

MINUTES OF COMMISSION I

59th ICCP Meeting, Victoria, 20 and 21/08/2007

Chair: Walter Pickel, Secretary: Deolinda Flores

Opening remarks

The commission I meeting was held on Monday afternoon and Tuesday morning and attended by 31 and 30 members and 8 and 6 guests respectively. The Chairman outlined the programme for the sessions and presented the opening remarks.

The working groups and items presented and discussed in the meeting were:

- SCAP - Single Coal Accreditation Program
- Standardization Working Group
- Handbook Editorial Group
- New Methodologies and Techniques in Organic Petrology
- Degradinite Working Group
- Peat Petrography Working Group
- Temporal variations of coals
- Microscope session

Commission I activities also included a presentation by Carl Hilgers of the latest developments and improvements of the “Hilgers” instrument for reflectance and maceral analysis.

The Chair of Com I reminded the audience of the service to calibrate standards against the ICCP Reflectance Standard available from Dave Pearson and Walter Pickel to interested laboratories (USD50 and free for ICCP members). Until now more than 30 laboratories had their standards checked against the ICCP standards.

The opening remarks covered the progress in the revision of the ISO 7404 Standards (Parts 2, 3 and 5). They are ready as CD version and, at this moment, are circulated within the ISO country members for voting. The Chair requested ICCP members to approach the institutions of their countries for voting.

Concerning the Standardization WG, Walter informed that during next year two round robin exercises will be performed, one on one or two lignite samples, one on a bituminous coal sample. It was also noted that the convener should be careful with instructions and purpose of the exercise that must be clearly written and presented. A subsequent discussion arose regarding the way to approach the exercise. The convener took the advice on board.

Nine members enrolled for the exercise on the bituminous coal at the meeting. Members, who were not able to attend the meeting but are interested to participate, are welcome and can request a sample from Walter Pickel.

The lignite round robin will be held among the same members as the last lignite exercise. Additional members are of course welcome.

SCAP - Single Coal Accreditation Program - Kimon Christanis

The Convener presented the report summarising the 2006 SCAP Exercise sent to the Chair of the Accreditation Sub-committee on 21st February 2007 as follows:

The 2006 SCAP exercise began in February 2006, when the Organizer invited by e-mail all participants of the 2004 exercise, as well as all the ICCP members to take part. The exercise was also announced in ICCP NEWS 37 (March 2006).

The participants

About 82 analysts (plus two Automatic Image Systems) working at 42 laboratories located in 17 countries expressed their willingness to participate in the 2006 exercise. The numbers of both the analysts and the laboratories by country are as follows:

- 21* from Australia (9 labs),
- 4 from Brazil (1 lab),
- 4* from Canada (2 labs),
- 2 from Colombia (1 lab),
- 1 from Czech Republic,
- 2 from Denmark (1 lab),
- 13 from Germany (7 labs),
- 4 from India (1 lab),
- 2 from New Zealand (1 lab),
- 1 from Poland,
- 2 from Portugal (1 lab),
- 1 from Serbia,
- 4 from South Africa (3 labs),
- 2 from Spain (1 lab),
- 5 from The Netherlands (2 labs),
- 6 from United Kingdom (2 labs), and
- 10 from the United States of America (7 labs).

*One Automatic System is included.

About 49 participants are ICCP members, 33 are non-members, whereas both Automatic Systems are operated by ICCP members. Comparing with 2004, the 2006 exercise lost 15 participants and won 29 new participants. From the new beginners, 9 analysts were already ICCP members, while 4 others have applied for and gained, in the meantime, ICCP membership. Four participants are postgraduate students.

The timetable

After registration and invoicing, two or six coal samples have been mailed to the analysts in April. As the number of the available un-mounted samples was limited, also polished blocks from the SCAP collection have been distributed, preferably to the new participants.

The return of data was completed by mid of October. Typing and calculation errors found in the data received from the analysts have been corrected until the end of the month.

The assessment of the VR and VC data was completed by early December, the participants have been informed about their performance in early December, and the certificates were dispatched before the turn of the year. Since mid February 2007 the list of the accredited petrographers with all their contact details is available on ICCP's webpage.

It has to be noted that following the practice of the previous Organizer and according to the exercise timetable announced in February 2006, a preliminary assessment procedure

was scheduled. Due to difficulties and delays with the data assessment as described below, the preliminary assessment could not be carried out.

The assessment

From the 82+2 participants, 80 submitted results for both VR and VC accreditation, one for VR and two for VC accreditation only. One participant has not submitted results, although the deadline was extended till mid of October.

The main problem the 2006 exercise had to face, was the reconstruction of the full dataset of all the individual VR and VC results submitted by the participants in all the past exercises and, of course, the accurate calculation of the group mean (GM) and the standard deviation (SD) for each coal. For a few of the 17 coals used in the previous exercises, small differences (affecting the GM and SD values of some coals after the third decimal place) in the GM and SD values appeared among the files received from the previous Organizer.

In order to solve this problem, the Chair of the Accreditation Sub-Committee, the President and the Organizer cooperated 'digging' for several weeks in all the files of the past exercises. The dataset resulted in what can be seen as the possibly most complete.

Thereafter the calculations of the GMs and the SDs for each coal, as well as of the UMSD, SMSD, AUMSD and ASMSD values for each participant was carried out manually using EXCEL.

It is worthwhile to note that the software prepared by Paddy Ranasinghe for the SCAP assessment could not be used at this time as it prerequisites a correspondence between all the data loaded and participants; these were not known for some previous exercises. Paddy, however, made all necessary arrangements and the program can be used in the 2008 exercise.

The results

The results are as follows:

- from 81 VR candidates, 77 participants awarded accreditation (VR failure 4.9%),
- from 82 VC candidates, 77 participants awarded accreditation (VC failure 6.1%).

The failure percentages are higher than in the previous exercises but we have to take into account the great number of the new beginners this year. Three of them (among four, in total) failed in VR and three others (among five, in total) failed in VC accreditation, respectively. From the participants, who were not accredited, only one (new beginner) failed in both VR and VC analyses. It also needs mentioning that another new participant applied visual estimation of the maceral-group contents and thus was excluded from the evaluation.

One appeal has been submitted to the Accreditation Sub-Committee. This could not be accepted as there was not any problem with sample identification, result interpretation or data analysis.

After finishing the exercise, polished blocks have been distributed to a few failed participants upon request for training and improving their practice.

Remarks & Suggestions

After the experience gained from the 2006 exercise I would like to suggest the following issues concerning the future SCAP exercises:

- A broad advertisement of SCAP in combination with the other accreditation programs has to be attempted (among the TSOP and CSCOP members, maybe also through the *International Journal of Coal Geology* and other Journals) in order to gain more participants.
- Between the deadline announced in the SCAP timetable and the final acceptance of the results a smaller tolerance is needed. This was my intention at the beginning but given the complications of the data evaluation, I accepted results sent up to mid of October.
- Participants awarded accreditation but having either AUMSD values within the danger zone or individual UMSD values extremely high can apply for analyzing six samples in the next exercise. In this case their old data will be ignored and they can be treated as new starters. However, all their VR and VC data will be included in the dataset for the GM and SD values calculation of the certain coals.
- One question requiring decision concerns the retention or removal of the VR and VC values of the failed participants. Up to the 2004 exercise, the data provided by the failed participants were removed from the dataset making, of course, the next exercise more difficult, as the SDs became smaller.
- One decision might be also necessary regarding the retention in or the removal from the dataset (for calculation of the GM and SD values) of extreme high or low VR or VC data provided by participants. If the participant does not fail, there are three options:
 1. To retain the extreme data in the dataset. This results in making the next exercises easier for the participants analyzing the certain coal.
 2. To remove the extreme data from the dataset. In this case the next exercises will be more difficult for the participants analyzing the certain coal due to a smaller SD.
 3. To establish a limit value, e.g. $5 \times \text{SD}$. If the VR or VC values provided by the participant are higher than the limit set, then this limit value (e.g., $5 \times \text{SD}$) has to be included in the dataset for the GM and SD calculations for each coal.
- Finally, it is necessary to repeat that sub-bituminous and bituminous coal samples (> 2 kg, on a dry basis, low mineral matter content) are welcome in the SCAP Sample Bank!

Acknowledgements

I would like to express my gratitude to the following colleagues for the continuous and sound support for running the 2006 exercise during the last year: all the members of the Accreditation Sub-Committee, the President, the Honorary Treasurer, the Secretary General, the previous Organizer and Paddy Ranasinghe.

Kimon made an appeal to all ICCP members to send coals to be included in the SCAP coal bank to be used in the next exercises. Coal samples are urgently needed if the ICCP decided to send bulk samples for the next exercise. Suitable samples must be not blend coals, have a vitrinite reflectance between 0.5% and 1.8% and be low ash coals with a mineral matter content ranging 8% to 14% (preferentially < 10%). Isabel offered to provide a sample. Alan expects to send 3 coal samples.

Com I congratulated Kimon for all his work and effort spent during the last two years to reorganize the SCAP as well as the conducting the 2006 exercise.

New Handbook Editorial Group – Petra David

Petra David presented the progress until 2006 on the activities on the preparation of the New ICCP Handbook Edition.

The text from previous Handbook versions and publications has been scanned and is available in editable text format. The sheets have been formatted for a common layout and pre-checked for consistency and a number of sheets have also been corrected for spelling and language mistakes.

Draft Structure of New Handbook Edition has been defined on previous meetings as follows:

- 1 Introduction
- 2 Definitions
- 3 Lithotypes
- 4 Microlithotypes
- 5 Maceral groups
- 6 Classification of dispersed organic matter
- 7 Methods
- 8 Coal Utilization
- 9 Other terms
- 10 ICCP services
- 11 Glossary of terms
- 12 References

For major parts, the text for a new Handbook Edition is available and the first version of it will be published as a CD ROM or DVD. For the preparation of the first version, with a minimum of information included, the status of the material available is the following:

Definitions (Chapter 2)	Text available and reviewed
Lithotypes (Chapter 3)	Text available and reviewed
Microlithotypes (Chapter 4)	Version as approved by GA
Maceral groups (Chapter 5)	
Vitrinite	Published
Inertinite	Published
Liptinite	As approved by GA
Classification of dispersed organic matter (Chapter 6)	To be drafted and added at a later stage
Methods (Chapter 7)	Text is available but needs major revision
Coal utilization (Chapter 8)	Incomplete Chapter reviewed and partly revised Chapters on coke and combustion residues to be drafted
Other terms (Chapter 9)	Comprises published sheets on Graphite, Semi-

graphite, Natural coke, Natural char, sheets on bitumens and oxidation to be added later

ICCP services (Chapter 10) To be drafted, but information largely available

Glossary of terms (Chapter 11) To be drafted and added at a later stage

As it can be noted in the table there is some work that must be done. Following the recommendation drawn at Bandung last year it is necessary to extent the Editorial group and to find volunteers who coordinate different activities and/or review and revise existing texts and draft new ones. The assignment of volunteers to prepare work and to coordinate the preparation of the chapters that are not yet covered was also discussed. Some members agreed to incorporate a small group to do this hard work. These items and collaborators are:

Lithotypes – Peter Crosdale

Microlithotypes – Walter Pickel

Maceral groups – Kimon Christanis

Coal Utilization – Petra will contact Alan Davis

Other terms – Barbara Kwiecińska and Henrik Petersen

Concerning Classification of dispersed organic matter item Petra reported of an existing ICCP/TSOP classification but there are not definitions of the terms. Angeles will send the Table with the classification in order to arrange some members to prepare the texts of the missing definitions.

Major activities are not yet covered such as: (i) Compile references in a consistent format and create a small database; and, (ii) Collect photomicrographs and create a small database. Alan will prepare notes on desirable requirements needed for this ICCP publication concerning format and resolution of the pictures.

This year an email was sent to all ICCP members asking for assistance. Several members answered positively and were provided with a username and password. To proceed activities during the next year, Com I agreed to use the secure area of the ICCP website. The Forum will be used for discussion and uploading files. Petra showed to the audience how the secure area of the ICCP webpage works and how it can allow members to use it. An announcement will be made in the ICCP News letting ICCP members know that all the documentation is available on the secure area of the webpage and members interested to collaborate can ask for a username and password.

It was also discussed that it will be possible to make some changes on the published texts as the final version of them, even the one already published, must be approved by the General Assembly.

Finally Petra thanked all volunteers and those who have indicated their willingness to participate in this work to prepare the New Handbook Edition.

Degradinite Working Group – Peter Crosdale

A report of the 2006-2007 activities was summarized by the convenor. Background information about Degradinite and its microlithotype Hydrite was presented and a detailed discussion of this problem was already published in ICCP News No.26, July 2002. It was noted that it occurs in: (i) Tertiary of Japan (Asai and Tanno, 1956), New

Zealand (Crosdale, 1993, 1995 but called bituminite); Indonesia (Crosdale, pers. comm.) and Malaysia (Dr Wan Hassia, pers. comm.); (ii) Oligocene of Upper Bavaria (ICCP, 1963), and (iii) Jurassic of Surat Basin, Australia.

For the 2006 – 2007 exercise 2 polished thin sections of coal samples from the Miocene North Wanganui Basin of New Zealand were prepared by a commercial laboratory (unfortunately the sections turned out to be very poorly made). A series of photographs of the same field were made in transmitted, reflected and fluorescence modes (by Alan Cook) from 4 different areas (set A, B, C and D) and distributed to participants. A selection of 20 points was made on one of the photomicrographs (set B) and participants asked to identify all macerals – giving as much information as they thought useful.

3 members performed the exercise and the results showed that there are macerals that are undoubtedly identified (8 points) by all participants, others some agreements were reached (5 points) and others with no agreement (7 points). The critical aspects were discussed by the audience. A discussion within these aspects had already taken place during the microscope session where the polished thin sections were looked at and discussed.

The status of ‘degradinite’ still remains uncertain. Many would agree that it is synonymous to bituminite, in which case one of the terms is redundant. It was proposed to do more polished – thin sections as they proved to be extremely useful but need a good lab to prepare suitable samples.

It was pointed out the necessity to have a chemical composition of the maceral mainly some microprobe analysis. It was suggested to ask Lila Gurba or Colin Ward to perform this analysis. A discussion on this subject started during the microscope session and continued after the presentation of the 2006-2007 activities. It was agreed by those present that this maceral belongs to the liptinite group, thus the note on degradinite in the vitrinite definitions should be removed.

Peat Petrography Working Group –Kimon Christanis and Stavros Kalaitzidis

A report on the 2006 round robin exercise and a summary of the 2004-2005 round robin exercises were presented by Stavros. 2004-2005 round robin exercises were performed using a photo-gallery file distributed among the participants. Each participant was asked to use his own-understanding “terms”. In 2004 11 members conducted the exercise and in 2005 only 9 members replied.

In 2006 exercise 2 polished blocks were distributed to the 10 interested analysts. They were asked to perform qualitative assessment, description of macerals and to define possible problems. They were also invited to take photos from usual and specially unusual aspects. Only 4 participants replied, 2 participants sent only images, 1 participant gave images and qualitative description and 1 participant replied and included comments, maceral analyses, reflectance data and images. Selected photos of previous exercises were presented and discussed. A discussion arose about the identification and differences between densinite and atrinite shown in selected pictures.

Next year, the convenors will circulate all the material among WG participants and using webpage forum to discuss unusual and problematic aspects. When an agreement is reached a polished block will be sent to the participants of the WG to perform maceral analyses. Angeles suggested that Convenors should ask specific questions in the exercise. A final report of all activities will be prepared and presented in Oviedo.

Temporal Variation of Coals – Lopo Vasconcelos

Lopo presented the draft final results of the WG. Up to now the WG managed to produce a data base with 7623 data of world coals, which includes information on the following parameters: Sample identification (Continent, Country/Territory, Coal Basin, Coal Field, Sample Reference, Coal Seam, Type of Sample), Age (Era, Period, Stage), Type of Basin, Petrography (mmf, H,V,L,I, V%R), Maceral Groups (old and new classifications), Minerals, Microlithotypes, Carbominerites, Minerite, Literature.

The following steps are (i) to make a final revision of the data base to detect possible and probable errors/mistakes, (ii) to include the data base in the ICCP webpage in a protected area and (iii) to present a final report of the WG at the Oviedo Meeting in 2008. It was also decided that the WG will produce some interpretation diagrams that will be included in the website in an open area, with ICCP logo for copyrights.

New methodologies and techniques in Organic Petrology – Lila Gurba

On behalf of the Convenor Zhongsheng Li presented a talk on the new advances on Electron Microprobe Analysis and Micro-FTIR of macerals and their applications in coal utilization.

Variations in the elemental composition of macerals in coals over a wide range of coal ranks, using light-element electron microprobe techniques, to establish the coalification tracks of key macerals in a single coal-bearing interval from subbituminous through bituminous coal to anthracite have been studied and presented.

Attenuated total reflectance micro-Fourier transform infrared (ATR-FTIR) spectrometry has been used to characterise coal macerals, in particular telocollinite, changes in the aromatic and aliphatic functional groups, over a wide rank range in coals.

Inorganic elements were found in electron microprobe studies of coal macerals, especially vitrinite macerals, without any visible minerals or mineral inclusions in the macerals concerned. Detailed mapping of the concentration of these elements in macerals of several low-rank coals reveals that a majority of inorganic elements occur as non-mineral entities rather than discrete mineral particles, and are intimately distributed throughout the macerals; the distribution of these elements is very similar to that of organic S, particularly in the collotelinite and fusinite of the same coal samples.

Further, the applications of these studies in coal utilization such as: Coal Utilisation, Ultra-clean Coal Combustion, as well as CBM production and CO₂ sequestration in coal seams, were presented.

Finally, these studies permitted to draw the following conclusions:

Microprobe analysis can indicate:

- Coalification tracks of macerals with rank advance
- Rank in coals with suppressed reflectance
- Inorganic elements that may react unfavorably in combustion or gasification or may survive demineralisation processes
- Detailed nature of cleat and other mineralisation that may affect gas drainage or CO₂ sequestration

Micro-FTIR of macerals may further identify:

- Functional groups in different macerals, including aliphatic/aromatic characteristics of vitrinites with normal and suppressed reflectance

Applications

- Calcium may be a problem in slagging, but may have favorable catalytic effects in gasification processes

Presentation of a New Organic Petrology Microscope - Carl Hilgers

The “ Hilgers” petrologic microscope was first presented to the ICCP at the Patras meeting in 2005. Since then, the software “Diskus Fossil” has been considerably upgraded and modifications to the microscope have been introduced.

The system presented at the Victoria meeting included the microscope Leica DM 4000 M, a Märzhäuser scanning stage Ecostep with a coaxial manipulator, a Basler black and white camera and the software Diskus-Fossil version 4.80.

Light emitting diodes (LEDs) have replaced halogen and mercury lamps for illumination. The diodes require no warm-up time to reach stability and they do not generate heat. Reflectance can be measured as soon as the system is turned on and calibrated. According to the manufacturer the life cycle of an LED exceeds 10.000 hours by far.

The microscope can be simply switched to fluorescence mode at any time during maceral analysis and reflectance measurement, without a need to re-calibrate for reflectance analysis afterwards. Expensive mercury- or xenon-arc lamps have been replaced by economic and durable LED bulbs with an excitation wavelength of 440 nm. The change of illumination is done by a simple key stroke\click on the interactive screen surface and by pulling out the white light LED which is mounted on a barrier filter that blocks the blue light, when in the light path.

A new ‘live’ measurement mode enables the user to take measurements at any location on the image area continuously.

Rmin/max can be measured by means of a rotating stage. At present an automated rotating polariser is going to be developed to enable Rmin/max determination also with microscopes not equipped with rotating stages.

The option to combine maceral analysis and reflectance measurement in one operation provides quick information about the sample (rank, single coal or blend) and a saving of time for the operator.

The system provides the option to save all images during reflectance measurement and in fluorescence mode.

Analysis reports including all data of the reflectance measurement or maceral group and maceral analyses as required by ISO standards can be printed The data can alternatively

be exported into ASCII files, which can be easily imported in software like MS-Excel to create individual test reports.

For contact details of Carl Hilgers please contact Walter Pickel.

Microscope Session

The microscope session took place on Monday afternoon and Tuesday morning using Carl Hilgers system and was attended by a large number of members.

Samples from the Degradinite WG were looked at and discussed at the Microscope Session. Peter gave an introduction addressing the problem that was to be discussed and looked at as his presentation following the session.

It was consensual that by using only microscope observations it is not possible to advise if Degradinite belongs to the vitrinite group, liptinite group or elsewhere. It is necessary to approach the problem using other techniques to compare with other materials. On the sequence of this discussion, it was suggested to ask Lila Gurba for microprobe analysis. John Crelling volunteered to perform some microanalyses to have all the data on the material before deciding where it belongs.

Com I would like to thank Carl Hilgers for this opportunity and for all technical assistance during this microscope session.