How Did You Do that?

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Methods and results reported to our scientific, technical or law enforcement clients and/or presented in a court of law demand the assurance that all work can be duplicated precisely in every detail. The preparation of technical reports that meet high standards for accuracy and completeness are an ethical responsibility and professional obligation for managers of analytical laboratories.

As the extent or complexity of work increases, more methods and techniques may be employed, producing an array of results. The preparation of reports which accurately describe what was done, how it was done, and what was produced can become a significant challenge. The collection, handling, and analysis of physical evidence can seriously impact the search for the truth, as the O.J. Simpson trial has clearly illustrated.

Interrelated multiple method studies frequently produce voluminous quantities of data. It can seem like an overwhelming task to prepare a report which accurately describes all pertinent details about materials, methods, and results. Reports which combine electron microscopy data with electron diffraction, x-ray and/or electron energy loss spectrometry, and/or feature analysis may become quite involved. In order to prepare or judge technical reports, it is helpful to ask, "Could I reproduce and verify these results using the information given?"

A good way to start a report is to give date, time, and means by which specimens were received. Describing what was received, how things were packaged and labeled, the amounts, physical condition or appearance, storage, sub-sampling and subsequent labeling of materials is useful. Photographic documentation of materials received may be required.

Pertinent to specimen preparation is the accurate referencing of processing materials, suppliers, lot numbers, concentrations, and processing dates and times. Other important documentation includes the makes, models, and serial numbers of equipment, operating parameters such as pressure, vacuum, voltage, current, angles and/or distances, and the identities of persons who carried out or assisted with procedures or were observers.

High magnification imaging and analysis of structure is normally required when data is generated by electron microscopy. A series of images at progressively lower magnifications are, however, essential for locating analyzed small areas on larger structures. Low magnification images which document the analysis geometry and specimen feature orientation relative to the microscope stage and signal detector(s) help assure that an analysis can be reproduced with high precision. Application of graphic transfer letters, numbers, arrows and circles, or overlay of alphanumeric mapping grids on low magnification images help to precisely identify locations where spectral or diffraction data has been taken. These images constitute important parts of the report and should be accompanied by a record of examination date and time, instrument make, model, and serial number, operator, operating voltage and beam amperage, aperture diameter(s), spot size(s), specimen tilt(s), operating mode(s), chamber vacuum, photographic medium, processing and image collection conditions. The report should include any other variables which may impact the reproducibility of an examination.

Diffraction results should include the analysis mode, accelerating voltage, aperture diameter(s), spot size, and accurate camera constant information. For reproducible work, stage and specimen holder positions and orientations, and materials, methods, and parameters selected for pattern photography should also be given.

Spectroscopy with electron microscopy may be critical to the understanding or interpretation of structure or processes that influence structure. Spectroscopy requires that an analyst select dozens of parameters which will affect how data is generated, collected, and analyzed and how the spectral results look. Because of the many variables involved, reports which include spectroscopic results may be particularly difficult to do well. In presenting details of studies which include spectroscopy, anything unique about the preparation of specimens for chemical analysis should be described. It is useful to indicate the lightest element detected and nominal detection limit of the spectrometer being used. Also helpful to include are reference spectra from specimen support structures, mounting adhesives, and applied coatings, as well as spectra that document hole counts, background, and the accuracy of system calibrations. The date of the most recent spectrometer calibration and person who did that procedure should be reported. The report should also include the brand and version of analysis software used for the work and the identities of the analyst, participating assistants, and observers.

Because differences in spectral acquisition conditions influence the results obtained from different instruments or different laboratories, as much detail as possible about those conditions should be recorded. Spectral excitation conditions include instrument make, model, and serial number, accelerating voltage, beam current and stability, absorbed current, spot size, scan rate, image magnification and analyzed field size. Analysis geometry factors include distances between the examined surface and microscope pole piece and the examined surface and x-ray detector as well as angular values for the specimen stage tilt and detector position descriptors and diameter of the detector window. Detection facts include spectrum acquisition time, pulse shaping time constant, pulse processor dead time, and spectrometer resolution.

A list of the elements detected and the file names and file locations of spectra which have been saved on the disc should be reported for each specimen and feature analyzed. Also report the values of horizontal and vertical scales selected for spectral plots and the method and step-by-step protocol by which quantitative data was acquired.

When element distribution mapping or line profile methods have been employed, an explanation of how region of interest "windows" were set (e.g., FWHM) should be given along with the mapping rate and integration time or scans used for each map, or the line time, gain, and signal rise time values used for profiles.

Documentation of feature analysis methods should include the method and equipment used for image production, digitization, and analysis. Report the make, model and serial numbers, operator, software version, calibration date and procedures, calibration verifications, the file names and location of raw and processed image files saved to disc, the step-by-step protocol used to extract relevant data, and the parameters evaluated.

Time required to prepare detailed reports may be conserved by using forms, form letters, and computer based word processing. Well structured forms and letters simplify preparing subsequent reports. By using forms and word processing techniques, relevant information can be changed or inserted efficiently without a need to re-enter every parameter, value, or statement. A hazard in using computer based word processing to modify entries on forms and letters, however, is that values or statements for a previous analysis may inadvertently lead to omissions, inaccuracies, and/or inappropriate statements, values, or details being incorporated into the current report. It is, therefore, imperative that every detail in the completed report be carefully checked before its release.

The amount of time needed for the preparation of in-depth professional quality technical reports may seem exorbitant. Authors or editors of scientific and technical publications, concerned with space limitations and publication costs, seldom require as necessary much of the detail suggested herein. The goal in publishing research papers is to report new findings concisely, with enough technical material to allow readers to judge appropriateness of materials and methods relative to the results obtained. However, as the technical issues concerning materials and methodology which surfaced in the O.J. Simpson trail demonstrate, when results or accuracy are questioned and the need arises to reproduce or verify data, a complete record of every detail concerning the analysis is irrefutably the best defense and strongest resource. The goal of a good technical report *does* differ from the goals for a research paper. A technical report should allow someone to reproduce or verify every minute and seemingly trivial detail, and to answer exactly the question, "How did you do that?"