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Abstract: Since environmental geochemical behaviour of antimony (Sb), especially Sb speciation in aquatic system were largely unknown, studies were conducted in various waters and sediments from the world's largest antimony mine area at Xikuangshan (XKS). Based on samples collection, total and speciation of Sb and several aquatic environmental parameters were determined in waters from river, well, reservoir, wastewater and sediments. Sb(V) was found as the predominant speciation in the waters and sediments. The environmental geochemical behavior of Sb speciations were mainly controlled by the process of oxidation and adsorption/combination with environmental matrix, mainly as Fe/Al (hydr)oxide, and oxidation may has higher priority than adsorption in the aquatic system. Spatial distribution of decreased Sb concentrations in some surface waters resulted from the dilution effect of river/reservoir/tributary water and adsorption of

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