

POSSIBILITY OF ANALYZING CALCIUM QUALITATIVE  
LY IN THIN SOILS SECTIONS BY CONTACT  
MICRORADIOGRAPHY

by

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Contact microradiography is a technique that provides interesting information for the micromorphological study on soils, as we have pointed out in various studies published on the subject (G. Vicente- 1.967, H. Carbajal 1.967.....).

In this study an attempt is made to broaden its analytical power to the locating of calcium in soils samples prepared by the thin section technique. The interpretation of the microradiographs obtained leads us to the conclusion that in the samples analyzed, the calcium is almost exclusively located in the mineral grains of the sample, and that the gradient of its concentration is decreasing towards the periphery of the grain. It is significant to observe that mineral grains, where the presence of calcium is quite evident, examined with the petrographic microscope, have the appearance of quartz fragments.

MATERIAL AND METHODS

Preparations of two classes of soils were analyzed: concretions rich in iron proceeding from Guadalajara (Spain) and a coarse moder formation from Huesca (Spain). In accordance with the usual methods, the samples were included in plastic "Chronolita" material and then, thin sections of a 40 $\mu$  thickness were obtained from the block.

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The bases of the microradiographic technique have been developed in other publications (H. Carbajal - 1.969 Müller, K. - 1.965).

Continuous filtered X-ray spectra have been used for obtaining the microradiographs. In order to obtain the band width spectra appropriate, we have based ourselves on the paper of H. Carbajal - 1.968.

The operating conditions were:

5kv. for the generator tube voltage,  $50\mu$  of Be +  $45\mu$  of Al of filter thickness, resulting band width of the continuous spectrum,  $0.5\text{\AA}$ . In order to obtain the microradiographs before the Ca absorption coefficient edge,

4kv. for the generator tube voltage,  $50\mu$  of Be of filter thickness, band width,  $1.5\text{\AA}$ . In order to obtain the microradiographs after the Ca absorption coefficient edge.

The thin sections were situated directly on the gelatine of the photographic plates (23D50 Gevaert Scientia) and in close contact by means of a highly tightened mylar sheet.

### DISCUSSION OF THE RESULTS

Figures 1 and 7 represent X-ray positives, i.e. lighter sites mean a greater X-ray transparency. The figures of the microphotographies 2-6 and 8, on the contrary are X-ray negatives on which a greater X-ray permeability results in a more intensive blackening.

Figure 1 shows the radiograms of a loose humus soil with great quantities of mineral fragments of different size. The direct microscopic analysis of the thin sections shows an incomplete decomposition of the plant rest. This material is generally intermixed with loose detached mineral particles. On the other side, these mineral grains are included in a well humified basal mass, rich in organic particles. In this survey microradiogra

phies the distribution of Calcium in the different mineral particles can already be well recognized. The microphotographies 2 - 6 show some specially marked examples on the sample with a 16 fold microscopic magnification of the microradiogramme. On figures 2 and 3 we see mineral grains with an inhomogeneous calcium distribution. Here, this element concentrates in different places in well defined regions. The distinct concentration in the different regions show up by different blackenings. Figures 4, 5 and 6 are examples for an homogeneous Ca distribution. Here is striking that calcium is to be found substantially only in the interior of the particles, while it lacks more or less in the borders. This marginal zone, free of Ca is specially wide in the grains that are stronger decomposed (fig. 5 and 6). The problem with regard to the relations between decomposition and calcium distribution can not yet be answered. We shall try to elucidate it in further investigations with this method.

Figure 7 shows a concretion rich in iron. It contains abundant isolated mineral grains, which show a sharp demarcation and others which are firmly included in the basal mass. Most of the larger mineral particles contain calcium in different concentrations. We recognize, here too, at a closer examination homogeneous distributions of this element, however always in such a way, that a determined external region remains free of them. An example for an inhomogeneous Ca distribution in this soil sample is given in figure 8. Here Ca is accumulated in varying quantities in some regions, but mainly in the centre of the mineral grain.

### CONCLUSIONS

The results obtained show that contact microradiography is a valuable technique for the micromorphological analysis of soils. The qualitative determination of ele -

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ments with a low atomic number is possible, as well as the possibility of simultaneously establishing in which microscopic structures they are located.

In this study, it is shown that mineral fragments, whose identification under the petrographic microscope would correspond to quartz grains, are impregnated with calcium. This fact has been observed on other occasion by us (unpublished studies), which has led us to carry out localized diffraction in such grains, thus obtaining diffraction effects corresponding to calcium silicate. In this case we point out that it will be a solid solution of a compound of calcium in quartz or the calcium is found as an impurity in the  $\text{SiO}_2$  structure.

On examination of the microradiographs obtained, the location of the calcium is found in mineral grains; the remainder of the structures present in the preparations do not seem to contain such element.

Acknowledgments.

We are indebted to Dr. J. Garcia-Vicente for his assistance in the accomplishment of this research work.

### SUMMARY

In this study is shown the possibility of locating calcium in thin section soils preparations by the contact microradiography technique by using continuous filtered X-ray spectra. The filters used are specified in thickness in order to obtain a band width of the spectrum appropriate for the study.

The calcium is found preferably distributed in the mineral grains of the preparation with a gradient decreasing towards the periphery of the grain as well as towards the regions of maximum alteration.

From the results obtained, it is concluded that contact microradiography is a useful method for the morphological study of soils.

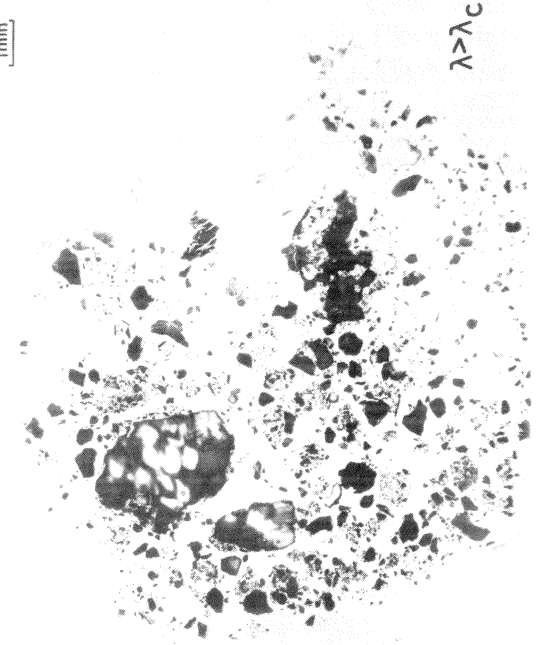


Figure 1

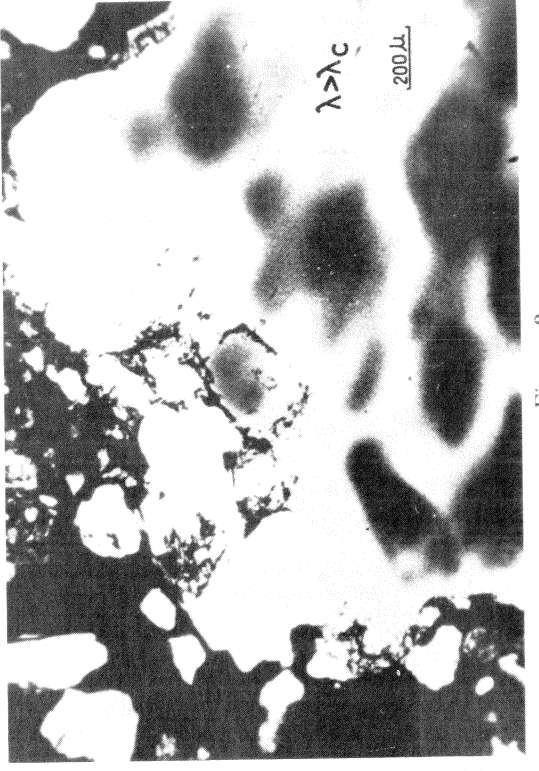
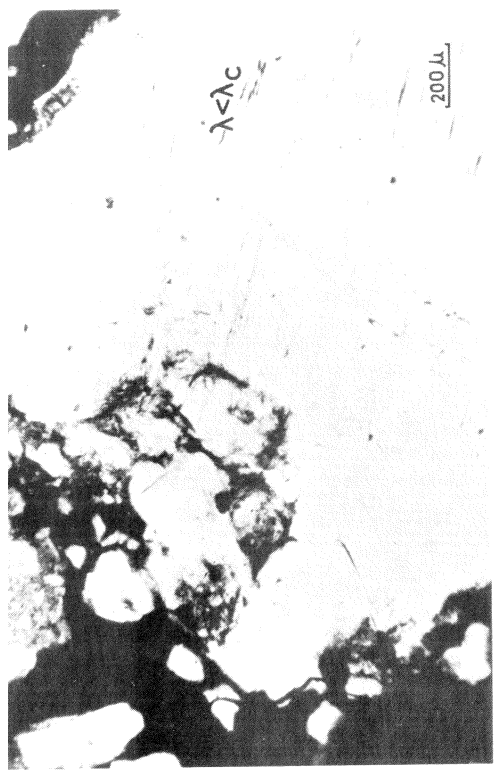


Figure 2



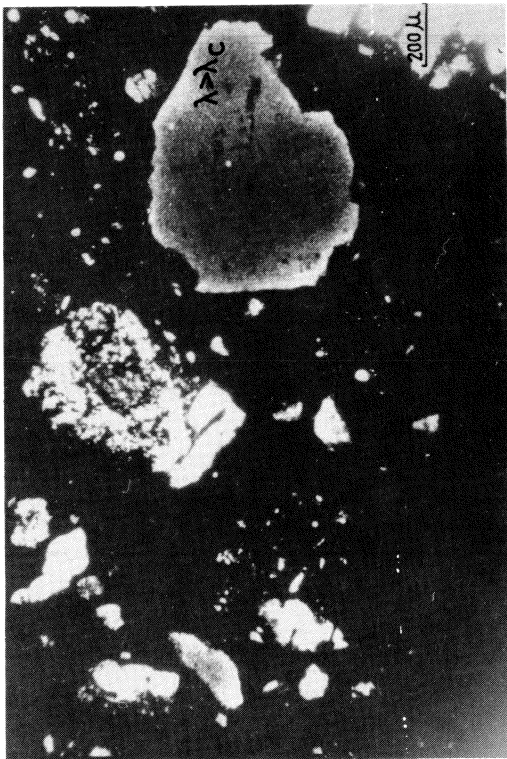
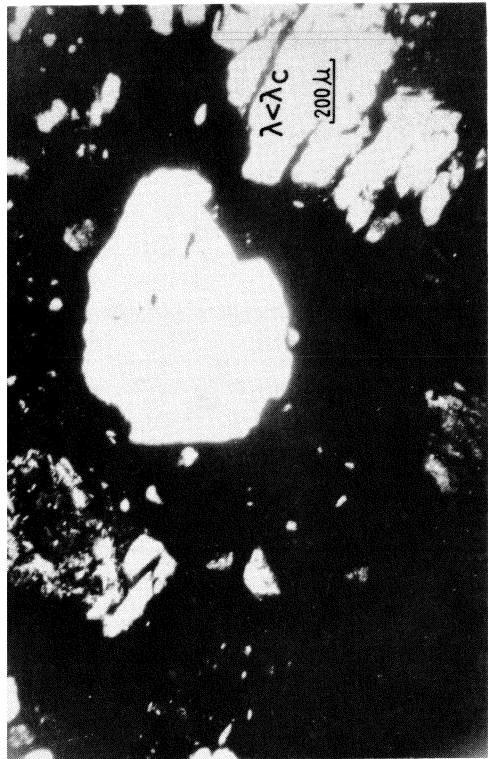


Figure 4

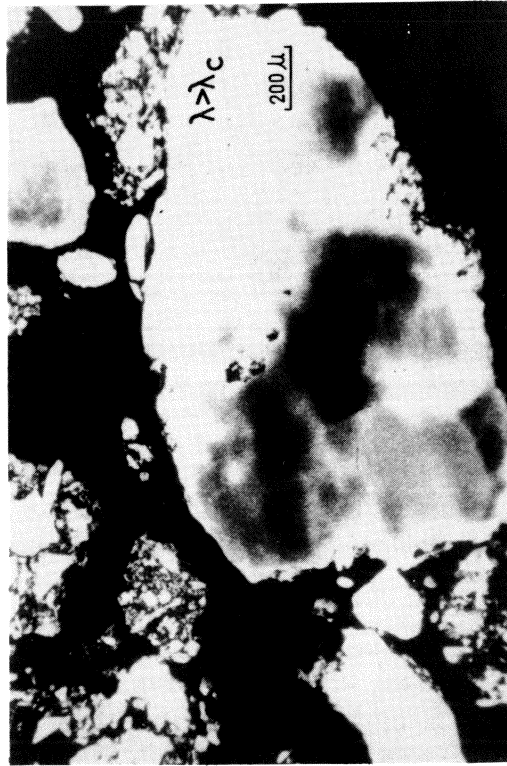


Figure 3





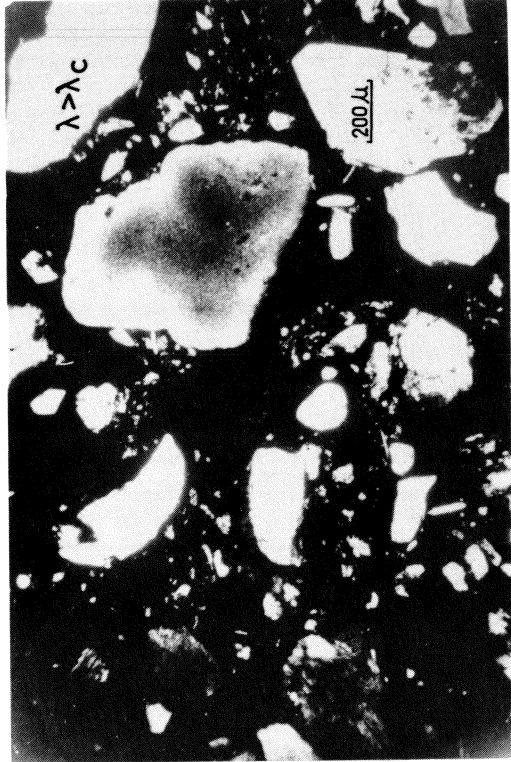


Figure 6

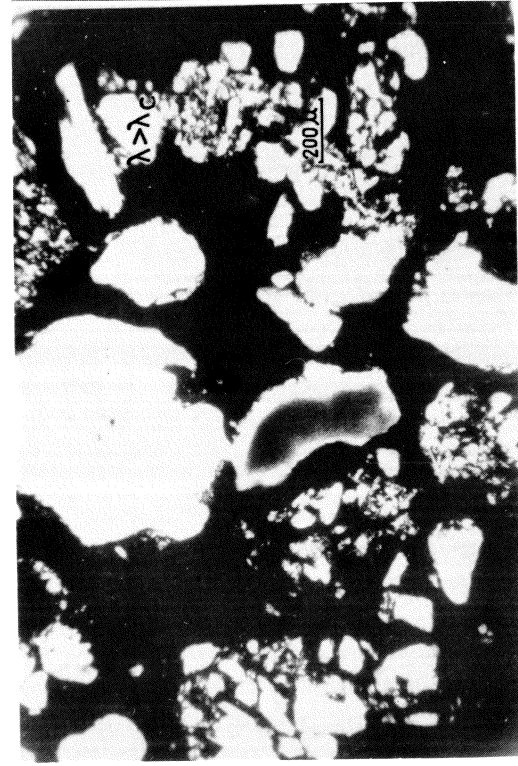


Figure 5



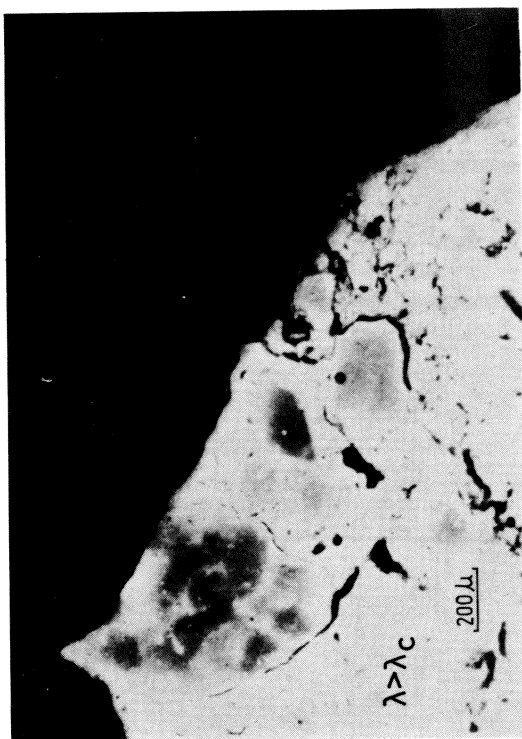
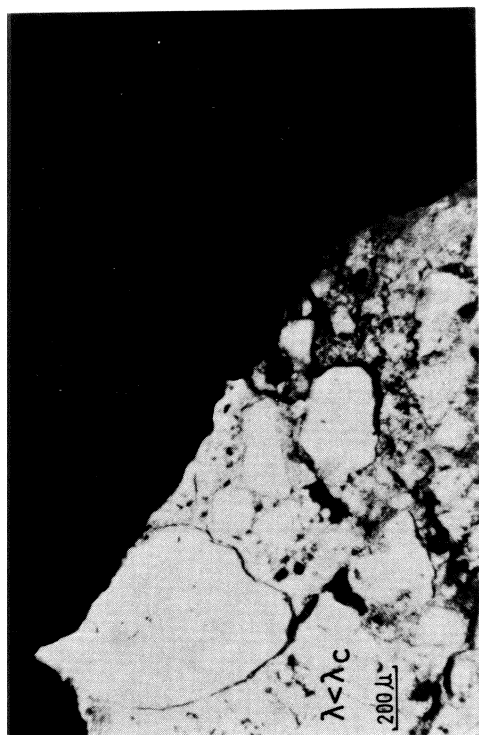


Figure 8

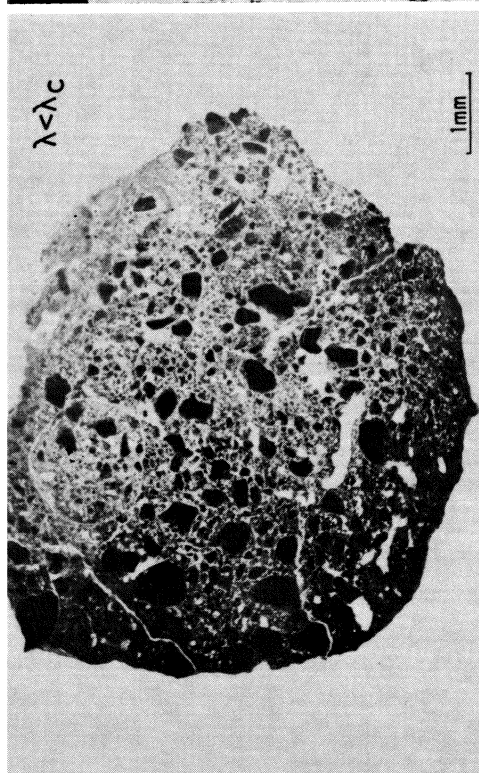


Figure 7



FIGURES .-

- 1 .- X- ray positives of a loose humus soil from Huesca/Pyrenees (total preparation). The light zones within the grains are those with a high level in calcium, (lower picture).
- 2 .- Large mineral fragment of a coarse modern formation. The trituration of the mineral grains is to be attributed, above all, to the decomposing effect of the humus substances. The rest of the grains so trituated remain involved in the humus substances. The dark zones within the grains are those with a high level in calcium, (lower picture).
- 3 .- Decomposition of a mineral grain, especially due to the influence of organic substances; besides there are humus products with trituated mineral fragments. The dark zones within the grains are those with a high level in calcium, (lower picture).
- 4 .- Humus formation, little advanced, (coarse modern) with loose edged mineral grains. The dark zones within the grains are those with a high level in calcium, (lower picture).
- 5 .- Mineral grains of different size included in organic substances, fairly well humified. There are also decomposed grains, unfixed among the humified particles. The dark zones within the grains are those with a high level in calcium, (lower picture).
- 6 .- Coarse modern formation with loose, edged mineral grains. The dark zones within the grains are those with a high level in calcium, (lower picture).
- 7 .- X- ray positive of a concretion rich in iron from Guadalajara/Spain (total preparation). The light zones within the grains are those with a high level in calcium (lower picture).

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- 8 .- Mineral grains firmly included in the basal mass with a sharp boundary. The dark zones within the grains are those with a high level in calcium (lower picture).

### BIBLIOGRAPHY

García Vicente, J. and Carbajal, H. La microradiografía de contacto en el estudio micromorfológico del suelo. Rev. Ciencia Apl. 114, 1967.

Carbajal, H., Kress, M., García Vicente, J. Localización topográfica del hierro en preparaciones en lámina delgada de suelos por la técnica de la microradiografía de contacto. An. Edaf. y Agro. XXVI, 115, 1967.

Carbajal, H. Microradiographie de contact avec spectre de rayon X filtrés. Verr. et Refr. 22, 272, 1968.

Müller, K. Mikroradiographie—ein Untersuchungsverfahren in der biophysikalischen Forschung. "25 Jahre Max Planck Institut für Biophysik". Georg Thieme Verlag, Stuttgart 1965.