

ANL/CMT/CP-92716
CONF-970805--

**"IN-SITU" INFRARED SPECTROSCOPY OF ELECTRODE
SURFACES WITH A SYNCHROTRON SOURCE***

C.A. Melendres
Argonne National Laboratory
Materials Science and Chemical Technology Divisions
Argonne, IL 60439 USA

G.A. Bowmaker
University of Auckland
Department of Chemistry
Auckland, New Zealand

J.M. Leger and B. Beden
University of Poitiers
Laboratoire de Chimie
Poitiers, France

A. Tadjeddine
LURE, Orsay
France

RECEIVED
SEP 22 1997
OSTI

Joint Meeting of the Electrochemical Society and the International Society of Electrochemistry
Paris, France
August 31 to September 5, 1997

The submitted manuscript has been created by the University of Chicago as Operator of Argonne National Laboratory ("Argonne") under Contract No. W-31-109-ENG-38 with the U.S. Department of Energy. The U.S. Government retains for itself, and others acting on its behalf, a paid-up, nonexclusive, irrevocable worldwide license in said article to reproduce, prepare derivative works, distribute copies to the public, and perform publicly and display publicly, by or on behalf of the Government.

*Work supported by the U.S. Department of Energy, Office of Basic Energy Sciences, Division of Materials Science, under Contract W-31-109-ENG-38; work done at the National Synchrotron Light Source, Brookhaven National Laboratory, beamline U4IR.

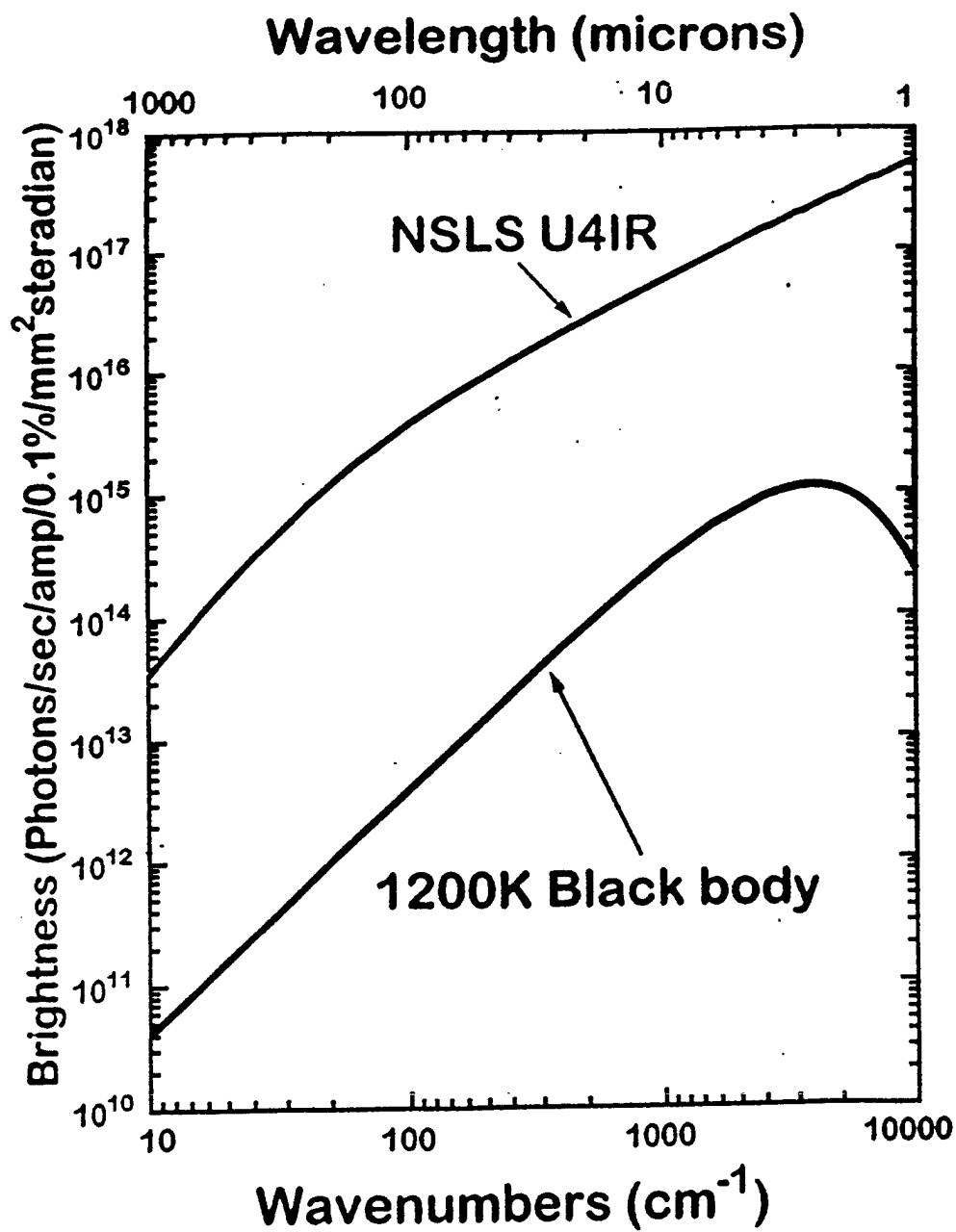
DISTRIBUTION OF THIS DOCUMENT IS UNLIMITED

MASTER

19980416 056

DISCLAIMER

This report was prepared as an account of work sponsored by an agency of the United States Government. Neither the United States Government nor any agency thereof, nor any of their employees, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness, or usefulness of any information, apparatus, product, or process disclosed, or represents that its use would not infringe privately owned rights. Reference herein to any specific commercial product, process, or service by trade name, trademark, manufacturer, or otherwise does not necessarily constitute or imply its endorsement, recommendation, or favoring by the United States Government or any agency thereof. The views and opinions of authors expressed herein do not necessarily state or reflect those of the United States Government or any agency thereof.



Brightness comparison of the NSLS U4IR
and a 1200K standard thermal source.

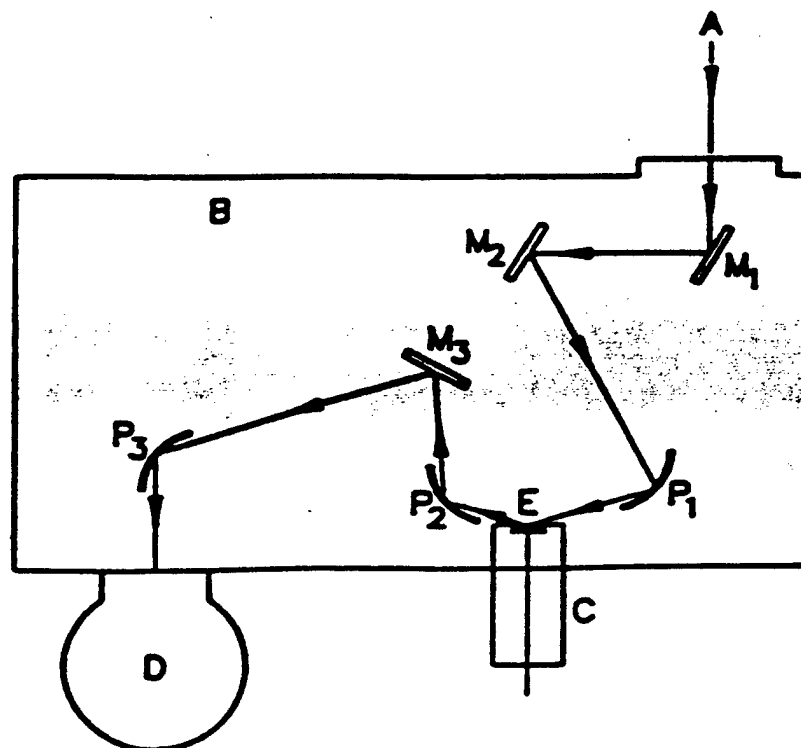
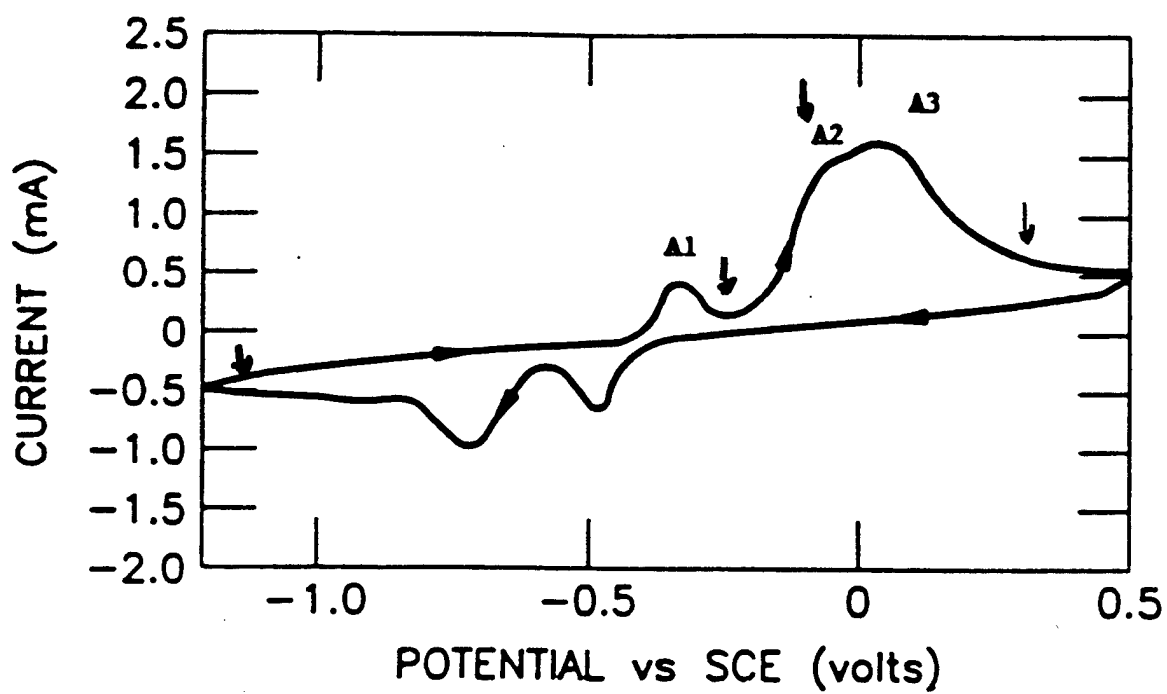
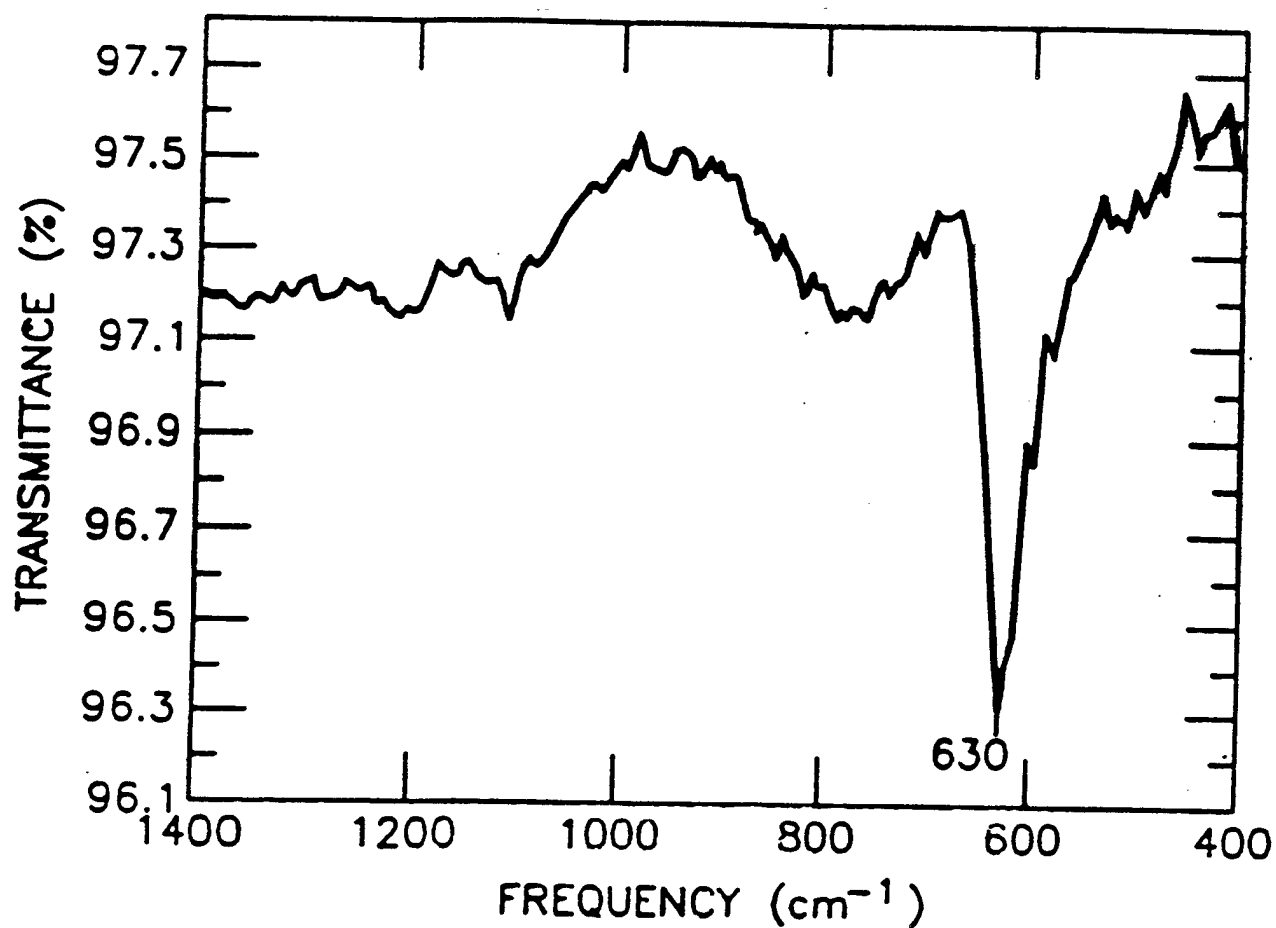


Fig. 1. Schematic of Optical Layout for Synchrotron Far IR Spectroscopy

- A - Collimated IR beam from synchrotron (through Nicolet Impact 400)
- B - Purgeable chamber
- C - Electrochemical cell
- D - Detector
- E - Platinum electrode
- M_1, M_2, M_3 , are plain mirrors
- P_1, P_2, P_3 , are off-axis paraboloid reflectors



Cyclic voltammogram of copper in 0.1 M NaOH solution at scan rate of 20 mV/sec.



"In-Situ" Synchrotron IR spectrum of copper in 0.01 M NaOH solution at -0.05 V vs. SCE.

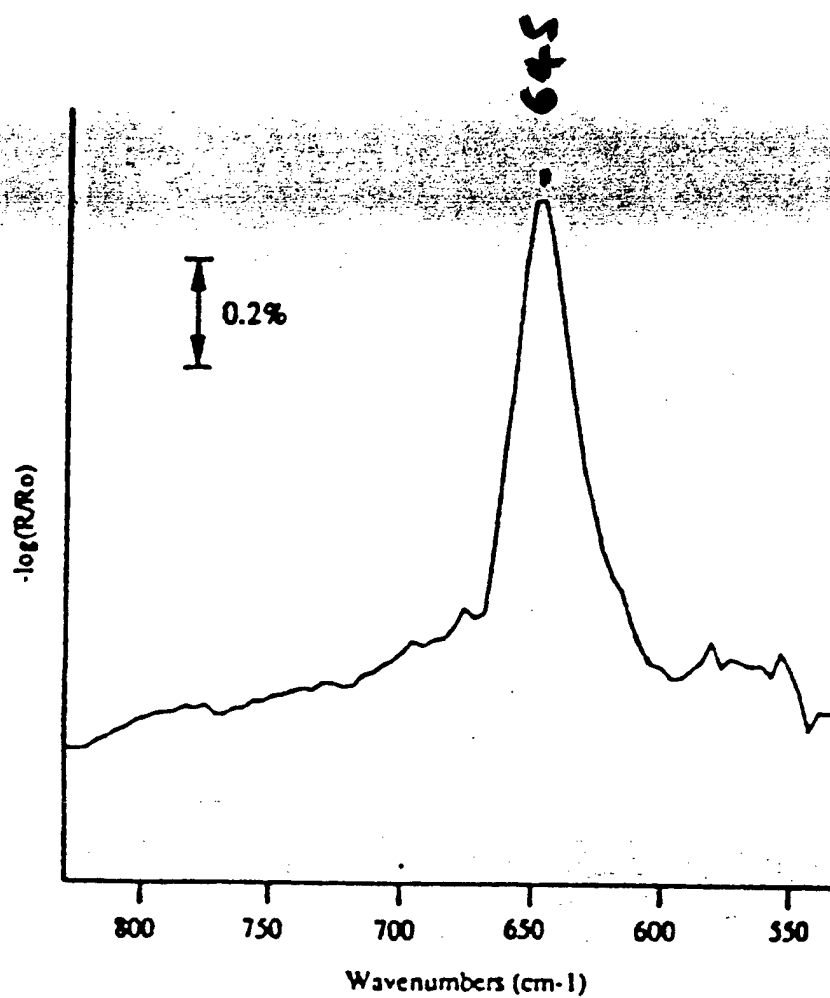


Fig. 4. *In situ* IRAS spectrum in the region from 830 to 530 cm⁻¹ for copper exposed to flowing air of 90% humidity.

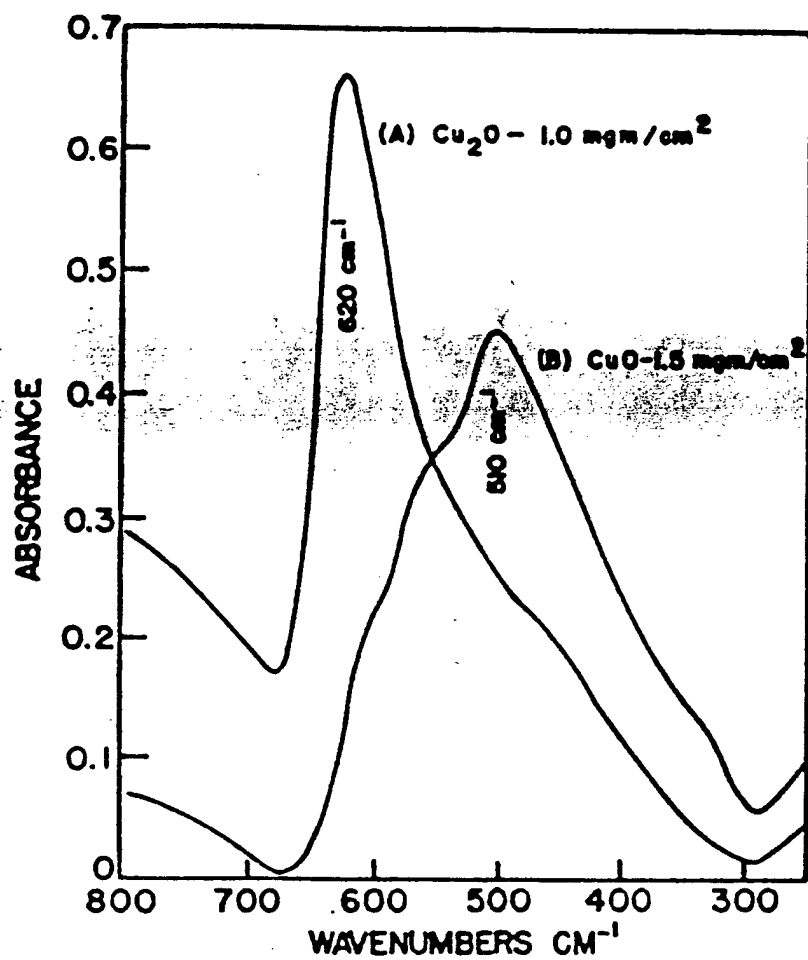


Fig. 1. Transmission spectra of Cu_2O and CuO (in KI pellets)

Poling (JECS '69)

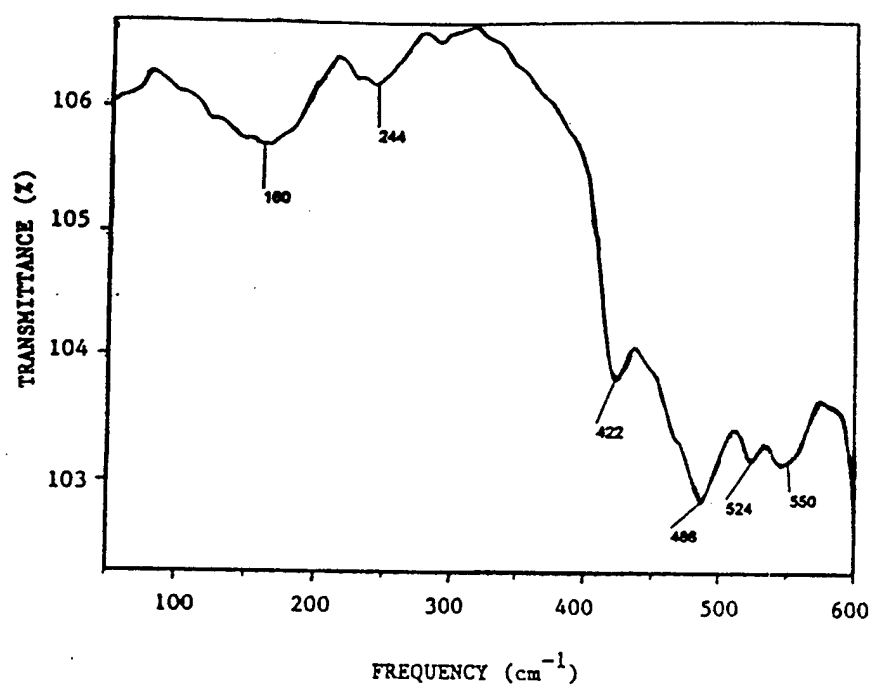


Figure 3. "In-situ" far IR spectrum of Cu in 0.1 M NaOH at 0.3 V vs. SCE (referenced to spectrum at -1.0 V).

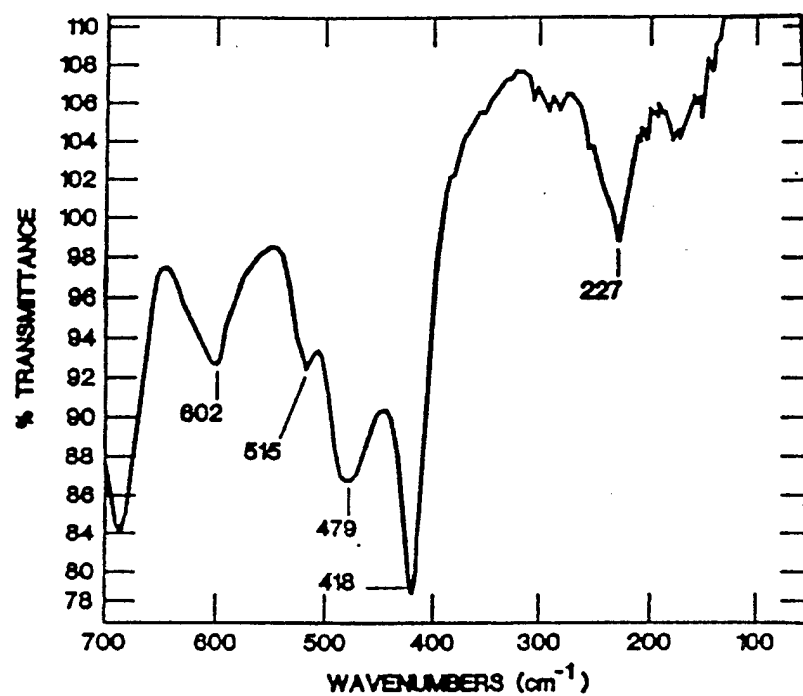


Figure 4. Far IR spectrum of $\text{Cu}(\text{OH})_2$ powder in a Nujol mull.

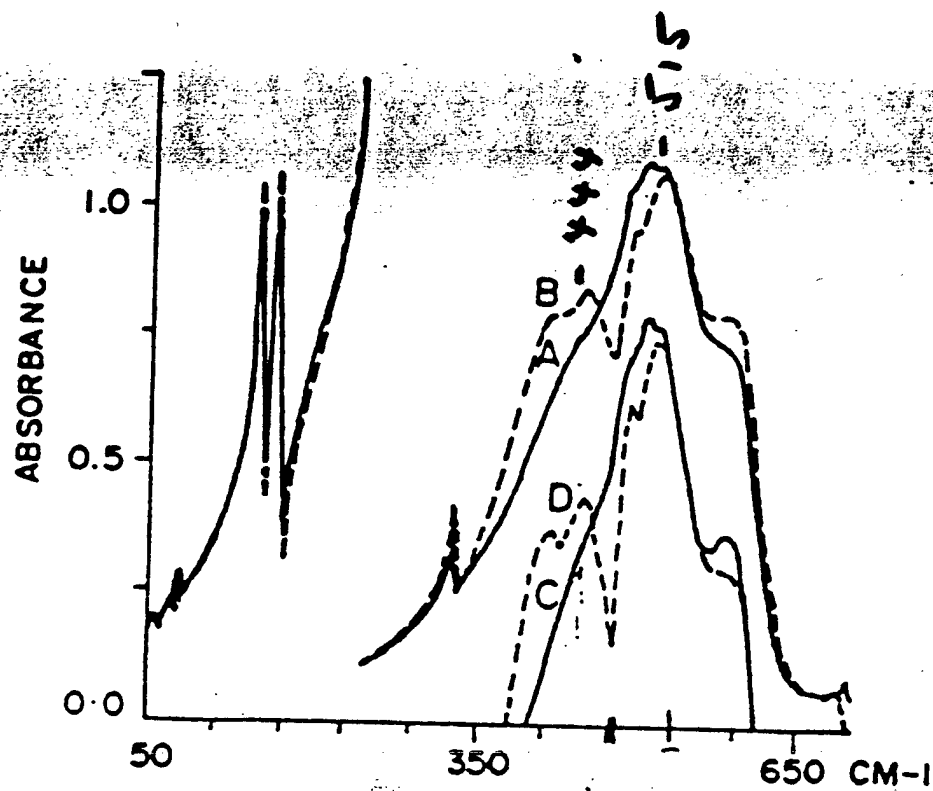
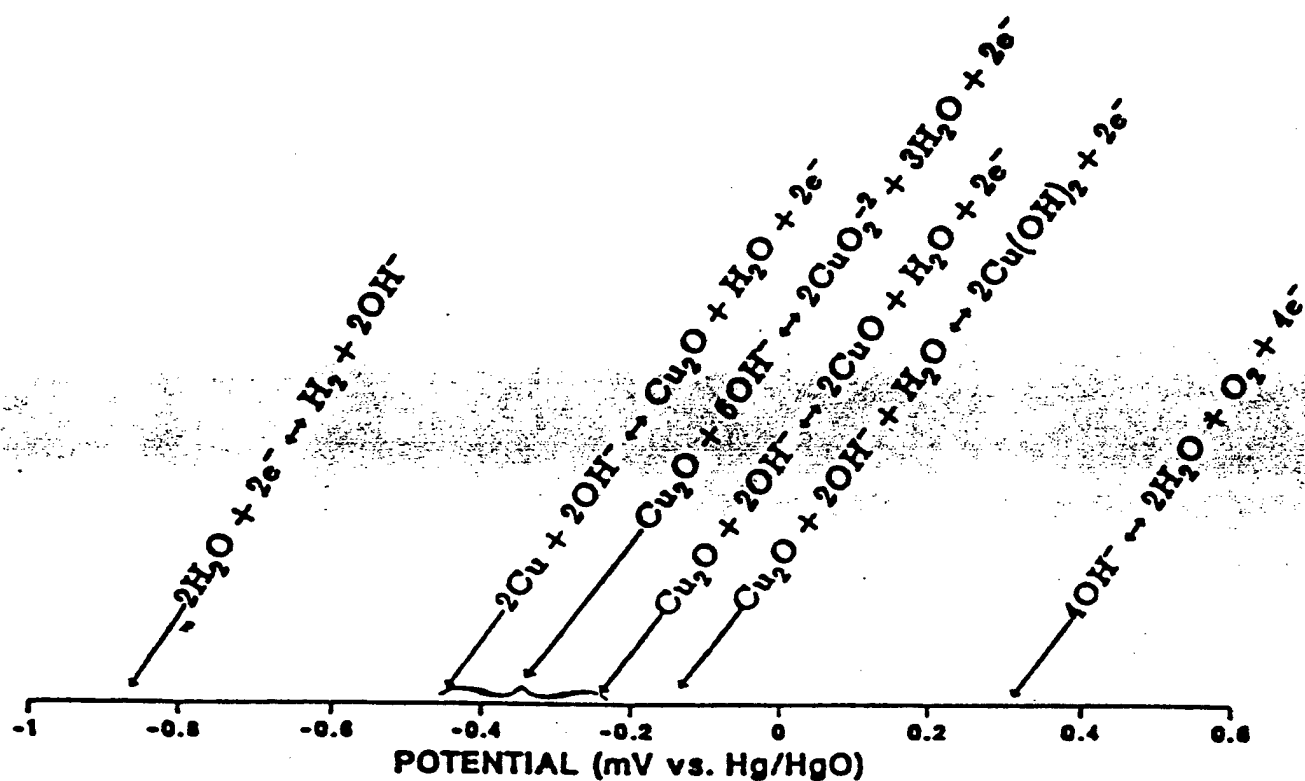


Fig. 1. Absorption spectra of CuO (paraffin mull) at 90 K and 300 K. (A) (solid line) 300 K. (B) (broken line) 90 K. (C) Deconvolution of A. (D) Deconvolution of B.



Thermodynamic potentials of various reactions of copper in 1 M KOH vs. Hg/HgO reference electrode.

Mayer (JECS '92)

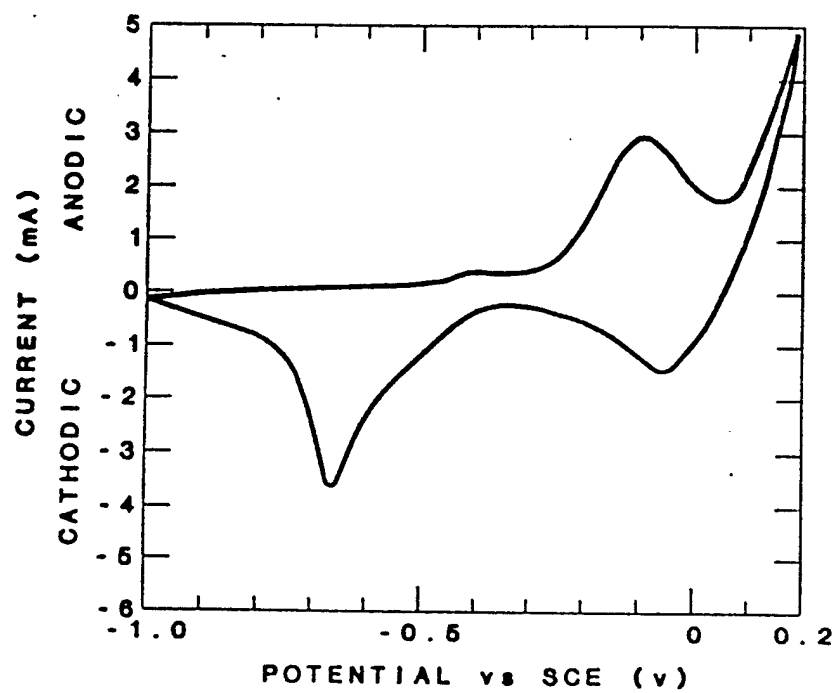


Figure 5. Cyclic voltammogram of Cu in 0.01 M KSCN + 0.1 M KClO₄ solution at a scan rate of 100 mV/sec.

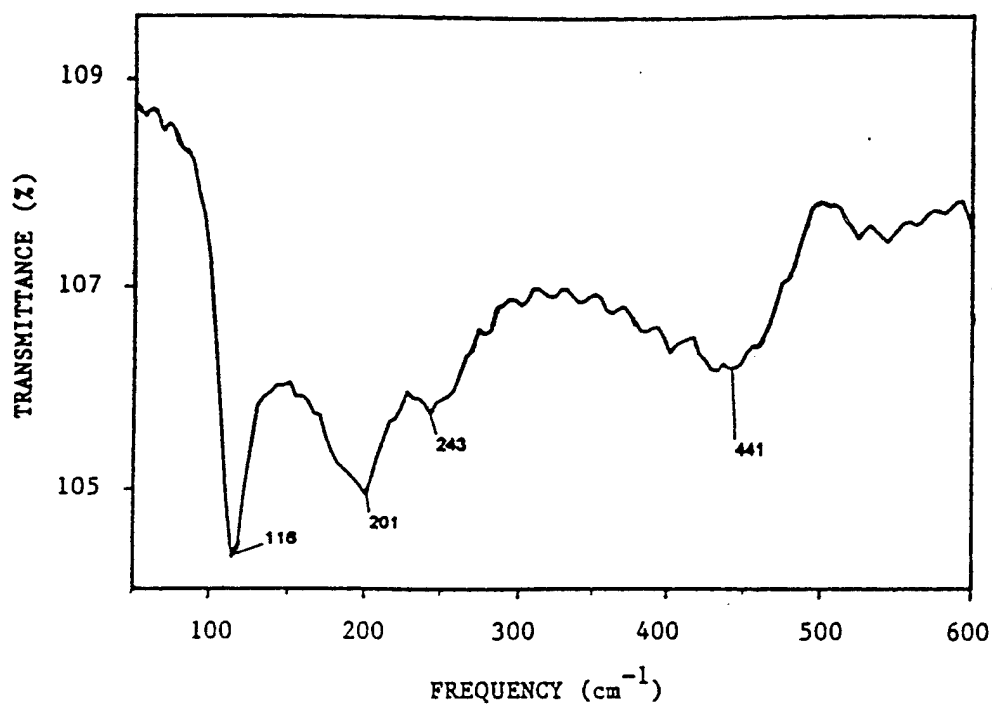


Figure 6. "In-situ" synchrotron far IR spectrum of Cu in 0.025 M KSCN + 0.1 M KClO₄ solution at 0.05 V vs. SCE (referenced to spectrum at 1.10 V).

M97054280



Report Number (14) ANL/CMT/CP- 92716

CONF-970805--

Publ. Date (11) 199709

Sponsor Code (18) DOE/ER, XF

UC Category (19) UC-404, DOE/ER

DOE