# Crystal structure of (acridine)dichloro(dimethyl sulfoxide-$\kappa S$)-palladium(II) — dimethyl sulfoxide (1:2), $PdCl_2(C_{13}H_9N)(C_2H_6SO) \cdot 2C_2H_6SO$

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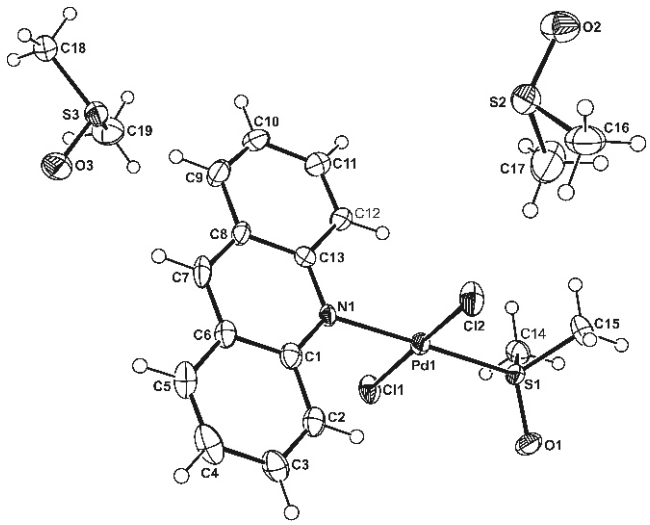


**Abstract**

$C_{19}H_{27}Cl_2NO_3PdS_3$, triclinic, $P\bar{1}$ (no. 2), $a$ = 9.6245(6) Å, $b$ = 10.9445(6) Å, $c$ = 12.7679(7) Å, $\alpha$ = 101.015(1)°, $\beta$ = 111.510(1)°, $\gamma$ = 93.545(1)°, $V$ = 1215.3 Å$^3$, $Z$ = 2, $R_{gt}(F)$ = 0.046, $wR_{ref}(F^2)$ = 0.107, $T$ = 200 K.

**Source of material**

To a solution of $Na_2PdCl_4$ (0.2014 g, 0.685 mmol) in $H_2O$ (20 ml) was added acridine (acr, 0.2561 g, 1.429 mmol), and the mixture was refluxed for 7 h. The precipitate was then separated by filtration, washed with acetone and pentane, and dried at 50 °C, to give a yellow powder (0.3369 g). Crystals suitable for X-ray diffraction analysis were obtained by slow evaporation from a dimethyl sulfoxide (DMSO) solution at 90 °C.

**Experimental details**

Hydrogen atoms were positioned geometrically and allowed to ride on their parent atoms with $d$(C—H) = 0.95 Å (CH) or 0.98 Å ($CH_3$) and $U_{iso}$(H) = 1.2 $U_{eq}$(C) or 1.5 $U_{eq}(C_{methyl})$. The highest peak (0.87 e Å$^{-3}$) and the deepest hole (–0.83 e Å$^{-3}$) in the difference Fourier map are located 1.11 Å and 0.83 Å from the Pd1 atom, respectively.

**Discussion**

Single crystals of the title compound were obtained from a dimethyl sulfoxide solution of $[PdCl_2(acr)_2]$. It seems that an acridine ligand of the complex $[PdCl_2(acr)_2]$ was replaced by a DMSO molecule during crystallization, whereas the analogous Pt complex $[PtCl_2(acr)_2]$ crystallized without substitution from a DMSO solution at 80 °C [1]. It was previously reported that single crystals of the complex $[PdCl_2(acr)(CH_3)_2NH]$ were acquired from an *N*,*N*-dimethylformamide solution of the Pd complex $[PdCl_2(acr)_2]$ at 50 °C [2].

The title compound consists of a neutral Pd(II) complex and two DMSO solvent molecules. In the complex, the Pd(II) ion is four-coordinated in a slightly distorted square-planar environment by one N atom from an acridine ligand, one S atom of the DMSO molecule and two chloride ions. The Cl atoms are in *trans* conformation with respect to each other (∠Cl1–Pd1–Cl2 = 176.46(6)°) and almost perpendicular to the nearly planar acridine ligand, with the bond angles ∠N1–Pd1–Cl1 = 88.6(1)° and ∠N1–Pd1–Cl2 = 89.7(1)°. In the crystal structure, the complex and the solvent molecules are linked by C–H···O and C–H···Cl hydrogen bonds with $d$(C···O) = 3.114(7) - 3.504(8) Å and $d$(C···Cl) = 3.170(6) - 3.666(6) Å, forming a three-dimensional network structure. In addition, several intermolecular $\pi$-$\pi$ interactions between adjacent six-membered rings are present. The shortest distance between Cg1 (the centroid of ring N1-C13) and $Cg1^{i}$ (symmetry code i: 1–$x$,1–$y$,1–$z$) is 3.769(3) Å; the ring planes are parallel and shifted by 1.400 Å.

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

| | |
|---|---|
| Crystal: | yellow block, size 0.06 × 0.12 × 0.24 mm |
| Wavelength: | Mo $K_\alpha$ radiation (0.71073 Å) |
| $\mu$: | 12.61 cm$^{-1}$ |
| Diffractometer, scan mode: | Bruker SMART 1000 CCD, $\varphi/\omega$ |
| $2\theta_{max}$: | 51.96° |
| $N(hkl)_{measured}$, $N(hkl)_{unique}$: | 7673, 4694 |
| Criterion for $I_{obs}$, $N(hkl)_{gt}$: | $I_{obs} > 2\ \sigma(I_{obs})$, 3402 |
| $N(param)_{refined}$: | 268 |
| Programs: | SHELXS-97, SHELXL-97 [3], ORTEP-3 [4], PLATON [5] |

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

| Atom | Site | $x$ | $y$ | $z$ | $U_{iso}$ |
|---|---|---|---|---|---|
| H(2) | 2*i* | 0.0067 | 0.4407 | 0.3680 | 0.041 |
| H(3) | 2*i* | –0.0336 | 0.6336 | 0.4519 | 0.049 |
| H(4) | 2*i* | 0.1565 | 0.8100 | 0.5198 | 0.058 |
| H(5) | 2*i* | 0.3744 | 0.7949 | 0.4883 | 0.051 |
| H(7) | 2*i* | 0.5326 | 0.6643 | 0.4087 | 0.043 |
| H(9) | 2*i* | 0.6816 | 0.5325 | 0.3191 | 0.050 |
| H(10) | 2*i* | 0.7087 | 0.3399 | 0.2270 | 0.054 |
| H(11) | 2*i* | 0.5189 | 0.1651 | 0.1749 | 0.053 |
| H(12) | 2*i* | 0.3143 | 0.1838 | 0.2246 | 0.044 |

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**Table 2.** Continued.

| Atom | Site | $x$ | $y$ | $z$ | $U_{iso}$ |
|---|---|---|---|---|---|
| H(14A) | 2*i* | –0.1824 | –0.1167 | 0.0668 | 0.052 |
| H(14B) | 2*i* | –0.0132 | –0.0629 | 0.0886 | 0.052 |
| H(14C) | 2*i* | –0.1499 | 0.0084 | 0.0270 | 0.052 |
| H(15A) | 2*i* | –0.0135 | –0.0200 | 0.3882 | 0.062 |
| H(15B) | 2*i* | 0.0667 | –0.0819 | 0.3054 | 0.062 |
| H(15C) | 2*i* | –0.1053 | –0.1327 | 0.2785 | 0.062 |
| H(16A) | 2*i* | 0.5147 | 0.0638 | 0.9100 | 0.083 |
| H(16B) | 2*i* | 0.4917 | 0.2080 | 0.9150 | 0.083 |
| H(16C) | 2*i* | 0.6459 | 0.1731 | 0.9993 | 0.083 |

**Table 2.** Continued.

| Atom | Site | $x$ | $y$ | $z$ | $U_{iso}$ |
|---|---|---|---|---|---|
| H(17A) | 2*i* | 0.4961 | 0.0758 | 0.6227 | 0.122 |
| H(17B) | 2*i* | 0.4014 | 0.1495 | 0.6878 | 0.122 |
| H(17C) | 2*i* | 0.4310 | 0.0096 | 0.7000 | 0.122 |
| H(18A) | 2*i* | 0.9703 | 0.6653 | 0.0754 | 0.059 |
| H(18B) | 2*i* | 1.0249 | 0.5442 | 0.1231 | 0.059 |
| H(18C) | 2*i* | 1.0354 | 0.6729 | 0.2116 | 0.059 |
| H(19A) | 2*i* | 0.5999 | 0.4794 | –0.0505 | 0.084 |
| H(19B) | 2*i* | 0.7574 | 0.4373 | –0.0476 | 0.084 |
| H(19C) | 2*i* | 0.7198 | 0.5736 | –0.0673 | 0.084 |

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

| Atom | Site | $x$ | $y$ | $z$ | $U_{11}$ | $U_{22}$ | $U_{33}$ | $U_{12}$ | $U_{13}$ | $U_{23}$ |
|---|---|---|---|---|---|---|---|---|---|---|
| Pd(1) | 2*i* | 0.08368(5) | 0.22413(4) | 0.27753(4) | 0.0232(2) | 0.0232(2) | 0.0285(2) | 0.0003(2) | 0.0079(2) | 0.0075(2) |
| Cl(1) | 2*i* | –0.0355(2) | 0.2701(1) | 0.1008(1) | 0.0394(9) | 0.0332(8) | 0.0284(8) | –0.0013(6) | 0.0034(7) | 0.0133(7) |
| Cl(2) | 2*i* | 0.1996(2) | 0.1897(1) | 0.4595(1) | 0.043(1) | 0.0408(9) | 0.0322(9) | –0.0092(7) | –0.0018(7) | 0.0159(7) |
| S(1) | 2*i* | –0.0891(2) | 0.0516(1) | 0.2224(1) | 0.0227(7) | 0.0203(7) | 0.0341(8) | 0.0015(5) | 0.0125(6) | 0.0056(6) |
| O(1) | 2*i* | –0.2373(4) | 0.0796(3) | 0.2205(4) | 0.030(2) | 0.031(2) | 0.066(3) | 0.001(2) | 0.027(2) | 0.001(2) |
| N(1) | 2*i* | 0.2408(5) | 0.3849(4) | 0.3231(4) | 0.024(2) | 0.014(2) | 0.026(2) | –0.001(2) | 0.009(2) | 0.005(2) |
| C(1) | 2*i* | 0.2172(6) | 0.4975(5) | 0.3739(5) | 0.033(3) | 0.030(3) | 0.027(3) | 0.003(3) | 0.009(3) | 0.012(3) |
| C(2) | 2*i* | 0.0823(7) | 0.5116(5) | 0.3917(5) | 0.039(4) | 0.033(3) | 0.030(3) | –0.001(3) | 0.012(3) | 0.013(3) |
| C(3) | 2*i* | 0.0590(8) | 0.6254(5) | 0.4424(5) | 0.056(4) | 0.025(3) | 0.040(4) | 0.006(3) | 0.021(3) | 0.000(3) |
| C(4) | 2*i* | 0.1719(9) | 0.7319(6) | 0.4812(5) | 0.092(6) | 0.027(3) | 0.032(4) | 0.012(4) | 0.033(4) | 0.001(3) |
| C(5) | 2*i* | 0.3006(8) | 0.7225(5) | 0.4635(5) | 0.060(5) | 0.029(3) | 0.038(4) | –0.007(3) | 0.020(4) | 0.006(3) |
| C(6) | 2*i* | 0.3287(6) | 0.6055(5) | 0.4080(5) | 0.031(3) | 0.026(3) | 0.023(3) | –0.003(2) | 0.004(3) | 0.006(2) |
| C(7) | 2*i* | 0.4572(7) | 0.5932(5) | 0.3855(5) | 0.039(4) | 0.029(3) | 0.030(3) | –0.010(3) | 0.002(3) | 0.016(3) |
| C(8) | 2*i* | 0.4781(6) | 0.4790(5) | 0.3297(5) | 0.021(3) | 0.029(3) | 0.034(3) | –0.005(2) | 0.002(3) | 0.014(3) |
| C(9) | 2*i* | 0.6063(7) | 0.4625(6) | 0.2999(6) | 0.031(4) | 0.045(4) | 0.050(4) | –0.003(3) | 0.015(3) | 0.020(3) |
| C(10) | 2*i* | 0.6223(7) | 0.3495(6) | 0.2453(6) | 0.028(4) | 0.055(4) | 0.065(5) | 0.011(3) | 0.024(3) | 0.028(4) |
| C(11) | 2*i* | 0.5087(7) | 0.2443(6) | 0.2151(6) | 0.033(4) | 0.038(4) | 0.062(5) | 0.006(3) | 0.019(3) | 0.012(3) |
| C(12) | 2*i* | 0.3866(6) | 0.2559(5) | 0.2431(5) | 0.026(3) | 0.027(3) | 0.056(4) | 0.000(3) | 0.014(3) | 0.008(3) |
| C(13) | 2*i* | 0.3650(6) | 0.3718(5) | 0.2987(5) | 0.025(3) | 0.027(3) | 0.034(3) | 0.007(2) | 0.009(3) | 0.013(3) |
| C(14) | 2*i* | –0.1111(7) | –0.0401(5) | 0.0860(5) | 0.037(4) | 0.026(3) | 0.036(4) | 0.004(3) | 0.011(3) | 0.002(3) |
| C(15) | 2*i* | –0.0286(8) | –0.0579(5) | 0.3081(5) | 0.056(4) | 0.029(3) | 0.033(4) | 0.007(3) | 0.008(3) | 0.014(3) |
| S(2) | 2*i* | 0.6435(2) | 0.1514(2) | 0.8159(2) | 0.035(1) | 0.0388(9) | 0.073(1) | 0.0015(8) | 0.0121(9) | 0.0190(9) |
| O(2) | 2*i* | 0.7437(5) | 0.0518(4) | 0.8228(4) | 0.044(3) | 0.047(3) | 0.057(3) | 0.010(2) | 0.013(2) | –0.002(2) |
| C(16) | 2*i* | 0.5652(8) | 0.1488(6) | 0.9220(6) | 0.035(4) | 0.049(4) | 0.070(5) | 0.010(3) | 0.010(4) | 0.006(4) |
| C(17) | 2*i* | 0.4740(9) | 0.0897(8) | 0.6928(7) | 0.053(5) | 0.110(7) | 0.072(6) | –0.009(5) | 0.000(4) | 0.057(6) |
| S(3) | 2*i* | 0.7909(2) | 0.5714(1) | 0.1256(2) | 0.0387(9) | 0.0323(8) | 0.046(1) | 0.0063(7) | 0.0206(8) | 0.0101(7) |
| O(3) | 2*i* | 0.7243(5) | 0.6884(4) | 0.1475(4) | 0.051(3) | 0.042(3) | 0.074(4) | 0.014(2) | 0.030(3) | 0.003(3) |
| C(18) | 2*i* | 0.9762(7) | 0.6188(5) | 0.1350(5) | 0.040(4) | 0.034(3) | 0.044(4) | 0.004(3) | 0.015(3) | 0.012(3) |
| C(19) | 2*i* | 0.7076(8) | 0.5083(7) | –0.0272(6) | 0.047(4) | 0.061(5) | 0.046(4) | 0.008(4) | 0.008(4) | 0.003(4) |

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## References

1. Ha, K.: Crystal structure of dichlorobis(acridine)platinum(II), $PtCl_2(C_{13}H_9N)_2$. Z. Kristallogr. NCS **225** (2010) 323-324.
2. Ha, K.: Crystal structure of dichloroacridinedimethylaminepalladium(II), $PdCl_2(C_{13}H_9N)(C_2H_7N)$. Z. Kristallogr. NCS **225** (2010) 655-656.
3. Sheldrick, G. M.: A short history of SHELX. Acta Crystallogr. **A64** (2008) 112-122.
4. Farrugia, L. J.: ORTEP-3 for Windows - a version of ORTEP-III with a Graphical User Interface (GUI) J. Appl. Crystallogr. **30** (1997) 565.
5. Spek, A. L.: Single-crystal structure validation with the program PLATON. J. Appl. Crystallogr. **36** (2003) 7-13.