Received: 30 October 2019 | Revised: 26 November 2019 | Accepted: 26 November 2019
DOI: 10.1002/jccs.201900463

SPECIAL ISSUE PAPER

JOURNAL OF THE CHINESE CHEMICAL SOCIETY

Reaction of small molecules O_2, NO, CO, and the Naldini salt $(PPh_3)_2MnBr_2$: Characterized by infrared spectroscopy, elemental analysis, and single-crystal X-ray diffraction

Bo-Wei Huang[1] | **Minh-Trung Nguyen**[1,2] | **Chung-Hung Hsieh**[1]

[1]Department of Chemistry, Tamkang University, New Taipei City, Taiwan

[2]Department of Biology, Faculty of Natural Science and Technology, Tay Nguyen University, Buon Ma Thuot City, Vietnam

Correspondence
Chung-Hung Hsieh, Department of Chemistry, Tamkang University, New Taipei City 25137, Taiwan.
Email: chsieh@mail.tku.edu.tw

Funding information
Ministry of Science and Technology, Taiwan, Grant/Award Number: MOST 107-2113-M-032-001-; 105-2113-M-032-004-MY2

Abstract

Addition of 2 equiv. of PPh_3 to $MnBr_2$ in tetrahydrofuran (THF) solution under N_2 atmosphere results in the formation of Naldini salt $(PPh_3)_2MnBr_2$ (**1**). Reaction of Complex **1** and O_2, NO, and CO (with reducing agent) leads to Complex $(OPPh_3)_2MnBr_2$ (**2**), $(PPh_3)_2Mn(NO)Br_2$ (**3**), and $(PPh_3)_2Mn(CO)_3Br$ (**4**), respectively. Both Complexes **2** and **4** crystallize in the triclinic space group P-1 with $a = 9.94$ Å, $b = 10.11$ Å, $c = 10.53$ Å; $\alpha = 65.42°$, $\beta = 63.16°$, and $\gamma = 89.22°$ of **2** and $a = 10.23$ Å, $b = 12.26$ Å, $c = 14.44$ Å and $\alpha = 97.03°$, $\beta = 104.34°$, and $\gamma = 106.33°$ of **4**. The isoelectronic replacement of 3CO with 2NO yields the $\{Mn(NO)_2\}^8$ species $(PPh_3)_2Mn(NO)_2Br$ (**5**). The single crystal of **5** is in the monoclinic space group C2/c with a = 23.17 Å, b = 9.62 Å, c = 15.92 Å, and β = 114.91°. In the THF solution, Complex **5** serves as an NO source in the presence of NO trapping, Co(TPP), Co(TPP) = 5,10,15,20-tetraphenyl-21*H*,23*H*-porphine cobalt(II).

KEYWORDS

bromide, carbon monoxide, manganese, Naldini salt, nitric oxide, oxygen, triphenyl phosphine

1 | INTRODUCTION

The Naldini salt $(PPh_3)_2MnBr_2$ (**1**) was first prepared under the air and ambient condition in 1960.[1] However, the reaction product was considered to be an oxygenated and unoxygenated mixture based on elemental analysis.[2] The preparation was then improved by Dumitru in 1965. The stoichiometric portions of $MnBr_2$ and PPh_3 were dissolved in dried, freshly distilled THF and then refluxed for 4 hr under nitrogen to generate Complex **1** (Equation 1).[3]

In a recent study, the triphenyl phosphine ligand of Naldini salt was modified to several dibenzofuran-based phosphines. These unique derivatives exhibit intense green phosphorescence with high photoluminescence quantum yields (PLQYs).[4] These manganese complexes are simple to synthesize and have a stable structure, bipolar characteristics, and good photo physics properties, which reduce the manufacturing cost of the luminescent material and can replace the precious metal ruthenium complex as the luminescent layer material to prepare the organic light-emitting diode.[4]

Also in the biological system, the manganese complexes were involved in the light-dependent reaction of oxygenic photosynthesis (photosystem II) and are known as the oxygen-evolving center (OEC). Photosynthetic water splitting to generate molecule oxygen was catalyzed by four manganese-oxo units with an OEC, which may contribute to effective sunlight as an alternative energy source and O_2 for human daily use.[5]

The reaction/activation of small molecules (H_2, N_2, O_2, NO, N_2O, CO_2, CO, and CH_4) by metal-mediated binding has been the highlight of fundamental chemistry in the past 10–15 years,[6] but little is known about the reactivity of these small molecules and the Naldini salt in the literature.[1–3] Here, we have examined the characteristics of O_2, NO, and CO toward the Naldini salt (PPh$_3$)$_2$MnBr$_2$ (**1**).

2 | RESULTS AND DISCUSSION

The Naldini salt (PPh$_3$)$_2$MnBr$_2$ (**1**) was prepared using an improved procedure. Complex **1** is a bright yellow solid with good solubility in the THF or CH_2Cl_2 at room temperature. Reaction of Complex **1** and freshly prepared O_2 gas in the THF solution leads to the formation of greenish yellow Complex (OPPh$_3$)$_2$MnBr$_2$ (**2**) (Scheme 1a). The single crystal of Complex **2** was obtained by slow diffusion of the diethyl ether/hexanes solution into a concentrated THF solution at 0°C. Full crystallographic data of Complex **2** are shown in Tables S1 and S2, and the selected bond lengths and angles are listed in Table 1. Greenish yellow crystals of **2** are stable in the air. The structure was first published in 1966 in the monoclinic system, with $a = 24.33$, $b = 14.33$, $c = 13.18$ Å, and β 93°; $Z = 7$.[7] In this work, the triclinic system was observed with $a = 9.94$, $b = 10.11$, $c = 10.53$ Å and $\alpha = 65.4°$, $\beta = 63.2°$, $\gamma = 89.2°$; $Z = 1$. The distorted tetrahedral geometries in both structures are largely the same (Figure 1)

In the reaction of Complex **1** and freshly prepared NO gas in the THF solution, the color of the solution turned from bright yellow to orange in about 5 min (Scheme 1b). The reaction product was identified by FT-IR (ν(NO): 1,737 s cm^{-1}), which was assigned as a mononitrosyl complex (PPh$_3$)$_2$Mn(NO)Br$_2$ (**3**) with $\{Mn(NO)\}^6$ electronic configuration according to the Enemark-Feltham notation.[8]

No reaction occurred when CO gas was bubbled into the THF solution of Complex **1**, which may be attributed to the lack electron density of the Mn^{II} center, partially transferred from a d-orbital of the metal to antibonding

TABLE 1 Selected bond distances (Å) and angle (deg) for Complexes **2**, **4**, and **5**

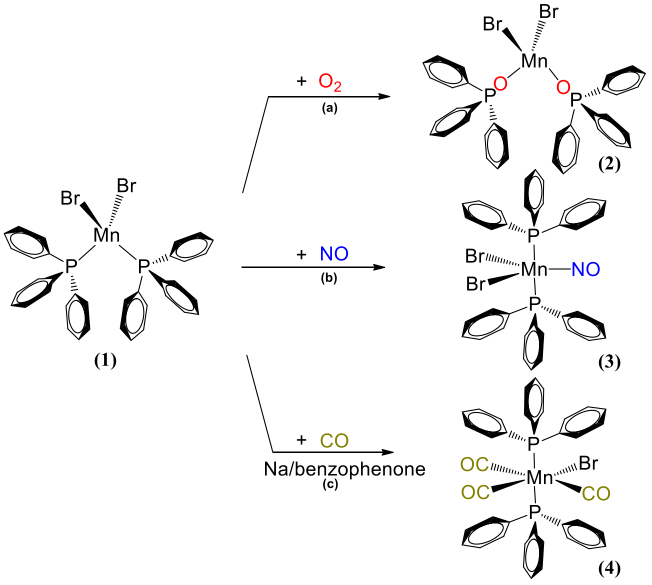
	2	**4**	**5**
Mn-PPh$_{3avg.}$	—	2.323(5)	2.329(6)
Mn-OPPh$_{3avg.}$	2.389(4)	—	—
Mn-Br	2.4739(14)^a	2.496(7)	2.4838(14)
Mn-CO$_{avg.}$	—	1.7174(19)	—
Mn-NO$_{avg.}$	—	—	1.687(8)
∠Ph$_3$P-Mn-PPh$_3$	—	176.48(2)	164.70(4)
∠O-Mn-O	100.89(17)	—	—
∠OC-Mn-CO$_{avg.}$	—	175.7(2)^b 89.5(2)^c	—
∠ON-Mn-NO	—	—	120.00(16)
∠Mn-N-O$_{avg.}$			174.6(7)

Note: The maximum deviations from the average distances and angles are shown in the table.

[a]The average distance of Mn-Br.

[b]The angle of *trans*-CO.

[c]The average angle of *cis*-CO. Full lists of metric parameters are given in the SI.



SCHEME 1 Reaction of (PPh$_3$)$_2$MnBr$_2$ (**1**) and O_2, NO, and CO

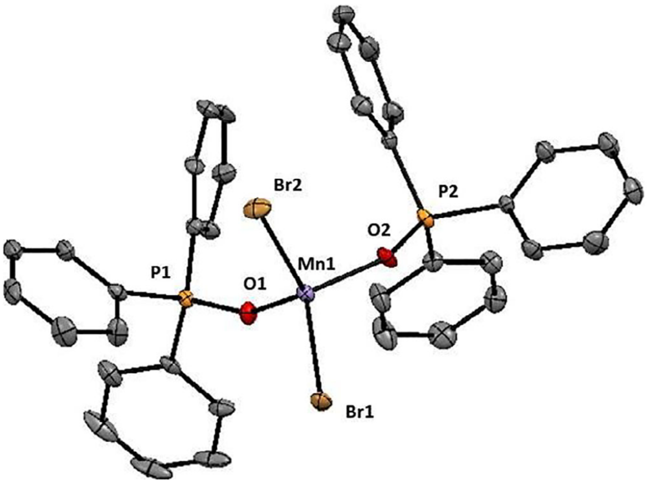


FIGURE 1 View of the thermal ellipsoid plot at 50% probability of Complex **2**. Hydrogen atoms have been removed for clarity. Full metric data of Complex **2** are listed in the Supporting Information

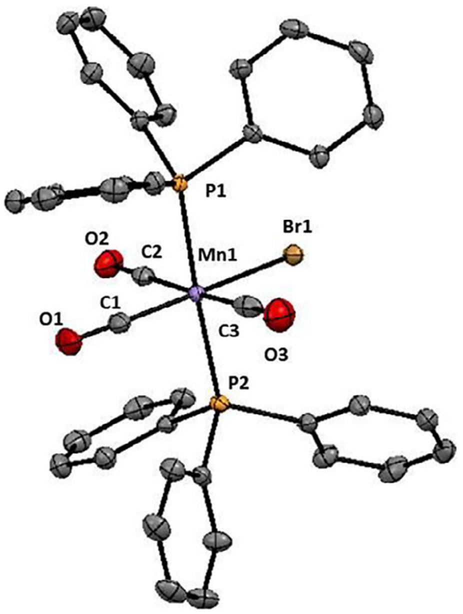
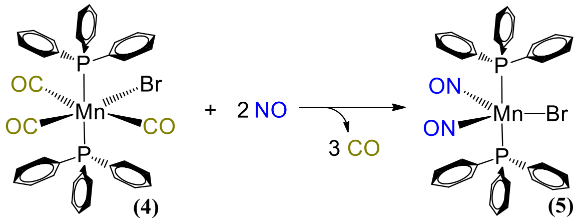


FIGURE 2 View of the thermal ellipsoid plot at 50% probability of Complex **4**. Hydrogen atoms have been removed for clarity. Full metric data of Complex **4** are listed in the Supporting Information. The site occupancy factor of the Br1 and the C1O1 groups in Complex **4** is found at disordered positions (2/3:1/3)

molecular orbitals of CO.[9] Addition of the reducing agent (sodium/benzophenone) in an equimolar portion of 1 under CO atmosphere in THF at room temperature overnight results in the formation of complex $(PPh_3)_2Mn(CO)_3Br$ (**4**) (Scheme 1c, FT-IR ν(CO): 1,950 vs, 1,916 w cm^{-1}). Complex **4** was isolated as a light orange semisolid, and single crystals suitable for the X-ray analysis were obtained by slow vaporization of the diethyl ether/hexanes mixed solution in the refrigerator. The structural determination data are shown in the SI (Tables S2 and S3), and selected bond lengths/angles are summarized in Table 1. The structure of **4** is described as an octahedral with two PPh_3 ligands located in the *trans*- position of the Mn^I center (Figure 2). Another synthetic option of Complex **4** is to react $Mn(CO)_5Br$ and 2 equiv. of PPh_3 in THF solution and reflux under N_2[10]

We also test the reactivity of Complex **4** and NO gas. The freshly prepared dried NO was bubbled into the solution until the reaction mixture turned from yellow to orange (about 5 min) at ambient temperature (Scheme 2). The FT-IR characterization (ν(NO): 1,712 m, 1,668 s cm^{-1} (THF)) was assigned to the formation of complex $(PPh_3)_2Mn(NO)_2Br$ (**5**). Single crystals were obtained by slow diffusion of the diethyl ether/hexane solution into a concentrated THF solution of Complex **5** at −20°C. The full crystal data of **5** are shown in the SI (Tables S4 and S5), and selected bond distances and angles are listed in Table 1. The ORTEP plot of five coordinate Complex **5** with distorted trigonal bipyramidal (TBP) geometry is shown in Figure 3. The structure is best described as two NO and one bromide located in the equatorial and two PPh_3 occupying the axial position. Complex **5** was first synthesized by Henri and Manfred in 1973, which was characterized as an impurity from the reaction of $[(C_5H_5)(PPh_3)Mn(CO)(NO)][PF_6]$ and PhLi (where PhLi is prepared from PhBr) and lack of X-ray structural evidence.[11] In this study, the isoelectronic replacement of 3CO by 2NO is presented, which leads to the formation of pure product **5**. The Mn-$PPh_{3avg.}$ and Mn-Br distances of Complex **5** are 2.329 (6) and 2.4838(14) Å, respectively, which are approximately identical to Complex **4** (2.323(5) and 2.496(7) Å). The Mn-$NO_{avg.}$ angle of **5** is close to linear (174.6(7)°). Therefore, we assign the electronic configuration of **5** as a composition of Mn^I and two NO radicals, that is, $\{Mn^I(\bullet NO)_2\}^8$[8]



SCHEME 2 Reaction of $(PPh_3)_2Mn(CO)_3Br$ (**4**) and NO

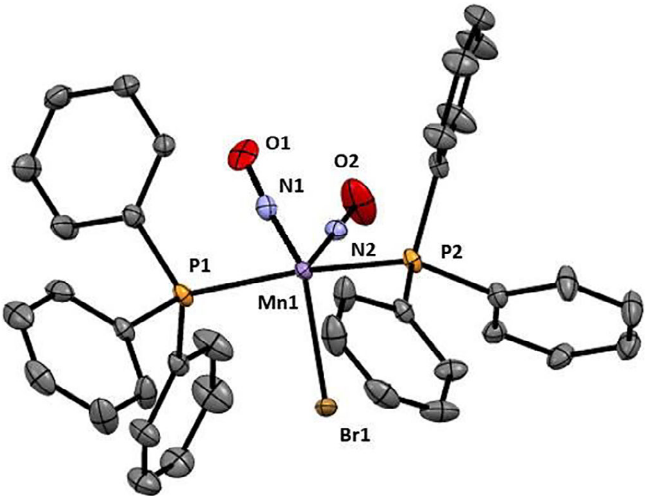


FIGURE 3 View of the thermal ellipsoid plot at 50% probability of Complex **5**. Hydrogen atoms have been removed for clarity. Full metric data of Complex **5** are listed in the Supporting Information. The site occupancy factor of the Br1 and the N1O1 groups in Complex **5** is found at disordered positions (1/2:1/2)

To probe the ability of the *Di*nitrosyl *M*anganese *C*omplex **5** (DNMC **5**) to release or transfer NO, the DNMC **5** was combined with 2 equiv. of NO trapping reagent Co(TPP), Co(TPP) = 5,10,15,20-tetraphenyl-21*H*,23*H*-porphine cobalt(II)), in THF solution. Coupled with an almost immediate color change, the FT-IR band that grew at 1,683 cm^{-1}, concomitant with the loss of NO bands from the DNMC **5**, indicated (NO)Co(TPP) formation (Equation 2).[12] This reactivity of the $\{Mn(NO)_2\}^8$ DNMC **5** was consistent with the oxidized $\{Fe(NO)_2\}^9$ *D*initrosyl *I*ron *C*omplexes, DNICs, which showed NO-releasing ability upon mixing with THF solutions of Co(TPP).[13] The preliminary result of this study suggests that DNMC in the $\{Mn(NO)_2\}^8$ oxidation level is able to act as a 2NO donor in the presence of a suitable NO-trapping agent. Nevertheless, the mechanism of NO transfer is unknown, similar to the fate of the DNIC following loss of NO.[14]

3 | EXPERIMENTAL

3.1 | Synthesis

Manipulations, reactions, and transfers were conducted under nitrogen according to Schlenk techniques or in a glovebox (nitrogen gas). Solvents were distilled under nitrogen from appropriate drying agents (diethyl ether, hexanes, and tetrahydrofuran (THF) from sodium benzophenone) and stored in dried, N_2-filled flasks over 4 Å molecular sieves. Nitrogen was purged through these solvents before use. The solvent was transferred to the reaction vessel via stainless cannula under positive pressure of N_2. The reagents $MnBr_2$, triphenylphosphine (PPh_3) (TCI), sodium, and benzophenone (Sigma-Aldrich) and infrared spectra of the carbonyl ν(CO) and ν(NO) stretching frequencies were recorded on a Thermo *iS5* FT-IR spectrophotometer. Analyses of carbon, hydrogen, and nitrogen were conducted with a CHN analyzer (Heraeus)

3.1.1 | Reaction of Complex $(PPh_3)_2MnBr_2$ (1) and O_2

A 50 ml Schlenk flask was loaded with Complex **1** (0.296 g, 0.4 mmole) and dissolved in THF (10 ml). The freshly prepared dried O_2 was bubbled until the reaction mixture turned from light yellow to greenish yellow (about 30 min) at ambient temperature. The solution was filtered through celite to separate the insoluble solid. After the resulting solution was reduced under vacuum, diethyl ether and hexanes (10 and 20 ml, respectively) were added to precipitate the solid and dried under vacuum to afford the solid of Complex $(OPPh_3)_2MnBr_2$ (**2**) (yield 0.234 g, 76%). The greenish yellow crystals, suitable for single-crystal X-ray diffraction analysis were obtained by slow diffusion of the diethyl ether/hexanes solution into a concentrated THF solution of Complex **2** in the refrigerator at 0°C. Anal. Calcd. for $C_{36}H_{30}Br_2Mn_1O_2P_2$: C, 56.06; H, 3.92. Found: C, 56.00; H, 3.75

3.1.2 | Reaction of Complex 1 and NO

A 50 ml Schlenk flask was loaded with Complex **1** (0.296 g, 0.4 mmole) and dissolved in THF (10 ml). The freshly prepared dried NO was bubbled until the mixed solution turned from light yellow to orange (about 5 min) at ambient temperature. The product was identified by FT-IR to confirm the formation of Complex $(PPh_3)_2Mn(NO)Br_2$ (**3**). The solution was filtered through celite to separate the insoluble solid. After the resulting solution was reduced under vacuum, diethyl ether and hexanes (10 and 20 ml, respectively) were added to precipitate the solid and dried under vacuum to afford the solid of Complex **3** (yield 0.189 g, 64%). The orange crystals, suitable for single-crystal X-ray diffraction analysis, were obtained by slow diffusion of the diethyl ether/hexanes solution into a concentrated THF solution of Complex **3** in the refrigerator at −20°C. IR ν(NO): 1,737 s cm^{-1} (THF). Anal. Calcd. for $C_{36}H_{30}Br_2MnNOP_2$: C, 56.20; H, 3.93; N, 1.82. Found: C, 56.13; H, 4.10; N, 2.00

3.1.3 | Reaction of Complex 1 and CO with reducing agent Na/benzophenone

To a stirred solution of Complex **1** (0.296 g, 0.4 mmole) in THF (15 ml) under $CO_{(g)}$ atmosphere at room temperature, a portion of Na/benzophenone THF solution (0.011 g and 0.48 mmole of metal Na and 0.073 g of benzophenone in 10 ml THF) was added dropwise by syringe. The reaction solution was stirred for 10 hr at room temperature and then dried under vacuum to afford the semisolid. After diethyl ether and hexane (5 and 15 ml, respectively) were added to dissolve the semisolid, the reaction mixture was filtered through celite to remove the insoluble solid. The filtrate was then dried under vacuum to afford the light orange semisolid of complex $(PPh_3)_2Mn(CO)_3Br$ (**4**) (yield 0.139 g, 47%). The light orange crystals, suitable for single-crystal X-ray diffraction analysis, were obtained by slow vaporization of the diethyl ether/hexanes solution in the refrigerator at −20°C. IR ν(CO): 1,950 vs 1,916 w/cm (THF). Anal. Calcd. for $C_{39}H_{30}BrMnO_3P_2$: C, 62.75; H, 4.46. Found: C, 63.01; H, 4.07

3.1.4 | Reaction of Complex 4 and NO

A 50 ml Schlenk flask was loaded with Complex **4** (0.297 g, 0.4 mmole) and dissolved in THF (10 ml). The freshly prepared dried NO was bubbled into the solution until the reaction mixture turned from yellow to orange (about 5 min) at ambient temperature. The product was identified by FT-IR to confirm the formation of

Complex $(PPh_3)_2Mn(NO)_2Br$ (**5**). The solution was filtered through celite to separate the insoluble solid. After the resulting solution was reduced under vacuum, diethyl ether and hexanes (10 and 20 ml, respectively) were added to precipitate the solid and dried under vacuum to afford the solid of Complex **5** (yield 0.189 g, 58%). The dark orange crystals, suitable for single-crystal X-ray diffraction analysis, were obtained by slow diffusion of the diethyl ether/hexanes solution into a concentrated THF solution of Complex **5** in the refrigerator at −20°C. IR ν(NO): 1,712 m, 1,668 s/cm (THF). Anal. Calcd. for $C_{36}H_{30}BrMnN_2O_2P_2$: C, 60.10; H, 4.20; N, 3.89. Found: C, 59.83; H, 3.98; N, 4.01

3.1.5 | NO trapping experiments

Orange Complex **5** was formed in situ according to the procedure described above (0.05 mmole) and transferred via a cannula to a Schlenk flask containing Co(TPP) (0.067 g, 0.1 mmole in 10 ml of THF, Co(TPP) = 5,10,15,20-tetraphenyl-21*H*,23*H*-porphine cobalt(II)).Within minutes, an FT-IR absorption band at 1,683 cm^{-1} assignable to (NO) Co(TPP) appeared and continued to grow over the course of 2 hr, along with a distinct color change of the solution from dark orange-red to red. A decrease of Complex **5** FT-IR bands at 1,712 and 1,668 cm^{-1} and an increase of the band at 1,683 cm^{-1} were considered an indication of NO transfer[12,13]

3.2 | Crystallography

Crystallographic data and structure refinements parameters of Complexes **2, 4,** and **5** are summarized in the Supporting Information (Tables S1–S6). The crystals of Complexes **2, 4,** and **5** chosen for X-ray diffraction studies are measured in sizes of 0.30 × 0.03 × 0.3 mm, 0.12 × 0.10 × 0.04 mm, and 0.09 × 0.03 × 0.02 mm, respectively. Each crystal was mounted on a glass fiber and quickly coated in epoxy resin. Unit-cell parameters were obtained by least-squares refinement. Diffraction measurements for Complexes **2**, **4**, and **5** were carried out on a Bruker SMART Apex CCD diffractometer using graphite-monochromated Mo Kα radiation (λ = 0.7107 Å), with values between 3.60° and 28.34° for Complex **2**, between 2.54° and 2,747° for Complex **4**, and between 3.33 and 28.34° for Complex **5**. The space groups were determined on the basis of systematic absences and intensity statistics. The structures were solved by direct methods and refined by full-matrix least-squares on F^2. Anisotropic displacement parameters were determined for all nonhydrogen atoms. Hydrogen atoms were placed at idealized positions and refined with fixed isotropic displacement parameters. The following is a list of programs used: data collection and cell refinement, APEX2;[15] data reductions, SAINTPLUS Version 6.63;[16] absorption correction, SADABAS;[17] structural solutions, SHELXS-97;[18] structural refinement, SHELXL-97;[19] and graphics and publication materials, Mercury Version 2.3.[20] Full crystal data have been submitted to the Chambridge Crystallographic Data Centre (CCDC).

4 | CONCLUSIONS

We have synthesized the Naldini salt $(PPh_3)_2MnBr_2$ (**1**) and reacted it with O_2, NO, and CO. We have given an account of the characterization of each product, including the IR, elemental analysis, single-crystal X-ray diffraction, and the NO transferring from DNMC **5** in the presence of NO trapping agent Co(TPP). This study may provide information for sensing/releasing of these gas molecules for future applications.

ACKNOWLEDGMENTS
This work was supported by the Ministry of Science and Technology, Taiwan (MOST 107-2113-M-032-001-; 105-2113-M-032-004-MY2). We thank Dr. Gene-Hsiang (Instrumentation Canter, National Taiwan University) for the discussions of crystallographic analyses.

ORCID
Chung-Hung Hsieh https://orcid.org/0000-0003-1509-8146

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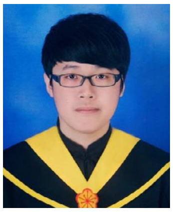
AUTHOR BIOGRAPHIES



Bo-Wei Huang received his B.S. and M.S. degree in Chemistry from Tamkang University, Taiwan, in 2014 and 2016, respectively. He undertook research on synthesis of the organometallic manganese complexes under the supervision of Professor Chung-Hung Hsieh.

Minh-Trung Nguyen is currently a Lecturer at Tay Nguyen University, Vietnam. He received his M.S. degree of Science in Chemical Biology, Tamkang University, Taiwan, under the supervision of Professor Chung-Hung Hsieh. His scientific interests include synthesis/isolation of bioactive complexes and drug target development.

Chung-Hung Hsieh is a Taiwanese, with a Ph.D. from the National Tsing Hua University, Taiwan. Following as associated research scantiest at Taxes A&M University, College Station, USA, he joined the faculty at Department of Chemistry, Tamkang University, Taiwan, in 2013. Trained as a bioorganometallic chemist, his research focuses on the small molecules (H_2O, O_2, CO, NO or H_2) activation via organometallic compounds and metal in medicine.

SUPPORTING INFORMATION

Additional supporting information may be found online in the Supporting Information section at the end of this article.

How to cite this article: Huang B-W, Nguyen M-T, Hsieh C-H. Reaction of small molecules O_2, NO, CO, and the Naldini salt $(PPh_3)_2MnBr_2$: Characterized by infrared spectroscopy, elemental analysis, and single-crystal X-ray diffraction. *J Chin Chem Soc*. 2019;1–6. https://doi.org/10.1002/jccs.201900463