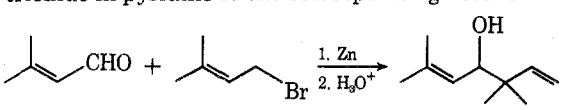
nesium halides.[4] A noteworthy feature of the zinc column method is the absence of Wurtz coupling product derived from the allylic halide. Successive addition to the column of different mixtures of carbonyl components with allylic bromides gave, in each case, a product with no detectable contamination from preceding alcohols. Thus, the zinc in the column can simply be replenished as it is consumed. The method is especially well suited to the large-scale preparation of homoallylic alcohols and, as judged from **10**, is applicable to esters as well as aldehydes and ketones. However, the method is ineffective with allylic chlorides and with saturated bromides.

Product alcohols were purified by short-path distillation and were identified by means of infrared, NMR, and mass spectral data (Table II). (See paragraph at end of paper regarding supplementary material.) In the case of the alcohol derived from crotyl bromide and tetralone, dehydration occurred upon distillation, leading to diene **13**. The structures of products **11–15** from crotyl and geranyl halides reveal that attack on the allylic moiety takes place solely at the β carbon in this reaction, in agreement with similar observations made with allylmagnesium[5] and allylzinc halides.[6] This feature ("allylic transposition") lends itself to an efficient, one-step synthesis of (±)-artemisia alcohol (**16**)[7]. Thus, passage through a heated zinc column of a mixture of 3-methyl-2-butenal[8] and 1-bromo-3-methyl-2-butene[9] gave, after hydrolysis, a 91% yield of **16**. The structure of artemisia alcohol was confirmed by oxidation with chromium trioxide in pyridine to the corresponding ketone.[10]



16

The continuous-flow, zinc column procedure appears to be a generally useful method for allylation at carbonyl functions where mild reaction conditions are necessary. The operational simplicity and high efficiency of the method afford significant advantages over the Grignard reaction in certain cases.

Experimental Section

Materials. Geranyl bromide was prepared by the method of Eschenmoser.[11] 1-Bromo-3-methyl-2-butene was prepared by addition of hydrogen bromide to isoprene.[9] 3-Methyl-2-butenal was prepared by oxidation of 3-methyl-2-buten-1-ol with manganese dioxide in petroleum ether. 2-Methyl-5-isopropylcyclopent-1-enecarboxaldehyde was prepared by the method of van Tamelen.[12] All other materials were obtained from commercial sources. Granular zinc (10 mesh) was activated prior to use by the method described previously.[1] GLC analysis was carried out using (1) a 10 ft × 0.375 in. column of 30% Carbowax 20M on Chromosorb W, or (2) a 5 ft × 0.25 in. column of 20% SE-30 on Chromosorb W with an Aerograph Autoprep 700 instrument. Infrared spectra were measured on neat liquids using a Perkin-Elmer Model 137 spectrophotometer. NMR spectra were measured on $CDCl_3$ solutions using a Varian EM-360 spectrometer.

Reactions Using Zinc Column. The following procedure for the reaction of 3-pentanone with crotyl bromide to yield **11** is representative. To a heated column charged with activated zinc (10 mesh)[1] was added dropwise 25 ml of anhydrous tetrahydrofuran followed by a mixture of 3.05 g (35.4 mmol) of 3-pentanone and 7.16 g (53.0 mmol) of 1-bromo-2-butene in 50 ml of tetrahydrofuran. A low reflux was maintained at the head of the column during addition, which took 1 hr. The column was then flushed with 25 ml of tetrahydrofuran and the combined eluate, after dilution with 50 ml of ether, was treated with ice-cold 5% sulfuric acid, followed by sodium bicarbonate solution and saturated brine. The organic layer was dried ($MgSO_4$), the solvent was removed in vacuo, and the residue was purified by short-path distillation to give 4.81 g (95.6%) of **11**, bp 70–73° (16 mm).

Artemisia Alcohol (16). A mixture of 1.15 g (13.6 mmol) of 3-methylbut-2-enal and 3.05 g (20.4 mmol) of 1-bromo-3-methylbut-2-ene in 20 ml of tetrahydrofuran was passed through the heated column of granular zinc. After flushing the column, the eluate was diluted with 50 ml of ether and washed with 30 ml of cold 5% sulfuric acid, sodium bicarbonate solution, and brine. After drying and removal of the solvent in vacuo, distillation afforded 1.68 g (91.3%) of artemisia alcohol (**16**).

Acknowledgments. We are indebted to Mr. Mitchell Avery for the preparation of 2-methyl-5-isopropylcyclopent-1-enecarboxaldehyde. Financial support was provided by the National Science Foundation.

Registry No.—**16**, 29887-38-5; 3-methylbut-2-enal, 107-86-8; 1-bromo-3-methylbut-2-ene, 870-63-3.

Supplementary Material Available. Table II (2 pages). Ordering information is given on any current masthead page.

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Crystal and Molecular Structure of Cephalotaxine

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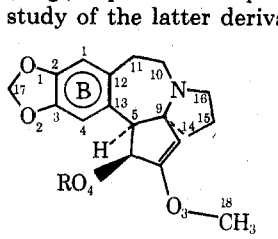
The natural antileukemic esters of caphalotaxine (**1**) include homoharringtonine (**2**), which is undergoing preclinical testing.[1] As these esters are unfortunately noncrystalline, x-ray studies to reveal the conformational preferences of the cephalotaxine portion are limited to other derivatives, e.g., cephalotaxine *p*-bromobenzoate (**3**).[2] Prior to our study of the latter derivative (**3**), we had initiated an



	R
1	H
2	$C_9H_{17}O_4$
3	*p*-BrC_6H_4

Table I
Fractional Coordinates and Estimated Standard Deviations in Molecules 1a and 1b

Atom	x/a		y/b		z/c	
	1a	1b	1a	1b	1a	1b
01	0.0676 (3)	0.2427 (2)	−0.7615 (7)	0.7842 (6)	0.8860 (3)	0.5325 (2)
02	0.1050 (3)	0.1730 (2)	−0.5379 (7)	0.5584 (6)	0.9689 (3)	0.5008 (2)
03	−0.1110 (2)	0.2329 (2)	0.0883 (6)	0.4723 (7)	0.6009 (2)	0.9492 (2)
04	−0.0946 (2)	0.1988 (2)	−0.1699 (6)	0.6433 (6)	0.7088 (2)	0.8074 (2)
N	0.0802 (2)	0.3768 (2)	−0.2399 (7)	0.4027 (6)	0.6729 (2)	0.8377 (2)
C1	0.0219 (3)	0.2948 (3)	−0.5985 (9)	0.7432 (8)	0.7651 (4)	0.6742 (3)
C2	0.0526 (4)	0.2538 (3)	−0.6162 (9)	0.7009 (8)	0.8426 (4)	0.5997 (3)
C3	0.0758 (3)	0.2135 (3)	−0.4850 (9)	0.5654 (8)	0.8927 (3)	0.5807 (3)
C4	0.0686 (3)	0.2127 (3)	−0.3273 (9)	0.4637 (8)	0.8656 (3)	0.6353 (3)
C5	0.0282 (3)	0.2493 (3)	−0.1268 (7)	0.3975 (7)	0.7564 (3)	0.7739 (3)
C6	−0.0440 (2)	0.2116 (3)	−0.0581 (8)	0.4708 (8)	0.7193 (3)	0.8166 (3)
C7	−0.0522 (3)	0.2573 (3)	0.0116 (7)	0.4318 (8)	0.6444 (3)	0.9000 (3)
C8	−0.0007 (3)	0.3125 (3)	−0.0041 (8)	0.3599 (8)	0.6322 (3)	0.9124 (3)
C9	0.0557 (3)	0.3177 (3)	−0.0917 (7)	0.3319 (8)	0.6977 (3)	0.8387 (3)
C10	0.0302 (3)	0.3936 (3)	−0.3560 (9)	0.5740 (8)	0.6213 (3)	0.8613 (3)
C11	−0.0190 (3)	0.3352 (3)	−0.4088 (8)	0.6904 (8)	0.6498 (3)	0.8162 (3)
C12	0.0139 (3)	0.2940 (3)	−0.4374 (8)	0.6443 (8)	0.7360 (3)	0.7322 (3)
C13	0.0367 (3)	0.2532 (3)	−0.3038 (7)	0.5039 (7)	0.7863 (3)	0.7134 (3)
C14	0.1191 (3)	0.3279 (3)	0.0087 (9)	0.1469 (9)	0.7331 (3)	0.8259 (3)
C15	0.1520 (4)	0.4027 (3)	−0.0181 (11)	0.1200 (9)	0.6814 (5)	0.8676 (4)
C16	0.1196 (3)	0.4318 (3)	−0.1755 (10)	0.2929 (9)	0.6362 (4)	0.8888 (4)
C17	0.0948 (6)	0.1983 (4)	−0.7137 (13)	0.6846 (12)	0.9640 (6)	0.4711 (4)
C18	−0.1182 (4)	0.2746 (4)	0.1529 (11)	0.4315 (11)	0.5280 (4)	1.0292 (4)
HO4	−0.111 (2)	0.160 (2)	0.875 (7)	0.644 (7)	0.730 (2)	0.781 (3)
HC1	0.007 (2)	0.327 (2)	0.315 (7)	0.818 (7)	0.728 (2)	0.681 (2)
HC4	0.085 (2)	0.182 (2)	0.773 (6)	0.382 (6)	0.896 (2)	0.619 (2)
HC5	0.058 (2)	0.224 (2)	0.938 (6)	0.300 (6)	0.802 (2)	0.749 (2)
HC6	−0.048 (2)	0.160 (2)	0.029 (6)	0.405 (6)	0.750 (2)	0.802 (3)
HC8	0.001 (2)	0.349 (2)	0.022 (6)	0.325 (7)	0.583 (2)	0.962 (3)
H1C10	0.059 (2)	0.441 (2)	0.562 (7)	0.613 (7)	0.615 (2)	0.857 (3)
H2C10	0.004 (2)	0.410 (2)	0.689 (7)	0.586 (7)	0.565 (3)	0.912 (3)
H1C11	−0.061 (2)	0.299 (2)	0.665 (7)	0.680 (7)	0.632 (3)	0.841 (3)
H2C11	−0.049 (2)	0.361 (2)	0.505 (7)	0.800 (7)	0.618 (3)	0.812 (3)
H1C14	0.150 (2)	0.308 (2)	0.978 (7)	0.063 (7)	0.788 (3)	0.856 (3)
H2C14	0.110 (2)	0.311 (2)	0.127 (7)	0.119 (7)	0.741 (3)	0.776 (3)
H1C15	0.203 (2)	0.425 (2)	0.977 (8)	0.056 (8)	0.717 (3)	0.828 (3)
H2C15	0.153 (2)	0.412 (2)	0.083 (8)	0.054 (8)	0.646 (3)	0.924 (3)
H1C16	0.157 (2)	0.451 (2)	0.754 (7)	0.316 (7)	0.629 (3)	0.946 (3)
H2C16	0.089 (2)	0.475 (2)	0.839 (7)	0.323 (7)	0.580 (3)	0.886 (3)
HlC17	0.147 (3)	0.230 (3)	0.247 (10)	0.603 (8)	0.982 (4)	0.449 (3)
H2C17	0.055 (3)	0.169 (3)	0.277 (10)	0.709 (8)	0.983 (4)	0.420 (3)
H1C18	−0.078 (3)	0.327 (2)	0.222 (8)	0.487 (8)	0.532 (3)	1.053 (3)
H2C18	−0.142 (3)	0.279 (2)	0.202 (8)	0.327 (8)	0.520 (3)	1.039 (3)
H3C18	−0.129 (3)	0.250 (2)	0.063 (8)	0.472 (8)	0.493 (3)	1.062 (3)

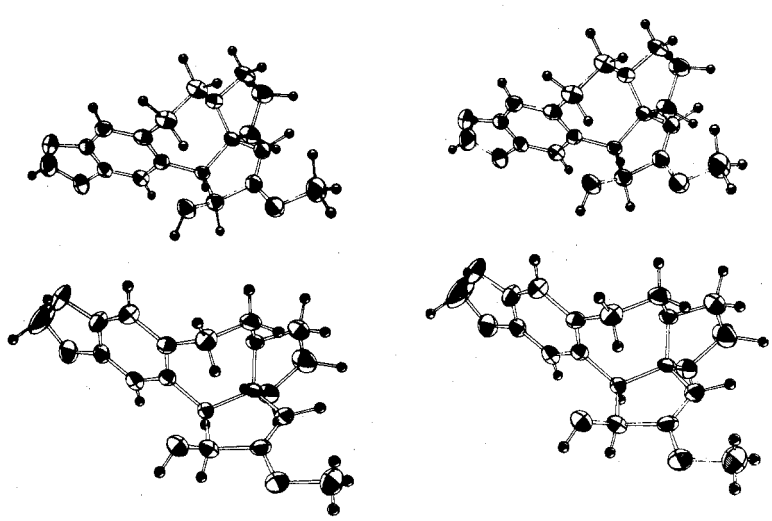


Figure 1. Stereoscopic view of cephalotaxine molecules **1a** (above) and **1b** (below). Hydrogen atoms are shown as spheres, and other atoms as 50% probability ellipsoids.

x-ray study on cephalotaxine (1) itself; after an uneventful data collection, we were unable to solve the phase problem by direct methods. Recently, vector search methods have revealed the structure, and we now wish to report the conformations of the two independent cephalotaxine molecules (**1a** and **1b**) in these crystals.

Table II
Bond Lengths in Molecules 1a and 1b

Atoms	Distance, Å 1a	1b	Atoms	Distance, Å 1a	1b
01–C2	1.402 (9)	1.393 (8)	C12–C13	1.396 (8)	1.411 (8)
01–C17	1.409 (10)	1.414 (8)	C14–C15	1.529 (11)	1.529 (9)
02–C3	1.387 (7)	1.397 (5)	C15–C16	1.539 (10)	1.533 (9)
02–C17	1.447 (11)	1.428 (10)	04–H	0.82 (6)	0.80 (5)
03–C7	1.362 (6)	1.362 (9)	C1–H	0.95 (5)	0.92 (5)
03–C18	1.457 (9)	1.442 (7)	C4–H	0.98 (5)	0.90 (5)
04–C6	1.410 (7)	1.432 (8)	C5–H	0.98 (4)	0.97 (4)
N–C9	1.502 (8)	1.478 (8)	C6–H	0.97 (5)	1.19 (6)
N–C10	1.463 (7)	1.466 (8)	C8–H	0.99 (6)	0.98 (4)
N–C16	1.484 (10)	1.487 (7)	C10–H1	0.99 (6)	1.15 (6)
C1–C2	1.350 (9)	1.359 (7)	C10–H2	1.05 (5)	0.89 (5)
C1–C12	1.410 (9)	1.397 (9)	C11–H1	1.05 (5)	1.14 (6)
C2–C3	1.381 (9)	1.373 (9)	C11–H2	0.97 (5)	1.09 (6)
C3–C4	1.371 (9)	1.360 (9)	C14–H1	1.01 (5)	1.12 (6)
C4–C13	1.386 (8)	1.406 (6)	C14–H2	1.02 (6)	0.89 (5)
C5–C6	1.564 (7)	1.570 (9)	C15–H1	1.05 (5)	1.18 (7)
C5–C9	1.569 (9)	1.577 (6)	C15–H2	1.08 (7)	1.15 (6)
C5–C13	1.536 (8)	1.502 (9)	C16–H1	1.09 (6)	1.01 (5)
C6–C7	1.499 (8)	1.501 (7)	C16–H2	0.99 (5)	1.05 (6)
C7–C8	1.311 (9)	1.306 (9)	C17–H1	1.11 (8)	1.18 (7)
C8–C9	1.509 (6)	1.514 (9)	C17–H2	1.18 (8)	0.93 (5)
C9–C14	1.520 (8)	1.565 (9)	C18–H1	1.06 (7)	1.15 (6)
C10–C11	1.531 (10)	1.536 (8)	C18–H2	0.74 (6)	0.86 (6)
C11–C12	1.508 (8)	1.514 (7)	C18–H3	0.95 (6)	1.08 (7)

Table III
Bond Angles in Molecules 1a and 1b with Estimated Standard Deviations[a]

Atoms	Angle, deg 1a	1b	Atoms	Angle, deg 1a	1b
01–C2–C1	128.5	128.2	C14–C15–C16	104.2	104.5
01–C2–C3	108.8	109.4	01–C17–H1	97.6	104.8
C2–01–C17	106.2	105.3	01–C17–H2	104.9	121.5
01–C17–02	108.9	109.6	02–C17–H1	98.9	104.4
02–C3–C2	110.7	110.4	02–C17–H2	99.5	111.8
02–C3–C4	128.2	127.5	03–C18–H1	115.5	113.3
C3–02–C17	104.8	104.0	03–C18–H2	99.6	114.6
03–C7–C6	115.0	114.1	03–C18–H3	107.5	108.4
03–C7–C8	129.7	130.7	04–C6–H	104.5	106.8
C7–03–C18	114.0	115.5	C6–04–H	101.3	100.8
04–C6–C5	116.9	116.0	N–C10–H1	109.1	111.1
04–C6–C7	112.4	110.1	N–C10–H2	112.6	111.7
N–C9–C5	115.0	115.2	N–C16–H1	124.1	115.4
N–C9–C8	114.0	115.4	N–C16–H2	107.8	110.3
N–C9–C14	99.5	99.8	C2–C1–H	125.9	115.5
C9–N–C10	117.1	118.4	C12–C1–H	116.9	125.3
N–C10–C11	113.7	112.2	C3–C4–H	126.8	117.1
C9–N–C16	105.6	104.4	C13–C4–H	115.2	124.7
C10–N–C16	110.5	110.3	C5–C6–H	111.6	115.3
N–C16–C15	104.8	104.3	C6–C5–H	116.8	103.4
C1–C2–C3	122.7	122.4	C9–C5–H	106.5	104.5
C2–C1–C12	117.1	117.3	C13–C5–H	95.5	108.7
C1–C12–C11	119.8	119.9	C7–C6–H	109.1	105.7
C1–C12–C13	120.5	120.8	C7–C8–H	125.8	127.3
C2–C3–C4	121.1	122.0	C9–C8–H	121.4	120.2
C3–C4–C13	117.9	117.8	C9–C14–H1	113.4	112.2
C4–C13–C5	117.6	117.9	C9–C14–H2	111.2	113.7
C4–C13–C12	120.6	119.6	C10–C11–H1	115.6	108.8
C5–C6–C7	102.4	102.5	C10–C11–H2	113.2	101.0
C6–C5–C9	106.9	106.5	C11–C10–H1	107.7	112.2
C5–C9–C8	103.4	103.2	C11–C10–H2	108.7	112.3
C5–C9–C14	112.6	111.3	C12–C11–H1	111.2	103.3
C6–C5–C13	115.6	116.5	C12–C11–H2	114.6	102.7
C9–C5–C13	115.2	115.5	C14–C15–H1	108.3	114.8
C5–C13–C12	121.8	122.4	C14–C15–H2	118.5	104.5
C6–C7–C8	115.2	115.1	C15–C14–H1	110.9	104.7
C7–C8–C9	112.2	112.5	C15–C14–H2	116.2	109.7
C8–C9–C14	112.8	112.4	C15–C16–H1	108.4	112.8
C9–C14–C15	106.1	105.4	C15–C16–H2	115.8	120.1
C10–C11–C12	112.3	113.9	C16–C15–H1	114.4	108.0
C11–C12–C13	119.7	119.2	C16–C15–H2	95.7	107.6

[a] The average estimated standard deviations in angles are 0.3° for angles involving nonhydrogen atoms and 2.0° for angles involving hydrogens.

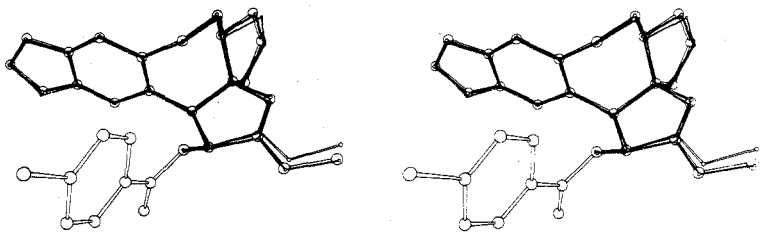


Figure 2. A stereoview comparing **1a**, **1b** (smallest atoms and bonds), and **3** (largest atoms and bonds) after least-squares fitting of the B rings.[5]

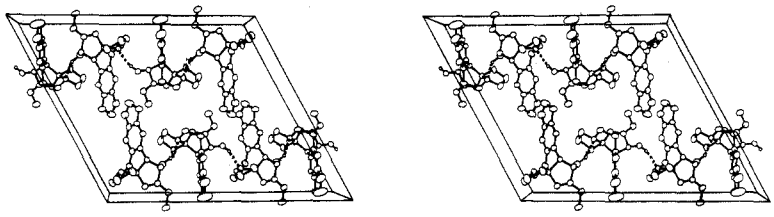


Figure 3. Stereoscopic view of a unit cell, *b* axis projection, with the *a* axis horizontal, the *c* axis approximately vertical, and dashed lines representing hydrogen bonds.

Experimental Section

Collection and Reduction of the Data. Oscillation and Weissenberg photographs of a 0.3 × 0.3 × 0.5 mm needle of **1** grown from ether indicated space group *C*2. The cell parameters were found by least-squares fitting of the settings for four angles of eight reflections on a Picker-FACS-I diffractometer (Cu Kα, λ = 1.54178 Å, graphite monochromator) to be a = 22.834 (5), b = 8.158 (3), c = 19.534 (4) Å, β = 117.7 (1)°, ρ_c = 1.298 g/ml (ρ_0 = 1.291 g/ml), and Z = 8. Intensity data were collected using a scintillation counter with pulse-height analyzer, θ–2θ scan technique, 2°/min scan rate, 10-sec background counts, attenuators when the count rate exceeded 10^4 counts/sec, and 2° scan range with a dispersion factor allowing for α_1-α_2 splitting at large 2θ values. Of 2910 independent reflections measured, $2690 > 3\sigma(I)$ were considered observed. Three standard reflections were monitored every 50 measurements to check crystal alignment and the stability; no decrease in the intensity of standards was observed. Lorentz and polarization corrections were applied to the data, but no correction was made for absorption.

Solution and Refinement. The structure was solved by the vector search method using the coordinates of the cephalotaxine part of cephalotaxine *p*-bromobenzoate;[2] two minima were obtained, corresponding to the two molecules in the asymmetric unit. The initial calculations of structure factors gave an R value of 0.252. Two cycles of isotropic least-squares refinement of nonhydrogen atoms reduced R to 0.122 and two more cycles of anisotropic least-squares refinement of nonhydrogen atoms brought R to 0.077. A difference map at this stage revealed all the hydrogen atoms. One more cycle of least-squares refinement using anisotropic temperature factors for nonhydrogen atoms and isotropic temperature factors (of nonhydrogen atoms to which they were attached) for hydrogen atoms reduced R to 0.052. The refinement was terminated at this stage with the ratios of shifts in parameters to estimated standard deviations all less than 0.3. Refinement was based on F_0, the quantity minimized being $\Sigma\omega(F_0 - F_c)^2$. Unit weights were used throughout the refinement. The scattering factors used were those of Hanson et al.[3] No correction was applied for extinction.

Results and Discussion

Table I gives the observed fractional coordinates in **1a** and **1b**, and Figure 1 shows ORTEP[4] drawings of both molecules. Table II gives bond lengths and Table III bond angles. In Figure 2, the conformations of cephalotaxine molecules **1a** and **1b** and cephalotaxine *p*-bromobenzoate (**3**)[2] are compared after least-squares fitting of the aromatic ring carbons.[5] *The extreme similarity of the conformations of the cephalotaxine portion of these three molecules provides strong support for the view that the antileukemic esters of cephalotaxine, e.g.,* **2**, *share this conformation.*[2] It should be noted that Dreiding models permit considerable flexibility for the seven-membered ring, and from these models, it is not obvious that the observed conformation should be preferred.

Figure 3 shows the molecular packing, governed by hydrogen bonds between the alcohol proton in **1a** and the nitrogen in **1b** (2.92 Å between O4 and N) and the alcohol proton in **1b** and the nitrogen in **1a** (2.94 Å between O4 and N); these bonds form chains of molecules parallel to the diagonals of the *ab* face. Other short intermolecular distances between nonhydrogen atoms are O3–O2 (3.12 Å), O3–C3 (3.25 Å), O4–C16 (3.29 Å), and O3–C2 (3.21 Å).

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Supplementary Material Available. Tables of temperature factors and torsion angles (4 pages). Ordering information is given on any current masthead page.

References and Notes

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Crystal Structure of 1-(*o*-Chlorophenyl)-1-(*p*-chlorophenyl)-2,2-dichloroethane

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1,1-Dichloro-2-(*o*-chlorophenyl)-2-(*p*-chlorophenyl)ethane (I, *o,p'*-DDD), strikingly close in structure to the insecticides *o,p'*- (II) and *p,p'*-DDT (III), is used in the treatment of tumors of the adrenal cortex.[1] However, the mech-