# WESTERN SOIL AND OVERBURDEN TASK GROUP ROUND ROBIN ANALYSIS PROGRAMS[1]

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Abstract.--Analytical data from soil and overburden is used extensively in reclamation planning. The quality of the data is often strongly related to the quality of the final reclamation product. Round robin analysis programs are one way in which an individual laboratory can monitor and improve quality control. The data from several round robin programs is used in this report to indicate where improvements in analytical methods and procedures are needed.

## INTRODUCTION

Accuracy and precision of analytical data affect reclamation planning and the potential for construction of quality minesoils. Poor accuracy and precision could result in, 1) unnecessary special handling of suitable overburden material, 2) incorporation of potentially toxic or acid-forming material into active ground water recharge zones, and 3) utilization of unsuitable material to construct minesoils. Because of the extensive use made of analytical data in reclamation planning, efforts have been made to develop programs to assess and improve laboratory quality control (Sandoval, 1979; Carlstrom and Harrington, 1983, 1984; Severson and Fisher, 1985, 1986, 1987). These efforts are mainly round robin programs; a round robin analysis program is an informal interlaboratory comparison of analytical precision based on the analysis of uncertified sample splits. The data resulting from these programs, 1) allow an individual laboratory to compare their results with other laboratories as one means of evaluating quality control, 2) show the range in values reported by several laboratories for individual parameters and this suggests how sensitive a method is to differences in laboratory techniques, and 3) suggests how differences in analytical methods and proceedures affect the reported results.

The objective of this report is to illustrate the types of differences that can be expected when several laboratories analyze a split of single sample by the same or different laboratory methods. Recommendations, based on the results of the six studies cited, should improve the quality of future round-robin programs.

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## ANALYTICAL TECHNIQUES

Procedures for the determination of each parameter are recommended to the laboratories participating in the round robin program. Many of the participating laboratories follow the recommended procedures with minor modifications in sample size, soil to solution ratio, or length of extraction time. In other instances, laboratories replace the recommended procedure with a different one, or do not report the procedure that was used. These are, perhaps, the greatest problems in interpreting the data generated by soil and overburden analysis and associated round robin programs. It is difficult to determine what effect the procedural modifications, or differing procedures, have on the reported analytical values. As an example, effects of reaction time and different extractants are illustrated (fig. 1) for four parameters from a recent round robin program (Severson and Fisher 1987). Reaction time appears to have no predictable effect on soluble Na or extractable Cu. The values reported by several laboratories using the same reaction time show a wider range than the values reported by laboratories using different reaction times. Few predictable trends can be observed for using different extractants and reaction times for extractable Cu, Mo, or Se. The limiteddata for Mo suggests that as extraction time is increased, acid ammonium oxalate extracts increasing amounts of Mo. This same trend, however, is not evident for AB-DTPA as an extractant for Cu, Mo, or Se. This suggests that differences in operational procedures, instruments, and personnel among laboratories may have as great an effect on the reported analytical values as do the type of extractant used or the length of reaction time used for these samples analyzed by these laboratories.

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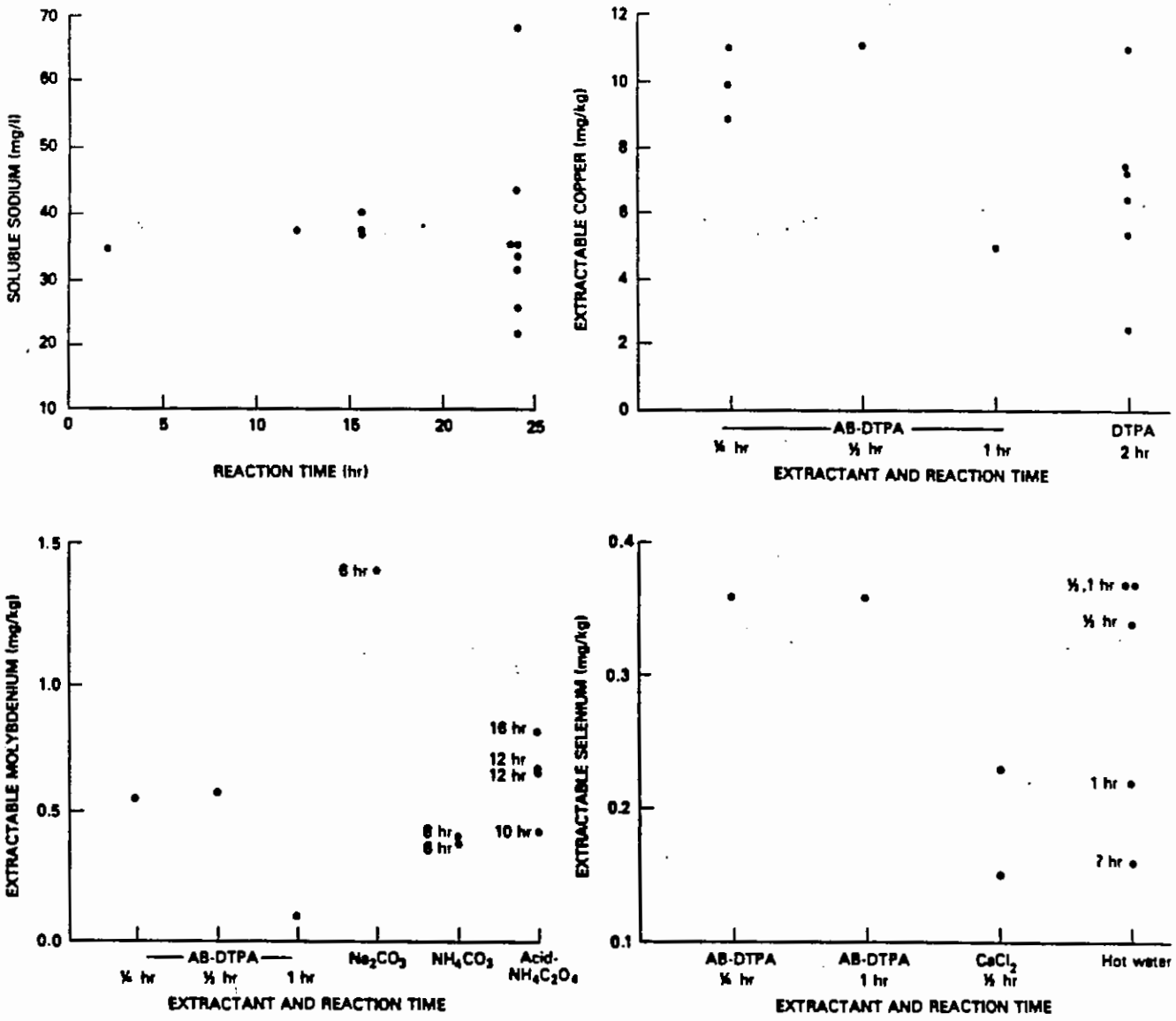


Figure 1.--Effects of differing reaction times and extractants on four parameters.

## SAMPLE CHARACTER

The physical and chemical characteristics of soil and overburden materials, and the way in which they are prepared for chemical analyses can affect the consistency of analytical results within and among laboratories. Average soil or overburden samples; those with typical contents of clay, organic matter, and trace metals, and of normal salinity or sodicity, are much easier to analyze than are abnormal samples. Analyses of average soil or overburden samples by several laboratories result in more consistent results than analyses of abnormal samples. However, obtaining accurate and precise results from abnormal samples is critical in reclamation planning. Those analyses largely determine whether or not, 1) special handling of soil or overburden material is required, 2) toxic or acid-forming materials are present, and 3) the

Table 1.--Subjective classification for reproducability of the analytical techniques used to measure parameters as based on the results of six round robin analysis programs.

| Relative Reproducability | | | | |
|---|---|---|---|---|
| Good | | Fair | | Poor |
| pH | SAR | Saturation % | Carbon-organic | AP |
| EC | Soluble Ca | Carbon-ash | Cu | CEC |
| Texture | | Soluble Mg | NP | ESP |
| | | Soluble Na | ABP | Exchangeable Na |
| | | | | B |
| | | | | Se |
| | | | | Mo |
| | | | | Available N |

soil and overburden material which is best suited for use in constructing quality minesoil is identified. Much work needs to be done to modify existing methods or to develop new methods so that consistent results can be obtained from abnormal samples.

Approximately twenty parameters are typically evaluated in the round robin programs cited above. To obtain data for all parameters, many different laboratory methods and analytical instruments are required. An attempt has been made to classify the reproducability of results obtained for each parameter based on the data and discussions from the six round robin analysis programs (table 1). In general, those parameters which are determined from a water extract of a sample tend to give results of good to fair reproducability. Parameters which require the determination of exchangeable cations, special extracting solutions for trace elements, or the determination of acid-forming potential tend to give results of fairly poor to poor reproducability. These observations are subjective and based on limited results. We speculate that, for samples of average composition, more parameters would be classified as providing fair to good reproducability, and for samples of abnormal composition, more parameters would be classified as fairly poor to poor in their reproducability.

The way in which samples are prepared for analysis also affects the results generated within and among laboratories. Generally, a sample is disaggregated and sieved through a 2mm screen. The portion passing the screen is saved for analyses. If the 2mm material is further ground to pass a 60 or 100 mesh sieve the results of analyses are more consistent than for the 2mm fraction. While this grinding increases the reproducability of data from the round robin analysis program, it may not provide suitable data for making agronomic and reclamation decisions. The way in which a finely ground sample responds to extraction methods and analysis in the laboratory may be much different than the way unground overburden material responds to natural environmental conditions in the field.

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