Table 3. *Intermolecular distances* (Å) *less than* 3·5 Å *between the non-hydrogen atoms*

$C(6)\cdots O(3^{i})$	3·286 (12)	$C(11)\cdots O(4^{ii})$	3·308 (10)
$C(10)\cdots O(3^{i})$	3·206 (9)	$O(2)\cdots C(11^{iii})$	3·493 (23)
$C(9)\cdots O(3^{i})$	3·248 (13)		

Symmetry code: (i) $x, \frac{1}{2}-y, \frac{1}{2}+z$; (ii) $x, -\frac{1}{2}-y, \frac{1}{2}+z$; (iii) $1-x, \frac{1}{2}+y, \frac{3}{2}-z$.

In addition, there is a close contact of 2·523 (13) Å between the phenolic O(3) atom and the O(4) carbonyl atom, which no doubt represents an intramolecular hydrogen bond. In spite of several attempts, the position of the H atom, as in 2-(4′-chloro-2′-hydroxybenzoyl)benzoic acid (Skrzat, 1980), could not be determined from a difference map.

The packing of dimers in the crystal is achieved by van der Waals forces. There are some short contacts but none is significantly different from the sum of the van der Waals radii (Table 3).

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2′-Acetamido-2-chloro-4′-diethylamino-4-mesylazobenzene and 2-Chloro-4′-diethylamino-4-mesyl-2′-propionamidoazobenzene

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Abstract. $C_{19}H_{23}ClN_4O_3S$: M_r = 422·9, monoclinic, $P2_1/c$, a = 4·913 (1), b = 16·139 (2), c = 25·485 (3) Å, β = 93·25 (2)°, V = 2017·5 Å^3, Z = 4, D_x = 1·392, D_m = 1·37 Mg m^{-3}. $C_{20}H_{25}ClN_4O_3S$: M_r = 437·0, monoclinic, $P2_1/c$, a = 8·994 (2), b = 17·039 (3), c = 14·610 (3) Å, β = 109·89 (2)°, V = 2105·4 Å^3, Z = 4, D_x = 1·379, D_m = 1·35 Mg m^{-3}. For both crystals the densities were measured by flotation at 298 K, the structures were solved by the heavy-atom method, and refinement was carried out by block-diagonal least-squares methods using diffractometer-measured intensity data. For the first compound 3114 observed independent reflections gave a final R = 0·077 and R_w = 0·082; for the second compound 1429 observed reflections led to R = 0·048, R_w = 0·050. No major structural differences were observed between the geometries of the two molecules.

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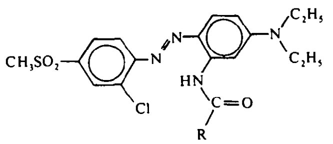
Introduction. The title compounds, designated ACLSA and CLSPA, are two members of a group of disperse dyes. Their formulas are shown in (I), where R is CH_3 for ACLSA and C_2H_5 for CLSPA. These dyes actually dissolve in polyester fibres, but their affinities for the fibers are markedly different, that of ACLSA being much greater than that of CLSPA. A theory of solubility has been proposed (Gerber, 1976), in which the dye–fiber affinity is a critical function of the dye's molecular volume. In order to test this theory, the above compounds and also 2′-acetamido-6-bromo-2-cyano-4′-diethylamino-4-nitroazobenzene (ABRCA) and 6-bromo-2-cyano-4′-diethylamino-4-nitro-2′-propionamidoazobenzene (BRCPA) were synthesized and studied (Gerber, Moriconi, Groeke & Altermann, 1976). The second pair also differ by one CH_2 group, but the situation is reversed, *i.e.* the larger molecule has the greater fiber affinity. X-ray investigations of all four compounds have been carried out, in order to ascertain whether any conformational changes might play a part in modifying physical properties, and the crystal structures of ACLSA and CLSPA are described in the present paper.



(I)

X-ray data were collected using monochromated Cu $K\alpha$ radiation on an Enraf–Nonius CAD-4 automated diffractometer. Cell parameters were derived from a least-squares fit of the angular data for 15 high-angle reflections. Reflections were considered observed when $I > 1{\cdot}5\sigma(I)$. The crystal of ACLSA measured $0{\cdot}40 \times 0{\cdot}14 \times 0{\cdot}04$ mm and absorption corrections [μ(Cu $K\alpha$) = 2·85 mm^{-1}] ranging from 1·23 to 1·90 were applied to the intensity data (Busing & Levy, 1957). In the range $2\theta < 150°$, 3632 reflections were measured, of which 3114 were considered observed. For CLSPA the crystal measured $0{\cdot}25 \times 0{\cdot}20 \times 0{\cdot}20$ mm, absorption corrections were not made and maximum absorption errors [μ(Cu $K\alpha$) = 2·72 mm^{-1}] are calculated to be $\sim\pm 4\%$ in F_o. The crystallinity was poorer than for ACLSA and only 1429 reflections were observed out of 3278 measured.

Both structures were solved by the heavy-atom method and, after refinement of the positional and isotropic thermal parameters by block-diagonal least-squares methods, the H atoms were located from difference electron density maps. Refinement of all positional parameters and anisotropic thermal parameters for the non-hydrogen atoms was then carried out, with the H atoms being assigned the equivalent isotropic B of the heavier atom to which each was bonded. The function minimized was $\sum w(|F_o| - k|F_c|)^2$ where w was obtained from the counting statistics. Unobserved reflections were weighted zero. For ACLSA, in the final cycle, $R = 0{\cdot}077$, $R_w = 0{\cdot}082$ (observed reflections only), and the mean and maximum shift/e.s.d. = 0·07 and 0·33. For CLSPA, $R = 0{\cdot}048$, $R_w = 0{\cdot}050$ and the mean and maximum shift/e.s.d. = 0·08 and 0·30. Including the unobserved reflections with F_c above threshold, $R = 0{\cdot}078$ for ACLSA and 0·087 for CLSPA. The scattering factors, including anomalous-dispersion corrections for Cl, S and O, were taken from *International Tables for X-ray Crystallography* (1968). All calculations were carried out using programs of the NRC system (Ahmed, Hall, Pippy & Huber, 1973). The refined positional parameters for all atoms in ACLSA and CLSPA are given in Tables 1 and 2 respectively.* The equivalent isotropic B's for the heavier atoms (Willis & Pryor, 1975) and the B's used for the H atoms are included.

Discussion. The bond distances (uncorrected for thermal-vibration effects) are shown in Fig. 1. The geometries of the two molecules are remarkably similar. In both structures N(1) and N(4) are *cis* with respect to rotation about C(7)–N(2). Short interactions, indicative of strong intramolecular hydrogen bonding which stabilizes the *cis* against the *trans* configuration, are observed between N(4) and N(1). The distances N(4)···N(1) are 2·663 (3) and 2·657 (6) Å, HN(4)···N(1) are 1·89 (5) and 1·99 (5) Å, and the angles N(4)–HN(4)···N(1) are 143 (5) and 131 (4) Å in ACLSA and CLSPA respectively. In both compounds the ethyl groups bonded to N(3) are roughly perpendicular to, and on opposite

* Lists of structure factors, anisotropic thermal parameters and bond angles for both compounds have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 35613 (51 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

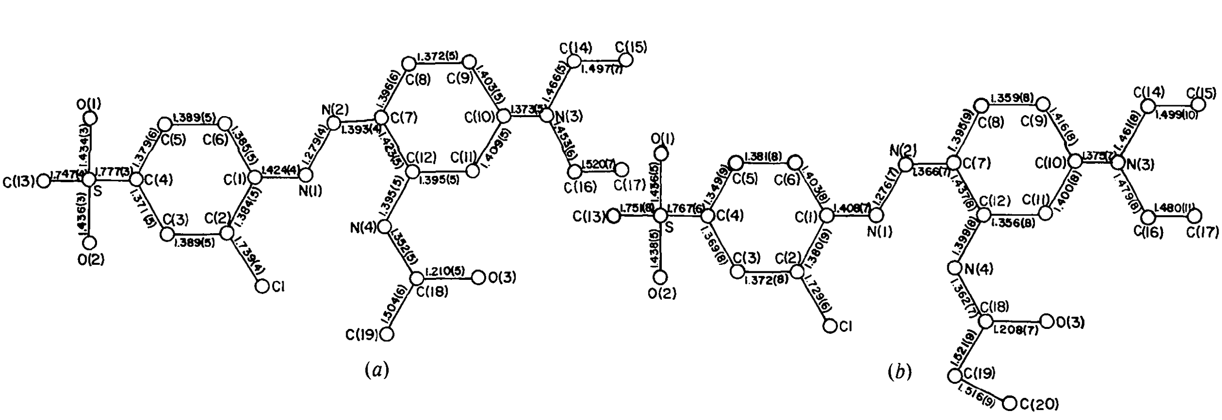


Fig. 1. Bond distances (Å) (*a*) in ACLSA and (*b*) in CLSPA. The distances are uncorrected for thermal-vibration effects.

Table 1. *Fractional atomic coordinates for ACLSA (those for the non-hydrogen atoms are* $\times 10^4$ *and for the* H *atoms,* $\times 10^2$ *for x, and* $\times 10^3$ *for y and z)*

The equivalent isotropic *B*'s are given for the non-hydrogen atoms, and the *B*'s used in isotropic temperature factors of the form $\exp(-\sin^2\theta/\lambda^2)$ are given for the H atoms.

	x	*y*	*z*	B_{eq}/B (Å^2)*
S	11062 (2)	1445 (1)	138 (0)	3·7 (1)
Cl	8334 (3)	3008 (1)	1900 (0)	5·8 (1)
O(1)	12577 (5)	689 (2)	107 (1)	4·5 (2)
O(2)	12505 (6)	2217 (2)	113 (1)	5·3 (2)
O(3)	2007 (8)	3052 (2)	3639 (1)	7·3 (2)
N(1)	4869 (6)	1562 (2)	2082 (1)	3·3 (1)
N(2)	3276 (6)	948 (2)	2145 (1)	3·4 (1)
N(3)	−3548 (6)	651 (2)	3815 (1)	3·8 (2)
N(4)	3246 (6)	2267 (2)	2956 (1)	3·6 (2)
C(1)	6370 (7)	1485 (2)	1623 (1)	3·1 (2)
C(2)	8033 (7)	2140 (2)	1497 (1)	3·3 (2)
C(3)	9469 (7)	2133 (2)	1043 (1)	3·6 (2)
C(4)	9317 (7)	1441 (2)	730 (1)	3·2 (2)
C(5)	7757 (8)	765 (3)	850 (1)	3·9 (2)
C(6)	6274 (8)	797 (3)	1297 (1)	3·8 (2)
C(7)	1693 (7)	959 (3)	2582 (1)	3·3 (2)
C(8)	3 (8)	267 (3)	2612 (1)	3·6 (2)
C(9)	−1694 (7)	149 (3)	3016 (1)	3·7 (2)
C(10)	−1849 (7)	750 (3)	3410 (1)	3·4 (2)
C(11)	−171 (7)	1455 (3)	3388 (1)	3·4 (2)
C(12)	1565 (7)	1573 (2)	2980 (1)	3·2 (2)
C(13)	8473 (8)	1422 (3)	−361 (1)	4·8 (2)
C(14)	−3882 (8)	1292 (3)	4214 (2)	4·4 (2)
C(15)	−2142 (13)	1163 (4)	4709 (2)	6·7 (3)
C(16)	−5136 (8)	−102 (3)	3866 (2)	4·4 (2)
C(17)	−3461 (10)	−830 (3)	4082 (2)	5·7 (2)
C(18)	3374 (8)	2949 (3)	3264 (2)	4·2 (2)
C(19)	5436 (11)	3586 (3)	3117 (2)	5·5 (2)
HN(4)	40 (1)	226 (3)	265 (2)	3·6
HC(3)	106 (1)	261 (2)	97 (1)	3·6
HC(5)	77 (1)	27 (3)	65 (2)	3·9
HC(6)	51 (1)	38 (3)	138 (2)	3·8
HC(8)	1 (1)	−13 (2)	234 (1)	3·6
HC(9)	−28 (1)	−35 (3)	302 (2)	3·7
HC(11)	−1 (1)	186 (2)	366 (1)	3·4
H1C(13)	70 (1)	186 (3)	−35 (2)	4·8
H2C(13)	92 (1)	146 (2)	−65 (1)	4·8
H3C(13)	75 (1)	98 (3)	−32 (2)	4·8
H1C(14)	−60 (1)	129 (3)	427 (2)	4·4
H2C(14)	−37 (1)	186 (3)	405 (2)	4·4
H1C(15)	−25 (1)	59 (3)	486 (2)	6·7
H2C(15)	−1 (2)	106 (5)	459 (3)	6·7
H3C(15)	−26 (1)	156 (4)	498 (3)	6·7
H1C(16)	−58 (1)	−29 (2)	349 (2)	4·4
H2C(16)	−67 (1)	3 (3)	405 (2)	4·4
H1C(17)	−44 (1)	−130 (4)	413 (2)	6·7
H2C(17)	−19 (2)	−89 (5)	394 (3)	5·7
H3C(17)	−29 (1)	−74 (3)	444 (2)	5·7
H1C(19)	53 (1)	366 (4)	280 (3)	5·5
H2C(19)	64 (1)	380 (4)	344 (3)	5·5
H3C(19)	50 (2)	417 (6)	330 (4)	5·5

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

sides of the adjacent benzene rings. A real though minor difference exists in the conformation of one benzene ring with respect to the −N=N− group. The

Table 2. *Fractional atomic coordinates for CLSPA (those for the non-hydrogen atoms are* $\times 10^4$ *and for the* H *atoms* $\times 10^3$*)*

The equivalent isotropic *B*'s are given for the non-hydrogen atoms, and the *B*'s used in isotropic temperature factors of the form $\exp(-\sin^2\theta/\lambda^2)$ are given for the H atoms.

	x	*y*	*z*	B_{eq}/B (Å^2)*
S	2752 (2)	1161 (1)	4465 (1)	4·9 (1)
Cl	465 (2)	1327 (1)	562 (1)	4·3 (1)
O(1)	4353 (5)	1107 (3)	5120 (3)	4·6 (2)
O(2)	1981 (6)	1913 (3)	4329 (3)	5·4 (3)
O(3)	1121 (5)	290 (2)	−2977 (3)	4·3 (2)
N(1)	2732 (6)	95 (3)	591 (3)	3·1 (2)
N(2)	3613 (6)	−499 (3)	624 (3)	3·5 (2)
N(3)	4025 (6)	−2110 (3)	−2570 (3)	3·7 (2)
N(4)	1933 (6)	116 (3)	−1335 (3)	3·1 (2)
C(1)	2765 (7)	322 (3)	1525 (4)	3·3 (3)
C(2)	1729 (7)	892 (3)	1607 (4)	3·0 (3)
C(3)	1674 (7)	1150 (4)	2484 (4)	3·4 (3)
C(4)	2708 (7)	823 (3)	3312 (4)	3·3 (3)
C(5)	3733 (8)	258 (4)	3269 (4)	4·1 (3)
C(6)	3803 (8)	−1 (4)	2389 (4)	3·7 (3)
C(7)	3670 (7)	−805 (3)	−228 (4)	2·9 (3)
C(8)	4618 (8)	−1472 (4)	−100 (4)	4·0 (3)
C(9)	4753 (7)	−1906 (3)	−848 (4)	3·8 (3)
C(10)	3915 (7)	−1662 (3)	−1813 (4)	3·5 (3)
C(11)	2998 (7)	−978 (3)	−1972 (4)	3·3 (3)
C(12)	2863 (7)	−560 (3)	−1214 (4)	3·4 (3)
C(13)	1607 (9)	480 (5)	4831 (5)	8·4 (5)
C(14)	4694 (8)	−2901 (4)	−2428 (5)	4·5 (4)
C(15)	6436 (8)	−2916 (4)	−2254 (5)	5·2 (4)
C(16)	3440 (8)	−1823 (4)	−3585 (5)	5·2 (4)
C(17)	1825 (9)	−2115 (5)	−4114 (6)	7·3 (5)
C(18)	1131 (7)	492 (3)	−2182 (4)	3·5 (3)
C(19)	220 (7)	1199 (4)	−2019 (4)	3·9 (3)
C(20)	−681 (8)	1623 (4)	−2956 (5)	5·2 (4)
HN(4)	172 (5)	30 (3)	−83 (3)	3·1
HC(3)	80 (5)	152 (3)	253 (3)	3·4
HC(5)	422 (4)	12 (2)	362 (3)	4·1
HC(6)	467 (7)	−31 (3)	238 (4)	3·7
HC(8)	513 (7)	−159 (4)	61 (4)	4·0
HC(9)	541 (6)	−239 (3)	−75 (4)	3·8
HC(11)	229 (7)	−79 (3)	−260 (4)	3·3
H1C(13)	37 (7)	47 (3)	437 (4)	8·4
H2C(13)	211 (6)	1 (3)	491 (4)	8·4
H3C(13)	166 (7)	70 (3)	558 (4)	8·4
H1C(14)	418 (6)	−320 (3)	−198 (4)	4·5
H2C(14)	387 (6)	−322 (3)	−311 (4)	4·5
H1C(15)	724 (7)	−260 (4)	−162 (4)	5·2
H2C(15)	681 (8)	−261 (4)	−290 (5)	5·2
H3C(15)	688 (7)	−337 (4)	−221 (4)	5·2
H1C(16)	418 (6)	−200 (3)	−393 (3)	5·2
H2C(16)	320 (8)	−124 (4)	−380 (5)	5·2
H1C(17)	110 (9)	−185 (4)	−481 (5)	7·3
H2C(17)	188 (6)	−259 (3)	−413 (4)	7·3
H3C(17)	86 (8)	−183 (4)	−381 (5)	7·3
H1C(19)	109 (6)	159 (3)	−145 (3)	3·9
H2C(19)	−48 (7)	98 (3)	−161 (4)	3·9
H1C(20)	9 (7)	172 (3)	−332 (4)	5·2
H2C(20)	−145 (6)	127 (3)	−335 (4)	5·2
H3C(20)	−113 (7)	208 (4)	−269 (4)	5·2

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

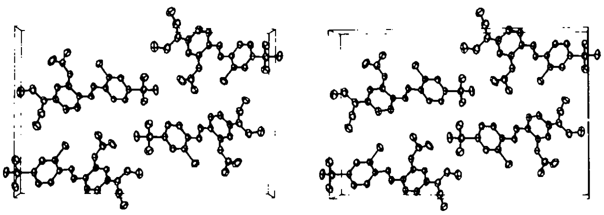


Fig. 2. A stereoview of the contents of one unit cell for ACLSA.

torsion angles C(6)–C(1)–N(1)–N(2) and C(2)–C(1)–N(1)–N(2) are −4·2 (4) and 176·0 (3)° in ACLSA and 8·0 (6) and −172·0 (5)° in CLSPA.

In both structures the molecular packing involves normal van der Waals contacts, with no intermolecular hydrogen bonding. A stereoview of the contents of one unit cell (Johnson, 1965) is shown for ACLSA in Fig. 2.

The results of the crystal structure analyses of these two dyes do not reveal any marked changes in molecular geometry which could be the cause of their different fiber affinity, nor have any such changes been observed between ABRCA (Handal, Gruska & White, 1980) and BRCPA (Handal & White, 1980). The differing affinities may be due to a critical molecular volume (Gerber, 1976) or to an important change in physical properties caused by the subtle chemical change.

We thank Dr E. J. Moriconi and Sandoz Ltd for crystals of the title compounds.

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[(1*RS*,2*RS*,3*SR*)-3-Hydroxy-2-methylcyclopentyl]trimethylammonium Chloride

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Abstract. $C_9H_{20}NO^+.Cl^-$, $M_r = 193·72$, orthorhombic, $P2_12_12_1$, $a = 7·440$ (2), $b = 11·609$ (2), $c = 12·295$ (3) Å, $U = 1061·9$ Å, $Z = 4$, $D_x = 1·212$ Mg m^{-3}, μ(Mo $K\alpha$) = 0·32 mm^{-1}. The structure was refined to $R = 0·0415$ for 985 independent reflexions. The five-membered ring adopts the envelope conformation; the three substituents are *cis*. The two ions are linked by an O—H···Cl hydrogen bond.

Introduction. The structure of the title compound has been determined to elucidate the arrangement of substituents on the five-membered ring. As frequently found for saturated C_5 rings, rapid conformational changes in solution (pseudorotation) ruled out an unambiguous structural assignment on the basis of NMR data alone.

Hygroscopic crystals were obtained from ethanol/diethyl ether and sealed in Lindemann capillaries. Data were collected on a Stoe–Siemens four-circle diffractometer with a control program written by Clegg (1981) and a crystal 0·3 × 0·3 × 0·4 mm. 1093 data were recorded for $7 < 2\theta < 50°$; after averaging

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