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A web-based library of XAFS data on model compounds

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An archive of XAFS data collected on standard model compounds of transition metals has been constructed and made available by the world wide web. The data in this library have all been taken in transmission on powder samples free of thickness effects. Data are stored in individual files in a standardized ASCII column format. Fields describing the contents of files can be searched easily. The data are intended to provide standards for comparison of EXAFS analysis procedures, for empirical analysis of XANES features, and to test theoretical XANES calculations. Emphasis has been placed on inorganic transition metal compounds to assist the analysis of environmentally relevant samples.

Keywords: EXAFS Data; web-based archive.

1. Introduction

As the interest in and use of x-ray absorption spectroscopy (XAS) to characterize environmentally-relevant transition metal complexes increases, there has been renewed interest in having XAS spectra on model compounds easily available through a standard archive. Despite the realization that such an archive would have a variety of uses, there has not been a significant effort made to construct an archive that contains data of sufficient quality and documentation to be useful to the broad XAFS community. The only current such archive publicly available is that containing data from Farrel Lytle, housed at the web-site of the International XAFS Society. This archive has a large amount of raw XAFS data, but is poorly documented, and contains data of varying quality in different formats with little description of sample preparation or collection details.

To address this need, we have begun a publicly available archive of EXAFS data that contains data only on well-characterized "model compounds" with sample preparation and collection techniques as close to ideal (Lytle *et al.*, 1989; Stern & Heald, 1983) as possible. The data are kept in files with a consistent ASCII column format and descriptions of sample information, including known crystal structure, sample preparation techniques, sample temperature, and data collection conditions (x-ray source, monochromator, etc), in format allowing the files to be searched and sorted.

With the predominance of XAFS analysis for structural information now using theoretical standards such as FEFF (Zabinsky *et al.*, 1995), one might question the utility and need for of an archive of XAFS data. The motivation for this work is threefold. First, the library is intended to reduce the need to collect XAFS data on model compounds, which is still useful in assessing the reliability of theoretical XAFS standards, and for empirically determining reasonable fitting parameters such as E_0 and S_0^2. Due to differences in beamline characteristics, this need will not be completely eliminated, but it is expected that having data easily accessible will be more efficient and convenient than re-measuring model compounds for most users. Second, the data in the library serves as a basis set for empirical XANES analysis, either using edge shifts and peak heights or more elaborate analysis methods based on combinations of spectra (Wasserman, 1997). Third, the library provides data that can be used as test cases for comparisons to theoretical XANES calculations, which are not currently adequate for quantitative analysis.

2. Sample Preparation and Data Collection

At this writing there are more that 120 data files in the archive, representing 27 model compounds. Samples are primarily transition metal oxides and sulfides, with emphasis on toxic metals relevant for environmental sciences. In addition, there has been a large effort to collect data on these samples at cryogenic as well as room temperature so as to assess the importance of anharmonicity on the local structures of these materials.

All data in the library have been collected in transmission mode using ion chambers. To date, all samples have been fine powders and all measurements have been made at the Stanford Synchrotron Radiation Laboratory with either Si (111) or (220) monochromator crystals, detuned so as to minimize harmonics, and with no focusing optics.

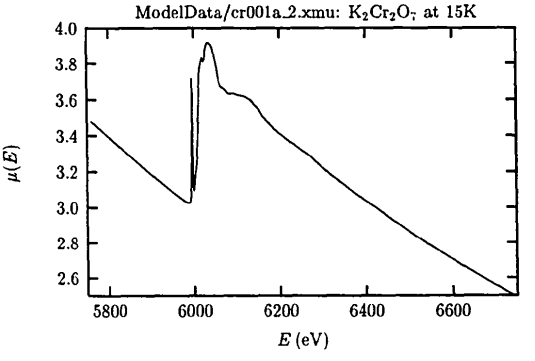

Figure 1
A sample $\mu(E)$ spectra from the Model Compound Library. Shown is a single scan through the Cr K-edge of $K_2Cr_2O_7$ at 15K.

For most compounds, EXAFS data was collected to at least 15 $Å^{-1}$ at three different temperatures (room temperature and 2 cryogenic temperature – usually 80K and 15K). Two or three successive spectra were typically collected at each temperature. Most spectra were collected with a simultaneous measure of a standard reference foil

to aid calibration of edge position and energy resolution. The absorption coefficient of the reference foil was also measured in transmission with an ion chamber placed downstream of the sample. A typical raw EXAFS spectra is shown in Figure 1 for the Cr K-edge of $K_2Cr_2O_7$ at 15K. The quality of the data is generally very high, with differences in $\chi(k)$ for successive scans on the order of 10^{-3}.

3. Data Cataloging and File Formats

To avoid beamline-specific data formats and the need to convert raw signals into absorption $\mu(E)$ data, all data in the archive are in plain text files with a common format easily used by many data analysis programs. All numerical data is listed in space-delimited columns of Energy, Absorption Coefficient $\ln(I_1/I_0)$, Absorption Coefficient of a reference foil $\ln(I_2/I_1)$, and I_0 for the three ion chamber configuration with the sample between I_0 and I_1 and a reference foil between I_1 and I_2.

For each compound, there is also a crystallographic description of the structure in the form of an input file to the ATOMS program (Ravel, 1994), which generates an input file for the FEFF program (Zabinsky *et al.*, 1995). In addition, there are fields giving the sample name, absorbing atom, excited core level, details of sample preparation, temperature, reference compound, fill gases and lengths of ion chambers, beamline used, monochromator used, date of collection, and number of data points. A sample data file is shown in Figure 2. The information in the files is arranged so that the different fields can be searched and sorted by a web-searching program.

```
#%name: K2Cr2O7
#%atom: Cr
#%edge: K
#%xtal: K2Cr2O7.inp
#%prep: powder on tape (4 layers)
#%ref:  Cr foil
#%misc: exafs to K16
#%det:  I0=N2 15cm; I1=N2 30cm; I2=N2 15cm
#%temp: 15K
#%beam: ssrl 2-3,  vert slits = 0.6mm
#%mono: si(111) unfocused, detuned 75%, clockwise at E=5990eV
#%date: Fri Apr 18 12:31:25 1997
#%cols: 396  E XMU XMUR I0
#--------------
# energy     log(i0/it)   log(it/ir)  i0
  5759.989   3.479599     2.054440    159822.8
  5769.980   3.457835     2.043293    159302.8
  5780.007   3.436509     2.041864    158831.8
  5790.003   3.414928     2.036382    158086.8
  5799.965   3.393809     2.029856    157403.8
  5810.034   3.371727     2.024143    156616.8
  5820.000   3.350557     2.019564    156073.8
  5830.002   3.329300     2.011395    155303.8
  5839.971   3.308482     2.007845    154619.8
  5849.975   3.287252     1.999791    153914.8
```

Figure 2
A sample data file from the XAFS Model Compound Library. Sample and data collection descriptions are given in a file header denoting sample name, crystal structure, temperature, monochromator, and so on. Numerical data are stored in plain text columns of energy, sample absorption coefficient, reference foil absorption coefficient, and I_0.

4. Conclusion

We have described the content of the Web-based Model Compound Library of EXAFS Data. The library is accessible at

http://cars.uchicago.edu/~newville/ModelLib/

We encourage use of this library and welcome contributions of data on well-characterized model compounds.

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