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SPECIAL COMMUNICATION

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Dr. Walter C. McCrone's Contribution to the Characterization and Identification of Explosives*

ABSTRACT: Dr. McCrone was an amazing individual, possessing many talents and having many interests. He especially loved applying polarized light microscopy (PLM) to answering the question-at-hand and solving problems. He applied PLM to many different fields including the identification of air pollution particles, asbestos identification, art conservation, pharmaceuticals, industry problems and forensic sciences. A field that I believe he enjoyed the most was the characterization and identification of explosives. Throughout his life he worked on, gave presentations and published articles on the characterization and identification of explosives. Also, he encouraged other scientists to give presentations and publish on the subject by providing "behind the scene" advice and/or be a co-author on a paper. He unselfishly taught others how to apply PLM and incorporate this invaluable tool into their analytical scheme.

KEYWORDS: Dr. Walter C. McCrone, McCrone, polarized light microscopy, explosives, fusion methods, crystallographic and optical properties

Dr. McCrone loved applying polarized light microscopy (PLM) to solving the problem-at-hand. This is why he and his microscope were well suited for the characterization and identification of explosives. Dr. McCrone's ability to characterize and understand the behavior of chemical compounds by PLM is what got him involved with explosives while a graduate student at Cornell University in the early 40's. A sample of RDX was given to him to characterize and Dr. McCrone quickly determined by PLM that it was composed of approximately 90% RDX along with another component. The other compound turned out to be HMX, a high explosive in its own right. RDX was believed to be safe to produce in large quantities but the presence of the unstable crystalline form of HMX was responsible for several explosions of the RDX compound. Continued work revealed that a new process of producing RDX was responsible for the presence of HMX as a by-product. Further work by Dr. McCrone showed there were four polymorphic forms of HMX and two polymorphs of RDX. Only the stable form of RDX could be produced on a large scale but three of the four forms of HMX could also be produced during the large-scale production of RDX. Two of these forms were extremely unstable and rivaled the sensitivity of lead azide. Dr. McCrone helped develop a process for the large-scale production and plant monitoring of RDX with the stable crystal from of HMX.

While at Cornell, Dr. McCrone and co-workers Jack Andreen and Sien-Mao Tsang characterized other high explosive compounds and the outcome of their work was the "bible" on the subject entitled "The Microscopic Examination of High Explosives and Boosters" published August 1, 1944. The wealth of information on the crystallographic and optical properties of the high explosives contained within this report is just astonishing. Dr. McCrone described it as "the most interesting two years of his life".

Dr. McCrone continued to work on explosive compounds after leaving Cornell. This on-going work led to an interesting incident late one night in his apartment in Chicago. Lucy was out of town and Dr. Jack Dodd, a good friend and professional associate of the McCrones, was visiting on business. Dr. McCrone was working on a project to grow single, large crystals of lead azide. Dr. Dodd, staying in the guestroom, was awakened in the early hours of the morning by a noise. He saw Dr. McCrone running down the hallway to the room where the lead azide experiment was being conducted, whereupon he discovered a small explosion had taken place. Since there were some un-reacted lead azide crystals scattered about the room, he and Dr. McCrone had to clean-up and neutralize the remaining lead azide before another mishap occurred. Dr. Dodd has vivid memories of the incident and in his own words "The sight of two naked (neither of us wore pajamas) middle-aged men sweeping oil and water from the floor into a dustpan is an image, not recorded except in my memory, that will stay with me until I die". It was learned from that event why lead azide spontaneously detonates during crystal growth and how to control the phenomenon. As it turns out, strain develops during crystal growth and is relieved by spontaneous fracturing. If the crystal is large enough, sufficient energy will be released to cause detonation. It was found that by raising the mean temperature of the seed crystal or by slowing the growth, or both, one could give the crystal strains time enough to anneal thus postponing the inevitable event. The result was larger single crystals could be grown and in Dr. Dodd's words, "In the world of experimental science nothing goes to waste – no experiment is a failure. It is at least a data point."

One of Dr. McCrone's favorite techniques for the characterization of explosives was the fusion method. He published his first book "Fusion Methods in Chemical Microscopy" in 1954. The

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FIG. 1—*Fusion preparation of TNT viewed with crossed polars.*

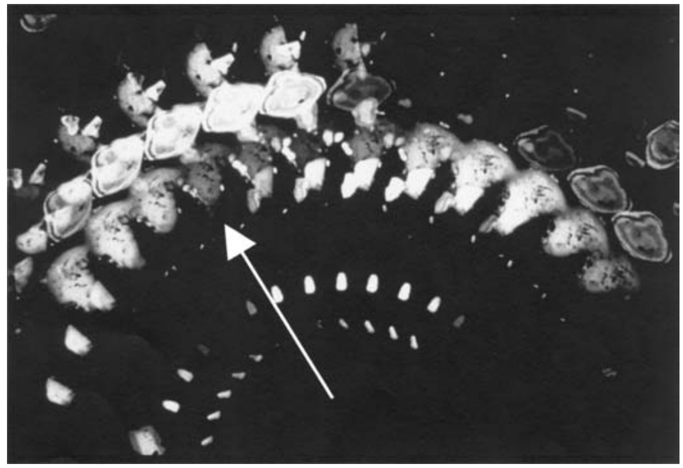

FIG. 2—*A photomicrograph of RDX by Dr. McCrone showing anomalous extinction due to the strong dispersion of the optic axes. The photomicrograph is a series of 12 exposures with the stage being rotated several degrees between each exposure. Anomalous extinction is observed when a crystal is oriented so you are looking more or less parallel to the dispersed optic axis. Under crossed polars, a crystal properly oriented will not show "good" extinction during rotation of the stage but will show an array of blue-purple-yellow colors (arrow). This feature is very distinctive for RDX. The RDX crystal above the crystal displaying anomalous extinction is oriented so you are not looking down the dispersed optic axis and therefore shows good extinction.*

book contains data on explosives as well as other compounds. No one could take a PLM course from Dr. McCrone without a "fusion methods" lecture with his special insight to phase diagrams. He then would allow the students to prepare some fusion preparations of TNT and let them watch with amazement as the crystals develop from the melt (Fig. 1).

Throughout his life, Dr. McCrone published articles, co-authored articles, and encouraged other microscopists to publish articles on the characterization and identification of explosives by PLM. Some of his last articles on the subject were the "Identification of Organic High Explosives" provided in three parts. These articles were published in *The Microscope* and were up-dates of some of the original work he did at Cornell University. Everyone knows that Dr. McCrone was not a selfish person but dedicated his career to teaching others how to observe, understand, and solve problems using PLM. His lectures would always start out simple and he would describe crystal systems, the arrangement of atoms, crystal form, typical crystal distortions, and crystal habits and then provide identification characteristics based on the optical properties. He would always demonstrate the wealth of information that one can "glean" from a PLM analysis and share with us any unique identification features of a compound as shown in Fig. 2. For all his contributions he provided to the scientific community I know we all thank him and will continue to "spread the word" on the analytical value of PLM.

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