Development of spectroscopic detection and neutralization strategies for biological threats crossing our border

Michael Pravica, Ph.D.
Professor of Physics
University of Nevada Las Vegas

CBTS Presentation
October 27, 2022
Development of spectroscopic detection and neutralization strategies for biological threats crossing our border

Michael Pravica, Ph.D.
Professor of Physics
University of Nevada Las Vegas
1. We are developing a hybrid Raman and UV/Vis spectrometer for rapid detection of some threats.
2. We have some ideas to share.
3. We are also developing ideas pertaining to eradication of immediate threats.

We examined 4 types of threats:

a. Viral (Tobacco Mosaic Virus/TMV)
b. Bacterial (K12 Ecoli)
c. Mold (Yeast)
d. Chemical/poison (ibuprofen, acetaminophen, children’s aspirin, e. pure aspirin, nicotine)
There are largely two types of detection strategies:

A. Detection via chemical alteration/reaction (e.g. PCR); i.e. ACTIVE

B. Detection via minimal or no chemical alteration (e.g. Raman/IR); i.e. PASSIVE

ACTIVE examples:

Factors to consider/boundary conditions:

1. Does the interrogation method damage the sample?
2. Is it dangerous to the operator?
3. How much training is required for the operator?
4. How long does it take to perform?
5. Expense
6. Reliability/ruggedness of equipment
7. Possibility for remote detection?
8. Reagents/chemicals/repeated measurements/supplies
9. Measurements in extreme conditions (e.g. high temperature)

We decided to focus on PASSIVE/spectroscopic methods for rapid testing.
THE ELECTROMAGNETIC SPECTRUM

Energy (eVs)→

https://socratic.org/questions/what-is-the-electromagnetic-spectrum-used-for
Spectroscopic methods we tried:

1. Raman spectroscopy
2. UV/Visible absorption spectroscopy
3. NMR
4. Cyclic voltammetry
5. Fluorescence spectroscopy
Raman spectra of aspirin (top), acetaminophen (2nd from top), and ibuprofen (3rd from top) powders. The lower 2 traces are Raman spectra of various mixtures of these 3 constituents.
UV/Vis absorption spectra

- Acetaminophen UV/Visible spectrum from Q-tip
- K12 Ecoli bacteria UV/Vis spectrum
- Uracil UV/Vis spectrum
- Tobacco Mosaic Virus (TMV) UV/Vis spectrum
- Torula yeast RNA UV/Vis spectrum
- Herring sperm DNA UV/Vis spectrum
Proton NMR spectra of yeast (left) and TMV (right).
One suggested approach:

Raman $\rightarrow$ UV/Vis $\rightarrow$ NMR $\rightarrow$ ACTIVE
Portable Raman spectrometer (example)

Technical Information

ACE-ID™
NON-CONTACT EXPLOSIVES & NARCOTICS IDENTIFIER WITH ORS TECHNOLOGY

ACE-ID is a next-generation, handheld Raman identifier for explosives and narcotics that analyzes solids, powders, liquids, and water based solutions as well as performs moisture analysis.

Utilizing Raman spectroscopy, ACE-ID enables non-contact analysis, yielding rapid results in seconds. Materials can be identified through translucent and semi-translucent containers such as plastic and glass. In addition, analysis is also supported by a software kit for remote operation.

ACE-ID is MIL-STD-810G compliant for rugged use in harsh conditions and operation in extreme temperatures (-20C to +50C). It is lightweight and can be operated with just one hand.

An intuitive software interface guides users through the entire identification process making it easy-to-use with minimal training.

ACE-ID utilizes an advanced Orbital Raster Scan (ORS) optical platform to diffuse laser energy, reducing the risk of heating samples and igniting energetic materials. It also provides operation using an off-the-shelf lithium battery.

ACE-ID is backed by ReachBack®, a first-rate 24/7/365 service and support program to ensure optimum product performance.

ACE-ID is a product from Smiths Detection, a leading worldwide provider of government regulated technology products and advanced services that aid in the detection and identification of chemical, biological, radiological, nuclear and explosives (CBRNE) material and other dangerous or illegal substances.

Fast and easy analysis of multi-layered liquids, no sampling required.

Ergonomically designed for one handed operation with touchscreen interface.

Orbital Raster Scan (ORS) technology diffuses laser energy, reducing the risk of heating samples and igniting energetic materials.

Technical Data ACE-ID

**General Specifications**
- **Technology**: Raman
- **Size**: 12.7 x 8.9 x 5.6 cm (5 x 3.5 x 2.2 in)
- **Weight**: 6.05kg (13lbs)
- **Sampling**
  - Point and shoot
- Library
  - Approximately 200 substances consisting of explosives, preservers, narcotics, and toxic chemicals
- User Library
  - Ability to add user defined samples via laptop
- **Start-up time**: Less than 20 sec at 20°C (68°F)
- **Detection time**: Less than 20 sec at 20°C (68°F)
- **Power**
  - One lithium battery (CR123A) or USB power source
- **Display**
  - Touchscreen display, compliant with level 1 PPE gloved
- **Connectivity**
  - Micro USB
- **Operating temperature**
  - -20°C to +50°C (-4°F to 122°F)
- **Storage temperature range**
  - -40°C to +70°C (-40°F to 158°F)
- **Operating humidity**
  - 95%
- **Color**
  - Olive drab

www.smithsdetection.com
Portable UV/Visible spectrometer (example)

Portable Spectrometer UV-VIS with LCD screen

US $ 3,000.00

SRI Width
10 (um)

Spectral Range (nm)
250-860nm

Additional Features
- mini-USB cable provided
- 4 AA batteries
- Compatible with Windows XP, Vista, Windows 7
- SMA male-fiber connector
- Including: USB cable, Software and DLL
- LED external

Specifications

<table>
<thead>
<tr>
<th>Item</th>
<th>Specification</th>
</tr>
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<tbody>
<tr>
<td>Spectral Resolution</td>
<td>1.3 – 5.0 nm-PeakM (varies by configuration)</td>
</tr>
<tr>
<td>Wavelength Range</td>
<td>300-1000nm, 250-800nm, 360-760nm, 700-1100nm, see table</td>
</tr>
<tr>
<td>LCD Size</td>
<td>4.3 inches</td>
</tr>
<tr>
<td>A/D</td>
<td>148Hz</td>
</tr>
<tr>
<td>Interface</td>
<td>USB 2.0 or SD card</td>
</tr>
<tr>
<td>Power supply</td>
<td>Battery or USB powered</td>
</tr>
</tbody>
</table>

CBTS Presentation
October 27, 2022
Portable NMR/benchtop spectrometer (example)

Case Study
Rapid screening of street drugs by benchtop NMR

The spread of illicit drugs has become a global problem, not only causing direct harm to people's physical and mental health, but also seriously affecting social and economic development. In addition to traditional drugs, many new psychoactive substances (NPS) have appeared in recent years, and they are becoming increasingly popular. New psychoactive substances are also known as "designer drugs" or "synthetic drugs," which are chemicals that imitate the effects of traditional drugs but are not controlled by law. These substances can be obtained in many forms at prices similar to or even lower than those of the controlled drugs. Due to the "new structure" of these substances, some traditional detection methods have become less effective in detecting and controlling the spread of these drugs. However, the rapid screening of street drugs is often an important component of NPS detection and control. The application of NMR technology in rapid drug detection can effectively solve this problem.

Oxford Instruments has developed a new instrument platform (Oxford Instruments In-Sight) that can effectively detect and identify new psychoactive substances. In this application, the In-Sight instrument is capable of identifying a wide range of psychoactive substances, such as psychoactive substances that can be used in the production of street drugs.

Table 1: Analysis results of a batch of suspicious samples

<table>
<thead>
<tr>
<th>Sample Match Criteria</th>
<th>Quantity</th>
<th>Percentage</th>
</tr>
</thead>
<tbody>
<tr>
<td>Total number of samples analyzed</td>
<td>432</td>
<td></td>
</tr>
<tr>
<td>Unable to verify GC-MS does not produce peak</td>
<td>13</td>
<td>3.0%</td>
</tr>
<tr>
<td>No API or adnentries</td>
<td>3</td>
<td>0.7%</td>
</tr>
<tr>
<td>Cannot match</td>
<td>4</td>
<td>0.9%</td>
</tr>
<tr>
<td>Cannot match single component sample</td>
<td>374</td>
<td>86.6%</td>
</tr>
<tr>
<td>Cannot match two-component sample</td>
<td>13</td>
<td>3.0%</td>
</tr>
<tr>
<td>Partial matching two-component or multi-component samples</td>
<td>25</td>
<td>5.8%</td>
</tr>
<tr>
<td>Verification of samples containing API or adnentries</td>
<td>416</td>
<td></td>
</tr>
<tr>
<td>Exact match</td>
<td>387</td>
<td>90.0%</td>
</tr>
<tr>
<td>Exact partial match</td>
<td>412</td>
<td>99.0%</td>
</tr>
</tbody>
</table>

A batch of suspicious samples seized by public security agencies was analyzed by In-Sight and gas chromatography-mass spectrometry (GC-MS) at the same time. A total of 432 samples were analyzed, of which 374 (86.6%) could not be detected by GC-MS due to the presence of unknown substances, and 13 (3.0%) contained no active ingredients (API) or adnentries. Among the remaining 416 samples that were confirmed to contain API or adnentries, 387 (93.3%) of the NMR and GC-MS test results were matched exactly. On this basis, if the partially matched samples are added, the total number reached 422 (97.9%). Of equal importance, no false positives were found. This data demonstrates that In-Sight can detect and identify street drugs with high accuracy and reliability.

Identifying street drugs - data for judicial enforcement

Through comparison and matching with the In-Sight drug database, it was found that more than 400 questionable samples contained a variety of different drugs. Among these drugs, a large number of single-component compounds, such as cocaine, were most often detected. Due to the low cost of cocaine and the lack of effective controls for these drugs, the police and forensic science agencies are often faced with the challenge of identifying the substances contained in these suspicious samples. The In-Sight instrument has the ability to identify substances contained in these samples with high accuracy and reliability.

References:

Visit nmr.oxinst.com

CBTS Presentation
October 27, 2022
Visit to the San Ysidro and Otay Mesa DHS Border Facilities

CBTS Presentation
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Controlled/selected decomposition with monochromatic hard x-rays
Decomposition of materials using tuned hard x-rays.

**Measurement of the Energy and High-Pressure Dependence of X-ray-Induced Decomposition of Crystalline Strontium Oxalate**

David Goldberger,* Egor Evlyukhin, Petrika Cifigliu, Yonggang Wang, and Michael Pravica*†

1High-Pressure Science and Engineering Center (HIPSEC) and Department of Physics, University of Nevada Las Vegas (UNLV), Las Vegas, Nevada 89154-4002, United States
2HPCAT, Geophysical Laboratory, Carnegie Institution of Washington, 9700 South Cass Avenue, Argonne, Illinois 60437, United States

ICIP Talk
July 11, 2022
K-edge and L-edge absorption of x-rays
Evidence for L-edge induced chemistry
Observation of molecular decomposition “resonances” in the hard x-ray regime.

**Fig 1**: (Left): Schematic of x-ray irradiation of strontium oxalate just above the K-edge of Sr$^6$. (Middle): photos of x-ray induced reaction of SrC$_2$O$_4$ (Right): X-ray induced decay of KClO$_3$ into KCl and O$_2$ as a function of x-ray energy.

25 keV ~ 0.5 Å
Nucleic acids and their incorporation into DNA/RNA

TAT

Melamine
Virus schematic

- Membrane glycoprotein (peplomer)
- Capsid
- Genetic material
- Matrix
- Lipid envelope
Patent application: VACCINES PRODUCED USING HARD X-RAYS

Before the Irradiation

After the Irradiation

Tuned Hard X-rays (> 7 keV)
About Mail Irradiation
In October 2001, the infectious disease anthrax was found in mail sent to several news agencies and the offices of two United States Senators. Anthrax is a species of bacteria (scientific name: Bacillus anthracis) that forms spores, which when inhaled, can make people sick. It is very rare that you would come in contact with anthrax during normal daily activities. However, after the anthrax mailings in 2001, the U.S. Postal Service began to irradiate mail addressed to certain government agencies. This was done with help from the Federal Bureau of Investigation (FBI) and public health experts.

Irradiating mail can make it dry, brittle or discolored. During the irradiation process, mail must pass through a high energy beam of ionizing radiation in order to kill harmful bacteria. The beam penetrates deep into the mail to destroy viruses and bacteria—like anthrax. Mail irradiation can also be used on thicker postal materials like letter trays and packages.

The ionizing radiation used in the mail irradiation process can cause chemical changes in paper. The mail might come out brittle and discolored, looking and smelling like it has been baked in an oven. Irradiation also might turn plastics brown and warp CD cases or other plastic storage containers. Even though it causes physical changes, irradiating mail does not make the mail radioactive.

Radiation levels are closely monitored at mail irradiation facilities to ensure that workers are safe. The facilities have thick concrete or lead lined walls to shield employees and visitors from radiation.

What You Can Do
There are no radiation concerns with handling irradiated mail. Irradiation does not make the mail radioactive.
Future Plans

• Surface Enhanced Raman Spectroscopy
• Demonstration of enhancement collection idea for fiber optics and remote detection
• Fluorescence detection
• Visit of DHS border facility
• Visit of CBTS
• Design of portable remote detection UV/Vis + Raman system
Acknowledgements:

We are grateful to

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Angelica Diaz Tremillo
Petrika Cifligu

Outside of UNLV:
Jenny Ligon
Matt Cochran
Gregory Pompelli
Chris Scarmardo
Sipra Daripa
Cedricka Harris
Beth White
ORISE SRT program
CBTS
DHS
Texas A&M
Progress Report

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ACTIVE examples:

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Herring sperm DNA UV/Vis spectrum

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October 27, 2022
Proton NMR spectra of yeast (left) and TMV (right).
One suggested approach:

Raman → UV/Vis → NMR → ACTIVE
Proposed setup (sketch):
Portable Raman spectrometer (example)

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For product information, sales or service, please go to www.smithsdetection.com/handheld

Portable Raman spectrometer

Technical Data ACE-ID

<table>
<thead>
<tr>
<th>General Specifications</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Technology</td>
<td>Raman</td>
</tr>
<tr>
<td>Size</td>
<td>12.7 x 8.9 x 5.4 cm (5 x 3.5 x 2.2 in)</td>
</tr>
<tr>
<td>Weight</td>
<td>0.65kg (1.4lb)</td>
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<td>Sampling</td>
<td>Point and shoot</td>
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<td>User library</td>
<td>Ability to add user defined samples via laptop</td>
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<td>Start-up time</td>
<td>Less than 20 sec at 20°C (68°F)</td>
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<tr>
<td>Detection time</td>
<td>Less than 20 sec at 20°C (68°F)</td>
</tr>
<tr>
<td>Power</td>
<td>One lithium battery (12V/2000mAh) or USB power source</td>
</tr>
<tr>
<td>Display</td>
<td>Touchscreen display, compatible with a PFG gloved hand</td>
</tr>
<tr>
<td>Connectivity</td>
<td>Micro USB</td>
</tr>
<tr>
<td>Operating temperature</td>
<td>-20°C to +60°C (4°F to 140°F)</td>
</tr>
<tr>
<td>Storage temperature range</td>
<td>-40°C to +70°C (-40°F to 158°F)</td>
</tr>
<tr>
<td>Operating humidity</td>
<td>5%</td>
</tr>
<tr>
<td>Color</td>
<td>Olive drab</td>
</tr>
</tbody>
</table>

Fast and easy analysis of multi-layered liquids, no sampling required.

Ergonomically designed for one-handed operation with touchscreen interface.

Orbital Raster Scan (ORS) technology diffuses laser energy, reducing the risk of heating samples and igniting energetic materials.

www.smithsdetection.com

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October 27, 2022
Portable UV/Visible spectrometer (example)

This completely independent and portable spectrometer comes with a full LCD screen and built-in analysis software making it possible to make measurements at any location. It also has battery, no local power supply is required.

Data from either measurements or analysis can then be loaded onto flash memory or plugged straight into your computer for further evaluation at a later stage. Applications for this new type of spectrometer include:

- Spectroscopy
- Absorption measurements
- Reflectance measurements
- Light source testing (e.g., LEDs)
- Detection of excipient additives in food
- Taste analyzers
- Fluorescence measurement
- Industrial color measurement
- Tooth decay analysis
- Semiconductor inspection
- Chromatography
- DNA analysis

Additional Features
- miniUSB cable provided
- 4 AA batteries
- Compatible with Windows XP, Vista, Windows 7
- SMA male-fiber connector
- Including: USB cable, Software and DLL
- LCD external

<table>
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<th>Items</th>
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<tr>
<td>Spectral Resolution</td>
<td>1.3 – 5.0 nm-PW-HM (varies by configuration)</td>
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<td></td>
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<tr>
<td>Wavelength Range</td>
<td>390-1000nm, 250-800nm, 360-760nm, 700-1100nm, see table</td>
<td></td>
<td></td>
</tr>
<tr>
<td>LCD Size</td>
<td>4.3 inches</td>
<td>Size</td>
<td>14(W) x 9.5(D) x 31.4(H)-mm</td>
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<tr>
<td>A/D</td>
<td>162fps</td>
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<td></td>
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<tr>
<td>Interface</td>
<td>USB 2.0 or SD-card</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Power supply</td>
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</table>

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October 27, 2022
Portable NMR/benchtop spectrometer (example)

**Case Study**

**Rapid screening of street drugs by benchtop NMR**

The spread of illicit drugs has become a global problem, not only causing direct harm to people’s physical and mental health, but also seriously affecting social and economic development. In addition to traditional drugs, many new psychoactive substances (NPS) have appeared in recent years, and they are becoming increasingly popular. New psychoactive substances are also known as “designer drugs” or “club drugs.” They are often used in nightclubs and parties. These substances can lead to unexpected and unpredictable effects. Furthermore, the use of new psychoactive substances is more difficult to detect, as they may not be present in the usual drug tests. This makes it challenging to detect new drugs. To address this challenge, portable NMR/benchtop spectrometers are used to identify street drugs. These instruments can provide quick and accurate results, helping law enforcement officers to detect and identify new psychoactive substances. The rapid screening of street drugs by benchtop NMR can help to identify new psychoactive substances and provide valuable information for public security and judicial enforcement agencies.

**Identifying street drugs - data for judicial enforcement**

Through comparison and matching with the NMR spectra of traditional substances, it was found that more than 40% of questionable samples contained a variety of different drugs. Among street drugs containing single-component compounds, the most often detected were ecstasy (MDMA), cocaine and ketamine. In the two-component mixture samples, cocaine/levorotac, cocaine/paracetamol, and other substances were mainly found. The results show that using NMR can provide valuable references for public security judicial identification agencies to understand the types and the prevalence of illicit drugs, and to formulate corresponding control strategies and action plans.

**References**


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**Visit nmr.oxinstr.com**

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**Sample Match Criteria**

<table>
<thead>
<tr>
<th>Criteria</th>
<th>Quantity</th>
<th>Percentage</th>
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<tr>
<td>Total number of samples analysed</td>
<td>432</td>
<td>3.0%</td>
</tr>
<tr>
<td>Unable to verify GC-MS does not produce peak</td>
<td>13</td>
<td>3.0%</td>
</tr>
<tr>
<td>No API or admixtures</td>
<td>3</td>
<td>0.7%</td>
</tr>
<tr>
<td>Connex match</td>
<td>4</td>
<td>0.9%</td>
</tr>
<tr>
<td>Connex match single component sample</td>
<td>374</td>
<td>86.6%</td>
</tr>
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<td>416</td>
<td></td>
</tr>
<tr>
<td>Exact match</td>
<td>387</td>
<td>93.0%</td>
</tr>
<tr>
<td>Exact + partial match</td>
<td>412</td>
<td>99.0%</td>
</tr>
</tbody>
</table>

---

A batch of questionable samples collected by public security agencies was analysed by 1- and 2-Pulse and gas chromatography-mass spectrometry (GC-MS) at the same time. A total of 500 questioned samples were tested in this way. The number of samples analysed was 432, of which 1- and 2-Pulse could not be verified by NMR as they were no GC-MS peaks, and 3.0% contained no active ingredients (API) or admixtures. Among the remaining 258 samples that were confirmed to contain API or admixtures, 267 (93.0%) of the NMR and GC-MS test results were matched exactly. On this basis, if the partially matched samples are added, the total number reached 6.8%. Of equal importance, no false positives were found. This data demonstrates that 1- and 2-Pulse can detect and identify street drugs with high accuracy and reliability.

---

Visit to the San Ysidro and Otay Mesa DHS Border Facilities
Controlled/selected decomposition with monochromatic hard x-rays
Decomposition of materials using tuned hard x-rays.

**Measurement of the Energy and High-Pressure Dependence of X-ray-Induced Decomposition of Crystalline Strontium Oxalate**

David Goldberger,*,† Egor Evlyukhin,*,‡ Petrika Cifiglu,*, Yonggang Wang,* and Michael Pravica*,†

†High-Pressure Science and Engineering Center (HiPSEC) and Department of Physics, University of Nevada Las Vegas (UNLV), Las Vegas, Nevada 89154-4002, United States

‡HPCAT, Geophysical Laboratory, Carnegie Institution of Washington, 9700 South Cass Avenue, Argonne, Illinois 60437, United States

ICIP Talk
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K-edge and L-edge absorption of x-rays
Evidence for L-edge induced chemistry
Observation of molecular decomposition “resonances” in the hard x-ray regime.

**Fig 1:** (Left): Schematic of x-ray irradiation of strontium oxalate just above the K-edge of Sr$^6$. (Middle): photos of x-ray induced reaction of SrC$_2$O$_4$ (Right): X-ray induced decay of KClO$_3$ into KCl and O$_2$ as a function of x-ray energy.

25 keV ~ 0.5 Å
Nucleic acids and their incorporation into DNA/RNA

TATB

Melamine
Virus schematic

- membrane glycoprotein (peplomer)
- capsid
- genetic material
- matrix
- lipid envelope
Patent application: VACCINES PRODUCED USING HARD X-RAYS

Tuned Hard X-rays (> 7 keV)

Before the Irradiation

DNA/RNA

After the Irradiation

Damaged DNA/RNA

Capsid Largely Undamaged

Capsid Remained Intact

Potential Vaccine
In October 2001, the infectious disease anthrax was found in mail sent to several news agencies and the offices of two United States Senators. Anthrax is a species of bacteria (scientific name: Bacillus anthracis) that forms spores, which when inhaled, can make people sick. It is very rare that you would come in contact with anthrax during normal daily activities. However, after the anthrax mailings in 2001, the U.S. Postal Service began to irradiate mail addressed to certain government agencies. This was done with help from the Federal Bureau of Investigation (FBI) and public health experts.

Irradiating mail can make it dry, brittle or discolored. During the irradiation process, mail must pass through a high energy beam of ionizing radiation in order to kill harmful bacteria. The beam penetrates deep into the mail to destroy viruses and bacteria—like anthrax. Mail irradiation can also be used on thicker postal materials like letter trays and packages.

The ionizing radiation used in the mail irradiation process can cause chemical changes in paper. The mail might come out brittle and discolored, looking and smelling like it has been baked in an oven. Irradiation also might turn plastics brown and warp CD cases or other plastic storage containers. Even though it causes physical changes, irradiating mail does not make the mail radioactive.

Radiation levels are closely monitored at mail irradiation facilities to ensure that workers are safe. The facilities have thick concrete or lead lined walls to shield employees and visitors from radiation.

What You Can Do
There are no radiation concerns with handling irradiated mail. Irradiation does not make the mail radioactive.
Future Plans

• Surface Enhanced Raman Spectroscopy
• Demonstration of enhancement collection idea for fiber optics and remote detection
• Fluorescence detection
• Visit of DHS border facility
• Visit of CBTS
• Design of portable remote detection UV/Vis + Raman system
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