Recycling of mining waste has become more important since global market prizes of metals have increased. Optimization of extraction procedures needs careful determination of elemental amounts in the waste, the extracts and the residue. Inductively-coupled plasma optical emission spectrometry (ICP-OES) and (total-reflection X-ray fluorescence spectrometry) TXRF were used to determine metals leached by sulfuric acid extraction. Comparison showed good agreement of both methods. Nonetheless, efficiency of continuous extraction is ideally determined by loss of metals in the solid residue comparing it to the original content. A determination directly from the solid sample is favorable to ensure a quick determination and prevent from digestion procedures involving concentrated HF. Here we present the optimization of determination of elements in suspensions of mining waste with TXRF. Special focus was on the determination of Si, because if Si can be determined probably the leaching of other elements can be determined by their decreased relative content to Si, assuming Si is not leached to the extract. The optimization was done by using acrylic glass reflectors (usually quartz reflectors are used) and using different certified reference material. The results show, determination of Si can be successfully done using P as internal standard.