## <sup>57</sup>Fe Mössbauer study of the oxidation state of iron in stilpnomelane from granite pegmatites in Poland

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## ABSTRACT

The effects of heat treatment on stilpnomelane from granite pegmatites at Zółkiewka (Lower Silesia, Poland) were studied using 57Fe Mössbauer spectroscopy. Samples of naturally altered stilpnomelane and chlorite were also studied. The Mössbauer spectrum of an untreated sample was fitted to two  $Fe^{2+}$ doublets and two  $Fe^{3+}$  doublets. The  $Fe^{2+}$  doublets had similar isomer shifts (1.13 and 1.16 mm/s for the octahedral M1 and M2 sites), different quadrupole splitting values (QS = 2.28 and 2.66 mm/s, respectively), and an M1:M2 area ratio of nearly 1:3. For ferric iron, the first doublet was assigned to the combined M1 + M2 + M4 octahedral positions and had IS = 0.41 and QS = 1.18 mm/s. The second doublet was assigned to the M3 site, with parameters IS = 0.36 and QS = 2.07 mm/s. This is characteristic for stilpnomelane. The oxidation of  $Fe^{2+}$  is fast at low temperatures, with a maximum at 360 °C. Near 1050 °C, the stilpnomelane structure broke down completely, and the spectrum consisted of two sextets and two ferric iron doublets. The predominant phase in this new material is hematite, as represented by the two sextets. The abnormally high fractional  $Fe^{2+}$  content in the altered stilpnomelane (0.70) as compared to the untreated sample (0.45) indicates that the sample was subjected to local hydrothermal processes. Nearly identical divalent iron contents and hyperfine parameters for the  $Fe^{2+}$ and  $Fe^{3+}$  doublets (except  $Fe^{3+}$  in M3) in the altered stilpnomelane and associated chlorite strongly suggest that chlorite is transformed into stilpnomelane.

Keywords: Stilpnomelane, Mössbauer spectroscopy, iron, oxidation state, heat treated