CORRECTION FOR LOSS OF METALLIC IRON IN "FUSED-BEAD" ANALYSIS OF METAL-BEARING SILICATE SAMPLES; Randy L. Korotev, Dept. of Earth & Planetary Sciences and the McDonnell Center for the Space Sciences, Washington University, St. Louis, MO, 63130.

Two common methods for determining the bulk chemical composition of small geologic samples are instrumental neutron activation analysis (INAA), which provides data primarily for trace elements but also for Fe, and electron microprobe analysis of fused powders (FBA = "fused bead" analysis), which is used to determine major element concentrations [1]. Occasionally both analytical methods are applied to the same samples and Fe concentrations are obtained by both techniques. For samples that contain a substantial quantity of metallic iron, such as Apollo 16 impact melt rocks and soils [2,3], results obtained by the two methods may disagree substantially because INAA determines total iron whereas FBA determines only the oxidized iron contained in the silicate glass [1]. Presumably, the metal either segregates and settles within the mostly-silicate bead or is extracted into the Mo-foil strip used to heat and support the sample. The results of Ryder and Seymour [4] for a suite of thirteen Apollo 16 impact melt rocks demonstrate the problem. FeO concentrations obtained by FBA are lower than those obtained by INAA for all samples except one by 1.1% FeO on the average, i.e., about 16% of the total FeO in the average sample (Fig. 1a).

A simple test shows that this disagreement results from the metallic Fe contained in the samples. In Apollo 16 impact melt rocks the metallic Fe is carried by Fe-Ni metal with Fe/Ni and Fe/Co ratios of about 16 and 280 [3]. The concentration of Fe contributed by the Fe-Ni metal can be estimated from the Ni or Co concentration obtained by INAA. Ni has the advantage that a much smaller fraction of the total concentration in the sample is contained in the nonmetal phases, but it is not as precisely determined by INAA as is Co and no values for Ni are given by Ryder & Seymour [2]. The Co concentration of the silicate phases can be estimated from the Sc concentration obtained by INAA. The Sc/Co ratio in the silicate portion of the melt found in Apollo 16 dimict breccias is 0.97 ([3], Table 4). The dimict-breccia melt is a typical type of impact melt and the Sc/Co ratio in other types of Apollo 16 impact melt rocks are unlikely to be substantially different unless the pyroxene/olivine ratio differs significantly. Subtracting the concentration of Co contained in the silicates from the total concentration obtained by INAA gives the concentration carried by the metal phase. Multiplying this by the Fe/Co ratio in the metal gives the Fe concentration carried by the metal phase. In Fig. 1b, this concentration (converted to FeO) has been added to the concentration of the silicate glass determined by FBA to give an estimated total FeO concentration. For all but two samples, the agreement with the INAA results is good. These two are the samples with the lowest and highest Fe concentrations and include the one sample for which the INAA value was less than FBA value.

In metal-bearing samples, INAA and FBA determine different quantities. For some applications, e.g., comparing Mg/Fe ratios of mafic silicates [4, Fig. 2], the Fe concentration determined by FBA may be preferred. When information about the metal phase is needed [2,3], the total Fe concentration obtained by INAA is useful.

REFERENCES

Fig. 1. FeO concentrations in Apollo 16 impact melt rocks [4]. a) Data (raw) obtained by the electron microprobe (EMP) on fused beads against that obtained by instrumental neutron activation analysis (INAA). The data plot below the diagonal (1:1) line because the Fe in the metal portion of the samples is not detected by the EMP. b) Estimated total FeO concentrations obtained by summing the concentration in the fused bead with the estimate (based on the Sc and Co) of the concentration carried by the metal.