

Fig. 1.—Distribution patterns between borate buffer at pH 7.8 and pentasol: A, 3450 units of streptothricin; B, 4095 units of S VI; C, a mixture of 1735 units of streptothricin and 1810 units of S VI.

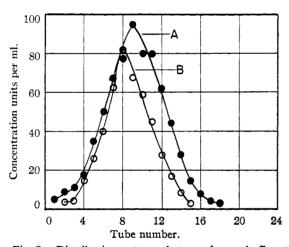


Fig. 2.—Distribution patterns between borate buffer at pH 7.6 and pentasol: A, 4650 units streptomycin; B, 3440 units streptothricin.

coefficient of 0.54. Streptomycin showed a peak at tube 21 with a corresponding distribution coefficient of 6.38. The distribution curves for streptothricin, streptomycin, and a mixture of these two antibiotics are shown in Fig. 3. It is interesting to note that the distribution of streptomycin in the bicarbonate buffer is considerably different from that in borate buffer at the same pH (7.6), while the distribution of streptothricin is the same in both systems. This might be explained on the basis of interaction of borate with the sugar moiety of streptomycin. The importance of using more than one system to characterize an antibiotic by the distribution analysis technique is clearly demonstrated by these results.

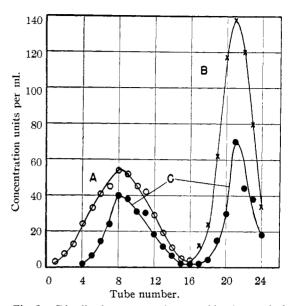


Fig. 3.—Distribution patterns between bicarbonate buffer and pentasol: A, 3630 units streptothricin; B, 4933 units streptomycin; C, a mixture of 2160 units of streptothricin and 2427 units streptomycin.

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This method is being investigated further as a possible means of characterizing other antibiotics produced by actinomycetes.

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The trans Isomer of 4-Amino-4'-hydroxy- $\alpha$ , $\alpha'$ -

## diethylstilbene By Philip Weiss

In a publication from this Laboratory,<sup>1</sup> the preparation of 4-amino-4'-hydroxy- $\alpha$ , $\alpha$ '-diethylstilbene was described and its melting point was given as 155–156°. From a crude sample which was prepared at that time I have now obtained by several recrystallizations from methanol a compound which melts at 180–183°.<sup>2</sup>

M. Rubin and H. Wishinsky, This Journal, 66, 1948 (1944).
 All melting points are uncorrected.

Anal. Calcd. for  $C_{18}H_{21}ON$ : N, 5.24. Found: N, 5.20.

This higher melting substance was converted into 4,4'-dihydroxy- $\alpha,\alpha'$ -diethylstilbene as follows: A solution of 50 mg. of it in dilute sulfuric acid was diazotized with sodium nitrite in the usual manner. To this solution 10 ml. of 18 N sulfuric acid was added and the resulting solution was refluxed for half an hour. The crude material obtained was recrystallized from dilute methanol; yield 0.013 g.; m. p. 169.5–170.5°. This substance mixed with authentic trans 4,4'-dihydroxy- $\alpha,\alpha'$ -diethylstilbene showed no depression of the melting point.

Thus it appears that the recently isolated higher melting substance is the *trans* isomer and that the previously described lower melting substance was probably the *cis* isomer.

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## Physical Adsorption from Mixtures of Gases. II. Oxygen-Argon on Silica Gel at 0°1

By Locke White, Jr., and Charles H. Schneider

Lambert and Heaven<sup>2</sup> have reported experiments which led to the conclusion that, in adsorption from binary mixtures by silica gel at 0°, "the composition of the adsorbed phase is greatly affected by the order in which the two gases reach the gel surface." In their apparatus, the gaseous mixture was not circulated through the bed of adsorbent and the rate of approach to equilibrium was presumably limited by diffusion; however, their diffusion paths were short and they allowed very long periods for the attainment of equilibrium. In view of the unexpected nature of their results, it seemed worthwhile to repeat some of their measurements, using forced circulation.

## Experimental

Apparatus and Materials.—The apparatus was that of a previous investigation. For this use, an ice-bath was substituted for the temperature-control system described. Gas analysis was accomplished by readings of total pressure on a manometer and of partial pressure of oxygen by Pauling's magnetic device. Oxygen, argon and helium (for calibrating the dead space in the sample cell) were prepared by the procedures used previously.

Lambert and Heaven used a sample from the Silica Gel Corporation of America. This company is no longer in existence, having been taken over by the Davison Chemical Corporation, which has no record of the sample supplied to Lambert and Heaven, but which contributed a

sample of its 14-20 mesh silica gel, grade code 08, for this investigation. Approximately 40 g. was used.

**Procedure.**—Before each measurement, the surface of the sample was cleaned by evacuation to less than  $10^{-3}$  mm. of mercury at  $150^{\circ}$  for at least an hour. After the sample was cooled to  $0^{\circ}$ , a measured quantity of one component was added and allowed to equilibrate, and the pressure was recorded. Then an approximately equal quantity ( $\pm 2\%$ ) of the other component was added and allowed to equilibrate, and both total pressure and partial pressure of oxygen were recorded. The surface of the sample was then cleaned and the procedure was repeated, except that the components were added in the reverse order. A pair of such runs was ordinarily made each day.

To avoid the possibility that a regular pattern as to which gas was added first was giving deceptive results, an extra pair of runs (nos. 9 and 10 in Table I) was made with the daily pattern reversed.

In none of the first ten runs was there any attempt to "wash out" the gel in the manner which Lambert and Heaven found to be essential for reproducibility. In run no. 11, on the contrary, the "washing out" was performed. That is, an unmeasured quantity of argon was adsorbed to equilibrium on the clean surface at 0° and was then removed by heating and evacuation. Another unmeasured quantity was similarly adsorbed and removed. Then the adsorption of argon was measured in the usual way.

## Results

The adsorption data obtained are recorded in Table I. Because no data have been omitted, it is possible to tell from the table which adsorbate was in contact with the gel immediately preceding any run under consideration.

TABLE I
ADSORPTION DATA

|     |                    | ADS             | SURPLION         | DATA               |                   |               |
|-----|--------------------|-----------------|------------------|--------------------|-------------------|---------------|
|     | Volume<br>ads      | -Oxygen-        |                  | Volume<br>ads.,    | — Argon——         |               |
| Run | ml. at<br>S. T. P. | Pressur<br>Obs. | e, mm.<br>Calcd. | ml. at<br>S. T. P. | Pressure,<br>Obs. | mm.<br>Calcd. |
| 1   | 5.77               | 42.0            | 42.4             |                    |                   |               |
|     | 5.23               | 40.0            | 38.4             | 5.40               | 40.5              | 39.2          |
| 2   |                    |                 |                  | 5.88               | 42.5              | 42.7          |
|     | 5.35               | 40.0            | 39. <b>3</b>     | 5.43               | 38.5              | 39.4          |
| 3   | 10.33              | 75.5            | 75.9             |                    |                   |               |
|     | 10.31              | 76.0            | 75.8             | 10.11              | 72.5              | 73.4          |
| 4   |                    |                 |                  | 10.89              | 78.0              | 79.1          |
|     | 10.37              | 76.0            | 76.2             | 9.97               | 72.5              | 72.4          |
| 5   | 16.91              | 123.5           | 124.3            |                    |                   |               |
|     | 15.50              | 114.0           | 113.9            | 15.73              | 113.0             | 114.2         |
| 6   |                    |                 |                  | 17.04              | 123.0             | 123.7         |
|     | 15.54              | 114.0           | 114.1            | 15.48              | 113.0             | 112.4         |
| 7   | 24.64              | 180.5           | 181.0            |                    |                   |               |
|     | 22.60              | 166.0           | 166.1            | 22.67              | 166.5             | 164.6         |
| 8   |                    |                 |                  | 24.72              | 179.5             | 179.5         |
|     | 22.80              | 166.0           | 167.6            | 22.62              | 166.5             | 164.2         |
| 9   |                    |                 |                  | 20.61              | 149.0             | 149.2         |
|     | 19.19              | 142.0           | 141.0            | 18.89              | 137.5             | 137.1         |
| 10  | 20.91              | 154.0           | 153.7            |                    |                   |               |
|     | 19.14              | 142.0           | 140.7            | 19.18              | 137.5             | 139.2         |
| 11  |                    |                 |                  | 17.13              | 123.5             | 124.4         |
|     |                    |                 |                  |                    |                   |               |

The measured points of each run, whether for oxygen or for argon, are so nearly superposed that it is impractical to distinguish between them on a graph. To show how nearly the volume

<sup>(1)</sup> This research was supported jointly by the Office of Naval Research and the Institute-Sponsored Research Fund of Southern Research Institute.

<sup>(2)</sup> B. Lambert and H. S. Heaven, Proc. Roy. Soc. (London), ▲153, 584 (1936).

L. White and C. H. Schneider, THIS JOURNAL, 71, 2593 (1949).
 L. Pauling, R. E. Wood and J. H. Sturdivant, ibid., 68, 795 (1948).

<sup>(5)</sup> B. Lambert and A. M. Clark, Proc. Roy. Soc. (London), ▲117, 183 (1927)