Tracing the maceral origin in combustion chars. The inertinite in Combustion WG of ICCP.

Report of the 1999 Round Robin exercise on CD optical microscopy images of coal chars

ICCP

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REPORT OF THE INERTINITE IN COMBUSTION 1999 CD ROUND ROBIN EXERCISE

by Angeles Gómez Borrego

Introduction

In 1997 a coal char of a South African inertinite-rich high volatile bituminous coal (Rr=0.73%, coal K) was analysed using manual microscopy analysis. In 1998 a virtually vitrinite-free coal of similar rank was analysed (Rr=0.66%, coal N). The agreement was relatively poor (Borrego et al., INCWG97 and INCWG99) and it was decided to go further with the activities including both further chars to analyse manually (Borrego et al., INCWG99) and a CD exercise to classify fields selected in optical microscopy images. The present report deals with the results of the CD exercise.

Classification system of carbonaceous material in chars

The classification system used was the same as in the manual char exercises, which essentially distinguishes between vitrinite and inertinite-derived materials, and, within the latter, porous *vs.* dense, isotropic *vs.* anisotropic and fused *vs.* unfused. This system is addressed at distinguishing those materials which differ in aspects which are thought to be of the major relevance during combustion: plastic behaviour, optical texture and porosity development.

Group 1 would include all kind of domains which could derive from vitrinite, and these can be isotropic or anisotropic, depending on the rank of the coal.

Group 2 would include the char materials which have been presumably formed from inertinites, but have been highly altered during pyrolysis, showing both anisotropic texture and an important porosity development ($\rho > 50\%$).

Group 3 would include those domains formed from inertinites which have developed an appreciable anisotropy without further porosity development ($\rho < 50\%$).

Conversely, the **Group 4** materials would be those which have passed though a plastic stage and generated a porous structure ($\rho > 50\%$) but do not show any signs of anisotropy.

Group 5 would include the isotropic dense domains with evidence of having fused (i.e.: small spherical degassing pores) ($\rho < 50\%$).

Group 6 would include all the least altered inertinites not showing cellular structure. They will be mainly massive isotropic material without any sign of having passed through a plastic stage.

Group 7 would only include the unchanged fusinites. They will be typically isotropic but might also exhibit wavy-like anisotropy.

<table>
<tr><td colspan="3">VITRINITE</td><td colspan="2">GROUP 1</td></tr>
<tr><td colspan="3"></td><td>POROUS</td><td>DENSE</td></tr>
<tr><td rowspan="4">INERTINITE</td><td rowspan="2">FUSED</td><td>ANISO</td><td>GROUP 2</td><td>GROUP 3</td></tr>
<tr><td>ISO</td><td>GROUP 4</td><td>GROUP 5</td></tr>
<tr><td rowspan="2">UNFUSED</td><td>MASSIVE</td><td colspan="2">GROUP 6</td></tr>
<tr><td>FUSINOID</td><td colspan="2">GROUP 7</td></tr>
</table>

The 1999 CD exercise occurred after discussion at the meeting in Wellington (1997) and Porto (1998). In the slide session following the presentation of the results, some of the problems with the assignments were addressed, specially those dealing with how to classify fusinites exhibiting wavy-like anisotropy, which is likely to be original and not generated during the pyrolysis process. It was agreed that this material should be included in the group 7 corresponding to unfused fusinites. Some other subjects discussed dealt with the criteria to distinguish between fused-dense and fused-porous inertinites. As experience shows that, after discussion of the micrographs, all participants tend to approach each other´s criterion, and as char preparation and distribution is rather expensive, it was decided to analyse for the 1999 Round Robin exercise the BB2 (coal N) sample already analysed in 1998, the sample used by the combustion working group of ICCP in their 1998 round robin exercise. The latter is a char from a medium rank (1.28 % vitrinite reflectance), inertinite-rich (54.2%) coal (coal L).

It was also decided that colour plates showing examples should be distributed in order to aid in the identification of the optical textures. Instead of micrographs a CD was prepared with micrographs showing examples of the different textures and structures, which should be included in each category. In addition, a CD exercise was arranged in which participants were asked to classify a set of images.

The 1999 RR CD Exercise content:

- A CD with two PowerPoint documents, the first one containing the examples of the materials to be assigned to each classification group, and the second one containing images with marked fields to be classified by the participants.
- Data of petrographic characterisation of the parent coals from which chars were obtained.

Instructions to carry out the exercise. The classification system was the same as in last years exercises. The only difference was the porosity limit (50%) between G2-G3 and G4-G5. Participants had to consider all the material around the mark exhibiting the same optical characteristics when taking a decision.

Participants:

A CD was sent to all participants in last year exercise and two other persons who manifested their interest.

12 Individuals from 6 different institutions took part in the exercise.

Henrik Petersen (DK)	Barbara Kwiecinska (PO)
Deolinda Flores (PT)	Manuela Marques (PT)
Bruno Valentin (PT)	Elizabeth Gawronski (AU)
Edward Lester (UK)	Dave Clift (UK)
Richelieu Barranco (UK)	M. Jesús García (ES)
Diego Álvarez (ES)	Angeles G. Borrego (ES)

Results from CD images were received from all participants but only 7 results of manual petrographic analysis

Results of the CD Round Robin exercise

Overall the results were very good. A summary of the percentage of identifications with indication of the level of agreement is shown in Figure 1. Ten or more out of 12 participants (>80 % agreement) agreed in the identification of 60% of the particles.

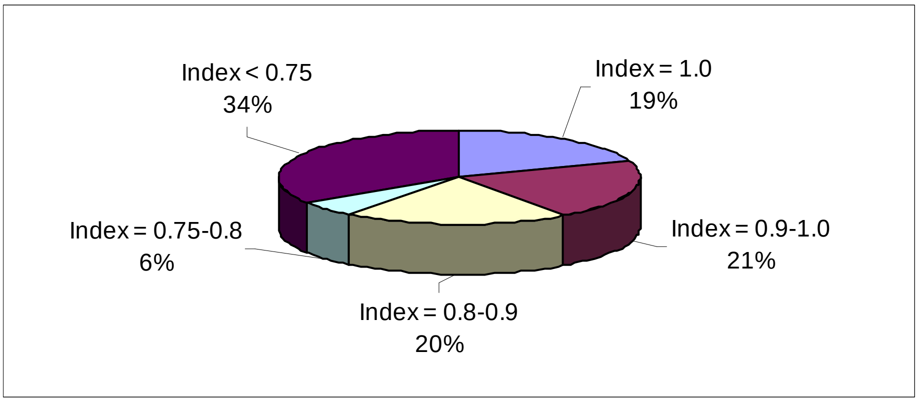


Figure 1. Percentage of fields with their corresponding Index of agreement.

In some cases, it was difficult to assign a texture to some of the fields due to the variety of assignments (see table of results). Despite this, modal values were calculated for all the fields, and Table 1 shows that all participants identified at least 70% of the fields according to modal values.

Table 1. Percentage of fields identified by each analyst according to modal values.

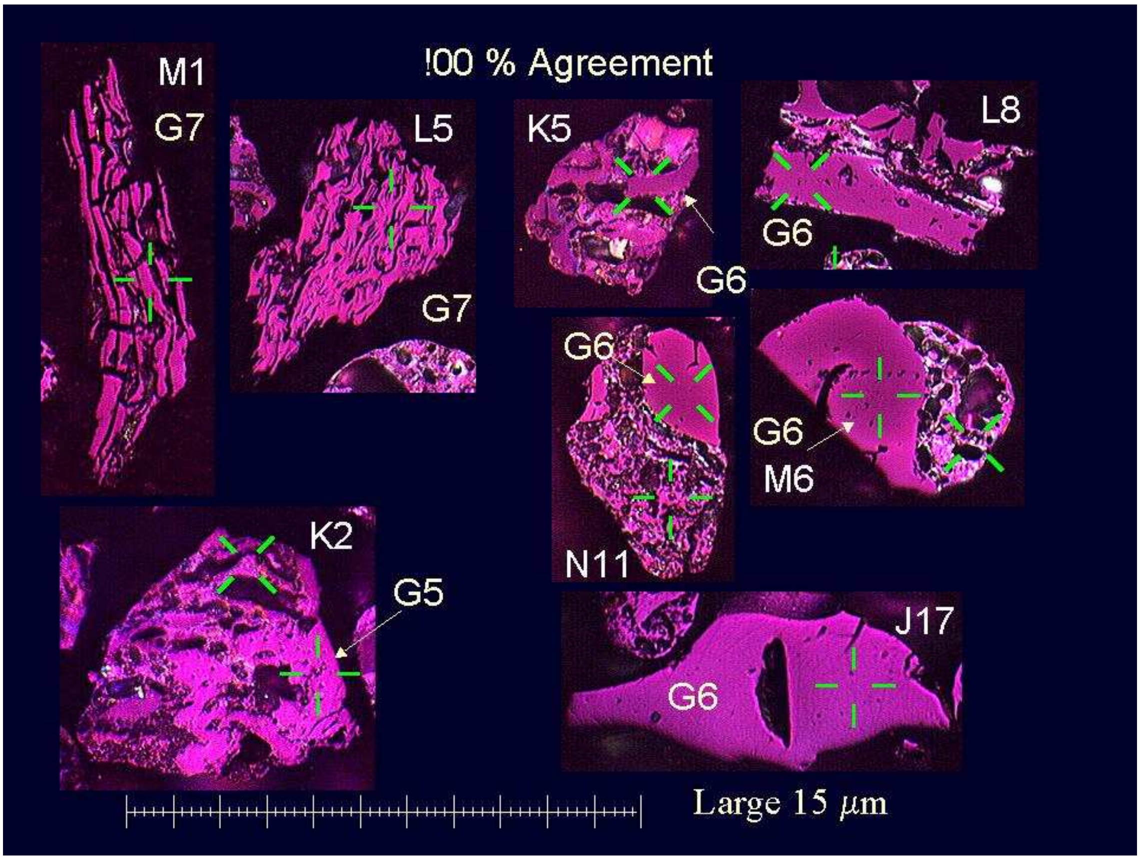
Participant	Identification according to modal values (%)
1	77.0
3	79.0
4	78.2
5	82.3
6	86.3
8	78.2
9	70.2
10	83.9
11	70.2
13	75.8
14	77.4
15	78.2

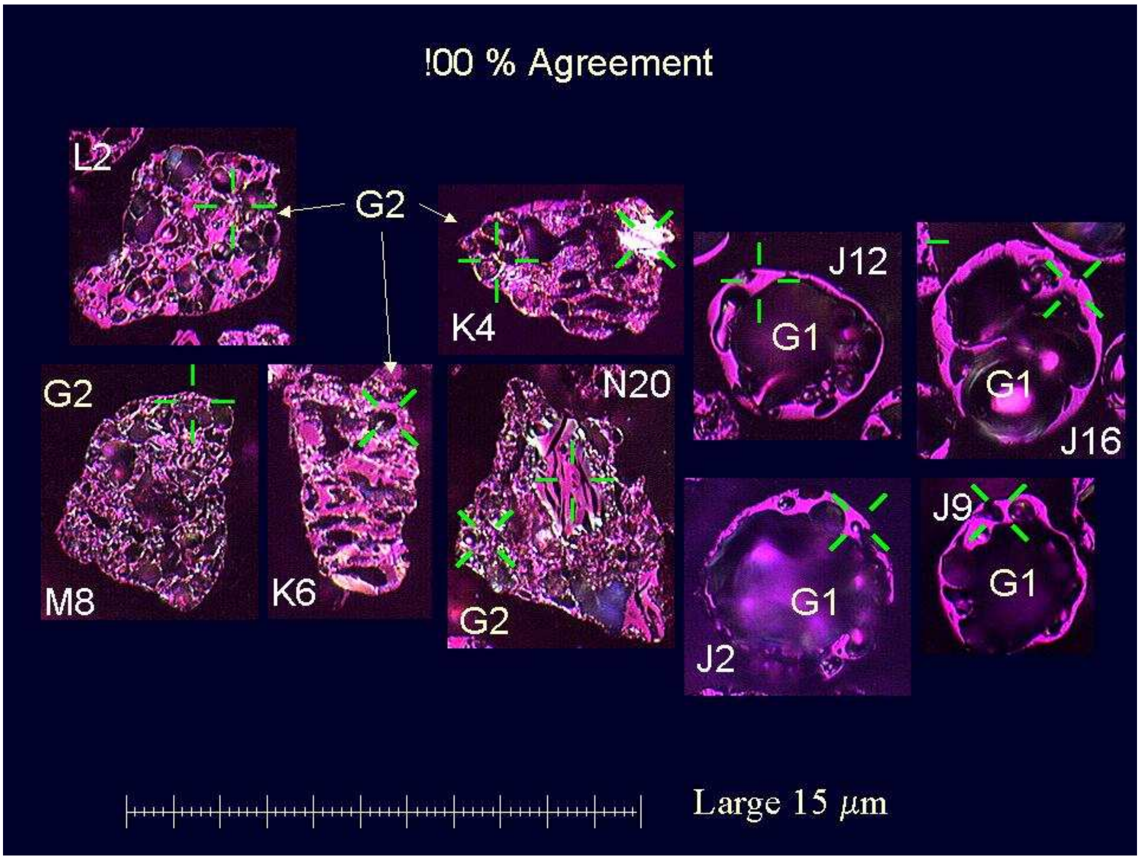
The reasons for dubious identifications are summarised in Table 2. It has been assumed that when the level of agreement was higher than 75%, this is, when at least 9 out of 12 participants agreed in the identification, the reasons for dubious identifications can be attributed to random distribution of mistakes. This occurred in 65% of the fields. In the remaining 35% there are usually two options, which concentrate most of the assignments and they are detailed in the table 2 along with the source of the discrepancy.

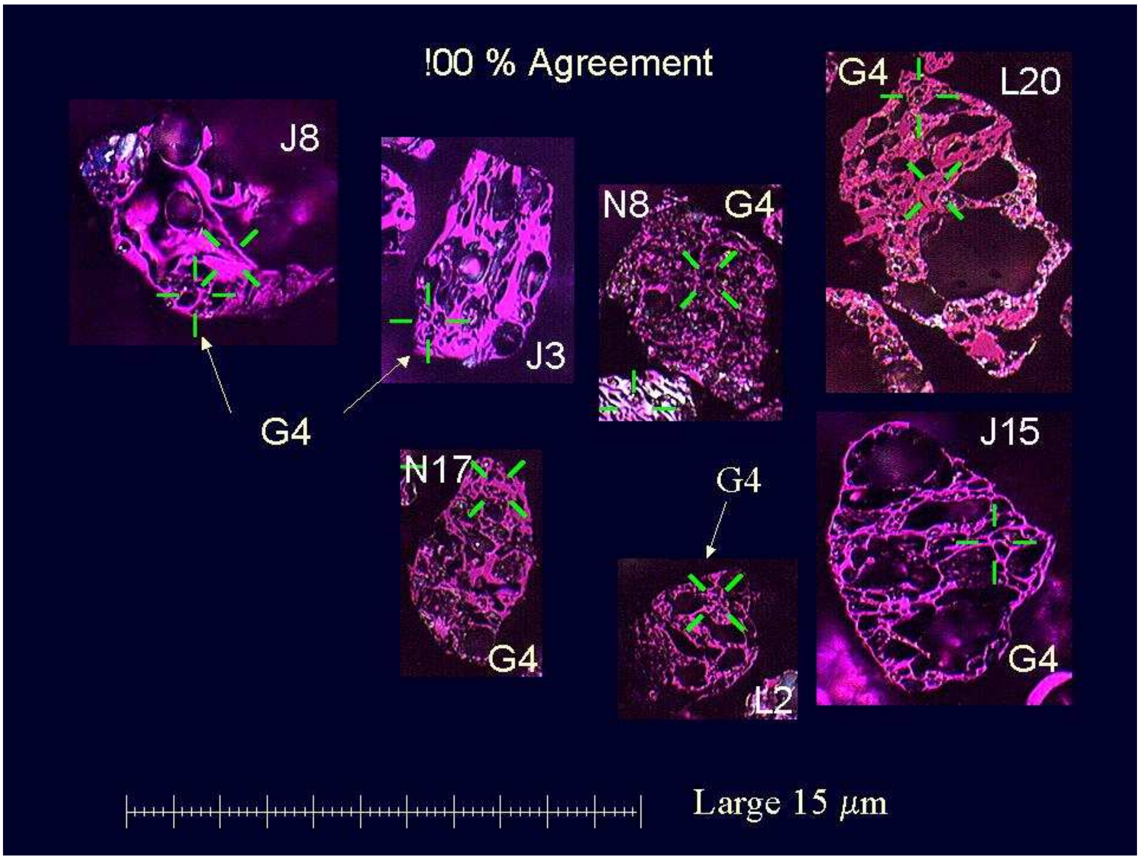
Table 2. Reasons for dubious identifications

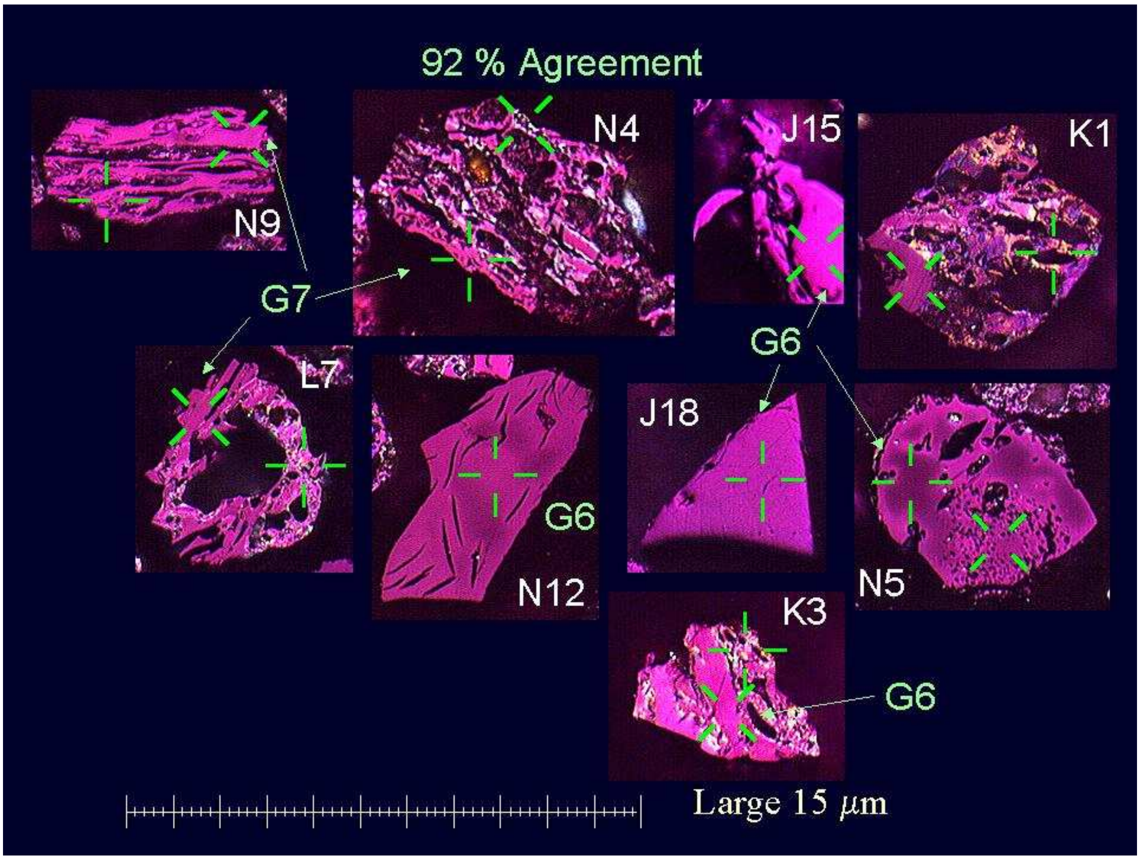
Class	Amount (%)	Sort of doubt
>75% Ag	65	
G1-G2	2	Vitrinite-derived in mixed particles
G1-G4	1	Vitrinite-derived in mixed particles
G2-G4	2	Isotropic/Anisotropic
G3-G5	2	Isotropic/Anisotropic
G2-G3	6	Porous or dense
G4-G5	8	Porous or dense
G5-G6	3	Did isotropic massive inertinite fuse?
G5-G7	2	Did isotropic fusinite fuse?
G4-G7	2	Did isotropic fusinite fuse?
G3-G7	3	Did anisotropic fusinite fuse?
G6-G7	4	Has unfused material fusinite structure?

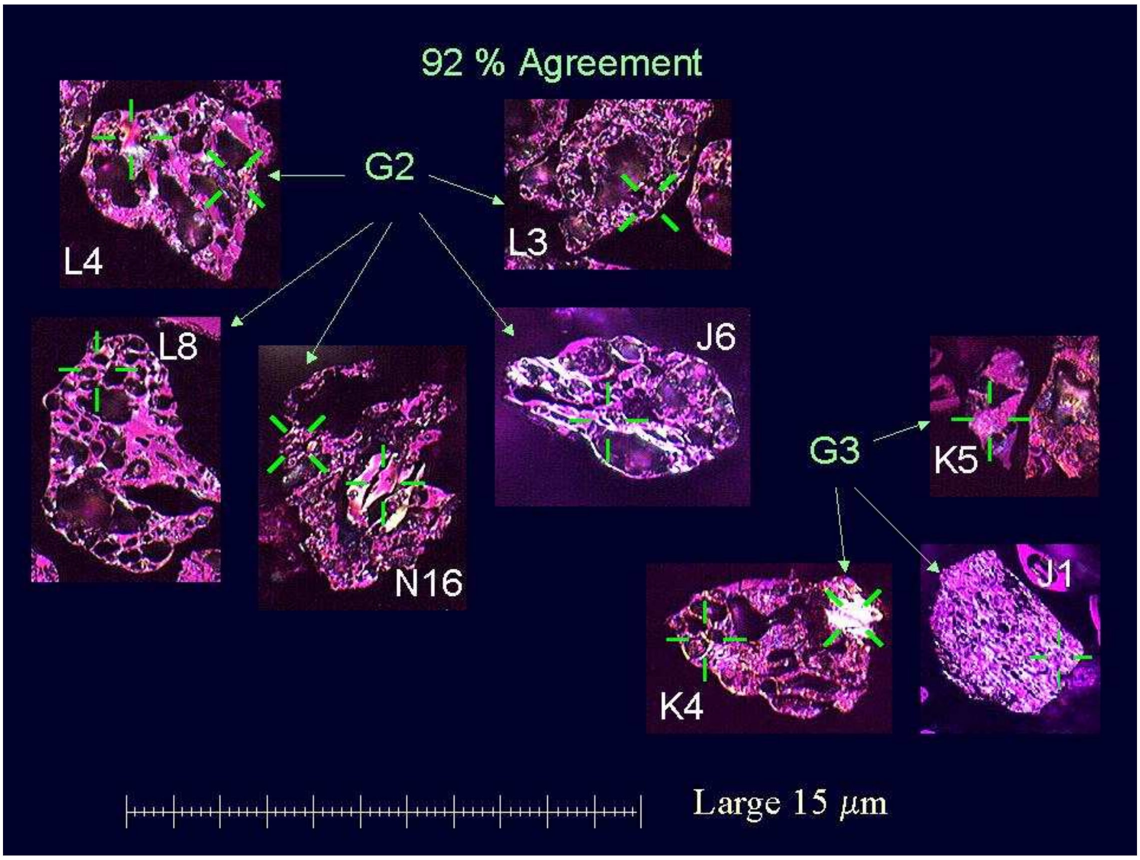
The following images are those classified during the 1999 CD exercises. They are ordered by level of agreement and therefore the first plates can be considered as very reliable assignments. The label of the images are those of the original exercises and do not necessarily coincide with those of the exercises which are now available in the website. The latter have been re-labelled for consistency. The equivalence between both labelling systems is available at the website (image.labelling.xls).

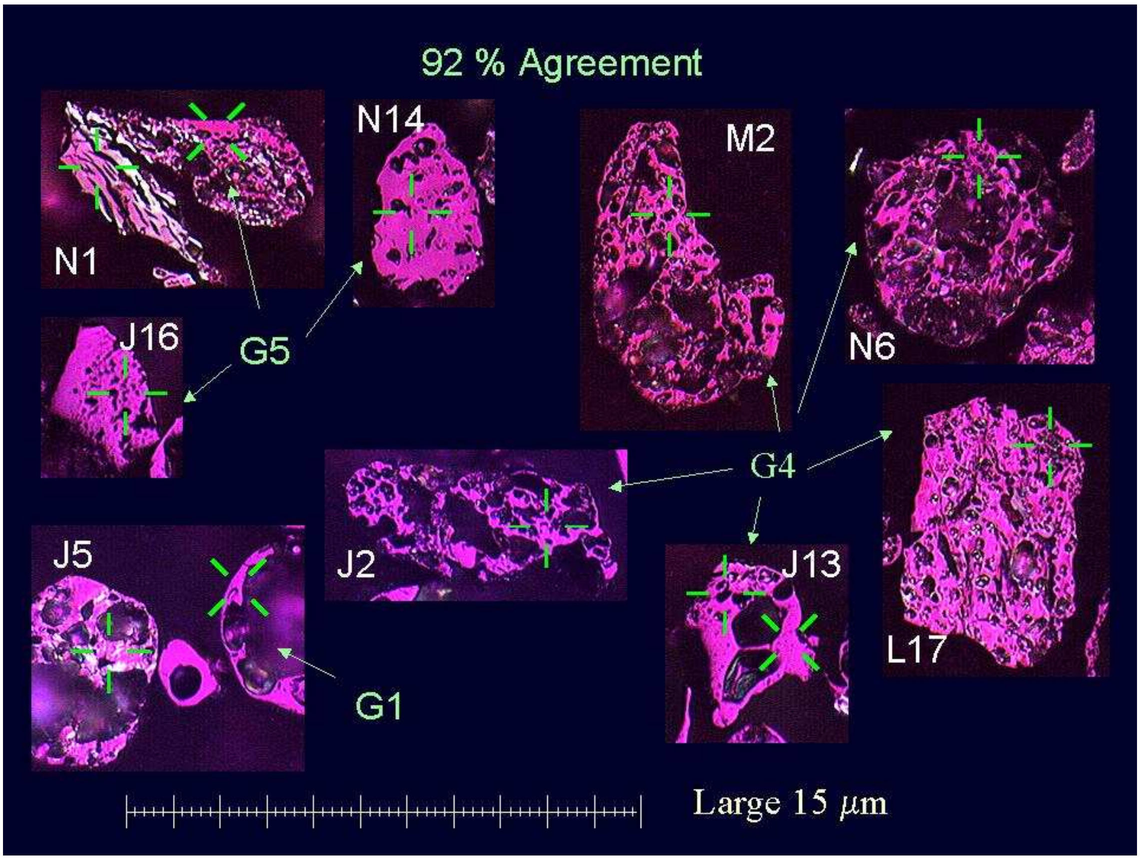


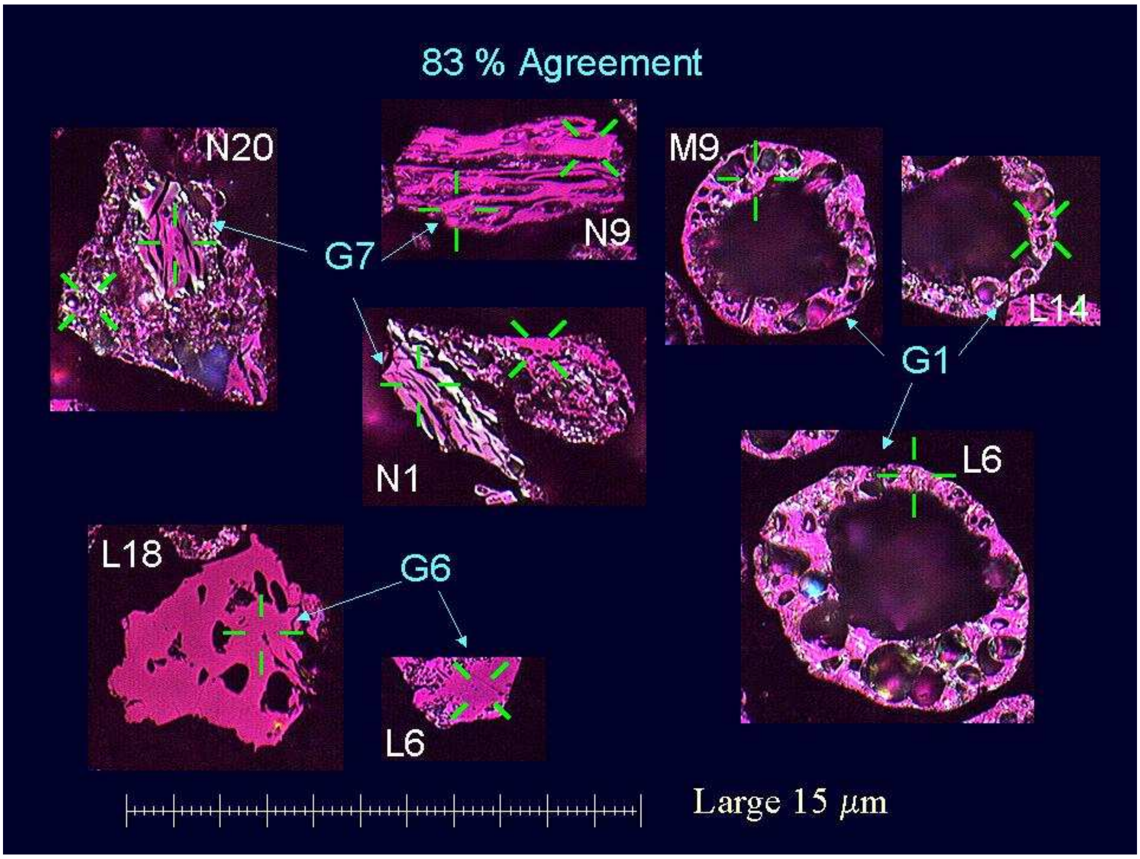

!00 % Agreement
L2
G2
K4
J12
G1
J16
G2
N20
M8
K6
G2
J2
J9
Large 15 μm

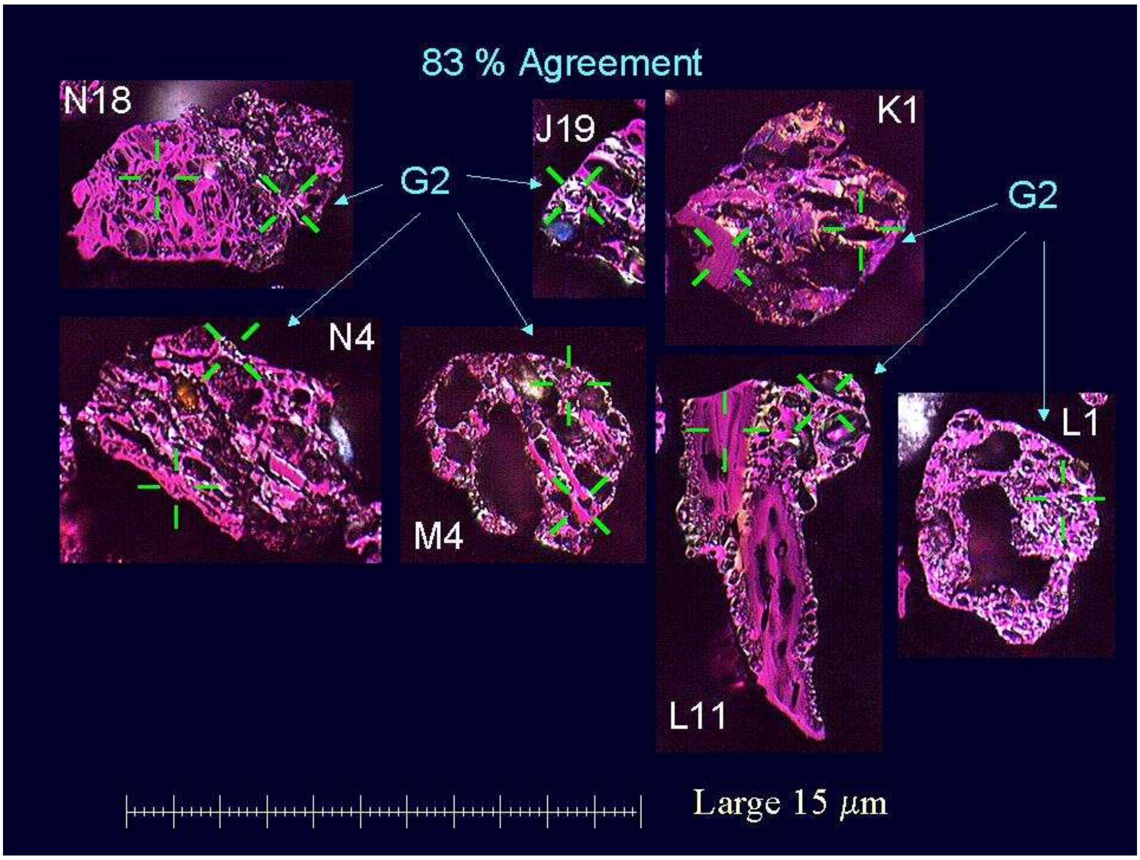

!00 % Agreement
G4
L20
J8
N8
J3
G4
J15
N17
L2
Large 15 μm

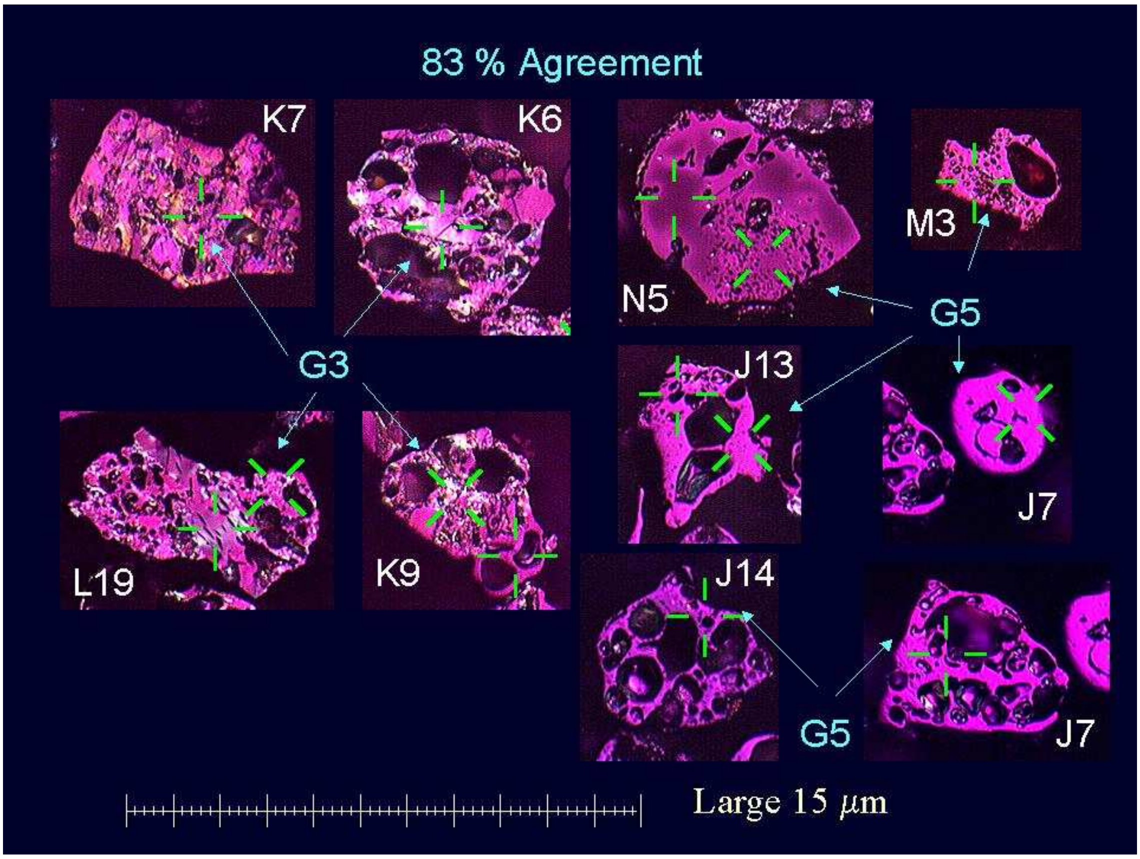

92 % Agreement
N9
N4
J15
K1
G7
G6
L7
J18
G6
N12
N5
K3
G6
Large 15 μm

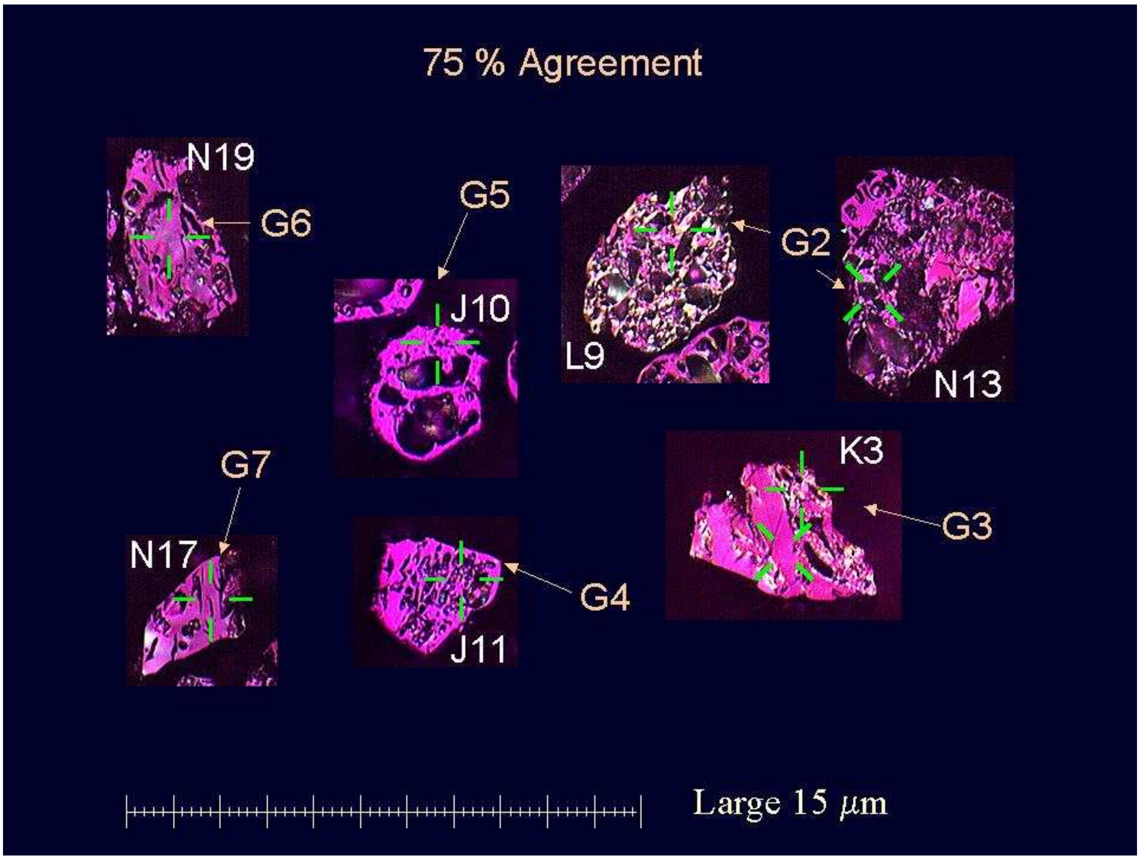

92 % Agreement
G2
L4
L3
L8
J6
N16
G3
K5
J1
K4
Large 15 μm

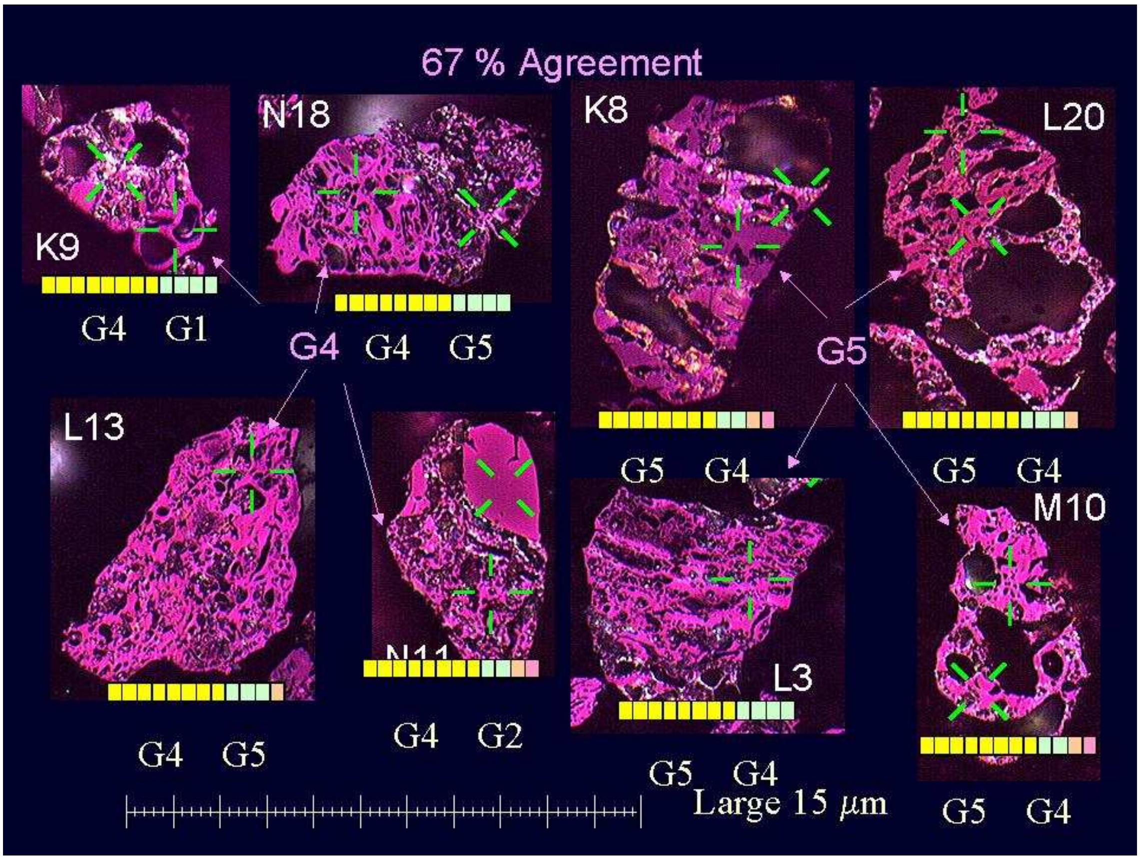

92 % Agreement
N1
N14
M2
N6
J16
G5
G4
L17
J5
J2
J13
G1
Large 15 µm

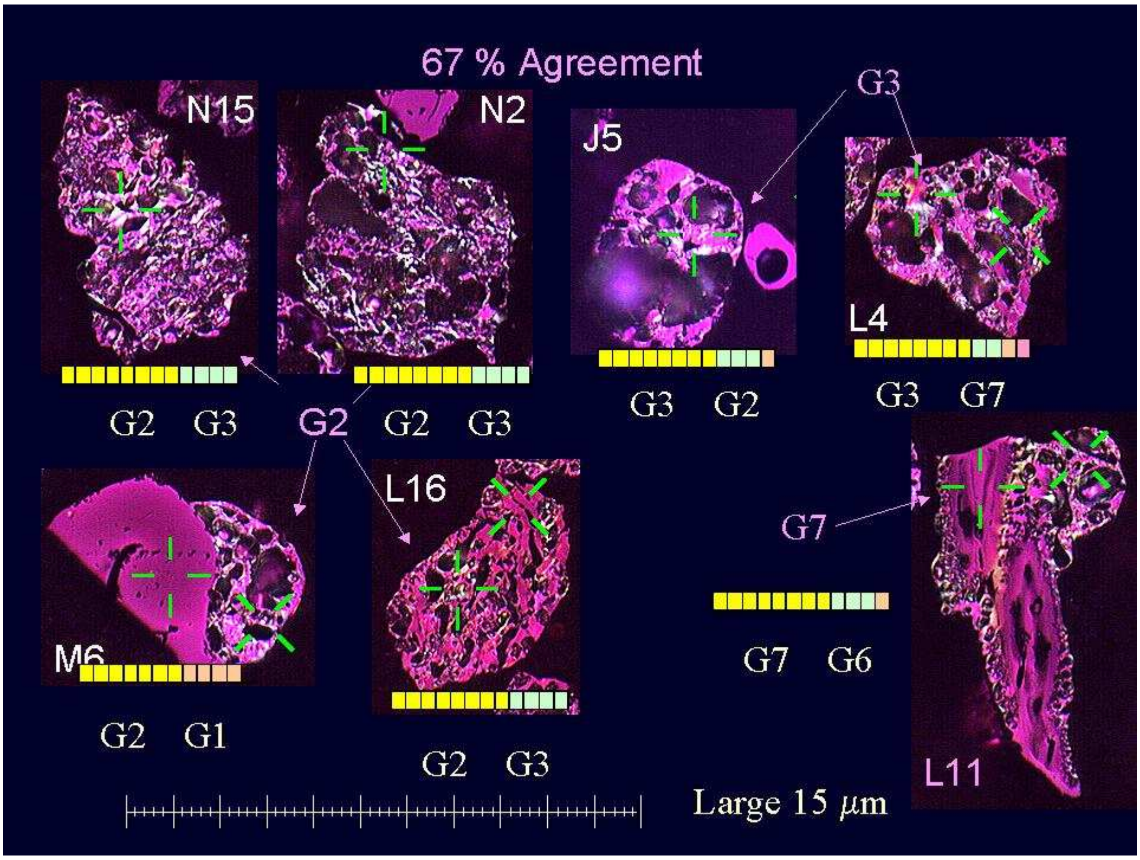

83 % Agreement
N20
N9
M9
L14
G7
N1
G1
L6
L18
G6
L6
Large 15 µm

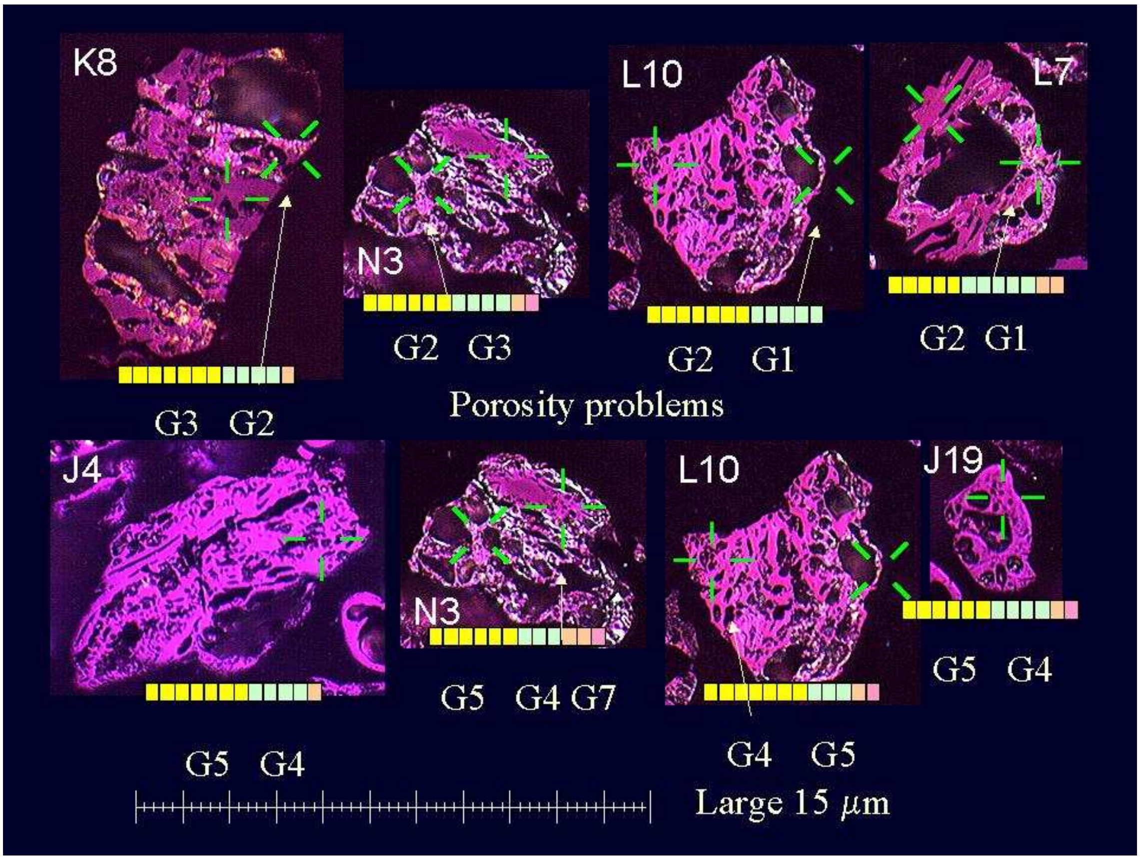

83 % Agreement
N18
J19
K1
G2
G2
N4
M4
L1
L11
Large 15 μm

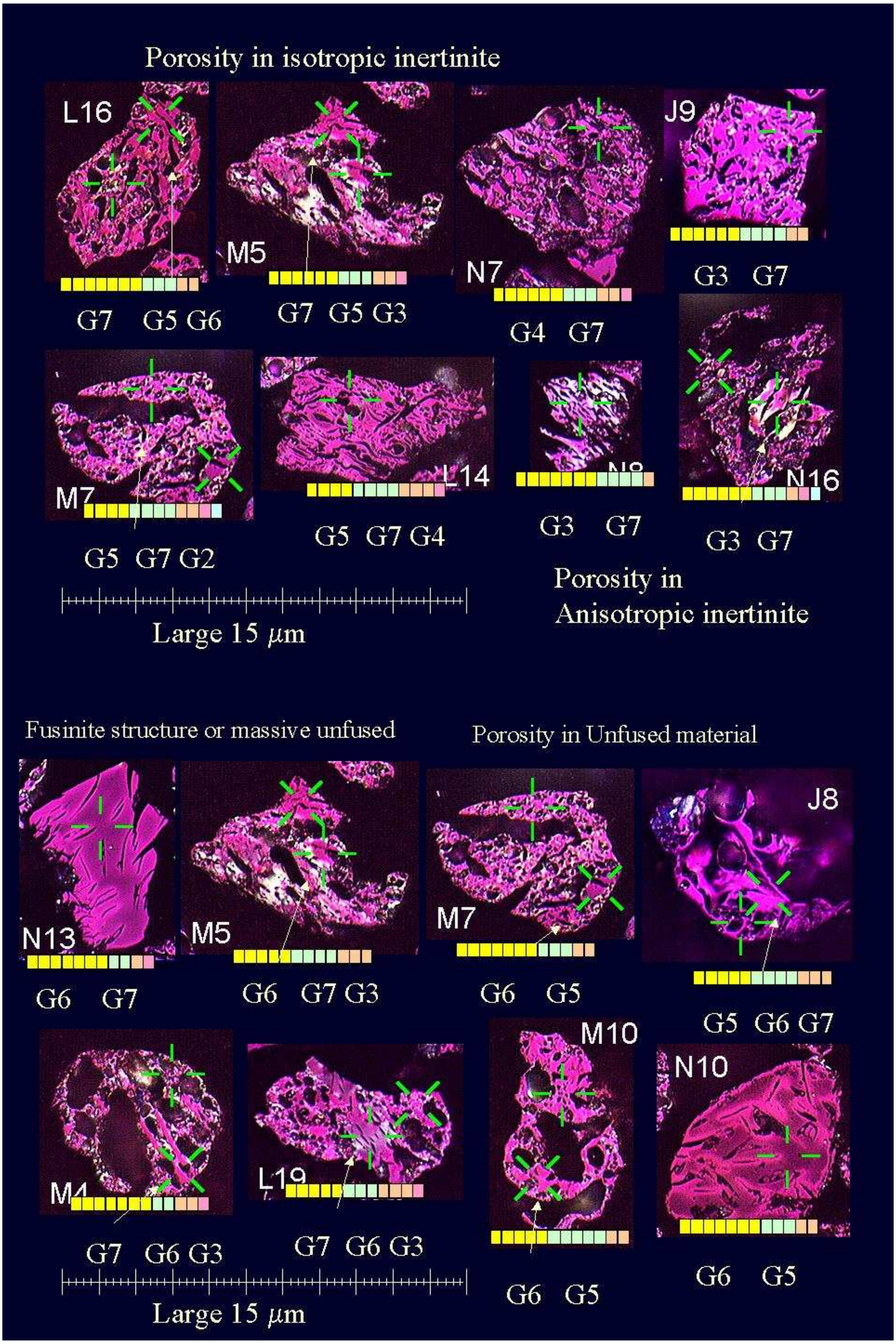

83 % Agreement
K7
K6
N5
M3
G5
G3
J13
L19
K9
J7
J14
G5
J7
Large 15 μm

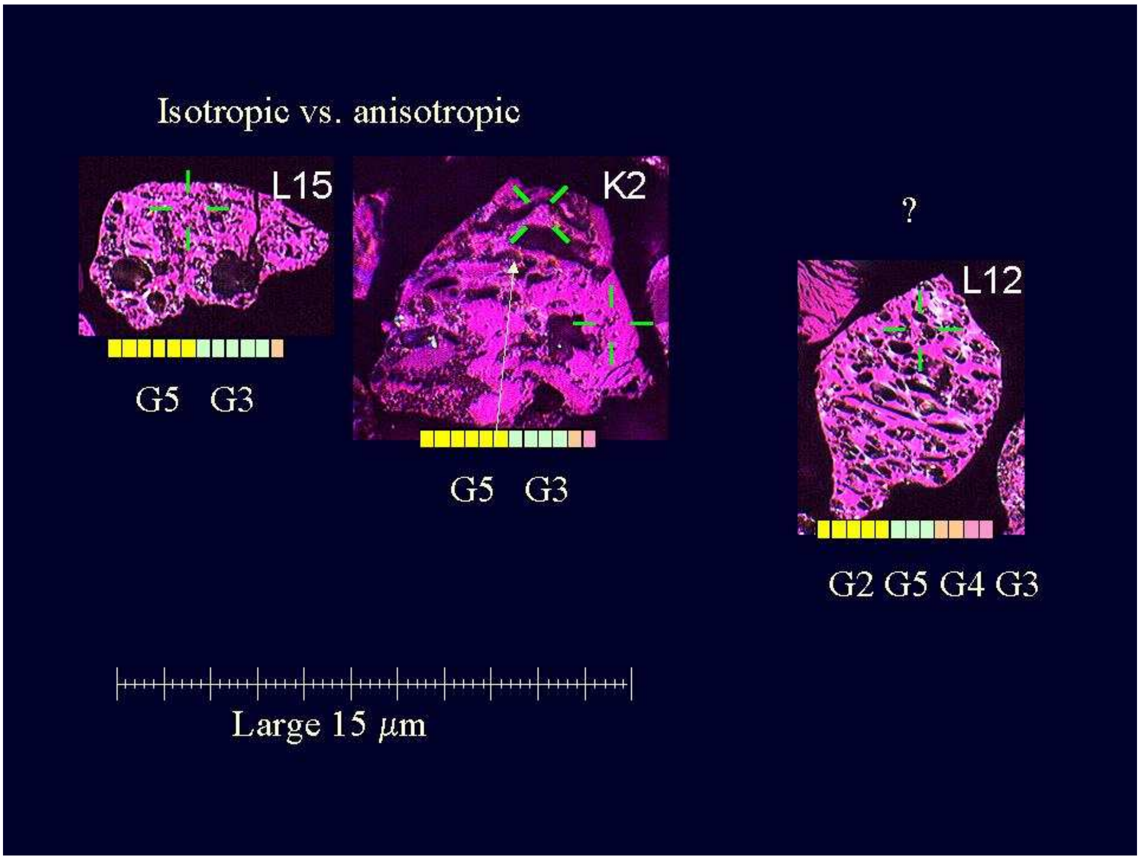

75 % Agreement
N19
G6
G5
J10
L9
G2
N13
K3
G3
G7
N17
G4
J11
Large 15 µm


67 % Agreement
K9
G4 G1
N18
G4 G4 G5
K8
G5
L20
G5 G4
G5 G4
L13
G4 G5
N11
G4 G2
L3
G5 G4
M10
G5 G4
Large 15 µm


67 % Agreement
N15
N2
J5
G3
L4
G2 G3
G2
G2 G3
G3 G2
G3 G7
L16
M6
G7
G2 G1
G7 G6
G2 G3
L11
Large 15 μm


K8
L10
L7
N3
G2 G3
G2 G1
G2 G1
G3 G2
Porosity problems
J4
L10
J19
N3
G5 G4 G7
G5 G4
G5 G4
G4 G5
Large 15 μm


Porosity in isotropic inertinite
L16
G7 G5 G6
M5
G7 G5 G3
N7
G4 G7
J9
G3 G7
M7
G5 G7 G2
L14
G5 G7 G4
G3 G7
N16
G3 G7
Porosity in Anisotropic inertinite
Large 15 µm
Fusinite structure or massive unfused
Porosity in Unfused material
N13
G6 G7
M5
G6 G7 G3
M7
G6 G5
J8
G5 G6 G7
M4
G7 G6 G3
L19
G7 G6 G3
M10
G6 G5
N10
G6 G5
Large 15 µm



The results in Table 2 illustrated by the images above can be summarised as follows:

- Great agreement was found in the identification of vitrinite-derived material, although there were no instructions about how to identify vitrinite-derived char in the classification system and no pellets were distributed. Only information on coal rank was provided.
  - When particles had cenospheric shape more than 92% participants identified them as vitrinite-derived
  - More discrepancies were found when material, likely vitrinite-derived, was included in mixed particles.
- Major differences in the identification of the fields related to:
  - Porosity level. Mainly between G2-G3 and G4-G5.
    - In some cases, the reason for the discrepancy was that there is a gradual change in the porosity, whose limit is difficult to establish. This occurred in few cases and it is difficult to solve by improving the definitions. Anyway it is a fact common to maceral occurrences that usually grade from one maceral to another.
    - In some other cases, the reason was that participants did not follow the rule "consider the material around the mark exhibiting the same optical characteristics". Some of us exclusively took into account the material within the mark that usually was centered in the wall-material, without considering the pores around.
  - Behaviour of the least plastic inertinites. Does the porosity belong to the fusinite structure or was it generated during pyrolysis? (G3-G7, G4-G7, G5-G7 and G6-G7).

  - In many cases, these differences appeared to be due to variable criteria. Some participants considered the porosity in fusinites as cellular structure and some others thought it was generated during the thermal treatment.
- Surprisingly (after last year manual results), few difficulties were found to identify isotropic and anisotropic material, although different level of resolution in the computer screens might change the appearance of the particles (G2-G4, G3-G5). In addition the definition of the image is critical when deciding about anisotropy and the fact of having a `frozen´ image, where the stage cannot be rotated, affects particularly the distinction between isotropic or anisotropic fields. This problem would not occur using a microscope.

Table 3. Summary of the reasons for dubious identifications

Sort of doubts	Amount (%)
Vitrinite-derived material	3
Isotropic/Anisotropic	4
Porosity problems	14
Fusinite structure and behaviour	14

Table 3 indicates that most of the doubts are related to the estimation of porosity and to the assessment of fusinite structure or behaviour.

The relationship between the characteristics of the coal and the level of agreement in the identification of the fields has been also checked. The maceral analysis and vitrinite reflectance of the parent coal from which the chars were obtained are listed in Table 4. In this table, Coal L is Collinsville and coal N is BB2 which are the parent coals of the chars analysed manually (Borrego et al., INCWG99). Table 5 shows the percentage of fields identified with a given level of agreement. The crossed comparison of the data in Tables 4 and 5 indicates that the lowest levels of agreement were found for the chars from coals of medium rank (Rr=1.28% and 1.05%) and for the inertinite-richest coal (N).

Table 4. Petrographic analysis of the coals from which chars were prepared

Coal	J	K	L	M	N
Ro (%)	0.68	0.73	1.28	1.05	0.66
Vitrinite (%)	84.2	27.4	54.2	55.0	1.6
Inertinite (%)	10.4	67.6	45.8	45.0	97.2
Liptinite (%)	5.4	5.0			1.2

Table 5. Percentage of fields identified with various level of agreement for the different coal chars

Coal	J	K	L	M	N
100 % AG	29	24	16	20	12
90 % AG	25	24	16	7	25
80 % AG	21	24	22	20	19
<80% AG	25	28	47	53	44

Summary

In summary, the results of the CD exercise are very promising. It seems that the WG will be able of solve the few problems related to the definition of the textures to be grouped under each classification heading and to the size of the fields to be considered when choosing the various options. Participants are invited to go back to the CD and re-consider the assignments after studying carefully the results and report back to the convener.

Suggestions for 2000 Round Robin exercise:

Considering the results summarised in this report, the suggestions for 2000 year exercise are:

- Improve the definitions of the material to be classified within each group
- Prepare an additional CD containing images taken with and without retarder plate in order to find out about the influence of observation conditions in the identifications.