UNCLASSIFIED

FEL
4.11.56

OAK RIDGE NATIONAL LABORATORY

Operated By
UNION CARBIDE NUCLEAR COMPANY POST OFFICE BOX P
OAK RIDGE, TENNESSEE

EXTERNAL TRANSMITTAL AUTHORIZED

ORNL
CENTRAL FILES NUMBER

56-3-151

COPY NO. 7

DATE: March 26, 1956

SUBJECT: Differential Infrared Absorption Techniques
for Characterizing the Surface of ThO₂

TO: F. L. Culler

FROM: V. D. Allred

LEGAL NOTICE

This report was prepared as an account of Government sponsored work. Neither the United States, nor the Commission, nor any person acting on behalf of the Commission:

A. Makes any warranty or representation, express or implied, with respect to the accuracy, completeness, or usefulness of the information contained in this report, or that the use of any information, apparatus, method, or process disclosed in this report may not infringe privately owned rights; or

B. Assumes any liabilities with respect to the use of, or for damages resulting from the use of any information, apparatus, method, or process disclosed in this report.

As used in the above, "person acting on behalf of the Commission" includes any employee or contractor of the Commission to the extent that such employee or contractor prepares, handles or distributes, or provides access to, any information pursuant to his employment or contract with the Commission.

Laboratory Records (B)

Photostat Price \$ 1.80
Microfilm Price \$ 1.80Available from the
Office of Technical Services
Department of Commerce
Washington 25, D. C.

NOTICE

This document contains information of a preliminary nature, and was prepared primarily for internal use at the Oak Ridge National Laboratory. It is subject to revision or correction and therefore does not represent a final report.

UNCLASSIFIED

398 001

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.

DISCLAIMER

Portions of this document may be illegible in electronic image products. Images are produced from the best available original document.

1.0 SUMMARY

The differential infrared absorption technique for studying the surface of solids has been investigated. This method was tested on samples of thorium oxide at the University of Utah and was found to have excellent sensitivity in the infrared region. It is recommended that the differential infrared absorption spectra be used as a tool in the characterization of solids in laboratory and engineering pumping studies.

2.0 DISCUSSION

The value of infrared absorption study of solids has been demonstrated.¹⁻⁵ The differential method of studying the infrared spectra of surfaces has been a recent development at the University of Utah.⁶ In this procedure finely divided suspensions of solids are uniformly suspended in a KI or KBr matrix in such a way that only the absorption bands associated with surface are obtained. Advantages of such techniques are that displacement of surface products, adsorption of new products, and differences in the chemical nature of the surfaces may be detected.

To determine the applicability of the method to the thorium oxide slurry problems a trip was made to the University of Utah by V. D. Allred, March 1-4, 1956. Dr. Milton E. Wadsworth, an ORNL consultant, was contacted there. He has had extensive experience in this work at the university. The necessary dies and other equipment were available so that typical thorium oxide samples could be readily examined.

Examination of thorium oxide showed it to be particularly well

suited for study in the infrared region. Oxides having specific surface areas from 2 to 35 m²/g and of concentrations up to 36 mg in a 2 gram potassium iodide matrix were used without difficulty in transmittance. Absorption bands characteristic of water or hydroxyl groups on the surface were observed at wave lengths of 2.85 and 6.15 microns. Additional bands characteristic of the thorium oxide surface, but not identified, were found at 6.5, 7.6, 9.5, and 11.7 microns.

Infrared absorption curves typical of those obtained are included as Fig. 1. Curve A represents the curve obtained for an uncompensated 2 gram window of KI. The bands at 2.85 and 6.15 microns are from water absorption. The bands at 7.1, 7.3, 9.0, 9.9, 7.9, 12.0, 12.5, 14.25, 3.4, and 4.85 appear to be characteristic of this particular matrix material. As noted in the curves C and D these bands are well compensated by the differential technique and have largely disappeared.

Curve B is a typical absorption curve for thorium oxide. The sample consisted of 12 mg of 650°C fired oxide in a typical 2 g KI matrix. The reference was the same KI window used for Curve A.

Curves C and D are typical differential absorption curves where both the matrix and the thorium oxide are compensated by the reference beam. These data are from 12 mg samples of oxide with approximately equivalent surface area but a different preparation or treatment. Of particular interest in these curves are the characteristic inversions seen at 3.15, 6.3-6.5, and 7.5; however, their significance is not known at this time.

In view of the favorable results obtained at the University of Utah it appears desirable to incorporate the differential infrared absorption technique into the present slurry program. A relatively common, compensating type, spectrometer such as the Perkin-Elmer

398 004

Fig. 1

UNCLASSIFIED

- 5 -

Model 21 can be used. A unit of this type is available in Bldg. 4501. To begin work of this type will require only the fabrication of suitable dies by which the sample and matrix may be pressed into spectrometer windows.

V. D. Allred

V. D. Allred

VDA/jr

3.0 REFERENCES

1. Hunt, J. M., Wisherd, M. P., Bonhom, L. C., Anal. Chem., 22, 1478-97 (1950).
2. Miller, F. A., Wilkens, C. H., Anal. Chem., 24, 1253-94 (1952).
3. Stimson, M. M., O'Donnell, M. J., J. Am. Chem. Soc., 74, 1805 (1952).
4. Scheidt, U., Z. Naturforsch., 76, No. 5, 270 (1953).
5. French, R. O., Wadsworth, M. E., Cook, M. A., Cutler, I. B., J. Phy. Chem., 58, 805-11 (1954).
6. Wadsworth, M. E., private communication.