# Kristallstrukturdaten zur Dissertation „Nass- und elektrochemische Synthese von Metallsalzen der *closo*-Dodekaborate $[B_{12}X_{12}]^{2-}$ (X = H, F, Cl, Br, I) und $[Me_3NB_{12}X_{11}]$ (X = F, Cl)“

vorgelegt von:
Bianca Wegener
29.05.1981 in Duderstadt

# Inhaltsverzeichnis

# 1 Kristallstrukturdaten

## 1.1 [Ag($\eta^2$-Acenaphthen][$Me_3NB_{12}Cl_{11}$]

**Tabelle 1** Kristalldaten und Strukturverfeinerung von [Ag($\eta^2$-Acenaphthen)][$Me_3NB_{12}Cl_{11}$].

| | |
|---|---|
| Probenkürzel | BW23 |
| Summenformel | $C_{15}H_{17}AgB_{12}Cl_{11}N$ |
| Molmasse | 838.84 |
| Temperatur (in K) | 150(1) |
| Kristallsystem | Orthorhombisch |
| Raumgruppe | *Pbca* |
| *a* (in Å) | 11.6524(6) |
| *b* (in Å) | 18.2149(9) |
| *c* (in Å) | 28.0167(16) |
| α (in °) | 90 |
| β (in °) | 90 |
| γ (in °) | 90 |
| Zellvolumen (in $Å^3$) | 5946.5(5) |
| *Z* | 8 |
| Berechnete Dichte (in $g/cm^3$) | 1.874 |
| Absorptionskoeffizient (in $mm^{-1}$) | 1.682 |
| F(000) | 3264.0 |
| Kristallgröße (in $mm^3$) | 0.8 × 0.13 × 0.12 |
| Strahlung | MoKα (λ = 0.71073) |
| 2Θ Bereich der Datenmessung (in °) | 4.396 bis 52 |
| Indexbereich | $-14 \leq h \leq 13$, $-22 \leq k \leq 15$, $-24 \leq l \leq 34$ |
| Gemessene Reflexe | 26898 |
| Unabhängige Reflexe | 5826 [$R_{int}$ = 0.0265, $R_{sigma}$ = 0.0198] |
| Daten/Beschränkungen/Parameter | 5826/0/364 |
| Anpassungsgüte $F^2$ | 1.063 |
| Finaler *R* Wert [I>=2s (I)] | $R_1$ = 0.0240, $wR_2$ = 0.0605 |
| Finaler *R* Wert [alle Daten] | $R_1$ = 0.0290, $wR_2$ = 0.0626 |

| | |
|---|---|
| Größtes Maximum und Minimum (in $Å^{-3}$) | 0.75/-0.65 |

**Tabelle 2** Atomkoordinaten ($\times 10^4$) und äquivalente, isotrope Auslenkungsparameter ($Å^2 \times 10^3$) von [Ag($\eta^2$-Acenaphthen)][$Me_3NB_{12}Cl_{11}$]. $U_{äq}$ ist als ein Drittel der Spur des orthogonalisierten $U_{IJ}$ Tensors definiert.

| Atom | x | y | z | U(äq) |
|---|---|---|---|---|
| Ag(1) | 4171.6(2) | 5355.5(2) | 6337.7(2) | 27.17(6) |
| Cl(7) | 1678.0(4) | 1538.2(3) | 5642.2(2) | 18.03(12) |
| Cl(11) | 2230.1(4) | 1347.5(3) | 6915.8(2) | 17.16(11) |
| Cl(5) | 4424.9(4) | 4272.3(3) | 5596.1(2) | 17.11(11) |
| Cl(4) | 5048.3(4) | 4068.9(3) | 6879.6(2) | 18.15(11) |
| Cl(6) | 4140.2(4) | 2379.1(3) | 5071.0(2) | 20.46(12) |
| Cl(8) | 1705.9(4) | 3536.1(3) | 5364.2(2) | 21.06(12) |
| Cl(12) | 493.0(4) | 2894.5(3) | 6497.9(2) | 20.60(12) |
| Cl(2) | 4353.3(5) | 988.1(3) | 6051.4(2) | 21.70(12) |
| Cl(9) | 2313.9(5) | 4523.0(3) | 6463.1(2) | 21.05(12) |
| Cl(3) | 5054.4(5) | 2008.2(3) | 7171.0(2) | 23.50(13) |
| Cl(10) | 2638.1(5) | 3176.4(3) | 7425.3(2) | 24.37(13) |
| N(18) | 6206.6(14) | 2682.8(10) | 6028.1(6) | 14.0(4) |
| C(7) | 7619.0(18) | 5143.0(12) | 6330.6(8) | 16.7(4) |
| C(13) | 8591.8(19) | 4683.8(12) | 6314.2(8) | 16.8(5) |
| C(6) | 7392.0(19) | 5399.7(12) | 6795.0(9) | 20.9(5) |
| C(12) | 8961.9(19) | 4432.6(13) | 5879.2(9) | 20.8(5) |
| C(8) | 7006.1(19) | 5346.3(13) | 5920.2(9) | 23.1(5) |
| C(15) | 8268(2) | 5082.8(14) | 7133.3(9) | 26.0(5) |
| C(9) | 6092(2) | 5853.3(14) | 5998.7(11) | 29.6(6) |
| C(14) | 9056(2) | 4601.9(14) | 6815.6(9) | 23.7(5) |
| B(3) | 4256(2) | 2410.8(13) | 6694.7(8) | 12.3(5) |
| C(10) | 7406(2) | 5074.0(14) | 5478.3(9) | 27.1(6) |
| B(5) | 3976.0(19) | 3477.1(13) | 5931.3(8) | 11.0(4) |
| B(4) | 4264.9(19) | 3375.9(13) | 6556.4(8) | 11.4(4) |
| C(11) | 8359(2) | 4636.4(14) | 5462.3(9) | 26.5(5) |
| B(11) | 2836.2(19) | 2087.9(13) | 6575.7(8) | 10.9(4) |
| B(6) | 3795.7(19) | 2585.2(13) | 5680.3(8) | 11.6(4) |
| B(12) | 1981.2(19) | 2831.0(13) | 6371.2(8) | 11.8(5) |

| | | | | |
|---|---|---|---|---|
| B(1) | 4873.4(19) | 2728.7(13) | 6139.5(8) | 10.9(4) |
| B(7) | 2558.0(19) | 2186.6(13) | 5956.0(8) | 10.8(4) |
| B(2) | 3950(2) | 1920.3(13) | 6151.5(8) | 12.1(5) |
| B(9) | 2869(2) | 3617.1(13) | 6356.7(8) | 12.4(5) |
| C(5) | 6509(2) | 5888.7(14) | 6861.0(11) | 30.7(6) |
| C(4) | 5874.4(19) | 6115.5(14) | 6450.6(12) | 33.3(7) |
| C(1) | 6555(2) | 1926.4(14) | 5880.4(11) | 31.3(6) |
| B(10) | 3034(2) | 2971.7(13) | 6826.8(8) | 12.2(5) |
| B(8) | 2570.8(19) | 3136.7(13) | 5819.4(8) | 12.2(4) |
| C(3) | 6550(2) | 3184.2(17) | 5628.5(11) | 37.1(7) |
| C(2) | 6944(2) | 2885(2) | 6444.1(10) | 40.1(8) |

## 1.2 [Ag($\eta^1$-Mesitylen)][$Me_3NB_{12}Cl_{11}$]

**Tabelle 3** Kristalldaten und Strukturverfeinerung von [Ag($\eta^1$-Mesitylen)][$Me_3NB_{12}Cl_{11}$].

| | |
|---|---|
| Probenkürzel | BW37 |
| Summenformel | $C_{12}H_{21}AgB_{12}Cl_{11}N$ |
| Molmasse | 806.84 |
| Temperatur (in K) | 150.00(10) |
| Kristallsystem | Monoklin |
| Raumgruppe | $P2_1/n$ |
| *a* (in Å) | 9.2852(6) |
| *b* (in Å) | 30.766(2) |
| *c* (in Å) | 10.1831(7) |
| α (in °) | 90 |
| β (in °) | 93.975(6) |
| γ (in °) | 90 |
| Zellvolumen (in $Å^3$) | 2902.0(3) |
| *Z* | 4 |
| Berechnete Dichte (in $g/cm^3$) | 1.847 |
| Absorptionskoeffizient (in $mm^{-1}$) | 1.719 |
| F(000) | 1576.0 |
| Kristallgröße (in $mm^3$) | 0.14 × 0.13 × 0.09 |

| | |
|---|---|
| Strahlung | MoKα (λ = 0.71073) |
| 2Θ Bereich der Datenmessung (in °) | 5.134 bis 53.994 |
| Indexbereich | -9 ≤ h ≤ 11, -38 ≤ k ≤ 38, -12 ≤ l ≤ 13 |
| Gemessene Reflexe | 15345 |
| Unabhängige Reflexe | 6202 [$R_{int}$ = 0.0320, $R_{sigma}$ = 0.0462] |
| Daten/Beschränkungen/Parameter | 6202/0/340 |
| Anpassungsgüte $F^2$ | 1.106 |
| Finaler $R$ Wert [I>=2s (I)] | $R_1$ = 0.0401, $wR_2$ = 0.0770 |
| Finaler $R$ Wert [alle Daten] | $R_1$ = 0.0517, $wR_2$ = 0.0803 |
| Größtes Maximum und Minimum (in $Å^{-3}$) | 0.51/-0.58 |

**Tabelle 4** Atomkoordinaten ($\times 10^4$) und äquivalente, isotrope Auslenkungsparameter ($Å^2 \times 10^3$) von [Ag($\eta^1$-Mesitylen)][$Me_3NB_{12}Cl_{11}$]. $U_{äq}$ ist als ein Drittel der Spur des orthogonalisierten $U_{IJ}$ Tensors definiert.

| Atom | x | y | z | U(äq) |
|---|---|---|---|---|
| Ag(1) | 441.4(3) | 5899.1(2) | 3607.2(3) | 26.99(9) |
| Cl(8) | 8495.2(8) | 6608.0(3) | 3702.6(8) | 18.75(18) |
| Cl(4) | 1614.9(8) | 6334.4(3) | 5867.9(8) | 21.55(19) |
| Cl(7) | 5433.3(9) | 7251.7(3) | 2425.8(7) | 19.33(18) |
| Cl(11) | 2170.0(9) | 6589.1(3) | 2611.6(8) | 21.80(19) |
| Cl(12) | 5504.8(9) | 6074.2(3) | 1741.0(7) | 22.10(19) |
| Cl(5) | 3008.9(9) | 7417.3(3) | 4987.6(8) | 23.6(2) |
| Cl(3) | 4806.2(9) | 5692.6(3) | 7324.2(8) | 21.37(18) |
| Cl(10) | 3214.0(10) | 5539.5(3) | 4013.2(8) | 23.9(2) |
| Cl(6) | 7051.2(9) | 7421.6(3) | 5712.5(8) | 23.7(2) |
| Cl(2) | 8182.2(9) | 6343.2(3) | 7000.1(8) | 24.6(2) |
| Cl(9) | 7172.5(10) | 5547.2(3) | 4706.1(8) | 27.5(2) |
| N(1) | 4682(3) | 6856.1(9) | 7921(2) | 15.2(6) |
| C(4) | -62(4) | 5074.0(11) | 2231(3) | 20.3(7) |
| C(8) | -39(4) | 5745.8(12) | 960(3) | 20.9(8) |
| C(9) | -693(4) | 5471.7(12) | 1832(3) | 19.9(7) |
| C(6) | 1911(4) | 5228.3(12) | 872(3) | 21.5(8) |
| C(7) | 1273(4) | 5616.2(12) | 500(3) | 22.5(8) |
| C(5) | 1240(4) | 4959.2(12) | 1741(3) | 21.5(8) |
| C(1) | 5905(4) | 6715.2(13) | 8859(3) | 26.4(9) |

| | | | | |
|---|---|---|---|---|
| C(12) | -777(4) | 6150.8(14) | 429(3) | 31.8(9) |
| C(10) | -795(4) | 4763.6(13) | 3106(4) | 33.3(9) |
| C(2) | 4612(4) | 7343.5(11) | 8016(3) | 24.4(8) |
| B(12) | 5308(4) | 6285.5(12) | 3351(3) | 13.0(7) |
| B(11) | 3699(4) | 6534.1(12) | 3774(3) | 11.8(7) |
| B(5) | 4097(4) | 6943.4(12) | 4996(3) | 12.4(7) |
| B(7) | 5260(4) | 6854.9(12) | 3684(3) | 12.3(7) |
| B(10) | 4204(4) | 6021.5(12) | 4458(3) | 14.5(8) |
| C(11) | 3325(4) | 5096.2(14) | 369(4) | 30.3(9) |
| B(6) | 6020(4) | 6945.5(12) | 5331(3) | 12.4(7) |
| B(8) | 6735(4) | 6536.4(12) | 4310(3) | 12.4(7) |
| B(3) | 4954(4) | 6107.5(12) | 6110(3) | 13.6(7) |
| B(4) | 3453(4) | 6423.1(12) | 5463(3) | 12.9(7) |
| B(2) | 6545(4) | 6427.5(12) | 6002(3) | 12.5(7) |
| B(9) | 6104(4) | 6025.0(12) | 4788(3) | 15.0(8) |
| B(1) | 4888(4) | 6679.9(12) | 6468(3) | 12.9(7) |
| C(3) | 3318(4) | 6690.6(14) | 8459(4) | 36.6(11) |

## 1.3 [Ag(Pyridin)$_4$][Me$_3$NB$_{12}$Cl$_{11}$]

**Tabelle 5** Kristalldaten und Strukturverfeinerung von [Ag(Pyridin)$_4$][Me$_3$NB$_{12}$Cl$_{11}$].

| | |
|---|---|
| Probenkürzel | bw25 |
| Summenformel | $C_{21.33}H_{27.33}AgB_{12}Cl_{11}N_{4.74}$ |
| Molmasse | 977.67 |
| Temperatur (in K) | 150.0(10) |
| Kristallsystem | Monoklin |
| Raumgruppe | $P2_1/n$ |
| *a* (in Å) | 13.9243(3) |
| *b* (in Å) | 11.3502(2) |
| *c* (in Å) | 25.9610(5) |
| α (in °) | 90.00 |
| β (in °) | 93.2781(19) |
| γ (in °) | 90.00 |

| | |
|---|---|
| Zellvolumen (in $Å^3$) | 4096.25(15) |
| *Z* | 4 |
| Berechnete Dichte (in $g/cm^3$) | 1.585 |
| Absorptionskoeffizient (in $mm^{-1}$) | 1.236 |
| F(000) | 1930.0 |
| Kristallgröße (in $mm^3$) | 0.16 × 0.1 × 0.1 |
| Strahlung | MoKα (λ = 0.71073) |
| 2Θ Bereich der Datenmessung (in °) | 3.92 bis 52 |
| Indexbereich | $-17 \leq h \leq 17$, $-14 \leq k \leq 10$, $-32 \leq l \leq 30$ |
| Gemessene Reflexe | 20109 |
| Unabhängige Reflexe | 7999 [$R_{int}$ = 0.0263, $R_{sigma}$ = 0.0323] |
| Daten/Beschränkungen/Parameter | 7999/36/580 |
| Anpassungsgüte $F^2$ | 1.071 |
| Finaler *R* Wert [I>=2s (I)] | $R_1$ = 0.0491, $wR_2$ = 0.1326 |
| Finaler *R* Wert [alle Daten] | $R_1$ = 0.0648, $wR_2$ = 0.1417 |
| Größtes Maximum und Minimum (in $Å^{-3}$) | 1.22/-0.54 |

**Tabelle 6** Atomkoordinaten ($\times 10^4$) und äquivalente, isotrope Auslenkungsparameter ($Å^2 \times 10^3$) von $[Ag(Pyridin)_4][Me_3NB_{12}Cl_{11}]$. $U_{äq}$ ist als ein Drittel der Spur des orthogonalisierten $U_{IJ}$ Tensors definiert.

| Atom | x | y | z | U(eq) |
|---|---|---|---|---|
| Ag(1) | 2516.5(3) | 5053.3(4) | 3341.76(16) | 44.94(14) |
| Cl(11) | 1119.9(6) | 698.1(8) | 7074.5(3) | 31.8(2) |
| Cl(7) | 3757.3(6) | 716.7(8) | 7002.5(3) | 30.5(2) |
| Cl(6) | 2471.9(7) | 3231.9(7) | 7403.3(3) | 34.8(2) |
| Cl(12) | 2271.4(7) | -805.5(7) | 6014.2(3) | 34.7(2) |
| Cl(9) | 2058.2(9) | 1127.3(9) | 4877.8(3) | 46.6(3) |
| Cl(10) | 71.7(7) | 916.7(9) | 5743.2(4) | 46.0(3) |
| Cl(8) | 4298.3(8) | 995.4(10) | 5651.1(4) | 47.6(3) |
| Cl(5) | 4473.2(8) | 3480(1) | 6522.5(4) | 50.3(3) |
| Cl(2) | 154.8(8) | 3437.2(10) | 6603.2(4) | 50.3(3) |
| Cl(3) | 700.5(10) | 3645.2(9) | 5207.0(4) | 55.9(3) |
| Cl(4) | 3385.8(10) | 3772.7(11) | 5163.3(4) | 60.6(3) |
| N(1) | 2231(3) | 5186(3) | 6237.8(13) | 47.8(9) |
| N(3) | 1224(3) | 3692(4) | 3419.8(18) | 60.3(11) |

| | | | | |
|---|---|---|---|---|
| N(2) | 3847(3) | 3631(4) | 3458(2) | 65.8(13) |
| N(4A) | 2724(3) | 6096(4) | 4083(2) | 37.6(10) |
| B(6) | 2357(3) | 2802(3) | 6740.2(14) | 24.8(8) |
| B(10) | 1189(3) | 1603(3) | 5942.4(15) | 27.7(8) |
| B(11) | 1702(3) | 1485(3) | 6584.9(14) | 23.1(7) |
| B(12) | 2258(3) | 765(3) | 6069.9(14) | 22.9(7) |
| B(7) | 2973(3) | 1506(3) | 6554.8(14) | 22.7(7) |
| B(8) | 3248(3) | 1641(4) | 5895.6(14) | 28.0(8) |
| B(9) | 2147(3) | 1703(3) | 5520.3(14) | 29.1(8) |
| C(15A) | 2187(14) | 7490(12) | 4703(7) | 52(4) |
| C(17A) | 3869(9) | 7001(19) | 4720(5) | 50(3) |
| C(18A) | 3600(7) | 6212(10) | 4292(3) | 38(2) |
| B(5) | 3312(3) | 2909(3) | 6314.7(15) | 28.8(8) |
| B(2) | 1247(3) | 2877(3) | 6355.7(15) | 30.0(8) |
| B(1) | 2239(3) | 3783(3) | 6186.7(15) | 33.1(9) |
| B(3) | 1518(3) | 3002(3) | 5690.7(15) | 32.5(9) |
| C(12) | 470(4) | 2371(4) | 3903(2) | 67.3(15) |
| C(8) | 3850(4) | 3080(6) | 3880(2) | 71.2(16) |
| C(13) | 1072(4) | 3263(5) | 3842(2) | 64.4(14) |
| C(11) | -30(4) | 1888(5) | 3478(2) | 62.6(14) |
| C(5) | 5147(4) | 2420(6) | 3208(2) | 69.3(15) |
| C(4) | 4494(4) | 3317(5) | 3105(2) | 70.2(16) |
| B(4) | 2793(3) | 3045(4) | 5668.2(15) | 32.3(9) |
| C(16A) | 3105(8) | 7648(8) | 4900(3) | 52(2) |
| C(6) | 5128(4) | 1868(5) | 3671(2) | 68.7(15) |
| C(14A) | 2022(5) | 6725(8) | 4285(3) | 50(2) |
| C(7) | 4468(4) | 2220(6) | 4008(2) | 75.3(16) |
| N(5A) | 2476(10) | 5494(13) | 2478(4) | 30(3) |
| C(10) | 67(4) | 2326(6) | 3000(2) | 88(2) |
| C(1) | 1489(5) | 5746(4) | 5875(2) | 82(2) |
| C(9) | 735(5) | 3281(6) | 2974(3) | 98(2) |
| C(3) | 1989(7) | 5587(5) | 6756(2) | 106(2) |
| C(2) | 3151(6) | 5734(5) | 6124(5) | 158(5) |

| | | | | |
|---|---|---|---|---|
| C(21) | 2422(8) | 4710(10) | 1434(4) | 34(2) |
| C(19A) | 3222(12) | 5403(11) | 2225(5) | 54(3) |
| C(20A) | 3177(14) | 5011(14) | 1678(7) | 87(7) |
| C(23A) | 1579(9) | 5354(9) | 2203(4) | 42(3) |
| C(22A) | 1526(11) | 5029(11) | 1693(5) | 54(3) |
| Ag(1A) | 2387.1(9) | 4255.9(12) | 4079.3(5) | 49.3(3) |
| C(19B) | 2577(10) | 3839(11) | 2292(5) | 58(3) |
| C(20B) | 2661(12) | 3674(15) | 1773(6) | 71(4) |
| C(21B) | 2443(12) | 5498(17) | 1594(7) | 73(4) |
| C(22B) | 2487(11) | 5726(12) | 2206(9) | 62(5) |
| N(5B) | 2549(9) | 4976(11) | 2449(5) | 39(3) |
| N(4B) | 2718(9) | 6008(10) | 4408(5) | 29(3) |
| C(14B) | 2023(13) | 6793(18) | 4529(8) | 27(4) |
| C(18B) | 3636(17) | 6290(20) | 4505(8) | 24(4) |
| C(16B) | 3240(20) | 8170(20) | 4873(10) | 51(7) |
| C(17B) | 3740(40) | 7200(50) | 4660(20) | 70(20) |
| C(15B) | 2270(30) | 7890(30) | 4730(20) | 32(7) |

## 1.4 $[Cu(PPh_3)_3(CH_3CN)]_2[B_{12}H_{12}]\cdot 2PPh_3\cdot 2CH_3CN$

**Tabelle 7** Kristalldaten und Strukturverfeinerung von $[Cu(PPh_3)_3(CH_3CN)]_2[B_{12}H_{12}]\cdot 2PPh_3\cdot 2CH_3CN$.

| | |
|---|---|
| Probenkürzel | acremt033 |
| Summenformel | $C_{152}H_{144}B_{12}Cu_2N_4P_8$ |
| Molmasse | 2531.64 |
| Temperatur (in K) | 150.0(10) |
| Kristallsystem | Monoklin |
| Raumgruppe | *C*2/c |
| *a* (in Å) | 28.6012(11) |
| *b* (in Å) | 12.7352(4) |
| *c* (in Å) | 38.0186(15) |
| α (in °) | 90 |
| β (in °) | 106.402(4) |
| γ (in °) | 90 |

| | |
|---|---|
| Zellvolumen (in $Å^3$) | 13284.4(9) |
| *Z* | 4 |
| Berechnete Dichte (in $g/cm^3$) | 12.656 |
| Absorptionskoeffizient (in $mm^{-1}$) | 0.471 |
| F(000) | 5295.6 |
| Kristallgröße (in $mm^3$) | 0.21 × 0.12 × 0.11 |
| Strahlung | Mo Kα (λ = 0.71073) |
| 2Θ Bereich der Datenmessung (in °) | 5.14 bis 52 |
| Indexbereich | $-37 \leq h \leq 35$, $-17 \leq k \leq 15$, $-50 \leq l \leq 51$ |
| Gemessene Reflexe | 37443 |
| Unabhängige Reflexe | 13054 [$R_{int}$ = 0.0251, $R_{sigma}$ = 0.0381] |
| Daten/Beschränkungen/Parameter | 13054/0/764 |
| Anpassungsgüte $F^2$ | 1.056 |
| Finaler *R* Wert [I>=2s (I)] | $R_1$ = 0.0400, w$R_2$ = 0.0973 |
| Finaler *R* Wert [alle Daten] | $R_1$ = 0.0517, w$R_2$ = 0.1046 |
| Größtes Maximum und Minimum (in $Å^{-3}$) | 0.78/-0.42 |

**Tabelle 8** Atomkoordinaten (×$10^4$) und äquivalente, isotrope Auslenkungsparameter ($Å^2$×$10^3$) von $[Cu(PPh_3)_3(CH_3CN)]_2[B_{12}H_{12}]\cdot 2PPh_3\cdot 2CH_3CN$. $U_{äq}$ ist als ein Drittel der Spur des orthogonalisierten $U_{IJ}$ Tensors definiert.

| Atom | x | y | z | U(äq) |
|---|---|---|---|---|
| Cu(1) | 8018.68(9) | 4907.53(19) | 6292.40(6) | 18.04(7) |
| P(1) | 7816.64(19) | 6022.5(4) | 5794.35(14) | 17.26(11) |
| P(2) | 7878.45(19) | 3120.1(4) | 6193.09(14) | 18.06(12) |
| P(3) | 7798.6(2) | 5478.5(4) | 6803.73(14) | 20.08(12) |
| N(1) | 8764.1(7) | 4967.5(14) | 6495.1(5) | 26.4(4) |
| C(1) | 7980.6(5) | 7394.0(8) | 5901.0(4) | 20.2(4) |
| C(6) | 8443.4(4) | 7595.5(9) | 6131.9(4) | 25.5(5) |
| C(5) | 8603.2(4) | 8625.0(11) | 6203.9(4) | 33.2(5) |
| C(4) | 8300.2(6) | 9453.0(8) | 6045.0(4) | 34.7(6) |
| C(3) | 7837.5(5) | 9251.5(9) | 5814.1(4) | 34.4(6) |
| C(2) | 7677.7(4) | 8222.0(10) | 5742.1(4) | 26.8(5) |
| C(7) | 8088.0(5) | 5770.8(10) | 5420.0(3) | 18.3(4) |
| C(12) | 8273.0(5) | 6577.8(7) | 5252.5(4) | 23.0(4) |
| C(11) | 8478.8(5) | 6353.6(9) | 4970.7(4) | 28.2(5) |

| | | | | |
|---|---|---|---|---|
| C(10) | 8499.4(6) | 5322.3(11) | 4856.3(3) | 30.5(5) |
| C(9) | 8314.4(6) | 4515.2(8) | 5023.7(4) | 33.2(5) |
| C(8) | 8108.7(5) | 4739.5(8) | 5305.6(4) | 27.5(5) |
| C(13) | 7165.2(3) | 6067.5(11) | 5556.0(3) | 19.4(4) |
| C(14) | 6983.6(4) | 5843.7(11) | 5184.5(3) | 23.2(4) |
| C(15) | 6483.4(5) | 5782.1(12) | 5024.2(3) | 29.7(5) |
| C(16) | 6164.8(3) | 5944.3(13) | 5235.5(4) | 35.2(6) |
| C(17) | 6346.3(4) | 6168.2(13) | 5607.1(4) | 35.1(6) |
| C(18) | 6846.6(5) | 6229.8(12) | 5767.3(3) | 27.3(5) |
| C(19) | 8092.5(5) | 2347.8(11) | 6615.2(3) | 22.4(4) |
| C(24) | 7801.5(4) | 2341.6(12) | 6852.6(4) | 28.3(5) |
| C(23) | 7962.0(6) | 1840.5(14) | 7190.2(4) | 38.9(6) |
| C(22) | 8413.6(6) | 1345.7(14) | 7290.3(3) | 46.6(7) |
| C(21) | 8704.6(5) | 1352.0(13) | 7052.8(4) | 45.1(7) |
| C(20) | 8544.0(5) | 1853.0(13) | 6715.3(4) | 33.4(5) |
| C(25) | 8192.7(4) | 2562.3(11) | 5878.0(3) | 20.7(4) |
| C(26) | 7942.2(4) | 2003.2(12) | 5568.1(4) | 25.0(5) |
| C(27) | 8187.4(5) | 1637.8(12) | 5324.6(3) | 32.6(5) |
| C(28) | 8683.1(5) | 1831.3(13) | 5391.0(4) | 35.2(6) |
| C(29) | 8933.6(4) | 2390.4(13) | 5701.0(4) | 34.1(6) |
| C(30) | 8688.4(4) | 2755.8(12) | 5944.5(3) | 27.4(5) |
| C(31) | 7259.9(3) | 2636.3(10) | 6002.1(4) | 19.8(4) |
| C(32) | 6919.0(5) | 3293.3(8) | 5772.4(4) | 23.8(5) |
| C(33) | 6441.9(4) | 2953.0(11) | 5625.0(4) | 30.6(5) |
| C(34) | 6305.6(4) | 1955.6(11) | 5707.3(4) | 32.3(5) |
| C(35) | 6646.5(5) | 1298.6(9) | 5937.0(4) | 30.2(5) |
| C(36) | 7123.6(4) | 1638.9(9) | 6084.3(4) | 24.6(5) |
| C(37) | 7639.8(5) | 6871.4(8) | 6816.3(4) | 25.0(5) |
| C(38) | 7175.9(5) | 7202.6(10) | 6814.0(5) | 33.6(5) |
| C(39) | 7070.1(5) | 8268.8(12) | 6808.8(5) | 46.6(7) |
| C(40) | 7428.3(7) | 9003.8(8) | 6806.0(5) | 48.2(8) |
| C(41) | 7892.3(6) | 8672.6(10) | 6808.4(5) | 42.7(7) |
| C(42) | 7998.0(4) | 7606.4(12) | 6813.5(4) | 33.3(6) |

| | | | | |
|---|---|---|---|---|
| C(43) | 8257.5(5) | 5290.6(11) | 7243.4(3) | 24.7(5) |
| C(48) | 8540.3(6) | 4385.9(10) | 7289.3(3) | 28.8(5) |
| C(47) | 8879.0(5) | 4182.1(10) | 7624.4(4) | 35.4(6) |
| C(46) | 8935.0(6) | 4883.0(13) | 7913.6(3) | 40.2(6) |
| C(45) | 8652.3(6) | 5787.7(12) | 7867.7(3) | 42.0(7) |
| C(44) | 8313.5(6) | 5991.5(10) | 7532.6(4) | 33.1(5) |
| C(49) | 7265.2(4) | 4790.7(11) | 6861.1(3) | 20.7(4) |
| C(50) | 6901.6(5) | 4534.1(12) | 6544.3(3) | 26.1(5) |
| C(51) | 6487.5(4) | 4002.0(13) | 6568.6(3) | 32.8(5) |
| C(52) | 6437.1(5) | 3726.4(12) | 6909.7(4) | 34.4(6) |
| C(53) | 6800.7(6) | 3983.0(13) | 7226.6(3) | 35.1(6) |
| C(54) | 7214.8(5) | 4515.2(12) | 7202.3(3) | 29.0(5) |
| C(75) | 9152.2(9) | 5029.3(18) | 6674.4(7) | 30.9(5) |
| P(4) | 5633.9(2) | 8863.5(5) | 5934.31(18) | 32.16(15) |
| C(55) | 5299.0(5) | 8159.0(12) | 5518.2(3) | 30.9(5) |
| C(56) | 5125.4(6) | 7141.7(12) | 5524.3(3) | 31.5(5) |
| C(57) | 4908.3(6) | 6622.2(11) | 5197.4(4) | 40.2(6) |
| C(58) | 4864.8(6) | 7120.2(15) | 4864.3(3) | 48.1(8) |
| C(59) | 5038.4(7) | 8137.5(16) | 4858.2(3) | 54.4(8) |
| C(60) | 5255.4(6) | 8657.0(12) | 5185.1(5) | 45.5(7) |
| C(61) | 5229.8(5) | 9965.5(10) | 5949.8(4) | 27.6(5) |
| C(66) | 5445.7(4) | 10822.2(12) | 6160.1(4) | 32.1(5) |
| C(65) | 5160.7(6) | 11662.7(11) | 6209.6(5) | 48.2(7) |
| C(64) | 4659.8(6) | 11646.4(13) | 6048.8(5) | 53.9(8) |
| C(63) | 4443.9(4) | 10789.7(15) | 5838.5(5) | 48.1(7) |
| C(62) | 4728.9(5) | 9949.2(12) | 5789.0(4) | 36.8(6) |
| C(67A) | 5588(3) | 7991(7) | 6282(3) | 31(2) |
| C(72A) | 5192(3) | 7978(6) | 6426(3) | 37.1(19) |
| C(71A) | 5164(2) | 7220(5) | 6683(2) | 47.8(19) |
| C(70A) | 5531(2) | 6476(4) | 6795.0(15) | 49(3) |
| C(69A) | 5927.0(18) | 6489(4) | 6651.0(16) | 47(2) |
| C(68A) | 5955(2) | 7246(7) | 6395(2) | 38.7(19) |
| C(67B) | 5470(3) | 8020(7) | 6301(3) | 28.6(19) |

| | | | | |
|---|---|---|---|---|
| C(72B) | 5054(3) | 8163(6) | 6415(3) | 37.0(18) |
| C(71B) | 4965(2) | 7512(5) | 6683(2) | 47.2(19) |
| C(70B) | 5293(2) | 6718(4) | 6836.0(14) | 50.2(19) |
| C(69B) | 5709(2) | 6575(4) | 6721.5(15) | 46(3) |
| C(68B) | 5798(2) | 7226(6) | 6454(2) | 38.8(19) |
| N(2) | 4574.2(14) | 5409(3) | 4151.0(11) | 86.0(10) |
| C(73) | 4887.8(15) | 5337(3) | 4024.1(10) | 60.4(9) |
| C(74) | 5278(2) | 5213(4) | 3869.4(15) | 109.5(17) |
| B(1) | 9454(3) | 8801(7) | 7442(2) | 37.5(16) |
| B(2) | 9859(3) | 8812(6) | 7888(2) | 39.2(17) |
| B(3) | 9715(2) | 7605(6) | 7652(2) | 33.7(15) |
| B(4) | 9677(8) | 7943(16) | 7179(5) | 52(5) |
| B(5) | 9789(3) | 9190(8) | 7140(2) | 38(2) |
| B(6) | 9923(3) | 9775(4) | 7571.4(16) | 42.4(19) |
| B(7) | 9353(2) | 8201(7) | 7404.1(17) | 34.9(14) |
| B(8) | 9769(2) | 8182(8) | 7847.1(18) | 36.5(15) |
| B(9) | 9809(2) | 7240(6) | 7518.4(18) | 31.8(14) |
| B(10) | 9652(7) | 7766(13) | 7101(4) | 28(2) |
| B(11) | 9544(4) | 9175(7) | 7141(2) | 43(2) |
| B(12) | 9592(2) | 9370(6) | 7611.7(16) | 31.8(13) |
| C(76A) | 9614(2) | 5068(5) | 6967.0(19) | 45.1(17) |
| C(76B) | 9671(2) | 5116(5) | 6861.2(17) | 33.1(14) |

## 1.5 $[Cu(PPh_3)_2(CH_3CN)_2]_2[B_{12}Cl_{12}]$

**Tabelle 9** Kristalldaten und Strukturverfeinerung von $[Cu(PPh_3)_2(CH_3CN)_2]_2[B_{12}Cl_{12}]$.

| | |
|---|---|
| Probenkürzel | bw51 |
| Summenformel | $C_{80}H_{72}B_{12}Cl_{12}Cu_2N_4P_4$ |
| Molmasse | 1895.49 |
| Temperatur (in K) | 150.0(10) |
| Kristallsystem | Monoklin |
| Raumgruppe | C2/c |
| *a* (in Å) | 25.4199(18) |

| | |
|---|---|
| *b* (in Å) | 15.8245(10) |
| *c* (in Å) | 21.9072(13) |
| α (in °) | 90 |
| β (in °) | 94.429(5) |
| γ (in °) | 90 |
| Zellvolumen (in $Å^3$) | 8786.0(10) |
| *Z* | 4 |
| Berechnete Dichte (in $g/cm^3$) | 1.433 |
| Absorptionskoeffizient (in $mm^{-1}$) | 0.968 |
| F(000) | 3848.0 |
| Kristallgröße (in $mm^3$) | 0.47 × 0.19 × 0.15 |
| Strahlung | MoKα (λ = 0.71073) |
| 2Θ Bereich der Datenmessung (in °) | 4.904 bis 53.998 |
| Indexbereich | -31 ≤ h ≤ 31, -20 ≤ k ≤ 20, -27 ≤ l ≤ 27 |
| Gemessene Reflexe | 23658 |
| Unabhängige Reflexe | 9420 [$R_{int}$ = 0.0232, $R_{sigma}$ = 0.0245] |
| Daten/Beschränkungen/Parameter | 9420/0/516 |
| Anpassungsgüte $F^2$ | 1.043 |
| Finaler *R* Wert [I>=2s (I)] | $R_1$ = 0.0316, w$R_2$ = 0.0769 |
| Finaler *R* Wert [alle Daten] | $R_1$ = 0.0399, w$R_2$ = 0.0805 |
| Größtes Maximum und Minimum (in $Å^{-3}$) | 0.68/-0.43 |

**Tabelle 10** Atomkoordinaten ($\times 10^4$) und äquivalente, isotrope Auslenkungsparameter ($Å^2 \times 10^3$) von $[Cu(PPh_3)_2(CH_3CN)_2]_2[B_{12}Cl_{12}]$. $U_{äq}$ ist als ein Drittel der Spur des orthogonalisierten $U_{IJ}$ Tensors definiert.

| Atom | x | y | z | U(äq) |
|---|---|---|---|---|
| Cu1 | 2076.6(2) | 5037.8(2) | 5514.7(2) | 21.57(6) |
| Cl5 | 5059.6(2) | 5092.9(3) | 913.1(2) | 23.25(10) |
| Cl6 | 4660.0(2) | 3194.2(3) | 1751.7(2) | 26.81(10) |
| Cl1 | 3813.7(2) | 5048.5(3) | 1600.0(2) | 28.11(11) |
| P2 | 1576.5(2) | 4301.1(3) | 4795.0(2) | 17.95(9) |
| P1 | 1761.8(2) | 6155.4(3) | 6009.0(2) | 20.1(1) |
| Cl2 | 3982.9(2) | 3928.0(3) | 3067.0(2) | 28.80(11) |
| Cl3 | 3978.5(2) | 6219.0(3) | 3065.2(2) | 28.87(11) |
| Cl4 | 4654.1(2) | 6942.4(3) | 1730.5(2) | 30.08(11) |

| | | | | |
|---|---|---|---|---|
| N1 | 2451.2(7) | 4195.0(11) | 6105.7(8) | 30.7(4) |
| N2 | 2729.8(7) | 5356.4(12) | 5077.4(8) | 34.6(4) |
| C19 | 1024.4(7) | 4863.0(11) | 4405.7(8) | 21.7(4) |
| C31 | 1995.0(7) | 4003.8(11) | 4192.3(8) | 20.3(3) |
| C13 | 1379.3(7) | 5855.0(12) | 6645.0(8) | 22.7(4) |
| C7 | 1332.7(7) | 6864.0(11) | 5546.6(8) | 22.7(4) |
| C25 | 1285.1(7) | 3306.6(11) | 5008.2(8) | 20.6(3) |
| C1 | 2258.2(7) | 6888.1(12) | 6345.0(8) | 22.3(4) |
| C30 | 1048.6(7) | 3270.5(12) | 5557.7(9) | 27.4(4) |
| C6 | 2747.5(7) | 6570.2(14) | 6548.9(9) | 30.0(4) |
| C14 | 1315.1(8) | 5004.9(12) | 6764.9(9) | 27.4(4) |
| C20 | 517.3(8) | 4547.9(14) | 4342.3(10) | 33.8(5) |
| C37 | 2693.7(8) | 3768.3(13) | 6428.5(9) | 30.1(4) |
| C32 | 2447.7(7) | 3540.3(13) | 4362.1(9) | 29.6(4) |
| C12 | 1551.1(8) | 7301.8(13) | 5077.0(9) | 31.0(4) |
| C24 | 1121.0(8) | 5669.9(12) | 4188.1(9) | 29.1(4) |
| C23 | 716.6(9) | 6137.9(13) | 3901.7(9) | 36.2(5) |
| C22 | 213.0(9) | 5818.6(14) | 3841.3(10) | 36.9(5) |
| C28 | 781.6(8) | 1844.2(13) | 5353.1(10) | 33.1(5) |
| C36 | 1904.4(8) | 4260.3(13) | 3592.6(8) | 29.4(4) |
| B6 | 4831.5(7) | 4165.0(12) | 2131.9(8) | 16.7(4) |
| B2 | 4504.1(7) | 4514.0(12) | 2773.5(8) | 16.9(4) |
| B5 | 5033.6(7) | 5074.7(12) | 1733.4(8) | 16.5(4) |
| C39 | 3090.5(9) | 5325.7(13) | 4811(1) | 35.7(5) |
| C29 | 797.3(8) | 2542.2(13) | 5727.2(10) | 33.2(5) |
| C26 | 1262.0(8) | 2601.4(13) | 4635.7(9) | 31.3(4) |
| C18 | 1136.2(9) | 6442.6(14) | 7001.1(9) | 34.9(5) |
| B1 | 4420.6(7) | 5070.5(12) | 2069.5(9) | 17.4(4) |
| C27 | 1012.4(9) | 1871.6(13) | 4809.6(10) | 35.9(5) |
| B4 | 4830.5(8) | 5983.8(12) | 2128.8(8) | 18.1(4) |
| C15 | 1003.2(8) | 4740.5(14) | 7218.1(9) | 34.4(5) |
| C5 | 3137.2(8) | 7100.9(16) | 6797.9(10) | 38.2(5) |
| C33 | 2798.7(8) | 3334.5(16) | 3935.5(11) | 41.2(6) |

| | | | | |
|---|---|---|---|---|
| B3 | 4504.3(7) | 5634.9(12) | 2771.6(8) | 17.8(4) |
| C8 | 806.6(8) | 6989.7(14) | 5636.7(9) | 34.2(5) |
| C21 | 113.3(9) | 5031.2(16) | 4060.7(11) | 43.0(6) |
| C2 | 2167.5(8) | 7752.0(13) | 6392.2(10) | 33.3(4) |
| C11 | 1244.8(10) | 7863.7(14) | 4717.6(9) | 40.5(5) |
| C34 | 2705.3(8) | 3597.5(17) | 3339.7(10) | 43.9(6) |
| C4 | 3046.6(9) | 7951.2(16) | 6837.8(10) | 42.6(6) |
| C16 | 749.9(9) | 5330.0(16) | 7553.6(10) | 40.0(5) |
| C35 | 2263.1(9) | 4055.3(16) | 3166.8(10) | 40.4(5) |
| C10 | 725.4(10) | 7990.9(15) | 4821.2(10) | 44.5(6) |
| C3 | 2560.9(9) | 8282.1(15) | 6634.1(11) | 43.5(5) |
| C38 | 3013.4(10) | 3229.4(15) | 6846.0(11) | 47.9(6) |
| C17 | 819.6(10) | 6171.4(16) | 7449.5(10) | 43.4(6) |
| C9 | 506.1(9) | 7554.3(17) | 5275.9(11) | 46.0(6) |
| C40 | 3554.7(12) | 5259.8(16) | 4467.0(15) | 62.0(9) |

## 1.6 $[Cu(PPh_3)_3(CH_3CN)]_2[B_{12}Br_{12}]\cdot 6CH_3CN$

**Tabelle 11** Kristalldaten und Strukturverfeinerung von $[Cu(PPh_3)_3(CH_3CN)]_2[B_{12}Br_{12}]\cdot 6CH_3CN$.

| | |
|---|---|
| Probenkürzel | acremt039 |
| Summenformel | $C_{62}H_{57}B_6Br_6CuN_4P_3$ |
| Molmasse | 1559.00 |
| Temperatur (in K) | 150.0(10) |
| Kristallsystem | Triklin |
| Raumgruppe | $P\bar{1}$ |
| *a* (in Å) | 10.1019(4) |
| *b* (in Å) | 18.8350(8) |
| *c* (in Å) | 18.8967(8) |
| α (in °) | 67.278(4) |
| β (in °) | 77.447(3) |
| γ (in °) | 83.664(3) |
| Zellvolumen (in Å$^3$) | 3235.7(3) |
| *Z* | 2 |

| | |
|---|---|
| Berechnete Dichte (in g/cm$^3$) | 15.999 |
| Absorptionskoeffizient (in mm$^{-1}$) | 4.158 |
| F(000) | 1540.5 |
| Kristallgröße (in mm$^3$) | 0.15 × 0.11 × 0.11 |
| Strahlung | MoKα (λ = 0.71073) |
| 2Θ Bereich der Datenmessung (in °) | 4.7 bis 54 |
| Indexbereich | -13 ≤ h ≤ 13, -25 ≤ k ≤ 20, -25 ≤ l ≤ 25 |
| Gemessene Reflexe | 33025 |
| Unabhängige Reflexe | 13864 [$R_{int}$ = 0.0384, $R_{sigma}$ = 0.0570] |
| Daten/Beschränkungen/Parameter | 13864/0/743 |
| Anpassungsgüte $F^2$ | 1.040 |
| Finaler $R$ Wert [I>=2s (I)] | $R_1$ = 0.0355, w$R_2$ = 0.0744 |
| Finaler $R$ Wert [alle Daten] | $R_1$ = 0.0537, w$R_2$ = 0.0830 |
| Größtes Maximum und Minimum (in Å$^{-3}$) | 0.78/-0.75 |

**Tabelle 12** Atomkoordinaten (×10$^4$) und äquivalente, isotrope Auslenkungsparameter (Å$^2$×10$^3$) von $[Cu(PPh_3)_3(CH_3CN)]_2[B_{12}Br_{12}]\cdot 6CH_3CN$. $U_{äq}$ ist als ein Drittel der Spur des orthogonalisierten $U_{IJ}$ Tensors definiert.

| Atom | x | y | z | U(äq) |
|---|---|---|---|---|
| Br(1) | 6429.2(3) | 4584.39(18) | 8253.79(19) | 21.19(8) |
| Br(2) | 3338.9(3) | 5918.19(19) | 8329.86(19) | 23.37(8) |
| Br(3) | 3064.5(3) | 3763.32(18) | 9554.4(2) | 23.99(8) |
| Br(4) | 6371.5(3) | 3053.23(17) | 10311.7(2) | 23.24(8) |
| Br(5) | 8675.0(3) | 4786.92(18) | 9588.4(2) | 22.45(8) |
| Br(6) | 6813.5(3) | 6546.26(17) | 8341.18(19) | 21.27(8) |
| B(1) | 5678(3) | 4803.1(19) | 9188(2) | 15.6(7) |
| B(2) | 4226(3) | 5428.0(19) | 9225(2) | 15.5(7) |
| B(3) | 4104(3) | 4425.6(19) | 9795(2) | 14.9(7) |
| B(4) | 5642(3) | 4093.6(19) | 10146(2) | 14.5(7) |
| B(5) | 6702(3) | 4894.1(19) | 9801(2) | 15.2(7) |
| B(6) | 5841(3) | 5723.2(19) | 9228(2) | 15.0(7) |
| Cu(1) | 2622.4(4) | 1804.7(2) | 6792.8(2) | 16.75(9) |
| P(1) | 2015.6(8) | 3076.3(4) | 6111.6(5) | 18.03(18) |
| P(2) | 3536.4(8) | 1049.9(4) | 6085.6(5) | 16.66(17) |
| P(3) | 1052.5(8) | 1211.0(4) | 7900.0(5) | 15.40(17) |

| | | | | |
|---|---|---|---|---|
| N(1) | 4154(3) | 2021.6(15) | 7266.5(17) | 24.4(6) |
| C(1) | 2698(3) | 3775.1(17) | 6379.6(19) | 19.5(7) |
| C(2) | 2579(3) | 3642.5(18) | 7159(2) | 26.3(8) |
| C(3) | 3108(4) | 4146(2) | 7385(2) | 34.8(9) |
| C(4) | 3770(4) | 4790(2) | 6838(2) | 31.2(8) |
| C(5) | 3872(4) | 4934(2) | 6056(2) | 37.5(9) |
| C(6) | 3343(3) | 4433.7(18) | 5823(2) | 29.5(8) |
| C(7) | 229(3) | 3355.4(17) | 6149.4(19) | 21.3(7) |
| C(8) | -283(4) | 4067(2) | 6166(2) | 37.3(10) |
| C(9) | -1657(4) | 4242(2) | 6199(3) | 49.4(12) |
| C(10) | -2519(4) | 3729(2) | 6199(3) | 42.9(10) |
| C(11) | -2016(4) | 3028(2) | 6183(3) | 40.5(10) |
| C(12) | -662(3) | 2840(2) | 6161(2) | 29.3(8) |
| C(13) | 2717(3) | 3358.5(16) | 5075.8(19) | 18.5(7) |
| C(14) | 1936(4) | 3613.7(18) | 4501(2) | 25.9(8) |
| C(15) | 2545(4) | 3732.2(19) | 3732(2) | 33.5(9) |
| C(16) | 3928(4) | 3607.9(19) | 3532(2) | 32.8(9) |
| C(17) | 4707(4) | 3370.6(18) | 4100(2) | 27.9(8) |
| C(18) | 4106(3) | 3242.9(17) | 4865(2) | 22.1(7) |
| C(19) | 5117(3) | 1370.4(16) | 5391.0(19) | 18.2(7) |
| C(20) | 6152(3) | 1602.9(19) | 5626(2) | 24.4(7) |
| C(21) | 7357(3) | 1854(2) | 5117(2) | 31.8(9) |
| C(22) | 7546(3) | 1891.2(19) | 4359(2) | 27.0(8) |
| C(23) | 6528(3) | 1664.4(18) | 4119(2) | 24.7(7) |
| C(24) | 5323(3) | 1400.9(17) | 4628.9(19) | 20.7(7) |
| C(25) | 3937(3) | 48.9(17) | 6665.0(18) | 18.3(7) |
| C(26) | 2923(3) | -393.0(18) | 7238(2) | 25.1(8) |
| C(27) | 3180(4) | -1158.0(19) | 7697(2) | 29.6(8) |
| C(28) | 4450(4) | -1480.8(19) | 7580(2) | 30.2(8) |
| C(29) | 5473(4) | -1051.5(19) | 7020(2) | 31.5(9) |
| C(30) | 5224(3) | -290.5(18) | 6555(2) | 24.1(7) |
| C(31) | 2466(3) | 960.3(17) | 5466.2(19) | 18.2(7) |
| C(32) | 2326(3) | 279.9(18) | 5377(2) | 22.6(7) |

| | | | | |
|---|---|---|---|---|
| C(33) | 1625(3) | 274(2) | 4827(2) | 28.2(8) |
| C(34) | 1071(3) | 952(2) | 4355(2) | 29.0(8) |
| C(35) | 1191(3) | 1629(2) | 4446(2) | 31.9(9) |
| C(36) | 1879(3) | 1634.3(18) | 5000(2) | 27.1(8) |
| C(37) | 1664(3) | 337.2(17) | 8615.4(19) | 18.3(7) |
| C(38) | 2993(3) | 311.7(19) | 8702(2) | 30.5(9) |
| C(39) | 3535(4) | -338.2(19) | 9219(2) | 35.2(9) |
| C(40) | 2767(4) | -976.0(19) | 9640(2) | 29.0(8) |
| C(41) | 1458(4) | -965.7(19) | 9557(2) | 31.8(9) |
| C(42) | 890(3) | -310.5(18) | 9052(2) | 26.9(8) |
| C(43) | -557(3) | 928.4(17) | 7815.6(19) | 19.2(7) |
| C(44) | -629(3) | 766.9(17) | 7167(2) | 22.3(7) |
| C(45) | -1830(4) | 525(2) | 7105(2) | 33.3(9) |
| C(46) | -2965(4) | 451(2) | 7678(2) | 34.6(9) |
| C(47) | -2909(3) | 615(2) | 8321(2) | 31.9(9) |
| C(48) | -1717(3) | 844.5(19) | 8396(2) | 25.9(8) |
| C(49) | 567(3) | 1865.7(16) | 8419.1(18) | 16.2(6) |
| C(50) | 1336(3) | 1896.5(17) | 8930.7(19) | 20.5(7) |
| C(51) | 1065(3) | 2459.6(18) | 9251(2) | 25.4(8) |
| C(52) | 31(4) | 2992.7(19) | 9070(2) | 30.0(8) |
| C(53) | -744(3) | 2966.6(19) | 8565(2) | 29.9(8) |
| C(54) | -468(3) | 2418.7(18) | 8230(2) | 22.9(7) |
| C(55) | 4736(3) | 2239.8(19) | 7586(2) | 27.3(8) |
| C(56) | 5436(4) | 2527(2) | 8018(3) | 47.1(11) |
| N(2) | 3972(4) | 1374(2) | -169(3) | 59.6(11) |
| C(57) | 3626(4) | 1493(2) | 385(3) | 45.1(11) |
| C(58) | 3210(5) | 1635(2) | 1102(3) | 59.5(13) |
| N(3) | 8900(5) | 3660(3) | 3879(3) | 99.4(18) |
| C(59) | 9234(4) | 4050(3) | 3244(4) | 59.5(13) |
| C(60) | 9679(5) | 4546(3) | 2445(3) | 74.0(16) |
| N(4) | 10020(6) | 2015(4) | 2560(3) | 154(3) |
| C(61) | 9921(5) | 2426(4) | 1963(3) | 71.0(16) |
| C(62) | 9824(4) | 2959(3) | 1205(3) | 57.4(13) |

## 1.7 $[Cu(PPh_3)_3(CH_3CN)]_2[B_{12}I_{12}]\cdot 2CH_3CN$

**Tabelle 13** Kristalldaten und Strukturverfeinerung von $[Cu(PPh_3)_3(CH_3CN)]_2[B_{12}I_{12}]\cdot 2CH_3CN$.

| | |
|---|---|
| Probenkürzel | acremt044 |
| Summenformel | $C_{116}H_{102.11}B_{12}Cu_2I_{11.9}N_4P_6$ |
| Molmasse | 3504.21 |
| Temperatur (in K) | 150.0(10) |
| Kristallsystem | Monoklin |
| Raumgruppe | *P*21/c |
| *a* (in Å) | 10.7471(3) |
| *b* (in Å) | 16.8794(4) |
| *c* (in Å) | 34.5816(10) |
| α (in °) | 90 |
| β (in °) | 96.562(2) |
| γ (in °) | 90 |
| Zellvolumen (in $Å^3$) | 6232.2(3) |
| *Z* | 2 |
| Berechnete Dichte (in $g/cm^3$) | 1.867 |
| Absorptionskoeffizient (in $mm^{-1}$) | 3.412 |
| F(000) | 3329.0 |
| Kristallgröße (in $mm^3$) | 0.15 × 0.14 × 0.1 |
| Strahlung | MoKα (λ = 0.71073) |
| 2Θ Bereich der Datenmessung (in °) | 4.716 to 51.998 |
| Indexbereich | $-12 \leq h \leq 13$, $-20 \leq k \leq 17$, $-42 \leq l \leq 42$ |
| Gemessene Reflexe | 39911 |
| Unabhängige Reflexe | 12242 [$R_{int}$ = 0.0508, $R_{sigma}$ = 0.0597] |
| Daten/Beschränkungen/Parameter | 12242/0/688 |
| Anpassungsgüte $F^2$ | 1.048 |
| Finaler *R* Wert [I>=2s (I)] | $R_1$ = 0.0375, w$R_2$ = 0.0632 |
| Finaler *R* Wert [alle Daten] | $R_1$ = 0.0548, w$R_2$ = 0.0683 |
| Größtes Maximum und Minimum (in $Å^{-3}$) | 0.65/-0.72 |

**Tabelle 14** Atomkoordinaten ($\times 10^4$) und äquivalente, isotrope Auslenkungsparameter ($Å^2 \times 10^3$) von $[Cu(PPh_3)_3(CH_3CN)]_2[B_{12}I_{12}]\cdot 2CH_3CN$. $U_{äq}$ ist als ein Drittel der Spur des orthogonalisierten $U_{IJ}$ Tensors definiert.

| Atom | x | y | z | U(äq) |
|---|---|---|---|---|
| I6 | 1545.5(3) | 9403.0(2) | 5020.3(2) | 21.84(8) |
| I1 | 4060.5(3) | 9319.8(2) | 5996.2(2) | 21.50(8) |
| I3 | 6610.3(3) | 11077.5(2) | 5906.6(2) | 21.38(8) |
| I5 | 4339.0(3) | 7774.7(2) | 5115.4(2) | 22.34(8) |
| I2 | 2943.0(3) | 11454.2(2) | 5512.4(2) | 23.51(9) |
| I4 | 7492.1(3) | 8802.0(2) | 5638.2(2) | 19.63(12) |
| Cu1 | 1582.9(5) | 5200.1(3) | 6620.6(2) | 15.74(13) |
| P3 | 997.1(10) | 5105.8(7) | 5957.8(4) | 14.9(3) |
| P1 | 3149.2(11) | 6121.5(7) | 6803.2(4) | 16.4(3) |
| P2 | 1815.8(11) | 3993.6(7) | 6940.1(4) | 16.5(3) |
| N1 | 44(4) | 5770(3) | 6817.4(13) | 26.6(10) |
| B3 | 5701(4) | 10472(3) | 5397.4(15) | 13.2(12) |
| B5 | 4708(5) | 9023(3) | 5048.6(15) | 14.6(12) |
| B1 | 4587(4) | 9696(3) | 5438.6(15) | 13.1(11) |
| C7 | 4254(4) | 6259(3) | 6445.4(13) | 15.3(10) |
| B4 | 6087(4) | 9477(3) | 5289.8(16) | 12.5(11) |
| B2 | 4091(4) | 10626(3) | 5231.2(15) | 13.4(11) |
| B6 | 3486(4) | 9733(3) | 5011.3(15) | 10.8(11) |
| C13 | 4190(4) | 5914(3) | 7246.5(14) | 17.1(11) |
| C31 | 3201(4) | 3461(3) | 6828.2(14) | 17.9(11) |
| C38 | 2846(4) | 4056(3) | 5781.1(14) | 20.6(11) |
| C8 | 4794(4) | 6986(3) | 6377.0(15) | 24.5(12) |
| C44 | -1347(4) | 4519(3) | 6064.3(15) | 24.1(12) |
| C49 | 516(4) | 6038(3) | 5720.7(13) | 15.4(10) |
| C37 | 2145(4) | 4724(3) | 5654.9(13) | 15.7(10) |
| C54 | 1376(4) | 6660(3) | 5740.1(14) | 20.3(11) |
| C48 | -429(4) | 3899(3) | 5543.2(14) | 22.5(12) |
| C43 | -359(4) | 4464(3) | 5839.1(13) | 17.6(11) |
| C25 | 560(4) | 3258(3) | 6871.6(14) | 18.6(11) |
| C32 | 3208(4) | 2701(3) | 6673.4(15) | 22.9(12) |
| C51 | -959(4) | 6877(3) | 5332.9(16) | 26.7(13) |

| | | | | |
|---|---|---|---|---|
| C52 | -90(4) | 7477(3) | 5351.8(15) | 26.4(12) |
| C12 | 4584(4) | 5605(3) | 6239.4(14) | 22.1(12) |
| C46 | -2432(4) | 3470(3) | 5703.3(15) | 25.0(12) |
| C1 | 2648(4) | 7134(3) | 6891.4(14) | 20.2(11) |
| C40 | 3956(4) | 4122(3) | 5220.5(15) | 24.7(12) |
| C45 | -2388(4) | 4038(3) | 5995.3(15) | 25.5(12) |
| C47 | -1461(4) | 3403(3) | 5480.0(15) | 24.8(12) |
| C36 | 4323(4) | 3878(3) | 6875.4(15) | 24.3(12) |
| C18 | 3663(4) | 5856(3) | 7595.2(14) | 21.2(11) |
| C19 | 2054(4) | 4090(3) | 7465.3(14) | 22.7(12) |
| C42 | 2369(4) | 5080(3) | 5309.5(14) | 21.5(12) |
| C53 | 1080(4) | 7376(3) | 5558.8(15) | 25.2(12) |
| C50 | -660(4) | 6154(3) | 5513.9(14) | 22.0(12) |
| C39 | 3744(5) | 3761(3) | 5564.3(16) | 27.5(13) |
| C11 | 5451(4) | 5675(3) | 5970.3(15) | 29.3(13) |
| C17 | 4398(5) | 5677(3) | 7939.7(16) | 30.2(13) |
| C14 | 5472(4) | 5777(3) | 7250.8(15) | 24.7(12) |
| C26 | 213(4) | 2951(3) | 6499.7(15) | 26.0(13) |
| C9 | 5644(5) | 7041(3) | 6109.9(16) | 33.5(14) |
| C20 | 1282(5) | 4600(3) | 7638.4(16) | 33.0(14) |
| C33 | 4300(5) | 2385(3) | 6557.7(16) | 34.1(14) |
| C27 | -700(5) | 2370(3) | 6442.5(17) | 31.1(14) |
| C10 | 5992(5) | 6396(3) | 5912.5(16) | 32.6(14) |
| C28 | -1288(5) | 2103(3) | 6747.9(19) | 36.6(15) |
| C41 | 3275(4) | 4785(3) | 5091.8(15) | 26.2(12) |
| C2 | 1581(4) | 7430(3) | 6670.4(16) | 27.8(13) |
| C24 | 2975(5) | 3680(3) | 7703.8(16) | 34.0(14) |
| C34 | 5394(5) | 2801(3) | 6607.8(16) | 32.8(14) |
| C30 | -60(5) | 2990(3) | 7171.8(17) | 35.4(14) |
| C35 | 5422(5) | 3545(3) | 6770.7(16) | 31.6(14) |
| C57 | 9038(5) | 5565(4) | 8605(2) | 40.5(16) |
| C5 | 2976(5) | 8417(3) | 7185.0(17) | 39.1(15) |
| C6 | 3330(5) | 7641(3) | 7150.0(16) | 28.4(13) |

| | | | | |
|---|---|---|---|---|
| N2 | 8660(5) | 5786(3) | 8307(2) | 64.5(19) |
| C3 | 1233(5) | 8212(3) | 6707.6(17) | 36.7(15) |
| C22 | 2341(7) | 4302(4) | 8266.6(18) | 58(2) |
| C16 | 5666(5) | 5548(3) | 7938.6(16) | 33.2(14) |
| C55 | -666(5) | 6209(4) | 6899(2) | 42.6(16) |
| C15 | 6188(5) | 5599(3) | 7597.7(17) | 34.6(14) |
| C29 | -989(5) | 2415(4) | 7112(2) | 47.2(17) |
| C4 | 1929(6) | 8704(3) | 6966.3(19) | 44.1(16) |
| C23 | 3107(6) | 3789(4) | 8100.6(19) | 51.1(18) |
| C21 | 1418(6) | 4702(4) | 8037.4(18) | 48.8(18) |
| C58 | 9558(6) | 5267(4) | 8979(2) | 62(2) |
| C56 | -1572(6) | 6800(4) | 6998(3) | 94(3) |

## 1.8 $[Cu(PPh_3)_2(CH_3CN)_2]_2[B_{12}I_{12}]\cdot 2CH_3CN$

**Tabelle 15** Kristalldaten und Strukturverfeinerung von $[Cu(PPh_3)_2(CH_3CN)_2]_2[B_{12}I_{12}]\cdot 2CH_3CN$.

| | |
|---|---|
| Probenkürzel | exp_3712 |
| Summenformel | $C_{84}H_{78}B_{12}Cu_2I_{12}N_6P_4$ |
| Molmasse | 3075.00 |
| Temperatur (in K) | 150.0(10) |
| Kristallsystem | Triklin |
| Raumgruppe | $P\bar{1}$ |
| *a* (in Å) | 11.1571(3) |
| *b* (in Å) | 13.6514(4) |
| *c* (in Å) | 17.2205(5) |
| α (in °) | 74.462(3) |
| β (in °) | 85.307(2) |
| γ (in °) | 84.077(2) |
| Zellvolumen (in $Å^3$) | 2509.49(13) |
| *Z* | 1 |
| Berechnete Dichte (in $g/cm^3$) | 2.035 |
| Absorptionskoeffizient (in $mm^{-1}$) | 4.223 |
| F(000) | 1438.0 |

| | |
|---|---|
| Kristallgröße (in $mm^3$) | 0.1 × 0.1 × 0.07 |
| Strahlung | MoKα (λ = 0.71073) |
| 2Θ Bereich der Datenmessung (in °) | 4.608 bis 58.97 |
| Indexbereich | -15 ≤ h ≤ 14, -18 ≤ k ≤ 18, -22 ≤ l ≤ 23 |
| Gemessene Reflexe | 24862 |
| Unabhängige Reflexe | 11651 [$R_{int}$ = 0.0261, $R_{sigma}$ = 0.0341] |
| Daten/Beschränkungen/Parameter | 11651/0/544 |
| Anpassungsgüte $F^2$ | 1.054 |
| Finaler *R* Wert [I>=2s (I)] | $R_1$ = 0.0291, w$R_2$ = 0.0670 |
| Finaler *R* Wert [alle Daten] | $R_1$ = 0.0384, w$R_2$ = 0.0721 |
| Größtes Maximum und Minimum (in $Å^{-3}$) | 1.11/-0.62 |

**Tabelle 16** Atomkoordinaten ($\times 10^4$) und äquivalente, isotrope Auslenkungsparameter ($Å^2 \times 10^3$) von $[Cu(PPh_3)_2(CH_3CN)_2]_2[B_{12}I_{12}]\cdot 2CH_3CN$. $U_{äq}$ ist als ein Drittel der Spur des orthogonalisierten $U_{IJ}$ Tensors definiert.

| Atom | x | y | z | U(äq) |
|---|---|---|---|---|
| I2 | 10808.2(2) | 1541.8(2) | 2818.4(2) | 26.98(6) |
| I4 | 8484.4(2) | 1491.0(2) | 6491.2(2) | 25.90(6) |
| I6 | 8023.2(2) | -275.5(2) | 3339.2(2) | 26.37(6) |
| I1 | 7746.8(2) | 2288.2(2) | 4091.7(2) | 27.52(6) |
| I3 | 11058.9(2) | 2682.8(2) | 4765.2(2) | 25.80(6) |
| I5 | 6605.5(2) | -309.9(2) | 5566.7(2) | 27.21(6) |
| Cu1 | 6020.1(4) | 3641.7(3) | 8135.5(2) | 20.05(9) |
| P2 | 4320.2(8) | 2819.5(7) | 8428.0(5) | 19.15(18) |
| P1 | 7672.2(8) | 3213.6(7) | 8878.5(5) | 20.13(18) |
| N1 | 5753(3) | 5223(2) | 7846.2(19) | 25.0(7) |
| N2 | 6580(3) | 3705(2) | 6954.4(19) | 26.3(7) |
| B2 | 10352(3) | 689(3) | 4040(2) | 16.3(7) |
| C19 | 3525(3) | 2885(3) | 9387(2) | 20.5(7) |
| B3 | 10461(3) | 1178(3) | 4897(2) | 18.1(8) |
| B5 | 8512(4) | -140(3) | 5256(2) | 19.1(8) |
| B4 | 9333(4) | 672(3) | 5649(2) | 19.0(8) |
| B1 | 9015(4) | 1009(3) | 4598(2) | 17.9(8) |
| C25 | 4546(3) | 1460(3) | 8477(2) | 22.4(7) |
| B6 | 9145(4) | -126(3) | 4267(2) | 17.8(7) |

| | | | | |
|---|---|---|---|---|
| C31 | 3198(3) | 3275(3) | 7673(2) | 20.4(7) |
| C1 | 8796(3) | 4140(3) | 8569(2) | 23.1(7) |
| C13 | 8451(3) | 1982(3) | 8843(2) | 23.7(7) |
| C37 | 6939(4) | 3971(3) | 6305(2) | 28.6(8) |
| C7 | 7462(3) | 3116(3) | 9965(2) | 24.4(8) |
| C18 | 7740(4) | 1178(3) | 8911(2) | 31.2(9) |
| C39 | 5817(3) | 6066(3) | 7512(2) | 26.5(8) |
| C33 | 1535(4) | 3043(3) | 6949(2) | 30.3(9) |
| C2 | 8831(4) | 4760(3) | 7785(2) | 29.4(8) |
| C32 | 2370(3) | 2659(3) | 7531(2) | 27.6(8) |
| C36 | 3183(4) | 4280(3) | 7212(2) | 29.2(8) |
| C30 | 5221(4) | 1191(3) | 7835(2) | 32.1(9) |
| C34 | 1513(4) | 4046(3) | 6512(2) | 33.9(9) |
| C14 | 9690(3) | 1806(3) | 8766(3) | 32.3(9) |
| C6 | 9598(4) | 4288(3) | 9104(2) | 33.5(9) |
| C26 | 4104(4) | 697(3) | 9102(3) | 34.8(9) |
| C8 | 6907(4) | 3965(3) | 10189(2) | 33.1(9) |
| C16 | 9521(4) | 46(3) | 8848(3) | 35.8(9) |
| C3 | 9637(4) | 5507(3) | 7539(3) | 33.8(9) |
| C4 | 10430(4) | 5632(3) | 8073(3) | 34.4(9) |
| C17 | 8288(4) | 212(3) | 8916(3) | 35.7(10) |
| C12 | 7819(4) | 2257(4) | 10560(2) | 37.4(10) |
| C20 | 4146(4) | 3201(3) | 9923(2) | 34.4(9) |
| C22 | 2432(4) | 3074(3) | 10865(2) | 35.2(10) |
| C29 | 5432(4) | 185(4) | 7820(3) | 43.8(11) |
| C15 | 10226(4) | 846(3) | 8773(3) | 40.6(11) |
| C24 | 2349(4) | 2648(4) | 9610(3) | 37.3(10) |
| N3 | 5268(5) | 6116(4) | 5625(3) | 65.3(13) |
| C40 | 5931(5) | 7129(3) | 7089(3) | 44.5(11) |
| C10 | 7069(4) | 3100(4) | 11586(3) | 46.7(13) |
| C5 | 10416(4) | 5015(3) | 8850(3) | 39.1(10) |
| C9 | 6711(4) | 3965(4) | 10999(3) | 42.6(11) |
| C23 | 1813(4) | 2733(4) | 10343(3) | 44.1(11) |

| | | | | |
|---|---|---|---|---|
| C41 | 5360(4) | 6754(4) | 5056(3) | 43.8(11) |
| C35 | 2344(4) | 4665(3) | 6636(3) | 38.6(10) |
| C38 | 7414(5) | 4313(4) | 5470(3) | 49.0(13) |
| C21 | 3604(4) | 3295(4) | 10656(3) | 42.3(11) |
| C11 | 7616(4) | 2254(4) | 11368(3) | 48.4(13) |
| C28 | 4989(5) | -569(3) | 8440(3) | 47.5(12) |
| C42 | 5481(5) | 7582(4) | 4322(3) | 53.8(13) |
| C27 | 4317(5) | -318(3) | 9081(3) | 49.1(12) |

## 1.9 $[Ag(PPh_3)_4]_2[B_{12}H_{12}]$

**Tabelle 17** Kristalldaten und Strukturverfeinerung von $[Ag(PPh_3)_4]_2[B_{12}H_{12}]$.

| | |
|---|---|
| Probenkürzel | bw49 |
| Summenformel | $C_{144}H_{132}Ag_2B_{12}P_8$ |
| Molmasse | 2455.71 |
| Temperatur (in K) | 150.0(10) |
| Kristallsystem | Triklin |
| Raumgruppe | $P\bar{1}$ |
| *a* (in Å) | 13.2972(6) |
| *b* (in Å) | 14.4137(5) |
| *c* (in Å) | 18.8908(5) |
| α (in °) | 110.409(3) |
| β (in °) | 99.200(3) |
| γ (in °) | 91.792(3) |
| Zellvolumen (in $Å^3$) | 3334.9(2) |
| *Z* | 1 |
| Berechnete Dichte (in $g/cm^3$) | 1.223 |
| Absorptionskoeffizient (in $mm^{-1}$) | 0.438 |
| F(000) | 1270.0 |
| Kristallgröße (in $mm^3$) | 0.16 × 0.13 × 0.09 |
| Strahlung | MoKα (λ = 0.71073) |
| 2Θ Bereich der Datenmessung (in °) | 5.204 bis 53.996 |
| Indexbereich | $-16 \leq h \leq 16$, $-17 \leq k \leq 18$, $-23 \leq l \leq 23$ |

| | |
|---|---|
| Gemessene Reflexe | 34677 |
| Unabhängige Reflexe | 14255 [$R_{int}$ = 0.0476, $R_{sigma}$ = 0.0595] |
| Daten/Beschränkungen/Parameter | 14255/0/802 |
| Anpassungsgüte $F^2$ | 1.067 |
| Finaler $R$ Wert [I>=2s (I)] | $R_1$ = 0.0631, $wR_2$ = 0.1498 |
| Finaler $R$ Wert [alle Daten] | $R_1$ = 0.0898, $wR_2$ = 0.1613 |
| Größtes Maximum und Minimum (in Å$^{-3}$) | 1.30/-0.85 |

**Tabelle 18** Atomkoordinaten (×10$^4$) und äquivalente, isotrope Auslenkungsparameter (Å$^2$×10$^3$) von $[Ag(PPh_3)_4]_2[B_{12}H_{12}]$. $U_{äq}$ ist als ein Drittel der Spur des orthogonalisierten $U_{IJ}$ Tensors definiert.

| Atom | x | y | z | U(äq) |
|---|---|---|---|---|
| Ag(1) | 7910.2(2) | 3385.2(2) | 6957.0(2) | 25.66(10) |
| P(1) | 8831.9(8) | 4130.4(8) | 6112.8(5) | 24.9(2) |
| P(4) | 6040.8(8) | 3900.3(8) | 6931.1(6) | 28.0(2) |
| P(2) | 7925.0(9) | 1444.5(8) | 6474.8(6) | 29.8(2) |
| P(3) | 8898.8(8) | 4112.3(8) | 8347.1(6) | 28.2(2) |
| C(67) | 5253(3) | 3441(3) | 5961(2) | 30.2(9) |
| C(12) | 9040(3) | 5494(3) | 6439(2) | 26.3(8) |
| C(1) | 10115(3) | 3738(3) | 6034(2) | 26.9(8) |
| C(18) | 8207(3) | 3767(3) | 5100(2) | 26.9(8) |
| C(43) | 10186(3) | 4674(3) | 8441(2) | 27.6(9) |
| C(11) | 8663(3) | 6043(3) | 5989(2) | 32.5(9) |
| C(19) | 7239(4) | 807(3) | 6966(2) | 33.0(9) |
| C(54) | 7937(4) | 5011(4) | 9620(2) | 40.3(11) |
| C(2) | 10265(3) | 2764(3) | 5952(2) | 32.1(9) |
| C(13) | 8719(3) | 3369(3) | 4496(2) | 31.5(9) |
| C(6) | 10935(3) | 4361(4) | 6030(2) | 32.6(9) |
| C(16) | 6651(3) | 3568(3) | 4184(3) | 35.4(10) |
| C(49) | 8266(3) | 5089(3) | 8973(2) | 31.3(9) |
| C(48) | 10828(3) | 4110(3) | 7971(2) | 33.6(10) |
| C(61) | 5268(3) | 3480(3) | 7495(2) | 30.7(9) |
| C(56) | 6601(4) | 5834(4) | 7098(3) | 39.5(11) |
| C(5) | 11879(3) | 4006(4) | 5935(3) | 38.1(10) |
| C(30) | 7471(3) | 1339(3) | 4964(2) | 32.9(9) |

| | | | | |
|---|---|---|---|---|
| C(44) | 10537(3) | 5650(3) | 8916(2) | 34.2(10) |
| C(25) | 7353(4) | 847(3) | 5463(2) | 34.1(10) |
| C(7) | 9568(4) | 6013(3) | 7183(2) | 37.3(10) |
| C(24) | 7614(4) | 5(3) | 7143(3) | 43.2(11) |
| C(17) | 7166(3) | 3855(3) | 4937(2) | 31.2(9) |
| C(38) | 10028(4) | 3028(3) | 9149(2) | 41.3(11) |
| C(72) | 4637(3) | 4042(4) | 5669(3) | 37.9(10) |
| C(55) | 5902(3) | 5231(3) | 7261(3) | 34.9(10) |
| C(14) | 8200(4) | 3072(4) | 3740(2) | 38.6(10) |
| C(20) | 6346(4) | 1168(3) | 7207(3) | 38.6(10) |
| C(3) | 11212(4) | 2402(4) | 5847(3) | 40.5(11) |
| C(15) | 7178(4) | 3173(4) | 3585(2) | 38.3(11) |
| C(4) | 12014(4) | 3028(4) | 5831(3) | 41.7(11) |
| C(62) | 4342(4) | 2923(4) | 7203(3) | 41.1(11) |
| C(37) | 9070(4) | 3197(3) | 8812(2) | 35.0(10) |
| C(10) | 8810(4) | 7076(4) | 6277(3) | 39.5(11) |
| C(31) | 9162(4) | 942(3) | 6543(3) | 35.5(10) |
| C(68) | 5320(3) | 2468(4) | 5496(3) | 36.9(10) |
| C(22) | 6219(4) | -36(4) | 7808(3) | 48.3(13) |
| C(50) | 8029(4) | 5899(4) | 8757(3) | 40.6(11) |
| C(29) | 7040(4) | 929(3) | 4187(3) | 37.5(10) |
| C(21) | 5838(4) | 744(4) | 7622(3) | 43.2(11) |
| C(45) | 11493(4) | 6058(4) | 8904(2) | 40.9(11) |
| C(71) | 4087(4) | 3660(4) | 4924(3) | 45.9(12) |
| C(70) | 4148(4) | 2681(4) | 4467(3) | 44.9(12) |
| C(23) | 7093(5) | -407(4) | 7571(3) | 50.0(13) |
| C(47) | 11787(4) | 4523(4) | 7976(3) | 39.7(11) |
| C(46) | 12122(4) | 5490(4) | 8437(3) | 44.4(12) |
| C(60) | 5142(4) | 5650(4) | 7651(3) | 51.6(13) |
| C(8) | 9725(4) | 7028(4) | 7465(3) | 46.3(12) |
| C(9) | 9341(4) | 7574(4) | 7016(3) | 44.0(11) |
| C(66) | 5665(4) | 3722(4) | 8278(3) | 49.3(13) |
| C(69) | 4752(4) | 2086(4) | 4749(3) | 42.5(11) |

| | | | | |
|---|---|---|---|---|
| C(32) | 9814(4) | 1287(4) | 7258(3) | 46.1(12) |
| C(57) | 6538(4) | 6843(4) | 7328(4) | 54.2(14) |
| C(36) | 9469(5) | 183(4) | 5946(3) | 53.1(14) |
| C(42) | 8207(4) | 2608(3) | 8786(3) | 43.4(12) |
| C(39) | 10108(5) | 2265(4) | 9438(3) | 55.5(15) |
| C(65) | 5133(5) | 3390(5) | 8733(3) | 64.1(17) |
| C(28) | 6481(5) | 29(4) | 3909(3) | 57.4(15) |
| C(51) | 7486(5) | 6630(4) | 9188(3) | 55.9(14) |
| C(63) | 3808(4) | 2595(4) | 7662(3) | 50.7(13) |
| C(59) | 5082(5) | 6672(4) | 7886(4) | 68.9(19) |
| C(58) | 5779(5) | 7262(5) | 7721(5) | 76(2) |
| C(53) | 7382(5) | 5742(4) | 10033(3) | 54.8(14) |
| C(64) | 4211(5) | 2822(4) | 8421(3) | 55.8(15) |
| C(41) | 8301(5) | 1863(4) | 9096(3) | 59.9(17) |
| C(40) | 9250(6) | 1692(4) | 9412(3) | 62.8(18) |
| C(35) | 10393(5) | -203(5) | 6075(4) | 69.2(19) |
| C(26) | 6793(6) | -73(4) | 5168(3) | 75(2) |
| C(33) | 10729(4) | 879(5) | 7382(3) | 58.0(15) |
| C(52) | 7158(5) | 6543(5) | 9824(3) | 66.4(17) |
| C(34) | 11004(5) | 124(5) | 6785(4) | 66.0(18) |
| C(27) | 6358(7) | -473(5) | 4396(4) | 91(3) |
| B(10) | 2931(8) | 10050(8) | 10095(6) | 38(2) |
| B(8) | 3164(9) | 8387(8) | 8737(7) | 44(3) |
| B(9) | 3433(9) | 8835(10) | 9729(7) | 46(3) |
| B(12) | 2251(9) | 9025(9) | 9276(7) | 44(3) |
| B(3) | 4381(10) | 9000(9) | 9186(7) | 50(3) |
| B(7) | 2431(9) | 9261(9) | 8418(7) | 45(3) |
| B(5) | 3525(11) | 10865(9) | 9738(7) | 50(3) |
| B(4) | 4246(10) | 9991(9) | 10034(7) | 45(3) |
| B(1) | 4424(11) | 10256(10) | 9207(7) | 54(3) |
| B(11) | 2295(11) | 10283(10) | 9279(8) | 55(3) |
| B(6) | 3215(11) | 10428(10) | 8740(7) | 53(3) |
| B(2) | 3728(11) | 9240(9) | 8368(7) | 51(3) |

## 1.10 $[Ag(PPh_3)_4]_2[B_{12}F_{12}]\cdot 2PPh_3$

**Tabelle 19** Kristalldaten und Strukturverfeinerung von $[Ag(PPh_3)_4]_2[B_{12}F_{12}]\cdot 2PPh_3$.

| | |
|---|---|
| Probenkürzel | bw53 |
| Summenformel | $C_{180}H_{150}Ag_2B_{12}F_{12}P_{10}$ |
| Molmasse | 3196.09 |
| Temperatur (in K) | 150.0(10) |
| Kristallsystem | Kubisch |
| Raumgruppe | $Pa\bar{3}$ |
| *a* (in Å) | 24.9424(3) |
| *b* (in Å) | 24.9424(3) |
| *c* (in Å) | 24.9424(3) |
| α (in °) | 90 |
| β (in °) | 90 |
| γ (in °) | 90 |
| Zellvolumen (in $Å^3$) | 15517.3(6) |
| *Z* | 400.008 |
| Berechnete Dichte (in $g/cm^3$) | 1.368 |
| Absorptionskoeffizient (in $mm^{-1}$) | 0.425 |
| F(000) | 6568.0 |
| Kristallgröße (in $mm^3$) | 0.15 × 0.13 × 0.12 |
| Strahlung | MoKα (λ = 0.71073) |
| 2Θ Bereich der Datenmessung (in °) | 4.62 bis 51.98 |
| Indexbereich | $-18 \leq h \leq 30$, $-23 \leq k \leq 20$, $-10 \leq l \leq 29$ |
| Gemessene Reflexe | 15950 |
| Unabhängige Reflexe | 5093 [$R_{int}$ = 0.0353, $R_{sigma}$ = 0.0384] |
| Daten/Beschränkungen/Parameter | 5093/30/391 |
| Anpassungsgüte $F^2$ | 1.048 |
| Finaler *R* Wert [I>=2s (I)] | $R_1$ = 0.0492, $wR_2$ = 0.1290 |
| Finaler *R* Wert [alle Daten] | $R_1$ = 0.0691, $wR_2$ = 0.1390 |
| Größtes Maximum und Minimum (in $Å^{-3}$) | 0.67/-0.98 |

**Tabelle 20** Atomkoordinaten ($\times 10^4$) und äquivalente, isotrope Auslenkungsparameter ($Å^2 \times 10^3$) von $[Ag(PPh_3)_4]_2[B_{12}F_{12}] \cdot 2PPh_3$. $U_{äq}$ ist als ein Drittel der Spur des orthogonalisierten $U_{IJ}$ Tensors definiert.

| Atom | x | y | z | U(äq) |
|---|---|---|---|---|
| Ag(1) | 2350.6(2) | 2649.4(2) | 7350.6(2) | 27.20(14) |
| P(2) | 2949.2(3) | 2050.8(3) | 7949.2(3) | 28.1(3) |
| P(4) | 4118.4(6) | 881.6(6) | 9118.4(6) | 39.5(5) |
| P(1) | 2124.4(3) | 2127.6(3) | 6474.8(3) | 28.4(2) |
| P(3) | 4638.9(11) | 361.1(11) | 9638.9(11) | 40.8(10) |
| F(1) | 1159.6(14) | 5386(2) | 4876(2) | 131.0(19) |
| F(2) | 704.0(16) | 4296.0(16) | 5704.0(16) | 209(5) |
| F(3A) | 963(3) | 4237(3) | 4963(4) | 29.7(18) |
| F(3B) | 686(5) | 4074(5) | 4576(5) | 68(4) |
| F(3C) | 837(5) | 4446(5) | 4292(5) | 61(3) |
| F(3D) | 1140(4) | 4600(5) | 4702(5) | 58(3) |
| B(1) | 625(2) | 5225(3) | 4930(3) | 63.3(18) |
| B(2) | 377(2) | 4623(2) | 5377(2) | 100(4) |
| B(3) | 492(4) | 4624(4) | 4806(4) | 115(3) |
| C(19) | 3264.5(12) | 1488.7(13) | 7603.5(12) | 30.5(7) |
| C(23) | 3507.5(17) | 564.2(15) | 7508.6(17) | 50.1(10) |
| C(11) | 1406.8(15) | 2793.5(14) | 5149.9(14) | 41.5(8) |
| C(21) | 3753.9(14) | 1185.3(15) | 6826.3(15) | 42.1(8) |
| C(1) | 2664.0(12) | 1907.8(13) | 6034.6(13) | 31.1(7) |
| C(25) | 4720(4) | 288(3) | 8917(3) | 38(3) |
| C(30) | 4860(5) | -224(3) | 8749(4) | 56(4) |
| C(29) | 4934(6) | -330(4) | 8207(5) | 75(7) |
| C(28) | 4867(6) | 77(5) | 7833(3) | 78(6) |
| C(27) | 4727(5) | 590(5) | 8000(3) | 61(4) |
| C(26) | 4653(3) | 695(3) | 8543(3) | 50(3) |
| C(2) | 3075.8(14) | 2266.4(15) | 5918.7(15) | 43.3(8) |
| C(3) | 3487.1(16) | 2121.3(18) | 5574.4(16) | 54.0(10) |
| C(4) | 3496.5(15) | 1621.6(17) | 5345.5(16) | 50.8(10) |
| C(10) | 979.5(15) | 3058.9(14) | 5367.8(16) | 44.9(9) |
| C(5) | 3092.9(15) | 1265.4(16) | 5457.4(15) | 46.1(9) |
| C(12) | 1756.1(14) | 2504.2(13) | 5471.0(13) | 35.2(7) |

| | | | | |
|---|---|---|---|---|
| C(6) | 2676.9(14) | 1408.5(14) | 5797.5(14) | 37.4(8) |
| C(20) | 3500.9(13) | 1590.0(14) | 7105.7(13) | 35.9(8) |
| C(13) | 1772.0(12) | 1504.6(12) | 6617.3(12) | 29.5(7) |
| C(22) | 3755.0(16) | 670.6(16) | 7027.6(16) | 49.6(10) |
| C(14) | 1996.7(13) | 1160.0(12) | 6993.9(13) | 33.1(7) |
| C(24) | 3263.6(14) | 968.6(13) | 7801.4(14) | 37.5(8) |
| C(15) | 1738.7(15) | 693.3(13) | 7141.0(14) | 39.7(8) |
| C(16) | 1262.7(15) | 555.8(15) | 6907.1(16) | 47.2(9) |
| C(17) | 1040.8(15) | 885.0(16) | 6528.0(16) | 50.1(10) |
| C(31) | 4480.8(16) | 339.4(17) | 8781.4(18) | 41.1(16) |
| C(36) | 4603.5(17) | -144.8(19) | 9028.5(15) | 48.0(15) |
| C(35) | 4878(2) | -539.5(18) | 8747(2) | 65(2) |
| C(34) | 5030(3) | -450(3) | 8218(2) | 80(4) |
| C(33) | 4908(3) | 34(3) | 7971.3(18) | 70(3) |
| C(32) | 4633(2) | 429(2) | 8252.8(19) | 56(2) |
| C(18) | 1290.4(14) | 1357.8(14) | 6381.0(14) | 41.3(8) |
| C(7) | 1673.5(12) | 2477.0(12) | 6018.7(13) | 29.4(7) |
| C(8) | 1231.8(14) | 2742.4(14) | 6236.8(14) | 40.7(8) |
| C(9) | 887.5(15) | 3033.9(15) | 5914.1(16) | 46.2(9) |

## 1.11 $[Ag(PPh_3)_3(CH_3CN)]_2[B_{12}Cl_{12}]\cdot 2CH_3CN$

**Tabelle 21** Kristalldaten und Strukturverfeinerung von $[Ag(PPh_3)_3(CH_3CN)]_2[B_{12}Cl_{12}]\cdot 2CH_3CN$.

| | |
|---|---|
| Probenkürzel | bw52 |
| Summenformel | $C_{116}H_{102}Ag_2B_{12}Cl_{12}N_4P_6$ |
| Molmasse | 2508.69 |
| Temperatur (in K) | 150.0(10) |
| Kristallsystem | Monoklin |
| Raumgruppe | $P2_1/c$ |
| *a* (in Å) | 10.5611(3) |
| *b* (in Å) | 16.1733(6) |
| *c* (in Å) | 34.7996(12) |
| α (in °) | 90 |

| | |
|---|---|
| β (in °) | 95.707(3) |
| γ (in °) | 90 |
| Zellvolumen (in Å$^3$) | 5914.6(3) |
| *Z* | 2 |
| Berechnete Dichte (in g/cm$^3$) | 1.409 |
| Absorptionskoeffizient (in mm$^{-1}$) | 0.733 |
| F(000) | 2548.0 |
| Kristallgröße (in mm$^3$) | 0.23 × 0.15 × 0.13 |
| Strahlung | MoKα (λ = 0.71073) |
| 2Θ Bereich der Datenmessung (in °) | 5.172 bis 53.998 |
| Indexbereich | -13 ≤ h ≤ 13, -20 ≤ k ≤ 20, -39 ≤ l ≤ 44 |
| Gemessene Reflexe | 30846 |
| Unabhängige Reflexe | 12618 [$R_{int}$ = 0.0321, $R_{sigma}$ = 0.0376] |
| Daten/Beschränkungen/Parameter | 12618/18/706 |
| Anpassungsgüte $F^2$ | 1.237 |
| Finaler *R* Wert [I>=2s (I)] | $R_1$ = 0.0502, $wR_2$ = 0.0951 |
| Finaler *R* Wert [alle Daten] | $R_1$ = 0.0564, $wR_2$ = 0.0970 |
| Größtes Maximum und Minimum (in Å$^{-3}$) | 0.96/-0.80 |

**Tabelle 22** Atomkoordinaten (×10$^4$) und äquivalente, isotrope Auslenkungsparameter (Å$^2$×10$^3$) von $[Ag(PPh_3)_3(CH_3CN)]_2[B_{12}Cl_{12}]\cdot 2CH_3CN$. $U_{äq}$ ist als ein Drittel der Spur des orthogonalisierten $U_{IJ}$ Tensors definiert.

| Atom | x | y | z | U(eq) |
|---|---|---|---|---|
| Ag(1) | 8396.4(2) | 73.8(2) | 3363.0(2) | 16.54(6) |
| Cl(5) | 2809.3(8) | 3846.0(5) | 4400.7(2) | 23.73(17) |
| Cl(3) | 4025.5(8) | 5563.9(5) | 5906.4(2) | 26.85(18) |
| P(1) | 6734.6(8) | 1136.4(5) | 3154.5(2) | 17.07(16) |
| P(3) | 8221.9(8) | -1253.3(5) | 2997.8(2) | 17.63(17) |
| Cl(6) | 3500.8(9) | 6010.2(5) | 4188.3(2) | 27.28(18) |
| P(2) | 9108.5(7) | 81.5(5) | 4073.3(2) | 15.30(15) |
| Cl(2) | 4238.8(9) | 7086.8(5) | 5107.7(2) | 26.80(18) |
| Cl(1) | 1782.8(8) | 5483.8(6) | 5028.3(3) | 29.95(19) |
| Cl(4) | 3154.2(9) | 3555.4(6) | 5444.2(3) | 29.01(19) |
| C(27) | 7871(3) | -321.9(19) | 4356.0(9) | 16.4(6) |
| C(33) | 10482(3) | -592(2) | 4208.0(9) | 17.5(6) |

| | | | | |
|---|---|---|---|---|
| C(32) | 7170(3) | -1012(2) | 4213.6(10) | 21.5(7) |
| C(9) | 5788(3) | 1324(2) | 3559.1(9) | 17.7(6) |
| C(45) | 9495(3) | -2022(2) | 3041.7(10) | 21.1(7) |
| C(51) | 6847(3) | -1784(2) | 3163.0(9) | 18.6(6) |
| C(21) | 9537(3) | 1084(2) | 4294.1(8) | 17.0(6) |
| C(28) | 7600(3) | 31(2) | 4704.0(9) | 21.4(7) |
| C(39) | 7871(3) | -1113(2) | 2477.6(9) | 20.9(7) |
| C(3) | 7289(3) | 2171(2) | 3043.8(9) | 19.8(7) |
| C(14) | 5473(3) | 2118(2) | 3675.6(10) | 21.6(7) |
| C(34) | 10465(3) | -1213(2) | 4485.3(9) | 21.9(7) |
| C(31) | 6239(3) | -1348(2) | 4420.9(10) | 25.0(7) |
| C(10) | 5387(3) | 639(2) | 3761.4(9) | 22.5(7) |
| C(15) | 5532(3) | 908(2) | 2755.5(9) | 19.8(7) |
| C(22) | 8619(3) | 1713(2) | 4264.4(9) | 22.8(7) |
| C(56) | 6966(3) | -2522(2) | 3371.0(10) | 23.2(7) |
| C(46) | 9740(4) | -2547(2) | 2739.4(10) | 28.3(8) |
| N(1) | 10417(4) | 810(3) | 3146.9(12) | 50.0(10) |
| B(3) | 4533(3) | 5284(2) | 5436.5(10) | 17.5(7) |
| C(38) | 11557(3) | -506(2) | 4004.4(10) | 22.1(7) |
| C(26) | 10737(3) | 1258(2) | 4479.6(10) | 23.2(7) |
| C(29) | 6641(3) | -301(2) | 4904.0(10) | 25.2(7) |
| C(16) | 4233(3) | 973(3) | 2784.2(11) | 33.2(9) |
| C(25) | 11011(3) | 2040(2) | 4633.6(10) | 27.2(8) |
| C(30) | 5966(3) | -993(2) | 4764.0(10) | 25.1(7) |
| C(52) | 5664(3) | -1376(2) | 3122.3(10) | 26.9(8) |
| C(4) | 8267(3) | 2512(2) | 3292.3(11) | 29.0(8) |
| C(35) | 11509(3) | -1743(2) | 4555.6(10) | 27.2(8) |
| C(13) | 4766(3) | 2224(2) | 3988.1(10) | 27.1(8) |
| C(44) | 6826(4) | -1456(2) | 2261.4(10) | 29.8(8) |
| B(1) | 3442(3) | 5234(2) | 5011.8(10) | 18.0(7) |
| C(12) | 4355(3) | 1543(3) | 4182.7(10) | 29.0(8) |
| C(20) | 5950(3) | 690(2) | 2402.2(10) | 25.5(7) |
| C(55) | 5930(4) | -2841(2) | 3540.5(10) | 29.8(8) |

| | | | | |
|---|---|---|---|---|
| C(50) | 10232(3) | -2094(2) | 3393.4(11) | 29.7(8) |
| C(36) | 12560(3) | -1652(2) | 4352.7(10) | 26.7(8) |
| C(40) | 8699(4) | -597(2) | 2298.0(10) | 26.9(8) |
| C(37) | 12592(3) | -1026(2) | 4079.6(10) | 24.4(7) |
| C(24) | 10089(4) | 2656(2) | 4600.9(11) | 28.2(8) |
| C(23) | 8887(4) | 2493(2) | 4418.5(11) | 28.5(8) |
| C(8) | 6733(3) | 2659(2) | 2743.7(10) | 26.8(8) |
| B(5) | 3943(3) | 4433(2) | 4707.0(10) | 17.4(7) |
| B(4) | 4107(4) | 4302(2) | 5220.8(10) | 18.4(7) |
| C(54) | 4767(4) | -2438(3) | 3498.0(10) | 31.4(9) |
| B(2) | 4638(3) | 6013(2) | 5056.3(10) | 16.9(7) |
| C(47) | 10715(4) | -3123(2) | 2789.4(11) | 33.9(9) |
| C(53) | 4636(3) | -1710(3) | 3284.1(10) | 30.6(8) |
| B(6) | 4275(3) | 5491(2) | 4606.2(10) | 17.4(7) |
| C(11) | 4672(3) | 755(2) | 4071.3(10) | 27.0(8) |
| C(48) | 11443(4) | -3189(2) | 3141.2(12) | 33.1(9) |
| C(1) | 10956(4) | 1359(3) | 3286.0(13) | 43.3(11) |
| C(5) | 8677(4) | 3316(3) | 3249.5(12) | 35.9(9) |
| C(49) | 11196(4) | -2683(3) | 3444.7(12) | 34.8(9) |
| C(41) | 8486(4) | -434(3) | 1904.4(11) | 35.7(9) |
| N(2) | 8919(4) | 5603(3) | 3491.2(13) | 55.6(11) |
| C(7) | 7147(4) | 3465(2) | 2697.4(12) | 35.7(9) |
| C(6) | 8109(4) | 3800(2) | 2949.7(12) | 35.6(9) |
| C(18) | 3796(4) | 617(3) | 2112.4(12) | 37.8(10) |
| C(43) | 6612(4) | -1269(3) | 1868.4(11) | 40.8(10) |
| C(17) | 3366(4) | 816(3) | 2462.8(13) | 40.3(10) |
| C(19) | 5079(4) | 552(2) | 2080.1(11) | 34.0(9) |
| C(42) | 7436(5) | -763(3) | 1691.2(11) | 41.1(11) |
| C(57) | 9240(20) | 5480(10) | 3788(7) | 30(4) |
| C(2) | 11611(5) | 2062(4) | 3472.8(18) | 70.3(17) |
| C(58) | 9783(18) | 5352(11) | 4180(5) | 81(5) |
| C(58A) | 9834(16) | 5912(12) | 4197(4) | 81(5) |
| C(57A) | 9350(20) | 5733(10) | 3805(8) | 35(4) |

## 1.12 $[Ag(PPh_3)_3(CH_3CN)]_2[B_{12}Br_{12}]\cdot 2CH_3CN$

**Tabelle 23** Kristalldaten und Strukturverfeinerung von $[Ag(PPh_3)_3(CH_3CN)]_2[B_{12}Br_{12}]\cdot 2CH_3CN$.

| | |
|---|---|
| Probenkürzel | bw58 |
| Summenformel | $C_{58}H_{51}AgB_6Br_6N_2P_3$ |
| Molmasse | 1521.11 |
| Temperatur (in K) | 150.0(10) |
| Kristallsystem | Monoklin |
| Raumgruppe | $P2_1/c$ |
| *a* (in Å) | 10.5525(2) |
| *b* (in Å) | 16.3444(4) |
| *c* (in Å) | 34.6326(7) |
| α (in °) | 90 |
| β (in °) | 95.454(2) |
| γ (in °) | 90 |
| Zellvolumen (in $Å^3$) | 5946.2(2) |
| *Z* | 4 |
| Berechnete Dichte (in $g/cm^3$) | 1.699 |
| Absorptionskoeffizient (in $mm^{-1}$) | 4.491 |
| F(000) | 2980.0 |
| Kristallgröße (in $mm^3$) | 0.11 × 0.1 × 0.09 |
| Strahlung | MoKα (λ = 0.71073) |
| 2Θ Bereich der Datenmessung (in °) | 4.728 bis 53.996 |
| Indexbereich | $-13 \leq h \leq 12$, $-20 \leq k \leq 19$, $-44 \leq l \leq 40$ |
| Gemessene Reflexe | 35455 |
| Unabhängige Reflexe | 12863 [$R_{int}$ = 0.0385, $R_{sigma}$ = 0.0479] |
| Daten/Beschränkungen/Parameter | 12863/12/697 |
| Anpassungsgüte $F^2$ | 1.030 |
| Finaler *R* Wert [I>=2s (I)] | $R_1$ = 0.0320, $wR_2$ = 0.0552 |
| Finaler *R* Wert [alle Daten] | $R_1$ = 0.0493, $wR_2$ = 0.0587 |
| Größtes Maximum und Minimum (in $Å^{-3}$) | 0.48/-0.54 |

**Tabelle 24** Atomkoordinaten ($\times 10^4$) und äquivalente, isotrope Auslenkungsparameter ($Å^2 \times 10^3$) von $[Ag(PPh_3)_3(CH_3CN)]_2[B_{12}Br_{12}]\cdot 2CH_3CN$. $U_{äq}$ ist als ein Drittel der Spur des orthogonalisierten $U_{IJ}$ Tensors definiert.

| Atom | x | y | z | U(eq) |
|---|---|---|---|---|
| Ag(1) | 8392.6(2) | 4913.1(2) | 3359.2(2) | 16.77(5) |
| Br(3) | 2717.2(3) | 1187.3(2) | 4384.4(2) | 20.06(7) |
| Br(4) | 3482.7(3) | -1023.5(2) | 4146.5(2) | 22.65(7) |
| Br(2) | 5757.0(3) | 2146.1(2) | 4896.5(2) | 22.64(7) |
| Br(1) | 6014.9(3) | 613.2(2) | 4066.5(2) | 23.07(7) |
| Br(6) | 8336.2(3) | 519.3(2) | 4980.3(2) | 23.34(7) |
| Br(5) | 6951.4(3) | -1455.5(2) | 4530.7(2) | 24.03(7) |
| P(3) | 6760.7(7) | 3860.2(5) | 3148.2(2) | 17.11(16) |
| P(1) | 8210.0(7) | 6221.9(5) | 3000.7(2) | 16.86(16) |
| P(2) | 9086.3(7) | 4916.4(5) | 4066.8(2) | 15.90(16) |
| C(19) | 10446(3) | 5575.5(18) | 4194.8(7) | 16.7(6) |
| C(25) | 9520(3) | 3935.5(17) | 4289.8(7) | 16.1(6) |
| B(5) | 5900(3) | -668(2) | 4776.7(8) | 14.1(7) |
| C(37) | 5778(3) | 3698.0(18) | 3543.4(7) | 16.9(6) |
| C(31) | 7863(3) | 5308.7(17) | 4353.0(7) | 15.8(6) |
| B(1) | 5466(3) | 289(2) | 4563.9(8) | 14.4(7) |
| B(6) | 6550(3) | 239(2) | 4990.5(9) | 15.4(7) |
| B(3) | 3955(3) | 550(2) | 4711.4(9) | 14.8(7) |
| C(13) | 6807(3) | 6723.0(17) | 3155.2(7) | 17.6(6) |
| C(24) | 10454(3) | 6182.4(19) | 4473.9(8) | 22.3(7) |
| C(27) | 10975(3) | 3017.5(19) | 4642.5(8) | 25.7(7) |
| B(2) | 5345(3) | 995(2) | 4949.3(8) | 14.3(7) |
| C(20) | 11501(3) | 5492.1(18) | 3984.7(8) | 20.9(7) |
| B(4) | 4298(3) | -477(2) | 4604.7(9) | 15.3(7) |
| C(14) | 6866(3) | 7454.0(19) | 3352.3(8) | 21.7(7) |
| C(49) | 7290(3) | 2831.6(18) | 3048.9(8) | 19.3(6) |
| C(1) | 7901(3) | 6084.6(18) | 2480.8(8) | 19.6(7) |
| C(18) | 5663(3) | 6298.9(19) | 3108.1(8) | 23.3(7) |
| C(7) | 9451(3) | 6989.2(18) | 3051.4(8) | 19.3(6) |
| C(38) | 5399(3) | 4376.5(19) | 3745.3(8) | 23.1(7) |
| C(28) | 10070(3) | 2407.5(19) | 4609.6(8) | 25.7(7) |

| | | | | |
|---|---|---|---|---|
| C(30) | 8616(3) | 3314.3(18) | 4257.6(8) | 21.7(7) |
| C(26) | 10696(3) | 3781.2(19) | 4485.1(8) | 21.0(7) |
| C(29) | 8893(3) | 2555.0(19) | 4417.2(9) | 26.9(7) |
| C(32) | 7610(3) | 4964.3(18) | 4702.4(8) | 20.5(7) |
| C(43) | 5616(3) | 4085.1(17) | 2739.1(8) | 19.4(6) |
| C(22) | 12524(3) | 6606.8(19) | 4330.9(8) | 24.6(7) |
| N(1) | 10393(3) | 4198(2) | 3140.2(9) | 49.4(9) |
| C(23) | 11493(3) | 6700(2) | 4541.6(9) | 28.7(8) |
| C(42) | 5410(3) | 2925.9(19) | 3657.1(8) | 21.2(7) |
| C(36) | 7163(3) | 5981.3(18) | 4213.6(8) | 22.5(7) |
| C(41) | 4686(3) | 2840(2) | 3964.2(9) | 27.4(7) |
| C(35) | 6229(3) | 6305.9(19) | 4421.2(9) | 24.6(7) |
| C(44) | 6082(3) | 4274.0(18) | 2390.8(8) | 23.6(7) |
| C(21) | 12536(3) | 6005.3(19) | 4057.6(8) | 24.1(7) |
| C(40) | 4310(3) | 3513(2) | 4159.2(8) | 28.0(8) |
| C(15) | 5795(3) | 7755(2) | 3509.8(8) | 29.5(8) |
| C(54) | 8274(3) | 2511(2) | 3294.1(9) | 29.0(8) |
| C(10) | 11355(3) | 8155(2) | 3160.1(10) | 32.0(8) |
| C(12) | 10131(3) | 7091.7(19) | 3409.8(9) | 28.1(8) |
| C(50) | 6706(3) | 2340.3(19) | 2762.9(9) | 28.7(8) |
| C(16) | 4674(3) | 7331(2) | 3464.0(8) | 29.9(8) |
| C(6) | 8732(3) | 5578.1(19) | 2306.5(8) | 27.1(7) |
| C(2) | 6885(3) | 6430(2) | 2258.1(8) | 28.4(8) |
| C(8) | 9741(3) | 7488(2) | 2750.5(9) | 32.9(8) |
| C(48) | 4315(3) | 4062(2) | 2755.5(9) | 31.3(8) |
| C(34) | 5977(3) | 5952.5(19) | 4765.4(8) | 25.3(7) |
| C(39) | 4671(3) | 4282(2) | 4052.5(8) | 26.6(7) |
| C(17) | 4601(3) | 6611(2) | 3259.3(8) | 28.6(8) |
| C(45) | 5265(3) | 4423(2) | 2061.3(9) | 31.7(8) |
| C(33) | 6658(3) | 5286(2) | 4906.3(8) | 24.9(7) |
| N(2) | 1196(3) | 4324(2) | 1567.9(10) | 50.7(9) |
| C(11) | 11073(3) | 7678(2) | 3463.0(10) | 34.8(9) |
| C(9) | 10694(3) | 8066(2) | 2803.6(10) | 37.6(9) |

| | | | | |
|---|---|---|---|---|
| C(51) | 7094(3) | 1540(2) | 2729.5(10) | 36.8(9) |
| C(55) | 10943(3) | 3658(3) | 3261.3(11) | 45.1(11) |
| C(46) | 3981(4) | 4398(2) | 2084.7(9) | 36.6(9) |
| C(52) | 8057(3) | 1222(2) | 2977.8(10) | 36.4(9) |
| C(53) | 8652(3) | 1710(2) | 3261.6(10) | 37.7(9) |
| C(5) | 8543(4) | 5418(2) | 1912.9(9) | 37.3(9) |
| C(4) | 7522(4) | 5752(2) | 1697.3(9) | 40.7(10) |
| C(3) | 6702(4) | 6254(2) | 1866.1(9) | 39.6(9) |
| C(47) | 3499(3) | 4225(2) | 2426.7(10) | 41.1(9) |
| C(57) | 790(3) | 4431(2) | 1267.7(11) | 39.5(9) |
| C(56) | 11623(4) | 2961(3) | 3437.9(15) | 83.7(17) |
| C(58A) | 160(18) | 4354(9) | 876(5) | 66(5) |
| C(58) | 302(18) | 4778(8) | 892(5) | 59(4) |

## 1.13 $[Ag(PPh_3)_4]_2[B_{12}I_{12}]\cdot 2CH_3CN$

**Tabelle 25** Kristalldaten und Strukturverfeinerung von $[Ag(PPh_3)_4]_2[B_{12}I_{12}]\cdot 2CH_3CN$.

| | |
|---|---|
| Probenkürzel | bw57 |
| Summenformel | $C_{148}H_{126}Ag_2B_{12}I_{12}N_2P_8$ |
| Molmasse | 4048.52 |
| Temperatur (in K) | 150.0(10) |
| Kristallsystem | Triklin |
| Raumgruppe | $P\bar{1}$ |
| *a* (in Å) | 14.0010(6) |
| *b* (in Å) | 14.2095(5) |
| *c* (in Å) | 21.0292(8) |
| α (in °) | 106.305(3) |
| β (in °) | 108.744(4) |
| γ (in °) | 91.447(3) |
| Zellvolumen (in $Å^3$) | 3771.7(3) |
| *Z* | 1 |
| Berechnete Dichte (in $g/cm^3$) | 1.782 |
| Absorptionskoeffizient (in $mm^{-1}$) | 2.849 |

| | |
|---|---|
| F(000) | 1938.0 |
| Kristallgröße (in $mm^3$) | 0.18 × 0.04 × 0.04 |
| Strahlung | MoKα (λ = 0.71073) |
| 2Θ Bereich der Datenmessung (in °) | 5.792 bis 52 |
| Indexbereich | -17 ≤ h ≤ 16, -17 ≤ k ≤ 17, -23 ≤ l ≤ 25 |
| Gemessene Reflexe | 31786 |
| Unabhängige Reflexe | 14376 [$R_{int}$ = 0.0362, $R_{sigma}$ = 0.0544] |
| Daten/Beschränkungen/Parameter | 14376/0/830 |
| Anpassungsgüte $F^2$ | 1.012 |
| Finaler *R* Wert [I>=2s (I)] | $R_1$ = 0.0361, $wR_2$ = 0.0726 |
| Finaler *R* Wert [alle Daten] | $R_1$ = 0.0571, $wR_2$ = 0.0782 |
| Größtes Maximum und Minimum (in $Å^{-3}$) | 1.17/-0.87 |

**Tabelle 26** Atomkoordinaten (×$10^4$) und äquivalente, isotrope Auslenkungsparameter ($Å^2$×$10^3$) von $[Ag(PPh_3)_4]_2[B_{12}I_{12}]\cdot 2CH_3CN$. $U_{äq}$ ist als ein Drittel der Spur des orthogonalisierten $U_{IJ}$ Tensors definiert.

| Atom | x | y | z | U(äq) |
|---|---|---|---|---|
| I(4) | 2833.6(2) | 1107.6(2) | 10751.6(2) | 26.50(8) |
| I(6) | -1998.9(2) | 1727.9(2) | 9479.9(2) | 27.07(8) |
| I(3) | -1363.2(3) | -470.5(3) | 7999.3(2) | 30.22(9) |
| I(2) | 1586.1(3) | -809.0(3) | 8780.5(2) | 29.72(9) |
| I(1) | -584.3(3) | -2724.2(2) | 8824.9(2) | 36.26(10) |
| I(5) | 701.9(3) | 1951.2(3) | 9202.5(2) | 32.68(9) |
| Ag(1) | 3437.0(3) | 3194.2(3) | 6747.5(2) | 21.43(9) |
| P(3) | 5162.0(9) | 3779.9(9) | 6642.4(6) | 19.5(3) |
| P(1) | 3056.7(9) | 1274.5(9) | 6283.9(6) | 19.0(3) |
| P(4) | 1916.8(9) | 3973.2(9) | 6029.8(6) | 21.7(3) |
| P(2) | 3605.4(10) | 3746.8(9) | 8063.2(6) | 20.4(3) |
| B(4) | 1250(4) | 500(4) | 10338(2) | 18.0(11) |
| B(2) | 702(4) | -359(4) | 9465(2) | 15.9(11) |
| B(5) | 318(4) | 852(4) | 9647(2) | 18.1(11) |
| B(3) | -606(4) | -203(4) | 9124(2) | 16.7(11) |
| C(67) | 645(3) | 3520(3) | 5964(2) | 21.7(10) |
| B(6) | -884(4) | 763(4) | 9773(3) | 19.1(12) |
| C(19) | 3831(4) | 2771(3) | 8484(2) | 20.4(10) |

| | | | | |
|---|---|---|---|---|
| B(1) | -265(4) | -1203(4) | 9479(2) | 18.1(11) |
| C(6) | 5078(4) | 1091(3) | 6895(2) | 23.8(11) |
| C(33) | 5569(4) | 6264(4) | 8751(2) | 29.2(12) |
| C(62) | 1944(4) | 5926(4) | 5918(2) | 28.8(12) |
| C(1) | 4091(3) | 667(3) | 6722(2) | 18.0(10) |
| C(7) | 1977(3) | 670(3) | 6387(2) | 21.1(10) |
| C(43) | 5267(3) | 3350(3) | 5766(2) | 20.8(10) |
| C(37) | 5460(4) | 5117(3) | 6892(2) | 19.4(10) |
| C(60) | 2712(4) | 3796(4) | 4951(2) | 28.6(12) |
| C(22) | 4160(4) | 1170(4) | 8998(3) | 35.7(13) |
| C(42) | 6448(4) | 5616(4) | 7190(2) | 23.9(11) |
| C(13) | 2873(4) | 741(3) | 5351(2) | 22.2(10) |
| C(68) | 443(4) | 2541(4) | 5928(2) | 29.9(12) |
| C(25) | 2466(4) | 4206(3) | 8201(2) | 23.6(11) |
| C(21) | 4815(4) | 1451(4) | 8697(2) | 32.2(13) |
| C(26) | 1537(4) | 3649(4) | 7770(2) | 28.6(12) |
| C(14) | 2338(4) | 1220(4) | 4880(2) | 26.2(11) |
| C(0AA) | 3936(4) | -166(3) | 6921(2) | 26.9(11) |
| C(48) | 4877(4) | 2375(4) | 5358(2) | 26.0(11) |
| C(36) | 5375(4) | 4673(4) | 9239(2) | 27.4(12) |
| C(35) | 6212(4) | 5391(4) | 9588(2) | 36.7(13) |
| C(31) | 4629(4) | 4730(3) | 8626(2) | 20.4(10) |
| C(61) | 1939(3) | 5320(3) | 6337(2) | 23.5(11) |
| C(66) | 1953(4) | 5753(4) | 7021(2) | 33.5(13) |
| C(39) | 4827(4) | 6695(4) | 7015(2) | 31.0(12) |
| C(34) | 6324(4) | 6182(4) | 9346(2) | 33.4(12) |
| C(38) | 4648(4) | 5673(3) | 6809(2) | 23.1(11) |
| C(45) | 5842(5) | 3522(4) | 4837(3) | 44.4(15) |
| C(53) | 7891(4) | 2710(4) | 7361(3) | 31.6(12) |
| C(20) | 4641(4) | 2238(3) | 8435(2) | 25.9(11) |
| C(41) | 6621(4) | 6624(4) | 7391(2) | 27.3(11) |
| C(55) | 1814(4) | 3725(3) | 5101(2) | 21.6(11) |
| C(18) | 3290(4) | -99(4) | 5095(2) | 32.1(13) |

| | | | | |
|---|---|---|---|---|
| C(4) | 5722(4) | -113(4) | 7467(2) | 28.5(12) |
| C(15) | 2205(4) | 867(4) | 4173(2) | 31.3(12) |
| C(56) | 894(4) | 3442(4) | 4552(2) | 28.8(12) |
| C(23) | 3377(4) | 1698(4) | 9070(2) | 34.2(13) |
| C(32) | 4750(4) | 5546(4) | 8399(2) | 26.2(11) |
| C(49) | 6331(4) | 3448(3) | 7186(2) | 22.5(11) |
| C(44) | 5752(4) | 3923(4) | 5498(2) | 34.0(13) |
| C(54) | 7023(4) | 2946(4) | 6916(2) | 27.3(11) |
| C(69) | -518(4) | 2144(4) | 5855(3) | 38.3(14) |
| N(1) | 5714(5) | 1594(4) | 1540(3) | 71.8(18) |
| C(16) | 2624(4) | 31(4) | 3920(2) | 34.6(13) |
| C(57) | 858(4) | 3232(4) | 3853(3) | 40.2(14) |
| C(30) | 2472(4) | 5104(4) | 8685(2) | 29.7(12) |
| C(58) | 1750(4) | 3305(4) | 3713(3) | 35.8(13) |
| C(12) | 1899(4) | 930(4) | 7058(2) | 30.0(12) |
| C(8) | 1264(4) | -60(4) | 5862(3) | 34.4(13) |
| C(50) | 6541(4) | 3715(4) | 7910(2) | 31.6(13) |
| C(64) | 1995(4) | 7365(4) | 6866(3) | 38.9(14) |
| C(5) | 5901(4) | 701(4) | 7266(2) | 27.4(11) |
| C(17) | 3171(4) | -441(4) | 4385(3) | 40.6(15) |
| C(3) | 4751(4) | -545(4) | 7291(2) | 30.9(12) |
| C(65) | 1985(4) | 6764(4) | 7280(3) | 38.3(14) |
| C(70) | -1288(4) | 2716(4) | 5806(3) | 38.9(14) |
| C(59) | 2669(4) | 3584(4) | 4257(3) | 37.9(14) |
| C(74) | 5892(5) | 1824(4) | 1119(3) | 42.4(15) |
| C(24) | 3210(4) | 2500(4) | 8814(2) | 25.5(11) |
| C(47) | 4977(4) | 1984(4) | 4707(2) | 31.2(13) |
| C(27) | 633(4) | 3971(4) | 7825(3) | 44.6(15) |
| C(72) | -144(4) | 4098(4) | 5928(2) | 32.0(13) |
| C(11) | 1165(4) | 462(4) | 7200(3) | 34.7(13) |
| C(10) | 464(4) | -280(5) | 6670(3) | 46.7(15) |
| C(9) | 507(5) | -533(5) | 5997(3) | 50.8(16) |
| C(40) | 5805(4) | 7171(4) | 7310(2) | 31.1(12) |

| | | | | |
|---|---|---|---|---|
| C(46) | 5469(4) | 2557(4) | 4453(3) | 38.2(14) |
| C(63) | 1985(4) | 6944(4) | 6190(3) | 36.1(13) |
| C(73) | 6136(5) | 2168(5) | 599(3) | 58.5(19) |
| C(51) | 7401(4) | 3474(4) | 8344(3) | 41.6(15) |
| C(28) | 654(5) | 4853(5) | 8301(3) | 54.6(18) |
| C(52) | 8079(4) | 2976(4) | 8074(3) | 37.9(14) |
| C(29) | 1558(5) | 5438(4) | 8727(3) | 48.1(16) |
| C(71) | -1100(4) | 3693(5) | 5850(3) | 40.9(15) |

## 1.14 $[HPPh_3]_2[B_{12}Cl_{12}]$

**Tabelle 27** Kristalldaten und Strukturverfeinerung von $[HPPh_3]_2[B_{12}Cl_{12}]$.

| | |
|---|---|
| Probenkürzel | bw55 |
| Summenformel | $C_{36}H_{32}B_{12}Cl_{12}P_2$ |
| Molmasse | 1081.67 |
| Temperatur (in K) | 150.0(10) |
| Kristallsystem | Trigonal |
| Raumgruppe | $R\bar{3}$ |
| *a* (in Å) | 15.0147(12) |
| *b* (in Å) | 15.0147(12) |
| *c* (in Å) | 17.9690(14) |
| α (in °) | 90 |
| β (in °) | 90 |
| γ (in °) | 120 |
| Zellvolumen (in $Å^3$) | 3508.2(6) |
| *Z* | 3 |
| Berechnete Dichte (in $g/cm^3$) | 1.536 |
| Absorptionskoeffizient (in $mm^{-1}$) | 0.810 |
| F(000) | 1626.0 |
| Kristallgröße (in $mm^3$) | 0.08 × 0.06 × 0.05 |
| Strahlung | MoKα (λ = 0.71073) |
| 2Θ Bereich der Datenmessung (in °) | 5.426 bis 58.798 |

| | |
|---|---|
| Indexbereich | $-17 \leq h \leq 8, -19 \leq k \leq 18, -15 \leq l \leq 24$ |
| Gemessene Reflexe | 3208 |
| Unabhängige Reflexe | 1779 [$R_{int}$ = 0.0322, $R_{sigma}$ = 0.0620] |
| Daten/Beschränkungen/Parameter | 1779/0/96 |
| Anpassungsgüte $F^2$ | 1.023 |
| Finaler $R$ Wert [I>=2s (I)] | $R_1$ = 0.0405, $wR_2$ = 0.0714 |
| Finaler $R$ Wert [alle Daten] | $R_1$ = 0.0589, $wR_2$ = 0.0776 |
| Größtes Maximum und Minimum (in $Å^{-3}$) | 0.60/-0.29 |

**Tabelle 28** Atomkoordinaten ($\times 10^4$) und äquivalente, isotrope Auslenkungsparameter ($Å^2 \times 10^3$) von $[HPPh_3]_2[B_{12}Cl_{12}]$. $U_{äq}$ ist als ein Drittel der Spur des orthogonalisierten $U_{IJ}$ Tensors definiert.

| Atom | x | y | z | U(äq) |
|---|---|---|---|---|
| Cl1 | 1640.6(4) | 2604.3(4) | 5379.2(3) | 21.17(16) |
| Cl2 | 1632.5(5) | 599.3(5) | 6546.1(3) | 22.29(16) |
| P1 | 3333.33 | 6666.67 | 5121.4(6) | 15.8(2) |
| C1 | 2028.9(17) | 5888.8(17) | 5428.1(12) | 17.2(5) |
| C5 | 565.7(17) | 5638.8(18) | 6140.5(12) | 21.5(5) |
| C2 | 1487.9(17) | 4855.0(18) | 5209.7(12) | 19.8(5) |
| C6 | 1560.8(18) | 6282.2(18) | 5892.4(12) | 19.1(5) |
| C3 | 500.8(18) | 4221.0(18) | 5476.1(13) | 22.3(5) |
| C4 | 42.2(18) | 4610.5(19) | 5942.4(12) | 23.2(5) |
| B1 | 800.8(19) | 1269(2) | 5177.1(14) | 14.7(5) |
| B2 | 784.1(19) | 290(2) | 5749.4(14) | 15.3(5) |

## 1.15 $[(H_3O)(OPPh_3)_3]_2[B_{12}Cl_{12}]$

**Tabelle 29** Kristalldaten und Strukturverfeinerung von $[(H_3O)(OPPh_3)_3]_2[B_{12}Cl_{12}]$.

| | |
|---|---|
| Probenkürzel | bw69_full |
| Summenformel | $C_{108}H_{96}B_{12}Cl_{12}O_8P_6$ |
| Molmasse | 2262.78 |
| Temperatur (in K) | 293(2) |
| Kristallsystem | Triklin |

| | |
|---|---|
| Raumgruppe | $P\bar{1}$ |
| *a* (in Å) | 12.7554(4) |
| *b* (in Å) | 14.8134(4) |
| *c* (in Å) | 15.7123(5) |
| α (in °) | 79.624(3) |
| β (in °) | 81.823(3) |
| γ (in °) | 85.471(2) |
| Zellvolumen (in $Å^3$) | 2886.27(15) |
| *Z* | 1 |
| Berechnete Dichte (in $g/cm^3$) | 1.302 |
| Absorptionskoeffizient (in $mm^{-1}$) | 0.424 |
| F(000) | 1162.0 |
| Kristallgröße (in $mm^3$) | 0.17 × 0.12 × 0.1 |
| Strahlung | MoKα (λ = 0.71073) |
| 2Θ Bereich der Datenmessung (in °) | 5.102 bis 48 |
| Indexbereich | -14 ≤ h ≤ 14, -16 ≤ k ≤ 16, -17 ≤ l ≤ 17 |
| Gemessene Reflexe | 23320 |
| Unabhängige Reflexe | 9043 [$R_{int}$ = 0.0354, $R_{sigma}$ = 0.0510] |
| Daten/Beschränkungen/Parameter | 9043/126/682 |
| Anpassungsgüte $F^2$ | 1.046 |
| Finaler *R* Wert [I>=2s (I)] | $R_1$ = 0.0555, $wR_2$ = 0.1296 |
| Finaler *R* Wert [alle Daten] | $R_1$ = 0.0888, $wR_2$ = 0.1428 |
| Größtes Maximum und Minimum (in $Å^{-3}$) | 0.31/-0.28 |

**Tabelle 30** Atomkoordinaten ($\times 10^4$) und äquivalente, isotrope Auslenkungsparameter ($Å^2 \times 10^3$) von $[(H_3O)(OPPh_3)_3]_2[B_{12}Cl_{12}]$. $U_{äq}$ ist als ein Drittel der Spur des orthogonalisierten $U_{IJ}$ Tensors definiert.

| Atom | x | y | z | U(äq) |
|---|---|---|---|---|
| Cl1 | -874.4(7) | 9503.6(7) | 2242.4(6) | 54.9(3) |
| Cl2 | 473.3(8) | 7736.9(6) | 1016.3(6) | 56.6(3) |
| Cl3 | 1952.1(7) | 9641.3(6) | 1404.7(6) | 55.2(3) |
| Cl5 | -2381.4(7) | 11124.8(7) | 728.4(6) | 58.4(3) |
| Cl4 | 193.0(8) | 11726.5(6) | 1212.5(6) | 58.1(3) |
| Cl6 | -2198.6(7) | 8656.9(7) | 637.0(7) | 61.8(3) |
| P2 | 3897.9(7) | 7316.5(7) | 4480.7(7) | 50.0(3) |

| | | | | |
|---|---|---|---|---|
| P1 | 3800.8(8) | 4722.7(7) | 7795.9(7) | 57.5(3) |
| P3 | 7429.6(8) | 7463.0(8) | 6624.7(7) | 60.9(3) |
| O1 | 5578(3) | 5902(2) | 5915(2) | 62.0(8) |
| O3 | 4642.6(19) | 6488.5(18) | 4658.9(18) | 60.4(7) |
| O2 | 4253(2) | 5562(2) | 7238.7(19) | 79.7(9) |
| O4 | 6822(2) | 7092(2) | 6030(2) | 90.9(10) |
| C13 | 3001.6(18) | 4169.6(16) | 7217.4(14) | 47.7(9) |
| C18 | 3056.1(18) | 4437.1(16) | 6318.4(14) | 55.3(10) |
| C17 | 2439(2) | 4025(2) | 5851.2(13) | 72.1(12) |
| C16 | 1767(2) | 3346(2) | 6283(2) | 84.6(14) |
| C15 | 1713(2) | 3078.4(17) | 7182(2) | 83.4(14) |
| C14 | 2330(2) | 3490.2(18) | 7649.3(13) | 68.6(12) |
| C19 | 2855.6(18) | 7029.9(17) | 3943.0(17) | 48.3(9) |
| C20 | 2079(2) | 7670.4(15) | 3640(2) | 77.6(13) |
| C21 | 1286(2) | 7400(2) | 3233(2) | 97.4(17) |
| C22 | 1270(2) | 6489(2) | 3129(2) | 105.2(18) |
| C23 | 2047(3) | 5848.7(17) | 3432(2) | 106.4(19) |
| C24 | 2840(2) | 6119.0(15) | 3839(2) | 73.4(13) |
| C43 | 6636(2) | 7589(2) | 7638.9(17) | 57.6(10) |
| C44 | 5695(2) | 7136(3) | 7842(2) | 120(2) |
| C45 | 5025(2) | 7231(3) | 8602(2) | 159(3) |
| C46 | 5294(3) | 7779(3) | 9160.7(19) | 110.0(19) |
| C47 | 6235(3) | 8231(3) | 8958(2) | 121(2) |
| C48 | 6906(2) | 8136(3) | 8197(2) | 111(2) |
| B2 | 230(3) | 8905(3) | 498(2) | 37.5(9) |
| B3 | 951(3) | 9824(3) | 677(2) | 38.6(9) |
| B1 | -428(3) | 9761(3) | 1085(2) | 37.3(9) |
| C31 | 3289(2) | 7692(2) | 5467.3(16) | 59(1) |
| C36 | 2249(2) | 7506(2) | 5814.2(19) | 73.3(12) |
| C35 | 1824(2) | 7758(2) | 6608(2) | 99.1(17) |
| C34 | 2440(4) | 8195(3) | 7055.4(16) | 109(2) |
| C33 | 3481(3) | 8381(3) | 6709(2) | 136(3) |
| C32 | 3905(2) | 8129(3) | 5914(2) | 111(2) |

| | | | | |
|---|---|---|---|---|
| B5 | -1150(3) | 10543(3) | 357(2) | 38.2(9) |
| C25 | 4588(3) | 8242(3) | 3800(3) | 68.1(12) |
| C30A | 5639(3) | 8115(3) | 3516(3) | 84.1(15) |
| C29A | 6340(20) | 8683(16) | 3199(15) | 58(6) |
| C28A | 5935(17) | 9536(17) | 2954(18) | 77(6) |
| C27A | 4860(20) | 9771(17) | 3350(20) | 112(9) |
| C29 | 6118(9) | 8838(9) | 2844(10) | 90(4) |
| C28 | 5581(10) | 9605(7) | 2517(9) | 102(4) |
| C27 | 4560(9) | 9738(6) | 2811(10) | 110(4) |
| C26 | 4086(5) | 9081(4) | 3507(5) | 142(3) |
| C37 | 7918(2) | 8559.0(18) | 6101(2) | 67.0(11) |
| C38 | 7612(3) | 8949(3) | 5294(2) | 98.3(17) |
| C39 | 7992(3) | 9787(3) | 4863.7(19) | 118(2) |
| C40 | 8678(3) | 10234.6(19) | 5239(3) | 124(2) |
| C41 | 8984(2) | 9844(2) | 6046(3) | 103.4(18) |
| C42 | 8604(2) | 9007(2) | 6476.3(19) | 80.8(13) |
| C49 | 8592(2) | 6749(2) | 6833(2) | 66.7(11) |
| C50 | 8869(3) | 6454(3) | 7666(2) | 118(2) |
| C51 | 9770(3) | 5878(3) | 7784(3) | 156(3) |
| C52 | 10394(2) | 5597(2) | 7069(4) | 127(3) |
| C53 | 10116(3) | 5891(3) | 6236(3) | 130(3) |
| C54 | 9215(3) | 6467(3) | 6118(2) | 104.5(18) |
| B6 | -1069(3) | 9349(3) | 310(2) | 38.6(9) |
| C7 | 2921(3) | 5025(2) | 8727(2) | 67.9(11) |
| C8 | 2473(3) | 5918(2) | 8646(3) | 77.3(13) |
| C9 | 1668(5) | 6155(3) | 9274(4) | 96(6) |
| C10 | 1311(7) | 5499(5) | 9985(5) | 107(4) |
| C11 | 1759(8) | 4606(4) | 10066(4) | 114(4) |
| C12 | 2563(6) | 4369(3) | 9437(3) | 83(3) |
| C1 | 4760(9) | 3780(10) | 8176(10) | 60(5) |
| C2 | 4615(12) | 2846(10) | 8270(11) | 80(5) |
| C3 | 5420(14) | 2208(10) | 8525(11) | 105(6) |
| C4 | 6368(10) | 2505(12) | 8685(8) | 84(5) |

| | | | | |
|---|---|---|---|---|
| C5 | 6512(8) | 3440(12) | 8591(13) | 127(8) |
| C6 | 5708(11) | 4078(10) | 8337(14) | 137(9) |
| B4 | 94(3) | 10836(3) | 585(2) | 38.2(9) |
| C10A | 1791(17) | 5616(12) | 10140(12) | 95(5) |
| C11A | 2460(17) | 4819(12) | 10281(11) | 112(6) |
| C12A | 3164(15) | 4582(12) | 9557(11) | 105(6) |
| C9A | 1870(15) | 6135(10) | 9419(13) | 70(5) |
| C1A | 4840(7) | 4006(8) | 8207(7) | 74(4) |
| C2A | 5014(9) | 3102(8) | 8071(6) | 73(3) |
| C3A | 5864(10) | 2564(9) | 8387(5) | 90(5) |
| C4A | 6540(6) | 2931(11) | 8841(7) | 109(5) |
| C5A | 6365(9) | 3835(10) | 8977(9) | 152(6) |
| C6A | 5515(10) | 4373(8) | 8660(9) | 130(6) |

## 1.16 $[Au(PPh_3)_2]_2[B_{12}Cl_{12}]$

**Tabelle 31** Kristalldaten und Strukturverfeinerung von $[Au(PPh_3)_2]_2[B_{12}Cl_{12}]$.

| | |
|---|---|
| Probenkürzel | bw70 |
| Summenformel | $C_{72}H_{60}Au_2B_{12}Cl_{12}P_4$ |
| Molmasse | 1998.09 |
| Temperatur (in K) | 150.0(10) |
| Kristallsystem | Trigonal |
| Raumgruppe | $P\bar{3}$ |
| *a* (in Å) | 14.0658(7) |
| *b* (in Å) | 14.0658(7) |
| *c* (in Å) | 11.1977(7) |
| α (in °) | 90 |
| β (in °) | 90 |
| γ (in °) | 120 |
| Zellvolumen (in $Å^3$) | 1918.6(2) |
| *Z* | 100.002 |
| Berechnete Dichte (in $g/cm^3$) | 1.729 |

| | |
|---|---|
| Absorptionskoeffizient (in $mm^{-1}$) | 4.364 |
| F(000) | 974.0 |
| Kristallgröße (in $mm^3$) | 0.1 × 0.1 × 0.07 |
| Strahlung | MoKα (λ = 0.71073) |
| 2Θ Bereich der Datenmessung (in °) | 4.942 bis 59.076 |
| Indexbereich | -19 ≤ h ≤ 17, -17 ≤ k ≤ 19, -15 ≤ l ≤ 14 |
| Gemessene Reflexe | 5837 |
| Unabhängige Reflexe | 2958 [$R_{int}$ = 0.0237, $R_{sigma}$ = 0.0329] |
| Daten/Beschränkungen/Parameter | 2958/0/154 |
| Anpassungsgüte $F^2$ | 1.082 |
| Finaler *R* Wert [I>=2s (I)] | $R_1$ = 0.0248, $wR_2$ = 0.0572 |
| Finaler *R* Wert [alle Daten] | $R_1$ = 0.0292, $wR_2$ = 0.0592 |
| Größtes Maximum und Minimum (in $Å^{-3}$) | 0.83/-0.59 |

**Tabelle 32** Atomkoordinaten (×$10^4$) und äquivalente, isotrope Auslenkungsparameter ($Å^2$×$10^3$) von $[Au(PPh_3)_2]_2[B_{12}Cl_{12}]$. $U_{äq}$ ist als ein Drittel der Spur des orthogonalisierten $U_{IJ}$ Tensors definiert.

| Atom | x | y | z | U(äq) |
|---|---|---|---|---|
| Au1 | 3333.33 | 6666.67 | 1394.9(2) | 13.92(7) |
| Cl1 | 895.5(6) | 1752.2(6) | 7532.8(7) | 21.32(17) |
| Cl2 | -1372.5(6) | 1435.1(6) | 9409.3(7) | 21.74(17) |
| P2 | 3333.33 | 6666.67 | 3466.7(12) | 13.6(3) |
| P1 | 3333.33 | 6666.67 | -670.1(11) | 11.4(2) |
| C2 | 4992(3) | 6196(3) | -909(3) | 21.8(7) |
| C7 | 2412(2) | 5319(2) | 4062(3) | 15.9(6) |
| C1 | 3930(2) | 5888(2) | -1253(3) | 13.5(6) |
| C12 | 2288(3) | 4410(3) | 3430(3) | 19.2(6) |
| C6 | 3365(3) | 5003(3) | -2019(3) | 23.3(7) |
| C8 | 1823(3) | 5163(3) | 5107(3) | 26.0(7) |
| B2 | -669(3) | 698(3) | 9717(3) | 13.4(6) |
| C9 | 1133(3) | 4105(3) | 5515(3) | 32.8(9) |
| C3 | 5486(3) | 5620(3) | -1322(4) | 30.0(8) |
| C10 | 1019(3) | 3213(3) | 4892(3) | 26.9(8) |
| C11 | 1593(3) | 3361(3) | 3849(3) | 23.8(7) |
| C5 | 3863(3) | 4424(3) | -2406(4) | 33.1(9) |

| | | | | |
|---|---|---|---|---|
| B1 | 431(3) | 846(3) | 8797(3) | 14.0(6) |
| C4 | 4919(3) | 4736(3) | -2067(4) | 33.7(9) |

## 1.17 $[(H_3O)(OPPh_3)_3]_2[B_{12}Br_{12}]$

**Tabelle 33** Kristalldaten und Strukturverfeinerung von $[(H_3O)(OPPh_3)_3]_2[B_{12}Br_{12}]$.

| | |
|---|---|
| Probenkürzel | bw72b |
| Summenformel | $C_{108}H_{96}B_{12}Br_{12}O_8P_6$ |
| Molmasse | 2796.30 |
| Temperatur (in K) | 150.0(10) |
| Kristallsystem | Triklin |
| Raumgruppe | $P\bar{1}$ |
| *a* (in Å) | 12.6638(2) |
| *b* (in Å) | 14.8064(3) |
| *c* (in Å) | 15.8258(3) |
| α (in °) | 79.885(2) |
| β (in °) | 81.742(2) |
| γ (in °) | 85.465(2) |
| Zellvolumen (in $Å^3$) | 2886.59(10) |
| *Z* | 1 |
| Berechnete Dichte (in $g/cm^3$) | 1.609 |
| Absorptionskoeffizient (in $mm^{-1}$) | 4.299 |
| F(000) | 1378.0 |
| Kristallgröße (in $mm^3$) | 0.16 × 0.13 × 0.09 |
| Strahlung | MoKα (λ = 0.71073) |
| 2Θ Bereich der Datenmessung (in °) | 5.12 bis 52 |
| Indexbereich | $-15 \leq h \leq 15$, $-18 \leq k \leq 18$, $-19 \leq l \leq 19$ |
| Gemessene Reflexe | 26966 |
| Unabhängige Reflexe | 11340 [$R_{int}$ = 0.0331, $R_{sigma}$ = 0.0516] |
| Daten/Beschränkungen/Parameter | 11340/0/665 |
| Anpassungsgüte $F^2$ | 1.039 |
| Finaler *R* Wert [I>=2s (I)] | $R_1$ = 0.0348, $wR_2$ = 0.0660 |

| | |
|---|---|
| Finaler *R* Wert [alle Daten] | $R_1$ = 0.0602, $wR_2$ = 0.0719 |
| Größtes Maximum und Minimum (in Å$^{-3}$) | 0.77/-0.70 |

**Tabelle 34** Atomkoordinaten (×10$^4$) und äquivalente, isotrope Auslenkungsparameter (Å$^2$×10$^3$) von $[(H_3O)(OPPh_3)_3]_2[B_{12}Br_{12}]$. $U_{äq}$ ist als ein Drittel der Spur des orthogonalisierten $U_{IJ}$ Tensors definiert.

| Atom | x | y | z | U(äq) |
|---|---|---|---|---|
| Br2 | 9566.9(3) | 2366.6(2) | 8944.4(2) | 26.96(9) |
| Br1 | 10898.1(3) | 466.2(2) | 7676.3(2) | 25.77(9) |
| Br5 | 12060.0(3) | -410.6(2) | 11438.3(2) | 27.50(9) |
| Br4 | 10290.6(3) | 1805.9(2) | 11236.5(2) | 27.59(9) |
| Br6 | 12360.6(3) | 1338.7(2) | 9306.3(2) | 29.89(9) |
| Br3 | 7534.4(3) | 1233.1(2) | 10755.7(2) | 27.95(9) |
| P1 | 6132.7(7) | 2755.1(6) | 5509.4(7) | 31.6(2) |
| P3 | 6232.6(8) | 5248.3(6) | 2229.5(6) | 31.4(2) |
| P2 | 2619.8(7) | 2497.3(7) | 3430.8(7) | 34.1(2) |
| O2 | 5387.8(18) | 3595.0(16) | 5345.2(17) | 39.0(6) |
| O1 | 4443(2) | 4121.7(16) | 4071.1(18) | 40.8(7) |
| O3 | 3236(2) | 2869.5(19) | 4023.5(17) | 50.3(7) |
| O4 | 5800(2) | 4385.2(16) | 2751.8(18) | 41.6(7) |
| B2 | 9799(3) | 1099(2) | 9499(2) | 18.9(8) |
| B6 | 11092(3) | 627(2) | 9683(2) | 19.8(8) |
| C49 | 7013(3) | 5805(2) | 2830(2) | 27.2(8) |
| B1 | 10422(3) | 226(2) | 8920(2) | 21.3(8) |
| C54 | 6974(3) | 5509(2) | 3715(2) | 30.5(8) |
| B4 | 10131(3) | 839(2) | 10569(2) | 18.0(8) |
| B3 | 8854(3) | 574(2) | 10358(2) | 19.1(8) |
| B5 | 10968(3) | -194(2) | 10662(2) | 19.7(8) |
| C25 | 3410(3) | 2383(2) | 2420(2) | 31.0(8) |
| C1 | 7174(3) | 3017(2) | 6069(2) | 29.2(8) |
| C50 | 7682(3) | 6499(2) | 2431(2) | 35.2(9) |
| C31 | 1455(3) | 3233(2) | 3207(2) | 31.5(9) |
| C19 | 2126(3) | 1401(2) | 3951(3) | 36.6(9) |
| C48 | 7606(3) | 4126(2) | 1317(3) | 38.1(9) |
| C13 | 6756(3) | 2388(2) | 4524(2) | 31.5(9) |

| | | | | |
|---|---|---|---|---|
| C18 | 7805(3) | 2559(2) | 4189(2) | 35.8(9) |
| C53 | 7577(3) | 5917(3) | 4190(3) | 39.7(10) |
| C12 | 4339(3) | 1957(3) | 6396(3) | 42.8(10) |
| C36 | 822(3) | 3546(3) | 3901(3) | 53.2(12) |
| C51 | 8279(3) | 6905(2) | 2908(3) | 41.2(10) |
| C24 | 1401(3) | 960(2) | 3606(3) | 40(1) |
| C7 | 5407(3) | 1814(2) | 6140(3) | 39.5(10) |
| C47 | 8324(3) | 3922(3) | 629(3) | 45.9(11) |
| C43 | 7104(3) | 4992(2) | 1298(2) | 35.8(9) |
| C2 | 7163(3) | 3913(2) | 6235(3) | 46.3(11) |
| C14 | 6147(3) | 1985(3) | 4048(3) | 50.8(12) |
| C6 | 7972(3) | 2388(3) | 6339(3) | 43.8(10) |
| C16 | 7642(4) | 1900(3) | 2950(3) | 48.4(11) |
| C17 | 8243(3) | 2307(2) | 3412(3) | 43(1) |
| C37 | 5194(3) | 6054(3) | 1848(2) | 39.4(10) |
| C52 | 8225(3) | 6616(3) | 3781(3) | 44.3(10) |
| C32 | 1169(3) | 3509(3) | 2400(3) | 46.8(11) |
| C11 | 3760(3) | 1277(3) | 6923(3) | 57.0(13) |
| C5 | 8761(3) | 2643(3) | 6743(3) | 53.3(12) |
| C35 | -57(4) | 4134(3) | 3776(4) | 62.9(14) |
| C34 | -335(3) | 4408(3) | 2961(4) | 57.9(13) |
| C30 | 4288(4) | 2911(3) | 2146(3) | 61.5(13) |
| C23 | 1046(3) | 131(3) | 4030(3) | 48.4(11) |
| C28 | 4702(3) | 2262(3) | 856(3) | 51.2(11) |
| C3 | 7959(3) | 4159(3) | 6643(3) | 54.4(12) |
| C22 | 1367(4) | -258(3) | 4800(3) | 60.0(13) |
| C21 | 2068(4) | 162(3) | 5162(3) | 59.0(13) |
| C20 | 2466(3) | 999(3) | 4741(3) | 49.7(11) |
| C4 | 8758(3) | 3533(3) | 6895(3) | 51.6(12) |
| C40 | 3528(4) | 7210(4) | 1289(3) | 65.3(14) |
| C15 | 6588(4) | 1748(3) | 3264(3) | 57.4(12) |
| C26 | 3203(3) | 1798(3) | 1880(3) | 54.7(12) |
| C39 | 4273(4) | 7536(3) | 1650(3) | 62.7(13) |

| | | | | |
|---|---|---|---|---|
| C38 | 5119(3) | 6965(3) | 1932(3) | 51.0(11) |
| C33 | 275(3) | 4096(3) | 2275(3) | 61.2(13) |
| C27 | 3841(3) | 1741(3) | 1116(3) | 61.1(13) |
| C46 | 8572(4) | 4566(3) | -68(3) | 70.9(15) |
| C10 | 4259(4) | 451(3) | 7204(3) | 75.6(17) |
| C41 | 3591(4) | 6320(4) | 1169(4) | 86.6(18) |
| C42 | 4426(4) | 5727(3) | 1463(4) | 72.7(15) |
| C8 | 5896(4) | 961(3) | 6409(4) | 83.4(19) |
| C44 | 7359(5) | 5639(3) | 574(3) | 89(2) |
| C29 | 4924(4) | 2845(4) | 1373(3) | 76.8(17) |
| C45 | 8091(6) | 5431(3) | -100(4) | 125(3) |
| C9 | 5327(4) | 285(3) | 6947(4) | 105(2) |

## 1.18 $[Au(PPh_3)_2]_2[B_{12}Br_{12}]$

**Tabelle 35** Kristalldaten und Strukturverfeinerung von $[Au(PPh_3)_2]_2[B_{12}Br_{12}]$.

| | |
|---|---|
| Probenkürzel | bw72a |
| Summenformel | $C_{72}H_{60}Au_2B_{12}Br_{12}P_4$ |
| Molmasse | 2531.65 |
| Temperatur (in K) | 293(2) |
| Kristallsystem | Trigonal |
| Raumgruppe | $P\bar{3}$ |
| *a* (in Å) | 14.4300(14) |
| *b* (in Å) | 14.4300(14) |
| *c* (in Å) | 11.4021(10) |
| α (in °) | 90 |
| β (in °) | 90 |
| γ (in °) | 120 |
| Zellvolumen (in $Å^3$) | 2056.1(4) |
| *Z* | 1 |
| Berechnete Dichte (in $g/cm^3$) | 2.045 |
| Absorptionskoeffizient (in $mm^{-1}$) | 9.512 |
| F(000) | 1190.0 |

| | |
|---|---|
| Kristallgröße (in $mm^3$) | 0.1 × 0.08 × 0.05 |
| Strahlung | MoKα (λ = 0.71073) |
| 2Θ Bereich der Datenmessung (in °) | 4.836 bis 51.938 |
| Indexbereich | -13 ≤ h ≤ 17, -17 ≤ k ≤ 14, -11 ≤ l ≤ 14 |
| Gemessene Reflexe | 6740 |
| Unabhängige Reflexe | 2634 [$R_{int}$ = 0.0288, $R_{sigma}$ = 0.0370] |
| Daten/Beschränkungen/Parameter | 2634/0/154 |
| Anpassungsgüte $F^2$ | 0.972 |
| Finaler *R* Wert [I>=2s (I)] | $R_1$ = 0.0232, $wR_2$ = 0.0385 |
| Finaler *R* Wert [alle Daten] | $R_1$ = 0.0324, $wR_2$ = 0.0402 |
| Größtes Maximum und Minimum (in $Å^{-3}$) | 0.51/-0.66 |

**Tabelle 36** Atomkoordinaten (×$10^4$) und äquivalente, isotrope Auslenkungsparameter ($Å^2$×$10^3$) von $[Au(PPh_3)_2]_2[B_{12}Br_{12}]$. $U_{äq}$ ist als ein Drittel der Spur des orthogonalisierten $U_{IJ}$ Tensors definiert.

| Atom | x | y | z | U(äq) |
|---|---|---|---|---|
| Au1 | 6666.67 | 3333.33 | 8640.5(2) | 31.29(7) |
| Br1 | 1783.2(3) | 868.7(3) | 2542.7(3) | 48.76(11) |
| Br2 | 2869.1(3) | 1393.0(3) | -597.0(3) | 49.15(11) |
| P2 | 6666.67 | 3333.33 | 10681.7(10) | 26.9(3) |
| P1 | 6666.67 | 3333.33 | 6596.2(11) | 30.7(3) |
| C7 | 8009(2) | 3892(2) | 11267(2) | 30.7(7) |
| C1 | 5357(3) | 2945(2) | 6000(2) | 33.6(7) |
| C12 | 8297(3) | 3340(3) | 12046(3) | 52.3(10) |
| C6 | 4458(3) | 2147(3) | 6566(3) | 41.6(8) |
| B2 | 1335(3) | 645(3) | -290(3) | 30.7(8) |
| B1 | 824(3) | 398(3) | 1188(3) | 29.3(8) |
| C8 | 8774(3) | 4907(3) | 10924(3) | 52.1(9) |
| C5 | 3448(3) | 1822(3) | 6148(3) | 50.7(9) |
| C4 | 3317(3) | 2282(3) | 5163(3) | 57.5(11) |
| C9 | 9801(3) | 5372(3) | 11358(4) | 70.9(12) |
| C2 | 5211(3) | 3404(3) | 4994(3) | 55.5(10) |
| C10 | 10080(3) | 4822(4) | 12094(4) | 73.9(13) |
| C11 | 9340(4) | 3818(4) | 12448(4) | 75.1(13) |
| C3 | 4194(4) | 3069(4) | 4593(3) | 67.8(12) |

# 1.19 $[(H_3O)(OPPh_3)_3]_2[B_{12}I_{12}]$ (1. Polymorph)

**Tabelle 37** Kristalldaten und Strukturverfeinerung von $[(H_3O)(OPPh_3)_3]_2[B_{12}I_{12}]$ (1. Polymorph).

| | |
|---|---|
| Probenkürzel | bw73c |
| Summenformel | $C_{108}H_{96}B_{12}I_{12}O_8P_6$ |
| Molmasse | 3360.18 |
| Temperatur (in K) | 150.0(10) |
| Kristallsystem | Monoklin |
| Raumgruppe | *Cc* |
| *a* (in Å) | 19.7484(4) |
| *b* (in Å) | 33.8677(4) |
| *c* (in Å) | 19.3378(3) |
| α (in °) | 90 |
| β (in °) | 114.496(2) |
| γ (in °) | 90 |
| Zellvolumen (in $Å^3$) | 11769.6(4) |
| *Z* | 4 |
| Berechnete Dichte (in $g/cm^3$) | 1.896 |
| Absorptionskoeffizient (in $mm^{-1}$) | 3.292 |
| F(000) | 6376.0 |
| Kristallgröße (in $mm^3$) | 0.1 × 0.08 × 0.08 |
| Strahlung | MoKα (λ = 0.71073) |
| 2Θ Bereich der Datenmessung (in °) | 4.796 bis 54 |
| Indexbereich | $-25 \leq h \leq 23$, $-43 \leq k \leq 41$, $-24 \leq l \leq 24$ |
| Gemessene Reflexe | 32314 |
| Unabhängige Reflexe | 20496 [$R_{int}$ = 0.0290, $R_{sigma}$ = 0.0511] |
| Daten/Beschränkungen/Parameter | 20496/5/1334 |
| Anpassungsgüte $F^2$ | 0.984 |
| Finaler *R* Wert [I>=2s (I)] | $R_1$ = 0.0305, $wR_2$ = 0.0544 |
| Finaler *R* Wert [alle Daten] | $R_1$ = 0.0410, $wR_2$ = 0.0572 |
| Größtes Maximum und Minimum (in $Å^{-3}$) | 0.64/-0.49 |
| Flack Parameter | -0.039(12) |

**Tabelle 38** Atomkoordinaten ($\times 10^4$) und äquivalente, isotrope Auslenkungsparameter ($Å^2 \times 10^3$) von $[(H_3O)(OPPh_3)_3]_2[B_{12}I_{12}]$ (1. Polymorph). $U_{äq}$ ist als ein Drittel der Spur des orthogonalisierten $U_{IJ}$ Tensors definiert.

| Atom | x | y | z | U(äq) |
|---|---|---|---|---|
| I1 | 3196.3(3) | 4914.5(2) | 5409.2(3) | 24.59(12) |
| I2 | 4213.8(3) | 5739.1(2) | 4612.8(3) | 26.21(12) |
| I17 | 969.9(3) | 5180.7(2) | 4517.2(3) | 25.14(12) |
| I18 | 2611.7(3) | 6501.8(2) | 3250.1(3) | 27.31(13) |
| I19 | 3969.2(3) | 6811.4(2) | 5444.2(3) | 25.80(12) |
| I20 | 2815.1(3) | 6648.6(2) | 6731.5(3) | 27.26(13) |
| I21 | 756.0(3) | 6253.2(2) | 5366.1(3) | 24.59(12) |
| I16 | 2329.1(3) | 5474.3(2) | 6703.4(3) | 26.07(12) |
| I22 | 1755.0(3) | 7067.4(2) | 4551.5(3) | 24.29(12) |
| I23 | 630.3(3) | 6157.3(2) | 3207.7(3) | 27.45(13) |
| I24 | 2160.1(3) | 5331.7(2) | 3235.2(3) | 28.07(13) |
| I15 | 4319.5(3) | 5835.1(2) | 6766.3(3) | 28.21(13) |
| P31 | 1925.7(12) | 3566.8(6) | 5277.2(13) | 26.1(5) |
| P51 | 5686.2(12) | 4458.6(6) | 4725.2(14) | 27.7(5) |
| P71 | 7836.3(12) | 3464.6(6) | 4355.9(14) | 27.2(5) |
| P91 | 6633.3(12) | 6929.3(6) | 5535.5(14) | 27.4(5) |
| P111 | 8293.0(13) | 5129.5(6) | 4495.6(15) | 29.4(5) |
| P125 | 4407.7(12) | 2564.0(6) | 5759.1(14) | 27.5(5) |
| B12 | 3297(5) | 5918(2) | 5769(5) | 17.3(19) |
| O146 | 2874(4) | 2718.2(17) | 6287(4) | 34.7(16) |
| O52 | 6263(3) | 4199.5(15) | 4623(4) | 35.9(15) |
| B10 | 1706(5) | 6104(2) | 5143(5) | 13.1(17) |
| O92 | 7393(3) | 7077.0(16) | 5654(4) | 34.7(15) |
| O32 | 1875(3) | 3127.4(15) | 5369(4) | 34.4(15) |
| O112 | 7522(4) | 5004.0(16) | 4342(4) | 43.9(18) |
| O147 | 6868(4) | 4369.8(19) | 3768(4) | 33.8(15) |
| B6 | 2162(5) | 6463(2) | 4793(5) | 15.4(18) |
| B9 | 1816(5) | 5633(2) | 4780(5) | 17.1(18) |
| O72 | 7913(3) | 3889.2(15) | 4186(4) | 38.3(16) |
| B8 | 2414(5) | 5766(2) | 5745(5) | 16.9(18) |
| B11 | 2624(5) | 6284(2) | 5747(5) | 15.6(18) |

| | | | | |
|---|---|---|---|---|
| C40 | 3329(4) | 3786(2) | 5409(5) | 27.3(19) |
| B5 | 3135(5) | 6352(2) | 5185(5) | 16.0(18) |
| B13 | 2792(5) | 5516(2) | 5164(5) | 13.1(17) |
| B3 | 2335(5) | 5701(2) | 4207(5) | 17.0(18) |
| O133 | 4001(3) | 2846.8(15) | 6051(4) | 36.3(15) |
| C99 | 6027(5) | 6910(2) | 4538(5) | 26.1(19) |
| C34 | 822(5) | 3613(3) | 5772(5) | 30(2) |
| C103 | 5807(6) | 7053(3) | 3244(6) | 39(3) |
| B4 | 3243(5) | 5878(2) | 4821(5) | 18.8(19) |
| C119 | 8274(5) | 5620(2) | 4142(5) | 26.7(19) |
| B14 | 2544(5) | 6219(2) | 4223(5) | 15.7(18) |
| C59 | 4787(5) | 4224(2) | 4332(5) | 27.6(19) |
| C98 | 6198(5) | 6276(2) | 6151(6) | 33(2) |
| C37 | 1008(5) | 4407(3) | 6130(5) | 33(2) |
| C39 | 2852(4) | 3751(2) | 5769(5) | 25.0(18) |
| C44 | 3120(5) | 3842(2) | 6539(5) | 32(2) |
| C66 | 8252(5) | 2773(2) | 3810(5) | 31(2) |
| B7 | 1663(5) | 6063(2) | 4196(5) | 17.4(18) |
| C88 | 5671(5) | 7735(2) | 6796(6) | 36(2) |
| C79 | 6888(4) | 3300(2) | 3899(5) | 25.9(18) |
| C33 | 1362(5) | 3829(2) | 5650(5) | 27.6(19) |
| C41 | 4056(5) | 3911(2) | 5802(6) | 31(2) |
| C55 | 6828(5) | 4462(3) | 7020(6) | 43(3) |
| C83 | 5781(5) | 3024(2) | 3934(6) | 34(2) |
| C113 | 8736(5) | 4816(2) | 4062(5) | 29(2) |
| C89 | 6354(6) | 7552(3) | 7178(6) | 41(2) |
| C109 | 9794(8) | 5055(3) | 7026(7) | 67(4) |
| C53 | 5955(5) | 4555(2) | 5713(5) | 29(2) |
| C97 | 6290(5) | 5892(3) | 6426(6) | 39(2) |
| C118 | 9390(5) | 4614(2) | 4484(6) | 34(2) |
| C107 | 9581(5) | 5297(2) | 5796(5) | 35(2) |
| C136 | 4241(5) | 1744(2) | 5591(5) | 32(2) |
| C50 | 5970(5) | 5253(2) | 4631(5) | 31(2) |

| | | | | |
|---|---|---|---|---|
| C84 | 6519(5) | 3123(2) | 4297(5) | 30(2) |
| C73 | 8171(4) | 3383(2) | 5366(5) | 26.1(18) |
| C117 | 9710(5) | 4364(2) | 4141(5) | 33(2) |
| C137 | 4376(5) | 2066(2) | 6067(5) | 28.6(19) |
| C120 | 7659(5) | 5855(2) | 4057(6) | 35(2) |
| C69 | 9576(5) | 3101(3) | 3954(6) | 36(2) |
| C123 | 8763(5) | 6170(3) | 3698(6) | 36(2) |
| C43 | 3840(5) | 3968(2) | 6931(5) | 35(2) |
| C49 | 5886(5) | 5611(3) | 4241(6) | 35(2) |
| C127 | 5944(5) | 2490(2) | 6126(5) | 32(2) |
| C124 | 8820(5) | 5779(2) | 3954(5) | 31(2) |
| C28 | 1263(5) | 3845(3) | 2759(5) | 43(2) |
| C100 | 5333(5) | 6715(2) | 4274(5) | 31(2) |
| C102 | 5124(5) | 6862(2) | 2981(6) | 36(2) |
| C135 | 4178(5) | 1365(2) | 5841(6) | 34(2) |
| C74 | 8357(5) | 3011(3) | 5687(6) | 37(2) |
| C38 | 1463(5) | 4232(2) | 5842(5) | 25.5(18) |
| C81 | 5755(5) | 3286(3) | 2764(6) | 45(2) |
| C121 | 7620(6) | 6240(3) | 3792(6) | 48(3) |
| C29 | 1409(5) | 4150(3) | 3271(5) | 37(2) |
| C61 | 3456(5) | 4240(3) | 3816(5) | 35(2) |
| C67 | 8744(5) | 2553(2) | 3622(5) | 34(2) |
| C126 | 5351(4) | 2729(2) | 6065(4) | 22.3(17) |
| C78 | 8299(6) | 3715(3) | 5835(6) | 41(2) |
| C48 | 5433(5) | 5634(2) | 3490(6) | 37(2) |
| C60 | 4132(5) | 4437(3) | 4145(5) | 30(2) |
| C93 | 6711(5) | 6434(2) | 5905(5) | 29(2) |
| C82 | 5388(5) | 3110(3) | 3168(6) | 40(2) |
| C56 | 6401(5) | 4693(3) | 7271(6) | 42(2) |
| C62 | 3429(6) | 3842(3) | 3668(5) | 39(2) |
| C36 | 480(5) | 4192(3) | 6255(5) | 36(2) |
| C45 | 5571(5) | 4926(2) | 4251(5) | 31(2) |
| C142 | 4025(5) | 2545(2) | 4740(5) | 29.7(19) |

| | | | | |
|---|---|---|---|---|
| C87 | 5249(5) | 7660(2) | 6035(6) | 35(2) |
| C104 | 6257(6) | 7078(2) | 4019(6) | 36(2) |
| C70 | 9090(5) | 3325(3) | 4134(6) | 33(2) |
| C27 | 1302(6) | 3457(3) | 3007(6) | 50(3) |
| C86 | 5529(5) | 7414(2) | 5640(5) | 30(2) |
| C80 | 6496(5) | 3378(2) | 3139(5) | 36(2) |
| C101 | 4891(5) | 6699(2) | 3499(6) | 36(2) |
| C68 | 9402(5) | 2722(3) | 3684(6) | 39(2) |
| C57 | 5751(5) | 4853(2) | 6746(5) | 39(2) |
| C30 | 1601(5) | 4071(2) | 4043(5) | 29.5(19) |
| C58 | 5513(5) | 4787(2) | 5964(5) | 31(2) |
| C47 | 5035(6) | 5311(2) | 3109(5) | 42(2) |
| C128 | 6644(5) | 2647(3) | 6314(5) | 38(2) |
| C122 | 8160(6) | 6395(3) | 3608(6) | 44(3) |
| C65 | 8417(4) | 3165(2) | 4061(5) | 25.5(19) |
| C76 | 8791(5) | 3296(3) | 6935(6) | 43(2) |
| C131 | 5474(5) | 3130(2) | 6202(6) | 36(2) |
| C85 | 6214(5) | 7235(2) | 6016(5) | 25.5(19) |
| C64 | 4756(5) | 3824(2) | 4194(5) | 34(2) |
| C138 | 4431(6) | 2010(3) | 6792(6) | 44(3) |
| C94 | 7277(5) | 6199(3) | 5886(6) | 39(2) |
| C90 | 6624(5) | 7309(2) | 6790(5) | 32(2) |
| C106 | 8869(5) | 5127(2) | 5491(6) | 32(2) |
| C108 | 10046(7) | 5265(3) | 6563(6) | 49(3) |
| C77 | 8608(6) | 3663(3) | 6620(6) | 51(3) |
| C141 | 3289(5) | 2665(3) | 4331(6) | 46(3) |
| C143 | 4433(5) | 2414(2) | 4350(5) | 35(2) |
| C114 | 8405(6) | 4748(4) | 3295(7) | 64(4) |
| C129 | 6754(6) | 3048(3) | 6445(6) | 46(3) |
| C96 | 6873(6) | 5664(3) | 6422(6) | 46(3) |
| C25 | 1642(5) | 3685(2) | 4290(5) | 28.0(19) |
| C130 | 6180(6) | 3286(3) | 6408(7) | 51(3) |
| C105 | 8648(7) | 4926(3) | 5979(7) | 51(3) |

| | | | | |
|---|---|---|---|---|
| C54 | 6615(5) | 4392(2) | 6249(6) | 37(2) |
| C75 | 8660(5) | 2965(3) | 6468(6) | 40(2) |
| C46 | 5105(6) | 4955(2) | 3486(5) | 37(2) |
| C35 | 374(5) | 3797(3) | 6069(6) | 37(2) |
| C26 | 1488(5) | 3378(3) | 3771(5) | 36(2) |
| C95 | 7364(5) | 5811(3) | 6144(6) | 45(3) |
| C145 | 3377(6) | 2518(3) | 3158(6) | 55(3) |
| C116 | 9386(5) | 4304(3) | 3378(6) | 40(2) |
| C140 | 2968(7) | 2649(3) | 3537(7) | 60(3) |
| C134 | 4244(5) | 1311(3) | 6562(6) | 37(2) |
| C110 | 9111(9) | 4886(4) | 6754(8) | 70(4) |
| C139 | 4368(6) | 1625(3) | 7048(6) | 53(3) |
| C42 | 4314(5) | 4002(2) | 6561(5) | 31(2) |
| C63 | 4075(6) | 3635(3) | 3867(6) | 41(3) |
| C144 | 4115(6) | 2395(3) | 3565(6) | 49(3) |
| C115 | 8728(7) | 4489(4) | 2954(7) | 83(5) |

## 1.20 $[(H_3O)(OPPh_3)_3]_2[B_{12}I_{12}]$ (2. Polymorph)

**Tabelle 39** Kristalldaten und Strukturverfeinerung von $[(H_3O)(OPPh_3)_3]_2[B_{12}I_{12}]$ (2. Polymorph).

| | |
|---|---|
| Probenkürzel | bw87a |
| Summenformel | $C_{108}H_{96}B_{12}I_{12}O_8P_6$ |
| Molmasse | 3360.18 |
| Temperatur (in K) | 150.0(10) |
| Kristallsystem | Triklin |
| Raumgruppe | $P\bar{1}$ |
| *a* (in Å) | 12.7631(4) |
| *b* (in Å) | 15.0721(4) |
| *c* (in Å) | 16.1196(4) |
| α (in °) | 79.304(2) |
| β (in °) | 81.788(2) |
| γ (in °) | 85.183(2) |

| | |
|---|---|
| Zellvolumen (in $Å^3$) | 3010.49(15) |
| *Z* | 1 |
| Berechnete Dichte (in $g/cm^3$) | 1.853 |
| Absorptionskoeffizient (in $mm^{-1}$) | 3.217 |
| F(000) | 1594.0 |
| Kristallgröße (in $mm^3$) | 0.1 × 0.08 × 0.07 |
| Strahlung | MoKα (λ = 0.71073) |
| 2Θ Bereich der Datenmessung (in °) | 5.114 bis 59.224 |
| Indexbereich | $-16 \leq h \leq 17$, $-20 \leq k \leq 20$, $-21 \leq l \leq 22$ |
| Gemessene Reflexe | 31272 |
| Unabhängige Reflexe | 14184 [$R_{int}$ = 0.0320, $R_{sigma}$ = 0.0481] |
| Daten/Beschränkungen/Parameter | 14184/3/670 |
| Anpassungsgüte $F^2$ | 1.036 |
| Finaler *R* Wert [I>=2s (I)] | $R_1$ = 0.0312, $wR_2$ = 0.0556 |
| Finaler *R* Wert [alle Daten] | $R_1$ = 0.0476, $wR_2$ = 0.0613 |
| Größtes Maximum und Minimum (in $Å^{-3}$) | 0.83/-0.61 |

**Tabelle 40** Atomkoordinaten ($\times 10^4$) und äquivalente, isotrope Auslenkungsparameter ($Å^2 \times 10^3$) von $[(H_3O)(OPPh_3)_3]_2[B_{12}I_{12}]$ (2. Polymorph). $U_{äq}$ ist als ein Drittel der Spur des orthogonalisierten $U_{IJ}$ Tensors definiert.

| Atom | x | y | z | U(äq) |
|---|---|---|---|---|
| I1 | 10327.7(2) | 1880.5(2) | 6258.0(2) | 21.40(6) |
| I2 | 9067.5(2) | -463.3(2) | 7421.5(2) | 20.38(6) |
| I3 | 10436.0(2) | -2465.4(2) | 6124.8(2) | 22.04(6) |
| I4 | 7502.0(2) | -1364.0(2) | 5739.4(2) | 23.69(6) |
| I5 | 7415.3(2) | 1302.5(2) | 5766.2(2) | 21.98(6) |
| I6 | 7837.7(2) | 438.4(2) | 3497.1(2) | 21.75(6) |
| B1 | 10136(3) | 825(2) | 5550(2) | 14.7(8) |
| B2 | 9578(3) | -210(2) | 6063(2) | 15.7(8) |
| B3 | 10195(3) | -1084(2) | 5509(2) | 14.5(8) |
| B4 | 8899(3) | -605(2) | 5322(2) | 15.6(8) |
| B5 | 8862(3) | 575(2) | 5346(2) | 13.9(8) |
| B6 | 9036(3) | 199(3) | 4345(2) | 15.1(8) |
| P1 | 7360.9(8) | 7428.7(7) | 1543.4(6) | 27.1(2) |
| O2 | 6763(2) | 7033(2) | 980.2(18) | 42.5(8) |

| | | | | |
|---|---|---|---|---|
| C1 | 7802(3) | 8509(3) | 1000(2) | 29.5(9) |
| C2 | 8547(3) | 8961(3) | 1286(3) | 34.1(10) |
| C3 | 8868(4) | 9785(3) | 840(3) | 47.4(13) |
| C4 | 8457(4) | 10153(3) | 100(3) | 55.7(15) |
| C5 | 7725(4) | 9717(3) | -194(3) | 52.8(14) |
| C6 | 7394(4) | 8894(3) | 254(3) | 40.1(11) |
| C7 | 8539(3) | 6732(2) | 1776(3) | 26.9(9) |
| C8 | 9210(4) | 6472(3) | 1095(3) | 43.8(12) |
| C9 | 10082(4) | 5899(3) | 1227(4) | 52.1(14) |
| C10 | 10313(4) | 5589(3) | 2044(4) | 52.9(14) |
| C11 | 9676(4) | 5863(3) | 2718(3) | 49.0(13) |
| C12 | 8775(4) | 6421(3) | 2590(3) | 37.8(11) |
| C13 | 6573(3) | 7545(2) | 2537(2) | 24.4(8) |
| C14 | 6781(3) | 8147(3) | 3040(3) | 34.8(10) |
| C15 | 6165(4) | 8187(3) | 3811(3) | 40.9(11) |
| C16 | 5353(3) | 7627(3) | 4098(3) | 37.7(10) |
| C17 | 5133(4) | 7038(3) | 3600(3) | 52.0(14) |
| C18 | 5727(4) | 7000(3) | 2828(3) | 43.5(12) |
| P2 | 3726.5(8) | 4768.7(6) | 2714.4(6) | 22.8(2) |
| P3 | 3835.1(8) | 7216.5(6) | -492.2(6) | 21.1(2) |
| O1 | 5501(2) | 5875.0(17) | 934.2(17) | 25.8(6) |
| O3 | 4139(2) | 5640.9(16) | 2213.1(16) | 27.3(6) |
| O4 | 4595(2) | 6404.4(16) | -330.3(16) | 26.6(6) |
| C19 | 4766(3) | 3979(2) | 3073(2) | 26.7(9) |
| C20 | 5529(4) | 4298(3) | 3451(3) | 52.9(14) |
| C21 | 6353(4) | 3728(3) | 3738(4) | 57.2(14) |
| C22 | 6420(4) | 2841(3) | 3645(3) | 42.9(11) |
| C23 | 5668(4) | 2515(3) | 3283(3) | 44.4(12) |
| C24 | 4837(3) | 3081(3) | 2993(3) | 36.1(10) |
| C25 | 2863(3) | 4974(2) | 3645(2) | 25.9(9) |
| C26 | 2609(5) | 4296(3) | 4328(3) | 56.9(16) |
| C27 | 1891(6) | 4465(3) | 5015(3) | 80(2) |
| C28 | 1454(4) | 5337(3) | 5031(3) | 54.9(15) |

| | | | | |
|---|---|---|---|---|
| C29 | 1714(4) | 6008(3) | 4374(3) | 39.6(11) |
| C30 | 2411(3) | 5835(2) | 3671(3) | 28.7(9) |
| C31 | 2978(3) | 4242(2) | 2097(2) | 20.0(8) |
| C32 | 2317(3) | 3549(2) | 2478(2) | 27.0(9) |
| C33 | 1776(3) | 3139(3) | 1985(3) | 30.8(9) |
| C34 | 1861(3) | 3434(3) | 1118(3) | 34.8(10) |
| C35 | 2493(3) | 4131(3) | 737(3) | 33.1(10) |
| C36 | 3056(3) | 4539(2) | 1224(2) | 25.0(8) |
| C37 | 3238(3) | 7581(2) | 472(2) | 21.3(8) |
| C38 | 3837(3) | 8023(3) | 902(3) | 35.1(10) |
| C39 | 3409(4) | 8259(3) | 1674(3) | 43.9(12) |
| C40 | 2388(4) | 8078(3) | 2009(3) | 39.1(11) |
| C41 | 1788(4) | 7642(3) | 1590(3) | 38.2(11) |
| C42 | 2208(3) | 7387(3) | 830(3) | 32.5(10) |
| C43 | 2794(3) | 6937(2) | -1014(2) | 21.9(8) |
| C44 | 2841(3) | 6070(2) | -1197(2) | 28.5(9) |
| C45 | 2037(4) | 5801(3) | -1567(3) | 40.1(11) |
| C46 | 1181(4) | 6387(3) | -1751(3) | 40.9(11) |
| C47 | 1137(4) | 7255(3) | -1590(3) | 40.9(11) |
| C48 | 1934(3) | 7530(3) | -1222(3) | 33.8(10) |
| C49 | 4513(3) | 8152(2) | -1132(2) | 24.9(8) |
| C50 | 5599(3) | 8026(3) | -1369(3) | 34.3(10) |
| C51 | 6145(4) | 8707(3) | -1884(3) | 46.1(13) |
| C52 | 5632(4) | 9511(3) | -2182(3) | 50.3(14) |
| C53 | 4566(4) | 9654(3) | -1947(3) | 59.7(16) |
| C54 | 4006(4) | 8974(3) | -1414(3) | 46.6(13) |

# 1.21 $[Au(PPh_3)_3]_2[B_{12}I_{12}]$

**Tabelle 41** Kristalldaten und Strukturverfeinerung von $[Au(PPh_3)_3]_2[B_{12}I_{12}]$.

| | |
|---|---|
| Probenkürzel | bw73a_2 |
| Summenformel | $C_{108}H_{90}Au_2B_{12}I_{12}P_6$ |
| Molmasse | 3620.07 |

| | |
|---|---|
| Temperatur (in K) | 293(2) |
| Kristallsystem | Trigonal |
| Raumgruppe | $R\bar{3}$ |
| *a* (in Å) | 19.0679(8) |
| *b* (in Å) | 19.0679(8) |
| *c* (in Å) | 27.2696(14) |
| α (in °) | 90 |
| β (in °) | 90 |
| γ (in °) | 120 |
| Zellvolumen (in $Å^3$) | 8586.5(8) |
| *Z* | 3 |
| Berechnete Dichte (in $g/cm^3$) | 2.100 |
| Absorptionskoeffizient (in $mm^{-1}$) | 5.926 |
| F(000) | 5046.0 |
| Kristallgröße (in $mm^3$) | 0.05 × 0.04 × 0.03 |
| Strahlung | MoKα (λ = 0.71073) |
| 2Θ Bereich der Datenmessung (in °) | 5.154 bis 51.994 |
| Indexbereich | $-8 \leq h \leq 23$, $-23 \leq k \leq 20$, $-21 \leq l \leq 33$ |
| Gemessene Reflexe | 6599 |
| Unabhängige Reflexe | 3766 [$R_{int}$ = 0.0297, $R_{sigma}$ = 0.0584] |
| Daten/Beschränkungen/Parameter | 3766/0/211 |
| Anpassungsgüte $F^2$ | 0.982 |
| Finaler *R* Wert [I>=2s (I)] | $R_1$ = 0.0358, $wR_2$ = 0.0511 |
| Finaler *R* Wert [alle Daten] | $R_1$ = 0.0616, $wR_2$ = 0.0562 |
| Größtes Maximum und Minimum (in $Å^{-3}$) | 0.64/-0.58 |

**Tabelle 42** Atomkoordinaten ($\times 10^4$) und äquivalente, isotrope Auslenkungsparameter ($Å^2 \times 10^3$) von $[Au(PPh_3)_3]_2[B_{12}I_{12}]$. $U_{äq}$ ist als ein Drittel der Spur des orthogonalisierten $U_{IJ}$ Tensors definiert.

| Atom | x | y | z | U(äq) |
|---|---|---|---|---|
| Au1 | 6666.67 | 3333.33 | 5381.4(2) | 28.91(10) |
| I1 | 6698.1(2) | 4580.9(2) | 7213.4(2) | 50.41(13) |
| I2 | 8657.0(2) | 5306.1(2) | 8058.1(2) | 49.00(13) |
| P1 | 7408.7(8) | 4765.9(7) | 5355.6(5) | 28.6(3) |
| C13 | 7626(3) | 5113(3) | 4726.3(19) | 30.9(12) |

| | | | | |
|---|---|---|---|---|
| C18 | 7605(3) | 4597(3) | 4364(2) | 41.0(14) |
| C1 | 6917(3) | 5297(3) | 5595(2) | 34.8(13) |
| C7 | 8376(3) | 5231(3) | 5662.7(19) | 32.0(13) |
| C14 | 7782(3) | 5879(3) | 4591(2) | 41.6(14) |
| B2 | 7540(3) | 4196(3) | 8216(2) | 29.0(14) |
| B1 | 6678(3) | 3882(3) | 7839(2) | 26.7(13) |
| C8 | 8434(3) | 4914(3) | 6101(2) | 47.1(15) |
| C2 | 6228(4) | 5192(4) | 5377(2) | 58.5(18) |
| C15 | 7910(3) | 6113(4) | 4103(2) | 52.1(17) |
| C16 | 7862(3) | 5580(5) | 3752(2) | 58.6(18) |
| C12 | 9038(3) | 5891(3) | 5478(2) | 56.8(18) |
| C9 | 9159(4) | 5276(4) | 6349(2) | 58.8(18) |
| C6 | 7217(4) | 5796(4) | 5992(2) | 61.0(18) |
| C10 | 9823(4) | 5943(4) | 6159(2) | 57.7(18) |
| C17 | 7721(3) | 4835(4) | 3881(2) | 59.5(19) |
| C11 | 9762(4) | 6242(4) | 5722(2) | 67(2) |
| C3 | 5835(4) | 5581(4) | 5555(3) | 74(2) |
| C4 | 6146(5) | 6084(4) | 5944(3) | 80(3) |
| C5 | 6827(5) | 6187(4) | 6162(3) | 86(3) |

## 1.22 $[Pyr_4][PF_6]_2 \cdot 2Pyr \cdot 2CH_2Cl_2$

**Tabelle 43** Kristalldaten und Strukturverfeinerung von $[Pyr_4][PF_6]_2 \cdot 2Pyr \cdot 2CH_2Cl_2$.

| | |
|---|---|
| Probenkürzel | bw41 |
| Summenformel | $C_{90}H_{59}Cl_4F_{12}P_2$ |
| Molmasse | 1572.11 |
| Temperatur (in K) | 150.0(10) |
| Kristallsystem | Triklin |
| Raumgruppe | $P\bar{1}$ |
| *a* (in Å) | 14.3652(9) |
| *b* (in Å) | 16.7147(7) |
| *c* (in Å) | 16.8891(10) |
| α (in °) | 76.704(4) |

| | |
|---|---|
| β (in °) | 66.555(6) |
| γ (in °) | 69.943(5) |
| Zellvolumen (in $Å^3$) | 3474.2(4) |
| *Z* | 2 |
| Berechnete Dichte (in $g/cm^3$) | 1.503 |
| Absorptionskoeffizient (in $mm^{-1}$) | 0.301 |
| F(000) | 1610.0 |
| Kristallgröße (in $mm^3$) | 0.18 × 0.08 × 0.06 |
| Strahlung | MoKα (λ = 0.71073) |
| 2Θ Bereich der Datenmessung (in °) | 4.828 bis 54 |
| Indexbereich | $-18 \leq h \leq 18$, $-20 \leq k \leq 21$, $-21 \leq l \leq 21$ |
| Gemessene Reflexe | 35188 |
| Unabhängige Reflexe | 14959 [$R_{int}$ = 0.0320, $R_{sigma}$ = 0.0486] |
| Daten/Beschränkungen/Parameter | 14959/6/1065 |
| Anpassungsgüte $F^2$ | 1.054 |
| Finaler *R* Wert [I>=2s (I)] | $R_1$ = 0.0559, $wR_2$ = 0.1310 |
| Finaler *R* Wert [alle Daten] | $R_1$ = 0.0912, $wR_2$ = 0.1457 |
| Größtes Maximum und Minimum (in $Å^{-3}$) | 1.00/-0.83 |

**Tabelle 44** Atomkoordinaten ($\times 10^4$) und äquivalente, isotrope Auslenkungsparameter ($Å^2 \times 10^3$) von $[Pyr_4][PF_6]_2 \cdot 2Pyr \cdot 2CH_2Cl_2$. $U_{äq}$ ist als ein Drittel der Spur des orthogonalisierten $U_{IJ}$ Tensors definiert.

| Atom | x | y | z | U(äq) |
|---|---|---|---|---|
| P(2) | 2930.3(5) | 1068.5(4) | 9078.6(4) | 32.59(16) |
| P(1) | 2787.4(5) | 4188.5(4) | 6029.6(4) | 33.87(16) |
| Cl(1) | 2615.5(7) | 1488.6(5) | 6343.1(7) | 75.4(3) |
| Cl(2) | 4879.7(7) | 735.0(6) | 5756.4(6) | 78.9(3) |
| Cl(3A) | 236(4) | 3602(5) | 8617(3) | 78.2(13) |
| Cl(4B) | 1701(4) | 4065(2) | 9253(3) | 75.0(11) |
| F(14) | 3486.3(12) | 324.9(9) | 8459.7(10) | 47.2(4) |
| F(11) | 2379.9(13) | 1818.4(9) | 9689.0(10) | 54.3(4) |
| F(9) | 1974.8(13) | 1392.2(10) | 8735.1(11) | 59.6(5) |
| F(8) | 1823.2(13) | 3808.8(11) | 6322.5(12) | 64.9(5) |
| F(3) | 3493.8(13) | 3480.4(10) | 5385.0(11) | 62.1(5) |
| F(6) | 2087.1(13) | 4869.3(11) | 6704.9(12) | 68.0(5) |

| | | | | |
|---|---|---|---|---|
| F(10) | 3507.7(16) | 1688.7(11) | 8327.6(11) | 67.6(5) |
| F(13) | 2336.7(16) | 468.2(10) | 9828.7(11) | 71.1(5) |
| F(12) | 3883.0(14) | 752.8(12) | 9423.4(12) | 74.8(6) |
| F(5) | 3765.7(14) | 4556.1(12) | 5737.4(14) | 80.8(6) |
| C(78) | 703.9(16) | 9494.2(12) | 4080.2(12) | 18.9(4) |
| F(7) | 3167.9(16) | 3548.1(14) | 6760.0(12) | 89.7(7) |
| C(89) | 796.5(16) | 8757.7(12) | 4683.8(12) | 18.5(4) |
| C(81) | 1627.1(16) | 8505.1(13) | 5017.1(13) | 21.4(4) |
| C(61) | 2588.7(17) | 6962.2(13) | 3681.8(13) | 24.8(5) |
| C(62) | 2546.8(16) | 7690.5(12) | 3062.0(13) | 21.4(4) |
| C(86) | 76.8(16) | 8268.3(12) | 4943.3(13) | 21.9(4) |
| C(73) | -121.4(17) | 9745.1(12) | 3737.9(13) | 22.9(5) |
| C(22) | 8112.4(17) | 1951.7(13) | 1933.0(13) | 23.2(5) |
| C(65) | 1017.6(17) | 7486.2(13) | 2922.5(14) | 26.1(5) |
| C(70) | 1757.7(17) | 7953.0(13) | 2683.2(13) | 23.1(5) |
| C(77) | 1429.3(17) | 9980.5(13) | 3816.7(13) | 22.4(4) |
| C(10) | 1978.0(17) | 3134.8(13) | 2992.3(14) | 24.1(5) |
| C(25) | 6527.4(17) | 2131.8(14) | 3248.3(14) | 26.5(5) |
| C(26) | 7365.7(17) | 2441.0(13) | 2622.4(13) | 22.1(4) |
| C(6) | 2654.3(17) | 2400.6(13) | 2562.7(13) | 23.8(5) |
| C(46) | 3725.5(17) | 6978.0(13) | 1034.9(13) | 24.6(5) |
| C(50) | 3817.5(17) | 6260.4(12) | 1659.2(13) | 23.3(5) |
| C(83) | 1724.0(18) | 7763.4(14) | 5597.4(14) | 27.0(5) |
| C(57) | 3296.7(18) | 8151.2(14) | 2811.6(14) | 28.2(5) |
| C(54) | 3105.3(17) | 5759.6(13) | 1922.8(14) | 26.0(5) |
| C(87) | -752.1(17) | 8541.7(13) | 4599.1(14) | 26.8(5) |
| F(4) | 2405.3(19) | 4798.9(13) | 5307.9(14) | 98.9(7) |
| C(1) | 3441.5(17) | 1815.0(14) | 2882.9(15) | 29.0(5) |
| C(21) | 8014.6(18) | 1156.1(13) | 1873.2(15) | 27.4(5) |
| C(69) | 1720.3(18) | 8676.6(13) | 2058.9(14) | 28.4(5) |
| C(79) | 2246.2(17) | 9716.7(14) | 4169.5(14) | 26.7(5) |
| C(80) | 2345.8(17) | 9005.0(13) | 4741.7(14) | 25.1(5) |
| C(5) | 2546.2(18) | 2250.3(14) | 1818.6(14) | 27.6(5) |

| | | | | |
|---|---|---|---|---|
| C(9) | 1186.5(18) | 3714.4(13) | 2679.7(15) | 28.8(5) |
| C(85) | 213.1(17) | 7524.5(13) | 5525.3(14) | 26.7(5) |
| C(49) | 4606.8(18) | 6052.5(14) | 2028.0(14) | 29.0(5) |
| C(30) | 7451.2(18) | 3246.8(13) | 2674.7(14) | 27.0(5) |
| C(14) | 2079.4(18) | 3276.9(15) | 3746.6(15) | 30.6(5) |
| C(76) | 1323.2(19) | 10701.8(13) | 3210.7(14) | 30.0(5) |
| C(41) | 2921.7(19) | 7192.1(14) | 680.6(14) | 30.3(5) |
| C(24) | 6476.6(19) | 1308.7(14) | 3178.6(16) | 33.0(5) |
| C(63) | 1833.0(18) | 6514.4(13) | 3916.8(14) | 28.9(5) |
| C(74) | -189.2(19) | 10472.0(14) | 3133.3(15) | 31.2(5) |
| C(17) | 8955.5(18) | 2261.8(14) | 1308.6(14) | 30.6(5) |
| C(88) | -843.7(17) | 9249.8(14) | 4024.5(15) | 28.0(5) |
| C(84) | 1022.1(18) | 7283.1(13) | 5850.0(14) | 28.9(5) |
| C(64) | 1073.4(18) | 6768.0(14) | 3549.5(15) | 30.5(5) |
| C(29) | 6688(2) | 3724.8(14) | 3338.3(16) | 33.1(6) |
| C(45) | 4428.7(19) | 7482.0(14) | 781.5(15) | 31.8(5) |
| C(75) | 525(2) | 10942.9(14) | 2872.5(15) | 33.8(6) |
| C(7) | 1742.3(19) | 2852.8(15) | 1514.3(15) | 34.2(6) |
| C(60) | 3392.5(19) | 6713.2(15) | 4040.7(15) | 33.8(6) |
| C(28) | 5862.6(19) | 3424.2(15) | 3934.8(15) | 34.7(6) |
| C(71) | 2470(2) | 9132.0(14) | 1830.4(15) | 35.6(6) |
| C(55) | 2327.2(18) | 5984.4(15) | 1542.7(15) | 33.0(5) |
| C(4) | 3227(2) | 1523.8(15) | 1406.4(15) | 35.9(6) |
| C(27) | 5780.7(18) | 2638.9(15) | 3897.6(15) | 32.7(5) |
| C(56) | 2234.6(19) | 6677.8(16) | 944.5(15) | 34.0(6) |
| C(23) | 7174(2) | 855.7(14) | 2523.2(16) | 33.5(6) |
| C(20) | 8755(2) | 692.1(15) | 1187.5(16) | 36.1(6) |
| C(72) | 3229(2) | 8883.5(15) | 2191.8(15) | 34.8(6) |
| C(13) | 1379(2) | 3986.2(16) | 4174.4(16) | 39.9(6) |
| C(53) | 3201(2) | 5059.9(14) | 2557.7(16) | 36.0(6) |
| C(8) | 1103.9(19) | 3547.3(15) | 1922.1(16) | 35.0(6) |
| C(15) | 2876(2) | 2670.7(17) | 4052.6(16) | 39.3(6) |
| C(16) | 3514(2) | 1981.5(17) | 3645.5(16) | 38.9(6) |

| | | | | |
|---|---|---|---|---|
| C(68) | 936(2) | 8910.6(16) | 1681.4(16) | 41.2(7) |
| C(66) | 251.9(19) | 7753.8(16) | 2523.9(16) | 37.9(6) |
| C(11) | 507(2) | 4413.1(15) | 3136.1(17) | 38.8(6) |
| C(48) | 5311.2(19) | 6560.0(16) | 1746.9(16) | 36.6(6) |
| C(58) | 4090.0(19) | 7865.4(16) | 3184.7(16) | 36.9(6) |
| C(32) | 9021(2) | 3068.5(16) | 1392.7(16) | 40.5(6) |
| C(59) | 4129.5(19) | 7157.9(17) | 3791.2(16) | 39.2(6) |
| C(3) | 4000(2) | 966.1(15) | 1719.5(16) | 39.6(6) |
| C(2) | 4104.5(19) | 1104.0(15) | 2441.8(16) | 37.8(6) |
| C(19) | 9581(2) | 997.1(16) | 589.9(16) | 42.4(7) |
| C(51) | 4661(2) | 5349.7(15) | 2667.2(16) | 39.4(6) |
| C(18) | 9683(2) | 1765.3(17) | 643.9(16) | 41.7(6) |
| C(31) | 8315(2) | 3532.4(15) | 2038.7(16) | 38.0(6) |
| C(47) | 5225.6(19) | 7250.6(16) | 1141.9(17) | 38.8(6) |
| C(52) | 3961(2) | 4863.3(15) | 2921.9(17) | 43.2(7) |
| C(12) | 599(2) | 4535.3(16) | 3870.4(17) | 44.2(7) |
| C(95) | 4100(5) | 5564(4) | 9362(5) | 25.5(13) |
| C(67) | 219(2) | 8454.3(17) | 1912.3(17) | 44.2(7) |
| C(42) | 2836(2) | 7921.0(16) | 70.6(15) | 42.8(7) |
| C(44) | 4284(2) | 8217.6(16) | 181.2(16) | 45.7(7) |
| C(96) | 4924(3) | 5362(2) | 9695(2) | 20.2(9) |
| C(43) | 3512(2) | 8419.0(17) | -174.0(17) | 50.8(8) |
| C(99) | 3801(2) | 1464.0(18) | 6395.5(18) | 45.8(7) |
| C(91) | 5619(3) | 5892(3) | 9406(3) | 24.4(9) |
| C(98) | 1324(2) | 3658.7(19) | 8681(2) | 54.9(8) |
| C(90) | 6442(4) | 5671(3) | 9753(3) | 30.7(11) |
| C(97) | 3421(3) | 5014(3) | 9669(3) | 31.9(10) |
| C(93) | 4635(3) | 6787(2) | 8496(2) | 39.1(10) |
| C(33) | 7128(7) | 4645(5) | 678(6) | 35(2) |
| Cl(4A) | 1057(8) | 4356(4) | 9471.8(18) | 102(2) |
| Cl(3B) | -77(3) | 4013(3) | 8929(3) | 70.8(11) |
| C(92) | 5445(4) | 6607(3) | 8813(2) | 37.0(10) |
| C(94) | 3971(3) | 6275(2) | 8769(2) | 37.7(10) |

| | | | | |
|---|---|---|---|---|
| C(37) | 5416(8) | 4946(6) | 135(6) | 22(2) |
| C(39) | 6955(8) | 5315(6) | 37(6) | 28(2) |
| C(36) | 4900(7) | 3726(6) | 1155(5) | 25(2) |
| C(34) | 6467(7) | 4134(5) | 1040(5) | 30(2) |
| C(40) | 5902(9) | 6150(7) | -900(7) | 27(2) |
| C(38) | 6102(8) | 5472(6) | -246(6) | 21(2) |
| C(35) | 5595(13) | 4262(11) | 788(11) | 28(3) |

# 2 Tabellenverzeichnis