NATIONAL BUREAU OF STANDARDS REPORT

7829

VACUUM ULTRAVIOLET ABSORPTION SPECTRA OF OXYGEN IN LIQUID AND CRYSTALLINE ARGON AND NITROGEN

by

Arnold M. Bass

Technical Report
to
National Aeronautics and Space Administration
Washington, D. C.

NBS

U. S. DEPARTMENT OF COMMERCE
NATIONAL BUREAU OF STANDARDS

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Preface

The work described in this report was done jointly by the author and by Dr. H. P. Broida. The results of this research will be presented at the Sixth International Symposium on Free Radicals, to be held in Cambridge, England, July 1963. A manuscript based upon this report will be submitted for publication in the Journal of Molecular Spectroscopy under the joint authorship of Arnold M. Bass and H. P. Broida.

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ABSTRACT

The ultraviolet absorption spectra of oxygen have been observed in liquid and solid media at low temperature. By freezing slowly from the liquid phase, samples of crystalline argon and nitrogen were prepared with added amounts of oxygen ranging between 0.01% and 10%. The spectra were recorded photoelectrically, through fused silica windows, over the wavelength range from ~~1600~~ 1750 to ~~3100~~ 2900 A. The Schumann-Runge bands of oxygen (B $^3\Sigma_u^- \leftarrow X\ ^3\Sigma_g^-$, 1750 to ~~1980~~ 2000 A) were observed in solid and liquid mixtures of oxygen in argon or nitrogen at concentrations less than 5%, over the temperature range 60 to 90°K. The bands are broad, with half-intensity width of 150 to 550 cm^{-1} and show no structure. The bands show a slight shading toward the violet in contrast to the bands in the gas phase, ~~which~~ and are displaced toward lower frequencies by 50 to 400 cm^{-1} as compared with the positions in the gas phase. These shifts appear to be slightly dependent upon temperature. The changes in position or shape of the bands between liquid and solid or between argon and nitrogen media are remarkably small. At concentration of oxygen above 5% the Herzberg bands of oxygen (A $^3\Sigma_u^+ \leftarrow X\ ^3\Sigma_g^-$) were observed in the region 2400 to 3000 A. These bands are also broad (200 to 600 cm^{-1}) and show some partially resolved structure. No differences were observed for changes either in temperature or matrix material.

INTRODUCTION

The ultraviolet absorption of oxygen in the region 2400 to 3000 A in condensed phases has been studied previously. McLennan, Smith, and Wilhelm[1] studied the spectrum of liquid oxygen in the region 2400 to 3000 A and observed a number of bands corresponding to the "high-pressure" bands in gaseous oxygen reported by Finkelnberg and Steiner[2]. Prikhotko[3] studied the absorption spectrum of oxygen in crystals of solid solutions of $O_2 - N_2$ and $O_2 - Ar$ at liquid hydrogen temperature. In the region 2400 to 2800 A she found that the spectra of all the mixtures consist of a series of triplet absorption bands. The triplet group spacing decreased toward shorter wavelengths from about 700 cm^{-1} to about 350 cm^{-1}. These bands coincide with the absorption spectra of solid oxygen, and have a structure identical with the "high-pressure" bands. From ~~his~~ her data she calculated a potential energy curve for the upper state of the transition which is very similar to the $A\ ^3\Sigma_u^+$ state of the Herzberg bands. Romand and Granier-Mayence[4] studied the spectrum of solid oxygen (condensed from the gas phase on a surface at 20° K) in the region from 1500 to 2000 A and observed a continuous absorption, increasing in strength toward shorter wavelengths. They did not observe any discrete absorption in the region 2000 to 2400 A. Hörl[5] prepared thin polycrystalline layers of solid α-oxygen by deposition from the gas phase onto surfaces cooled to near 4.2° K by liquid helium with film thicknesses estimated to be a few hundredths of a millimeter. Between 2400 and 2700 A a number of broad and diffuse bands were observed with halfwidths of the order of 200 to

300 cm^{-1}. Hörl suggested that the bands in the solid are the Herzberg bands of oxygen, ($A\ {}^3\Sigma_u^+ \leftarrow X\ {}^3\Sigma_g^-$) shifted to shorter wavelengths by about 90 to 120 cm^{-1}. Dressler and Schnepp[6,7] studied thin layers of oxygen deposited in mixtures with argon and nitrogen on a surface at 4.2° K. Between 1750 and 2000 A 11 bands, 50 to 150 cm^{-1} broad, were observed and identified as the Schumann-Runge bands of oxygen ($B\ {}^3\Sigma_u^- \leftarrow X\ {}^3\Sigma_g^-$). By measuring the isotope shift produced with O_2^{18} they were able to establish that the bands in the solid are shifted toward longer wavelengths by roughly 300 to 400 cm^{-1}.

In the present work absorption spectra of oxygen in condensed phases have been recorded over the wavelength range 1750 to 2900 A. Both the Schumann-Runge bands of O_2 and the diffuse oxygen bands in the region of 2400 to 3000 A have been studied. Observations of the oxygen have been made in liquid and crystalline argon and nitrogen at temperatures between 60 and 90° K. Some small variations with change of parameters were observed. However the surprising result has been that the observed spectra show little change between the solid and liquid matrix and are quite similar to the spectra observed at 4.2° K.

2. EXPERIMENTAL

Solid crystalline samples were produced in a glass Dewar vessel (Figure 1), by the method described by Bolz, et al[8]. Argon or nitrogen gas, with varying concentrations of oxygen, was admitted to the central chamber at a pressure of 1 atmosphere. When liquid coolant (nitrogen or oxygen) was added slowly to the surrounding compartment it was possible

to produce, between the two fused silica windows (25 mm path length), a transparent solid. Solid argon was grown at temperatures from 60 to 82° K. This solid was similar to that described by Bolz, et al.[8], in which they measured grain-sizes of up to 4 mm. By pumping on liquid oxygen, the temperature of the coolant bath could be adjusted as desired in the range 60 to 90° K. Thus the same sample could be observed in the liquid phase as well as in the solid.

The concentration of oxygen in the sample was controlled by adjusting the ratio of oxygen to argon (or nitrogen) in the gas prior to condensation. The concentrations used in this way ranged from about 0.01% to 10% (and also 100% oxygen). However, the concentration of oxygen in the condensed material was probably different from that in the input gas. This was verified in one case where mass spectrometric analysis indicated the "input" gas composition to be 10.3% O_2, 89.7% N_2, while the composition of the gas evaporating from the condensed sample to be 4.2% O_2, 95.6%.

The temperature of the sample was assumed to be the same as that of the surrounding bath, as determined from the vapor pressure of the bath refrigerant. Boiling points and triple points of the sample obtained in this way generally agree with the known values of these fixed points. However, the assumption that the sample and the surrounding bath are at same temperature is not always reliable. Occasionally we have found that, even with vapor pressure of the bath corresponding to a temperature well below that of the triple point of argon (83.78° K), a sample of argon in

the inner chamber will remain liquid for some time. Thermocouple measurements of the liquid argon show that the liquid is not supercooled. We have not been able to explain this lack of thermal equilibrium, but it does cause some uncertainty as to the accuracy of our knowledge of the sample temperature.

A side-arm cooled by liquid hydrogen was used to trap out any substances which might otherwise condense on the outside of the fused silica windows of the sample chamber. Prior to the use of this trap, in the region below 1900 A, a continuum absorption increased with the time that coolant was in the cell.

The absorption spectra of the samples were obtained by using a vacuum grating-spectrograph of 2-meter focal length. The grating has 1200 grooves/mm, providing a reciprocal dispersion at the focal plane of about 4.2 A/mm. Spectra were obtained both photographically and photoelectrically. Photographs with relatively narrow slits (10 to 20 microns) showed that wider slits could be used with no loss of information. Thus in the photoelectric measurements, entrance and exit slits of 100 microns (6 cm^{-1} at 2600 A, 12 cm^{-1} at 1800 A) were used. The detector was an EMI 6256S photomultiplier tube with a fused silica window. The background continuum was provided by a water-cooled hydrogen discharge lamp operated at about 1 kw and a pressure of 14 mm Hg. It was found that the continuum intensity was enhanced if the hydrogen gas flowed through liquid nitrogen-cooled charcoal traps. The hydrogen lamp radiation was collimated through the sample chamber by means of a lithium fluoride lens. A thin quartz plate before the lens

prevented the discoloration of the lithium fluoride through formation of color centers by the far ultraviolet radiation from the lamp.

3. RESULTS AND DISCUSSION

In the wavelength range 1750 to 2000 A the Schumann-Runge bands of oxygen ($B\ ^3\Sigma_u^- \leftarrow X\ ^3\Sigma_g^-$) were observed in liquid and solid media ranging in temperature from about 60 to 90° K. The bands could be observed over a range of concentrations of about 0.01% to 3%. At higher concentrations there was total absorption in this region and no band structure could be observed. A typical absorption curve is shown in Figure 2(c) with comparison spectra of gas phase absorption at 90° K and at 295° K. These spectra were recorded with the same slit widths of 100 microns so that the resolution conditions are the same for all. The broadening of the bands and the disappearance of the rotational structure in solid argon are apparent. Furthermore, the bands in the solid appear to be degraded slightly toward shorter wavelengths in contrast to the red-shading of the gas phase bands. In Table I are collected representative measurements of the positions of absorption maxima of the Schumann-Runge bands. The vibrational numbering of the bands (v' v") follows that suggested by Dressler and Schnepp[6,7] and is based upon their work using O_2^{18}.

The values of relative intensity in Table I represent the fractional absorption below the baseline which results from the general continuous background. The half-intensity widths of the bands range from about 150 to about 550 cm^{-1}, and seem to be substantially greater than the widths

observed by Dressler[6] at 4.2° K. Both the half-widths and the peak intensities tend to increase and reach a maximum value in the neighborhood of v' = 8 - 10. Similar behavior is reported by Bethke[9] for the absolute integrated absorption coefficients of the Schumann-Runge band of O_2 in the gas phase, and is attributed by him to the effect of overlapping of the bands for large v'. Over the range of conditions covered in our work the half-widths appear to be somewhat smaller in the solid than in the liquid, with the maximum difference being of the order of 30%.

Wavelength measurements of the absorption maxima in Table I are uncertain to about 1 A because of the width of the bands. There are small but real differences in band positions in liquid argon, solid argon, and liquid or solid nitrogen. Comparisons of the positions of the absorption maxima in the condensed gases with the position of the gas phase band heads show shifts toward lower frequencies (longer wavelengths) from 50 to 400 cm^{-1}. The shift appears also to increase linearly with temperature with a coefficient of roughly 2 $\mathrm{cm}^{-1}/°\mathrm{K}$ in the range 60 to 90° K. The wavelength shift of the absorption maxima in condensed phases with respect to the gas-phase band heads, is large for small values of v' (approx. 400 cm^{-1}) and decreases slowly as v' increases. The decrease in the shift is somewhat more rapid for oxygen in solid argon than in liquid argon, and more rapid in liquid argon than in liquid nitrogen.

In the wavelength range 2400 to 3000 A a number of absorption bands were observed in pure liquid oxygen as well as in condensed mixtures of oxygen in argon or nitrogen. In contrast to the Schumann-Runge system,

the longer wavelength bands could be observed only in concentrations greater than a few percent (in the 25 mm path length used). The bands are broad (200 to 600 cm^{-1}) and show some partially resolved structure. Figure 3 shows typical traces in this region. By way of comparison, for sample (b) with 4% oxygen, the region of the Schumann-Runge bands, 1750 to 2000 A, was completely absorbed, while for sample (a), with 1.6% oxygen, the Schumann-Runge bands could be observed superimposed on a very strong continuous background. Table II lists the absorption maxima observed in this region. Because the bands are quite broad the positions of the maxima are not as well known as in the case of the Schumann-Runge bands, and the uncertainty in the wavelength of the maxima may be as large as 2A. No differences were observed for changes either in temperature or matrix material.

Although our instrumental resolving power was adequate for resolution of the triplets as observed by Prikhotko[3], the bands observed in the 60 to 90° K range were not fully resolved. It is possible that the lower temperature at which Prikhotko made measurements (~ 20° K) is necessary to produce bands narrow enough for complete separation. However, Hörl's spectra at 4.2° K show only broad unresolved bands, indicating that the temperature is not the only factor that controls this aspect of the spectrum. The measurements of both Hörl (15) and Prikhotko suggest that the bands in the 2400 to 2900 A region should be correlated with the Herzberg system ($A\ {}^3\Sigma_u^+ \leftarrow X\ {}^3\Sigma_g^-$) of O_2. The triplet structure of the bands in condensed media -- which is identical to that of the "high-pressure" bands -- is

presumed to arise from splitting of the $^3\Sigma_g^-$ ground state by the perturbing influence of the matrix molecules. The "high-pressure" bands have sometimes been attributed to O_4, based in part on their occurrence in systems having a high density of oxygen. Since what appears to be the same spectrum is observed for concentrations of oxygen in liquid argon of as little as 2%, as for liquid oxygen, the assignment of these bands to O_4, rather than to O_2, is open to question.

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1900 27.7

1800 30.8

Table I. Absorption maxima of the Schumann-Runge Bands of O_2 in liquid and solid argon and nitrogen

v', v"	Relative Intensity	Liquid Argon 85° K A_{vac}	Liquid Argon 85° K cm^{-1}_{vac}	Solid Argon 76° K A_{vac}	Solid Argon 76° K cm^{-1}_{vac}	Liquid Nitrogen A_{vac}	Liquid Nitrogen cm^{-1}_{vac}	Solid Nitrogen A_{vac}	Solid Nitrogen cm^{-1}_{vac}	Gas Phase* (band head) A_{vac}	Gas Phase* (band head) cm^{-1}_{vac}
2,0	1	(1985.6)	50347.4 (50362.6) 613.2							1971.9	50712.5
			598.0	[illegible]							640.7
3,0	4	1962.3	50960.6	1964.2	50911.3	1961.2	50989.2	1961.9	50971.0	1947.3	51353.2
			620.4		653.7		653.0		631.2		616.4
4,0	8	1938.7	51581.0	1939.3	51565.0	1936.4	51642.2	1937.9	51602.2	1924.2	51969.6
			589.3		583.5		598.9		608.9		592.8
5,0	18	1916.8	52170.3	1917.6	52148.5	1914.2	52241.1	1915.3	52211.1	1902.5	52562.4
			577.9	[illegible]	616.4		543.3	569	595.6		561.3
6,0	38	1895.8	52748.2	1895.2	52764.9	1894.5	52784.4	1893.7	52806.7	1882.4	53123.7
			553.9	557	534.3		563.2	539	526.6		535.90
7,0	57	1876.1	53302.1	1876.2	53299.2	1874.5	53347.6	1875.0	53333.3	1863.6	53659.6
			501.8	526	525.0		540.4	511			499.8
8,0	67	1858.6	53803.9	1857.9	53824.2	1855.7	53888.0			1846.4	54159.4
			499.7	548	538.4		483.4	525			464.5
9,0	85	1841.5	54303.6	1839.5	54362.6	1839.2	54371.4			1830.7	54623.9
			466.5	[illegible]	474.0		444.1	463			430.1
10,0	102	1825.8	54770.5	1823.6	54836.6	1824.3	54815.5			1816.4	55054.0
			417.1	[illegible]	464.0		430.1	437			387.6
11,0	106	1812.0	55187.6	1808.3	55300.6	1810.1	55245.6			1803.7	55441.6
			380.3	[illegible]	363.2		387.2	404			346.4
12,0	79	1799.6	55567.9	1796.5	55663.8	1797.5	55632.8			1792.5	55788.0
			360.5	373	380.6		333.2	372			300.4
13,0	51	1788.0	55928.4	1784.3	56044.4	1786.8	55966.0			1782.9	56088.4
				432				376			

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*L. Wallace, Astrophys. J., Suppl. No. 68, 7, 165 (1962).

Table II. Absorption maxima of oxygen in liquid oxygen, liquid argon, and solid nitrogen in the region 2400-3000 A.

Relative Intensity	Liquid Oxygen 56 to 90° K		O_2 in Liquid Argon		O_2 in Solid Nitrogen		Herzberg Bands (gas phase)*		
	A_{air}	cm^{-1}_{vac}	A_{air}	cm^{-1}_{vac}	A_{air}	cm^{-1}_{vac}	v', v"	A_{air}	cm^{-1}_{vac}
1	2952.7	33857							
2	2919.8	34239							
7	2857.9	34980					0,0	2855.9	35008
14	2798.6	35722							
11	2783.0	35922					1.0	2794.0	35780
6	2759.8	36224	2762.7	38186					
15	2740.4	36486	2745.4	36414			2,0	2737.1	36526
18	2728.7	36637							
17	2720.0	36754							
37	2689.5	37171	2683.0	37261			3,0	2684.9	37325
44	2674.0	37386							
88	2642.7	37829	2644.5	37803			4,0	2637.0	37910
78	2632.5	37975	2632.0	37983					
156			2601.2	38432					
			2591.5	38576	2588.9	38615	5,0	2593.5	38546
202			2563.2	39004					
			2555.6	39118	2553.5	39150	6,0	2554.2	39138
148			2522.2	39636	2520.6	39661	7,0	2519.3	39681
75			2488.0	40180			8,0	2488.8	40167
-			2459.3	40650			9,0	2463.3	40585
							10,0	2443.1	40920

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*L. Wallace, Astrophys. J. Suppl. No. 68, 7, 165 (1962).

③

Method

Slide 1

Dewar - Liquid O_2 or N_2 coolant ~60 - 90°K.

Argon, Nitrogen, Oxygen.

S-R bands in concentrations up to ~3%. have different contour V-shaded are shifted. Dressler + Schnepp with O_2^{18} used to number nds and to define a red shift

Lines observed in liquid CH_4 solution at b.p.

2483 Å	2535	2598, 2609	2675
40,274 cm⁻¹	39,448	38,491, 38,329	37,383

Med. Quartz Hilger
103a-O plates
High Pressure Xe lamp source

Intro

In work done previously on the spectra of small molecular species trapped in rigid matrices there has always been some uncertainty as to the interactions of the trapped species with lattice, because of the unknown nature of the polycrystalline solid formed by rapid freezing from the gas phase.

In an attempt to clarify this ~~situation~~ aspect of the problem, Dr. Broida devised a method for preparing solid~~s~~ ~~samples in which~~ matrices of the rare gases in which absorbing molecular species are dispersed, and in which the single-crystal

domains are relatively large – with dimensions of up to several mm.

We have applied this technique to a study of the Schumann-Runge bands of Oxygen, since considerable work has been done previously by Dressler and Schnepp on this system in ~~pt~~ solids formed by rapid freezing at 4°K and it was ~~hoped~~ assumed that striking changes related to the differences in temperature or crystal structure would be readily apparent.

FIGURE CAPTIONS

Figure 1. Dewar vessel for preparation of crystalline solids of argon suitable for spectroscopic studies.

Figure 2. Schumann-Runge absorption bands of oxygen ($B\ ^3\Sigma_u^- \leftarrow X\ ^3\Sigma_g^-$)

a) Room temperature, 760 mm Hg pressure

b) 90° K, 760 mm Hg pressure

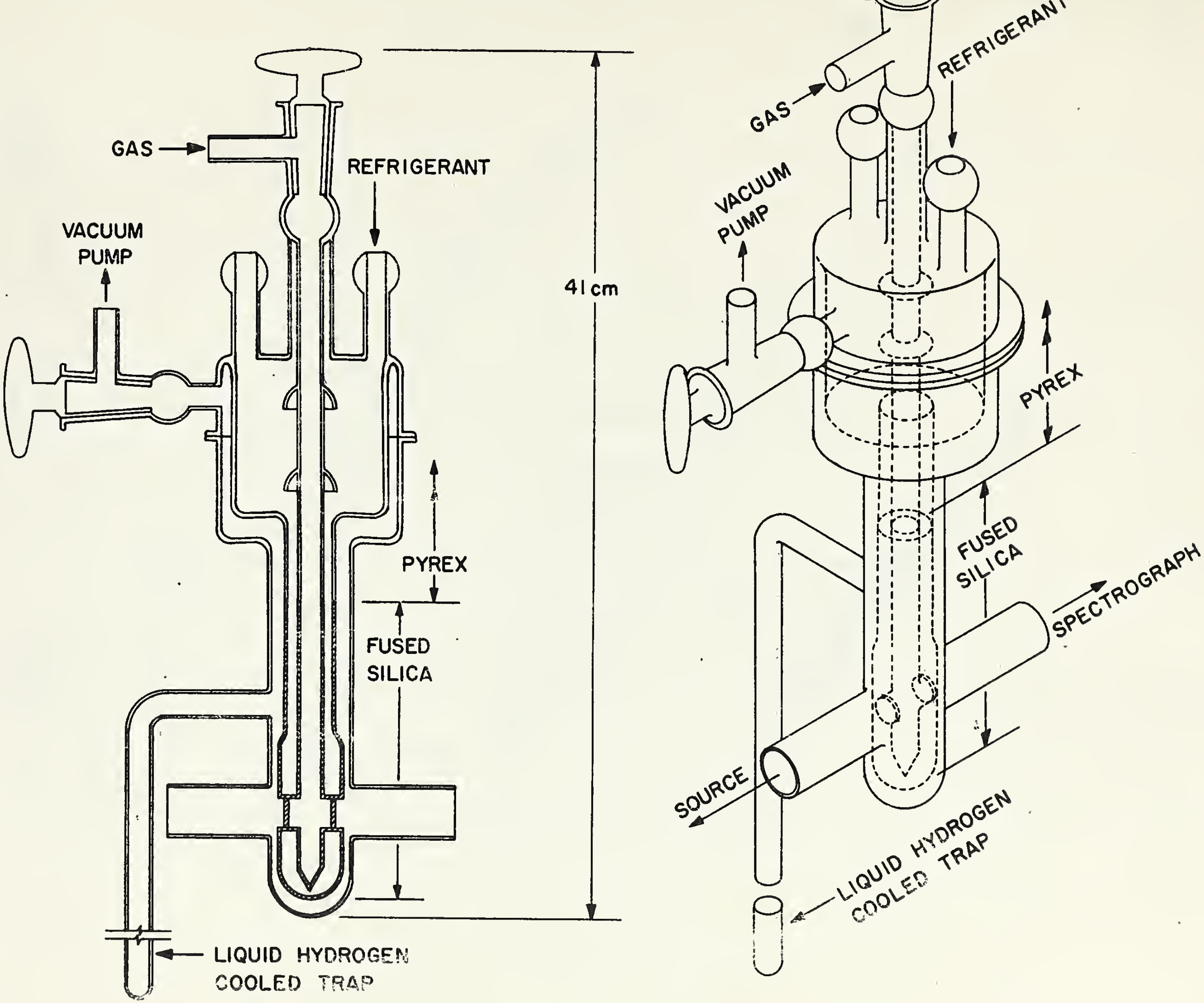
c) Oxygen (0.2%) in solid argon, 77° K; dashed curve is the transmission of the empty sample cell.

Figure 3. Absorption bands of oxygen in the near ultraviolet

a) Oxygen (1.6%) in liquid argon, 84° K

b) Oxygen (4%) in liquid argon, 84° K

c) Liquid oxygen, 84° K.


GAS
REFRIGERANT
VACUUM PUMP
41 cm
PYREX
FUSED SILICA
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GAS
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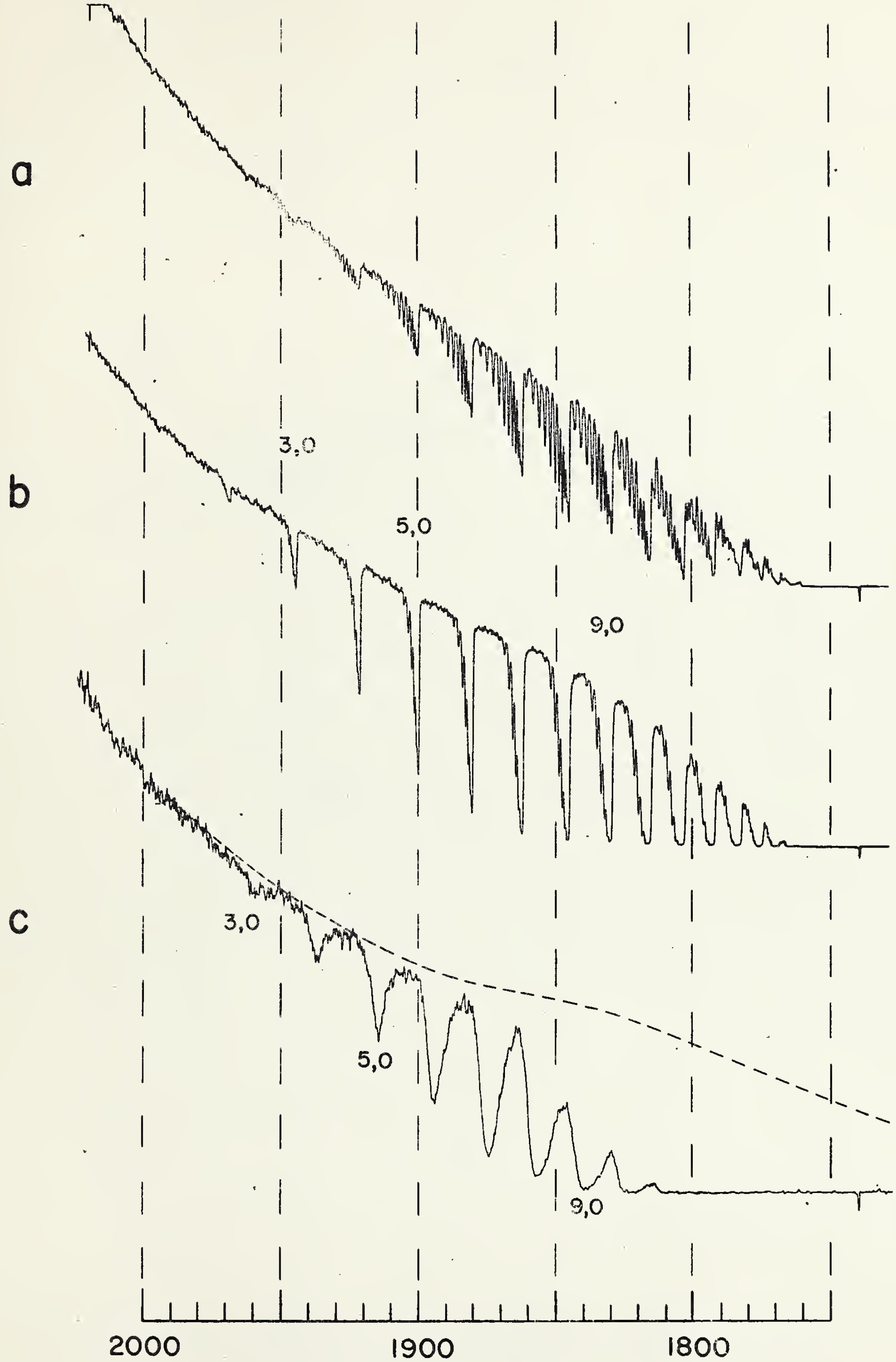
VACUUM PUMP
PYREX
FUSED SILICA
SPECTROGRAPH
SOURCE
LIQUID HYDROGEN COOLED TRAP


a
b
c
3,0
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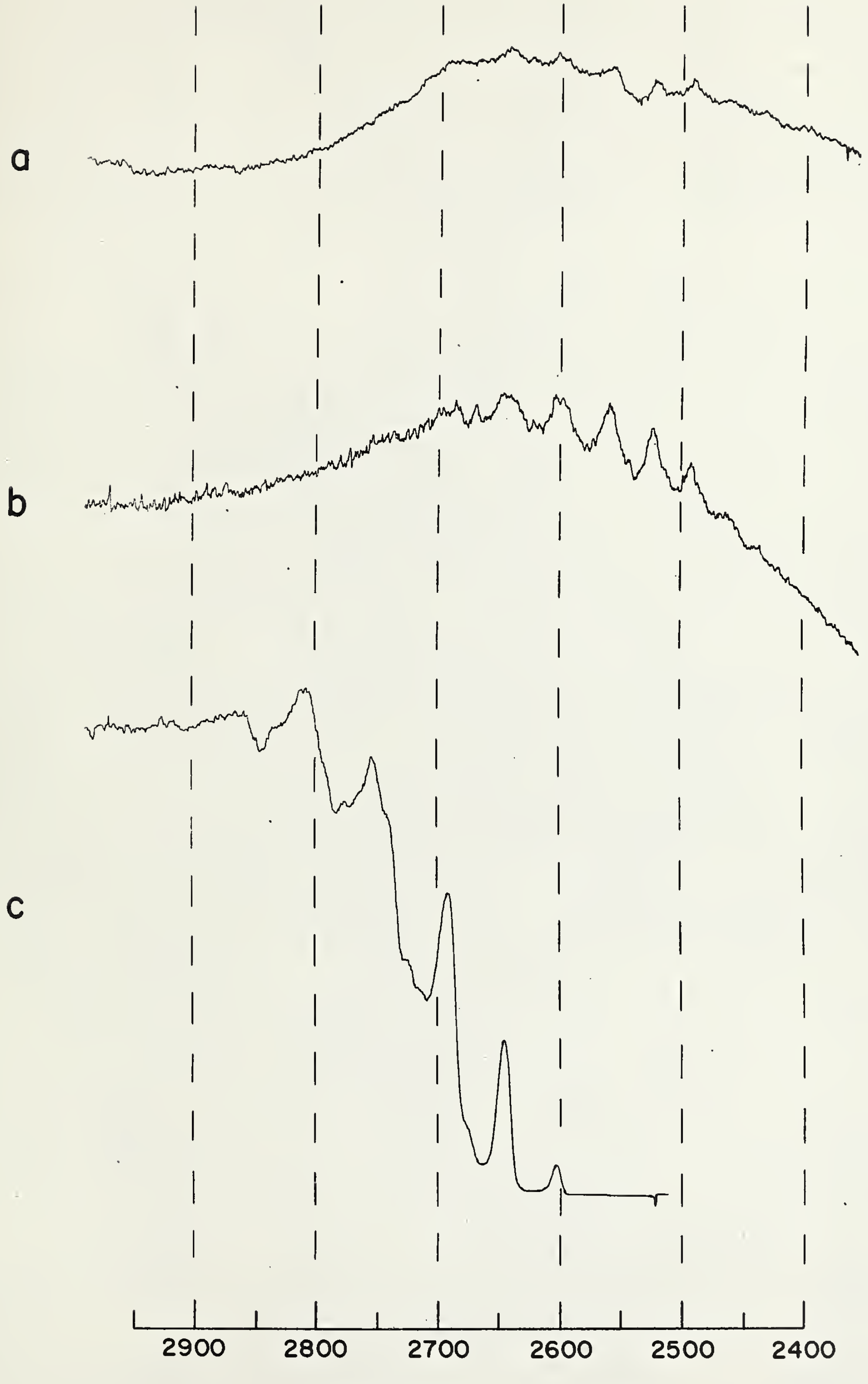
1800
WAVELENGTH (Å, vacuum)


a
b
c
2900
2800
2700
2600
2500
2400
WAVELENGTH (A, air)

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