Predicting the Utility of Non-Resonant Inelastic X-ray Scattering (NRIXS) for Standoff Explosives Detection

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Background

The research reported here was a component of a three-year program at the University of Washington to improve fundamental knowledge that is important for developing new technologies and approaches to counter improvised explosive devices (IEDs). The focus of our component was to develop methods for identifying explosive materials using X-ray spectroscopy.

All chemical explosives store energy in specific, high-energy chemical bonds. Detecting and classifying explosives is a matter of analyzing the structure of these bonds by some method, either direct or indirect. One direct method is to measure the X-ray emission spectrum of the relevant elements involved in the chemical bonds, such as nitrogen or oxygen in a typical explosive. A complementary technique is provided by a particular type of non-resonant inelastic X-ray scattering (NRIXS).

Introduction

Non-resonant inelastic X-ray scattering (NRIXS), also referred to as Compton scattering, was first observed by Arthur Holly Compton in 1923. He was awarded the Nobel Prize in Physics in 1927 for the discovery. NRIXS is distinguished from resonant inelastic X-ray scatter (RIXS) by the fact that the incident X-ray energy is well away from the binding energy of any of the electronic levels in the scattering atom. The energy of the scattered X-ray is shifted downward by an amount that depends on both the incident energy and the scattering angle. This energy shift is referred to as the Compton shift. Compton scatter only occurs from electrons that are not bound to the atom, or are bound weakly. Weakly bound electrons can scatter when the Compton shift is larger than the binding energy of the electron. This dependence on binding energy gives NRIXS access to the electronic structure of the scattering atom. At sufficiently high energy resolution (for both the incident and scattered X-rays), absorption features in the NRIXS spectrum can be observed and used to investigate chemical effects in a material [Fister et al., 2006].

The incident X-rays in NRIXS can have very high energies and therefore can penetrate many materials (such as suitcases, soil, etc.). The binding energies investigated, however, are at energies comparable to the Compton shift, which is much less than the incident energy. This allows access to the electronic transitions of the light elements, such as carbon, nitrogen, and oxygen that are typical components of explosives. The electronic binding energies of these light elements are below 1 keV. X-rays of this low energy cannot penetrate air for any significant distance. The unique combination of penetrating power and chemical information about light elements makes NRIXS a good
candidate for finding and identifying explosives within containers, buildings, or other concealment.

NRIXS, however, is a weak effect. Its cross-section is small compared to the photoelectric effect (at the energies of interest here). It must be measured at high resolution in both angle and energy to provide the necessary chemical information. Thus a doubly-differential cross-section must be measured. Because the count rate is proportional to the resolution, and a resolution of about 1 eV is required, the count rates are very low. Its intensity and practicality must be investigated for realistic situations and feasible hardware. It is worth investigating because NRIXS is the only approach that combines sensitivity to the low atomic number elements in explosives with the penetrating power of high-energy X-rays.

To investigate the feasibility of using NRIXS to detect and identify explosives, we have attempted to predict the signal to noise from NRIXS for typical applications. To achieve this, a new Atomic Inelastic X-ray Scatter (AXIS) computer code was developed, tested, and its accuracy verified by synchrotron and laboratory measurements.

Explosives are unique in that they typically have a very high density of nitrogen (employed chemically to bind the oxygen needed for the explosive reaction while yielding some additional energy during its release). For this reason, we focused on investigating the nitrogen K absorption edge. The absorption fine structure at this edge is related to the local chemical bonds and should provide a unique signature for explosive compounds.

The characteristic X-rays from nitrogen at 392 eV are considered low energy or soft X-rays. They do not pass through air, soil, plastic, or any housing of an explosive device. For standoff detection of an improvised explosive device or bomb contained in a suitcase, for example, high-energy X-rays are needed to penetrate the enclosure. However, for the chemical information necessary to prevent false positives, one needs access to the nitrogen electronic states. NRIXS is ideal in that it provides both of these requirements, if it provides enough signal to work in a realistic scenario.

As an example of a possible application scenario, one would first acquire a backscatter image. From this image, one could focus on any suspicious portion of the image. The NRIXS spectrum for this spot could be measured in a reasonable time (say 100 seconds). From this spectrum, one would then determine the chemical information about the material in the spot and identify it as explosive or not. Because NRIXS is a weak effect, the main question is whether it can provide enough signal to get a spectrum in a reasonable time and with enough energy resolution to access the nitrogen absorption features.

Figure 1 shows an inelastic scatter spectrum from boron nitride taken with a monochromatized synchrotron source and very high resolution (about 1 eV) for the
scattered radiation. The monochromator for the scattered radiation operates at a fixed energy of 9.9 keV and the incident energy varies. The inset shows the same spectrum plotted as the energy loss — the difference between the incident beam energy and the scattered beam energy. The momentum transfer direction in the scatter is parallel to the c-axis of the boron nitride crystal. Note the absorption edge features for the boron and nitrogen K edges at 195 and 404 eV, respectively. It is these features that provide the chemical information in NRIXS. For reference, the traditional Compton shift energy is at the center of the Compton peak labeled in the figure.

A number of theoretical methods are available to calculate the effect of chemical bonds on the NRIXS spectrum. These methods are based on solid-state physics codes. They fall into two broad categories: momentum space methods (such as band structure calculations) and real space methods. The calculations are very similar to X-ray absorption fine structure (XAFS) calculations. Figure 2 shows examples of the results of these types of calculations for boron nitride. The upper panel is for the region around the boron K edge and the lower panel is for the nitrogen K edge. The points are the measured spectrum from the same data in Figure 1. The blue curve is a band structure method that includes the Bethe–Salpeter approach for unoccupied states above the Fermi level [Soininen, 2001]. This is essential for calculating scattering at energy loss greater
than the binding energy, where the electron is scattered into an unoccupied electronic state. The red curve is a real-space calculation using the FEFF8 code [Ankudinov, 1998; Rehr, 2000]. This code uses a cluster of atoms, in this case 293 atoms. As can be seen in the figure, the chemical effects are well predicted by either of these methods.

![Figure 2. Calculations of the fine structure in NRIXS due to chemical effects. An experimental spectrum from boron nitride is plotted as dots with error bars. Two different theoretical methods are plotted as the colored lines.](image)

However, neither of these codes gives the absolute intensity of the signal. They only provide the structure relative to the atomic absorption edge. The purpose of this study was to develop reliable predictions of the absolute signal level from NRIXS in specific application scenarios.
Approach

First, we measured the NRIXS spectrum of selected compounds using a synchrotron source. The use of a synchrotron provides a strong signal and thus very good signal to noise at very high measurement resolution (better than 1 eV). This yields very good data to evaluate NRIXS and make comparisons to calculations.

Second, we developed a computer code to calculate the size and chemical dependence of the NRIXS scatter cross-section. This new code was named AXIS for Atomic X-ray Inelastic Scatter code. AXIS incorporates a fundamental parameters code to include the effects of self-absorption, absorption in the air path and any other obstructions, and geometry. An algorithm to calculate the absolute output of an X-ray tube was also included. This gives an absolute calculation of the rate of photons expected in the detector.

The calculations were validated using the synchrotron data and additional data collected in the laboratory. The laboratory data did not have the energy resolution to determine chemical effects, but it did validate the absolute intensity of the scatter and the absolute output of the X-ray tube. These validations were important in establishing the reliability of the signal strength predictions.

Finally, we calculated the signal strength for a number of relevant geometries. These geometries were based as closely as could be approximated to application scenarios expected in the field. One scenario focused on typical suitcase inspection that may occur in airports while a second scenario focused on standoff detection from a vehicle.

Experimental Measurements

Powders of NaNO$_2$, NaNO$_3$, hexagonal and cubic BN, and NH$_4$NO$_3$ were obtained from commercial vendors and pressed into pellets. The NRIXS spectra for these compounds were measured using the LERIX (Low-Energy Resolution Inelastic X-ray) apparatus [Fister, 2006] at beamline 20-ID at the Advanced Photon Source [Cross, 2007]. The scattered beam monochromators were operated at an energy of 9.9 keV and the incident energy was varied above this value to measure the energy lost during scatter. Multiple scatter angles (each with a different momentum transfer) were measured simultaneously. The first experiments measured 10 angles simultaneously and later experiments benefitted from the addition of 10 new detectors installed under this project, bringing the total up to 20 simultaneous angles (Figure 3).

Laboratory measurements were made using a small transmission-target X-ray tube with a silver anode (Comet Model MTI-40-2-AG-500). It was typically operated at 35
kV and 10 microamps. The tube had a 3-mm aperture at its output to confine the beam and form a well-defined solid angle acceptance of the tube output. The detector was an Amptek XR-100CR Si-PIN diode collimated to 7 mm$^2$ active area and with 0.4 mm thickness. For direct measurements of the X-ray tube output a pinhole of 0.4 mm diameter was placed in front of the detector and the path from the tube to the detector was filled with helium. The scatter measurements were made in air using Kapton® as the scatter target. This material was chosen because it is similar to explosives in density and contains significant nitrogen.

Figure 3. LERIX non-resonant inelastic X-ray scattering setup at beamline ID-20 of the Advanced Photon Source. This early version measures 10 scatter angles simultaneously as can be seen by the semicircle of scattered beam monochromators at the top of the picture. The incident beam enters from the left and the specimen is at the center of the circle. The scattered beams are diffracted by the monochromator crystals at the top and travel down to a set of detectors just below the center of the apparatus. The backscatter geometry of the scattered beam crystal monochromators gives very high energy resolution for the scattered beams, but at a fixed energy. Energy loss is varied by changing the incident energy using the beamline monochromator.
Calculations

The AXIS calculations begin with the X-ray source, where the code determines the spectrum and intensity output (Figure 4). The code accommodates an X-ray tube or synchrotron source. Because the synchrotron source was used only to verify the high-resolution scatter calculations, it was treated simply as a monochromatic source with the intensity from the beamline characterization measurements. This gave good agreement with the measurements (see Validation, next section). The X-ray tube was treated in more detail because it is the source likely to be used in any realistic scenario. We also wanted to be able to vary the voltage and anode material to optimize performance. For an X-ray tube, the power, anode material, geometry, and vacuum containment window are considered. An algorithm for tube output and new formulae for an end-window, transmission-target X-ray tube developed for this project followed the methods of Ebel [1999].

NRIXS Absolute Intensity Calculation

![Diagram of NRIXS Absolute Intensity Calculation]

Figure 4. Stages included in the AXIS calculation.

Losses in the air path to and from the scatterer (the explosive in actual scenarios) have absorption losses in both directions that are taken into account. Likewise any container for the device will have absorption losses going in and out. These absorption losses are calculated using an exponential dependence where the linear absorption coefficient is calculated from the photoelectric and scatter cross-sections and the density
of the material. Cross-sections were taken from the database of Elam, Ravel, and Sieber [Elam et al., 2002] throughout these calculations.

For realistic scenarios (and for the synchrotron measurements) a NaI scintillator and photomultiplier tube were assumed for the detector. The efficiency for this detector was calculated with a simplified model including only absorption in the active NaI volume. This gives results within about 10% of the actual efficiency. For the Si-PIN diode used in the lab measurements, a complete model with the window transmission, a dead layer, and the active layer absorption were used.

The scattered beam monochromator is the heart of the apparatus for these measurements. To compute response, the solid angle, energy resolution, and efficiency must be included. Its description has the largest uncertainty in the realistic calculations. We assumed a perfect Si crystal that has unit reflectivity within its bandwidth. The validity of this assumption was proven by the agreement with the synchrotron NRIXS measurements.

Estimating the scatter cross-section for the explosive (or simulant in the case of the validation measurements) was the main thrust of this project. A scatter cross-section theory was developed to yield absolute units and enable an absolute calculation of the NRIXS signal. The density and composition of explosives is readily available.

Self-absorption effects were included using the formulas of van Sprang and Bekkers [1998]. The actual scatter calculation was based on the methods of Alm Carlsson et al. [1982]. Numerical values of the doubly-differential Compton cross-section were estimated using a method suggested by Brusa et al. [1996].

**Validation**

A plot of the high-resolution synchrotron measurements of NRIXS for the nitrogen compound sodium nitrate (NaNO₃) shows that the feature due to the nitrogen edge is clearly visible near 400 eV of energy loss (Figure 5). The inset is an expansion of this region. Note the sharp feature at 405 eV below the increase in absorption at about 412 eV. Features of this type can be used to distinguish different chemical forms of nitrogen and can be predicted using the theoretical methods described in the previous section.
Figure 5. High-resolution synchrotron measurements of NRIXS measurements of sodium nitrate.

Similar data were also collected for sodium nitrite (NaNO₂; Figure 6). Both of these measurements were used to validate the absolute signal calculations from the AXIS code. These are *ab-initio* calculations with no adjustable parameters. The agreement in absolute signal level is very good for all momentum transfers (Figure 7).

Figures 8 and 9 compare the details of the calculation and measurement for the features due to the nitrogen and oxygen absorption edges, respectively. These features are reproduced in the calculation with the inclusion of the doubly-differential Compton cross section with the effects of electron binding energies properly accounted for. The amplitude of these features in the calculations also agrees very well with the measurement. Observation of these features is important to enable explosives to be distinguished from common materials.
Figure 6. High-resolution synchrotron measurements of NRIXS for sodium nitrite.

Figure 7. AXIS calculation of the absolute signal level expected in the high-resolution synchrotron measurements of NRIXS of sodium nitrite (Figure 6).
Figure 8. Comparison of NRIXS calculation and measurement for the region of the nitrogen K edge feature.

Figure 9. Comparison of NRIXS calculation and measurement for the region of the oxygen K edge feature.

To provide a more realistic comparison to the kind of apparatus that would be employed in field situations, more validations were performed using an X-ray tube source. Both the direct spectrum of the X-ray tube and the scatter from a nitrogen-containing plastic target were obtained. In these cases the energy resolution is not adequate to observe the features in the Compton scatter from the nitrogen and oxygen absorption edges. However, the physical setup and all other conditions could be controlled carefully. This provided a better opportunity for absolute measurements that could be compared directly with the calculations and obtain more quantitative agreement.
The agreement for the laboratory scatter measurement is good overall, within about 40% relative error, and the prediction of the absolute intensity of the NRIXS scatter peak is excellent. This agreement between the model and the measurements for these cases provides great confidence in the predictions of the signal intensity in the scenarios described in the next section. Figure 10 shows the comparison of the measured and calculated spectrum from the X-ray tube and Figure 11 shows the comparison for the scatter spectrum measured in the lab.

![Figure 10. Comparison of calculated and measured spectrum directly emitted by the X-ray tube source. The peaks between 5 and 8 keV are contaminant emission lines in X-ray tube output, which were not included in the simulation.](image-url)
Investigation of Specific Scenarios

To evaluate the capability of NRIXS to perform the desired detection and identification of explosives in realistic situations, two scenarios were used. The first is typical of suitcase inspection in airports and the second is related to full standoff capability. In the first scenario, only increments to existing technology were included in the predictions. No assumptions of new breakthroughs or emerging technologies were assumed. For the second, more demanding scenario, some emerging technologies were allowed in the simulations.

Suitcase Inspector Scenario

In this scenario a computed tomography scan would first be performed on the item being inspected (Figure 12). This three-dimensional image could be computer processed to identify any volumes that could possibly contain explosives in sufficient quantity to be a threat. Any such volumes would then be investigated by NRIXS to determine their content.

Figure 11. Comparison of calculated and measured scatter from a plastic target using an X-ray tube source.
Only existing technology with some straightforward increments were used to evaluate this scenario. The source was a rotating anode X-ray tube with a uranium target operated at 250 kilovolts and 1 ampere (250 kilowatts). The explosive volume used the RDX composition and was located within a brass container of 0.32 cm thickness. The incident beam and scattered beam monochromators were assumed to be perfect crystals at 1000 cm distance and an effective area of 7800 cm². The scatter angle was 20 degrees and the X-ray detector was a sodium iodide scintillator 10 cm thick. A totally controlled geometry was assumed such that complete access to all scattering angles at once could be employed and the apparatus placed at optimal locations with respect to the item being inspected.

The results of the simulation for this scenario (Figure 13) show that the energy on the abscissa is absolute energy with the incident energy of the uranium Kα characteristic emission line at 98.4 keV. The locations of the three features due to NRIXS of carbon, nitrogen, and oxygen are noted in the figure. With the signal intensities from this simulation, it is estimated that these three elements could be roughly quantified in one second. This would be adequate to eliminate the majority of suspect volumes in typical consumer materials. Any volumes not eliminated by the rough quantification would then be measured for 100 seconds to yield composition to within 1% relative. This would again eliminate many materials. The remaining materials that had composition within 1% of an explosive would be subject to longer analysis to obtain the full NRIXS.
spectrum and positive identification by spectral fingerprint techniques backed up by theoretical calculations.

![Image of a graph showing energy vs. intensity for Oxygen, Nitrogen, and Carbon]

**Figure 13. Results from simulation of the suitcase inspection scenario.**

**True Standoff Scenario**

True standoff capability can only be achieved if the measurements can be made at 60 miles per hour and completed within 100 yards distance from a vehicle. To determine what would be necessary to achieve this, a simulation of this scenario was performed. Sixty miles per hour is about 27 m s\(^{-1}\) and 100 m distance implies about 2 s. For quantitative analysis in the time limit, at least 1000 counts s\(^{-1}\) would be required. The scenario is limited to about 1 m for the size of the detectors, sources, optics, etc. The explosive was assumed covered by a 1/8-in brass container and the backscatter geometry was appropriate for a vehicle-mounted system looking ahead.

Unfortunately the simulation for this scenario indicated it was unfeasible. The signal intensity was on the order of 10\(^{-15}\) photons per second for equipment similar to that for the suitcase scenario above.
Feasible Standoff Scenario

Since the ideal standoff scenario was not feasible, but the suitcase scenario was workable, another series of simulations was undertaken to determine the limits of this approach. These simulations yielded a scenario that is shown via an artist’s concept in Figure 14. Some emerging technologies were allowed for this scenario that were judged to be ready in the next few years.

Figure 14. Feasible standoff inspection scenario.

One major change was necessary to obtain adequate scatter signal for any scenario: a small scatter angle is required. A maximum scatter angle of 20 degrees yields a useable signal. This requires the source and the detector to be located on opposite sides of the area being investigated. However, in this geometry an adequate NRIXS signal could be obtained that allows detection and identification of explosives that are completely concealed inside buildings or improvised devices. Direct measurement of the bulk explosive is obtained.

The X-ray source used for this scenario is called the Mirricle source. This new source is made in Japan as an alternative to synchrotron sources. It is reasonably compact and uses very high-energy electrons to bombard a metal target. The X-ray production mechanism is conventional, just as in an X-ray tube. However, the use of a
compact electron accelerator to produce high-energy electrons to bombard the target allows very high-intensity X-ray production at high photon energies. The manufacturer claims measured intensities of $10^{13}$ photons per second per square milliradian [Mirrorcle, 2007], with design intensities of $10^{15}$. Because the electron energy is well above the pair-production threshold, the X-ray source calculations used here would not give reliable figures for X-ray production intensity for this source. Consequently, the design figure from the manufacturer was used for these calculations. The change to a more forward scattering angle in the AXIS calculations is straightforward.

The other innovation that is necessary for the above scenario is the use of X-ray optics to monochromatize the photon beam. These optics are necessary for the Mirrorcle source, because it produces continuum X-rays over a broad band of energies. To observe the characteristic scatter from C, N, and O in explosives requires at least 5 eV resolution in the energy loss. Thus the incident beam must be monochromatized to this level, as well as the scattered beam. This requires specialized X-ray optics that have both a large collection area and high energy resolution. The large area is necessary to collect a reasonable signal from a scatterer at 100 m distance, simply due to solid angle. This conflicts with the resolution requirement, because the energy selection is achieved by diffraction from a single crystal. The acceptance angles of the crystal to achieve high-energy resolution, as well as crystal defects, preclude the use of very large single crystals. The optic must be a composite of smaller crystals arranged to achieve the required energy resolution and build up a large active area.

Such optics are under active investigation [Nakajima et al., 2004]. They require a significant fabrication effort, so tests on such optics are expensive. Detailed calculations that take into account all aspects of geometric ray tracing and crystal dynamical diffraction are required for accurate estimation of their performance. Such calculations would provide a cost-effective method to allow exploration of possible configurations and how they perform, but were beyond the scope of this work. For the results shown below, the monochromators were again assumed to be prefect crystals with 7800 cm$^2$ active area located at a distance of 10,000 cm. The same detector and other conditions as the suitcase scenario were also used.

The results of the simulation for this scenario are given in Figure 15. Under these assumptions the nitrogen content can be quantified to within 10 percent in a 100-s measurement time. Longer times would give better results.
Figure 15. Simulation results for the feasible standoff scenario. The energy plotted on the abscissa is energy loss referred to the 100 keV incident energy.

Discussion

The results of this work indicate that NRIXS certainly has a role in identifying hidden explosives. Additional research and development are needed before the technique will be ready for applications. Future work may make standoff detection with this technique feasible. It remains the only likely near-future standoff detection technique that will directly identify bulk quantities of explosives hidden underground, in containers, or inside buildings. Areas where improvement is needed to make standoff detection possible are a lighter and more compact X-ray source and better X-ray optics. The restriction to nearly-forward scattering angles remains a significant limitation for this approach.

The suitcase inspector has significantly lower technological risk than standoff detection, but has mostly domestic applications rather than military usefulness. It uses only currently available technology that is pushed a bit further along than is currently available. These advances are rather straightforward and could be implemented with relative ease. This application would, of course, be considerably lower in cost than development of a standoff detection apparatus.

Standoff detection is possible in the limit where a forward scattering geometry is possible, such as in buildings or where the source and detector can be located in separate
vehicles. This possibility has higher risk than suitcase inspection, but also has much higher potential payoff. It requires new technology that would take a significant investment in research and development. Without a major improvement in technology, it would also be large and perhaps unwieldy as well as expensive. This work is aimed at helping evaluate the potential payoffs as well as the risks of developing such a standoff detection system using NRIXS. The calculation tools and methods developed under this project can provide valuable insight as technology improves and military needs evolve.

Two issues need to be addressed by future work. The first is to verify the predicted performance of the high-energy X-ray sources that are required. At X-ray energies of 50–100 keV the conventional formulas published to calculate X-ray tube spectra are not fully applicable and need to be adapted and validated. The actual performance of the novel, compact, high-intensity Japanese X-ray source assumed for the standoff scenario will have to be checked as this source comes to market. Second, novel optic designs and detailed simulations of optic performance will be necessary. Such optics might be based on Laue or Boorman effects and can take advantage of work being done in X-ray astronomy. Absorption edge filter foils coupled with spatial filters have very large areas and have the potential to provide high energy resolution if coupled with derivative approaches.

Theoretical methods to model and predict solid-state effects are developing rapidly and will be important for data analysis. These methods have the ability to replace large measurement data sets for training of spectral fingerprint methods and for identification based on physical understanding of the features in the spectrum. Recent advances in theory can be integrated into the AXIS code developed here. AXIS would also benefit from further improvements in the approximations used to calculate the Compton cross-section. Finally, volume scatter integrals using realistic shapes for the explosive material would produce more accurate and realistic predictions.

Conclusions

An atomic inelastic X-ray scatter (AXIS) computer code was developed and tested. The calculations were absolute in that no adjustable parameters were employed and the actual observed intensity of the signal was predicted. Calculation accuracy was verified by comparison with high-resolution synchrotron measurements as well as laboratory measurements with more realistic sources. Agreement of the absolute signal was within 40% relative error at worst and often much better. AXIS simulations of several scenarios where explosives detection and identification are encountered were also performed.

Our AXIS simulation shows that a suitcase or package inspection system based on non-resonant inelastic X-ray scattering is clearly feasible with existing technology plus
some enhancements that are fairly straightforward. AXIS simulation of a standoff inspector indicates feasibility for distances somewhere between 10 and 100 m with emerging technology that is as yet untested. Standoff detection via this approach is limited to situations were the scatter angle can be 20 degrees or less.
References


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