

Instead of helium, oxygen-free nitrogen might be used.

The gel tube \* was 100 mm long and had an outer diam. of 4 mm. It was packed to a length of 10 mm with the carbon oxide indicating gel (silica gel impregnated with a mixture of palladium sulphate and ammonium molybdate, *cf.* Ref.<sup>1</sup>). On the inlet side of the indicating layer was placed a 40 mm length and on the outlet side a 10 mm length of silica gel.

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\* We have used tubes manufactured and sold by LKB-Produkter Fabriksaktiebolag, Stockholm.

## The Crystal Structure of Pd<sub>3</sub>P

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An X-ray investigation of the palladium-phosphorus system was recently begun at this Institute. Preliminary results show that the system is far more complex than is indicated in the equilibrium diagram given by Wiehage *et al.*<sup>1</sup> It is especially noteworthy that at least four intermediate phases seem to exist in the range 0–25 atom % phosphorus. The existence of a phase near the composition Pd<sub>3</sub>P, as observed by Wiehage *et al.*<sup>1</sup>, was confirmed by our results. A single-crystal investigation showed that Pd<sub>3</sub>P has the cementite

(*D* O<sub>11</sub>) structure. The homogeneity range, determined from alloys quenched from 740°C, was found to extend from approximately Pd<sub>3</sub>P<sub>0.75</sub> to Pd<sub>3</sub>P<sub>1.0</sub> with the following lattice parameters (accuracy ± 0.05 %): phosphorus-rich limit  $a = 5.980$  Å;  $b = 7.440$  Å;  $c = 5.164$  Å and palladium-rich limit  $a = 5.645$  Å;  $b = 7.558$  Å;  $c = 5.071$  Å.

The crystal structure was determined by examination of single-crystals selected from an alloy with the approximate composition Pd<sub>3</sub>P<sub>0.95</sub>. 62  $F(h0l)$ -values and 63  $F(0kl)$ -values were recorded with niobium-filtered MoK-radiation in a multiple-film Weissenberg camera with thin iron foils between successive films. Atomic parameters were refined from electron density projections and difference syntheses. Fourier summations and structure factor calculations were made on the electronic digital computer BESK with programs devised by Åsbrink *et al.*<sup>2</sup>

The final  $R$ -value for the recorded  $F(h0l)$ -values was 0.087, applying an isotropic temperature factor with  $B = 0.72$  Å<sup>2</sup>; the corresponding figures for the  $F(0kl)$ -values were  $R = 0.088$ ,  $B = 0.67$  Å<sup>2</sup>. (The five largest  $F(0kl)$ -values, which were obviously weakened owing to extinction, were omitted in the final stages of refinement).

The following structural data for Pd<sub>3</sub>P<sub>0.95</sub> were obtained: Space group  $Pnma - (D_{2h}^{16})$ ;  $Z = 4$ . (Table 1.)

The standard deviations quoted were calculated using Cruickshank's formula<sup>3</sup>.

Interatomic distances are listed in Table 2.

It is of interest to note that in addition to Pd<sub>3</sub>P described above, the compounds Pd<sub>3</sub>Si and Pd<sub>3</sub>B also possess the cementite structure<sup>4,5</sup>. A case of isomorphism between a boride, a silicide and a phosphide of the same metal is thus demonstrated.

Further reports of the work on palladium phosphides will be published later in this journal.

Table 1. Structural data for Pd<sub>3</sub>P<sub>0.95</sub>.

$$a = 5.947 \text{ \AA}; b = 7.451 \text{ \AA}; c = 5.170 \text{ \AA}; U = 229.1 \text{ \AA}^3$$

		$x$	$\sigma(x)$	$y$	$\sigma(y)$	$z$	$\sigma(z)$
8 Pd <sub>I</sub>	in 8( <i>d</i> )	0.1783	0.0002	0.0636	0.0003	0.3373	0.0003
4 Pd <sub>II</sub>	in 4( <i>c</i> )	0.0264	0.0004	—	—	0.8700	0.0004
4 P	in 4( <i>c</i> )	0.8834	0.0011	—	—	0.4550	0.0011

Table 2. Interatomic distances in  $\text{Pd}_3\text{P}_{0.95}$  (Å). (Distances shorter than 3.5 Å listed).

	Pd <sub>I</sub>	Pd <sub>II</sub>	P
Pd <sub>I</sub>	2.77 <sub>8</sub> , 2.85 <sub>1</sub> , 2.88 <sub>9</sub> (2), 3.10 <sub>7</sub> (2)	2.83 <sub>9</sub> , 2.92 <sub>5</sub> , 2.92 <sub>8</sub> , 2.93 <sub>5</sub> , 3.21 <sub>0</sub>	2.30 <sub>9</sub> , 2.39 <sub>4</sub> , 2.59 <sub>8</sub>
Pd <sub>II</sub>	2.83 <sub>9</sub> (2), 2.92 <sub>5</sub> (2) 2.92 <sub>8</sub> (2), 2.93 <sub>5</sub> (2) 3.21 <sub>0</sub> (2)	3.22 <sub>2</sub> (2)	2.30 <sub>9</sub> , 2.30 <sub>8</sub> 3.14 <sub>2</sub>
P	2.30 <sub>9</sub> (2), 2.39 <sub>4</sub> (2) 2.59 <sub>8</sub> (2)	2.30 <sub>8</sub> , 2.30 <sub>8</sub> , 3.14 <sub>2</sub>	—

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## Studies on Rennin

### V. Chromatographic Purification of Prorennin

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The first paper in this series<sup>1</sup> described the preparation of partly purified prorennin obtained from dried calf stomachs by extraction with a 2% solution of sodium bicarbonate. After clarification with alu-

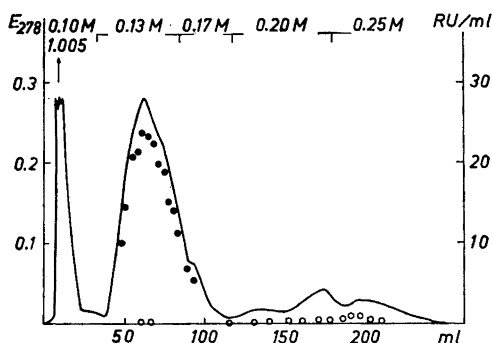


Fig. 1. Chromatography of partly purified prorennin on DEAE-cellulose involving stepwise elution with phosphate buffers of pH 5.8–5.7; 0.10 M, 0.13 M, 0.17 M, 0.20 M, and 0.25 M. Load 20 mg, column 0.9 × 11 cm. Flow rate 4 ml/h; fractions of 2 ml. O preformed milk-clotting activity (RU/ml). ● milk-clotting activity after activation. Experimental points have been omitted from the extinction curve of  $E_{278}$  for the sake of clarity.

minium sulphate and disodium hydrogen phosphate, the extract was precipitated twice by saturation with sodium chloride. Paper electrophoresis indicated that approximately 50% of the protein in the second precipitate was prorennin, but attempts at further purification by salt fractionation failed.

The investigations have been continued by chromatographic fractionation of the partly purified prorennin on columns of diethylaminoethylcellulose (DEAE-cellulose) as described by Peterson and Sober<sup>2</sup>.

Commercial products of DEAE-cellulose were used (Bio-Rad lot No. CXD 59-1 and Eastman-Kodak lot No. 7392). The partly purified prorennin was prepared as earlier described. After removal of the NaCl by dialysis the sample was applied to a column of DEAE-cellulose previously equilibrated with 0.10 M phosphate buffer of pH 5.8. Fig. 1 illustrates such an experiment with elution carried out by stepwise increase of the phosphate concentration from 0.10 to 0.25 M. The concentration of protein is expressed by the optical density at 278  $\mu\mu$ . For assay of the potential milk-clotting activity, 200  $\mu\text{l}$  of each fraction was mixed with 200  $\mu\text{l}$  0.4 M acetate buffer of pH 4.1 and kept 24 h at 25°C to convert the prorennin to active enzyme before the