the Pt—Cl distances are similar [2·306 (2) and 2·308 (2) Å] but vary significantly [from 2·284 (2) to 2·334 (2) Å] in the three independent molecules of the *B* form of the complex. In *cis*-$[PtCl_2(NH_3)_2]$ (Milburn & Truter, 1966) these distances are equally elongated [2·328 (9) and 2·333 (9) Å], whereas in the two independent molecules of the *cis*-$Pt[(CH_3)_2NH]_2Cl_2$ complex (Arpalahti, Lippert, Schollhorn & Thewalt, 1988), the Pt—Cl bond lengths have values ranging from 2·294 (5) to 2·312 (5) Å.

The Pt—N(1) and Pt—N(2) distances [2·048 (6) and 2·038 (6) Å respectively] are slightly longer than the analogous distances in the *A* form of *cis*-$Pt(CH_3NH_2)_2Cl_2$ (Wimmer *et al.*, 1989) [2·027 (7), 2·040 (7) Å] and lie within the interval of 2·034 (6)–2·084 (6) Å for bond lengths in the alternative *B* form. In *cis*-$[PtCl_2(NH_3)_2]$ (Milburn & Truter, 1966), the less accurate values are 1·95 (3) and 2·05 (4) Å and in *cis*-$Pt[(CH_3)_2NH]_2Cl_2$ (Arpalahti *et al.*, 1988) these distances range from 2·05 (1) to 2·06 (2) Å.

The C—N distances have normal lengths. The endocyclic P—C bonds [average 1·801 (8) Å] are slightly longer than the exocyclic bond [1·759 (7) Å]. The P—O distance of 1·493 (4) Å corresponds to the P=O double bond which participates in hydrogen bonding (Blessing, 1988). The conformation of the six-membered chelate is a slightly distorted chair (Boeyens, 1978), which follows from the values of the ring-puckering parameters (Cremer & Pople, 1975), $q = 0{\cdot}701$ Å, $\theta = 10{\cdot}6°$, $\varphi = 203{\cdot}1°$.

The shortest Pt···Pt distances are between the complex molecules related by the centre of symmetry at $\frac{1}{4},\frac{1}{4},\frac{1}{4}$ [4·634 (1) Å] and the twofold axis at $\frac{1}{4},y,0$ [5·037 (1) Å]. In *cis*-$[PtCl_2(NH_3)_2]$ the Pt···Pt distances are much shorter [3·372 (2) and 3·409 (2) Å] and Milburn & Truter (1966) suggested that there was an interaction between the orbitals of the Pt atoms. In *cis*-$Pt[(CH_3)_2NH]_2Cl_2$ (Arpalahti *et al.*, 1988), the Pt separation distances are 5·514 and 5·576 Å.

The complex molecules are linked by a system of hydrogen bonds. Both amino groups and the water molecule perform a hydrogen-donating function and all electron-rich sites (the Cl, phosphoryl and water O atoms) act as hydrogen-bond acceptors.

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Structure of (4α)-Kaura-9(11),16-dien-18-oic Acid (Grandiflorenic Acid), an Active Ingredient of the Mexican Medicinal Plant Zoapatle

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Abstract. Grandiflorenic acid, $C_{20}H_{28}O_2$, $M_r = 300{\cdot}44$, monoclinic, $P2_1$, $a = 10{\cdot}697$ (1), $b = 11{\cdot}476$ (2), $c = 14{\cdot}229$ (2) Å, $\beta = 98{\cdot}86$ (1)°, $V = 1725{\cdot}8$ Å^3, $Z = 4$, $D_x = 1{\cdot}159$ g cm^{-3}, λ(Mo $K\alpha$) = 0·71069 Å, $\mu = 0{\cdot}7$ cm^{-1}, $F(000) = 656$, $T = 298$ K, $R(wR) = 0{\cdot}0478$ (0·0576) for 2007 reflections with $I \geq$

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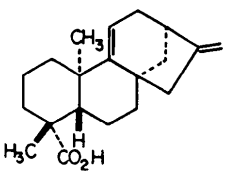
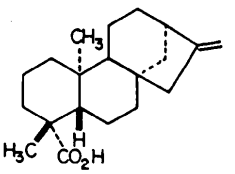
2·5σ. The two independent molecules in the asymmetric unit form a strongly hydrogen-bonded dimer involving the C19 carboxylic acid groups [O2*A*···H1 = 1·63 (7), O1*B*···H2 = 1·51 (5) Å; O2*B*–H1···O2*A* = 170 (6), O1*A*–H2···O1*B* = 166 (6)°] with the dimers having an extended *anti* overall conformation. Stacking interactions between carbon skeletons determine the overall crystal packing. The introduction of the 9(11)-double bond in grandiflorenic acid has a drastic effect on the overall molecular conformation, shifting the C(17) atom by over 4 Å from the corresponding position in kaurenoic acid. Detailed investigations of NMR vicinal ^1H–^1H coupling constants and nuclear Overhauser enhancement factors confirm that grandiflorenic acid has very similar solution and crystal conformations.

Introduction. Grandiflorenic acid (GF) [(4α)-kaura-9(11),16-dien-18-oic acid] and (4α)-kaur-16-en-18-oic acid (KA) are among the secondary metabolites isolated from *Montanoa tomentosa* (Caballero & Walls, 1970; Enriquez, Escobar, Romero, Chavez & Lozoya, 1983).



GF



KA

There is an extensive history in Mexican traditional medicine of the use of aqueous extracts of the leaves of this plant (popularly known as zoapatle) as an abortifacient and for inducing labor in late stages of pregnancy (Gallegos, 1983). Furthermore, it has been shown that GF (but not KA) induces uterine contractions (Lozoya, Enriquez, Bejar, Estrada, Giron, Ponce-Monter & Gallegas, 1983; Bejar, Enriquez & Lozoya, 1984). We have previously used concerted two-dimensional NMR techniques to assign fully the ^1H and ^{13}C spectra of grandiflorenic acid (Reynolds, Enriquez, Escobar & Lozoya, 1984). However, the ^1H spectra at 200 MHz showed serious spectral overlap, preventing the deduction of the solution conformation from vicinal ^1H–^1H coupling constants. In view of the interesting pharmacological activity of GF and the corresponding inactivity of KA, we felt it would be useful to determine the crystal structures of these two compounds since this information should be of value for understanding structure–activity relationships. At the same time, we undertook a higher-field (400 MHz) ^1H NMR spectral investigation of GF in order to compare solution and crystal structures. Results of this investigation are presented below. After submission of this manuscript we were informed of a recent independent study of the crystal structure of KA isolated from the exudate of ferns of the genus *Notholaena* (*N. peninsularis* and *N. Pallens*) (Brassy, Bachet & Wollenweber, 1988). Since this structure is in close agreement with our structure, no details will be reported here for KA other than a comparison of the conformations of GF and KA.

Experimental. Large well formed colorless crystals of GF were cleaved into smaller fragments. Preliminary precession photographs were used to check crystal qualities and to obtain preliminary cell and space-group information. Further work, obtained on an Enraf–Nonius CAD-4 diffractometer, gave the crystal data summarized in Table 1, which also contains details of the intensity measurements. Lorentz and polarization corrections were applied to all data collected.

The structure was solved using the program *MITHRIL* (Gilmore, 1983). In the final cycles of least-squares refinement ($\Sigma 2\Delta F^2$ minimized), all non-H atoms were refined with anisotropic thermal parameters and carboxylic acid H atoms were refined with isotropic thermal parameters (all other H atoms in calculated positions with U values common for each molecule). Agreement indices upon convergence of least-squares refinement are given in Table 1. Calculations were on PDP 11/23 and Gould 9705 computers using the programs *MITHRIL*, *SHELX*76 (Sheldrick, 1976) and the Enraf–Nonius *Structure Determination Package* (Frenz, 1980). Scattering factors stored in the programs were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV).

The final atomic positional and thermal parameters for GF are given in Table 2. In view of the close similarity of the two determinations of the kaurenoic acid structure (this work and Brassy, Bachet & Wollenweber, 1988) positional, thermal and other parameters for the present determination of KA have been deposited. The results of least-squares molecular fits and other comparisons of the GF and KA structures are based on these deposited coordinates. The hydrogen-bonded dimers in GF are illustrated in Fig. 1, while torsional angles are summarized in Fig. 2 (where atom numbering is also illustrated).*

* Lists of final structure-factor amplitudes and anisotropic thermal parameters, the results of best molecular fits between the various independent molecules in two structures and figures displaying bond lengths and bond angles in both compounds along with a view of the pairs of dimers in KA and of the geometries of hydrogen bonding in the two compounds have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53426 (45 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. *Experimental details*

Crystal size	Irregular approx. triangular *ca* 0·237 × 0325 × 0·375 mm
Number of reflections for cell parameters, θ range (°)	25, 8·0 < θ < 13·6
ω-scan range (°)	0·80 + 0·35 tanθ
Maximum scan time (s)*	100
Max. 2θ (°)	50
hkl range	−12–12; 0–13; 0–16
Number of standard reflections/ interval(s)/intensity variation	3/12000/1%
Number of data collected	3496
Number of observed data	2007 [$I \geq 2{\cdot}5\sigma(I)$]
Final R (wR)	0·0478 (0·0576)†
R (wR) for non-zero data	0·0748 (0·0844)
Maxiumum Δ/σ in final cycle	0·35‡
Weights	$[\sigma^2(F) - 0{\cdot}01050F^2]^{-1}$
Maximum peak in final ΔF map (eÅ^{-3})	0·16

* Prescan at 10° min^{-1}. Prescan intensity accepted if $I/\sigma(I) \geq 25$, otherwise scan speeds chosen to give this ratio within max. scan time specified. Backgrounds by extending scan by 25% on either side of peak were measured for half the time taken to collect peak.

† Two blocks (all C + O anisotropic − 425 variables).

‡ For U(H) for the CH_3 groups (< 0·03 otherwise).

Table 2. *Positional and thermal parameters for GF and their e.s.d.'s*

$U_{eq} = (U_{11} + U_{22} + U_{33})/3.$

	x	y	z	U_{eq}/U_{iso}(Å^2)
C1*A*	1·0183 (5)	0·1834 (9)	0·3229 (5)	0·053 (3)
C2*A*	1·1603 (6)	0·2003 (8)	0·3436 (5)	0·063 (4)
C3*A*	1·1990 (5)	0·2634 (8)	0·4377 (5)	0·055 (4)
C4*A*	1·1311 (5)	0·3787 (7)	0·4448 (4)	0·047 (3)
C5*A*	0·9853 (5)	0·3559 (7)	0·4232 (4)	0·039 (3)
C6*A*	0·9042 (5)	0·4621 (7)	0·4444 (4)	0·046 (3)
C7*A*	0·7726 (5)	0·4634 (7)	0·3874 (4)	0·049 (3)
C8*A*	0·7149 (5)	0·3433 (7)	0·3714 (4)	0·038 (3)
C9*A*	0·7995 (5)	0·2637 (7)	0·3217 (4)	0·039 (3)
C10*A*	0·9398 (5)	0·2951 (7)	0·3257 (3)	0·038 (3)
C11*A*	0·7477 (6)	0·1724 (7)	0·2738 (4)	0·049 (3)
C12*A*	0·6127 (6)	0·1376 (8)	0·2672 (4)	0·055 (4)
C13*A*	0·5397 (5)	0·2193 (8)	0·3242 (4)	0·057 (4)
C14*A*	0·5818 (6)	0·3441 (8)	0·3122 (4)	0·057 (4)
C15*A*	0·6887 (5)	0·2851 (8)	0·4637 (4)	0·049 (3)
C16*A*	0·5802 (5)	0·1994 (8)	0·4298 (4)	0·052 (3)
C17*A*	0·5279 (6)	0·1266 (9)	0·4831 (5)	0·067 (4)
C18*A*	1·1749 (5)	0·4692 (8)	0·3795 (4)	0·048 (3)
C19*A*	1·1681 (6)	0·4274 (9)	0·5471 (4)	0·065 (4)
C20*A*	0·9546 (6)	0·3709 (8)	0·2392 (4)	0·056 (4)
O1*A*	1·2739 (4)	0·4444 (7)	0·3416 (3)	0·062 (3)
O2*A*	1·1230 (4)	0·5656 (6)	0·3670 (4)	0·070 (3)
H2	1·313 (7)	0·527 (8)	0·315 (5)	0·09 (2)
C1*B*	0·6209 (6)	0·3151 (9)	1·0032 (4)	0·062 (4)
C2*B*	0·7488 (6)	0·3232 (9)	0·9684 (5)	0·065 (4)
C3*B*	0·7357 (7)	0·3855 (8)	0·8739 (5)	0·064 (4)
C4*B*	0·6420 (6)	0·3243 (8)	0·7983 (4)	0·051 (3)
C5*B*	0·5125 (5)	0·3081 (8)	0·8341 (4)	0·045 (3)
C6*B*	0·4071 (6)	0·2604 (8)	0·7580 (4)	0·060 (4)
C7*B*	0·2979 (6)	0·2035 (8)	0·7993 (4)	0·059 (4)
C8*B*	0·2700 (5)	0·2639 (7)	0·8893 (4)	0·050 (3)
C9*B*	0·3882 (6)	0·2676 (7)	0·9648 (4)	0·051 (3)
C10*B*	0·5186 (6)	0·2502 (7)	0·9338 (4)	0·047 (3)
C11*B*	0·3715 (7)	0·2806 (10)	1·0544 (4)	0·074 (5)
C12*B*	0·2467 (8)	0·2910 (11)	1·0883 (5)	0·099 (6)
C13*B*	0·1388 (7)	0·2962 (9)	1·0050 (6)	0·077 (5)
C14*B*	0·1626 (7)	0·2051 (9)	0·9304 (6)	0·076 (5)
C15*B*	0·2175 (6)	0·3891 (9)	0·8706 (4)	0·059 (4)
C16*B*	0·1416 (6)	0·4094 (9)	0·9516 (5)	0·065 (4)
C17*B*	0·0875 (8)	0·5074 (10)	0·9676 (7)	0·094 (6)
C18*B*	0·7005 (6)	0·2136 (8)	0·7650 (4)	0·051 (4)
C19*B*	0·6242 (7)	0·4037 (8)	0·7081 (5)	0·076 (5)
C20*B*	0·5520 (6)	0·1195 (8)	0·9378 (5)	0·061 (4)
O1*B*	0·6427 (5)	0·1232 (7)	0·7459 (4)	0·077 (3)
O2*B*	0·8168 (5)	0·2264 (7)	0·7514 (5)	0·098 (4)
H1	1·158 (10)	0·662 (11)	0·287 (7)	0·14 (4)

assumed absolute stereochemistry is identical to that previously determined (Karle, 1972) for (−)-kaur-15-en-19-al.

All NMR special data for GF were obtained on a Varian XL-400 spectrometer at the University of Toronto, operating at 400 MHz for ^{1}H. Spectra were obtained for solutions of *ca* 10 mg of GF in 0·5 ml of the particular solvent (or solvent mixture, see below).

Discussion.

Comparison of crystal structures of GF and KA. Crystals of GF contain two independent molecules in the asymmetric unit linked by hydrogen-bonding interactions between the carboxylic acid groups to give dimeric units. The oxygen–oxygen separations between molecules are O1*A*···O2*B* = 2·627 (10) and O2*A*···O2*B* = 2·645 (10) Å. The plane of the carbox-

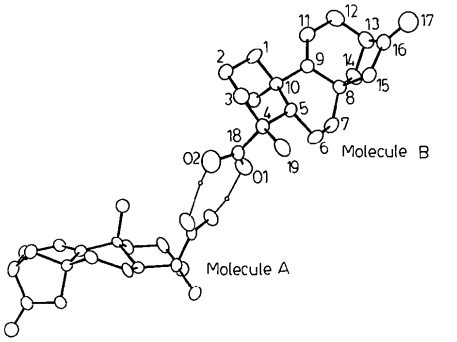


Fig. 1. *ORTEP* view (Johnson, 1971) of the hydrogen-bonded dimeric unit in the asymmetric unit of grandiflorenic acid. Thermal ellipsoids are drawn at the 40% probability level. All H atoms are drawn with uniform isotropic thermal parameters. The stereochemistry is consistent with that reported (Karle, 1972) for (−)-kaur-15-en-19-al.

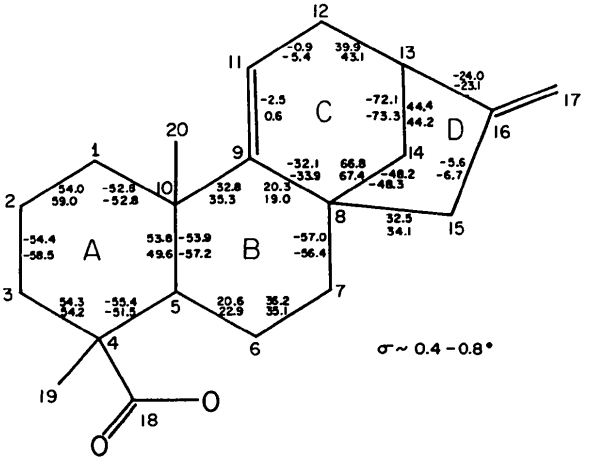


Fig. 2. Torsion angles (°) in grandiflorenic acid. Upper values refer to molecule *A*. Atomic numbering is indicated outside the rings which are designed as *A*, *B etc.*

ylic acid groups is approximately orthogonal to mean planes through the kaurane skeletons such that the dimer has an overall *anti* conformation (Fig. 1).

Bond lengths and bond angles in the independent molecules* and the torsion angles given in Fig. 2 indicate that the differences between the two independent molecules in GF and between the four independent molecules in KA are minimal. This can also be seen in the results of least-squares fits* using the program *BMFIT* (Yuen & Nyburg, 1979). Thus, the atoms C(1) to C(18) in the two molecules of GF fit with a sum of differences squared of 0·085 Å^2 while a small rotational difference in the orientations of the CO_2H groups leads to a maximum difference of *ca* 1 Å in position. However, the introduction of the 9–11 double bond in GF has a substantial effect on the overall conformation of this compound as can be seen from the fit over all non-H atoms for KA and GF or, better, the deviations of the unmatching atoms after atoms C(1)—C(6), C(9).* Deviations of up to 4 Å [for C(17)] in corresponding atom positions are observed as the bicyclo[3.2.1]octane/octene system is rotated by *ca* 27° about the C8—C9 bond towards the β face in GF. This puts the *B* ring of GF in a boat conformation compared with the fairly regular chair conformation of this ring in KA. This boat conformation can be clearly seen for molecule *B* in Fig. 1 (C5–C10).

Comparison of solution and crystal conformations of GF. The most unusual feature of the crystal structure of GF is the boat conformation for ring *B*. Therefore, we were particularly interested in determining whether the molecule also favored this conformation in solution. The key features for determining this are the vicinal ^{1}H–^{1}H coupling constants involving the C5, C6 and C7 protons. Due to the extreme spectral complexity, even at 400 MHz not all necessary multiplet patterns could be observed in any one solvent. However, by determining spectra in $CDCl_3$, C_6D_6, $CDCl_3$:C_6D_6 mixtures and $(CD_3)_2CO$, it was possible to observe each multiplet pattern in one or more of the solutions. Fortunately the multiplet for H5 was observable in all solutions and showed no change in coupling to either $H6_\alpha$ or $H6_\beta$, indicating that the solution conformation was not solvent dependent. Vicinal couplings were determined by iterative spectral analysis, using the spectral analysis package supplied with the spectrometer. Results are summarized in Table 3, along with the corresponding dihedral H—C—C—H angles, φ, as determined from the crystal structures.† Vicinal coupling constants are also given in Table 3 for the sequence of protons from H11 to $H14_\alpha$ and $H14_\beta$.

Table 3. *Vicinal* ^{1}H–^{1}H *coupling constants,* $^3J_{HH}$, *compared with* H—C—C—H *dihedral angles,* φ

Coupling constant	$^3J_{HH}$(Hz)	φ(°)*
H5–$H6_\alpha$	11·0	149
H5–$H6_\beta$	8·7	30
$H6_\alpha$–$H7_\alpha$	9·1	36
$H6_\alpha$–$H7_\beta$	1·1	82
$H6_\beta$–$H7_\alpha$	10·8	154
$H6_\beta$–$H7_\beta$	8·9	36
H11–$H12_\alpha$	2·7	63
H11–$H12_\beta$	4·0	57
$H12_\alpha$–H13	4·6	46
$H12_\beta$–H13	2·0	74
H13–$H14_\alpha$	5·2	49
H13–$H14_\beta$	2·7	74

* Average value from two non-equivalent molecules.

The various coupling constants all show the behavior predicted by the Karplus relationships, *i.e.* they show a maximum value as φ approaches 180°, a minimum for φ *ca* 90° and a smaller maximum as φ approaches 0°. This strongly suggests that GF adopts very similar conformations in solution and in the crystal. In particular, the near-zero coupling for $H6_\alpha$–$H7_\beta$ (φ = 82°) indicates that there is little conformational averaging since, in the alternative chair form, φ should approach 180°, giving a maximum value for this coupling. Further support for the boat conformation of ring *B* was provided by the observation of small positive nuclear Overhauser enhancements for $H7_\alpha$ on irradiation of the C20 methyl protons and for H5 upon irradiation of $H15_\beta$. The former interaction is particularly indicative of the boat conformation since $H7_\alpha$ would be spatially close to the methyl group only in this conformation. The large conformational differences between GF and KA may, at least in part, account for the significantly different pharmacological activities of these two compounds.

Finally, the detailed investigation of vicinal coupling constants and nuclear Overhauser effects has allowed unambiguous assignment of individual methylene protons as α or β. With the exception of ring *A* protons, this assignment was previously, in many cases, ambiguous (Reynolds *et al.*, 1984). Correct assignments (and ^{1}H chemical shifts) in $CDCl_3$ are given in Table 4.

The observed rotation for GF is $[\alpha]_D^{25°C} = +46°$[$CHCl_3$], in reasonable agreement with the original value of +32° (Brieskorn & Pohlman, 1969) while the rotation of KA is −102°, close to the original report of −110° (Henrick & Jefferies, 1964). It is noteworthy that the opposite rotation for the two compounds does not imply that they exist as opposite enantiomers. It has been shown (Piozzi, Passannanti, Marino & Spiro, 1972) that catalytic hydrogenation of the exocyclic double bond of GF only changed the rotation from +38 to +43°. However, subsequent further reduction of the 9–11 double bond changed the rotation from +43 to −80°. These results can be rationalized in terms of

* See deposition footnote.

† H atoms in calculated positions.

Table 4. *Assigned chemical shifts for methylene protons in rings B, C and D of GF (on δ scale in* $CDCl_3$)

Proton	δ	Proton	δ
$H6_\alpha$	2·48	$H14_\alpha$	1·52
$H6_\beta$	1·84	$H14_\beta$	1·45
$H7_\alpha$	1·99	$H15_\alpha$	2·18
$H7_\beta$	1·45	$H15_\beta$	2·60
$H12_\alpha$	2·42	$H17_c$*	4·89
$H12_\beta$	1·98	H17,†	4·78

* *cis* to C13.
† *trans* to C13.

the dramatic conformational differences between GF and KA. The adoption of the boat conformation by ring *B* of GF must change the rotation of the (−)-kaurene skeleton from negative to positive.

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Structure of Bis(*p*-methoxyphenyltelluro)ethyne

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Abstract. $C_{16}H_{14}O_2Te_2$, $M_r = 493{\cdot}3$, monoclinic, $P2_1/n$, $a = 7{\cdot}911$ (3), $b = 6{\cdot}671$ (6), $c = 15{\cdot}443$ (4) Å, $\beta = 95{\cdot}4$ (4)°, $V = 811{\cdot}1$ Å^3, $Z = 2$, $D_x = 2{\cdot}02$ g cm^{-3}, Mo $K\alpha$, $\lambda = 0{\cdot}70926$ Å, $\mu = 36{\cdot}02$ cm^{-1}, $F(000) = 460$, room temperature, $R = 0{\cdot}051$, $wR = 0{\cdot}053$ for 1429 observed reflections [$I \geq 2{\cdot}5\sigma(I)$]. The structure is characterized by centrosymmetric binuclear molecules linked through the acetylenic moiety, with four atoms almost linear: Te(1)—C(1)—C(1*)—Te(1*).

Introduction. The use of organotellurium derivatives as reagents and intermediates in organic synthesis has been reviewed (Petragnani & Comasseto, 1986). We have been interested in the insertion reaction of reactive intermediates like carbenes and diacetylides in the Te—Te bond of diaryl ditellurides. In an attempt to understand their chemistry more fully and especially to use the new products in organic synthesis, we report here the synthesis of the title compound, prepared for the first time, by reaction of arenetellurenyl bromides, generated *in situ*, with di-Grignard reagents.

Procedures described in the literature were followed to synthesize all the reagents: bis(*p*-methoxyphenyl) ditelluride (Reichel & Kirschbaum, 1943), *p*-methoxyphenyltellurenyl bromide (Petragnani, Torres, Wynne & Maxwell, 1973;

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