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Key indicators

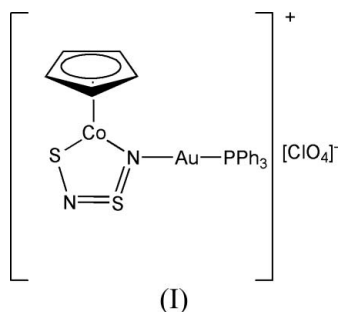
Single-crystal X-ray study
 $T = 133$ K
Mean $\sigma(\text{C}-\text{C}) = 0.007$ Å
 R factor = 0.030
 wR factor = 0.054
Data-to-parameter ratio = 14.9For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.**($1\eta^5$ -Cyclopentadienyl)(μ -disulfur dinitrido)(triphenyl-
phosphino- $2\kappa P$)cobalt(II)gold(I) perchlorate**The title compound, $[\{\text{Au}(\text{C}_{18}\text{H}_{15}\text{P})\}\text{Co}(\text{C}_5\text{H}_5)(\text{N}_2\text{S}_2)]\text{ClO}_4$, has a planar CoS_2N_2 ring and a close-to-linear $\text{N}-\text{Au}-\text{P}$ angle $[176.54(11)^\circ]$.

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Comment

The disulfur dinitride dianion is not known as a simple species but can be isolated in metal complexes (Kelly & Woollins, 1986; Jones *et al.*, 1985*a,b*; Bates *et al.*, 1986). These complexes may be protonated at the metal-coordinated N (Jones *et al.*, 1986) and we have previously commented on the structural consequences of this protonation (Jones *et al.*, 1987, 1988). Recently, we developed a new route to disulfur dinitrido complexes (Aucott *et al.*, 2002) and we examined the metallation of IrS_2N_2 rings using the AuPR_3 cation as a species which is isolobal with a proton (Aucott *et al.*, 2003). A comparison of metallacycle bond lengths for $[(\eta^5\text{-C}_5\text{Me}_5)\text{Ir}(\text{S}_2\text{N}_2)]$ and $[(\text{C}_5\text{Me}_5)_2\text{Ir}_2(\text{S}_2\text{N}_2)\text{Cl}(\text{PPh}_3)][\text{PF}_6]$ indicates that metallation appears to change the IrS_2N_2 bond lengths and angles in a similar fashion to protonation: both enlarge the $\text{M}-\text{S}_2$, N_1-S_1 and N_2-S_2 distances. We have also recently carried out detailed studies of CpCoS_2N_2 (Van Droogenbroeck *et al.*, 2005). This led us to synthesize the title compound, (I), in order to allow us to investigate the effects of metallation on the CoS_2N_2 ring.



Compound (I) (Fig. 1) has a planar CoS_2N_2 ring and a close-to-linear $\text{N}-\text{Au}-\text{P}$ angle $[176.54(11)^\circ]$. Compared with the non-metallated parent, CpCoS_2N_2 (Van Droogenbroeck *et al.*, 2005), we note that (I) has statistically invariant $\text{Co}-\text{N}$, $\text{Co}-\text{S}$ and S_2-N_2 distances, whilst the N_1-S_1 distance is longer in (I) than in the parent compound $[1.599(4)$ versus $1.556(1)$ Å] and the S_1-N_2 distance is slightly shorter in (I) than in the parent molecule $[1.580(4)$ versus $1.597(2)$ Å]. Within the CoS_2N_2 ring, it is noticeable that metallation results in an almost perfect trigonal $\text{Co}-\text{N}-\text{S}$ internal angle $[120.1(2)^\circ$ in (I) versus $118.32(8)^\circ$ in the parent compound]. In general, all internal angles in the CoS_2N_2 ring in (I) are closer to the idealized tetrahedral values at S and trigonal values at N

compared with the parent molecule. This work illustrates the difficulties in rationalizing bond lengths in S–N compounds and the continuing need for structural work in this area.

Experimental

Triphenylphosphinogold(disulfur dinitrido)(cyclopentadienyl)cobalt(II) perchlorate was prepared as described in the literature (Aucott *et al.*, 2003) and was crystallized by vapour diffusion of diethyl ether into a dichloromethane solution, to give small dark-reddish–violet plates.

Crystal data

[AuCo(C ₅ H ₅)(N ₂ N ₂)(C ₁₈ H ₁₅ P)]·ClO ₄	$V = 2591.2 (11) \text{ \AA}^3$
$M_r = 774.85$	$Z = 4$
Monoclinic, $P2_1/c$	$D_x = 1.986 \text{ Mg m}^{-3}$
$a = 14.646 (3) \text{ \AA}$	Mo $K\alpha$ radiation
$b = 14.186 (3) \text{ \AA}$	$\mu = 6.65 \text{ mm}^{-1}$
$c = 13.377 (3) \text{ \AA}$	$T = 133 (2) \text{ K}$
$\beta = 111.20 (3)^\circ$	Block, red-violet
	$0.26 \times 0.25 \times 0.20 \text{ mm}$

Data collection

Rigaku SCXmini diffractometer	15021 measured reflections
ω scans	4718 independent reflections
Absorption correction: multi-scan (ABSCOR; Higashi, 1995)	3953 reflections with $I > 2\sigma(I)$
$T_{\min} = 0.203$, $T_{\max} = 0.274$	$R_{\text{int}} = 0.048$
	$\theta_{\text{max}} = 25.4^\circ$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0176P)^2 + 3.2524P]$
$R[F^2 > 2\sigma(F^2)] = 0.030$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.054$	$(\Delta/\sigma)_{\text{max}} = 0.007$
$S = 1.04$	$\Delta\rho_{\text{max}} = 0.70 \text{ e \AA}^{-3}$
4718 reflections	$\Delta\rho_{\text{min}} = -0.77 \text{ e \AA}^{-3}$
317 parameters	
H-atom parameters constrained	

All H atoms were included in calculated positions and refined as riding, with C–H = 0.95 Å and with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$.

Data collection: *SCXmini Benchtop Crystallography System Software* (Rigaku, 2006); cell refinement: *PROCESS-AUTO* (Rigaku, 1998); data reduction: *PROCESS-AUTO*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *CrystalStructure* (Rigaku/MS, 2004); software used to prepare material for publication: *CrystalStructure*.

References

Aucott, S. M., Bhattacharyya, P., Milton, H. L., Slawin, A. M. Z. & Woollins, J. D. (2003). *New J. Chem.* **27**, 1466–1469.

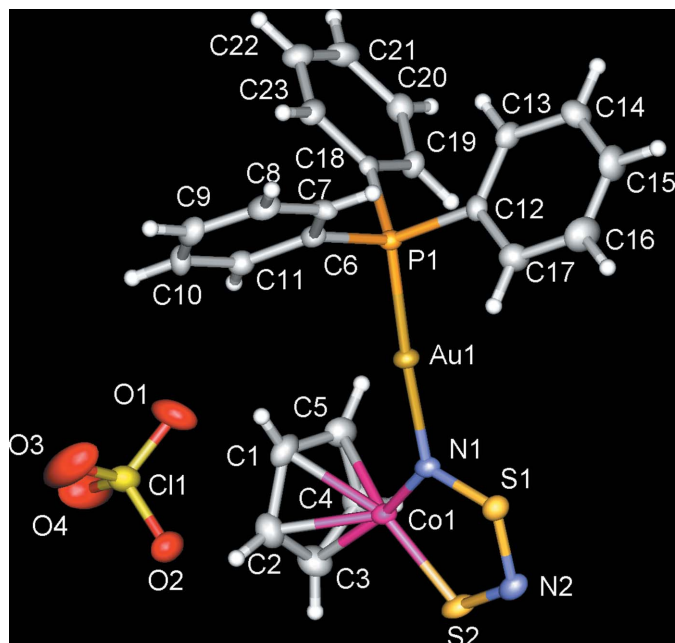


Figure 1

The structure and atom-labelling scheme for (I), with displacement ellipsoids drawn at the 50% probability level.

- Aucott, S. M., Slawin, A. M. Z. & Woollins, J. D. (2002). *Can. J. Chem.* **80**, 1481–1487.
- Bates, P. A., Hursthouse, M. B., Kelly, P. F. & Woollins, J. D. (1986). *J. Chem. Soc. Dalton Trans.* pp. 2367–2370.
- Higashi, T. (1995). *ABSCOR*. Rigaku Corporation, Tokyo, Japan.
- Jones, R., Kelly, P. F., Warrens, C. P., Williams, D. J. & Woollins, J. D. (1986). *J. Chem. Soc. Chem. Commun.* pp. 711–713.
- Jones, R., Kelly, P. F., Williams, D. J. & Woollins, J. D. (1985a). *J. Chem. Soc. Chem. Commun.* pp. 1325–1326.
- Jones, R., Kelly, P. F., Williams, D. J. & Woollins, J. D. (1985b). *Polyhedron*, **4**, 1947–1950.
- Jones, R., Kelly, P. F., Williams, D. J. & Woollins, J. D. (1988). *J. Chem. Soc. Dalton Trans.* pp. 803–807.
- Jones, R., Warrens, C. P., Williams, D. J. & Woollins, J. D. (1987). *J. Chem. Soc. Dalton Trans.* pp. 907–914.
- Kelly, P. F. & Woollins, J. D. (1986). *Polyhedron*, **5**, 607–632.
- Rigaku (1998). *PROCESS-AUTO*. Rigaku Corporation, 3-9-12 Matsubara, Akishima, Tokyo 196-8666, Japan.
- Rigaku (2006). *SCXmini Benchtop Crystallography System Software*. Version 1.0. Rigaku Americas Corporation, 9009 New Trails Drive, The Woodlands, TX 77381-5209, USA.
- Rigaku/MS (2004). *CrystalStructure*. Version 3.6.0. Rigaku/MS, 9009 New Trails Drive, The Woodlands, TX 77381-5209, USA.
- Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
- Van Droogenbroeck, J., Van Alsenoy, C., Aucott, S. M., Woollins, J. D., Hunter, A. D. & Blockhuys, F. (2005). *Organometallics*, **24**, 1004–1011.