# Crystal structure of dichlorido[tris(pyrazolyl)borate]triphenylphosphine-rhenium(III), $C_{27}H_{25}BCl_2N_6PRe$

Ran Zhang, Jing Nie and Li-Fang Zhang*

School of Chemical Engineering and Technology, China University of Mining and Technology, Xuzhou 221116, Jiangsu Province, P. R. China

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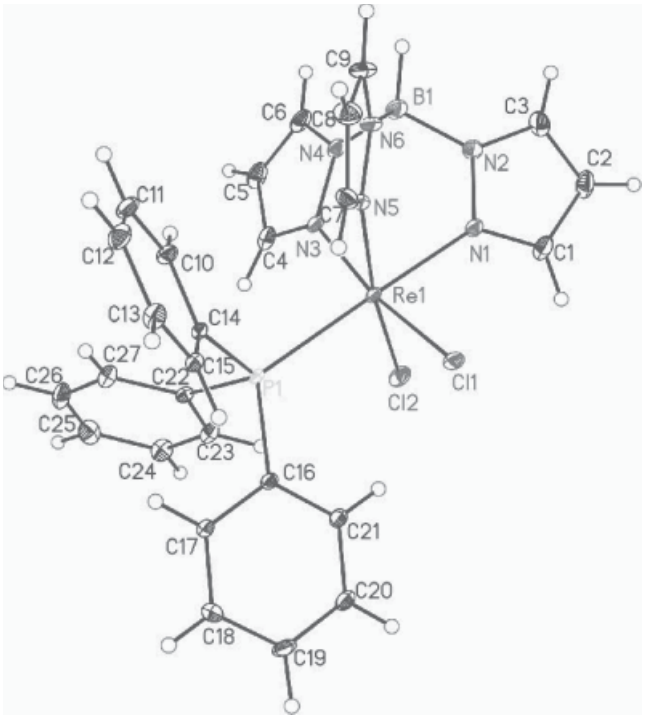


**Abstract**

$C_{27}H_{25}BCl_2N_6PRe$, monoclinic, $P2_1/c$ (no. 14), $a$ = 10.404(2) Å, $b$ = 20.006(4) Å, $c$ = 13.391(3) Å, $\beta$ = 92.74(3)°, $V$ = 2784.2 Å$^3$, $Z$ = 4, $R_{gt}(F)$ = 0.0205, $wR_{ref}(F^2)$ = 0.0449, $T$ = 123 K.

**Table 1.** Data collection and handling.

| | |
|---|---|
| Crystal: | red blocks, size 0.12×0.14×0.20 mm |
| Wavelength: | Mo $K_\alpha$ radiation (0.71073 Å) |
| $\mu$: | 46.43 cm$^{-1}$ |
| Diffractometer, scan mode: | Bruker APEX II CCD, $\varphi$ and $\omega$ |
| $2\theta_{max}$: | 51.38° |
| $N(hkl)_{measured}$, $N(hkl)_{unique}$: | 29584, 5293 |
| Criterion for $I_{obs}$, $N(hkl)_{gt}$: | $I_{obs} > 2\ \sigma(I_{obs})$, 4957 |
| $N(param)_{refined}$: | 347 |
| Programs: | SHELX [2], PLATON [3] |

## Source of material

The title complex was prepared according to the literature [1]. Red, block-shaped crystals were obtained by the slow evaporation of methanol solution at room temperature. **Elemental analysis** –found: C, 44.36%; H, 3.48%; N, 11.36%; calcd. for $ReC_{27}H_{25}BCl_2N_6P$: C, 44.27%; H, 3.44%; N, 11.47%.

## Experimental details

The coordinates of the H atom bound to the boron atom was found from difference Fourier maps and refined freely. All H atoms bound to C atoms were placed using the AFIX commands in SHELXL-97 [2], with $d$(C–H) = 0.93 Å. All H atoms were allowed for as riding atoms with $U_{iso}$(H) = 1.2 $U_{eq}$(C). The structure was checked with PLATON [3].

## Discussion

In the past two decades, the tris(pyrazolyl)borate anion ($Tp^-$) and its derivatives have been employed as tridentate ligands to assemble molecular functional materials [4, 5], and also their coordination modes have been studied [6, 7]. Furthermore, some mononuclear rhenium(III), rhenium(V) and rhenium(VII) complexes containing the $Tp^-$ ligand have been synthesized [8, 9]. To our knowledge, the crystal structures of rhenium complexes are relatively limited. The molecular structure of title compound comprises of one $Tp^-$ ligand, two chlorido ligands and one triphenylphosphine ligand coordinating the central rhenium(III) ion. In the molecular structure, the central metal rhenium(III) ion is coordinated by three pyrazole nitrogen atoms from the capping $Tp^-$ ligand, two chlorine atoms and one phosphorous atom, yielding a distorted bipyramidal [$ReN_3Cl_2P$] geometry. The averaged Re–N bond length is 2.118(2) Å. The Re–Cl bond lengths are 2.3548(8) Å for Re1–Cl1 and 2.4063(8) Å for Re1–Cl2, respectively. The Re–P bond length is 2.4352(8) Å. The N1–Re1–P1 bond angle is 178.82(6)°. The bond angles of N3–Re1–Cl1 and N5–Re1–Cl2 are 170.51(7) Å and 171.13(6) Å, respectively. All the bond lengths and bond angles are in the typical ranges for rhenium(III) compounds.

**Table 2.** Atomic coordinates and displacement parameters (in Å$^2$).

| Atom | Site | $x$ | $y$ | $z$ | $U_{iso}$ |
|---|---|---|---|---|---|
| H(1A) | 4*e* | 0.1086 | 0.2799 | 0.5791 | 0.029 |
| H(1) | 4*e* | 0.5016 | 0.2269 | 0.7968 | 0.030 |
| H(2) | 4*e* | 0.5104 | 0.3435 | 0.7356 | 0.036 |
| H(3) | 4*e* | 0.3122 | 0.3605 | 0.6348 | 0.032 |
| H(4) | 4*e* | 0.2881 | 0.0434 | 0.5058 | 0.028 |
| H(5) | 4*e* | 0.2394 | 0.1058 | 0.3494 | 0.035 |
| H(6) | 4*e* | 0.1608 | 0.2169 | 0.4021 | 0.033 |
| H(7) | 4*e* | –0.0107 | 0.1256 | 0.8793 | 0.024 |
| H(8) | 4*e* | –0.1829 | 0.2098 | 0.8466 | 0.033 |
| H(9) | 4*e* | –0.1106 | 0.2735 | 0.6988 | 0.034 |
| H(10) | 4*e* | –0.0268 | 0.0379 | 0.6381 | 0.022 |

* Correspondence author (e-mail: zhanglifang@cumt.edu.cn)

**Table 2.** continued.

| Atom | Site | *x* | *y* | *z* | $U_{iso}$ |
|---|---|---|---|---|---|
| H(11) | 4*e* | –0.2446 | 0.0501 | 0.6646 | 0.027 |
| H(12) | 4*e* | –0.3353 | 0.0235 | 0.8161 | 0.029 |
| H(13) | 4*e* | –0.2057 | –0.0165 | 0.9459 | 0.025 |
| H(15) | 4*e* | 0.0119 | –0.0303 | 0.9186 | 0.020 |
| H(17) | 4*e* | 0.1174 | –0.1199 | 0.8469 | 0.024 |
| H(18) | 4*e* | 0.2092 | –0.1897 | 0.9657 | 0.029 |
| H(19) | 4*e* | 0.4083 | –0.1618 | 1.0334 | 0.029 |
| H(20) | 4*e* | 0.5115 | –0.0666 | 0.9839 | 0.024 |
| H(21) | 4*e* | 0.4177 | 0.0050 | 0.8684 | 0.019 |
| H(23) | 4*e* | 0.4165 | –0.0474 | 0.6501 | 0.026 |
| H(24) | 4*e* | 0.4641 | –0.1231 | 0.5257 | 0.030 |
| H(25) | 4*e* | 0.2977 | –0.1793 | 0.4441 | 0.032 |
| H(26) | 4*e* | 0.0889 | –0.1580 | 0.4885 | 0.033 |
| H(27) | 4*e* | 0.0389 | –0.0830 | 0.6138 | 0.026 |

**Table 3.** Atomic coordinates and displacement parameters (in Å$^2$).

| Atom | Site | *x* | *y* | *z* | $U_{11}$ | $U_{22}$ | $U_{33}$ | $U_{12}$ | $U_{13}$ | $U_{23}$ |
|---|---|---|---|---|---|---|---|---|---|---|
| Re(1) | 4*e* | 0.26504(1) | 0.117604(5) | 0.734948(8) | 0.01183(6) | 0.01510(6) | 0.01256(7) | –0.00202(4) | –0.00381(4) | 0.00292(4) |
| P(1) | 4*e* | 0.19128(6) | 0.00269(3) | 0.75005(5) | 0.0107(3) | 0.0149(3) | 0.0127(3) | 0.0001(3) | –0.0044(3) | 0.0010(3) |
| N(1) | 4*e* | 0.3331(2) | 0.2176(1) | 0.7234(2) | 0.021(1) | 0.018(1) | 0.019(1) | –0.005(1) | –0.007(1) | 0.006(1) |
| N(2) | 4*e* | 0.2701(2) | 0.2642(1) | 0.6693(2) | 0.029(1) | 0.019(1) | 0.019(1) | –0.004(1) | –0.005(1) | 0.007(1) |
| N(3) | 4*e* | 0.2304(2) | 0.1260(1) | 0.5811(2) | 0.017(1) | 0.019(1) | 0.018(1) | –0.0036(9) | –0.007(1) | 0.005(1) |
| N(4) | 4*e* | 0.1859(2) | 0.1855(1) | 0.5450(2) | 0.023(1) | 0.022(1) | 0.019(1) | –0.007(1) | –0.008(1) | 0.008(1) |
| N(5) | 4*e* | 0.0837(2) | 0.1639(1) | 0.7594(2) | 0.018(1) | 0.013(1) | 0.021(1) | –0.0018(9) | –0.006(1) | 0.0045(9) |
| N(6) | 4*e* | 0.0512(2) | 0.2154(1) | 0.6978(2) | 0.021(1) | 0.016(1) | 0.025(1) | 0.003(1) | –0.007(1) | 0.007(1) |
| Cl(1) | 4*e* | 0.28559(7) | 0.12492(3) | 0.91050(5) | 0.0253(4) | 0.0194(4) | 0.0134(3) | 0.0036(3) | –0.0055(3) | 0.0006(3) |
| Cl(2) | 4*e* | 0.47862(6) | 0.08218(4) | 0.69801(5) | 0.0120(3) | 0.0306(4) | 0.0242(4) | –0.0009(3) | –0.0013(3) | 0.0027(3) |
| B(1) | 4*e* | 0.1478(3) | 0.2426(2) | 0.6171(3) | 0.028(2) | 0.023(2) | 0.021(2) | –0.002(2) | –0.008(2) | 0.010(1) |
| C(1) | 4*e* | 0.4383(3) | 0.2473(2) | 0.7559(2) | 0.023(2) | 0.032(2) | 0.020(2) | –0.009(1) | –0.005(1) | 0.003(1) |
| C(2) | 4*e* | 0.4447(3) | 0.3127(2) | 0.7225(2) | 0.037(2) | 0.030(2) | 0.023(2) | –0.019(2) | –0.006(1) | 0.003(1) |
| C(3) | 4*e* | 0.3372(3) | 0.3215(2) | 0.6681(2) | 0.040(2) | 0.020(2) | 0.019(2) | –0.011(1) | –0.000(1) | 0.007(1) |
| C(4) | 4*e* | 0.2567(3) | 0.0869(2) | 0.5015(2) | 0.021(2) | 0.030(2) | 0.018(2) | –0.007(1) | –0.004(1) | –0.002(1) |
| C(5) | 4*e* | 0.2302(3) | 0.1207(2) | 0.4144(2) | 0.034(2) | 0.039(2) | 0.013(2) | –0.013(2) | –0.006(1) | 0.003(1) |
| C(6) | 4*e* | 0.1864(3) | 0.1823(2) | 0.4450(2) | 0.029(2) | 0.035(2) | 0.018(2) | –0.011(1) | –0.010(1) | 0.012(1) |
| C(7) | 4*e* | –0.0121(3) | 0.1573(1) | 0.8284(2) | 0.021(2) | 0.015(1) | 0.025(2) | –0.001(1) | 0.002(1) | 0.003(1) |
| C(8) | 4*e* | –0.1084(3) | 0.2034(2) | 0.8120(2) | 0.021(2) | 0.026(2) | 0.035(2) | 0.004(1) | 0.004(1) | 0.004(1) |
| C(9) | 4*e* | –0.0654(3) | 0.2384(2) | 0.7293(3) | 0.024(2) | 0.023(2) | 0.037(2) | 0.008(1) | –0.009(1) | 0.008(1) |
| C(10) | 4*e* | –0.0596(3) | 0.0262(1) | 0.6991(2) | 0.018(1) | 0.023(2) | 0.014(2) | –0.000(1) | –0.003(1) | 0.003(1) |
| C(11) | 4*e* | –0.1909(3) | 0.0337(2) | 0.7165(2) | 0.018(1) | 0.028(2) | 0.020(2) | 0.001(1) | –0.007(1) | 0.001(1) |
| C(12) | 4*e* | –0.2476(3) | 0.0176(2) | 0.8090(2) | 0.014(1) | 0.029(2) | 0.029(2) | 0.000(1) | –0.001(1) | –0.003(1) |
| C(13) | 4*e* | –0.1729(3) | –0.0059(2) | 0.8845(2) | 0.019(2) | 0.026(2) | 0.018(2) | –0.003(1) | 0.002(1) | 0.000(1) |
| C(14) | 4*e* | 0.0171(2) | 0.0015(1) | 0.7741(2) | 0.011(1) | 0.014(1) | 0.016(1) | 0.000(1) | –0.004(1) | –0.001(1) |
| C(15) | 4*e* | –0.0413(3) | –0.0138(1) | 0.8665(2) | 0.018(1) | 0.016(1) | 0.015(1) | –0.002(1) | –0.005(1) | 0.001(1) |
| C(16) | 4*e* | 0.2577(3) | –0.0496(1) | 0.8478(2) | 0.018(1) | 0.014(1) | 0.014(1) | 0.004(1) | –0.005(1) | –0.003(1) |
| C(17) | 4*e* | 0.1973(3) | –0.1091(1) | 0.8766(2) | 0.023(2) | 0.015(2) | 0.021(2) | 0.001(1) | –0.011(1) | –0.002(1) |
| C(18) | 4*e* | 0.2511(3) | –0.1511(2) | 0.9461(2) | 0.032(2) | 0.015(2) | 0.026(2) | –0.001(1) | –0.007(1) | 0.004(1) |
| C(19) | 4*e* | 0.3681(3) | –0.1343(2) | 0.9855(2) | 0.030(2) | 0.023(2) | 0.019(2) | 0.010(1) | –0.011(1) | –0.001(1) |
| C(20) | 4*e* | 0.4302(3) | –0.0763(1) | 0.9559(2) | 0.019(2) | 0.022(2) | 0.017(2) | 0.006(1) | –0.006(1) | –0.006(1) |
| C(21) | 4*e* | 0.3756(3) | –0.0336(1) | 0.8873(2) | 0.015(1) | 0.017(1) | 0.017(2) | 0.002(1) | –0.002(1) | –0.003(1) |
| C(22) | 4*e* | 0.2205(3) | –0.0557(1) | 0.6449(2) | 0.018(1) | 0.017(1) | 0.013(1) | 0.000(1) | –0.004(1) | 0.002(1) |
| C(23) | 4*e* | 0.3490(3) | –0.0699(2) | 0.6167(2) | 0.017(2) | 0.026(2) | 0.022(2) | –0.002(1) | –0.005(1) | –0.001(1) |
| C(24) | 4*e* | 0.3792(3) | –0.1158(2) | 0.5418(2) | 0.022(2) | 0.030(2) | 0.023(2) | 0.001(1) | 0.002(1) | –0.001(1) |
| C(25) | 4*e* | 0.2822(3) | –0.1485(2) | 0.4942(2) | 0.036(2) | 0.026(2) | 0.018(2) | –0.001(1) | 0.004(1) | –0.006(1) |
| C(26) | 4*e* | 0.1558(3) | –0.1352(2) | 0.5220(2) | 0.029(2) | 0.031(2) | 0.023(2) | –0.008(1) | –0.006(1) | –0.007(1) |
| C(27) | 4*e* | 0.1239(3) | –0.0895(2) | 0.5975(2) | 0.018(2) | 0.027(2) | 0.020(2) | –0.003(1) | –0.003(1) | –0.002(1) |

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