A151 Analysis of Trace Hydrogen Peroxide by HPLC-ED and HPLC-FD

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After attending this presentation, attendees will have be introduced to new methods for the detection of trace levels of hydrogen peroxide.

This presentation will impact the forensic community by serving the advanced level of knowledge in the field of post-blast crime scene investigation.

Recently, there have been an increased number of terrorist attacks which utilize improvised explosive mixtures. Some of these mixtures contain concentrated hydrogen peroxide mixed with a carbonaceous fuel source. When these materials are combined in the correct proportion, they can be detonated. It is therefore desirable to have methods in place which can be used to detect trace amounts of hydrogen peroxide which may be present at a post-blast crime scene.

Two methods have been developed for the analysis of hydrogen peroxide: high performance liquid chromatography with electrochemical detection (HPLC-ED) and high performance liquid chromatography with fluorescence detection (HPLC-FD). The HPLC-ED method is a direct method, meaning that hydrogen peroxide is detected as is, without further treatment of the compound. In contrast, the HPLC-FD method is an indirect method. Hydrogen peroxide is detected after interacting with a hemin enzyme and p-hydroxyphenylacetic acid in a post-column reaction. The result of this interaction is the formation of a fluorescent dimer which can readily be detected with a standard fluorescence detector. Given that neither of these HPLC methods alone permits unequivocal detection of hydrogen peroxide, concomitant use of these two analytical approaches provides a greater level of certainty.

For the HPLC-ED system, the parameters which required optimization included the flow rate and composition of the mobile phase, the column packing material, the mode of detection, the flow cell settings, and the temperature of the column and flow cell. It was theorized that optimal detection of peroxide in a matrix environment would occur when the detector was operated in pulsed amperometric detection (PAD) mode, as this mode of detection minimizes the build-up of insoluble material on the surface of the working electrode. For the HPLC-FD system, the parameters which required optimization included the flow rate and composition of the mobile phase, reagent solution, and base solution, the column packing material, and the wavelength settings of the fluorescence detector. The set-up of the HPLC-FD system was complicated, involving three pumps, three reagent solutions, and a post column reactor. Following method optimization, the linear range of the HPLC-ED system was 50 ppb to 25 ppm hydrogen peroxide. The HPLC-FD system was able to detect hydrogen peroxide over the range of 100 ppb to 5 ppm; the fluorescence detector became saturated at peroxide concentrations greater than 5 ppm. Initial studies have demonstrated that both methods are robust, with neither method readily affected by matrix components.

A limited series of field tests using improvised explosive mixtures were conducted to determine the ability of the HPLC-ED and HPLC-FD

methods to detect trace levels of hydrogen peroxide. Application of the methods to the analysis of a limited number of post-blast samples resulted in positive detection of hydrogen peroxide on post-blast material aged nine months. Additional studies will test various types of materials including metal and plastic fragments, metal witness plates, and cotton swabs. Some of these materials may be more successful at retaining trace levels of hydrogen peroxide than others; this may influence what materials/evidence will be collected by investigators at post-blast crime scenes.

Peroxide, Explosives, HPLC

 ** Presenting Author*