

³¹P NMR STUDY OF PHOSPHORUS IN SOILS

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INTRODUCTION

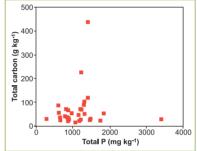
To understand the movement and biological availability of P in soils, knowledge of the chemical forms of P present in soil is required. Organic P can be the dominant fraction of the total P in temperate soils and exists in many different forms. This poster describes our studies to identify and quantify the P forms present in soils from the UK.

EXPERIMENTAL: The study involved a range of 32 soils with differing carbon to phosphorus ratios (Fig. 1).

 31P NMR measurements were made using 0.25 M NaOH / 0.05 M EDTA extracts of the soils. The extracts were freeze-dried and 100 mg of the solid dissolved in 1 ml of 1M NaOH / 0.05 M EDTA for NMR analysis. Spectra

were obtained using a Bruker Avance 500 II instrument.

- To provide a reference signal for quantification of P species, a capillary containing 50 µL of 4 mM methylene diphosphonic acid (MDPA) was placed in the NMR tube. This procedure provides a reference signal near16 ppm.
- To aid identification of myo-inositol hexakisphosphate (Fig 2), an organic P compound known to occur in soil, the extracts were spiked with a small amount of its sodium salt.



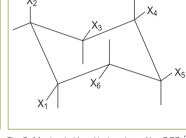


Fig. 1. Phosphorus and C contents of the soils

Fig. 2. Myo-inositol hexakisphosphate, X = -0-P03²-

RESULTS: A plot of concentration of P in the soil extract vs total integrated peak area relative to the internal MDPA standard (Fig. 3) demonstrates that the NMR conditions we used gives quantitative data.

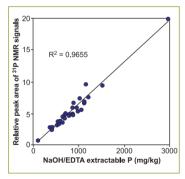


Fig. 3. Concentration of P in the extract vs total integrated peak area of all signals relative to the internal MDPA standard (peak area = 1).

A typical example of the ^{31}P NMR spectra of the soil extracts is shown in Fig 4. The lower trace is the unspiked extract; the upper trace is with a d d e d myo-inositol hexakisphosphate. Because of shifts caused by co-extracted substances, using external standards may result in misidentification of signals.

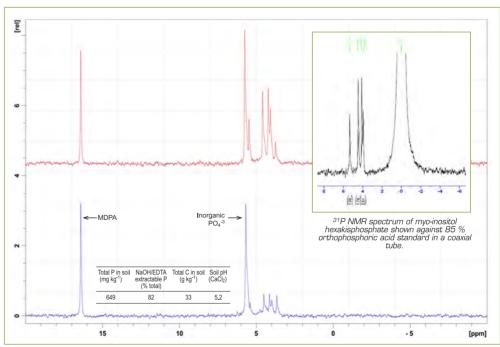


Fig. 4. Typical ³¹P NMR spectra of the soil extracts. The peak area of the spectra of the unspiked soil indicates that 41% of the total P is in organic form. The spike clearly identifies those peaks due to myo inositol hexakisphosphate.

CONCLUSION

- The use of methylene diphosphonic acid in a capillary tube as internal standard and spiking the soil extract with a small amount of *myo*-inositol hexakisphosphate allows quantification of this important organic P species in soil.
- For the 32 soils studied organic P ranged between 11 and 77 % of the total P.