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# Crystal structure of lead uranyl carbonate mineral widenmannite: Precession electron-diffraction and synchrotron powder-diffraction study†

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## ABSTRACT

The crystal structure of the lead uranyl-carbonate mineral widenmannite has been solved from precession electron-diffraction data and refined using both electron-diffraction data and synchrotron powder-diffraction data. Widenmannite is orthorhombic, *Pmmn*, with $a = 4.9744(9)$, $b = 9.3816(16)$, $c = 8.9539(15)$ Å, and $V = 417.86(12)$ Å$^3$. The structure was solved by charge-flipping and refined to an $R_1 = 0.1911$ on the basis of 301 unique, observed reflections from electron diffraction data, and to $R_p$ of 0.0253 and $R_F$ of 0.0164 from X-ray powder data. The idealized structure formula of widenmannite is $Pb_2(OH)_2[(UO_2)(CO_3)_2]$, $Z = 2$. However, both data sets suggest that the widenmmanite structure is not that simple. There are two symmetrically independent, partly occupied U sites. The substitution mechanism can be written as $U(1)O_2 + Pb(OH)_2 \leftrightarrow U(2)O_2$. When the U(2) site is occupied, the U(1) $O_2$ group is absent, the two OH groups are substituted by $O^{2-}$ and one $Pb^{2+}$-vacancy. The chemical formula of the real structure should be written as $Pb_{2-x}(OH)_{2-2x}[(UO_2)(CO_3)_2]$, where $x$ is the probability of the substitution U(2) → U(1). The probability of occurrence of U(2) refines to $x = 0.074(15)$ from the powder-diffraction data and to $x = 0.176(4)$ from the electron-diffraction data. There is one Pb site (nearly fully occupied), which is coordinated by 11 anions (up to the distance of 3.5 Å), including O and $OH^-$. The shorter Pb-O bonds form a sheet structure, which is linked by the weaker bonds to the uranyl-carbonate chains to form a three-dimensional framework structure.

**Keywords:** Widenmannite, uranyl bicarbonate, crystal structure, precession electron diffraction, synchrotron powder diffraction