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ORIGINAL ARTICLE

Synthesis and structural characterization of a gold(I) complex containing 1,3-dimethylcyanurate ligand

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Abstract The gold(I) complex [Au(DMC)(PPh_3)] (**1**) (DMC = 1,3-dimethylcyanurate) was synthesized and characterized by elemental analysis, FT-IR, ^{1}H, ^{13}C, ^{31}P NMR and FAB mass spectroscopies. The structure of the complex was determined by X-ray diffraction and crystallizes in the triclinic space group $P{-}1$ with $Z = 2$. The gold(I) ion in complex **1** is coordinated by DMC ligand and PPh_3 ligand, resulting in a slightly distorted linear structure. Molecules of **1** are connected *via* intermolecular C—H···O hydrogen bonds, which lead to the formation of supramolecular network. Other intramolecular interactions which include Au···O contacts [Au···O25 3.045(1) and Au···O29 3.164(1)Å] were observed.

1. Introduction

Cyanuric acid (CYH_3), which can exist in equilibrium with its tautomer either triol (a) or trione (b) forms, has played a diverse role in the oriented organic synthesis (Wojtowicz, 1993). The X-ray structures of its potassium, (Nichol et al., 2006; Sysova et al., 1990; Marsh et al., 2002) calcium (Lin et al., 1995; Chekhlov, 2006) and rubidium (Nichol et al., 2006) salts have been studied and suggested that the hard metals of these main group elements are affected by HSAB concept (Pearson, 1997) and coordinated to the cyanurate ligand at the deprotonated enol oxygen atom. On the other hand, soft and medium-hard transition metal complexes of cyanurates have also been prepared (Branzburg et al., 1986; Falvello et al., 1997, 1995; Palade and Nutiu, 1986; Shao et al., 1996; Sysova et al., 1991; Hart et al., 1992; Chen et al., 1995; Server et al., 1998; Wu et al., 2005; Yang et al., 2006; Yue et al., 2006; Zhang et al., 2006; Du and Kang, 2007;

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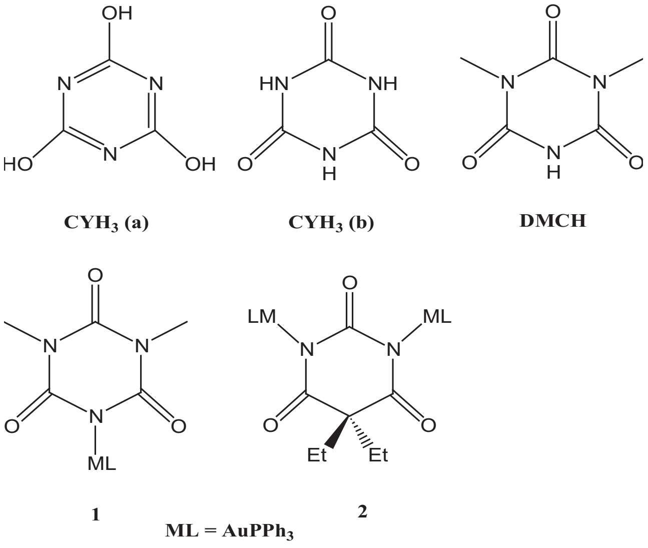
Sivashankar et al., 2000; Brunner et al., 2001), and the donor site of the deprotonated cyanurate anion CYH$_2^-$ is the deprotonated nitrogen atom. Furthermore di- and tri-anionic cyanurate ligands were found to be N,O-chelated (Chen et al., 1994; Müller, 1976; Elschenbroich et al., 2007; Pérez et al., 2007; Antiñolo et al., 2007; Cotton et al., 2003).

Among cyanurates, 1,3-dimethylcyanuric acid (DMCH, synonymous to 1,3-dimethyl-2,4,6-trioxo-1,3,5-triazine) was known since 1881 (Hofmann, 1881). The presence of several potential donor sites such as one amine nitrogen atom and three carbonyl oxygen atoms makes DMCH a very interesting polyfunctional ligand in coordination chemistry. By comparing with CYH$_3$, DMCH (Scheme 1) has two methyl groups which lower the tendency for forming intra- and intermolecular hydrogen bonding (Desiraju and Steiner, 1999). This has prompted us to investigate its coordination behavior. Proton loss from the amine nitrogen atom to form an amide is necessary for the complexation of the 1,3-dimethylcyanurate monoanion (DMC$^-$). The present work was undertaken as a part of a research project on the synthesis and structural characterization of metal complexes of 1,3-dimethylcyanurate, in our earlier work, we synthesized various metal-DMC complexes with different metal ions (Kuhn et al., 2008; Abu-Salem et al., 2008a,b, 2012). However, a literature survey indicates that few studies have concerned cyanuric acid complexes of group d^{10} metals (Kuhn et al., 2008; Abu-Salem et al., 2008a). To the best of our knowledge, anionic cyanurate ligands have rarely been used with gold(I) and hardly any chemistry has been described. Gold(I) complexes with N-donor ligands are much less common than those with P-donor ligands, the affinity of gold for nitrogen can be increased if a phosphine ligand is attached to gold, because of the efficient π-acceptor nature of the phosphine (Grohman and Schmidbaur, 1995). In the course of our studies on cyanurate–metal ion interactions, we have succeeded in preparing the first new DMC-gold(I) complex containing an ancillary triphenylphosphine ligand. Herein, we report the synthesis, characterization and crystal structure of [Au(DMC)(PPh$_3$)] (**1**).



Scheme 1 Triazine rings, complexes **1** and **2**.

2. Experimental section

2.1. General remarks

Lithium 1,3-dimethylcyanurate (LiDMC) was obtained according to a published procedure (Kuhn et al., 2008). Other chemicals were purchased by commercial providers (Aldrich Company) and used as received.

2.2. Physical measurements

The elemental analysis for C, H and N was performed using a Carlo Erba 1106 elemental analyzer. IR spectra were recorded on a VERTEX 70 FT-IR spectrometer using the drift spectroscopy method in the range 4000–400 cm^{-1}. The high resolution NMR spectra were acquired by a Bruker DRX 400 NMR spectrometer (^{1}H 400 MHz; ^{13}C 100 MHz, using TMS as external standard; ^{31}P 162 MHz, using 85% H$_3$PO$_4$ as external standard). FAB mass spectra were carried out on a TSQ 70 quadrupol mass spectrometer with xenon atoms as bombarding particles (kinetic energy ca. 8 keV). Unless otherwise stated, 3-nitrobenzyl alcohol (3-NBA) was used as matrix material at an ion source temperature of *ca.* 60 °C.

2.3. Synthesis of [Au(DMC)(PPh$_3$)] (1)

To a solution of LiDMC (82.0 mg, 0.503 mmol) in methanol (30 mL) Ph$_3$PAuCl (0.250 g, 0.503 mmol) was added. The mixture was warmed to 50 °C and stirred for 4 h to yield a white precipitate. Then the mixture was stirred overnight at room temperature and evaporated to dryness under reduced pressure, the product was extracted with dichloromethane (30 mL) and filtered. The filtrate was reduced in volume to *ca.* 5 mL and light petroleum (25 mL) was added. After one day, white crystals at 32 °C were obtained and were suitable for X-ray diffraction. Yield: 68%. Anal. Calc. for C$_{23}$H$_{21}$AuN$_3$O$_3$P (615.37 g/mol): C, 44.89; H, 3.44; N, 6.83. Found: C, 44.59; H, 3.14; N, 6.46%. ^{1}H NMR (400 MHz, CDCl$_3$): δ = 3.27 (s, 6 H, NCH$_3$), 7.37–7.54 (m, 15 H, Ph) ppm. ^{13}C NMR (100 MHz, CDCl$_3$): δ = 29.62 (CH$_3$), 128.66 (J = 19.0), 129.66 (J = 11.6), 132.63, 134.63 (J = 14.0) (Ph), 152.49 (C$^2_{DMC}$), 155.43 (C$^{4,6}_{DMC}$) ppm. ^{31}P NMR (162 MHz, CDCl$_3$): δ = 31.73 (s) ppm. IR(DRIFT): ν_{CO} = 1727 (vs), 1714 (s), 1641 (vs) cm^{-1}. MS [FAB, (3-NBA matrix)]: m/z (%) = 1074.0 (8%) [M + AuPPh$_3$]$^+$, 721.0 (7%) [(PPh$_3$)$_2$Au]$^+$, 616.0 (46%) [M + H]$^+$, 458.9 (100%) [M – DMC].

2.4. Solid-state structure of [Au(DMC)(PPh$_3$)] (1)

Crystals of compound **1** were grown from a solution of dichloromethane and light petroleum at −32 °C. The crystal of **1** was mounted on a glass fiber with epoxy cement at room temperature, and cooled in a cold stream of liquid nitrogen. Intensity data of the complex were collected using a STOE IPDS 2 diffractometer with graphite-monochromated MoK_α radiation (λ = 0.71073 Å). The structure was solved by direct methods and refined by full-matrix least-square procedures on F^2 with the SHELX-97 and SHELXL-97 programs (Sheldrick, 1997). All non-hydrogen atoms were refined anisotropically and were added on calculated positions. The details of data collection, refinement and crystallographic data are summarized in Table 1.

3. Results and discussion

3.1. Synthesis and characterization

Complex **1** was synthesized by the ligand displacement of 1:1 ratio in stoichiometric reaction upon mixing of the precursor [PPh_3AuCl] with the anhydrous lithium salt of 1,3-dimethylcyanuric acid, Li[DMC] (Abu-Salem, 2009). The complex was isolated as colorless crystals, air-stable solid and non-hygroscopic at room temperature in moderate yield (over 50%), which can be isolated in pure state from the mixture of products by recrystallization from chlorinated solvents such as light petroleum. It is soluble in most organic solvents at room temperature.

Complex **1** was characterized by elemental analysis and IR spectroscopy. The molecular structure of **1** in the solid state was determined by the single X-ray structure analysis.

Table 1 Crystal data and structure refinement for **1**.

Empirical formula	$C_{23}H_{21}AuN_3O_3P$
Formula weight	615.36
Temperature, K	173(2) K
λ, Å	0.71073
Crystal system	Triclinic
Space group	$P-1$
a (Å)	9.2791(9)
b (Å)	10.8619(11)
c (Å)	12.5511(12)
α (°)	96.456(8)
β (°)	109.949(7)
γ (°)	107.048(8)
Volume, $Å^3$	1104.63(19)
Z	2
D_{calcd}, g/cm^3	1.850
μ (MoK_α) (mm^{-1})	6.761
F(000), e	596
Crystal size (mm^3)	0.20 × 0.10 × 0.05
Θ, deg	3.42–26.37
Index ranges	$-11 \leqslant h \leqslant 11$, $-13 \leqslant k \leqslant 13$, $-15 \leqslant l \leqslant 15$
Reflections collected	15,963
Independent reflections	4503
Refinement method	Full-matrix least-squares on F^2
Data parameters	4503/283
Goodness-of-fit on F^2	1.035
Final $R_1/wR2$ [$I \geqslant 2\sigma(I)$]	0.0227/0.0492
Final $R_1/wR2$ (all data)	0.0263/0.0503
$\Delta\rho_{fin}$ (max/min), e·$Å^{-3}$	+0.751/−1.021

Table 2 Selected FTIR spectral data[a] for **1**.

Assignment	1	LiDMC[16]
ν(CH)	3057m, 3026w, 2989w, 2949w	3020m, 3002w, 2964m
ν(CO)	1727vs, 1714s, 1641vs	1729vs, 1651vs, 1635sh
ν(CC)	1572w, 1527w	
ν(C=N)	1587w	1558s
ν(CH)	1482s	1460s
ν(CN)	1257s	1275s

[a] Frequencies in cm^{-1}; m: medium; w: weak; vs: very strong; s: strong; sh: shoulder.

Selected FT-IR spectroscopic data for **1** are listed in Table 2, together with the data for the lithium salt of 1,3-dimethylcyanuratic acid. The medium and weak bands in the 2949–3057 cm^{-1} range are characteristic of aromatic and aliphatic ν(C—H) vibrations. The gold complex of DMC is well characterized by the presence of sharp IR bands due to the absorption of the carbonyl groups. The DMC ligand exists in triketo form, as indicated by three types of strong carbonyl absorption bands, observed in the frequency range 1641–1727 cm^{-1}. The position of the absorption band at 1558 cm^{-1} of C=N in 1,3-dimethylcyanurate lithium salt (Abu-Salem, 2009) as a result of the enolization process, did show a little change upon complexation and shifted to a higher wave number at1587 cm^{-1}, this is also a supportive of the metal nitrogen complex formation. This assignment appears reasonable, because in the case of gold(I) complexes with pyridine and its derivatives such vibrations are often found in this spectral region (Munakata et al., 1997).

The weak bands in the frequency range 1527–1572 cm^{-1} can be assigned to C=C vibrations of the phenyl groups. The band with medium intensity at 1482 cm^{-1} correspond to the ν(C—H) deformation vibration and the strong band centered around 1257 cm^{-1} is attributed to the ν(C—N) stretching vibrations (Levi and Hubley, 1956).

The ^{31}P NMR spectrum of **1**, measured at room temperature in $CDCl_3$ has displayed only one resonance at 31.73 ppm, the chemical shift of which is in agreement with

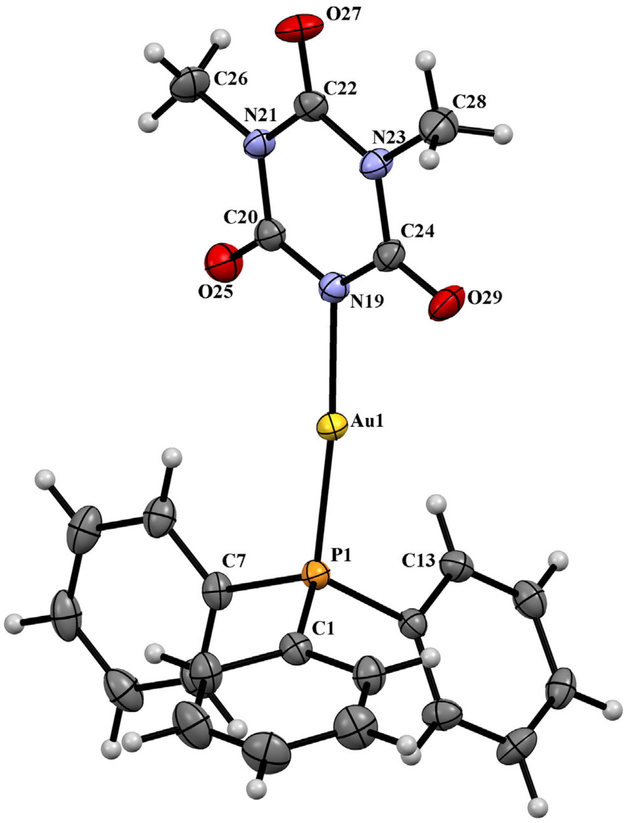


Figure 1 Molecular structure of 1 with the atomic numbering scheme. Thermal ellipsoids drawn at the 50% probability level.

the data for the PPh_3 ligand, coordinated to the gold(I) center and can be compared with those of related gold(I) complexes; e.g. at 33.40 ppm for $[AuCl(PPh_3)]$ (Nomiya et al., 1998) and at 32.84 ppm for $[1,3\text{-}(Ph_3PAu)_2(debarb)]$ (**2**) (debarb: 5,5-diethylbarbiturate) (Bonati et al., 1986).

The ^1H NMR spectrum of complex **1** has displayed one signal for the methyl groups at 3.27 ppm and multiplets in the range 7.37–7.54 ppm for the phenyl ring protons.

The ^{13}C NMR spectrum of **1** has displayed, in addition to the expected resonances for the PPh_3 ligand, three signals one for the methyl groups at 29.62 ppm and two for the carbonyl groups in an approximate 2:1 intensity ratio. The more intense resonance for the two carbonyl groups near the N atom (deprotonation site) was observed downfield (155.43 ppm) to the smaller signal assigned to the carbonyl group close to both the *N*-methyl groups at 152.49 ppm.

The fast-atom bombardment (FAB) mass spectrum of **1** in 3-nitrobenzylalcohol matrix shows a relatively medium parent (protonated) molecular ion $[(PPh_3)AuDMC + H]^+$ at m/z 616.4 (in 46% relative intensity). However, the cation of the highest mass is the dinuclear species $[(PPh_3)_2Au_2DMC]^+$ at m/z 1074.6 (8%) indicating the stability of the diaurated cation (Thwaite et al., 2004). $[(PPh_3)_2Au]^+$ (m/z 721.0, 7%) is the product of ligand redistribution, and $[(PPh_3)Au]^+$ is the most abundant ion at m/z 458.9 (100%).

3.2. Molecular structure of 1,3-dimethylcyanurate(triphenylphosphine) gold(I)

The molecular structure of complex **1** with the atom labeling is shown in Fig. 1. Selected bond distances and angles are listed in Table 3. Complex **1** crystallizes in the triclinic space group $P{-}1$, with $Z = 2$ formula units in the unit cell. There are no solvent molecules in the crystal.

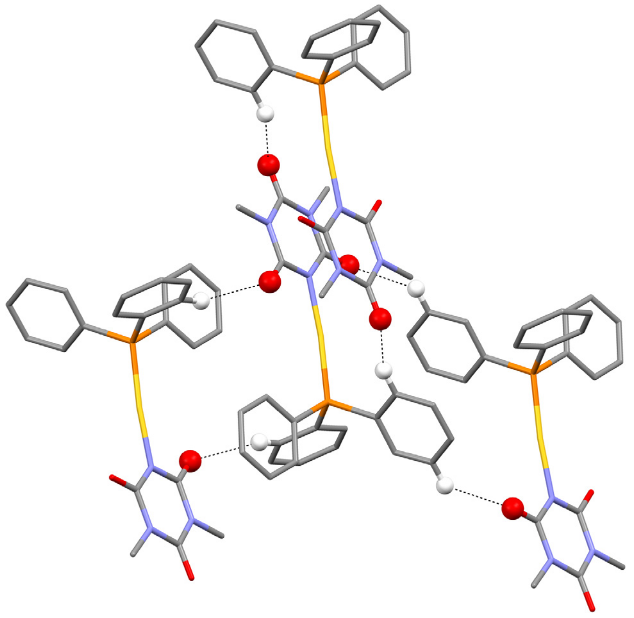


Figure 2 Each tetramer is built *via* six C—H···O interactions (three types of C—H···O). Contact atoms are shown in ball representation (Other C—H atoms were omitted for clarity).

The neutral complex **1** consisting of the $AuN(PPh_3)$ core coordinated by a monodentate *N*-bonded 1,3-dimethylcyanurate anion, in which the geometry around the gold(I) atom is described as distorted linear coordination with a P—Au—N angle of 174.59(8)°. This is obviously related to the steric effect of the DMC ligand. In fact the P—Au—N angle [179.3(2)°] in $[Au(PPh_3)NMe_3]^+$ having NMe_3 ligand of small steric hindrance is almost linear (Munakata et al.,1997). The Au—N

Table 3 Selected bond lengths (Å) and angles (°), and hydrogen bonding geometry for **1**.[a]

Bond lengths				
Au1—N19	2.063(3)	P—C	1.814–1.816(4)	
Au1—P1	2.228(9)	C20—O25	1.209(5)	
N19—C20	1.364(5)	C22—O27	1.208(4)	
N19—C24	1.369(5)	C24—O29	1.213(5)	
C20—N21	1.400(4)	N21—C26	1.468(5)	
C22—N21	1.380(5)	N23—C28	1.463(5)	
N23—C24	1.393(4)	C22—N23	1.376(5)	
Bond angles				
C24—N19—C20	123.5(3)	C24—N19—Au1	120.3(2)	
N21—C20—N19	116.5(3)	O(25)—C(20)—N(19)	122.9(3)	
N21—C22—N23	115.1(3)	O(25)—C(20)—N(21)	120.6(3)	
C22—N23—C24	124.3(3)	O(27)—C(22)—N(23)	122.6(4)	
N23—C24—N19	116.6(3)	O(27)—C(22)—N(21)	122.3(3)	
C20—N21—C22	124.3(3)	O(29)—C(24)—N(19)	122.6(3)	
N19—Au1—P1	174.59(8)	O(29)—C(24)—N(23)	120.8(3)	
C20—N19—Au1	115.7(2)	Au1—P1—C1	114.4(11)	
Au1—P1—C7	110.59(12)	Au1—P1—C13	113.65(11)	
D—H···A	D—H (Å)	H···A (Å)	D···A (Å)	D—H···A (°)
C2—H2^{i}···O27	0.95	2.301(1)	3.162	151
C5—H5^{ii}···O29	0.95	2.431(1)	3.131	130
C10—H10^{iii}···O29	0.95	2.529(1)	3.463	167
C18—H18^{iv}···O25	0.95	2.584(1)	3.446	151

[a] Symmetry operations: (i) $2-x, -y, 2-z$; (ii) $1-x, -y, 1-z$; (iii) $-1+x, -1+y, z$; (iv) $2-x, 1-y, 2-z$.

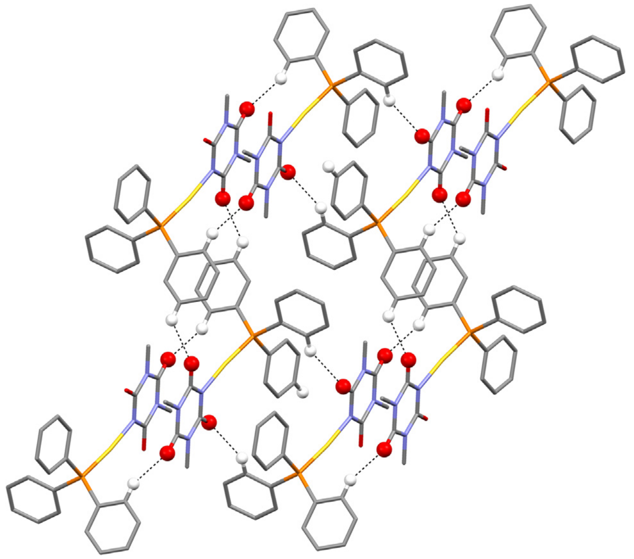


Figure 3 Partial packing diagram showing the two tetramers connected *via* additional C—H···O interactions. Contact atoms are shown in balls representation (Other C—H atoms were omitted for clarity).

bond distance, 2.063(3) Å, in **1** is longer than that in the corresponding barbiturate complex **2** [2.022(12) Å] containing a P—Au^{I}—N linkage, while the Au—P bond distance, 2.228(8) Å, is in line with the value found for the related complex **2** [2.233(5) Å] (Bonati et al., 1986).

The *van der Waals* radii of gold and oxygen are 1.70 and 1.50 Å, respectively (Huheey, 1983), and the deviation of P—Au—N angle from the ideal 180°, as discussed above, can be attributed to the close approach of the two oxygen atoms O29 and O25 to gold as weak interactions. The structure contains intramolecular Au···O interactions [Au1···O29 3.164(1) Å, Au1···O25 3.045(1) Å] that fall in a range lower than the sum of *van der Waals* radii, and exert a great influence on the magnitude of the P—Au—N angle. Evidence is also found for the narrowing of Au—N19—C20 angle [115.7(2)°] with concomitant widening of the Au—N19—C24 angle [120.3(2)°]. The Au···O bond distances are much longer than Au1—N19, 2.063(3) Å, bond distance, further indication of weak interaction of the two carbonyl groups of DMC. The maximization of the Au···O distances leads to the deviation of gold(I) ion (±0.295(1) Å) out of the mean plane passing through the triazine ring.

The C—N bond distances at the deprotonation site, C20—N19, 1.364(5), and N19—C24, 1.369(5) Å are as expected for sp^2-hybridized C—N bonds (Bryndza and Tam, 1988), and are almost equal resulting in a symmetric π-electron distribution in the sense of an effective delocalization within the triazine ring. Also the exocyclic C—O bond distances, 1.209(5), 1.212(4) and 1.208(4) Å, in **1** are little affected as a result of ligand coordination and hydrogen bond association.

In the phosphine ligand the P—C distances and the C—P—C or Au—P—C angles are in the range reported for gold(I) phosphine complex **2** (Bonati et al., 1986). However, it should be noted that of the three Au—P—C angles that involve C7, Au—P—C7 is smaller than the other two: 110.59(12)° compared with 113.65(11)° and 114.4(11)°.

The triazine ring, the two methyl groups and the three carbonyl groups in complex **1** are approximately planar from the mean plane passing through the triazine ring (and including N21, N23, N19).

3.3. Supramolecular structure in 1,3-dimethylcyanurate(triphenylphosphine) gold(I)

The possibility of stabilization by a stacking interaction between one heterocyclic ligand and one of the phenyl rings in the PPh$_3$ group has been suggested (Nomiya et al., 2000). The examination of the packing diagram for **1** reveals a three-dimensional network. The building unit of this network composed of tetramer units of molecules assembled through hydrogen bonding interactions (see Table 3). Within these units the molecules are connected *via* three types of C—H···O hydrogen bonding, involving hydrogen atoms of the CH groups from aromatic rings and the carbonyl groups of the DMC anion, [(Ph)C—H···O(DMC) of 2.301(1), 2.431(1) and 2.584(1) Å] as shown in Fig. 2. These interactions and the symmetry related ones result in the formation of two tetrameric units of eight molecules, Fig. 3. These supramolecular arrays are also further connected *via* other hydrogen bonding interactions [C10—H10···O29; 2.529(1) Å], and help to reinforcing the three-dimensional network as shown in Fig. 4. In complex **1**, containing a closely 1,3-dimethylcyanurate ring, it should be noted that the aurophilic interactions were absent, the lowest value for this separation being 6.79(1) Å.

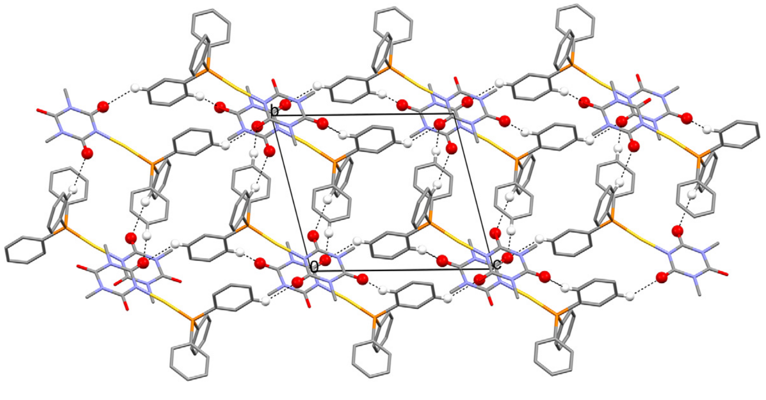


Figure 4 Crystal packing diagram viewed down the *b* crystallographic axis showing the overall three dimensional network structure of the title compound. Contact atoms are shown in balls representation (Other C—H atoms were omitted for clarity).

4. Conclusions

The 1,3-dimethylcyanurate ion (DMC) has the ability to form stable complex with soft metal center in low oxidation state similar to pyridine and 5,5-diethylbarbiturate (debarb) ligands. The stability of complex **1** is thus a reflection of the donor properties of the N-DMC ligand both from steric and electronic effects. Apparently, the location of partial negative charge at the amide nitrogen atom of DMC is compensated by the presence of strongly electronegative carbonyl groups. The tendency for gold(I) ions to aggregate in the solid state is expected to contribute to the formation of supramolecular structures through hydrogen bonds. In contrast with the previous studies, the complexes given in reference Abu-Salem et al., 2012 and including a Pt(II) complex does not form such a network.

5. Supplementary data

CCDC 906233 contains the supplementary crystallographic data for the compound reported in this communication. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre *via* http://www.ccdc.cam.ac.uk/conts/retrieving.html, or from the Cambridge Crystallographic Data Centre, 12 Union Road, Cambridge CB2 1EZ, UK; fax: (+44) 1223-336-033; or e-mail: deposit@ccdc.cam.ac.uk.

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