

## Very low-grade metamorphism of the Seo Formation in the Orri Dome, South-Central Pyrenees

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### Abstract

The metamorphic grade of the Seo Formation, South-Central Pyrenees, has been determined using illite crystallinity measurements. The total range of the illite crystallinity indices of the K<sup>+</sup>-saturated fraction < 2 μm is 0.21–0.31 °2θ and averages 0.26 ± 0.03 °2θ, which indicates metamorphism in the high-grade part of the anchizone. This is in agreement with mineralogical data, such as the absence of expandable clay minerals and of metamorphic minerals such as biotite and epidote. Illite crystallinity indices of the detrital micas suggest that they were derived from igneous rocks or from rocks with at least epizonal grade of metamorphism.

### Introduction

The Seo Formation in the Orri Dome, South-Central Pyrenees (Fig. 1), is assumed to be non-metamorphic to very low-grade metamorphic (Hartvelt 1970; Zwart, 1979; Speksnijder, 1987b). This assumption is based on the relatively poorly developed cleavages, but has not yet been supported by any mineralogical data (Zwart, 1979). The present paper reports the results of a more detailed investigation into the metamorphism of the Seo Formation.

The determination of the metamorphic grade in very low-grade pelitic and quartzitic rocks presents some special problems. In meta-grauwackes and meta-volcanic rocks a metamorphic zonation can be made, based on the occurrence of metamorphic minerals such as zeolites, pumpellyite, prehnite, lawsonite, etc. (e.g. Winkler, 1979; Liou et al.,

1987). However, such minerals rarely occur in pelitic and quartzitic rocks. Therefore the determination of metamorphic grade in pelitic and quartzitic rocks between diagenesis and the middle greenschist facies can be quite difficult and other indicators than mineral assemblage must often be used, such as illite crystallinity or coalification (Frey, 1987).

Various subdivisions of the realm of diagenesis and incipient metamorphism exist (Kisch, 1983, 1987; Frey, 1987). Each subdivision is based on one or more metamorphic indicators, such as metamorphic minerals (Winkler, 1979), porosity and microstructure (Kossovskaya & Shutov, 1970), coalification (Teichmüller & Teichmüller, 1981; Teichmüller, 1987) or illite crystallinity (Kubler, 1964, 1967, 1968). Correlations between the various subdivisions are very difficult (Teichmüller et al., 1979; Kisch, 1980a, b, 1981, 1987). Therefore

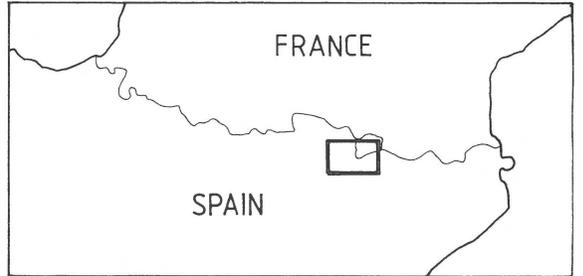
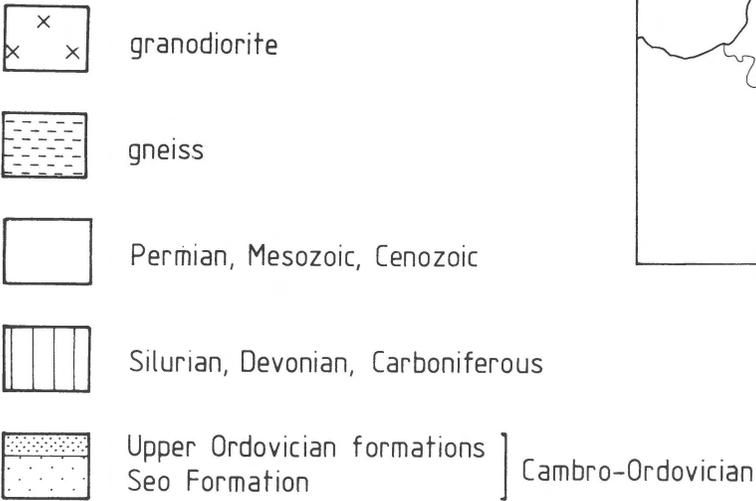
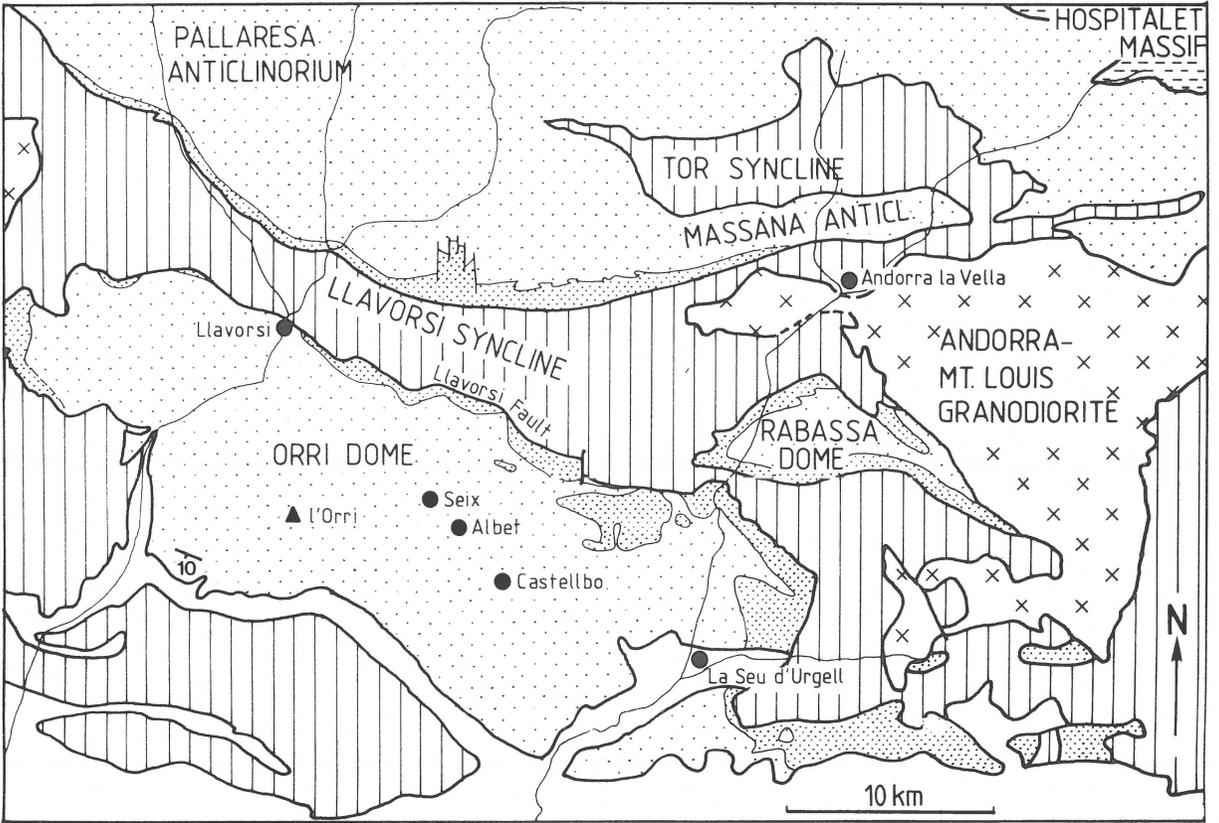


Fig. 1. Geologic map of the study area (After Hartevelt, 1970)

the terminology one uses to describe the metamorphic grade should depend on the indicators used.

In this study illite crystallinity is used as the main indicator of metamorphic grade and therefore the terminology of Kubler (1964, 1967, 1968) will be used. Near the onset of metamorphism three zones are distinguished: diagenesis, anchizone and epizone (see Table 1). In some cases the metamorphic grade was determined using mineral paragenesis. In that case terms like greenschist facies will also be employed.

### Geologic setting

The Orri Dome, named after the Orri Mountain in the centre of the structure (Fig. 1), is one of the large anticlinoria in the Axial Zone of the Pyrenees (Zwart, 1979). It is bounded in the north by the Llavorsi Syncline and the Llavorsi Fault. To the south the boundary is formed by the unconformable contact with Permian sediments (Speksnijder, 1985). An excellent map and a general description of the Orri Dome and surrounding structures have been presented by Hartevelt (1970). The structural geology has been studied in detail by Speksnijder (1986, 1987a, b).

The Orri Dome is formed mainly by Cambro-Ordovician rocks. The lowest rock unit consists of

an unfossiliferous sequence of thin-bedded slates and siltstones of at least 2 km thickness, the Seo Formation. The Seo Formation is probably deposited in a shallow sea (Hartevelt, 1970). The formations overlying the Seo Formation are found along the northern border of the Orri Dome. They show a much more diverse lithology than the Seo Formation, including conglomerates, sandstones and slates, ranging in age from Upper Ordovician to Carboniferous (see Hartevelt, (1970).

Several generations of deformation structures have been described in the Orri Dome (Hartevelt, 1970; Zwart, 1979; Speksnijder, 1987a, b). The most evident structures are south-facing folds, developed on various scales from centimetres to kilometres, associated with a north-dipping axial plane cleavage. These structures have been termed main-phase folds and -cleavage (Speksnijder, 1987a, b; Bons, 1988). Two generations of very open pre-mainphase folds have been described (Speksnijder, 1987a), as well as several post-mainphase deformations structures (Hartevelt, 1970; Zwart, 1979; Speksnijder, 1987b).

### Mineralogical indicators of metamorphic grade

The main minerals of the rocks of the Orri Dome are muscovite, chlorite, quartz and albite. This mineral assemblage is stable from the diagenetic

Table 1. Metamorphic zonation in non-metamorphic to low-grade metamorphic pelitic rocks based on illite crystallinity, and correlation with mineral facies (after Kisch, 1987)

Zonation based on illite crystallinity (Kubler, 1968)	Boundary values used in the present study (Kisch, 1986, pers. comm.)	Mineral facies (Winkler, 1979)
diagenesis		zeolite facies
-----	0.0370 °θ	----- prehnite- pumpellyite facies
anchizone		pumpellyite- actinolite facies
-----	0.205 °θ	----- greenschist facies
epizone		

field up to the middle greenschist facies (Winkler, 1979; Frey, 1987). Careful analysis of the samples, using optical microscopy, X-ray diffraction, electron microscopy and energy-dispersive X-ray microanalysis has revealed no typical low-grade metamorphic minerals such as pumpellyite or epidote.

Detrital minerals such as biotite, K-feldspar and Ca-plagioclase are entirely absent. These minerals are unstable during diagenesis and disappear completely near the anchizone (Kisch, 1983). X-ray

diffraction analyses have shown that muscovite and chlorite are the only phyllosilicates present, with minor quantities of kaolinite. The absence of clay minerals with expandable layers, such as smectite, confirms that the high-grade boundary of the diagenetic field has been reached (Kisch, 1983).

Along the southern border of the Pallaresa Anticlinorium and in the Massana Anticline (see Fig. 1) the slates of the Seo Formation show the mineral assemblage muscovite + chlorite + quartz + al-

Table 2. Major element oxide data (wt%) from pelitic rocks from the Seo Formation, Massana Anticline and Orri Dome

A. Massana Anticline

Sample	SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	FeO	MgO	CaO	Na <sub>2</sub> O	K <sub>2</sub> O
P-HH1	53.4	19.5	4.5	0.9	6.5	0.9	0.5
P-HH2	57.7	21.2	8.1	2.2	0.2	1.2	3.9
P-HH3	63.5	18.6	7.2	2.0	0.6	1.5	3.2
P-HH4	61.4	20.0	6.9	1.9	0.2	1.9	3.4
P-HH5	58.5	16.0	6.0	1.4	5.4	0.4	3.4
P-HH6	74.4	10.2	3.2	0.8	1.8	0.8	2.0
P-HH7	54.2	21.1	8.6	2.5	1.0	1.5	3.4
P-HH10	53.6	21.6	8.4	2.3	0.2	1.6	3.6
average	59.6	18.5	6.6	1.8	2.0	1.2	2.9

B. Orri Dome

Sample	SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	FeO	MgO	CaO	Na <sub>2</sub> O	K <sub>2</sub> O
P-HH11	59.9	20.2	8.0	2.2	0.1	1.3	3.5
P-HH12	59.3	19.4	7.8	1.9	0.0	0.7	3.7
P-HH13	60.0	22.4	8.6	2.7	2.8	0.9	4.4
P-HH14	62.5	17.9	7.3	2.3	0.1	1.3	3.1
P-HH15	51.7	25.1	5.2	2.1	0.0	0.8	5.0
P-HH16	47.4	27.7	8.9	2.2	0.2	0.7	5.7
P-HH17	58.2	21.2	7.0	2.2	0.0	1.1	3.8
P-HH19	60.4	19.2	8.7	0.6	0.4	0.5	5.2
P-HH20	65.0	14.9	5.7	1.3	0.2	1.9	2.8
P-HH21	58.4	17.6	4.5	1.5	0.4	0.7	3.5
P-HH22	58.2	20.8	7.1	1.7	0.8	0.5	4.6
P-HH23	59.4	20.1	7.8	2.2	0.2	1.1	4.0
P-HH24	60.2	21.2	8.0	2.0	0.2	0.7	4.2
P-HH25	59.6	21.3	12.7	2.2	2.5	1.2	3.6
P-HH26	68.0	15.8	6.0	2.1	0.2	1.6	2.6
P-HH27	64.7	17.4	6.8	2.2	0.3	1.2	3.2
P-HH28	61.4	19.7	7.2	2.3	0.2	1.3	3.4
P-HH29	61.2	18.4	6.7	1.9	0.3	1.2	3.1
average	59.8	20.0	7.4	2.0	0.5	1.0	3.8

bite + epidote. Metamorphic chloritoid has been found in the Llavorsi Syncline and along the southern border of the Massana Anticline (Zwart, 1959). The presence of epidote and chloritoid in these rocks indicates greenschist facies metamorphism. The bulk chemistry of the Orri Dome slates does not differ significantly from the Cambro-Ordovician slates of the Pallaresa and Massana Anticlines, which contain epidote even in the samples low in Ca (see Table 2). Therefore it seems likely that epidote could also have been formed in the slates of the Orri Dome if the degree of metamorphism had reached the greenschist facies zone. Thus, the absence of epidote in the Seo Slates of the Orri Dome can be used as an indication that the greenschist facies zone has not been reached in the Orri Dome.

One striking feature of the slates of the Seo Formation is the occurrence of monazite porphyroblasts up to 400  $\mu\text{m}$  in size (Bons, 1988). Because metamorphic monazite has not yet been described in (very) low-grade rocks, it can not be used as an indicator of metamorphic grade (Overstreet, 1967; Mohr, 1984). The occurrence of monazite porphyroblasts will be described in detail in a separate paper.

### Illite crystallinity

Another method to determine the metamorphic grade in (very) low-grade pelitic rocks is the measurement of illite crystallinity (Weaver, 1960; Kubler, 1964; Weber, 1972a, b).

With increasing metamorphic grade the crystallinity of illite improves due to increasing size of the crystallites and increasing ordering of the crystal layers; the high-grade end member is muscovite. The increase of illite crystallinity is reflected in the sharpness of the X-ray diffraction peaks of illite/muscovite. Thus the sharpness of the illite 10 Å-peak can be used as a measure of metamorphic grade; the first to use this parameter was Weaver (1960). The peak sharpness can be measured as the width of the peak at half height; this quantity is called the illite crystallinity index (Kubler, 1964), henceforward abbreviated to 'IC index'. The IC

index can be expressed in mm on the paper chart of the X-ray diffractometer (Kubler, 1964, 1967, 1968), or in degrees of the angular separation ( $^{\circ}2\theta$ ) (Kisch, 1980a, b, 1981, 1983). A small value of the IC index indicates a narrow peak and thus a high degree of diagenesis or metamorphism.

The samples for the illite crystallinity measurements were collected from the pelitic layers in fresh roadside outcrops. One to three samples per outcrop were measured. Four samples from the Rabassa Dome, an outcrop of the Seo Formation north of the Orri Dome, have also been measured. The IC-index is strongly influenced by sample preparation and measurement techniques (Kisch & Frey, 1987). In this study the preparation techniques and X-ray diffractometer settings of Kisch (1980a, 1981, 1983) are used (see Appendix).

The crystallinity of detrital micas is usually better than that of the authigenic micas. The mean IC index of samples consisting of a mixture of detrital and authigenic micas will therefore be smaller than that of the authigenic micas that indicate the true degree of diagenesis or metamorphism. As the clay-size fraction ( $< 2 \mu\text{m}$ ) is generally free of detrital micas, this fraction is usually used for determination of the crystallinity index. Following Kisch (1980a, b, 1981, 1983), the fraction  $< 2 \mu\text{m}$  is used in this study, while the fractions 2–6 and 6–50  $\mu\text{m}$  were also measured, in order to monitor the influence of the detrital micas.

To check for the presence of clay minerals like smectite and vermiculite, and to compensate for loss of  $\text{K}^+$ -ions during weathering and sample preparation, the fraction  $< 2 \mu\text{m}$  was saturated with  $\text{K}^+$ -ions. A parallel set of samples was saturated with  $\text{Mg}^{2+}$ -ions. If smectite or vermiculite is present, the width of the 10 Å-peak will decrease after  $\text{K}^+$ -saturation, while  $\text{Mg}^{2+}$ -saturation does not affect the 10 Å-peak.

In order to interpret the IC indices measured in Utrecht in terms of metamorphic zonation, using the boundary values of Kisch (see Table 1), the X-ray diffractometer in Utrecht was calibrated against the diffractometer at the laboratory of Kisch (Ben Gurion University of the Negev, Beer-Sheva, Israel). This was done using polished rock slabs with varying illite crystallinity. As the IC-

Table 3. Internal illite crystallinity standards

Zone	Sample no.	<2 $\mu\text{m}$ K	<2 $\mu\text{m}$ Mg	2–6 $\mu\text{m}$ [ $^{\circ}2\theta$ ]
1. diagenesis	Mo2	0.530	0.530	0.580
1. greenschist facies (epizone)	P251	0.190	0.190	0.190
3. muscovite single crystal M1 (1 cm <sup>2</sup> ): 0.105 $^{\circ}2\theta$				

1. Shale from the North Sea, maximum palaeotemperature ca. 130 $^{\circ}$  C.

2. Phyllite from the Massana Anticline: epidote + allanite porphyroblasts

3. Flake from a 10 cm large crystal from a pegmatite (value quoted by Kisch, 1983 p. 348–350, for 'maximum' illite crystallinity: 0.11  $^{\circ}2\theta$ ).

indices measured in Utrecht and in Beer-Sheva were exactly the same, the boundary values of the anchizone cited by Kisch can also be used in this study.

A set of house standards with known diagenetic or metamorphic grade was used to check the validity of the illite crystallinity results. These standards include a diagenetic shale (Mo2) and a greenschist facies phyllite (P251) (see Table 3). The IC-index of a large single crystal of muscovite from a pegmatite (M1) has also been measured, in order to measure the maximum crystallinity (narrowest peak) of muscovite. The IC-indices of the standards are in good agreement with their degree of diagenesis or metamorphism, while the crystallinity of the muscovite crystal is the same as the value quoted by Kisch (1983, p. 348–350).

The measured IC indices are given in Table 4 and Fig. 2. There is no significant difference between the K<sup>+</sup> and Mg<sup>2+</sup> saturated samples. This means that clay minerals like smectite and vermiculite are absent, and that there was no significant loss of K<sup>+</sup>-ions during weathering and sample preparation.

Weathering can reduce the crystallinity of illite, although the effect of weathering is usually within the standard deviation of the IC measurements (Schramm, 1981). The IC indices of the samples from the Pallaresa Anticlinorium and the Massana Anticline fall within the epizone field; this is in

good agreement with a greenschist facies metamorphic grade, which has been determined using mineral assemblages. Apparently the IC indices of these rocks have not been affected by weathering. As the samples from the Orri Dome were collected in outcrops of similar quality, it can be assumed that weathering did not significantly affect their IC index either.

The grainsize of the detrital micas in the slates is in the range 10–100  $\mu\text{m}$ . In the fractions 2–6  $\mu\text{m}$  and 6–50  $\mu\text{m}$  there will be an increased contribution of the detrital micas. The IC index decreases with increasing grainsize. The fraction 6–50  $\mu\text{m}$ , which consists almost entirely of detrital micas, shows IC indices of 0.15–0.22  $^{\circ}2\theta$ , largely within the epizone field. This suggests that the detrital micas were derived from igneous rocks or from rocks with at least an epizonal grade of metamorphism.

The total range of IC indices of the K<sup>+</sup>-saturated grainsize fraction <2  $\mu\text{m}$  of the slate samples of the Orri Dome is 0.21–0.31  $^{\circ}2\theta$ ; the average is 0.26  $\pm$  0.03  $^{\circ}2\theta$ . The low-grade boundary of the anchizone lies at 0.37  $^{\circ}2\theta$ , while the anchizone-epizone boundary lies at 0.205  $^{\circ}2\theta$  (Kisch, 1986, pers. comm; Table 1). Therefore it can be concluded that the Seo Formation of the Orri Dome has been metamorphosed in the high-grade part of the anchizone. Within the Orri Dome, there is no significant trend in the IC indices with geographical position.

It is very difficult to connect illite crystallinity data to a temperature scale. Based on correlations between illite crystallinity, oxygen-isotope data, fluid inclusions and mineralogical zonations Kisch (1987, p. 291–293, 296) suggests that the anchizone represents a temperature range from ca. 200–250 $^{\circ}$  C to ca. 300 $^{\circ}$  C (Table 1). This would suggest paleotemperatures between 250 $^{\circ}$  C and 300 $^{\circ}$  C for the rocks of the Orri Dome. For a geothermal gradient of 30 $^{\circ}$  C/km this would imply pressures on the order of 200–300 MPa. However, this is a maximum estimate, as the geothermal gradient during Variscan metamorphism has probably been much higher (Zwart, 1962, 1979).

Table 4. Illite crystallinity indices measured in the Seo Formation, Orri Dome (all values in  $^{\circ}2\theta$ . See Fig. 2 for the location of the sections

	km along section	sample no.	$<2\ \mu\text{m K}$	$<2\ \mu\text{m Mg}$	$2\text{--}6\ \mu\text{m}$	$6\text{--}50\ \mu\text{m}$
Section A–A'	1.100	P113	0.213	0.218	0.200	0.170
	3.450	P106	0.218	0.195	0.200	0.218
	5.550	P99	0.243	0.275	0.213	0.158
	7.950	P98	0.230	0.225	0.200	0.155
	9.500	P89	0.205	0.213	0.188	0.155
	10.550	P78	0.300	0.325	0.213	0.195
	11.650	P77	0.230	0.230	0.213	0.163
	12.500	P74	0.225	0.213	0.213	0.170
	23.000	P189	0.213	0.213	0.193	0.138
23.750	P185	0.205	0.213	0.200	0.150	
Section B–B'	3.650	P18	0.210	0.260	0.180	–
	6.950	P31	0.250	0.263	0.200	0.163
	7.900	P33	0.243	0.225	0.195	0.150
	8.400	P34	0.250	0.255	0.200	0.175
	8.900	P35	0.243	0.250	0.200	0.150
	9.550	P37	0.238	0.238	0.205	0.188
	9.650	P48	0.238	0.263	–	0.193
	10.600	P50	0.305	0.300	0.225	0.180
	10.650	P59	0.268	0.275	0.238	0.188
	10.750	P60	0.218	0.205	0.200	0.168
	11.050	P51	0.288	0.250	0.205	0.188
	11.200	P52	0.263	0.313	0.218	0.200
	11.750	P55	0.225	0.250	0.213	0.175
	11.750	P57	0.225	0.283	0.225	0.168
	11.850	P54	0.225	0.250	0.213	0.175
	11.850	P61	0.243	0.263	0.213	0.163
23.000	P192	0.230	0.230	0.205	0.155	
Section C–C'	2.500	P150	0.243	0.263	0.225	0.205
	2.500	P151	0.275	0.263	0.213	0.168
	2.500	P152	0.263	0.258	0.218	0.163
	3.050	P153	0.275	0.293	0.238	0.188
	3.050	P154	0.300	–	0.238	0.175
	3.050	P155	0.300	0.305	0.238	0.188
	5.100	P217	0.268	0.275	0.225	0.183
	5.100	P218	0.288	0.300	0.225	0.205
	6.450	P215	0.313	0.275	0.193	0.155
	6.750	P220	0.305	0.268	0.225	0.195
	6.750	P221	0.313	0.293	0.250	0.205
	6.750	P222	0.300	0.288	0.213	0.200
	7.300	P213	0.263	0.288	0.218	0.195
	7.300	P214	0.275	0.275	0.200	0.180
	7.800	P208	0.318	0.318	0.250	0.195
	8.350	P211	0.288	0.263	0.238	0.200
	11.300	P226*	0.238	0.305	0.230	0.188
	11.550	P236*	0.263	0.280	0.250	0.193
	12.150	P230*	0.208	0.230	0.193	0.155
12.300	P231*	0.225	0.230	0.200	0.145	

\* Seo Formation, Rabassa Dome, Andorra.

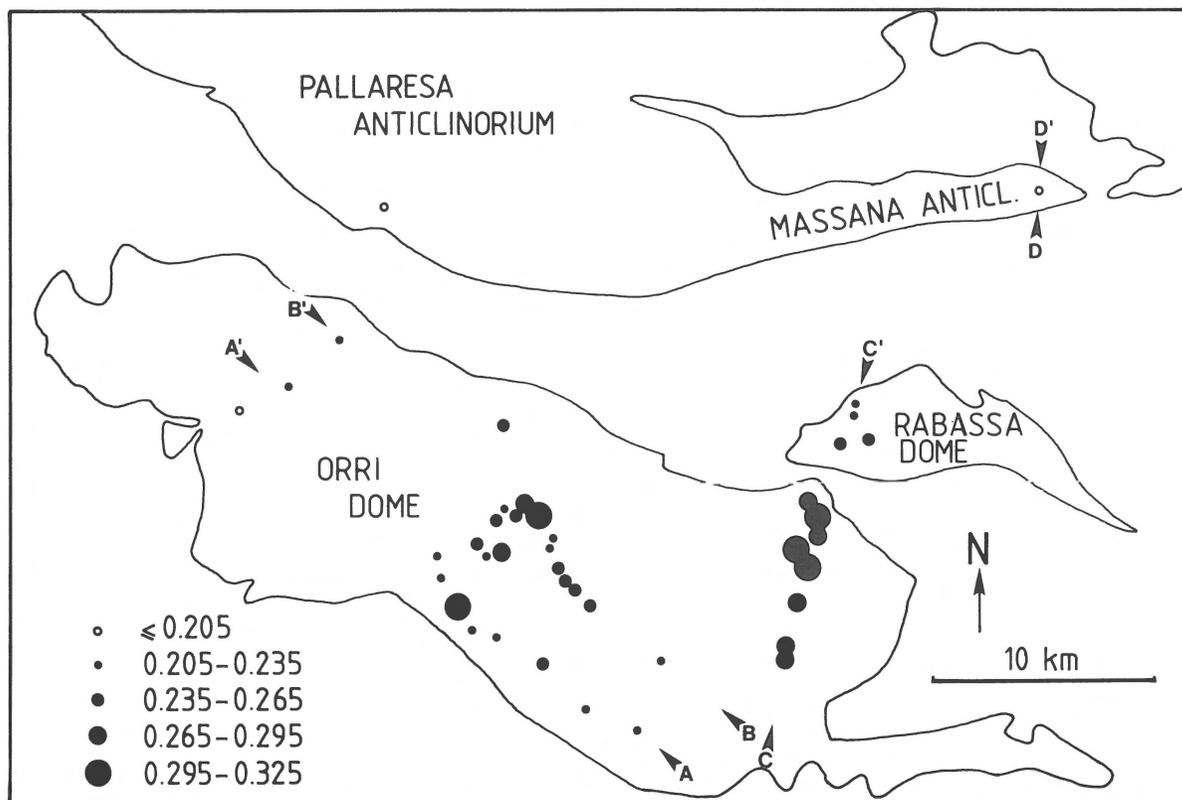


Fig. 2. Schematic map of the Cambro-Ordovician outcrops in the study area, with the illite crystallinity indices measured on the  $K^+$ -saturated fraction  $< 2\mu m$ . More data on the samples taken along the sections A-A', B-B' and C-C' are given in Table 4.

## Conclusions

Illite crystallinity measurements on slates of the Seo Formation in the Orri Dome indicate metamorphism in the high-grade part of the anchizone. This is confirmed by the absence of detrital minerals such as K-feldspar and Ca-plagioclase and of clay minerals such as smectite, and by the absence of epidote. An estimate for the palaeotemperature is 250–300°C. The palaeopressure probably did not exceed 300 MPa. The high degree of crystallinity of the detrital micas indicates that they originate from igneous or metamorphic rocks with at least epizonal grade of metamorphism.

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## Appendix

Illite crystallinity measurement: sample preparation and instrument settings (after Kisch, 1980a, b, 1983, pers. comm. 1986)\*

### A. Sample preparation

#### *Desintegration, removal of organic matter and calcium carbonate*

1. crush sample in a mortar to pass a 0.2 mm sieve
2. transfer 5 g to a 250 ml Erlenmeyer flask containing 50 ml 6% H<sub>2</sub>O<sub>2</sub> and put flask in an ultrasonic bath for 30 minutes
3. add 5 ml 1N HCl and put flask in a 70°C water bath for 30 minutes
4. wash the suspension with distilled water until the pH of the water is neutral

#### *Separation of size fractions*

5. use a 50 µm sieve to remove the size fraction > 50 µm
6. transfer the suspension to a 20 cm high sedimentation cylinder, stir well; after 70 minutes pipette out the upper 15 cm; this contains the fraction < 6 µm. Repeat once, most of the fraction < 6 µm is thus removed and the remaining suspension contains the fraction 6–50 µm.
7. transfer the fraction < 6 µm to a 30 cm high sedimentation cylinder, stir, and let settle for 17 hours 10 minutes; pipette out the upper 25 cm of liquid, this contains the fraction < 2 µm. Repeat this step once; most of the fraction < 2 µm is now removed and the remaining suspension contains the fraction 2–6 µm.
8. concentrate all size fractions using a centrifuge

#### *Saturation with K and Mg*

9. transfer 10 ml of the suspension < 2 µm to a 50 ml tube and add 10 ml 0.5N KCl; transfer another 10 ml to a second tube and add 10 ml 1N MgCl<sub>2</sub>
10. stir for 20 minutes, concentrate the suspension with the centrifuge; repeat step 9 and 10 twice
11. wash once with water and at least three times with alcohol (check with AgNO<sub>3</sub>)
12. concentrate the suspensions with the centrifuge, pipette out the alcohol and add a few ml water

#### *Preparation of slides*

13. freeze-dry the four suspensions < 2 µm K<sup>+</sup>, < 2 µm Mg<sup>2+</sup>, 2–6 µ and 6–50 µm
14. dilute 0.020 g of each dry fraction in 0.500 ml water and put the suspension on a glass slide
15. let the sample settle and the water evaporate at room temperature

### B. X-ray diffractometer settings

1. Philips X-ray diffractometer, CuKα radiation, 40 kV/30 mA
2. monochromator, discriminator

3. sample spinner on
4. slits: 1° – 0.2 mm – 1°
5. time constants: TC = 2 at range > 1 · 10<sup>3</sup>; TC = 4 at range 4 · 10<sup>2</sup>
6. goniometer speed 0.5 °2θ/min
7. paper speed 20 mm/min
8. usually only the range 7–11 °2θ is measured

\* Recently Kisch has modified his procedure (Kisch & Frey, 1987).

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