Interaction of polynuclear palladium (I) carbonyl carboxylates with gaseous NO: X-ray structure of eight-nuclear clusters $Pd_8(\mu\text{-}CO)_4(\mu\text{-}OOCR)_4[\mu\text{-}N(=O)O-]_4$

Oleg N. Shishilov [a,*], Tatiana A. Stromnova [a,‡], Andrei V. Churakov [a], Lyudmila G. Kuz'mina [a], Judith A.K. Howard [b]

[a] N.S. Kurnakov Institute of General and Inorganic Chemistry, Russian Academy of Science, Leninsky pr., 31, Moscow 119991, Russian Federation
[b] Department of Chemistry, University of Durham, South Road, Durham DH1 3LE, UK

ARTICLE INFO

ABSTRACT

The reaction of palladium carbonyl carboxylates $Pd_6(CO)_6(RCOO)_6$ with gaseous nitrogen monoxide was investigated. These clusters were found to promote the NO disproportionation into N_2 and NO_2 under mild conditions. The reaction is accompanied with the oxidation of coordinated carbon monoxide. These processes result in unusual eight-nuclear palladium carboxylate clusters $Pd_8(CO)_4(NO_2)_4(RCOO)_8$.

© 2009 Elsevier B.V. All rights reserved.

1. Introduction

Transformations of nitric monoxide within the coordination sphere of transition metal complexes lead to the formation of species such as NO_2, N_2, and N_2O which are of great importance for biological and environmental processes [1]. The following reaction represents an important way for the purification of exhaust industrial gases and exhaust gases of combustion engines [2]:

$$CO + NO \rightarrow CO_2 + N_2O + N_2$$

The reaction is very slow at room temperature but accelerates over catalysts based on platinum metals including palladium [3]. Studying the chemical behaviour of polynuclear carbonyl and nitrosyl palladium complexes should allow us to understand the mechanism of the catalytic effect.

Palladium carbonyl carboxylates $Pd_6(\mu\text{-}CO)_6(\mu\text{-}RCO_2)_6$ (**1**: R = ^{tert}Bu (**a**), ^{i}Pr (**b**), and ^{n}Pr (**c**)) have a planar cyclic *hexagonal* metal core with alternating pairs of bridging carbonyl and carboxylate ligands [4]. Herein, we describe our investigation of the reaction of clusters **1** with nitric monoxide which leads to palladium carbonyl nitrite carboxylate clusters.

2. Results and discussion

A vast majority of nitrosyl clusters can be obtained from the reaction of carbonyl compounds with nitrosonium cations or nitrite anions as a source of the NO ligand. The direct reaction with gaseous NO usually leads to the decomposition of the carbonyl cluster frameworks [5]. When neutral NO is used, the main problem is that the resultant complex decomposes because NO should donate three electrons to metal frames, one of them being responsible for metal–metal bond cleavage. We have found that the reaction of clusters **1** proceeds quite differently. Upon treatment of complexes **1** with gaseous nitric monoxide at 50 °C for 4–5 h or at r.t. for 2–3 days, the color of the solution changed from bright yellow to dark vinous. In this case, partial decomposition of the complexes into Pd black was observed, and isolated Pd-containing products involve NO_2-groups. According to elemental analysis, these complexes have the total composition $Pd_2(CO)(NO_2)(RCO_2)_2$:

$$Pd_6(CO)_6(RCO_2)_6 + NO \rightarrow Pd_8(CO)_4(NO_2)_4(RCO_2)_8$$
$$+ \ldots R = {}^{tert}Bu, {}^{i}Pr, {}^{n}Pr$$
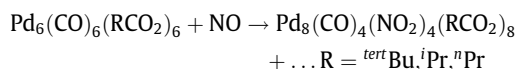
The IR-spectra of the complexes display bands of stretching vibrations of carbonyls (1944–1936 cm^{-1}), nitrites (1560 and 1304–1312 cm^{-1}) and carboxylates (1544–1548, 1404–1412 cm^{-1} – asymmetric and symmetric). Their structures were determined by X-ray crystallography. In general, the molecular structure of $Pd_8(\mu\text{-}CO)_4(\mu\text{-}NO_2)_4(\mu\text{-}RCO_2)_8$ (**2**: R = ^{tert}Bu (**a**), ^{i}Pr (**b**), and ^{n}Pr (**c**)) consists of two bent four-member palladium chains. The ends of

* Corresponding author.
E-mail addresses: oshishilov@gmail.com (O.N. Shishilov), j.a.k.howard@durham.ac.uk (J.A.K. Howard).
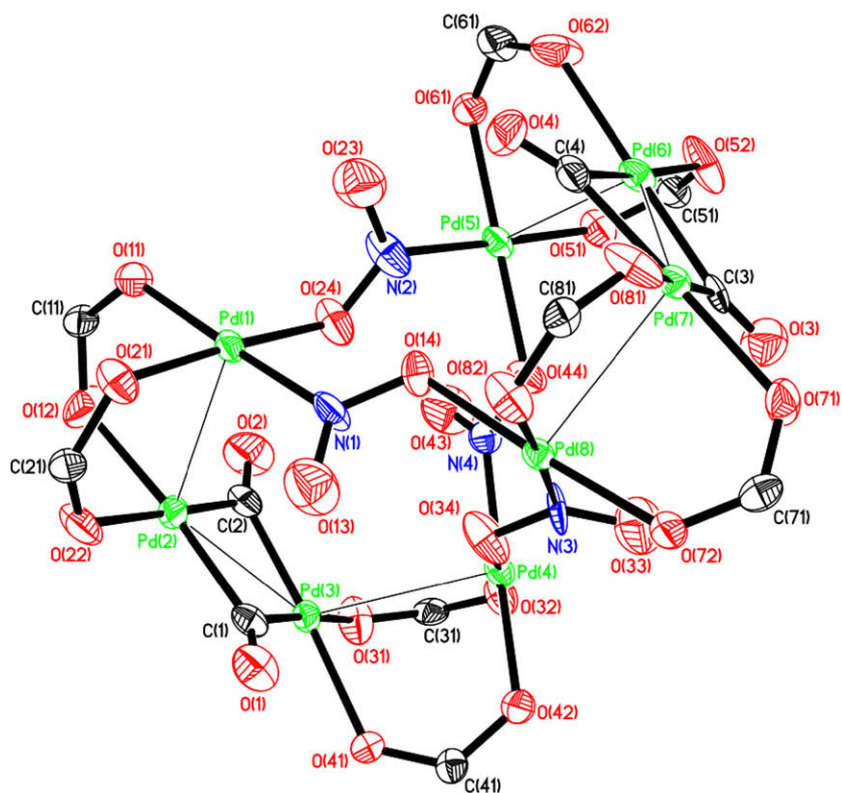‡ Deceased.

Fig. 1. The molecular structure of $Pd_8(\mu\text{-}CO)_4(\mu\text{-}OOCCMe_3)_8(\mu\text{-}NO_2)_4$. Hydrogen atoms and *t*-Bu groups are omitted for clarity.
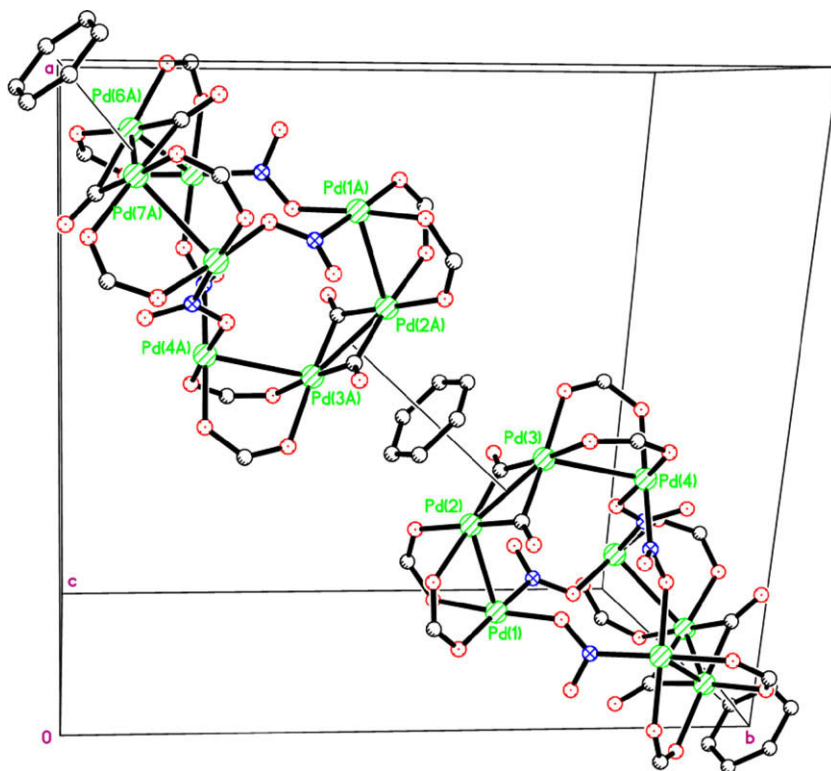

Fig. 2. Cluster–benzene chains in the structure **2a**. Hydrogen atoms and ^{t}Bu groups are omitted for clarity. Thin lines indicate the distances between the centers of $Pd_2(CO)_2$ units and benzene molecules.

the opposite metal chains are connected by four nitrito-groups forming the entire Pd_8 cluster. All nitrito-groups are coordinated by central nitrogen atoms to one metal chain and by terminal oxygen atoms to another. Meanwhile, all terminal Pd atoms capture one N atom and one O atom from bridging nitrito-*N,O* ligands.

Earlier we described complex $Pd_8(\mu\text{-CO})_4(\mu\text{-NO}_2)_4(\mu\text{-}^{tert}BuCO_2)_8$ (**2a**) [6]. Herein, we report another crystal modification of **2a**

(Fig. 1). The structure of **2a** also consists of two bent four-membered palladium chains, Pd(1)–Pd(4) and Pd(5)–Pd(8). Both metal chains are planar within 0.0592(4) Å and are almost perpendicular to each other (88.94(1)°). In these chains, the central metal pairs Pd(2)–Pd(3) and Pd(6)–Pd(8) are linked by two approximately symmetrical μ_2-CO ligands forming near planar $Pd_2(CO)_2$ fragments, while the terminal palladium atoms are linked to central
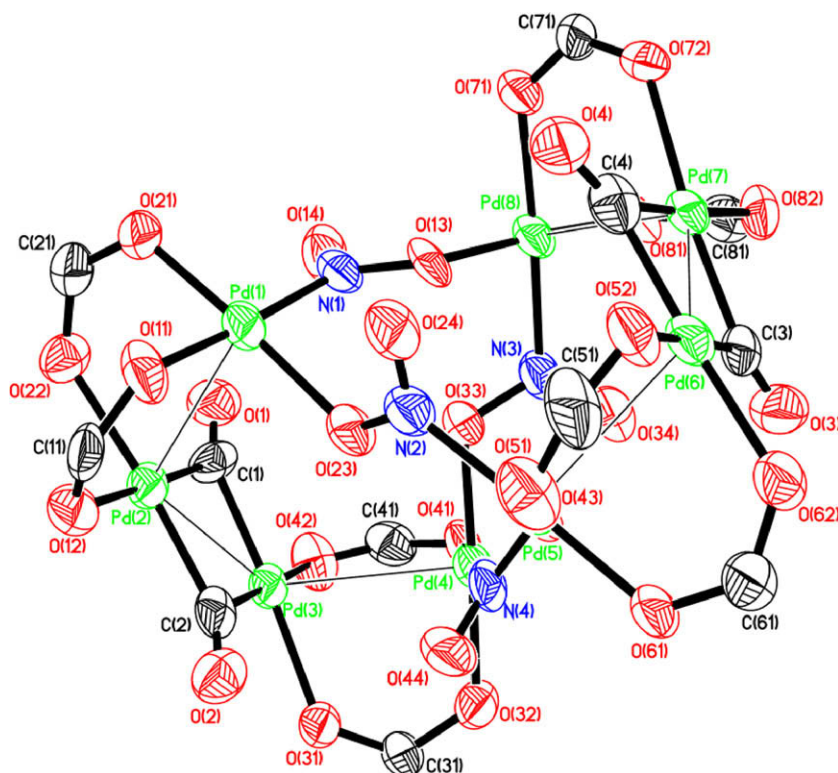

Fig. 3. The molecular structure of $Pd_8(\mu\text{-CO})_4(\mu\text{-OOCCHMe}_2)_8(\mu\text{-NO}_2)_4$. Hydrogen atoms and ^iPr groups are omitted for clarity.
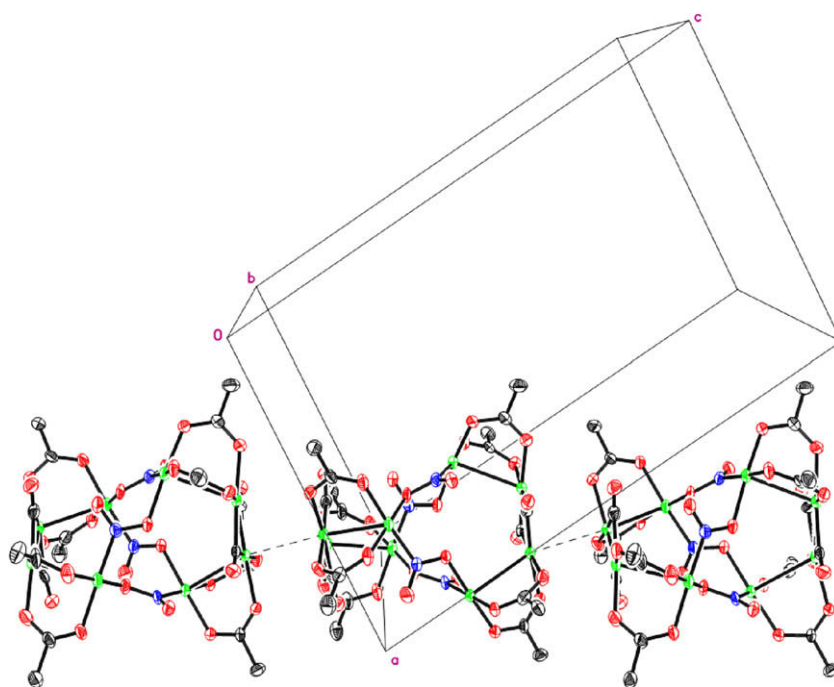

Fig. 4. Chains in the structure of **2b**. Hydrogen atoms and Me groups are omitted for clarity. Thin lines indicate Pd-Pd interactions.

atoms by μ_2-pivalato groups. The coordination environment of all Pd atoms is square–planar with *cis* X-Pd-Y (X, Y = C, N, O) angles ranging within 84.6(2)–94.4(3)°. The Pd–Pd distances range from 2.6723(8) to 2.6747(8) Å for the $Pd_2(\mu\text{-}CO)_2$ fragment and from 2.8479(8) to 2.8853(8) Å for the $Pd_2(\mu\text{-}O_2CR)_2$ unit Table 1. This results from different sterical requirements of bridging CO and carboxylate ligands. In general, the geometry of $Pd_2(\mu_2\text{-}CO)_2$ and $Pd_2(\mu_2\text{-}O_2CMe)_2$ frames is close to that found previously in the structures of the parent carbonyl carboxylate Pd clusters [4]. All four Pd–O–N(=O)–Pd units are planar within 0.155(5) Å. All μ-N and μ-O atoms may be treated as sp^2-hybridizied, since the angles around these atoms are close to 120° (115.4(6)–124.3(5)°).

In crystal, Pd_8 clusters are combined in chains running along the *ab*-diagonal. In a chain, the adjacent clusters are linked by centro-symmetric solvate benzene molecules (Fig. 2). The intermolecular distances between the centers of $Pd_2(CO)_2$ units and benzene molecules are 3.063 and 3.130 Å. These values indicate the presence of strong π–π stacking interactions. We observed the same motif previously in the structures of the parent carbonyl carboxylates **1a,b** $Pd_6(CO)_6(RCO_2)_6 \cdot C_6H_6$ (R = CMe$_3$, CHMe$_2$) [4]. The chains are separated by wide channels filled with additional solvent benzene and hexane molecules lying in general positions. It should be noted that all ^tBu groups are directed towards these channels. The observed high thermal rotation motion of alkyl substituents may result from such a specific crystal packing.

The presented structure of **2a** may be treated as isostructural with the previously determined one [7]. The only difference is that all solvent toluene molecules in the previous structure are replaced by benzene molecules.

The structure of the new complex $Pd_8(\mu\text{-}CO)_4(\mu\text{-}NO_2)_4(\mu\text{-}^iPr\text{-}CO_2)_8$ (**2b**) was also determined by the X-ray diffraction analysis (see Fig. 3). The structure of metal core in **2b** is very similar to that found for **2a**. The crystal of **2b** does not contain any solvent molecules.

In the crystal of **2b**, short intermolecular Pd–Pd interactions combine the adjacent molecules in chains parallel to the *ac*-diagonal (Fig. 4). These interactions are formed by the parallel $Pd_2(CO)_2$ units. The Pd–Pd distance is equal to 3.173(1) Å. We observed the same type of intermolecular metal–metal contacts previously in the structure of the parent cluster **1b** $Pd_6(CO)_6(Me_2HCCO_2)_6 \cdot C_6H_6$ [4a].

On treatment of **2b** with NO at 50 °C, we did increase the yield of complex **2b** and reduce the reaction time. Under these conditions, a partial decomposition of the reaction mixture into palladium black was observed. The reaction time was only 5 h, and the yield was about 10%. Analogous reaction of **1c** under the same conditions resulted in $Pd_8(\mu\text{-}CO)_4(\mu\text{-}NO_2)_4(\mu\text{-}^nPrCO_2)_8$ (**2c**) which was also characterized by X-ray crystallography (see Fig. 5). The molecular structure of **2c** is also very similar to that of **2a** and **2b**.

In structure **2c,** as in structure **2b**, the adjacent clusters are combined in chains by analogous short Pd–Pd interactions (3.117(1) and 3.188(1) Å). However, **2b** and **2c** are not isostructural.

The most interesting feature is the simultaneous presence of CO and NO_2^- ligands in clusters **2**. According to the literature data, the reaction of transition metal carbonyl complexes with nitric monoxide can proceed in different pathways:
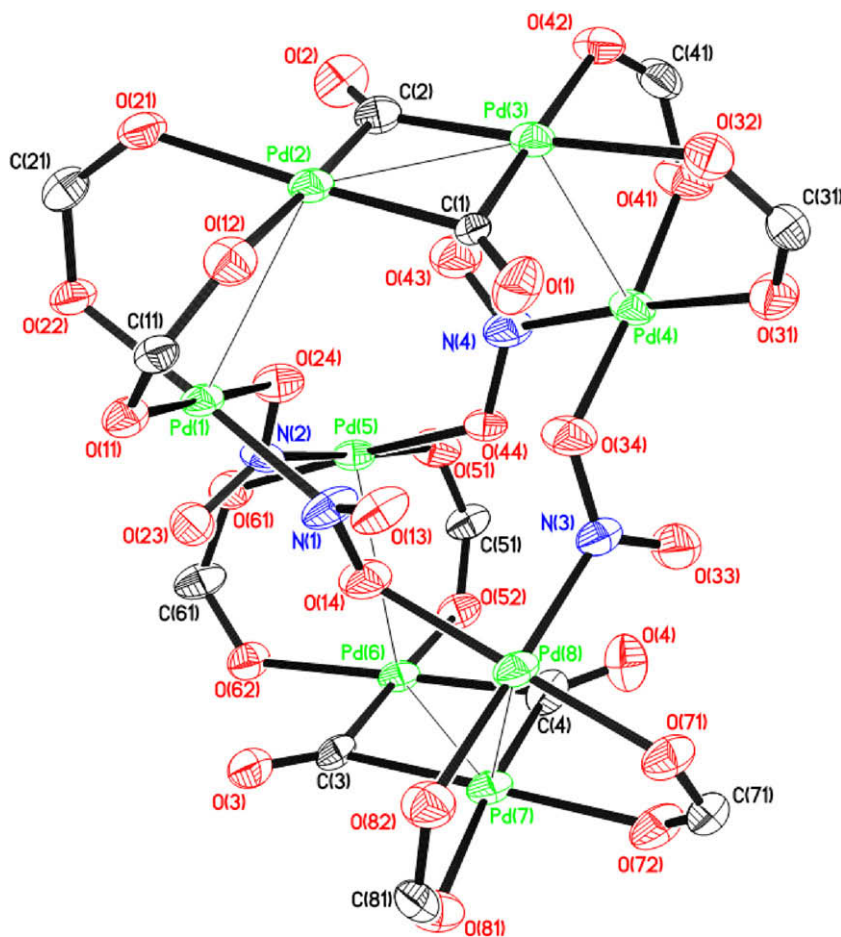

Fig. 5. The molecular structure of $Pd_8(\mu\text{-}CO)_4(\mu\text{-}OOCCH_2CH_2Me)_8(\mu\text{-}NO_2)_4$. Hydrogen atoms and *n*-Pr groups are omitted for clarity.

Table 1
Selected bond lengths (Å) for **2a**, **2b** and **2c**.

	2a	2b	2c
Pd(1)–Pd(2)	2.8479(8)	2.8993(11)	2.8942(9)
Pd(2)–Pd(3)	2.6723(8)	2.6718(11)	2.6769(11)
Pd(3)–Pd(4)	2.8699(8)	2.9118(11)	2.8943(10)
Pd(5)–Pd(6)	2.8853(8)	2.8805(11)	2.9114(9)
Pd(6)–Pd(7)	2.6747(8)	2.6688(12)	2.6901(10)
Pd(7)–Pd(8)	2.8522(8)	2.9026(11)	2.8671(10)
Pd–C	1.964(9)–1.987(8)	1.944(10)–2.012(11)	1.962(10)–2.000(10)
Pd–N	1.974(7)–1.989(7)	1.948(8)–1.993(9)	1.963(8)–1.992(7)
Pd–O(–N)	2.005(6)–2.035(5)	2.016(7)–2.034(7)	2.011(7)–2.044(7)
Pd–O(carboxyl)	1.987(5)–2.133(5)	1.977(7)–2.119(8)	1.986(7)–2.129(7)
C–O(carbonyl)	1.133(9)–1.160(9)	1.111(12)–1.146(12)	1.132(12)–1.171(11)
N–O(terminal)	1.230(9)–1.274(9)	1.203(11)–1.250(10)	1.210(10)–1.241(10)
N–O(–Pd)	1.217(8)–1.246(8)	1.268(10)–1.298(9)	1.271(10)–1.286(9)

1. The partial or full replacement of CO by NO [7].
2. The replacement of CO by NO followed by a nitrite complex formation. In these cases, the nitrite complexes never contain CO [8].
3. The replacement of CO by NO with the nitrite *nitrosyl* rather than carbonyl complex formation [9].

The nitrite carbonyl complex formation has been observed only once [10]. The reason is that carbon monoxide is a typical reducing agent, whereas the nitrite anion possesses strong oxidizing properties. Therefore, CO reduces NO_2 into NO, whereas $[NO_2]$ is a source of nitrosyl ligands of a high-usage (see, for example, [11]). According to [12], CO reduces NO_2 and NO to N_2O rather than to N_2. We determined the composition of the gaseous phase with GC and found that the total amounts of gases after 72 h are the following: 0.15–0.16 mol CO_2/1 mol Pd, 0.03–0.035 mol N_2O/1 mol Pd, and 1.1–1.3 mol N_2/1 mol Pd. Thus, the main product of NO/NO_2 reduction is molecular nitrogen, and clusters **1** being the first complexes that react with NO yielding N_2.

Thus, we detected N_2 in gaseous phase as a product of NO reduction and found NO_2-ligand in clusters **2** as a product of NO oxidation. This means that the NO disproportionation into N_2 and NO_2 proceeds on palladium clusters.

3. Conclusions

The reaction of palladium carbonyl carboxylates with gaseous nitric monoxide is accompanied by the oxidation of coordinated CO and the disproportionation of NO into N_2 and NO_2 under mild conditions. This reaction resulted in eight-nuclear nitrite clusters of the novel type $Pd_8(\mu\text{-CO})_4(\mu\text{-NO}_2)_4(\mu\text{-RCO}_2)_8$, simultaneously containing potentially oxidative and reductive ligands. Up to date, these complexes are the largest palladium carboxylate clusters characterized by an X-ray analysis.

4. Experimental

4.1. General techniques and procedures

All organic solvents and liquid organic reagents were purified and dried according to the standard procedures. Microanalyses were performed on a Carlo Erba Analyzer CHND-OEA 1108. The Carl Zeiss SPECORD-M82 was used for IR-spectroscopy. *Cyclo*-[$Pd_4(\mu\text{-CO})_4(\mu\text{-OOCMe})_4$] was prepared according to a published procedure [13] by reductive carbonylation of palladium diacetate in glacial acetic acid. Palladium carbonyl carboxylates Pd_6-($\mu\text{-CO}$)$_6$($\mu\text{-RCO}_2$)$_6$ were prepared according to a published

procedure [4]. Solid carboxylic acids were commercially supplied. Synthesis of nitric monoxide was carried out according to a standard method by reduction of $NaNO_2$ by KI in the presence of H_2SO_4 in water solution.

4.2. Syntheses of $Pd_8(\mu\text{-NO}_2)_4(\mu\text{-CO})_4(\mu\text{-}^nPrCO_2)_8$

$Pd_6(CO)_6(PrCO_2)_6$(220 mg) was first dissolved in 30 ml benzene and filtered. The solution was placed in a 100 ml two-necked round-bottomed flask and was stirred by a magnetic stirrer under NO atmosphere at 50 °C for 5 h. After that NO was removed, and the dark vinous solution was filtered to remove metal palladium. The filtrate was evaporated on an oil pump to 5–7 ml, benzene or toluene and hexane were added up to dimness. The crystals were obtained by storing the solution in a refrigerator at 4 °C. Crystals were filtered off and dried under vacuum. The yield was 10% based on palladium. Element Anal. calc. for $Pd_8(NO_2)_4(CO)_4(PrCO_2)_8$: C, 23.44; H, 3.04; N, 3.04. Found: C, 23.86; H, 3.12; N, 3.02%. IR-spectrum: 1944, 1560, 1544, 1496, 1404, 1312, 1200, 668 cm^{-1}.

Single crystals of $Pd_8(NO_2)_4(CO)_4(^nPrCO_2)_8$ were obtained from benzene/hexane mixture.

4.3. Synthesis of $Pd_8(\mu\text{-NO}_2)_4(\mu\text{-CO})_4(\mu\text{-}^iPrCO_2)_8$

$Pd_6(CO)_6(^iPrCO_2)_6$(220 mg) was first dissolved in 30 ml toluene and filtered. The solution was placed in a 100 ml two-necked round-bottomed flask and was stirred by a magnetic stirrer under NO atmosphere at 50 °C for 4 h. After that NO was removed, and the dark vinous solution was filtered to remove metal palladium. The filtrate was evaporated on an oil pump to 3–4 ml, toluene and hexane were added up to dimness. The crystals were obtained by storing the solution in a refrigerator at 4 °C, and were filtered off and dried under vacuum. The yield is 10% based on palladium. Element Anal. calc. for $Pd_8(NO_2)_4(CO)_4(OCOCHMe_2)_8$: C, 23.44; H 3.04; N, 3.04. Found: C, 23.91; H, 3.28; N, 2.97%. IR-spectrum: 1944, 1560, 1548, 1496, 1412, 1304, 1200, 1096, 688, 668 cm^{-1}.

Single crystals of $Pd_8(NO_2)_4(CO)_4(^iPrCO_2)_8$ were crystallized from benzene/hexane mixture.

4.4. Syntheses of $Pd_8(\mu\text{-NO}_2)_4(\mu\text{-CO})_4(\mu\text{-}^{tert}BuCO_2)_8$

$Pd_6(CO)_6(^{tert}BuCO_2)_6$(600 mg) was first dissolved in 100 ml benzene and filtered. The solution was placed in a 250 ml three-necked round-bottomed flask and was stirred by a magnetic stirrer under NO atmosphere for 72 h. After that NO was removed, and dark vinous solution was filtered to remove small amount of impurities. The filtrate was evaporated on an oil pump to 5–7 ml, benzene or toluene and hexane were added to precipitate vinous powder, which was filtered off and dried under vacuum. The crystals of $Pd_8(NO_2)_4(CO)_4(^{tert}BuCO_2)_8$ were obtained by storing the solution in a refrigerator at 4 °C. The yield is 12% based on palladium.

4.5. Gas chromatography

Gas chromatography was performed on a 3700 instrument for N_2O and CO_2 analyses equipped with a Porapak Q packed column ($l = 1$ m) and a LHM-80 instrument for N_2, CO and NO analyses equipped with molecular sieve packed column ($l = 3$ m). Both instruments were equipped with a thermal conductivity (TC) detector. The column flow rate of He carrier gas was 30 ml/h at 25 °C.

4.6. Crystal structure determination

The experimental intensities for compounds **2a**, **2b** and **2c** were measured on a Bruker SMART CCD diffractometer (graphite mono-

Table 2
Crystal data, data collection, structure solution and refinement parameters for **2a**, **2b** and **2c**.

Empirical formula	$C_{62}H_{94}N_4O_{28}Pd_8$ (2a)	$C_{36}H_{56}N_4O_{28}Pd_8$ (2b)	$C_{36}H_{56}N_4O_{28}Pd_8$ (2c)
Formula weight	2194.61	1844.05	1844.05
Colour, habit	Orange block	Yellow prism	Yellow needle
Crystal size/mm^3	$0.15 \times 0.06 \times 0.04$	$0.08 \times 0.06 \times 0.06$	$0.30 \times 0.05 \times 0.05$
Crystal system	Monoclinic	Monoclinic	Triclinic
Space group	$P2_1/n$	$P2_1/n$	$P{-}1$
Unit cell dimensions			
$a/Å$	20.2315(5)	12.8280(4)	11.6680(4)
$b/Å$	19.2364(5)	22.1844(8)	12.0687(5)
$c/Å$	23.9185(6)	20.0187(7)	21.5554(8)
$\alpha/(°)$	90	90	101.623(2)
$\beta/(°)$	114.364(1)	98.172(1)	102.066(2)
$\gamma/(°)$	90	90	98.262(2)
Volume/Å^3	8479.6(4)	5639.1(3)	2852.22(19)
Z	4	4	2
Density (calculated)/ g cm^{-3}	1.719	2.172	2.147
Absorption coefficient/ mm^{-1}	1.728	2.576	2.547
$F(000)$	4344	3568	1784
θ Range for data collection/(°)	1.41–27.00	1.84–28.00	0.99–27.50
Index ranges	$-21 \leqslant h \leqslant 25$ $-24 \leqslant k \leqslant 24$ $-29 \leqslant l \leqslant 30$	$-16 \leqslant h \leqslant 16$ $-29 \leqslant k \leqslant 29$ $-17 \leqslant l \leqslant 26$	$-13 \leqslant h \leqslant 15$ $-15 \leqslant k \leqslant 13$ $-28 \leqslant l \leqslant 27$
Reflections collected	52 176	43 079	17 161
Independent reflections	18 505 [$R_{int} = 0.0827$]	13 590 [$R_{int} = 0.1075$]	12 690 [$R_{int} = 0.0497$]
Data/restraints/ parameters	18 505/0/734	13 590/1/621	12 690/12/695
Final R indices [$I > 2\sigma(I)$]	$R_1 = 0.0606$, $wR_2 = 0.1227$	$R_1 = 0.0656$, $wR_2 = 0.1554$	$R_1 = 0.0598$, $wR_2 = 0.1406$
R indices (all data)	$R_1 = 0.1124$, $wR_2 = 0.1366$	$R_1 = 0.1497$, $wR_2 = 0.1831$	$R_1 = 0.1200$, $wR_2 = 0.1597$
Goodness-of-fit on F^2	1.028	0.844	0.951
Largest diffraction peak/hole (e Å^{-3})	2.004/−0.932	2.108/−1.138	2.608/−1.665

chromatized Mo Kα radiation, $\lambda = 0.71073$ Å) at 120 K. The structures were solved by direct methods [14] and refined by full matrix least-squares on F^2 [15][18] with anisotropic thermal parameters for all non-hydrogen atoms except for all terminal methyl groups in **2a** and **2b**. These methyl groups showed high thermal motion due to possible rotation along O_2C–CMe_n bonds. In **2a,** carbon atoms of solvent benzene and hexane molecules were refined with isotropic thermal parameters. In **2c,** two ^nPr groups were found to be disordered over two positions with occupancies ratio of 0.60/0.40and 0.65/0.35 and were refined isotropically. All H atoms were placed in the calculated positions and were refined using a riding model. Crystal data, data collection, structure solution and refinement

parameters are listed in Table 2. Poor crystallinity of all the investigated samples resulted in relatively high values of final R indices and residual electron density.

Appendix A. Supplementary data

CCDC-278722 and -278723 contain the supplementary crystallographic data for this paper. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre via http://www.ccdc.cam.ac.uk/data_request/cif

Supplementary data associated with this article can be found, in the online version, at doi:10.1016/j.jorganchem.2008.12.045.

References

[1] (a) See for example T. Nagano, Chem. Rev. 102 (2002) 1235;
(b) I.M. Wasser, S. Vries, P. Moënne-Loccoz, I. Schröder, K.D. Karlin, Chem. Rev. 102 (2002) 1200;
(c) J.A. McCleverty, Chem. Rev. 104 (2004) 403. and references therein..
[2] M. Shelef, G.W. Graham, Catal. Rev.-Sci. Eng. 36 (3) (1994) 433.
[3] (a) W.B. Hughes, J. Chem. Soc., Chem. Commun. 1126 (1969);
(b) P.J. Gans, Chem. Soc. A 943 (1967);
(c) M. Rossi, A. Sacco, J. Chem. Soc., Chem. Commun. 694 (1971);
(d) D. Gwost, K.G. Caulton, Inorg. Chem. 13 (1974) 414.
[4] (a) T.A. Stromnova, O.N. Shishilov, L.I. Boganova, N.A. Minaeva, A.V. Churakov, L.G. Kuz'mina, J.A.K. Howard, Russ. J. Inorg. Chem. 50 (2) (2005) 179;
(b) T.A. Stromnova, O.N. Shishilov, M.V. Dayneko, K.Yu. Monakhov, A.V. Churakov, L.G. Kuz'mina, J.A.K. Howard, J. Organomet. Chem. 691 (2006) 3730.
[5] W.L. Gladfelter, Adv. Organomet. Chem. 24 (1985) 41. and references therein..
[6] T.A. Stromnova, O.N. Shishilov, A.V. Churakov, L.G. Kuz'mina, J.A.K. Howard, J. Chem. Soc., Chem. Commun. 45 (2007) 4800.
[7] (a) W. Hieber, H.Z. Beutner, Anorg. Allg. Chem. 320 (1963) 101;
(b) N. Tsumori, Q. Xu, Inorg. Chem. 42 (2003) 4519.
[8] (a) S.C. Srivastava, A.K. Shrimal, P. Tiwari, Polyhedron 11 (1992) 1181;
(b) R.K. Afshar, A.K. Patra, E. Bill, M.M. Olmstead, P.K. Mascharak, Inorg. Chem. 45 (2006) 3774.
[9] T.W. Hayton, P. Legzdins, W.B. Sharp, Chem. Rev. 102 (2002) 935.
[10] T. Chihara, K. Sawamura, H. Ikezawa, H. Ogawa, H. Wakatsuki, Organometallics 15 (1996) 415.
[11] (a) R.E. Stevens, D.E. Fjare, W.L.J. Gladfelter, Organomet. Chem. 347 (1988) 373;
(b) R.J. Clark, S.E. Whiddon, R.E. Serfass, J. Organomet. Chem. 11 (1968) 637;
(c) B.F.G. Johnson, J. Lewis, J. Chem. Soc., Dalton Trans. (1983) 2447.
[12] (a) K.M. Kadish, V.A. Adamian, E.V. Caemelbecke, Z. Tan, P. Tagliatesta, P. Bianco, T. Boschi, G.-B. Yi, M.A. Khan, G.B. Richter-Addo, Inorg. Chem. 35 (1996) 1343;
(b) D.S. Bohle, P.A. Goodson, B.D. Smith, Polyhedron 15 (1996) 3147;
(c) G.-B. Yi, M.A. Khan, G.B. Richter-Addo, J. Chem. Soc., Chem. Commun. 2045 (1996);
(d) S.J. Hodge, L.-S. Wang, M.A. Khan, V.G. Young, G.B. Richter-Addo, J. Chem. Soc., Chem. Commun. (1996) 2283.
[13] I.I. Moiseev, T.A. Stromnova, M.N. Vargaftik, G.Ja. Mazo, L.G. Kuz'mina, Yu.T. Struchkov, J. Chem. Soc., Chem. Commun. 27 (1978).
[14] G.M. Sheldrick, Acta Crystallogr. A A46 (1990) 467.
[15] G.M. Sheldrick, SHELXL-97. Program for the Refinement of Crystal Structures, University of Göttingen, Germany, 1997.