Table 1. *Atomic coordinates and equivalent isotropic thermal parameters with e.s.d.'s in parentheses*

$U_{eq} = \frac{1}{3}(U_{11} + U_{22} + U_{33})$.

	x	y	z	U_{eq}(Å^2)
Br1	0·27071 (4)	0	0·17509 (3)	0·058
Br2	0·16982 (4)	0·0254 (4)	0·1217 (3)	0·048
N(*K*1)	0·3408 (3)	0·034 (2)	0·07781 (19)	0·041
C*A*(*K*1)	0·4063 (3)	0·0679 (18)	0·1204 (3)	0·038
C*B*(*K*1)	0·4602 (4)	0·098 (2)	0·0931 (4)	0·071
C*G*(*K*1)	0·5186 (6)	0·057 (4)	0·1163 (6)	0·195
C*D*(*K*1)	0·5691 (6)	0·017 (5)	0·0850 (6)	0·173
C*E*(*K*1)	0·6334 (6)	−0·002 (4)	0·1238 (4)	0·116
N*Z*(*K*1)	0·6879 (3)	0·019 (2)	0·0998 (2)	0·060
C(*K*1)	0·4007 (4)	0·3091 (18)	0·1519 (3)	0·042
O(*K*1)	0·3674 (3)	0·4991 (17)	0·12995 (19)	0·060
N(*F*2)	0·4346 (3)	0·2937 (14)	0·2034 (2)	0·036
C*A*(*F*2)	0·4345 (3)	0·504 (2)	0·2418 (2)	0·037
C*B*(*F*2)	0·4090 (4)	0·3850 (18)	0·2853 (3)	0·044
C*G*(*F*2)	0·4089 (4)	0·5764 (18)	0·3279 (3)	0·047
C*D*1(*F*2)	0·4606 (4)	0·584 (2)	0·3760 (3)	0·063
C*E*1(*F*2)	0·4601 (6)	0·759 (3)	0·4147 (4)	0·086
C*Z*(*F*2)	0·4099 (6)	0·931 (3)	0·4078 (4)	0·092
C*E*2(*F*2)	0·3578 (6)	0·928 (2)	0·3613 (4)	0·084
C*D*2(*F*2)	0·3580 (4)	0·755 (2)	0·3213 (4)	0·059
C(*F*2)	0·5045 (4)	0·6201 (16)	0·2645 (3)	0·038
O(*F*2)	0·5139 (3)	0·8579 (11)	0·2633 (2)	0·041
N(*F*3)	0·5523 (3)	0·4478 (12)	0·2854 (2)	0·036
C*A*(*F*3)	0·6222 (3)	0·525 (3)	0·3009 (2)	0·039
C*B*(*F*3)	0·6659 (4)	0·3314 (19)	0·3418 (3)	0·048
C*G*(*F*3)	0·6564 (4)	0·3586 (18)	0·3955 (3)	0·044
C*D*1(*F*3)	0·6899 (5)	0·562 (3)	0·4291 (3)	0·072
C*E*1(*F*3)	0·6814 (6)	0·591 (2)	0·4799 (4)	0·083
C*Z*(*F*3)	0·6415 (6)	0·426 (3)	0·4957 (4)	0·086
C*E*3(*F*3)	0·6090 (6)	0·236 (3)	0·4626 (4)	0·090
C*D*2(*F*3)	0·6161 (5)	0·201 (2)	0·4122 (4)	0·061
C(*F*3)	0·6443 (4)	0·540 (3)	0·2521 (3)	0·053
O(*F*3)	0·6114 (3)	0·464 (2)	0·2088 (2)	0·081
OH(*F*3)	0·7038 (3)	0·6376 (17)	0·2627 (2)	0·076

edge-to-face interaction between phenyl rings is enthalpically favorable (Burley & Petsko, 1986).

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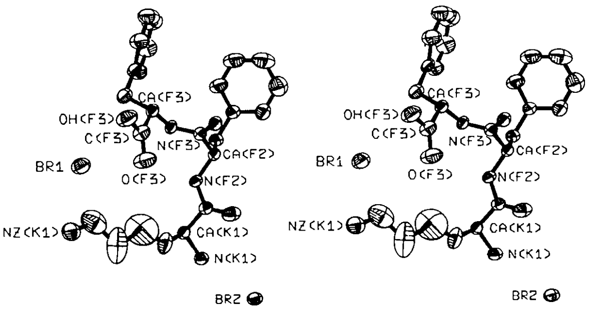

Fig. 1. Stereodrawing of the molecular structure showing the numbering scheme. The thermal ellipsoids are drawn at the 50% level.

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Structure of 4-Chloro-7*H*-benz[*de*]anthracen-7-one

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Abstract. $C_{17}H_9ClO$, $M_r = 264·7$, orthorhombic, $P2_12_12_1$, $a = 16·153$ (6), $b = 14·564$ (5), $c = 5·003$ (2) Å, $V = 1177·0$ (7) Å^3, $Z = 4$, $D_m = 1·49$, $D_x = 1·494$ Mg m^{-3}, λ(Mo $K\alpha$) = 0·71069 Å, $\mu = 3·14$ mm^{-1}, $F(000) = 544$, $T = 298$ K, final $R = 0·047$ for 1126 independent reflections. The title compound

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was isolated from the mixture synthesized by glycerol condensation of chloroanthrones *via* 2-chloroanthraquinone. The analysis was performed in order to assign the position of Cl. The molecule is planar within 0·042 (4)~−0·072 (4) Å.

Experimental. Greenish yellow plate-like crystals from xylene solution; D_m by flotation in $ZnCl_2$ solution; systematic absences: $h00$, $h = 2n+1$, $0k0$, $k = 2n+1$, $00l$, $l = 2n+1$; crystal dimensions 0·45 × 0·45 × 0·20 mm; Rigaku AFC-6 diffractometer; graphite monochromator; cell parameters refined by least-squares method on the basis of 20 independent 2θ values, $18 < 2\theta < 25°$; intensity measurement performed up to $2\theta = 55°$; range of *hkl* 0 to 21, 0 to 18 and 0 to 6; ω–2θ scan, scan speed 4° min^{-1} (2θ), scan width $(1{\cdot}48 + 0{\cdot}5\tan\theta)°$; background 5 s before and after each scan; three standard reflections monitored every 100 reflections, no significant variation in intensities; 1661 reflections measured, 1126 with $|F_o| > 3\sigma(|F_o|)$ considered observed and used for structure determination; corrections for Lorentz and polarization, absorption ignored; direct methods (*MULTAN*78; Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978) and subsequent difference Fourier calculation; block-diagonal least squares (*HBLS*; Ohashi, 1975) with anisotropic thermal parameters for all non-H atoms; H atoms derived geometrically (C—H 1·08 Å) and refined; $\sum w(|F_o| - |F_c|)^2$ minimized with $w =$

Table 1. *Final atomic coordinates* (× 10^4) *and equivalent isotropic thermal parameters* ($Å^2$ × 10)

$$B_{eq} = \tfrac{4}{3}\textstyle\sum_i\sum_j B_{ij}\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	B_{eq}
C(1)	153 (2)	1425 (3)	−2524 (9)	41 (1)
C(2)	−439 (2)	1644 (3)	−4433 (9)	46 (1)
C(3)	−365 (2)	2391 (3)	−6038 (8)	42 (1)
C(4)	433 (2)	3792 (3)	−7235 (8)	42 (1)
C(5)	1085 (3)	4362 (3)	−6872 (9)	48 (1)
C(6)	1674 (2)	4141 (3)	−4997 (9)	43 (1)
C(7)	2278 (2)	3138 (2)	−1587 (8)	38 (1)
C(8)	2799 (3)	2079 (3)	1906 (9)	44 (1)
C(9)	2725 (3)	1317 (3)	3487 (9)	47 (1)
C(10)	2037 (3)	760 (3)	3222 (9)	47 (1)
C(11)	1441 (2)	969 (3)	1406 (8)	42 (1)
C(12)	1487 (2)	1736 (2)	−233 (7)	32 (1)
C(13)	850 (2)	1964 (2)	−2185 (7)	33 (1)
C(14)	937 (2)	2760 (2)	−3800 (7)	32 (1)
C(15)	328 (2)	2977 (2)	−5735 (7)	36 (1)
C(16)	1626 (2)	3356 (2)	−3499 (8)	35 (1)
C(17)	2186 (2)	2301 (2)	45 (9)	34 (1)
O	2892 (2)	3630 (2)	−1352 (7)	55 (1)
Cl	−306 (1)	4080 (1)	−9605 (2)	57 (0)

Table 2. *Bond lengths* (Å) *and bond angles* (°) *with their estimated standard deviations*

C(1)–C(2)	1·389 (6)	C(7)–O	1·230 (5)
C(1)–C(13)	1·383 (6)	C(8)–C(9)	1·368 (6)
C(2)–C(3)	1·358 (6)	C(8)–C(17)	1·397 (6)
C(3)–C(15)	1·416 (5)	C(9)–C(10)	1·383 (6)
C(4)–C(5)	1·354 (6)	C(10)–C(11)	1·359 (6)
C(4)–C(15)	1·415 (5)	C(11)–C(12)	1·388 (5)
C(4)–Cl	1·734 (4)	C(12)–C(13)	1·457 (5)
C(5)–C(6)	1·375 (6)	C(12)–C(17)	1·404 (5)
C(6)–C(16)	1·370 (6)	C(13)–C(14)	1·420 (5)
C(7)–C(16)	1·458 (5)	C(14)–C(15)	1·416 (5)
C(7)–C(17)	1·474 (6)	C(14)–C(16)	1·419 (5)

C(2)–C(1)–C(13)	121·0 (4)	C(11)–C(12)–C(17)	117·2 (4)
C(1)–C(2)–C(3)	122·0 (4)	C(13)–C(12)–C(17)	120·1 (4)
C(2)–C(3)–C(15)	119·3 (4)	C(1)–C(13)–C(12)	121·9 (4)
C(5)–C(4)–C(15)	122·5 (4)	C(1)–C(13)–C(14)	118·3 (4)
C(5)–C(4)–Cl	118·6 (4)	C(12)–C(13)–C(14)	119·8 (4)
C(15)–C(4)–Cl	118·9 (3)	C(13)–C(14)–C(15)	120·2 (4)
C(4)–C(5)–C(6)	119·1 (4)	C(13)–C(14)–C(16)	121·1 (4)
C(5)–C(6)–C(16)	121·9 (4)	C(15)–C(14)–C(16)	118·7 (4)
C(16)–C(7)–C(17)	118·1 (4)	C(3)–C(15)–C(4)	122·9 (4)
C(16)–C(7)–O	121·2 (4)	C(3)–C(15)–C(14)	119·2 (4)
C(17)–C(7)–O	120·7 (4)	C(4)–C(15)–C(14)	117·9 (4)
C(9)–C(8)–C(17)	120·7 (4)	C(6)–C(16)–C(7)	120·0 (4)
C(8)–C(9)–C(10)	119·5 (4)	C(6)–C(16)–C(14)	119·9 (4)
C(9)–C(10)–C(11)	120·1 (4)	C(7)–C(16)–C(14)	120·2 (4)
C(10)–C(11)–C(12)	122·5 (4)	C(7)–C(17)–C(8)	119·3 (4)
C(11)–C(12)–C(13)	122·7 (4)	C(7)–C(17)–C(12)	120·7 (4)
		C(8)–C(17)–C(12)	120·0 (4)

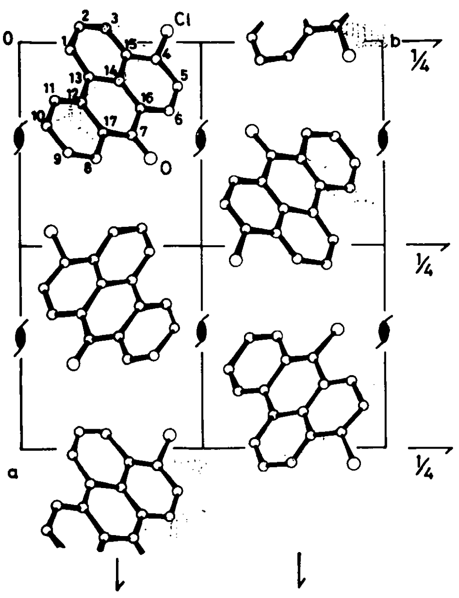

Fig. 1. A projection of the crystal structure along the *c* axis and the numbering scheme.

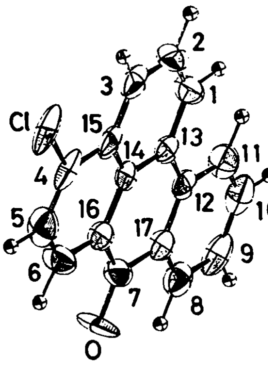

Fig. 2. Perspective drawing of the molecule.

$[\sigma^2(F_o) + (0\cdot03F)^2]^{-1}$; max. (Δ/σ) 0·32; final $R = 0\cdot047$ and $wR = 0\cdot051$, $S = 0\cdot64$; $\Delta\rho$ excursions in final difference map 0·3 e Å^{-3}; atomic scattering factors from *International Tables for X-ray Crystallography* (1974); calculations carried out on HITAC M-280 computer at the Computer Center of the University of Tokyo. The final atomic parameters for non-H atoms are in Table 1.* A projection of the structure along the *c* axis is shown in Fig. 1, and a perspective drawing of the molecule with the numbering scheme is shown in Fig. 2. The bond lengths and angles are listed in Table 2.

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* Lists of the structure factors, anisotropic thermal parameters for non-H atoms, positional and thermal parameters for H atoms, and equations of mean planes have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 43876 (8 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Structure of 13,13-Dimethyl-1,2-didehydrocrotalanine Picrate: A Pyrrolizidine Alkaloid Analogue

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Abstract. $C_{15}H_{22}NO_4^+.C_6H_2N_3O_7^-$, $M_r = 508\cdot4$, monoclinic, $P2_1$, $a = 12\cdot714$ (1), $b = 6\cdot754$ (3), $c = 14\cdot504$ (1) Å, $\beta = 103\cdot97$ (1)°, $V = 1208\cdot6$ Å^3, $Z = 2$, $D_x = 1\cdot397$ Mg m^{-3}, Mo $K\alpha$, $\lambda = 0\cdot71073$ Å, $\mu = 0\cdot11$ mm^{-1}, $F(000) = 532$, $T = 293$ (1) K, $R = 0\cdot085$ for 1579 reflections. The ester carbonyl bonds in the 11-membered macrocyclic pyrrolizidine alkaloid analogue are antiparallel and the pyrrolizidine nucleus has an *exo* conformation. The picrate ion and the alkaloid analogue are linked through the phenolic O atom, O(7), of the picrate and the N atom, N(4), of the alkaloid analogue [O(7)···N(4) 2·62 (1), O(7)···H(4) 1·73 (1) Å; N(4)—H(4)···O(7) 136 (1)°].

Experimental. Yellow rod-like crystals grown from EtOH/MeOH, dimensions 0·08 × 0·11 × 0·46 mm. Stoe–Siemens AED four-circle diffractometer, monochromated Mo $K\alpha$ radiation, ω/θ scan mode, $2\theta_{\max} = 50°$, $\pm h$ $+k$ $+l$, four check reflections with no intensity change, 2277 unique reflections of which 1586 with $F > 3\sigma(F)$ used for all calculations, program system *SHELX76* (Sheldrick, 1976). Index ranges $|h| \leq 14$, k 0→8, l 0→17. Cell constants refined from $\pm\omega$ values of ten reflections and their equivalents in the range $30 < 2\theta < 40°$. No absorption or extinction corrections applied. Structure solution using the *DIRDIF* system (Beurskens *et al.*, 1984) (after introducing coordinates for a picrate ion followed by the use of the programs *ORIENT, TRADIR* and *DIRDIF*). Blocked full-matrix least-squares refinement to $R = 0\cdot085$, $wR = 0\cdot081$ for 1579 reflections (seven reflections probably suffering from extinction removed), all non-H atoms anisotropic, phenyl group as rigid hexagon (C—C 1·395 Å), H atoms included using a riding model [C—H 1·08, N—H 1·01 Å, overall $U(\mathrm{H})_{\mathrm{iso}}$ refined, benzene type 0·081, CH_2 type 0·072, CH_3 type 0·098 Å^2], 326 parameters, weighting scheme $w^{-1} = \sigma^2(F) + 0\cdot0023F^2$ which gave a featureless analysis of variance, max. $(\Delta/\sigma) = 0\cdot85$, ave. $(\Delta/\sigma) < 0\cdot2$, max. and min. heights in the final $\Delta\rho$ map 0·33 and −0·40 e Å^{-3}, respectively. Origin definition implicit in blocked refinement. The probable cause of the relatively high R indices was the small crystal (for Mo $K\alpha$ radiation), hence the low reflection:parameter ratio (4·8). Atomic scattering factors from *International Tables for X-ray Crystallography* (1974). The atomic

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