Crystal structure of dirubidium hydrogenphosphide, $\text{Rb}_2[\text{PH}]$

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Source of material: Single crystals of $\text{Rb}_2[\text{PH}]$ were first obtained from the reaction of a mixture of Rb with HPN_2 (ratio 5:1) in a sealed steel ampoule at 950 K. The compound can also be synthesized via direct reaction of hydrogen with a mixture of Rb_4P_6 and Rb (ratio 1:10) at 950 K. Rb_4P_6 and Rb were placed in a Nb crucible, which in turn was sealed within a steel ampoule with ZrH_2 present as H_2 source, but separated from the $\text{Rb}_4\text{P}_6/\text{Rb}$ mixture. In both cases, excess Rb was removed at 473 K in high vacuum.

Table 2. Final atomic coordinates and displacement parameters (in $\text{\AA}^2$)

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Rb(1)	4c	0.8440(2)	1/4	0.0769(1)	0.0716(8)	0.0368(6)	0.0364(6)	0	-0.0023(6)	0
Rb(2)	4c	0.9787(2)	1/4	0.6835(1)	0.0500(7)	0.0605(9)	0.0570(8)	0	-0.0051(6)	0
P(1)	4c	0.2470(4)	1/4	0.1043(3)	0.051(2)	0.044(2)	0.040(2)	0	-0.007(1)	0

$\text{Rb}_2[\text{PH}]$ crystallizes in the Co_2P type (see ref. 1). The $[\text{PH}]^{2-}$ anions center the tricapped trigonal prisms built up by the Rb atoms. It was not possible to determine the position of the H atoms. However, the IR spectra of a single crystal as well as of a powdered sample exhibit the characteristic P-H absorption at $\nu(\text{P-H}) = 2122 \text{ cm}^{-1}$.

HPRb_2 , orthorhombic, $Pnma$ (No. 62), $a = 8.415(1) \text{ \AA}$, $b = 5.546(2) \text{ \AA}$, $c = 10.349(2) \text{ \AA}$, $V = 483.0 \text{ \AA}^3$, $Z = 4$, $R(F) = 0.047$, $R_w(F) = 0.046$.

Table 1. Parameters used for the X-ray data collection

Crystal:	orange prism, size 0.12 x 0.10 x 0.13 mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 $\text{\AA}$)
μ :	198.50 cm^{-1}
Diffractometer:	Siemens P4
Scan mode:	ω
$T_{\text{measurement}}$:	293 K
$2\theta_{\text{max}}$:	55°
$N(hkl)_{\text{unique}}$:	611
Criterion for F_o :	$F_o > 3 \sigma(F_o)$
$N(\text{param})_{\text{refined}}$:	20
Program:	SHELXTL-plus

References

1. Rundqvist, S.: The Structures of Co_2P , Ru_2P and Related Phases. Acta Chem. Scand. 14 (1960) 1961-1979.
2. Sheldrick, G. M.: Program Package SHELXTL-plus. Release 4.1. Siemens Analytical X-Ray Instruments Inc., Madison (WI 53719), USA 1990.