Crystal structure of *trans*-((2-aminophenolato-κ*O*)-(2-hydroxyphenylimido-κ^2*N*,*O*)-bromido-bis(triphenylphosphane)-rhenium(V)) bromide – acetonitrile – water (1:2:1), $C_{52}H_{49}Br_2N_4O_3P_2Re$

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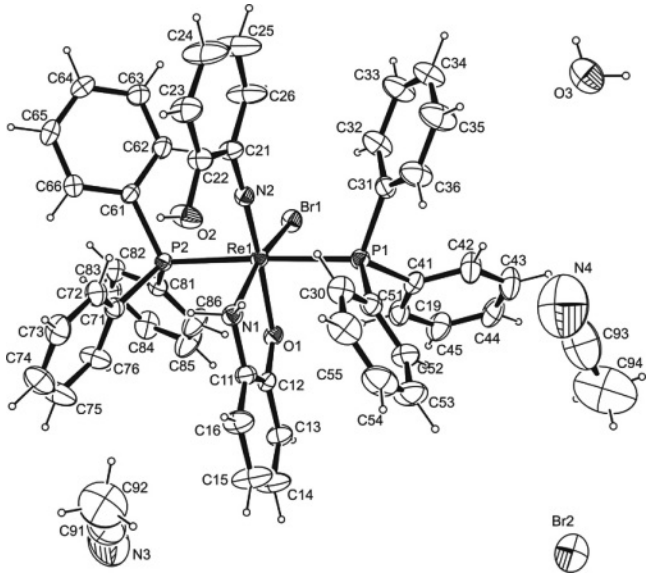

Abstract

$C_{52}H_{49}Br_2N_4O_3P_2Re$, triclinic, $P\bar{1}$ (no. 2), a = 13.5222(9) Å, b = 14.1956(9) Å, c = 14.3102(9) Å, α = 92.096(3)°, β = 114.726(2)°, γ = 96.966(2)°, V = 2456.0 Å^3, Z = 2, $R_{gt}(F)$ = 0.0221, $wR_{ref}(F^2)$ = 0.0501, T = 200 K.

Table 1. Data collection and handling.

Crystal:	red platelets, size 0.148×0.24×0.557 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	41.97 cm^{-1}
Diffractometer, scan mode:	Bruker APEX-II CCD, φ and ω
$2\theta_{max}$:	56.78°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	41881, 12257
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 11004
$N(param)_{refined}$:	584
Programs:	SHELX, WinGX, MERCURY, PLATON [10–13]

Source of material

The title compound was prepared upon reacting $ReOBr_3(PPh_3)_2$ with the Schiff-base derived from bis(pentamethylene)urea and 2-aminophenol in acetonitrile. Crystals suitable for the diffraction study were obtained upon free evaporation of the solvent.

Experimental details

Carbon-bound H atoms were placed in calculated positions (C–H 0.95 Å) and were included in the refinement in the riding model approximation, with U_{iso}(H) set to 1.2U_{eq}(C). The H atoms of the methyl groups were allowed to rotate with a fixed angle around the C–C bond to best fit the experimental electron density (HFIX 137 in the SHELX program suite [10]), with U_{iso}(H) set to 1.5U_{eq}(C). Both nitrogen bound hydrogen atoms were located on a DFM and refined with individual thermal parameters. Both oxygen bound hydrogen atoms were located on a DFM and refined with individual thermal parameters.

Discussion

Next to cardiovascular diseases, cancer has become one of the main fatal diseases in industrialized countries. Apart from classical surgery, chemo- and radiotherapeutic treatments have entered the arsenal of possible cures for certain types of cancer. All methods, however, suffer from their own set of problematic side-effects and, as a consequence, the development of radiopharmaceuticals – combining the advantages of chemotherapy as well as radiation methods while at the same time avoiding their unique respective undesired side-effects – has been a topic of research [1, 2]. Tailoring and fine-tuning of the envisioned radiopharmaceuticals' properties such as lipophilicity and, in particular, inertness is of paramount importance with respect to possible future *in vivo* applications in contemporary medicine and requires sound knowledge about structural parameters of the ligands applied if a more heuristic approach in the synthesis is to triumph over pure trial-and-error as it is encountered in this specific field of coordination chemistry up to the present day. In continuation of our interest in rhenium-based coordination compounds that might serve as radiopharmaceuticals, the title compound was synthesized. The molecular and crystal structure of a similar compound – namely the iodo derivative – has been reported earlier by us [3]. The title compound is a rhenium(V) coordination compound. The octahedral coordination sphere around the central atom consists of two triphenylphosphane ligands in *trans* configuration, one bromido ligand, one 2-hydroxyphenylimido moiety as well as one chelating 2-aminophenolate ligand. In addition, two molecules of acetonitrile as well as one molecule of water are present in the crystal structure. A puckering analysis of the five-membered chelate ring according to Cremer & Pople [4] shows the latter to adopt a $^{Re(1)}E$ conformation [5]. The two Re–N bond lengths are measured at 1.7330(19) Å and 2.1819(19) Å with the shorter of the two established towards the phenylimido ligand. The former value is in good agreement with the most common bond lengths reported for similar compounds featuring a Re=N bond whose metrical parameters have been deposited with the Cambridge Structural Database [6]. Both Re–P bond lengths of 2.4895(6) Å and 2.4939(6) Å are found in a smaller interval than the ones reported for the comparable iodido derivative [3]. The rhenium atom is displaced by 0.005(1) Å from the least-squares

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plane as defined by itself as well as the bromido ligand and the coordinating oxygen and nitrogen atoms. The least-squares planes as defined by the atoms of the chelate ring on the one hand and the non-hydrogen atoms of the phenyl moiety supporting this chelate ring enclose an angle of 3.39(14)°. The latter least-squares plane intersects with the least-squares plane as defined by the carbon atoms of the phenylimido ligand at an angle of 17.69(18)°. In the crystal, classical hydrogen bonds of the O–H···O and O–H···Br type as well as the N–H···O type are observed next to C–H···N contacts whose range falls below the sum of van-der-Waals radii of the atoms participating in them [7]. While the O–H···O hydrogen bond is established between the phenolic hydroxyl group as donor and the oxygen atom of the water molecule as acceptor, the water molecule exclusively gives rise to O–H···Br hydrogen bonds involving the anionic bromide. The C–H···N contacts are apparent in between one of the hydrogen atoms on a triphenylphosphane ligand as donor and the nitrogen atom of one of the acetonitrile molecules present in the crystal structure as acceptor. In terms of graph-set analysis [8, 9], the descriptor for the classical hydrogen bonds is *DDD* on the unary level while the C–H···N contacts can be described by a *D* descriptor on the same level. Cyclic $Br_2(H_2O)_2$ units ($R^2_4(8)$ graph set) connect two adjacent complexes. In total, the entities of the crystal structure are connected to planes perpendicular to the crystallographic *a* axis. The shortest intercentroid distance between two centers of gravity was measured at 4.0405(15) Å and is found between one of the phenyl groups on a triphenylphosphane ligand as well as the five-membered chelate ring.

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

Atom	Site	*x*	*y*	*z*	U_{iso}
H(2)	2*i*	–0.0396	0.0872	0.1938	0.057
H(3B)	2*i*	0.0136	0.0185	0.8952	0.08(2)
H(3A)	2*i*	0.1038	–0.0101	0.9666	0.07(1)
H(1A)	2*i*	0.1893	0.1815	0.3395	0.016(6)
H(1B)	2*i*	0.1690	0.2303	0.2580	0.028(7)
H(13)	2*i*	0.5284	0.4283	0.4639	0.038
H(14)	2*i*	0.6198	0.3213	0.4147	0.059
H(15)	2*i*	0.5275	0.1730	0.3267	0.064
H(16)	2*i*	0.3407	0.1281	0.2897	0.045
H(19)	2*i*	0.3954	0.4463	0.6089	0.041
H(23)	2*i*	–0.2070	0.0331	0.2002	0.048
H(24)	2*i*	–0.3270	0.0689	0.2698	0.075
H(25)	2*i*	–0.2839	0.2038	0.3843	0.087
H(26)	2*i*	–0.1230	0.3097	0.4241	0.060
H(30)	2*i*	0.1501	0.0904	0.4565	0.042
H(32)	2*i*	0.0763	0.3626	0.5982	0.048
H(33)	2*i*	–0.0542	0.3236	0.6626	0.057
H(34)	2*i*	–0.0767	0.1733	0.7162	0.052
H(35)	2*i*	0.0300	0.0620	0.7053	0.063
H(36)	2*i*	0.1628	0.1002	0.6433	0.053
H(42)	2*i*	0.3243	0.2647	0.7910	0.050
H(43)	2*i*	0.4571	0.3577	0.9368	0.058
H(44)	2*i*	0.5550	0.4969	0.9196	0.052
H(45)	2*i*	0.5250	0.5409	0.7566	0.051
H(52)	2*i*	0.4555	0.2116	0.6627	0.044

Table 2. continued.

Atom	Site	*x*	*y*	*z*	U_{iso}
H(53)	2*i*	0.5238	0.0746	0.6377	0.060
H(54)	2*i*	0.4083	–0.0513	0.5201	0.062
H(55)	2*i*	0.2212	–0.0443	0.4300	0.056
H(62)	2*i*	–0.0479	0.4684	0.3272	0.038
H(63)	2*i*	–0.2372	0.4681	0.2627	0.045
H(64)	2*i*	–0.3498	0.4264	0.0875	0.046
H(65)	2*i*	–0.2716	0.3943	–0.0249	0.047
H(66)	2*i*	–0.0826	0.3967	0.0375	0.039
H(72)	2*i*	0.0114	0.2482	0.1227	0.036
H(73)	2*i*	0.0403	0.1620	–0.0029	0.047
H(74)	2*i*	0.1662	0.2284	–0.0623	0.061
H(75)	2*i*	0.2657	0.3787	0.0054	0.067
H(76)	2*i*	0.2411	0.4651	0.1339	0.048
H(82)	2*i*	0.0339	0.5893	0.1275	0.034
H(83)	2*i*	0.1186	0.7422	0.1268	0.040
H(84)	2*i*	0.2983	0.7958	0.2474	0.049
H(85)	2*i*	0.3940	0.6964	0.3675	0.064
H(86)	2*i*	0.3111	0.5428	0.3684	0.050
H(92A)	2*i*	0.3436	0.0900	0.0006	0.150
H(92B)	2*i*	0.3130	0.1220	0.0924	0.150
H(92C)	2*i*	0.4200	0.0720	0.1169	0.150
H(94A)	2*i*	0.6902	0.1205	0.9606	0.217
H(94B)	2*i*	0.6567	0.1803	1.0370	0.217
H(94C)	2*i*	0.6494	0.2212	0.9319	0.217

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)	2*i*	0.165548(7)	0.348548(6)	0.405846(7)	0.01667(4)	0.01906(4)	0.01808(4)	0.00123(3)	0.00577(3)	–0.00075(3)
Br(1)	2*i*	0.16374(2)	0.50288(2)	0.49935(2)	0.0297(1)	0.0229(1)	0.0242(1)	0.00378(9)	0.00896(9)	–0.00365(9)
Br(2)	2*i*	0.82182(3)	0.03122(2)	0.84701(3)	0.0570(2)	0.0387(2)	0.0585(2)	–0.0056(1)	0.0162(2)	0.0057(2)
P(1)	2*i*	0.23922(5)	0.27360(4)	0.57167(4)	0.0206(3)	0.0218(3)	0.0204(3)	0.0020(2)	0.0077(2)	0.0024(2)
P(2)	2*i*	0.10187(4)	0.42828(4)	0.24311(4)	0.0170(3)	0.0202(3)	0.0177(3)	0.0011(2)	0.0060(2)	–0.0001(2)
O(1)	2*i*	0.3219(1)	0.3983(1)	0.4330(1)	0.0197(7)	0.0204(7)	0.0210(8)	0.0007(6)	0.0058(6)	–0.0009(6)
O(2)	2*i*	–0.0116(2)	0.1293(1)	0.2441(2)	0.038(1)	0.034(1)	0.046(1)	–0.0082(8)	0.0266(9)	–0.0173(8)
O(3)	2*i*	0.0747(2)	0.0031(2)	0.9052(2)	0.069(2)	0.046(1)	0.045(1)	0.003(1)	0.031(1)	–0.012(1)
N(1)	2*i*	0.2099(2)	0.2346(1)	0.3312(2)	0.0212(9)	0.023(1)	0.023(1)	0.0025(8)	0.0076(8)	–0.0003(8)
N(2)	2*i*	0.0348(2)	0.2877(1)	0.3736(1)	0.0230(9)	0.0206(9)	0.0204(9)	0.0044(7)	0.0095(8)	–0.0004(7)
N(3)	2*i*	0.4883(4)	0.2790(3)	0.0975(4)	0.117(4)	0.100(3)	0.096(3)	–0.018(3)	0.053(3)	0.022(3)
N(4)	2*i*	0.4387(6)	0.0783(4)	0.8694(4)	0.164(6)	0.102(4)	0.108(4)	–0.022(4)	0.026(4)	–0.031(3)
C(11)	2*i*	0.3256(2)	0.2530(2)	0.3529(2)	0.020(1)	0.027(1)	0.026(1)	0.0035(9)	0.0101(9)	0.0012(9)
C(12)	2*i*	0.3798(2)	0.3413(2)	0.4061(2)	0.019(1)	0.026(1)	0.020(1)	0.0060(9)	0.0062(9)	0.0056(9)
C(13)	2*i*	0.4904(2)	0.3677(2)	0.4287(2)	0.022(1)	0.032(1)	0.038(1)	–0.001(1)	0.011(1)	–0.000(1)
C(14)	2*i*	0.5440(2)	0.3041(2)	0.3991(3)	0.024(1)	0.051(2)	0.072(2)	0.001(1)	0.022(1)	–0.011(2)
C(15)	2*i*	0.4892(2)	0.2155(2)	0.3469(3)	0.028(2)	0.046(2)	0.086(3)	0.005(1)	0.026(2)	–0.011(2)
C(16)	2*i*	0.3786(2)	0.1889(2)	0.3242(2)	0.031(1)	0.032(1)	0.048(2)	0.005(1)	0.017(1)	–0.009(1)
C(19)	2*i*	0.4068(2)	0.4282(2)	0.6755(2)	0.031(1)	0.039(1)	0.025(1)	–0.003(1)	0.006(1)	0.003(1)
C(21)	2*i*	–0.0634(2)	0.2293(2)	0.3441(2)	0.020(1)	0.025(1)	0.031(1)	0.0007(9)	0.011(1)	–0.0032(9)
C(22)	2*i*	–0.0883(2)	0.1472(2)	0.2761(2)	0.026(1)	0.026(1)	0.032(1)	0.002(1)	0.013(1)	–0.003(1)
C(23)	2*i*	–0.1881(2)	0.0881(2)	0.2475(2)	0.031(1)	0.035(1)	0.048(2)	–0.005(1)	0.015(1)	–0.011(1)
C(24)	2*i*	–0.2594(3)	0.1100(3)	0.2883(3)	0.031(2)	0.053(2)	0.100(3)	–0.017(1)	0.033(2)	–0.025(2)

Table 3. continued.

Atom	Site	*x*	*y*	*z*	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
C(25)	2*i*	–0.2345(3)	0.1907(3)	0.3557(4)	0.041(2)	0.064(2)	0.123(4)	–0.012(2)	0.054(2)	–0.035(2)
C(26)	2*i*	–0.1382(2)	0.2521(2)	0.3813(3)	0.030(1)	0.043(2)	0.081(2)	–0.006(1)	0.032(2)	–0.029(2)
C(30)	2*i*	0.2267(2)	0.0880(2)	0.4928(2)	0.039(2)	0.027(1)	0.038(2)	0.004(1)	0.016(1)	0.004(1)
C(31)	2*i*	0.1333(2)	0.2355(2)	0.6146(2)	0.026(1)	0.029(1)	0.023(1)	0.0016(9)	0.011(1)	0.0033(9)
C(32)	2*i*	0.0678(3)	0.3011(2)	0.6204(2)	0.055(2)	0.031(1)	0.050(2)	0.010(1)	0.037(2)	0.008(1)
C(33)	2*i*	–0.0100(3)	0.2779(2)	0.6585(3)	0.060(2)	0.045(2)	0.061(2)	0.017(2)	0.046(2)	0.010(2)
C(34)	2*i*	–0.0234(3)	0.1891(2)	0.6901(2)	0.048(2)	0.046(2)	0.051(2)	0.002(1)	0.036(2)	0.005(1)
C(35)	2*i*	0.0398(3)	0.1237(2)	0.6840(3)	0.065(2)	0.038(2)	0.080(2)	0.010(2)	0.052(2)	0.022(2)
C(36)	2*i*	0.1187(3)	0.1463(2)	0.6468(3)	0.049(2)	0.036(2)	0.064(2)	0.015(1)	0.038(2)	0.021(1)
C(41)	2*i*	0.3463(2)	0.3461(2)	0.6850(2)	0.023(1)	0.028(1)	0.023(1)	0.0043(9)	0.0039(9)	0.0006(9)
C(42)	2*i*	0.3654(2)	0.3207(2)	0.7834(2)	0.045(2)	0.044(2)	0.026(1)	–0.001(1)	0.006(1)	0.008(1)
C(43)	2*i*	0.4437(3)	0.3763(2)	0.8699(2)	0.050(2)	0.057(2)	0.023(1)	0.002(2)	0.003(1)	0.003(1)
C(44)	2*i*	0.5022(2)	0.4582(2)	0.8598(2)	0.035(2)	0.047(2)	0.028(1)	0.004(1)	–0.004(1)	–0.006(1)
C(45)	2*i*	0.4843(2)	0.4843(2)	0.7634(2)	0.033(1)	0.042(2)	0.036(2)	–0.006(1)	0.002(1)	–0.001(1)
C(51)	2*i*	0.2960(2)	0.1658(2)	0.5614(2)	0.030(1)	0.025(1)	0.028(1)	0.006(1)	0.015(1)	0.0067(9)
C(52)	2*i*	0.4073(2)	0.1599(2)	0.6153(2)	0.031(1)	0.041(2)	0.041(2)	0.010(1)	0.018(1)	0.005(1)
C(53)	2*i*	0.4479(3)	0.0786(2)	0.6000(3)	0.043(2)	0.054(2)	0.063(2)	0.024(2)	0.029(2)	0.010(2)
C(54)	2*i*	0.3793(3)	0.0036(2)	0.5310(3)	0.067(2)	0.041(2)	0.067(2)	0.023(2)	0.043(2)	0.011(2)
C(55)	2*i*	0.2686(3)	0.0078(2)	0.4774(3)	0.061(2)	0.029(1)	0.055(2)	0.004(1)	0.031(2)	–0.001(1)
C(61)	2*i*	–0.0459(2)	0.4304(2)	0.1896(2)	0.018(1)	0.025(1)	0.021(1)	0.0033(9)	0.0048(9)	0.0027(9)
C(62)	2*i*	–0.0931(2)	0.4527(2)	0.2555(2)	0.026(1)	0.043(2)	0.021(1)	0.010(1)	0.005(1)	–0.003(1)
C(63)	2*i*	–0.2056(2)	0.4522(2)	0.2171(2)	0.028(1)	0.056(2)	0.031(1)	0.015(1)	0.012(1)	0.002(1)
C(64)	2*i*	–0.2723(2)	0.4288(2)	0.1131(2)	0.020(1)	0.054(2)	0.035(1)	0.008(1)	0.006(1)	0.006(1)
C(65)	2*i*	–0.2260(2)	0.4089(2)	0.0470(2)	0.027(1)	0.057(2)	0.023(1)	0.005(1)	0.001(1)	0.001(1)
C(66)	2*i*	–0.1138(2)	0.4100(2)	0.0840(2)	0.028(1)	0.047(2)	0.022(1)	0.007(1)	0.010(1)	0.003(1)
C(71)	2*i*	0.1232(2)	0.3658(2)	0.1410(2)	0.025(1)	0.026(1)	0.022(1)	0.0058(9)	0.0084(9)	0.0004(9)
C(72)	2*i*	0.0638(2)	0.2751(2)	0.0993(2)	0.034(1)	0.028(1)	0.025(1)	0.002(1)	0.011(1)	–0.001(1)
C(73)	2*i*	0.0805(2)	0.2240(2)	0.0244(2)	0.044(2)	0.032(1)	0.033(1)	0.006(1)	0.009(1)	–0.008(1)
C(74)	2*i*	0.1552(3)	0.2631(2)	–0.0103(3)	0.060(2)	0.055(2)	0.047(2)	0.017(2)	0.032(2)	–0.010(2)
C(75)	2*i*	0.2141(3)	0.3521(3)	0.0300(3)	0.062(2)	0.054(2)	0.071(2)	–0.000(2)	0.052(2)	–0.011(2)
C(76)	2*i*	0.1992(2)	0.4038(2)	0.1060(2)	0.041(2)	0.035(1)	0.054(2)	–0.002(1)	0.032(1)	–0.008(1)
C(81)	2*i*	0.1643(2)	0.5504(2)	0.2476(2)	0.023(1)	0.021(1)	0.023(1)	0.0022(9)	0.0105(9)	0.0007(9)
C(82)	2*i*	0.1076(2)	0.6106(2)	0.1764(2)	0.029(1)	0.029(1)	0.023(1)	0.003(1)	0.008(1)	0.0013(9)
C(83)	2*i*	0.1578(2)	0.7016(2)	0.1762(2)	0.041(2)	0.030(1)	0.029(1)	0.008(1)	0.014(1)	0.008(1)
C(84)	2*i*	0.2641(2)	0.7333(2)	0.2474(2)	0.045(2)	0.028(1)	0.043(2)	–0.006(1)	0.014(1)	0.007(1)
C(85)	2*i*	0.3206(3)	0.6744(2)	0.3184(3)	0.036(2)	0.044(2)	0.054(2)	–0.014(1)	–0.003(1)	0.017(2)
C(86)	2*i*	0.2714(2)	0.5831(2)	0.3190(2)	0.029(1)	0.034(1)	0.047(2)	–0.004(1)	0.001(1)	0.015(1)
C(91)	2*i*	0.4378(4)	0.2064(4)	0.0872(4)	0.078(3)	0.080(3)	0.080(3)	0.003(2)	0.043(3)	0.020(3)
C(92)	2*i*	0.3736(5)	0.1155(4)	0.0732(5)	0.109(4)	0.076(3)	0.121(5)	–0.006(3)	0.059(4)	0.011(3)
C(93)	2*i*	0.5265(6)	0.1153(4)	0.9104(4)	0.147(5)	0.063(3)	0.068(3)	0.001(3)	0.046(3)	–0.008(2)
C(94)	2*i*	0.6401(6)	0.1633(5)	0.9644(6)	0.149(6)	0.113(5)	0.187(8)	–0.017(5)	0.100(6)	–0.034(5)

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