$wR = 0.030$
$S = 0.231$
1924 reflections
161 parameters
Unit weights applied
$(\Delta/\sigma)_{\max} = 0.15$ [$B$ of H(2C1)]

Extinction correction: none
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

Table 3. *Fractional atomic coordinates and equivalent isotropic thermal parameters* (Å$^2$) *for glycylglycinium phosphite*

$$B_{eq} = (4/3)\Sigma_i\Sigma_j\beta_{ij}\mathbf{a}_i.\mathbf{a}_j.$$

| | $x$ | $y$ | $z$ | $B_{eq}$ |
|---|---|---|---|---|
| P | 0.81589 (3) | 0.0 | 0.4513 (4) | 2.151 (5) |
| O(1) | 0.6675 (1) | −0.0179 (4) | 0.4636 (1) | 2.97 (2) |
| O(2) | 0.8971 (1) | −0.0580 (3) | 0.5847 (1) | 2.90 (2) |
| O(3) | 0.8501 (1) | 0.2869 (3) | 0.3895 (2) | 4.14 (3) |
| N(1) | 0.5247 (1) | 0.7745 (4) | 0.6866 (2) | 2.65 (2) |
| C(1) | 0.4634 (2) | 0.9916 (5) | 0.7714 (2) | 3.03 (3) |
| C(2) | 0.3577 (2) | 0.8801 (4) | 0.8681 (2) | 2.37 (3) |
| O(4) | 0.3452 (1) | 0.6349 (3) | 0.8894 (2) | 3.21 (2) |
| N(2) | 0.2803 (2) | 1.0666 (3) | 0.9278 (2) | 2.98 (3) |
| C(3) | 0.1627 (2) | 0.9868 (6) | 1.0038 (2) | 3.57 (3) |
| C(4) | 0.1914 (2) | 0.8472 (4) | 1.1443 (2) | 2.58 (3) |
| O(5) | 0.2921 (1) | 0.8792 (3) | 1.2150 (2) | 3.19 (2) |
| O(6) | 0.0938 (1) | 0.6886 (5) | 1.1804 (2) | 4.96 (3) |
| H | 0.858 (2) | −0.145 (5) | 0.352 (2) | 1.2 (4)* |
| H(O3) | 0.927 (3) | 0.334 (6) | 0.397 (3) | 2.8 (6)* |

* $B_{iso}$.

Table 4. *Main interatomic distances* (Å) *and bond angles* (°) *in the atomic arrangement of glycylglycinium phosphite*

$C_4H_9N_2O_3$ group

| | | | |
|---|---|---|---|
| N(1)—C(1) | 1.470 (3) | N(1)—C(1)—C(2) | 111.1 (2) |
| C(1)—C(2) | 1.506 (2) | C(1)—C(2)—N(2) | 114.8 (2) |
| C(2)—O(4) | 1.230 (2) | C(1)—C(2)—O(4) | 121.9 (2) |
| C(2)—N(2) | 1.329 (2) | O(4)—C(2)—N(2) | 123.3 (2) |
| N(2)—C(3) | 1.438 (2) | C(2)—N(2)—C(3) | 120.3 (2) |
| C(3)—C(4) | 1.506 (3) | N(2)—C(3)—C(4) | 114.1 (1) |
| C(4)—O(5) | 1.206 (2) | C(3)—C(4)—O(5) | 124.3 (2) |
| C(4)—O(6) | 1.299 (2) | C(3)—C(4)—O(6) | 111.5 (1) |
| | | O(5)—C(4)—O(6) | 124.1 (2) |

$PO_3H$ tetrahedron

| P | O(1) | O(2) | O(3) | H |
|---|---|---|---|---|
| O(1) | 1.493 (1) | 2.553 (1) | 2.472 (2) | 2.27 (2) |
| O(2) | 116.73 (7) | 1.505 (1) | 2.531 (2) | 2.24 (2) |
| O(3) | 107.74 (9) | 111.01 (8) | 1.566 (2) | 2.15 (3) |
| H | 112 (1) | 109 (1) | 99 (1) | 1.25 (2) |

P—O(3)—H(O3) 116 (2)

Hydrogen bonds

| O(N)—H···O | O(N)—H | H···O | O(N)···O | O(N)—H···O |
|---|---|---|---|---|
| O(3)—H(O3)···O(2) | 0.81 (3) | 1.84 (3) | 2.649 (2) | 180 (3) |
| O(6)—H(O6)···O(2) | 0.80 (3) | 1.73 (3) | 2.508 (2) | 166 (3) |
| N(1)—H(1N1)···O(1) | 0.92 (2) | 1.83 (2) | 2.742 (2) | 172 (2) |
| N(1)—H(2N1)···O(5) | 0.86 (3) | 2.00 (3) | 2.818 (2) | 157 (2) |
| N(1)—H(3N1)···O(1) | 0.80 (2) | 1.97 (2) | 2.766 (2) | 171 (3) |
| N(2)—H(N2)···O(4) | 0.72 (3) | 2.18 (3) | 2.896 (2) | 176 (2) |

The two title compounds were prepared by the slow evaporation (over a few days) at room temperature of diluted aqueous solutions containing $H_2(PO_3H)$ and glycine or glycylglycine in stoichiometric ratios.

The H atoms were found by difference Fourier techniques and refined with $B_{iso}$. The structure was determined using *MULTAN77* (Main, Lessinger, Woolfson, Germain & Declercq, 1977) and refined with *SDP* (Enraf-Nonius, 1977). The figures were drawn using *STRUPLO* (Fischer, 1985).

Lists of structure factors, anisotropic thermal parameters, H-atom coordinates and main interatomic bond distances and angles involving H atoms have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55726 (40 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: PA1020]

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# X-ray Structure of a New Pyrethroid, RU 52259

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## Abstract

The X-ray structure of this intermediate compound, *tert*-butyl α-(*tert*-butylsulfonyl)-β-(methoxysulfinyl)-3,3-dimethyl-2-[(3-phenoxybenzyl)oxycarbonyl]-cyclopropanepropionate, shows the configuration of the asymmetric atoms at sites C1, C2 and S1 ($C^\alpha$,$C^\beta$ and β-S) and allows the mechanism of its formation to be established. The bond distances in the molecule are in the expected range.

## Comment

Biological activity in pyrethroids is related to molecular structure and strongly dependent on the stereochemistry at the asymmetric centre. For example, the $Z$ isomer of norpyrethrates is generally far more active than the $E$ isomer (Tessier, Teche & Demoute, 1982).

0108-2701/93/040818-03$06.00

We discuss here a compound with the general formula given in Fig. 1. To prepare *Z* derivatives a new methodology was developed (Babin, Demassey, Demoute, Dutheil, Terrie & Tessier, 1992) and RU52259 is an intermediate of a key reaction in this new approach to synthesis. The X-ray structure determination was necessary to define the three asymmetric centres (C1, C2, S1).

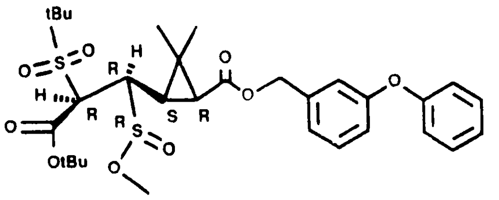

Fig. 1. General formula for pyrethroid compounds with only important H atoms represented for clarity.

Fractional coordinates and equivalent isotropic thermal parameters for non-H atoms are given in Table 1, selected interatomic distances and angles in Table 2. The conformation of the molecule and the atomic numbering are depicted in Fig. 2. The C—C distances in the phenoxybenzyl rings range between 1.369 (20) and 1.4201 (20) Å; there is no shortening of the bonds in the terminal ring (thermal libration) as observed in similar compounds (Baert, Guelzim & Germain, 1991).

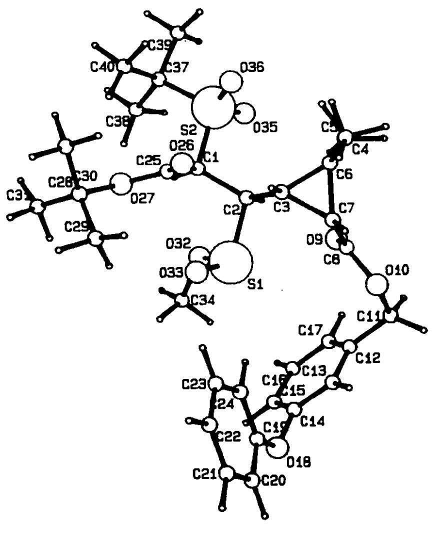

Fig. 2. Perspective view of the molecule and key to the numbering of the molecule.

The cyclopropane ring has a mean bond length of 1.542 (11) Å.

Knowledge of the stereochemistry of this compound has enabled chemists to describe precisely the reaction path of the reduction of sulfonylacrylates with sodium dithionite (Babin *et al.*, 1992).

## Experimental

### *Crystal data*

$C_{31}H_{42}O_9S_2$
$M_r = 622.7$
Orthorhombic
$P2_12_12_1$
$a = 12.010\ (3)$ Å
$b = 6.681\ (5)$ Å
$c = 42.090\ (8)$ Å
$V = 3377.4$ Å$^3$
$Z = 4$
$D_x = 1.2$ Mg m$^{-3}$

Cu $K\alpha$ radiation
$\lambda = 1.5419$ Å
Cell parameters from 25 reflections
$\theta = 30\text{-}50°$
$\mu = 1.79$ mm$^{-1}$
$T = 293\ (2)$ K
Plate
0.5 × 0.4 × 0.2 mm
Colourless

### *Data collection*

Nonius CAD-4 rotating anode diffractometer
$\omega/2\theta$ scans, width $(1.0 + 0.35\tan\theta)°$, prescan speed $16.48/8°\text{min}^{-1}$
Absorption correction: none
3203 measured reflections

2838 observed reflections $[I \geq 3\sigma(I)]$
$\theta_{\max} = 60°$
$h = 0 \rightarrow 13$
$k = 0 \rightarrow 7$
$l = 0 \rightarrow 47$
5 standard reflections
frequency: 120 min
intensity variation: none

### *Refinement*

Refinement on $F$
Final $R = 0.062$
$wR = 0.062$
$S = 7.4$
2838 reflections
407 parameters
H atoms: constrained refinement
$w = 1$

$(\Delta/\sigma)_{\max} = 0.012$
$\Delta\rho_{\max} = 0.21$ e Å$^{-3}$
$\Delta\rho_{\min} = -0.41$ e Å$^{-3}$
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV); for H from Stewart, Davidson & Simpson (1965)

Table 1. *Fractional atomic coordinates and equivalent isotropic thermal parameters* (Å$^2$)

$$U_{\text{eq}} = \tfrac{1}{3}\textstyle\sum_i\sum_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

| | $x$ | $y$ | $z$ | $U_{\text{eq}}$ |
|---|---|---|---|---|
| S1 | 0.7097 (2) | 0.3840 (3) | 0.1422 (1) | 0.044 (2) |
| S2 | 0.6884 (2) | 0.4458 (3) | 0.0386 (1) | 0.047 (2) |
| C1 | 0.6400 (6) | 0.4375 (14) | 0.0800 (2) | 0.042 (10) |
| C2 | 0.7475 (6) | 0.4588 (13) | 0.1012 (2) | 0.038 (9) |
| C3 | 0.7974 (6) | 0.6647 (12) | 0.1043 (2) | 0.039 (9) |
| C4 | 0.9039 (8) | 0.9500 (13) | 0.0787 (2) | 0.067 (12) |
| C5 | 0.9691 (7) | 0.5943 (14) | 0.0661 (2) | 0.056 (11) |
| C6 | 0.9024 (7) | 0.7327 (13) | 0.0876 (2) | 0.043 (9) |
| C7 | 0.9046 (6) | 0.6948 (13) | 0.1242 (2) | 0.038 (9) |
| C8 | 0.9649 (6) | 0.5208 (14) | 0.1375 (2) | 0.044 (10) |
| O9 | 0.9726 (5) | 0.3547 (9) | 0.1272 (1) | 0.050 (7) |
| O10 | 1.0183 (4) | 0.5836 (9) | 0.1646 (1) | 0.043 (6) |
| C11 | 1.0771 (6) | 0.4225 (15) | 0.1820 (2) | 0.052 (11) |
| C12 | 0.9960 (6) | 0.2908 (14) | 0.1995 (2) | 0.049 (10) |
| C13 | 0.9329 (6) | 0.3740 (13) | 0.2245 (2) | 0.044 (10) |
| C14 | 0.8570 (6) | 0.2512 (15) | 0.2402 (2) | 0.057 (11) |
| C15 | 0.8425 (7) | 0.0549 (14) | 0.2320 (2) | 0.057 (12) |
| C16 | 0.9071 (4) | −0.0240 (14) | 0.2077 (2) | 0.062 (13) |
| C17 | 0.9821 (7) | 0.0932 (14) | 0.1915 (2) | 0.052 (11) |
| O18 | 0.7985 (5) | 0.3298 (11) | 0.2662 (1) | 0.085 (9) |
| C20 | 0.6940 (9) | 0.5889 (20) | 0.2882 (2) | 0.086 (18) |
| C21 | 0.6159 (11) | 0.7462 (24) | 0.2840 (3) | 0.095 (23) |
| C22 | 0.5778 (10) | 0.7963 (20) | 0.2537 (4) | 0.122 (24) |
| C23 | 0.6115 (8) | 0.6951 (7) | 0.2271 (3) | 0.098 (16) |

| | | | | |
|---|---|---|---|---|
| C24 | 0.6877 (7) | 0.5434 (15) | 0.2308 (2) | 0.067 (12) |
| C19 | 0.7282 (7) | 0.4884 (15) | 0.2611 (2) | 0.058 (12) |
| C25 | 0.5488 (7) | 0.5927 (19) | 0.0871 (2) | 0.061 (13) |
| O26 | 0.5607 (5) | 0.7691 (12) | 0.0882 (2) | 0.069 (10) |
| O27 | 0.4542 (4) | 0.4880 (11) | 0.0915 (1) | 0.065 (9) |
| C28 | 0.3482 (7) | 0.5921 (26) | 0.1011 (3) | 0.108 (21) |
| C29 | 0.3654 (10) | 0.7025 (32) | 0.1330 (3) | 0.153 (33) |
| C30 | 0.3101 (9) | 0.7422 (26) | 0.0753 (3) | 0.140 (26) |
| C31 | 0.2700 (9) | 0.4105 (25) | 0.1033 (3) | 0.125 (26) |
| O32 | 0.6692 (5) | 0.1603 (8) | 0.1322 (1) | 0.058 (7) |
| O33 | 0.6112 (5) | 0.5038 (10) | 0.1512 (1) | 0.075 (8) |
| C34 | 0.6354 (9) | 0.0363 (15) | 0.1587 (2) | 0.070 (13) |
| O35 | 0.7816 (5) | 0.3106 (10) | 0.0372 (1) | 0.072 (8) |
| O36 | 0.7030 (6) | 0.6496 (9) | 0.0296 (1) | 0.069 (8) |
| C37 | 0.5788 (8) | 0.3359 (16) | 0.0142 (2) | 0.052 (13) |
| C38 | 0.5448 (13) | 0.1344 (19) | 0.0272 (3) | 0.081 (24) |
| C39 | 0.6359 (9) | 0.3161 (22) | −0.0190 (2) | 0.080 (19) |
| C40 | 0.4817 (9) | 0.4808 (23) | 0.0119 (2) | 0.093 (21) |

Table 2. *Geometric parameters* (Å, °)

| | | | |
|---|---|---|---|
| S1—C2 | 1.854 (7) | C14—C15 | 1.367 (13) |
| S1—O32 | 1.627 (6) | C14—O18 | 1.399 (10) |
| S1—O33 | 1.476 (7) | C15—C16 | 1.387 (12) |
| S2—C1 | 1.839 (7) | C16—C17 | 1.375 (13) |
| S2—O35 | 1.439 (7) | O18—C19 | 1.371 (11) |
| S2—O36 | 1.424 (7) | C28—C30 | 1.547 (20) |
| S2—C37 | 1.824 (10) | C28—C31 | 1.537 (21) |
| C1—C2 | 1.575 (10) | O32—C34 | 1.446 (10) |
| C1—C25 | 1.538 (13) | C37—C38 | 1.509 (17) |
| C2—C3 | 1.507 (11) | C37—C39 | 1.563 (12) |
| C3—C6 | 1.514 (10) | C37—C40 | 1.519 (16) |
| C3—C7 | 1.550 (10) | C20—C21 | 1.420 (19) |
| C4—C6 | 1.499 (12) | C21—C22 | 1.395 (22) |
| C6—C7 | 1.564 (10) | C22—C23 | 1.369 (20) |
| C7—C8 | 1.479 (12) | C23—C24 | 1.374 (14) |
| C8—O9 | 1.195 (11) | C24—C19 | 1.414 (12) |
| C8—O10 | 1.375 (8) | C25—O26 | 1.187 (15) |
| O10—C11 | 1.480 (10) | C25—O27 | 1.347 (11) |
| C11—C12 | 1.506 (11) | O27—C28 | 1.506 (13) |
| C12—C13 | 1.410 (10) | C28—C29 | 1.547 (19) |
| C12—C17 | 1.373 (13) | C20—C19 | 1.386 (14) |
| C13—C14 | 1.394 (11) | | |
| C2—S1—O32 | 94.6 (3) | C3—C7—C8 | 120.6 (7) |
| O32—S1—O33 | 108.9 (3) | C7—C8—O9 | 129.1 (8) |
| C1—S2—O36 | 108.7 (4) | O9—C8—O10 | 123.3 (7) |
| O35—S2—O36 | 119.6 (4) | O10—C11—C12 | 111.0 (6) |
| O36—S2—C37 | 108.8 (4) | C11—C12—C17 | 121.3 (7) |
| S2—C1—C25 | 112.9 (6) | C13—C14—C15 | 121.8 (8) |
| S1—C2—C1 | 107.6 (5) | C15—C14—O18 | 119.6 (7) |
| C1—C2—C3 | 117.2 (7) | C15—C16—C17 | 121.1 (8) |
| C2—C3—C7 | 119.7 (7) | C14—O18—C19 | 118.6 (7) |
| C3—C6—C4 | 114.6 (7) | C21—C20—C19 | 116.8 (11) |
| C3—C6—C7 | 60.5 (5) | C21—C22—C23 | 122.1 (13) |
| C4—C6—C7 | 113.7 (7) | C23—C24—C19 | 121.5 (9) |
| C3—C7—C6 | 58.2 (5) | O18—C19—C24 | 123.5 (8) |
| C6—C7—C8 | 120.5 (6) | C1—C25—O26 | 126.1 (9) |
| C7—C8—O10 | 107.6 (7) | O26—C25—O27 | 127.8 (9) |
| C8—O10—C11 | 114.2 (6) | O27—C28—C29 | 110.0 (10) |
| C11—C12—C13 | 118.8 (7) | O27—C28—C31 | 99.7 (9) |
| C13—C12—C17 | 119.9 (5) | C29—C28—C31 | 113.9 (11) |
| C12—C13—C14 | 118.3 (7) | S1—O32—C34 | 114.3 (5) |
| C13—C14—O18 | 118.5 (7) | S2—C37—C39 | 102.8 (7) |
| C14—C15—C16 | 118.7 (8) | C38—C37—C39 | 111.6 (10) |
| C12—C17—C16 | 120.3 (8) | C39—C37—C40 | 109.5 (8) |
| C2—S1—O33 | 106.7 (3) | C20—C21—C22 | 120.6 (13) |
| C1—S2—O35 | 105.4 (4) | C22—C23—C24 | 118.0 (10) |
| C1—S2—C37 | 107.1 (4) | O18—C19—C20 | 115.5 (8) |
| O35—S2—C37 | 106.6 (4) | C20—C19—C24 | 120.9 (9) |
| S2—C1—C2 | 105.9 (5) | C1—C25—O27 | 106.1 (7) |
| C2—C1—C25 | 114.4 (7) | C25—O27—C28 | 120.6 (8) |
| S1—C2—C3 | 105.3 (5) | O27—C28—C30 | 111.1 (10) |
| C2—C3—C6 | 124.5 (7) | C29—C28—C30 | 109.9 (11) |
| C6—C3—C7 | 61.4 (5) | C30—C28—C31 | 111.9 (11) |
| C3—C6—C5 | 122.2 (7) | S2—C37—C38 | 110.5 (7) |
| C4—C6—C5 | 115.7 (8) | S2—C37—C40 | 109.5 (7) |
| C5—C6—C7 | 118.7 (7) | C38—C37—C40 | 112.6 (9) |

The structure was solved using direct methods (*SHELXS*86; Sheldrick, 1986); 50 phase sets with $E \geq 1.20$. The best solution had a combined figure of merit of 0.057. Non-H atoms were refined anisotropically (*SHELX*76; Sheldrick, 1976). H atoms, found from $\Delta\rho$ synthesis and theoretically adjusted, were refined isotropically.

We thank D. Babin and J. P. Demoute from Roussel Uclaf Company (Romainville, France) for suggesting this work and providing the samples.

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Lists of structure factors, anisotropic thermal parameters, H-atom coordinates and complete geometry have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55761 (22 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: PA1024]

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# Structures of Tribenzylmethanol and 1,2,3-Triphenyl-2-propanol

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## Abstract

The tribenzylmethanol molecule, $(PhCH_2)_3COH$, has approximate threefold symmetry in the solid state. The hydroxyl H atom is disordered unequally over three orientations and is not involved in hydrogen bonding. The 1,2,3-

0108-2701/93/040820-05$06.00