

Table 1. Positional parameters and equivalent isotropic displacement parameters (\AA^2) for phthalimide
$$B_{eq} = (4/3)[a^2B_{1,1} + b^2B_{2,2} + c^2B_{3,3} + ab(\cos\gamma)B_{1,2} + ac(\cos\beta)B_{1,3} + bc(\cos\alpha)B_{2,3}].$$

	x	y	z	B_{eq}
O1	0.2685 (5)	-0.0061 (3)	0.16896 (8)	4.49 (4)
O2	-0.2157 (6)	-0.2170 (3)	-0.00158 (7)	4.44 (4)
N3	0.0403 (6)	-0.0734 (3)	0.07739 (8)	3.48 (4)
C4	0.1201 (7)	-0.1060 (3)	0.1361 (1)	3.13 (5)
C5	-0.1207 (7)	-0.2130 (3)	0.0498 (1)	3.16 (5)
C6	-0.1533 (6)	-0.3506 (3)	0.0949 (1)	2.76 (5)
C7	-0.2873 (7)	-0.5174 (3)	0.0912 (1)	3.40 (5)
C8	-0.2748 (7)	-0.6176 (3)	0.1417 (1)	3.92 (6)
C9	-0.1317 (7)	-0.5534 (4)	0.1934 (1)	3.73 (6)
C10	-0.0034 (7)	-0.3858 (3)	0.1971 (1)	3.26 (5)
C11	-0.0094 (6)	-0.2860 (3)	0.1471 (1)	2.65 (4)

Table 2. Bond distances (\AA) and angles ($^\circ$) for phthalimide

O1—C4	1.202 (3)	O2—C5	1.218 (3)
N3—C4	1.388 (3)	N3—C5	1.376 (3)
C4—C11	1.485 (4)	C5—C6	1.478 (4)
C6—C7	1.374 (4)	C6—C11	1.389 (4)
C7—C8	1.385 (4)	C8—C9	1.377 (4)
C9—C10	1.382 (4)	C10—C11	1.373 (4)
C4—N3—C5	112.8 (2)	O1—C4—N3	125.2 (3)
O1—C4—C11	129.4 (3)	N3—C4—C11	105.3 (2)
O2—C5—N3	125.3 (3)	O2—C5—C6	128.6 (3)
N3—C5—C6	106.1 (2)	C5—C6—C7	130.8 (3)
C5—C6—C11	107.8 (2)	C7—C6—C11	121.3 (3)
C6—C7—C8	117.1 (3)	C7—C8—C9	121.4 (3)
C8—C9—C10	121.5 (3)	C9—C10—C11	117.2 (3)
C4—C11—C6	108.0 (2)	C4—C11—C10	130.5 (3)
C6—C11—C10	121.4 (3)		

Related literature. A search through the Cambridge Structural Database yielded the structure of phthalimide; this had been abstracted by *Chemical Abstracts* as the mineral kladnoite (Matzat, 1972a). The results of the present structure determination are

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Structure of a Densely Oxygenated Carbocycle*

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Abstract. (3a*R*-(3a α ,4 α ,5 β [*R**(*S**)),6 α ,6a α))-Methyl α -(cyclohexylhydroxymethyl)-4-[[[(1,1-dimethyl-ethyl)dimethylsilyloxy]tetrahydro-6-hydroxy-2,2-dimethyl-4*H*-cyclopenta-1,3-dioxole-5-acetate, $C_{24}H_{44}O_7Si$, (I), $M_r = 472.69$, orthorhombic, $P2_12_12_1$, $a = 9.496$ (1), $b = 12.508$ (2), $c = 23.317$ (4) \AA , $V =$

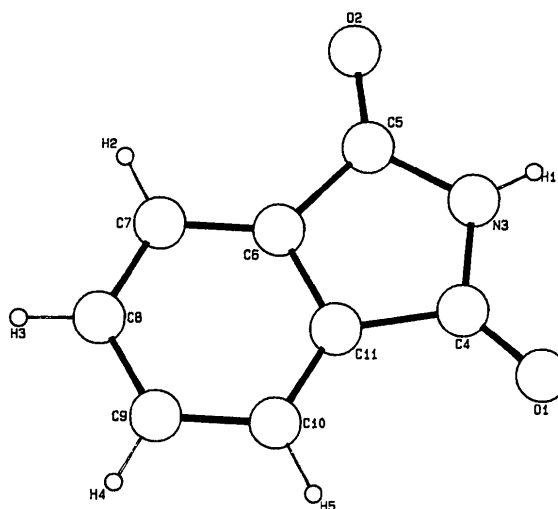


Fig. 1. Molecular structure of phthalimide.

an improvement of the published structure, which had been refined to $R = 0.072$ (Matzat, 1972b).

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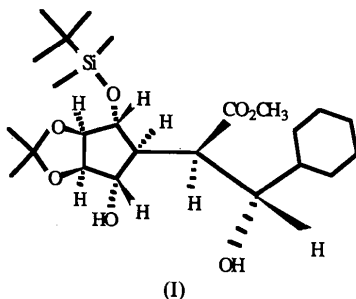
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* Part 2. Part 1: Abboud, Enholm & Trivellas (1992).

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molecules are involved in two kinds of hydrogen bonding: an intramolecular hydrogen bond exists between O6 and O8—H8' [O8—H8'...O6; O8—H8' = 0.76 (4), H8'...O6 = 2.04 (4), O8...O6 = 2.768 (4) Å and O8—H8'...O6 = 161 (5)°]; H6' bonds together two molecules related by a 2₁ screw axis along the *a* axis [O6—H6'...O8 (*x* - 0.5, -*y* + 1.5, -*z*); O6—H6' = 0.84 (5), H6'...O8 = 2.08 (5), O6...O8 = 2.917 (4) Å and O6—H6'...O8 = 175 (4)°]. The latter interaction results in infinite chains of hydrogen-bonded molecules in the *a*-axis direction.

Experimental. Compound (I) was purified by flash chromatography over silica gel. This was followed by treatment of the colorless thick oil with hot pentanes.



Subsequent cooling of the solution to 273 K produced colorless needles which had an m.p. of 365–366.5 K (uncorrected). The crystal used in the X-ray analysis was obtained by slow evaporation from methanol and had dimensions 0.27 × 0.31 × 0.46 mm. Data were collected at 298 K on a Siemens *P3m/E* diffractometer equipped with a graphite monochromator utilizing Mo *K*α radiation. 25 reflections with 20 ≤ 2θ ≤ 22° were used to refine the cell parameters. 7237 reflections were collected using the ω-scan method [two equivalent sets: (*h* 0 → 12, *k* 0 → 16, *l* 0 → 30) and (*h* 0 → 12, *k* -16 → 0, *l* -30 → 0)], of which 3599 were unique, *R*_{int} = 0.0275; 2θ range 3 → 55°; 1.2° ω scan at 3–6° min⁻¹, depending on intensity. Four reflections (331, 323, 323, 222) were measured every 96 reflections to monitor instrument and crystal stability (maximum correction on *I* was < 1.01%). Absorption corrections were applied based on measured crystal faces using *SHELXTL-Plus* (Sheldrick, 1986); minimum and maximum transmission 0.911 and 0.946, respectively.

The structure was solved by direct methods in *SHELXTL-Plus* from which the locations of all non-H atoms were obtained. The structure was refined (*SHELX76*; Sheldrick, 1976) using full-matrix least squares and the positions of all non-methyl H atoms were determined from a difference Fourier map; the methyl H atoms were calculated in idealized positions and their isotropic thermal parameters fixed. The non-H atoms were

treated anisotropically, whereas the non-methyl H atoms were refined with isotropic thermal parameters. During the anisotropic refinement, it became evident from the thermal parameters of Si, C17, C18, C21 and C22 that the *tert*-butylsilyl group is disordered (TBS). Si, C17 and C18 exhibit large and parallel displacements in the plane containing them; C21 and C22 have similar displacements parallel to this plane. The best model that describes this disorder has two sets of the TBS group with C19 and C20 approximately in ordered positions (see Fig. 1). 415 parameters were refined and $\sum w(|F_o| - |F_c|)^2$ was minimized; $w = 1/(\sigma|F_o|)^2$, $\sigma(F_o) = 0.5 kI^{-1/2}\{[\sigma(I)]^2 + (0.02I)^2\}^{1/2}$, $I(\text{intensity}) = (I_{\text{peak}} - I_{\text{background}}) \times (\text{scan rate})$, and $\sigma(I) = (I_{\text{peak}} + I_{\text{background}})^{1/2} \times (\text{scan rate})$, k is the correction for decay and Lp effects, 0.02 is a factor used to down-weight intense reflections and to account for instrument instability. Final *R* = 0.0475, *wR* = 0.0579 (*R*_{all} = 0.0744, *wR*_{all} = 0.0650) for 2409 reflections having $I \geq 3\sigma(I)$, and goodness of fit = 1.97. Maximum $\Delta/\sigma = 0.001$ in the final refinement cycle and the minimum and maximum peaks in the ΔF map were -0.17 and 0.21 e Å⁻³, respectively. The linear absorption coefficient was calculated using values from *International Tables for X-ray Crystallography* (1974, Vol. IV, p. 55). Scattering factors for non-H atoms were taken from Cromer & Mann (1968) with anomalous-dispersion corrections from Cromer & Liberman (1970), while those of H atoms were from Stewart, Davidson & Simpson (1965). The positional parameters and the equivalent isotropic thermal parameters of the non-H atoms are listed in Table 1; * bond lengths and angles are in Table 2. The thermal ellipsoids drawing (*SHELXTL-Plus*; Sheldrick, 1986) of the molecule with the atom-labelling scheme is given in Fig. 1.

Related literature. Similar densely oxygenated cyclopentane rings have been prepared by treatment of an acyclic carbohydrate template with SmI₂ (Enholm & Trivellas, 1989). These highly functionalized rings have also been applied to natural products synthesis (Enholm, Satici & Trivellas, 1989). The crystal structure of a related carbocycle has been studied in this laboratory (Abboud, Enholm & Trivellas, 1992).

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* Lists of anisotropic thermal parameters, H-atom positional parameters, bond lengths and angles involving H atoms and structure-factor amplitudes have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55073 (20 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: ST0553]

Table 1. Fractional coordinates and equivalent isotropic thermal parameters (\AA^2) for the non-H atoms of compound (I)

$U_{eq} = (1/3)\sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{A}_{ij}$, where \mathbf{A}_{ij} is the dot product of the i th and j th direct-space unit-cell vectors.

	<i>x</i>	<i>y</i>	<i>z</i>	U_{eq}
Si	0.7732 (7)	0.4057 (3)	0.18831 (14)	0.056 (2)
Si'	0.8463 (8)	0.4165 (3)	0.1884 (2)	0.0535 (15)
O2	0.7797 (3)	0.4863 (2)	0.13238 (10)	0.0577 (10)
O3	0.7841 (4)	0.3253 (2)	0.01900 (11)	0.0743 (14)
O4	0.8508 (3)	0.4144 (2)	-0.05990 (11)	0.0647 (11)
O6	0.8707 (3)	0.6784 (2)	-0.03995 (11)	0.0509 (10)
O8	1.0692 (3)	0.7905 (2)	0.02280 (12)	0.0530 (9)
O15a	1.1662 (4)	0.5315 (3)	0.0578 (2)	0.0911 (14)
O15b	1.2184 (4)	0.6470 (3)	0.1247 (2)	0.1001 (12)
C1	0.8743 (4)	0.5957 (3)	0.05615 (13)	0.0398 (11)
C2	0.8451 (5)	0.4818 (3)	0.07746 (14)	0.0480 (14)
C3	0.7517 (4)	0.4333 (3)	0.0311 (2)	0.0476 (13)
C4	0.7995 (5)	0.3146 (3)	-0.0413 (2)	0.062 (2)
C5	0.7880 (4)	0.4926 (3)	-0.02435 (14)	0.0433 (12)
C6	0.8920 (4)	0.5819 (3)	-0.00908 (13)	0.0400 (11)
C7	0.9857 (4)	0.6549 (3)	0.0905 (2)	0.0483 (13)
C8	0.9999 (4)	0.7768 (3)	0.0769 (2)	0.0505 (13)
C9	0.8662 (4)	0.8404 (3)	0.0827 (2)	0.0469 (12)
C10	0.7920 (6)	0.8227 (4)	0.1401 (2)	0.063 (2)
C11	0.6635 (6)	0.8907 (4)	0.1475 (3)	0.077 (2)
C12	0.6952 (7)	1.0082 (4)	0.1398 (3)	0.083 (2)
C13	0.7659 (6)	1.0294 (4)	0.0832 (2)	0.069 (2)
C14	0.8966 (5)	0.9592 (3)	0.0749 (2)	0.0555 (14)
C15	1.1308 (5)	0.6023 (3)	0.0873 (2)	0.0602 (14)
C16	1.3591 (6)	0.6070 (5)	0.1268 (4)	0.136 (3)
C17	0.608 (2)	0.3196 (12)	0.1868 (6)	0.115 (6)
C17'	0.773 (3)	0.2806 (11)	0.1793 (7)	0.18 (2)
C18	0.933 (2)	0.3250 (14)	0.1878 (6)	0.146 (9)
C18'	1.0405 (14)	0.434 (2)	0.2000 (6)	0.144 (8)
C19	0.7535 (6)	0.4843 (5)	0.2518 (2)	0.079 (2)
C20	0.7488 (6)	0.4130 (5)	0.3048 (2)	0.099 (3)
C21	0.635 (2)	0.5607 (11)	0.2490 (6)	0.092 (6)
C21'	0.591 (2)	0.469 (3)	0.2328 (8)	0.20 (2)
C22	0.888 (2)	0.568 (2)	0.2589 (7)	0.153 (10)
C22'	0.793 (5)	0.583 (2)	0.2613 (11)	0.034 (3)
C23	0.9106 (7)	0.2304 (4)	-0.0546 (3)	0.111 (3)
C24	0.6601 (6)	0.2892 (4)	-0.0685 (2)	0.087 (2)

Table 2. Bond lengths (\AA) and angles ($^\circ$) for the non-H atoms of compound (I)

<i>A</i>	<i>B</i>	<i>C</i>	<i>A—B</i>	<i>A—B—C</i>
O2	Si	C17	1.650 (4)	111.3 (5)
O2	Si	C18		107.6 (6)
C17	Si	C18	1.90 (2)	111.8 (7)
C17	Si	C19		103.9 (5)
C18	Si	C19	1.82 (2)	113.4 (6)
C19	Si	O2	1.787 (6)	108.8 (3)
O2	Si'	C17'	1.693 (5)	104.2 (7)
O2	Si'	C18'		114.8 (6)
C17'	Si'	C18'	1.85 (2)	119.3 (12)
C17'	Si'	C19		108.8 (8)
C18'	Si'	C19	1.88 (2)	106.8 (6)
C19	Si'	O2	1.918 (7)	101.2 (3)
C2	O2	Si'	1.424 (4)	120.7 (3)
C2	O2	Si		134.7 (3)
C3	O3	C4	1.413 (4)	108.1 (3)
C4	O3		1.420 (5)	
C4	O4		1.407 (5)	
C4	O4	C5	1.414 (4)	
C6	O6		1.420 (4)	
C8	O8		1.433 (5)	
C15	O15a		1.170 (6)	
C15	O15b	C16	1.329 (6)	117.4 (4)
C16	O15b		1.427 (7)	
C2	C1	C6	1.534 (5)	103.6 (3)
C6	C1	C7	1.540 (4)	120.0 (3)
C7	C1	C2	1.519 (5)	114.0 (3)
C3	C2	O2	1.524 (5)	113.6 (3)
C3	C2	C1		104.2 (3)
O2	C2	C1		109.5 (3)
C5	C3	O3	1.530 (5)	104.2 (3)
C5	C3	C2		106.0 (3)
O3	C3	C2		113.4 (3)
C23	C4	C24	1.523 (7)	112.2 (4)
C23	C4	O3		109.7 (4)
C23	C4	O4		108.1 (4)
C24	C4	O3	1.502 (7)	110.3 (4)
C24	C4	O4		111.2 (4)
O3	C4	O4		104.9 (3)
C6	C5	O4	1.533 (5)	111.6 (3)
C6	C5	C3		107.6 (3)
O4	C5	C3		104.8 (3)
O6	C6	C1		112.9 (3)
O6	C6	C5		114.2 (3)
C1	C6	C5		103.9 (3)
C8	C7	C15	1.564 (5)	109.4 (3)
C8	C7	C1		115.3 (3)
C15	C7	C1	1.529 (6)	113.1 (3)
C9	C8	O8	1.504 (6)	113.8 (3)
C9	C8	C7		115.2 (3)
O8	C8	C7		109.5 (3)
C10	C9	C14	1.529 (6)	109.4 (3)
C10	C9	C8		113.0 (3)
C14	C9	C8	1.524 (5)	110.2 (3)
C11	C10	C9	1.497 (8)	113.2 (4)
C12	C11	C10	1.511 (8)	112.1 (5)
C13	C12	C11	1.503 (8)	111.4 (5)
C14	C13	C12	1.532 (7)	111.8 (4)
C9	C14	C13		113.0 (4)
O15a	C15	O15b		121.6 (4)
O15a	C15	C7		127.8 (4)
O15b	C15	C7		110.5 (4)
C20	C19	C21	1.525 (7)	113.2 (7)
C20	C19	C22		108.1 (7)
C20	C19	Si		110.6 (4)
C21	C19	C22	1.47 (2)	100.5 (10)
C21	C19	Si		113.6 (6)
C22	C19	Si	1.66 (2)	110.4 (7)
C20	C19	C21'	1.525 (7)	97.2 (9)
C20	C19	C22'		115.0 (11)
C20	C19	Si'		112.3 (4)
C21'	C19	C22'	1.62 (2)	116. (2)
C21'	C19	Si'		100.1 (8)
C22'	C19	Si'	1.31 (2)	115. (2)

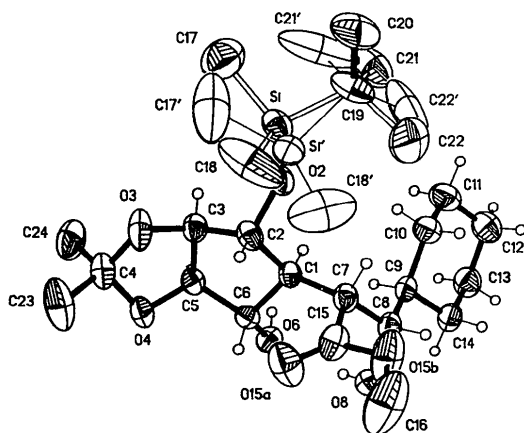


Fig. 1. Molecular structure of (I), with 50% probability ellipsoids and showing the atom-numbering scheme. The disordered TBS group is represented by two subgroups: (Si, C17–C22) and (Si', C17'–C22'). The site-occupation factor of the Si subgroup is 0.54 (1) and that of the Si' subgroup is 0.46 (1 – 0.54).

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Structure of 3-Methoxytyramine Perchlorate

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Abstract. 2-(4-Hydroxy-3-methoxyphenyl)ethylammonium perchlorate, C₉H₁₄NO₂⁺.ClO₄⁻, *M_r* = 267.67, triclinic, *P* $\bar{1}$, *a* = 8.075 (2), *b* = 10.086 (3), *c* = 7.465 (2) Å, α = 92.17 (3), β = 93.91 (3), γ = 96.58 (2)°, *V* = 601.9 (3) Å³, *Z* = 2, *D_m* = 1.483 (1), *D_x* = 1.477 g cm⁻³, λ (Mo *K*α) = 0.71069 Å, μ = 3.29 cm⁻¹, *F*(000) = 278, *T* = 296 K, final *R* = 0.068 for 891 reflections [*I* > 3σ(*I*)]. The molecule has a fully extended amino side chain of a *trans* configuration, and the plane of the side chain is oriented nearly perpendicular to the phenyl ring plane. There is a hydrogen-bonding network involving the 3-methoxy group, the 4-hydroxy group, the protonated amino group and O atoms of the perchlorate ions.

Experimental. Platelets of the title compound were crystallized from 50% methanol solution. A crystal 0.2 × 0.05 × 0.5 mm was used for data collection on a Rigaku AFC-5R automated four-circle diffractometer with graphite-monochromated Mo *K*α radiation. Lattice parameters were determined from 2θ values of 24 reflections (20.4 < 2θ < 34.4°). Intensity data were collected to 2θ = 50.0°, using ω–2θ scan, scan speed 32.0°(ω) min⁻¹, scan width (1.05 + 0.30tanθ)°, the ratio of peak counting time to background counting time was 2:1; *h* 0–8, *k* –11–11, *l* –8–8. 1900 reflections were measured of which 1844 were unique and 891 with *I* > 3σ(*I*) were used for the analysis. Three reference reflections monitored every 100 reflections showed no crystal deterioration. Lorentz, polarization and absorption (maximum and minimum transmission factors 0.87, 1.11) corrections were applied. The structure was solved by direct methods with *MITHRIL* (Gilmore, 1984) and *DIRDIF* (Beurskens, 1984), and refined by least squares with anisotropic thermal parameters for all non-H atoms; H atoms were located from a

Table 1. Atomic coordinates and equivalent isotropic thermal parameters (Å²)

$$B_{\text{eq}} = (B_{11}a^2 + B_{22}b^2 + B_{33}c^2 + B_{12}ac\cos\beta).$$

	<i>x</i>	<i>y</i>	<i>z</i>	<i>B_{eq}</i>
Cl(1)	0.7885 (5)	0.0854 (3)	0.2555 (4)	4.2 (1)
O(1)	0.447 (1)	0.7971 (6)	0.274 (1)	4.8 (4)
O(2)	0.685 (1)	0.6431 (6)	0.3247 (8)	4.1 (4)
O(3)	0.900 (1)	0.055 (1)	0.140 (1)	14 (1)
O(4)	0.751 (2)	–0.007 (1)	0.362 (2)	23 (1)
O(5)	0.836 (2)	0.197 (1)	0.353 (2)	16 (1)
O(6)	0.637 (2)	0.080 (1)	0.142 (2)	12 (1)
N(1)	0.255 (2)	0.0181 (9)	0.241 (2)	4.4 (6)
C(1)	0.337 (1)	0.3897 (8)	0.164 (1)	3.1 (5)
C(2)	0.493 (2)	0.4423 (9)	0.228 (1)	3.0 (5)
C(3)	0.531 (1)	0.5775 (8)	0.264 (1)	2.8 (5)
C(4)	0.411 (2)	0.6617 (9)	0.241 (1)	3.1 (5)
C(5)	0.254 (2)	0.607 (1)	0.180 (2)	4.7 (7)
C(6)	0.211 (2)	0.474 (1)	0.141 (1)	3.5 (5)
C(7)	0.300 (2)	0.241 (1)	0.123 (1)	4.0 (6)
C(8)	0.291 (2)	0.1639 (9)	0.288 (1)	3.5 (5)
C(9)	0.815 (2)	0.565 (2)	0.368 (2)	5.0 (7)

Table 2. Bond lengths (Å), bond angles (°) and hydrogen-bond geometry (Å)

Cl(1)—O(3)	1.339 (9)	C(1)—C(2)	1.36 (1)
Cl(1)—O(4)	1.27 (1)	C(1)—C(6)	1.40 (1)
Cl(1)—O(5)	1.326 (9)	C(1)—C(7)	1.51 (1)
Cl(1)—O(6)	1.43 (1)	C(2)—C(3)	1.37 (1)
O(1)—C(4)	1.37 (1)	C(3)—C(4)	1.37 (1)
O(2)—C(3)	1.38 (1)	C(4)—C(5)	1.36 (2)
O(2)—C(9)	1.41 (1)	C(5)—C(6)	1.37 (2)
N(1)—C(8)	1.49 (1)	C(7)—C(8)	1.49 (1)
O(3)—Cl(1)—O(4)	113 (1)	O(2)—C(3)—C(2)	126.5 (9)
O(3)—Cl(1)—O(5)	113.8 (7)	O(2)—C(3)—C(4)	112.9 (9)
O(3)—Cl(1)—O(6)	102.7 (6)	C(2)—C(3)—C(4)	121 (1)
O(4)—Cl(1)—O(5)	108.1 (9)	O(1)—C(4)—C(3)	122 (1)
O(4)—Cl(1)—O(6)	102 (1)	O(1)—C(4)—C(5)	121 (1)
O(5)—Cl(1)—O(6)	117.0 (8)	C(3)—C(4)—C(5)	118 (1)
C(3)—O(2)—C(9)	118.0 (9)	C(4)—C(5)—C(6)	124 (1)
C(2)—C(1)—C(6)	119.5 (9)	C(1)—C(6)—C(5)	117 (1)
C(2)—C(1)—C(7)	120 (1)	C(1)—C(7)—C(8)	112.7 (8)
C(6)—C(1)—C(7)	121 (1)	N(1)—C(8)—C(7)	110.7 (9)
C(1)—C(2)—C(3)	121.0 (9)		
<i>D</i>	<i>A</i>	Symmetry	<i>D</i> ... <i>A</i>
O(1)	O(2)	(i)	2.62 (1)
N(1)	O(1)	(ii)	2.87 (1)
N(1)	O(3)	(iii)	2.98 (2)
N(1)	O(4)	(iv)	2.98 (2)

Symmetry code: (i) *x*, *y*, *z*; (ii) *x*, –1 + *y*, *z*; (iii) –1 + *x*, *y*, *z*; (iv) –1 – *x*, –*y*, –1 – *z*.