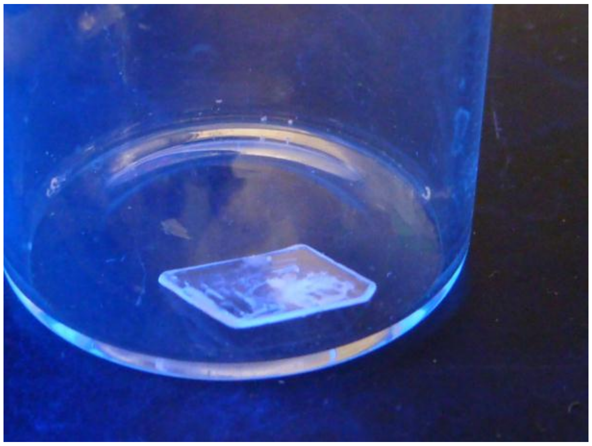


Figure 7. Azaboranaphthalene platelet grown from chlorobenzene shown under UV light.

Figures 5 and 6 show the result of azaboranaphthalene in acetonitrile and methanol, respectively. From the photographs it is clear that no crystals were formed and powder was recovered. This is an indication that the rate of evaporation might have been too high, or that the difference between the dipole moment of the solvent and that of the azaborine compound is too large.[4] Numerous attempts at the crystallization of azaboraphenanthrene did not yield any crystals of size. However, in the case of azaboranaphthalene and phenyl-azaboranaphthalene we were successful in growing small platelets from diethylether and chlorobenzene, see **Figure 7**.

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[4] Yoshiaki Tanaka, Masakuni Matsuoka, "Selection of solvents for organic crystal growth from solution", J. Crystal Growth **99** (1990) 1130 – 1133.

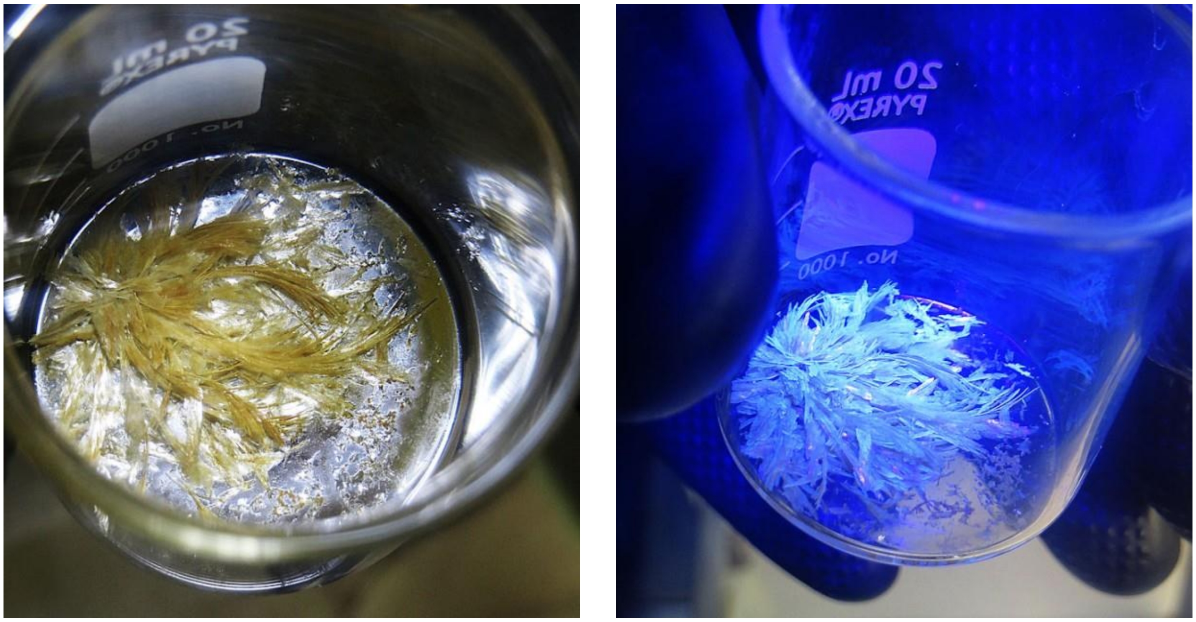

Figure 8. Azaboraanthracene grown from toluene under ambient (left) and UV (right) illumination.

Figure 8 shows the result of azaboraanthracene grown from toluene under ambient (left) and UV (right) illumination. Similar results were obtained with pyridine and ethanol. From the photographs it is clear that no multi-dimensional crystals were formed and either needles were obtained or powder was recovered. This is an indication that the rate of evaporation might have been too high, or that the difference between the dipole moment of the solvent and that of the azaborine compound is too large. Numerous attempts at the crystallization of azaboraanthracene did not yield any crystals of size.

Next we attempted the crystal growth of brominated azaboraanthracene, the precursor to phenylazaboraanthracene and diphenylazaboraanthracene. **Figures 9 and 10** show the result of brominated azaboraanthracene grown from isopropanol and xylene, respectively, under ambient and ultraviolet illumination.

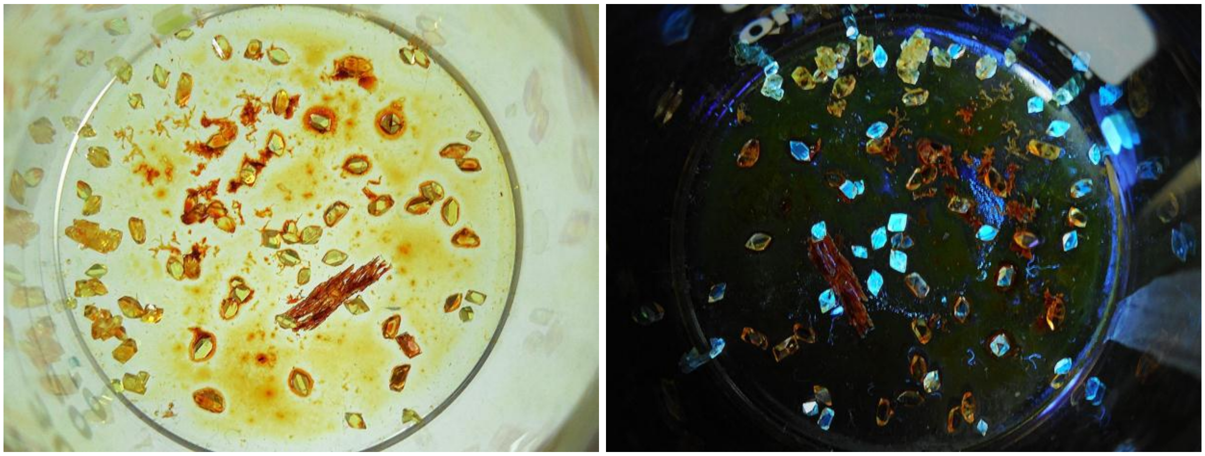

Figure 9. Brominated azaboraanthracene grown from isopropanol under ambient (left) and ultraviolet (right) illumination.

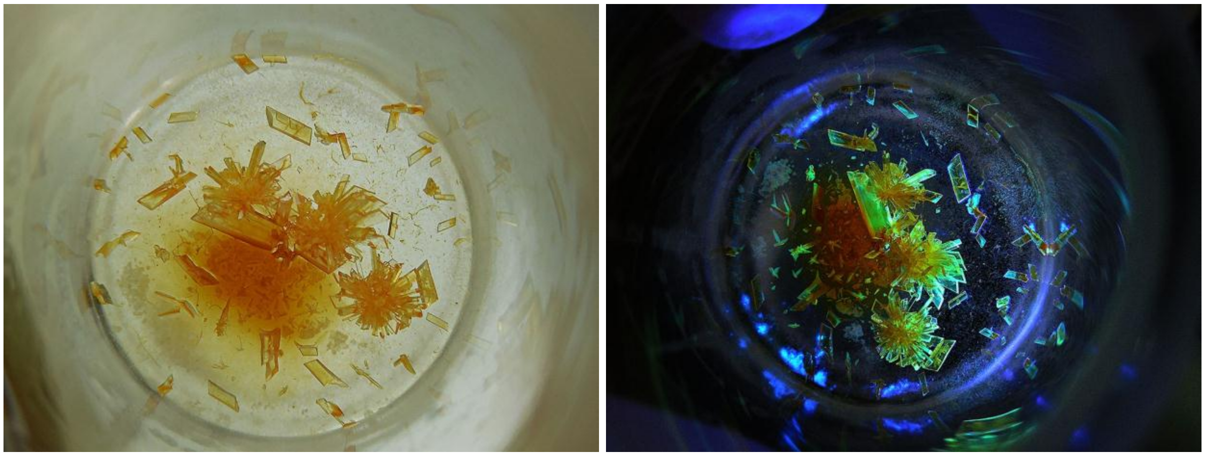

Figure 10. Brominated azaboraanthracene grown from xylene under ambient (left) and ultraviolet (right) illumination.

Brominated azaboraanthracene grown from isopropanol shows defined crystallites that have a hexagonal habitus similar to diphenylanthracene crystals.[5] However, under ultraviolet illumination there appear to be four distinct families of crystallites that have dark red, yellow, cyan and blue coloration under ultraviolet illumination. We surmise that these are formed due to interaction of the solvent with bromo-azaboraanthracene and the presence of impurities in the powder. As will be shown later, only the cyan-colored crystallites show a pulse height spectrum.

Figure 11. Phenylazaboraanthracene grown from toluene.

In contrast to isopropanol, brominated azaboraanthracene crystals obtained from xylene have a platelet-style habitus. And in this case, only two distinct families of crystallites can be distinguished: one that glows dark red under ultraviolet illumination, and one that glows green-blue under ultraviolet illumination. Due to the inertness of xylene, we assume that one of these two families of crystallites is due to impurities in the powder.

Several attempts to grow phenylazaboraanthracene from chlorobenzene, pyridine or toluene only yielded very thin needles or platelets. For instance, **figure 11** shows the result of phenylazaboraanthracene grown from toluene. As can be seen in the figure, the growth beaker is covered with very small and fragile platelets that are rather deliquescent. Attempts to remove the platelets from the beaker resulted in breakage and was therefore unsuccessful.

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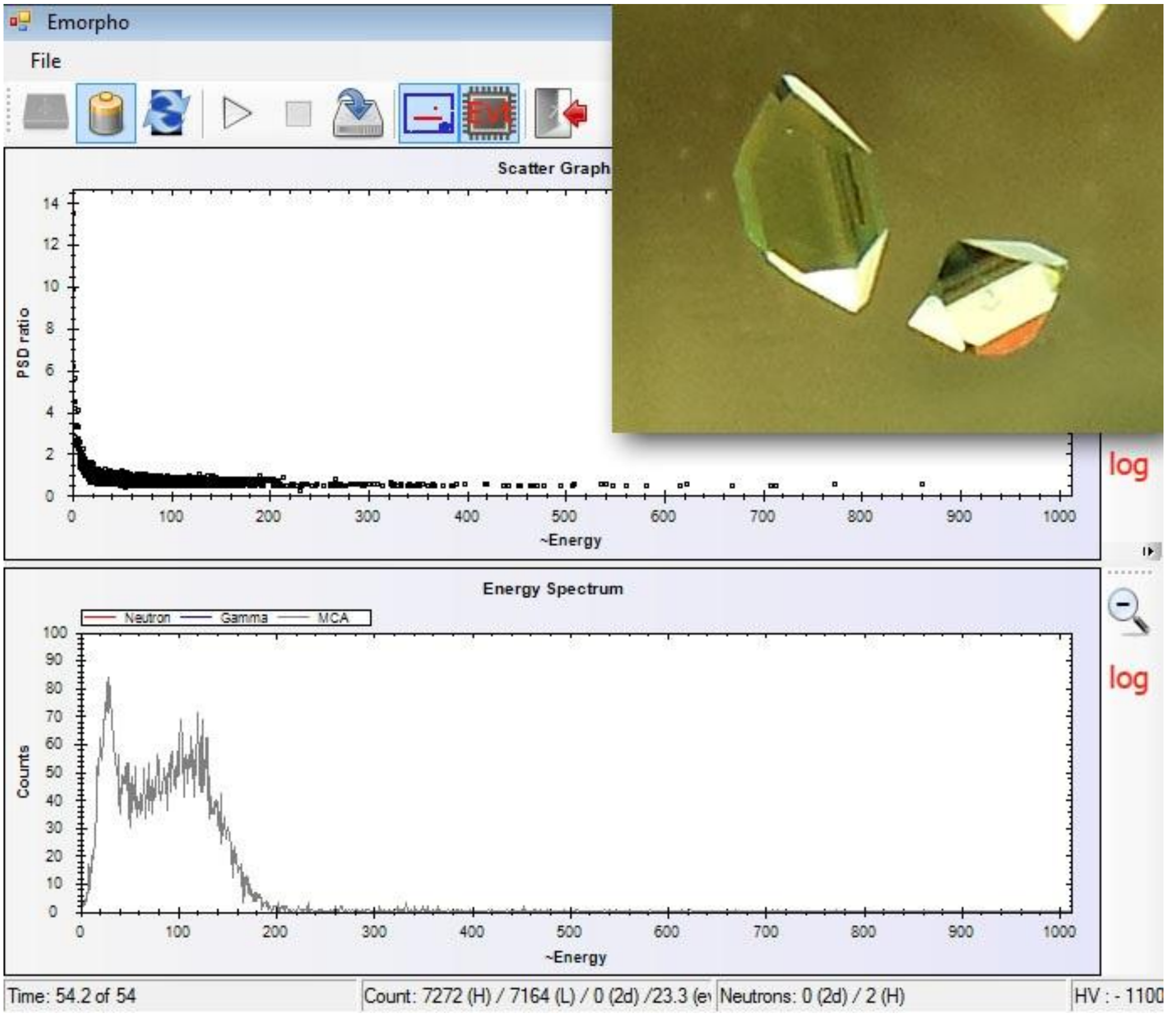
[5] Edgar V. van Loef, Sharmistha Mukhopadhyay, Natalia Zaitseva, Steve Payne, Kanai S. Shah, "Crystal growth and characterization of 9,10-diphenylanthracene," J. Crystal Growth **352** (2012) 103 – 105.

4. Characterization of the Scintillation Properties

When large enough crystalline samples of the azaborines were obtained, we characterized their scintillation properties including emission under X-ray excitation, light yield, and pulse shape discrimination. **Figure 12** shows the pulse height spectrum of bromo-azaboraanthracene under 662 keV gamma-ray excitation. The inset in the figure shows a macro photograph of two bromo-azaboraanthracene crystallites. Note that due to the low density and effective Z of the azaborines, no photopeak is visible in the gamma-ray spectra and only Compton events are present. For brominated azaboraanthracene the Compton Edge can be found at 120 keV cut-off energy.

Continuing with phenylazaboraanthracene, **Figure 13** shows its radioluminescence spectrum. The spectrum is dominated by a broad structured band between 300 and 550nm, peaking at 360 nm. We attribute this band to electronic transitions of the conjugated molecule. Due to the very broad nature of



Time: 54.2 of 54

Count: 7272 (H) / 7164 (L) / 0 (2d) /23.3 (e

Neutrons: 0 (2d) / 2 (H)

HV : - 1100

Figure 12. Pulse height spectrum of bromo-azaboraanthracene under 662 keV gamma-ray excitation.

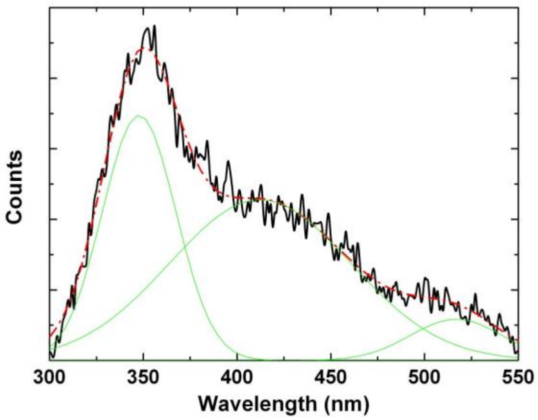


Figure 13. Radioluminescence spectrum of phenylazaboraanthracene.

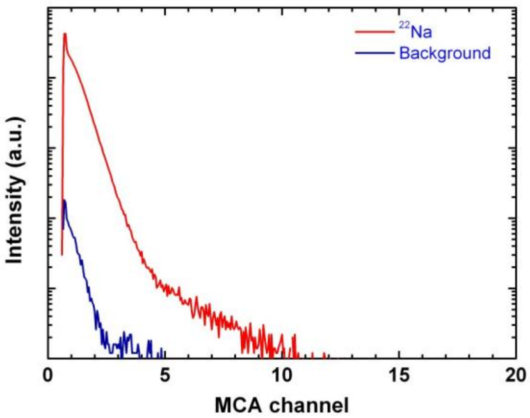


Figure 14. Pulse height spectrum of phenylazaboraanthracene.

this band, we believe that in fact it is composed of multiple emissions centered at 348 nm, 412 nm, and 517 nm, respectively. In the figure this is shown by the green Gaussian curve fits. Note that these emissions corresponds relatively well with the fluorescence spectrum shown in **Figure 4**. The emissions can probably be ascribed to a combination of $\pi^* \rightarrow \pi$ (slow) and $\sigma^* \rightarrow \sigma$ (fast) transitions.

z`When the same needles were exposed to gamma-ray sources (^{22}Na and ^{137}Cs) scintillation was observed. Exposure to a ^{22}Na source and collecting the scintillation light yielded the pulse height spectrum shown in **Figure 14**. These measurements involved optical coupling the crystal to a photomultiplier tube (Hamamatsu R2059), irradiating the material with 511 keV gamma-rays from the ^{22}Na source and recording the resulting pulse height spectrum. Background measurements were collected employing the same setup but without using the ^{22}Na source. The results are shown in **Figure 14**, with the red curve denoting the gamma-ray pulse height spectrum and the blue curve the background spectrum. Note that although the light yield of **3b** is relatively low, the signal can clearly be discerned from the background.

VI. Training and Professional Development

A large part of the effort was executed in collaboration with the team of Professor Liu where two graduate students were engaged to work exclusively on the azaborine synthesis. Laboratory training and developing the synthetic chemistry of the azaborine scintillators was their main task. To aid the students in their effort, internships were created at Radiation Monitoring Devices, Inc. (RMD) where students were taught about radiation detection, crystal growth, and characterization of scintillation properties, while exposing the student to a business-like environment.

Several internships were held over the course of the 3-year program with the final internship planned for June 2014. Interns at RMD are fulltime employees and be paid a generous $15 per hour.

VII. Presentation and Publications

Two posters were presented by the PI. One at the 2012 SORMA West Conference in Oakland, CA, entitled “Azaborines as Novel Boron-containing Scintillators”, and one at the SCINT 2013 Conference in Shanghai, China, entitled “Novel Organic Scintillators for Fast -and Thermal-Neutron Detection”.

The poster “Synthesis and Neutron Detection of Boron Nitrogen Containing Acenes” was presented by Jonathan Marshall, a student from the group of Professor Liu, at the 2013 American Chemical Society National Meeting & Exposition in New Orleans, LA. Two papers were published by the PI:

- Edgar V. van Loef, Jonathan Marshall, Shih-Yuan Liu, Kanai S. Shah, “Azaborines as Novel Boron-containing Scintillators”, SORMA West 2012 Conference Record, Oakland, CA.
- E. V. van Loef, J. Marshall, L. N. Zakharov, S.-Y. Liu, K. S. Shah, “Novel Boron-Containing Organic Scintillators: Azaboranaphthalene and Phenylazaboranaphthalene”, IEEE Trans. Nucl. Sci. 60 (2013) 952 – 954.

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5. Edgar V. van Loef, Sharmistha Mukhopadhyay, Natalia Zaitseva, Steve Payne, Kanai S. Shah, “Crystal growth and characterization of 9,10-diphenylanthracene,” J. Crystal Growth 352 (2012) 103 – 105.

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