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Structures of Three Products Derived from the Tin- and Aluminium-Mediated Coupling of Aldehydes and Cinnamyl Chloride

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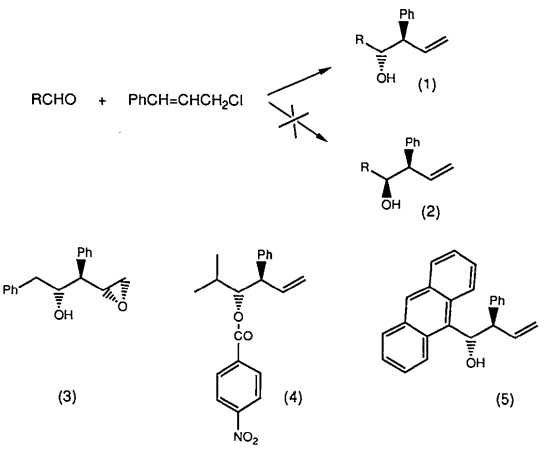
Abstract. (3) (2*RS*,3*RS*,4*RS*)-1,3-Diphenyl-4,5-epoxy-2-pentanol, $C_{17}H_{18}O_2$, $M_r = 254{\cdot}3$, monoclinic, $P2_1/n$, $a = 15{\cdot}243\ (4)$, $b = 9{\cdot}736\ (2)$, $c = 19{\cdot}066\ (4)$ Å, $\beta = 103{\cdot}03\ (2)°$, $V = 2757$ Å^3, $Z = 8$, $D_x = 1{\cdot}23$ Mg m^{-3}, Mo $K\alpha$, $\lambda = 0{\cdot}71069$ Å, $\mu = 0{\cdot}07$ mm^{-1}, $F(000) = 1088$, $T = 150$ K, $R = 0{\cdot}047$ for 2339 independent observed reflections. (4) (3*RS*,4*RS*)-2-Methyl-4-phenyl-5-hexen-3-yl 4-nitrobenzoate, $C_{20}H_{21}NO_4$, $M_r = 339{\cdot}4$, monoclinic, $P2_1/c$, $a = 19{\cdot}521\ (6)$, $b = 6{\cdot}014\ (2)$, $c = 15{\cdot}454\ (5)$ Å, $\beta = 96{\cdot}78\ (3)°$, $V = 1802$ Å^3, $Z = 4$, $D_x = 1{\cdot}25$ Mg m^{-3}, Mo $K\alpha$, $\lambda = 0{\cdot}71069$ Å, $\mu = 0{\cdot}08$ mm^{-1}, $F(000) = 720$, $T = 170$ K, $R = 0{\cdot}044$ for 1182 independent observed reflections. (5) (1*RS*,2*SR*)-1-(9-Anthryl)-2-phenyl-3-buten-1-ol, $C_{24}H_{20}O$, $M_r = 324{\cdot}4$, tetragonal, $P4_22_12$, $a = b = 19{\cdot}920\ (4)$, $c = 9{\cdot}095\ (5)$ Å, $V = 3609$ Å^3, $Z = 8$, $D_m = 1{\cdot}17$, $D_x = 1{\cdot}19$ Mg m^{-3}, Mo $K\alpha$, $\lambda = 0{\cdot}71069$ Å, $\mu = 0{\cdot}08$ mm^{-1}, $F(000) = 1376$, $T = 295$ K, $R = 0{\cdot}046$ for 1756 independent observed reflections. The three structures confirm the stereoselective preference for formation of the *threo* diastereoisomer in Sn/Al-mediated coupling of aldehydes and cinnamyl chloride.

Introduction. We have recently reported the regioselective and diastereoselective coupling of a variety of aldehydes with cinnamyl chloride mediated by tin and aluminium (Coxon, van Eyk & Steel, 1989). A key feature of these reactions is the highly selective formation of the *threo* diastereoisomer (1) in preference to the *erythro* isomer (2). Distinction between the two isomers could not always be made by spectroscopic methods and it was necessary to carry out X-ray structure determinations on a number of the products. Here we report the crystal structures of the major products from reaction of cinnamyl chloride with phenylacetaldehyde (and subsequent epoxidation), with methylpropanal (and subsequent conversion to its 4-nitrobenzoate) and with 9-anthraldehyde.



Experimental. The three compounds (3)–(5) were prepared as previously reported (Coxon, van Eyk & Steel, 1989). Crystal sizes $0{\cdot}49 \times 0{\cdot}23 \times 0{\cdot}08$ mm for (3), $0{\cdot}65 \times 0{\cdot}26 \times 0{\cdot}07$ mm for (4), $0{\cdot}72 \times 0{\cdot}68 \times 0{\cdot}24$ mm for (5). Measured density by flotation in aqueous potassium iodide solution. Nicolet *R3m* diffractometer; lattice parameters from 25 reflections with $2\theta > 25°$. $\theta/2\theta$ data collection to 50 and 55° for (3) and (5) respectively and ω collection to 48° for (4). Standard reflections (and intensity variations) 008, 031, 600 (4·0%) for (3), 004, 031, 500 (2·1%) for (4), 006, 080, 12,0,0 (2·2%) for (5) monitored every

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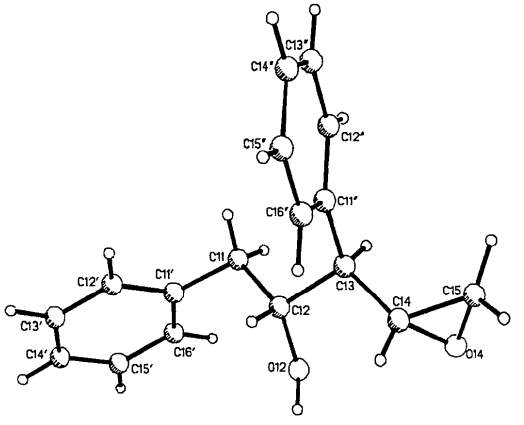
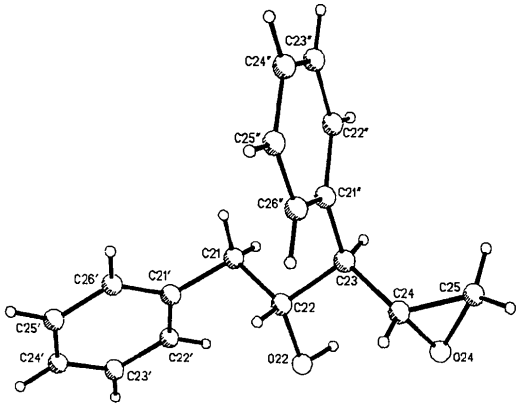
100 measurements. Reflections measured, unique reflections, R_{int}, observed reflections $[I > 3\sigma(I)]$: 5365, 4882, 0·024, 2339 for (3), 2859, 2758, 0·043, 1182 for (4), 2520, 2421, 0·030, 1756 for (5). Corrections for Lorentz and polarization effects but not for absorption.

All structures solved by direct methods and refined on $|F|$ by blocked-cascade least-squares procedures. All non-H atoms anisotropic, H atoms in calculated positions with isotropic thermal parameters equal to the isotropic equivalent of their carrier carbon (C—H = 0·96 Å), except for the O—H hydrogens and the epoxide-ring hydrogens of (3) which were located from difference Fourier syntheses and their positions refined. $w^{-1} = [\sigma^2(F) + g(F)^2]$. $R = 0{\cdot}047$, $wR = 0{\cdot}052$, $g = 0{\cdot}0007$, $S = 1{\cdot}17$, 367 parameters for (3). $R = 0{\cdot}044$, $wR = 0{\cdot}050$, $g = 0{\cdot}0007$, $S = 1{\cdot}14$, 226 parameters for (4). $R = 0{\cdot}046$, $wR = 0{\cdot}059$, $g = 0{\cdot}00045$, $S = 1{\cdot}93$, 226 parameters for (5). $(\Delta/\sigma)_{max}$ 0·191 for (3), 0·064 for (4), 0·030 for (5). $(\Delta\rho)_{max} =$ 0·17, 0·16, 0·20, $(\Delta\rho)_{min} = -0{\cdot}19$, $-0{\cdot}19$,



Molecule 1



Molecule 2

Fig. 1. Perspective view and atom labelling of each of the two independent molecules of (2*RS*,3*RS*,4*RS*)-1,3-diphenyl-4,5-epoxy-2-pentanol (3).

Table 1. *Fractional atomic coordinates and equivalent isotropic temperature factors*

U_{eq} is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	*y*	*z*	U_{eq}(Å²)
Compound (3)				
O(12)	0·7736 (1)	0·5555 (2)	0·2123 (1)	0·026 (1)
O(14)	0·6049 (1)	0·4960 (3)	0·0813 (1)	0·037 (1)
C(11)	0·8754 (2)	0·7169 (3)	0·1793 (2)	0·029 (1)
C(12)	0·8226 (2)	0·5845 (3)	0·1582 (2)	0·023 (1)
C(13)	0·7581 (2)	0·5961 (3)	0·0837 (2)	0·022 (1)
C(14)	0·6942 (2)	0·4755 (3)	0·0693 (2)	0·026 (1)
C(15)	0·6170 (2)	0·4793 (4)	0·0092 (2)	0·033 (1)
C(11′)	0·9471 (2)	0·7044 (3)	0·2482 (2)	0·026 (1)
C(12′)	1·0337 (2))	0·6616 (4)	0·2471 (2)	0·031 (1)
C(13′)	1·0981 (2))	0·6447 (4)	0·3101 (2)	0·036 (1)
C(14′)	1·0778 (2))	0·6703 (4)	0·3755 (2)	0·033 (1)
C(15′)	0·9927 (2)	0·7148 (3)	0·3779 (2)	0·031 (1)
C(16′)	0·9277 (2)	0·7313 (3)	0·3149 (2)	0·027 (1)
C(11″)	0·8098 (2)	0·6067 (3)	0·0248 (2)	0·024 (1)
C(12″)	0·8151 (2)	0·7300 (4)	−0·0108 (2)	0·029 (1)
C(13″)	0·8612 (2)	0·7370 (4)	−0·0653 (2)	0·039 (1)
C(14″)	0·9033 (2)	0·6226 (4)	−0·0849 (2)	0·043 (1)
C(15″)	0·8988 (2)	0·4996 (4)	−0·0501 (2)	0·039 (1)
C(16″)	0·8522 (2)	0·4912 (4)	0·0043 (2)	0·032 (1)
O(22)	0·6968 (2)	0·7669 (2)	0·2854 (1)	0·027 (1)
O(24)	0·6377 (2)	0·8623 (2)	0·1382 (1)	0·036 (1)
C(21)	0·6107 (2)	0·5808 (3)	0·3226 (2)	0·031 (1)
C(22)	0·6071 (2)	0·7182 (3)	0·2836 (2)	0·025 (1)
C(23)	0·5520 (2)	0·7105 (3)	0·2057 (2)	0·023 (1)
C(24)	0·5582 (2)	0·8424 (4)	0·1663 (2)	0·030 (1)
C(25)	0·5520 (2)	0·8442 (4)	0·0887 (2)	0·036 (1)
C(21′)	0·6521 (2)	0·5905 (3)	0·4021 (2)	0·027 (1)
C(22′)	0·7442 (2)	0·5878 (4)	0·4275 (2)	0·041 (1)
C(23′)	0·7806 (3)	0·6013 (5)	0·5001 (3)	0·069 (2)
C(24′)	0·7295 (4)	0·6161 (5)	0·5483 (3)	0·087 (2)
C(25′)	0·6379 (4)	0·6163 (4)	0·5243 (2)	0·074 (2)
C(26′)	0·5992 (3)	0·6046 (4)	0·4512 (2)	0·048 (2)
C(21″)	0·4548 (2)	0·6754 (3)	0·2026 (2)	0·024 (1)
C(22″)	0·4188 (2)	0·5510 (3)	0·1745 (2)	0·029 (1)
C(23″)	0·3302 (2)	0·5171 (4)	0·1720 (2)	0·037 (1)
C(24″)	0·2759 (2)	0·6070 (4)	0·1992 (2)	0·037 (1)
C(25″)	0·3103 (2)	0·7301 (4)	0·2281 (2)	0·034 (1)
C(26″)	0·3988 (2)	0·7647 (4)	0·2292 (2)	0·029 (1)
Compound (4)				
C(1)	0·2706 (2)	0·9888 (8)	0·1050 (3)	0·051 (2)
C(1*a*)	0·3262 (2)	0·6163 (8)	0·0937 (3)	0·051 (2)
C(2)	0·3086 (2)	0·8005 (7)	0·1546 (3)	0·040 (2)
C(3)	0·2694 (2)	0·7182 (7)	0·2288 (3)	0·035 (1)
C(4)	0·3035 (2)	0·5329 (7)	0·2871 (3)	0·035 (1)
C(5)	0·2625 (2)	0·4938 (8)	0·3616 (3)	0·038 (2)
C(6)	0·2283 (2)	0·3137 (8)	0·3757 (3)	0·053 (2)
C(11)	0·3781 (2)	0·5820 (8)	0·3210 (3)	0·039 (2)
C(12)	0·3977 (2)	0·7782 (8)	0·3634 (3)	0·050 (2)
C(13)	0·4663 (3)	0·8119 (11)	0·3974 (3)	0·073 (2)
C(14)	0·5145 (3)	0·6516 (11)	0·3887 (3)	0·085 (3)
C(15)	0·4953 (2)	0·4554 (10)	0·3475 (4)	0·081 (2)
C(16)	0·4274 (2)	0·4230 (9)	0·3127 (3)	0·057 (2)
O(1)	0·2038 (1)	0·6240 (5)	0·1896 (2)	0·035 (1)
O(2)	0·1430 (1)	0·9001 (5)	0·2429 (2)	0·057 (1)
C(20)	0·1457 (2)	0·7273 (7)	0·2048 (3)	0·037 (2)
C(21)	0·0833 (2)	0·6002 (7)	0·1668 (2)	0·030 (1)
C(22)	0·0190 (2)	0·7005 (7)	0·1686 (2)	0·032 (1)
C(23)	−0·0403 (2)	0·5962 (7)	0·1330 (2)	0·033 (2)
C(24)	−0·0346 (2)	0·3920 (7)	0·0964 (2)	0·031 (1)
C(25)	0·0279 (2)	0·2843 (7)	0·0939 (2)	0·033 (1)
C(26)	0·0873 (2)	0·3928 (7)	0·1296 (2)	0·035 (1)
N(1)	−0·0976 (2)	0·2789 (6)	0·0556 (2)	0·040 (1)
O(11)	−0·1511 (1)	0·3856 (5)	0·0448 (2)	0·051 (1)
O(12)	−0·0935 (1)	0·0844 (5)	0·0333 (2)	0·058 (1)
Compound (5)				
O(1)	0·0867 (1)	−0·0069 (1)	0·0666 (2)	0·045 (1)
C(1)	0·1578 (1)	−0·0008 (1)	0·0807 (3)	0·039 (1)

Table 1 (*cont.*)

	x	*y*	*z*	U_{eq}(Å^2)
C(2)	0·1768 (1)	0·0338 (1)	0·2295 (3)	0·039 (1)
C(3)	0·1436 (1)	0·1014 (1)	0·2398 (3)	0·047 (1)
C(4)	0·0914 (2)	0·1155 (2)	0·3217 (4)	0·073 (1)
C(1′)	0·2677 (2)	−0·0271 (2)	−0·1328 (3)	0·048 (1)
C(2′)	0·3178 (2)	−0·0378 (2)	−0·2313 (3)	0·057 (1)
C(3′)	0·3481 (2)	−0·1019 (2)	−0·2438 (4)	0·061 (1)
C(4′)	0·3261 (2)	−0·1529 (2)	−0·1575 (4)	0·058 (1)
C(4a′)	0·2733 (1)	−0·1438 (1)	−0·0543 (3)	0·047 (1)
C(5′)	0·1778 (2)	−0·2436 (2)	0·2237 (4)	0·057 (1)
C(6′)	0·1288 (2)	−0·2360 (2)	0·3256 (4)	0·060 (1)
C(7′)	0·0958 (2)	−0·1733 (2)	0·3395 (3)	0·055 (1)
C(8′)	0·1138 (1)	−0·1198 (1)	0·2546 (3)	0·048 (1)
C(8a′)	0·1674 (1)	−0·1245 (1)	0·1497 (3)	0·041 (1)
C(9′)	0·1899 (1)	−0·0698 (1)	0·0637 (3)	0·038 (1)
C(9a′)	0·2425 (1)	−0·0786 (1)	−0·0391 (3)	0·042 (1)
C(10′)	0·2505 (2)	−0·1965 (1)	0·0318 (3)	0·051 (1)
C(10*a*)	0·1989 (1)	−0·1890 (1)	0·1331 (3)	0·046 (1)
C(1″)	0·2522 (1)	0·0386 (1)	0·2467 (3)	0·038 (1)
C(2″)	0·2854 (1)	0·0036 (1)	0·3570 (3)	0·044 (1)
C(3″)	0·3546 (2)	0·0067 (2)	0·3715 (3)	0·053 (1)
C(4″)	0·3919 (2)	0·0439 (2)	0·2745 (4)	0·060 (1)
C(5″)	0·3603 (2)	0·0792 (2)	0·1639 (4)	0·057 (1)
C(6″)	0·2912 (2)	0·0772 (1)	0·1509 (3)	0·048 (1)

Table 2. *Bond lengths* (Å) *with e.s.d.'s in parentheses*

Compound (3)

	Mol. 1	Mol. 2		Mol. 1	Mol. 2
O(12)—C(12)	1·432 (4)	1·441 (4)	O(14)—C(14)	1·443 (4)	1·443 (4)
O(14)—C(15)	1·437 (4)	1·439 (4)	C(11)—C(12)	1·524 (4)	1·525 (5)
C(11)—C(11′)	1·513 (4)	1·508 (4)	C(12)—C(13)	1·538 (4)	1·535 (4)
C(13)—C(14)	1·511 (4)	1·502 (5)	C(13)—C(11″)	1·514 (4)	1·509 (4)
C(14)—C(15)	1·447 (4)	1·460 (5)	C(11′)—C(12′)	1·390 (5)	1·377 (5)
C(11′)—C(16′)	1·393 (5)	1·372 (6)	C(12′)—C(13′)	1·378 (4)	1·377 (6)
C(13′)—C(14′)	1·374 (5)	1·340 (8)	C(14′)—C(15′)	1·379 (5)	1·368 (8)
C(15′)—C(16′)	1·383 (4)	1·391 (5)	C(11″)—C(12″)	1·390 (5)	1·386 (4)
C(11″)—C(16″)	1·397 (5)	1·391 (5)	C(12″)—C(13″)	1·381 (5)	1·381 (5)
C(13″)—C(14″)	1·378 (6)	1·384 (5)	C(14″)—C(15″)	1·379 (6)	1·373 (5)
C(15″)—C(16″)	1·384 (5)	1·386 (5)			

Compound (4)

C(1)—C(2)	1·512 (6)	C(1a)—C(2)	1·518 (6)
C(2)—C(3)	1·536 (6)	C(3)—C(4)	1·535 (6)
C(3)—O(1)	1·463 (4)	C(4)—C(5)	1·496 (6)
C(4)—C(11)	1·518 (5)	C(5)—C(6)	1·304 (7)
C(11)—C(12)	1·382 (6)	C(11)—C(16)	1·374 (6)
C(12)—C(13)	1·393 (6)	C(13)—C(14)	1·366 (8)
C(14)—C(15)	1·372 (8)	C(15)—C(16)	1·384 (6)
O(1)—C(20)	1·339 (5)	O(2)—C(20)	1·198 (5)
C(20)—C(21)	1·499 (5)	C(21)—C(22)	1·395 (5)
C(21)—C(26)	1·380 (6)	C(22)—C(23)	1·374 (5)
C(23)—C(24)	1·362 (6)	C(24)—C(25)	1·385 (5)
C(24)—N(1)	1·480 (5)	C(25)—C(26)	1·387 (5)
N(1)—O(11)	1·219 (4)	N(1)—O(12)	1·225 (4)

Compound (5)

O(1)—C(1)	1·428 (3)	C(1)—C(2)	1·564 (4)
C(1)—C(9′)	1·524 (4)	C(2)—C(3)	1·503 (4)
C(2)—C(1″)	1·514 (4)	C(3)—C(4)	1·310 (5)
C(1′)—C(2′)	1·358 (4)	C(1′)—C(9a′)	1·425 (4)
C(2′)—C(3′)	1·416 (5)	C(3′)—C(4′)	1·356 (5)
C(4′)—C(4a′)	1·421 (4)	C(4a′)—C(9a′)	1·444 (4)
C(4a′)—C(10′)	1·387 (4)	C(5′)—C(6′)	1·354 (5)
C(5′)—C(10a)	1·429 (4)	C(6′)—C(7′)	1·416 (5)
C(7′)—C(8′)	1·364 (4)	C(8′)—C(8a′)	1·434 (4)
C(8a′)—C(9′)	1·413 (4)	C(8a′)—C(10a)	1·438 (4)
C(9′)—C(9a′)	1·415 (4)	C(10′)—C(10a)	1·389 (4)
C(1″)—C(2″)	1·390 (4)	C(1″)—C(6″)	1·397 (4)
C(2″)—C(3″)	1·386 (4)	C(3″)—C(4″)	1·371 (5)
C(4″)—C(5″)	1·380 (5)	C(5″)—C(6″)	1·382 (4)

$-0·15$ e Å^{-3} for (3), (4), (5) respectively. Scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV). All calculations, including diagrams, on a Nova 4*X* computer using *SHELXTL* (Sheldrick, 1983).

Discussion. Fig. 1 shows perspective views and atom labelling of the two independent molecules of (2*RS*,3*RS*,4*RS*)-1,3-diphenyl-4,5-epoxy-2-pentanol (3) in the asymmetric unit. The structure determination thus confirms that the coupling reaction produces the *threo* product and, in addition, distinguishes the relative stereochemistry of the two epoxides produced in the subsequent epoxidation reaction. Tables 1* and 2 list atomic coordinates and bonding parameters respectively for (3). The two independent molecules have similar bonding geometries and exist in similar conformations except for small torsional differences; for example the mean planes through the two phenyl rings of molecule 1

* Lists of structure factors, anisotropic thermal parameters, bond angles and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53245 (42 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

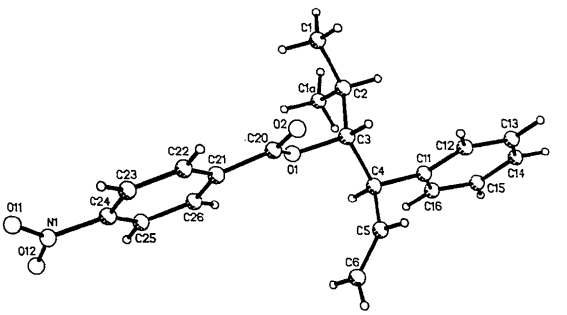


Fig. 2. Perspective view and atom labelling of (3*RS*,4*RS*)-2-methyl-4-phenyl-5-hexen-3-yl 4-nitrobenzoate (4).

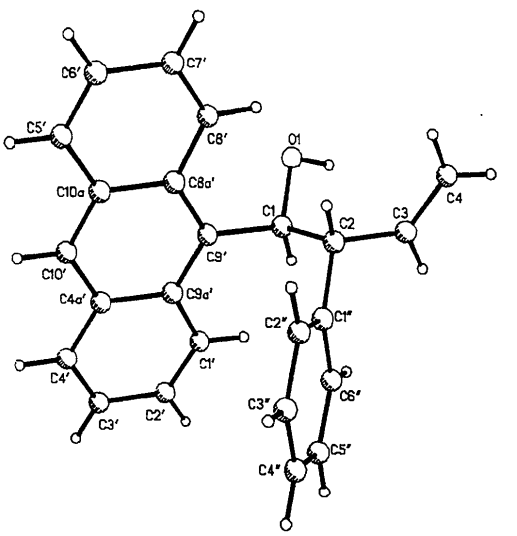


Fig. 3. Perspective view and atom labelling of (1*RS*,2*SR*)-1-(9-anthyl)-2-phenyl-3-buten-1-ol (5).

are mutually inclined at an angle of 66·3 (3)° while the corresponding value for molecule 2 is 59·4 (3)°. All four independent phenyl rings are planar to within 0·006 Å. Steric interactions are minimized by the adoption of staggered conformations about the C—C single bonds. The crystal packing is determined by a network of hydrogen bonding wherein the hydroxyl groups form hydrogen-bonded chains with two independent O—H···O distances of 2·877 (4) and 2·845 (4) Å.

Fig. 2 shows a perspective view and atom labelling of the structure of (3*RS*,4*RS*)-2-methyl-4-phenyl-5-hexen-3-yl 4-nitrobenzoate (4), which again confirms the structure as the *threo* product. Tables 1 and 2 list atomic coordinates and bonding parameters respectively. Both phenyl rings are planar to within 0·008 Å and are inclined to one another at an angle of 5·9 (3)°; the carbonyl group lies in the plane of the nitrophenyl ring. Although no short intermolecular contacts (< 3·1 Å) exist, the molecules pack in layers with the nitrophenyl groups stacked down the *c* axis.

Fig. 3 shows a perspective view and atom labelling of the structure of (1*RS*,2*SR*)-1-(9-anthryl)-2-phenyl-3-buten-1-ol (5) which again confirms the structure as the *threo* product. Tables 1 and 2 list atomic coordinates and bonding parameters respectively. The anthryl ring is planar to within 0·02 Å. In the solid state the molecules form a hydrogen-bonded cyclic tetramer wherein four hydroxyl groups are hydrogen bonded in a cyclic manner about the four-fold axis with an O—H···O distance of 2·733 (3) Å.

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Structure of 4-[(1-Amidiniohydrazono)ethyl]-3-methyl-1-pyrazolecarboxamidinium Dichloride Monohydrate

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Abstract. 4-Acetyl-1-amidino-3-methylpyrazole amidinohydrazone dihydrochloride monohydrate, $C_8H_{16}N_8^{2+}.2Cl^-.H_2O$, $M_r = 313{\cdot}19$, triclinic, $P\bar{1}$, $a = 5{\cdot}488$ (2), $b = 9{\cdot}191$ (4), $c = 15{\cdot}013$ (3) Å, $\alpha = 72{\cdot}09$ (3), $\beta = 85{\cdot}51$ (3), $\gamma = 85{\cdot}36$ (4)°, $V = 717$ (6) Å^3, $Z = 2$, $D_x = 1{\cdot}448$ Mg m^{-3}, λ(Mo $K\alpha$) = 0·7107 Å, $\mu = 0{\cdot}45$ mm^{-1}, $F(000) = 328$, $T = 293$ K, $R = 0{\cdot}057$ for 1699 independent reflections. The structural likeness of the title compound and methylglyoxal bis(guanylhydrazone) dihydrochloride (MGBG), which is clearly apparent from this study, should help in interpreting the similarity of some of their biological properties.

Introduction. Among anticancer drugs, mitoguazone or methylglyoxal bis(guanylhydrazone) dihydrochloride (MGBG) (Mihich, 1975), (1), has the striking feature of being structurally related to spermidine (French, Freedlander, Hosking & French, 1960), and is a good inhibitor of the enzyme *S*-adenosylmethioninedecarboxylase (SAM-DC, 4.1.1.50; *Enzyme Nomenclature*, 1978) (Porter, Dave & Mihich, 1981). In our search for new analogs of MGBG, we prepared a homogeneous series of isomeric 1-amidino-4-acylpyrazole amidinohydrazones (Menichi, Naciri, Kokel & Hubert-Habart, 1984; Menichi, Boutar, Kokel, Takagi & Hubert-Habart,

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