The objectives of this study were to determine volatile organic compound (VOC) emission signatures of nine typical building materials by using proton transfer reaction-mass spectrometry (PTR-MS) and to explore the correlation between the PTR-MS measurements and the measurements of acceptability by human subjects. VOC emissions from each material were measured in a 50-l small-scale chamber. Chamber air was sampled by PTR-MS to determine emission signatures. Sorbent tube sampling and TD-GC/MS analysis were also performed to identify the major VOCs emitted and to compare the resulting data with the PTR-MS emission signatures. The data on the acceptability of air quality assessed by human subjects were obtained from a previous experimental study in which the emissions from the same batch of materials were determined under the same area-specific ventilation rates as in the case of the measurements with PTR-MS. Results show that PTR-MS can be an effective tool for establishing VOC emission signatures of material types and that there were reasonable correlations between the PTR-MS measurements and the acceptability of air quality for the nine materials tested when the sum of selected major individual VOC odor indices was used to represent the emission level measured by PTR-MS.