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# Single-crystal X-ray and Raman investigation on melanophlogite from Varano Marchesi (Parma, Italy)

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## Abstract

The results of a new single-crystal structure refinement and of a Raman spectroscopy investigation on melanophlogite, a clathrate structure of $SiO_2$, are reported. The studied sample comes from a new finding at Varano Marchesi (Parma, Italy), and occurs in small veins and pockets along fractures in a siliceous marl from a chaotic complex. Melanophlogite is invariably separated from the host rock by a thin crust of opal-CT.

Raman spectroscopy was done to investigate the guest molecules that are hosted in the cages of the structure. In the Varano Marchesi melanophlogite, only $CH_4$ is present in the clathrate structure. During a comparative investigation of melanophlogite from different geological setting (Racalmuto, Sicily, Italy), $H_2S$ also was found, together with $CH_4$, in the cavities of the structure.

A single-crystal X-ray refinement of the Varano Marchesi sample was done using the $Pm\bar{3}n$ symmetry of β-melanophlogite [$a$ = 13.399(2), $R_{4\sigma}$ = 4.7%]. According to the site refinement from X-ray diffraction results, $CH_4$ occupies 71 and 91% of the $5^{12}$ and $5^{12}6^2$ site cavities, respectively.

The Varano Marchesi melanophlogite formed as a result of low-temperature hydrothermal activity. The mineral growth occurred at the expense of opal, in connection with $CH_4$ flux through the porous material.

**Keywords:** Melanophlogite, single-crystal X-ray diffraction, Raman spectroscopy, phase transition, $CH_4$