

# Preparation and Crystal Structure of the Calcium Rhenate(VI, VII)



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The title compound was prepared by reaction of elemental calcium with the calcium metaperhenate  $\text{Ca}(\text{ReO}_4)_2$ . Its crystal structure was determined from single-crystal X-ray data: *Amm*2,  $a = 560.31(5)$  pm,  $b = 1572.4(1)$  pm,  $c = 719.91(6)$  pm,  $Z = 2$ ,  $R = 0.033$  for 930 structure factors and 46 variable parameters. The calcium atoms occupy three atomic sites, all with seven oxygen neighbors. Of the two different rhenium atoms one has square-pyramidal oxygen coordination with an average oxidation number +6.25. The other rhenium site (oxidation number +7) was refined as a split position with trigonal-bipyramidal (75%) and tetrahedral oxygen coordination (25%). One oxygen site remains unoccupied, whenever the tetrahedral rhenium site is occupied, resulting in the composition  $\text{Ca}_5\text{Re}_3\text{O}_{14.75}$ . A test for superconductivity of this black compound down to 1.5 K was negative.