2C (2)	0.0953 (1)	0.4707 (2)	1.0653 (1)	0.0480 (11)
2C (3)	0.0930 (1)	0.5837 (2)	1.0842 (1)	0.0503 (12)
2C (4)	0.0744 (1)	0.6704 (2)	1.0312 (1)	0.0671 (11)
2C (5)	0.0593 (1)	0.6432 (2)	0.9602 (1)	0.0535 (13)
2C (6)	0.0623 (1)	0.5304 (2)	0.9416 (1)	0.0473 (12)
3C (1)	0.1595 (1)	0.0385 (2)	0.9848 (1)	0.0417 (11)
3C (2)	0.2065 (1)	0.0994 (2)	0.9807 (1)	0.0549 (13)
3C (3)	0.2662 (1)	0.0488 (2)	1.0078 (1)	0.0674 (16)
3C (4)	0.2780 (1)	−0.0612 (3)	1.0365 (1)	0.0638 (16)
3C (5)	0.2313 (1)	−0.1216 (2)	1.0393 (1)	0.0654 (13)
3C (6)	0.1708 (1)	−0.0713 (2)	1.0137 (1)	0.0546 (13)

Table 2. *Selected geometric parameters* (Å, °)

N(1)—C(2)	1.373 (4)	C(2′)—2C(1)	1.465 (3)
N(1)—C(8)	1.369 (3)	1C(1)—1C(2)	1.391 (4)
C(2)—C(3)	1.349 (2)	1C(1)—1C(6)	1.389 (5)
C(2)—C(1′)	1.463 (3)	1C(2)—1C(3)	1.360 (6)
C(3)—C(9)	1.450 (4)	1C(3)—1C(4)	1.363 (10)
C(3)—S(10)	1.740 (5)	1C(4)—1C(5)	1.387 (7)
C(4)—C(5)	1.373 (5)	1C(5)—1C(6)	1.401 (5)
C(4)—C(9)	1.381 (2)	2C(1)—2C(2)	1.386 (3)
C(5)—C(6)	1.393 (6)	2C(1)—2C(6)	1.392 (3)
C(6)—C(7)	1.353 (2)	2C(2)—2C(3)	1.379 (3)
C(7)—C(8)	1.400 (4)	2C(3)—2C(4)	1.382 (3)
C(8)—C(9)	1.391 (3)	2C(4)—2C(5)	1.369 (3)
S(10)—1C(1)	1.763 (4)	2C(5)—2C(6)	1.378 (3)
S(11)—O(12)	1.427 (3)	3C(1)—3C(2)	1.390 (4)
S(11)—O(13)	1.443 (3)	3C(1)—3C(6)	1.372 (3)
S(11)—C(1′)	1.775 (3)	3C(2)—3C(3)	1.371 (3)
S(11)—3C(1)	1.756 (3)	3C(3)—3C(4)	1.372 (4)
O(14)—C(15)	1.423 (3)	3C(4)—3C(5)	1.366 (4)
O(14)—2C(4)	1.360 (3)	3C(5)—3C(6)	1.392 (3)
C(1′)—C(2′)	1.337 (3)		
C(2)—N(1)—C(8)	110.2 (2)	1C(3)—1C(4)—1C(5)	120.9 (5)
N(1)—C(2)—C(3)	109.2 (2)	1C(4)—1C(5)—1C(6)	118.2 (4)
C(2)—C(3)—C(9)	106.4 (2)	1C(5)—1C(6)—1C(1)	120.0 (3)
C(5)—C(4)—C(9)	118.3 (2)	2C(1)—2C(2)—2C(3)	121.3 (2)
C(4)—C(5)—C(6)	122.3 (4)	2C(2)—2C(3)—2C(4)	120.3 (2)
C(5)—C(6)—C(7)	120.7 (2)	2C(3)—2C(4)—2C(5)	119.3 (2)
C(6)—C(7)—C(8)	117.0 (2)	2C(4)—2C(5)—2C(6)	120.4 (2)
N(1)—C(8)—C(7)	130.3 (2)	2C(5)—2C(6)—2C(1)	121.4 (2)
C(7)—C(8)—C(9)	123.0 (2)	2C(6)—2C(1)—2C(2)	117.4 (2)
N(1)—C(8)—C(9)	106.7 (2)	3C(2)—3C(3)—3C(4)	119.9 (3)
C(4)—C(9)—C(8)	118.7 (2)	3C(3)—3C(4)—3C(5)	121.1 (3)
C(3)—C(9)—C(8)	107.5 (2)	3C(4)—3C(5)—3C(6)	120.1 (3)
C(3)—C(9)—C(4)	133.8 (2)	3C(1)—3C(6)—3C(5)	118.2 (2)
1C(2)—1C(1)—1C(6)	120.2 (3)	3C(6)—3C(1)—3C(2)	121.8 (2)
1C(1)—1C(2)—1C(3)	119.0 (4)	3C(1)—3C(2)—3C(3)	118.8 (3)
1C(2)—1C(3)—1C(4)	121.6 (5)		
N(1)—H(1)···O(13)	3.023 (3)	3C(6)—H3(6)···O(14^{i})	3.337 (4)
C(2′)—H(2′)···O(12)	2.827 (3)		

Symmetry code: (i) $x, y-1, z$.

Cell refinement and data reduction: *SDP* (Frenz, 1978). Program used to solve structure: *SHELXS86* (Sheldrick, 1985). Program used to refine structure: *SHELX76* (Sheldrick, 1976). Software used to prepare material for publication: *PARST* (Nardell, 1983) and *PLUTO* (Motherwell, 1976).

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates, complete geometry and least-squares-planes data have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71182 (22 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: HA1018]

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2-Benzoyl-3-(4-methoxyphenyl)-4-methyl-1-phenyl-2,3-dihydro-1*H*-pyrazolo[4,3-*c*]-[1,2]benzothiazine 5,5-Dioxide

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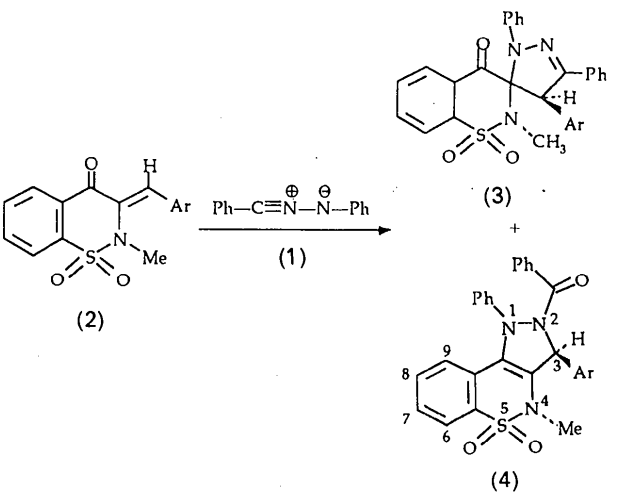
Abstract

Two molecules of the title compound, $C_{30}H_{25}N_3O_4S$, crystallize in the asymmetric unit. The phenyl, benzoyl and methoxyphenyl substituents point alternately up and down with respect to the dihydropyrazolo cycle (in agreement with steric effects). The conformation of the molecule is such that the thiazine cycle has an approximate plane of symmetry passing through the S atom (and the two O atoms), whereas the dihydropyrazolo cycle has a twofold axis passing between the two N atoms.

Comment

The reaction of diphenylnitrilimine (1) with 3-arylidene-2*H*-3,4-dihydro-1,2-benzothiazin-4-one

1,1-dioxides (2) leads to a mixture of two products, (3) and (4) (see scheme below), which were isolated by chromatography. Product (3) is a classical cycloadduct as identified by spectroscopic and chemical analysis. These methods, however, did not enable us to determine the structure of compound (4) (the title compound). Work is in progress to explain the chemical pathway which leads to compound (4).



The asymmetric unit in the triclinic cell contains two molecules which have similar bond distances and bond angles. Most of the stereochemical features of the two molecules are similar. Two cycles, $\Pi(3)$ and $\Pi(4)$, are defined as follows: $\Pi(3)$, N(5)—N(6)—C(7)—C(3)—C(4); $\Pi(4)$, N(2)—C(3)—C(7)—C(8)—C(13)—S(1).

Cycle $\Pi(3)$, which is approximately flat, has substituents C(14), C(28) and C(20), alternately, on opposite sides of the ring plane. This disposition minimizes steric effects.

The conformation of cycle $\Pi(3)$ may be described by examining the torsion angles Φ_i around the cycle, according to Duax & Norton (1975). This cycle exhibits an approximate twofold axis passing between the atoms N(5) and N(6) and through C(3); the parameter

$$\Delta C_2 = [(1/m)\sum_{i=1}^{i=m}(\Phi_i - \Phi_i')^2]^{1/2}$$

defined by these authors gives 1.4 and 0.4° for molecules *A* and *B*, respectively, which is fairly good.

In cycle $\Pi(4)$ the S(1) atom is the furthest out of the average plane, as might be expected. The plane passing through C(7) and S(1) and perpendicular to the cycle is approximately a mirror plane; the parameter

$$\Delta C_s = [(1/m)\sum_{i=1}^{i=m}(\Phi_i + \Phi_i')^2]^{1/2}$$

is 6.9 and 8.7° for molecules *A* and *B*, respectively. This symmetry plane passes approximately through the two O atoms.

The configuration of N(2) is approximately flat, as may be seen from the sum of the three bond angles around it [357.6 (7) and 359.4 (8)° for molecules *A* and *B*, respectively].

The carbonyl group, C(28)—O(29), conjugates with C(28)—N(5), as shown by the interatomic distances N(105)—C(128) 1.367 (4) and C(128)—O(129) 1.217 (4) Å, and N(205)—C(228) 1.359 (3) and C(228)—O(229) 1.232 (4) Å. [For molecule *A*, atom labels are prefixed by 1 or 10, while for molecule *B*, atom labels are prefixed by 2 or 20 (*cf.* Fig. 2 and Table 1).]

The planarity of the other (aromatic) cycles is satisfactory. For plane $\Pi(6)$ [C(20) to C(25)] the methoxy substituent is not far from the mean plane: C(227) is 0.066 (4) Å from plane $\Pi(26)$ and C(127) is 0.013 (5) Å from plane $\Pi(16)$. But the interatomic distances C(123)—O(126) 1.382 (5) and O(126)—C(127) 1.394 (5) Å, and C(223)—O(226) 1.368 (4) and O(226)—C(227) 1.432 (6) Å, show that the conjugation is less obvious.

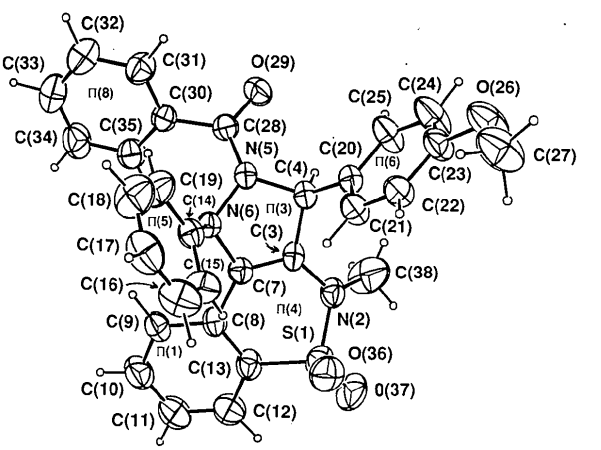


Fig. 1. *ORTEP* (Johnson, 1976) view of the title molecule. For molecule *A*, atom labels should be prefixed by 1 or 10; for molecule *B*, atom labels should be prefixed by 2 or 20 (*cf.* Table 1).

Experimental

Crystal data

$C_{30}H_{25}N_3O_4S$
M_r = 523.6
Triclinic
$P\bar{1}$
a = 11.947 (2) Å
b = 11.333 (2) Å
c = 21.114 (6) Å
α = 109.38 (2)°
β = 94.85 (2)°
γ = 90.33 (1)°
V = 2685 (2) Å^3
Z = 4
D_x = 1.295 Mg m^{-3}
D_m = 1.29 Mg m^{-3}
D_m measured by flotation
Cu $K\alpha$ radiation
λ = 1.5418 Å
Cell parameters from 25 reflections
θ = 12.64-23.97°
μ = 1.36 mm^{-1}
T = 295 (1) K
Largest face trapezoidal
0.15 × 0.12 × 0.08 mm
Pale beige

Data collection

Enraf–Nonius CAD-4 diffractometer
$\theta/2\theta$ scans
Absorption correction: none
7879 measured reflections
7879 independent reflections
5102 observed reflections [$I > 3\sigma(I)$]
$\theta_{max} = 60°$
$h = -13 \rightarrow 13$
$k = 0 \rightarrow 12$
$l = -23 \rightarrow 22$
3 standard reflections
frequency: 120 min
intensity variation: 2.3%

Refinement

Refinement on F
$R = 0.047$
$wR = 0.060$
$S = 1.91$
5102 reflections
835 parameters
Only coordinates of H atoms refined
$w = 1/\sigma^2(F)$
$(\Delta/\sigma)_{max} = 0.08$
$\Delta\rho_{max} = 0.33$ (4) e Å^{-3}
$\Delta\rho_{min} = -0.32$ (4) e Å^{-3}
Extinction correction: Stout & Jensen (1968)
Extinction coefficient: $6.2\,(1) \times 10^{-7}$
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

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

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

	x	y	z	B_{eq}
Molecule *A*				
S(101)	0.89765 (8)	0.98748 (8)	0.19768 (5)	5.40 (2)
N(102)	0.9265 (2)	0.8946 (2)	0.1224 (1)	4.54 (7)
C(103)	0.8696 (3)	0.7782 (3)	0.1008 (2)	3.88 (7)
C(104)	0.8471 (3)	0.6982 (3)	0.0286 (1)	3.92 (7)
N(105)	0.7997 (2)	0.5830 (2)	0.0389 (1)	3.80 (6)
N(106)	0.7706 (2)	0.6097 (2)	0.1072 (1)	3.84 (6)
C(107)	0.8311 (3)	0.7254 (3)	0.1430 (2)	3.87 (7)
C(108)	0.8593 (3)	0.7669 (3)	0.2149 (2)	4.08 (8)
C(109)	0.8540 (3)	0.6893 (3)	0.2539 (2)	5.00 (9)
C(110)	0.8986 (4)	0.7327 (4)	0.3204 (2)	6.5 (1)
C(111)	0.9500 (4)	0.8510 (4)	0.3485 (2)	7.0 (1)
C(112)	0.9553 (3)	0.9293 (4)	0.3114 (2)	6.2 (1)
C(113)	0.9069 (3)	0.8875 (3)	0.2453 (2)	4.74 (8)
C(114)	0.6507 (3)	0.6120 (3)	0.1134 (2)	4.06 (7)
C(115)	0.6013 (3)	0.7173 (4)	0.1533 (2)	5.6 (1)
C(116)	0.4871 (3)	0.7137 (4)	0.1618 (2)	6.8 (1)
C(117)	0.4232 (3)	0.6074 (4)	0.1294 (2)	6.9 (1)
C(118)	0.4712 (3)	0.5037 (4)	0.0891 (2)	7.6 (1)
C(119)	0.5852 (3)	0.5055 (4)	0.0820 (2)	6.3 (1)
C(120)	0.7718 (3)	0.7475 (3)	−0.0174 (1)	3.94 (7)
C(121)	0.6737 (3)	0.8056 (3)	0.0021 (2)	4.79 (8)
C(122)	0.6026 (3)	0.8445 (3)	−0.0423 (2)	4.72 (8)
C(123)	0.6324 (3)	0.8283 (3)	−0.1054 (2)	5.15 (9)
C(124)	0.7304 (3)	0.7722 (4)	−0.1253 (2)	7.4 (1)
C(125)	0.7988 (3)	0.7298 (4)	−0.0817 (2)	6.1 (1)
O(126)	0.5694 (2)	0.8653 (3)	−0.1531 (1)	7.65 (8)
C(127)	0.4687 (4)	0.9243 (5)	−0.1366 (2)	8.4 (1)
C(128)	0.8388 (3)	0.4689 (3)	0.0039 (2)	4.13 (8)
O(129)	0.8793 (2)	0.4538 (2)	−0.0491 (1)	5.83 (6)
C(130)	0.8223 (3)	0.3612 (3)	0.0285 (2)	4.04 (8)
C(131)	0.7594 (3)	0.2583 (3)	−0.0136 (2)	5.40 (9)
C(132)	0.7474 (3)	0.1525 (3)	0.0056 (2)	6.6 (1)
C(133)	0.7989 (4)	0.1512 (3)	0.0653 (2)	6.9 (1)
C(134)	0.8623 (4)	0.2524 (4)	0.1071 (2)	6.6 (1)
C(135)	0.8747 (3)	0.3587 (3)	0.0895 (2)	5.18 (9)
O(136)	0.7820 (2)	1.0186 (2)	0.1915 (1)	6.48 (7)
O(137)	0.9816 (3)	1.0859 (2)	0.2210 (1)	7.78 (8)
C(138)	1.0250 (4)	0.9173 (4)	0.0917 (3)	9.9 (1)
Molecule *B*				
S(201)	0.65115 (9)	1.0138 (1)	0.65056 (6)	6.81 (3)
N(202)	0.6086 (2)	0.8671 (3)	0.6344 (1)	5.13 (7)
C(203)	0.6595 (3)	0.7797 (3)	0.5835 (2)	4.04 (8)
C(204)	0.6650 (3)	0.6425 (3)	0.5753 (2)	3.94 (8)
N(205)	0.7078 (2)	0.5969 (2)	0.5066 (1)	3.64 (6)
N(206)	0.7517 (2)	0.6988 (2)	0.4880 (1)	3.62 (6)
C(207)	0.7020 (3)	0.8061 (3)	0.5326 (2)	4.00 (8)
C(208)	0.6861 (3)	0.9198 (3)	0.5184 (2)	4.48 (8)
C(209)	0.6938 (3)	0.9300 (3)	0.4550 (2)	5.43 (9)
C(210)	0.6612 (3)	1.0399 (3)	0.4432 (2)	7.0 (1)
C(211)	0.6192 (3)	1.1367 (3)	0.4929 (2)	7.9 (1)
C(212)	0.6120 (3)	1.1309 (3)	0.5559 (2)	7.2 (1)
C(213)	0.6477 (3)	1.0198 (3)	0.5703 (2)	5.9 (1)
C(214)	0.8727 (2)	0.7031 (3)	0.4905 (1)	3.45 (7)
C(215)	0.9351 (3)	0.8133 (3)	0.5236 (2)	4.93 (9)
C(216)	1.0507 (3)	0.8160 (4)	0.5206 (2)	5.9 (1)
C(217)	1.1040 (3)	0.7107 (4)	0.4861 (2)	5.7 (1)
C(218)	1.0428 (3)	0.6016 (3)	0.4540 (2)	5.11 (9)
C(219)	0.9275 (3)	0.5968 (3)	0.4556 (2)	4.32 (8)
C(220)	0.7335 (3)	0.6075 (3)	0.6292 (2)	4.07 (8)
C(221)	0.8262 (3)	0.6762 (3)	0.6670 (2)	4.87 (9)
C(222)	0.8860 (3)	0.6386 (3)	0.7152 (2)	5.4 (1)
C(223)	0.8528 (3)	0.5294 (3)	0.7264 (2)	4.86 (9)
C(224)	0.7609 (3)	0.4593 (3)	0.6891 (2)	5.77 (9)
C(225)	0.7024 (3)	0.4984 (3)	0.6400 (2)	5.31 (9)
O(226)	0.9165 (2)	0.4995 (2)	0.7755 (1)	6.38 (7)
C(227)	0.8807 (4)	0.3907 (4)	0.7896 (2)	7.3 (1)
C(228)	0.6584 (3)	0.4937 (3)	0.4586 (2)	3.85 (7)
O(229)	0.6122 (2)	0.4135 (2)	0.4760 (1)	4.92 (6)
C(230)	0.6635 (3)	0.4765 (3)	0.3858 (2)	4.51 (8)
C(231)	0.7087 (3)	0.3675 (4)	0.3447 (2)	6.5 (1)
C(232)	0.7048 (4)	0.3434 (5)	0.2766 (2)	9.1 (1)
C(233)	0.6542 (5)	0.4236 (5)	0.2490 (2)	9.7 (2)
C(234)	0.6074 (4)	0.5300 (4)	0.2871 (2)	8.2 (1)
C(235)	0.6137 (3)	0.5595 (4)	0.3580 (2)	6.0 (1)
O(236)	0.7657 (3)	1.0293 (3)	0.6780 (2)	8.55 (9)
O(237)	0.5682 (3)	1.0911 (3)	0.6881 (2)	9.6 (1)
C(238)	0.5351 (4)	0.8314 (4)	0.6777 (2)	7.6 (1)

Table 2. *Bond distances* (Å) *and angles* (°)

Molecule *A*		Molecule *B*	
S(101)—N(102)	1.655 (2)	S(201)—N(202)	1.650 (3)
S(101)—C(113)	1.745 (4)	S(201)—C(213)	1.715 (5)
S(101)—O(136)	1.433 (3)	S(201)—O(236)	1.426 (3)
S(101)—O(137)	1.426 (3)	S(201)—O(237)	1.435 (3)
N(102)—C(103)	1.396 (4)	N(202)—C(203)	1.384 (4)
N(102)—C(138)	1.451 (6)	N(202)—C(238)	1.466 (6)
C(103)—C(104)	1.493 (4)	C(203)—C(204)	1.508 (5)
C(103)—C(107)	1.336 (5)	C(203)—C(207)	1.344 (5)
C(104)—N(105)	1.509 (4)	C(204)—N(205)	1.504 (4)
C(104)—C(120)	1.510 (5)	C(204)—C(220)	1.507 (5)
N(105)—N(106)	1.443 (3)	N(205)—N(206)	1.446 (4)
N(105)—C(128)	1.367 (4)	N(205)—C(228)	1.359 (3)
N(106)—C(107)	1.433 (3)	N(206)—C(207)	1.435 (4)
N(106)—C(114)	1.450 (4)	N(206)—C(214)	1.443 (4)
C(107)—C(108)	1.441 (4)	C(207)—C(208)	1.428 (5)
C(108)—C(109)	1.393 (5)	C(208)—C(209)	1.393 (6)
C(108)—C(113)	1.396 (4)	C(208)—C(213)	1.401 (5)
C(109)—C(110)	1.383 (5)	C(209)—C(210)	1.399 (6)
C(110)—C(111)	1.388 (6)	C(210)—C(211)	1.374 (5)
C(111)—C(112)	1.369 (7)	C(211)—C(212)	1.363 (8)
C(112)—C(113)	1.389 (5)	C(212)—C(213)	1.449 (6)
C(114)—C(115)	1.380 (4)	C(214)—C(215)	1.387 (4)
C(114)—C(119)	1.371 (5)	C(214)—C(219)	1.388 (4)
C(115)—C(116)	1.394 (5)	C(215)—C(216)	1.388 (5)
C(116)—C(117)	1.362 (5)	C(216)—C(217)	1.368 (5)
C(117)—C(118)	1.364 (6)	C(217)—C(218)	1.369 (5)
C(118)—C(119)	1.383 (6)	C(218)—C(219)	1.381 (5)
C(120)—C(121)	1.374 (5)	C(220)—C(221)	1.377 (4)
C(120)—C(125)	1.373 (5)	C(220)—C(225)	1.383 (5)
C(121)—C(122)	1.391 (5)	C(221)—C(222)	1.377 (6)
C(122)—C(123)	1.362 (5)	C(222)—C(223)	1.396 (6)
C(123)—C(124)	1.366 (5)	C(223)—C(224)	1.373 (5)
C(123)—O(126)	1.382 (5)	C(223)—O(226)	1.368 (4)
C(124)—C(125)	1.384 (6)	C(224)—C(225)	1.395 (6)

O(126)—C(127)	1.394 (5)	O(226)—C(227)	1.432 (6)
C(128)—O(129)	1.217 (4)	C(228)—O(229)	1.232 (4)
C(128)—C(130)	1.494 (5)	C(228)—C(230)	1.491 (5)
C(130)—C(131)	1.380 (4)	C(230)—C(231)	1.395 (5)
C(130)—C(135)	1.393 (5)	C(230)—C(235)	1.375 (6)
C(131)—C(132)	1.397 (6)	C(231)—C(232)	1.370 (6)
C(132)—C(133)	1.359 (6)	C(232)—C(233)	1.353 (8)
C(133)—C(134)	1.369 (5)	C(233)—C(234)	1.362 (6)
C(134)—C(135)	1.384 (6)	C(234)—C(235)	1.417 (6)
N(102)—S(101)—C(113)	102.5 (1)	N(202)—S(201)—C(213)	100.5 (2)
N(102)—S(101)—O(136)	107.0 (1)	N(202)—S(201)—O(236)	109.0 (2)
N(102)—S(101)—O(137)	107.9 (2)	N(202)—S(201)—O(237)	106.9 (2)
C(113)—S(101)—O(136)	107.2 (2)	C(213)—S(201)—O(236)	107.7 (2)
C(113)—S(101)—O(137)	111.9 (2)	C(213)—S(201)—O(237)	111.6 (2)
O(136)—S(101)—O(137)	119.0 (2)	O(236)—S(201)—O(237)	119.5 (2)
S(101)—N(102)—C(103)	114.1 (2)	S(201)—N(202)—C(203)	115.2 (3)
S(101)—N(102)—C(138)	121.6 (2)	S(201)—N(202)—C(238)	121.7 (2)
C(103)—N(102)—C(138)	121.9 (3)	C(203)—N(202)—C(238)	122.5 (3)
N(102)—C(103)—C(104)	124.0 (3)	N(202)—C(203)—C(204)	124.3 (3)
N(102)—C(103)—C(107)	123.2 (3)	N(202)—C(203)—C(207)	123.4 (3)
C(104)—C(103)—C(107)	112.7 (3)	C(203)—C(204)—C(207)	112.1 (3)
C(103)—C(104)—N(105)	98.5 (2)	C(203)—C(204)—N(205)	98.4 (3)
C(103)—C(104)—C(120)	118.1 (2)	C(203)—C(204)—C(220)	117.5 (2)
N(105)—C(104)—C(120)	114.0 (2)	N(205)—C(204)—C(220)	115.3 (2)
C(104)—N(105)—N(106)	111.4 (2)	C(204)—N(205)—N(206)	111.8 (2)
C(104)—N(105)—C(128)	118.7 (3)	C(204)—N(205)—C(228)	119.6 (3)
N(106)—N(105)—C(128)	120.7 (3)	N(206)—N(205)—C(228)	120.3 (3)
N(105)—N(106)—C(107)	103.1 (2)	N(205)—N(206)—C(207)	102.8 (2)
N(105)—N(106)—C(114)	114.0 (2)	N(205)—N(206)—C(214)	113.0 (2)
C(107)—N(106)—C(114)	114.8 (2)	C(207)—N(206)—C(214)	116.0 (2)
C(103)—C(107)—N(106)	111.4 (2)	C(203)—C(207)—N(206)	111.5 (3)
C(103)—C(107)—C(108)	125.4 (3)	C(203)—C(207)—C(208)	124.7 (3)
N(106)—C(107)—C(108)	122.6 (3)	N(206)—C(207)—C(208)	123.2 (3)
C(107)—C(108)—C(109)	123.7 (3)	C(207)—C(208)—C(209)	123.6 (3)
C(107)—C(108)—C(113)	117.3 (3)	C(207)—C(208)—C(213)	115.9 (4)
C(109)—C(108)—C(113)	118.5 (3)	C(209)—C(208)—C(213)	120.1 (3)
C(108)—C(109)—C(110)	119.2 (3)	C(208)—C(209)—C(210)	119.5 (3)
C(109)—C(110)—C(111)	121.1 (4)	C(209)—C(210)—C(211)	120.9 (4)
C(110)—C(111)—C(112)	120.8 (3)	C(210)—C(211)—C(212)	121.2 (4)
C(111)—C(112)—C(113)	118.1 (3)	C(211)—C(212)—C(213)	119.3 (3)
S(101)—C(113)—C(108)	117.0 (2)	S(201)—C(213)—C(208)	120.3 (3)
S(101)—C(113)—C(112)	120.7 (3)	S(201)—C(213)—C(212)	120.8 (3)
C(108)—C(113)—C(112)	122.2 (4)	C(208)—C(213)—C(212)	118.8 (4)
N(106)—C(114)—C(115)	121.6 (3)	N(206)—C(214)—C(215)	121.4 (3)
N(106)—C(114)—C(119)	119.4 (3)	N(206)—C(214)—C(219)	119.2 (2)
C(115)—C(114)—C(119)	118.9 (3)	C(215)—C(214)—C(219)	119.2 (3)
C(114)—C(115)—C(116)	120.1 (3)	C(214)—C(215)—C(216)	119.7 (3)
C(115)—C(116)—C(117)	120.1 (3)	C(215)—C(216)—C(217)	120.7 (3)
C(116)—C(117)—C(118)	120.0 (4)	C(216)—C(217)—C(218)	119.7 (3)
C(117)—C(118)—C(119)	120.1 (4)	C(217)—C(218)—C(219)	120.7 (3)
C(114)—C(119)—C(118)	120.7 (3)	C(214)—C(219)—C(218)	119.9 (3)
C(104)—C(120)—C(121)	122.1 (3)	C(204)—C(220)—C(221)	123.8 (3)
C(104)—C(120)—C(125)	119.6 (3)	C(204)—C(220)—C(225)	117.7 (3)
C(121)—C(120)—C(125)	118.2 (3)	C(221)—C(220)—C(225)	118.5 (3)
C(120)—C(121)—C(122)	121.2 (3)	C(220)—C(221)—C(222)	121.0 (4)
C(121)—C(122)—C(123)	119.5 (3)	C(221)—C(222)—C(223)	120.0 (3)
C(122)—C(123)—C(124)	120.1 (4)	C(222)—C(223)—C(224)	120.0 (3)
C(122)—C(123)—O(126)	124.4 (3)	C(222)—C(223)—O(226)	116.0 (3)
C(124)—C(123)—O(126)	115.5 (3)	C(224)—C(223)—O(226)	124.0 (4)
C(123)—C(124)—C(125)	120.1 (4)	C(223)—C(224)—C(225)	119.0 (4)
C(120)—C(125)—C(124)	120.8 (3)	C(220)—C(225)—C(224)	121.5 (3)
C(123)—O(126)—C(127)	119.1 (3)	C(223)—O(226)—C(227)	116.7 (3)
N(105)—C(128)—O(129)	120.0 (3)	N(205)—C(228)—O(229)	119.2 (3)
N(105)—C(128)—C(130)	119.1 (3)	N(205)—C(228)—C(230)	120.2 (3)
O(129)—C(128)—C(130)	120.7 (3)	O(229)—C(228)—C(230)	120.6 (2)
C(128)—C(130)—C(131)	117.9 (3)	C(228)—C(230)—C(231)	118.6 (3)
C(128)—C(130)—C(135)	122.0 (3)	C(228)—C(230)—C(235)	120.7 (3)
C(131)—C(130)—C(135)	119.9 (3)	C(231)—C(230)—C(235)	120.4 (3)
C(130)—C(131)—C(132)	119.8 (3)	C(230)—C(231)—C(232)	119.9 (4)
C(131)—C(132)—C(133)	119.7 (3)	C(231)—C(232)—C(233)	119.9 (4)
C(132)—C(133)—C(134)	120.9 (4)	C(232)—C(233)—C(234)	122.0 (4)
C(133)—C(134)—C(135)	120.5 (4)	C(233)—C(234)—C(235)	119.3 (5)
C(130)—C(135)—C(134)	119.1 (3)	C(230)—C(235)—C(234)	118.5 (3)

The structure was solved by direct methods using *MULTAN*11/82 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1982). Other programs used were *ORTEP* (Johnson, 1976) and *SDP* (B. A. Frenz & Associates Inc., 1982). For H atoms, B was chosen as equal to $1.30 \times B_{eq}$ of the neighbouring heavy atom.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and geometry involving H atoms, least-squares-planes data and equations, dihedral angles between planes and torsion angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71671 (47 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: PA1044]

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A New Quassinoid Isolated from *Picrolemma pseudocoffea*

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Abstract

The rings in 15β-(acetyloxy)-13,20-epoxy-11β,12α-dihydroxy-16,2′-dioxo-2,2′-olide-picras-1(1′),2-dien-21-oic acid methyl ester, $C_{25}H_{28}O_{11}.H_2O$ (systematic name: methyl 4β-acetyloxy-14β,15α-dihydroxy-8α,12β-dimethyl-5,11-dioxo-3,3a,4,5,6a,7,7a,8,12b,-13-decahydro-3,13-ethano-1*H*,11*H*-furo[3,4-*c*]furo-

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