American Mineralogist, Volume 96, pages 1028–1038, 2011

Magnesium quantification in calcites [$(Ca,Mg)CO_3$] by Rietveld-based XRD analysis: Revisiting a well-established method

Jürgen Titschack,[1,*] Friedlinde Goetz-Neunhoeffer,[2] and Jürgen Neubauer[2]

[1]MARUM-Center for Marine Environmental Sciences, Loebener Strasse, D-28359 Bremen, Germany; Senckenberg am Meer, Abteilung für Meeresforschung, Südstrand 40, D-26382 Wilhelmshaven, Germany
[2]GeoZentrum Nordbayern, Mineralogie, Friedrich-Alexander-Universität Erlangen-Nürnberg, Schlossgarten 5A, D-91054, Erlangen, Germany

Abstract

Mg-calcites commonly occur in natural environments, with Mg-contents ranging between about 0 and 32 mol% (referring to $MgCO_3$). Often, different Mg-calcite phases occur within the same sample. The Mg-content in calcites permits the classification of the diagenetic environment (marine vs. meteoric), or the reconstruction of paleotemperatures from skeletal remains. Since the 1960s, there have been published various calibrations for Mg determination in calcites based on XRD measurements. Recently, this method has come to be superseded by wet chemical, laser ablation, and microprobe analysis, due to their higher accuracy and/or higher sample resolution of these latter methods.

This study presents a new calibration for the Mg determination in calcites using XRD measurements analyzed by means of the Rietveld refinement method. The calibration is based on lattice parameters and exhibits a reliable Mg-determination accuracy of more than 0.8 mol% between 0 and 15.5 mol%. The incorporation of the calibration into the Rietveld refinement software TOPAS permits a fast, standardized workflow. This, in turn, enhances the user-friendliness and the reproducibility of results, as well as allows the simultaneous analysis of multiple Mg-calcites in a sample. Mixtures of two Mg-calcites were analyzed using this new method. The Mg-content of two co-occurring Mg-calcite phases can be reliably quantified providing the Mg-calcites differ by at least 4.9 mol% and the minor Mg-calcite phase makes up more than 2.7 wt%. If the Mg-calcite phases differ by 3.4 mol% or less, the reliable identification of different Mg-calcite phases is questionable, due to the fact that differences involved here are too small. If such XRD patterns are refined with only one Mg-calcite phase, then the systematic misfit between measured and refined pattern can be used for the identification of a second Mg-calcite phase down to a difference in the Mg-content of about 3 mol%. However, a reliable phase quantification for these patterns cannot be achieved.

Keywords: Magnesium calcite, XRD, Rietveld refinement, calibration, phase quantification