

2-{4-[4-(Dimethylamino)phenyl]-1,3-butadienyl}-1-ethylpyridinium perchlorate

Jerry P. Jasinski,* John M. Jasinski and Daniel J. Crosby

Department of Chemistry, Keene State College,
229 Main Street, Keene, New Hampshire
03435-2001, USA

Correspondence e-mail: jjasinski@keene.edu

The title compound, $C_{19}H_{23}N_2^+ \cdot ClO_4^-$, also known as LDS 698, contains almost parallel pyridyl and phenyl ring systems and an in-plane dimethylamino group. The extended pyridyl aminoethyl group is nearly perpendicular to the dimethylamino group. The molecules stack along the *a* axis, with the ring systems oblique to this axis.

Received 4 October 2002

Accepted 16 October 2002

Online 25 October 2002

Key indicators

Single-crystal X-ray study

 $T = 296$ KMean $\sigma(C-C) = 0.005$ Å

Disorder in solvent or counterion

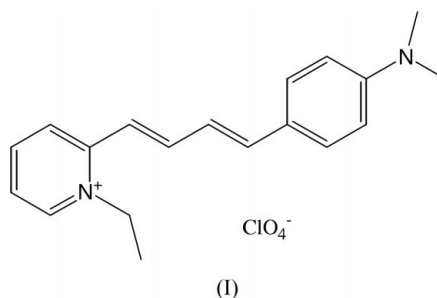
 R factor = 0.046 wR factor = 0.159

Data-to-parameter ratio = 15.5

For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

Comment

The title compound, (I) (Fig. 1), also known as LDS 698 (Exiton Inc.), is an efficient laser dye in the 675–930 nm region (Bado *et al.*, 1983). It has an absorption λ_{\max} at 476^m nm (*m* signifies a metastable state) and exhibits a lasing λ_{\max} at 684 nm (Broyer *et al.*, 1984) and 695 nm (Gomes & Taylor, 1985). The crystal structure determination of (I) was performed in order to examine the non-rigid nitrogen centers at opposite ends of the molecule, and the possible relationship between the structure and laser efficiency. The r.m.s. deviations from the least-squares planes for the pyridyl and phenyl rings are 0.0074 and 0.0035 Å, respectively. The dihedral angle between these planes is 2.7 (1)°.



Bond lengths and angles for the pyridyl and phenyl rings, the butadiene group and the dimethylamino group are normal. O atoms in the perchlorate anion are disordered. The amino N atom, N19, and the dimethyl groups, C20 and C21, are approximately coplanar with the phenyl ring, with deviations of -0.009 (3), 0.175 (7) and -0.116 (4) Å, respectively. The ethyl group is tilted somewhat, with respect to the plane of the pyridyl ring [$C2-N1-C7-C8 = 76.9$ (3)° and $C6-N1-C7-C8 = -102.3$ (3)°], and exhibits a slightly shortened $C7-C8$ [1.498 (4) Å] bond length. The distance between the N atoms, N1 and N19, is 11.34 (2) Å.

A packing diagram of the molecule (Fig. 2) indicates that the ions are stacked in layers along the *a* axis, with the parallel planes of the pyridyl and phenyl rings almost perpendicular to the axis and a closest interlayer contact of 3.347 (5) Å [$C10 \cdots C12(-x, 3 - y, 2 - z)$].

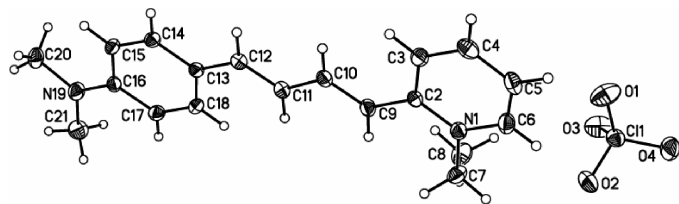


Figure 1
ORTEPII (Johnson, 1976) view of (I), with ellipsoids drawn at the 50% probability level, and the atomic numbering scheme.

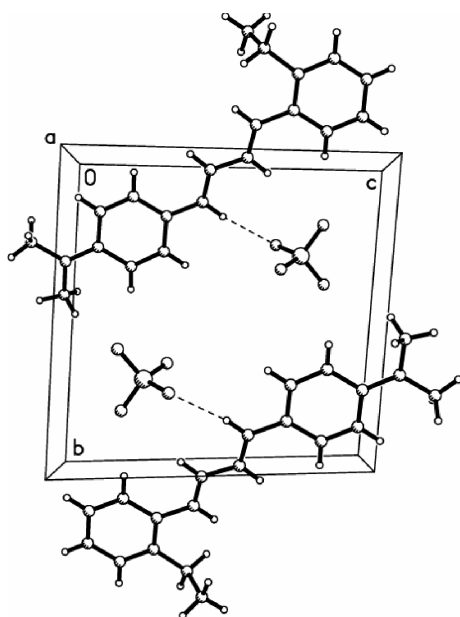


Figure 2
ORTEPII (Johnson, 1976) packing diagram, viewed down the *a* axis. The minor disorder component of the anion is not shown.

Experimental

Commercial LDS 698 (Exiton Inc., Ohio, USA) was crystallized by slow evaporation of a solution in acetonitrile.

Crystal data

$C_{19}H_{23}N_2^+ \cdot ClO_4^-$	$Z = 2$
$M_r = 378.84$	$D_x = 1.349 \text{ Mg m}^{-3}$
Triclinic, $P\bar{1}$	Mo $K\alpha$ radiation
$a = 7.8213 (14) \text{ \AA}$	Cell parameters from 20 reflections
$b = 11.1055 (13) \text{ \AA}$	$\theta = 6.1\text{--}9.4^\circ$
$c = 11.494 (2) \text{ \AA}$	$\mu = 0.23 \text{ mm}^{-1}$
$\alpha = 90.003 (18)^\circ$	$T = 296 (2) \text{ K}$
$\beta = 106.098 (15)^\circ$	Prism, dark purple
$\gamma = 102.960 (14)^\circ$	$0.50 \times 0.50 \times 0.20 \text{ mm}$
$V = 932.8 (3) \text{ \AA}^3$	

Data collection

Rigaku AFC-6S diffractometer
 $2\theta/\omega$ scans
 Absorption correction: ψ scan
 (North *et al.*, 1968)
 $T_{\min} = 0.893$, $T_{\max} = 0.955$
 4516 measured reflections
 4297 independent reflections
 1835 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.057$
 $\theta_{\text{max}} = 27.5^\circ$
 $h = -10 \rightarrow 9$
 $k = 0 \rightarrow 14$
 $l = -14 \rightarrow 14$
 3 standard reflections
 every 150 reflections
 intensity decay: 0.5%

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.046$
 $wR(F^2) = 0.159$
 $S = 1.00$
 4297 reflections
 277 parameters
 H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0668P)^2 + 0.0636P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} = 0.004$
 $\Delta\rho_{\text{max}} = 0.23 \text{ e \AA}^{-3}$
 $\Delta\rho_{\text{min}} = -0.23 \text{ e \AA}^{-3}$
 Extinction correction: SHELXL97
 Extinction coefficient: 0.030 (4)

H atoms were included in calculated positions and refined as riding atoms. All four perchlorate O atoms are disordered.

Data collection: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1998); cell refinement: *MSC/AFC Diffractometer Control Software*; data reduction: *TEXSAN* (Molecular Structure Corporation, 1998); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *TEXSAN*; software used to prepare material for publication: *TEXSAN*.

The authors gratefully acknowledge the donors of the Petroleum Research Fund, administered by the National Science Foundation, for support of this research, and the National Science Foundation Research in Undergraduate Institutions Instrumentation Program (grant No. 8818307) for creation of the New England Molecular Structure Center at Keene State College. JPJ thanks Exiton Inc. for their generosity and assistance.

References

- Bado, P., Dupuy, C., Wilson, K. R., Boggy, R., Bowen, J. & Westra, S. (1983). *Opt. Commun.* **46**, 241–245.
 Broyer, M., Chevaleyre, J., Delacretaz, G. & Woste, L. (1984). *Appl. Phys. B*, **35**, 31–35.
 Gomes, A. S. L. & Taylor, J. R. (1985). *Opt. Commun.* **55**, 435–437.
 Johnson, C. K. (1976). *ORTEPII*. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.
 Molecular Structure Corporation (1998). *MSC/AFC Diffractometer Control Software* and *TEXSAN* (Version 1.06). MSC, 3200 Research Forest Drive, The Woodlands, TX 77381, USA.
 North, A. C. T., Phillips, D. C. & Mathews, F. S. (1968). *Acta Cryst.* **A24**, 351–359.
 Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.