TECHNICAL ARTICLE

Mineral identification by elemental composition: a new tool within PDF-4 databases

T. G. Fawcett,[a] J. R. Blanton, S. N. Kabekkodu, and T. N. Blanton
International Centre for Diffraction Data, 12 Campus Blvd., Newtown Square, Pennsylvania 19073, USA

(Received 21 March 2018; accepted 29 March 2018)

The ICDD has developed a microanalysis tool to help scientists identify minerals from their elemental analyses, most typically micro-XRF or a microprobe analysis. Many minerals have characteristic elemental profiles that can often distinguish the mineral from others by their composition differences. In Release 2016 ICDD® PDF-4 databases 20 670 unique compositions have been identified out of 45 497 mineral and mineral-related entries. The application utilizes several common features of PDF® databases to enhance correct identification, most notably those formulas are expressed in weight and atomic percent, data sets are classified by mineral nomenclature and structural classifications, and most minerals have associated atomic and molecular structures. These crystal structures are very useful in determining compositional variants and solid solutions. The ICDD has developed algorithms that are analogous to the search/match processes used for powder diffraction identification. Data can be input as either the element or common oxide. To test the algorithm and graphics interfaces we compared results from the microanalysis module to published data from the Smithsonian Microbeam reference mineral collection. The software correctly identified 24/28 minerals by the highest merit score in the algorithm. In two cases, an isoelemental mineral was identified and in two other cases, the specimens had more elements than the reference standards hindering positive phase identification. © *2018 International Centre for Diffraction Data.* [doi:10.1017/S0885715618000404]

Key words: PDF-4 databases, mineral identification, microanalysis, EDS, XRF

I. INTRODUCTION

Many compounds have unique elemental signatures. ICDD databases now contain over 979 000 data entries and there are over 663 000 unique chemical formulas. Therefore, it is possible that an accurate compositional analysis should be able to identify a compound provided that (1) the references are significantly comprehensive, (2) the elemental determination is for a single compound, and (3) both experimental data and reference data were accurately determined. This is the basis for many handheld XRF data libraries, that allow researchers to do metal and alloy sorting for scrap metal recycling.

Mineral identifications are more difficult in that cation and anion substitutions are commonplace, especially in several species of clays and various carbonates, sulfates, and hydroxides. The comprehensive ICDD databases offer multiple solutions to this problem by providing the user the ability to do data mining and perform a multivariant analysis. In the ICDD databases, minerals are classified by their structural and elemental characteristics, locality, and color. The ICDD subfile and subclass system enables the user to focus on either minerals, gems or synthetics, or combinations. The extensive subfile system can also be used to discriminate against inorganic materials that are not minerals but might have similar compositions. Since the ICDD PDF-4 family of databases also contains atomic and molecular structures; the structures themselves can be used to identify logical substitutional variants and solid solutions. This turns a disadvantage in many XRF data library systems into an advantage if the same data are coupled with a PDF-4 database.

Many material and analytical laboratories have multiple analytical tools and are associated with major universities, government or industrial laboratories. ICDD customer surveys conducted in the past 20 years show that XRF, XRD, and electron microscopy are associated tools commonly used in inorganic analyses. Many microscopes are equipped with EDS-based microprobes. The development of microfocus X-ray tubes and microfocus optics has exploded in the past decade creating a new generation of handheld and microfocus XRF and XRD instruments. The ICDD has developed the PDF databases as chemical analysis tools that can be used with all of these associated analytical techniques (Fawcett *et al.*, 2017). The development of tools to identify and quantify materials using known associated analytical techniques was the main driving force for this project.

II. EXPERIMENTAL

The developments described in this publication have been bundled in a new “Microanalysis” application that is embedded in the Release 2018 PDF family of databases.

The application takes advantage of several features embedded in PDF databases. These features have been gradually developed with the PDF-4 family of databases that were first released in 2002, and designed to be used in various data

---

[a] Author to whom correspondence should be addressed. Electronic mail: dxcfawcett@outlook.com

mining applications. Key features used by the Microanalysis application are listed below:

- Every entry has a formula expressed in atomic and weight percent
- Most entries include linked atomic and molecular structures with graphic display
- Entries are classified (subfiles, prototypes, structural classifications)
- Nomenclature is standardized: IMA, IZA, IUPAC nomenclature used when available
- Entries are assigned subfiles by field experts
- Physical properties are included when available

Specific to the Microanalysis application, new tables were added that convert the elemental formula to oxide equivalents so that either oxides or elements (common XRF output) could be used as input into the application. The ICDD developers also utilized their experience with Goodness-of-Match (GOM) algorithms used for search and identification of X-ray powder patterns (Faber *et al.*, 2004). Several algorithms were evaluated and different functions, filters, and weighting schemes will be an area of future development. Two algorithms were developed using a weighted average of elements (WA and UWA). They differ in that one weighs the average vs. the reference elements (WA) and the other weighs the average vs. the input elements (UWA).

$$\mathrm{GOM(WA)} = \frac{1}{n_{\mathrm{ref}}}\sum\left(\frac{E_1\,\mathrm{exp}}{E_1\mathrm{ref}}+\frac{E_2\,\mathrm{exp}}{E_2\mathrm{ref}}+\frac{E_3\,\mathrm{exp}}{E_3\mathrm{ref}}+\cdots+\frac{E_n\,\mathrm{exp}}{E_n\mathrm{ref}}\right),$$

$$\mathrm{GOM(UWA)} = \frac{1}{n_{\mathrm{input}}}\sum\left(\frac{E_1\,\mathrm{exp}}{E_1\mathrm{ref}}+\frac{E_2\,\mathrm{exp}}{E_2\mathrm{ref}}+\frac{E_3\,\mathrm{exp}}{E_3\mathrm{ref}}+\cdots+\frac{E_n\,\mathrm{exp}}{E_n\mathrm{ref}}\right).$$

In the above formulae, E_1 refers to element 1, E_2 refers to element 2, etc., while the subscripts $_{\mathrm{exp}}$ and $_{\mathrm{ref}}$ refer to the experimental and reference determinations, respectively. The need for two algorithms will be explained in the "Results" section. All data were standardized and normalized so individual element results of 105 and 95% were not averaged to 100% but to 95%. In this example, each individual element deviates by 5%, so they are averaged to 95%. This allows an exact match of both elements to receive the highest possible score of 100%. The use of the number of elements as a denominator in both equations was to deliberately impose a severe penalty for an elemental mismatch between the experimental and reference data. For example, if one searched for an iron-containing compound with 70% iron and a 3% estimated standard deviation (ESD) there would be 1914 entries matching that criteria in PDF-4 + Release 2016. Selection of the mineral subfile reduces the entries to 321 references, eliminating many iron alloys. Limiting the selection to two elements, iron, and oxygen, further reduces the candidate list. The minerals, γ-maghemite, and hematite, would score highest by the WA goodness-of-merit formula. As the number of elements is increased the search becomes more selective. Adding titanium to the above search criteria quickly reduces the search to a choice of three titanium substituted magnetites, where the titanium ranges from 1.99 to 3.87 wt%.

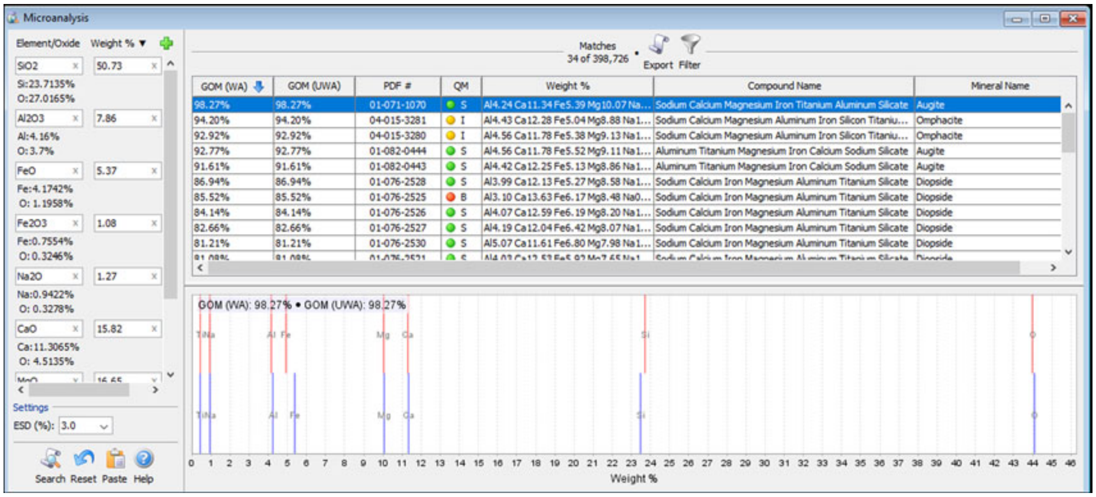
To test the algorithms the authors used reference experimental data that were available on known reference compounds. Trials were conducted using National Institute of Standards and Technology (NIST) Standard Reference Materials, United States Geological Society (USGS) reference standards and microbeam standards from the Smithsonian Institution. The NIST and USGS references were used to demonstrate the feasibility and initial development, fostered by the X-ray Fluorescence Subcommittee of the International Centre for Diffraction Data. This publication focuses on the Smithsonian Institution microbeam references that were used exclusively in the later stages of development. The microbeam reference analyses were publicly available on the internet (http://mineralsciences.si.edu/facilities/standards/datasheets.htm) and include references, mineral localities, photographs of the crystals analyzed, EDS spectra, and mass-balance composition. The collection contained 28 different mineral analyses with representation from both common and exotic minerals. These references were preferred since they had demonstrated high-quality analyses from single crystals. Trace elements were therefore clearly associated with the mineral and not impurities. While this represents an ideal case, our purpose was to test various evaluation algorithms vs. testing the purity of the specimens under analysis.

The algorithms were developed empirically using trial and error methods. The authors have extensive experience in material analysis and identification (Fawcett et al., 2011) and this experience was used to develop the algorithms and associated methods. All authors have extensive experience with the database that includes developing and integrating display formats and using the interrelated tables and data mining filters of the database. The authors consider these developments to be the initial application development. We anticipate that future user feedback will help optimize and further enhance the microanalysis capability.

III. RESULTS

Initial results on NIST and USGS reference materials were positive and logical. The ICDD database has the world's largest mineral reference collection and that collection has over 20 000 unique formulas. We knew that the algorithm would not be able to identify most polymorphic materials (e.g. rutile, anatase, and brookite forms of TiO_2) but might be able to differentiate between the different cation and anion substituents in clays. Reference data are typically high quality and accurate, and all data are editorially reviewed, so there was a reasonable expectation for positive results. The data accuracy helps in distinguishing between different compounds having the same elements, for example, wustite, magnetite, hematite, and goethite with 77.23, 72.36, 69.94, and 62.85 wt% iron respectively, and can be separately identified by an accurate iron analysis.

The UWA GOM algorithm compares all of the elemental concentrations in a sample to all of the elemental concentrations in a reference. As shown in Figure 1, data can be input as oxides or elements, in atomic percentages or in weight percentages. Conversion tables embedded in the database convert input oxide data into elemental weight percent or atomic percent. Comparisons are then made to the reference formula in the database. The ICDD uses a relational database (Faber and Fawcett, 2002) with Boolean operators and a Java point-and-click interface, so the searches are fast and user-friendly.



Element/Oxide	Weight %
SiO2	50.73
Al2O3	7.86
FeO	5.37
Fe2O3	1.08
Na2O	1.27
CaO	15.82

Si:23.7135%
O:27.0165%
Al:4.16%
O:3.7%
Fe:4.1742%
O: 1.1958%
Fe:0.7554%
O:0.3246%
Na:0.9422%
O: 0.3278%
Ca:11.3065%
O: 4.5135%

Settings
ESD (%): 3.0

Matches
34 of 398,726

GOM (WA): 98.27% • GOM (UWA): 98.27%

GOM (WA)	GOM (UWA)	PDF #	QM	Weight %	Compound Name	Mineral Name
98.27%	98.27%	01-071-1070	● S	Al4.24 Ca11.34 Fe5.39 Mg10.07 Na...	Sodium Calcium Magnesium Iron Titanium Aluminum Silicate	Augite
94.20%	94.20%	04-015-3281	● I	Al4.43 Ca12.28 Fe5.04 Mg8.88 Na1...	Sodium Calcium Magnesium Aluminum Iron Silicon Titaniu...	Omphacite
92.92%	92.92%	04-015-3280	● I	Al4.56 Ca11.78 Fe5.38 Mg9.13 Na1...	Sodium Calcium Magnesium Aluminum Iron Silicon Titaniu...	Omphacite
92.77%	92.77%	01-082-0444	● S	Al4.56 Ca11.78 Fe5.52 Mg9.11 Na1...	Aluminum Titanium Magnesium Iron Calcium Sodium Silicate	Augite
91.61%	91.61%	01-082-0443	● S	Al4.42 Ca12.25 Fe5.13 Mg8.86 Na1...	Aluminum Titanium Magnesium Iron Calcium Sodium Silicate	Augite
86.94%	86.94%	01-076-2528	● S	Al3.99 Ca12.13 Fe5.27 Mg8.58 Na1...	Sodium Calcium Iron Magnesium Aluminum Titanium Silicate	Diopside
85.52%	85.52%	01-076-2525	● B	Al3.10 Ca13.63 Fe6.17 Mg8.48 Na0...	Sodium Calcium Iron Magnesium Aluminum Titanium Silicate	Diopside
84.14%	84.14%	01-076-2526	● S	Al4.07 Ca12.59 Fe6.19 Mg8.20 Na1...	Sodium Calcium Iron Magnesium Aluminum Titanium Silicate	Diopside
82.66%	82.66%	01-076-2527	● S	Al4.19 Ca12.04 Fe6.42 Mg8.07 Na1...	Sodium Calcium Iron Magnesium Aluminum Titanium Silicate	Diopside
81.21%	81.21%	01-076-2530	● S	Al5.07 Ca11.61 Fe6.80 Mg7.98 Na1...	Sodium Calcium Iron Magnesium Aluminum Titanium Silicate	Diopside

Figure 1. (Colour online) Identification of Augite, National Museum of Natural History R6600, from input Smithsonian Microbeam reference data. The red bars are the experimental composition profile compared to the reference represented by blue bars. In this case, both experiment and reference were different specimens from Kakanui, New Zealand.

Tens of thousands of entries can be compared with the input data in a few seconds.

When oxide data are input, the program calculates an oxygen content to compare with the oxygen in the reference. Different oxidation states are acceptable so that FeO and Fe_2O_3 can both be input, as shown in Figure 1.

In Figure 1, the application display consists of three panels; left, top right, and bottom right. The left panel displays the input data and any conversion to elemental concentration. In this example, oxide formulae are input and elemental weight percentages are calculated and displayed. Data can also be input from Excel spreadsheets using the paste option in the lower left corner. The user can select atomic or weight percent; weight percent is shown in Figure 1. The right-hand panels are the main diagnostic panel for the application. Candidate entries are listed and sorted by their WA or UWA scores. The panel displays GOM scores, PDF identification number, a quality mark, composition, compound name, and mineral name. At the very top of this panel, the program shows that there were 34 matches that meet the acceptance criteria out of 398 726 PDF-4 + entries. The acceptance criteria are shown in the bottom of the left panel where a 3% ESD was applied to each input element. This ESD window is a user selectable option; the wider the window the more candidates that will be displayed. The bottom right panel graphs the weight percent of both the experimental input data (top red bars) and the reference data (bottom blue bars). The reference data display will change when various candidates are selected by highlighting with a left click of the computer mouse. In the top right panel, there is an icon labeled “Filter”. This is an important feature of this application in that the reference data can be filtered prior to analysis. PDF-4 + 2016 contains 66 fields that can be searched independently or in combination, with each search and combination potentially acting as a filter. The searches can include subfiles (i.e. minerals, gems), locality, color, density etc., that could help identify the mineral. In addition, if the highlighted entry is double-clicked with the computer mouse a full reference data entry is displayed. This connects the user to such features as the material’s diffraction pattern and graphics of the atomic and molecular structure.

The microanalysis results of the 28 Smithsonian references can be grouped into several case studies.

Case 1 – Top candidate, WA and UWA *both* > 90%

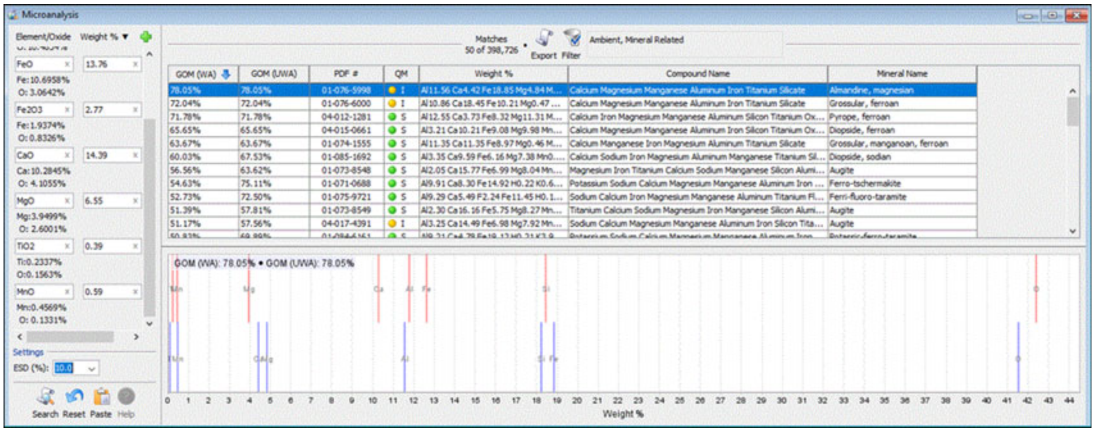
Sixteen out of 28 Smithsonian references met this criterion. In these cases (an example is shown in Figure 1) the input elements exactly match the reference elements and each element had a similar concentration. In many cases, the GOM numbers approached 100% as there were comparable references in the database. It should be mentioned that some of the Smithsonian samples were not perfectly pure, so 100% was never achieved. In a few cases, such as Figure 1, not only did the mineral match, but the mineral locality matched as well. These cases are fairly easy to interpret as the top candidate matched the Smithsonian reference mineral designation in each example. In the 16 identifications, the number of elements ranged from two to eight.

Case 2 – Top candidate, UWA > WA, UWA > 75%

In these cases, the number of elements input did not match the number of elements in the reference or there was a large discrepancy in the elemental concentrations. Is the match accurate or not? In the cases where UWA is >75% this denotes that a single element is missing in the reference, in a material with four or more elements. In fact, trying to decide whether a low WA score was a valid identification for the top candidate, led directly to the development of the UWA GOM, where the focus was shifted to the input elements. A UWA score >75% means that most elements have been accounted for by the reference.

The above criteria were met for eight additional identifications, where the top candidate was the correct mineral as identified by the Smithsonian reference analysis. In four of these cases, the UWA was >90%, making the identification easier. Such was the case for pyrope from Kakanui, New Zealand. The WA score was 84.16% because the ICDD reference pattern contained a small amount of Mn; however, the UWA score was 96.18% because of the match with the other seven elements and all input elements matched. This is also a case where the match identified the locality.

Another example is shown in Figure 2, where both the WA and UWA were 78.05%. In this case, both the calcium and iron concentrations deviated between the experimental data and the reference. The identification is for the mineral



FeO 13.76
Fe:10.6958%
O: 3.0642%
Fe2O3 2.77
Fe:1.9374%
O: 0.8326%
CaO 14.39
Ca:10.2845%
O: 4.1055%
MgO 6.55
Mg:3.9499%
O: 2.6001%
TiO2 0.39
Ti:0.2337%
O:0.1563%
MnO 0.59
Mn:0.4569%
O: 0.1331%

ESD (%): 10.0

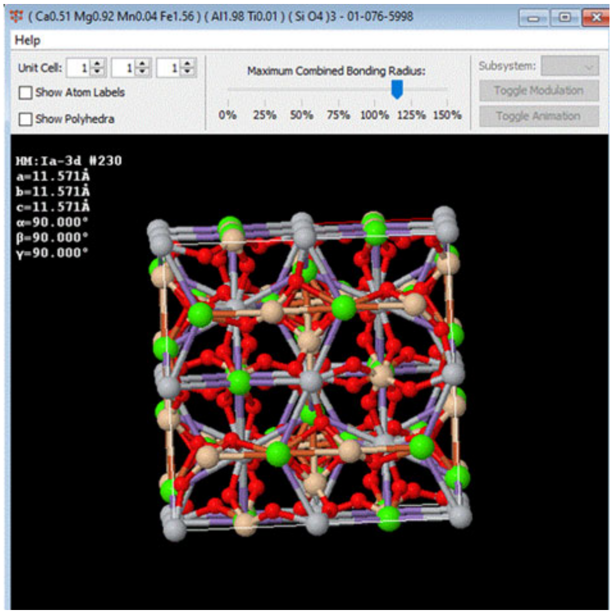
Ambient, Mineral Related

GOM (WA)	GOM (UWA)	PDF #	QM	Weight %	Compound Name	Mineral Name
78.05%	78.05%	01-076-5998	● I	Al11.56 Ca4.42 Fe18.85 Mg4.84 M...	Calcium Magnesium Manganese Aluminum Iron Titanium Silicate	Almandine, magnesian
72.04%	72.04%	01-076-6000	● I	Al10.86 Ca18.45 Fe10.21 Mg0.47 ...	Calcium Magnesium Manganese Aluminum Iron Titanium Silicate	Grossular, ferroan
71.78%	71.78%	04-012-1281	● S	Al12.55 Ca3.73 Fe8.32 Mg11.31 M...	Calcium Iron Magnesium Manganese Aluminum Silicon Titanium Ox...	Pyrope, ferroan
65.65%	65.65%	04-015-0661	● S	Al3.21 Ca10.21 Fe9.08 Mg9.98 Mn...	Calcium Magnesium Manganese Aluminum Iron Silicon Titanium Ox...	Diopside, ferroan
63.67%	63.67%	01-074-1555	● S	Al11.35 Ca11.35 Fe8.97 Mg0.46 M...	Calcium Manganese Iron Magnesium Aluminum Titanium Silicate	Grossular, manganoan, ferroan
60.03%	67.53%	01-085-1692	● S	Al3.35 Ca9.59 Fe6.16 Mg7.38 Mn0....	Calcium Sodium Iron Magnesium Aluminum Manganese Titanium Sil...	Diopside, sodian
56.56%	63.62%	01-073-8548	● S	Al2.05 Ca15.77 Fe6.99 Mg8.04 Mn...	Magnesium Iron Titanium Calcium Sodium Manganese Silicon Alumi...	Augite
54.63%	75.11%	01-071-0688	● S	Al9.91 Ca8.30 Fe14.92 H0.22 K0.6...	Potassium Sodium Calcium Magnesium Manganese Aluminum Iron ...	Ferro-tschermakite
52.73%	72.50%	01-075-9721	● S	Al9.29 Ca5.49 F2.24 Fe11.45 H0.1...	Sodium Calcium Iron Magnesium Manganese Aluminum Titanium Fl...	Ferri-fluoro-taramite
51.39%	57.81%	01-073-8549	● S	Al2.30 Ca16.16 Fe5.75 Mg8.27 Mn...	Titanium Calcium Sodium Magnesium Iron Manganese Silicon Alumi...	Augite
51.17%	57.56%	04-017-4391	● I	Al3.25 Ca14.49 Fe6.98 Mg7.92 Mn...	Sodium Calcium Magnesium Manganese Aluminum Iron Silicon Tita...	Augite

Figure 2. (Colour online) Microanalysis results on a garnet from the Roberts Victor Mine, South Africa.

almandine, which is in the garnet family. A key to understanding whether this is an appropriate identification comes from the single-crystal structure shown in Figure 3, and its corresponding table of atomic coordinates. Both are linked in the database through the PDF entry designation.

The ICDD PDF-4 + database contains tabulations of atomic positions in entries with atomic coordinates. These positions are then extracted and graphically displayed in molecular graphic options embedded in the database. The user has several options, they can examine the entry atom position table and see that the Ca and Fe atoms are in the same position, they can also look at the molecular graphics and see if the atoms are labeled (another option), or take their mouse and hover over an atom and see the specific atom label. In Figure 3, the atomic position table noted that the Ca and Fe atoms were in Wyckoff notation 24c, and then the graphics colored these in green. In an active display (not shown), the atoms could be labeled, or the label shown by the mouse positioned on the atom. The specific coordinates, site occupancy factors, and Wyckoff designations can be viewed as part of PDF entry 01-076-5598. Double-clicking on the entry, highlighted at the top of Figure 2, produces the full entry display and contents. The sum of Ca and Fe in the input data is very similar to the sum in the reference data, even though the concentrations of the individual atoms are different. A nearly identical situation with substituted cations was found for a hornblende mineral from Costa Rica, where hornblende was correctly identified as the top candidate. Once again the crystal structure and atomic positions identified allowed cation substitutions. The distinguishing feature between cases 2 and 3 is whether the crystallography supports the identification when lower scores (below 90%) are involved.



(Ca0.51 Mg0.92 Mn0.04 Fe1.56) (Al1.98 Ti0.01) (Si O4)3 - 01-076-5998

HM:Ia-3d #230
a=11.571Å
b=11.571Å
c=11.571Å
α=90.000°
β=90.000°
γ=90.000°

Figure 3. (Colour online) Structure of almandine, PDF entry 01-076-5998.

Case 3 – Top candidate, UWA > WA, UWA > 75%, incorrect results

In an example of anorthoclase, the Smithsonian reference material contained a small concentration of iron (0.2 wt.%). The ICDD mineral subfile has 15 reference data sets for anorthoclase but none have iron. The software identified an iron-containing aluminosilicate, marialite, as the top candidate. If iron is removed from the input data the software correctly identifies anorthoclase. Similarly, a Smithsonian reference of olivine contained a small concentration of manganese (0.3 wt.%), which led to the incorrect identification of the mineral chondrodite. Removal of the manganese from the input data resulted in the correct identification. In both cases, the mineral candidate was among the top listed but not the highest candidate.

Case 4 – No candidate identified

In two cases, hypersthene and ilmenite, there were no matches within the ESD elemental search limitations with the ICDD database. In both cases the Smithsonian references had more elements than the ICDD references, so scores were outside the match criteria. The ilmenite sample contained niobium and there are no corresponding Group 5 entries in the 89 ilmenite references in the ICDD database. The hypersthene reference data contained eight elements with four elements below 1.2 wt.%. The ICDD database has four hypersthene references, all contain just four elements.

IV. DISCUSSION

During the course of application testing, several variables were evaluated. We tested the sensitivity of the GOM algorithms toward light elements with the practical realization that many XRF and microprobe techniques are insensitive to light elements and that the detection limit for light elements can vary from instrument to instrument. We also wanted to test sensitivity toward low concentration elements. This comes from the realization that many single-crystal determinations, a source for much of our reference data, do not include independent trace elemental analysis determinations. In addition, single-crystal X-ray experiments often have difficulty distinguishing between close *Z* elements, for example, a small amount of Mn in an iron-containing mineral, or a small amount of Ti in a silicate.

The analysis of carbonates provided some insights. The Smithsonian references contained data on common carbonate minerals; calcite, dolomite, siderite, and strontianite. Each analysis contained a carbon dioxide determination, which when added to the elemental oxides produced a positive identification with high GOM values (>97%). We then considered the case that many XRF or EDS analyses would not have a carbon dioxide, carbon, or oxygen analysis. Just inputting the cation concentration did not work because the references would have C and the input data would not. If the oxide formulations were used, a program would expect a carbon dioxide concentration. Fundamentally this series of tests demonstrated that we had to be careful and not mix input formats so that the program does not penalize for missing elements or incorrectly calculate oxygen content. We are currently working on algorithms that can account for non-detectable light elements without a severe penalty.

As shown in cases 3 and 4 above, the presence or absence of elements below 0.5 wt%, caused a mismatch in input and reference elements and/or concentrations and often had a deleterious effect. This was particularly frustrating with case 3 where 99% of the composition of the mineral was accounted for, but the wrong phase identifications were obtained. We did trial an algorithm that did not include the number of elements in the denominator and focused on mass composition. While this algorithm exhibited improved GOM scores it also greatly increased the candidate list by opening the number of candidate chemistries and did not significantly improve results. We did find a practical limit on the concentration of input elements at approximately 0.2 wt.%. While this number was empirically determined using the Smithsonian references, it also made common sense. The Smithsonian microprobe analyses used an EDX technique and the spectra are available for each reference sample. Typically, concentrations above 0.2 wt.% are easily observed in the corresponding data. XRF and microprobe analytical techniques are commonly used and reported in both powder diffraction and single crystal crystallographic publications and they have similar *average* detection limits, especially if one considers the past 30 years of published data. While lower concentrations can be identified, particularly with newer instruments or trace analysis techniques (i.e. TXRF), the microprobe application is probably limited by the detection limits in the ICDD entries that reflect averages in the published data. This limit may be worth examining periodically as more low concentration determinations enter the public literature over time, and/ or we develop more accurate algorithms.

The results reported here were recorded after optimizing the method relative to (1) using pure elemental or oxide data, (2) setting a 0.2 wt.% limit on input element concentrations, and (3) confirming low GOM scores with associated crystallography. Those without crystallographic training and background might not be able to distinguish between positive and negative results when the GOM scores are below 90. The crystallographic data confirms elements that are located at the same atomic position and their likely concentration ranges. With element concentrations approaching 0.2% or lower, we often found it useful to run results both with and without the element and compare the data. The niobium doped magnetite was easy to identify without the niobium, but a mineralogist would probably know if they are working with Group 5 deposits and whether such identification was logical. In our analyses, we typically used wide ESD limits on the elemental concentrations. This is because we knew that we were analyzing minerals and that anion and cation substitutions were likely. This is a user adjustable parameter and can be changed for different chemical systems.

While minerals were used as a test case, the software works with all subfiles and references contained in the Powder Diffraction File™. While the application was designed for XRF and microprobe analyses, any elemental analysis input can be used.

V. SUMMARY

A microanalysis capability has been developed that is able to detect phase composition from elemental profiles. Matches between experimental data and reference data are made by using two GOM algorithms that evaluate the quality of the match. GOM scores are normalized to 100 and scores above 90 usually signify that the top candidate matches the experimental data.

This software application can tolerate a range of cation and anion substitutions in isostructural materials and still make a positive identification even with lower GOM scores. Positive identifications are confirmed using associated crystal structure and atomic coordinate tables, both of which are part of the PDF-4 database. The application takes advantage of data mining capabilities in the world's largest database of mineral atomic and molecular structures and world's largest database of minerals.

This application was developed using the mineral subfile because of its comprehensive content and the availability of suitable experimental test data. However, the capability itself is generic and can be used with any subfile, or user define query, or collection of entries, in the Release 2018 PDF-4™ database family (PDF-4+, PDF-4/Minerals, PDF-4/Organics).

ACKNOWLEDGEMENT

This work is part of a multi-phase effort by the XRF subcommittee of the International Centre for Diffraction Data (ICDD). The authors are working as a task group of the subcommittee. Development of a microanalysis capability that interfaces with the PDF-4 family of relational databases completes the initial phase. Future objectives include interactive XRD-XRF or XRD-Microscopy/EDS analyses with multiphase quantitative analyses using applications developed within the database. The authors would like to thank ICDD member scientists and PDF users of the database for their input in developing new capabilities.

Faber, J., and Fawcett, T. G. (**2002**). “The powder diffraction file: present and future”, Acta Crystallogr. B: Struct. Sci. **58**(3), 325–332.

Faber, J., Weth, C. A., and Bridge, J. (**2004**). “A plug-in program to perform hanawalt or fink search-indexing using organics entries in the ICDD PDF-4/organics 2003 database,” Adv. X-ray Anal. **51**, 183–189.

Fawcett, T. G., Crowder, C. E., Kabekkodu, S. N., and Kaduk, J. A. (**2011**). “Improved material identification methods through targeted data mining,” Adv. X-ray Anal. **54**, 149–161.

Fawcett, T. G., Kabekkodu, S. N., Blanton, J. R., and Blanton, T. N. (**2017**). “Chemical analysis by diffraction: the powder diffraction file™”, Powder Diffr. **32**(2), 63–71.