



Australian X-ray Analytical Association (AXAA)

AXAA E-Newsletter

Issue 2006/03 July 2006

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X-RAY ANALYSIS TECHNICAL NOTES SECTION

1. Clay Analysis by XRD. II Identification of Clays (Author: Tony Raftery, QUT)

CALL FOR TECHNICAL NOTES (ESPECIALLY XRF NOTES):

The newsletter Technical Notes initiative is proceeding well according to our readers. We have now published 7 notes since the first appeared in issue 2005/2 in September 2005. Thanks to Ned Blagojevic, our energetic Secretary, these are now available at the AXAA` website as downloadable Tutorial Notes.

While the response for submitting XRD Notes has been excellent (6 of the 7 notes), we are experiencing much difficulty in twisting arms for the production of XRF notes? How about it?

Remember that the concept is to produce ‘best-practice’ notes, e.g. on matrix corrections. These are not intended to be original research publications.

We require approximately 1 page of text (tables can be included, but not figures or photographs). We can accept several pages if needed.

1. EDITORIAL

The time has come for another issue. There are many items of interest but I think the membership should pay special attention to the plans for the next Conference outlined in this issue. Now is the time to convey your thoughts on what should happen to the Council. There is no need for what has come before to constrain what will happen. I was impressed by the fresh outlook of the last meeting but I am sure that further new concepts would be welcomed – look below for what is already planned.

Tony Raftery
E-Newsletter Editor

2. MESSAGE FROM THE PRESIDENT ON AXAA-2008

The next AXAA National Conference (AXAA-2008), to be held at the Grand Hyatt Hotel in Melbourne from 4 – 8 February 2008, will be our biggest and best conference.

Please read Item 7 of this newsletter for ideas on the conference format.

Very soon, many of our members will be approached to participate by (1) contributing to program development, (2) nominating invited speakers – Australian and international, (3) giving invited papers, and (4) speaking at an advanced workshop session.

Please send your ideas on the program to me as we will be largely completing the invited speaker line up by the end-of the year.

Brian O'Connor
AXAA President

3. LETTER TO THE EDITOR FROM NICK PEARSON, ALCOA, WA

Is AXAA still the best name for our Association?

Over the years a significant number of x-ray scientists have become increasingly involved in analyses other than laboratory based XRD and XRF - historically the core activities for AXAA members. These 'new' analyses employ specialised techniques that may utilise x-rays generated by advanced sources, or radiations other than x-rays.

I would like to propose that the AXAA National Council and members consider whether the name AXAA should be changed to reflect the broader analytical techniques that our members are working with. Considerations in selecting an alternative name include:

- Maintaining representation for laboratory based XRD and XRF.
- Reflecting the advanced radiation sources and techniques being used (synchrotron x-rays; other parts of the synchrotron spectrum; neutrons and other 'particle' radiations).
- Extension beyond diffraction to scattering techniques more generally (including SAXS and SANS).
- Surface analysis techniques (e.g. XPS).
- Catchy acronym, but avoiding a name that is long and cumbersome when written out in full.

There are various ways such an association could be administered. For example there might be one "umbrella" Association, but allowing members to select areas of interest administered by sub-committees. This would allow members to stay informed regarding techniques, facilities, and developments in areas related to, or complementary to, their primary field of interest.

Such an organisation would benefit not only from increased membership, but from the diversity of members and interaction between disciplines.

Regards,
Nick Pearson

4. AXAA (WA) AND WA SOCIETY FOR MICROSCOPY [WASM] ANNUAL CONFERENCE, MARGARET RIVER, WA. 22–24 SEP 2006 – INCLUDING AXAA (WA) STUDENT SEMINARS

The Call for Papers and Registration forms are accessible from the AXAA website – see item 12. The annual *X-ray & Microscopy Conference*, organised jointly by AXAA (WA) and WASM, will be held at the All Seasons Conference Centre, Margaret River from 22-24 September 2006 (Fri – Sun). The conference brings together the two local communities to show-case research in x-ray, electron, optical and confocal microscopy. This conference provides a unique opportunity for researchers and professionals with an interest in x-ray analysis and microscopy to interact and share knowledge of new and developing techniques at the largest local meeting on these topics in Australia.

Set in native and traditional gardens, the All Seasons Margaret River is the perfect springboard to the attractions of the area which include world-class wineries, the world's best surf beaches, and the breathtakingly beautiful karri forests. Margaret River is a comfortable 3 hours drive south of Perth, approximately 300 kilometres, along the West Coastal Highway passing through the coastal towns of Mandurah, Bunbury and Busselton.

For general information please contact: Jeanette Hatch, Centre for Microscopy and Microanalysis, The University of Western Australia, Crawley WA 6009. email: admin@cmm.uwa.edu.au. Web site: [http://cmm.uwa.edu.au/AXAA\(WA\)](http://cmm.uwa.edu.au/AXAA(WA)).

Technical questions about the x-ray analysis aspects of the program should be directed to Rob Hart (AXAA-WA Branch Chair) – see item 9 of the newsletter for contacts.

5. NSW STUDENT SEMINAR, SCHOOL OF CHEMISTRY, SYDNEY UNIVERSITY. TUESDAY 19 SEP 2006

Flyers for the seminar are downloadable from the AXAA website – see item 12. AXAA congratulates the NSW branch for their initiative in staging the first of our AXAA Student Seminars which are being launched the National Council in partnership with the Branches.

This first student seminar has been organised by the NSW Branch Two new committee members for NSW, Catherine Kealley (ANSTO) and Vanessa Peterson (Sydney University), are working with Ken Turner to develop the program. Special thanks to Cat.

PLEASE SUPPORT THIS EXCITING INITIATIVE BY MAKING EVERY EFFORT TO BE THERE

For further information, contact Cat Kealley, cky@ansto.gov.au.

6. XRD COURSE X-RAY POWDER DIFFRACTION ANALYTICAL METHODS

Venue: Department of Imaging and Applied Physics, Curtin University of Technology, Bentley (Perth), Western Australia

Dates: 26-29 August 2006 (Sat – Tue)

Course Presenters: Professor Brian O'Connor and Dr Robert Hart

Enquiries: B.O'Connor@curtin.edu.au

Cost: \$1,650 including GST

Overview: The course has been designed to give participants a theoretical and practical grounding in the principal characterisation methods which make use of x-ray powder diffractometry data. Approximately 60% of the course will involve hands-on instruction. Students will personally collect diffractometry data sets and then process these, both manually and with PC computers, in exercises on various analytical methods, including Rietveld analysis. While the course is relevant to the analysis of all classes of crystalline materials,

attention will be devoted mainly to the material relevant to the mining and mineral processing sector.

7. PROGRAM FOR AXAA-2008

The next AXAA National Conference (AXAA-2008) will be held at the Grand Hyatt Hotel in Melbourne from 4 – 8 February 2008. The national Council plans to give attention to the use of laboratory x-ray techniques, synchrotron radiation analysis and neutron beam characterisation methods. It is intended to attract substantial numbers from the synchrotron radiation and neutron scattering communities as well as our traditional laboratory XRF and XRD supporters.

Design Principles

We aim to achieve 250 registrants, with a break even figure of 200. To achieve this target, we must bring in substantial numbers of synchrotron and neutron scattering users and also some SEM/TEM people. We must also aim to attract international x-ray analysts from the region.

We will retain the initial 2 days of workshops and schools. Days 3 and 4 will comprise conference sessions, and day 5 will be a half-day visit to the Australian Synchrotron.

We wish to achieve much greater integration of laboratory XRF and XRD people. Therefore the School sessions will be for new practitioners, and we will encourage experienced practitioners to attend the advance workshops on days 1 and 2 rather than the School sessions. The School sessions will be run along traditional lines with a small team of Australian instructors. We do not want to use our plenary and invited speakers for these sessions.

The Conference sessions will not be partitioned into XRF and XRD sessions. Rather, there will be 4 parallel sessions on particular themes.

We will again seed the program with invited speakers, but with space being left for contributed papers.

We must ensure that students are well catered for in the advanced workshops and conference sessions.

The poster sessions will be more substantial than at past AXAA meetings.

Program Development and Management

The AXAA-2008 Organising Committee will comprise the AXAA National Council plus Jane Yeaman of Tulips.

Program development and management will be handled as follows.

Program Committee

Executive Group – Brian O'Connor (Program Chair), Ian Madsen, Ned Blagojevich
Additional Members – AXAA Council Members, Bragg Institute nominee, Australian Synchrotron nominee

International Advisory Program Committee

Brian O'Connor (Chair)

Ian Madsen

Ned Blagojevich

Other members by invitation – predominantly prospective plenary and keynote speakers

Prospective Advanced Workshops (18)

1. Laboratory information management (XRF and XRD)
2. Quality assurance in x-ray analysis (XRF and XRD)
3. Calibration for quantitative analysis (XRF and XRD)
4. Sample preparation in XRF – the state of the art
5. Emerging instrumentation - hardware and software (XRF and XRD) (2 sessions)
6. Complementary analytical methods (XRF)
7. Complementary analytical methods (XRD)
8. Energy dispersive XRF for bulk sample analysis
9. XRF analysis in electron microscopy

10. X-ray absorption methods
11. Micro-XRD and Micro-XRF
12. Neutrons in industry (2 sessions)
13. Synchrotron radiation in industry (2 sessions)
14. Rietveld XRD analysis at the cutting edge
15. Data mining in XRD analysis

Each workshop session will require 3 invited speakers. Therefore the total number of speakers for the advanced workshops will be 54.

Prospective Conference Thematic Sessions (21)

There will be 21 thematic sessions run via three parallel streams as for AXAA-2005.

Therefore we will require 63 speakers with a substantial number of these being invited.

Where possible we wish to avoid partitioning of the XRF and XRD people through there being XRF and XRD streams.

It is proposed that the thematic sessions be divided into the broad categories:

- Instrumentation
- Analytical methods
- Materials-focussed analytical science, e.g. alumina, iron ore, base materials, environmental, forensics, etc, etc
- X-ray analysis in the region
- Etc

Brian O'Connor

8. ICDD LUDO FREVEL CRYSTALLOGRAPHY SCHOLARSHIPS 2007: CALL FOR APPLICATIONS

To encourage promising graduate students to pursue crystallographically oriented research, the International Centre for Diffraction Data (ICDD) has established the Ludo Frevel Crystallography Scholarship Fund to honour the founder of the fund, Dr. Ludo Frevel. The ICDD has awarded 71 scholarships since 1991. The year 2005 Scholarship Awards were in the amount of \$2,500 (US) each. Since the scholarship's inception in 1991, over \$154,000 has been awarded to aspiring crystallographers. Applications for the year 2006 awards must be received by ICDD no later than 31 October 2006.

Qualifications for the applicant:

The applicant should be a graduate student enrolled in a graduate degree program during the 2007 calendar year, with major interest in crystallography e.g. crystal structure analysis, crystal morphology, modulated structures, correlation of atomic structure with physical properties, systematic classification of crystal structures, phase identification and materials characterisation. There are no restrictions on country, race, age or sex. The term of the scholarship is one year. The recipient may make application for one renewal at the end of the first year. Because a limited number of scholarships are awarded, renewal applications will be considered on a competitive basis in conjunction with all applications that have been submitted up to the closing date.

Application will consist of:

Letter of recommendation from the student's principal research supervisor. (limit 1 page). Letters which provide specifics on the student's distinguishing qualities and accomplishments as a researcher are helpful in evaluating the applications.

A description of the candidate's proposed research (limit 2 pages) including: purpose and rationale for the research, proposed methodology to be used in the study, references and/or descriptions of the scientific background for the proposed research

A CV that includes: educational preparation (institutions, dates, degrees obtained and in progress, and particularly pertinent coursework), awards, honours received, any research publications and/or presentations given, any work experience (dates, employers, positions), professional activities, memberships

Evaluation of applications:

The amount of available funding limits the number of scholarships that can be granted in any given year. A selection committee will evaluate the applications received to determine which are most deserving of a scholarship. These evaluations consider both the proposal (impact, innovativeness, originality, efficacy of approach, and relationship to crystallography) and the student (recommendation letter, educational track record, prior work and/or research, honors, awards, and professional activities) in determining which applicants will receive the award.

There is a limitation of one award per educational institution. In the event that two or more candidates from one institution are considered to be among the top applicants, only one will be given an award.

Restrictions on the scholarship fund:

The scholarship award is to be used by the graduate student to help defray tuition and laboratory fees. A portion of the award may be applied to registration fees to accredited scientific meetings related to crystallography.

No more than one scholarship will be awarded to applicants at any one accredited institution per year.

The funds of the scholarship are not to be used for travel.

Applications must be **received by 31 October 2006**. Fax transmittals will be accepted until 31 October 2006, however a hard copy of the application must be mailed and received by ICDD no later than 1 December 2006. E-mail applications are not acceptable. Please mail to:

Scholarship Awards Committee
C/O Secretary, International Centre for Diffraction Data
12 Campus Boulevard
Newtown Square, PA 19073-3273 U.S.A.
Fax: +610-325-9823

9. RECENT PUBLICATIONS ON X-RAY ANALYSIS

- X-ray Spectrometry: Volume 35, May/June 2006

Article of interest:

Chemical analysis of phosphate rock using different methods - advantages and disadvantages. Madian Jamil Safi et al

ABSTRACT:

Many analytical methods are considered for chemical analysis of phosphate rock because the accuracy of analysis is very important. In the present investigations, spectrophotometric and x-ray spectrometric methods were used to determine P₂O₅ whereas SiO₂, CaO, MgO, Al₂O₃ and Fe₂O₃ were determined by gravimetric and x-ray spectrometric methods. Volumetric and x-ray spectrometry methods are necessary for fluorine determination. The investigations revealed that x-ray spectrometry is a precise and accurate technique, which is cost effective and so better than other methods. Spectrophotometric, volumetric and gravimetric methods are expensive, time consuming and laborious. X-ray spectrometry is suggested for commercial companies using regular analyses to save chemicals, time and labour and to maintain quality.

- J. Appl. Cryst. Volume 39, June 2006

Article of interest:

SVDdiagnostic, a program to diagnose numerical conditioning of Rietveld refinements. P. H. J. Mercier et al.

SYNOPSIS:

Singular value decomposition tools developed here to assess the comparable numerical stability of crystallographic and crystal-chemical types of refinement described in the work by Mercier *et al.* [*J. Appl. Cryst.* (2006), **39**, 369-375] have general applicability for troubleshooting problematic Rietveld refinements.

{COMMENT FROM BRIAN O'CONNOR: This paper appears to usher in a breakthrough for Rietveld practitioners troubled by calculation instabilities. Who isn't?}.

- Powder Diffraction, Volume 21, June 2006

Article of interest:

X-ray backscatter imaging: Photography through barriers. J. Callerame.

ABSTRACT:

X-ray imaging techniques based on Compton backscatter permit inspection and screening of sea containers, a wide variety of vehicles, luggage, and even people. In contrast to more commonly used transmission images, backscatter imaging involves positioning both source and detection apparatus on only one side of a target object. This presents the user with inspection opportunities in situations that may be extremely difficult, if not impossible, for transmission systems that require access by the detector subsystem to the opposing side of the target. The backscatter image is somewhat akin to a photograph of the contents of a closed container, taken through the container walls. Techniques for producing X-ray images based on Compton scattering will be discussed, along with wide-ranging examples of how systems based on these principles are used to perform inspections for both security applications and for the detection of contraband materials at ports and borders. Potential applications in the area of nondestructive evaluation will also be considered. Differences in the type of information displayed by transmission and backscatter images will be highlighted, and tradeoffs between backscatter image quality and interpretability, scan speed, effective penetration, and X-ray tube voltage will also be discussed. The method used in scanning the target object results in an extremely low radiation dose, a result that significantly broadens the application spectrum for this imaging technique.

10. COMPANY NEWS

GBC Scientific Equipment

GBC wish to introduce you to eMMA

eMMA is the name given to our enhanced Mini Materials Analyser, the compact high performance diffractometer which has set a new standard in economical XRD instrumentation.

That so many advanced options are now available, gives testimony to the integrity and flexibility of the basic design. There are over 25 units installed and successfully operating world-wide (with more than 3 units installed in some countries).

Specifically, eMMA now offers, in addition to all the options available for the original MMA—

Theta/Theta geometry, better for environmental stages

- New high speed microprocessor, which allows 8 simultaneous axes, Ethernet comms, and motor driven autoslits.
- Bulk sample stages for very large and heavy samples.
- Confocal mirror optics for transmission methods.
- Capillary sample spinner for transmission methods
- X-ray Fluorescence attachment operating in TXRF or Secondary target polarised geometry for elemental identification and analysis.

If you would like to meet eMMA we would be pleased to introduce you at our Applications Laboratory in Melbourne, after the Denver X-ray Conference in mid August.

Application notes are available for your perusal at www.gbcsoci.com under "Publications".

CONTACT: Rod Clapp, GBC Scientific Equipment, 12 Monterey Road, Dandenong, Vic 3174 email:Rclapp@gbcsoci.com

PANalytical

PANalytical's MiniPal: Fast, cost-effective analysis of solid and powdered samples

PANalytical introduces the MiniPal QC, the latest addition to the MiniPal series of EDXRF spectrometers, which sets the standard for delivering rapid and accurate analysis exactly when and where you need it.

MiniPal QC makes on-site X-ray analysis even easier and more effective with a sample surface up presentation. With its Silicon Drift Detector, the MiniPal QC sets new standards for EDS systems in quality and process control. Its automated capability allows MiniPal QC to be integrated with remote systems and laboratories, and to act as a back-up for larger systems such as CubiX and MagiX. Users will enjoy high up-time due to the specially designed dust collection device for easy user maintenance.

MiniPal QC is robust, safe, reliable, and cost-effective – ideal for sectors such as cement, mining, and industrial minerals. And advanced software makes it extremely easy-to-use. With the touch of one button, non-expert personnel can achieve expert analysis for process and quality control, recycling, incoming material inspection and identification of unknown samples.

For more information go to: www.panalytical.com

XRF Scientific Ltd

WHO is XRF Scientific Ltd?

XRF Scientific Ltd is the parent company of some of the most significant analytical industry supply and manufacturing companies in Australia.

XRF Scientific Ltd combines the talents of:

- Automated Fusion Technology Pty Ltd
- Modutemp Furnaces Ltd
- Analytical Platinum Supplies Pty Ltd
- Precious Metals Engineering
- Laser Analysis Technologies Pty Ltd
- X-ray Flux Pty Ltd

Direct contact with those operators of each individual business within XRF Scientific Ltd is still only a phone call away with the advantage of a complete network across Australia.

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- Electric or Gas Fusion; Crucible/Mould or Mouldable combinations.
- Flux, pre-fused granular Lithium Tetraborate/ Lithium Metaborate
- Specialised Nickel Flux; designed to save you more through accurate analysis.
- Platinum Laboratory ware, manufactured in two locations Perth & Melbourne

Contact: XRF Scientific Perth (08) 9244 9600, Melbourne. (03) 97206339

Web www.xrfscientific.com Email info@xrfscientific.com

Thermo Electron Corporation

Thermo Electron Corporation announces the launch of a new Wavelength Dispersive XRF instrument series: the ARL ADVANT'X series with IntelliPower Technology. With the increased analytical power of the X-ray tube, this novel series is now the most powerful XRF available with output up to 4200 watts. The new ARL ADVANT'X series of sequential XRF instruments is designed to give increased performance with three versions enabling increased application capability via a number of different power levels.

Visit our website: <http://www.thermo.com/elemental>

Inel

Inel announces new goniometer less diffractometer – Equinox

X-ray diffraction has always been a slow [analytical technique](#) due mainly to the need to rotate a heavy X-ray source and detector in a circle around the sample which in turn has required complicated, expensive and very slow mechanical drive systems. The new X-ray diffractometer is called Equinox and Inel says its unique design has allowed it to eliminate all of the complex mechanical systems normally found in diffraction systems. In its place is a rugged and very high resolution position sensitive detector that can measure all angles of diffraction simultaneously and in real time.

More details: <http://www.laboratorytalk.com/news/spa/spa110.html>

11. CALENDAR OF EVENTS

Dates	Event	Location	Further information
19 July	AXAA National Council Teleconference	N/A	B.O'Connor@curtin.edu.au
7-11 Aug 2006	55 th Denver X-ray Conference	Denver, Colorado	http://www.dxcicdd.com/
26-29 Aug 2006	XRD Course <i>X-ray Powder Diffraction Analytical Methods</i> Coordinator: Brian O'Connor	Curtin University, Perth	B.O'Connor@curtin.edu.au
1-4 Sep 2006	EPDIC10 – European Powder Diffraction International Conference	Geneva, Switzerland	http://www.sgk-sscr.ch/EPDIC10/EPDIC10.html
19 Sep 2006	AXAA (NSW) Student Seminar	School of Chemistry, University of Sydney	Cat Kealley: cky@ansto.gov.au
22-24 Sep 2006	2006 AXAA(WA) – WASM Joint Conference	Margaret River Convention Centre at All Seasons, Margaret River, WA	http://cmm.uwa.edu.au/AXAA(WA)-WASM06
27-29 Sep 2006	ICDD Rietveld Refinement & Indexing Workshop	ICDD headquarters, PA, USA.	www.icdd.com
25-27 Oct 2006	ICDD Specimen Preparation for XRF Workshop	ICDD headquarters, PA, USA.	www.icdd.com
20-23 Nov 2006	Asian Crystallographic Association (AsCA)	Tsukuba, Japan	http://www.realize-at.jp/AsCA2006/AsCA2006-CrSJ.htm

	Conference (AsCA'06)		
5 Dec 2006	NSW Branch Christmas Technical Meeting	Macquarie University, Sydney	ken.elaine@bigpond.com
4 -8 Feb 2008	AXAA National Schools and Conference (AXAA-2008)	Grand Hyatt Hotel, Melbourne	http://www.axaa.org

12. AXAA WEBSITE AND CONTACTS

WEBSITE <http://www.axaa.org>

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13. SUBSEQUENT ISSUES OF THE NEWSLETTER AND ADVERTISING

NEXT ISSUE (2006/4) - TO BE PUBLISHED (LATE) SEPTEMBER 2006

Final date for submission of material: 31 August 2006

Material other than company news and advertising to be submitted to the Editor, Tony Raftery: see item 4 for contacts.

Company news and advertising to be submitted to Brian O'Connor: see item 4 for contacts.

Specifications for company news items:

Items to be non-sales focussed, eg staff appointments, product releases. There is no charge for companies which have exhibited at recent AXAA meetings and/or have sponsored recent AXAA events. Limit of 25 lines of 12-point text per company.

Specifications for advertisements

Text only

40 kB limit

Charge per advertisement = \$100/10kB plus GST, based on 1 page per 10 kB.

FURTHER ISSUES SCHEDULED FOR 2006: SEPTEMBER (late), NOVEMBER (late)

X-RAY ANALYSIS TECHNICAL NOTES SECTION

Clay Analysis by XRD II. Identification of Clays

Tony Raftery, Faculty of Science, Queensland University of Technology GPO Box 2434
Brisbane, QLD 4001. Australia. E-Mail: a.raftery@qut.edu.au

Preamble

Having prepared the (hopefully) highly orientated clay mounts, the procedure of clay identification involves a series of treatments. The basic ones are normal (air dried), glycolation and heat treatment (350C). There are other treatments that are used on the basis of what the standard treatments suggest – e.g. heating to higher temperatures (550C), ion exchange, and treatment with formamide. These will be touched on when specific clays are to be confirmed or identified. Glycolation can either be with ethylene glycol or glycerol but the standard treatment is ethylene glycol atmosphere at 80C for at least 1 hour (beyond heating up time), however sometimes 4 hours is required as some samples react more slowly than expected.

Basis of Identification

It is hoped that the clay preparation has produced a highly orientated specimen with the (00 ℓ) planes (basal spacing) parallel to the sample surface. If this is so, then a strong series of basal reflections - (001), (002), (003), (004), etc. – will be present for each clay. For some clays, some of these reflections are systematically absent or suppressed.

One advantage of clay identification is that there are only seven clay classes (depending on whose classification scheme you adopt) - some argue that some classes are not true clays. I'll settle for illite/mica, kaolinite/kandite, smectite, chlorite, palygorsite (or attapugite), vermiculite and sepiolite. However, there is a series of known {and suspected} mixed layers – the most widely studied is illite-smectite (but also kaolinite-smectite, chlorite-smectite, etc.). The following is directed to the identification of classes and interlayers. It is generally not possible to identify specific minerals within a class. It is usually necessary to prepare powder preparations (of the clay extracts) and use the other hkl lines to differentiate members within a class (or group) - for instance, there are many minerals within the chlorite group.

Illite

Illite is distinguished by the (00 ℓ) series 10A, 5A, 3.33A. It is unaffected by glycolation and heat treatment (350C). It is perhaps the easiest to identify. There may be a slight problem with quartz overlap at 3.34A. The peaks may be narrow (mica like) or broad (illite like) – and where the line should be drawn is open to debate. Iron rich clays such as glauconite and celadonite have severely suppressed 5A peaks. The only possible confusions are palygorsite at 10.4A and hydrated halloysite at 10A but these are distinguished by the absences of the other characteristic illite peaks and the presence of the other palygorsite or halloysite peaks.

Kaolinite/Kandite

It is a big class which ranges from the very ordered (narrow and intense diffraction peaks) to the very disordered (weak and broad diffraction peaks) and beyond to true halloysites.

Dehydrated halloysite is about 7.2A (hydrated halloysite at about 10A – it is difficult to keep the halloysite hydrated in my air-dried orientated clay preparations and I try usually to ensure that it is dehydrated). Halloysite can be distinguished from disordered kaolinite by the lack of high preferred orientation in the sample preparation (the 4.45A hkl is stronger than it ought to be) and by a test. Reaction to formamide within 30 minutes is indicative of halloysite (kaolinite will react to formamide but more slowly – a period of hours or days or weeks). Halloysite will expand to 10A. It is not unusual to have both halloysite and disordered kaolinite present. If the sample fails to react in 30 mins then there is little or no halloysite present and only disordered kaolinite is present.

The characteristic lines of kaolinite are 7.1A and 3.57A. Possible confusions are chlorite (14A, 7A and 3.53A). It is not unusual for both kaolinite and chlorite to be present in a particular sample.

Kaolinite survives heat treatment (350C) but not at 550C. Kaolinite is unaffected by glycolation.

Chlorite

Chlorite is a large diverse group – the characteristic lines are 14A, 7A, 4.72A and 3.53A. There is some variation in line position but not enough to confuse chlorite for kaolinite but enough to make mixtures of kaolinite and chlorite problematic. One could use formamide to expand the kaolinite (this is very slow for ordered kaolinite but the problems of overlap tend to be with the less ordered kaolinites that react within a period of hours). Chlorite is unaffected by formamide so any reaction indicate kaolinite.

So called iron chlorites have suppressed 14A and 4.72A peaks (hence problems with kaolinite confusion).

There is possible confusion with vermiculite.

Chlorites generally survive heat treatment (350C and 550C), but some chlorites do not and in particular, iron chlorites collapse. It should not be used to differentiate chlorites from kaolinite.

Chlorites are unaffected by glycolation.

Vermiculite

Vermiculite has spacings at 14.5 A, 7.2A, 4.80A and 3.58A. The ratio of peak intensities depends on ion saturation. If vermiculite is suspected, it is usually best to prepare a Mg-saturated air-dried preparation (which gives strong 14.5A and weak 7.2A), glycol with *glycerol* (not ethylene glycol) for the 14.4A does not expand (it will slightly expand with ethylene glycol).

Palygorsite

Palygorsite is fibrous and hence does not orientate. It is detected by its characteristic lines at 10.45A (100), 6.4A (15), 5.4A (10), 4.45A (15) and 3.65A (10). The ratio of intensities can vary sample to sample. It is not likely to be confused with any other clay.

Sepolite

Sepiolite is fibrous and hence does not orientate. It is detected by its characteristic lines at 12.8A (100), 7.6A (5), 5.1A (10), 4.4A (35) and 3.77A. (broad). The ratio of intensities can vary sample to sample. It is not likely to be confused with any other clay.

Smectite

Smectite is a diverse group. In air-dried samples it has a peak in the range 12A to 15A which on ethylene glycolation it expands uniformly to 17.2A (the peak usually sharpens and increases in intensity with glycolation - also an often observed 002 peak occurs at 8.5A – there is no 002 peak in the air-dried oriented samples). Confirmation (if required) is to K-saturate the air dried preparation and heat to 300C - the first diffraction peak collapses to an illite-like 10A.

Mixed Layers

The most difficult area is the identification of mixed layers. They tend to be mixed layers with smectite, eg. illite/smectite, kaolinite/smectite, chlorite/smectite but also chlorite/vermiculite, illite/vermiculite and chlorite/kaolinite. There may be others not as yet recognized.

It is beyond the scope here to consider all types but there are two main classes – ordered (or regular, R1) and disordered (or random, R0).

It is easy enough to think of the disordered types (R0) as some clay layers at irregular interval existing in other layer types. This give rise to broadening and shifting (sometimes it is very slight) of diffraction peaks. An example is a disordered kaolinite/smectite with 20% smectite. There is a slight shift to a higher d-spacing of the basal spacing [d(001)] on ethylene glycolation (with a slight sharpening) and a retreat to about the air-dried value on heating to 375C (with slight broadening). It is subtle and may be missed (one would need also to be confident of the equipment and sample positioning). The relationship of broadening, shift and intensity is dependant on the repeat distances involved – for a particular mixed layer, some peaks may be broadened others not, some enhanced in intensity others depressed.

The regular mixed layers (R1) have quite different diffraction peaks from the individual clays and the random mixed layers. The ordering leads to superstructures. For instance e-glycolated regular illite/smectite (50/50) [also called rectorite] – has spacings at 27A (10+17), 13.5A, etc. Regular chlorite/smectite has d(001)* at 14+17= 31A. Regular mixed layers are 50/50, however there may be a 50/50 ratio and no ordering (i.e. random mixed layer, R0). Certain mixed layer clays are found only in certain mixing ratios, eg. chlorite/smectite is found 50/50 (regular,R0) and also <10% smectite only (R0), kaolinite/smectite is only found as R0.

A useful reference is *X-ray Diffraction and the Identification and Analysis of Clay Minerals*. D M Moore, R C Reynolds. 2nd edition 1997 Oxford University Press.
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