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Synthesis and Characterization of *N*-(2,2-Diphenylacetyl)-*N*-Substituted Thiourea Derivatives: The Crystal Structure of *N*-(2,2-Diphenylacetyl)-*N*-(4-chloro phenyl)-thiourea

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Synthesis and Characterization of *N*-(2,2-Diphenylacetyl)-*N'*-Substituted Thiourea Derivatives: The Crystal Structure of *N*-(2,2-Diphenylacetyl)-*N'*-(4-chlorophenyl)-thiourea

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A series of new N-(2,2-diphenylacetyl)-N'-substituted thiourea derivatives (1–9) have been prepared and characterized by elemental analyses, IR and ¹H NMR spectroscopy. N-(2,2-diphenylacetyl)-N'-(4-chlorophenyl)-thiourea was also characterized by a single crystal X-ray diffraction study. The compound crystallizes in the monoclinic space group P2₁/c with Z = 4 and a = 9.6551(19) Å, b = 20.060(4) Å, c = 9.894(2) Å, β = 104.29(3)°. The molecular conformation of the compound is stabilized by an intramolecular (N1-H1...O1) hydrogen bond that forms a pseudo-six-membered ring.

Keywords Pseudo-six-membered ring; single crystal structure determination; synthesis; thiourea

INTRODUCTION

Thiourea derivatives have attracted much attention due to their chemical properties and their biological activities.¹ Some thiourea derivatives have been found to be useful as insecticides, fungicides, herbicides, and plant-growth regulators.^{2–4} In recent years, thiourea derivatives have been the subject of special attention because it has been shown that they have antitumor bioactivities, antifungal bioactivities, and

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inhibitor activities against viruses.^{2,5-7} In addition, thiourea derivatives have been widely applied in many research fields, such as the separation of platinum group metals and the extraction of trace elements.⁸⁻¹² The thiourea derivatives have also received much attention because the existence of a thiocarbonyl group in these compounds enhances their coordination ability. Particularly the presence of oxygen, nitrogen, and sulfur donor atoms provides a variety of bonding possibilities. These derivatives react with transition metals mostly in monoanionic and bidentate form by deprotonation, resulting in neutral complexes with sulfur and oxygen coordination.¹³

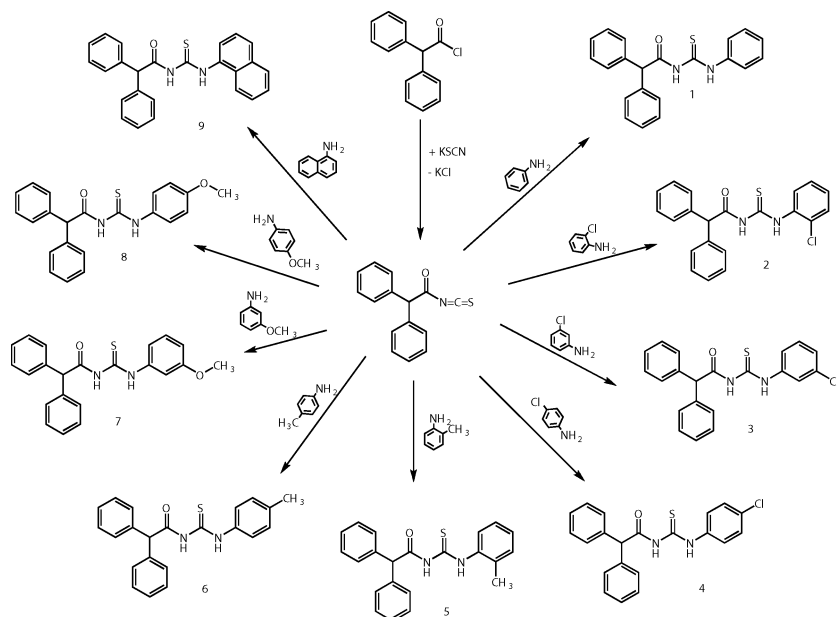
In the past few years, we have pursued investigations on the synthesis, characterization, crystal structure, thermal behavior, vibrational analysis, antifungal activity, and antibacterial activity of new thiourea derivatives.¹⁴⁻³³ Based upon a literature search, we found no data on the synthesis or characterization of thiourea derivatives of the type presented here.

In this article, we report the preparation and characterization of nine new thiourea derivatives and the crystal and molecular structure of *N*-(2,2-diphenylacetyl)-*N'*-(4-chlorophenyl)-thiourea.

RESULTS AND DISCUSSION

All thioureas **1-9** were prepared according to the method of Douglass and Dains³⁴ and were obtained in yields ranging from 85% to 94% (Scheme 1). The synthesis involves the reaction of a 2,2-diphenylacetyl chloride with potassium thiocyanate in acetone followed by condensation of the resulting 2,2-diphenylacetyl isothiocyanate with an appropriate primary amine. The compounds were purified by recrystallization from ethanol:dichloromethane mixtures and characterized by elemental analysis, ¹H NMR, and IR spectroscopy. The analytical and spectroscopic data are consistent with the proposed structures.^{34,35} The synthesis of intermediate and target compounds **1-9** were performed by the reactions illustrated in Scheme 1.

The main vibrational bands of the compounds **1-9** are given in the experimental section. The characteristic ν_{NH} stretching vibrations of *N,N*-dialkyl-*N'*-benzoylthiourea derivatives are expected to appear in the 3450–3100 cm⁻¹ frequency range.^{14-17,32-38} The room temperature attenuated total reflection Fourier transform infrared spectra of the new compounds show two bands at ~3250 cm⁻¹ and ~3180 cm⁻¹ due to ν_{NH} stretching vibrations.³⁶ This difference between the ν_{NH} stretching vibration frequencies is due to intramolecular hydrogen bonding (X-ray



SCHEME 1 Synthesis reaction of the compounds.

single crystal diffraction data). The carbonyl group is connected to the imine group via an intramolecular hydrogen bond. The characteristic region for the carbonyl stretching vibrations in thiourea derivatives is $1700\text{--}1650\text{ cm}^{-1}$.^{34–36} A strong absorption band at $1673\text{--}1689\text{ cm}^{-1}$ in the IR spectra of the new compounds is ascribed to the stretching vibration of the carbonyl group. A strong band at $\sim 1240\text{ cm}^{-1}$ is assigned to the stretching vibration of the thiocarbonyl group. This results agree with the data in the literature.^{37,38}

The ^1H NMR spectra of the new thioureas are consistent with their structures. Here only the N-H signals are discussed. The two N-H signals of the compounds **1–9** are observed at $12.00\text{--}12.42$ and at $11.80\text{--}11.99$ ppm. Because of the formation of an intramolecular hydrogen bond, the signal of the imine proton, which makes a hydrogen bond, appears at a lower field.³⁷ This observation agrees with the results of Li et al.³⁸ The signal of the other imine proton appears at higher field. Generally the NMR signals of NH protons for amides are observed in the range of $9\text{--}10$ ppm. The low-field shift of the signal for the imine proton for compounds **1–9** can be attributed to the deshielding effect of the electron-withdrawing carbonyl and thiocarbonyl group. All these

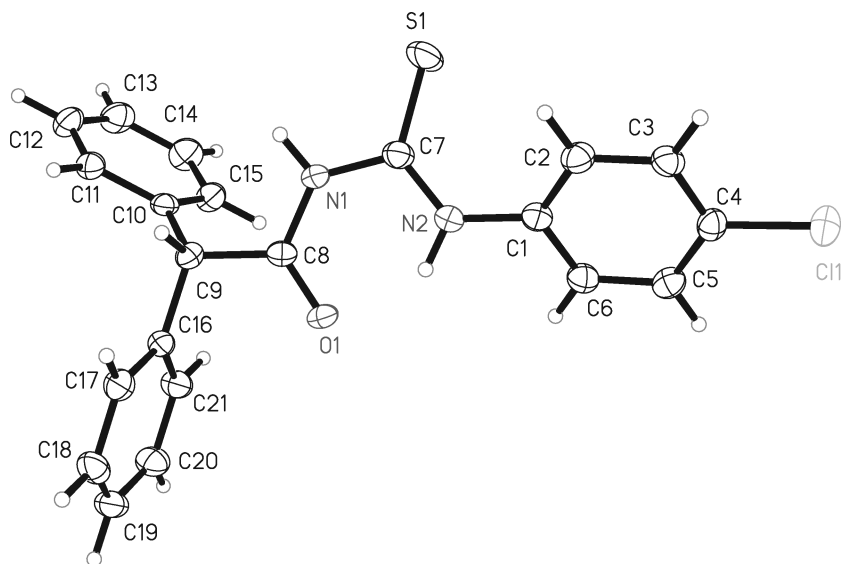


FIGURE 1 Molecular structure of **4**. Thermal ellipsoids are shown at the 50% probability level.

data agree with the results of FT-IR ATR investigations and single crystal X-ray diffraction.^{37,38}

The molecular structure of *N*-(2,2-diphenylacetyl)-*N'*-(4-chlorophenyl) thiourea is depicted in Figure 1. Selected bond lengths and angles are presented in Table I. All C-C bond lengths and angles of **4** are in the normal ranges.³⁹ The geometry of the central carbonyl thiourea group is also in agreement with those observed in substituted thiourea derivatives.^{32,33,40,41} The bond lengths of the carbonyl and thiocarbonyl groups in **4** are typical for double bonds, C8-O1 = 1.223(2) Å, C7-S1 = 1.6588(19) Å. The C-N bond lengths [N1-C8 = 1.370(2) Å, N1-C7 = 1.401(3) Å, N2-C7 = 1.335(3) Å, N2-C1 = 1.418(3) Å] differ significantly from one another but are shorter as compared with the typical value for a C-N single bond (1.479 Å).^{29,32,33,40-42} This information indicates partial electron delocalization within the C8-N1-C7-N2 fragment. The molecule adopts *cis-trans* configuration with respect to the position of the 4-chlorophenyl and 2,2-diphenylacetyl groups relative to the thiocarbonyl sulfur atom across the N1-C7 and N2-C7 bonds, respectively.⁴⁰⁻⁴² The conformation of the molecule with respect to the thiocarbonyl and carbonyl moieties is twisted, as reflected by the torsion angles O1-C8-N1-C7, C8-N1-C7-N2, and S1-C7-N1-C8 of 1.03°, -5.75°, and 173.09°, respectively.³¹⁻³³ The maximum deviation from the O1-C8-N1-C7-N2 plane is 0.035(2) Å.

TABLE I Selected Bond Lengths (Å) and Angles (°) of Compound 4

Bond Lengths			
O1-C8	1.223(2)	N2-C7	1.335(3)
C11-C4	1.738(2)	N2-C1	1.418(3)
S1-C7	1.659(2)	C8-C9	1.526(3)
N1-C8	1.370(2)	C9-C10	1.523(3)
N1-C7	1.401(3)	C9-C16	1.526(3)
Bond Angles			
C8-N1-C7	128.3(2)	O1-C8-N1	122.6(2)
C7-N2-C1	128.2(2)	O1-C8-C9	122.2(2)
N2-C7-N1	114.7(2)	N1-C8-C9	115.2(2)
N2-C7-S1	126.7(2)	C10-C9-C16	113.3(2)
N1-C7-S1	118.6(2)	C10-C9-C8	114.1(2)

However, the central carbonyl thiourea moiety (S1-C7-N1-N2-C8-O1) connecting the 4-chlorophenyl and 2,2-diphenylacetyl groups is almost planar, with atom O1 deviating by 0.070(2) Å. Intramolecular hydrogen bonding N2-H2...O1, N-H 0.90 Å, H...O 1.87 Å, N-H...O 139° results in the formation of a six-membered ring in the O1-C8-N1-C7-N2 plane (Figure 2). This intramolecular hydrogen bonding will influence the coordination behavior of the compounds. Thiourea derivatives such as *N,N*-dialkyl-*N'*-benzoylthiourea easily coordinate to a metal atom via both sulfur and oxygen atoms. However, for **4** and related compounds, it will be difficult for the carbonyl oxygen atom to participate in the coordination with transition metal atoms because it is involved in intramolecular hydrogen bonding. In the crystal structure, the molecules are linked by intermolecular hydrogen bonds: N1-H1...O1^{*i*}, with N-H 0.90 Å, H-O 2.02 Å, N-H-O 173° [symmetry code: (*i*) *x*, 1/2-*y*, 1/2+*z*] (Figure 2).

EXPERIMENTAL

Instrumentation

The room temperature attenuated total reflection Fourier transform infrared (FT-IR ATR) spectra of all synthesized compounds were recorded using a Varian FTS1000 FT-IR spectrometer with a Diamond/ZnSe prism (4000–525 cm⁻¹; number of scans: 250; resolution: 1 cm⁻¹). All ¹H NMR spectra were recorded with a Bruker DPX-400 spectrometer, using CDCl₃ as solvent and TMS as internal standard. C, H, and N analyses were carried out with a Carlo Erba MOD 1106 elemental analyzer. Melting points were determined on a digital melting point instrument

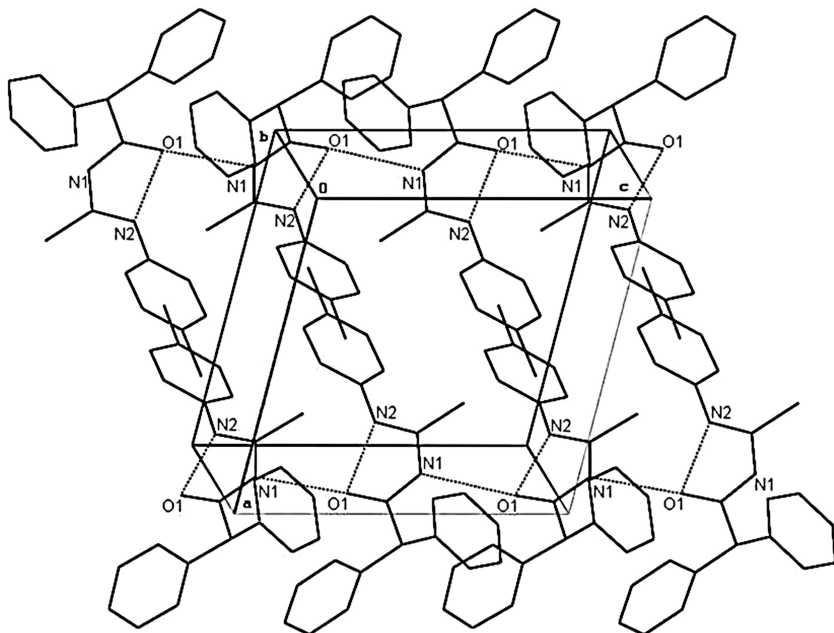


FIGURE 2 Packing diagram for **4** in the crystal, with hydrogen bonds as dotted lines; H atoms not involved are omitted.

(Electrothermal model 9200). Single crystal X-ray data were collected on a Rigaku Mercury AFC8S system with a Mercury CCD detector using graphite-monochromated MoK α radiation ($\lambda = 0.71073 \text{ \AA}$). The structure was solved by direct methods and refined using full-matrix least-squares techniques (on F^2).⁴³ Data were corrected for Lorentz and polarization effects and for absorption. The latter correction was made using REQABA, a multi-scan technique.⁴⁴ All non-hydrogen atoms were refined anisotropically. Further details concerning data collection and refinement are given in Table II.

Synthesis of Compounds 1–9: General Procedure

All chemicals used for the preparation were of reagent grade quality. Ethanol and acetone were distilled before use. The compounds were prepared by a procedure similar to that reported in the literature.^{31–33} A solution of 2,2-diphenylacetyl chloride (0.005 mol) in acetone (50 mL) was added dropwise to a suspension of potassium thiocyanate (0.005 mol) in acetone (50 mL). The reaction mixture was heated (35°C) under reflux for 30 min and then cooled to room temperature. A solution of

TABLE II Crystallographic Data and Refinement Parameters for Compound 4

Empirical formula	C ₂₁ H ₁₇ ClN ₂ OS
Formula weight	380.88
Temperature (K)	153(2)
Wavelength (Å)	0.71073
Crystal system	Monoclinic
Space group	P2 ₁ /c
Unit cell dimensions	
<i>a</i> (Å)	9.6551(19)
<i>b</i> (Å)	20.060(4)
<i>c</i> (Å)	9.894(2)
β (°)	104.29(3)
<i>V</i> (Å ³)	1857.0(6)
<i>Z</i>	4
<i>D_c</i> (g/cm ³)	1.362
Absorption coefficient (mm ⁻¹)	0.330
<i>F</i> (000)	792
Crystal size (mm ³)	0.36 × 0.29 × 0.19
θ range for data collection (°)	3.54 to 25.70
Index ranges	-11 ≤ <i>h</i> ≤ 11 -23 ≤ <i>k</i> ≤ 23 -9 ≤ <i>l</i> ≤ 11
Reflections collected	11273
Independent reflections (<i>R</i> _{int})	3280 (0.029)
Absorption correction	Multi scan
Refinement method	Full-matrix least-squares on <i>F</i> ²
Data/parameters	3280/235
Goodness-of-fit on <i>F</i> ²	1.065
Final <i>R</i> indices [<i>I</i> > 2σ(<i>I</i>)]	<i>R</i> 1 = 0.0394, <i>wR</i> 2 = 0.0941
<i>R</i> indices (all data)	<i>R</i> 1 = 0.0544, <i>wR</i> 2 = 0.1035
Largest diff. peak and hole (e.Å ⁻³)	0.370 and -0.470

the primary amine (0.005 mol) in acetone (30 mL) was added to the mixture during a period of 15 min at room temperature and stirred for 2 h. Hydrochloric acid (0.1 N, 300 mL) was added, and the solution was filtered. The solid product was washed with water and purified by recrystallization from ethanol:dichloromethane mixture (1:1).

N-(2,2-Diphenylacetyl)-N'-phenylthiourea (1)

White solid. Yield: 87% (1.5071 g). Mp: 160–162°C. Anal. Calcd for C₂₁H₁₈N₂OS: C 72.8; H 5.2; N 8.1. Found: C 72.4; H 5.2; N 8.0%. FT-IR (cm⁻¹): ν(N-H) 3254, 3179 (v), ν(C-H) 3061–3032 (v), ν(C=O) 1679 (s), ν(C=S) 1235 (s). ¹H NMR (CDCl₃): δ = 12.35 (s, 1H, CSNH), 11.85 (s, 1H, CONH), 7.65–7.23 (m, 15H, arom-H), 5.49 (s, 1H, CH).

***N*-(2,2-Diphenylacetyl)-*N'*-(2-chlorophenyl)-thiourea (2)**

White solid. Yield: 85% (1.6188 g). Mp: 134–136°C. Anal. Calcd for C₂₁H₁₇N₂O₂SCl: C 66.2; H 4.5; N 7.4. Found: C 66.1; H 4.5; N 7.3%. FT-IR (cm⁻¹): ν(N-H) 3262, 3212 (v), ν(C-H) 3060–3027 (v), ν(C=O) 1676 (s), ν(C=S) 1239 (s). ¹H NMR (CDCl₃): δ = 12.33 (s, 1H, CSNH), 11.99 (s, 1H, CONH), 8.01–7.28 (m, 14H, arom-H), 5.50 (s, 1H, CH).

***N*-(2,2-Diphenylacetyl)-*N'*-(3-chlorophenyl)-thiourea (3)**

White solid. Yield: 90% (1.7140 g). Mp: 165–167°C. Anal. Calcd for C₂₁H₁₇N₂O₂SCl: C 66.2; H 4.5; N 7.4. Found: C 66.1; H 4.5; N 7.2%. FT-IR (cm⁻¹): ν(N-H) 3252, 3168 (v), ν(C-H) 3056–3022 (v), ν(C=O) 1677 (s), ν(C=S) 1239 (s). ¹H NMR (CDCl₃): δ = 12.36 (s, 1H, CSNH), 11.90 (s, 1H, CONH), 7.89–7.30 (m, 14H, arom-H), 5.48 (s, 1H, CH).

***N*-(2,2-Diphenylacetyl)-*N'*-(4-chlorophenyl)-thiourea (4)**

White solid. Yield: 88% (1.6759 g). Mp: 135–137°C. Anal. Calcd for C₂₁H₁₇N₂O₂SCl: C 66.2; H 4.5; N 7.4. Found: C 65.9; H 4.4; N 7.3%. FT-IR (cm⁻¹): ν(N-H) 3264, 3156 (v), ν(C-H) 3064–3020 (v), ν(C=O) 1677 (s), ν(C=S) 1247 (s). ¹H NMR (CDCl₃): δ = 12.30 (s, 1H, CSNH), 11.90 (s, 1H, CONH), 7.66–7.28 (m, 14H, arom-H), 5.48 (s, 1H, CH).

***N*-(2,2-Diphenylacetyl)-*N'*-(2-methylphenyl)-thiourea (5)**

White solid. Yield: 87% (1.5680 g). Mp: 138–140°C. Anal. Calcd for C₂₂H₂₀N₂O₂S: C 73.3; H 5.6; N 7.8. Found: C 73.0; H 5.5; N 7.7%. FT-IR (cm⁻¹): ν(N-H) 3254, 3182 (v), ν(C-H) 3060–3032 (v), ν(C=O) 1673 (s), ν(C=S) 1243 (s). ¹H NMR (CDCl₃): δ = 12.00 (s, 1H, CSNH), 11.86 (s, 1H, CONH), 7.55–7.19 (m, 14H, arom-H), 5.49 (s, 1H, CH), 2.19 (s, 3H, CH₃).

***N*-(2,2-Diphenylacetyl)-*N'*-(4-methylphenyl)-thiourea (6)**

White solid. Yield: 90% (1.6221 g). Mp: 173–175°C. Anal. Calcd for C₂₂H₂₀N₂O₂S: C 73.3; H 5.6; N 7.8. Found: C 73.2; H 5.5; N 7.6%. FT-IR (cm⁻¹): ν(N-H) 3258, 3162 (v), ν(C-H) 3054–3031 (v), ν(C=O) 1677 (s), ν(C=S) 1243 (s). ¹H NMR (CDCl₃): δ = 12.30 (s, 1H, CSNH), 11.80 (s, 1H, CONH), 7.50–7.17 (m, 14H, arom-H), 5.49 (s, 1H, CH), 2.28 (s, 3H, CH₃).

***N*-(2,2-Diphenylacetyl)-*N'*-(3-methoxyphenyl)-thiourea (7)**

White solid. Yield: 89% (1.6753 g). Mp: 140–142°C. Anal. Calcd for C₂₂H₂₀N₂O₂S: C 70.2; H 5.4; N 7.4. Found: C 70.2; H 5.3; N 7.4%. FT-IR (cm⁻¹): ν(N-H) 3271, 3187 (v), ν(C-H) 3061–3029 (v), ν(C=O) 1682 (s), ν(C=S) 1264 (s). ¹H NMR (CDCl₃): δ = 12.39 (s, 1H, CSNH), 11.86 (s,

1H, CONH), 7.40–7.18 (m, 14H, arom-H), 5.49 (s, 1H, CH), 3.45 (s, 3H, CH₃).

N-(2,2-Diphenylacetyl)-N'-(4-methoxyphenyl)-thiourea (8)

White solid. Yield: 91% (1.7129 g). Mp: 143–145°C. Anal. Calcd for C₂₂H₂₀N₂O₂S: C 70.2; H 5.4; N 7.4. Found: C 70.2; H 5.3; N 7.3%. FT-IR (cm⁻¹): ν(N-H) 3237, 3181 (v), ν(C-H) 3058–3030 (v), ν(C=O) 1676 (s), ν(C=S) 1251 (s). ¹H NMR (CDCl₃): δ = 12.30 (s, 1H, CSNH), 11.80 (s, 1H, CONH), 7.50–7.17 (m, 14H, arom-H), 5.48 (s, 1H, CH), 2.28 (s, 3H, CH₃).

N-(2,2-Diphenylacetyl)-N'-(naphthalen-1-yl)-thiourea (9)

White solid. Yield: 94% (1.8636 g). Mp: 180–182°C. Anal. Calcd for C₂₅H₂₀N₂OS: C 75.7; H 5.1; N 7.1. Found: C 75.3; H 5.1; N 7.0%. FT-IR (cm⁻¹): ν(N-H) 3228, 3192 (v), ν(C-H) 3061–3018 (v), ν(C=O) 1689 (s), ν(C=S) 1243 (s). ¹H NMR (CDCl₃): δ = 12.42 (s, 1H, CSNH), 11.99 (s, 1H, CONH), 7.99–7.31 (m, 17H, arom-H), 5.57 (s, 1H, CH).

Supplementary Material

Crystallographic data for the structure reported in this article have been deposited at the Cambridge Crystallographic Data Centre (CCDC) with quotation number CCDC-662387 for compound **4** and can be obtained free of charge on application to CCDC 12 Union Road, Cambridge CB2 1EZ, UK [Fax: (internat.) + 44(1223)336-033, E-mail: deposit@ccdc.cam.ac.uk].

REFERENCES

- [1] S. Chuan-Wen and Z. Xiao-Dong, *Chin. J. Struct. Chem.*, **26**, 153 (2007).
- [2] Y. Bessard and R. Crettaz, *Tetrahedron*, **56**, 4739 (2000).
- [3] Y. F. Yuan, J. T. Wang, M. C. Gimeno, A. Laguna, and P. G. Jones, *Inorg. Chim. Acta*, **324**, 309 (2001).
- [4] Y. M. Zhang, T. B. Wei, L. Xian, and L. M. Gao, *Phosphorus, Sulfur, and Silicon*, **179**, 2007 (2004).
- [5] Y. M. Zhang, T. B. Wei, X. C. Wang, and S. Y. Yang, *Indian J. Chem.*, **B37**, 604 (1998).
- [6] T. K. Venkatachalam, C. Mao, and F. M. Uckun, *Bioorg. Med. Chem.*, **12**, 4275 (2004).
- [7] C. W. Sun, H. Huang, M. Q. Feng, X. L. Shi, X. D. Zhang, and P. Zhou, *Bioorg. Med. Chem. Lett.*, **1**, 162 (2006).
- [8] K. R. Koch, *Coord. Chem. Rev.*, **216–217**, 473 (2001).
- [9] P. Vest, M. Schuster, and K. H. Konig, *Fres. J. Anal. Chem.*, **341**, 566 (1991).
- [10] O. Foss, S. Husebye, K. W. Tornroos, and P. E. Fanwick, *Polyhedron*, **23**, 3021 (2004).
- [11] K. H. Konig, M. Schuster, G. Schneeweis, and B. Steinbrech, *Fres. J. Anal. Chem.*, **319**, 66 (1984).
- [12] P. Bombicz, I. Mutikainen, M. Krunks, T. Leskela, J. Madarasz, and L. Niinist, *Inorg. Chim. Acta*, **357**, 513 (2004).
- [13] T. Seshadri and H. J. Haupt, *J. Mater. Chem.*, **8**, 1345 (1998).

- [14] H. Arslan, N. Kulcu, and U. Florke, *Transition Met. Chem.*, **28**, 816 (2003).
- [15] G. Binzet, H. Arslan, U. Florke, N. Kulcu, and N. Duran, *J. Coord. Chem.*, **59**, 1395 (2006).
- [16] M. F. Emen, H. Arslan, N. Kulcu, U. Florke, and N. Duran, *Pol. J. Chem.*, **79**, 1615 (2005).
- [17] D. Ugur, H. Arslan, and N. Kulcu, *Russ. J. Coord. Chem.*, **32**, 669 (2006).
- [18] H. Arslan, N. Duran, N. O. Sahin, and N. Kulcu, *Asian J. Chem.*, **18**, 1710 (2006).
- [19] G. Avsar, N. Kulcu, and H. Arslan, *Turk. J. Chem.*, **26**, 607 (2002).
- [20] N. Ozpozan, H. Arslan, T. Ozpozan, M. Merdivan, and N. Kulcu, *J. Therm. Anal. Calorim.*, **61**, 955 (2000).
- [21] N. Ozpozan, H. Arslan, T. Ozpozan, N. Ozdes, and N. Kulcu, *Thermochim. Acta*, **343**, 127 (2000).
- [22] N. Ozpozan, T. Ozpozan, H. Arslan, F. Karipcin, and N. Kulcu, *Thermochim. Acta*, **336**, 97 (1999).
- [23] H. Arslan, U. Florke, and N. Kulcu, *Spectrochim. Acta A*, **67**, 936 (2007).
- [24] H. Arslan, F. M. Emen, and N. Kulcu, *Asian J. Chem.*, **19**, 1888 (2007).
- [25] H. Arslan, U. Florke, N. Kulcu, and G. Binzet, *Spectrochim. Acta A*, **68**, 1347 (2007).
- [26] H. Arslan, N. Kulcu, and U. Florke, *Spectrochimica Acta A*, **64**, 1065 (2006).
- [27] H. Arslan, U. Florke, N. Kulcu, and E. Kayhan, *Turk. J. Chem.*, **30**, 429 (2006).
- [28] H. Arslan, U. Florke, N. Kulcu, and M. F. Emen, *J. Coord. Chem.*, **59**, 223 (2006).
- [29] H. Arslan, U. Florke, and N. Kulcu, *J. Chem. Crystallogr.*, **33**, 919 (2003).
- [30] G. Avsar, H. Arslan, H. J. Haupt, and N. Kulcu, *Turk. J. Chem.*, **27**, 281 (2003).
- [31] T. Yesilkaynak, U. Florke, N. Kulcu, and H. Arslan, *Acta Crystallogr. E*, **62**, O3934 (2006).
- [32] H. Arslan, U. Florke, and N. Kulcu, *Turk. J. Chem.*, **28**, 673 (2004).
- [33] H. Arslan, U. Florke, and N. Kulcu, *Acta Chim. Slov.*, **51**, 787 (2004).
- [34] I. B. Douglass and F. B. Dains, *J. Am. Chem. Soc.*, **56**, 719 (1934).
- [35] C. Sacht and M. S. Datt, *Polyhedron*, **19**, 1347 (2000).
- [36] X. Shen, X. Shi, B. Kang, Y. Tong, Y. Liu, L. Gu, Q. Liu, and Y. Huang, *Polyhedron*, **18**, 33 (1998).
- [37] B. Q. Su, G. L. Liu, L. Sheng, X. Q. Wang, and L. Xian, *Phosphorus, Sulfur, and Silicon*, **181**, 745 (2006).
- [38] Z. Li, Y. Zhang, and Y. Wang, *Phosphorus, Sulfur, and Silicon*, **178**, 293 (2003).
- [39] F. H. Allen, O. Kennard, D. G. Watson, L. Brammer, A. G. Orpen, and R. Taylor, *J. Chem. Soc., Perkin Trans.*, **2**, S1 (1987).
- [40] B. M. Yamin and M. S. M. Yusof, *Acta Crystallogr. E*, **59**, O151 (2003).
- [41] M. S. M. Yusof, F. S. M. Asroh, M. A. Kadir, and B. M. Yamin, *Acta Crystallogr. E*, **63**, o1190 (2007).
- [42] M. K. Rauf, A. Badshah, and M. Bolte, *Acta Crystallogr. E*, **63**, O1676 (2007).
- [43] SHELXTL 6.10, *Bruker-AXS*, Madison, WS (2000).
- [44] R. A. Jacobson, *REQABA: Empirical Absorption Correction Version 1.1.*, Rigaku/Molecular Structure Corporation, The Woodlands, TX (1998).