X-ray fluorescence (XRF) is an established technology for elemental analysis of polyolefins. XRF is a matrix dependent analysis and accurate results require standards of similar matrices. Although the preparation of matrix-matched standards requires extensive effort and resources, characterized materials are needed for routine analysis of additives in polyolefins. The preparation of a large quantity (~1 Kg) of additive blends in a base resin using V-blending and triple extrusion will be described. After initial evaluation of homogeneity, the characterization of the blends was done using independent techniques: neutron activation analysis, inductively coupled plasma atomic emission, elemental combustion and total dissolution liquid chromatography. Finally, the protocol for evaluating molded plaques for XRF calibration will be discussed.