PROUT, C. K., ARMSTRONG, R. A., CARRUTHERS, J. R., FORREST, J. G., MURRAY-RUST, P. & ROSSOTTI, F. J. C. (1968). *J. Chem. Soc. A*, pp. 2791–2813.

STEWART, R. F., DAVIDSON, E. R. & SIMPSON, W. T. (1965). *J. Chem. Phys.* **42**, 3175–3187.
WALKER, N. & STUART, D. (1983). *Acta Cryst.* A39, 158–166.

cis-Dichloro[2-(4-ethoxyphenyltelluro)ethyl methyl sulfide-*S,Te*]platinum(II), cis-PtCl₂[Te(C₆H₄OC₂H₅)CH₂CH₂(CH₃)S]

BY AJAI K. SINGH*, VINOD SRIVASTAVA AND S. K. DHINGRA

Department of Chemistry, Indian Institute of Technology, New Delhi-110016, *India*

AND JOHN E. DRAKE* AND JANE H. E. BAILEY

Department of Chemistry and Biochemistry, University of Windsor, Windsor, Ontario, Canada N9B 3P4

Abstract. [PtCl₂(C₁₁H₁₆OSTe)], M_r = 589.9, triclinic, $P\bar{1}$, a = 9.185 (4), b = 11.179 (2), c = 8.396 (2) Å, α = 103.82 (2), β = 105.49 (3), γ = 99.38 (3)°, V = 783 (1) Å³, Z = 2, D_m = 2.48, D_x = 2.50 Mg m⁻³, λ(Mo $K\alpha$) = 0.71069 Å, μ = 11.346 mm⁻¹, $F(000)$ = 540, T = 297 (1) K, final R = 0.0345, wR = 0.0371 for 1937 unique observed reflections. The structure contains square-planar Pt with the Pt—Cl bond lengths being typical of those *trans* to a π-bonding ligand. The bond *trans* to Te is very slightly longer, 2.336 (3) Å, than that *trans* to S, 2.324 (4) Å. The Pt—Te bond is shorter, 2.514 (1) Å, than those reported previously where the Te atom was *trans* to another Te or to a P atom.

Introduction. An interest in the chemistry of Te compounds coupled with the recognition that, to our knowledge, there were only two reported structures of compounds containing a Pt—Te bond (Gysling & Luss, 1984; Kelly, Slawin, Williams & Woolins, 1990) led to this work describing the structure of cis-PtCl₂[Te(C₆H₄OC₂H₅)CH₂CH₂(CH₃)S], a complex of a hybrid organotellurium ligand of (Te,S) type. Only recently has the ligand chemistry of a few multidentate hybrid Te donors been investigated (Singh, Srivastava & Khandelwal, 1990; Singh & Srivastava, 1990*a,b*). Like their P analogues, such ligands can generate interesting chemistry and the 2-(4-ethoxyphenyltelluro)ethyl methyl sulfide present in this Pt complex is one of the two examples of the (Te,S) type of ligand investigated to date.

Experimental. Preparation as described recently (Singh & Srivastava, 1990*c*) by reaction of

---

* To whom correspondence should be addressed.

C₂H₅OC₆H₄TeCH₂CH₂SCH₃ with K₂PtCl₄ in an acetone/water solution followed by slow evaporation of CH₃CN solution; density measured by flotation; crystal 0.31 × 0.47 × 0.37 mm; Rigaku AFC-6S diffractometer, highly oriented graphite monochromator; cell parameters from 25 strong reflections (35.90 < 2θ < 40.86°); data collected at 297 (1) K using the ω–2θ scan technique to a maximum 2θ value of 50.0°; ω scans had an average width at half height of 0.31° with a take-off angle of 6.0°; scans of $(1.73 + 0.30\tan\theta)°$ at a speed of 32.0° min⁻¹. Weak reflections [$I < 10.0\sigma(I)$] rescanned (maximum of two rescans) and counts accumulated to assure good counting statistics. Diameter of incidence beam collimator was 0.5 mm and crystal to detector distance was 400.0 mm. Intensities of three representative reflections measured every 150 reflections changed by only 1.2% and a linear correction factor was applied; 2936 reflections; 2746 unique reflections (4 < 2θ < 50°; h = 0 to 10, k = −13 to 13, l = −9 to 9). The linear absorption coefficient for Mo $K\alpha$ was 11.346 mm⁻¹ and an empirical absorption correction, based on azimuthal scans of several reflections, was applied with transmission factors 0.5 to 1.0. The data were corrected for Lorentz and polarization effects. The structure was solved by direct methods (*SHELX*; Sheldrick & Egert, 1986). The non-H atoms, other than those in the phenyl ring, were refined anisotropically. The phenyl rings were constrained to a regular hexagon with bond distances of 1.40 Å and C—C—C angles of 120.0°. Phenyl and alkyl H atoms were also included in their idealized positions with C—H set at 0.95 Å and with the isotropic thermal parameters set at 0.007 Å² greater than that of the C atom to which they were attached. The final cycle of full-matrix least-squares refinement

Table 1. Final fractional coordinates and B_{eq} values ($\mathrm{\AA}^2 \times 10^2$) with e.s.d.'s in parentheses

$$B_{eq} = (8\pi^2/3)\Sigma_{i=1}^3 \Sigma_{j=1}^3 U_{ij} a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	B_{eq}
Pt	0.28960 (5)	0.44297 (5)	0.47174 (5)	2.40 (2)
Te	0.32420 (9)	0.29963 (7)	0.2111 (1)	2.83 (3)
Cl(1)	0.2927 (4)	0.2805 (3)	0.6003 (4)	4.2 (1)
Cl(2)	0.2698 (4)	0.5811 (3)	0.7179 (4)	3.6 (1)
S	0.2793 (3)	0.6011 (3)	0.3478 (4)	2.7 (1)
O	−0.358 (1)	0.0127 (8)	−0.249 (1)	4.2 (3)
C(1)	0.0908 (6)	0.2100 (7)	0.0580 (8)	3.14 (9)
C(2)	0.0620 (7)	0.1192 (7)	−0.1002 (9)	3.14 (9)
C(3)	−0.0906 (8)	0.0551 (6)	−0.2014 (7)	3.14 (9)
C(4)	−0.2144 (6)	0.0817 (7)	−0.1442 (8)	3.14 (9)
C(5)	−0.1856 (7)	0.1725 (7)	0.0141 (9)	3.14 (9)
C(6)	−0.0330 (8)	0.2366 (6)	0.1152 (7)	3.14 (9)
C(7)	−0.491 (2)	0.047 (1)	−0.216 (2)	5.0 (6)
C(8)	−0.631 (2)	−0.048 (1)	−0.345 (2)	5.4 (6)
C(9)	0.342 (1)	0.442 (1)	0.081 (1)	3.2 (4)
C(10)	0.236 (1)	0.526 (1)	0.115 (1)	3.6 (4)
C(11)	0.099 (1)	0.645 (1)	0.343 (2)	3.8 (5)

Table 2. Bond lengths ($\mathrm{\AA}$) and angles ($^\circ$) with e.s.d.'s in parentheses

Pt—Te	2.514 (1)	Te—Pt—Cl(1)	89.53 (9)
Pt—Cl(1)	2.324 (4)	Te—Pt—Cl(2)	177.31 (8)
Pt—Cl(2)	2.336 (3)	Te—Pt—S	91.45 (8)
Pt—S	2.258 (4)	Cl(1)—Pt—Cl(2)	91.1 (1)
Te—C(1)	2.113 (5)	Cl(1)—Pt—S	178.2 (1)
Te—C(9)	2.15 (1)	Cl(2)—Pt—S	88.0 (1)
C(9)—C(10)	1.48 (2)	Pt—Te—C(1)	101.6 (2)
C(10)—S	1.84 (1)	Pt—Te—C(9)	94.7 (3)
S—C(11)	1.80 (1)	C(1)—Te—C(9)	93.8 (4)
C(4)—O	1.352 (9)	Pt—S—C(10)	106.0 (4)
O—C(7)	1.42 (2)	Pt—S—C(11)	107.7 (5)
C(7)—C(8)	1.49 (2)	C(10)—S—C(11)	99.0 (6)
C(1)—C(2)	1.40 (1)	C(7)—O—C(4)	119.3 (8)
C(1)—C(6)	1.40 (1)	O—C(7)—C(8)	108.0 (1)
C(2)—C(3)	1.395 (8)	Te—C(9)—C(10)	110.1 (9)
C(3)—C(4)	1.40 (1)	S—C(10)—C(9)	111.9 (8)
C(4)—C(5)	1.40 (1)		
C(5)—C(6)	1.395 (8)		
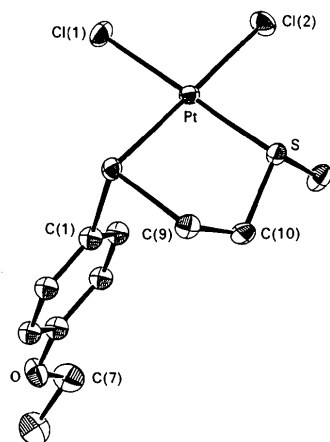
Fig. 1. *ORTEP*II (Johnson, 1976) plot of *cis*-PtCl$_2$[Te(C$_6$H$_4$-OC$_2$H$_5$)CH$_2$CH$_2$(CH$_3$)S]. The atoms are drawn with 20% probability ellipsoids. H atoms are omitted for clarity.

was based on 1937 observed reflections [$I > 3.00\sigma(I)$] and 107 variable parameters and converged (largest shift/e.s.d. = 0.0002) with $R = 0.0345$ and $wR = 0.0371$, where $w = 4F_o/\sigma^2(F_o^2)$. The difference maps had no features of chemical significance; maximum peak 1.23 e $\mathrm{\AA}^{-3}$ and minimum peak −1.62 e $\mathrm{\AA}^{-3}$. Neutral-atom scattering factors were taken from Cromer & Waber (1974). Anomalous-dispersion effects were included in F_c (Ibers & Hamilton, 1964); the values of f' and f'' were those of Cromer (1974). All calculations used the *TEXSAN* crystallographic software package (Molecular Structure Corporation, 1985), including *ORTEP*II (Johnson, 1976) and *PLUTO* (Motherwell & Clegg, 1978).

Discussion. The final atomic coordinates for non-H atoms are given in Table 1 and important distances and angles are combined in Table 2.*

The crystal structure analysis of *cis*-PtCl$_2$-[Te(C$_6$H$_4$OC$_2$H$_5$)CH$_2$CH$_2$(CH$_3$)S] confirms the predictions of the existence of a *cis*-planar Pt^{II} complex. This can be seen clearly in the *ORTEP*II diagram (Fig. 1). The Pt—Te bond length of 2.514 (1) $\mathrm{\AA}$ is less than in two other Pt^{II} compounds reported previously where the lengths were 2.575 (1) $\mathrm{\AA}$ for the ion [Pt{PhTe(O—PPh$_2$C$_6$H$_4$)}$_2$]$^{2+}$ where the Pt—Te bonds are *trans* to one another (Gysling & Luss, 1984) and 2.553 (1) $\mathrm{\AA}$ for [Pt(TeSN$_2$H)(PMe$_2$Ph)$_2$]$^+$ in which the Te atom is *trans* to a phosphine group (Kelly, Slawin, Williams & Woolins, 1990). The Pt—Cl bond lengths are typical of those *trans* to a π-bonding ligand. The Pt—Cl bond *trans* to Te is very slightly longer, 2.336 (3) $\mathrm{\AA}$, than that *trans* to S, 2.324 (4) $\mathrm{\AA}$, consistent with Te having a comparable, but slightly lower, *trans* effect than S.

References

Cromer, D. T. (1974). *International Tables for X-ray Crystallography*, Vol. IV, Table 2.3.1. Birmingham: Kynoch Press. (Present distributor: Kluwer Academic Publishers, Dordrecht.)

Cromer, D. T. & Waber, J. T. (1974). *International Tables for X-ray Crystallography*, Vol. IV, Table 2.2A. Birmingham: Kynoch Press. (Present distributor: Kluwer Academic Publishers, Dordrecht.)

Gysling, H. J. & Luss, H. R. (1984). *Organometallics*, 3, 496–598.

IBERS, J. A. & HAMILTON, W. C. (1964). *Acta Cryst.* **17**, 781–782.

JOHNSON, C. K. (1976). *ORTEPII*. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.

KELLY, P. F., SLAWIN, M. Z., WILLIAMS, D. J. & WOOLINS, J. D. (1990). *Polyhedron*, **9**, 2659–2662.

Molecular Structure Corporation (1985). *TEXSAN. TEXRAY Stucture Analysis Package.* MSC, 3200A Research Forest Drive, The Woodlands, TX 77381, USA.

MOTHERWELL, W. D. S. & CLEGG, W. (1978). *PLUTO.* Program for plotting molecular and crystal structures. Univ. of Cambridge, England.

SHELDRICK, G. M. & EGERT, E. (1986). *SHELX.* Program for general direct methods and automatic Patterson. Univ. of Göttingen, Germany.

SINGH, A. K. & SRIVASTAVA, V. (1990a). *Phosphorus Sulfur Silicon Relat. Elem.* **47**, 471.

SINGH, A. K. & SRIVASTAVA, V. (1990b). *J. Coord. Chem.* **21**, 39–42, and references therein.

SINGH, A. K. & SRIVASTAVA, V. (1990c). *J. Coord. Chem.* **21**, 269–273.

SINGH, A. K., SRIVASTAVA, V. & KHANDELWAL, B. L. (1990). *Polyhedron*, **9**, 495, 851, and references therein.

Structures of Three Sesquiterpene γ-Lactones from *Rudbeckia mollis*

BY MARTA VASQUEZ, FRANK R. FRONCZEK, LEOVIGILDO QUIJANO AND NIKOLAUS H. FISCHER

Department of Chemistry, Louisiana State University, Baton Rouge, Louisiana 70803, USA

Abstract. (1): 15-Acetoxyrudmollin, $C_{17}H_{24}O_5$, $M_r = 308.4$, orthorhombic, $P2_12_12_1$, $a = 9.0071$ (10), $b = 10.053$ (2), $c = 18.144$ (2) Å, $V = 1642.9$ (7) Å³, $Z = 4$, $D_x = 1.247$ g cm⁻³, $\lambda(\text{Mo } K\alpha) = 0.71073$ Å, $\mu = 0.85$ cm⁻¹, $F(000) = 664$, $T = 295$ K, $R = 0.049$ for 1540 observations having $I > 3\sigma(I)$ (of 2158 unique data). (2): Rudmollin diacetate, $C_{19}H_{26}O_6$, $M_r = 350.4$, monoclinic, $P2_1$, $a = 8.117$ (2), $b = 7.681$ (4), $c = 14.865$ (3) Å, $\beta = 92.47$ (2)°, $V = 926.0$ (6) Å³, $Z = 2$, $D_x = 1.257$ g cm⁻³, $\lambda(\text{Cu } K\alpha) = 1.54184$ Å, $\mu = 7.29$ cm⁻¹, $F(000) = 376$, $T = 295$ K, $R = 0.044$ for 3170 observations having $I > 3\sigma(I)$ (of 3747 unique data). (3): Dihydrorudmollin, $C_{15}H_{24}O_4$, $M_r = 268.4$, orthorhombic, $P2_12_12_1$, $a = 9.441$ (2), $b = 10.731$ (2), $c = 13.349$ (2) Å, $V = 1352.3$ (7) Å³, $Z = 4$, $D_x = 1.320$ g cm⁻³, $\lambda(\text{Mo } K\alpha) = 0.71073$ Å, $\mu = 0.88$ cm⁻¹, $F(000) = 584$, $T = 125$ K, $R = 0.043$ for 2293 observations having $I > 3\sigma(I)$ (of 3335 unqiue data). Crystals of the three compounds were isolated from *Rudbeckia mollis* Ell (Asteraceae) which was collected in Putman County, Florida. All three compounds are ambrosanolide-type pseudoguaianolides. Dihydrorudmollin (3) has its methyl group β-oriented on the lactone. All compounds have the seven-membered rings in twist-boat conformations with the methyl-bearing C atom on the pseudodiad and the cyclopentane ring in envelope conformations with the quaternary C atom C5 at the flap. Hydroxyl groups of the two OH-bearing compounds [(1) and (3)] are involved in hydrogen bonding.

Introduction. Recently, we have isolated a new sesquiterpene lactone, 11α*H*,13-dihydrorudmollin (3) from *R. mollis* (Vasquez, Quijano, Urbatsch &

Fischer, 1991). From the same plant we obtained two related sesquiterpene lactones, 15-acetoxyrudmollin (1) and rudmollin diacetate (2), which had been previously reported by Herz, Kumar & Blount (1981). The chemical structures of compounds (1), (2) and (3) were established on the basis of ¹H and ¹³C NMR spectroscopic studies and chemical transformations. The molecular structures of pseudoguaianolides (1), (2) and (3) were determined in order to learn about the influence of the presence of acetate groups upon the conformation of the rudmollin skeleton in lactones (1) and (2) as well as the effects of saturation of the 11,13-double bond found in (3), when compared to the molecular structure of rudmollin (Herz, Kumar & Blount, 1981).

(1) *R* = H
(2) *R* = Ac
(3)
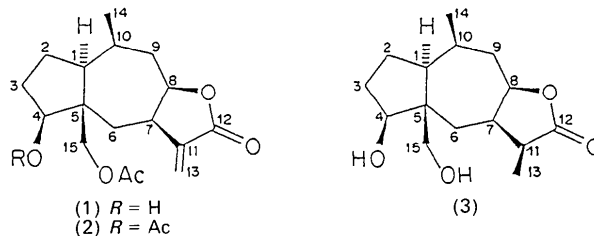
Experimental. Intensity data for all three compounds were obtained from fragments of colorless needles, on Enraf–Nonius CAD-4 diffractometers equipped with either Mo $K\alpha$ ($\lambda = 0.71073$ Å) or Cu $K\alpha$ ($\lambda = 1.54184$ Å) radiation and graphite monochromators. Data-collection parameters are summarized in Table 1. The cryogenic (125 K) data for (3) were collected using an N_2 gas stream cryostat. Variable scan rates were employed in the ω–2θ scans, and a maximum