UNIVERSITY OF RHODE ISLAND
Department of Chemistry
SEMINAR

2:00 PM, Friday, April 16, 2021
Please email dathomas@uri.edu for link

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Ph.D. Seminar
Chemistry Department
URI

"Analytical Methods for Tracing Illicit Picric Acid Synthesis"

HOST

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Analytical Methods for Tracing Illicit Picric Acid Synthesis

Home-made explosives (HME) have become a growing threat in recent years, and due to the wide range of explosive material functionality, it is difficult to prevent their illicit preparation. Tracking the production of HMEs from a synthetic method or starting material is of critical importance to law enforcement. In this work, the explosive compound 2,4,6-trinitrophenol (picric acid) was prepared using several synthetic routes. Both laboratory-grade chemicals and over-the-counter-style reagents were used to produce picric acid. Using consumer-grade chemicals and procedures allows for a controlled recreation of real-world clandestine conditions. For this study, such conditions were replicated by using over-the-counter sources of salicylic acid, sodium salicylate, and acetylsalicylic acid. These compounds were nitrated using either concentrated nitric acid or consumer-available nitrate salts. More traditional reactions were also tested, starting with phenol and 2,4-dinitrophenol to act as controls. Recovered products were analyzed using spectroscopic techniques (Raman and IR), differential scanning calorimetry (DSC), powder X-ray diffraction (XRD), and X-ray fluorescence (XRF). Identification of impurities or differences in the isolated products provide insight into the method or starting material used in the synthesis of picric acid. For compounds synthesized from clandestine starting materials, unique bands in the carbonyl stretching region of the IR and Raman spectra indicated the presence of remaining impurities not found utilizing traditional laboratory synthesis. In addition, metals from nitrating salts were seen using XRD and XRF, indicating the formation of highly sensitive metal picrates. General impurities and lesser nitrated species in the recovered samples were seen in the thermograms, giving insight to the purity of the recovered material. Once recrystallized the exothermic decomposition of picric acid changes allowing a reliable distinction between recrystallized and crude materials. Overall, the analysis presented here provides an improved assessment of the preparation method and starting materials used to create picric acid, enabling the synthesis condition to be identified by the impurities of the product.