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Reactions of chromium (methoxymethyl)carbene complexes with 3*H*-indoles

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Abstract

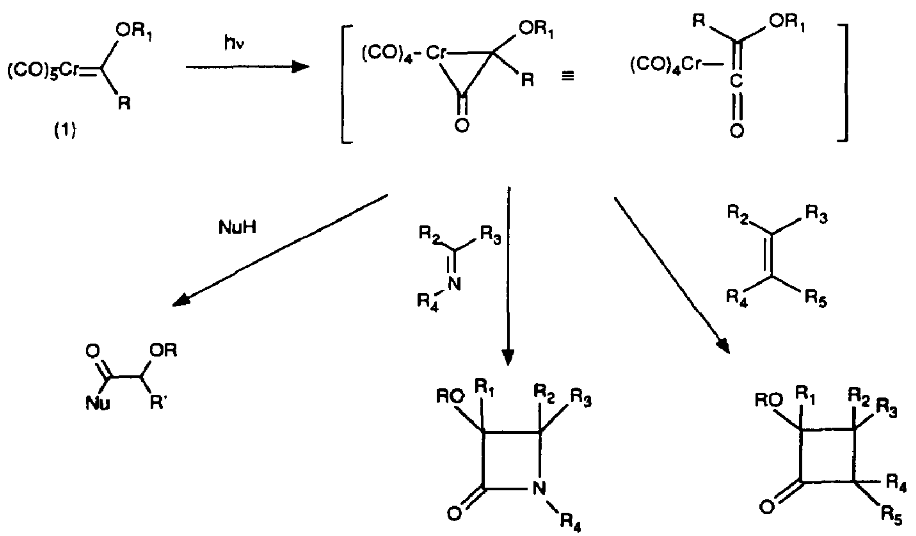
The reactions of chromium-complexed carbenes with 3,3-dimethyl 3*H*-indoles have been studied in the presence and absence of a Lewis acid as activator in sunlight or under irradiation from a Hg lamp. 1,3-Diazinone-diindolic derivatives have been obtained.

Introduction

Among the isolable metal-complexed carbenic species, those stabilized by heteroatoms, such as the Fischer carbenes (**1**), are particularly interesting and are becoming useful reagents for organic synthesis. The (C=M)OR unit behaves like an ester group and increases the acidity of the protons on the carbon bound to the carbenic center [1]. In addition, the $C(OR)R_1$ unit has been often used as a C_1 synthon in C–C bond formation [2].

Another important characteristic of this class of compounds is the ease of photochemical insertion of a C=O ligand into the metal carbenic-carbon bond. This produces metal-complexed ketenic species, which are highly reactive towards nucleophiles and very stereoselective in 2 + 2 cycloaddition reactions. These features have allowed the synthesis of β-lactams [3], α-aminoacids [3] and cyclobutanones [4] (Scheme 1).

Our aim in this work was to study the reactivity of chromium-complexed carbenes in 2 + 2 cycloaddition reactions. In particular, since many compounds which possess biological activity contain in their structure a 3,3-dialkyl-3*H*-indole framework, we decided to study the applicability of the cycloaddition reaction to this substrate. Furthermore, 3*H*-indoles are heterocyclic imines that do not undergo



Scheme 1

easily 2 + 2 cycloadditions, but seem to favour 1,4 dipolar cyclizations yielding products containing one or two indole units [5]. However, in one case, involving the reaction of 3,3-dimethyl-3*H*-indole with ynamines in the presence of BF_3, the formation of benzazepinic derivatives seems to proceed by a 2 + 2 cycloaddition mechanism [6]. Thus we were also interested in finding out whether chromium ketenic species, photogenerated from chromium carbene complexes, would react by a 2 + 2 cycloaddition rather than a 1,4 dipolar pathway.

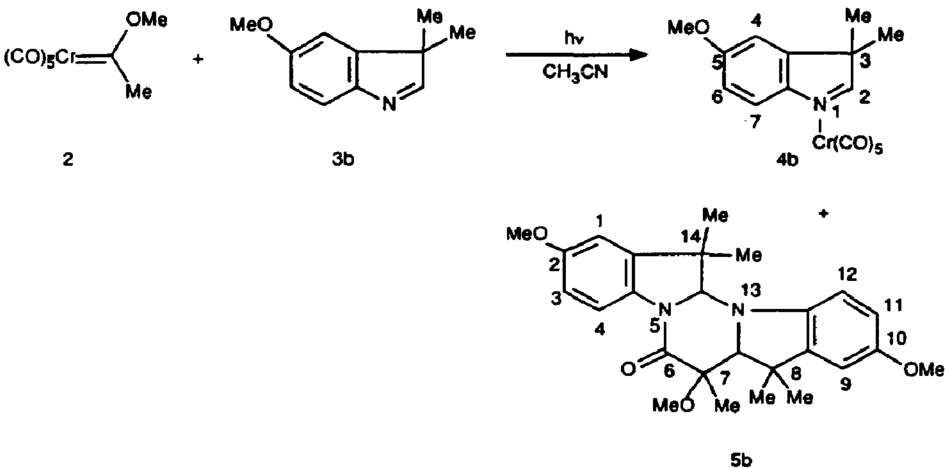
Results and discussion

The reaction of pentacarbonyl(methylmethoxycarbene)chromium(0) (**2**) with 3,3-dimethyl-3*H*-indole derivatives (**3a** and **3b**) was conducted in sunlight or under irradiation from a high vapor pressure Hg lamp. In both cases, reaction of **2** with **3b** yielded a complex mixture from which the following were isolated: unchanged starting material (**3b**, 20%), adduct (**4b**, 21%), and another product (ca. 10%). The last product, further purified by crystallization from acetone, was identified as 2,7,10-trimethoxy-7,8,8,14,14-pentamethyl-6-oxo-6*H*, 8*H*-[1,3]-diazino-[2,1-a:4,3-a′]-diindole (**5b**) from its analytical and spectroscopic data (Scheme 2).

The IR spectrum of **5b** shows a strong band at 1650 cm^{-1}, within the range characteristic of six-membered ring lactams. The chemical shift values for the protons of the group C(7)–CH–C(8) (3–4 ppm) and group N(5)–CH–N(13) (4.7–5.5 ppm) are consistent with those reported in the literature for similar compounds [5].

Analysis of the mother liquors from crystallization of **5b** revealed the presence of **5b** and three other possible diastereoisomers, and this was confirmed by NMR spectroscopy and mass spectrometry.

The diastereoisomeric ratio can be determined directly with the mixture from the integrals of the signals from the protons of the four diastereoisomeric N(5)–CH–N(13) groups.



Scheme 2

The exact stereochemistry of the major diastereoisomer was determined by a single crystal diffraction study, which revealed that both indole rings (Fig. 1) are, except for C(2) and C(2′), essentially planar; C(2) lies 0.39 Å out of the plane and C(2′) 0.58 Å. The rings are inclined by ca. 60° to each other. The central six-membered ring (Fig. 2) has an half-chair conformation, with the methoxy substituent, and the C(2) and C(2′) hydrogens axial. There are no short intermolecular contacts.

From the photochemical reaction of **2** with **3a** three products were isolated: starting material (30%), adduct (**4a**, 20%), and the indolenine trimer (**6**, 15%) [7] (Scheme 3).

The differences in reactivity between **3a** and **3b** may be due to the higher stability of **3b** than of (**3a**) as a monomer, and to its stronger nucleophilicity.

The formation of **5b** is consistent with the behaviour of 3*H*-indoles towards heterocumulenes [5]. Thus **5b** is formed from the reaction of one molecule of **3b** with

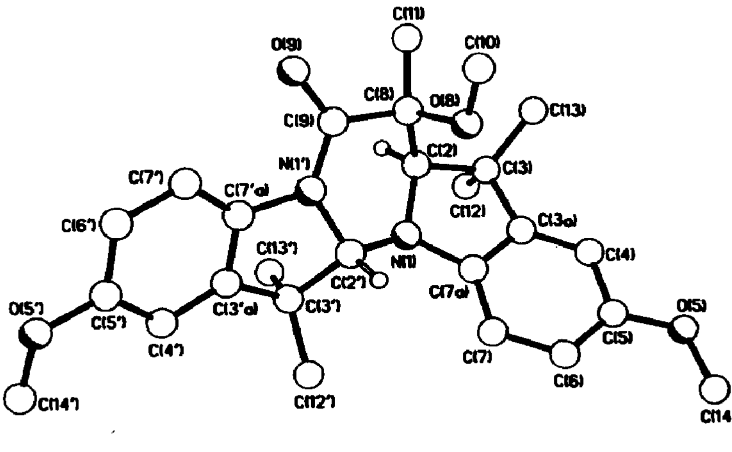


Fig. 1. The molecular structure of **5b** showing atom numbering.

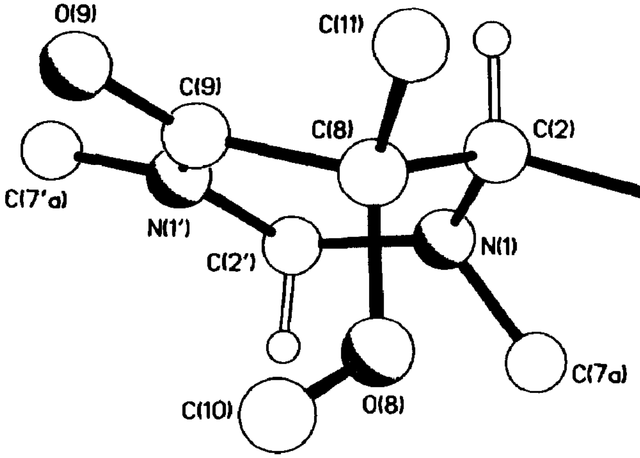
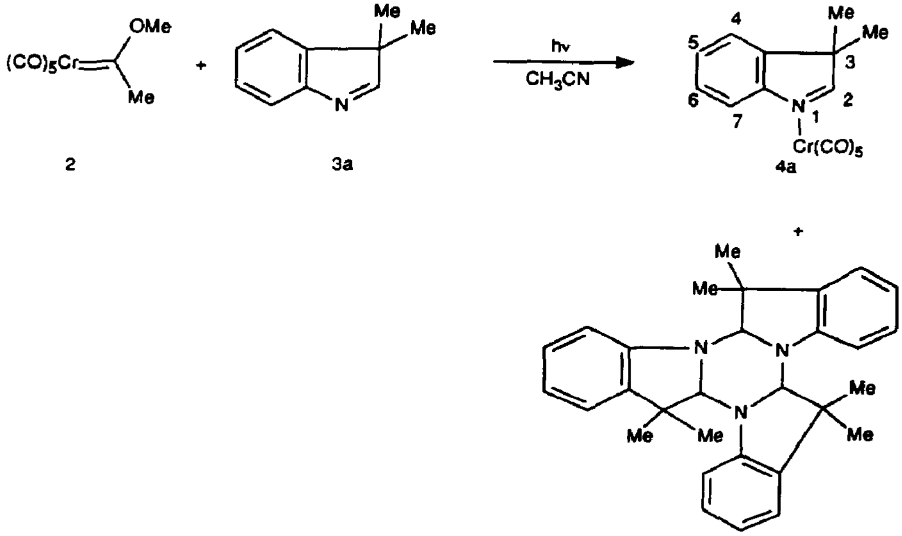


Fig. 2. Perspective view showing the half-chair conformation of the central ring.

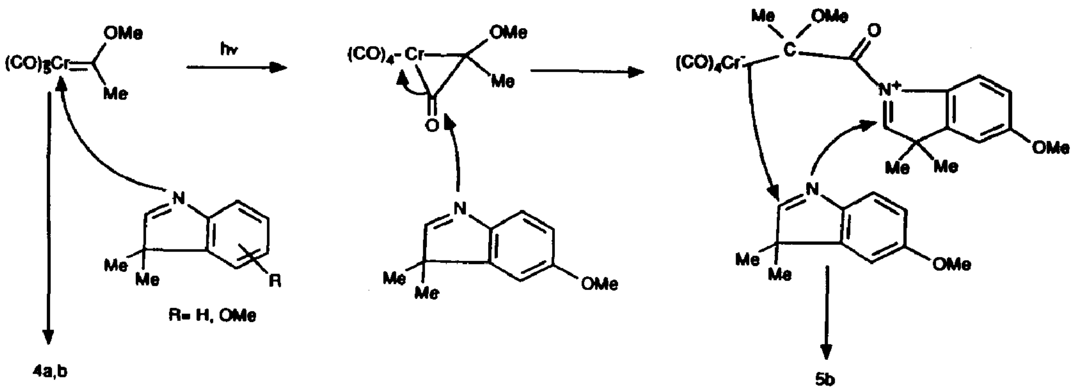
one chromium-complexed ketenic species photochemically generated from **2**, and subsequent 1,4-dipolar cyclization between the adduct and another molecule of **3b**.

The derivatives **4a** and **4b** arise from attack of the indole nitrogen on the carbenic metal center, followed by displacement of the carbenic ligand (Scheme 4). The latter pathway becomes predominant when strongly basic imines, such as aliphatic ones, are used [8].

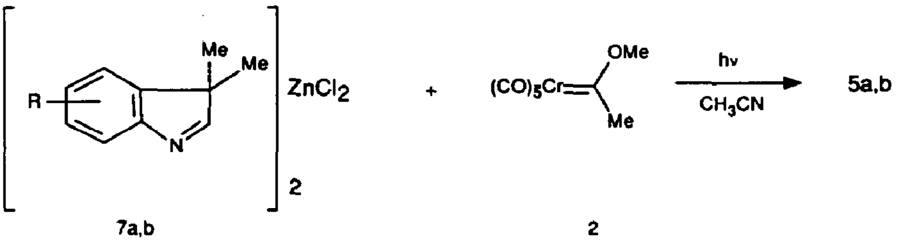
The basicity of imines thus imposes a serious limitation on this type of reaction. For this reason we have investigated the possibility of using Lewis acids to change



Scheme 3



Scheme 4



Scheme 5

the imine basicity. Furthermore a Lewis acid also stabilizes the monomeric form relative to the trimer [9], and could also activate the cycloaddition step.

In the presence of $ZnCl_2$, 3*H*-indoles form stable and isolable complexes in a monomeric form, thus affording a reliable way of evaluating the effects of the presence of a Lewis acid.

The reaction of **2** with $ZnCl_2$-complexed 3*H*-indoles **7a** and **7b** were performed under the conditions used previously with the non-complexed 3*H*-indoles. Crude yields for the 1,3-diazinone-diindolic derivatives were almost quantitative as measured by NMR spectroscopy; isolated product yields were high in all cases. Furthermore, the formation of derivatives **4a** and **4b** was completely inhibited (Scheme 5).

The improvement in yields reflects the sum of all the effects arising from the presence of the Lewis acid.

Further studies are in progress to ascertain whether the results can be generalized to those imines from which the corresponding β-lactams cannot be obtained in satisfactory yields owing to their high basicity.

Experimental

General remarks

Melting points were taken on a Büchi 510 apparatus and are uncorrected. NMR spectra were recorded on a Varian 390 (90 MHz) or Varian XL200 (200 MHz) spectrometers in $CDCl_3$ with TMS as internal standard. IR spectra were recorded on a Perkin Elmer 298 spectrometer. Elemental analyses were performed with a Perkin Elmer 240 analyzer. Ultraviolet irradiation was carried out with a Philips BA15D type 57302 B100 high vapor pressure Hg lamp operating at 125 W. Silica gel for flash chromatography was Merck type 60 (230–470 mesh). Acetonitrile was dried over CaH_2 just prior to use. All reactions were performed under argon.

General procedure

Equimolar amounts of carbene complexes **2** [10] and 3*H*-indoles complexed or not [11] were dissolved in dry and degassed acetonitrile (50 ml/mmol) and the flask was exposed to direct sunlight or in photoreactor. The disappearance of starting carbene **2** was monitored. Reaction times were approximately 8–10 h with sunlight irradiation and 1.5 h with Hg-lamp irradiation. The solvent was taken off on a rotary evaporator and the residue dissolved in methylene chloride. The solution was filtered through a Celite pad and exposed to air and sunlight to eliminate unstable chromium by-products. Filtration through a Celite pad and removal of the solvent under reduced pressure yielded a yellow residue. Purification was by flash chromatography with pentane/ethyl ether (8/2) as eluent.

*Reaction of **3b** with **2** in sunlight.* From 0.71 g (2.85 mmol) of **2** and 0.5 g (2.85 mmol) of **3b** the following products were obtained (yields %):

3b: 0.13 g (20%).

4b: 0.217 g (21%). M.p. 94–95 °C (pentane −78 °C). Found: C, 52.28; H, 3.52; N, 3.77. $C_{16}H_{13}CrNO_6$ calc.: C, 52.32; H, 3.57; N, 3.81%. MS: m/e 367 (M^+); in addition to the molecular peak the mass spectrum showed fragment ions at m/e values corrisponding to consecutive loss of five CO groups. ^1H NMR (ppm): 1.35 (s, 6H, CH_3); 3.85 (s, 3H, OCH_3); 6.8–7 (m, 2H, H(4), H(6)); 7.65–7.8 (d, 1H, H(7); 8.15 (s, 1H, H(2)). IR (cm^{-1}, Nujol): 2080, 1990–1880, ν(CO).

5b: 0.127 g (10%). M.p. 213–215 °C (acetone). Found: C, 71.78; H, 7.49; N, 6.33. $C_{26}H_{32}N_2O_4$ calc.: C, 71.53; H, 7.39; N, 6.42%. MS: m/e 436 (M^+). ^1H NMR (ppm): 1.1 (s, 3H, CH_3); 1.35 (s, 3H, CH_3); 1.55 (s, 3H, CH_3); 1.6 (s, 3H, CH_3); 1.65 (s, 3H, CH_3); 3.1 (s, 3H, C(7)OCH_3); 3.5 (s, 1H, C(7)–CH–C(8)); 3.75–3.85 (2s, 6H, C(10)OCH_3, C(2)OCH_3); 5.25 (s, 1H, N(5)–CH–N(13)); 6.37(d, 1H, C(12)H); 6.58 (bd, 1H, C(10)H); 6.63 (bs, 1H, C(9)H); 6.76 (bs, 1H, C(1)H); 6.79 (bd, 1H, C(3)H); 8.01 (d, 1H, C(4)H). ^{13}C NMR (ppm): 165.94 (C(6)); 157.38 (C(2)); 152.84 (C(10)); 144.00 (C(1)–*C*–C(14)); 140.99 (N(13)–*C*–C(12)); 139.80 (C(8)–*C*–C(9)); 132.68 (C(4)–*C*–N(5)); 118.51 (C(12)); 111.71 (C(11)); 110.21 (C(3)); 108.77 (C(9)); 108.55 (C(1)); 104.25 (C(4)); 81.75 (N(5)–*C*–N(13)); 78.86 (C(7)); 77.69 (N(13)–*C*–C(8)); 55.80–52.03 (C(2)–O–CH_3, C(7)–O–CH_3, C(10)–O–CH_3); 47.20–44.81 (C(14), C(8)); 33.53–23.20 (C(14)$(CH_3)_2$, C(8)$(CH_3)_2$); 15.18 (C(7)–CH_3). IR (cm^{-1}, Nujol): 1650, ν(CO).

*Reaction of **3b** under Hg lamp irradiation.* From 0.9 g (3.6 mmol) of **2** and 0.63 g (3.6 mmol) of **3b** were obtained:

4b in 5 and **5b** in 10% yield.

*Reaction of **3a** with **2** in sunlight.* From 0.251 g of **3a** and 0.433 g of **2** were obtained:

3a in 30% yield.

4a in 20% yield. M.p. 108–109 °C (pentane −78 °C). Found: C 3.20; N, 4.03. $C_{15}H_{11}CrNO_5$ calc.: C, 53.42; H, 3.29; N, 4.15%. MS: m/e 337 (M^+). ^1H NMR (ppm): 1.4 (s, 6H, C(3)(CH$_3$)$_2$; 7.1–7.5 (m, 3H arom.); 7.75–7.85 (d, 1H, C(7)H); 8.3 (s, 1H, C(2)H). IR (cm^{-1}, Nujol): 2080, 1990–1880, ν(CO).

6 in 15% yield. M.p. 212° C (lit. 214° C [7]).

*Reaction of **3a** with **2** under Hg-lamp irradiation.* From 0.73 g of **3a** and 1.3 g of **2** were obtained:

4a in 28 and **6** in 12% yield.

*Reaction of **7a** with **2** in sunlight.* From 0.36 g of (**7a**) and 0.2 g of (**2**) were obtained:

5a in 65% yield after flash chromatography. M.p. 255–256° C (acetone). Found: C, 76.31; H, 7.39; N, 7.29. $C_{24}H_{28}N_2O_2$ calc.: C, 76.56; H, 7.49; N, 7.44%). MS: m/e 376 (M^+). ^{1}H NMR (ppm): 1.15 (s, 3H, CH$_3$); 1.3 (s, 3H, CH$_3$); 1.4 (s, 3H, CH$_3$); 1.6 (s, 3H, CH$_3$); 1.65 (s, 3H, CH$_3$); 3.1 (s, 3H, C(7)OCH$_3$); 3.5 (s, 1H, C(7)–CH–C(8)); 5.4 (s, 1H, N(5)–CH–N(13)); 6.5–7.4 (m, 7H arom.); 8–8.1 (d, 1H, C(4)).

Table 1

Atom coordinates (×10^4) and temperature factors (Å^2 ×10^3) with estimated standard deviations in parentheses

Atom	x	y	z	U_{eq} [a]
N(1)	4889(2)	1290(1)	1387(1)	33(1)
C(2)	4320(2)	829(1)	1818(2)	34(1)
C(3)	3215(2)	974(1)	2881(2)	36(1)
C(3a)	3950(2)	1457(1)	3303(2)	33(2)
C(4)	3795(2)	1721(1)	4387(2)	38(1)
C(5)	4571(2)	2167(1)	4551(2)	39(1)
O()	4395(2)	2399(1)	5687(1)	52(1)
C(6)	5447(2)	2342(1)	3616(2)	41(1)
C(7)	5608(2)	2071(1)	2511(2)	39(1)
C(7a)	4880(2)	1626(1)	2382(2)	32(1)
C(8)	5876(2)	491(1)	2206(2)	36(1)
O(8)	7002(2)	705(1)	3304(1)	41(1)
C(9)	6888(2)	460(1)	1069(2)	37(1)
O(9)	7588(2)	96(1)	803(1)	55(1)
C(10)	8387(3)	417(1)	3959(2)	52(1)
C(11)	5294(3)	−8(1)	2474(2)	48(1)
C(12)	1324(3)	1024(1)	2147(2)	45(1)
C(13)	3246(3)	650(1)	4067(2)	48(1)
C(14)	5365(3)	2820(1)	5988(2)	62(1)
N(1′)	6962(2)	860(1)	367(1)	35(1)
C(2′)	6175(2)	1320(1)	573(2)	32(1)
C(3′)	5570(2)	1507(1)	−857(2)	34(1)
C(3a′)	7020(2)	1305(1)	−1476(2)	35(1)
C(4′)	7642(2)	1454(1)	−2573(2)	38(1)
C(5′)	8998(2)	1202(1)	−2945(2)	39(1)
O(5′)	9797(2)	1327(1)	−3963(1)	51(1)
C(6′)	9672(2)	802(1)	−2240(2)	41(1)
C(7′)	9060(2)	656(1)	−1144(2)	40(1)
C(7a′)	7741(2)	913(1)	−762(2)	34(1)
C(12′)	5436(3)	2042(1)	−914(2)	44(1)
C(13′)	3822(2)	1289(1)	−1475(2)	45(1)
C(14′)	9029(3)	1694(1)	−4796(2)	63(1)

[a] Equivalent isotropic U defined as one third of the trace of the orthogonalised U_{ij} tensor.

*Reaction of **7a** with **2** under Hg-lamp irradiation.* From 0.72 g of **7a** and 0.4 g of **2** were obtained:

5a in 69% yield.

*Reaction of **7b** with **2** in sunlight.* From 0.49 g of **7b** and 0.25 g of **2** were obtained:

5b in 75% yield.

*Reaction of **7b** with **2** under Hg-lamp irradiation.* From 0.97 g of **7b** and 0.5 g of **2** were obtained:

5b in 75% yield.

*Determination of the structure of **5b***

Crystal data: $C_{26}H_{32}N_2O_4$, $M = 436.6$, monoclinic, a 7.837(3), b 28.352(12), c 10.347(5) Å, β 100.95(3)°, U 2257 Å^3, space group $P2_1/n$, $Z = 4$, D_c 1.28 g cm^{-3}, Cu-radiation, λ 1.54178 Å, $\mu(\text{Cu-}K_\alpha)$ 7 cm^{-1}, $F(000) = 936$. Data were measured on a Nicolet R3m diffractometer with Cu-K_α radiation (graphite monochromator) using ω-scans. 3044 Independent reflections were measured ($2\theta \leqslant 116°$), of which 2831 had $|F_o| > 3\sigma(|F_o|)$ and were considered to be observed. The data were corrected for Lorentz and polarisation factors; no absorption correction was applied. The structure was solved by direct methods. The non-hydrogen atoms were refined anisotropically. The hydrogen atoms were placed in calculated positions, C–H 0.96 Å, and assigned isotropic thermal parameters, $U(\text{H}) = 1.2\ U_{eq}(\text{C})$, and allowed to ride on their parent carbon atoms. The methyl groups were refined as rigid bodies. Refinement was by block-cascade, full-matrix least-squares to $R = 0.041$, $R_w = 0.048$ ($w^{-1} = \sigma^2(F) + 0.000397F^2$). The maximum and minimum residual electron densities in the final ΔF map were 0.21 and -0.22 eÅ^{-3} respectively. The mean and maximum shift/error in the final refinement were 0.007 and 0.042 respectively. Computations were carried out on an Eclipse S140 computer using the SHELXTL program system. The atomic coordinates are listed in Table 1,

Table 2

Bond lengths (Å) with e.s.d.'s in parentheses

N(1)–C(2)	1.478(2)	N(1)–C(7a)	1.404(2)
N(1)–C(2′)	1.433(2)	C(2)–C(3)	1.578(3)
C(2)–C(8)	1.543(2)	C(3)–C(3a)	1.518(2)
C(3)–C(12)	1.538(2)	C(3)–C(13)	1.528(3)
C(3a)–C(4)	1.374(3)	C(3a)–C(7a)	1.391(3)
C(4)–C(5)	1.398(2)	C(5)–O(5)	1.377(2)
C(5)–C(6)	1.381(3)	O(5)–C(14)	1.419(3)
C(6)–C(7)	1.402(3)	C(7)–C(7a)	1.379(2)
C(8)–O(8)	1.435(2)	C(8)–C(9)	1.541(3)
C(8)–C(11)	1.527(3)	O(8)–C(10)	1.423(2)
C(9)–O(9)	1.227(2)	C(9)–N(1′)	1.353(2)
N(1′)–C(2′)	1.475(2)	N(1′)–C(7a′)	1.425(2)
C(2′)–C(3′)	1.559(2)	C(3′)–C(3a′)	1.519(3)
C(3′)–C(12′)	1.520(2)	C(3′)–C(13′)	1.528(2)
C(3a′)–C(4′)	1.383(3)	C(3a′)–C(7a′)	1.392(2)
C(4′)–C(5′)	1.394(3)	C(5′)–O(5′)	1.371(2)
C(5′)–C(6′)	1.397(3)	O(5′)–C(14′)	1.410(3)
C(6′)–C(7′)	1.376(3)	C(7′)–C(7′a)	1.383(3)

Table 3

Bond angles (°) with e.s.d.'s in parentheses

C(2)–N(1)–C(7a)	109.6(1)	C(2)–N(1)–C(2′)	121.0(1)
C(7a)–N(1)–C(2′)	119.5(1)	N(1)–C(2)–C(3)	102.5(1)
N(1)–C(2)–C(8)	111.1(1)	C(3)–C(2)–C(8)	119.4(1)
C(2)–C(3)–C(3a)	101.9(1)	C(2)–C(3)–C(12)	106.6(1)
C(3a)–C(3)–C(12)	109.6(1)	C(2)–C(3)–C(13)	118.5(1)
C(3a)–C(3)–C(13)	111.3(1)	C(12)–C(3)–C(13)	108.6(2)
C(3)–C(3a)–C(4)	129.8(2)	C(3)–C(3a)–C(7a)	109.5(1)
C(4)–C(3a)–C(7a)	120.6(2)	C(3a)–C(4)–C(5)	119.3(2)
C(4)–C(5)–O(5)	115.0(2)	C(4)–C(5)–C(6)	120.2(2)
O(5)–C(5)–C(6)	124.8(2)	C(5)–O(5)–C(14)	116.9(2)
C(5)–C(6)–C(7)	120.5(2)	C(6)–C(7)–C(7a)	118.7(2)
N(1)–C(7a)–C(3a)	110.1(1)	N(1)–C(7a)–C(7)	129.2(2)
C(3a)–C(7a)–C(7)	120.7(2)	C(2)–C(8)–O(8)	106.2(1)
C(2)–C(8)–C(9)	109.4(1)	O(8)–C(8)–C(9)	107.4(1)
C(2)–C(8)–C(11)	112.0(2)	O(8)–C(8)–C(11)	114.1(1)
C(9)–C(8)–C(11)	107.6(1)	C(8)–O(8)–C(10)	115.6(1)
C(8)–C(9)–O(9)	122.4(2)	C(8)–C(9)–N(1′)	116.4(1)
O(9)–C(9)–N(1′)	121.2(2)	C(9)–N(1′)–C(2′)	126.9(2)
C(9)–N(1′)–C(7a′)	126.7(1)	C(2′)–N(1′)–C(7a′)	106.4(1)
N(1)–C(2′)–N(1′)	113.2(1)	N(1)–C(2′)–C(3′)	117.1(1)
N(1′)–C(2′)–C(3′)	102.9(1)	C(2′)–C(3′)–C(3a′)	98.9(1)
C(2′)–C(3′)–C(12′)	112.5(1)	C(3a′)–C(3′)–C(12′)	114.3(2)
C(2′)–C(3′)–C(13′)	110.3(1)	C(3a′)–C(3′)–C(13′)	110.7(1)
C(12′)–C(3′)–C(13′)	109.8(1)	C(3′)–C(3a′)–C(4′)	129.7(2)
C(3′)–C(3a′)–C(7a′)	109.8(2)	C(4′)–C(3a′)–C(7a′)	120.5(2)
C(3a′)–C(4′)–C(5′)	118.5(2)	C(4′)–C(5′)–O(5′)	124.3(2)
C(4′)–C(5′)–C(6′)	120.2(2)	O(5′)–C(5′)–C(6′)	115.4(2)
C(5′)–O(5′)–C(14′)	117.1(2)	C(5′)–C(6′)–C(7′)	121.3(2)
C(6′)–C(7′)–C(7a′)	118.2(2)	N(1′)–C(7a′)–C(3a′)	109.0(2)
N(1′)–C(7a′)–C(7′)	129.7(2)	C(3a′)–C(7a′)–C(7′)	121.3(2)

and the bond lengths and angles in Tables 2 and 3. Tables of thermal parameters and torsion angles, and a list of observed and calculated structure factors are available from the authors.

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