

Related literature. As part of studies of the asymmetric 1,4-addition reactions of 2-propene-1-phosphonamides (Hua, Chan-Yu-King, McKie & Myer, 1987), the title compound was prepared for the stereospecific displacement reactions with various organometallic reagents. So far, the configurations of 2-substituted 1,3,2-oxazaphospholidin-2-ones were assigned by NMR (nuclear magnetic resonance) spectroscopy (Cooper, Hall, Harrison & Inch, 1977), and only structures of the 3-methyl 2-sulfides (Bartczak & Galdecki, 1983; Bartczak, Galdecki & Rutkowska, 1983), 3-methyl-2-(methylthio) 2-oxide (Bartczak, Galdecki, Antipin & Struchkov, 1984), and 3-methyl-2-phenoxy 2-oxide derivatives (Setzer, Black & Hubbard, 1990) were reported. Since the configuration of the starting amino alcohol is known, the structure reported herein also establishes the absolute configuration of related compounds prepared similarly.

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Structures of a Pair of Diastereomeric Salts of Ephedrine and Dichloro-Substituted Cyclic Phosphoric Acid

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Abstract. (1) (*R*)-(+)-4-(2,6-Dichlorophenyl)-2-hydroxy-5,5-dimethyl-1,3,2-dioxaphosphorinane 2-oxide (1*S*,2*R*)-(–)- α -[(1-methylamino)ethyl]benzyl alcohol, (–)-C₁₀H₁₆NO⁺.(+)-C₁₁H₁₂Cl₂O₄P[–], *M_r* = 476.335, monoclinic, *P*2₁, *a* = 10.063 (1), *b* = 8.206 (1), *c* = 14.856 (1) Å, β = 104.84 (1)°, *V* = 1185.8 (2) Å³, *Z* = 2, *D_x* = 1.334 Mg m^{–3}, Mo *K* α radiation (graphite monochromator), λ = 0.71073 Å, μ (Mo *K* α) = 0.3689 mm^{–1}, *F*(000) = 500, *T* = 130 K, final *R* = 0.053, *wR* = 0.056 for 3504 observed unique reflections and 281 variables. (2) (*S*)-(–)-4-(2,6-Dichlorophenyl)-2-hydroxy-5,5-dimethyl-1,3,2-

dioxaphosphorinane 2-oxide (1*R*,2*S*)-(–)- α -[(1-methylamino)ethyl]benzyl alcohol hydrate, (–)-C₁₀H₁₆NO⁺.(–)-C₁₁H₁₂Cl₂O₄P[–].H₂O, *M_r* = 494.351, orthorhombic, *P*2₁2₁, *a* = 6.502 (1), *b* = 11.393 (1), *c* = 32.739 (1) Å, *V* = 2425.2 (4) Å³, *Z* = 4, *D_x* = 1.354 Mg m^{–3}, Mo *K* α radiation (graphite monochromator), λ = 0.71073 Å, μ (Mo *K* α) = 0.3660 mm^{–1}, *F*(000) = 1040, *T* = 130 K, final *R* = 0.055, *wR* = 0.059 for 4225 observed unique reflections and 265 variables. For both (1) and (2), the phosphorinane ring is in the usual chair conformation and the ephedrine is in an extended form. The crystal packing of (1) can be regarded as chains parallel to the *b* axis consisting of phosphorinane

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Table 1. *Crystal and experimental data*

| | (1) | (2) |
|---|--------------------|--------------------|
| Crystal dimensions (mm) | 0.30 × 0.30 × 0.25 | 0.35 × 0.20 × 0.25 |
| No. of reflections for lattice parameters, θ range (°) | 25, 9.6–18.9 | 25, 10.1–15.2 |
| ($\sin\theta/\lambda$) _{max} (Å ⁻¹) | 0.745 | 0.767 |
| <i>hkl</i> range | | |
| <i>h</i> | 0–15 | 0–9 |
| <i>k</i> | 0–12 | 0–7 |
| <i>l</i> | –22–22 | –50–50 |
| No. of standard reflections | 3 | 3 |
| Drift correction range | 0.957–1.025 | 0.979–1.129 |
| Total data measured | 4368 | 5091 |
| Unique data [$I > 3\sigma(I)$] | 3535 | 4230 |
| Data used in the refinement | 3504 | 4225 |
| No. of parameters refined | 281 | 265 |
| <i>R</i> | 0.053 | 0.055 |
| <i>wR</i> | 0.056 | 0.059 |
| $w = [\sigma^2(F) + g^*F^2]^{-1}$ | | |
| Weighting scheme parameter, <i>g</i> | 0.002 | 0.001 |
| <i>S</i> | 1.32 | 6.07 |
| Max shift/e.s.d. in last cycle | | |
| for non-H atoms | 0.013 | 0.033 |
| for H atoms | 0.056 | 0.234 |
| Final difference Fourier map peaks min./max. (e Å ⁻³) | –0.49/0.33 | –0.66/0.79 |

anions and ephedrine cations linked by hydrogen bonds. In the crystal packing of (2), which can also be regarded as chains parallel to the *b* axis, the solvent water molecules are incorporated in the hydrogen-bonding pattern between the phosphorinane and ephedrine ions.

Experimental. Colorless block-shaped crystals prepared from equimolar amounts of (–)-ephedrine and (+)-phosphorinane dissolved in ethanol for (1) and equimolar amounts of (–)-ephedrine and (–)-phosphorinane dissolved in 2-propanol/water (3/1) for (2), by slow evaporation. Suitable crystals for (2) obtained after recrystallization from ethyl acetate/ethanol (4/1). Data sets collected on Enraf–Nonius CAD-4F κ -controlled diffractometer using graphite-monochromatized Mo $K\alpha$ radiation using θ – 2θ scan [$0 < \theta < 32^\circ$ for (1); $0 < \theta < 33^\circ$ for (2)] with scan angle of $0.75 + 0.35\tan\theta^\circ$ for (1), $0.85 + 0.35\tan\theta^\circ$ for (2) and variable scan rate with a max. scan time of 1.5 s per reflection at 130 K (van Bolhuis, 1971; Nes & van Bolhuis, 1978). Profile analysis not performed. Lorentz–polarization corrections applied, but no absorption corrections made. Data pertinent to the intensity data collection and refinement are given in Table 1.* Both structures solved by direct methods (*SDP/PDP* software, 1985) and difference Fourier syntheses used to find remaining non-H

Table 2. *Atomic coordinates and equivalent isotropic thermal parameters of non-H atoms*

U_{eq} is defined as one third of the trace of the orthogonalized U_{ij} tensor. The first line gives the values for (1), the second line values for (2).

| | <i>x</i> | <i>y</i> | <i>z</i> | U_{eq} (Å ²) |
|--------|--------------|--------------|--------------|----------------------------|
| Cl(1) | 0.55235 (13) | 0.7375 | 0.15903 (8) | 0.0809 (4) |
| | 0.94757 (16) | 0.47950 (8) | 0.05320 (3) | 0.0210 (2) |
| Cl(2) | 0.96401 (9) | 0.3055 (2) | 0.31621 (6) | 0.0478 (2) |
| | 0.83764 (17) | 0.06361 (8) | 0.13661 (4) | 0.0256 (3) |
| P(2) | 0.72991 (7) | 0.7740 (2) | 0.45196 (5) | 0.0325 (2) |
| | 0.83091 (14) | 0.49050 (7) | 0.18428 (3) | 0.0118 (2) |
| O(1) | 0.8417 (2) | 0.9074 (3) | 0.44502 (17) | 0.0414 (7) |
| | 0.5843 (4) | 0.4962 (2) | 0.18287 (9) | 0.0182 (7) |
| O(3) | 0.69722 (19) | 0.6924 (3) | 0.35093 (14) | 0.0356 (5) |
| | 0.8870 (4) | 0.4364 (2) | 0.14043 (8) | 0.0127 (6) |
| O(21) | 0.6029 (2) | 0.8625 (4) | 0.46071 (17) | 0.0418 (6) |
| | 0.9050 (5) | 0.6141 (2) | 0.18615 (9) | 0.0184 (7) |
| O(22) | 0.7901 (3) | 0.6481 (4) | 0.52278 (16) | 0.0475 (7) |
| | 0.8964 (4) | 0.4067 (2) | 0.21709 (8) | 0.0165 (7) |
| C(4) | 0.8147 (3) | 0.6249 (4) | 0.32449 (18) | 0.0307 (7) |
| | 0.7780 (5) | 0.3296 (3) | 0.13025 (12) | 0.0128 (8) |
| C(5) | 0.9204 (3) | 0.7612 (4) | 0.3219 (2) | 0.0377 (8) |
| | 0.5438 (5) | 0.3546 (3) | 0.12657 (12) | 0.0145 (9) |
| C(6) | 0.9614 (3) | 0.8407 (5) | 0.4187 (2) | 0.0442 (9) |
| | 0.4729 (6) | 0.3941 (3) | 0.16863 (12) | 0.0175 (9) |
| C(41) | 0.7650 (2) | 0.5247 (3) | 0.23359 (12) | 0.0342 (7) |
| | 0.8821 (4) | 0.26879 (18) | 0.09294 (7) | 0.0146 (9) |
| C(42) | 0.6555 (2) | 0.5726 (3) | 0.16002 (12) | 0.0496 (10) |
| | 0.9538 (4) | 0.32949 (18) | 0.05879 (7) | 0.0171 (9) |
| C(43) | 0.6205 (2) | 0.4800 (3) | 0.07858 (12) | 0.0665 (15) |
| | 1.0486 (4) | 0.26865 (18) | 0.02696 (7) | 0.0255 (11) |
| C(44) | 0.6949 (2) | 0.3397 (3) | 0.07071 (12) | 0.0662 (15) |
| | 1.0716 (4) | 0.14709 (18) | 0.02928 (7) | 0.0307 (12) |
| C(45) | 0.8044 (2) | 0.2918 (3) | 0.14428 (12) | 0.0551 (10) |
| | 0.9999 (4) | 0.08639 (18) | 0.06344 (7) | 0.0263 (11) |
| C(46) | 0.8395 (2) | 0.3844 (3) | 0.22572 (12) | 0.0391 (8) |
| | 0.9051 (4) | 0.14724 (18) | 0.09526 (7) | 0.0181 (9) |
| C(51) | 1.0535 (4) | 0.6911 (6) | 0.3054 (3) | 0.0564 (13) |
| | 0.4898 (6) | 0.4493 (4) | 0.09488 (13) | 0.0208 (10) |
| C(52) | 0.8648 (5) | 0.8892 (5) | 0.2484 (3) | 0.0567 (13) |
| | 0.4270 (7) | 0.2409 (4) | 0.11621 (15) | 0.0262 (13) |
| O(10) | 0.2841 (3) | 0.0737 (4) | 0.3244 (2) | 0.0525 (8) |
| | 0.6348 (4) | 0.7889 (2) | 0.16259 (9) | 0.0188 (8) |
| N(2) | 0.5326 (3) | 0.2032 (4) | 0.42275 (19) | 0.0400 (7) |
| | 0.3571 (5) | 0.7960 (3) | 0.22918 (9) | 0.0143 (8) |
| C(1) | 0.3200 (3) | 0.2316 (5) | 0.3014 (2) | 0.0426 (9) |
| | 0.4252 (6) | 0.7580 (3) | 0.15609 (11) | 0.0143 (8) |
| C(2) | 0.4750 (3) | 0.2469 (5) | 0.3226 (2) | 0.0425 (9) |
| | 0.2950 (6) | 0.8279 (3) | 0.18692 (11) | 0.0148 (8) |
| C(3) | 0.5389 (5) | 0.1383 (8) | 0.2623 (3) | 0.0660 (14) |
| | 0.3218 (7) | 0.9608 (3) | 0.18217 (13) | 0.0232 (10) |
| C(11) | 0.2537 (3) | 0.2745 (5) | 0.20107 (17) | 0.0609 (15) |
| | 0.3569 (4) | 0.7808 (2) | 0.11220 (6) | 0.0153 (9) |
| C(12) | 0.2488 (3) | 0.4371 (5) | 0.17295 (17) | 0.082 (2) |
| | 0.4894 (4) | 0.8357 (2) | 0.08466 (6) | 0.0216 (10) |
| C(13) | 0.1906 (3) | 0.4780 (5) | 0.08005 (17) | 0.127 (4) |
| | 0.4267 (4) | 0.8551 (2) | 0.04448 (6) | 0.0260 (11) |
| C(14) | 0.1373 (3) | 0.3563 (5) | 0.01528 (17) | 0.155 (6) |
| | 0.2315 (4) | 0.8195 (2) | 0.03183 (6) | 0.0262 (11) |
| C(15) | 0.1423 (3) | 0.1937 (5) | 0.04341 (17) | 0.143 (6) |
| | 0.0990 (4) | 0.7645 (2) | 0.05936 (6) | 0.0235 (11) |
| C(16) | 0.2005 (3) | 0.1529 (5) | 0.13630 (17) | 0.087 (2) |
| | 0.1618 (4) | 0.7452 (2) | 0.09955 (6) | 0.0205 (10) |
| C(20) | 0.6756 (3) | 0.2614 (6) | 0.4641 (3) | 0.0614 (13) |
| | 0.3172 (7) | 0.6718 (3) | 0.24202 (11) | 0.0176 (9) |
| O(101) | – | – | – | – |
| | 0.2284 (5) | 0.2656 (4) | 0.24511 (13) | 0.0359 (11) |

* Lists of structure factors, anisotropic thermal parameters, H-atom parameters and full bond lengths, angles and torsion angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54719 (56 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: AB0249]

atoms and all H atoms. Full-matrix least-squares refinement based on *F* (*SHELX76*; Sheldrick, 1976), with H atoms at constrained positions. Anisotropic thermal parameters for all non-H atoms; for (1), H

Table 3. Selected bond lengths (Å), bond angles (°) and torsion angles (°) for non-H atoms

| | (1) | (2) |
|-----------------------|-------------|-------------|
| Cl(1)—C(42) | 1.703 (2) | 1.719 (2) |
| Cl(2)—C(46) | 1.713 (2) | 1.713 (3) |
| P(2)—O(1) | 1.592 (3) | 1.605 (3) |
| P(2)—O(3) | 1.599 (2) | 1.604 (3) |
| P(2)—O(21) | 1.504 (3) | 1.490 (3) |
| P(2)—O(22) | 1.488 (3) | 1.499 (3) |
| O(1)—C(6) | 1.464 (4) | 1.447 (4) |
| O(3)—C(4) | 1.448 (4) | 1.447 (4) |
| C(4)—C(5) | 1.551 (5) | 1.554 (5) |
| C(4)—C(41) | 1.550 (3) | 1.559 (4) |
| C(5)—C(6) | 1.536 (4) | 1.520 (5) |
| C(5)—C(51) | 1.534 (5) | 1.537 (6) |
| C(5)—C(52) | 1.515 (5) | 1.539 (6) |
| O(10)—C(1) | 1.410 (5) | 1.424 (5) |
| N(2)—C(2) | 1.495 (4) | 1.486 (5) |
| N(2)—C(20) | 1.491 (5) | 1.499 (5) |
| C(1)—C(2) | 1.515 (4) | 1.539 (5) |
| C(1)—C(11) | 1.510 (4) | 1.526 (4) |
| C(2)—C(3) | 1.518 (6) | 1.532 (5) |
| O(1)—P(2)—O(3) | 101.89 (13) | 102.52 (15) |
| O(1)—P(2)—O(21) | 107.68 (17) | 106.62 (15) |
| O(1)—P(2)—O(22) | 110.90 (15) | 109.28 (15) |
| O(3)—P(2)—O(21) | 107.95 (13) | 109.05 (15) |
| O(3)—P(2)—O(22) | 108.76 (17) | 109.39 (14) |
| O(21)—P(2)—O(22) | 118.36 (16) | 118.74 (16) |
| P(2)—O(1)—C(6) | 113.8 (2) | 118.5 (2) |
| P(2)—O(3)—C(4) | 115.40 (17) | 114.7 (2) |
| O(3)—C(4)—C(5) | 110.1 (3) | 110.1 (3) |
| O(3)—C(4)—C(41) | 109.6 (2) | 110.0 (3) |
| C(5)—C(4)—C(41) | 115.5 (2) | 116.5 (3) |
| C(4)—C(5)—C(6) | 107.7 (2) | 106.3 (3) |
| C(4)—C(5)—C(51) | 111.5 (3) | 113.9 (3) |
| C(4)—C(5)—C(52) | 112.9 (3) | 110.3 (3) |
| C(6)—C(5)—C(51) | 105.8 (3) | 109.5 (3) |
| C(6)—C(5)—C(52) | 110.0 (3) | 107.4 (3) |
| C(51)—C(5)—C(52) | 108.7 (3) | 109.2 (3) |
| O(1)—C(6)—C(5) | 111.5 (2) | 112.2 (3) |
| C(4)—C(41)—C(42) | 123.1 (2) | 123.6 (2) |
| C(4)—C(41)—C(46) | 116.76 (19) | 116.4 (2) |
| Cl(1)—C(42)—C(41) | 126.01 (17) | 124.77 (18) |
| Cl(1)—C(42)—C(43) | 114.03 (16) | 115.14 (17) |
| Cl(2)—C(26)—C(41) | 123.16 (15) | 124.60 (18) |
| Cl(2)—C(46)—C(45) | 116.52 (18) | 115.28 (16) |
| C(2)—N(2)—C(20) | 114.9 (3) | 116.4 (3) |
| O(10)—C(1)—C(2) | 109.9 (3) | 107.5 (3) |
| O(10)—C(1)—C(11) | 112.2 (3) | 112.2 (3) |
| C(2)—C(1)—C(11) | 110.9 (3) | 111.7 (3) |
| N(2)—C(2)—C(1) | 107.6 (3) | 109.5 (3) |
| N(2)—C(2)—C(3) | 109.4 (3) | 107.8 (3) |
| C(1)—C(2)—C(3) | 113.4 (3) | 112.5 (3) |
| C(1)—C(11)—C(12) | 119.4 (3) | 120.3 (2) |
| C(1)—C(11)—C(16) | 120.6 (3) | 119.7 (2) |
| C(6)—O(1)—P(2)—O(3) | -55.7 (2) | 46.9 (3) |
| C(6)—O(1)—P(2)—O(21) | -169.1 (2) | 161.4 (3) |
| C(6)—O(1)—P(2)—O(22) | 59.9 (2) | -69.1 (3) |
| C(4)—O(3)—P(2)—O(1) | 56.9 (2) | -51.5 (2) |
| C(4)—O(3)—P(2)—O(21) | 170.2 (2) | -164.2 (2) |
| C(4)—O(3)—P(2)—O(22) | -60.3 (3) | 64.4 (3) |
| C(5)—C(4)—O(3)—P(2) | -61.6 (3) | 64.5 (3) |
| C(41)—C(4)—O(3)—P(2) | 170.26 (18) | -165.8 (2) |
| O(3)—C(4)—C(5)—C(6) | 57.6 (3) | -63.2 (4) |
| O(3)—C(4)—C(5)—C(51) | 173.3 (3) | 57.5 (4) |
| O(3)—C(4)—C(5)—C(52) | -64.0 (3) | -179.4 (3) |
| C(41)—C(4)—C(5)—C(6) | -177.6 (2) | 170.7 (3) |
| C(41)—C(4)—C(5)—C(51) | -61.9 (3) | -68.6 (4) |
| C(41)—C(4)—C(5)—C(52) | 60.8 (4) | 54.5 (4) |
| O(3)—C(4)—C(41)—C(42) | 38.8 (3) | -41.7 (4) |
| O(3)—C(4)—C(41)—C(46) | -144.1 (2) | 137.2 (3) |
| C(5)—C(4)—C(41)—C(42) | -86.3 (3) | 84.4 (4) |
| C(5)—C(4)—C(41)—C(46) | 90.9 (3) | -96.6 (3) |
| C(4)—C(5)—C(6)—O(1) | -58.4 (4) | 57.7 (4) |
| C(51)—C(5)—C(6)—O(1) | -177.7 (3) | -65.7 (4) |

Table 3 (cont.)

| | (1) | (2) |
|-------------------------|--------------|------------|
| C(52)—C(5)—C(6)—O(1) | 65.0 (4) | 175.8 (3) |
| C(5)—C(6)—O(1)—P(2) | 61.7 (3) | -54.8 (4) |
| C(4)—C(41)—C(42)—Cl(1) | -4.3 (3) | 2.5 (4) |
| C(4)—C(41)—C(42)—C(43) | 177.1 (2) | 178.8 (3) |
| C(46)—C(41)—C(42)—Cl(1) | 178.65 (17) | -176.4 (2) |
| C(4)—C(41)—C(46)—Cl(2) | 9.4 (3) | -2.9 (4) |
| C(4)—C(41)—C(46)—C(45) | -177.3 (2) | -178.9 (2) |
| C(42)—C(41)—C(46)—Cl(2) | -173.36 (18) | 176.0 (2) |
| Cl(1)—C(42)—C(43)—C(44) | -178.78 (17) | 176.8 (2) |
| C(44)—C(45)—C(46)—Cl(2) | 173.79 (17) | -176.4 (2) |
| C(44)—C(45)—C(46)—C(41) | 0.1 (3) | 0.0 (5) |
| O(10)—C(1)—C(2)—N(2) | -55.6 (4) | -60.7 (4) |
| O(10)—C(1)—C(2)—C(3) | 65.5 (4) | 59.1 (4) |
| C(11)—C(1)—C(2)—N(2) | 179.8 (4) | 175.8 (3) |
| C(11)—C(1)—C(2)—C(3) | -59.1 (5) | -64.3 (4) |
| O(10)—C(1)—C(11)—C(12) | 163.7 (3) | -5.3 (4) |
| O(10)—C(1)—C(11)—C(16) | -17.8 (4) | 174.0 (2) |
| C(2)—C(1)—C(11)—C(12) | -73.1 (4) | 115.5 (3) |
| C(2)—C(1)—C(11)—C(16) | 105.4 (4) | -65.3 (3) |
| C(1)—C(2)—N(2)—C(20) | -161.1 (3) | -64.9 (4) |
| C(3)—C(2)—N(2)—C(20) | 75.2 (4) | 172.5 (3) |
| C(1)—C(11)—C(12)—C(13) | 178.6 (3) | 179.3 (2) |
| C(1)—C(11)—C(16)—C(15) | -178.6 (3) | -179.3 (2) |

atoms refined isotropically; for (2), H atoms were assigned the isotropic thermal parameters of the atoms they are bonded to and kept fixed. Positional parameters and temperature factors are given in Table 2. Selected bond lengths, angles and torsion angles are given in Table 3. Scattering factors from *SHELX76* (Sheldrick, 1976). Geometry calculations and figures obtained using *EUCLID* (Spek, 1982). Calculations were performed on PDP11/23 and VAX-3800 computers.

Related literature. The present structure determinations were undertaken as part of an ongoing research project to gain insight into the processes involved in the resolution of enantiomers *via* diastereomeric salt formation. The synthesis of the chiral acidic resolving phosphorinane agent is described by van der Haest, Wynberg, Leusen & Bruggink (1990). Previous crystal structure determinations of related compounds include Kok, Wynberg, Smits, Beurskens & Parthasarathi (1987); Smits, Beurskens, Kok & Wynberg (1987); Smits, Beurskens, Parthasarathi, Rijk, Kok & Wynberg (1987); Kok, Wynberg, Parthasarathi, Smits & Beurskens (1987); Bruins Slot, Leusen, van der Haest & Smits (1992); Bruins Slot, Leusen, van der Haest & van Bolhuis (1992). Salt (1) may be called DICLINAM, and salt (2) in relation to these compounds DICLINAP.* The resolving power of the phosphorinane agents in these compounds is described by van der Haest *et al.* (1990). A detailed description of the crystal packing, to identify interactions which determine the differences in resolution efficiency of the resolving agent

* These names are composed of the general code INA, used for all compounds in the cited series, followed by *M* (minus) and *P* (plus) indicating the diastereomers of the pair.

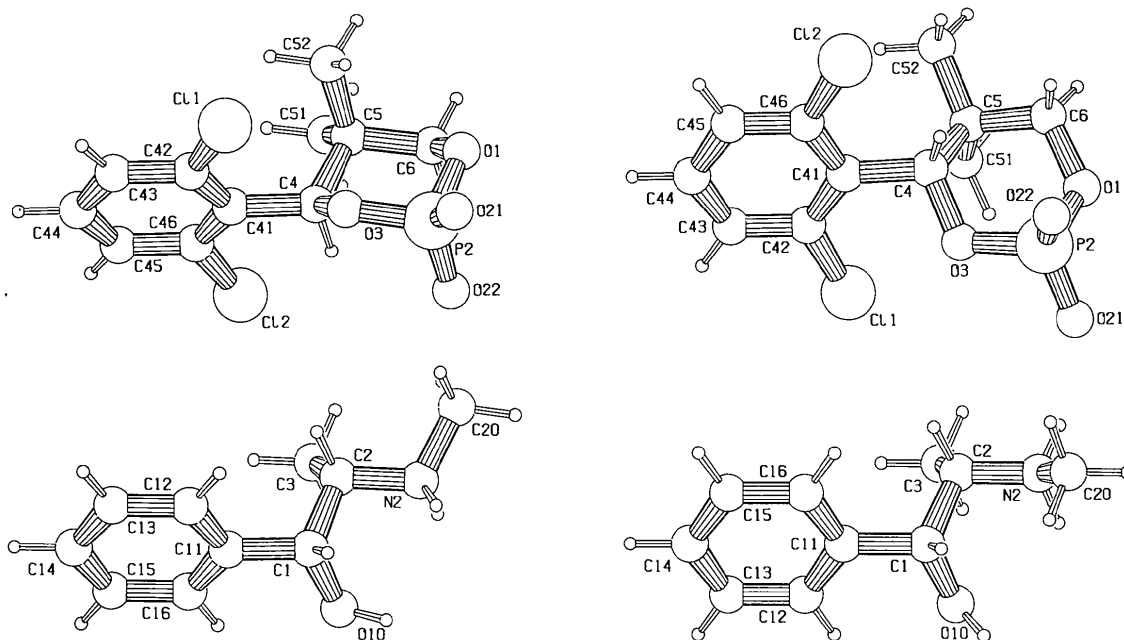


Fig. 1. Perspective drawings of the (+)-phosphorinane moiety of (1) (top left), the (-)-phosphorinane moiety of (2) (top right) and the (-)-ephedrine moieties [bottom left (1), right (2)] showing the atomic numbering of non-H atoms.

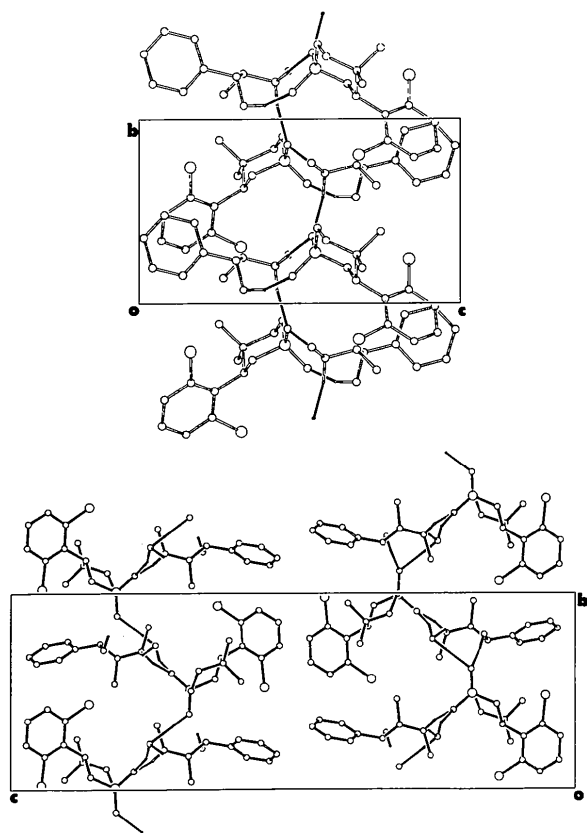


Fig. 2. Molecular packing, showing the hydrogen-bonding chains along the *b* axes [top (1), bottom (2)].

for a pair of diastereomeric salts, has been published (Leusen, Bruins Slot, Noordik, van der Haest, Wynberg & Bruggink, 1991). A second paper on this subject will be published by the same authors.

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Structure of 2,3,4,5-Tetra-*O*-acetyl-6-amino-6-deoxy-D-mannonolactam

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Abstract. C₁₄H₁₉NO₉, *M_r* = 345.31, orthorhombic, *P*2₁2₁2₁, *a* = 8.742 (1), *b* = 10.982 (1), *c* = 17.302 (2) Å, *V* = 1661.1 (2) Å³, *Z* = 4, *D_m* = 1.40, *D_x* = 1.38 Mg m⁻³, λ(Mo *K*α) = 0.71073 Å, μ = 0.11 mm⁻¹, *F*(000) = 728, *T* = 295 K, *R* = 0.051 for 1848 unique observed reflections. The seven-membered lactam ring adopts a chair conformation with C3, C6 and N displaced by 0.677 (6), -1.007 (7), -0.323 (7) Å, respectively, from the plane through C1, C2, C4 and C5. The molecules are linked together by N—H⋯O hydrogen bonds involving the amido N and the acetyl O9 atoms.

Experimental. 2,3,4,5-Tetra-*O*-acetyl-6-amino-6-deoxy-D-mannonolactam was prepared as described by Kefurt, Kefurtová & Jový (1989) and crystals obtained by recrystallization from a 1/1 mixture of chloroform/ethanol. The density was determined by flotation in iodomethane/toluene mixture at 298 K.

Data collection and structure refinement parameters are listed in Table 1.* Structure solved by direct

Table 1. *Data collection and structure refinement parameters*

| | |
|---|---|
| Crystal dimensions (mm) | 0.42 × 0.27 × 0.18 |
| Diffractometer | Enraf-Nonius CAD-4, graphite monochromator |
| Scan technique | ω/2θ |
| Number and θ range (°) for lattice parameters | 23, 13.07–19.23 |
| Range of <i>h</i> , <i>k</i> and <i>l</i> | 0 → 10, -13 → 13, -20 → 20 |
| Max. value of (sin θ)/λ (Å ⁻¹) | 0.595 |
| Standard reflections | 022, 130 |
| Monitored interval (min ⁻¹) | 120 |
| Intensity fluctuation (%) | -0.9 |
| Total reflections measured, 2θ range (°) | 6261, 2θ < 50 |
| <i>R</i> _{int} | 0.056 |
| Unique observed reflections | 1848 |
| Criterion for observed reflections | <i>I</i> > 1.96σ(<i>I</i>) |
| Function minimized | Σ(<i>F_o</i> - <i>F_c</i>) ² |
| Weighting scheme | Unit |
| Parameters refined | 195 |
| <i>R</i> | 0.051 |
| <i>S</i> | 1.12 |
| (Δ/ <i>σ</i>) _{max} | 0.004 |
| Max. and min. Δρ (e Å ⁻³) | 0.21, -0.26 |
| Source of atomic scattering factors | <i>SHELX76</i> (Sheldrick, 1976) |
| Programs used | <i>SHELXS86</i> (Sheldrick, 1986); <i>SHELX76</i> (Sheldrick, 1976); <i>PARST</i> (Nardelli, 1982); <i>SDP-Plus</i> (B. A. Frenz & Associates, Inc., 1985) |
| Computers used | PDP11/73, PC AT 286 |

* List of structure factors, anisotropic thermal parameters, H-atom positions and isotropic thermal parameters, and bond distances and angles involving H atoms, have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54718 (10 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: LI0112]

methods and anisotropically refined by block-diagonal least squares in three blocks. H atoms were localized from a difference Fourier synthesis and