

This article was downloaded by:

On: 23 January 2011

Access details: *Access Details: Free Access*

Publisher *Taylor & Francis*

Informa Ltd Registered in England and Wales Registered Number: 1072954 Registered office: Mortimer House, 37-41 Mortimer Street, London W1T 3JH, UK



Journal of Coordination Chemistry

Publication details, including instructions for authors and subscription information:

<http://www.informaworld.com/smpp/title~content=t713455674>

The Crystal and Molecular Structure of Bis[1,2-Bis(Diphenylphosphino)Ethane] Dichlororuthenium(II)

Tarlok S. Lobana^a; Randhir Singh^a; Edward R. T. Tiekink^b

^a Department of Chemistry, Guru Nanak Dev University, Amritsar, India ^b Jordan Laboratories, Department of Physical and Inorganic Chemistry, University of Adelaide, Adelaide, South Australia, Australia

To cite this Article Lobana, Tarlok S. , Singh, Randhir and Tiekink, Edward R. T.(1990) 'The Crystal and Molecular Structure of Bis[1,2-Bis(Diphenylphosphino)Ethane] Dichlororuthenium(II)', *Journal of Coordination Chemistry*, 21: 3, 225 – 229

To link to this Article: DOI: 10.1080/00958979009409719

URL: <http://dx.doi.org/10.1080/00958979009409719>

PLEASE SCROLL DOWN FOR ARTICLE

Full terms and conditions of use: <http://www.informaworld.com/terms-and-conditions-of-access.pdf>

This article may be used for research, teaching and private study purposes. Any substantial or systematic reproduction, re-distribution, re-selling, loan or sub-licensing, systematic supply or distribution in any form to anyone is expressly forbidden.

The publisher does not give any warranty express or implied or make any representation that the contents will be complete or accurate or up to date. The accuracy of any instructions, formulae and drug doses should be independently verified with primary sources. The publisher shall not be liable for any loss, actions, claims, proceedings, demand or costs or damages whatsoever or howsoever caused arising directly or indirectly in connection with or arising out of the use of this material.

THE CRYSTAL AND MOLECULAR STRUCTURE OF BIS[1,2-BIS(DIPHENYLPHOSPHINO)ETHANE] DICHLORORUTHENIUM(II)

TARLOK S. LOBANA, RANDHIR SINGH

Department of Chemistry, Guru Nanak Dev University, Amritsar-143005, India

and EDWARD R. T. TIEKINK

*Jordan Laboratories, Department of Physical and Inorganic Chemistry, University of Adelaide, Adelaide,
South Australia, 5001, Australia*

(Received November 25, 1989; in final form December 28, 1989)

The title compound, *trans*-[Ru(dppe)₂Cl₂], has $\bar{1}$ symmetry such that the chloride atoms are exactly *trans*. The coordination geometry is distorted octahedral owing, in part, to the restricted bite distance of the chelating dppe ligands. Important interatomic parameters are Ru-Cl 2.436(1), Ru-P(1), 2.389(1) Ru-P(2) 2.369(1) Å and P(1)-Ru-P(2) 82.1(1)°. The compound crystallizes as a dichloromethane solvate in the monoclinic space group *C2/c* with unit cell dimensions *a* = 27.995(3), *b* = 13.85(1), *c* = 13.265(3) Å, β = 105.38(2)° and *Z* = 4. The structure was refined by a full-matrix least-squares procedure to final *R* = 0.038 for 3522 reflections with $I \geq 2.5\sigma(I)$.

Keywords: Ruthenium(II), diphosphine, X-ray structure.

INTRODUCTION

The crystal structure of the title compound, *trans*-[Ru(dppe)₂Cl₂], was determined as a part of a continuing investigation of metal phosphine complexes.¹⁻³ Particular interest in the present system arises as a result of our study of the photochemistry of derivatives based on *trans*-[Ru(dppe)₂Cl₂]. Further interest in these compounds is related to their potential use as catalysts; for example, *trans*-[Ru(dppe)₂Cl₂] has been shown to catalytically hydrogenate olefins.⁴

EXPERIMENTAL

Crystallography

Trans-[Ru(dppe)₂Cl₂] is a known compound⁵ and crystals suitable for the X-ray analysis were grown as a CH₂Cl₂ solvate by the slow evaporation of a solution of the compound. Intensity data for 5693 reflections were measured on an Enraf-Nonius CAD4F diffractometer with the use of graphite monochromatized MoK α radiation and the ω : 2θ scan technique such that θ_{\max} was 25°. The data were corrected for Lorentz and polarization effects and for absorption.⁶ There were 4390 unique reflections (*R*_{amal} 0.019) and of these 3522 satisfied the $I \geq 2.5\sigma(I)$ criterion of observability. Crystal data and refinement details are given in Table I.

TABLE I
Crystal and refinement details for *trans*-[Ru(dppe)₂Cl₂].CH₂Cl₂.

Formula	C ₅₃ H ₅₀ Cl ₄ P ₄ Ru
<i>M_r</i>	1053.7
Crystal system	monoclinic
Space group	<i>C2/c</i>
<i>a</i> , Å	27.995(3)
<i>b</i> , Å	13.85(1)
<i>c</i> , Å	13.265(3)
β(°)	105.38(4)
Vol. (Å ³)	4959(2)
<i>Z</i>	4
<i>D_c</i> (g cm ⁻³)	1.411
<i>F</i> (000)	2328
μ, cm ⁻¹	7.44
Transmission factors (max./min.)	0.964; 0.785
θ limits (°)	1.5–25.0
No. of data collected	5693
No. of unique data	4390
No. of unique reflections used with <i>I</i> ≥ 2.5σ(<i>I</i>)	3522
<i>R</i>	0.038
<i>g</i>	0.004
<i>R_w</i>	0.043
ρ _{max} (e Å ⁻³)	0.76

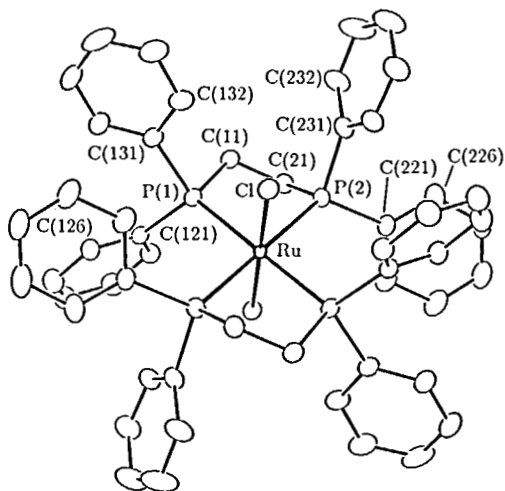


FIGURE 1 Molecular structure and crystallographic numbering scheme for *trans*-[Ru(dppe)₂Cl₂].

The structure was solved from the interpretation of the Patterson synthesis and refined by a full-matrix least-squares procedure based on *F*.⁶ Non-hydrogen atoms were refined with anisotropic thermal parameters and hydrogen atoms were included in the model at their calculated positions. The solvent CH₂Cl₂ molecule was found to

be disordered about a crystallographic 2-fold axis and was refined with three sites for the chloride atoms each with a fractional occupancy factor. Full details are given in Table S(5) of the supplementary material. Refinement with a weighting scheme of the form $w = [\sigma^2(F) + g(F)^2]^{-1}$ converged with final R 0.038, R_w 0.043, and g 0.004.

TABLE II
Fractional atomic coordinates ($\times 10^5$ for Ru, P, Cl; $\times 10^4$ others) for trans-[Ru(dppe)₂Cl₂].

Atom	x/a	y/b	z/c
Ru	25000(-)	25000(-)	50000(-)
P(1)	18105(3)	33012(6)	38559(6)
P(2)	29487(3)	34740(6)	41012(7)
Cl	24965(3)	10996(6)	38870(6)
C(11)	2052(1)	4302(3)	3184(3)
C(21)	2581(1)	4584(2)	3745(3)
C(121)	1381(1)	3901(2)	4487(3)
C(122)	1466(1)	4845(3)	4856(3)
C(123)	1153(2)	5278(3)	5388(3)
C(124)	762(2)	4777(4)	5546(4)
C(125)	671(2)	3844(4)	5178(4)
C(126)	979(1)	3399(3)	4665(3)
C(131)	1392(1)	2741(3)	2697(3)
C(132)	1600(2)	2116(3)	2101(3)
C(133)	1300(2)	1683(3)	1215(3)
C(134)	807(2)	1866(4)	892(4)
C(135)	597(2)	2503(4)	1451(4)
C(136)	890(2)	2942(3)	2338(3)
C(221)	3566(1)	3927(2)	4756(3)
C(222)	3661(2)	4236(3)	5793(3)
C(223)	4144(2)	4495(4)	6342(4)
C(224)	4519(2)	4475(4)	5885(4)
C(225)	4433(2)	4184(4)	4873(5)
C(226)	3960(2)	3924(3)	4298(4)
C(231)	3038(1)	3058(3)	2845(3)
C(232)	2906(2)	3607(3)	1940(3)
C(233)	3012(3)	3283(4)	1043(4)
C(234)	3266(3)	2420(3)	1033(4)
C(235)	3398(2)	1877(3)	1930(3)
C(236)	3277(1)	2179(3)	2824(3)

Fractional atomic coordinates are listed in Table II and the numbering scheme employed is shown in Figure 1 (drawn at 25% probability ellipsoids with ORTEP⁷). The refinement was performed with the SHELX76⁹ program system installed on a SUN 4/280 computer system. Scattering factors for Ru (corrected for f' and f'') were the *International Tables for X-ray Crystallography*⁸ and those for the remaining atoms were those incorporated in SHELX76.⁶ Listings of thermal parameters, hydrogen atom parameters, all bond distances and angles, details of the disordered CH₂Cl₂ molecule and the observed and calculated structure factors are available from the Editor.

RESULTS AND DISCUSSION

The crystal structure determination confirms the stoichiometry of the compound and the *trans* disposition of the chloride atoms. A diagram of the molecular structure is shown in Figure 1 and selected interatomic parameters are given in Table III. The ruthenium atom is situated on a crystallographic centre of inversion (located at 1/4, 1/4, 1/2) so that half the molecule comprises the asymmetric unit. In addition there is disordered CH_2Cl_2 in the crystal lattice as discussed in the Experimental section.

The ruthenium atom exists in a distorted octahedral geometry with the maximum deviation from the ideal geometry arising as a result of the restricted bite distance of the diphosphine ligand; $\text{P}(1)\text{-Ru-P}(2)$ $82.1(1)^\circ$. The Ru-Cl bond distance of $2.436(1)$ Å lies within the narrow range of Ru-Cl bond distances found in related *trans*-ruthenium(diphosphine) $_2\text{Cl}_2$ complexes. Thus Ru-Cl distances of $2 \times 2.426(1)$ Å (molecule has $\bar{1}$ symmetry) has been reported for *trans*-[Ru(dppm) $_2\text{Cl}_2$]⁹ [dppm is bis(diphenylphosphino)methane] and Ru-Cl distances of $2.443(4)$ and $2.446(4)$ Å were found in *trans*-[Ru(PhMePCH $_2\text{CH}_2\text{PPhMe}$) $_2\text{Cl}_2$].¹⁰ The Ru-Cl distances in *trans*-[Ru(dppe) $_2\text{Cl}_2$] may also be compared to the Ru-Cl distances found in *trans*-[Ru(PHMe $_2$) $_4\text{Cl}_2$] of $2 \times 2.440(1)$ Å (molecule has $\bar{1}$ symmetry).¹¹

TABLE III
Selected bond distances (Å) and angles ($^\circ$) for *trans*-[Ru(dppe) $_2\text{Cl}_2$].

<i>Distances</i>			
Ru-Cl	2.436(1)	Ru-P(1)	2.389(1)
Ru-P(2)	2.369(1)	P(1)-C(11)	1.868(3)
P(1)-C(121)	1.836(3)	P(1)-C(131)	1.839(4)
P(2)-C(21)	1.841(3)	P(2)-C(221)	1.827(3)
P(2)-C(231)	1.841(4)	C(11)-C(21)	1.522(5)
<i>Angles</i>			
Cl-Ru-P(1)	96.4(1)	Cl-Ru-P(2)	93.8(1)
Cl-Ru-P(1')	83.6(1)	Cl-Ru-P(2')	86.2(1)
P(1)-Ru-P(2)	82.1(1)	P(1)-Ru-P(2')	97.9(1)
Ru-P(1)-C(11)	108.3(1)	Ru-P(1)-C(121)	115.9(1)
Ru-P(1)-C(131)	124.3(1)	C(11)-P(1)-C(121)	104.2(2)
C(11)-P(1)-C(131)	98.3(2)	C(121)-P(1)-C(131)	102.9(2)
Ru-P(2)-C(21)	105.7(1)	Ru-P(2)-C(221)	121.1(1)
Ru-P(2)-C(231)	119.6(1)	C(21)-P(1)-C(221)	103.2(1)
C(21)-P(1)-C(231)	103.3(2)	C(221)-P(1)-C(231)	101.5(2)
P(1)-C(11)-C(21)	112.7(2)	P(2)-C(21)-C(11)	108.4(2)

The dppe ligands in *trans*-[Ru(dppe) $_2\text{Cl}_2$] coordinate in a bidentate fashion, there being a slight asymmetry in the Ru-P bond distances: Ru-P(1) $2.389(1)$ and $2.369(1)$ Å. The Ru-P(1) distance is significantly longer than other Ru-P bonds in the complexes cited above whereas the Ru-P(2) bond distance, while at the upper limit of Ru-P bond distances, has a precedent. In *trans*-[Ru(dppm) $_2\text{Cl}_2$]⁹ the Ru-P bond distances are $2.340(1)$ and $2.367(1)$ Å and in *trans*-[Ru(PhMePCH $_2\text{CH}_2\text{PPhMe}$) $_2\text{Cl}_2$] the Ru-P bonds were found to be $2.319(4)$, $2 \times 2.346(4)$ and $2.361(4)$ Å.¹⁰ In the *trans*-[Ru(PH $_2\text{Me}$) $_4\text{Cl}_2$] compound the two unique Ru-P bond distances are at the lower end of the range at $2.323(1)$ and $2.331(1)$ Å.¹¹ Bond lengths and angles associated with

the dppe ligand show no abnormal features. In conclusion there does not appear to be any systematic variation in the Ru–Cl and Ru–P bond distances as the nature of the diphosphine is varied; any differences that do occur in the ruthenium atom geometry may be related to the bite distance of the diphosphine.

ACKNOWLEDGEMENTS

Support from the Australian Research Council is acknowledged. Financial assistance from D.A.E., Bombay and laboratory facilities from the Guru Nanak Dev University are acknowledged.

REFERENCES

1. T.S. Lobana, M.K. Sandhu and E.R.T. Tiekink, *J. Chem. Soc., Dalton Trans.*, 1401 (1988).
2. T.S. Lobana, M.K. Sandhu, M.R. Snow and E.R.T. Tiekink, *Acta Cryst. C* **44**, 179 (1988).
3. T.S. Lobana, P.K. Bhatia and E.R.T. Tiekink, *J. Chem. Soc., Dalton Trans.*, 749 (1989).
4. T. Suárez, B. Fontal and D. Garcia, *J. Molec. Catal.*, **34**, 163 (1986).
5. R. Mason, D.W. Meek and G.R. Scollary, *Inorg. Chim. Acta*, **16**, L11 (1976).
6. G.M. Sheldrick, "SHELX76. Program for Crystal Structure Determination", (University of Cambridge, England, 1976).
7. C.K. Johnson, ORTEP, *Report ORNL-3794, Oak Ridge National Laboratory*, Tennessee (1971).
8. W.C. Hamilton and J.A. Ibers, Editors, "International Tables for X-ray Crystallography", Vol. IV, pp. 99, 149 (Birmingham, Kynoch Press, 1974).
9. A.R. Chakravarty, F.A. Cotton and W. Schwotzer, *Inorg. Chim. Acta*, **84**, 179 (1984).
10. R. Wartchow and H.J. Berthold, *Z. Krist.*, **145**, 240 (1977).
11. F.A. Cotton, B.A. Frenz and D.L. Hunter, *Inorg. Chim. Acta*, **16**, 203 (1976).