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Synthesis and spectroscopic and structural characterization of spiro[indoline-3,3′-indolizine]s formed by 1,3-dipolar cycloadditions between isatins, pipecolic acid and an electron-deficient alkene

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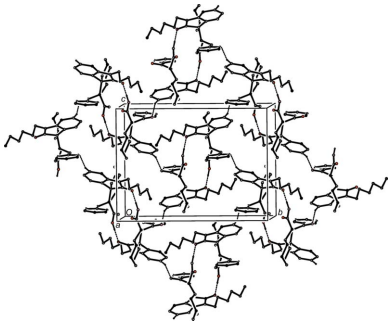
Five new spiro[indoline-3,3′-indolizine]s have been synthesized with high regio- and stereospecificity in one-pot three-component reactions between a substituted indole-2,3-dione, (*S*)-pipecolic acid and *trans*-3-benzoylacrylic acid, and subsequently characterized using a combination of elemental analysis, IR and ^{1}H and ^{13}C NMR spectroscopy, mass spectrometry and crystal structure analysis. (1′*SR*,2′*SR*,3*RS*,8a′*RS*)-2′-Benzoyl-5-fluoro-2-oxo-1′,5′,6′,7′,8′,8a′-hexahydro-2′*H*-spiro[indoline-3,3′-indolizine]-1′-carboxylic acid, $C_{23}H_{21}FN_2O_4$, (I), and (1′*SR*,2′*SR*,3*RS*,8a′*RS*)-2′-benzoyl-5-methyl-2-oxo-1′,5′,6′,7′,8′,8a′-hexahydro-2′*H*-spiro[indoline-3,3′-indolizine]-1′-carboxylic acid, $C_{24}H_{24}N_2O_4$, (II), are isomorphous, as are (1′*SR*,2′*SR*,3*RS*,8a′*RS*)-2′-benzoyl-1-methyl-2-oxo-1′,5′,6′,7′,8′,8a′-hexahydro-2′*H*-spiro[indoline-3,3′-indolizine]-1′-carboxylic acid, $C_{24}H_{24}N_2O_4$, (III), and (1′*SR*,2′*SR*,3*RS*,8a′*RS*)-2′-benzoyl-5-chloro-1-methyl-2-oxo-1′,5′,6′,7′,8′,8a′-hexahydro-2′*H*-spiro[indoline-3,3′-indolizine]-1′-carboxylic acid, $C_{24}H_{23}ClN_2O_4$, (IV). Within each isomorphous pair, the spiro ring systems show some conformational differences. In each of (I) and (II), the molecules are linked into complex sheets by a combination of four types of hydrogen bond, and in each of (III) and (IV), a combination of O—H···O and C—H···π(arene) hydrogen bonds links the molecules to form a chain of centrosymmetric rings. In (1′*SR*,2′*SR*,3*RS*,8a′*RS*)-2′-benzoyl-1-hexyl-2-oxo-1′,5′,6′,7′,8′,8a′-hexahydro-2′*H*-spiro[indoline-3,3′-indolizine]-1′-carboxylic acid, $C_{29}H_{34}N_2O_4$, (V), a combination of five hydrogen bonds links the molecules into sheets of alternating $R_2^2(16)$ and $R_6^6(46)$ rings. A mechanism is proposed for the formation of compounds (I)–(V) and some comparisons with related structures are made.

1. Introduction

Spirooxindoles are a privileged category of heterocycles containing a unique and versatile scaffold for novel drug discovery in fields as diverse as analgesics, anticancer, anti-inflammatory and antimicrobial agents, and antioxidants, whose structure–activity relationships and molecular mechanisms of action have recently been reviewed (Zhou *et al.*, 2020).

Multicomponent reactions can provide versatile and efficient routes to new heterocyclic systems, permitting the incorporation of a wide variety of functionalities by the combination of three or more simple building blocks (Dömling, 2002; Hulme & Gore, 2003; Orru & de Greef, 2003; Quiroga *et al.*, 2007, 2014). The spirooxindole core is readily obtained using 1,3-dipolar cycloadditions between electron-deficient alkenes and an azomethine ylide, generated *in situ* from an isatin (indole-2,3-dione) and an amino acid (Grigg *et al.*, 1984; Al-Majid *et al.*, 2020; Ghosh *et al.*, 2020). We have recently reported the regio- and stereospecific synthesis,



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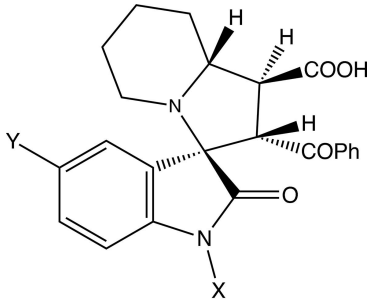
spectroscopic characterization and crystal structures of some spiro[indoline-3,3′-pyrrolizine]s (Quiroga *et al.*, 2017) and dispiro[indoline-3,3′-pyrrolizine-1′,5′-thiazolidine]s (Romo *et al.*, 2020), formed in a single step from mixtures of a substituted isatin, a cyclic amino compound and an electron-deficient alkene. As a development of these previous studies, we have now investigated the reactions between isatins, pipecolic acid [(*RS*)-piperidine-2-carboxylic acid] and *trans*-3-benzoylacrylic acid [(*E*)-4-oxo-4-phenylbut-2-enoic acid] to form spiro[indoline-3,3′-indolizine]s. Here we report the synthesis and spectroscopic charaterization, and the molecular and supramolecular structures of five representative examples, namely, (1′*SR*,2′*SR*,3*RS*,8a′*RS*)-2′-benzoyl-5-fluoro-2-oxo-1′,5′,6′,7′,8′,8a′-hexahydro-2′*H*-spiro[indoline-3,3′-indolizine]-1′-carboxylic acid, (I), (1′*SR*,2′*SR*,3*RS*,8a′*RS*)-2′-benzoyl-5-methyl-2-oxo-1′,5′,6′,-7′,8′,8a′-hexahydro-2′*H*-spiro[indoline-3,3′-indolizine]-1′-carboxylic acid, (II), (1′*SR*,2′*SR*,3*RS*,8a′*RS*)-2′-benzoyl-1-methyl-2-oxo-1′,5′,6′,7′,8′,8a′-hexahydro-2′*H*-spiro[indoline-3,3′-indolizine]-1′-carboxylic acid, (III), (1′*SR*,2′*SR*,3*RS*,8a′*RS*)-2′-benzoyl-5-chloro-1-methyl-2-oxo-1′,5′,6′,7′,8′,8a′-hexahydro-2′*H*-spiro[indoline-3,3′-indolizine]-1′-carboxylic acid, (IV), and (1′*SR*,2′*SR*,3*RS*,8a′*RS*)-2′-benzoyl-1-hexyl-2-oxo-1′,5′,6′,7′,8′,8a′-hexahydro-2′*H*-spiro[indoline-3,3′-indolizine]-1′-carboxylic acid, (V) (Scheme 1).



(I) X = H, Y = F
(II) X = H, Y = Me
(III) X = Me, Y = H
(IV) X = Me, Y = Cl
(V) X = n-hexyl, Y = H

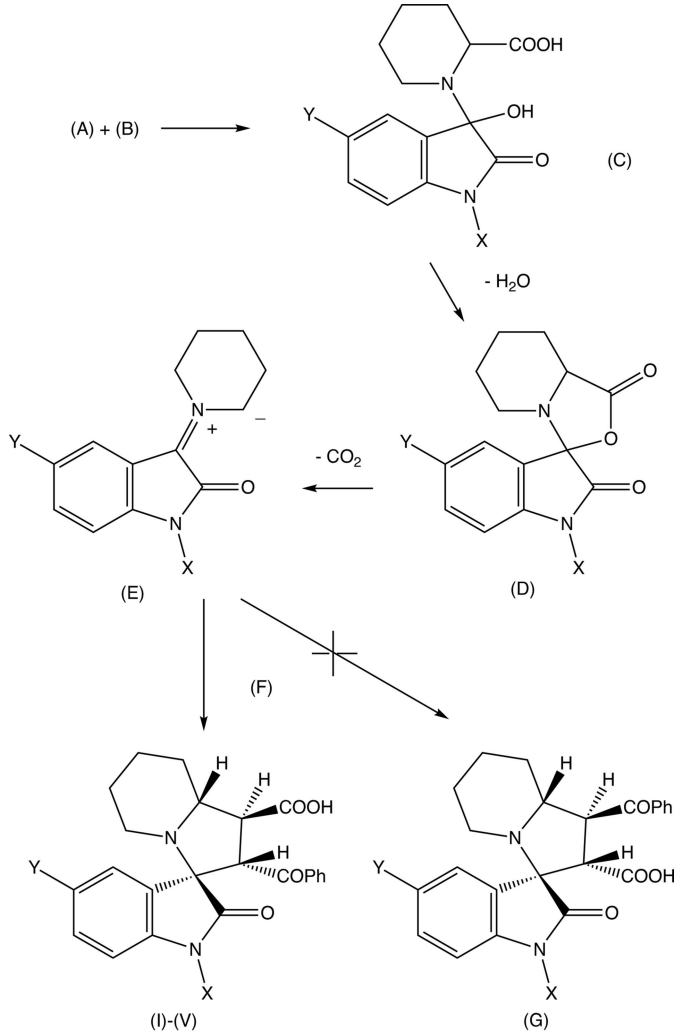
Scheme 1

Compounds (I)–(V) were formed in yields between 48 and 69% in one-pot reactions between an appropriately substituted isatin (see Scheme 2), pipecolic acid acting as the cyclic amine component and *trans*-3-benzoylacrylic acid acting as the electron-deficient alkene to give the products defined in Scheme 1 and Figs. 1–5. Products (I)–(V) were all isolated as single racemic stereoisomers and all have been characterized by a combination of elemental analysis, IR and ^{1}H and ^{13}C NMR spectroscopy, mass spectrometry and X-ray crystal structure analysis, which enables a complete definition of the stereochemistry.

2. Experimental

2.1. Synthesis and crystallization

All reagents and solvents were obtained commercially and all were used as recieved. For the synthesis of compounds (I)–(V), mixtures of pipecolic acid (64.6 mg, 0.5 mmol), the appropriately substituted isatin (0.5 mmol) [5-fluoroisatin (83.5 mg) for (I), 5-methylisatin (80.6 mg) for (II), 1-methylisatin (80.6 mg) for (III), 5-chloro-1-methylisatin (97.8 mg) for (IV) and 1-hexylisatin (115.6 mg) for (V)] and *trans*-3-benzoylacrylic acid [(*E*)-4-oxo-4-phenylbut-2-enoic acid] (88.1 mg, 0.5 mmol) in acetonitrile (10 ml) were heated under reflux until the reactions were complete, as judged by thin-layer chromatography (TLC) monitoring (reactions times were all in the range 8–12 h). The reaction mixtures were allowed to cool to ambient temperature, giving the crystalline products (I)–(V), which were collected by filtration and then dried in air. No further purification was required, as judged by TLC and spectroscopic examination, and crystals suitable for single-crystal X-ray diffraction were, in each case, selected directly from the synthesized samples.



Scheme 2

Compound (I): yield 68%; m.p. 508–509 K. Analysis found (%): C 67.6, H 5.2, N 6.8; calculated for $C_{23}H_{21}FN_2O_4$ (%): C 67.6, H 5.2, N 6.9. FT–IR (ATR, cm^{-1}): 3478, 3096, 2937, 1715, 1676. NMR (DMSO-d_6): δ(^{1}H) 1.11–1.23 (*m*, 2H, H8′, H7′), 1.23–1.33 (*m*, 1H, H6′), 1.42–1.54 (*m*, 1H, H7′), 1.66–1.79 (*m*, 1H, H8′), 2.05–2.19 (*m*, 2H, H5′, H6′), 2.20–2.32 (*m*, 1H, H5′), 3.25 (*t*, *J* = 10.0 Hz, 1H, H8a′), 3.45 (*t*, *J* = 9.7 Hz, 1H, H1′), 4.50

Table 1
Experimental details.

Experiments were carried out at 100 K with Mo $K\alpha$ radiation using a Bruker D8 Venture diffractometer. Absorption was corrected for by multi-scan methods (*SADABS*; Bruker, 2016). H atoms were treated by a mixture of independent and constrained refinement.

	(I)	(II)	(III)
Crystal data			
Chemical formula	$C_{23}H_{21}FN_2O_4$	$C_{24}H_{24}N_2O_4$	$C_{24}H_{24}N_2O_4$
M_r	408.42	404.45	404.45
Crystal system, space group	Triclinic, $P\overline{1}$	Triclinic, $P\overline{1}$	Triclinic, $P\overline{1}$
a, b, c (Å)	8.1440 (5), 8.4565 (5), 14.9945 (8)	8.1874 (6), 8.5015 (6), 15.5775 (12)	8.6535 (4), 9.2064 (4), 14.4327 (6)
α, β, γ (°)	87.549 (2), 79.926 (2), 68.467 (2)	85.775 (3), 77.641 (3), 68.022 (2)	72.660 (1), 74.539 (1), 65.930 (2)
V (Å^3)	945.52 (10)	982.15 (13)	988.16 (8)
Z	2	2	2
μ (mm^{-1})	0.11	0.09	0.09
Crystal size (mm)	0.26 × 0.21 × 0.12	0.16 × 0.12 × 0.07	0.19 × 0.19 × 0.12
Data collection			
T_{min}, T_{max}	0.939, 0.987	0.934, 0.993	0.944, 0.989
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	50206, 4727, 3889	40953, 4490, 3667	47689, 4917, 4145
R_{int}	0.068	0.069	0.057
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.668	0.650	0.667
Refinement			
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.037, 0.095, 1.04	0.040, 0.098, 1.03	0.039, 0.098, 1.04
No. of reflections	4727	4490	4917
No. of parameters	277	278	275
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.32, −0.26	0.34, −0.22	0.30, −0.33

	(IV)	(V)
Crystal data		
Chemical formula	$C_{24}H_{23}ClN_2O_4$	$C_{29}H_{34}N_2O_4$
M_r	438.89	474.58
Crystal system, space group	Triclinic, $P\overline{1}$	Monoclinic, $P2_1/n$
a, b, c (Å)	8.7914 (9), 9.3155 (10), 14.6188 (15)	11.0442 (4), 17.4707 (6), 13.0081 (4)
α, β, γ (°)	73.437 (4), 76.259 (4), 64.156 (3)	90, 90.215 (1), 90
V (Å^3)	1023.84 (19)	2509.89 (15)
Z	2	4
μ (mm^{-1})	0.22	0.08
Crystal size (mm)	0.41 × 0.32 × 0.14	0.23 × 0.13 × 0.12
Data collection		
T_{min}, T_{max}	0.934, 0.969	0.921, 0.990
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	41790, 5097, 4493	24313, 5765, 4619
R_{int}	0.055	0.059
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.668	0.650
Refinement		
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.033, 0.081, 1.03	0.043, 0.102, 1.03
No. of reflections	5097	5765
No. of parameters	284	320
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.33, −0.31	0.29, −0.26

Computer programs: *APEX3* (Bruker, 2018), *SAINT* (Bruker, 2017), *SHELXT2014* (Sheldrick, 2015*a*), *SHELXL2014* (Sheldrick, 2015*b*) and *PLATON* (Spek, 2020).

(*d*, *J* = 9.7 Hz, 1H, H2′), 6.41 (*dd*, *J* = 8.6, 4.3 Hz, 1H), 6.54 (*dd*, *J* = 8.2, 2.7 Hz, 1H), 6.78 (*td*, *J* = 9.0, 2.7 Hz, 1H), 7.32 (*t*, *J* = 7.6 Hz, 2H), 7.40–7.51 (*m*, 3H), 10.52 (*s*, 1H, NH), 12.69 (*s*, 1H, COOH); $\delta(^{13}C)$ 23.7 (C8′), 25.5 (C7′), 31.8 (C6′), 45.6 (C5′), 50.5 (C1′), 55.1 (C2′), 61.5 (C8a′), 71.6 (C3, C-spiro), 110.4 (*d*, J_{C-F} = 6.9 Hz, CH), 113.5 (*d*, J_{C-F} = 24.7 Hz, CH), 116.0 (*d*, J_{C-F} = 23.6 Hz, CH), 127.6 (CH), 129.0 (CH), 129.4 (*d*, J_{C-F} = 7.4 Hz, C), 133.8 (CH), 136.9 (C), 139.0 (C), 159.5 (C), 173.4 (COOH), 179.3 (C2), 197.4 (C—CO—C). MS (EI, 70 eV) *m*/*z* (%): 408 (M^+, 10), 368 (17), 336 (37), 313 (12), 275 (36), 259 (16), 231 (39), 141 (22), 105 (49), 77 (34).

Compound (II): yield 48%; m.p. 529–530 K. Analysis found (%): C 71.2, H 5.9, N 7.0; calculated for $C_{24}H_{24}N_2O_4$ (%): C 71.3, H 6.0, N 6.9. FT–IR (ATR, cm^{-1}): 3364, 3225, 2957, 1741, 1704, 1668. NMR (DMSO-d_6): $\delta(^1H)$ 1.10–1.20 (*m*, 2H, H8′, H7′), 1.21–1.32 (*m*, 1H, H6′), 1.43–1.50 (*m*, 1H, H7′), 1.67–1.74 (*m*, 1H, H8′), 2.05–2.18 (*m*, 5H, 5-CH_3, H5′, H6′), 2.23 (*td*, *J* = 10.9, 2.8 Hz, 1H, H5′), 3.22–3.29 (*m*, 1H, H8a′), 3.44 (*t*, *J* = 9.8 Hz, 1H, H1′), 4.47 (*d*, *J* = 9.6 Hz, 1H, H2′), 6.30 (*d*, *J* = 7.8 Hz, 1H, H7), 6.57 (*s*, 1H, H4), 6.71 (*d*, *J* = 7.8 Hz, 1H, H6), 7.28 (*t*, *J* = 7.7 Hz, 2H, H_m), 7.38 (*d*, *J* = 7.3 Hz, 2H, H_o), 7.43 (*t*, *J* = 7.3 Hz, 1H, H_p), 10.35 (*s*, 1H, NH), 12.62 (*s*, 1H, COOH);

δ(^{13}C) 21.0 (5-CH_3), 23.8 (C8′), 25.5 (C7′), 31.8 (C6′), 45.6 (C5′), 50.5 (C1′), 54.9 (C2′), 61.4 (C8a′), 71.4 (C-spiro), 109.1 (CH, C7), 126.7 (CH, C4), 127.5 (C), 127.6 (CH, C_o), 128.8 (CH, C_m), 129.7 (CH, C6), 130.9 (C), 133.4 (CH, C_p), 137.2 (C), 140.3 (C), 173.6 (COOH), 179.3 (C2), 197.6 (C—CO—C). MS (EI, 70 eV) *m*/*z* (%): 404 (M^+, 9), 368 (5), 332 (39), 315 (15), 271 (30), 255 (17), 227 (69), 141 (33), 105 (100), 77 (69).

Compound (III): yield 49%; m.p. 492–493 K. Analysis found (%): C 71.3, H 5.9, N 6.9; calculated for $C_{24}H_{24}N_2O_4$ (%): C 71.3, H 6.0, N 6.9. FT–IR (ATR, cm^{-1}): 2941, 2356, 1709, 1677. NMR (DMSO-d_6): δ(^{1}H) 1.09–1.21 (*m*, 2H, H8′, H7′), 1.21–1.36 (*m*, 1H, H6′), 1.38–1.52 (*m*, 1H, H7′), 1.66–1.81 (*m*, 1H, H8′), 2.01–2.18 (*m*, 2H, H5′, H6′), 2.23 (*t*, *J* = 10.2 Hz, 1H, H5′), 2.98 (*s*, 3H, N—CH_3), 3.22–3.29 (*m*, 1H, H8a′), 3.42 (*t*, *J* = 10.0 Hz, 1H, H1′), 4.47 (*d*, *J* = 9.8 Hz, 1H, H2′), 6.52 (*d*, *J* = 7.8 Hz, 1H), 6.79–6.90 (*m*, 2H), 7.01 (*t*, *J* = 7.5 Hz, 1H), 7.20–7.31 (*m*, 4H), 7.37–7.47 (*m*, 1H), 12.66 (*s*, 1H, COOH); δ(^{13}C) 23.7 (C8′), 25.4 (C7′), 26.2 (CH_3), 31.8 (C6′), 45.6 (C5′), 50.4 (C1′), 55.9 (C2′), 61.8 (C8a′), 71.1 (C3, C-spiro), 108.2 (CH), 122.9 (CH), 125.6 (CH), 127.0 (C), 127.4 (CH), 128.7 (CH), 129.6 (CH), 133.5 (CH), 137.0 (C), 144.0 (C), 173.4 (COOH), 177.4 (C2), 197.3 (C—CO—C). MS (EI, 70 eV) *m*/*z* (%): 404 (M^+, 1), 393 (12), 368 (22), 339 (26), 313 (70), 264 (34), 236 (16), 57 (100).

Compound (IV): yield 69%; m.p. 497–497 K. Analysis found (%): C 65.6, H 5.2, N 6.4; calculated for $C_{24}H_{23}ClN_2O_4$ (%): C 65.7, H 5.3, N 6.4. FT–IR (ATR, cm^{-1}): 3378, 3227, 2957, 1744, 1708, 1668. NMR (DMSO-d_6): δ(^{1}H) 1.11–1.22 (*m*, 2H), 1.22–1.36 (*m*, 1H), 1.40–1.50 (*m*, 1H), 1.66–1.79 (*m*, 1H), 2.05–2.19 (*m*, 2H), 2.19–2.29 (*m*, 1H), 2.97 (*s*, 3H, CH_3), 3.24 (*td*, *J* = 10.2, 2.5 Hz, 1H, H8a′), 3.43 (*t*, *J* = 10.0 Hz, 1H, H1′), 4.48 (*d*, *J* = 9.9 Hz, 1H, H2′), 6.56 (*d*, *J* = 8.3 Hz, 1H), 6.79 (*d*, *J* = 2.1 Hz, 1H), 7.09 (*dd*, *J* = 8.3, 2.2 Hz, 1H), 7.23–7.35 (*m*, 4H), 7.45 (*td*, *J* = 7.0, 1.6 Hz, 1H); δ(^{13}C) 23.6 (C8′), 25.4 (C7′), 26.4 (CH_3), 31.7 (C6′), 45.7 (C5′), 50.2 (C1′), 56.1 (C2′), 62.0 (C8a′), 71.1 (C3, C-spiro), 109.9 (CH), 125.5 (CH), 127.0 (C), 127.4 (CH), 128.9 (CH), 129.2 (C), 129.5 (CH), 133.8 (CH), 136.8 (C), 142.9 (C), 173.3 (COOH), 177.0 (C2), 197.3 (C—CO—C). MS (EI, 70 eV) *m*/*z* (%): 438 (M^+, 1), 336 (17), 313 (18), 275 (17), 231 (30), 141 (41), 105 (91), 77 (55), 57 (87), 43 (100).

Compound (V): yield 48%; m.p. 449–450 K. Analysis found (%): C 73.4, H 7.2, N 5.9; calculated for $C_{29}H_{34}N_2O_4$ (%): C 73.4, H 7.2, N 5.9. FT–IR (ATR, cm^{-1}): 2931, 2858, 1723, 1684, 1662. NMR (DMSO-d_6): δ(^{1}H) 0.79–0.91 (*m*, 3H, CH_3), 1.08–1.22 (*m*, 2H), 1.20–1.33 (*m*, 7H), 1.35–1.57 (*m*, 3H), 1.66–1.78 (*m*, 1H), 2.00–2.10 (*m*, 1H), 2.11–2.27 (*m*, 2H), 3.23–3.29 (*m*, 1H, H8a′), 3.37–3.51 (*m*, 2H, NCHH, H1′), 3.60 (*dt*, *J* = 14.5, 7.4 Hz, 1H, NCHH), 4.48 (*d*, *J* = 9.7 Hz, 1H, H2′), 6.60 (*d*, *J* = 7.8 Hz, 1H), 6.74–6.91 (*m*, 2H), 7.01 (*t*, *J* = 7.5 Hz, 1H), 7.19–7.32 (*m*, 4H), 7.42 (*t*, *J* = 7.2 Hz, 1H), 12.68 (*s*, 1H, COOH); δ(^{13}C) 14.3 (CH_3), 22.5 (CH_2), 23.7 (C8′), 25.5 (C7′), 26.4 (CH_2), 27.4 (CH_2), 31.3 (CH_2), 31.8 (C6′), 39.7 (CH_2), 45.5 (C5′), 50.5 (C1′), 55.4 (C2′), 61.6 (C8a′), 70.9 (C3, C-spiro), 108.4 (CH), 122.7 (CH), 126.0 (CH), 126.9 (C), 127.5 (CH), 128.8 (CH), 129.6 (CH), 133.5 (CH), 143.5 (C), 173.5 (COOH), 177.2 (C2), 197.4 (C—CO—C). MS (EI, 70 eV) *m*/*z* (%): 475 (M^+ + H, 3), 368 (22), 339 (35), 313 (75), 264 (39).

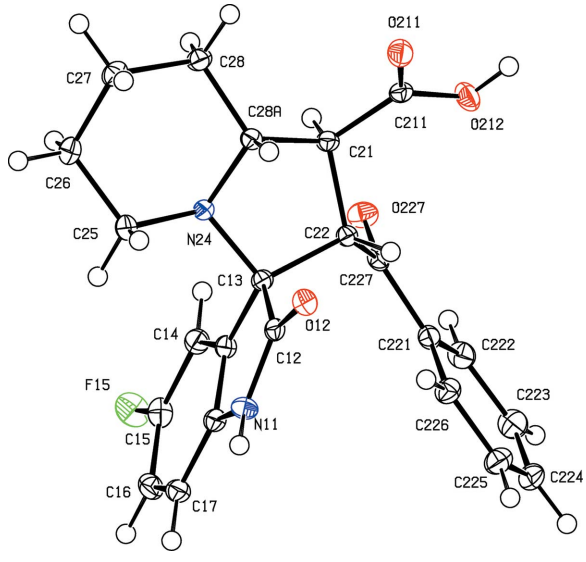


Figure 1
The molecular structure of the (1′*S*,2′*S*,3*RS*,8a′*R*) enantiomer of compound (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level.

2.2. Refinement

Crystal data, data collection and refinement details are summarized in Table 1. The crystallographic atom labelling followed the convention employed previously (Quiroga *et al.*, 2017; Romo *et al.*, 2020). For compound (II), five low-angle reflections which had been attenuated by the beam stop (101, 111, 0$\bar{1}$1, $\bar{1}$02 and $\bar{1}$03) were omitted from the data set. All H atoms were located in difference maps. H atoms bonded to C atoms were then treated as riding atoms in geometrically idealized positions, with C—H = 0.95 (aromatic), 0.98 (CH_3), 0.99 (CH_2) or 1.00 Å (aliphatic C—H) and U_{iso}(H) = kU_{eq}(C), where k = 1.5 for the methyl groups, which were permitted to rotate but not to tilt, and 1.2 for all other H atoms bonded to C atoms. For the H atoms bonded to N or O atoms, the atomic coordinates were refined with U_{iso}(H) = 1.2U_{eq}(N) or 1.5U_{eq}(O), giving the N—H and O—H distances shown in Table 2.

3. Results and discussion

All of the signals for the H and C atoms in compounds (I)–(V) were observed in their NMR spectra, with the sole exception of the carboxyl H-atom signal in compound (IV). All of the signals were assigned using one-dimensional spectra and two-dimensional COSY, HSQC and HMBC spectra. In terms of the formation of the spiro ring system, it is necessary to consider the NMR spectra only for compound (I), as those for (II)–(V) follow very similar lines, apart from the obvious differences arising from the differences in the peripheral substituents. The signals from atoms H1′ and H2′, bonded to atoms C1′ and C2′ (C21 and C22 in the crystallographic numbering scheme; see Fig. 1) which originated in the electron-deficient alkene, show a mutual coupling of 9.7 Hz, while

Table 2
Hydrogen-bond parameters (Å, °).

*Cg*1 represents the centroid of the C221–C226 ring.

	D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
(I)	N11—H11···O227^{i}	0.868 (18)	2.242 (17)	2.9756 (16)	142.2 (13)
	O212—H212···O12^{ii}	0.899 (19)	1.873 (19)	2.7723 (14)	178.4 (15)
	C22—H22···O211^{ii}	1.00	2.22	3.1930 (15)	164
	C16—H15···*Cg*1^{iii}	0.95	2.63	3.4966 (14)	152
(II)	N11—H11···O227^{i}	0.880 (18)	2.181 (18)	2.9598 (18)	147.3 (15)
	O212—H212···O12^{ii}	0.89 (2)	1.88 (2)	2.7691 (15)	179 (2)
	C22—H22···O211^{ii}	1.00	2.21	3.1894 (16)	165
	C16—H15···*Cg*1^{iii}	0.95	2.75	3.6366 (16)	155
(III)	O212—H212···O211^{ii}	0.920 (19)	1.798 (19)	2.7162 (14)	175.5 (16)
	C16—H16···*Cg*1^{iv}	0.95	2.70	3.5181 (17)	144
(IV)	O212—H212···O211^{ii}	0.863 (19)	1.854 (19)	2.7152 (14)	175.3 (16)
	C16—H16···*Cg*1^{iv}	0.95	2.52	3.3872 (14)	152
(V)	O212—H212···O12^{ii}	0.915 (18)	1.744 (18)	2.6589 (13)	178.5 (15)
	C113—H11*F*···O227^{v}	0.99	2.51	3.4738 (17)	163
	C16—H16···O227^{vi}	0.95	2.48	3.3947 (17)	162
	C22—H22···O211^{ii}	1.00	2.57	3.5693 (16)	177
	C226—H226···O211^{ii}	0.95	2.50	3.3854 (17)	155

Symmetry codes: (i) $x+1, y, z$; (ii) $-x+1, -y+1, -z+1$; (iii) $-x+2, -y+1, -z$; (iv) $-x, -y+1, -z$; (v) $-x+\frac{1}{2}, y+\frac{1}{2}, -z+\frac{1}{2}$; (vi) $x-\frac{1}{2}, -y+\frac{1}{2}, z-\frac{1}{2}$.

H1′ is similarly coupled to H8a′, bonded to C8a′ (C28*A*). These signals confirm the formation of the new ring and the magnitude of the coupling constants show (Karplus, 1959) that atom H1′ is *trans* to both H2′ and H8′, so establishing the relative stereochemistry at atoms C1′, C2′ and C8a′ (C21, C22 and C28*A*). However, the NMR data do not allow definition of the stereochemistry of the spiro C atom relative to these three centres, nor that of the relative location of the benzoyl and carbonyl substituents, both of which were determined from the single-crystal diffraction study.

Compounds (I) and (II) are isomorphous, as are compounds (III) and (IV) (Table 1). Each compound contains four contiguous stereogenic centres, at atoms C21, C22, C13 and C28*A* (Figs. 1–5), and the centrosymmetric space groups (Table 1) confirm that each compound has crystallized as a racemic mixture. For each compound, the reference molecule was selected as one having the *R* configuration at atom C13; on this basis, the configurations at atoms C21, C22 and C28*A* are *S*, *S* and *R*, respectively, with these atoms corresponding to locants C3, C1′, C2′ and C8a′ in the chemical numbering scheme, so that the overall configuration in each of (I)–(V) is (1′*SR*,2′*SR*,3*RS*,8a′*RS*). The structure analyses also show that for each compound, the carboxyl group is bonded to atom C21 and the benzoyl group is bonded to atom C22 (Figs. 1–5).

A plausible mechanism for the formation of compounds (I)–(V), based on previous work (Pardasani *et al.*, 2003; Quiroga *et al.*, 2017; Romo *et al.*, 2020), involves a condensation reaction between a substituted isatin (*A*) (Scheme 2) and pipecolic acid (*B*) to give intermediate (*C*), followed by dehydration to (*D*) and decarboxylation to give the ylide (*E*). The subsequent reaction between ylide (*E*) and *trans*-3-benzoylacrylic acid (*F*), neither of which contains any stereogenic centres, is both regio- and stereospecific. Compounds (I)–(V) were all formed as racemic mixtures of a single stereoisomer, and formation of the alternative regioisomers of type (*G*) was not detected in any of the reactions. The *endo* approach of the alkene to the ylide is preferred over the alternative *exo* approach, as its transition state is better stabilized by π–π interactions between the aryl groups in the two reaction components.

The synthetic pathway defined in Scheme 2 thus significantly amplifies the scope of the ylide/alkene route to novel spiro compounds. The product yields, which are comparable with, say, those of a three-step process with conversions of 80–85% at each stage, are regarded as entirely acceptable in view of the one-step nature of the procedure, the ready availability of starting materials which permit a very wide range of

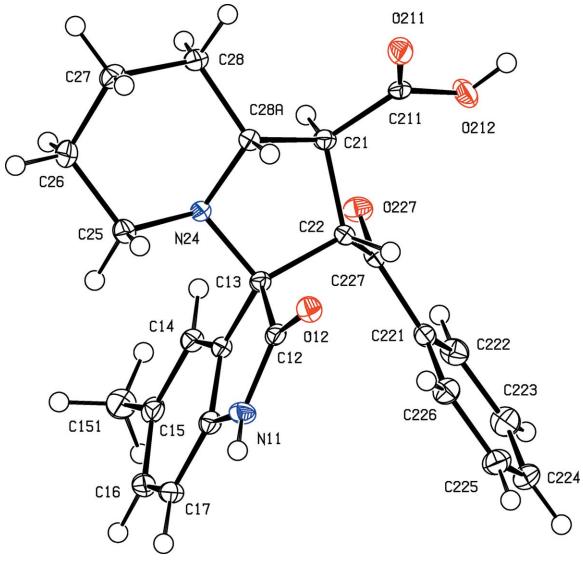


Figure 2
The molecular structure of the (1′*S*,2′*S*,3*RS*,8a′*R*) enantiomer of compound (II), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level.

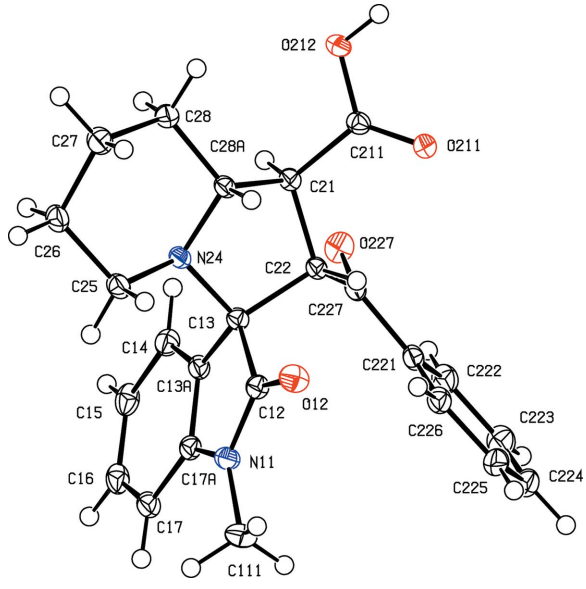


Figure 3
The molecular structure of the (1′*S*,2′*S*,3*RS*,8a′*R*) enantiomer of compound (III), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level.

Table 3
Ring-puckering parameters (Å, °).

Parameters for rings *A* and *B* are calculated for the atom sequences N24—C13—C22—C21—C28*A* and N24—C25—C26—C27—C28—C28*A*, respectively.

Ring *A*	Q_2	φ_2
(I)	0.4391 (11)	333.05 (17)
(II)	0.4363 (14)	332.26 (19)
(III)	0.4436 (13)	312.16 (17)
(IV)	0.4456 (13)	317.67 (17)
(V)	0.4125 (13)	327.81 (18)

Ring *B*	Q	θ	φ
(I)	0.5673 (14)	175.99 (14)	227.3 (19)
(II)	0.5670 (15)	176.31 (15)	226 (2)
(III)	0.5846 (14)	176.53 (14)	141 (3)
(IV)	0.5913 (14)	176.98 (14)	132 (3)
(V)	0.5778 (14)	179.45 (14)	219 (22)

substituent combinations, and the regio- and stereospecificity giving racemic mixtures of single stereoisomers.

The conformations (Evans & Boeyens, 1989) of the five-membered ring containing atom N24 show some unexpected variations. Thus, in the isomorphous pair (I) and (II), this ring adopts a half-chair conformation in (I), but an envelope conformation in (II). In (I), the ring is twisted about a line between atom C22 and the mid-point of the N24—C28*A* bond, such that atoms C13 and C21 are displaced to either side of the plane through atoms C22, N24 and C28*A* by 0.5324 (18) and 0.6374 (15) Å, respectively. In contrast, the corresponding ring in (II) adopts an envelope conformation, with the ring folded across the C21···N24 line and with atom C28*A* displaced by 0.6528 (19) Å from the plane through atoms C21, C22, C13 and N24. Similarly, in the isomorphous pair (III) and (IV), this ring adopts a half-chair conformation, but now twisted across the line between atom C13 and the mid-point of the C21—C28*A* bond, with atoms C22 and C24 displaced to either side of the plane through C13, C21 and C28*A* by 0.5410 (18) and 0.5819 (16) Å, respectively, while in (IV), this ring adopts the envelope conformation, folded across the C21···C24 line, with atom C28*A* displaced by 0.6709 (16) Å from the plane of the other four atoms. The same envelope conformation is found in (V), with a displacement of 0.6284 (19) Å for atom C28*A*. In each of (I)–(V), the six-membered ring containing atom N24 adopts an almost perfect chair conformation, with substituents

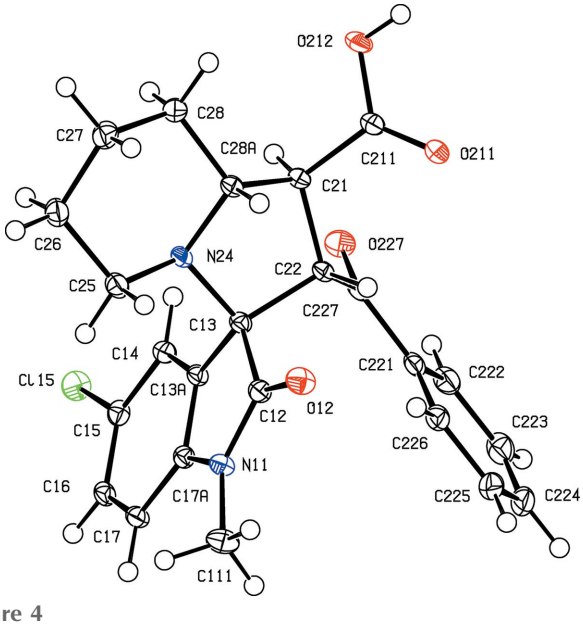


Figure 4
The molecular structure of the (1′*S*,2′*S*,3*RS*,8a′*R*) enantiomer of compound (IV), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level.

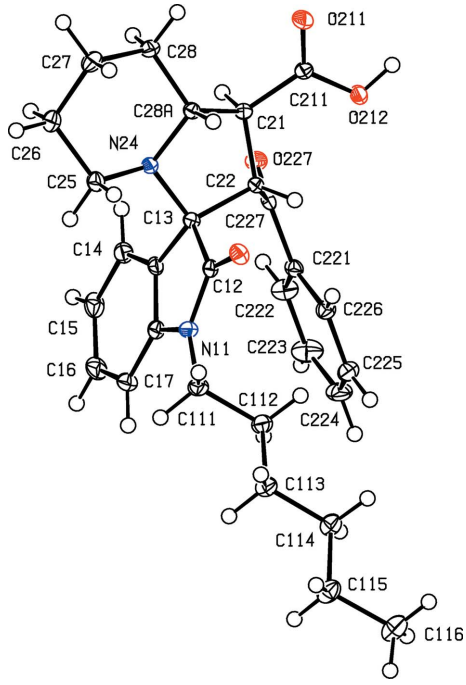


Figure 5
The molecular structure of the (1′*S*,2′*S*,3*RS*,8a′*R*) enantiomer of compound (V), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level.

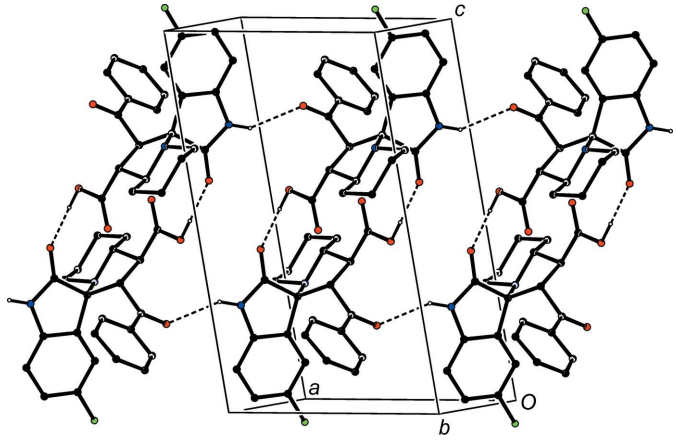


Figure 6
Part of the crystal structure of compound (I), showing the formation of a chain of rings along [100] built from O—H···O and N—H···O hydrogen bonds. For the sake of clarity, H atoms bonded to C atoms have all been omitted.

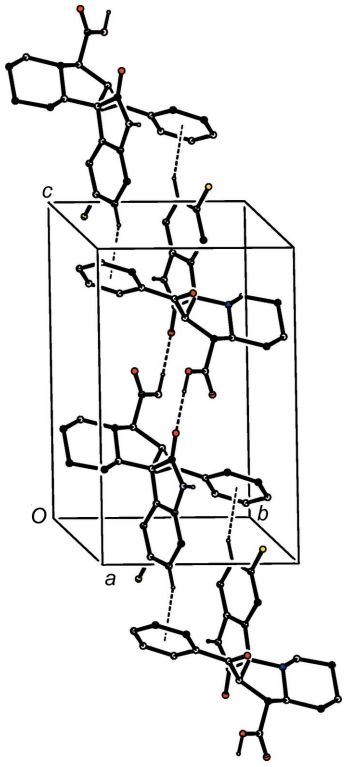


Figure 7
Part of the crystal structure of compound (I), showing the formation of a chain of rings along [10$\bar{1}$] built from O—H···O and C—H···π(arene) hydrogen bonds. For the sake of clarity, H atoms bonded to those C atoms not involved in the motif shown have been omitted.

C13 and C21 both in equatorial sites. The values of the ring-puckering parameters (Cremer & Pople, 1975; Boeyens, 1978) are summarized in Table 3. In view of the conformational differences within the isomorphous pairs (I)/(II) and (III)/(IV), it may not be appropriate to regard these pairs as strictly isostructural (Acosta *et al.*, 2009; Blanco *et al.*, 2012).

In the structure of compound (I), four hydrogen bonds (Table 2) link the molecules into complex sheets whose formation can, however, be readily analysed in terms of two one-dimensional substructures (Ferguson *et al.*, 1998*a*,*b*; Gregson *et al.*, 2000). A combination of O—H···O and N—H···O hydrogen bonds forms a ribbon in the form of a chain of edge-fused centrosymmetric rings running parallel to [100], in which $R_2^2(16)$ (Etter, 1990; Etter *et al.*, 1990; Bernstein *et al.*, 1995) rings centred at $(n + \frac{1}{2}, \frac{1}{2}, \frac{1}{2})$ alternate with $R_4^4(22)$ rings centred at $(n, \frac{1}{2}, \frac{1}{2})$, where n represents an integer in each case (Fig. 6). The formation of this ribbon is modestly enhanced by a C—H···O hydrogen bond involving a C—H bond of rather low acidity. In the second substructure, a combination of O—H···O and C—H···π(arene) hydrogen bonds forms a second chain of rings, this time running parallel to [10$\bar{1}$], in which $R_2^2(16)$ rings centred at $(n + \frac{1}{2}, \frac{1}{2}, -n + \frac{1}{2})$ alternate with rings formed by the C—H···π(arene) hydrogen bonds, which are centred at $(n, \frac{1}{2}, 1 - n)$, where n represents an integer in each case (Fig. 7). The combination of these two chain motifs generates a sheet lying parallel to (101), but there are no direction-specific interactions between adjacent sheets. The supramolecular assembly of the isomorphous compound (II) is entirely similar to that in (I).

In the isomorphous pair of compounds (III) and (IV), there are just two hydrogen bonds (Table 2), and these link the molecules into a chain of centrosymmetric rings running parallel to [101], in which $R_2^2(8)$ rings formed by the O—H···O hydrogen bonds and centred at $(n + \frac{1}{2}, \frac{1}{2}, n + \frac{1}{2})$ alternate with rings formed by C—H···π(arene) hydrogen bonds and centred at $(n, \frac{1}{2}, n)$, where n represents an integer in each case (Fig. 8). There are no direction-specific interactions between adjacent chains.

Five hydrogen bonds (Table 2) link the molecules of compound (V) into sheets lying parallel to (10$\bar{1}$), but the formation of the sheet can, in fact, be analysed in terms of just two of these interactions, those having atoms O212 and C16 as the donors. Inversion-related pairs of molecules are linked by paired O—H···O hydrogen bonds to form centrosymmetric $R_2^2(16)$ dimers, of the type seen also in compounds (I) and (II), although in (V) the dimer formation is weakly augmented by two C—H···O interactions. Linkage of these dimers by the C—H···O hydrogen bond involving atom C16 then generates a sheet in which centrosymmetric rings of $R_2^2(16)$ and $R_6^6(46)$ types alternate in a chessboard fashion (Fig. 9). There are no direction-specific interactions between adjacent sheets.

Overall, therefore, the supramolecular assembly is one-dimensional in each of compounds (III) and (IV), and two-dimensional in (I), (II) and (V); however, a three-dimensional assembly is not observed amongst the examples reported here.

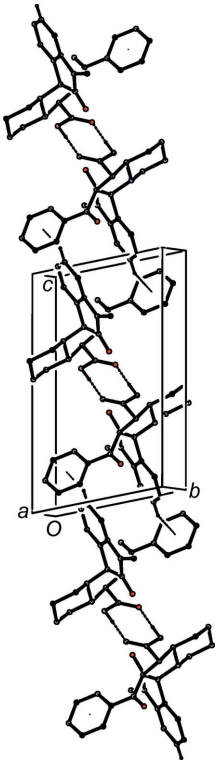


Figure 8
Part of the crystal structure of compound (III), showing the formation of a chain of rings along [101] built from O—H···O and C—H···π(arene) hydrogen bonds. For the sake of clarity, H atoms not involved in the motif shown have been omitted.

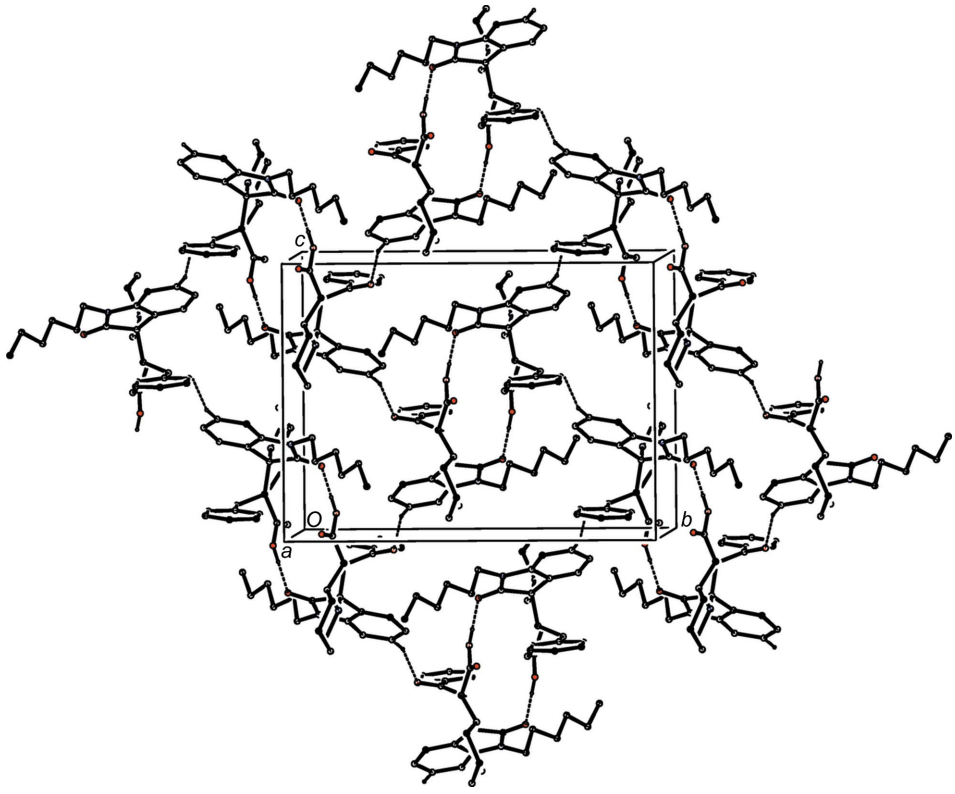


Figure 9
Part of the crystal structure of compound (V), showing the formation of a sheet lying parallel to (10$\bar{1}$) built from O—H···O and C—H···O hydrogen bonds. For the sake of clarity, H atoms bonded to those C atoms not involved in the motif shown have been omitted.

This may be contrasted with the behaviour observed in two spiro[indoline-3,3′-pyrrolizine]s (Quiroga *et al.*, 2017). In (1′*RS*,2′*RS*,3*SR*,7a′*RR*)-1′,2′-bis(4-chlorobenzoyl)-5,7-dichloro-2-oxo-1′,2′,5′,6′,7′,7a′-hexahydrospiro[indoline-3,3′-pyrrolizine], which crystallizes as a partial dichloromethane solvate, the heterocyclic molecules are linked by N—H···O hydrogen bonds to form $R_2^2(8)$ dimers, while in (1′*RS*,2′*RS*,3*SR*,7a′*SR*)-2′-benzoyl-1-hexyl-2-oxo-1′,2′,5′,6′,7′,7a′-hexahydrospiro[indoline-3,3′-pyrrolizine]-1′-carboxylic acid, the molecules are linked by O—H···O hydrogen bonds to form cyclic $R_6^6(48)$ hexamers with $\bar{3}$ (S_6) symmetry, which are further linked by C—H···O hydrogen bonds to form a three-dimensional framework structure.

In summary, therefore, we have developed a new application of the ylide/alkene procedure, which we have now used for the formation of spiro[indoline-3,3′-indolizine]s in a single step, using simple and readily available starting materials. This approach permits the incorporation of a wide variety of substituents and other functional groups for further elaboration. Five representative examples have been fully characterized spectroscopically and structurally, and their patterns of supramolecular assembly have been analysed, described and illustrated.

Acknowledgements

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supporting information

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Synthesis and spectroscopic and structural characterization of spiro-[indoline-3,3′-indolizine]s formed by 1,3-dipolar cycloadditions between isatins, pipecolic acid and an electron-deficient alkene

Pablo E. Romo, Jairo Quiroga, Justo Cobo and Christopher Glidewell

Computing details

For all structures, data collection: *APEX3* (Bruker, 2018); cell refinement: *SAINT* (Bruker, 2017); data reduction: *SAINT* (Bruker, 2017); program(s) used to solve structure: SHELXT2014 (Sheldrick, 2015a); program(s) used to refine structure: *SHELXL2014* (Sheldrick, 2015b); molecular graphics: *PLATON* (Spek, 2020); software used to prepare material for publication: *SHELXL2014* (Sheldrick, 2015b) and *PLATON* (Spek, 2020).

(1′*SR*,2′*SR*,3*RS*,8a′*RS*)-2′-Benzoyl-5-fluoro-2-oxo-1′,5′,6′,7′,8′,8a′-hexahydro-2′*H*-spiro[indoline-3,3′-indolizine]-1′-carboxylic acid (I)

Crystal data

$C_{23}H_{21}FN_2O_4$
M_r = 408.42
Triclinic, $P\overline{1}$
a = 8.1440 (5) Å
b = 8.4565 (5) Å
c = 14.9945 (8) Å
α = 87.549 (2)°
β = 79.926 (2)°
γ = 68.467 (2)°
V = 945.52 (10) Å^3
Z = 2
$F(000)$ = 428
D_x = 1.434 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 4727 reflections
θ = 2.6–28.4°
μ = 0.11 mm^{-1}
T = 100 K
Block, colourless
0.26 × 0.21 × 0.12 mm

Data collection

Bruker D8 Venture diffractometer
Radiation source: INCOATEC high brilliance microfocus sealed tube
Multilayer mirror monochromator
φ and ω scans
Absorption correction: multi-scan (SADABS; Bruker, 2016)
T_{min} = 0.939, T_{max} = 0.987
50206 measured reflections
4727 independent reflections
3889 reflections with $I > 2\sigma(I)$
R_{int} = 0.068
θ_{max} = 28.4°, θ_{min} = 2.6°
h = −10→10
k = −11→11
l = −20→19

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.037
$wR(F^2)$ = 0.095
S = 1.04
4727 reflections
277 parameters
0 restraints

Primary atom site location: dual
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0348P)^2 + 0.5208P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.32$ e Å^{-3}
$\Delta\rho_{min} = -0.25$ e Å^{-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.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
N11	1.08269 (14)	0.39724 (14)	0.25922 (7)	0.0158 (2)
H11	1.170 (2)	0.426 (2)	0.2691 (10)	0.019*
C12	0.95853 (15)	0.37142 (15)	0.32522 (8)	0.0134 (2)
O12	0.94302 (11)	0.39830 (11)	0.40642 (6)	0.01625 (18)
C13	0.84286 (15)	0.29780 (14)	0.27968 (8)	0.0126 (2)
C13A	0.92063 (16)	0.29951 (15)	0.18062 (8)	0.0139 (2)
C14	0.88091 (17)	0.24167 (16)	0.10478 (8)	0.0166 (2)
H14	0.7864	0.1999	0.1091	0.020*
C15	0.98626 (17)	0.24807 (16)	0.02246 (8)	0.0182 (3)
F15	0.94893 (11)	0.19433 (11)	−0.05352 (5)	0.02647 (19)
C16	1.12559 (17)	0.30658 (16)	0.01302 (8)	0.0188 (3)
H16	1.1924	0.3099	−0.0452	0.023*
C17	1.16756 (16)	0.36082 (16)	0.08971 (8)	0.0172 (2)
H17	1.2640	0.4001	0.0853	0.021*
C17A	1.06354 (16)	0.35527 (15)	0.17253 (8)	0.0143 (2)
C21	0.57206 (15)	0.27283 (15)	0.37179 (8)	0.0133 (2)
H21	0.5176	0.2197	0.3328	0.016*
C22	0.63827 (15)	0.40201 (15)	0.31493 (8)	0.0125 (2)
H22	0.6285	0.4960	0.3562	0.015*
N24	0.86385 (13)	0.12371 (12)	0.30761 (7)	0.0134 (2)
C25	1.04769 (16)	0.00583 (15)	0.30830 (9)	0.0177 (2)
H25A	1.0990	0.0425	0.3551	0.021*
H25B	1.1231	0.0044	0.2487	0.021*
C26	1.04418 (18)	−0.17129 (16)	0.32871 (9)	0.0208 (3)
H26A	1.1670	−0.2519	0.3317	0.025*
H26B	1.0009	−0.2105	0.2794	0.025*
C27	0.92145 (17)	−0.16920 (16)	0.41871 (9)	0.0193 (3)
H27A	0.9741	−0.1451	0.4688	0.023*
H27B	0.9130	−0.2827	0.4282	0.023*
C28	0.73338 (16)	−0.03508 (15)	0.42078 (8)	0.0160 (2)
H28A	0.6720	−0.0700	0.3782	0.019*
H28B	0.6627	−0.0266	0.4824	0.019*
C28A	0.74292 (16)	0.13729 (15)	0.39444 (8)	0.0134 (2)
H28C	0.7862	0.1818	0.4427	0.016*

C211	0.43487 (16)	0.35381 (15)	0.45420 (8)	0.0148 (2)
O211	0.45433 (12)	0.31384 (11)	0.53129 (6)	0.0196 (2)
O212	0.28836 (12)	0.47406 (12)	0.43253 (7)	0.0212 (2)
H212	0.212 (3)	0.517 (2)	0.4842 (12)	0.032*
C227	0.53320 (15)	0.47914 (15)	0.23910 (8)	0.0136 (2)
O227	0.44923 (12)	0.40598 (12)	0.20899 (6)	0.0192 (2)
C221	0.53110 (16)	0.64745 (15)	0.20304 (8)	0.0149 (2)
C222	0.42462 (17)	0.72116 (17)	0.13716 (9)	0.0192 (3)
H222	0.3617	0.6611	0.1145	0.023*
C223	0.41032 (19)	0.88090 (18)	0.10488 (9)	0.0229 (3)
H223	0.3363	0.9307	0.0610	0.028*
C224	0.50443 (19)	0.96829 (17)	0.13682 (9)	0.0219 (3)
H224	0.4940	1.0781	0.1149	0.026*
C225	0.61343 (19)	0.89555 (17)	0.20057 (9)	0.0207 (3)
H225	0.6797	0.9543	0.2212	0.025*
C226	0.62558 (17)	0.73600 (16)	0.23430 (8)	0.0174 (2)
H226	0.6985	0.6873	0.2788	0.021*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N11	0.0137 (5)	0.0190 (5)	0.0171 (5)	−0.0089 (4)	−0.0027 (4)	0.0011 (4)
C12	0.0106 (5)	0.0111 (5)	0.0172 (6)	−0.0025 (4)	−0.0027 (4)	0.0013 (4)
O12	0.0148 (4)	0.0182 (4)	0.0156 (4)	−0.0053 (3)	−0.0035 (3)	−0.0006 (3)
C13	0.0119 (5)	0.0116 (5)	0.0146 (5)	−0.0046 (4)	−0.0027 (4)	0.0016 (4)
C13A	0.0121 (5)	0.0127 (5)	0.0156 (5)	−0.0031 (4)	−0.0022 (4)	0.0017 (4)
C14	0.0155 (6)	0.0163 (6)	0.0183 (6)	−0.0058 (5)	−0.0036 (5)	0.0003 (4)
C15	0.0196 (6)	0.0184 (6)	0.0143 (6)	−0.0032 (5)	−0.0049 (5)	−0.0006 (4)
F15	0.0287 (4)	0.0362 (5)	0.0158 (4)	−0.0126 (4)	−0.0043 (3)	−0.0041 (3)
C16	0.0161 (6)	0.0197 (6)	0.0160 (6)	−0.0028 (5)	0.0001 (5)	0.0033 (5)
C17	0.0135 (6)	0.0168 (6)	0.0199 (6)	−0.0048 (5)	−0.0017 (5)	0.0045 (5)
C17A	0.0132 (5)	0.0129 (5)	0.0159 (6)	−0.0036 (4)	−0.0030 (4)	0.0028 (4)
C21	0.0121 (5)	0.0135 (5)	0.0151 (5)	−0.0058 (4)	−0.0020 (4)	0.0019 (4)
C22	0.0109 (5)	0.0121 (5)	0.0144 (5)	−0.0041 (4)	−0.0022 (4)	0.0015 (4)
N24	0.0117 (5)	0.0109 (4)	0.0163 (5)	−0.0036 (4)	−0.0006 (4)	0.0020 (4)
C25	0.0129 (6)	0.0145 (6)	0.0224 (6)	−0.0021 (5)	−0.0009 (5)	0.0016 (5)
C26	0.0190 (6)	0.0133 (6)	0.0252 (7)	−0.0018 (5)	−0.0009 (5)	0.0018 (5)
C27	0.0196 (6)	0.0138 (6)	0.0237 (6)	−0.0054 (5)	−0.0043 (5)	0.0047 (5)
C28	0.0165 (6)	0.0143 (6)	0.0182 (6)	−0.0068 (5)	−0.0033 (5)	0.0034 (4)
C28A	0.0118 (5)	0.0132 (5)	0.0146 (5)	−0.0043 (4)	−0.0012 (4)	0.0015 (4)
C211	0.0134 (5)	0.0130 (5)	0.0194 (6)	−0.0068 (4)	−0.0020 (4)	0.0019 (4)
O211	0.0209 (5)	0.0180 (4)	0.0168 (4)	−0.0043 (4)	−0.0013 (3)	0.0012 (3)
O212	0.0129 (4)	0.0230 (5)	0.0214 (5)	−0.0009 (4)	0.0001 (4)	0.0029 (4)
C227	0.0096 (5)	0.0153 (5)	0.0146 (5)	−0.0034 (4)	−0.0008 (4)	0.0002 (4)
O227	0.0187 (4)	0.0224 (5)	0.0214 (4)	−0.0112 (4)	−0.0081 (4)	0.0034 (4)
C221	0.0132 (5)	0.0149 (6)	0.0143 (5)	−0.0030 (4)	−0.0011 (4)	0.0009 (4)
C222	0.0160 (6)	0.0218 (6)	0.0194 (6)	−0.0057 (5)	−0.0052 (5)	0.0043 (5)
C223	0.0206 (6)	0.0242 (7)	0.0210 (6)	−0.0045 (5)	−0.0060 (5)	0.0083 (5)

C224	0.0242 (7)	0.0155 (6)	0.0213 (6)	−0.0040 (5)	−0.0005 (5)	0.0050 (5)
C225	0.0269 (7)	0.0171 (6)	0.0186 (6)	−0.0093 (5)	−0.0032 (5)	0.0017 (5)
C226	0.0201 (6)	0.0160 (6)	0.0161 (6)	−0.0064 (5)	−0.0038 (5)	0.0017 (4)

Geometric parameters (Å, º)

N11—C12	1.3576 (16)	C25—H25B	0.9900
N11—C17A	1.4111 (15)	C26—C27	1.5289 (18)
N11—H11	0.870 (17)	C26—H26A	0.9900
C12—O12	1.2243 (14)	C26—H26B	0.9900
C12—C13	1.5599 (16)	C27—C28	1.5312 (17)
C13—N24	1.4696 (15)	C27—H27A	0.9900
C13—C13A	1.5110 (16)	C27—H27B	0.9900
C13—C22	1.5767 (16)	C28—C28A	1.5199 (16)
C13A—C14	1.3891 (17)	C28—H28A	0.9900
C13A—C17A	1.3929 (17)	C28—H28B	0.9900
C14—C15	1.3858 (18)	C28A—H28C	1.0000
C14—H14	0.9500	C211—O211	1.2111 (15)
C15—F15	1.3652 (14)	C211—O212	1.3342 (15)
C15—C16	1.3793 (19)	O212—H212	0.898 (19)
C16—C17	1.3955 (18)	C227—O227	1.2234 (15)
C16—H16	0.9500	C227—C221	1.4960 (17)
C17—C17A	1.3849 (17)	C221—C226	1.3971 (17)
C17—H17	0.9500	C221—C222	1.4023 (17)
C21—C211	1.5074 (16)	C222—C223	1.3859 (19)
C21—C28A	1.5253 (16)	C222—H222	0.9500
C21—C22	1.5501 (16)	C223—C224	1.392 (2)
C21—H21	1.0000	C223—H223	0.9500
C22—C227	1.5225 (16)	C224—C225	1.3876 (19)
C22—H22	1.0000	C224—H224	0.9500
N24—C25	1.4643 (15)	C225—C226	1.3955 (18)
N24—C28A	1.4695 (15)	C225—H225	0.9500
C25—C26	1.5255 (17)	C226—H226	0.9500
C25—H25A	0.9900		

C12—N11—C17A	111.66 (10)	H25A—C25—H25B	108.4
C12—N11—H11	124.4 (10)	C25—C26—C27	110.53 (10)
C17A—N11—H11	123.6 (10)	C25—C26—H26A	109.5
O12—C12—N11	125.70 (11)	C27—C26—H26A	109.5
O12—C12—C13	126.21 (11)	C25—C26—H26B	109.5
N11—C12—C13	108.08 (10)	C27—C26—H26B	109.5
N24—C13—C13A	110.59 (9)	H26A—C26—H26B	108.1
N24—C13—C12	112.62 (9)	C26—C27—C28	111.69 (10)
C13A—C13—C12	101.43 (9)	C26—C27—H27A	109.3
N24—C13—C22	103.20 (9)	C28—C27—H27A	109.3
C13A—C13—C22	120.13 (10)	C26—C27—H27B	109.3
C12—C13—C22	109.18 (9)	C28—C27—H27B	109.3
C14—C13A—C17A	120.40 (11)	H27A—C27—H27B	107.9

C14—C13A—C13	130.03 (11)	C28A—C28—C27	110.73 (10)
C17A—C13A—C13	109.26 (10)	C28A—C28—H28A	109.5
C15—C14—C13A	116.59 (11)	C27—C28—H28A	109.5
C15—C14—H14	121.7	C28A—C28—H28B	109.5
C13A—C14—H14	121.7	C27—C28—H28B	109.5
F15—C15—C16	118.31 (11)	H28A—C28—H28B	108.1
F15—C15—C14	117.98 (12)	N24—C28A—C28	110.49 (9)
C16—C15—C14	123.71 (12)	N24—C28A—C21	100.03 (9)
C15—C16—C17	119.45 (12)	C28—C28A—C21	116.54 (10)
C15—C16—H16	120.3	N24—C28A—H28C	109.8
C17—C16—H16	120.3	C28—C28A—H28C	109.8
C17A—C17—C16	117.57 (12)	C21—C28A—H28C	109.8
C17A—C17—H17	121.2	O211—C211—O212	123.70 (11)
C16—C17—H17	121.2	O211—C211—C21	124.07 (11)
C17—C17A—C13A	122.25 (11)	O212—C211—C21	112.23 (10)
C17—C17A—N11	128.25 (11)	C211—O212—H212	107.8 (11)
C13A—C17A—N11	109.48 (10)	O227—C227—C221	120.37 (11)
C211—C21—C28A	113.48 (10)	O227—C227—C22	120.55 (11)
C211—C21—C22	112.77 (10)	C221—C227—C22	119.07 (10)
C28A—C21—C22	104.15 (9)	C226—C221—C222	118.98 (11)
C211—C21—H21	108.7	C226—C221—C227	122.84 (11)
C28A—C21—H21	108.7	C222—C221—C227	118.15 (11)
C22—C21—H21	108.7	C223—C222—C221	120.56 (12)
C227—C22—C21	113.74 (9)	C223—C222—H222	119.7
C227—C22—C13	113.26 (9)	C221—C222—H222	119.7
C21—C22—C13	103.95 (9)	C222—C223—C224	119.96 (12)
C227—C22—H22	108.6	C222—C223—H223	120.0
C21—C22—H22	108.6	C224—C223—H223	120.0
C13—C22—H22	108.6	C225—C224—C223	120.19 (12)
C25—N24—C28A	113.80 (9)	C225—C224—H224	119.9
C25—N24—C13	116.13 (9)	C223—C224—H224	119.9
C28A—N24—C13	107.07 (9)	C224—C225—C226	119.93 (12)
N24—C25—C26	108.45 (10)	C224—C225—H225	120.0
N24—C25—H25A	110.0	C226—C225—H225	120.0
C26—C25—H25A	110.0	C225—C226—C221	120.37 (12)
N24—C25—H25B	110.0	C225—C226—H226	119.8
C26—C25—H25B	110.0	C221—C226—H226	119.8

C17A—N11—C12—O12	−179.45 (11)	C12—C13—N24—C25	−45.23 (13)
C17A—N11—C12—C13	1.88 (13)	C22—C13—N24—C25	−162.84 (9)
O12—C12—C13—N24	−63.41 (15)	C13A—C13—N24—C28A	−164.16 (9)
N11—C12—C13—N24	115.26 (11)	C12—C13—N24—C28A	83.15 (11)
O12—C12—C13—C13A	178.37 (11)	C22—C13—N24—C28A	−34.46 (11)
N11—C12—C13—C13A	−2.96 (12)	C28A—N24—C25—C26	61.11 (13)
O12—C12—C13—C22	50.60 (15)	C13—N24—C25—C26	−173.87 (10)
N11—C12—C13—C22	−130.73 (10)	N24—C25—C26—C27	−57.14 (14)
N24—C13—C13A—C14	56.89 (16)	C25—C26—C27—C28	54.26 (14)
C12—C13—C13A—C14	176.57 (12)	C26—C27—C28—C28A	−51.69 (14)

C22—C13—C13A—C14	−63.11 (17)	C25—N24—C28A—C28	−59.49 (13)
N24—C13—C13A—C17A	−116.61 (11)	C13—N24—C28A—C28	170.79 (9)
C12—C13—C13A—C17A	3.07 (12)	C25—N24—C28A—C21	177.10 (9)
C22—C13—C13A—C17A	123.40 (11)	C13—N24—C28A—C21	47.38 (11)
C17A—C13A—C14—C15	−1.96 (18)	C27—C28—C28A—N24	52.77 (13)
C13—C13A—C14—C15	−174.84 (12)	C27—C28—C28A—C21	166.01 (10)
C13A—C14—C15—F15	−179.10 (11)	C211—C21—C28A—N24	−163.33 (9)
C13A—C14—C15—C16	0.47 (19)	C22—C21—C28A—N24	−40.31 (11)
F15—C15—C16—C17	−179.43 (11)	C211—C21—C28A—C28	77.61 (13)
C14—C15—C16—C17	1.0 (2)	C22—C21—C28A—C28	−159.38 (10)
C15—C16—C17—C17A	−0.95 (18)	C28A—C21—C211—O211	−1.72 (17)
C16—C17—C17A—C13A	−0.55 (18)	C22—C21—C211—O211	−119.85 (13)
C16—C17—C17A—N11	177.65 (12)	C28A—C21—C211—O212	178.78 (10)
C14—C13A—C17A—C17	2.07 (18)	C22—C21—C211—O212	60.65 (13)
C13—C13A—C17A—C17	176.29 (11)	C21—C22—C227—O227	−22.27 (16)
C14—C13A—C17A—N11	−176.43 (11)	C13—C22—C227—O227	96.13 (13)
C13—C13A—C17A—N11	−2.20 (13)	C21—C22—C227—C221	156.56 (10)
C12—N11—C17A—C17	−178.22 (12)	C13—C22—C227—C221	−85.04 (13)
C12—N11—C17A—C13A	0.16 (14)	O227—C227—C221—C226	−179.45 (12)
C211—C21—C22—C227	−92.63 (12)	C22—C227—C221—C226	1.71 (17)
C28A—C21—C22—C227	143.89 (10)	O227—C227—C221—C222	2.72 (17)
C211—C21—C22—C13	143.74 (10)	C22—C227—C221—C222	−176.12 (11)
C28A—C21—C22—C13	20.27 (11)	C226—C221—C222—C223	−1.31 (19)
N24—C13—C22—C227	−116.45 (10)	C227—C221—C222—C223	176.60 (11)
C13A—C13—C22—C227	7.17 (15)	C221—C222—C223—C224	1.0 (2)
C12—C13—C22—C227	123.56 (10)	C222—C223—C224—C225	0.4 (2)
N24—C13—C22—C21	7.49 (11)	C223—C224—C225—C226	−1.5 (2)
C13A—C13—C22—C21	131.11 (11)	C224—C225—C226—C221	1.2 (2)
C12—C13—C22—C21	−112.51 (10)	C222—C221—C226—C225	0.20 (18)
C13A—C13—N24—C25	67.46 (13)	C227—C221—C226—C225	−177.61 (11)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N11—H11···O227^{i}	0.868 (18)	2.242 (17)	2.9756 (16)	142.2 (13)
O212—H212···O12^{ii}	0.899 (19)	1.873 (19)	2.7723 (14)	178.4 (15)
C22—H22···O211^{ii}	1.00	2.22	3.1930 (15)	164
C16—H16···*Cg*1^{iii}	0.95	2.63	3.4966 (14)	152

Symmetry codes: (i) *x*+1, *y*, *z*; (ii) −*x*+1, −*y*+1, −*z*+1; (iii) −*x*+2, −*y*+1, −*z*.

(1′*SR*,2′*SR*,3*RS*,8a′*RS*)-2′-Benzoyl-5-methyl-2-oxo-1′,5′,6′,7′,8′,8a′-hexahydro-2′*H*-spiro[indoline-3,3′-indolizine]-1′-carboxylic acid (II)

Crystal data

$C_{24}H_{24}N_2O_4$
M_r = 404.45
Triclinic, $P\overline{1}$
a = 8.1874 (6) Å
b = 8.5015 (6) Å
c = 15.5775 (12) Å
α = 85.775 (3)°
β = 77.641 (3)°

γ = 68.022 (2)°
V = 982.15 (13) Å^3
Z = 2
F(000) = 428
D_x = 1.368 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å

Cell parameters from 4495 reflections
θ = 2.7–27.5°
μ = 0.09 mm^{-1}
T = 100 K
Block, colourless
0.16 × 0.12 × 0.07 mm

Data collection

Bruker D8 Venture diffractometer
Radiation source: INCOATEC high brilliance microfocus sealed tube
Multilayer mirror monochromator
φ and ω scans
Absorption correction: multi-scan (SADABS; Bruker, 2016)
T_{min} = 0.934, T_{max} = 0.993

40953 measured reflections
4490 independent reflections
3667 reflections with $I > 2\sigma(I)$
R_{int} = 0.069
θ_{max} = 27.5°, θ_{min} = 2.7°
h = −10→10
k = −11→11
l = −20→20

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.040
$wR(F^2)$ = 0.098
S = 1.03
4490 reflections
278 parameters
0 restraints
Primary atom site location: dual

Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0397P)^2 + 0.5075P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.34 e Å^{-3}
$\Delta\rho_{min}$ = −0.21 e Å^{-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.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	y	z	U_{iso}*/U_{eq}
N11	1.09701 (16)	0.39151 (15)	0.26240 (8)	0.0156 (2)
H11	1.187 (2)	0.415 (2)	0.2728 (11)	0.019*
C12	0.96755 (17)	0.37128 (16)	0.32786 (9)	0.0132 (3)
O12	0.94935 (13)	0.39794 (12)	0.40602 (6)	0.0162 (2)
C13	0.85035 (17)	0.30208 (16)	0.28577 (9)	0.0120 (2)
C13A	0.93032 (17)	0.30520 (16)	0.18913 (9)	0.0131 (3)
C14	0.88832 (18)	0.25617 (16)	0.11664 (9)	0.0150 (3)
H14	0.7896	0.2200	0.1236	0.018*
C15	0.99176 (19)	0.25999 (17)	0.03299 (9)	0.0167 (3)
C16	1.13690 (19)	0.31229 (17)	0.02505 (9)	0.0177 (3)
H16	1.2060	0.3168	−0.0318	0.021*
C17	1.18385 (18)	0.35820 (17)	0.09780 (9)	0.0174 (3)
H17	1.2845	0.3913	0.0915	0.021*
C17A	1.07816 (18)	0.35368 (16)	0.17934 (9)	0.0146 (3)

C21	0.57635 (17)	0.27666 (16)	0.37763 (9)	0.0127 (3)
H21	0.5232	0.2258	0.3403	0.015*
C22	0.64524 (17)	0.40613 (16)	0.32191 (8)	0.0118 (2)
H22	0.6353	0.4976	0.3618	0.014*
N24	0.87015 (15)	0.12758 (13)	0.31379 (7)	0.0127 (2)
C25	1.05406 (18)	0.00874 (17)	0.31350 (10)	0.0172 (3)
H25A	1.1040	0.0437	0.3581	0.021*
H25B	1.1316	0.0081	0.2552	0.021*
C26	1.04917 (19)	−0.16755 (17)	0.33436 (10)	0.0200 (3)
H26A	1.1718	−0.2488	0.3367	0.024*
H26B	1.0076	−0.2050	0.2872	0.024*
C27	0.92344 (19)	−0.16700 (17)	0.42207 (10)	0.0182 (3)
H27A	0.9740	−0.1444	0.4700	0.022*
H27B	0.9147	−0.2802	0.4319	0.022*
C28	0.73517 (18)	−0.03248 (16)	0.42473 (9)	0.0150 (3)
H28A	0.6756	−0.0657	0.3840	0.018*
H28B	0.6619	−0.0252	0.4848	0.018*
C28A	0.74617 (17)	0.13950 (16)	0.39850 (8)	0.0126 (3)
H28C	0.7881	0.1817	0.4447	0.015*
C151	0.9470 (2)	0.20877 (19)	−0.04700 (9)	0.0221 (3)
H51A	0.9463	0.2942	−0.0929	0.033*
H51B	0.8282	0.2002	−0.0313	0.033*
H51C	1.0375	0.0986	−0.0688	0.033*
C211	0.43611 (17)	0.35525 (16)	0.45817 (9)	0.0139 (3)
O211	0.45181 (14)	0.31099 (12)	0.53233 (6)	0.0192 (2)
O212	0.29169 (13)	0.47851 (13)	0.43809 (7)	0.0209 (2)
H212	0.216 (3)	0.518 (2)	0.4883 (13)	0.031*
C227	0.54194 (17)	0.48792 (16)	0.24979 (9)	0.0131 (3)
O227	0.45460 (13)	0.42065 (12)	0.22238 (7)	0.0179 (2)
C221	0.54491 (17)	0.65538 (16)	0.21388 (9)	0.0142 (3)
C222	0.44259 (19)	0.73206 (18)	0.15024 (9)	0.0190 (3)
H222	0.3791	0.6747	0.1291	0.023*
C223	0.4330 (2)	0.89047 (19)	0.11780 (10)	0.0219 (3)
H223	0.3616	0.9423	0.0754	0.026*
C224	0.5281 (2)	0.97386 (18)	0.14737 (10)	0.0211 (3)
H224	0.5215	1.0826	0.1251	0.025*
C225	0.6322 (2)	0.89864 (18)	0.20929 (9)	0.0203 (3)
H225	0.6983	0.9552	0.2288	0.024*
C226	0.64013 (19)	0.73990 (17)	0.24298 (9)	0.0171 (3)
H226	0.7106	0.6891	0.2859	0.021*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N11	0.0125 (5)	0.0182 (6)	0.0185 (6)	−0.0087 (5)	−0.0031 (5)	0.0008 (4)
C12	0.0111 (6)	0.0095 (6)	0.0186 (7)	−0.0034 (5)	−0.0036 (5)	0.0015 (5)
O12	0.0149 (5)	0.0176 (5)	0.0170 (5)	−0.0064 (4)	−0.0040 (4)	−0.0012 (4)
C13	0.0107 (6)	0.0109 (6)	0.0147 (6)	−0.0044 (5)	−0.0026 (5)	0.0007 (5)

C13A	0.0118 (6)	0.0103 (6)	0.0158 (6)	−0.0034 (5)	−0.0012 (5)	0.0012 (5)
C14	0.0142 (6)	0.0130 (6)	0.0171 (7)	−0.0048 (5)	−0.0025 (5)	0.0013 (5)
C15	0.0189 (7)	0.0120 (6)	0.0168 (7)	−0.0032 (5)	−0.0035 (5)	0.0021 (5)
C16	0.0174 (7)	0.0144 (6)	0.0164 (7)	−0.0034 (5)	0.0015 (5)	0.0032 (5)
C17	0.0135 (6)	0.0150 (6)	0.0218 (7)	−0.0056 (5)	−0.0005 (5)	0.0038 (5)
C17A	0.0133 (6)	0.0121 (6)	0.0175 (7)	−0.0037 (5)	−0.0032 (5)	0.0021 (5)
C21	0.0120 (6)	0.0129 (6)	0.0140 (6)	−0.0058 (5)	−0.0021 (5)	0.0011 (5)
C22	0.0097 (6)	0.0117 (6)	0.0133 (6)	−0.0041 (5)	−0.0010 (5)	0.0000 (5)
N24	0.0115 (5)	0.0104 (5)	0.0147 (5)	−0.0036 (4)	−0.0008 (4)	0.0015 (4)
C25	0.0121 (6)	0.0149 (6)	0.0214 (7)	−0.0030 (5)	−0.0009 (5)	0.0020 (5)
C26	0.0173 (7)	0.0132 (6)	0.0242 (7)	−0.0016 (5)	−0.0008 (6)	0.0013 (5)
C27	0.0182 (7)	0.0135 (6)	0.0219 (7)	−0.0056 (5)	−0.0039 (6)	0.0042 (5)
C28	0.0153 (6)	0.0136 (6)	0.0165 (6)	−0.0066 (5)	−0.0023 (5)	0.0021 (5)
C28A	0.0119 (6)	0.0124 (6)	0.0131 (6)	−0.0045 (5)	−0.0014 (5)	0.0002 (5)
C151	0.0271 (8)	0.0229 (7)	0.0158 (7)	−0.0089 (6)	−0.0042 (6)	0.0008 (5)
C211	0.0119 (6)	0.0118 (6)	0.0194 (7)	−0.0070 (5)	−0.0011 (5)	0.0007 (5)
O211	0.0216 (5)	0.0169 (5)	0.0154 (5)	−0.0049 (4)	−0.0002 (4)	−0.0001 (4)
O212	0.0128 (5)	0.0217 (5)	0.0207 (5)	−0.0006 (4)	0.0011 (4)	0.0023 (4)
C227	0.0094 (6)	0.0138 (6)	0.0138 (6)	−0.0031 (5)	0.0006 (5)	−0.0014 (5)
O227	0.0166 (5)	0.0207 (5)	0.0202 (5)	−0.0100 (4)	−0.0065 (4)	0.0016 (4)
C221	0.0122 (6)	0.0144 (6)	0.0135 (6)	−0.0031 (5)	−0.0006 (5)	0.0010 (5)
C222	0.0167 (7)	0.0217 (7)	0.0188 (7)	−0.0072 (5)	−0.0049 (5)	0.0021 (5)
C223	0.0201 (7)	0.0222 (7)	0.0208 (7)	−0.0045 (6)	−0.0071 (6)	0.0067 (6)
C224	0.0233 (7)	0.0148 (6)	0.0215 (7)	−0.0055 (6)	−0.0016 (6)	0.0052 (5)
C225	0.0255 (7)	0.0181 (7)	0.0185 (7)	−0.0106 (6)	−0.0024 (6)	0.0009 (5)
C226	0.0196 (7)	0.0170 (7)	0.0153 (7)	−0.0067 (5)	−0.0053 (5)	0.0022 (5)

Geometric parameters (Å, º)

N11—C12	1.3568 (17)	C26—C27	1.524 (2)
N11—C17A	1.4090 (18)	C26—H26A	0.9900
N11—H11	0.880 (18)	C26—H26B	0.9900
C12—O12	1.2223 (16)	C27—C28	1.5316 (18)
C12—C13	1.5637 (17)	C27—H27A	0.9900
C13—N24	1.4740 (16)	C27—H27B	0.9900
C13—C13A	1.5102 (18)	C28—C28A	1.5191 (17)
C13—C22	1.5736 (17)	C28—H28A	0.9900
C13A—C14	1.3820 (19)	C28—H28B	0.9900
C13A—C17A	1.3941 (18)	C28A—H28C	1.0000
C14—C15	1.3993 (19)	C151—H51A	0.9800
C14—H14	0.9500	C151—H51B	0.9800
C15—C16	1.396 (2)	C151—H51C	0.9800
C15—C151	1.507 (2)	C211—O211	1.2066 (17)
C16—C17	1.396 (2)	C211—O212	1.3328 (16)
C16—H16	0.9500	O212—H212	0.89 (2)
C17—C17A	1.3822 (19)	C227—O227	1.2221 (16)
C17—H17	0.9500	C227—C221	1.4977 (18)
C21—C211	1.5071 (18)	C221—C226	1.3958 (19)

C21—C28A	1.5240 (17)	C221—C222	1.4004 (19)
C21—C22	1.5484 (17)	C222—C223	1.383 (2)
C21—H21	1.0000	C222—H222	0.9500
C22—C227	1.5207 (18)	C223—C224	1.391 (2)
C22—H22	1.0000	C223—H223	0.9500
N24—C25	1.4652 (17)	C224—C225	1.386 (2)
N24—C28A	1.4667 (17)	C224—H224	0.9500
C25—C26	1.5234 (19)	C225—C226	1.3949 (19)
C25—H25A	0.9900	C225—H225	0.9500
C25—H25B	0.9900	C226—H226	0.9500

C12—N11—C17A	111.83 (11)	C27—C26—H26A	109.5
C12—N11—H11	122.4 (11)	C25—C26—H26B	109.5
C17A—N11—H11	125.5 (11)	C27—C26—H26B	109.5
O12—C12—N11	125.93 (12)	H26A—C26—H26B	108.1
O12—C12—C13	126.24 (12)	C26—C27—C28	111.64 (11)
N11—C12—C13	107.79 (11)	C26—C27—H27A	109.3
N24—C13—C13A	110.45 (10)	C28—C27—H27A	109.3
N24—C13—C12	112.14 (10)	C26—C27—H27B	109.3
C13A—C13—C12	101.50 (10)	C28—C27—H27B	109.3
N24—C13—C22	103.04 (10)	H27A—C27—H27B	108.0
C13A—C13—C22	120.34 (11)	C28A—C28—C27	110.56 (11)
C12—C13—C22	109.64 (10)	C28A—C28—H28A	109.5
C14—C13A—C17A	120.18 (12)	C27—C28—H28A	109.5
C14—C13A—C13	130.47 (12)	C28A—C28—H28B	109.5
C17A—C13A—C13	109.09 (11)	C27—C28—H28B	109.5
C13A—C14—C15	119.72 (12)	H28A—C28—H28B	108.1
C13A—C14—H14	120.1	N24—C28A—C28	110.52 (10)
C15—C14—H14	120.1	N24—C28A—C21	100.15 (10)
C16—C15—C14	118.78 (13)	C28—C28A—C21	116.59 (11)
C16—C15—C151	120.68 (13)	N24—C28A—H28C	109.7
C14—C15—C151	120.54 (13)	C28—C28A—H28C	109.7
C17—C16—C15	122.22 (13)	C21—C28A—H28C	109.7
C17—C16—H16	118.9	C15—C151—H51A	109.5
C15—C16—H16	118.9	C15—C151—H51B	109.5
C17A—C17—C16	117.40 (13)	H51A—C151—H51B	109.5
C17A—C17—H17	121.3	C15—C151—H51C	109.5
C16—C17—H17	121.3	H51A—C151—H51C	109.5
C17—C17A—C13A	121.67 (13)	H51B—C151—H51C	109.5
C17—C17A—N11	128.78 (12)	O211—C211—O212	123.76 (13)
C13A—C17A—N11	109.55 (12)	O211—C211—C21	124.00 (12)
C211—C21—C28A	113.54 (11)	O212—C211—C21	112.24 (11)
C211—C21—C22	112.94 (10)	C211—O212—H212	107.0 (13)
C28A—C21—C22	104.05 (10)	O227—C227—C221	120.06 (12)
C211—C21—H21	108.7	O227—C227—C22	120.97 (12)
C28A—C21—H21	108.7	C221—C227—C22	118.95 (11)
C22—C21—H21	108.7	C226—C221—C222	118.97 (12)
C227—C22—C21	113.74 (10)	C226—C221—C227	122.95 (12)

C227—C22—C13	113.22 (10)	C222—C221—C227	118.05 (12)
C21—C22—C13	104.21 (10)	C223—C222—C221	120.67 (13)
C227—C22—H22	108.5	C223—C222—H222	119.7
C21—C22—H22	108.5	C221—C222—H222	119.7
C13—C22—H22	108.5	C222—C223—C224	119.92 (13)
C25—N24—C28A	113.82 (10)	C222—C223—H223	120.0
C25—N24—C13	116.16 (10)	C224—C223—H223	120.0
C28A—N24—C13	107.24 (10)	C225—C224—C223	120.16 (13)
N24—C25—C26	108.39 (11)	C225—C224—H224	119.9
N24—C25—H25A	110.0	C223—C224—H224	119.9
C26—C25—H25A	110.0	C224—C225—C226	120.00 (13)
N24—C25—H25B	110.0	C224—C225—H225	120.0
C26—C25—H25B	110.0	C226—C225—H225	120.0
H25A—C25—H25B	108.4	C225—C226—C221	120.27 (13)
C25—C26—C27	110.73 (11)	C225—C226—H226	119.9
C25—C26—H26A	109.5	C221—C226—H226	119.9

C17A—N11—C12—O12	−178.65 (12)	C12—C13—N24—C25	−44.53 (15)
C17A—N11—C12—C13	3.25 (14)	C22—C13—N24—C25	−162.34 (11)
O12—C12—C13—N24	−64.87 (16)	C13A—C13—N24—C28A	−163.53 (10)
N11—C12—C13—N24	113.22 (12)	C12—C13—N24—C28A	84.05 (12)
O12—C12—C13—C13A	177.24 (12)	C22—C13—N24—C28A	−33.76 (12)
N11—C12—C13—C13A	−4.66 (13)	C28A—N24—C25—C26	60.85 (14)
O12—C12—C13—C22	48.94 (17)	C13—N24—C25—C26	−173.85 (11)
N11—C12—C13—C22	−132.96 (11)	N24—C25—C26—C27	−57.02 (15)
N24—C13—C13A—C14	59.54 (18)	C25—C26—C27—C28	54.41 (16)
C12—C13—C13A—C14	178.63 (13)	C26—C27—C28—C28A	−51.88 (15)
C22—C13—C13A—C14	−60.28 (19)	C25—N24—C28A—C28	−59.53 (14)
N24—C13—C13A—C17A	−114.56 (12)	C13—N24—C28A—C28	170.55 (10)
C12—C13—C13A—C17A	4.53 (13)	C25—N24—C28A—C21	176.92 (10)
C22—C13—C13A—C17A	125.62 (12)	C13—N24—C28A—C21	47.01 (12)
C17A—C13A—C14—C15	−1.81 (19)	C27—C28—C28A—N24	52.98 (14)
C13—C13A—C14—C15	−175.36 (13)	C27—C28—C28A—C21	166.42 (11)
C13A—C14—C15—C16	0.56 (19)	C211—C21—C28A—N24	−163.56 (10)
C13A—C14—C15—C151	−179.36 (12)	C22—C21—C28A—N24	−40.38 (12)
C14—C15—C16—C17	1.0 (2)	C211—C21—C28A—C28	77.24 (14)
C151—C15—C16—C17	−179.03 (13)	C22—C21—C28A—C28	−159.58 (11)
C15—C16—C17—C17A	−1.3 (2)	C28A—C21—C211—O211	−2.64 (18)
C16—C17—C17A—C13A	0.05 (19)	C22—C21—C211—O211	−120.80 (14)
C16—C17—C17A—N11	179.21 (13)	C28A—C21—C211—O212	177.92 (11)
C14—C13A—C17A—C17	1.5 (2)	C22—C21—C211—O212	59.76 (14)
C13—C13A—C17A—C17	176.34 (12)	C21—C22—C227—O227	−21.03 (17)
C14—C13A—C17A—N11	−177.78 (11)	C13—C22—C227—O227	97.69 (14)
C13—C13A—C17A—N11	−2.97 (14)	C21—C22—C227—C221	157.53 (11)
C12—N11—C17A—C17	−179.51 (13)	C13—C22—C227—C221	−83.75 (14)
C12—N11—C17A—C13A	−0.26 (15)	O227—C227—C221—C226	179.25 (13)
C211—C21—C22—C227	−91.97 (13)	C22—C227—C221—C226	0.68 (19)
C28A—C21—C22—C227	144.45 (11)	O227—C227—C221—C222	1.31 (19)

C211—C21—C22—C13	144.27 (11)	C22—C227—C221—C222	−177.26 (12)
C28A—C21—C22—C13	20.69 (13)	C226—C221—C222—C223	−1.2 (2)
N24—C13—C22—C227	−117.31 (11)	C227—C221—C222—C223	176.82 (13)
C13A—C13—C22—C227	6.13 (16)	C221—C222—C223—C224	1.1 (2)
C12—C13—C22—C227	123.13 (11)	C222—C223—C224—C225	0.0 (2)
N24—C13—C22—C21	6.78 (12)	C223—C224—C225—C226	−0.8 (2)
C13A—C13—C22—C21	130.22 (12)	C224—C225—C226—C221	0.7 (2)
C12—C13—C22—C21	−112.78 (11)	C222—C221—C226—C225	0.3 (2)
C13A—C13—N24—C25	67.89 (14)	C227—C221—C226—C225	−177.60 (13)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N11—H11···O227^{i}	0.880 (18)	2.181 (18)	2.9598 (18)	147.3 (15)
O212—H212···O12^{ii}	0.89 (2)	1.88 (2)	2.7691 (15)	179 (2)
C22—H22···O211^{ii}	1.00	2.21	3.1894 (16)	165
C16—H16···*Cg*1^{iii}	0.95	2.75	3.6366 (16)	155

Symmetry codes: (i) *x*+1, *y*, *z*; (ii) −*x*+1, −*y*+1, −*z*+1; (iii) −*x*+2, −*y*+1, −*z*.

(1′*SR*,2′*SR*,3*RS*,8a′*RS*)-2′-Benzoyl-1-methyl-2-oxo-1′,5′,6′,7′,8′,8a′-hexahydro-2′*H*-spiro[indoline-3,3′-indolizine]-1′-carboxylic acid (III)

Crystal data

$C_{24}H_{24}N_2O_4$
M_r = 404.45
Triclinic, $P\overline{1}$
a = 8.6535 (4) Å
b = 9.2064 (4) Å
c = 14.4327 (6) Å
α = 72.660 (1)°
β = 74.539 (1)°
γ = 65.930 (2)°
V = 988.16 (8) Å^3

Z = 2
F(000) = 428
D_x = 1.359 Mg m^{-3}
Mo *Kα* radiation, *λ* = 0.71073 Å
Cell parameters from 4917 reflections
θ = 2.5–28.3°
μ = 0.09 mm^{-1}
T = 100 K
Block, colourless
0.19 × 0.19 × 0.12 mm

Data collection

Bruker D8 Venture diffractometer
Radiation source: INCOATEC high brilliance microfocus sealed tube
Multilayer mirror monochromator
φ and *ω* scans
Absorption correction: multi-scan (SADABS; Bruker, 2016)
T_{min} = 0.944, T_{max} = 0.989

47689 measured reflections
4917 independent reflections
4145 reflections with $I > 2\sigma(I)$
R_{int} = 0.057
θ_{max} = 28.3°, θ_{min} = 2.5°
h = −11→11
k = −12→12
l = −19→19

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.039
$wR(F^2)$ = 0.098
S = 1.03

4917 reflections
275 parameters
0 restraints
Primary atom site location: dual
Hydrogen site location: mixed

H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0359P)^2 + 0.5593P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.30$ e Å^{-3}
$\Delta\rho_{min} = -0.33$ e Å^{-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.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
N11	−0.14571 (13)	0.47390 (13)	0.23697 (8)	0.0172 (2)
C12	−0.07277 (16)	0.47854 (15)	0.30841 (9)	0.0164 (2)
O12	−0.07700 (12)	0.39560 (11)	0.39176 (6)	0.0216 (2)
C13	0.01728 (15)	0.60589 (14)	0.26485 (8)	0.0145 (2)
C13A	−0.00831 (15)	0.65569 (14)	0.15853 (8)	0.0154 (2)
C14	0.04073 (17)	0.76671 (15)	0.08045 (9)	0.0191 (2)
H14	0.1111	0.8184	0.0872	0.023*
C15	−0.01538 (18)	0.80155 (16)	−0.00889 (9)	0.0220 (3)
H15	0.0201	0.8750	−0.0640	0.026*
C16	−0.12246 (18)	0.72949 (16)	−0.01737 (9)	0.0223 (3)
H16	−0.1613	0.7565	−0.0781	0.027*
C17	−0.17467 (17)	0.61819 (16)	0.06124 (9)	0.0202 (3)
H17	−0.2492	0.5699	0.0556	0.024*
C17A	−0.11268 (15)	0.58157 (15)	0.14768 (9)	0.0162 (2)
C21	0.21876 (15)	0.65461 (14)	0.33316 (8)	0.0150 (2)
H21	0.2492	0.7401	0.2785	0.018*
C22	0.20367 (15)	0.52841 (14)	0.29027 (8)	0.0143 (2)
H22	0.2110	0.4281	0.3434	0.017*
N24	−0.06205 (13)	0.75017 (12)	0.30930 (7)	0.0150 (2)
C25	−0.24787 (15)	0.80117 (15)	0.34287 (9)	0.0182 (2)
H25A	−0.2748	0.7184	0.3998	0.022*
H25B	−0.3061	0.8119	0.2894	0.022*
C26	−0.31119 (16)	0.96420 (16)	0.37253 (10)	0.0209 (3)
H26A	−0.4351	0.9954	0.4002	0.025*
H26B	−0.2962	1.0493	0.3136	0.025*
C27	−0.21203 (17)	0.95411 (17)	0.44903 (10)	0.0227 (3)
H27A	−0.2403	0.8806	0.5113	0.027*
H27B	−0.2475	1.0635	0.4625	0.027*
C28	−0.01792 (16)	0.89056 (15)	0.41296 (10)	0.0195 (3)
H28B	0.0130	0.9698	0.3550	0.023*
H28C	0.0438	0.8767	0.4655	0.023*
C28A	0.03369 (15)	0.72830 (14)	0.38539 (8)	0.0148 (2)
H28A	0.0075	0.6468	0.4448	0.018*
C111	−0.22772 (18)	0.35784 (17)	0.25008 (10)	0.0235 (3)
H1A	−0.2570	0.3144	0.3205	0.035*

H1B	−0.1490	0.2684	0.2179	0.035*
H1C	−0.3325	0.4124	0.2207	0.035*
C211	0.34802 (15)	0.58198 (15)	0.40254 (9)	0.0153 (2)
O211	0.41199 (11)	0.43600 (11)	0.43615 (6)	0.01863 (19)
O212	0.38354 (12)	0.69396 (11)	0.42394 (7)	0.0206 (2)
H212	0.450 (2)	0.646 (2)	0.4728 (13)	0.031*
C227	0.34105 (15)	0.47991 (15)	0.20275 (9)	0.0163 (2)
O227	0.41306 (12)	0.57337 (11)	0.15069 (7)	0.0230 (2)
C221	0.38702 (16)	0.31597 (15)	0.18205 (9)	0.0175 (2)
C222	0.50576 (17)	0.27607 (17)	0.09776 (10)	0.0219 (3)
H222	0.5523	0.3538	0.0541	0.026*
C223	0.55561 (18)	0.12322 (18)	0.07792 (11)	0.0269 (3)
H223	0.6352	0.0973	0.0202	0.032*
C224	0.48988 (18)	0.00793 (17)	0.14186 (11)	0.0258 (3)
H224	0.5255	−0.0969	0.1284	0.031*
C225	0.37201 (18)	0.04642 (16)	0.22556 (10)	0.0232 (3)
H225	0.3269	−0.0323	0.2693	0.028*
C226	0.31981 (17)	0.19981 (15)	0.24544 (9)	0.0193 (2)
H226	0.2381	0.2259	0.3024	0.023*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N11	0.0174 (5)	0.0186 (5)	0.0179 (5)	−0.0074 (4)	−0.0045 (4)	−0.0044 (4)
C12	0.0154 (5)	0.0161 (5)	0.0174 (5)	−0.0040 (4)	−0.0031 (4)	−0.0051 (4)
O12	0.0257 (5)	0.0239 (5)	0.0164 (4)	−0.0122 (4)	−0.0047 (4)	0.0000 (3)
C13	0.0148 (5)	0.0150 (5)	0.0139 (5)	−0.0045 (4)	−0.0037 (4)	−0.0034 (4)
C13A	0.0153 (5)	0.0154 (5)	0.0145 (5)	−0.0022 (4)	−0.0043 (4)	−0.0045 (4)
C14	0.0211 (6)	0.0167 (6)	0.0181 (6)	−0.0050 (5)	−0.0040 (5)	−0.0035 (4)
C15	0.0263 (7)	0.0172 (6)	0.0158 (6)	−0.0016 (5)	−0.0044 (5)	−0.0020 (5)
C16	0.0249 (7)	0.0205 (6)	0.0170 (6)	0.0013 (5)	−0.0087 (5)	−0.0063 (5)
C17	0.0190 (6)	0.0206 (6)	0.0205 (6)	−0.0015 (5)	−0.0074 (5)	−0.0081 (5)
C17A	0.0155 (5)	0.0153 (5)	0.0165 (5)	−0.0020 (4)	−0.0036 (4)	−0.0058 (4)
C21	0.0151 (5)	0.0149 (5)	0.0153 (5)	−0.0040 (4)	−0.0047 (4)	−0.0035 (4)
C22	0.0144 (5)	0.0145 (5)	0.0139 (5)	−0.0035 (4)	−0.0050 (4)	−0.0029 (4)
N24	0.0135 (5)	0.0164 (5)	0.0158 (5)	−0.0032 (4)	−0.0046 (4)	−0.0056 (4)
C25	0.0135 (6)	0.0208 (6)	0.0202 (6)	−0.0039 (5)	−0.0042 (4)	−0.0064 (5)
C26	0.0150 (6)	0.0221 (6)	0.0240 (6)	−0.0006 (5)	−0.0057 (5)	−0.0093 (5)
C27	0.0192 (6)	0.0250 (6)	0.0237 (6)	−0.0014 (5)	−0.0056 (5)	−0.0124 (5)
C28	0.0182 (6)	0.0190 (6)	0.0229 (6)	−0.0027 (5)	−0.0070 (5)	−0.0092 (5)
C28A	0.0140 (5)	0.0164 (5)	0.0144 (5)	−0.0038 (4)	−0.0051 (4)	−0.0038 (4)
C111	0.0234 (7)	0.0278 (7)	0.0259 (6)	−0.0146 (6)	−0.0053 (5)	−0.0062 (5)
C211	0.0132 (5)	0.0182 (6)	0.0154 (5)	−0.0057 (4)	−0.0016 (4)	−0.0054 (4)
O211	0.0188 (4)	0.0163 (4)	0.0213 (4)	−0.0037 (3)	−0.0079 (3)	−0.0045 (3)
O212	0.0221 (5)	0.0181 (4)	0.0261 (5)	−0.0074 (4)	−0.0126 (4)	−0.0031 (4)
C227	0.0143 (5)	0.0173 (6)	0.0157 (5)	−0.0028 (4)	−0.0054 (4)	−0.0027 (4)
O227	0.0228 (5)	0.0225 (5)	0.0214 (5)	−0.0090 (4)	0.0007 (4)	−0.0039 (4)
C221	0.0157 (6)	0.0175 (6)	0.0175 (6)	−0.0010 (5)	−0.0067 (4)	−0.0050 (4)

C222	0.0186 (6)	0.0245 (6)	0.0204 (6)	−0.0045 (5)	−0.0029 (5)	−0.0068 (5)
C223	0.0217 (7)	0.0301 (7)	0.0259 (7)	−0.0011 (6)	−0.0027 (5)	−0.0148 (6)
C224	0.0265 (7)	0.0197 (6)	0.0305 (7)	0.0015 (5)	−0.0124 (6)	−0.0117 (5)
C225	0.0273 (7)	0.0172 (6)	0.0252 (6)	−0.0050 (5)	−0.0110 (5)	−0.0032 (5)
C226	0.0202 (6)	0.0192 (6)	0.0172 (6)	−0.0032 (5)	−0.0062 (5)	−0.0048 (5)

Geometric parameters (Å, º)

N11—C12	1.3633 (15)	C26—C27	1.5307 (17)
N11—C17A	1.4136 (16)	C26—H26A	0.9900
N11—C111	1.4517 (16)	C26—H26B	0.9900
C12—O12	1.2179 (15)	C27—C28	1.5325 (18)
C12—C13	1.5562 (17)	C27—H27A	0.9900
C13—N24	1.4754 (15)	C27—H27B	0.9900
C13—C13A	1.5142 (16)	C28—C28A	1.5181 (16)
C13—C22	1.5703 (16)	C28—H28B	0.9900
C13A—C14	1.3809 (17)	C28—H28C	0.9900
C13A—C17A	1.3955 (17)	C28A—H28A	1.0000
C14—C15	1.4005 (17)	C111—H1A	0.9800
C14—H14	0.9500	C111—H1B	0.9800
C15—C16	1.387 (2)	C111—H1C	0.9800
C15—H15	0.9500	C211—O211	1.2230 (15)
C16—C17	1.3974 (19)	C211—O212	1.3235 (15)
C16—H16	0.9500	O212—H212	0.918 (19)
C17—C17A	1.3844 (17)	C227—O227	1.2229 (15)
C17—H17	0.9500	C227—C221	1.4970 (17)
C21—C211	1.5129 (16)	C221—C226	1.3997 (18)
C21—C22	1.5316 (16)	C221—C222	1.4009 (17)
C21—C28A	1.5389 (16)	C222—C223	1.3883 (19)
C21—H21	1.0000	C222—H222	0.9500
C22—C227	1.5202 (16)	C223—C224	1.389 (2)
C22—H22	1.0000	C223—H223	0.9500
N24—C25	1.4646 (15)	C224—C225	1.389 (2)
N24—C28A	1.4663 (14)	C224—H224	0.9500
C25—C26	1.5243 (17)	C225—C226	1.3902 (18)
C25—H25A	0.9900	C225—H225	0.9500
C25—H25B	0.9900	C226—H226	0.9500
C12—N11—C17A	111.16 (10)	C27—C26—H26A	109.5
C12—N11—C111	122.90 (11)	C25—C26—H26B	109.5
C17A—N11—C111	125.54 (10)	C27—C26—H26B	109.5
O12—C12—N11	125.34 (12)	H26A—C26—H26B	108.1
O12—C12—C13	126.26 (11)	C26—C27—C28	111.24 (10)
N11—C12—C13	108.40 (10)	C26—C27—H27A	109.4
N24—C13—C13A	109.58 (9)	C28—C27—H27A	109.4
N24—C13—C12	114.07 (10)	C26—C27—H27B	109.4
C13A—C13—C12	101.40 (9)	C28—C27—H27B	109.4
N24—C13—C22	103.18 (9)	H27A—C27—H27B	108.0

C13A—C13—C22	119.93 (10)	C28A—C28—C27	109.13 (10)
C12—C13—C22	109.16 (9)	C28A—C28—H28B	109.9
C14—C13A—C17A	120.01 (11)	C27—C28—H28B	109.9
C14—C13A—C13	130.66 (11)	C28A—C28—H28C	109.9
C17A—C13A—C13	109.04 (10)	C27—C28—H28C	109.9
C13A—C14—C15	118.65 (12)	H28B—C28—H28C	108.3
C13A—C14—H14	120.7	N24—C28A—C28	109.13 (9)
C15—C14—H14	120.7	N24—C28A—C21	100.22 (9)
C16—C15—C14	120.39 (12)	C28—C28A—C21	117.69 (10)
C16—C15—H15	119.8	N24—C28A—H28A	109.8
C14—C15—H15	119.8	C28—C28A—H28A	109.8
C15—C16—C17	121.63 (12)	C21—C28A—H28A	109.8
C15—C16—H16	119.2	N11—C111—H1A	109.5
C17—C16—H16	119.2	N11—C111—H1B	109.5
C17A—C17—C16	116.84 (12)	H1A—C111—H1B	109.5
C17A—C17—H17	121.6	N11—C111—H1C	109.5
C16—C17—H17	121.6	H1A—C111—H1C	109.5
C17—C17A—C13A	122.42 (12)	H1B—C111—H1C	109.5
C17—C17A—N11	127.90 (12)	O211—C211—O212	123.38 (11)
C13A—C17A—N11	109.65 (10)	O211—C211—C21	123.98 (11)
C211—C21—C22	113.57 (10)	O212—C211—C21	112.63 (10)
C211—C21—C28A	112.59 (9)	C211—O212—H212	109.6 (11)
C22—C21—C28A	101.53 (9)	O227—C227—C221	120.83 (11)
C211—C21—H21	109.6	O227—C227—C22	120.29 (11)
C22—C21—H21	109.6	C221—C227—C22	118.88 (10)
C28A—C21—H21	109.6	C226—C221—C222	119.04 (12)
C227—C22—C21	114.71 (10)	C226—C221—C227	122.34 (11)
C227—C22—C13	112.82 (9)	C222—C221—C227	118.59 (12)
C21—C22—C13	104.68 (9)	C223—C222—C221	120.17 (13)
C227—C22—H22	108.1	C223—C222—H222	119.9
C21—C22—H22	108.1	C221—C222—H222	119.9
C13—C22—H22	108.1	C222—C223—C224	120.42 (13)
C25—N24—C28A	113.12 (9)	C222—C223—H223	119.8
C25—N24—C13	116.19 (9)	C224—C223—H223	119.8
C28A—N24—C13	109.17 (9)	C225—C224—C223	119.82 (13)
N24—C25—C26	108.98 (10)	C225—C224—H224	120.1
N24—C25—H25A	109.9	C223—C224—H224	120.1
C26—C25—H25A	109.9	C224—C225—C226	120.16 (13)
N24—C25—H25B	109.9	C224—C225—H225	119.9
C26—C25—H25B	109.9	C226—C225—H225	119.9
H25A—C25—H25B	108.3	C225—C226—C221	120.38 (12)
C25—C26—C27	110.78 (10)	C225—C226—H226	119.8
C25—C26—H26A	109.5	C221—C226—H226	119.8

C17A—N11—C12—O12	−178.10 (12)	C13A—C13—N24—C25	81.00 (12)
C111—N11—C12—O12	−4.9 (2)	C12—C13—N24—C25	−31.88 (14)
C17A—N11—C12—C13	1.37 (13)	C22—C13—N24—C25	−150.16 (10)
C111—N11—C12—C13	174.54 (11)	C13A—C13—N24—C28A	−149.64 (10)

O12—C12—C13—N24	−67.14 (16)	C12—C13—N24—C28A	97.48 (11)
N11—C12—C13—N24	113.38 (11)	C22—C13—N24—C28A	−20.80 (12)
O12—C12—C13—C13A	175.18 (12)	C28A—N24—C25—C26	60.70 (13)
N11—C12—C13—C13A	−4.30 (12)	C13—N24—C25—C26	−171.86 (10)
O12—C12—C13—C22	47.67 (16)	N24—C25—C26—C27	−54.77 (14)
N11—C12—C13—C22	−131.81 (10)	C25—C26—C27—C28	53.79 (15)
N24—C13—C13A—C14	58.53 (16)	C26—C27—C28—C28A	−55.03 (14)
C12—C13—C13A—C14	179.42 (12)	C25—N24—C28A—C28	−63.03 (13)
C22—C13—C13A—C14	−60.41 (17)	C13—N24—C28A—C28	165.94 (10)
N24—C13—C13A—C17A	−115.11 (11)	C25—N24—C28A—C21	172.75 (9)
C12—C13—C13A—C17A	5.78 (12)	C13—N24—C28A—C21	41.72 (11)
C22—C13—C13A—C17A	125.94 (11)	C27—C28—C28A—N24	58.10 (13)
C17A—C13A—C14—C15	−0.44 (18)	C27—C28—C28A—C21	171.32 (10)
C13—C13A—C14—C15	−173.50 (12)	C211—C21—C28A—N24	−166.70 (9)
C13A—C14—C15—C16	2.01 (19)	C22—C21—C28A—N24	−44.91 (10)
C14—C15—C16—C17	−1.4 (2)	C211—C21—C28A—C28	75.21 (13)
C15—C16—C17—C17A	−0.81 (19)	C22—C21—C28A—C28	−163.00 (10)
C16—C17—C17A—C13A	2.43 (18)	C22—C21—C211—O211	−12.78 (17)
C16—C17—C17A—N11	−179.96 (12)	C28A—C21—C211—O211	101.91 (13)
C14—C13A—C17A—C17	−1.85 (19)	C22—C21—C211—O212	167.84 (10)
C13—C13A—C17A—C17	172.59 (11)	C28A—C21—C211—O212	−77.47 (13)
C14—C13A—C17A—N11	−179.84 (11)	C21—C22—C227—O227	−25.39 (16)
C13—C13A—C17A—N11	−5.40 (13)	C13—C22—C227—O227	94.37 (13)
C12—N11—C17A—C17	−175.33 (12)	C21—C22—C227—C221	154.21 (10)
C111—N11—C17A—C17	11.7 (2)	C13—C22—C227—C221	−86.03 (13)
C12—N11—C17A—C13A	2.52 (14)	O227—C227—C221—C226	173.53 (12)
C111—N11—C17A—C13A	−170.43 (12)	C22—C227—C221—C226	−6.07 (17)
C211—C21—C22—C227	−81.62 (12)	O227—C227—C221—C222	−4.40 (18)
C28A—C21—C22—C227	157.27 (9)	C22—C227—C221—C222	176.00 (11)
C211—C21—C22—C13	154.19 (10)	C226—C221—C222—C223	−0.08 (19)
C28A—C21—C22—C13	33.08 (11)	C227—C221—C222—C223	177.92 (12)
N24—C13—C22—C227	−134.20 (10)	C221—C222—C223—C224	−0.7 (2)
C13A—C13—C22—C227	−12.06 (15)	C222—C223—C224—C225	0.8 (2)
C12—C13—C22—C227	104.14 (11)	C223—C224—C225—C226	0.0 (2)
N24—C13—C22—C21	−8.81 (11)	C224—C225—C226—C221	−0.79 (19)
C13A—C13—C22—C21	113.33 (11)	C222—C221—C226—C225	0.83 (18)
C12—C13—C22—C21	−130.47 (10)	C227—C221—C226—C225	−177.09 (11)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O212—H212···O211^{i}	0.920 (19)	1.798 (19)	2.7162 (14)	175.5 (16)
C16—H16···*Cg*1^{ii}	0.95	2.70	3.5181 (17)	144

Symmetry codes: (i) −*x*+1, −*y*+1, −*z*+1; (ii) −*x*, −*y*+1, −*z*.

(1′*SR*,2′*SR*,3*RS*,8a′*RS*)-2′-Benzoyl-5-chloro-1-methyl-2-oxo-1′,5′,6′,7′,8′,8a′-hexahydro-2′*H*-spiro[indoline-3,3′-indolizine]-1′-carboxylic acid (IV)

Crystal data

$C_{24}H_{23}ClN_2O_4$
$M_r = 438.89$
Triclinic, $P\overline{1}$
$a = 8.7914$ (9) Å
$b = 9.3155$ (10) Å
$c = 14.6188$ (15) Å
$\alpha = 73.437$ (4)°
$\beta = 76.259$ (4)°
$\gamma = 64.156$ (3)°
$V = 1023.84$ (19) Å^3
$Z = 2$
$F(000) = 460$
$D_x = 1.424$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 5097 reflections
$\theta = 2.5$–28.3°
$\mu = 0.22$ mm^{-1}
$T = 100$ K
Plate, colourless
0.41 × 0.32 × 0.14 mm

Data collection

Bruker D8 Venture diffractometer
Radiation source: INCOATEC high brilliance microfocus sealed tube
Multilayer mirror monochromator
φ and ω scans
Absorption correction: multi-scan (SADABS; Bruker, 2016)
$T_{min} = 0.934$, $T_{max} = 0.969$
41790 measured reflections
5097 independent reflections
4493 reflections with $I > 2\sigma(I)$
$R_{int} = 0.055$
$\theta_{max} = 28.3°$, $\theta_{min} = 2.5°$
$h = -11\rightarrow11$
$k = -12\rightarrow12$
$l = -19\rightarrow19$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.033$
$wR(F^2) = 0.081$
$S = 1.03$
5097 reflections
284 parameters
0 restraints
Primary atom site location: dual
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0282P)^2 + 0.5813P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.33$ e Å^{-3}
$\Delta\rho_{min} = -0.31$ e Å^{-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.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
N11	−0.14842 (12)	0.46216 (12)	0.25503 (7)	0.01402 (19)
C12	−0.06065 (14)	0.46906 (14)	0.31797 (8)	0.0140 (2)
O12	−0.05142 (11)	0.39188 (11)	0.40072 (6)	0.01929 (18)
C13	0.02210 (14)	0.59386 (14)	0.26669 (8)	0.0126 (2)
C13A	−0.01677 (14)	0.63206 (14)	0.16485 (8)	0.0126 (2)
C14	0.02219 (14)	0.73536 (14)	0.08315 (8)	0.0141 (2)
H14	0.0952	0.7866	0.0827	0.017*

C15	−0.04979 (15)	0.76167 (14)	0.00131 (8)	0.0145 (2)
Cl15	−0.00313 (4)	0.89173 (4)	−0.10324 (2)	0.02027 (8)
C16	−0.15833 (14)	0.69000 (14)	0.00037 (8)	0.0151 (2)
H16	−0.2052	0.7113	−0.0567	0.018*
C17	−0.19881 (14)	0.58679 (14)	0.08303 (8)	0.0149 (2)
H17	−0.2739	0.5376	0.0840	0.018*
C17A	−0.12529 (14)	0.55880 (14)	0.16370 (8)	0.0127 (2)
C21	0.22086 (14)	0.65003 (14)	0.33088 (8)	0.0133 (2)
H21	0.2476	0.7343	0.2779	0.016*
C22	0.21110 (14)	0.52179 (14)	0.28854 (8)	0.0127 (2)
H22	0.2297	0.4202	0.3396	0.015*
N24	−0.05893 (12)	0.74457 (12)	0.30519 (7)	0.01319 (19)
C25	−0.24257 (14)	0.79743 (15)	0.33804 (9)	0.0167 (2)
H25A	−0.2640	0.7157	0.3940	0.020*
H25B	−0.3028	0.8073	0.2859	0.020*
C26	−0.30912 (15)	0.96175 (15)	0.36651 (9)	0.0197 (2)
H26A	−0.4314	0.9944	0.3927	0.024*
H26B	−0.2984	1.0455	0.3088	0.024*
C27	−0.20907 (16)	0.95248 (16)	0.44219 (9)	0.0201 (2)
H27A	−0.2314	0.8790	0.5028	0.024*
H27B	−0.2478	1.0622	0.4558	0.024*
C28	−0.01738 (15)	0.88889 (15)	0.40660 (9)	0.0175 (2)
H28B	0.0071	0.9674	0.3496	0.021*
H28C	0.0465	0.8764	0.4577	0.021*
C28A	0.03774 (14)	0.72514 (14)	0.38034 (8)	0.0138 (2)
H28A	0.0166	0.6451	0.4386	0.017*
C111	−0.24901 (16)	0.36509 (16)	0.27979 (9)	0.0209 (3)
H1A	−0.2506	0.3143	0.3485	0.031*
H1B	−0.1988	0.2801	0.2418	0.031*
H1C	−0.3658	0.4351	0.2659	0.031*
C211	0.34812 (14)	0.57889 (14)	0.40220 (8)	0.0140 (2)
O211	0.40115 (11)	0.43658 (10)	0.44479 (6)	0.01692 (17)
O212	0.39375 (12)	0.68921 (11)	0.41496 (7)	0.02076 (19)
H212	0.459 (2)	0.644 (2)	0.4597 (13)	0.031*
C227	0.34164 (14)	0.47834 (14)	0.20089 (8)	0.0141 (2)
O227	0.40243 (11)	0.57494 (11)	0.15070 (6)	0.02011 (19)
C221	0.39422 (14)	0.31536 (15)	0.17815 (8)	0.0155 (2)
C222	0.51533 (15)	0.27592 (16)	0.09744 (9)	0.0201 (2)
H222	0.5603	0.3528	0.0584	0.024*
C223	0.56952 (16)	0.12514 (17)	0.07448 (10)	0.0250 (3)
H223	0.6519	0.0989	0.0199	0.030*
C224	0.50392 (17)	0.01235 (16)	0.13093 (10)	0.0252 (3)
H224	0.5413	−0.0907	0.1148	0.030*
C225	0.38392 (17)	0.04960 (15)	0.21088 (10)	0.0220 (3)
H225	0.3391	−0.0278	0.2493	0.026*
C226	0.32927 (15)	0.20059 (15)	0.23469 (9)	0.0175 (2)
H226	0.2475	0.2257	0.2896	0.021*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N11	0.0148 (4)	0.0167 (5)	0.0131 (5)	−0.0087 (4)	−0.0029 (4)	−0.0019 (4)
C12	0.0130 (5)	0.0150 (5)	0.0143 (5)	−0.0050 (4)	−0.0023 (4)	−0.0040 (4)
O12	0.0248 (4)	0.0215 (4)	0.0132 (4)	−0.0121 (4)	−0.0051 (3)	0.0008 (3)
C13	0.0133 (5)	0.0139 (5)	0.0116 (5)	−0.0054 (4)	−0.0036 (4)	−0.0023 (4)
C13A	0.0124 (5)	0.0135 (5)	0.0123 (5)	−0.0037 (4)	−0.0036 (4)	−0.0040 (4)
C14	0.0143 (5)	0.0146 (5)	0.0148 (5)	−0.0062 (4)	−0.0035 (4)	−0.0028 (4)
C15	0.0162 (5)	0.0136 (5)	0.0115 (5)	−0.0036 (4)	−0.0024 (4)	−0.0024 (4)
Cl15	0.02631 (15)	0.02091 (15)	0.01277 (13)	−0.01030 (12)	−0.00413 (10)	0.00057 (11)
C16	0.0144 (5)	0.0164 (5)	0.0137 (5)	−0.0020 (4)	−0.0052 (4)	−0.0057 (4)
C17	0.0128 (5)	0.0170 (5)	0.0167 (5)	−0.0049 (4)	−0.0037 (4)	−0.0065 (4)
C17A	0.0117 (5)	0.0131 (5)	0.0125 (5)	−0.0036 (4)	−0.0017 (4)	−0.0038 (4)
C21	0.0143 (5)	0.0143 (5)	0.0131 (5)	−0.0055 (4)	−0.0047 (4)	−0.0033 (4)
C22	0.0127 (5)	0.0143 (5)	0.0123 (5)	−0.0050 (4)	−0.0044 (4)	−0.0028 (4)
N24	0.0125 (4)	0.0146 (5)	0.0135 (4)	−0.0041 (4)	−0.0046 (3)	−0.0043 (4)
C25	0.0129 (5)	0.0187 (6)	0.0182 (6)	−0.0046 (4)	−0.0037 (4)	−0.0048 (5)
C26	0.0163 (5)	0.0191 (6)	0.0215 (6)	−0.0024 (5)	−0.0051 (5)	−0.0062 (5)
C27	0.0206 (6)	0.0187 (6)	0.0195 (6)	−0.0031 (5)	−0.0041 (5)	−0.0081 (5)
C28	0.0186 (6)	0.0159 (5)	0.0188 (6)	−0.0043 (4)	−0.0061 (4)	−0.0060 (5)
C28A	0.0147 (5)	0.0149 (5)	0.0127 (5)	−0.0050 (4)	−0.0048 (4)	−0.0033 (4)
C111	0.0215 (6)	0.0266 (6)	0.0208 (6)	−0.0162 (5)	−0.0010 (5)	−0.0046 (5)
C211	0.0120 (5)	0.0179 (5)	0.0139 (5)	−0.0061 (4)	−0.0015 (4)	−0.0057 (4)
O211	0.0172 (4)	0.0170 (4)	0.0178 (4)	−0.0058 (3)	−0.0074 (3)	−0.0030 (3)
O212	0.0249 (5)	0.0192 (4)	0.0244 (5)	−0.0104 (4)	−0.0149 (4)	−0.0013 (4)
C227	0.0115 (5)	0.0178 (5)	0.0139 (5)	−0.0046 (4)	−0.0059 (4)	−0.0032 (4)
O227	0.0203 (4)	0.0233 (5)	0.0191 (4)	−0.0117 (4)	−0.0004 (3)	−0.0045 (4)
C221	0.0125 (5)	0.0181 (6)	0.0158 (5)	−0.0027 (4)	−0.0063 (4)	−0.0052 (4)
C222	0.0152 (5)	0.0249 (6)	0.0203 (6)	−0.0054 (5)	−0.0031 (4)	−0.0085 (5)
C223	0.0174 (6)	0.0294 (7)	0.0256 (7)	−0.0011 (5)	−0.0033 (5)	−0.0149 (6)
C224	0.0244 (6)	0.0186 (6)	0.0304 (7)	0.0020 (5)	−0.0124 (5)	−0.0116 (5)
C225	0.0253 (6)	0.0158 (6)	0.0244 (6)	−0.0046 (5)	−0.0114 (5)	−0.0026 (5)
C226	0.0182 (5)	0.0173 (6)	0.0164 (6)	−0.0038 (4)	−0.0065 (4)	−0.0042 (4)

Geometric parameters (Å, º)

N11—C12	1.3639 (14)	C26—C27	1.5319 (17)
N11—C17A	1.4070 (15)	C26—H26A	0.9900
N11—C111	1.4454 (15)	C26—H26B	0.9900
C12—O12	1.2194 (14)	C27—C28	1.5331 (17)
C12—C13	1.5570 (16)	C27—H27A	0.9900
C13—N24	1.4776 (14)	C27—H27B	0.9900
C13—C13A	1.5145 (15)	C28—C28A	1.5199 (16)
C13—C22	1.5719 (15)	C28—H28B	0.9900
C13A—C14	1.3811 (16)	C28—H28C	0.9900
C13A—C17A	1.3993 (15)	C28A—H28A	1.0000
C14—C15	1.3962 (16)	C111—H1A	0.9800

C14—H14	0.9500	C111—H1B	0.9800
C15—C16	1.3868 (17)	C111—H1C	0.9800
C15—Cl15	1.7469 (12)	C211—O211	1.2210 (15)
C16—C17	1.3939 (17)	C211—O212	1.3253 (14)
C16—H16	0.9500	O212—H212	0.861 (19)
C17—C17A	1.3844 (15)	C227—O227	1.2208 (15)
C17—H17	0.9500	C227—C221	1.4941 (16)
C21—C211	1.5138 (15)	C221—C226	1.3987 (17)
C21—C22	1.5342 (15)	C221—C222	1.4036 (17)
C21—C28A	1.5354 (15)	C222—C223	1.3863 (18)
C21—H21	1.0000	C222—H222	0.9500
C22—C227	1.5214 (16)	C223—C224	1.388 (2)
C22—H22	1.0000	C223—H223	0.9500
N24—C25	1.4673 (14)	C224—C225	1.388 (2)
N24—C28A	1.4690 (14)	C224—H224	0.9500
C25—C26	1.5249 (17)	C225—C226	1.3931 (17)
C25—H25A	0.9900	C225—H225	0.9500
C25—H25B	0.9900	C226—H226	0.9500

C12—N11—C17A	111.19 (9)	C27—C26—H26A	109.5
C12—N11—C111	123.70 (10)	C25—C26—H26B	109.5
C17A—N11—C111	125.09 (10)	C27—C26—H26B	109.5
O12—C12—N11	125.38 (11)	H26A—C26—H26B	108.1
O12—C12—C13	126.20 (10)	C26—C27—C28	110.63 (10)
N11—C12—C13	108.41 (9)	C26—C27—H27A	109.5
N24—C13—C13A	109.50 (9)	C28—C27—H27A	109.5
N24—C13—C12	112.67 (9)	C26—C27—H27B	109.5
C13A—C13—C12	101.12 (9)	C28—C27—H27B	109.5
N24—C13—C22	103.27 (8)	H27A—C27—H27B	108.1
C13A—C13—C22	120.84 (9)	C28A—C28—C27	108.82 (10)
C12—C13—C22	109.74 (9)	C28A—C28—H28B	109.9
C14—C13A—C17A	120.08 (10)	C27—C28—H28B	109.9
C14—C13A—C13	130.54 (10)	C28A—C28—H28C	109.9
C17A—C13A—C13	108.96 (10)	C27—C28—H28C	109.9
C13A—C14—C15	117.48 (10)	H28B—C28—H28C	108.3
C13A—C14—H14	121.3	N24—C28A—C28	109.52 (9)
C15—C14—H14	121.3	N24—C28A—C21	100.39 (9)
C16—C15—C14	122.44 (11)	C28—C28A—C21	117.23 (10)
C16—C15—Cl15	118.92 (9)	N24—C28A—H28A	109.7
C14—C15—Cl15	118.63 (9)	C28—C28A—H28A	109.7
C15—C16—C17	120.11 (11)	C21—C28A—H28A	109.7
C15—C16—H16	119.9	N11—C111—H1A	109.5
C17—C16—H16	119.9	N11—C111—H1B	109.5
C17A—C17—C16	117.40 (10)	H1A—C111—H1B	109.5
C17A—C17—H17	121.3	N11—C111—H1C	109.5
C16—C17—H17	121.3	H1A—C111—H1C	109.5
C17—C17A—C13A	122.48 (11)	H1B—C111—H1C	109.5
C17—C17A—N11	127.79 (10)	O211—C211—O212	123.38 (11)

C13A—C17A—N11	109.70 (10)	O211—C211—C21	124.14 (10)
C211—C21—C22	113.64 (9)	O212—C211—C21	112.47 (10)
C211—C21—C28A	111.18 (9)	C211—O212—H212	108.4 (12)
C22—C21—C28A	101.78 (9)	O227—C227—C221	120.95 (11)
C211—C21—H21	110.0	O227—C227—C22	120.35 (10)
C22—C21—H21	110.0	C221—C227—C22	118.70 (10)
C28A—C21—H21	110.0	C226—C221—C222	119.04 (11)
C227—C22—C21	114.31 (9)	C226—C221—C227	122.62 (11)
C227—C22—C13	112.89 (9)	C222—C221—C227	118.34 (11)
C21—C22—C13	104.88 (9)	C223—C222—C221	120.25 (12)
C227—C22—H22	108.2	C223—C222—H222	119.9
C21—C22—H22	108.2	C221—C222—H222	119.9
C13—C22—H22	108.2	C222—C223—C224	120.23 (12)
C25—N24—C28A	112.33 (9)	C222—C223—H223	119.9
C25—N24—C13	115.84 (9)	C224—C223—H223	119.9
C28A—N24—C13	108.07 (8)	C223—C224—C225	120.21 (12)
N24—C25—C26	109.23 (10)	C223—C224—H224	119.9
N24—C25—H25A	109.8	C225—C224—H224	119.9
C26—C25—H25A	109.8	C224—C225—C226	119.93 (13)
N24—C25—H25B	109.8	C224—C225—H225	120.0
C26—C25—H25B	109.8	C226—C225—H225	120.0
H25A—C25—H25B	108.3	C225—C226—C221	120.34 (12)
C25—C26—C27	110.77 (10)	C225—C226—H226	119.8
C25—C26—H26A	109.5	C221—C226—H226	119.8

C17A—N11—C12—O12	−176.56 (11)	C12—C13—C22—C21	−124.92 (9)
C111—N11—C12—O12	2.39 (19)	C13A—C13—N24—C25	78.27 (12)
C17A—N11—C12—C13	4.44 (12)	C12—C13—N24—C25	−33.43 (13)
C111—N11—C12—C13	−176.60 (10)	C22—C13—N24—C25	−151.76 (9)
O12—C12—C13—N24	−69.41 (14)	C13A—C13—N24—C28A	−154.71 (9)
N11—C12—C13—N24	109.58 (10)	C12—C13—N24—C28A	93.59 (11)
O12—C12—C13—C13A	173.79 (11)	C22—C13—N24—C28A	−24.75 (11)
N11—C12—C13—C13A	−7.22 (11)	C28A—N24—C25—C26	60.42 (12)
O12—C12—C13—C22	45.06 (15)	C13—N24—C25—C26	−174.72 (9)
N11—C12—C13—C22	−135.95 (9)	N24—C25—C26—C27	−55.47 (13)
N24—C13—C13A—C14	60.82 (15)	C25—C26—C27—C28	54.63 (14)
C12—C13—C13A—C14	179.93 (11)	C26—C27—C28—C28A	−55.81 (13)
C22—C13—C13A—C14	−58.85 (17)	C25—N24—C28A—C28	−62.97 (12)
N24—C13—C13A—C17A	−111.53 (10)	C13—N24—C28A—C28	168.01 (9)
C12—C13—C13A—C17A	7.57 (11)	C25—N24—C28A—C21	173.08 (9)
C22—C13—C13A—C17A	128.80 (11)	C13—N24—C28A—C21	44.06 (11)
C17A—C13A—C14—C15	−0.28 (16)	C27—C28—C28A—N24	59.01 (12)
C13—C13A—C14—C15	−171.92 (11)	C27—C28—C28A—C21	172.44 (10)
C13A—C14—C15—C16	0.77 (17)	C211—C21—C28A—N24	−165.95 (9)
C13A—C14—C15—Cl15	179.98 (8)	C22—C21—C28A—N24	−44.61 (10)
C14—C15—C16—C17	−0.27 (17)	C211—C21—C28A—C28	75.60 (13)
Cl15—C15—C16—C17	−179.48 (9)	C22—C21—C28A—C28	−163.06 (9)
C15—C16—C17—C17A	−0.72 (16)	C22—C21—C211—O211	−20.85 (16)

C16—C17—C17A—C13A	1.22 (17)	C28A—C21—C211—O211	93.26 (13)
C16—C17—C17A—N11	178.99 (11)	C22—C21—C211—O212	160.36 (10)
C14—C13A—C17A—C17	−0.72 (17)	C28A—C21—C211—O212	−85.52 (12)
C13—C13A—C17A—C17	172.57 (10)	C21—C22—C227—O227	−24.47 (15)
C14—C13A—C17A—N11	−178.86 (10)	C13—C22—C227—O227	95.31 (12)
C13—C13A—C17A—N11	−5.57 (12)	C21—C22—C227—C221	155.15 (10)
C12—N11—C17A—C17	−177.39 (11)	C13—C22—C227—C221	−85.07 (12)
C111—N11—C17A—C17	3.68 (18)	O227—C227—C221—C226	179.08 (11)
C12—N11—C17A—C13A	0.62 (13)	C22—C227—C221—C226	−0.54 (16)
C111—N11—C17A—C13A	−178.32 (11)	O227—C227—C221—C222	−0.08 (16)
C211—C21—C22—C227	−86.04 (12)	C22—C227—C221—C222	−179.70 (10)
C28A—C21—C22—C227	154.35 (9)	C226—C221—C222—C223	−0.09 (18)
C211—C21—C22—C13	149.80 (9)	C227—C221—C222—C223	179.10 (11)
C28A—C21—C22—C13	30.18 (11)	C221—C222—C223—C224	0.25 (19)
N24—C13—C22—C227	−129.63 (10)	C222—C223—C224—C225	−0.2 (2)
C13A—C13—C22—C227	−6.93 (14)	C223—C224—C225—C226	−0.10 (19)
C12—C13—C22—C227	110.01 (10)	C224—C225—C226—C221	0.26 (18)
N24—C13—C22—C21	−4.57 (11)	C222—C221—C226—C225	−0.17 (17)
C13A—C13—C22—C21	118.14 (11)	C227—C221—C226—C225	−179.32 (11)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O212—H212···O211^{i}	0.863 (19)	1.854 (19)	2.7152 (14)	175.3 (16)
C16—H16···*Cg*1^{ii}	0.95	2.52	3.3872 (14)	152

Symmetry codes: (i) −*x*+1, −*y*+1, −*z*+1; (ii) −*x*, −*y*+1, −*z*.

(1′*SR*,2′*SR*,3*RS*,8a′*RS*)-2′-Benzoyl-1-hexyl-2-oxo-1′,5′,6′,7′,8′,8a′-hexahydro-2′*H*-spiro[indoline-3,3′-indolizine]-1′-carboxylic acid (V)

Crystal data

$C_{29}H_{34}N_2O_4$
M_r = 474.58
Monoclinic, $P2_1/n$
a = 11.0442 (4) Å
b = 17.4707 (6) Å
c = 13.0081 (4) Å
β = 90.215 (1)°
V = 2509.89 (15) Å^3
Z = 4
$F(000)$ = 1016
D_x = 1.256 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 5765 reflections
θ = 2.2–27.5°
μ = 0.08 mm^{-1}
T = 100 K
Block, colourless
0.23 × 0.13 × 0.12 mm

Data collection

Bruker D8 Venture diffractometer
Radiation source: INCOATEC high brilliance microfocus sealed tube
Multilayer mirror monochromator
φ and ω scans
Absorption correction: multi-scan (SADABS; Bruker, 2016)
T_{min} = 0.921, T_{max} = 0.990
24313 measured reflections
5765 independent reflections
4619 reflections with $I > 2\sigma(I)$
R_{int} = 0.059

θ_{max} = 27.5°, θ_{min} = 2.2°
h = −14→14
k = −21→22
l = −16→16

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.043
$wR(F^2)$ = 0.102
S = 1.03
5765 reflections
320 parameters
0 restraints
Primary atom site location: dual
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0368P)^2 + 0.9168P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.29 e Å^{-3}
$\Delta\rho_{min}$ = −0.26 e Å^{-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.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
N11	0.22287 (10)	0.48035 (7)	0.18134 (8)	0.0148 (2)
C12	0.32622 (12)	0.49126 (8)	0.23582 (9)	0.0129 (3)
O12	0.36289 (9)	0.55415 (5)	0.26592 (7)	0.0164 (2)
C13	0.39048 (11)	0.41293 (7)	0.25035 (10)	0.0118 (3)
C13A	0.30516 (11)	0.35997 (8)	0.19371 (10)	0.0136 (3)
C14	0.31398 (12)	0.28250 (8)	0.17408 (10)	0.0173 (3)
H14	0.3824	0.2541	0.1969	0.021*
C15	0.21988 (13)	0.24655 (9)	0.11982 (11)	0.0215 (3)
H15	0.2235	0.1931	0.1069	0.026*
C16	0.12169 (13)	0.28869 (9)	0.08490 (11)	0.0238 (3)
H16	0.0585	0.2635	0.0486	0.029*
C17	0.11376 (13)	0.36756 (9)	0.10203 (11)	0.0210 (3)
H17	0.0468	0.3965	0.0773	0.025*
C17A	0.20708 (12)	0.40190 (8)	0.15632 (10)	0.0149 (3)
C21	0.55038 (11)	0.39258 (7)	0.37985 (10)	0.0120 (3)
H21	0.5733	0.3375	0.3719	0.014*
C22	0.41156 (11)	0.39777 (7)	0.36885 (9)	0.0112 (3)
H22	0.3819	0.4430	0.4087	0.013*
N24	0.51257 (9)	0.40998 (6)	0.20792 (8)	0.0126 (2)
C25	0.52881 (12)	0.44460 (9)	0.10668 (10)	0.0172 (3)
H25A	0.5194	0.5008	0.1114	0.021*
H25B	0.4669	0.4248	0.0583	0.021*
C26	0.65518 (12)	0.42485 (9)	0.06784 (10)	0.0200 (3)
H26A	0.6692	0.4504	0.0010	0.024*
H26B	0.6615	0.3689	0.0572	0.024*
C27	0.75124 (12)	0.45069 (9)	0.14523 (11)	0.0200 (3)
H27A	0.7505	0.5072	0.1504	0.024*

H27B	0.8323	0.4347	0.1210	0.024*
C28	0.72742 (12)	0.41587 (8)	0.25140 (10)	0.0163 (3)
H28A	0.7371	0.3596	0.2482	0.020*
H28B	0.7868	0.4362	0.3016	0.020*
C28A	0.59937 (11)	0.43555 (8)	0.28612 (9)	0.0122 (3)
H18C	0.5917	0.4920	0.2968	0.015*
C111	0.13937 (12)	0.54202 (8)	0.15437 (10)	0.0177 (3)
H11A	0.1002	0.5298	0.0878	0.021*
H11B	0.1860	0.5899	0.1453	0.021*
C112	0.04138 (12)	0.55524 (8)	0.23478 (11)	0.0178 (3)
H11C	0.0792	0.5723	0.3000	0.021*
H11D	−0.0019	0.5067	0.2481	0.021*
C113	−0.04843 (12)	0.61569 (8)	0.19747 (11)	0.0175 (3)
H11E	−0.0890	0.5966	0.1345	0.021*
H11F	−0.0031	0.6625	0.1788	0.021*
C114	−0.14507 (13)	0.63668 (8)	0.27627 (11)	0.0191 (3)
H11G	−0.1825	0.5892	0.3029	0.023*
H11H	−0.1063	0.6635	0.3348	0.023*
C115	−0.24331 (13)	0.68768 (9)	0.23080 (12)	0.0231 (3)
H11I	−0.2861	0.6592	0.1758	0.028*
H11J	−0.2048	0.7330	0.1990	0.028*
C116	−0.33563 (14)	0.71456 (9)	0.30990 (12)	0.0273 (3)
H11K	−0.3758	0.6700	0.3404	0.041*
H11L	−0.3961	0.7471	0.2762	0.041*
H11M	−0.2943	0.7438	0.3639	0.041*
C211	0.60196 (12)	0.41976 (7)	0.48092 (10)	0.0126 (3)
O211	0.70490 (8)	0.44278 (6)	0.49134 (7)	0.0193 (2)
O212	0.52409 (8)	0.41326 (6)	0.55863 (7)	0.0159 (2)
H212	0.5642 (15)	0.4242 (10)	0.6185 (14)	0.024*
C227	0.34460 (12)	0.32642 (8)	0.40512 (9)	0.0124 (3)
O227	0.39880 (8)	0.26668 (5)	0.42060 (7)	0.0170 (2)
C221	0.20965 (12)	0.33008 (8)	0.41731 (10)	0.0141 (3)
C222	0.14638 (13)	0.26082 (9)	0.41690 (12)	0.0214 (3)
H222	0.1892	0.2141	0.4087	0.026*
C223	0.02173 (14)	0.25984 (9)	0.42841 (13)	0.0270 (3)
H223	−0.0206	0.2125	0.4284	0.032*
C224	−0.04133 (13)	0.32800 (9)	0.43998 (12)	0.0240 (3)
H224	−0.1268	0.3273	0.4479	0.029*
C225	0.02029 (13)	0.39690 (9)	0.43991 (11)	0.0202 (3)
H225	−0.0232	0.4434	0.4476	0.024*
C226	0.14587 (12)	0.39867 (8)	0.42859 (10)	0.0162 (3)
H226	0.1877	0.4462	0.4285	0.019*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N11	0.0116 (5)	0.0188 (6)	0.0139 (5)	0.0019 (4)	−0.0010 (4)	0.0009 (5)
C12	0.0128 (6)	0.0169 (7)	0.0090 (6)	−0.0003 (5)	0.0016 (5)	0.0008 (5)

O12	0.0202 (5)	0.0146 (5)	0.0143 (4)	0.0002 (4)	−0.0022 (4)	0.0002 (4)
C13	0.0108 (6)	0.0131 (6)	0.0115 (6)	0.0002 (5)	−0.0009 (5)	0.0003 (5)
C13A	0.0125 (6)	0.0187 (7)	0.0096 (6)	−0.0031 (5)	0.0008 (5)	−0.0011 (5)
C14	0.0172 (7)	0.0192 (7)	0.0154 (6)	−0.0017 (5)	0.0006 (5)	−0.0017 (5)
C15	0.0234 (7)	0.0218 (8)	0.0194 (7)	−0.0072 (6)	0.0023 (6)	−0.0068 (6)
C16	0.0180 (7)	0.0334 (9)	0.0201 (7)	−0.0098 (6)	−0.0016 (6)	−0.0079 (6)
C17	0.0133 (7)	0.0335 (9)	0.0163 (7)	−0.0009 (6)	−0.0019 (5)	−0.0011 (6)
C17A	0.0132 (6)	0.0202 (7)	0.0114 (6)	−0.0009 (5)	0.0016 (5)	−0.0001 (5)
C21	0.0111 (6)	0.0115 (6)	0.0133 (6)	0.0004 (5)	−0.0006 (5)	0.0007 (5)
C22	0.0105 (6)	0.0129 (6)	0.0102 (6)	−0.0002 (5)	−0.0008 (5)	−0.0004 (5)
N24	0.0104 (5)	0.0166 (6)	0.0107 (5)	−0.0005 (4)	0.0001 (4)	0.0001 (4)
C25	0.0162 (7)	0.0237 (7)	0.0118 (6)	−0.0009 (5)	0.0010 (5)	0.0012 (5)
C26	0.0178 (7)	0.0278 (8)	0.0145 (6)	−0.0001 (6)	0.0037 (5)	0.0000 (6)
C27	0.0137 (6)	0.0268 (8)	0.0195 (7)	−0.0017 (6)	0.0053 (5)	0.0015 (6)
C28	0.0113 (6)	0.0209 (7)	0.0167 (6)	0.0005 (5)	0.0007 (5)	0.0007 (5)
C28A	0.0116 (6)	0.0133 (6)	0.0117 (6)	−0.0005 (5)	−0.0007 (5)	0.0000 (5)
C111	0.0148 (7)	0.0226 (7)	0.0158 (6)	0.0044 (5)	−0.0004 (5)	0.0040 (5)
C112	0.0152 (7)	0.0204 (7)	0.0178 (7)	0.0020 (5)	0.0004 (5)	0.0021 (6)
C113	0.0161 (7)	0.0167 (7)	0.0196 (7)	0.0009 (5)	0.0015 (5)	0.0024 (5)
C114	0.0190 (7)	0.0182 (7)	0.0202 (7)	0.0016 (5)	0.0033 (5)	0.0019 (6)
C115	0.0228 (8)	0.0213 (8)	0.0252 (8)	0.0062 (6)	0.0048 (6)	0.0036 (6)
C116	0.0252 (8)	0.0259 (8)	0.0308 (8)	0.0059 (6)	0.0098 (7)	0.0054 (7)
C211	0.0135 (6)	0.0102 (6)	0.0140 (6)	0.0022 (5)	−0.0006 (5)	0.0008 (5)
O211	0.0138 (5)	0.0261 (6)	0.0180 (5)	−0.0034 (4)	−0.0024 (4)	−0.0023 (4)
O212	0.0151 (5)	0.0209 (5)	0.0116 (4)	−0.0021 (4)	−0.0004 (4)	−0.0006 (4)
C227	0.0143 (6)	0.0145 (6)	0.0084 (6)	−0.0005 (5)	−0.0017 (5)	−0.0009 (5)
O227	0.0166 (5)	0.0149 (5)	0.0194 (5)	0.0010 (4)	−0.0010 (4)	0.0022 (4)
C221	0.0132 (6)	0.0180 (7)	0.0112 (6)	−0.0015 (5)	−0.0003 (5)	0.0036 (5)
C222	0.0187 (7)	0.0168 (7)	0.0287 (8)	−0.0007 (6)	−0.0006 (6)	0.0059 (6)
C223	0.0188 (7)	0.0229 (8)	0.0393 (9)	−0.0078 (6)	−0.0012 (6)	0.0087 (7)
C224	0.0127 (7)	0.0342 (9)	0.0250 (8)	−0.0019 (6)	−0.0003 (6)	0.0072 (7)
C225	0.0163 (7)	0.0251 (8)	0.0193 (7)	0.0039 (6)	0.0022 (5)	0.0011 (6)
C226	0.0146 (6)	0.0177 (7)	0.0162 (6)	−0.0003 (5)	0.0009 (5)	0.0008 (5)

Geometric parameters (Å, º)

N11—C12	1.3547 (17)	C28—H28B	0.9900
N11—C17A	1.4194 (18)	C28A—H18C	1.0000
N11—C111	1.4601 (17)	C111—C112	1.5255 (19)
C12—O12	1.2343 (16)	C111—H11A	0.9900
C12—C13	1.5527 (18)	C111—H11B	0.9900
C13—N24	1.4599 (16)	C112—C113	1.5268 (19)
C13—C13A	1.5106 (17)	C112—H11C	0.9900
C13—C22	1.5804 (17)	C112—H11D	0.9900
C13A—C14	1.381 (2)	C113—C114	1.5272 (19)
C13A—C17A	1.3938 (19)	C113—H11E	0.9900
C14—C15	1.4025 (19)	C113—H11F	0.9900
C14—H14	0.9500	C114—C115	1.522 (2)

C15—C16	1.386 (2)	C114—H11G	0.9900
C15—H15	0.9500	C114—H11H	0.9900
C16—C17	1.399 (2)	C115—C116	1.525 (2)
C16—H16	0.9500	C115—H11I	0.9900
C17—C17A	1.3839 (19)	C115—H11J	0.9900
C17—H17	0.9500	C116—H11K	0.9800
C21—C211	1.5076 (17)	C116—H11L	0.9800
C21—C28A	1.5322 (18)	C116—H11M	0.9800
C21—C22	1.5419 (17)	C211—O211	1.2130 (16)
C21—H21	1.0000	C211—O212	1.3345 (16)
C22—C227	1.5251 (18)	O212—H212	0.914 (18)
C22—H22	1.0000	C227—O227	1.2195 (16)
N24—C25	1.4608 (17)	C227—C221	1.5007 (18)
N24—C28A	1.4648 (16)	C221—C222	1.3974 (19)
C25—C26	1.5256 (19)	C221—C226	1.3979 (19)
C25—H25A	0.9900	C222—C223	1.385 (2)
C25—H25B	0.9900	C222—H222	0.9500
C26—C27	1.528 (2)	C223—C224	1.388 (2)
C26—H26A	0.9900	C223—H223	0.9500
C26—H26B	0.9900	C224—C225	1.383 (2)
C27—C28	1.5327 (19)	C224—H224	0.9500
C27—H27A	0.9900	C225—C226	1.3956 (19)
C27—H27B	0.9900	C225—H225	0.9500
C28—C28A	1.5255 (18)	C226—H226	0.9500
C28—H28A	0.9900		

C12—N11—C17A	111.00 (11)	H28A—C28—H28B	108.2
C12—N11—C111	123.51 (12)	N24—C28A—C28	109.31 (10)
C17A—N11—C111	125.48 (11)	N24—C28A—C21	99.85 (10)
O12—C12—N11	124.47 (12)	C28—C28A—C21	117.11 (11)
O12—C12—C13	126.63 (11)	N24—C28A—H18C	110.0
N11—C12—C13	108.88 (11)	C28—C28A—H18C	110.0
N24—C13—C13A	111.67 (10)	C21—C28A—H18C	110.0
N24—C13—C12	114.04 (10)	N11—C111—C112	113.33 (11)
C13A—C13—C12	101.33 (10)	N11—C111—H11A	108.9
N24—C13—C22	103.29 (9)	C112—C111—H11A	108.9
C13A—C13—C22	117.54 (11)	N11—C111—H11B	108.9
C12—C13—C22	109.40 (10)	C112—C111—H11B	108.9
C14—C13A—C17A	120.39 (12)	H11A—C111—H11B	107.7
C14—C13A—C13	130.27 (12)	C111—C112—C113	110.39 (11)
C17A—C13A—C13	109.32 (12)	C111—C112—H11C	109.6
C13A—C14—C15	118.65 (13)	C113—C112—H11C	109.6
C13A—C14—H14	120.7	C111—C112—H11D	109.6
C15—C14—H14	120.7	C113—C112—H11D	109.6
C16—C15—C14	120.29 (14)	H11C—C112—H11D	108.1
C16—C15—H15	119.9	C112—C113—C114	114.05 (11)
C14—C15—H15	119.9	C112—C113—H11E	108.7
C15—C16—C17	121.37 (13)	C114—C113—H11E	108.7

C15—C16—H16	119.3	C112—C113—H11F	108.7
C17—C16—H16	119.3	C114—C113—H11F	108.7
C17A—C17—C16	117.48 (13)	H11E—C113—H11F	107.6
C17A—C17—H17	121.3	C115—C114—C113	112.26 (12)
C16—C17—H17	121.3	C115—C114—H11G	109.2
C17—C17A—C13A	121.77 (13)	C113—C114—H11G	109.2
C17—C17A—N11	128.78 (13)	C115—C114—H11H	109.2
C13A—C17A—N11	109.44 (11)	C113—C114—H11H	109.2
C211—C21—C28A	113.97 (11)	H11G—C114—H11H	107.9
C211—C21—C22	115.77 (11)	C114—C115—C116	113.29 (12)
C28A—C21—C22	104.56 (10)	C114—C115—H11I	108.9
C211—C21—H21	107.4	C116—C115—H11I	108.9
C28A—C21—H21	107.4	C114—C115—H11J	108.9
C22—C21—H21	107.4	C116—C115—H11J	108.9
C227—C22—C21	113.98 (11)	H11I—C115—H11J	107.7
C227—C22—C13	111.65 (10)	C115—C116—H11K	109.5
C21—C22—C13	104.07 (10)	C115—C116—H11L	109.5
C227—C22—H22	109.0	H11K—C116—H11L	109.5
C21—C22—H22	109.0	C115—C116—H11M	109.5
C13—C22—H22	109.0	H11K—C116—H11M	109.5
C13—N24—C25	116.29 (10)	H11L—C116—H11M	109.5
C13—N24—C28A	109.25 (10)	O211—C211—O212	123.36 (12)
C25—N24—C28A	114.68 (10)	O211—C211—C21	123.56 (12)
N24—C25—C26	108.69 (11)	O212—C211—C21	113.04 (11)
N24—C25—H25A	110.0	C211—O212—H212	108.4 (11)
C26—C25—H25A	110.0	O227—C227—C221	120.41 (12)
N24—C25—H25B	110.0	O227—C227—C22	120.81 (12)
C26—C25—H25B	110.0	C221—C227—C22	118.73 (11)
H25A—C25—H25B	108.3	C222—C221—C226	119.37 (12)
C25—C26—C27	110.42 (11)	C222—C221—C227	117.35 (12)
C25—C26—H26A	109.6	C226—C221—C227	123.28 (12)
C27—C26—H26A	109.6	C223—C222—C221	120.49 (14)
C25—C26—H26B	109.6	C223—C222—H222	119.8
C27—C26—H26B	109.6	C221—C222—H222	119.8
H26A—C26—H26B	108.1	C222—C223—C224	120.02 (14)
C26—C27—C28	110.81 (11)	C222—C223—H223	120.0
C26—C27—H27A	109.5	C224—C223—H223	120.0
C28—C27—H27A	109.5	C225—C224—C223	119.97 (13)
C26—C27—H27B	109.5	C225—C224—H224	120.0
C28—C27—H27B	109.5	C223—C224—H224	120.0
H27A—C27—H27B	108.1	C224—C225—C226	120.57 (14)
C28A—C28—C27	109.86 (11)	C224—C225—H225	119.7
C28A—C28—H28A	109.7	C226—C225—H225	119.7
C27—C28—H28A	109.7	C225—C226—C221	119.57 (13)
C28A—C28—H28B	109.7	C225—C226—H226	120.2
C27—C28—H28B	109.7	C221—C226—H226	120.2
C17A—N11—C12—O12	178.71 (12)	C13A—C13—N24—C28A	−157.26 (11)

C111—N11—C12—O12	−2.2 (2)	C12—C13—N24—C28A	88.61 (12)
C17A—N11—C12—C13	0.36 (14)	C22—C13—N24—C28A	−30.02 (13)
C111—N11—C12—C13	179.47 (11)	C13—N24—C25—C26	−170.95 (11)
O12—C12—C13—N24	−57.46 (17)	C28A—N24—C25—C26	59.88 (15)
N11—C12—C13—N24	120.85 (12)	N24—C25—C26—C27	−56.02 (16)
O12—C12—C13—C13A	−177.58 (12)	C25—C26—C27—C28	55.76 (16)
N11—C12—C13—C13A	0.73 (13)	C26—C27—C28—C28A	−55.23 (15)
O12—C12—C13—C22	57.63 (16)	C13—N24—C28A—C28	167.41 (11)
N11—C12—C13—C22	−124.06 (11)	C25—N24—C28A—C28	−60.00 (14)
N24—C13—C13A—C14	54.77 (18)	C13—N24—C28A—C21	43.98 (12)
C12—C13—C13A—C14	176.56 (13)	C25—N24—C28A—C21	176.57 (11)
C22—C13—C13A—C14	−64.33 (18)	C27—C28—C28A—N24	55.48 (14)
N24—C13—C13A—C17A	−123.36 (12)	C27—C28—C28A—C21	168.00 (11)
C12—C13—C13A—C17A	−1.57 (13)	C211—C21—C28A—N24	−166.73 (10)
C22—C13—C13A—C17A	117.54 (12)	C22—C21—C28A—N24	−39.35 (12)
C17A—C13A—C14—C15	−2.7 (2)	C211—C21—C28A—C28	75.50 (14)
C13—C13A—C14—C15	179.32 (13)	C22—C21—C28A—C28	−157.13 (11)
C13A—C14—C15—C16	1.4 (2)	C12—N11—C111—C112	−89.99 (15)
C14—C15—C16—C17	0.3 (2)	C17A—N11—C111—C112	88.98 (16)
C15—C16—C17—C17A	−0.8 (2)	N11—C111—C112—C113	−175.28 (11)
C16—C17—C17A—C13A	−0.6 (2)	C111—C112—C113—C114	−176.02 (12)
C16—C17—C17A—N11	177.99 (13)	C112—C113—C114—C115	−171.33 (12)
C14—C13A—C17A—C17	2.4 (2)	C113—C114—C115—C116	−175.52 (13)
C13—C13A—C17A—C17	−179.29 (12)	C28A—C21—C211—O211	−33.95 (18)
C14—C13A—C17A—N11	−176.46 (12)	C22—C21—C211—O211	−155.29 (13)
C13—C13A—C17A—N11	1.89 (14)	C28A—C21—C211—O212	148.37 (11)
C12—N11—C17A—C17	179.86 (13)	C22—C21—C211—O212	27.03 (16)
C111—N11—C17A—C17	0.8 (2)	C21—C22—C227—O227	−14.10 (17)
C12—N11—C17A—C13A	−1.42 (15)	C13—C22—C227—O227	103.47 (13)
C111—N11—C17A—C13A	179.49 (12)	C21—C22—C227—C221	168.39 (11)
C211—C21—C22—C227	−89.51 (14)	C13—C22—C227—C221	−74.03 (14)
C28A—C21—C22—C227	144.23 (11)	O227—C227—C221—C222	−18.16 (18)
C211—C21—C22—C13	148.63 (11)	C22—C227—C221—C222	159.35 (12)
C28A—C21—C22—C13	22.37 (12)	O227—C227—C221—C226	162.00 (12)
N24—C13—C22—C227	−120.05 (11)	C22—C227—C221—C226	−20.49 (18)
C13A—C13—C22—C227	3.40 (16)	C226—C221—C222—C223	−0.5 (2)
C12—C13—C22—C227	118.14 (12)	C227—C221—C222—C223	179.61 (13)
N24—C13—C22—C21	3.34 (12)	C221—C222—C223—C224	0.3 (2)
C13A—C13—C22—C21	126.79 (11)	C222—C223—C224—C225	0.1 (2)
C12—C13—C22—C21	−118.47 (11)	C223—C224—C225—C226	−0.2 (2)
C13A—C13—N24—C25	71.00 (14)	C224—C225—C226—C221	−0.1 (2)
C12—C13—N24—C25	−43.13 (15)	C222—C221—C226—C225	0.4 (2)
C22—C13—N24—C25	−161.76 (11)	C227—C221—C226—C225	−179.75 (12)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O212—H212···O12^{i}	0.915 (18)	1.744 (18)	2.6589 (13)	178.5 (15)

C113—H11*F*···O227^{ii}	0.99	2.51	3.4738 (17)	163
C16—H16···O227^{iii}	0.95	2.48	3.3947 (17)	162
C22—H22···O211^{i}	1.00	2.57	3.5693 (16)	177
C226—H226···O211^{i}	0.95	2.50	3.3854 (17)	155

Symmetry codes: (i) −*x*+1, −*y*+1, −*z*+1; (ii) −*x*+1/2, *y*+1/2, −*z*+1/2; (iii) *x*−1/2, −*y*+1/2, *z*−1/2.