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### Specific Surface Area of Arsenic Sulphide

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Low temperature gas adsorption measurements have been carried out on two samples of arsenic sulphide at different degassing temperatures. The adsorption data have been analysed by BET method. For the first time in literature the adsorption data of sulphides have been analysed on the basis of  $t$  and  $\alpha_s$  methods to calculate the specific surface area of the samples. An explanation has been offered regarding the shape of the  $t$  and  $\alpha_s$  curves.

IN the present study low temperature gas adsorption method<sup>1</sup> has been used to measure the surface areas of arsenic sulphide samples, prepared by two different methods. The choice of the system, arsenic sulphide is based on its use as a pigment, catalyst, lubricant and pharmaceutical powder. In principle, the method involves the determination of  $V_m$  (monolayer capacity), the volume of gas necessary to form a complete adsorbed layer of one molecule thickness on the surface to be measured.

Two samples A and B were prepared for the present studies. Sample A was obtained by dissolving arsenious oxide (10 g) in 100 ml of conc. HCl at 45-50°. The solution was diluted to 1000 ml by adding water in small portions while maintaining the temperature in the range 45-50°. Hydrogen sulphide was passed for about 13-15 min with occasional shaking of the container. The precipitate formed was allowed to settle for 2 hr, filtered, washed repeatedly with water to remove acid completely and then dried for 2 hr at 100-104°. Sample B was obtained by mixing sodium arsenite (500 ml, 0.5M) and sodium hydrosulphide (1000 ml, 0.5M) at room temperature. The solution was neutralized by adding 5N HCl drop by drop and stirring the solution vigorously. The precipitate was allowed to settle, filtered, washed until free from acid and dried at 100-104° for 2 hr.

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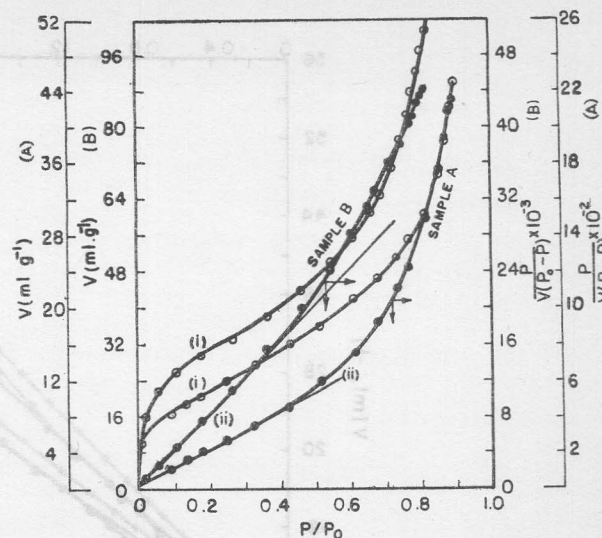


Fig. 1 — Adsorption of  $N_2$  on samples A and B of arsenic sulphide [(i) Adsorption isotherm curve; (ii) BET plot]

The adsorption isotherms were determined using the volumetric method of Emmett<sup>2</sup>. The usual procedure followed for the measurements was described in an earlier communication<sup>3</sup>. The adsorption measurements were carried out on samples A and B after degassing at 27°, 60° and 110°. The experiments were carried out at the temperature of the boiling point of nitrogen, -196°.

Fig. 1 (curve i) represents the adsorption isotherm of  $N_2$  gas on the solids A and B at the degassing temperature of 27°. The isotherms in both the cases are of type II of Brunauer's classification. The BET plots for both the samples are shown by curve (ii). The deviation of the BET plot from linearity was observed at a relative pressure of 0.35 and 0.4 respectively for samples A and B. Identical isotherms and BET plots were obtained for both the samples at other degassing temperatures. The surface area values obtained at different degassing temperatures of the samples are given in Table 1. The BET constant,  $C$ , was found to be 39 for sample A and 101 for sample B.

In order to know the nature of the powders, an attempt was made to analyse the adsorption data on the basis of the  $t$  method of de Boer<sup>4</sup> and  $\alpha_s$  method of Sing<sup>5</sup>. The survey of literature shows that the  $t$  and  $\alpha_s$  methods were used to analyse the adsorption data of oxides, carbons, silica, alumina and other powders but not of sulphides. In Fig. 2 are given the  $t$  and  $\alpha_s$  plots of samples A and B. Similar  $t$  and  $\alpha_s$  plots were obtained at other degassing temperatures. The areas obtained from the  $t$  and  $\alpha_s$  methods are given in Table 1.

The surface areas of the samples obtained by BET,  $t$  and  $\alpha_s$  methods at different degassing temperatures are in good agreement. From the values obtained at three degassing temperatures, it is clear that degassing at room temperature itself removes the condensable gas or vapours and surfaces are free from foreign gases. Table 1 shows that the surface structure of the solid is not affected by higher degassing temperatures.

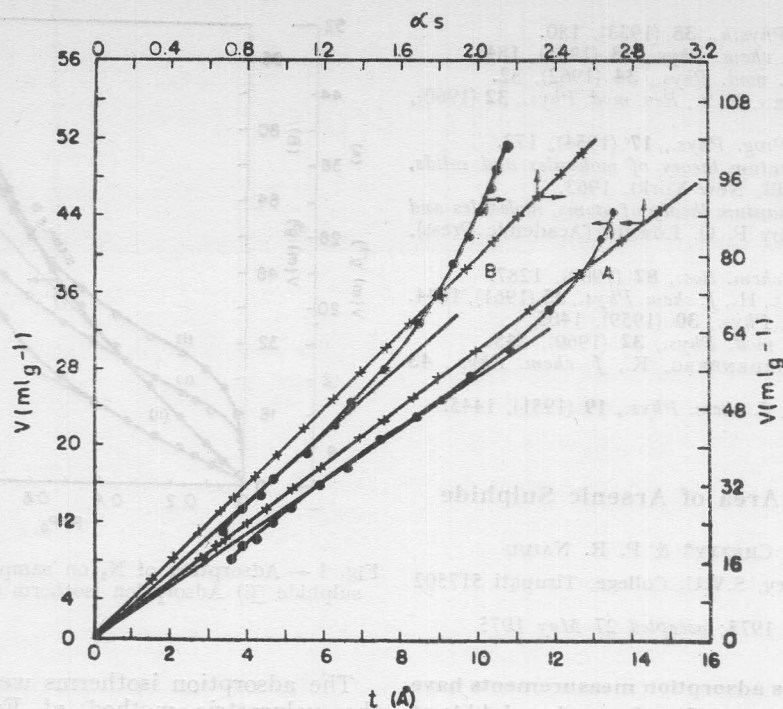

 Fig. 2—V versus  $t$  and  $\alpha_s$  plots for samples A and B of arsenic sulphide

 TABLE 1—SURFACE AREA VALUES BY BET,  $t$  AND  $\alpha_s$  METHODS

Degas- sing temp. (°C)	Specific surface area (m <sup>2</sup> g <sup>-1</sup> )					
	SBET		$t$		$\alpha_s$	
	SAMPLE A	SAMPLE B	SAMPLE A	SAMPLE B	SAMPLE A	SAMPLE B
27	42.0	42.7	44.2	113.2	109.5	114.8
60	40.7	41.3	42.5	108.9	107.6	114.8
110	40.7	41.8	44.2	108.9	109.5	114.8

The  $\alpha_s$  plot is linear over the entire relative pressure range and passes through the origin. This indicates that the sample is non-porous or contains pores of width more than 200 Å (ref. 5). The  $t$  plot which also passes through the origin is linear up to a certain range only. At  $t$  values of 10 Å for sample A and 6 Å for sample B, the experimental curve deviates upwards from linearity indicating that more nitrogen is taken up than corresponding with multilayer adsorption. This indicates that the capillary condensation starts setting in at this point in the intermediate pores<sup>6</sup>.

The agreement of surface area of the samples between  $t$  and  $\alpha_s$  methods and BET method indicates that the former two methods can be used to analyse the adsorption data of the present sulphides.

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#### Photo-stabilization of Butyl Rubber in Solid State by Phenyl Allyl Mercury

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**The incorporation of 0.5% PAM or more in the matrix of butyl rubber film irradiated by 366 nm exercises a protective influence on the photo-oxidative degradation of butyl rubber.**

**T**HE kinetics of photo-oxidative degradation of butyl rubber in the presence of oxygen and its photo-stabilization by the addition of 0.1% (wt/wt) phenyl allyl mercury<sup>1,2</sup> (PAM) were reported earlier<sup>3</sup>. In the present note, the physico-chemical studies have been conducted to find out an optimum limit of [PAM] which would impart the saturation photo-stabilization to butyl rubber in solid state.