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VAPOR PRESSURES. I. PARA-DICHLOROBENZENE; GLYCOL DIACETATE

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In keeping with the policy of giving to chemistry students some introduction to the principles and methods of research, a program of vapor pressure studies on various substances has been projected in our laboratory. Since there are many substances for which vapor pressure data are not available, this program offers abundant opportunity to a large number of students for each to do a little independent research with the hope that the data obtained may sometime prove useful.

The first problem that required attention in connection with this projected series of studies was, of course, the setting up of a suitable apparatus within the limitations of our equipment. The work reported here has been largely on the developing of that apparatus--which is not yet totally satisfactory. Just enough actual determinations have been made to prove the practicability of the apparatus and to show the desirability of certain modifications.

It has not been our aim to achieve the highest possible accuracy, but rather to develop a practicable technique within the ability of the under-graduate student and capable of yielding usable data. It is to be expected that impurities in the materials tested may sometimes introduce greater errors than those that might be eliminated by refinements of apparatus or procedure. It sometimes is not desirable to remove those impurities even if it is possible, because the vapor pressure of a material as it is used may be more important than that of the strictly pure substance.

Of the two substances on which we have taken data, p-dichlorobenzene and glycol diacetate, the former was selected because of its wide use as an insecticide, for which its volatility is an important characteristic. The latter was chosen simply because some of the compound was available and a preliminary search of the literature did not bring to light any data on its vapor pressures. Further investigation, however, disclosed vapor pressures in the International Critical Tables for that compound at 10° intervals from 100°C to 190°C. The critical tables give a formula for the vapor pressure of p-dichlorobenzene for the range 30°C to 50°C.

The apparatus used in these studies consists of a 5-mm glass tube bent into a long, narrow U with one arm sealed and the upper end of the sealed arm inclosed in a jacket. Before the end of the tube was sealed, mercury was introduced to a height of about 40 cm. in each arm and the test material was introduced into one arm above the mercury. That tube arm was then connected to a vacuum pump and the material was heated enough to produce boiling under the vacuum. While the material was still boiling the tube was sealed off, leaving the space above the mercury filled with liquid and vapor.

After the tube was sealed the upper portion was enclosed in a glass jacket about 4 cm. in diameter. The jacket was filled with glycol which was kept stirred by a stream of air bubbles escaping from a jet into the lower end of the jacket. A thermometer placed with its bulb just above the level of the mercury in the vapor pressure tube was used to determine the temperature. Frequent checking of the temperatures at different

levels indicated thorough mixing of the glycol. Heating was done by a controlled electric current in a tight coil of bare chromel wire suspended directly in the glycol so that it lay in a loose spiral against the inside of the jacket wall.

Measurements were made first at room temperature or near it, then at progressively higher temperatures. Except for a reading at room temperature the apparatus was always heated a little above, and then allowed to cool slowly to, the desired temperature, where it was held for a time to allow equilibrium to be established. Readings were made of upper and lower mercury levels, level of liquid above the mercury, barometric pressure, and temperature. At temperatures below the melting point of p-dichlorobenzene that substance remained in the highest part of the tube where it was frozen before the jacket was placed around the tube.

The upper mercury level was corrected for the column of liquid floating on it and for expansion of the heated portion of the mercury column. Then the vapor pressure was found by subtracting the corrected height of the upper mercury level from the sum of the barometric height and the height of the lower mercury level.

The p-dichlorobenzene used was that taken directly from a stock bottle with no attempt at purification. The glycol diacetate was obtained by twice fractionating a material of somewhat uncertain purity, discarding each time that portion which distilled over with rising temperature and reserving the middle fraction that distilled at nearly constant temperature. There were indications, however, that some decomposition accompanied the distillation, among them the fact that the final distillate with a range of half a degree showed a boiling point two degrees below that range, so that the purity of the final product is still open to serious question. There is also the question whether decomposition occurs in the vapor pressure tube.

The results of these rather preliminary measurements are given in the following table.

Vapor pressure data

Temperature Centigrade	Vapor pressure in millimeters: p-Dichloro-benzene	Glycol diacetate
25	.31	.24
35	.55	.31
45	.77	.63
55	1.17	.96
65	1.94	1.29
75	2.98	1.91
85	4.56	2.99
95	6.37	4.27
105	9.54	6.24
110		7.27
115	13.67	
120		10.39
125	18.25	
135	19.50	
140		14.17

These results are only tentative, but they do serve to show the order of magnitude of the vapor pressures of these substances over a temperature range of something more than 100°C. It is hoped that further studies will confirm or correct them with greater certainty. In any case, a beginning has been made on the project of adding to the available data on vapor pressures, and it is confidently expected that the accumulation of that data by the method described will proceed at an accelerated pace.