PLANT ANALYSIS : A MEASURE OF SUCCESSFUL RECLAMATION[1]

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Abstract.--A comprehensive evaluation of reclamation success may require analysis of vegetation for trace elements such as Se, Mo, Cu, B, etc. Phenomena of spoil weathering and subsequent release of trace elements needs further study. The effect of plant growth stage on elemental content needs to be evaluated for a greater number of reclamation species.

INTRODUCTION

Regulatory agencies of the western U.S. currently evaluate reclamation success in part by determining whether an operator has established a diverse and vigorous vegetative cover - consistant with the intended post-mining land use. The mining of coal, uranium, bentonite and other minerals may place earthen materials containing elevated levels of trace elements within the root zone of reestablished vegetation.

The National Research Council (1980) has identified the elements of greatest concern in coal and coal resource development as arsenic (As), boron (B), cadmium (Cd), lead (Pb), mercury (Hg), molybdenum (Mo) and selenium (Se). High levels of As, B, Cd, Hg, and Pb may be toxic to biological systems including plants. Enriched levels of available Mo and Se may lead to accumulation of these elements in vegetation rendering it toxic to livestock.

Uranium mining and milling involves the handling of materials often containing high concentrations of As, Mo, Se and pyrite (Harshman, 1974).

Bentonite mining in Wyoming involves handling of marine sedimentary deposits containing potentially elevated levels of B, Se and pyrite (N.L. Baroid, 1984).

Prevention of trace element toxicities and nutrient deficiencies involves adequate chemical characterization of overburden, regraded spoil and topsoil (Munshower, 1983). Analysis of mine spoils for plant available trace elements may not adequately address spoil weathering and subsequent release of potentially toxic amounts of these trace elements. Increases in plant availability of Se and B have been observed in uranium and coal spoils over time.

Plant analysis to assess vegetative quality prior to bond release of reclaimed lands has been suggested (Munshower, 1983; Boon, 1984).

PLANT UPTAKE OF TRACE ELEMENTS ON RECLAIMED LANDS

The presence of elevated levels of Se and Mo in mine spoils will be used to demonstrate the need for more detailed regraded spoil and plant tissue analysis.

Selenium

The history of Se poisoning of livestock in Wyoming is well documented (Beath, 1982; Anderson et al., 1961). Selenium is considered essential in trace amounts for most animals. In excess, poisoning results and is locally known as "blind staggers" or "alkali disease" and results from the ingestion of contaminated plants and water supplies. The availability of Se to plants increases under oxidized/alkaline conditions which prevail in the western U.S. soils and overburden. Selenium generally occurs as the selenate ion under these conditions (Williams and Byers, 1930; Workman, 1983).

Plant samples containing greater than 5 ppm Se are considered toxic to livestock (National Research Council, 1976). The maxiumum recommended concentration reported by the Nuclear Regulatory Commission (1980) is 3 ppm.

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Wyoming (1984) and Montana (1983) have recently lowered the "suspect" level for extractable Se in soils and overburden to 0.1 ppm. This value is based on the work of Olsen et al. (1942) and Soltanpour and Workman (1980). Both studies suggested that hot water extractable Se in excess of 0.1 ppm could produce toxic plants.

The value of 0.1 ppm has been proposed for topsoil, overburden and regraded spoils due to the possibility of subsoil feeding. Grasses and some other shallow rooted plants may obtain Se from the 2nd or 3rd foot of the soil profile (James and Shupe, 1984). Olsen et al. (1942) observed that subsoil feeding of soil Se by plants was necessary to account for plant Se concentrations. Kubota and Cary (1982) suggested that subsoil feeding may contribute to the lack of correlations between soil Se and plant concentrations. Soltanpour et al. (1982) found that the Se concentration in wheat grain could only be predicted if soil samples were taken to a 90 cm depth. Similarly, the Se indicator plants (Astragalus pectinatus and Oonopsis multicaulis) were found growing on the Samsil soil characterized in Table 1 below. Selenium concentration increased abruptly below the 40 cm depth.

Table 1.--Distribution of Se within a soil profile

Horizon	Depth (cm)	pH	EC (mmhos/cm)	SAR	Se (ppm)	Texture (USDA)
A1	0 - 5	6.7	0.3	0.5	0.02	l
C1	5 - 28	7.2	2.4	0.1	0.02	sl
C2cs	28 - 40	7.6	4.5	0.5	0.03	sl
C3rcs	40 - 75	7.2	6.2	0.5	0.22	sil
C4r	75 - 120	6.5	4.2	0.7	0.50	sil

Total Se concentrations in sedimentary rocks of the western U.S. range from 0.1 to 890 ppm (Lakin, 1961). Naturally occurring seleniferous areas of Wyoming include the Chalk Buttes east of Rock River, the Poison Basin northwest of Baggs, and the Alkali Basin north of Sinclair (Beath, 1982). Total Se concentrations in excess of 500 ppm have been reported in overburden from the Gas Hills, Shirley Basin, and the Southern Powder River Basin in Wyoming (Harshman, 1974; Sharp and Gibbons, 1964). Table 2 illustrates a substantial difference between total and extractable forms of trace elements in an overburden core from the Shirley Basin Uranium district.

Table 2.--Uranium overburden analysis - Shirley Basin, Wyoming

Interval (ft)	ppm As	ppm Se	ppm Mo
0 - 52	87 (0.02)*	5.0 (0.05)*	25 (1.6)*
52 - 81.5	89 (0.02)	5.0 (0.05)	25 (1.5)
81.5 - 83	93 (0.02)	410 (1.0)	140 (14.3)
83 - 120.6	97 (0.02)	30 (0.05)	190 (79.9)
120.6 - 134	87 (0.02)	5 (0.05)	200 (82.5)

* numbers in parentheses indicate extractable values.

As the spoils are introduced into an oxidized environment the plant availability of many trace elements becomes increased. Abandoned uranium spoils near Baggs, Wyoming contained extractable Se as high as 76 ppm (Wyoming, 1985).

Jump (1985) found extremely high concentrations of Se in plants growing at an abandoned uranium mine in the Southern Powder River Basin, Wyoming (Table 3). It is interesting to note that traditional Se indicator species such as Astragalus bisulcatus were not present on the site.

Table 3.--Southern Powder River Basin, Wyoming.

# of samples	Species	Range of Se (ppm)
7	Oryzopsis hymenoides	37.2 - 189
6	Agropyron smithii	46.5 - 175
1	Stipa comata	123
1	Populus deltoides	114

The mining of coal also involves handling materials containing high levels of Se (Boon and Smith, 1985). Several coal mines in Wyoming have encountered sufficient quantities of seleniferous overburden to warrant special handling operations. Numerous studies conducted by the mining industry seem to implicate carbonaceous materials such as rider seams, partings and top-of-coal cleanings as sources of selenium enrichment.

Elevated levels of Se can also be found in bentonite overburden (Table 4). The extensive investigations into retorted oil shale have demonstrated Se concentrations of 1.24 ppm extractable from the Piceance Basin of Colorado (Klusman, 1979).

Table 4.--Bentonite overburden analysis - Colony, Wyoming.

Lithology	pH	SAR	Se*	B*	ABP
silty coal	7.5	28.2	0.21	3.49	-7.4
gray shale	6.3	15.3	0.29	1.97	-7.3
bentonite shale	8.3	18.3	0.27	6.39	-3.3
shale/siderite	7.2	14.7	0.19	1.69	-3.7

* extractable Se and B ppm.

Table 5.--Uranium overburden analysis - Gas Hills, Wyoming

	Interval (ft)					
	40-50	50-60	60-64	64-68	68-75	76-80
Mo*	16.1	18.3	23.6	90.4	288	143
Cu*	0.05	0.04	0.03	0.05	0.05	0.13

* extractable Mo and Cu (ppm)

Molybdenum

Molybdenum (Mo) is generally considered essential in trace amounts for plants and animals. However, forage plants with Mo concentrations toxic to cattle have been identified in the Western United States (Kubota, 1975; Stone et al., 1983). Kubota (1975) indicates that the Mo induced copper (Cu) deficiency known as molybdenosis may be a more widespread problem than has been generally recognized. Disruption of rock and soil during mining can mobilize enough Mo to cause pronounced molybdenosis in cattle (Stone et al., 1983).

The availability of Mo to plants increases under conditions of high pH (Alloway, 1973). Therefore, Mo levels in forage could be increased by the alkaline conditions that prevail in soils and overburden in the west. Conversely, the solubility of Cu is greatest under acid conditions and is greatly reduced under calcareous or alkaline conditions (Lindsay, 1979).

High levels of available Mo do not generally result in an observed effect on plant growth. This renders detection of high levels of plant tissue Mo difficult without laboratory analysis. Webb and Atkinson (1965) consider forage containing 5 ppm Mo to be toxic to livestock. A Cu to Mo ratio of 2:1 is considered the lower critical value in forage whereas a ratio of at least 5:1 is considered the minimum to meet long-term nutritional needs of livestock (Miltimore and Mason, 1971).

Wyoming (1984) currently considers a soil or overburden extractable Mo level of 1.0 ppm to be "suspect" of producing vegetation with a Cu to Mo ratio of less than 2.0. High levels of Mo are commonly found in association with uranium deposits. Table 5 demonstrates high Mo concentrations in uranium overburden from the Gas Hills, Wyoming.

Dressen and Marple (1979) found levels of Mo in alkali sacaton (Sporobolus airoides) and fourwing saltbush (Atriplex canescens) to exceed 170 and 270 ppm, respectively. Copper to molybdenum ratios were less than 0.5. It is worth noting that neither of these species are legumes, hence are not considered Mo accumulators. This is not meant to imply that toxic vegetation will be produced on all uranium spoil reclamation. Table 6 lists plant analysis data from two successfully reclaimed areas in the Gas Hill uranium district.

Table 6.--Plant analysis - Gas Hills, Wyoming

Species	Site	Al	As	Cu	Mo	Se	Cu/Mo
		ppm					
Astragalus cicer	A	111*	0.89	9.0	6.1	1.1	1.5
Oryzopsis hymenoides	A	65	0.14	8.3	2.0	0.7	4.3
Medicago sativa	A	313*	4.67	11.3	6.4	1.0	1.8
Stipa viridula	A	29	0.50	8.6	1.3	0.6	6.7
Astragalus bisulcatus	B	29	0.23	10.6	0.8	313	13.8
Elymus junceus	B	43	0.39	10.5	0.6	1.8	17.0
Lupinus sp.	B	60	0.43	11.1	4.9	3.3	2.2
Atriplex canescens	B	11	5.0	8.7	0.8	4.2	11.2
Medicago sativa	B	140*	2.2	14.7	2.4	4.7	6.1

* Indicates soil contamination of sample.

In only one case is 5 ppm Se exceeded. The fact that the Astragalus bisulcatus, a known primary selenium accumulator plant, contained only hundreds rather than thousands of ppm Se indicates appropriate placement of unsuitable overburden. The Cu to Mo ratios are near the critical value for cicer milkvetch (Astragalus cicer), 'Ladak' alfalfa (Medicago sativa) and Lupinus sp. Only 'Ladak' alfalfa appeared to have been grazed to any extent by wildlife and they made up a small portion of the overall plant community.

Revegetation of retorted oil shale may also involve materials high in available Mo. Schwab et al. (1983) concluded that at least a 90 cm soil cover over retorted oil shale was necessary to prevent the production of vegetation with a Cu to Mo ratio less than 2:1.

Surface coal mining in the western United States may also involve handling overburden materials with elevated Mo levels. Erdman et al. (1978) identified leguminous species growing on mine spoils of a southeastern Montana coal mine as having Cu to Mo ratios less than 2:1. Munshower and Neuman (1978a, 1978b) also point out that low Cu status is typical of both native vegetation and plants growing on reclaimed coal spoils. This situation further skews the Cu to Mo imbalance.

Timing of Plant Tissue Sampling

It is important to recognize that the elemental content of vegetation does not remain constant throughout the growing season. Gough and Erdman (1980) found that Wyoming big sagebrush (Artemisia tridentata subsp. wyomingensis) displayed significant seasonal variability for many major and trace elements. The authors concluded that "in general the element content of young tissue (predominantly leaves, stems, and influorescences) fluctuates more, has higher concentrations, and shows greater differences between seasonal means than older sagebrush tissue (predomiantly stems)." Munshower and Neuman (1978b) studied the seasonal variability of several elements in native range grasses from the Northern Great Plains of Montana. Tissue concentrations of Zn and Cu in five range grasses were generally highest in spring samples and declined throughout the summer, fall, and winter. Halvorson and White (1982) found that forage Zn and Cu values declined throughout the growing season for nitrogen fertilized western wheatgrass and green needlegrass. Iron and Mn levels decreased throughout the growing season until maxiumum forage production was attained. Iron then accumulatd in the grasses while Mn concentration remained constant throughout the remainder of the growing season. Ferguson et al. (1943) concluded that forage containing high levels of Mo in the spring have been shown to lose their toxicity when cut as hay or dried in the field. The seasonal variability of trace elements for a wide variety of revegetation species is not well known. If determination of specific element toxicities or deficiencies is necessary, then sampling at various periods of the growing season may be of value.

ANALYSIS OF PLANT TISSUE

Decontamination of Plant Samples

Plant materials from reclaimed areas will always be contaminated with soil particles especially if grazing has occured (Fleming, 1965; Healy et al., 1974). Plants tend to concentrate most elements relative to the soil concentration (Gough and Erdman, 1980). An exception to this rule would be vegetation growing on some uranium overburden and mill tailings. Geological materials associated with uranium ore bodies can be highly enriched with respect to As, Se, and Mo. Shacklette (1965) and Cherney and Robinson (1983) note that elements such as Al, Si, Ti, Fe and Zr are normally found in greater concentations in soil than in tissues and are commonly used to identify soil contamination of plant tissue. As an example, the values with an asterisk (*) in Table 6 show elevated aluminum concentration which is indictive of soil contamination. Two of these plants had been heavily grazed. Prior knowledge of the elemental content of the plants substrate is essential in determining whether soil contamination will bias the plant analysis.

Steyn (1959) showed that washing plant tissue with 0.1 to 0.3% detergent solution was satisfactory in removing soil contamination. A variety of detergents and dilute acids have been evaluated for removing soil contamination prior to plant analysis (Arkley et al., 1960; Baker et al., 1964; Labanauskas, 1968; Ashby, 1969). The effect of washing plant materials of the trace element content aside from soil contamination is not well known. However, if it is suspected that the plant concentration of a given element is elevated well above the soil concentration then washing is probably not necessary.

Plant Digestion Procedures

The two most common digestion procedures for the oxidation and dissolution of plant tissue for element analysis are the dry and wet methods. A controversy exists between which method is the best. The choice is limited by the element being determined since volatile elements (Hg, Cd, As, Se, and Pb) may be lost during dry ashing. Piper (1942) and Johnson and Ulrich (1959) describe wet and dry ashing techniques in detail. Advantages of wet ashing are: 1) low temperature which limits volatilization; 2) speed of oxidation; and 3) less elemental retention because of liquid condition during digestion.

Wet ashing can be accomplished by using a variety of different mixtures of acids including HNO_3, $HClO_4$ and H_2SO_4. Nitric-perchloric acids have been widely used for the digestion of plant tissue (Blanchar et al., 1965; Behan and Kincaide, 1970; Zososki and Boran, 1977). Wet digestion procedures utilizing perchloric acid have two disadvantages: first, $HClO_4$ requires special hoods and very careful handling due to its explosive nature and, secondly, needle-like KC10$_4$ crystals often form during digestion due to high plant potassium concentrations. To overcome these problems a wet digestion procedure using nitric acid was developed by Havlin and Soltanpour (1980) for simultaneous analysis of P, K, Ca, Mg, Na, Zn, Fe, Mn and Cu using ICP-AES. The nitric acid digestion procedure may not completely oxidize organic matter. Any incomplete oxidation of organic matter is overcome due to the high temperature (6000-10,000° K) of the argon plasma (Fassel and Knisley, 1974). More recently this same procedure has been used in assessing boron concentrations in plants (Gestring and Soltanpour, 1981a, 1981b). Distribution of organic matter in plant digests is of primary concern if hydride generation is being utilized to determine As and Se concentration. Wet ashing plant tissue with perchloric acid should ensure that all organics are destroyed prior to hydride generation.

CONCLUSIONS AND RECOMMENDATIONS

1. Elemental analysis of reclaimed vegetation may be appropriate for active and abandoned uranium, coal, and other mines that handle geologic materials containing elevated levels of trace elements.

2. The phenomena of spoil weathering and subsequent release of available forms of trace elements warrants further study.

3. Limited data indicates that grasses can take up dangerous levels of Mo and Se. This may occur on sites not containing the traditional leguminous indicator species. This phenomenon indicates the need to reassess our thinking on accumulator species.

4. Appropriate time of sampling as well as the proper plant part to be sampled needs to be evaluated for a broader range of revegetation species.

5. Additional work needs to be done in developing appropriate plant washing techniques.

6. Further work needs to be done correlating Se, Mo, B, etc. with specific geologic materials.

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