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The Analysis of Explosives by Isotope Ratio Mass Spectrometry - an Australian Perspective

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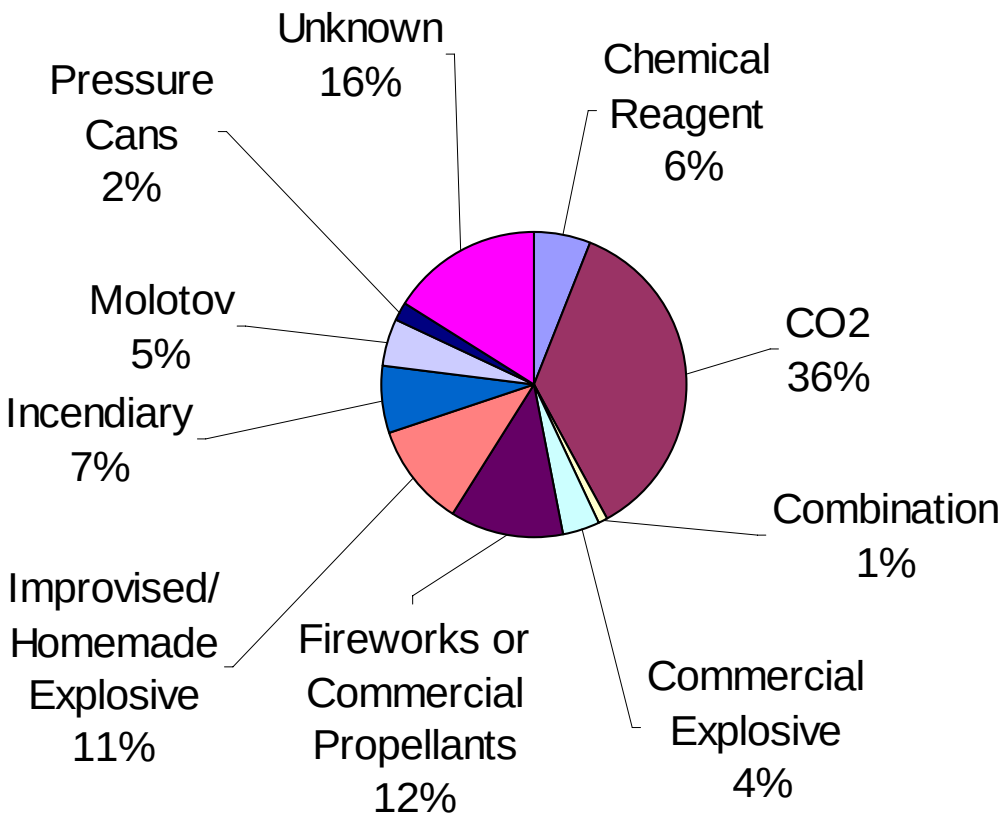
(AFP)

Conduct Examinations For:

- ACT Community Policing
- AFP National Operations
- External agencies on request:
  - Marine Incident Investigation Unit (MIIU)
  - Director of Public Prosecutions (DPP)
  - Other government agencies
  - International law enforcement agencies, including: Solomon Islands, Fiji, Indonesia, and the Philippines.
- Increasing proportion of work is providing forensic support in relation to national and international suspected terrorist incidents.

Australian Incidents, 2003



Bombing Incidents

- 37 actual bombings
- 242 attempted
- 15 hoax
- 137 recoveries
- 12 thefts
- TOTAL: 443

Statistics from Australian Bomb Data Centre (ABDC) 2003 Annual Report

South East Asian Devices

Initiators

- Non-electric (plain detonators)
- Improvised electric detonators
- Electric detonators

Main Charges

- Military ordnance
- TNT
- Plastic explosives
- Home made explosives (HME) and home made incendiary (HMI)

Boosters

- TNT
- Bulk detonators
- Detonating cord
- Commercial

AFP Research Plan

- Differentiation between batches/lots of the same explosive from the one manufacturer.
  - evaluate the relationship between the isotopic composition of the raw materials and the isotopic composition of the end product.
  - evaluate potential causes of fractionation.
  - monitor batches/lots over time.

- Differentiation between same explosive type from different manufacturers.
  - consider the effect of different manufacturing processes/environments on the isotopic composition of the end product.

AFP Research Plan

- Build a database consisting of isotope ratio values of explosive compounds and components manufactured in and imported into Australia.
- Expand to include explosives available in the South East Asian region.
- Monitor changes in the isotopic composition of the raw materials and end products over time during storage.
- Evaluate potential for comparison of traces and bulk samples and also pre and post blast samples.

AFP Progress

- July 2002 - compiled business case for the purchase of an IRMS instrument. October 2002 - funding for the project was approved by the AFP Science and Technology Steering Committee (STSC) ($AUD550K).

- February 2003 - order placed for a Thermo Finnigan DELTA$_{plus}$XP IRMS with dual inlet and continuous flow capabilities (both BSIA and CSIA for C, N, O and H).

- June 2003 - laboratory refurbishment. Issues encountered regarding appropriate gas cylinder storage and set-up creating significant delays in the project. Late October 2003 - issues resolved with the manufacturer.

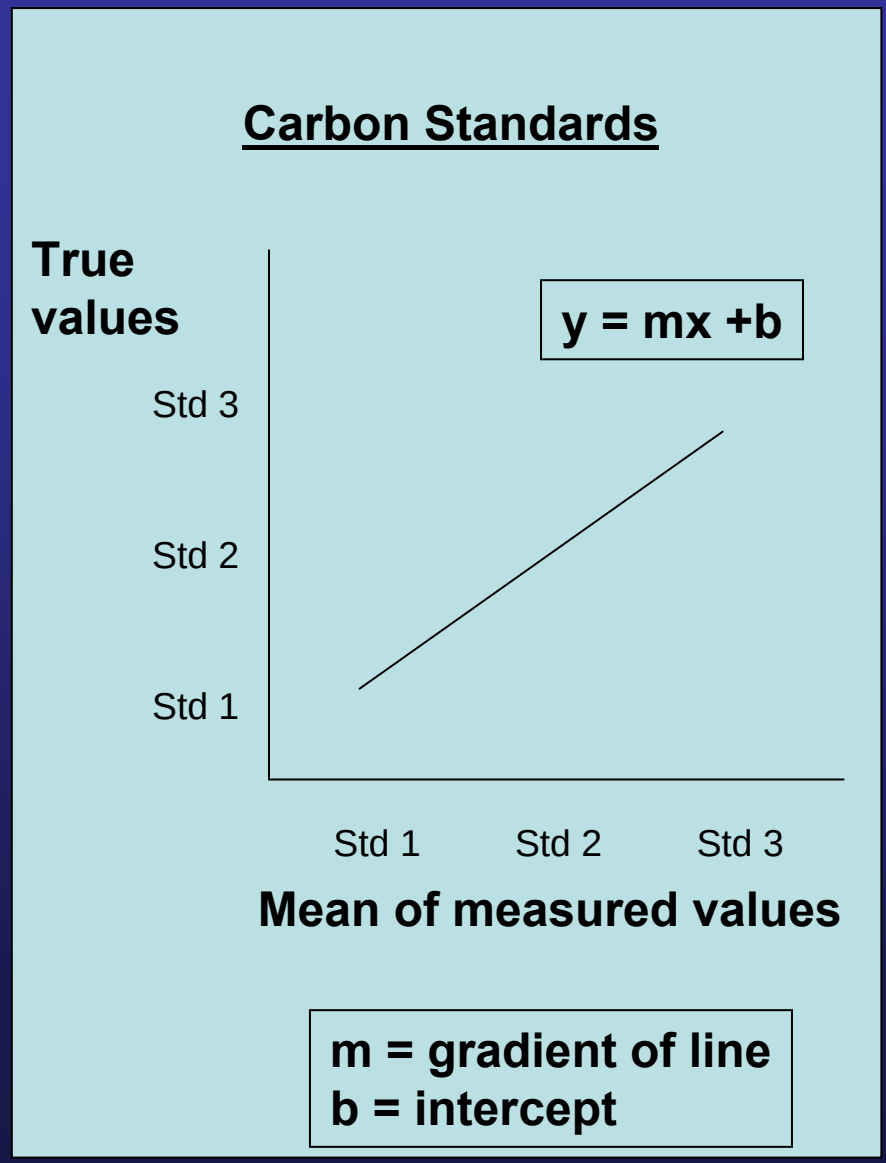
- March 2004 - instrument installation and acceptance testing. Range of International Standards purchased from the IAEA.

AFP Progress

- November 2004 - sample collection from ADI (Australian Defence Industries).
- December 2004 - ammonium nitrate (AN) samples received (Australia and overseas).
- Experimental work delayed - faulty vacuum pumps, gas leaks from the fittings to the gas cylinders, blown filaments and excessive moisture in compressed air lines.
- January 2005 - commenced selection and evaluation of working standards for analysis of ammonium nitrate samples using EA-IRMS. Initial plans for calibration of standards, method development and validation.
- February 2005 - installation of air conditioning unit to stabilise room temperature.
- March 2005 - further vacuum pump issues. Bulk nitrogen isotope ratios of ammonium nitrate samples analysed (preliminary only).

Calibration of Standards

- International Standards (Secondary Reference Materials)
  - Select suitable International Standards
  - Wash and dry tin capsules
  - Weigh standard
  - Analyse 10 samples of each
  - Calculate mean of measured values
  - Plot calibration graph for each element
  - Analyse International Standard as unknown to check calibration graph

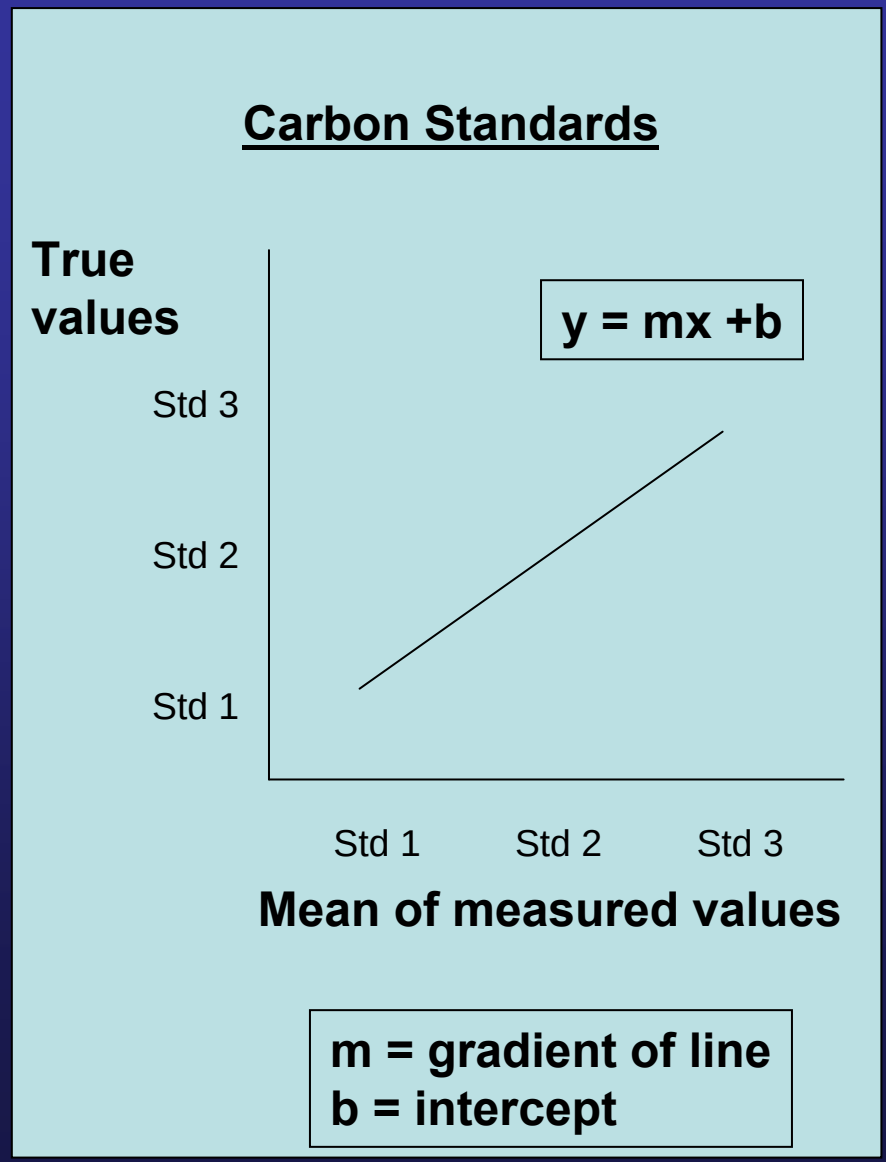


Calibration of Standards

- Working Standards
  - Select and evaluate suitable working standards
  - Wash and dry tin capsules
  - Ground and weigh standard
  - Analyse 10 samples of each
  - Calculate mean of measured values
  - Use calibration graph for International Standards to calculate true values of working standards

Analysis of Samples

- Analyse working standards in triplicate each sequence/run (e.g. at start and then every 10 samples)
- Calculate mean of measured values
- Use calibration graph for International Standards to calculate true values of working standards
- Plot measured values vs. true values of working standards
- Analyse samples (triplicate)
- Calculate true values of samples using the working standards calibration graph formed for each sequence/run



Method Validation

- NATA - ISO 17025 & Technical Note 17
- Evaluate performance characteristics:
    - Selectivity (interferences & matrix effects)
    - Linearity range
    - Limit of detection (sensitivity)
    - Accuracy
    - Precision (repeatability)
    - Recovery
    - Ruggedness
    - Stability of samples/standards & sample homogeneity
    - Measurement uncertainty

Explosive Manufacturers

- Explosive Manufacturers in Australia:
  - Orica Australia
  - Dyno Nobel
  - UEE Explosives
  - Roach Mining Services
  - Quinn Investments
  - Johnson Explosives
  - Some smaller companies and ammunition and fireworks companies
  - Australian Defence Industries (ADI) (not commercial)

ADI Samples

- Propellants
  - four different single and double based propellants.
  - samples of propellant from the current lots and lots from the last 5 years.
  - following through a current lot in production - samples of the starting materials, residual materials and end products.

- High Explosives
  - TNT, RDX, RDX/TNT and PE4.
  - samples of each from current lots and lots manufactured in last 5 years.
  - will follow future production lots to collect starting materials, residual acids and final products.

ADI Samples

Issues to consider during analysis and interpretation:

- Complex continuous manufacturing processes - no distinct batches.
- Different batches mixed to form lots - reference samples kept of each lot.
- Different batches may use different starting materials - starting materials from all over the world.
- Starting materials tested prior to use, however, non-solid materials not archived long term (e.g. toluene, nitric acid, sulphuric acid, ammonia).
- Synthetic pathways
- Recycling of residual materials (e.g. acids, solvents).
- Quality (purity) of starting materials and additives.
- Quality control throughout manufacturing process.
- Evaluate the need to separate additives prior to analysis.

Ammonium Nitrate

- Australian production - over 1 million tonnes annually.
- Consumption in Australia divided between mining (~90%) and fertilisation.
- Three grades: fertiliser, explosive and chemical.
- Major manufacturers of explosive grade ammonium nitrate in Australia, include:
  - Orica Australia ~ 685 000 tonnes produced annually
  - CSBP ~ 230 000 tonnes produced annually
  - Queensland Nitrates (partnership of CSBP and Dyno Nobel) ~ 180 000 tonnes
- Various suppliers of chemical grade AN in Australia, many importing from overseas.

Current Regulations

- The availability and the recognised potential for the use of AN in improvised explosive devices resulted in a Council of Australian Governments (COAG) Review of Hazardous Materials in June 2004.

- Australian states are modifying their regulations and legislation to reflect the tighter regulations with respect to the production, supply, use, storage and transportation of AN.

  - No longer available for household use or everyday use as a fertiliser
  - Background ASIO checks/police monitoring on buyers and manufacturers
  - Customers must be known to or recorded by the sellers
  - Quantity available is limited
  - Must demonstrate a legitimate need and approved ability to use
  - Must demonstrate safe and secure storage area
  - Authority required to manufacture and import AN

AN Manufacture

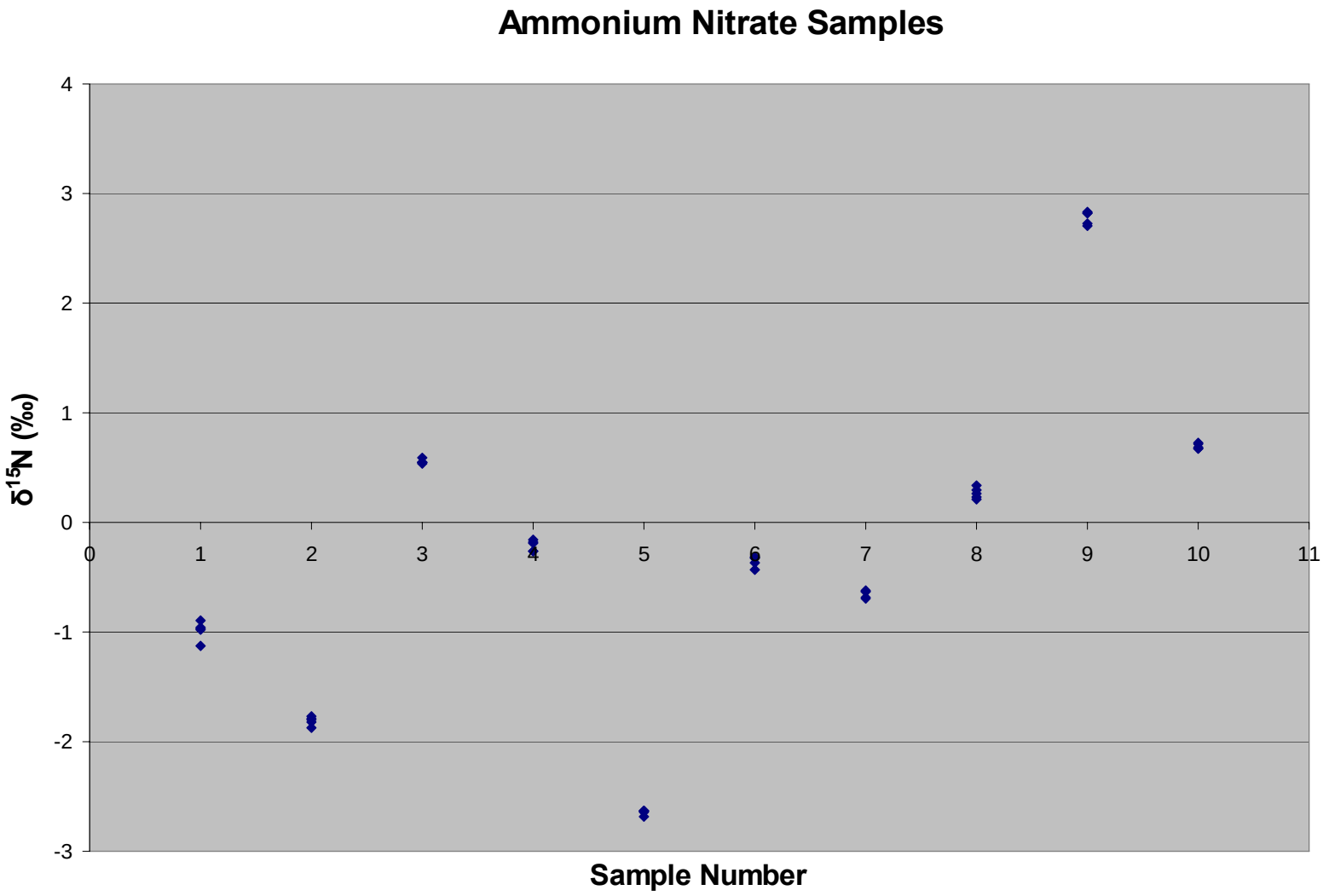
Issues to consider during analysis and interpretation:

- Majority of manufacturers utilise a continuous production line.
- Consider effect of:
  - different synthetic pathway.
  - type and purity of starting materials.
  - type and purity of stabilisers, additives and coating agents.
  - quality control throughout manufacturing process.
- Consider the need to extract additives prior to analysis.

- Process unique to each plant, hence we can expect to observe both physical and chemical variations.

- Current techniques generally do not detect the expected chemical variations between AN samples from different manufacturers.

Preliminary AN Results



Database Options

- Microsoft Access database
  - Accessible by multiple users
  - Secure, password protected and/or encrypted if required
  - Easily backed up
  - Updates sent to other agencies on CD
  - Development costs minimal
  - BUT live access to data from outside the AFP's international network would not be possible
- MS SQL Server or Oracle database
  - Potential to be connected to other agencies via secure web session (with appropriate hardware, including firewalls and a dedicated server)
  - Require AFP's IT support
  - Potentially require funding for a contractor to develop the database
  - Initial software costs
  - Hardware costs to provide access from outside the AFP network

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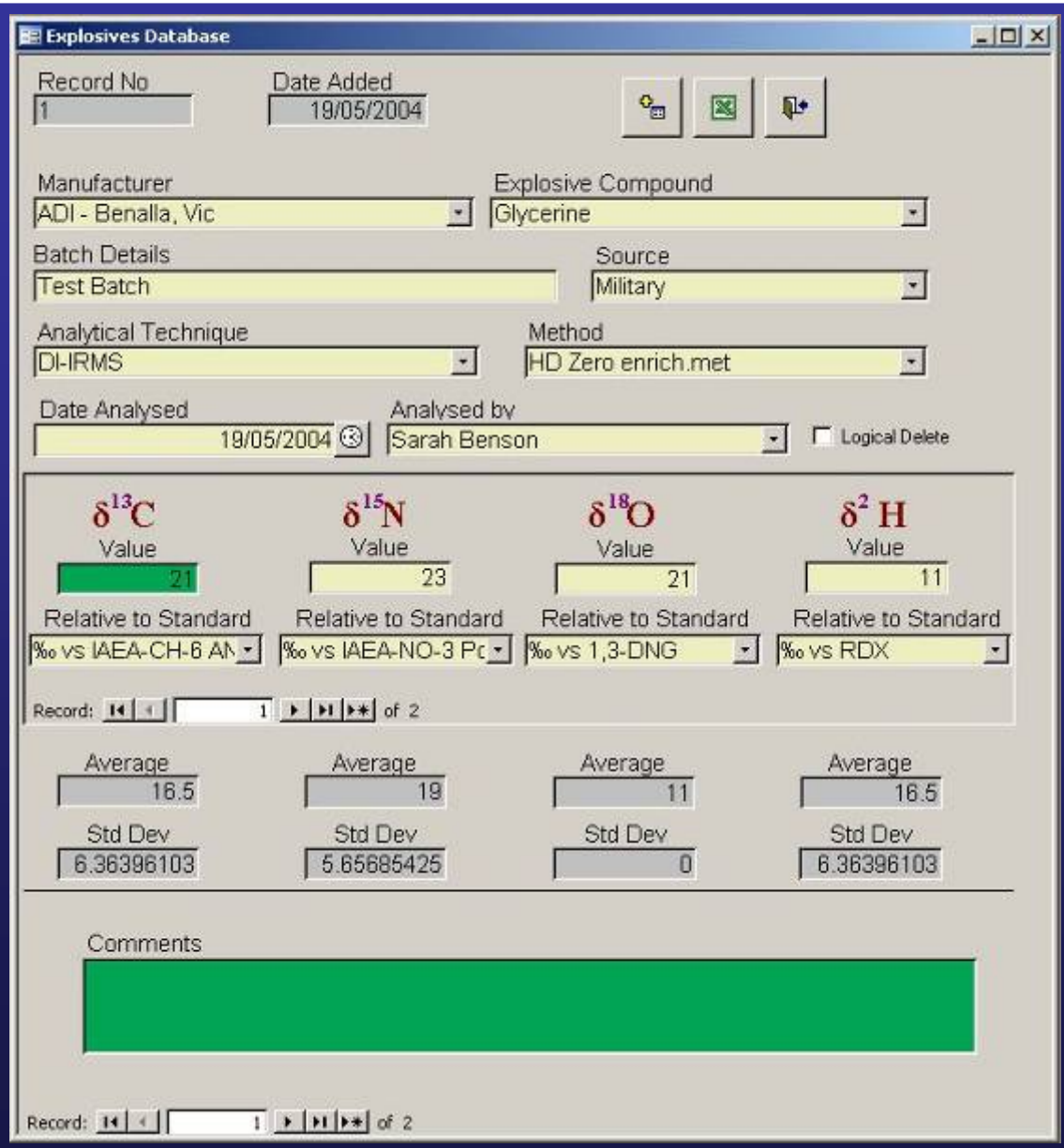
IRMS Database



Future Plans

- Continue evaluation of working standards for analysis of ammonium nitrate samples using EA-IRMS.
- Calibrate standards for nitrogen isotope.
- Validate EA-IRMS method for nitrogen isotope.
- Analyse nitrogen isotopes of AN samples using EA-IRMS.

- Conduct above for EA-IRMS analysis of oxygen isotope in AN samples.

- Obtain and analyse further AN samples from Australian and overseas manufacturers.

- Commence work on next priority explosive.

Any Questions?

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