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# Bis($\mu$-2,2′-4,4′-6,6′-hexamethyl-$\mu$-terphenyl-2′-phosphanido)bis[(*N*-methylimidazole)-rubidium(I)]

**Gerd W. Rabe,[a]* Louise M. Liable-Sands,[b] James A. Golen[c] and Arnold L. Rheingold[d]**

[a]Technische Universität München, Anorganisch-chemisches Institut, Lichtenbergstraße 4, 85747 Garching, Germany, [b]University of Delaware, Department of Chemistry and Biochemistry, Newark, Delaware 19716, USA, [c]University of Massachusetts Dartmouth, Department of Chemistry and Biochemistry, North Dartmouth, MA 02747, USA, and [d]University of California, San Diego, Department of Chemistry and Biochemistry, La Jolla, CA 92093, USA

Correspondence e-mail: g.rabe@lrz.tu-muenchen.de

**Key indicators**

Single-crystal X-ray study
$T$ = 198 K
Mean $\sigma$(C–C) = 0.004 Å
$R$ factor = 0.038
$wR$ factor = 0.109
Data-to-parameter ratio = 14.8

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

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The solid-state structure of $[\{(C_{24}H_{25})P(H)\}Rb(C_4H_6N_2)]_2$ or $[Rb_2(C_{24}H_{26}P)_2(C_4H_6N_2)_2]$, a novel base adduct of tetrameric $[DmpP(H)Rb]_4$ (Dmp = 2,6-dimesitylphenyl), is reported. It exhibits a dimeric centrosymmetric arrangement.

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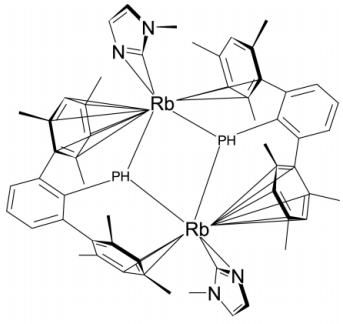
## Comment

Until now the number of structurally authenticated molecular phosphanide compounds of the heavier alkali metals rubidium and caesium is still relatively small (Frenzel *et al.*, 2001; Izod, 2000; Izod *et al.*, 2001; Rabe, Heise *et al.*, 1998; Rabe, Kheradmandan *et al.*, 1998; Rabe *et al.*, 1999, 2000; Smith, 1998). The previously reported tetrameric rubidium phosphanide compound $[DmpP(H)Rb]_4$ (Rabe, Kheradmandan *et al.*, 1998; Smith, 1998) is not complexed by classical Lewis bases such as tetrahydrofuran, but achieves coordinative saturation instead through $\pi$-arene interactions exclusively. We were interested in investigating the possibility of breaking up the $Rb_4P_4$-framework and coordinating Lewis bases to the Rb atom using bases stronger than tetrahydrofuran.




(I)

The molecular structure of the title compound, (I), features a dimeric arrangement. In the centrosymmetic $Rb_2P_2$ unit, the Rb atom is coordinated by an *N*-MeIm ligand (*N*-MeIm = *N*-methylimidazole) in an unprecedented $\eta^2$-bonding mode [Rb—N1 = 2.927 (3) Å and Rb—C25 = 3.441 (3) Å]. The N atom deviates by 2.89 Å from the $Rb_2P_2$ plane and the sum of the P—Rb—P and the two N—Rb—P angles is 254.5°. Additionally, the metal atom is $\pi$-coordinated by two mesityl rings of different terphenyl ligands; one aryl ring coordinates to the Rb atom in an approximate $\eta^6$-fashion, with Rb—C distances ranging from 3.297 (3) (for C2) to 3.700 (3) Å (for C5), the other in an $\eta^3$-mode [Rb—C = 3.393 (3) Å for C16, 3.533 (3) Å for C15, and 3.625 (3) Å for C17]. Also, one of the methyl groups (C24) of the mesityl ring that is $\eta^3$-bonded to a Rb atom shows an additional short interatomic contact to the other metal atom, at a distance of 3.476 (3) Å. A very short

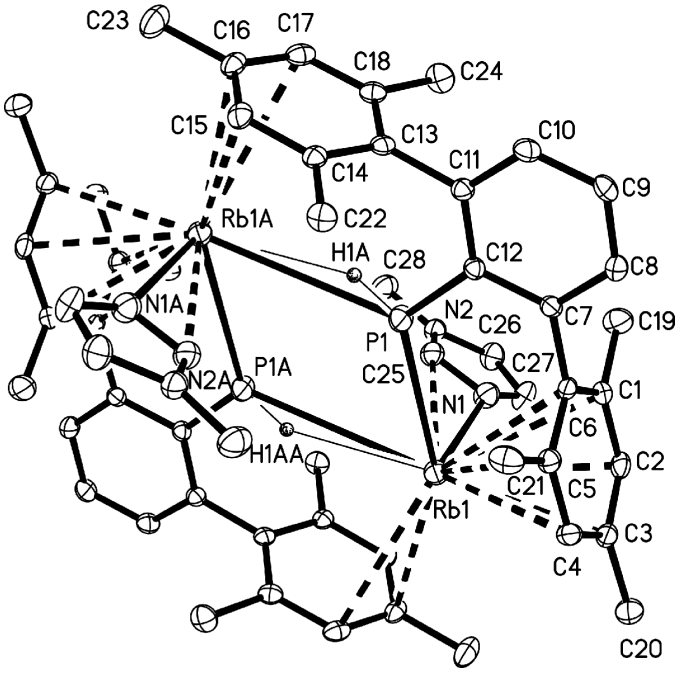



**Figure 1**
The molecular structure of the title compound, showing the atom-labeling scheme for the symmetry-independent half of the molecular system. Displacement ellipsoids are drawn at the 30% probability level and H atoms, except for H1*A* and H1*AA*, have been omitted for clarity. Non-H atoms labeled with A and atom H1AA are related by the symmetry transformation $(1 - x, 1 - y, 1 - z)$.

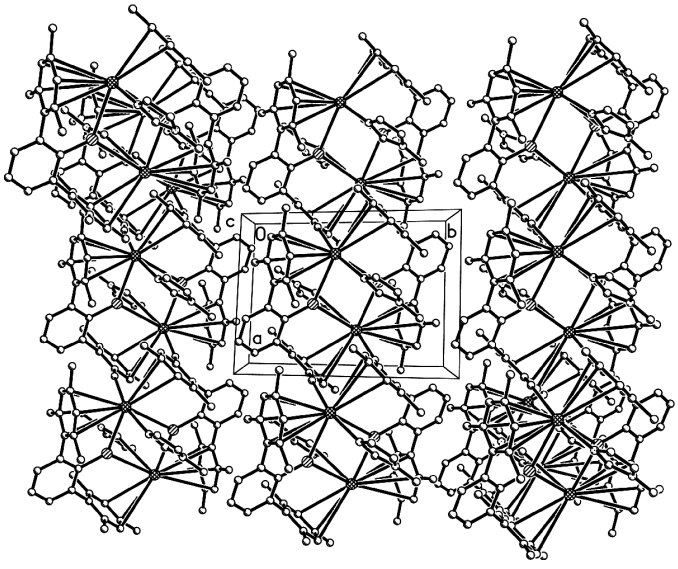



**Figure 2**
Unit-cell diagram, viewed along the *c* axis. All H atoms have been omitted for clarity.

P1—H1*A* distance of 1.09 (4) Å was found for H1*A* along with an interatomic interaction with the symmetry-generated Rb atom; Rb1$^{i}$—H1*A* at 2.85 (4) Å, with a P1—H1*A*—Rb1$^{i}$ angle of 151 (3)° [symmetry code: (i) $1 - x$, $1 - y$, $1 - z$]. The Rb1$^{i}$—P1 distance of 3.8448 (8) Å is greater than the Rb1—P1 distance of 3.3185 (8) Å. The longer Rb1$^{i}$—P1 bond interaction may explain the acute H1*A*—P1—Rb1$^{i}$ and H1*A*—Rb1$^{i}$—P1 angles which are 21 (2) and 9 (2)°, respectively.

## Experimental

Crystalline material of orange–yellow [DmpP(H)Rb(*N*-MeIm)]$_2$ was obtained by slow evaporation of a toluene solution of unsolvated [DmpP(H)Rb]$_4$ (Rabe, Kheradmandan *et al.*, 1998) in the presence of a couple of drops of *N*-MeIm.

*Crystal data*

$[Rb_2(C_{24}H_{26}P)_2(C_4H_6N_2)_2]$
$M_r$ = 1026.00
Triclinic, $P\overline{1}$
$a$ = 8.6940 (3) Å
$b$ = 12.3225 (4) Å
$c$ = 13.6724 (5) Å
$\alpha$ = 67.228 (1)°
$\beta$ = 75.138 (1)°
$\gamma$ = 85.917 (1)°
$V$ = 1304.75 (8) Å$^3$
$Z$ = 1
$D_x$ = 1.306 Mg m$^{-3}$
Mo $K\alpha$ radiation
Cell parameters from 3500 reflections
$\theta$ = 2.0–25.0°
$\mu$ = 1.98 mm$^{-1}$
$T$ = 198 (2) K
Block, orange
0.50 × 0.40 × 0.40 mm

*Data collection*

Siemens–Bruker *P*4 1K CCD-detector diffractometer
$\omega$ scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2001)
$T_{min}$ = 0.393, $T_{max}$ = 0.454
7167 measured reflections
4422 independent reflections
3920 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.025
$\theta_{max}$ = 25.0°
$h = -9 \rightarrow 10$
$k = -14 \rightarrow 14$
$l = -14 \rightarrow 16$

*Refinement*

Refinement on $F^2$
$R[F^2 > 2\sigma(F^2)]$ = 0.039
$wR(F^2)$ = 0.110
$S$ = 1.05
4422 reflections
299 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0741P)^2 + 0.1125P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.91 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.86 e Å$^{-3}$

**Table 1**
Selected geometric parameters (Å, °).

| | | | |
|---|---|---|---|
| Rb1—N1 | 2.927 (3) | Rb1—C25 | 3.441 (3) |
| Rb1—C2 | 3.297 (3) | Rb1—C15$^{i}$ | 3.533 (3) |
| Rb1—P1 | 3.3185 (8) | Rb1—C4 | 3.565 (3) |
| Rb1—C16$^{i}$ | 3.393 (3) | Rb1—C17$^{i}$ | 3.625 (3) |
| Rb1—C3 | 3.403 (3) | Rb1—C6 | 3.647 (2) |
| Rb1—C1 | 3.429 (3) | Rb1—C5 | 3.700 (3) |
| Rb1—C24 | 3.476 (3) | P1—Rb1$^{i}$ | 3.8448 (8) |
| N1—Rb1—P1 | 98.17 (6) | P1—Rb1—P1$^{i}$ | 66.92 (2) |
| Rb1—P1—Rb1$^{i}$ | 113.08 (2) | Rb1—N1—C25 | 101.61 (19) |

Symmetry code: (i) $1 - x, 1 - y, 1 - z$.

**Table 2**
Agostic interaction (Å, °).

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| P1—H1*A*···Rb1$^{i}$ | 1.09 (4) | 2.85 (4) | 3.8448 (8) | 151 (3) |

Symmetry code: (i) $1 - x, 1 - y, 1 - z$.

Atom H1*A* was found in a Fourier difference map and was allowed to refine. All other H atoms were placed in calculated positions (C—H = 0.95–1.00 Å), with isotropic displacement parameters fixed at 1.2 or 1.5 times $U_{eq}$ of the parent atom, and were refined as riding atoms.

Data collection: *SMART* (Bruker, 2001); cell refinement: *SMART*; data reduction: *SAINT* (Bruker, 2001); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 2001); software used to prepare material for publication: *SHELXTL*.

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