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# Notes on the use of extractable iron and clay minerals for determination of soil age

William C. Mahaney and K. Sanmugadas

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Changes in soil morphologic properties assist in dating and correlating Quaternary deposits in the Rocky Mountain glacial sequence. Determinations of the ratio of acid oxalate to citrate-dithionite extractable Fe (Fe ratio) gives a trend with age that promises to add another relative dating (RD) method to the field of soil stratigraphy.

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Soil morphology has been used to differentiate deposits in the Rocky Mountain Quaternary glacial sequence as described by Mahaney and Fahey (1976), Mahaney (1978), and Mahaney et al. (1984). A number of soil chemical parameters are useful in age differentiation, but most lose their time-dependent character after a few millenia (Mahaney, 1974, 1978). Our research reveals that the ratio of oxalate extractable to dithionite extractable Fe may be used for age differentiation over fairly long periods of time (e.g. several hundred thousand years).

A sequence of Pleistocene glacial deposits along the east flank of Fremont Lake, Wyoming (Figure 1) was chosen for detailed measurements of the Fe ratio (Feo/Fed) in a well drained sequence, from youngest to oldest, of an Inceptisol, and two Alfisols. The Inceptisol, tentatively classified as a Dystric Cryochrept, (post-Pinedale soil), is 15,000 yrs. BP; the youngest Alfisol, tentatively classified as a Typic Cryoboralf (post-Bull Lake soil), is older than 100,000 yrs. BP; and the oldest Alfisol, tentatively classified as a Typic Eutroboralf (pre-Bull Lake soil), is probably 0.5 million yrs. BP or older (Mahaney et al., 1982). Climatic details are unknown, but all sites are located in the montane forest of Douglas fir (Pseudotsuga menziesii) and aspen Populus tremuloides). Deposits have been mapped by Richmond (1974) and Mahaney et al. (1984), and samples were collected at sites shown on Figure 2. Sample

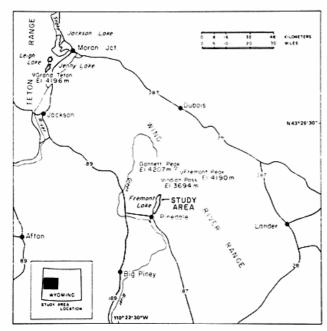


Fig. 1. Map of western Wyoming showing the location of the study area near Fremont Lake in the Wind River Mountains.

sites (FL7 and FL8, Figure 2) are located in deposit crests in order to exclude topographic effects and the influence of soil erosion, as well as to maximize the degree to which air-fall materials (loess) might be seen in the profiles. Site FL9 (Figure 2) is at the bottom of a slope below Fortification Mountain, and has been subjected to input of material either by slope wash or Aeolian processes.

# Deposit Chronology

Mahaney et al. (1984) and Richmond (1974) have established that these deposits, consisting mainly of granodioritic clasts, were built up by Quaternary glaciations. The oldest pre-Bull Lake soil contains argillic horizons considerably finer in texture than B horizons in the post-Bull Lake and post-Pinedale profiles. Many soil characteristics, especially increases in clay size materials, loess cover, thickness, percent Fe<sub>2</sub>O<sub>3</sub>, and clay mineral composition are all important age differentiation criteria.

### METHODS

Soil descriptions follow the Soil Survey Staff (1951, 1975) and Birkeland (1974). Particle size analysis is based on the Wentworth scale as defined by Folk (1968); coarse grade sizes (2mm-63µm) were separated by dry sieving (Day, 1965) and fine grade sizes ( $<63\mu m$ ) were calculated by sedimentation (Bouyoucos, 1962). Soil and sediment samples were dispersed in a solution of sodium pyrophosphate and clay plus silt were separated by sedimentation. Following procedures outlined by Whittig (1965) a clay

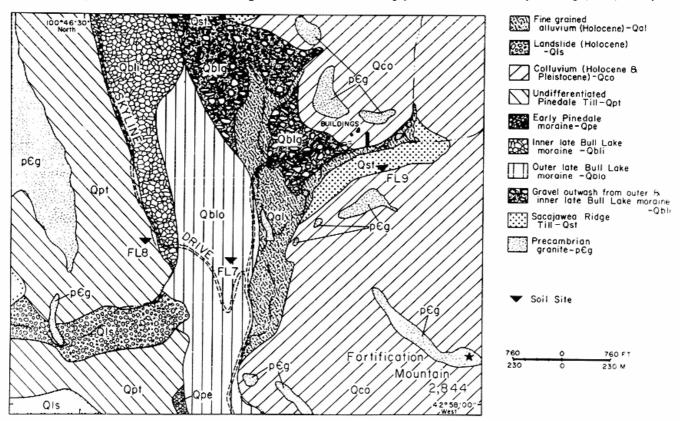


Fig. 2. Map of surficial deposits and location of sites FL7 (post-Bull Lake soil), FL8 (post-Pinedale soil) and FL9 (pre-Bull Lake soil).

Table 1. Physicala, chemical and mineral properties of post-Pinedale soils in montane and sagebrush steppe vegetation zones, Wind River Mountains, Wyoming.

| Site | Age and<br>Parent<br>Material | Elevation<br>(m) | Vegetation | Soil<br>Horizon | Depth<br>(cm) | %<br>Sand<br>(2mm-63µm) |      | g<br>Clay<br>(<4µm) | pH<br>(1:1) |     | Clay Minerals<br>(< 2µm) |     |     |     |     |    |  |
|------|-------------------------------|------------------|------------|-----------------|---------------|-------------------------|------|---------------------|-------------|-----|--------------------------|-----|-----|-----|-----|----|--|
|      |                               |                  |            |                 |               |                         |      |                     |             | К   | н                        | ı   | S   | M×  | ν   | С  |  |
| L8   | Pinedale                      | 2450             | Montane    | A11             | 0-6           | 43.0                    | 29.0 | 28.0                | 6.2         | -   | -                        | tr  | -   | -   | -   | -  |  |
|      | +111                          |                  | Forest     | A12             | 6-22          | 52.5                    | 34.0 | 13.5                | 6.4         | ×   | -                        | ××  | -   | -   | -   | -  |  |
|      |                               |                  |            | B21             | 22-28         | 55.9                    | 32.1 | 12.0                | 6.4         | ×   | -                        | ××  | -   | -   | tr  | -  |  |
|      |                               |                  |            | 11822           | 28-38         | 63.9                    | 27.1 | 9.0                 | 6.4         | tr  | -                        | tr  | -   | tr  | -   | -  |  |
|      |                               |                  |            | IICox           | 38-102        | 65.8                    | 25.2 | 9.0                 | 6.3         | ?   | -                        | ×   | -   | -   | ?   | -  |  |
|      |                               |                  |            | I ICn           | 102+          | 67.9                    | 25.1 | 7.0                 | 6.3         | ?   | -                        | tr  | -   | -   | -   | -  |  |
| L7   | Bull Lake                     | 2445             | Montane    | A2              | 0-5           | 55.9                    | 35.6 | 8.5                 | 5.3         | tr  | -                        | -   | tr  | -   | -   | -  |  |
|      | 1111                          |                  | Forest     | 821t            | 5-23          | 56.4                    | 32.6 | 11.0                | 5.9         | tr  | -                        | -   | ×   | ×   | -   | +- |  |
|      |                               |                  |            | 822ir           | 23-54         | 61.7                    | 28.8 | 9.5                 | 5.9         | -   | -                        | -   | -   | -   | -   | -  |  |
|      |                               |                  |            | Clox            | 54-87         | 58.4                    | 34.6 | 7.0                 | 6.5         | -   | -                        | -   | ×   | tr  | -   | -  |  |
|      |                               |                  |            | 11C2ox          | 87-119        | 67.2                    | 25.8 | 7.0                 | 6.6         | -   | -                        | -   | tr  | tr  | -   | tr |  |
|      |                               |                  |            | IIC3ox          | 119-193       | 64.4                    | 26.6 | 9.0                 | 7.3         | -   | -                        | -   | xxx | ××  | -   | XX |  |
|      |                               |                  |            | IICn            | 193+          | 68.8                    | 24.2 | 7.0                 | 7.3         | -   | -                        | -   | ×   | ×   | -   | ×  |  |
| L9   | pre-Bull                      | 2400             | Montane    | A1              | 0-4           | 44.6                    | 37.9 | 17.5                | 5.7         | tr  | -                        | ×   | -   | ×   | ×   | -  |  |
|      | Lake till                     |                  | Forest     | 821†            | 4-10          | 44.0                    | 34.0 | 22.0                | 5.5         | ×   | -                        | ×   | -   | ×   | ×   | ×  |  |
|      |                               |                  |            | B22†            | 10-36         | 43.4                    | 22.1 | 34.5                | 5.6         | xx  | -                        | xxx | xxx | xxx | -   | хх |  |
|      |                               |                  |            | 823t            | 36-60         | 48.2                    | 22.8 | 29.0                | 5.5         | ×   | -                        | ××× | ××× | ××× | xxx | -  |  |
|      |                               |                  |            | B24†            | 60-95         | 50.0                    | 25.5 | 24.5                | 5.7         | ××  | ×                        | xxx | ××× | xxx | -   | -  |  |
|      |                               |                  |            | 11825†          | 95-122        | 63.6                    | 14.4 | 22.0                | 6.6         | xxx | -                        | ××× | ××× | xxx | -   | -  |  |
|      |                               |                  |            | 11826†          | 122-158       | 61.9                    | 10.1 | 28.0                | 5.7         | ×   | -                        | xxx | ××× | xxx | -   | -  |  |
|      |                               |                  |            | IIC1ox          | 158-175       | 73.5                    | 10.5 | 16.0                | 6.0         | ×   | -                        | ××  | xxx | ××  | -   | -  |  |
|      |                               |                  |            | IIC2ox          | 175-200       | 77.4                    | 13.6 | 9.0                 | 5.7         | -   | -                        | xxx | xxx | xxx | -   | -  |  |
|      |                               |                  |            | IICnm           | 200+          | 80.2                    | 10.8 | 9.0                 | 6.9         | tr  | -                        | xxx | xxx | xxx | -   | -  |  |

<sup>&</sup>lt;sup>a</sup>Data are given in weight-percentages of sand, silt and clay (<2mm). Coarse particle sizes (2000-63µm) determined by sieving; fine particle sizes (63-1.95µm) determined by hydrometer.

paste was oriented on a ceramic tile by centrifugation, air-dried, and X-rayed using a Toshiba ADG-301H diffractometer with Ni-filtered Cukaradiation.

# Oxalate Extractable Fe

The procedure adopted for the determination of oxalate extractable iron is that of McKeague and Day (1966). The

soil sample was ground to pass a 100 mesh sieve and shaken with acid ammonium oxalate of pH 3.0 for four hours in the dark. The soil suspension was centrifuged and iron in the clear centrifugate was estimated by atomic absorption spectroscopy using a Perkin Elmer Model 373 AA Spectrophotometer. Standards of a similar matrix were used and the standards as well as the samples contained 2000 ppm sodium to suppress interferences.

bMineral abundance is based on peak height: nil (-); minor amount (tr); small amount (x); moderate amount (xx); abundant (xxx). Clay minerals are kaolinite (K), halloysite (H), illite (I), smectite (S), vermiculite (V), mixedlayer illite-smective (Mx) and chlorite (C).

Table 2. Soil Color and iron in the pyrophosphate, citrate-dithionite, and acid oxalate extracts for sites in the Wind River Range.

| Site | Age      | Horizon | Depth<br>(cm) | Hue   |                   | Fe Ratio |                                     |  |                                       |                                     |
|------|----------|---------|---------------|-------|-------------------|----------|-------------------------------------|--|---------------------------------------|-------------------------------------|
| 3,10 |          |         |               |       | value/ch<br>moist | dry      | Pyrophosphate<br>(Fe <sub>p</sub> ) | Citrate-<br>Dithionite<br>(Fe <sub>d</sub> ) | Acid<br>Oxalate<br>(Fe <sub>0</sub> ) | (Fe <sub>o</sub> /Fe <sub>d</sub> ) |
| FL8  | post-    | A11     | 0-6           | 10YR  | 2/3               | 3/2,4/2  | 0.16                                | 1.05   | 0.52                                  | 0.50                                |
|      | Pinedale | A12     | 6-22          | 10YR  | 3/2               | 4/2      | 0.24                                | 1.35   | 0.47                                  | 0.35                                |
|      |          | 821     | 22-28         | 10YR  | 4/3               | 4/3      | 0.22                                | 1.36   | 0.44                                  | 0.32                                |
|      |          | 11822   | 28-38         | 10YR  | 5/3               | 6/3      | 0.12                                | 1.34   | 0.63                                  | 0.47                                |
|      |          | HCox    | 28-102        | 10YR  | 6/3               | 7/3      | 0.09                                | 1.07   | 0.77                                  | 0.72                                |
|      |          | HCn     | 102+          | 2.5Y  | 5/3               | 8/3      | 0.07                                | 1.04   | 0.72                                  | 7 0.69                              |
| FL7  | post-    | A2      | 0-5           | 10YR  | 5/2               | 6/2      | 0.20                                | 1.12   | 0.82                                  | 0.73                                |
|      | Bull     | B21†    | 5-23          | 10YR  | 4/4               | 7/3      | 0.16                                | 1.18   | 0.96                                  | 0.81                                |
|      | Lake     | B22     | 23-54         | 10YR  | 6/4,6/3           | 8/2,7/2  | 0.09                                | 0.97   | 0.77                                  | 0.79                                |
|      |          | Clox    | 54-87         | 10YR  | 5/3,5/4,6/3       | 8/2      | 0.06                                | 0.87   | 0.69                                  | 0.79                                |
|      |          | IIC2ox  | 87-119        | 10YR  | 5/3               | 8/3      | 0.05                                | 0.83   | 0.69                                  | 0.83                                |
|      |          | 1103ox  | 119-193       | 10YR  | 5/4,5/6,6/4       | 8/4      | 0.06                                | 0.91   | 0.64                                  | 0.70                                |
|      |          | IICn    | 193+          | 2.5Y  | 6/4               | 8/3      | 0.06                                | 0.88   | 0.66                                  | 0.75                                |
| FL9  | pre-     | A1      | 0-4           | 10YR  | 2/2,2/3           | 4/2      | 0.42                                | . 1.70                                       | 0.82                                  | 0.48                                |
|      | Bull     | B21†    | 4-10          | 10YR  | 5/4               | 5/3      | 0.32                                | 0.81   | 0.71                                  | 0.88                                |
|      | Lake     | B22+    | 10-36         | IOYR  | 6/4               | 7/3      | 0.07                                | 1.83   | 0,43                                  | 0.23                                |
|      |          | 823†    | 36-60         | 10YR  | 6/4               | 7/4      | 0.08                                | 2.69   | 0.63                                  | 0.23                                |
|      |          | 824†    | 60-95         | 10YR  | 5/4               | 6/4      | 0.18                                | 1.86   | 0.77                                  | 0.41                                |
|      |          | 11825+  | 95-122        | 7.5YR | 4/4               | 6/4      | 0.13                                | 4.58   | 0.70                                  | 0.15                                |
|      |          | 11826†  | 122-158       | 10YR  | 4/4               | 5/4,6/6  | 0.12                                | 5.98   | 0.37                                  | 0.06                                |
|      |          | 11Clox  | 158-175       | 10YR  | 5/4               | 6/4      | 0.09                                | 3.18   | 0.58                                  | 0.18                                |
|      |          | HC2ox   | 175-200       | 7.5YR | 5/6,4/4           | 6/4,7/4  | 0.10                                | 2.30   | 0.45                                  | 0.20                                |
|      |          | 11Cnm   | 200+          | 10YR  | 8/1               |          | 0.08                                | 3.24   | 0.36                                  | 0.11                                |
|      |          |         |               | 2.5Y  | 7/3               | 6/4,7/4  |                                     |  |                                       |                                     |

#### Dithionite Extractable Fe

The extraction procedure used is that of Coffin (1963). One gram of the soil sample (<2mm) was shaken with 2 g sodium dithionite and 40 ml citrate buffer (sodium citrate and citric acid) for a period of 16 hours. The iron in the clear filtrate was determined by atomic absorption spectroscopy using appropriate standards.

### Sodium Phyrophosphate Extractable Iron

Following the procedure of McKeague (1976) 30 ml of 0.1M sodium pyrophosphate was added to 0.300 g of soil and ground to pass a 100 mesh sieve. The suspension was shaken overnight. Thereafter, 0.5 ml of 0.1 percent superfloc was added to the suspension and centrifuged at 1500 rpm for 10 minutes. The clear centrifugate was analyzed for Fe by atomic absorption spectroscopy using appropriate standards.

## RESULTS AND DISCUSSION

As shown in Table 1 soil depth increases with time, especially between the post-Pinedale and post-Bull Lake pedons. Further age differentiation between soils is achieved using loess thickness which increases from 28 cm in FL8 to 87 cm in FL7. The high silt content in the surface of FL9 (upper 95 cm) may have been emplaced by aeolian or slope wash processes. While the weight-percentages of silt do not show any time-dependent relationship, the overall

thickness in the profiles is attributed primarily to airfall influx. The lower percentages of silt in the lower B horizons of the oldest profile (FL9) suggest that silt in older soils may weather to clay or to solutes. The higher percentages of clay in the sola of the three profiles may also result partly from airfall influx and partly from in situ weathering processes. Soil pH values are uniform throughout the younger post-Pinedale soil, and they tend to increase slightly with depth in the older post-Bull Lake and pre-Bull Lake soils, suggesting little movement of H+ ions downward in the profiles.

The mineralogical composition (Table 1) of the three profiles suggests that clay minerals, especially relative amounts of kaolinite and smectite, provide an important means of differentiating soils in this stratigraphic succession. The absence of smectite and trace amounts of mixed-layer lattices in the post-Pinedale soil contrast sharply with the moderate to large amounts found in the post-Bull Lake and pre-Bull Lake soils. Similarly, kaolinite which is located randomly in small to trace amounts in the younger profiles increases to abundant proportions in the 11B25t horizon of the pre-Bull Lake soil, a trend reported elsewhere in the Rocky Mountains (Mahaney and Fahey, 1980). The decrease in illite between post-Pinedale and post-Bull Lake soils suggests it is weathering to form smectite, mixed hyphenate layer lattices or chlorite. The high illite content in the pre-Bull Lake soil may be a result of input by slope wash processes.

As shown in Table 2 organically-bound Fe (pyrophosphate extractable Fe<sub>p</sub>) has somewhat similar concentrations and distributions in the two youngest profiles (FL8 and 7), increasing in the lower solum of the older soil (FL9). The data indicate that organically complexed Fe increases slightly with greater age, and tends to become dispersed through the profiles to considerable depth.

Oxalate extractable Fe (Fe<sub>o</sub>) forms slowly reaching the highest values in the post-Bull Lake soil (nearly 1.0%), then decreasing, especially in the lower subsoil of the oldest profile. This trend suggests that over time more and more amorphous iron may be converted to crystalline Fe<sub>2</sub>O<sub>3</sub>.

Dithionite extractable Fe (Fe<sub>d</sub>) decreases from a maximum of 1.36% in the post-Pinedale soil to 1.18% in the post-Bull Lake soil, and rises dramatically to nearly 6.0% in the pre-Bull Lake soil. The slight drop in the post-Bull Lake soil may be due to lower overall clay values (Table 1) which may reflect variations in aeolian influx with FL8 receiving more airfall input (it is the first ridge to encounter air masses moving across Fremont Lake). There are no known lithologic or biotic factors that could account for the drop in dithionite extractable iron in the post-Bull Lake soil.

Comparison of the soil colors in each profile with the dithionite extractable Fe (Fe<sub>d</sub>) are given in Table 2. The data show that over time soils tend to become redder as

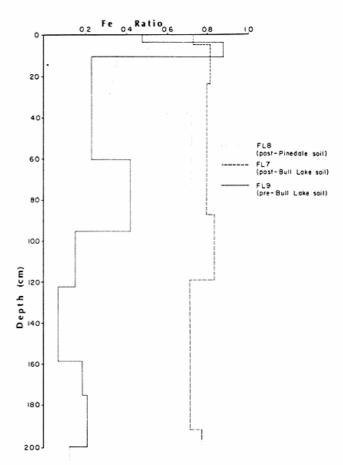


Fig. 3. Fe ratio (Fe<sub>o</sub>/Fe<sub>d</sub>) distribution with depth in the Fremont Lake soil chronosequence.

total Fe<sub>2</sub>O<sub>3</sub> increases. This relationship is most apparent in the FL9 profile (pre-Bull Lake soil) where 10YR hues strengthen to 7.5YR in the lower solum. That high dithionite extractable Fe (Fe<sub>d</sub>) is not always accompanied by reddish hues, however, is shown in the llCnm horizon of FL9. Here total Fe<sub>2</sub>O<sub>3</sub> reaches 3.24, whereas color is light gray (10YR 8/1m) and light yellow (2.5Y 7/3m). Clearly changes in soil color provide only a general indication of the development of Fe oxides. Moreover, Fe<sub>d</sub> values for post-Pinedale and post-Bull Lake soils reported herein compare favorably with values obtained in other areas of the Rocky Mountains (Mahaney 1978; Mahaney and Fahey, 1976; and Mahaney et al. 1984).

The data in Figure 3 show the distribution of the Fe ratio with depth in the three profiles. Initially it is quite variable, increasing in the subsoil as oxalate extractable Fe exceeds dithionite Fe development. In the post-Bull Lake soil the ratio is more uniformly distributed with depth. The pre-Bull Lake soil undergoes an overall reduction in the ratio, especially in the IlB26t horizon where it falls to <0.10. Here the rate of production of dithionite extractable Fe far exceeds the rate of development of oxalate extractable Fe.

The data herein support the findings of Alexander (1974) who reported on variations in the Fe ratio in a sequence of Quaternary soils formed in stream deposits along the east flank of the Sierra Nevada, California. While his ratios were somewhat lower, possibly due to lower rainfall, the overall trend is similar. Here, as in this study, the ratio rises slowly over time, and then falls. Clearly the Fe ratio is an important age indicator, especially for Pleistocene soil sequences. The degree to which the Fe ratio might be an important age indicator for younger Holocene soil sequences in the Rocky Mountain alpine zone is presently under investigation in our laboratory.

#### CONCLUSIONS

The Fe ratio (oxalate extractable to dithionite extractable Fe) in well-drained soils across a sequence of glacial deposits increases over the first hundred thousand years, and then decreases. The Fe ratio will not stand alone as an age indicator, but as noted by Alexander (1974) it provides a useful quantitative supplement to other soil morphologic criteria useful in defining soil stratigraphic units. Changes in the clay mineral composition of the three profiles suggest that weathering over time produces clay mineral assemblages that are useful in differentiating soils in a stratigraphic succession.

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#### Resumé

Changes in soil morphologic properties assist in dating and correlating Quaternary deposits in the Rocky Mountain glacial sequence. Determinations of the ratio of acid oxalate to citrate-dithionite extractable Fe (Fe ratio) gives a trend with age that promises to add another relative dating (RD) method to the field of soil stratigraphy. Iron extractions from the horizons of a sequence of an Inceptisol → Alfisols in glacial deposits in the Wind River Range, Wyoming, were made to determine relative amounts of oxalate and dithionite extractable iron. The data show that dithionite extractable iron increases slowly from the post-Pinedale soil reaching nearly 6% in the pre-Bull Lake soil (0.5) million years old). Oxalate extractable iron develops more slowly in the early stages of soil development, slowing after 100,000 years. The Fe ratio appears useful in differentiating mid-to-late Pleistocene soils, where it

increases in the B and C horizons for the first hundred thousand years, and then declines. Differences in clay mineral content between the three pedons indicate that relative amounts of 1:1 and 2:1 clays can be used to distinguish soils of different age.

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The data herein support the findings of Alexander (1974) who reported on variations in the Fe ratio in a sequence of Quaternary soils formed in stream deposits along the east flank of the Sierra Nevada, California. While his ratios were somewhat lower, possibly due to lower rainfall, the overall trend is similar. Here, as in this study, the ratio rises slowly over time, and then falls. Clearly the Fe ratio is an important age indicator, especially for Pleistocene soil sequences. The degree to which the Fe ratio might be an important age indicator for younger Holocene soil sequences in the Rocky Mountain alpine zone is presently under investigation in our laboratory.

#### CONCLUSIONS

The Fe ratio (oxalate extractable to dithionite extractable Fe) in well-drained soils across a sequence of glacial deposits increases over the first hundred thousand years, and then decreases. The Fe ratio will not stand alone as an age indicator, but as noted by Alexander (1974) it provides a useful quantitative supplement to other soil morphologic criteria useful in defining soil stratigraphic units. Changes in the clay mineral composition of the three profiles suggest that weathering over time produces clay mineral assemblages that are useful in differentiating soils in a stratigraphic succession.

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#### Resumé

Changes in soil morphologic properties assist in dating and correlating Quaternary deposits in the Rocky Mountain glacial sequence. Determinations of the ratio of acid oxalate to citrate-dithionite extractable Fe (Fe ratio) gives a trend with age that promises to add another relative dating (RD) method to the field of soil stratigraphy. Iron extractions from the horizons of a sequence of an Inceptisol → Alfisols in glacial deposits in the Wind River Range, Wyoming, were made to determine relative amounts of oxalate and dithionite extractable iron. The data show that dithionite extractable iron increases slowly from the post-Pinedale soil reaching nearly 6% in the pre-Bull Lake soil (0.5) million years old). Oxalate extractable iron develops more slowly in the early stages of soil development, slowing after 100,000 years. The Fe ratio appears useful in differentiating mid-to-late Pleistocene soils, where it

increases in the B and C horizons for the first hundred thousand years, and then declines. Differences in clay mineral content between the three pedons indicate that relative amounts of 1:1 and 2:1 clays can be used to distinguish soils of different age.

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