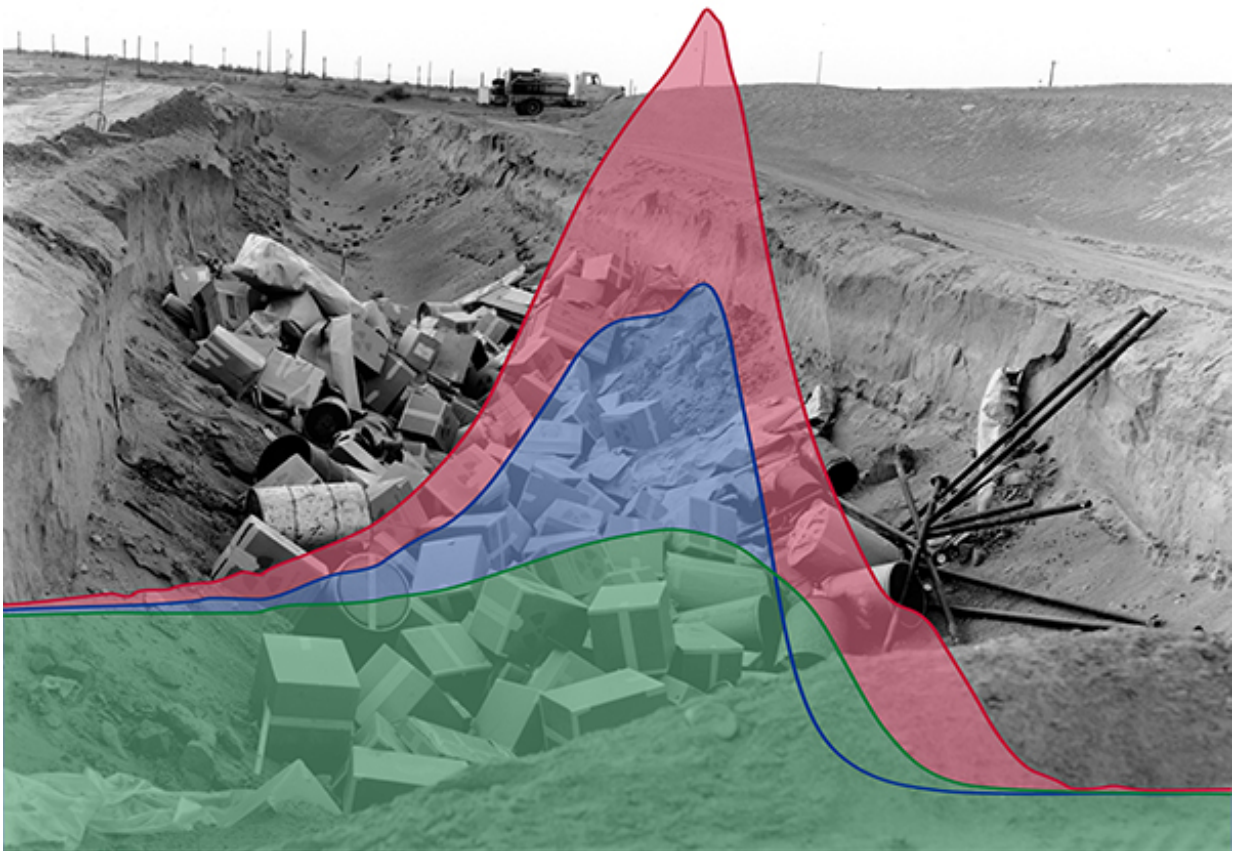


# Scientists precisely measure the charge state of manganese

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Keeping chromium stationary often requires a better understanding of manganese and its oxidation state. The team's rigorous work to determine the oxidation state using x-ray photoelectron spectroscopy offers new insights. Credit: Nathan Johnson, Pacific Northwest National Laboratory

Most atoms lose or gain just a few negatively charged electrons from their surroundings, but not manganese. This element can donate up to seven electrons or wrench as many as three electrons away, abilities that can have implications for water and soil quality. Scientists need to accurately count the number of electrons manganese has when present in materials and minerals. Led by Pacific Northwest National Laboratory (PNNL), a team from the Smithsonian Institution and Penn State devised an easy-to-implement method that does just that. Their approach, based on detailed, controlled experiments and careful analysis, lets others with access to an x-ray photoelectron spectrometer conduct their own measurements.

If you saw the movie "Erin Brockovich," you know the concerns around chromium (Cr) contamination. The element is part of wood preservatives, metal treatments, and more. Chromium that has lost three [electrons](#) ( $\text{Cr}^{3+}$ ) can stay put in the soil and deep underground, but if it loses more electrons and transforms to  $\text{Cr}^{6+}$ , it becomes mobile and can enter water supplies. Manganese oxyhydroxides (a combination of [manganese](#), oxygen, and hydrogen) are the only minerals in the environment capable of taking those electrons away from  $\text{Cr}^{3+}$  and transforming it to  $\text{Cr}^{6+}$ . Preventing chromium from mobilizing would benefit from a more detailed understanding of manganese and its electron count, or oxidation state. The team's rigorous work to determine the oxidation state using [x-ray photoelectron spectroscopy](#) (XPS), a technique widely available in the scientific community, will offer new insights.

The goal was to quantify the oxidation state of manganese in birnessite, a mineral that contains manganese in three different oxidation states, having lost two, three, or four electrons (noted as  $\text{Mn}^{2+}$ ,  $^{3+}$ ,  $^{4+}$ , respectively). The samples came from the Department of Mineral Sciences at the Smithsonian Institution in Washington, DC.

The team analyzed the samples with the XPS at the Environmental Molecular Sciences Laboratory (EMSL) in Washington State. For each sample, they obtained spectra, showing peaks that signal the oxidation states present, but in a convoluted, not easily-extractable manner. How does one determine the contribution from each oxidation state?

"This question has been notoriously difficult to answer," said Dr. Sebastien Kerisit, a PNNL chemist on the project. "How do you assign peaks, what do they mean, how do they relate to the composition of the minerals? We wanted to provide a rigorous approach that let scientists really be able to quantify the different oxidation states of manganese."

The team performed an in-depth analysis of the spectra using as standards samples in which manganese was known to be present in a single oxidation state and exploiting correlations between the different peaks for samples with multiple oxidation states. As part of this work, they showed that a more detailed approach is vital to get the correct oxidation state for manganese oxyhydroxides. The simple approach of relying on a single peak does not work with the oxyhydroxide systems with three possible oxidation states.

"You have to be careful when doing XPS studies, and we've laid out how careful you have to be," said Dr. Eugene Ilton, who led the study. Their technique is now available for others to use. Scientists at EMSL and elsewhere are using this approach to calculate the oxidation state of manganese.

Scientists can use this new approach to gain an accurate understanding of manganese [oxidation states](#), a benefit to those designing new energy solutions and those working on environmental cleanup. At PNNL, scientists are already using this method to understand how manganese-containing minerals nucleate and grow.

**More information:** Eugene S. Ilton et al. XPS determination of Mn oxidation states in Mn (hydr)oxides, *Applied Surface Science* (2016).  
[DOI: 10.1016/j.apsusc.2015.12.159](https://doi.org/10.1016/j.apsusc.2015.12.159)

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