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Wavelength and energy-dispersive X-ray microanalysis with EMA and SEM-EDXRA on thin sections of soils

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Summary

Organic matter, minerals and iron-manganese nodules were studied in thin sections of soils with an electron microprobe analyzer (EMA) and a combination of a scanning electron microscope (SEM) and an energy-dispersive X-ray analyzer (EDXRA). Both instruments were used to estimate the presence and nature of chemical elements in two selected areas, one containing a combination of organic and mineral material and another inside an iron-manganese nodule. This allowed comparison of the performance of both instruments on two types of soil materials.

The detection of organic matter proved problematic. Of the light elements, N could not be detected with EMA and O was detected but is not specific to organic matter. EMA could not be used for C in this case because the insulating soil material was covered with carbon for conductivity. SEM-EDXRA only detected heavier elements.

EMA produced somewhat better X-ray images of heavier elements, especially from an iron-manganese nodule. However, with organic material, SEM-EDXRA X-ray images were similar to or slightly better than EMA.

An advantage of SEM-EDXRA over EMA is that the soil material can be analysed at various magnifications with a much higher limit and point analysis can be made of loose material.

For soil material, SEM-EDXRA was better as a routine instrument by which the majority of problems could be solved. EMA can be applied as a complementary instrument. Other microanalytical techniques like the ion microprobe mass analyzer (IMMA) were necessary to analyse light elements in organic material of soils.

Acronyms

ED: energy-dispersive
EDXRA: energy-dispersive X-ray analyzer/analysis
EMA: electron microprobe analyzer
IMMA: ion microprobe mass analyzer
SEM: scanning electron microscope
WD: wavelength-dispersive
WDXRA: wavelength-dispersive X-ray analysis.

Introduction

Two instruments are in common use for the study of unconsolidated soil samples or thin sections, the scanning electron microscope (SEM) and the electron microprobe analyzer (EMA). By SEM, one can make secondary electron images of loose soil material or the surface of a thin section but one cannot detect chemical elements. But EMA measures chemical elements on polished surfaces of thin sections or small impregnated blocks of soil material.

Published studies on EMA and SEM

EMA and SEM, alone or in combination with other instruments, have been used on soils by many workers. A list of papers, subdivided according to the instruments used, is given below.

EMA

Brewer et al. 1973; Cescas et al. 1968; Childs, 1975; Delvigne & Martin, 1970; Gillespie & Elrick, 1968; Gillespie & Protz, 1972; Hill & Sawhney, 1971; Innes & Pluth, 1970; Jeanson, 1966, 1969; Leroux et al. 1970; Quershi et al. 1969; Rutherford, 1969; Sawhney, 1968; Seddoh & Pedro, 1974; Sweatmann & Long, 1969; Veen & Maaskant, 1971.

SEM

Amiel, 1975; Benayas et al, 1974; Bjørlykke, 1975; Bresson, 1974a; Bruckert et al. 1974; Chang Wang et al. 1974; Clanton et al. 1974; Dormaar, 1974; Driessen & Schoorl, 1973; Espinoza et al. 1975; Eswaran, 1971, 1972; Eswaran & de Coninck, 1971; Gallavan & Greene-Kelly, 1974; Gillott, 1974; Hetier, 1975; Hetier et al. 1974; Hugenroth & Meyer, 1974; Ingersoll, 1974; Jackson & Sridhar, 1974; Kilbertus et al. 1973; Legigan & Le Ribault, 1974; Leneuf, 1972, 1973; Low & Stuart, 1974; McHardy & Birnie, 1975 (these authors used a SEM with crystal spectrometer attachment); Miedema et al. 1974; Moinereau & Roger, 1975; Nahon, 1976; Nahon et al. 1975; Nixon, 1969; Ogunbadejo & Quigley, 1974; Osman & Eswaran, 1974; Pierre et al. 1974; Quigley & Ogunbadejo, 1974; Riezebos, 1974; Riezebos & van der Waals, 1974; Schmidt-Lorenz, 1974a, b; Sheeran & Yong, 1974; Singer, 1975; Sridhar et al. 1975; Stoops, 1974; Tan et al. 1975; Tovey, 1974; Tovey & Kwong

Yan Wong, 1974; Verheye & Stoops, 1975; Wilding & Drees, 1973, 1974; Wilding & Geissinger, 1973; Wilson, 1975.

EMA and SEM
Bocquier & Nalovic, 1972; Bresson, 1974b; Righi, 1975; Smart, 1974; Wieder & Yaalon, 1974.

Purpose of this work

Our own work concentrated on investigating soil samples with a SEM to which an instrument was attached that could make chemical analyses. This instrument is an EDXRA, which can only analyse heavier elements ($Z \geqslant 11$). But EMA can analyse chemical elements from boron ($Z = 5$) on, and can therefore test elements like O, N and C. SEM-EDXRA, however, has a number of advantages over EMA:
1. SEM-EDXRA can analyse loose soil material and polished surfaces of thin sections and hardened blocks of soil;
2. SEM-EDXRA can measure various elements simultaneously;
3. EMA can analyse elements up to a magnification of about × 500, whereas with SEM-EDXRA analysis up to × 10 000 is possible.

The first results of our work with SEM-EDXRA in soil micromorphology were presented at the 'First EDAX European Users Meeting' at Liège, Belgium (Henstra et al., 1973). A description of the methods of analysis with different applications was given in a later publication (Bisdom et al., 1975). With this technique, elements such as Al, Si, Ca and Fe can be detected in soil material, but to study lighter elements, such as O, N and C, we decided to use EMA. Two different areas in thin sections were selected to evaluate EMA for analysis for light elements and to compare analysis for heavier elements by both EMA and SEM-EDXRA. The first area consisted of organic matter, mineral grains and clay-sized material, whereas the second one formed part of an iron-manganese nodule containing mineral grains. The results are described below.

Instruments

For these experiments, we used an EMA, type Cameca MS 46, and a SEM, type Jeol-JSM-U3 scanning electron microscope, with an EDAX (energy-dispersive X-ray analysis) system. Of these, the EMA is portrayed in Fig. 1, and the various components are indicated. The SEM-EDXRA combination of instruments has been described in a previous study (Bisdom et al., 1975).

Several technical details of EMA and SEM-EDXRA were discussed in the section 'Instruments' by Bisdom et al. (1975). The reader is referred to that paper for an introduction on instruments and some technical principles. The differences between the techniques were summarized in Table 1 of that article. The present paper discusses a few details relevant for comparison of the two instruments. More technical details are given by, for instance, Anderson (1973), Birks (1971), Goldstein & Yakowitz (1975), Hornsveld (1970), McCrone & Delly (1973), Reed (1975) and Seiler (1974).

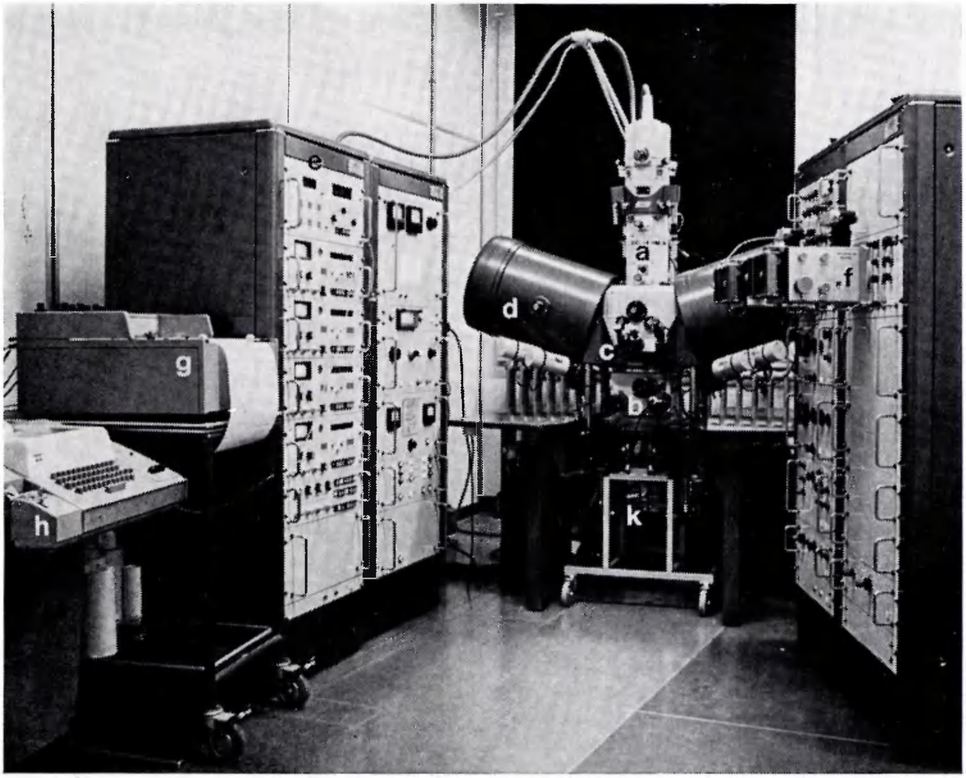


Fig. 1. For the experiments, an electron microprobe analyzer (EMA), type Cameca MS 46, was used. The basic instrument consists of a central optical column (a) with electron gun and electromagnetic lenses and deflexion coils, the specimen chamber (b), an optical microscope (c) for positioning of the area to be analyzed, four X-ray spectrometers (d) enabling analysis of four chemical elements simultaneously.

Four counting channels (e) are coupled to the X-ray spectrometers. Current absorbed by the specimen can be used to obtain a micrograph called an absorbed electron image, the contrast on which is determined by variations in the coefficient of back-scatter of electrons across the specimen surface. Absorbed electron and X-ray images can be displayed on two cathode ray tubes (f) provided with cameras for photography.

An X-Y recorder (g) is present for recording line scans. Moreover, the instrument is equipped with an automatic system of analysis digitalized on punched tape by a teletype (h).

This instrument is of a special type adapted for analysis of radioactive specimens by shielding of the specimen chamber and the spectrometers. A vehicle (k) introduces the sample into the specimen chamber.

Microprobes (EMA) are generally equipped with multiple crystal (or wavelength-dispersive, WD) spectrometers. These spectrometers have good spectral resolution in the order of a few electronvolts (eV), which means that neighbouring elements in the periodic system can be separated perfectly. On the other hand, the intensity of the X-ray beam is greatly diminished after reflection off the crystal. The sensitivity of a gas detector is lower than of a Si (Li) solid-state detector, so that the current from the electron beam onto the specimen must be relatively high. In thin sections from plastic-impregnated soils these high currents may cause heating and contaminate the plastic. During this experiment, a beam current of 10^{-7} A was used, without any observable damage to the specimen.

Another advantage of a WD spectrometer is that in principle elements with low atomic number ($Z \geqslant 5$) can be detected. The energy of this radiation, however, is low and is easily absorbed by the matrix material. This is one of the reasons that detection of elements with $5 \leqslant Z < 13$ can be difficult (Robinson, 1973).

Because the geometric demands for a WD detector assembly are quite severe, specimens for a microprobe must be highly polished. This presents no problem for metal samples, but for plastic-impregnated soil material, where we have substances of various hardness, it is impossible to make thin sections with highly polished scratch-free surfaces which are entirely flat.

With the energy-dispersive (ED) spectrometer from SEM-EDXRA, previously called non-dispersive spectrometer because there is no analysing crystal between the sample and the detector, the X-rays enter the detector unsorted and thus allow detection of all elements (from $Z = 11$) simultaneously.

Contemporary ED spectrometers have a Si (Li) solid-state detector (cooled by liquid nitrogen) with a spectral resolution of about 135 eV, which is worse than for a WD spectrometer. However sensitivity is very high so that the current from the electron beam can be greatly diminished (by a factor of about 1000) reducing chances of damage by the beam and contamination of the specimen. During these experiments, a practical beam current of 10^{-10} A was used.

Between this detector and the vacuum there is a thin (ca 8 μm) beryllium window. From Na ($Z = 11$) upwards, detection and separation of the elements, unless the concentration is too low, raises no problem. For the elements with $Z < 11$, the low-energy radiation is easily absorbed by the beryllium window. As an example: for the Na-Kα radiation this Be window has a transmission of 60 % whereas for O-Kα radiation this is only 2 %. This indicates that elements with $Z < 11$ cannot be detected. Windowless detectors can in principle detect elements with lower atomic numbers, but in practice they are still in the experimental stage.

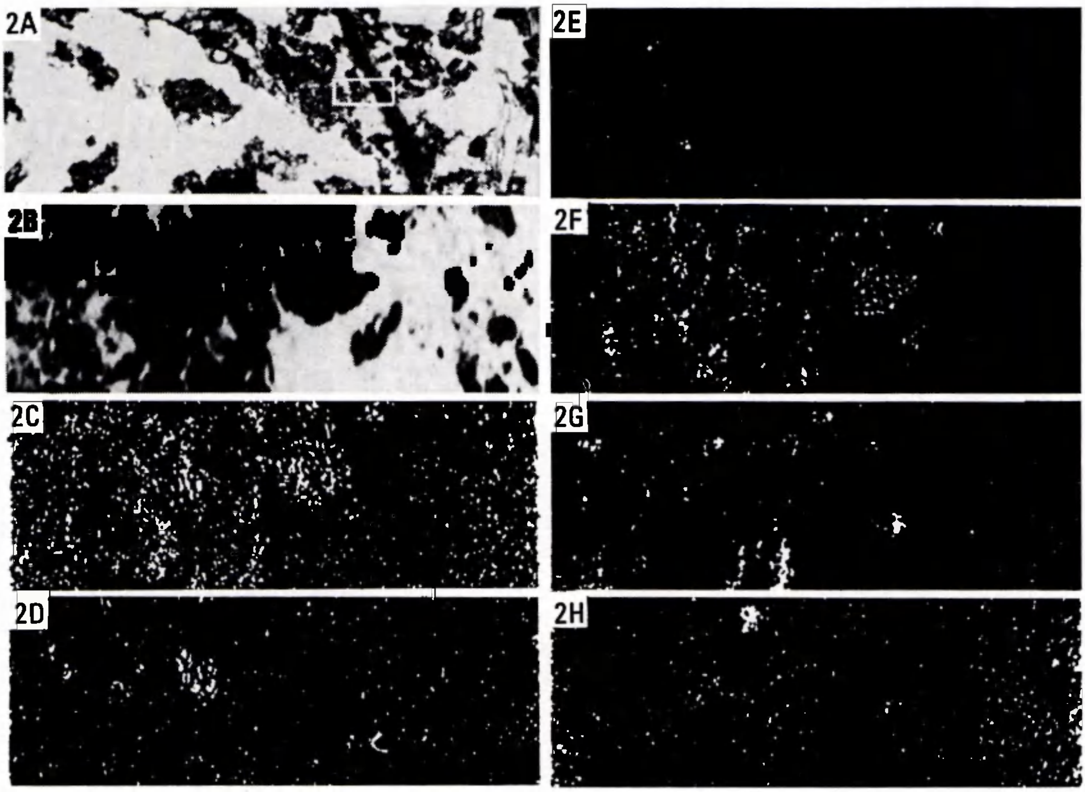
ED detectors have also a higher background level (mainly caused by 'Bremsstrahlung') which gives a much poorer signal-to-noise ratio so that small peaks are submerged into this background.

Because there are no geometrical demands, we can analyze at various magnifications, not only very high (which is impossible with EMA) but also at very low magnifications.

One of the main advantages of ED spectrometers, however, is that we can quickly make a complete qualitative analysis of all sorts of specimens to learn about the principal elements present in the sample.

Examples of the techniques

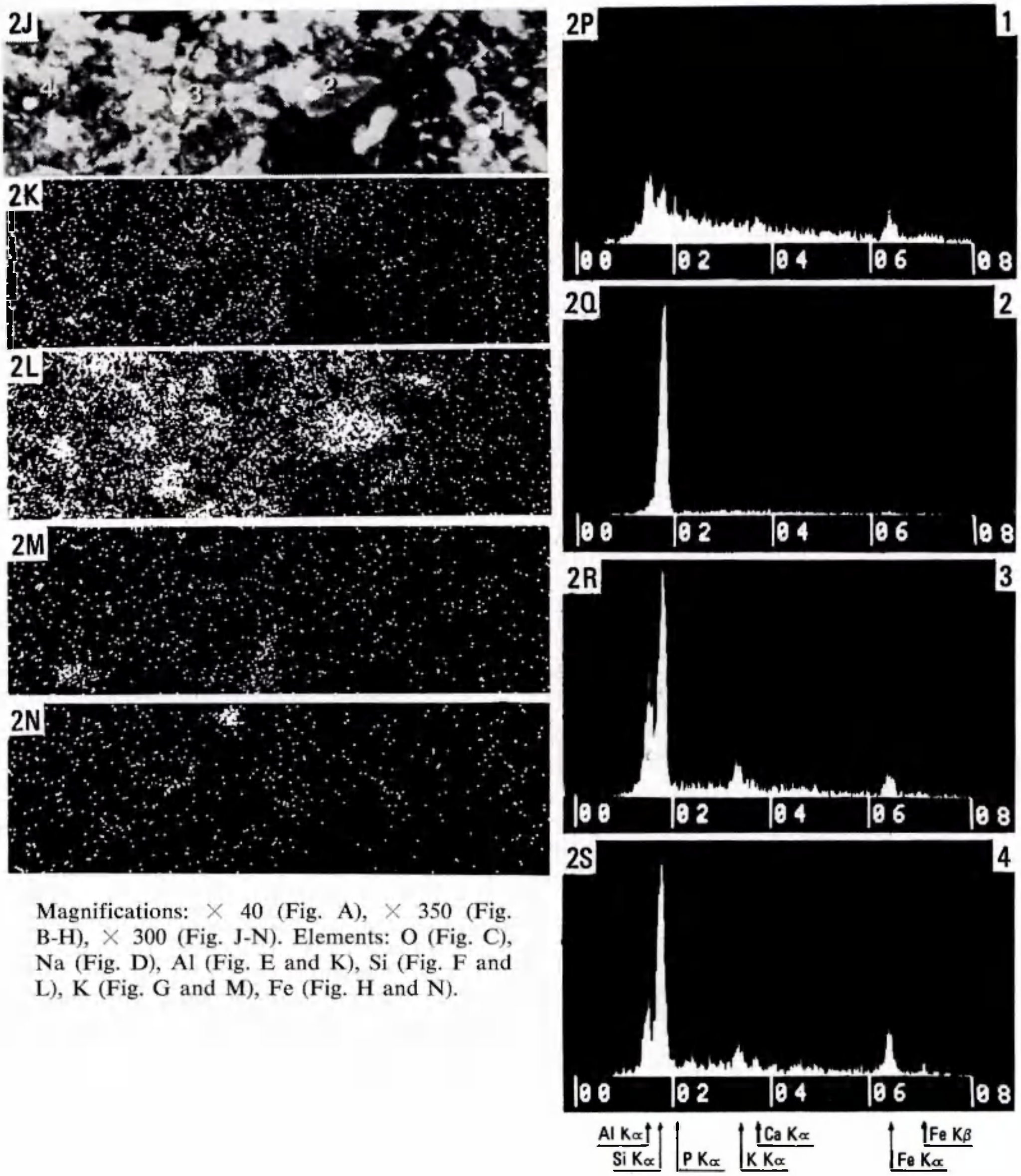
Example 1 concerns analysis of organic matter, excrements, mineral grains and clay-sized material in an Entic Haplorthod (Anon., 1975) near Doorwerth west of



Arnhem, Netherlands, which has been described by de Bakker & Edelman-Vlam (1976). The results of analysis are portrayed in Fig. 2. Of the light elements, only O could be detected with EMA in both organic and mineral material. N was not detected because this is always a difficult element to detect because of characteristic long-wave radiation, which is easily absorbed by the matrix of the specimen, by the crystal (after reflection) and by the detector window. C-coating of the thin section prohibited in this case EMA-analysis for this element. Therefore no light elements exclusively present in organic material could be demonstrated by EMA in this sample. Preliminary experiments with the ion microprobe mass analyzer (IMMA) seem promising. Some results will be published shortly in this journal.

EMA X-ray images of the heavier elements in Example 1 are given in Fig. 2C-2H, in which the image of O (Fig. 2C) has also been included. Na, Al, Si, K and Fe could be detected, while elements like P, S, Cl, Ca and Mn were present in small amounts and were not included in the figure. SEM-EDXRA X-ray images of virtually the same area – the magnification of EMA is × 350 and of SEM-EDXRA × 300 in Fig. 2 and 3 – show Al, Si, K and Fe in Fig. 2K-2N. Sodium was not detected.

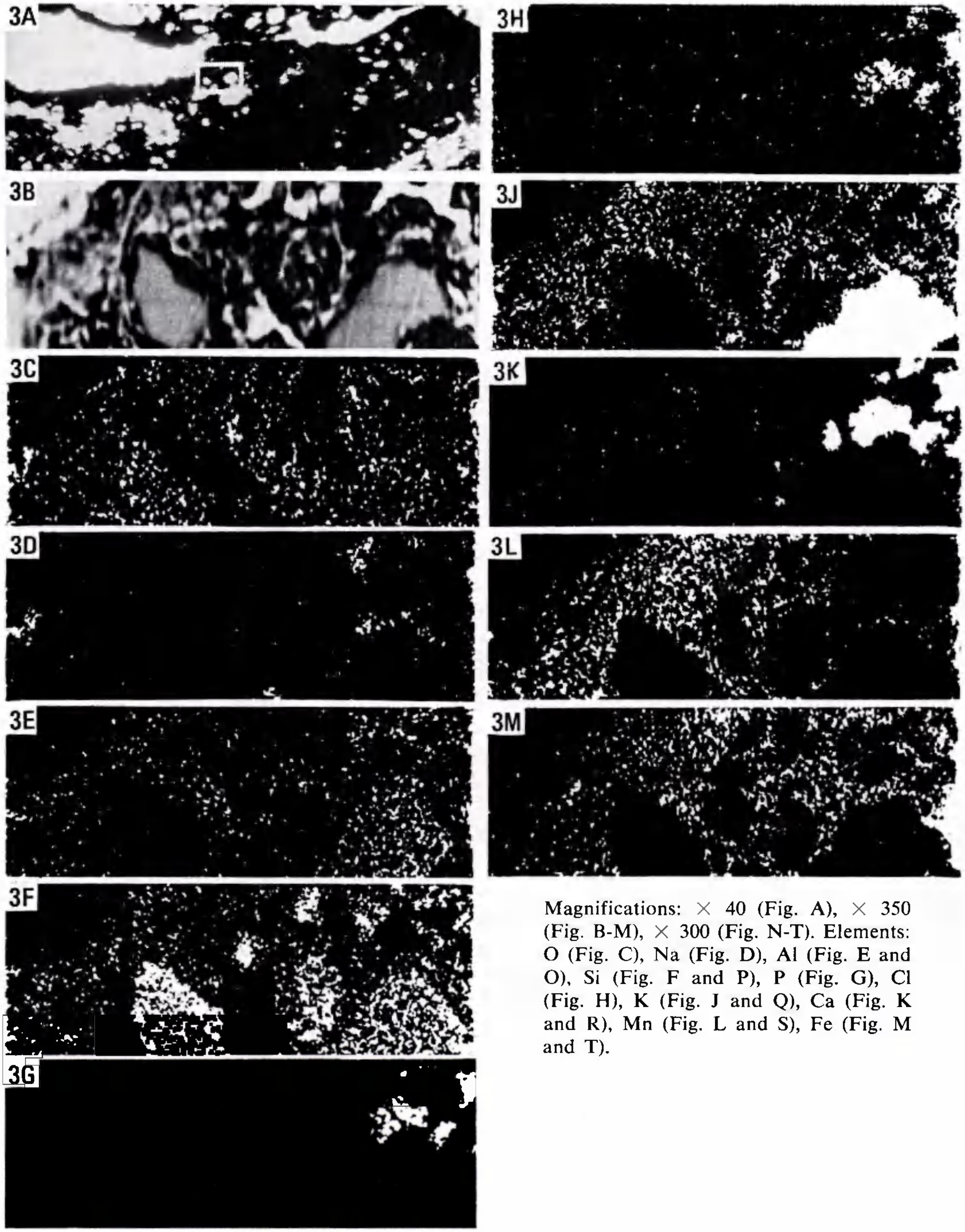
The comparison of the X-ray images of both instruments shows somewhat better



Magnifications: × 40 (Fig. A), × 350 (Fig. B-H), × 300 (Fig. J-N). Elements: O (Fig. C), Na (Fig. D), Al (Fig. E and K), Si (Fig. F and L), K (Fig. G and M), Fe (Fig. H and N).

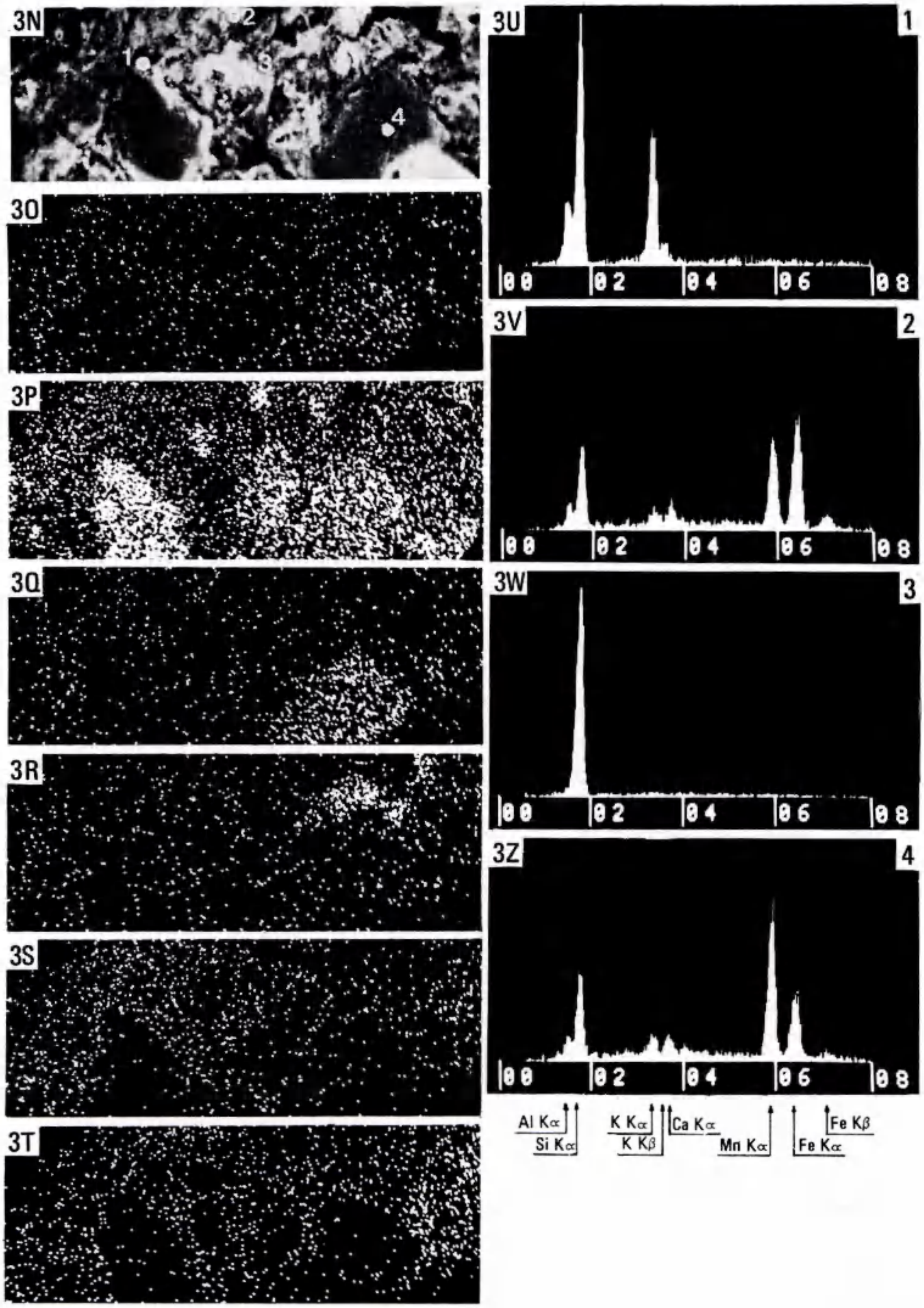
Fig. 2. EMA (Fig. B-H) and SEM-EDXRA (Fig. J-S) techniques with a thin section of a Moder Podzol (Entic Haplorthod) from near Doorwerth, Netherlands. A: Light-microscope photograph of uncovered thin section with area indication from which Fig. B-H are derived. B: Absorbed electron image. C-H: X-ray images of O, Na, Al, Si, K and Fe from soil material in Fig. B. J: Secondary electron image. Note the reversed contrast of the absorbed electron image (Fig. B) with regard to the SEM image. (Fig. J). K-N: X-ray images of Al, Si, K and Fe from soil material in Fig. J. P-S: Point analyses at four points (see text) in Fig. J. Point 1: Elements in a plant fragment; point 2: Si in quartz; points 3-4: analyses of the fine mass in the thin section.

Note: For technical reasons only strips of the originally analysed areas are given in Fig. 2 and 3.



Magnifications: × 40 (Fig. A), × 350 (Fig. B-M), × 300 (Fig. N-T). Elements: O (Fig. C), Na (Fig. D), Al (Fig. E and O), Si (Fig. F and P), P (Fig. G), Cl (Fig. H), K (Fig. J and Q), Ca (Fig. K and R), Mn (Fig. L and S), Fe (Fig. M and T).

Fig. 3. EMA (Fig. B-M) and SEM-EDXRA (Fig. N-Z) techniques with a thin section of a Poldervaaggrond (Typic Fluvaquent), near Bolsward, Netherlands. A: Light-microscopic photograph of uncovered thin section with area indication whence Fig. B-M are derived. B: Absorbed electron image. C-M: X-ray images of O, Na, Al, Si, P, Cl, K, Ca, Mn and Fe from nodule material in Fig. B.



N: Secondary electron image. Note the reversed contrast of the absorbed electron image (Fig. B) with respect to the SEM image (Fig. N). O-T: X-ray images of Al, Si, K, Ca, Mn and Fe from nodule material in Fig. N. U-Z: Point analyses at four points (see text) in Fig. N. Point 1: elements in a feldspar; points 2 and 4: analyses of a mixture of iron-manganese compounds and clayey material; point 3: Si in quartz.

results for Al and Si with SEM-EDXRA, a comparable outcome with Fe and a better image of K with EMA.

Some additional point analyses by SEM-EDXRA are given in Fig. 2P-2S. The position of the points can be found on the secondary electron image – made with SEM – in Fig. 2J. Fig. 2P demonstrates a point analysis in a plant fragment. The $K\alpha$ peaks of Al, Si, P and Fe are clearly discernable, whereas those of Mg, S, Cl and Ca are virtually part of the background. In Fig. 2Q, the silicon of quartz has been measured. Small differences in the fine mass of the thin section are demonstrated in Fig. 2R and 2S. $K\alpha$ peaks of Al, Si, K and Fe are present in both figures. Fig. 2S shows, however, a little more iron and traces of S, Cl and Ca in the point analyzed. Point analyses generally gave more accurate results than did X-ray images. Consequently we often detected small amounts of elements not discernable with X-ray images. With point analyses, we could compare the height of peaks of individual chemical elements, which allows distinction of minute changes in composition between points in the thin section.

No comparison of SEM-EDXRA and EMA for point analyses has been made. Theoretically, however, point analyses of both instruments are comparable, although for example in the case of Ba and Ti where the resolution of SEM-EDXRA is insufficient, EMA is invaluable.

Example 2 for comparison of the performance of the two instruments concerns a part of an iron-manganese nodule of a Typic Fluvaquent (Anon., 1975) near Bolsward, Netherlands, which has been described by de Bakker & Edelman-Vlam (1976). Light-microscopy showed the presence of predominantly quartz in the nodule and some feldspar. A considerable part of the mineral material in the nodule was masked by iron-manganese compounds.

EMA X-ray images demonstrated the presence of O, Na, Al, Si, P, Cl, K, Ca, Mn and Fe (Fig. 3C-3M). Not included are the images of Mg, S and Ti, which were present in small amounts.

SEM-EDXRA X-ray images show Al, Si, K, Ca, Mn and Fe (Fig. 3O-3T). A few point analyses are given in Fig. 3U-3Z. Points 1-4 are indicated in the secondary electron image of Fig. 3N. Al, Si and K were found in point analysis 1 made from a feldspar. The amounts of elements demonstrated by point analysis with SEM-EDXRA are not quantitative. Point 3 (Fig. 3W) is an analysis of quartz. Chemical composition varies slightly in point analyses 2 and 4 made from a mixture of iron-manganese compounds and clayey material. Point 2 contains more iron and less manganese than point 4, but both points have comparable amounts of Al, Si, K and Ca.

In this example, the quality of the EMA X-ray images was superior.

Conclusions

These experiments indicate that SEM-EDXRA gives somewhat better X-ray images than EMA in the thin section with organic matter. The opposite was true, however, with the iron-manganese nodule, perhaps because of differences in the applied beam current and in composition of the tested materials.

The detection of light elements in organic material with EMA gave poor results. Consequently another technique for analysis of light elements in thin sections had to be found, and we did some preliminary tests with IMMA.

SEM-EDXRA can analyse material for chemical elements up to a magnification of × 10 000, whereas EMA has a maximum of about × 500. Also useful is that SEM-EDXRA can analyse loose soil materials by point analysis.

For these reasons, SEM-EDXRA is preferred to EMA as a general instrument for routine soil analysis. EMA has advantages for certain uses. For analysis of light elements, one needs instruments like IMMA.

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