INFRARED SPECTRA OF VARIOUS METAL OXIDES IN THE REGION OF 2 TO 26 MICRONS

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NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

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SUMMARY

The infrared spectra of 52 metal oxides were determined through the sodium chloride and potassium bromide absorption regions. These spectra include the stable rare earth oxides and are the first comprehensive collection of infrared metal oxide spectra known to be published.

INTRODUCTION

This report presents a comprehensive set of infrared spectra of metal oxides which are of value in the identification of metal oxides by infrared spectroscopy. Prior to this report, the publication of infrared spectra of the oxides of the elements was rather restricted. The majority of oxide spectra published previously were those of the halogens, the various oxides of nitrogen, the nitrogen group, and carbon. This work excludes these oxides except those of the metals - antimony, arsenic, and bismuth - of the nitrogen group. It does not include oxide spectra of alkali metals and the alkaline earths.

The infrared spectra (figs. 1 to 52) of various oxides of 51 elements were determined through the absorption region of sodium chloride and potassium bromide (from 2 to 26 μ). The sodium chloride region, known as the rocksalt region, transmits infrared radiation in the wavelength range 2 to 16 microns. The potassium bromide region has a useful transmission range through 26 microns.

The metal oxides other than rare earth oxides for which spectra were obtained are listed in the following table:

*Instructor of Chemistry, Norwalk High School, Norwalk, Ohio.
<table>
<thead>
<tr>
<th>Oxide</th>
<th>Spectrum in fig. -</th>
<th>Oxide</th>
<th>Spectrum in fig. -</th>
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<td>Aluminum oxide (Al₂O₃)</td>
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<td>Bismuth trioxide (Bi₂O₃)</td>
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<td>Silicon dioxide (SiO₂)</td>
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<td>Zinc oxide (ZnO)</td>
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<td>Mercuric oxide (HgO)</td>
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<td>Zirconium dioxide (ZrO₂)</td>
<td>52</td>
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</table>

The potassium bromide (KBr) pellet technique was employed to obtain these spectra. Wherever possible the paraffin oil mull technique was used to verify the spectra, since the possibility of reaction between various oxides and the KBr due to the pressure required to make the pellets was not known.

The spectra of the rare earths were determined by the KBr pellet technique only, since the predominant absorption bands for the rare earth oxides occur in the same wavelength regions as the absorption bands of the paraffin oil. The rare earth oxides for which spectra were obtained are listed in the table at the left. Promethium is not included because it has no stable or long-lived isotopes.

The reader should be aware of the combination of bands at 2.9 to 3.0 and 6.1 to 6.2 microns in these spectra which may be due to water. The combination of bands at 4.3 and 14.3 microns should also be viewed with some suspicion since carbon dioxide may be present. The use of spectrographic grade oxides did not assure the absence of water or carbon dioxide. The KBr pellets were made under vacuum in an attempt to remove water.
and carbon dioxide. However, this treatment may not have completely removed these contaminants because these bands, principally the water bands, are observed in some of the spectra. These bands could represent water vapor or carbon dioxide adsorbed on the surface of the KBr pellets.

Figures 53 and 54 are composites of infrared absorption bands for the various oxide spectra presented in this report. Figure 53 lists the metal oxides exclusive of the rare earth oxides. The rare earth oxides are grouped together in figure 54 to show spectral similarities. Magnesium oxide, yttrium oxide, and scandium oxide are also listed with the rare earth oxides in figure 54 since their spectra show marked similarities to those of the rare earths. In figures 53 and 54, the horizontal lines represent the widths of the bands, while the vertical lines indicate relative absorption of infrared radiation at the different wavelengths for a particular oxide. When observing the broad absorption bands in figures 53 and 54, the reader should remember the spectra are the result of excitation of low energy molecular vibrations and crystal lattice vibrations. The results of the interactions between the molecular vibrations and the lattice vibrations are diffuse spectra. There are probably cases where large broad bands could have been resolved into several bands by further dilution of the sample with KBr. However, in most instances, if the broad bands did not tend to show significant definition after several dilutions, further dilutions were not attempted.

Before these spectra were prepared for publication, they were evaluated by Dr. Herman Syzmanski of Canisius College, Buffalo, New York.

EXPERIMENTAL DETAILS

The instrument used was a Baird-Atomic NK-1 double-beam recording spectrophotometer utilizing a cam and prism interchange to cover the wavelength region from 2 to 26 microns. With a sodium chloride prism the instrument covered the range from 2 to 16 microns; with the potassium bromide prism and cam interchange, the region from 12 to 26 microns was studied.

The majority of the oxides used for the work were spectrographic standards as obtained from Spex Industries, Inc., Scotch Plains, N. J. The oxides were used directly from the containers. They were neither dessicated nor heated to remove water. Other oxides used were obtained from Fisher Chemical and Mallincrodt Chemical. Samples for analyses were prepared as KBr pellets and as paraffin oil mulls.

The pellets were made by using spectrographic grade KBr (≈100 mg) and a small amount (10 to 20 mg) of the oxide. The two compounds were ground and mixed together with a mortar and pestle. Occasionally a "Wig-L-Bug" was used to mix samples. The pellet was formed by placing a small amount of the mixture in the pellet mold. The
thickness was normally 0.78 millimeter. The mold was then placed in a vacuum chamber to allow the release of carbon dioxide and water vapor from the sample mixture. A pressure of $1.03 \times 10^9$ dynes per square centimeter was then applied with a Carver press. After 5 minutes, the pellet was removed from the mold. The pellet was then placed in the sample holder and positioned in the sample beam of the instrument, and the recorder drum was rotated manually through the region studied to determine whether the oxide content of the pellet was of the proper concentration to give an acceptable absorption spectrum. If the spectrum appeared unsatisfactory, the sample mixture was either diluted or concentrated with respect to oxide content so that an absorption spectrum was obtained which showed bands in the greatest detail. In some cases, pellets of several concentrations were used to provide spectra of the oxide when intensities of the bands differed greatly. In many cases, the concentration of the pellets used for the sodium chloride region was not satisfactory for the best band detail in the potassium bromide region. These pellets were then placed in airtight containers until a prism and cam interchange was made, and the spectrum was determined through the longer wavelength region.

Paraffin oil mulls were made by grinding the oxide in the oil with a Mullite mortar and pestle. The mull was then placed between the plates of a demountable cell, either sodium chloride or potassium bromide, depending upon the region under study. The technique of hand rotating the recorder drum prior to recording the spectrum was also employed with the mulls.

All spectra were obtained under the same instrument operating conditions. The scanning speed was 1 micron per minute, and the slit width was set at the 1 normal position. (The slit width in the so-called normal position is actually continuously adjustable. A cam opens the slits as the recording drum rotates, and thus a constant energy level is maintained in the system throughout the wavelength range.) Instrument suppression was set at 5, and air was used as a reference.

**DISCUSSION OF SPECTRAL SIMILARITIES**

Similarities existing between these spectra and spectra in the literature or between spectra of the elements of the same group are noted in this section. No attempts are made to characterize bands observed in the spectra by assigning stretching frequencies of $M-O$, $M-O-M$, or $M-M$ (where $M$ indicates metal and $O$ indicates oxygen).

**Rare Earths**

The great similarity of the infrared spectra of the rare earth oxides having the gen-
eral formula $R_2O_3$ (sesquioxides) is shown in figure 54. This similarity is particularly
evident with $Dy_2O_3$ (fig. 13), $Er_2O_3$ (fig. 14), $Eu_2O_3$ (fig. 15), $Gd_2O_3$ (fig. 16), $Ho_2O_3$
(fig. 20), $La_2O_3$ (fig. 23), $Lu_2O_3$ (fig. 25), $Nd_2O_3$ (fig. 29), $Pr_6O_{11}$ (fig. 32), $Sm_2O_3$
(fig. 33), $Tm_2O_3$ (fig. 43), and $Yb_2O_3$ (fig. 49). Although $Pr_6O_{11}$ is not of the $R_2O_3$ for-
mula, the spectrum of $Pr_6O_{11}$ (fig. 32) is similar to the spectra of the rare earth oxides.
The exceptions to the similarity of spectra are $CeO_2$ (fig. 9), in which the cerium is in
the +4 oxidation state, and $Tb_4O_7$ (fig. 40), which is a peroxide thought to contain $Tb^{+3}$
and $Tb^{+4}$ in equal amounts. Scandium and yttrium, group IIIA elements of the $M_2O_3$
formula, also exhibit similar spectra, as might be expected from members of the same
group. The spectral similarity of $MgO$, a group IIA metal, is perhaps explained by the
phenomenon known as "diagonal relationship." However, relating the spectral similari-
 ties of $MgO$ to the rare earth oxides is probably an overextension of the principle.

Selenium

The spectrum for selenium dioxide ($SeO_2$, fig. 35) is similar to the spectra of am-
omium selenate and copper selenate-pentahydrate, which have a strong band at 11.7 mi-
crons (ref. 1, pp. 1281-1282). The selenates of sodium and potassium have sharper
bands which occur as multiple bands from 11.1 to 11.4 microns. The selenites show a
strong broad band similar to that of $SeO_2$, but it is shifted from the region of 11.1 to 11.4
microns to the region of 13 to 14 microns.

Silicon

The spectrum of silicon dioxide ($SiO_2$, fig. 36) is quite similar to that of silica gel,
$SiO_2 \cdot H_2O$, reported in reference 1 (p. 1266) and the $SiO_2$ spectrum reported in refer-
ence 2.

Antimony and Arsenic

The spectra of antimony oxide ($Sb_2O_3$, fig. 3) and arsenous oxide ($As_2O_3$, fig. 4)
show well pronounced bands at 10.6 and 9.6 microns, respectively. These bands were
recently characterized (ref. 3) as the Sb-O and the As-O stretching frequencies. Exami-
nation of the spectra published in reference 1 (p. 1275) reveals an indication of these
bands. Had the concentration of the oxides in the mulls been greater, their spectra
would undoubtedly have shown these same bands.
Iron

The spectrum of ferric oxide (Fe$_2$O$_3$, fig. 22) does not agree with the hematite (Fe$_2$O$_3$) spectrum reported in reference 4. The latter spectrum shows a strong band at 10.2 microns, which is characterized as the Fe-O stretching frequency. From the position of the band at 10.2 microns, the presence of sulfate impurity is indicated. Several rechecks of the infrared spectrum of pure Fe$_2$O$_3$ at different concentrations have not verified this absorption band.

Boron

The spectrum of boron oxide (B$_2$O$_3$, fig. 7) was obtained from a sample prepared by heating boric acid at 600° C for 1 hour. The composite spectrum was obtained from a flat plate of the cooled melt and from a ribbon pulled from the molten mass to obtain various thicknesses. The lower, more defined portion of the spectrum was from a thick sample. Spectra obtained from KBr pellets of this material at thicknesses of 0.78, 1.2, and 1.6 millimeters showed very little absorption in the 2- to 5-micron region. The spectrum is similar to that reported in reference 5.

Magnesium

The spectrum of magnesium oxide (MgO, fig. 26) compares favorably with the spectrum given in reference 6 except for the band at 2.9 microns and the shoulder at 6.1 microns (in the spectrum of this investigation), which are probably due to water. The doublet at 6.8 to 7.0 microns is shown as one band in reference 6. The bands from 9.0 to 21.0 microns also are indicated in both spectra, although band positions are not in good agreement. The spectra of reference 6 were obtained by fuming thin films of MgO onto potassium chloride plates.

Gallium

The spectrum of gallium sesquioxide (Ga$_2$O$_3$, fig. 17) is similar to that reported in reference 5. Both spectra show the broad band from 12 to 23 microns having a shoulder at 13 microns and maximum intensity at 14.5 microns. The spectrum of reference 5 (p. 50) does not show the less intense band at 7.2 microns, probably because the sample had less gallium oxide in the pellet.
Chromium

The chromic oxide (Cr$_2$O$_3$, fig. 10) spectrum is reported in reference 5 (p. 50). The spectrum in this reference shows a band at 9.0 microns which does not appear in figure 10. However, the broad band starting at 13.6 microns is present in both spectra.

Aluminum

The spectrum of aluminum oxide (Al$_2$O$_3$, fig. 1) is reported in reference 5 (p. 50). The two spectra are similar; both show a weak band at 7.2 microns and a broad band from 9.0 to 19.5 microns.

LITERATURE SEARCH

The literature searched for this report included Chemical Abstracts and Nuclear Science Abstracts from 1950 to 1966. The bibliographies of reference 7, which covers Chemical Abstracts from 1927 to 1962, and reference 8, which covers the literature from 1950 to 1960, were used.

Lewis Research Center,
National Aeronautics and Space Administration,
Cleveland, Ohio, July 14, 1966,
000-00-00-00-22.

REFERENCES


Figure 1. Infrared spectrum of aluminum oxide (Al₂O₃).

(a) Sodium chloride absorption region.

(b) Potassium bromide absorption region.
Figure 2. Infrared spectrum of antimony tetroxide ($\text{Sb}_2\text{O}_4$).

(a) Sodium chloride absorption region.

(b) Potassium bromide absorption region.
Figure 3. - Infrared spectrum of antimony oxide ($\text{Sb}_2\text{O}_3$).
Figure 4. – Infrared spectrum of arsenous oxide (As$_2$O$_3$).
(a) Sodium chloride absorption region.

(b) Potassium bromide absorption region.

Figure 5. Infrared spectrum of beryllium oxide (BeO).
Figure 6. Infrared spectrum of bismuth trioxide (Bi$_2$O$_3$).
Figure 7. - Infrared spectrum of boron oxide (B₂O₃).
Figure 8. - Infrared spectrum of cadmium oxide (CdO).

(a) Sodium chloride absorption region.

(b) Potassium bromide absorption region.
Figure 9. - Infrared spectrum of ceric oxide (CeO₂).

(a) Sodium chloride absorption region.

(b) Potassium bromide absorption region.
(b) Potassium bromide absorption region.

Figure 10. Infrared spectrum of chromic oxide (Cr₂O₃).
Figure 11. - Infrared spectrum of cobaltic oxide (CoO$_2$).
Figure 12. - Infrared spectrum of cupric oxide (CuO).
Figure 13. - Infrared spectrum of dysprosium oxide (Dy$_2$O$_3$).
Figure 14. - Infrared spectrum of erbium oxide (Er₂O₃).
Figure 15. - Infrared spectrum of europium oxide (Eu₂O₃).
Figure 16. - Infrared spectrum of gadolinium oxide (Gd$_2$O$_3$).

(a) Sodium chloride absorption region.
(b) Potassium bromide absorption region.
Figure 17. - Infrared spectrum of gallium sesquioxide (Ga$_2$O$_3$).
Figure 18. - Infrared spectrum of germanium dioxide (GeO₂).

(a) Sodium chloride absorption region.

(b) Potassium bromide absorption region.
Figure 19. - Infrared spectrum of hafnium oxide (HfO$_2$).
Figure 20. Infrared spectrum of holmium oxide (Ho₂O₃).
Figure 21. - Infrared spectrum of indium sesquioxide (In$_2$O$_3$).
Figure 22. - Infrared spectrum of ferric oxide (Fe$_2$O$_3$).

(a) Sodium chloride absorption region.

(b) Potassium bromide absorption region.
(a) Sodium chloride absorption region.

(b) Potassium bromide absorption region.

Figure 23. - Infrared spectrum of lanthanum sesquioxide (La₂O₃).
Figure 24. Infrared spectrum of lead monoxide (PbO).

(a) Sodium chloride absorption region.

(b) Potassium bromide absorption region.
(a) Sodium chloride absorption region.

(b) Potassium bromide absorption region.

Figure 25. - Infrared spectrum of lutetium oxide (Lu₂O₃).
Figure 26. - Infrared spectrum of magnesium oxide (MgO).

(a) Sodium chloride absorption region.

(b) Potassium bromide absorption region.
Figure 27. - Infrared spectrum of mercuric oxide (HgO).

(a) Sodium chloride absorption region.

(b) Potassium bromide absorption region.
Figure 28. - Infrared spectrum of molybdenum trioxide (MoO$_3$).
Figure 29. - Infrared spectrum of neodymium oxide (Nd₂O₃).
Figure 30. - Infrared spectrum of nickel monoxide (NiO).
Wave number, cm$^{-1}$

(a) Sodium chloride absorption region.

(b) Potassium bromide absorption region.

Figure 31. - Infrared spectrum of niobium pentoxide (Nb$_2$O$_5$).
Figure 32. - Infrared spectrum of praseodymium oxide (Pr$_6$O$_{11}$).
Figure 33. - Infrared spectrum of samarium oxide (Sm$_2$O$_3$).
Wave number, cm\(^{-1}\)

(a) Sodium chloride absorption region.

(b) Potassium bromide absorption region.

Figure 34. Infrared spectrum of scandium oxide (Sc\(_2\)O\(_3\)).
Figure 35. - Infrared spectrum of selenium dioxide (SeO₂).

(a) Sodium chloride absorption region.

(b) Potassium bromide absorption region.
Figure 36. - Infrared spectrum of silicon dioxide (SiO₂).
Figure 37. - Infrared spectrum of silver oxide (Ag₂O).

(a) Sodium chloride absorption region.

(b) Potassium bromide absorption region.
Figure 38. Infrared spectrum of tantalum pentoxide (Ta₂O₅).

(a) Sodium chloride absorption region.

(b) Potassium bromide absorption region.
Wave number, cm$^{-1}$

(b) Potassium bromide absorption region.

Figure 39. Infrared spectrum of tellurium dioxide (TeO$_2$).
Figure 40. - Infrared spectrum of terbium peroxide ($Tb_2O_7$).
Figure 41. Infrared spectrum of thallic oxide (Ti2O3).
Figure 42. Infrared spectrum of thorium dioxide (ThO₂).
Wave number, cm⁻¹
5000 4000 3000 2500 2000 1500 1400 1300 1200 1100 1000 900 800 700 625

(a) Sodium chloride absorption region.

Wave number, cm⁻¹
500 400

(b) Potassium bromide absorption region.

Figure 43. - Infrared spectrum of thulium oxide (Tm₂O₃).
Figure 44. - Infrared spectrum of stannic oxide ($SnO_2$).
Figure 45. Infrared spectrum of titanium dioxide (TiO$_2$).
Figure 46. - Infrared spectrum of tungsten trioxide (WO₃).

(a) Sodium chloride absorption region.

(b) Potassium bromide absorption region.
Figure 47. - Infrared spectrum of uranium oxide (UO₂).
Figure 48. - Infrared spectrum of vanadium pentoxide (V₂O₅).
Figure 49. - Infrared spectrum of ytterbium oxide (Yb₂O₃).

(a) Sodium chloride absorption region.
(b) Potassium bromide absorption region.
Wave number, cm⁻¹

(a) Sodium chloride absorption region.
(b) Potassium bromide absorption region.

Figure 50. - Infrared spectrum of yttrium oxide (Y₂O₃).
Figure 51. - Infrared spectrum of zinc oxide (ZnO).

(a) Sodium chloride absorption region.

(b) Potassium bromide absorption region.
Figure 52. - Infrared spectrum of zirconium dioxide (ZrO₂).
<table>
<thead>
<tr>
<th>Chemical Compound</th>
<th>Chemical Formula</th>
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<td>Aluminum oxide</td>
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Figure 53. - Absorption regions of metal oxides other than rare earth oxides. Horizontal lines represent band widths; vertical lines indicate relative absorption of infrared radiation.
Figure 54. Absorption regions of rare earth oxides. Horizontal lines represent band widths; vertical lines indicate relative absorption of infrared radiation. (Magnesium oxide, scandium oxide, and yttrium oxide shown because of similarity to rare earth oxides.)
"The aeronautical and space activities of the United States shall be conducted so as to contribute . . . to the expansion of human knowledge of phenomena in the atmosphere and space. The Administration shall provide for the widest practicable and appropriate dissemination of information concerning its activities and the results thereof."

—National Aeronautics and Space Act of 1958

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