

HANDLING OF SOIL SAMPLES PRIOR TO TESTING

G.A. Kruger, P.J. Doyle and R.E. Karamanos
Saskatchewan Soil Testing Laboratory
University of Saskatchewan
Saskatoon, Saskatchewan

INTRODUCTION

The SSTL installed drying facilities for handling wet soil samples during the summer of 1990 to improve the quality of service to its customers. For many farmers and dealers, the drying of soil samples was a serious constraint to soil sampling. The soil samples dried slowly in their facilities which increased the period of time between the sampling of a field and the printing of the soil test report. Drying the samples at the laboratory minimizes several potential problems related to sample handling: 1) contamination from fertilizer, 2) slow drying of soil samples, and 3) proper identification of samples. Many farmers do not have a suitable location for drying ten to fifteen soil samples. Soil sampling is an easier task if the drying of the samples is completed at the laboratory.

The handling of moist soil samples between the time of sampling and the analysis of that sample at the laboratory can affect the extractable levels of the soil nutrients. Of foremost concern is mineralization of organic nitrogen in the sample by the activity of microorganisms. The effect of a number of soil handling variables were tested in an experiment conducted during the fall of 1990. The objective of the experiment was to determine guidelines for submitting wet samples to the laboratory.

METHODOLOGY

A bulk soil sample was collected from a Thin Black Chernozemic soil near Rosthern, Saskatchewan in early November. The sample was passed through a 10 mm sieve to remove straw and stones and mixed several times by shovel. The bulk sample was divided into two portions - one retained at the moisture content of the field and a second which was moistened to simulate a rainfall event during the harvest season. The oven dry moisture content of the field was 16% which was essentially the wilting point for this loam soil. The moistened sample was mixed by shovel several more times to distribute the added moisture equally throughout the moistened sample. A pair of subsamples indicated that the oven dry moisture content of the moistened soil was 30%. The two bulk samples were again split in two to provide four bulk samples and the bulk samples were placed into soil bags. A set of samples of each moisture content were kept at room temperature overnight while the another two sets were placed in a refrigerated cooler for the same period of time. This treatment was imposed to simulate the effect of keeping the soil samples in a cooler while the sampling unit was working in the field compared to keeping the soil samples at the ambient temperature throughout the working day. The soil samples were subjected to a variety of treatments after 20 hours to simulate possible scenarios that may occur during the shipment of the soil sample to the laboratory. Each treatment was replicated three times. The ten main treatments can be summarized as follows:

- 1) drying in the forced-air dryer for 24, 48, and 72 hours,
- 2) drying at room temperature for 24, 48, and 72 hours,
- 3) incubating the wet samples at 5°C, 15°C, 25°C, and 35°C for 24, 48, and 72 hours and drying in the forced-air dryer for 24 hours, and
- 4) freezing the samples for 24 hours, incubating the frozen samples at 5°C, 15°C, 25°C, and 35°C for 24, 48, and 72 hours and drying in the forced-air dryer for 24 hours.

RESULTS AND DISCUSSION

A. Effect of Keeping Samples Cool During the Day of Sampling

Over the entire experiment, there was no difference in available nitrate between these two treatments (Figure 1). It should be noted, however, that the samples were kept out of direct sunlight at all times. If the sun's radiation was allowed to strike the soil sample in a sealed plasticized bag for several hours, the soil would have been warmed significantly and the possibility for mineralization of nitrogen in these samples would be high.

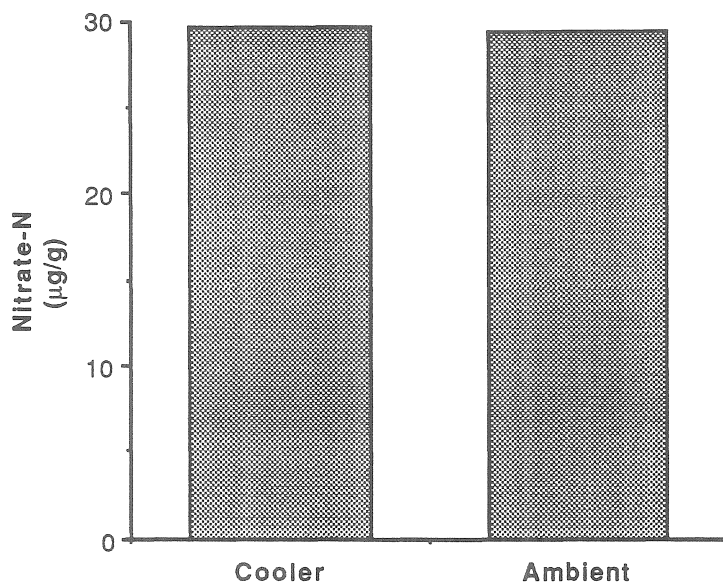


Figure 1: Effect of field handling on nitrate content of soil samples ($\mu\text{g N0}_3\text{-N/g soil}$)

B. Effect of Moisture Content of the Sample

All of the treatments were conducted at two oven dry moisture contents - 16% and 30%. Over the entire experiment, the level of available nitrate in the wetter samples averaged $2.7 \mu\text{g N0}_3\text{-N per g}$ of soil higher than the corresponding drier samples at essentially the wilting point (Figure 2). This corresponds to 5.4 lb of N for a 6" sample which would translate into a difference in fertilizer recommendations of 15 lb N per acre. Among the main treatments, the smallest change in available $\text{N0}_3\text{-N}$ occurred in the samples stored in the moist state (Figure 3). Samples dried immediately in the SSTL dryer or at room temperature increased an average of $2.0 \mu\text{g N0}_3\text{-N per g soil}$ at 30% moisture compared to 16% moisture. The average increase of $\text{N0}_3\text{-N}$ over all four thawing temperatures was $4.0 \mu\text{g/g}$ for the frozen samples which would reduce nitrogen recommendations by up to 24 lb N/ac. Among the samples kept wet for an equivalent period of time, only the samples at the 30% moisture content stored at 35°C had a large increase in available nitrate relative to that found in the frozen samples thawed at 5°C , 15°C , 25°C , and 35°C . El-Hout and Blackmer (1990) found that soil samples maintained their integrity provided that moist samples were received at the soil testing facility within 48 hours and kept at less than 25°C during that time.

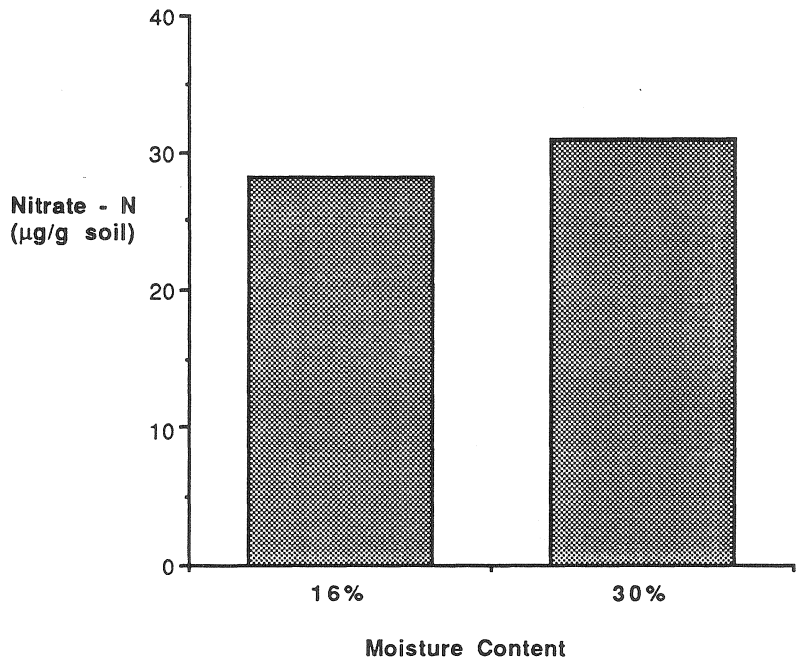


Figure 2: Effect of moisture content of the soil sample on level of available nitrate

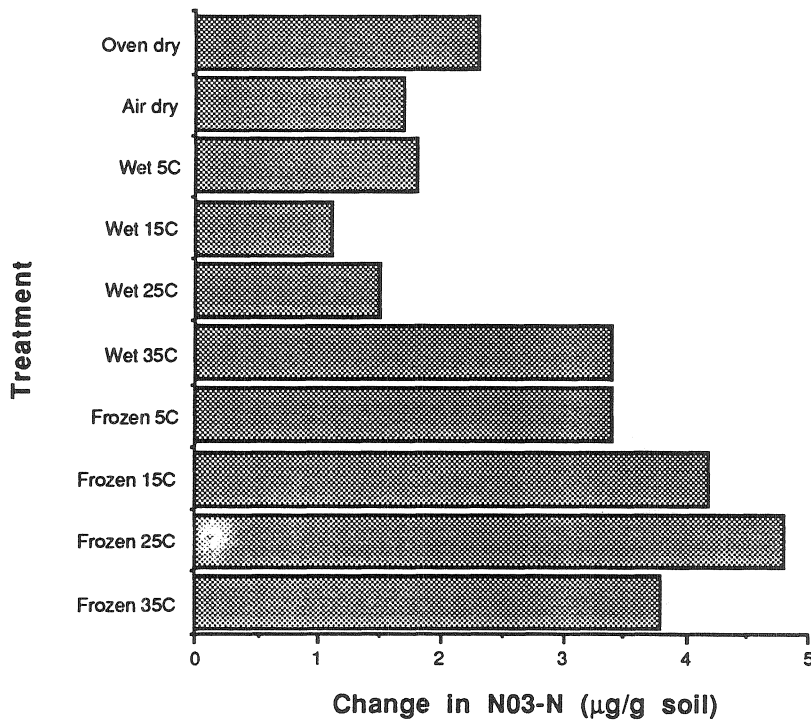


Figure 3: Average increase in N03-N from moistening of soil samples

C. Effect of Drying Samples in Different Environments

Two drying environments were compared for their effect on the available nitrate content of the samples. Soil samples were dried 1) at room temperature without supplemental ventilation and 2) in the SSTL dryer equipped with forced air heated to 32°C. The 36 samples dried in the SSTL oven averaged 27.8 µg NO₃-N/g soil compared to 27.1 µg NO₃-N/g soil for those dried at room temperature.

The method of drying the soil samples can influence the level of available nitrate in a sample. Factors such as the temperature of the drying air, depth of soil in the soil tray, type and shape of container and the presence of forced air can introduce small changes in the level of available nitrogen in the sample (Selmer-Olsen et. al., 1971). The effect of these parameters on the level of available nitrogen in the sample is small, however, when compared with the precision of the analytical instrumentation. The difference of 0.7 µg NO₃-N per g soil observed in this experiment between drying the samples in the forced air oven and at room temperature is not practically significant.

D. Effect of Freezing Soil Samples Prior to Drying Them

Over the entire experiment, the average nitrate level in samples kept moist at 5°C, 15°C, 25°C and 35°C was slightly higher than in samples that were frozen and allowed to thaw at the same temperatures (Figure 4). However, when the data for individual treatments is compared, freezing the soil samples had a profound effect on the level of available nitrate. The average level of available nitrate was lower in the unfrozen samples at 5°C, 15°C, and 25°C than in frozen samples (Figure 5). Only when the temperature during shipment exceeded 25°C do moist samples have a higher nitrate content than the frozen samples.

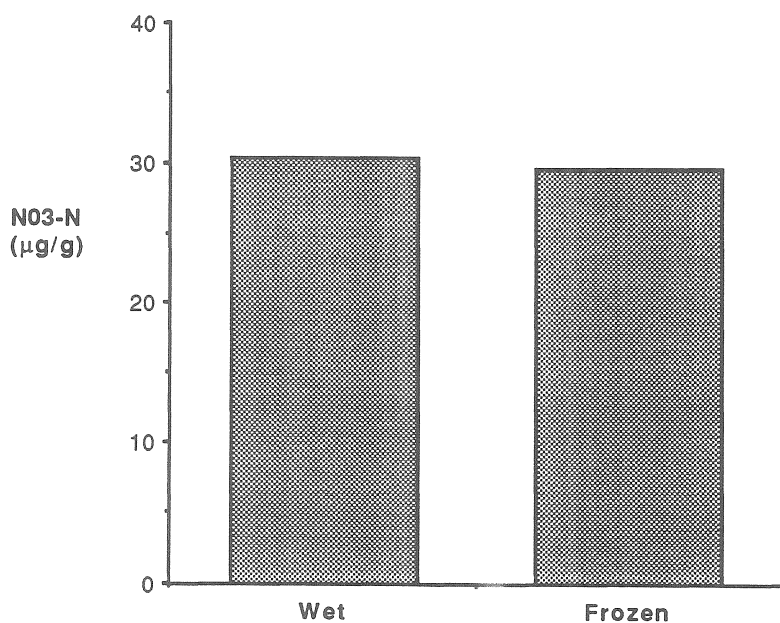


Figure 4: Comparison of Average N03-N Levels for Wet and Frozen Samples

The freezing of the soil samples, itself, has little effect on the level of available nitrate. If the samples were lightly frozen and analyzed after a short thawing period of several minutes, no change in nitrate level would be observed. Nelson and Bremmer

(1972) found that samples frozen at -5°C , thawed rapidly and analyzed immediately had nitrate levels equivalent to field moist samples. Westfall et. al. (1978) observed that the content of nitrate in air-dried samples was similar to frozen samples over the course of 7 days. Mack (1963) found, however, that freezing stimulated the activity of microorganisms during subsequent incubation at 24°C . The rate of respiration and evolution of CO_2 is increased after thawing (Soulides and Allison, 1961). The effect of freezing on the soil microorganisms is greatest in samples that are frozen slowly and thawed quickly. Soil samples submitted to a laboratory for analysis are usually frozen quickly and thawed slowly while in transit. The size of the ice crystals formed during these events would cause the least possible injury to the microbial population. Still, the large increase in nitrate in frozen samples that incubate for a period of time prior to analysis is a greater risk than the shipment of field-moist samples that are kept cool.

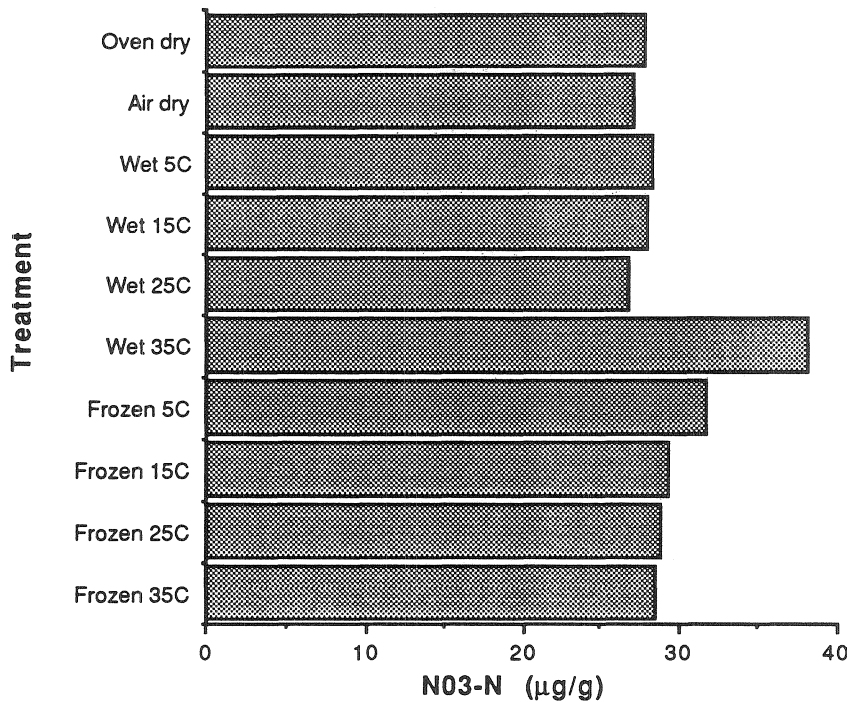


Figure 5: Effect of sample handling on N03-N content of sample

A comparison of the levels in moist and frozen samples at the two moisture contents is shown in Figure 6 and 7. Frozen samples that have a higher moisture content have a large increase in nitrate relative to drier samples. With samples that are retained moist without freezing, the moisture content has no effect on the level of nitrate at 25°C or less.

E. The Effect of Time on the Level of Nitrate in Soil Samples

The level of nitrates in soil samples increase with time as incubation increases. The typical effect of time on nitrate level is shown in Figure 8. Up to 48 hours, the increase in nitrate content of the sample is small at less than 25°C and would not affect nitrogen recommendations significantly. This work verified that the results of El-Hout and Blackmer (1990) for Iowa soils also are applicable to this Saskatchewan soil.

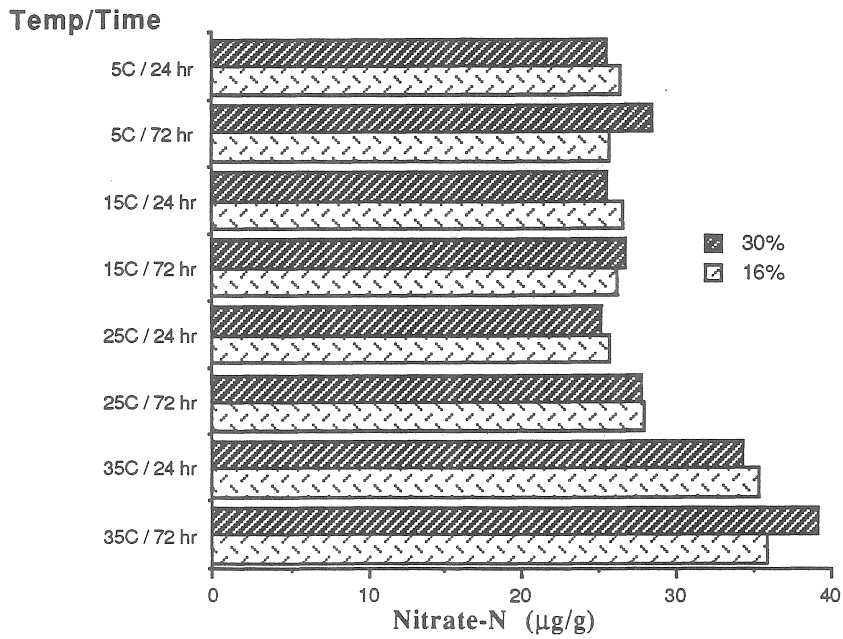


Figure 6: The effect of soil moisture on the nitrate level in moist samples

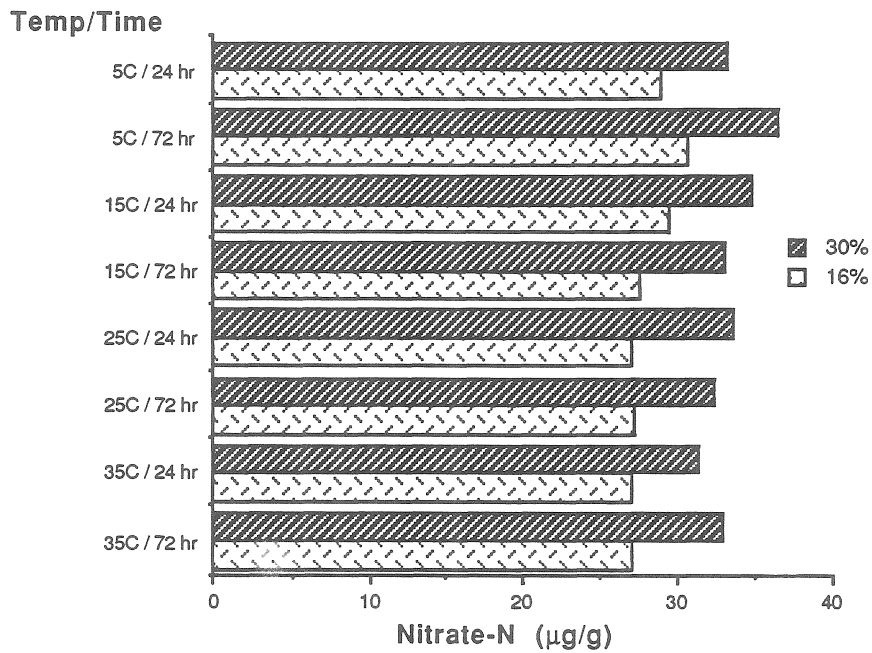


Figure 7: The effect of soil moisture on nitrate levels in frozen samples

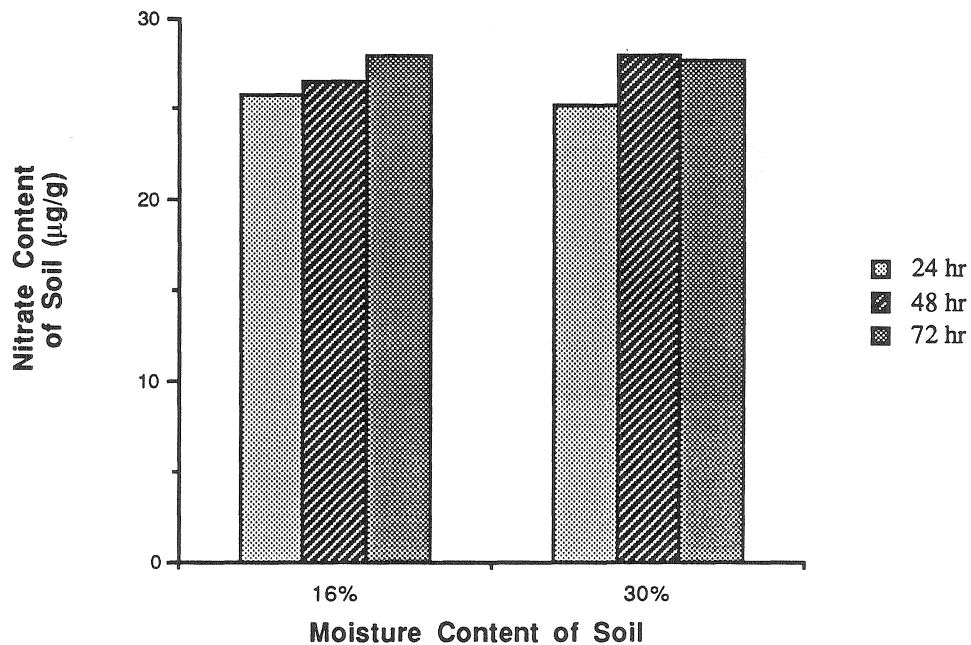


Figure 8: Effect of Time on the Level of Nitrate in Unfrozen Soil Samples at 25°C

CONCLUSION

The proper handling of soil samples in the field and during shipment to the soil testing laboratory is crucial for maintaining the sample in a state that is representative of the field. Any precaution which reduces the possibility of mineralization in the sample should be followed. This includes:

- 1) keeping the sample out of the sun while sampling in the field, preferably in a cooler.
- 2) cooling the sample to 5°C before shipment if possible.
- 3) shipping the sample as quickly as possible to the laboratory (within 48 hours).
- 4) ship the samples in field-moist state without freezing.

Freezing soil samples increases the mineralization of nitrogen once the sample thaws. The effect is most pronounced in moister samples.

REFERENCES:

- El-Hout, N.M. and A.M. Blackmer. 1990. Handling soil samples for the late-spring nitrate test. Agronomy Abstracts, 1990 Annual Meeting, ASA, CSSA, SSSA, San Antonio, Texas
- Mack, A.R. 1963. Biological activity and mineralization of nitrogen in three soils as induced by freezing and drying. *Can. J. Soil Sci* 43:316-324.
- Nelson, D.W. and J.M. Bremner. 1972. Preservation of soil samples for inorganic nitrogen analyses. *Agron. J.* 64:196-199.

Selmer-Olsen, A.R., A. Oien, R. Baerug and I. Lyngstad. 1971. Pre-treatment and storage of soil samples prior to mineral nitrogen determination. *Acta. Agric. Scandinavica* 21:57-63.

Soulides, D.A. and F.E. Allison. 1961. Effect of drying and freezing soils on carbon dioxide production, available mineral nutrients, aggregation, and bacterial population. *Soil Sci.* 91:291-298

Westfall, D.G., M.A. Henson, and E.P. Evans. 1978. The effect of soil sample handling between collection and drying on nitrate concentration. *Com. Soil Sci. Pl. Anal.* 9:169-185.