Table 2. *Bond distances* (Å), *bond angles* (°) *and selected torsion angles* (°)

O(1)–C(13)	1·205 (3)	C(3)–C(4)	1·510 (3)
O(2)–C(13)	1·330 (4)	C(3)–C(9)	1·467 (3)
O(2)–C(14*A*)	1·510 (13)	C(4)–C(5)	1·519 (4)
O(2)–C(14*B*)	1·37 (2)	C(4)–C(12)	1·537 (4)
O(3)–C(9)	1·210 (3)	C(5)–C(6)	1·347 (4)
O(4)–C(9)	1·341 (3)	C(5)–C(13)	1·468 (4)
O(4)–C(10)	1·446 (3)	C(6)–C(7)	1·499 (4)
N(1)–C(2)	1·376 (3)	C(10)–C(11)	1·481 (4)
N(1)–C(6)	1·371 (3)	C(14*A*)–C(15*A*)	1·32 (2)
C(2)–C(3)	1·358 (3)	C(14*B*)–C(15*B*)	1·52 (2)
C(2)–C(8)	1·501 (4)		
C(13)–O(2)–C(14*A*)	122·8 (6)	C(4)–C(5)–C(13)	118·5 (2)
C(13)–O(2)–C(14*B*)	107·1 (8)	C(6)–C(5)–C(13)	120·9 (3)
C(9)–O(4)–C(10)	117·4 (2)	N(1)–C(6)–C(5)	119·2 (2)
C(2)–N(1)–C(6)	124·8 (2)	N(1)–C(6)–C(7)	112·9 (2)
N(1)–C(2)–C(3)	118·6 (2)	C(5)–C(6)–C(7)	127·8 (2)
N(1)–C(2)–C(8)	112·1 (2)	O(3)–C(9)–O(4)	121·1 (2)
C(3)–C(2)–C(8)	129·3 (2)	O(3)–C(9)–C(3)	123·5 (2)
C(2)–C(3)–C(4)	120·8 (2)	O(4)–C(9)–C(3)	115·3 (2)
C(2)–C(3)–C(9)	124·7 (2)	O(4)–C(10)–C(11)	108·1 (2)
C(4)–C(3)–C(9)	114·5 (2)	O(1)–C(13)–O(2)	121·3 (3)
C(3)–C(4)–C(5)	112·0 (2)	O(1)–C(13)–C(5)	126·9 (3)
C(3)–C(4)–C(12)	110·6 (2)	O(2)–C(13)–C(5)	111·8 (3)
C(5)–C(4)–C(12)	109·7 (2)	O(2)–C(14*A*)–C(15*A*)	105·4 (9)
C(4)–C(5)–C(6)	120·5 (2)	O(2)–C(14*B*)–C(15*B*)	109 (2)
C(6)–N(1)–C(2)–C(3)	−10·5 (5)	C(3)–C(4)–C(5)–C(6)	−20·5 (4)
C(2)–N(1)–C(6)–C(5)	9·7 (5)	C(4)–C(5)–C(6)–N(1)	7·2 (5)
C(2)–C(3)–C(4)–C(5)	19·7 (4)	C(2)–C(3)–C(4)–C(12)	−103·0 (4)
N(1)–C(2)–C(3)–C(4)	−5·7 (5)	C(12)–C(4)–C(5)–C(6)	102·8 (4)
C(4)–C(3)–C(9)–O(4)	−170·7 (5)	C(4)–C(5)–C(13)–O(2)	−18·5 (5)

Gluziński, 1977) and diethyl 1,4-dihydro-2,6-dimethyl-4-phenyl-3,5-pyridinedicarboxylate (Hempel & Gupta, 1978). The two ethoxycarbonyl groups are twisted in opposite directions so that the molecule does not have a mirror symmetry. The C(4) methyl substituent is approximately perpendicular to the dihydropyridine ring.

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Structure of 2-(*o*-Ammoniobenzyl)-3-(3-indolyl)-1*H*-quinolinium Bis(ethanesulfonate), $C_{24}H_{21}N_3^{2+}.2C_2H_5SO_3^-$

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Abstract. $M_r = 569{\cdot}7$, triclinic, $P\bar{1}$, $a = 13{\cdot}492\ (1)$, $b = 14{\cdot}789\ (1)$, $c = 8{\cdot}109\ (1)$ Å, $\alpha = 90{\cdot}17\ (1)$, $\beta = 113{\cdot}38\ (1)$, $\gamma = 108{\cdot}85\ (1)$°, $V = 1389{\cdot}23\ (1)$ Å^3, $Z = 2$, $D_x = 1{\cdot}36$ g cm^{-3}, $\lambda(\mathrm{Cu}\ K\alpha) = 1{\cdot}54178$ Å, $\mu = 20{\cdot}7$ cm^{-1}, $F(000) = 600$, $T = 295$ K, $R = 0{\cdot}052$ for 3665 unique significant reflections. This is the first reported occurrence of this compound which is the product of an indole condensation reaction. The aminobenzyl ring is twist-skewed with respect to the quinoline ring and the indolyl ring is twisted −42·6 (4)° from the plane of the quinoline ring. As a 1:2 salt of ethanesulfonic acid, the parent molecule is protonated at the quinoline and amino nitrogen atoms. There is an extensive network of hydrogen bonds formed with the ethanesulfonate oxygen atoms.

Introduction. Protonation and electrophilic substitution are the most important processes in indole chemistry (Remers, 1972). In the presence of strong acids, indoles become protonated and, depending upon acid concentration, can form a mixture of dimer or trimer salts (Bocchi & Palla, 1983). Electrophilic substitution with

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ring expansion to quinolines can also occur. It has been further shown that irradiation of indolines in alcohol will result in either reduction or addition products, depending on the alcohol and wavelength.

During crystallization experiments involving diaminopteridines, red crystals were grown from a methanol/water solution containing ethanesulfonic acid and indole. This paper reports the crystal-structure determination of the hitherto unreported indole condensation product, 2-(*o*-aminobenzyl)-3-(3-indolyl)-quinoline, crystallized as a 1:2 salt of ethanesulfonic acid.

Experimental. Red crystals of title compound grown at room temperature; cell dimensions from least-squares refinement of 50 reflections, 2θ range 50·16 to 72·45°; Nicolet *P*3 diffractometer, Ni-filtered Cu $K\alpha$ radiation, θ–2θ scan, $2\theta_{\max} = 116°$ ($0 < h < 16$, $-18 < k < 18$, $-10 < l < 10$); 0·12 × 0·30 × 0·74 mm; four standard reflections monitored at every 59 reflections, no crystal decomposition, Lp correction applied but no corrections for extinction or absorption, 3795 unique reflections, 3665 with $I > 2\sigma(I)$ (Stout & Jensen, 1968); direct methods (*MULTAN*: Germain, Main & Woolfson, 1971; *NQEST*: De Titta, Edmonds, Langs & Hauptman, 1975), refinement on *F* by full-matrix least squares, anisotropic thermal parameters, H positions located in difference Fourier syntheses, refined isotropically; final difference Fourier maps showed no peaks $> 0·15$ e Å^{-3}, $\sum w(|F_o| - |F_c|)^2$ minimized, $w = 1/[\sigma^2(I) + (0·02I)^2]$, final $R = 0·052$, $R_w = 0·073$, max. Δ/σ 0·01; atomic scattering factors from *International Tables for X-ray Crystallography* (1974); all calculations performed on a VAX 11/780 computer using Enraf–Nonius (1978) *SDP*. The ethyl carbon atoms of one ethanesulfonate molecule are disordered (50%) and only their isotropic thermal parameters were refined.

Table 1. *Atomic coordinates* ($\times 10^4$) *and equivalent isotropic thermal parameters* ($\times 10$) *for the title compound*

	x	*y*	*z*	B_{eq}(Å^2)*
C(1)	11455 (2)	5637 (2)	8357 (4)	43 (1)
C(2)	12527 (3)	5894 (2)	9729 (4)	48 (1)
C(3)	12807 (3)	5309 (2)	11052 (5)	50 (1)
C(4)	11993 (3)	4459 (2)	11026 (4)	44 (1)
N(5)	10024 (2)	3367 (1)	9607 (3)	35 (1)
C(6)	8950 (2)	3044 (2)	8320 (3)	33 (1)
C(7)	8644 (2)	3542 (2)	6822 (3)	33 (1)
C(8)	9460 (2)	4412 (2)	6863 (4)	36 (1)
C(9)	10583 (2)	4760 (2)	8276 (4)	36 (1)
C(10)	10875 (2)	4191 (2)	9640 (3)	35 (1)
C(11)	8118 (2)	2199 (2)	8695 (4)	37 (1)
C(12)	8277 (2)	1235 (2)	8557 (3)	34 (1)
C(13)	7917 (2)	526 (2)	9517 (3)	35 (1)
C(14)	8047 (3)	−356 (2)	9409 (4)	47 (1)
C(15)	8551 (3)	−548 (2)	8324 (5)	52 (1)
C(16)	8909 (3)	143 (2)	7354 (4)	49 (1)
C(17)	8769 (2)	1024 (2)	7464 (4)	41 (1)
N(18)	7426 (2)	712 (2)	10748 (3)	38 (1)
C(19)	7508 (2)	3147 (2)	5245 (3)	36 (1)
C(20)	6794 (2)	3657 (2)	4167 (3)	37 (1)
C(21)	6824 (3)	4609 (2)	4223 (4)	48 (1)
C(22)	5946 (3)	4826 (3)	2963 (5)	59 (1)
C(23)	5016 (3)	4120 (3)	1581 (5)	62 (2)
C(24)	4958 (3)	3189 (3)	1464 (5)	52 (1)
C(25)	5842 (2)	2957 (2)	2758 (4)	41 (1)
N(26)	5987 (2)	2090 (2)	2978 (4)	46 (1)
C(27)	6972 (3)	2204 (2)	4434 (4)	43 (1)
S(1*A*)	4617 (1)	903 (1)	7843 (1)	381 (3)
O(2*A*)	4523 (2)	−87 (1)	7617 (3)	68 (1)
O(3*A*)	3775 (2)	1015 (1)	8443 (3)	59 (1)
O(4*A*)	5774 (2)	1538 (1)	9029 (3)	48 (1)
C(2*A*)	4326 (4)	1299 (3)	5698 (5)	64 (2)
C(3*A*)	4494 (5)	2349 (4)	5797 (9)	80 (2)
S(1*B*)	361 (1)	2493 (1)	3985 (1)	444 (3)
O(2*B*)	−647 (2)	1775 (2)	3933 (3)	84 (1)
O(3*B*)	543 (2)	2356 (2)	2378 (3)	69 (1)
O(4*B*)	421 (3)	3457 (2)	4341 (5)	90 (2)
C(2*B*)	1604 (4)	2352 (3)	5699 (6)	69 (2)
C(3*B*)	1747 (4)	2586 (4)	7605 (6)	69 (2)
C(2*Bx*)	1340	2777	6355	83 (6)
C(3*Bx*)	1509	1822	6116	1 (6)

$*B_{eq} = \frac{4}{3}\sum_i\sum_j\beta_{ij}(\mathbf{a}_i.\mathbf{a}_j)$.

Table 2. *Bond lengths* (Å) *and bond angles* (°) *for the non-hydrogen atoms with e.s.d.'s in parentheses*

C(1)–C(2)	1·356 (4)	C(1)–C(9)	1·421 (4)
C(2)–C(3)	1·392 (4)	C(3)–C(4)	1·370 (4)
C(4)–C(10)	1·400 (3)	N(5)–C(6)	1·329 (3)
N(5)–C(10)	1·370 (3)	C(6)–C(7)	1·412 (4)
C(6) C(11)	1·510 (4)	C(7)–C(8)	1·388 (3)
C(7)–C(19)	1·478 (3)	C(8)–C(9)	1·410 (3)
C(9)–C(10)	1·400 (4)	C(11)–C(12)	1·520 (4)
C(12)–C(13)	1·385 (4)	C(12)–C(17)	1·387 (5)
C(13)–C(14)	1·377 (4)	C(13)–N(18)	1·467 (5)
C(14)–C(15)	1·385 (6)	C(15)–C(16)	1·371 (5)
C(16)–C(17)	1·383 (5)	C(19)–C(20)	1·438 (4)
C(19)–C(27)	1·366 (4)	C(20)–C(21)	1·395 (4)
C(20)–C(25)	1·413 (3)	C(21)–C(22)	1·355 (5)
C(22)–C(23)	1·397 (4)	C(23)–C(24)	1·355 (6)
C(24)–C(25)	1·383 (5)	C(25)–N(26)	1·360 (4)
N(26)–C(27)	1·340 (4)		
S(1*A*)–O(2*A*)	1·432 (2)	S(1*A*)–O(3*A*)	1·454 (3)
S(1*A*)–O(4*A*)	1·449 (2)	S(1*A*)–C(2*A*)	1·771 (4)
C(2*A*)–C(3*A*)	1·491 (8)		
S(1*B*)–O(2*B*)	1·414 (3)	S(1*B*)–O(3*B*)	1·444 (3)
S(1*B*)–O(4*B*)	1·424 (3)	S(1*B*)–C(2*B*)	1·780 (5)
C(2*B*)–C(3*B*)	1·504 (7)	S(1*B*)–C(2*Bx*)	1·799
C(2*Bx*)–C(3*Bx*)	1·524		
C(2)–C(1)–C(9)	119·8 (2)	C(1)–C(2)–C(3)	121·5 (3)
C(2)–C(3)–C(4)	120·6 (3)	C(3)–C(4)–C(10)	118·6 (3)
C(6)–N(5)–C(10)	124·6 (2)	N(5)–C(6)–C(7)	119·6 (2)
N(5)–C(6)–C(11)	115·2 (2)	C(7)–C(6)–C(11)	125·0 (2)
C(6)–C(7)–C(8)	117·5 (2)	C(6)–C(7)–C(19)	121·6 (2)
C(8)–C(7)–C(19)	120·9 (2)	C(7)–C(8)–C(9)	121·8 (2)
C(1)–C(9)–C(8)	123·8 (2)	C(1)–C(9)–C(10)	117·9 (2)
C(8)–C(9)–C(10)	118·2 (2)	C(4)–C(10)–N(5)	120·4 (2)
C(4)–C(10)–C(9)	121·6 (2)	N(5)–C(10)–C(9)	117·9 (2)
C(6)–C(11)–C(12)	115·8 (2)	C(11)–C(12)–C(13)	120·5 (2)
C(11)–C(12)–C(17)	122·0 (2)	C(13)–C(12)–C(17)	117·5 (2)
C(12)–C(13)–C(14)	121·6 (2)	C(12)–C(13)–N(18)	120·4 (2)
C(14)–C(13)–N(18)	117·9 (2)	C(13)–C(14)–C(15)	119·9 (2)
C(14)–C(15)–C(16)	119·4 (3)	C(15)–C(16)–C(17)	120·3 (3)
C(12)–C(17)–C(16)	121·3 (2)	C(7)–C(19)–C(20)	128·6 (2)
C(7)–C(19)–C(27)	125·2 (2)	C(20)–C(19)–C(27)	106·0 (2)
C(19)–C(20)–C(21)	136·1 (2)	C(19)–C(20)–C(25)	106·1 (2)
C(21)–C(20)–C(25)	117·8 (2)	C(20)–C(21)–C(22)	119·6 (3)
C(21)–C(22)–C(23)	121·6 (3)	C(22)–C(23)–C(24)	120·8 (3)
C(23)–C(24)–C(25)	118·1 (3)	C(20)–C(25)–C(24)	122·2 (2)
C(20)–C(25)–N(26)	107·6 (2)	C(24)–C(25)–N(26)	130·2 (2)
C(25)–N(26)–C(27)	109·6 (2)	C(19)–C(27)–N(26)	110·6 (2)
O(2*A*)–S(1*A*)–O(3*A*)	111·9 (1)	O(2*A*)–S(1*A*)–O(4*A*)	112·9 (1)
O(2*A*)–S(1*A*)–C(2*A*)	107·8 (2)	O(3*A*)–S(1*A*)–O(4*A*)	111·0 (1)
O(3*A*)–S(1*A*)–C(2*A*)	107·5 (2)	O(4*A*)–S(1*A*)–C(2*A*)	105·4 (2)
S(1*A*)–C(2*A*)–C(3*A*)	112·5 (4)		
O(2*B*)–S(1*B*)–O(3*B*)	112·2 (1)	O(2*B*)–S(1*B*)–O(4*B*)	114·2 (2)
O(2*B*)–S(1*B*)–C(2*B*)	109·7 (2)	O(2*B*)–S(1*B*)–C(2*Bx*)	104·1
O(3*B*)–S(1*B*)–O(4*B*)	111·0 (2)	O(3*B*)–S(1*B*)–C(2*B*)	101·2 (2)
O(3*B*)–S(1*B*)–C(2*Bx*)	131·2	O(4*B*)–S(1*B*)–C(2*B*)	107·7 (2)
O(4*B*)–S(1*B*)–C(2*Bx*)	80·6	S(1*B*)–C(2*B*)–C(3*B*)	114·0 (3)
S(1*B*)–C(2*Bx*)–C(3*Bx*)	86·8		

Discussion. Final fractional coordinates and equivalent B values for the title compound are listed in Table 1* and the geometry is described in Table 2. Fig. 1 shows the atom-labeling scheme of the molecule. The aminobenzyl ring is twist-skewed with respect to the quinoline ring as defined by the torsion angles C(7)—C(6)—C(11)—C(12) = 113·4 (3) and C(6)—C(11)—C(12)—C(17) = −26·4 (4)°. The *o*-amino group is distal to the quinoline ring and is protonated, as is the quinoline nitrogen atom. The indolyl ring is twisted −42·6 (4)° [C(6)—C(7)—C(19)—C(27)] from the quinoline ring. The molecule is involved in a network of hydrogen bonds formed between the hydrogen atoms of N(5), N(18), and N(26) and the ethanesulfonate oxygen atoms. The geometry of the complex is similar to that observed in other indole and ethanesulfonate structures (Andrianov, Struchkov & Babievsky, 1982; Cody & Zakrzewski, 1982). The only exception is the N(26)—C(27) indole-ring bond length [1·340 (4) Å] which is somewhat shortened when compared to the indole bond lengths [1·377 (3) Å] observed in other indole structures. This shortening may be indicative of a partial positive charge on the nitrogen atom.

This is the first reported occurrence of this unusual compound which may have been formed by the condensation of two indole molecules in the presence of the strong acid, ethanesulfonic acid, which then reacted in UV light and methanol to form the observed product.

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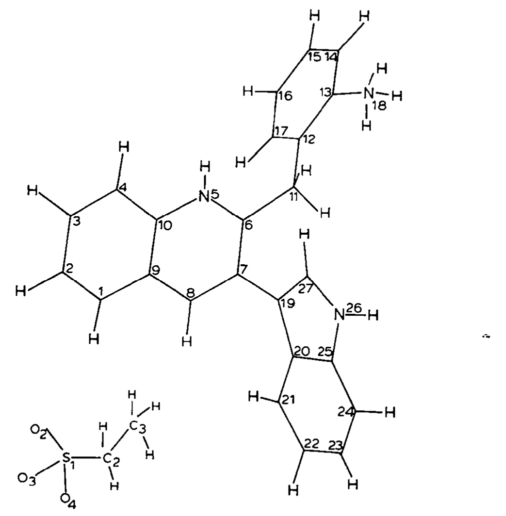

Fig. 1. Molecular structure and numbering scheme for the title compound.

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* Lists of structure factors, anisotropic thermal parameters, hydrogen-atom coordinates and intermolecular contacts have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 39842 (22 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Structures of 3,8-Dihydroxy-3-methyl-3,4-dihydroisocoumarin (I), $C_{10}H_{10}O_4$, and 2-Acetonyl-6-methoxybenzoic Acid (II), $C_{11}H_{12}O_4$

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Abstract. (I): $M_r = 194{\cdot}19$, monoclinic, $P2_1/c$, $a = 7{\cdot}011$ (2), $b = 9{\cdot}493$ (2), $c = 13{\cdot}943$ (3) Å, $\beta = 93{\cdot}87$ (2)°, $V = 926{\cdot}1$ (3) Å^3, $Z = 4$, $D_x = 1{\cdot}39$ Mg m^{-3}, λ(Mo $K\alpha$) = 0·71070 Å, $\mu = 0{\cdot}10$ mm^{-1}, $F(000) = 408$, $T = 293$ K, final $R = 0{\cdot}068$ for 1041 unique observed reflections. (II): $M_r = 208{\cdot}22$, orthorhombic, $P2_12_12_1$, $a = 12{\cdot}954$ (12), $b = 15{\cdot}043$ (9), $c = 5{\cdot}218$ (1) Å, $V = 1016{\cdot}9$ (11) Å^3, Z

0108-2701/85/030415-03$01.50