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Tricoccin R2. Erratum

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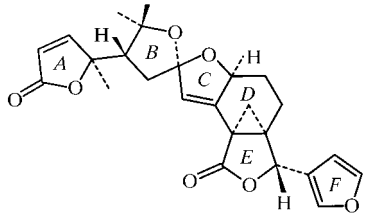
The crystal structure of the title compound, $C_{25}H_{26}O_7$, was published with erroneous positions for a C atom and the O atom in ring *F* [Sekar *et al.* (1996). *Acta Cryst.* (1996), C**52**, 92–94]. This has now been corrected and leads to a more sensible bond length and angle geometry.

Comment

During a comparative study of the molecular structure of Tricoccin R6 (Abdul Ajees *et al.*, 2001) with that of the related compound Tricoccin R2 (Sekar *et al.*, 1996) it was found that



Tricoccin R2

the geometry of the molecules in the two structures agreed well except in the region of ring *F* of Tricoccin R2. This could be traced to a wrong assignment of two of the atoms in ring *F* of Tricoccin R2. That is, the neighbours of atoms C21 and C22 in ring *F* of Tricoccin R2 are to be taken as O and C atoms, respectively, instead of C and O as in the original report. The structure of Tricoccin R2 thus modified was refined and converged to a lower *R* value and the final difference Fourier was better. There is now better agreement of the geometry of ring *F* of Tricoccin R2 with that of Tricoccin R6.

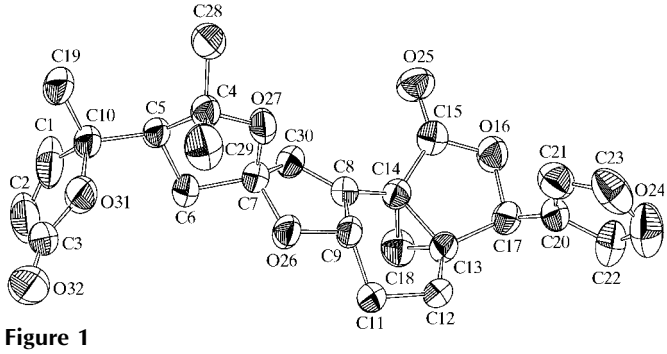


Figure 1
The molecular structure of the title compound with 30% probability displacement ellipsoids

Experimental

Crystal data

$C_{25}H_{26}O_7$
M_r = 438.46
Monoclinic, *C*2
a = 22.939 (1) Å
b = 6.574 (2) Å
c = 16.481 (2) Å
β = 114.67 (1)°
V = 2258.5 (7) Å^3
Z = 4
D_x = 1.289 Mg m^{-3}
Cu $K\alpha$ radiation
Cell parameters from 25 reflections
θ = 20–30°
μ = 0.78 mm^{-1}
T = 293 (2) K
Needle, colourless
0.30 × 0.25 × 0.20 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: empirical ψ scan (North *et al.*, 1968)
T_{min} = 0.961, T_{max} = 0.991
2312 measured reflections
2230 independent reflections
2054 reflections with $I > 2\sigma(I)$
R_{int} = 0.027
θ_{max} = 70.3°
h = −25 → 27
k = 0 → 8
l = −19 → 0
2 standard reflections
frequency: 120 min
intensity decay: <1%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.036
$wR(F^2)$ = 0.103
S = 1.07
2230 reflections
293 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0588P)^2 + 0.6138P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.27 e Å^{-3}
$\Delta\rho_{min}$ = −0.12 e Å^{-3}
Extinction correction: *SHELXL*97
Extinction coefficient: 0.0018 (2)
Absolute structure: Flack (1983)
Flack parameter = 0.1 (3)

Data collection: *CAD*-4 *Software* (Enraf–Nonius, 1989); cell refinement: *SDP* (Frenz, 1978); data reduction: *CAD*-4 *Software*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997).

Supplementary data for this paper are available from the IUCr electronic archives (Reference: VJ1131). Services for accessing these data are described at the back of the journal.

References

Abdul Ajees, A., Sekar, K., Parthasarathy, S., Schenk, H., Epe, B. & Mondon, A. (2001). *Acta Cryst.* E**57** o116–117.
Enraf–Nonius (1989). *CAD*-4 *Software*. Version 5.0. Enraf–Nonius, Delft, The Netherlands.
Flack, H. D. (1983). *Acta Cryst.* A**39**, 876–881.
Frenz, B. A. (1978). *The Enraf–Nonius CAD*-4 *SDP*. *Computing in Crystallography*, edited by H. Schenk, R. Olthof-Hazekamp, H. van Koningsveld & G. C. Bassi, pp. 64–71. Delft University Press.
North, A. C. T., Phillips, D. C. & Mathews, F. S. (1968). *Acta Cryst.* A**24**, 351–359.
Sekar, K., Parthasarathy, S., Schenk, H., Epe, B. & Mondon, A. (1996). *Acta Cryst.* C**52**, 92–94.
Sheldrick, G. M. (1997). *SHELXS*97 and *SHELXL*97. Release 97–2. University of Göttingen, Germany.

supporting information

Acta Cryst. (2001). C**57**, 499 [https://doi.org/10.1107/S010827010002045X]

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Computing details

Data collection: *CAD-4 Software* (Enraf-Nonius, 1989); cell refinement: *SDP* (Frenz, 1978); data reduction: *CAD-4 Software*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997).

(I)

Crystal data

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Monoclinic, $C2$
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$c = 16.481\ (2)$ Å
$\beta = 114.67\ (1)°$
$V = 2258.5\ (7)$ Å^3
$Z = 4$
$F(000) = 928$
$D_x = 1.289$ Mg m^{-3}
Cu $K\alpha$ radiation, $\lambda = 1.54180$ Å
Cell parameters from 25 reflections
$\theta = 20–30°$
$\mu = 0.78$ mm^{-1}
$T = 293$ K
Needle, colourless
0.30 × 0.25 × 0.20 mm

Data collection

Enraf-Nonius CAD-4 diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
$\omega/2\theta$ scans
Absorption correction: empirical (using intensity measurements) ψ scan
$T_{min} = 0.961$, $T_{max} = 0.991$
2312 measured reflections
2230 independent reflections
2054 reflections with $I > 2\sigma(I)$
$R_{int} = 0.027$
$\theta_{max} = 70.3°$, $\theta_{min} = 3.0°$
$h = -25\rightarrow27$
$k = 0\rightarrow8$
$l = -19\rightarrow0$
2 standard reflections every 120 min
intensity decay: <1%

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.036$
$wR(F^2) = 0.103$
$S = 1.07$
2230 reflections
293 parameters
1 restraint
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0588P)^2 + 0.6138P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.27$ e Å^{-3}
$\Delta\rho_{min} = -0.12$ e Å^{-3}
Extinction correction: SHELXL97, $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.0018 (2)
Absolute structure: Flack (1983)
Absolute structure parameter: 0.1 (3)

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted *R*-factor *wR* and goodness of fit *S* are based on F^2, conventional *R*-factors *R* are based on *F*, with *F* set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on *F*, and *R*- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	0.5001 (2)	0.3359 (8)	0.1535 (2)	0.0884 (13)
H1	0.4821	0.4590	0.1589	0.106*
C2	0.5380 (3)	0.3143 (15)	0.1124 (3)	0.138 (3)
H2	0.5515	0.4171	0.0854	0.165*
C3	0.55499 (19)	0.0986 (16)	0.1168 (2)	0.136 (3)
C4	0.51095 (12)	−0.0319 (5)	0.35079 (16)	0.0495 (6)
C5	0.51555 (12)	0.1521 (4)	0.29493 (16)	0.0451 (6)
H5	0.4918	0.2628	0.3072	0.054*
C6	0.58606 (12)	0.2103 (6)	0.34162 (16)	0.0543 (7)
H6A	0.5919	0.3549	0.3360	0.065*
H6B	0.6114	0.1356	0.3170	0.065*
C7	0.60510 (12)	0.1507 (5)	0.44081 (16)	0.0506 (7)
C8	0.66748 (11)	0.2699 (4)	0.58414 (15)	0.0432 (6)
C9	0.69709 (11)	0.0774 (4)	0.56993 (15)	0.0453 (6)
H9	0.6873	−0.0349	0.6013	0.054*
C10	0.48957 (12)	0.1400 (5)	0.19060 (16)	0.0529 (7)
C11	0.76878 (11)	0.0952 (5)	0.60159 (16)	0.0517 (7)
H11A	0.7789	0.2061	0.5709	0.062*
H11B	0.7859	−0.0296	0.5888	0.062*
C12	0.79923 (11)	0.1359 (5)	0.70458 (16)	0.0516 (7)
H12A	0.7954	0.0136	0.7349	0.062*
H12B	0.8446	0.1639	0.7240	0.062*
C13	0.76875 (11)	0.3117 (5)	0.73265 (16)	0.0465 (6)
C14	0.70042 (11)	0.3743 (4)	0.67166 (16)	0.0431 (6)
C15	0.66908 (13)	0.4280 (4)	0.73307 (17)	0.0493 (6)
O16	0.71301 (9)	0.4229 (4)	0.82068 (12)	0.0587 (5)
C17	0.77399 (12)	0.3345 (5)	0.82997 (16)	0.0507 (6)
H17	0.8085	0.4311	0.8623	0.061*
C18	0.75748 (14)	0.5110 (5)	0.6846 (2)	0.0596 (7)
H18A	0.7700	0.5225	0.6354	0.072*
H18B	0.7630	0.6341	0.7195	0.072*
C19	0.42104 (14)	0.0723 (7)	0.14383 (18)	0.0683 (9)
H19A	0.4078	0.0792	0.0804	0.102*
H19B	0.3943	0.1595	0.1605	0.102*
H19C	0.4172	−0.0652	0.1606	0.102*

C20	0.78499 (13)	0.1459 (5)	0.88397 (16)	0.0528 (7)
C21	0.74541 (17)	−0.0300 (6)	0.8694 (2)	0.0687 (8)
H21	0.7064	−0.0541	0.8211	0.082*
C22	0.83659 (18)	0.1101 (6)	0.9619 (2)	0.0756 (10)
H22	0.8707	0.2000	0.9880	0.091*
O24	0.83239 (17)	−0.0739 (5)	0.99729 (18)	0.0999 (10)
C23	0.7759 (2)	−0.1549 (7)	0.9400 (3)	0.0874 (12)
H23	0.7601	−0.2800	0.9479	0.105*
O25	0.61416 (9)	0.4682 (4)	0.71455 (14)	0.0655 (6)
O26	0.66502 (8)	0.0429 (4)	0.47372 (11)	0.0558 (5)
O27	0.55533 (8)	0.0248 (4)	0.44137 (11)	0.0595 (6)
C28	0.44694 (13)	−0.0560 (6)	0.3563 (2)	0.0662 (8)
H28A	0.4516	−0.1489	0.4036	0.099*
H28B	0.4158	−0.1082	0.3007	0.099*
H28C	0.4330	0.0738	0.3682	0.099*
C29	0.5324 (2)	−0.2330 (6)	0.3265 (3)	0.0832 (11)
H29A	0.5747	−0.2179	0.3286	0.125*
H29B	0.5031	−0.2720	0.2673	0.125*
H29C	0.5330	−0.3359	0.3681	0.125*
C30	0.61627 (12)	0.3144 (5)	0.50991 (16)	0.0512 (7)
H30	0.5909	0.4297	0.5017	0.061*
O31	0.52773 (10)	−0.0018 (5)	0.16438 (13)	0.0861 (9)
O32	0.58593 (15)	0.0036 (14)	0.08401 (18)	0.219 (4)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.098 (3)	0.100 (3)	0.0397 (16)	−0.021 (2)	0.0024 (17)	0.0130 (19)
C2	0.090 (3)	0.257 (9)	0.047 (2)	−0.057 (5)	0.009 (2)	0.037 (4)
C3	0.056 (2)	0.315 (11)	0.0362 (17)	0.033 (4)	0.0190 (14)	0.017 (4)
C4	0.0511 (13)	0.0536 (16)	0.0412 (12)	0.0019 (13)	0.0167 (11)	−0.0039 (12)
C5	0.0413 (12)	0.0560 (16)	0.0351 (12)	0.0055 (12)	0.0130 (10)	−0.0039 (11)
C6	0.0451 (13)	0.076 (2)	0.0357 (12)	−0.0020 (14)	0.0111 (10)	0.0050 (13)
C7	0.0429 (12)	0.0677 (19)	0.0373 (12)	0.0046 (13)	0.0130 (10)	0.0030 (13)
C8	0.0417 (11)	0.0492 (15)	0.0380 (12)	0.0029 (11)	0.0160 (10)	0.0047 (11)
C9	0.0441 (12)	0.0532 (16)	0.0335 (11)	0.0063 (12)	0.0110 (10)	0.0024 (11)
C10	0.0482 (13)	0.071 (2)	0.0347 (12)	0.0096 (14)	0.0128 (10)	−0.0019 (13)
C11	0.0433 (12)	0.0691 (19)	0.0422 (12)	0.0093 (13)	0.0174 (10)	0.0024 (13)
C12	0.0365 (11)	0.0706 (19)	0.0441 (12)	0.0059 (13)	0.0132 (10)	0.0039 (14)
C13	0.0392 (11)	0.0554 (16)	0.0398 (12)	−0.0049 (11)	0.0114 (10)	0.0011 (12)
C14	0.0412 (11)	0.0427 (14)	0.0424 (12)	−0.0001 (10)	0.0144 (10)	0.0031 (11)
C15	0.0547 (14)	0.0418 (14)	0.0486 (13)	0.0029 (12)	0.0186 (11)	−0.0009 (12)
O16	0.0614 (11)	0.0674 (14)	0.0441 (9)	0.0082 (10)	0.0188 (8)	−0.0053 (10)
C17	0.0443 (12)	0.0588 (17)	0.0410 (13)	−0.0051 (12)	0.0098 (10)	−0.0067 (12)
C18	0.0637 (16)	0.0551 (18)	0.0582 (16)	−0.0135 (15)	0.0235 (13)	0.0033 (14)
C19	0.0568 (15)	0.095 (3)	0.0409 (13)	0.0041 (17)	0.0088 (11)	−0.0081 (16)
C20	0.0542 (14)	0.0654 (18)	0.0383 (12)	0.0036 (14)	0.0188 (11)	−0.0014 (13)
C21	0.0706 (18)	0.073 (2)	0.0688 (19)	−0.0051 (18)	0.0352 (16)	0.0017 (18)

C22	0.082 (2)	0.079 (3)	0.0464 (16)	0.007 (2)	0.0073 (15)	0.0028 (17)
O24	0.135 (2)	0.091 (2)	0.0618 (14)	0.025 (2)	0.0296 (15)	0.0204 (15)
C23	0.131 (4)	0.067 (2)	0.091 (3)	0.007 (2)	0.072 (3)	0.014 (2)
O25	0.0542 (11)	0.0703 (14)	0.0711 (13)	0.0134 (11)	0.0251 (9)	−0.0040 (12)
O26	0.0495 (9)	0.0735 (14)	0.0363 (8)	0.0124 (10)	0.0099 (7)	−0.0062 (9)
O27	0.0553 (10)	0.0818 (15)	0.0365 (8)	−0.0099 (11)	0.0144 (8)	0.0048 (10)
C28	0.0594 (16)	0.082 (2)	0.0561 (16)	−0.0122 (17)	0.0236 (13)	−0.0050 (18)
C29	0.111 (3)	0.062 (2)	0.078 (2)	0.028 (2)	0.041 (2)	0.0074 (19)
C30	0.0467 (13)	0.0605 (18)	0.0420 (13)	0.0115 (13)	0.0142 (10)	0.0068 (13)
O31	0.0720 (13)	0.142 (3)	0.0431 (10)	0.0408 (16)	0.0228 (10)	−0.0083 (14)
O32	0.100 (2)	0.500 (12)	0.0690 (16)	0.110 (5)	0.0482 (16)	0.015 (4)

Geometric parameters (Å, º)

C1—C2	1.312 (7)	C9—C11	1.508 (3)
C1—C10	1.488 (5)	C10—O31	1.462 (4)
C2—C3	1.464 (11)	C10—C19	1.501 (4)
C3—O32	1.228 (7)	C11—C12	1.566 (3)
C3—O31	1.360 (7)	C12—C13	1.520 (4)
C4—O27	1.461 (3)	C13—C18	1.497 (4)
C4—C28	1.517 (4)	C13—C14	1.523 (3)
C4—C29	1.522 (5)	C13—C17	1.565 (3)
C4—C5	1.550 (4)	C14—C15	1.508 (4)
C5—C6	1.522 (4)	C14—C18	1.527 (4)
C5—C10	1.569 (3)	C15—O25	1.195 (3)
C6—C7	1.556 (3)	C15—O16	1.374 (3)
C7—O27	1.413 (3)	O16—C17	1.463 (3)
C7—O26	1.437 (3)	C17—C20	1.485 (4)
C7—C30	1.509 (4)	C20—C22	1.355 (4)
C8—C30	1.328 (3)	C20—C21	1.427 (5)
C8—C14	1.487 (3)	C21—C23	1.355 (5)
C8—C9	1.500 (4)	C22—O24	1.364 (5)
C9—O26	1.461 (3)	O24—C23	1.354 (6)
C2—C1—C10	112.0 (5)	C9—C11—C12	108.48 (19)
C1—C2—C3	107.4 (5)	C13—C12—C11	114.0 (2)
O32—C3—O31	119.7 (9)	C18—C13—C12	120.3 (2)
O32—C3—C2	131.4 (8)	C18—C13—C14	60.76 (18)
O31—C3—C2	108.8 (4)	C12—C13—C14	118.8 (2)
O27—C4—C28	104.1 (2)	C18—C13—C17	112.4 (2)
O27—C4—C29	109.1 (3)	C12—C13—C17	122.5 (2)
C28—C4—C29	110.7 (3)	C14—C13—C17	105.6 (2)
O27—C4—C5	102.5 (2)	C8—C14—C15	124.6 (2)
C28—C4—C5	115.1 (2)	C8—C14—C13	118.8 (2)
C29—C4—C5	114.3 (2)	C15—C14—C13	105.5 (2)
C6—C5—C4	102.5 (2)	C8—C14—C18	117.7 (2)
C6—C5—C10	113.8 (2)	C15—C14—C18	112.9 (2)
C4—C5—C10	121.5 (2)	C13—C14—C18	58.75 (18)

C5—C6—C7	103.9 (2)	O25—C15—O16	120.4 (2)
O27—C7—O26	110.9 (3)	O25—C15—C14	128.9 (2)
O27—C7—C30	107.2 (2)	O16—C15—C14	110.7 (2)
O26—C7—C30	103.5 (2)	C15—O16—C17	111.70 (19)
O27—C7—C6	106.3 (2)	O16—C17—C20	108.1 (2)
O26—C7—C6	109.0 (2)	O16—C17—C13	105.65 (19)
C30—C7—C6	119.9 (3)	C20—C17—C13	117.1 (2)
C30—C8—C14	133.5 (3)	C13—C18—C14	60.49 (17)
C30—C8—C9	109.0 (2)	C22—C20—C21	105.1 (3)
C14—C8—C9	117.4 (2)	C22—C20—C17	125.1 (3)
O26—C9—C8	104.17 (19)	C21—C20—C17	129.7 (3)
O26—C9—C11	111.71 (19)	C23—C21—C20	106.4 (3)
C8—C9—C11	112.3 (2)	C20—C22—O24	111.7 (4)
O31—C10—C1	102.1 (3)	C23—O24—C22	105.6 (3)
O31—C10—C19	106.7 (3)	O24—C23—C21	111.2 (4)
C1—C10—C19	111.2 (3)	C7—O26—C9	107.59 (18)
O31—C10—C5	110.9 (2)	C7—O27—C4	111.29 (18)
C1—C10—C5	110.7 (3)	C8—C30—C7	110.1 (3)
C19—C10—C5	114.5 (2)	C3—O31—C10	109.7 (5)