Table 5 (cont.)

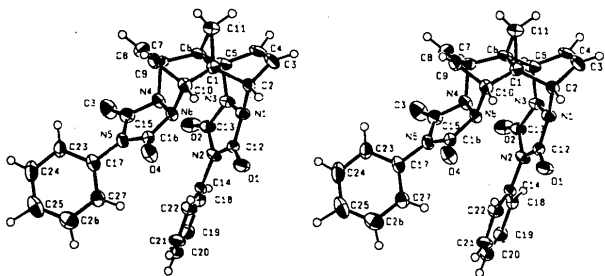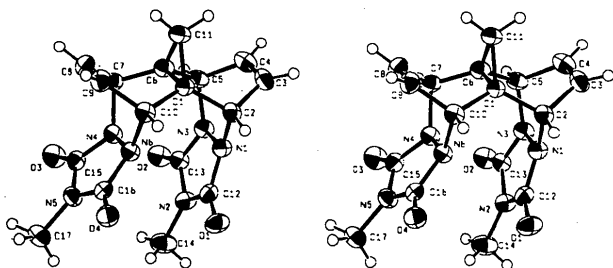|	(1)	(2)	(3)
O(2)—C(13)—N(2)	126.9 (1)	128.3 (4)	127.8 (4)
O(2)—C(13)—N(3)	127.9 (1)	127.5 (4)	127.5 (3)
N(2)—C(13)—N(3)	105.1 (1)	104.1 (3)	104.6 (3)
O(3)—C(15)—N(4)	126.9 (2)	126.4 (4)	125.8 (3)
O(3)—C(15)—N(5)	126.8 (1)	127.5 (4)	127.1 (4)
N(4)—C(15)—N(5)	106.2 (1)	106.1 (3)	106.9 (3)
O(4)—C(16)—N(5)	127.7 (1)	127.0 (4)	127.1 (3)
O(4)—C(16)—N(6)	126.9 (1)	126.2 (4)	126.2 (4)
N(5)—C(16)—N(6)	105.3 (1)	106.7 (3)	106.6 (3)
N(2)—C(14)—C(18)		120.2 (3)	120.1 (3)
N(2)—C(14)—C(22)		118.6 (3)	119.7 (4)
N(5)—C(17)—C(23)		120.0 (4)	120.4 (3)
N(5)—C(17)—C(27)		119.1 (4)	118.3 (3)

Fig. 1. Stereoscopic view of (1).

Fig. 2. Stereoscopic view of (2).

Compounds (2) and (3) also differ in the rotation of the phenyl ring with respect to the plane of the triazolinedione fragment: 49.1 (4) and 38.2 (4)° at N(2) for (2) and (3), respectively, and 61.0 (4) and 51.7 (4)° at N(5).

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9-Chloro-2-ethoxy-6-nitroacridine

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Abstract. 9-Chloro-2-ethoxy-6-nitroacridine (CENA) chloroform solvate, $C_{15}H_{11}ClN_2O_3.CHCl_3$, $M_r = 422.10$, triclinic, $P\bar{1}$, $a = 7.349$ (1), $b = 10.613$ (2), $c = 11.721$ (2) Å, $\alpha = 84.42$ (1), $\beta = 79.42$ (1), $\gamma = 83.23$ (1)°, $V = 889.7$ (4) Å³, $Z = 2$, $D_x = 1.576$ g cm⁻³, $\lambda(Mo\ K\alpha) = 0.71073$ Å, $\mu = 6.85$ cm⁻¹, $F(000) = 428$, $T = 293$ K, final $R = 0.062$ for 3027 observed reflections. The chloroform solvate crystals are stable at standard conditions, desolvate around 335 K and melt at 464 K. The acridine rings are arranged in stacks with a ring–ring stacking distance of 3.6 Å.

Introduction. This report is part of our ongoing investigation of acridine derivatives substituted at the 9 position (Toma, Ray, Stowell & Byrn, 1990;

Stowell, Toma & Byrn, 1991). These acridines are being linked to oligonucliotides in the hope of synthesizing new anti-AIDS or anti-cancer agents. The biological activity of acridine derivatives is hypothesized to be related to the intercalation of the planar chromophore between the planar pyrimidine–purine base pairs (Lerman, 1961). The crystal structure of the title compound has been determined so that it may be used for molecular modeling studies. In addition, other 9-chloroacridine derivatives have been studied crystallographically for potential DNA intercalation (Berman, Stallings, Carrell, Glusker, Neidle, Achari & Taylor, 1979; Achari & Neidle, 1977).

Experimental. The title compound was prepared following a procedure by Albert & Linnell (1936). A yellow needle (0.48 × 0.28 × 0.19 mm), obtained by slow evaporation from chloroform, was used for data collection on an Enraf–Nonius CAD-4 diffractometer, using *SDP* (Frenz, 1978), with graphite-monochromated Mo $K\alpha$ radiation at 293 (1) K and the ω–2θ scan technique (ω varied from 1 to $20°\ \mathrm{min}^{-1}$) to a maximum θ of 27.5°. A total of 4131 unique reflections were measured within the range $-9 \le h \le 9$, $-13 \le k \le 13$, $0 \le l \le 15$, of which 3027 with $I > 3\sigma$ were used for all calculations. Three standard reflections were measured every 97 reflections; no crystal decay was detected. Cell constants were refined with 25 reflections in the range $22 < \theta < 25°$. Lorentz and polarization corrections were applied to the data; no corrections for absorption were necessary. The phase problem was solved by direct methods using *SHELXS*86 (Sheldrick, 1986) and the remaining atoms were located in subsequent difference Fourier syntheses. H atoms were located and their positions and isotropic displacement parameters were refined. The structure was refined by full-matrix least squares using *SHELX*76 (Sheldrick, 1976) where the function minimized was $\sum w(||F_o| - |F_c||)^2$ and the weight w was defined according to the Killean & Lawrence (1969) method with terms of $P = 0.04$ in $w = 4F^2/[\sigma(I)^2 + (PF^2)^2]$.

Atomic scattering factors and the values for f' and f'' were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV, Tables 2.2B and 2.3.1). Anomalous-dispersion effects were included in F_c (Ibers & Hamilton, 1964). The final cycle of refinement included 275 variable parameters and converged with an unweighted agreement factor R of 0.062, a weighted agreement factor wR of 0.091, and $(\Delta/\sigma)_{\mathrm{max}}$ of 0.02. The standard deviation of an observation of unit weight was 1.866. The final difference Fourier map showed no significant residual electron density and the highest peak had a maximum ρ of $0.84\ \mathrm{e\ Å}^{-3}$ with an e.s.d. based on a ΔF of 0.07 (Cruickshank, 1949). Plots of $\sum w(||F_o| - |F_c||)^2$

versus $|F_o|$, reflection order in data collection, $\sin\theta/\lambda$, and various classes of indices, showed no unusual trends.

Discussion. Final positional parameters for all atoms of CENA and the solvent are listed in Table 1.* An *ORTEP*II (Johnson, 1976) thermal-ellipsoid drawing is given in Fig. 1, depicting the numbering scheme. Bond lengths are given in Table 2. The crystal structure of CENA is very similar to the structure of 6,9-dichloro-2-methoxyacridine (DCMA; Neidle, 1982); the two molecules have the same substitution pattern on the ring system. In common with DCMA (Neidle, 1982) and other simple acridines (Jones & Neidle, 1975), the acridine ring of CENA does not show exact mirror symmetry along the C(9)–N(10) axis. This results from the electronic effect of the non-symmetrical ring-substitution pattern. The bond lengths and angles of CENA and DCMA are very similar and the majority of valence angles have the expected trigonal-hybridization values. A significant distortion around C(2) is also observed, with angle C(3)—C(2)—O(2) having a low value (114.0°) and C(1)—C(2)—O(2) a high value (125.8°). A similar distortion was observed by Neidle (1982) for DCMA and can be attributed to repulsion of the nitro substituent of one ring and the ethoxy group of the next ring in a stack.

The CENA molecules form centrosymmetric stacks in the crystal, with a π–π interaction distance of 3.6 Å. As shown in the packing diagram (Fig. 2), the overlap between two molecules involves all three rings of the acridine. Table 3 shows that the outer rings, which do not contain an N atom, are planar. The center ring is slightly buckled making an angle of 0.6° with the two outer rings. This buckling of the acridine ring is a common phenomenon and has been observed previously (Jones & Neidle, 1975). The chloroform molecule is alongside the acridine moiety and the C—H vector points in the direction of the Cl atom which is attached to C(9) of the acridine. The H⋯Cl distance is 3.51 (2) Å, which is larger than the sum of the van der Waals radii, yet the packing arrangement may still indicate a weak polar interaction.

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* Lists of structure factors, anisotropic thermal paramters, bond angles and torsion angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55770 (26 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: GR0204]

$C_{15}H_{11}ClN_2O_3.CHCl_3$

Table 1. Positional parameters and isotropic or equivalent isotropic displacement parameters ($\text{\AA}^2$)

H atoms were refined isotropically. For non-H atoms
$U_{eq} = (4/3)[a^2\beta(1,1) + b^2\beta(2,2) + c^2\beta(3,3) + ab(\cos\gamma)\beta(1,2) + ac(\cos\beta)\beta(1,3) + bc(\cos\alpha)\beta(2,3)]$.

	x	y	z	U_{iso}/U_{eq}
Cl(9)	0.1926 (1)	0.4338 (1)	0.7506 (1)	4.34 (2)
O(2)	0.3804 (3)	0.9042 (2)	0.6544 (2)	3.69 (4)
O(61)	0.0852 (4)	0.0419 (2)	0.3144 (3)	5.91 (6)
O(62)	0.1603 (4)	0.1828 (3)	0.1750 (2)	6.22 (7)
N(6)	0.1353 (4)	0.1464 (3)	0.2771 (3)	4.16 (5)
N(10)	0.2857 (3)	0.5505 (2)	0.3643 (2)	3.02 (4)
C(1)	0.3025 (4)	0.6920 (3)	0.6395 (2)	2.99 (5)
C(2)	0.3530 (4)	0.8089 (3)	0.5935 (2)	3.04 (5)
C(3)	0.3813 (4)	0.8385 (3)	0.4707 (2)	3.21 (5)
C(4)	0.3591 (4)	0.7534 (3)	0.3981 (2)	3.34 (5)
C(5)	0.2139 (4)	0.3496 (3)	0.3232 (3)	3.25 (5)
C(6)	0.1630 (4)	0.2313 (3)	0.3630 (3)	3.37 (5)
C(7)	0.1324 (4)	0.1882 (3)	0.4805 (3)	3.59 (6)
C(8)	0.1525 (4)	0.2665 (3)	0.5602 (3)	3.37 (5)
C(9)	0.2274 (4)	0.4783 (3)	0.6033 (2)	2.88 (5)
C(4a)	0.3064 (3)	0.6299 (2)	0.4411 (2)	2.71 (5)
C(8a)	0.2054 (3)	0.3923 (2)	0.5262 (2)	2.83 (5)
C(9a)	0.2792 (3)	0.6001 (2)	0.5649 (2)	2.66 (5)
C(10a)	0.2363 (3)	0.4333 (2)	0.4052 (2)	2.86 (5)
C(21)	0.3436 (4)	0.8871 (3)	0.7794 (3)	3.85 (6)
C(22)	0.3818 (6)	1.0067 (3)	0.8246 (3)	4.97 (8)
H(1)	0.281 (4)	0.681 (3)	0.712 (3)	1.2 (6)
H(3)	0.407 (4)	0.919 (3)	0.441 (3)	1.6 (6)
H(4)	0.364 (5)	0.770 (4)	0.319 (3)	2.5 (8)
H(5)	0.231 (5)	0.375 (4)	0.245 (3)	3.1 (8)
H(7)	0.104 (6)	0.102 (4)	0.496 (3)	3.2 (8)
H(8)	0.123 (5)	0.241 (3)	0.639 (3)	1.5 (6)
H(211)	0.429 (5)	0.815 (3)	0.803 (3)	2.5 (8)
H(212)	0.215 (4)	0.870 (3)	0.803 (3)	1.3 (6)
H(221)	0.352 (6)	1.001 (4)	0.910 (4)	5 (1)
H(222)	0.524 (6)	1.028 (4)	0.793 (4)	4 (1)
H(223)	0.292 (6)	1.081 (4)	0.791 (4)	3.6 (9)
Cl(101)	0.1416 (2)	0.8296 (1)	1.1179 (1)	8.22 (4)
Cl(102)	0.1817 (2)	0.6014 (2)	0.9995 (1)	9.60 (3)
Cl(103)	0.5002 (3)	0.7112 (2)	1.0399 (1)	8.16 (4)
C(100)	0.2677 (7)	0.6855 (4)	1.0936 (3)	5.57 (9)
H(100)	0.267 (6)	0.645 (4)	1.164 (4)	5 (1)

Table 2. Bond distances ($\text{\AA}$)

|			
Cl(9)—C(9)	1.725 (2)	C(6)—C(7)	1.395 (3)
O(2)—C(2)	1.348 (3)	C(7)—C(8)	1.348 (3)
O(2)—C(21)	1.438 (3)	C(7)—H(7)	0.95 (3)
O(61)—N(6)	1.225 (3)	C(8)—C(8a)	1.433 (3)
O(62)—N(6)	1.209 (3)	C(8)—H(8)	0.93 (3)
N(6)—C(6)	1.471 (3)	C(9)—C(9a)	1.400 (3)
N(10)—C(4a)	1.332 (3)	C(9)—C(8a)	1.387 (3)
N(10)—C(10a)	1.355 (3)	C(4a)—C(9a)	1.436 (3)
C(1)—C(2)	1.368 (3)	C(8a)—C(10a)	1.427 (3)
C(1)—C(9a)	1.417 (3)	C(21)—C(22)	1.496 (3)
C(1)—H(1)	0.83 (3)	C(21)—H(211)	0.98 (3)
C(2)—C(3)	1.425 (3)	C(21)—H(212)	0.97 (3)
C(3)—C(4)	1.342 (3)	C(22)—H(221)	0.99 (3)
C(3)—H(3)	0.92 (3)	C(22)—H(222)	1.09 (3)
C(4)—C(4a)	1.428 (3)	C(22)—H(223)	1.06 (3)
C(4)—H(4)	0.93 (3)	C(100)—Cl(101)	1.713 (4)
C(5)—C(6)	1.366 (3)	C(100)—Cl(102)	1.730 (3)
C(5)—C(10a)	1.414 (3)	C(100)—Cl(103)	1.753 (4)
C(5)—H(5)	0.92 (3)	C(100)—H(100)	0.89 (3)
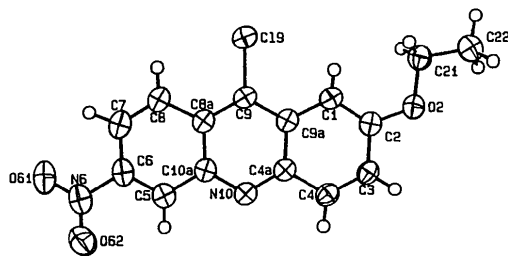
Fig. 1. Thermal ellipsoid (50% probability) depiction of 9-chloro-2-ethoxy-6-nitroacridine showing the numbering scheme.

Table 3. Deviations of atoms ($\text{\AA}$) from, and planarity of various least-squares planes

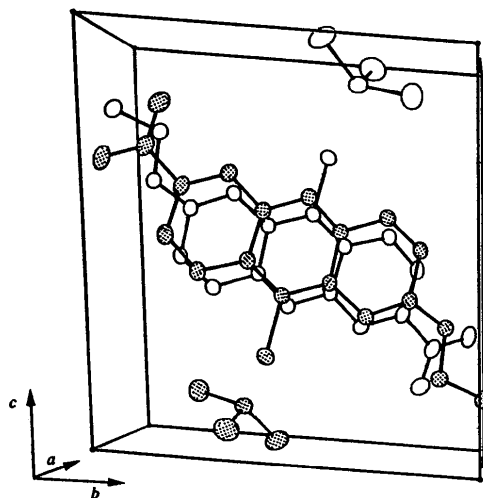|	Plane 1	Plane 2	Plane 3
N(10)			0.001 (2)
C(1)	−0.002 (2)		−0.008 (3)
C(2)	−0.000 (3)		−0.008 (3)
C(3)	0.002 (3)		−0.000 (3)
C(4)	−0.000 (3)		0.004 (3)
C(5)		0.000 (3)	−0.004 (3)
C(6)		−0.002 (3)	−0.009 (3)
C(7)		0.002 (3)	−0.002 (3)
C(8)		−0.001 (3)	0.003 (3)
C(9)			0.002 (3)
C(4a)	−0.003 (3)		0.004 (3)
C(8a)		−0.000 (3)	0.007 (3)
C(9a)	0.004 (2)		0.005 (2)
C(10a)		0.000 (3)	0.004 (3)
$(\chi)^2$	4.8 (1)	1.0 (1)	50.4 (2)

Fig. 2. Molecular packing of 9-chloro-2-ethoxy-6-nitroacridine.

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