# From Student to Scholar – A 30-year Journey of Transformation

Nicholas H. Snow, Ph.D.
Professor of Chemistry and Biochemistry
Director, Center for Academic Industry Partnership
Interim Director, Office of Grants and Research Services

## Journeys of Transformation

- "Rapid Lifetime Determination"
- "Numerical Extrapolations"
- "Doing Drugs"

# Rapid Lifetime Determination

- Light sticks
- How long do they glow?
- Lifetime

# Seton Hall's First PhD Program

SETON HALL UNIVERSITY

MINUTES OF UNIVERSITY COUNCIL MEETING

1962 REPORT NUMBER 1

The Council discussed the following matters:

1. Proposal for the establishment of a program leading to the Ph. D. in chemistry.

| | |
|---|---|
| Sponsor | Rev. Albert B. Hakim, Dean<br>College of Arts and Sciences |
| Presentation | Rev. Alfred V. Celiano, Chairman<br>Department of Chemistry |

# Seton Hall's First PhD Program

SETON HALL UNIVERSITY

SOUTH ORANGE, NEW JERSEY

REPORT OF THE PRESIDENT

TO

THE BOARD OF TRUSTEES

MONDAY, MAY 6, 1963

EXECUTIVE OFFICE

Very Rev. Msgr. Edward J. Fleming
Executive Vice President

The Executive Vice President, acting as the deputy of the President, has administered duly approved and established policies of the University. His principal function, in accordance with University Statutes, has been the administration of policies proposed by the University Council and approved by the President.

D. The University Council approved a curriculum leading to a Ph.D. in Chemistry, effective September 1963.

# "Light sticks" The Cyalume Reaction

- Mohan, Journal of Chemical Education, 1974

**Arthur G. Mohan**
American Cyanamid Company
Bound Brook, New Jersey 08805
**Nicholas J. Turro**
Columbia University
New York, New York 10027

## A Facile and Effective Chemiluminescence Demonstration Experiment

(1) (a) Rauhut, M. M., *Accounts Chem. Res.*, 2, 80 (1969) and references cited therein; (b) Rauhut, M. M., and Hollyky, L. J., U.S. Patent 3597362, 1971; Hollyky, L. J., U.S. Patent 3709231, 1971; Zweig, A., and Maulding, D. R., U.S. Patent 3729862, 1973; Roberts, B. G., and Rauhut, M. M., U.S. Patent 3700738, 1972.
(2) For a recent general review of chemiluminescence see: McCapra, F., "Progress in Organic Chemistry," 8, 231 1971; Demonstration of chemiluminescence, White, E., J. CHEM. EDUC., 34, 275 (1957); Schneider, H. W., *J. Chem. Educ.*, 47, 518 (1970).
(3) Baker, J. W., and Schumaker, L., *J. Amer. Eng. Data*, 9, 564 (1964).
(4) Rauhut, M. M., et al., *J. Amer. Chem. Soc.*, 89, 6516 (1967).
(5) Reid, W., Donner, W., and Schlegelmilch, W., *Ber.*, 94, 1051 (1961).

[1] "Cyalume" (Trademark of American Cyanamid Company) chemical light available from the Aldrich Chemical Company and the Edmund Scientific and Chemical Dynamics Corporation.

# How long does the light last?

- Cline Love and Ashworth, SHU 1984

Anal. Chem. 1984, 56, 1395-1400

Transient Digitizer for the Determination of Microsecond Luminescence Lifetimes

R. J. Woods, Stephen Scypinski, and L. J. Cline Love*
*Department of Chemistry, Seton Hall University, South Orange, New Jersey 07079*
H. A. Ashworth
*Department of Physics, Seton Hall University, South Orange, New Jersey 07079*

**"Rapid Lifetime Determination – RLD"**

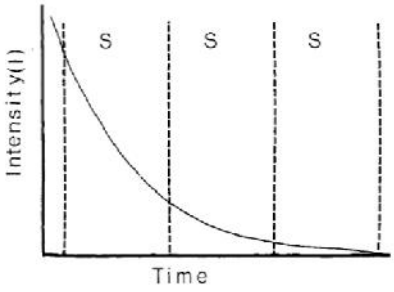
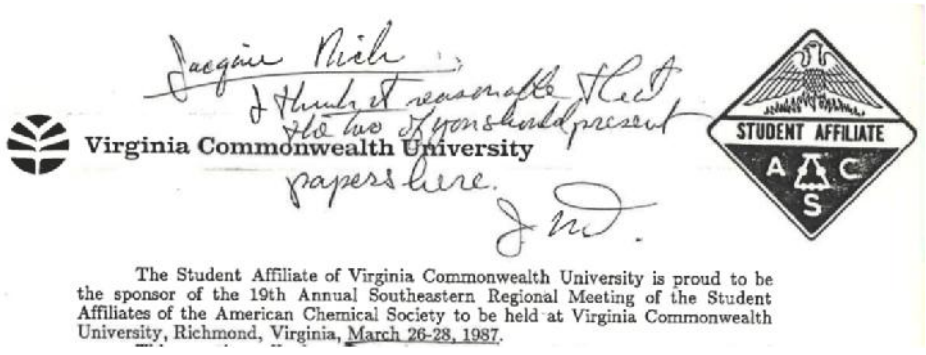



**Figure 1.** Graphical representation of the RLD method.

# Snow's first conference presentation - 1987



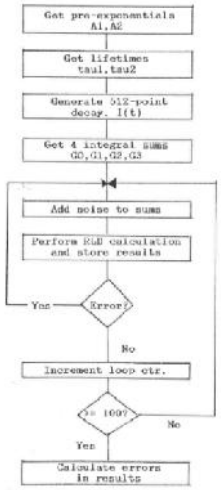

The Student Affiliate of Virginia Commonwealth University is proud to be the sponsor of the 19th Annual Southeastern Regional Meeting of the Student Affiliates of the American Chemical Society to be held at Virginia Commonwealth University, Richmond, Virginia, March 26-28, 1987.

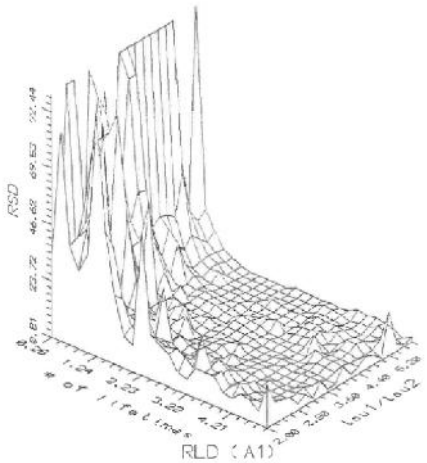
# Snow, ACS 1987

ERROR ANALYSIS OF THE RAPID LIFETIME DETERMINATION METHOD. N. H. Snow, J. N. Demas, University of Virginia, Charlottesville, VA 22901 and B. A. DeGraff, James Madison University, Harrisonburg, VA 22807

Using numerical simulations, we analyzed the speed, precision, and accuracy of the Rapid Lifetime Determination (RLD) method applied to single and bi-exponential decays. We show the ranges of parameters and the fitting regions for which the RLD is useful. The RLD was found to be 100's to 1000's of times faster than nonlinear least squares methods. For single exponential fits the RLD compares favorably in precision with least squares methods, but for biexponential fits, the precision is much poorer. We feel that the RLD will be particularly useful as a fast method for providing seed data for much slower non-linear least squares methods. The speed of convergence of double exponential least squares methods is quite sensitive to the accuracy of the initial parameter guesses, and for the majority of cases the RLD will quickly provide good initial estimates.

# RLD Data - 1987



# Error Analysis of RLD is Published

30 Anal. Chem. 1989, 61, 30–33

## An Error Analysis of the Rapid Lifetime Determination Method for the Evaluation of Single Exponential Decays

Richard M. Ballew and J. N. Demas*
*Chemistry Department, University of Virginia, Charlottesville, Virginia 22901*

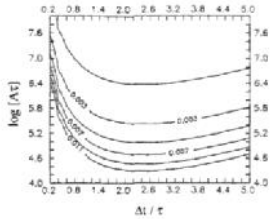



**Figure 2.** Contour map, generated by propagation of errors, of relative standard deviations ($\sigma_\tau/\tau$) in lifetime determination by RLD. Each contour is separated by 0.002.

# Numerical Extrapolations in Gas Chromatography

Journal of Chromatographic Science, Vol. 30, July 1992

## A Numerical Simulation of Temperature-Programmed Gas Chromatography*

**N.H. Snow**
University of Virginia, Department of Pathology, Toxicology Laboratory, Medical Center Box 168, Charlottesville, Virginia 22908
and
**H.M. McNair†**
Virginia Polytechnic Institute and State University, Department of Chemistry, Blacksburg, Virginia 24061

2. **N.H. Snow**, "Determination of Free Energy Relationships Using Gas Chromatography," Journal of Chemical Education, **1996**, *73*(7), 592-597.

## Determination of Free-Energy Relationships Using Gas Chromatography

**Nicholas H. Snow**
Department of Chemistry, Seton Hall University, South Orange, NJ 07079

# RLD - Seton Hall, 1999

## Error Analysis of the Rapid Lifetime Determination Method for Double-Exponential Decays and New Windowing Schemes

**Kristin K. Sharman and Ammasi Periasamy***

*Center for Cellular Imaging, Department of Biology, Gilmer Hall, University of Virginia, Charlottesville Virginia 22903*

**Harry Ashworth***

*Department of Physics, Seton Hall, South Orange, New Jersey 07079*

**J. N. Demas***

*Department of Chemistry, University of Virginia, Charlottesville, Virginia 22904*

**N. H. Snow**

*Department of Chemistry, Seton Hall University, South Orange, New Jersey 07079*

# Retention Projection - 2014

analytical chemistry

Article

pubs.acs.org/ac

## "Retention Projection" Enables Reliable Use of Shared Gas Chromatographic Retention Data Across Laboratories, Instruments, and Methods

Brian B. Barnes,[†] Michael B. Wilson,[†] Peter W. Carr,[‡] Mark F. Vitha,[§] Corey D. Broeckling,[⊥] Adam L. Heuberger,[⊥] Jessica Prenni,[⊥] Gregory C. Janis,[¶] Henry Corcoran,[¶] Nicholas H. Snow,[∥] Shilpi Chopra,[∥] Ramkumar Dhandapani,[∥] Amanda Tawfall,[#] Lloyd W. Sumner,[#] and Paul G. Boswell*[,†]

[†]Department of Horticultural Science, University of Minnesota, 1970 Folwell Ave., St. Paul, Minnesota 55108, United States

[‡]Department of Chemistry, University of Minnesota, 207 Pleasant St. SE, Minneapolis, Minnesota 55455, United States

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[¶]MedTox Laboratories, Laboratory Corporation of America, Holdings, St. Paul, Minnesota 55112, United States

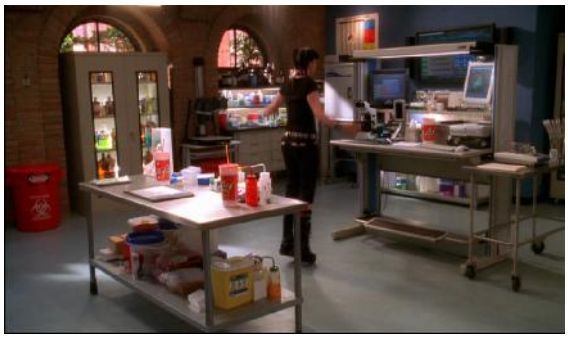
[∥]Department of Chemistry and Biochemistry, Center for Academic Industry Partnership, Seton Hall University, 400 South Orange Ave., South Orange, New Jersey 07079, United States

[#]The Samuel Roberts Noble Foundation, Ardmore, Oklahoma 73401, United States

# "Doing Drugs" by Gas Chromatography

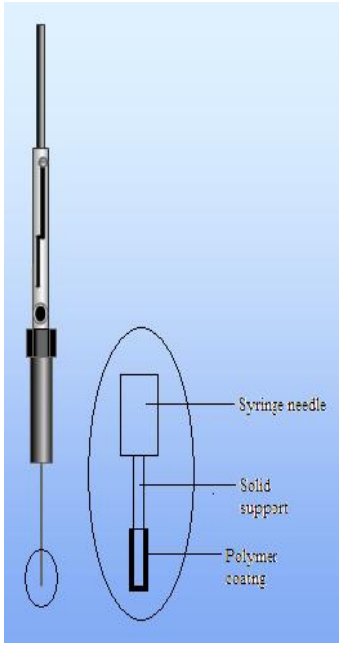
- A story of analytical chemistry, sports and the law

- 1992 – 1993 Postdoctoral work – University of Virginia Department of Pathology
  - DAU confirmations
  - NCIS – "Abby's Lab"
  - Major Mass Spectrometer

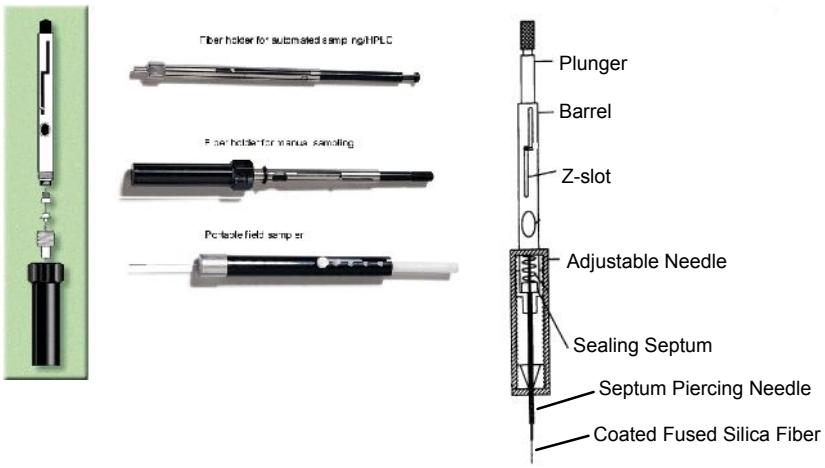


## Solid phase micro extraction

- SPME device consists of a silica fiber coated with  phase material. The fiber is mounted into a microsyringe, which protects the fiber.
- Coating on the fiber is non-volatile polymeric phase, e.g PDMS, PA
- An equilibrium technique - analyte is distributed between the fiber coating and sample.
- After the extraction concentrated analytes are desorbed into the analytical instrument for separation and quantitation.

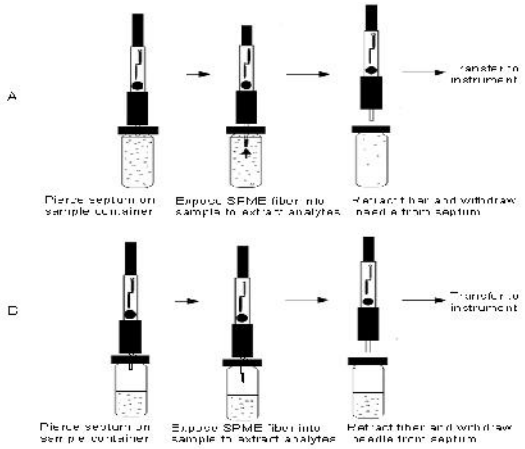


# Solid phase microextraction (SPME)




## Solid phase micro extraction

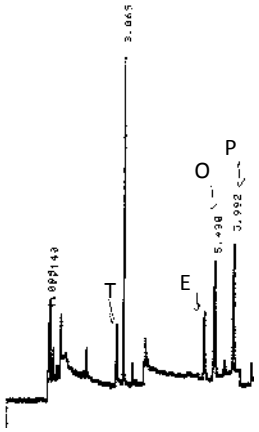
SPME procedures for:
A. direct ext.
B. headspace ext.



The distribution coefficient determines the amount of analyte that is extracted.

$$n = \frac{K_{fs} V_f C_0 V_s}{K_{fs} V_f + V_s} \qquad K_{fs} V_f \prec\prec V \qquad n = K_{fs} V_f C_0$$

# SPME Trace Analysis




400 part per trillion

toluene, ethylbenzene, o-xylene, p-xylene

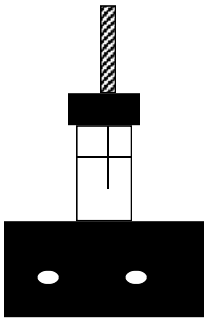
extraction: 3 mL, 30 min, direct

desorption: 220°C, 5 min

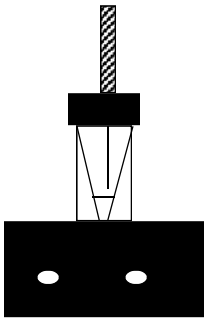
Column: 30m x 0.32mm x 1 mm SPB-1, 40°C (5min), 10°C/min.

Detector: FID, 250°C.

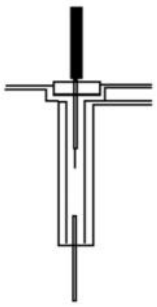
## SPME DERIVATIZATION: *A SIMPLE THREE STEP PROCEDURE*



Extract -
Direct, 30 min

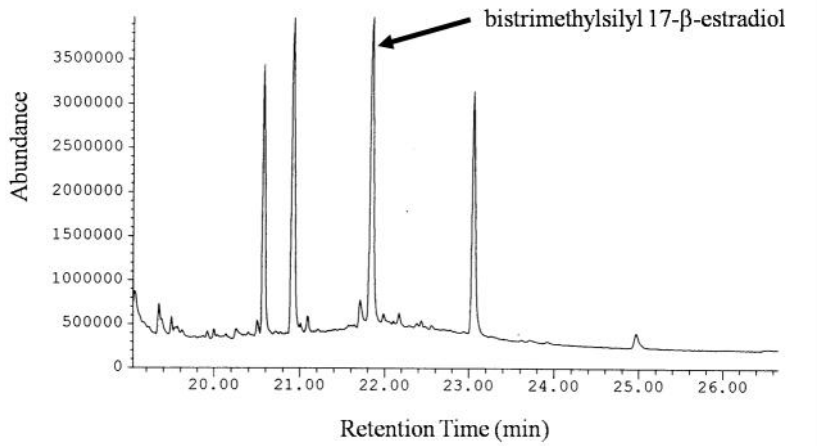


Derivatize-
Headspace, 1 hr.



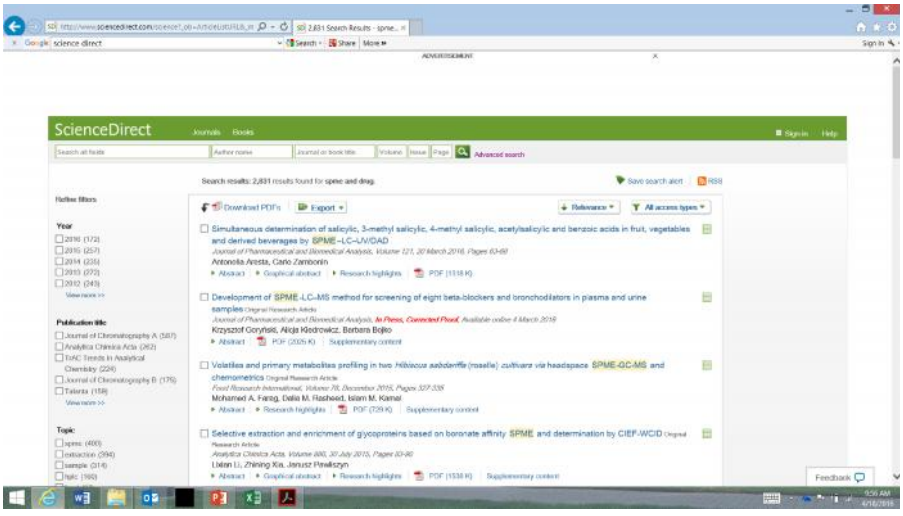
Inject -
Splitless,
280°C

# RESULTS - 17-β-ESTRADIOL
## TOTAL ION CHROMATOGRAM




P. Okeyo, S. Rentz and **N.H. Snow**, "Analysis of Steroids from Human Serum by SPME with Headspace Derivatization and GC/MS" Journal of High Resolution Chromatography, **1997**, *20*, 171-173.

# Science Direct – SPME and Drug



# We're not derivatizing any more…

Trends in Analytical Chemistry

Making the case for QuEChERS-gas chromatography of drugs

Michelle L. Schmidt, Nicholas H. Snow

Journal of Chromatography A

Short communication

Determination of steroids, caffeine and methylparaben in water using solid phase microextraction-comprehensive two dimensional gas chromatography–time of flight mass spectrometry

Paulo C.F. Lima Gomes, Brian B. Barnes, Álvaro J. Santos-Neto, Fernando M. Lancas, Nicholas H. Snow

18 orders of magnitude

Scientia Chromatographica 2014; 6(2):105-116

GC-MS(MS)

Analysis of Steroids using Solid Phase Microextraction-Gas Chromatography-Mass Spectrometry-Mass Spectrometry (SPME-GC-MS-MS)

Shilpi Chopra, Paulo C. F. L. Gomes, Ramkumar Dhandapani, Nicholas H. Snow

Abstract

Direct immersion SPME-GC-MS-MS was used for the analysis of steroids in water at part-per-trillion(ppt) and lower concentrations. The method was validated and extended to real sample analysis. The method were linear from 0.01 to 5 ng/ml with precision less than 10% relative standard deviation for a steroid mixture at 1 ng/ml. Limit of quantitation and limit of detection was found to be 200- 1200 pg/L and 30-200 pg/L respectively and recoveries ranged from 88-103 %. To understand the extraction efficiency of the fiber, a depletion study was performed. The fiber/ sample partition coefficients for the steroids were determined to be $1.0 \times 10^4$ to $1.5 \times 10^5$. The extraction was performed without derivatization or the use of an internal standard. SPME-GC-MS-MS effectively demonstrated ultra-trace level detection of steroids in water.

# A few takeaways

- Science is the most social scholarly pursuit
- A career is in interwoven journey
  - Actions today do impact tomorrow
- Even a “bad” job (or class) can be a great learning experience
- We can measure 18 orders of magnitude of mass

# Movie Quotes

- “We’ll make you better…” Top Gun, 1987
- “You have fungus on your shower shoes…” Bull Durham, 1989
- “Cheater’s justice…” Casino, 1995

# Acknowledgments

1. P. Okeyo, "Solid Phase Micro-extraction: Optimization and Interface with GC/MS for Trace Analysis of Steroids in Biological Fluids" May, 1997
2. T. O'Brien, "Insight into the Selectivity of Molecular Imprinted Polymer Phases" May, 1999.
3. S. Patel, "Analytical SFE: Optimization and Applications" May, 1999.
4. C. Bitzer, "Fundamental Capillary Electrophoresis: An Evaluation of Electrokinetic Sampling" May, 2000
5. K. Flood, "Cyclodextrin Inclusion Complexes of Betamethasone-related Steroids Studies Using HPLC" August, 2000.
6. K. Karasz, "Determination of Degradation Products of Cosmetics and Toiletries Preservatives" August, 2000
7. W. Fish, "Characterization and Design of a Molecular Imprinted Polymer for Cinchona Alkaloids" May, 2003.
8. J.K. Lokhnauth, "Sorptive Micro-extraction coupled to Ion Mobility Spectrometry", May, 2005.
9. I.L. Kazakevich, "Mechanism of Ion-pair Chromatography and Retention Behavior of Lipophilic Ions", December, 2005.
10. B.J. Forsatz, "Understanding Charged Aerosol Detection with High Performance Liquid Chromatography" August, 2007.
11. G.P. Bullock, "Measuring Partition and Activity Coefficients using Headspace Gas Chromatography" May, 2009.
12. B.B. Barnes, "Analysis of Street Drugs Using comprehensive Two-dimensional Gas Chromatography", May, 2012.
13. F. Tavlarakis, "Chemical Analysis of Opthalmic Solutions", May, 2012.
14. K. Dhandapani, "Ionic Liquids in Microextractions and Gas Chromatographic Stationary Phases", May, 2014.
15. S. Chopra, "Extending the Limits of Solid Phase Microextraction", August, 2014.
16. Michelle L. Schmidt, QuEChERS-Gas Chromatography of Drugs, May, 2015

**Research Students (undergraduate)**

| | |
|---|---|
| Maureen Dunn | Class of 1996 |
| Shannon Berg | Class of 1996 |
| Diane Johnson | Class of 1997 |
| Carlos Santos | Class of 1997 |
| Michele Pereira | Class of 2002 |
| Gisela Rios | Class of 2004 |
| Jember Trinidad | Class of 2004 |
| Nima Ansari | Class of 2006 |
| Maudelle Drawer | Class of 2008 |
| Jessan Sintx | Class of 2010 |
| Catherine Andreski | Class of 2010 |
| Leanne Mocniak | Class of 2016 |

**Freshman Research Projects Supervised**

Leotha Francis (1995)
Carlos Santos (1995)
Michelle Vincent (1996)
Shelley Bennison (1997)
Carlos Escobar (1998)
Michele Pereira (1999)
John McCormick (2000)
Nima Ansari (2004)
Eric Moore (2006)
Jessan Sintx (2007)
Catherine Andreski (2007)
Erin Hartman (2009)
John Ann Mobley (2009)

**Secondary Students Supervised Under Project SEED**

Tricia Huggins (1996,1997)
Nicole Quellis (1998, 1999)
Ashley Perez (2007)
Ramon Cruz (2008, 2009)
Yannette Perez (2009)
Monique Inonbolique (2013-14)
Abdul Fraser, (2014)
Badjessa Bilmouda (2014, 2015)
Danielson Joseph (2015)

| Name | Year | Status | Expected completion |
|---|---|---|---|
| Vrushali Bhawtankar | 5th | Matriculated/FT | 2015 |
| Thomas DelMastro | 5th | Matriculated/PT | 2017 |
| Arsu Apedo | 7th | Matriculated/PT | 2016 |
| Michael Sidhersingh | 6th | Matriculated/FT | 20160 |
| Cierra Green | 6th | Matriculated/FT | 2015 |
| Anumeha Muthal | 2nd | Non-matriculated/PT | 2017 |
| Shipra Patel | 2nd | Non-matriculated/PT | 2018 |
| Nicole Haemuth (MS) | 2nd | PT | 2015 |

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