NIST DTSA-II ("Son of DTSA"): Step-by-Step

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NIST-NIH Desktop Spectrum Analyzer (DTSA)
For 16 years, I have heard: “When will you have DTSA for the pc?”
NIST DTSA-II

- Created by Nicholas Ritchie of NIST (nicholas.ritchie@nist.gov), inspired by NIST-NIH Desktop Spectrum Analyzer (DTSA) invented 1990-92 by Chuck Fiori (NIH and NIST) and Carol Swyt-Thomas (NIH), and then further developed by Carol and Bob Myklebust (NIST).
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- DTSA-II is written in Java and operates on Mac, pc, UNIX, Linux.
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INTRODUCTION

DTSA-II is a multiplatform software package for quantitative x-ray microanalysis. DTSA-II was inspired by the popular Desktop Spectrum Analyzer (DTSA) package developed by Chuck Fiori, Carol Swyt-Thomas, and Bob Myklebust at NIST and NIH in the ‘80s and early ‘90s.

DTSA-II has been designed with the goal of making standards-based microanalysis more accessible for the novice microanalyst. We want to encourage standards-based analysis by making it as easy as possible to get reliable results. Many operations which had previously required user intervention under DTSA now are performed entirely by the software. Furthermore, the software attempts to guide the user step-by-step through common processes while performing quality control checks. While this might not provide the flexibility that some sophisticated users may desire, we feel that this philosophy is more consistent with the way laboratories are moving towards technicians responsible for multiple techniques and away from experts in single techniques. We encourage users who desire the additional power and flexibility available in the EPQ library to learn to script using Jython or to create their own alternative user interface. EPQ is much more capable than the fraction exposed via DTSA-II.

DTSA-II is based on an entirely new code base written by Nicholas W. M. Ritchie. The codebase has been carefully divided into a shared algorithm library which forms the basis for a handful of software products and a user interface shell. DTSA-II is the user interface shell and the EPQ library is the algorithm library.
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- DTSA-II is written in Java and operates on Mac, pc, UNIX, Linux.
- DTSA-II is **NOT** DTSA! Nicholas started from scratch and used DTSA as a guide to develop DTSA-II.
- Tools currently embedded in DTSA-II:
MAJOR FEATURES:

Basic IO and Display

- Read energy dispersive x-ray spectra in a variety of different commercial and non-commercial formats including the industry standard EMSA format
- Display and overlay spectra with various scaling options on linear/log/sqrt axes
- Copy/save/print the spectrum display as a bitmap/PNG file
- Output the spectra as a GNUPlot file for publication quality output
- Overlay labeled x-ray emission lines and x-ray absorption edges
- Define and integrate regions-of-interest
- View spectrum contextual information
- Archive spectra to a searchable database
- Sub-sampling of spectral data to simulate shorter acquisition times
Basic operations

- Opening and manipulating spectral files
Display and overlay spectra with various scaling options on linear/log/sqrt axes

Basic DTSA-II display window
Spectrum file selection window
A single spectrum is selected.
Spectrum preview opens in this window.
Display and overlay spectra with various scaling options on linear/log/sqrt axes

“OK” or double click loads spectrum
Basic display of single spectrum

Intensity (x-ray counts)
Energy scale in eV

Opened spectra are listed here
Spectrum information from header
But wait!

- A brilliant feature, the “Report” is going to record your actions. A daily diary of actions (file named by date) is automatically saved.
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The Report is a great feature for those of us approaching geezerhood.

Now, what was I saying?
Open several spectra at a time:
Holding down "SHIFT" key gives a continuous run

Preview of spectra in this window
To open several spectra at a time:
Holding down “CTRL” key allows multiple separate selections
3 spectrum files have opened
Note data from header

List of loaded spectra.
Highlighted are displayed

When spectra have differences in the header data, only the terms in common will be displayed when more than one spectrum is selected.
Basic operations

- Opening and manipulating spectral files
- Display of spectra
Changing the vertical axis scale:

Method 1: Use arrows: x2 or x5 /2 or /5

This arrow button restores full spectrum display

Method 2: Mouse in y-axis number field; click and drag up or down
Basic display of single spectrum

Changing the vertical axis scale:
Method 3
Right click in spectrum display brings up this Window; choose zoom in or zoom out; this operation can be repeated.
Note: “Zoom to all” restores full spectrum
Basic display of single spectrum

Changing the vertical axis scale from linear to log:
1. Right click in spectrum window.
2. Select “Ordinate scale”
3. Select “Log”
Basic display of single spectrum

Changing the vertical axis scale from linear to log
1. Right click in spectrum window.
2. Select “Ordinate scale”
3. Select “Log”
Basic display of single spectrum

Changing the vertical axis scale from linear to square root:
1. Right click in spectrum window.
2. Select “Ordinate scale”
3. Select “Square root”
Basic display of single spectrum

Changing the horizontal axis scale: Method 1
1. Click a preset to show 0-5, 0–10, 0-15, or 0-20 keV
Basic display of single spectrum

Changing the horizontal axis scale: Method 1
- Click a preset to show 0-5, 0–10, 0-15, or 0-20 keV
- 0 – 5 keV is shown
Basic display of single spectrum

Changing the horizontal axis scale: method 2
- Right click in spectrum field
- Choose “create an ROI”
Basic display of single spectrum

Changing the horizontal axis scale: Method 2
1. Right click in spectrum field
2. Choose “create an ROI”
3. Define start and end energies
Basic display of single spectrum

Changing the horizontal axis scale: Method 2
1. Right click in spectrum field
2. Choose “create an ROI”
3. Define start and end energies
4. “OK” This action creates yellow ROI
5. Click this icon to fully expand this ROI
6. Click within ROI and select "Zoom to region"
Basic display of single spectrum

Changing the horizontal axis scale: Method 2

6. Final display of 3.0 – 5.0 keV range
Basic display of single spectrum

Changing the horizontal axis scale: Method 3
1. Double click and sweep an ROI (low to high energy) creating yellow ROI
2. Immediate display of ROI parameters
Basic display of single spectrum

Changing the horizontal axis scale: Method 3
1. Double click and sweep an ROI (low to high energy) creating yellow ROI
2. Immediate display of ROI parameters
3. Click icon to expand
   OR
4. Right click within yellow ROI
5. Select “zoom to region”
Basic display of single spectrum

Changing the horizontal axis scale: Method 3
1. Double click and sweep an ROI (low to high energy) creating yellow ROI
2. Immediate display of ROI parameters
3. Click icon to expand
OR
4. Right click within yellow ROI
5. Select “zoom to region”
6. Final display of expanded ROI
Basic operations

- Opening and manipulating spectral files
- Display of spectra
- Peak labeling (manual only)
Peak Labeling

One spectrum selected from list.

KLM selection
To make labels “stick” click these check boxes
Peak Labeling

Labeled Spectrum
Peak Labeling

Choosing the peak label style
Peak Labeling

Choosing the peak label style IUPAC Long labels
Choosing the peak label style
Siegbahn labels
Basic operations

• Opening and manipulating spectral files
• Peak labeling (manual only)
• Exporting spectra for publication (Gnuplot)
Exporting spectrum for publication as gnuplot
Publication-quality graphics from Gnuplot
Comparing spectra from different EDS spectrometers, or from different dates from the same EDS:
The issue of EDS calibration

- A spectrum is recorded with calibration data: eV/channel, zero offset, number of channels (depending on manufacturer, this data may or may not be embedded in the .msa header).
- When a spectrum is read into DTSA-II, the calibration information is checked against the current calibration. If there is a mismatch, a message prompts the analyst.
When attempting to open a spectrum file with a different calibration, this message appears:

- Spectrum Calibration

When opening a spectrum file with a different calibration, the software may display a message indicating that the calibration does not seem to be similar to the default detector. The user is prompted to apply the default detector nonetheless.
The issue of EDS calibration

• A spectrum is recorded with calibration data: eV/channel, zero offset, number of channels (depending on manufacturer, this data may or may not be embedded in the .msa header).

• When a spectrum is read into DTSA-II, the calibration information is checked against the current calibration. If there is a mismatch, a message prompts the analyst.

• The analyst then has two choices:
  - 1. Change the detector selection to match the incoming spectrum
  - 2. Accept the incoming spectrum but display it according to the current calibration information. (Note: the incoming spectrum will retain its calibration data so that when it is the only spectrum being displayed, its own calibration will be applied.)
Comparing multiple spectra

• Matching to a particular ROI
Comparing multiple spectra

We wish to match the spectra for the integral of SiKα,β
Step 1: Click and swipe across SiKα,β peak to define ROI for matching.
Comparing multiple spectra

We wish to match the spectra for the integral of SiKα,β

Step 2: Right click and select “Spectrum Comparison”, then “Region integral”
Comparing multiple spectra

Result shows Si peaks matched
Spectrum sub-sampling tool

• Take an experimentally measured spectrum and create one or more “sub-samples”, that is, equivalent spectra that would have been collected at lower dose.
• Sub-sampled spectra are useful for statistical studies. e.g., how does detection limit vary with dose.
Spectrum “sub-sampling”: creating equivalent spectra for other doses

Starting measured spectrum
Spectrum “sub-sampling”: creating equivalent spectra for other doses

- Equivalent dose factor; set to 5% for this calculation
- Number of synthesized spectra, each with different statistics
- Random Number “seed”
Spectrum “sub-sampling”: creating equivalent spectra for other doses

Note: for this example, 5% dose lowered the intensity scale by a factor of 20, as expected.
Spectrum “sub-sampling”: creating equivalent spectra for other doses

Each sub-sampled spectrum is prepared with different random statistics.
Background fitting tool
Background Fitting
Background Fitting

Enter material composition
Background Fitting

Fit with automatic placement of background ROIs
Background Fitting

Fit with manual placement of background ROIs
Background Fitting

Comparison of manual and automatic placement of background ROIs
DTSA-II Simulation Mode

• EDS spectra calculated from
  – 1. First principles, using best available cross sections and physical data (flat, bulk target only)
Simulation Alien
Simulation Alien: selecting Analytical Simulation
Simulation Alien: specifying composition
Simulation Alien: target composition
Simulation Alien: instrument configuration
Simulation Alien: Other options not invoked
Simulation Alien

Hit “Finish” when “Progress” bar is filled
Simulation Alien

Physics result
Simulation Alien: Other options invoked

Counting statistics appropriate to the dose and spectrometer efficiency are applied for the specified number of replicates.
Counting statistics appropriate to the dose and spectrometer efficiency are applied to the physics result for the specified number of replicates.
Simulation Alien

Comparison of Physics Result and after applying counting statistics

Simulation Alien
DTSA-II Simulation Mode

• EDS spectra calculated from
  – 1. First principles, using best available cross sections and physical data (flat, bulk target only)
  – 2. Monte Carlo electron trajectory simulation for various specimen configurations:
    • 1. Flat, bulk
    • 2. Layer on bulk
    • 3. Inclusion (hemisphere) embedded in bulk
    • 4. Spherical particle on substrate
    • 5. Cubic particle on substrate
Simulation Alien: Monte Carlo simulation of a 1 μm K411 glass sphere on a C substrate
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"Report" tab: Generated and emitted intensities
Simulation Alien: Monte Carlo simulation of a 1 \( \mu \text{m} \) K411 glass sphere on a C substrate

X-ray production images under "Report" tab.
Simulation Alien: Monte Carlo simulation of a 1 μm K411 glass sphere on a C substrate
Simulation Alien: Monte Carlo simulation trajectories can be viewed with Cosmo Player
Simulation Alien: Monte Carlo simulation of a 1 μm K411 glass sphere on a C substrate

View along X-axis (rotated view from Y-axis)
Simulation Alien: Monte Carlo simulation of a 1 μm K411 glass sphere on a C substrate
View along the beam
View along the beam
View from bottom of particle
DTSA-II Simulation Mode

• EDS spectra calculated from
  – 1. First principles, using best available cross sections and physical data (flat, bulk target only)
  – 2. Monte Carlo electron trajectory simulation for various specimen configurations:
    • 1. Flat, bulk
    • 2. Layer on bulk
    • 3. Inclusion (hemisphere) embedded in bulk
    • 4. Spherical particle on substrate
    • 5. Cubic particle on substrate
E0 = 20 keV

Simulation Alien: Monte Carlo simulation of a 1 μm SiO2 hemisphere in FeS2
Simulation Alien: Monte Carlo simulation of a 1 µm SiO₂ hemisphere in FeS₂

$E_0 = 20$ keV

1-µm inclusion of SiO₂ in FeS₂
Simulation Alien: Monte Carlo simulation of a 1 μm SiO₂ hemisphere in FeS₂
Simulation Alien: Monte Carlo simulation of a 1 μm SiO$_2$ hemisphere in FeS$_2$
DTSA-II: Quantitative Analysis

- ZAF analysis against standards
- Standards are used to extract needed peak references for MLLS fit.
- Report contains pertinent data (ZAF factors, weight\%, atom\%, normalized weight\%; $1\sigma$ statistics)
Select a quantification mode

Select the mode which best describes the operation you wish to perform. The mode you select will determine what information you will be asked to provide and what information will be computed.

- Determine the composition of an 'unknown' spectrum by MLSQ fitting to standards
- Determine the composition from k-ratios
- Determine the composition of an 'unknown' spectrum by fitting using a simplex method
- Estimate measured k-ratios from composition

Message: Select an analysis mode.
Specify the instrument

Instrument: 6600 Probe
Detector: EDAX_35ms
using the EDAX_35ms
with calibration: FWHM[Mn Kα]=134.0 eV - initial
Setting: at a beam energy of 20.0 keV.
Choose a spectrum file
Specify standard spectra

Select the element(s) for which Cu5-20K750pA35mu-s200s is a reference.

Sulfur - Selected

Antimony

Message: Specify standard spectra and the associated elements and compositions.
Continue specifying needed spectra
Oxygen & other elements

- No extra element
- Element by difference: Oxygen
- Oxygen by stoichiometry

Element | Cation | Anion | As
---------|--------|-------|---
Sulfur   | 1      | 3     | SO₂
Iron     | 1      | 1     | FeO

Message: Specify an element to be handled specially

Back | Next | Finish | Cancel
Access Finder to select spectra
The results

Normalization:
- Weight percent
- Normalized weight percent
- Atomic percent

<table>
<thead>
<tr>
<th>Spectrum</th>
<th>Sum</th>
<th>S</th>
<th>Fe</th>
</tr>
</thead>
<tbody>
<tr>
<td>FeS 20k'750pA35mu...</td>
<td>100.10 ± 0.26</td>
<td>36.41 ± 0.09</td>
<td>63.59 ± 0.25</td>
</tr>
</tbody>
</table>
The results

Normalization:
- Weight percent
- Normalized weight percent
- Atomic percent

<table>
<thead>
<tr>
<th>Spectrum</th>
<th>Sum</th>
<th>Si</th>
<th>Fe</th>
</tr>
</thead>
<tbody>
<tr>
<td>FeS-200°C/50μm...</td>
<td>100.10 ± 0.26</td>
<td>49.89 ± 0.22</td>
<td>50.11 ± 0.26</td>
</tr>
</tbody>
</table>

Finish

More...
Report contains ZAF details
Residuals after fitting peaks

Note: symmetric structure of residuals at peak positions implies that the peak references used are appropriate for this unknown.
Comparison of Measured and Simulated FeS spectra
Note good fit for FeK and S K, but poor fit for FeL
Comparison of Measured and Simulated FeS spectra
Comparison of Measured and Simulated FeS spectra after region integral scaling
Adding a new detector to the list under "Preferences"
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