A novel high-energy explosive material, FOX-7 (1,1-diamino-2,2-dinitroethylene), was studied using a combination of laser-induced breakdown spectroscopy (LIBS) and selected ion flow tube mass spectrometry (SIFT-MS). The LIBS technique uses short laser pulses (an ArF excimer laser) as the energy source to convert small quantities of a sample into plasma and to induce the emission of its molecular fragments or atoms. SIFT-MS is a novel method for absolute quantification based on chemical ionization using three reagent ions, with the ability to determine concentrations of trace gases and vapors of volatile organic compounds in real time. SIFT-MS was used to study the release of NO, NO2, HCN, HONO, HCHO, CH3CH2OH, and C2H2 after laser ablation of the explosive compound FOX-7 in solid crystalline form. The radiation emitted after excitation was analyzed using a time-resolved UV-vis spectrometerwith an ICCDdetector.The electronic bands ofCN(388nm),OH(308.4 nm), andNO(237.1 nm) radicals and the atomic lines of C, N, and H were identified.