Extra Laue Reflections of Tetraphenylethylene Crystal

M. N. Dutt

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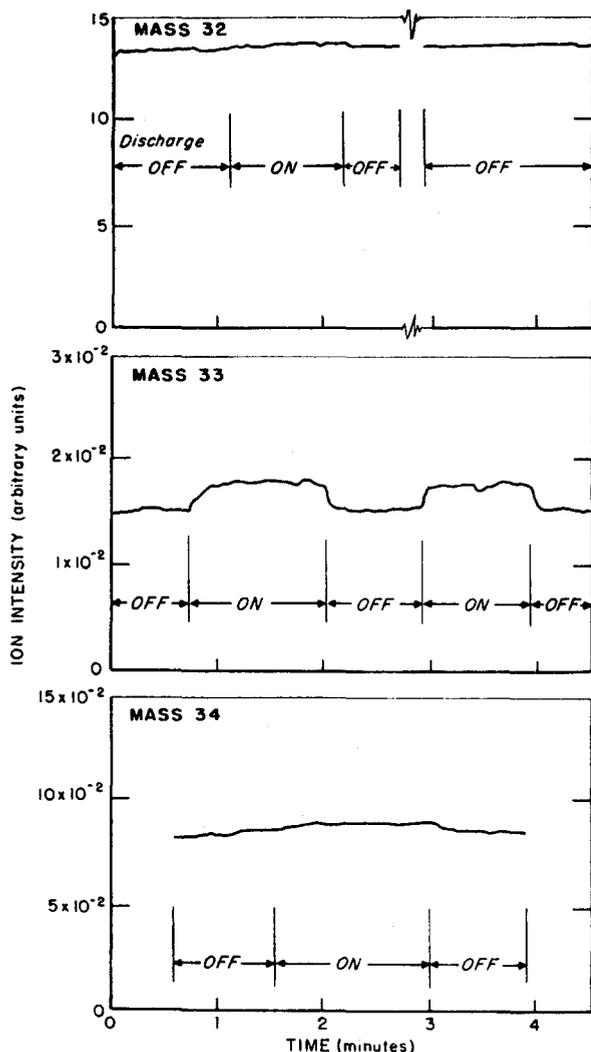Rev. Sci. Instrum. **39**, 124 (1968); 10.1063/1.1683087

FIG. 2. Ion intensities of masses 32, 33, and 34 at 60 volts electron energy for the reaction of hydrogen atoms with oxygen molecules. The ordinate scale for mass 32 should be multiplied by 1.6. Measurements were made in the sequence: mass 32, mass 33, mass 34, followed by a check on mass 32 indicated by the break in the abscissa.

jected into the center of the atomic hydrogen stream. A constriction in the glass system prevents back diffusion of oxygen molecules into the discharge tube. The reaction products enter a special mass spectrometer, previously described,[1] which employs a molecular beam sampling system. The inlet orifice is a pinhole 0.010 cm diameter in a foil 0.0025 cm thick.

The ion intensities at the oxygen isotopic masses 32, 33, and 34 were observed to determine if any HO_2 was being produced. Measurement of mass 34 intensity is necessary to rule out isotopic diffusion effects and to monitor the production of H_2O_2 which yields some HO_2^+ as an ionization fragment. At pressures of a few cm of Hg. we have found that the intensity of the mass 33 peak increased abruptly when the discharge tube was turned on, while under the same conditions the ion intensities at masses 32 and 34 remained substantially constant. In Fig. 2 are shown tracings of the records for a run at a pressure of 3.7 cm of Hg when CO_2 was mixed with the oxygen. The gas velocity in the reaction region was about 500 cm/sec. Generation of hydrogen atoms in the discharge tube in clearly correlated with a stepwise rise in the mass 33 ion intensity well above the $O^{16}O^{17}$ isotopic level. In this experiment a slight rise in the 34 peak, which might be due to H_2O_2, was ob-

served with the discharge on. The contribution of such a small amount of H_2O_2 to the mass 33 peak would, however, be less than the noise level. Increases in mass 33 intensity have similarly been observed with argon and helium serving as the inert gas mixed with the oxygen. In 16 out of 18 observations made under various experimental conditions no increase in the 34 peak was observed, while in all cases an increase, averaging 9.2 ± 2.1 percent, in the 33 peak was obtained.

The ion intensity at mass 33 cannot come from any impurity in the system as turning off the oxygen supply completely removes the 33 peak. The presence of HO_2 when operating the system at a few cm pressure is in accord with the prediction that HO_2 is formed in intermolecular collisions:

$$H+O_2+M\rightarrow HO_2+M, \qquad (1)$$
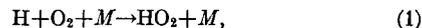
where the third body, M, removes excess energy.

Some rough values of ion intensities in one of the experiments are given in Table I. It will be observed that considerable amounts

TABLE I. Ion intensities at 60 volts electron energy. Total pressure =3 cm, $CO_2:O_2$ ratio 2:1. The OH intensity has been corrected for the contribution from H_2O.

Component	Ion intensity (arbitrary units)
H	0.45×10^{-2}
H (O_2 off)	1.42×10^{-2}
H_2	18.2×10^{-2}
OH	3.0×10^{-2}
H_2O	9.2×10^{-2}
HO_2	0.14×10^{-2}
O_2	11.9
He	6.4

of OH and H_2O are produced in the reaction. The OH is formed presumably by the reaction,

$$HO_2+H_2\rightarrow OH+H_2O, \qquad (2)$$
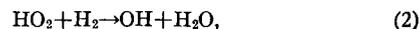
since the endothermic reaction,

$$H+O_2\rightarrow OH+H, \qquad (3)$$

at room temperature has small probability of occurring in the $\sim10^{-2}$ sec residence time in the reaction zone. In the experiments described we estimate that roughly 1 percent of the hydrogen atoms end up in HO_2 at the sampling orifice.

Experiments on the HO_2 problem are in progress and a complete report on this research will be presented later.

Extra Laue Reflections of Tetraphenylethylene Crystal

M. N. DUTT
Physics Department, Calcutta University, Calcutta, India

TETRAPHENYLETHYLENE was dissolved in alcohol which was heated till it dissolved completely, then benzene was added. The whole mixture was allowed to evaporate slowly; then the crystal of tetraphenylethylene was obtained. Morphological data indicates that it belongs to the holo axial monoclinic system with the axial ratio $a:b:c=1.213:1:1.078$, $\beta=68°20'$. The rotation photographs about the three crystallographic axes using Ni radiation show $a=11.36$ Aμ, $b=9.37$ Aμ, and $c=10.10$ Aμ. The density (ρ) was found to be 1.057. The number of molecules per unit cell was calculated to be one. A systematic study of x-ray reflections from a large number of planes, indicates that the space group of this crystal is P^2/m. The crystal was set with the needle axis (b-axis) vertical and x-ray (unfiltered Ni-radiation) falls normal to (001) face. Laue photographs were taken in this position at an interval of 5°.

The planes (312), (514), (200), (210), (400), (523), and (627) show diffuse spots. The diffuse spots appear on layer lines in the first photographs (Fig. 1) which vanish after 6° orientations.
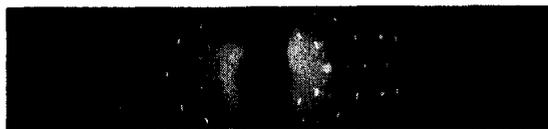

FIG. 1. X-ray (unfiltered), Ni radiation passes through C axis, b axis vertical. The arrangements of diffuse spots on layer lines are to be noted.

This shows that extra Laue spots are of low intensity and of thermal origin, according to the Faxen Waller theory. When exposed to cobalt radiation, some of the extra spots are relatively displaced (Fig. 2), but the others remain practically, in the same
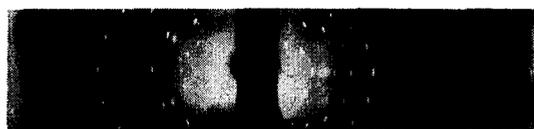

FIG. 2. The same position as the previous one. X-ray co-radiation passes, diffuse spots are displaced slightly to one side.

position. The author expresses his grateful thanks to Professor S. N. Bose, Head of the Department of Physics, for his valuable advice.

The Solubility Diagram and the Temperature-Composition Diagram at Atmospheric Pressure of the Liquid System Ozone-Oxygen*

HANS JOACHIM SCHUMACHER
Instituto de Investigaciones Facultad de Quimica Universidad Nacional de La Plata, Argentina
(Received May 15, 1953)

IT is well known that liquid oxygen and liquid ozone do not dissolve each other in all proportions.[1] If for example a gaseous mixture of oxygen with 20 percent of ozone is liquified at the temperature of liquid air two phases will appear: a light blue one, representing the solution of some percent of ozone in oxygen and a dark violet one of a much higher density, representing the solution of some percent of oxygen in ozone.

After Riesenfeld[2] the oxygen phase consists at liquid air temperature of approximately 70 percent of oxygen and 30 percent of ozone and the ozone phase of approximately 70 percent of ozone and 30 percent of oxygen. However, these figures represent only a very rough estimation because there exist no analytical determinations of the concentrations in question.

The critical temperature of solubility is said to be at $-158°C$; but there are many reasons to believe that this is a too high value. The experimental technic applied by Riesenfeld did not permit to determine the correct temperature. The temperature measured

TABLE I. The ozone concentrations of the "oxygen" and the "ozone" phase at different temperatures.

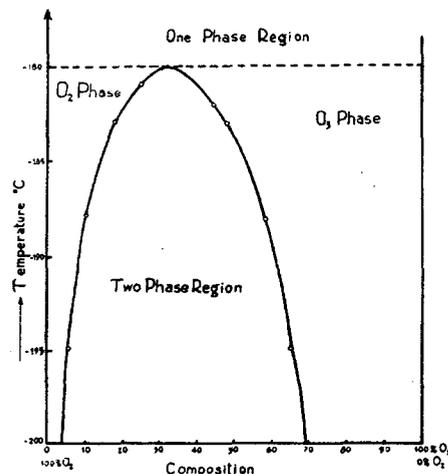|	Mole percent O_3	
$T°C$	O_2 phase	O_3 phase
−195	5.3	65
−188	10.0	58
−183	18.0	48
−182	...	44.5
−181	24.5	...

FIG. 1. The solubility diagram of the system ozone-oxygen.

by him corresponded to the temperature of a liquid considerably superheated. In fact the critical temperature derived from the complete solubility diagram is found to be at a much lower temperature, at -180 ± 0.2 C.

In order to be able to construct the entire solubility diagram of the system ozone-oxygen we determine the concentrations of the components of the two phases at different temperatures. As liquids of the thermostat we used for the measurements, we had liquid nitrogen, liquid oxygen, a mixture of liquid nitrogen and oxygen and liquid methane cooled down by liquid oxygen.

The experimental results are represented in Table I and Fig. 1.

The temperature-composition diagram of ozone-oxygen was not known until now. We measured it using a thermostat which kept the temperature in the range between the boiling points of oxygen and ozone for several hours within a tenth of a degree constant. The apparatus for measuring the ozone concentrations of the liquid and the gas phase at different temperatures and at the constant pressure of one atmosphere, was constructed in such a way that we could be certain that the two phases were always in perfect equilibrium.
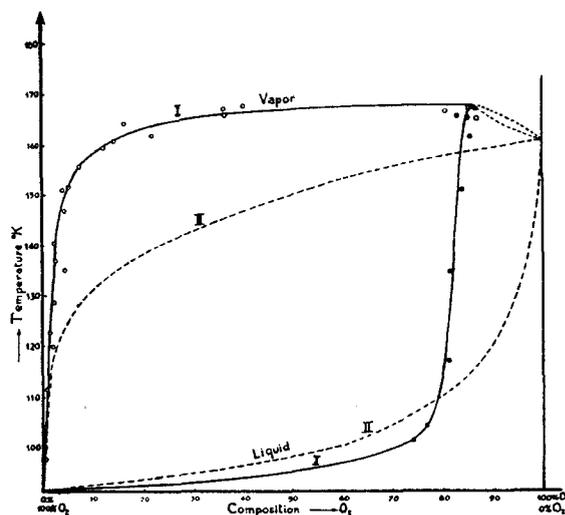

FIG. 2. The temperature-composition diagram of the system ozone-oxygen at atmospheric pressure. ——— experimental curve; – – – – theoretical curve; O measured values of the gas phase; ● measured values of the liquid phase.