# Crystal structure of *trans*-4-[4-(*N*,*N*-diethylamino)-2-hydroxystyryl]-*N*-methylpyridinium iodide monohydrate acetonitrile hemisolvate, $(C_{18}H_{23}N_2O)I \cdot H_2O \cdot \frac{1}{2}CH_3CN$

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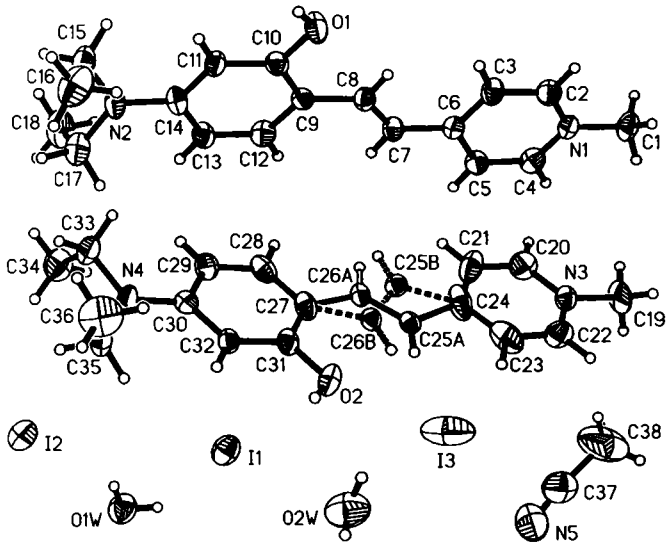


**Abstract**

$C_{19}H_{26.50}IN_{2.50}O_2$, monoclinic, *C*12/*c*1 (no. 15), $a = 35.970(4)$ Å, $b = 11.627(2)$ Å, $c = 22.081(3)$ Å, $\beta = 114.341(7)°$, $V = 8414.0$ Å$^3$, $Z = 16$, $R_{gt}(F) = 0.050$, $wR_{ref}(F^2) = 0.124$, $T = 293$ K.

**Source of material**

A mixture of 4-diethylamino-2-hydroxybenzaldehyde (1 g, 0.0052 mol) and 4-methyl-*N*-methylpyridinium iodide (1.1 g, 0.0052 mol) in ethanol (100 ml) was heated under reflux for 8 h. Then the mixture was concentrated and purified by column chromatography on silica gel using chloroform/petroleum ether (5:1 *v*/*v*) as eluent. After removal of the solvent, dark-red platelet-like crystals were obtained (yield 68 %, 1.45 g). A sample for the structure analysis was obtained by recrystallization from acetonitrile. Elemental analysis: found – C, 52.51 %; H, 5.68 %; N, 6.36 %; calc. for $C_{18}H_{23}IN_2O$ – C, 52.69 %; H, 5.65 %; N, 6.83 %. $^1$H NMR data are available in the CIF.

**Experimental details**

The structure shows three different disordered atoms or molecules: I3, one substituted pyridinium cation and the acetonitrile molecule. Only C25 and C26 were split into to two positions each due to their large Fourier difference peaks. The I3 anion probably has eight different positions. Therefore, it was refined with large anisotropic displacement parameters. The acetonitrile shows a typical solvent disorder which also was not resolved. The final residual electron density maximum in the Fourier difference analysis had a value of 0.55 e Å$^{-3}$.

**Discussion**

Recently, optical limiting effects attracted considerable research interests due to growing needs in eye protection from intense laser exposure and for protective application in optical sensors [1]. Pyridinium dyes are a type of important optical limiting materials for their strong nonlinear absorption [2] and weak fluorescence emission. From the viewpoint of molecular engineering, structural features such as $\pi$-conjugation style, molecular planarity, and characteristics of substituted group play important roles in tuning nonlinear optical properties of materials [3]. According to the concept of molecular engineering, *trans*-4-[4-(*N*,*N*-diethylamino)-2-hydroxystyryl]-*N*-methylpyridinium iodide was synthesized and studied.

In the crystal structure of the title compound the cation can be described within a *D*-$\pi$-*A* model with diethylamino, pyridinium and styrene groups as the electron donor (*D*), the electron acceptor (*A*) and the $\pi$ conjugated bridge, respectively. There are two independent molecules in the asymmetric unit. The cation backbone is perfectly planar, as indicated by the dihedral angle of 4.1(3)° between the planes of the phenyl (C9 to C14) and pyridyl (C2 to C6, N1) groups in the first molecule. This planarity can be further proved by the dihedral angle of 1.6(3)° between the planes of the phenyl (C27 to C32) and pyridyl (C20 to C24, N3) groups in the second one. The maximum deviations for the non-hydrogen atoms from the mean planes are 0.083 Å for N2 and 0.055 Å for N4, respectively. The perfect planarity of the backbones of cation contributes to the high $\pi$-conjugation, a necessary condition for non-linear optical effects. The two molecules in one asymmetric unit are nearly perpendicular, with dihedral angles of 88.5(3)° between two phenyl rings and 84.3(3)° between two pyridyl rings. A similar compound with non-substituted hydroxyl group at the 2-position (DEASPI) was reported [4] and claimed to have strong two-photon absorption. Comparing with the title structure, the planarity of the cation backbone of DEASPI is much worse. The dihedral angle between the phenyl and pyridyl groups is 8.8(4)°. Based on the structure data above, we think that the planarity of the substituted pyridyl salt is strongly affected by the substituted 2-hydroxyl group. The packing of the molecules can be described by layers perpendicular to the *b* axis, in which the molecules are either parallel or perpendicular, and the neighboring two molecules are packed in a head-to-tail manner. Two water molecules and an acetonitrile molecule were co-crystallized in the asymmetric unit. The water molecules perhaps come from moist air and impurity of acetonitrile solvent.

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**Table 1.** Data collection and handling.

| | |
|---|---|
| Crystal: | red plate, size 0.04 × 0.30 × 0.50 mm |
| Wavelength: | Mo $K_\alpha$ radiation (0.71073 Å) |
| $\mu$: | 15.36 $cm^{-1}$ |
| Diffractometer, scan mode: | Bruker P4, $\omega$ |
| $2\theta_{max}$: | 52° |
| $N(hkl)_{measured}$, $N(hkl)_{unique}$: | 9210, 7946 |
| Criterion for $I_{obs}$, $N(hkl)_{gt}$: | $I_{obs} > 2\,\sigma(I_{obs})$, 3200 |
| $N(param)_{refined}$: | 472 |
| Programs: | SIR97 [5], SHELXL-97 [6] |

**Table 2.** Atomic coordinates and displacement parameters (in Å$^2$).

| Atom | Site | Occ. | $x$ | $y$ | $z$ | $U_{iso}$ |
|---|---|---|---|---|---|---|
| H(1D) | 8*f* | | 0.1017 | 0.3790 | 0.3956 | 0.087 |
| H(1A) | 8*f* | | 0.3439 | 0.2349 | 0.3490 | 0.102 |
| H(1B) | 8*f* | | 0.3298 | 0.1163 | 0.3130 | 0.102 |
| H(1C) | 8*f* | | 0.3640 | 0.1205 | 0.3855 | 0.102 |
| H(2A) | 8*f* | | 0.2720 | 0.2668 | 0.3252 | 0.073 |
| H(3A) | 8*f* | | 0.2229 | 0.2563 | 0.3643 | 0.067 |
| H(4A) | 8*f* | | 0.3344 | 0.0215 | 0.4441 | 0.068 |
| H(5A) | 8*f* | | 0.2874 | 0.0059 | 0.4882 | 0.063 |
| H(7A) | 8*f* | | 0.2224 | 0.0610 | 0.4893 | 0.058 |
| H(8A) | 8*f* | | 0.1815 | 0.2420 | 0.4117 | 0.057 |
| H(11A) | 8*f* | | 0.0705 | 0.3030 | 0.4573 | 0.060 |
| H(12A) | 8*f* | | 0.1769 | 0.0428 | 0.5310 | 0.065 |
| H(13A) | 8*f* | | 0.1296 | 0.0366 | 0.5744 | 0.064 |
| H(15A) | 8*f* | | 0.0075 | 0.1985 | 0.5276 | 0.091 |
| H(15B) | 8*f* | | 0.0216 | 0.2531 | 0.4758 | 0.091 |
| H(16A) | 8*f* | | 0.0099 | 0.3935 | 0.5400 | 0.157 |
| H(16B) | 8*f* | | 0.0556 | 0.3960 | 0.5497 | 0.157 |
| H(16C) | 8*f* | | 0.0438 | 0.3397 | 0.6040 | 0.157 |
| H(17A) | 8*f* | | 0.1011 | 0.0928 | 0.6367 | 0.091 |
| H(17B) | 8*f* | | 0.0582 | 0.1310 | 0.6324 | 0.091 |
| H(18A) | 8*f* | | 0.0605 | −0.0672 | 0.6269 | 0.156 |
| H(18B) | 8*f* | | 0.0709 | −0.0587 | 0.5647 | 0.156 |
| H(18C) | 8*f* | | 0.0279 | −0.0200 | 0.5593 | 0.156 |
| H(2B) | 8*f* | | 0.3102 | 0.6111 | 0.3486 | 0.085 |
| H(19A) | 8*f* | | 0.4517 | 0.3994 | 0.0653 | 0.133 |
| H(19B) | 8*f* | | 0.4836 | 0.3941 | 0.1396 | 0.133 |
| H(19C) | 8*f* | | 0.4705 | 0.5132 | 0.1038 | 0.133 |
| H(20A) | 8*f* | | 0.3907 | 0.3427 | 0.0613 | 0.085 |
| H(21A) | 8*f* | | 0.3391 | 0.3486 | 0.0965 | 0.105 |
| H(22A) | 8*f* | | 0.4565 | 0.5480 | 0.2055 | 0.084 |
| H(23A) | 8*f* | | 0.4068 | 0.5522 | 0.2436 | 0.105 |
| H(25A) | 8*f* | 0.54 | 0.3542 | 0.5246 | 0.2560 | 0.048 |
| H(26A) | 8*f* | 0.54 | 0.2981 | 0.3845 | 0.1598 | 0.049 |
| H(25B) | 8*f* | 0.46 | 0.3037 | 0.3825 | 0.1399 | 0.055 |
| H(26B) | 8*f* | 0.46 | 0.3418 | 0.5071 | 0.2588 | 0.050 |
| H(28A) | 8*f* | | 0.2425 | 0.3425 | 0.1586 | 0.073 |
| H(29A) | 8*f* | | 0.1887 | 0.3397 | 0.1870 | 0.066 |
| H(32A) | 8*f* | | 0.2488 | 0.5539 | 0.3394 | 0.057 |
| H(33A) | 8*f* | | 0.1362 | 0.4103 | 0.1908 | 0.077 |
| H(33B) | 8*f* | | 0.1176 | 0.4417 | 0.2416 | 0.077 |
| H(34A) | 8*f* | | 0.1078 | 0.2492 | 0.2160 | 0.130 |
| H(34B) | 8*f* | | 0.1370 | 0.2626 | 0.2914 | 0.130 |
| H(34C) | 8*f* | | 0.1549 | 0.2312 | 0.2397 | 0.130 |
| H(35A) | 8*f* | | 0.2075 | 0.5043 | 0.3752 | 0.078 |
| H(35B) | 8*f* | | 0.1638 | 0.4561 | 0.3595 | 0.078 |
| H(36A) | 8*f* | | 0.1641 | 0.6535 | 0.3705 | 0.177 |
| H(36B) | 8*f* | | 0.1351 | 0.6197 | 0.2975 | 0.177 |
| H(36C) | 8*f* | | 0.1788 | 0.6679 | 0.3133 | 0.177 |
| H(38A) | 8*f* | | 0.0345 | 0.4478 | 0.1994 | 0.317 |
| H(38B) | 8*f* | | −0.0004 | 0.4322 | 0.1281 | 0.317 |
| H(38C) | 8*f* | | 0.0447 | 0.4018 | 0.1412 | 0.317 |
| H(101) | 8*f* | | 0.2003 | 0.2072 | 0.0613 | 0.132 |
| H(102) | 8*f* | | 0.1645 | 0.2345 | 0.0571 | 0.132 |
| H(103) | 8*f* | | 0.0613 | 0.5538 | 0.3453 | 0.173 |
| H(104) | 8*f* | | 0.0765 | 0.5593 | 0.4096 | 0.173 |

**Table 3.** Atomic coordinates and displacement parameters (in Å$^2$).

| Atom | Site | Occ. | $x$ | $y$ | $z$ | $U_{11}$ | $U_{22}$ | $U_{33}$ | $U_{12}$ | $U_{13}$ | $U_{23}$ |
|---|---|---|---|---|---|---|---|---|---|---|---|
| I(1) | 4*c* | | ¼ | ¼ | 0 | 0.0690(5) | 0.0835(5) | 0.0957(5) | −0.0023(4) | 0.0354(4) | −0.0175(4) |
| I(2) | 8*f* | | 0.09457(2) | 0.34692(5) | 0.03218(2) | 0.0629(3) | 0.0805(4) | 0.0879(4) | −0.0031(3) | 0.0262(3) | −0.0146(3) |
| I(3) | 8*f* | 0.50 | −0.0089(3) | 0.6962(1) | 0.2574(3) | 0.262(7) | 0.0809(7) | 0.074(3) | 0.007(2) | 0.009(2) | 0.002(1) |
| O(1) | 8*f* | | 0.1195(1) | 0.3313(4) | 0.4000(2) | 0.070(3) | 0.081(4) | 0.087(3) | 0.026(3) | 0.054(3) | 0.037(3) |
| N(1) | 8*f* | | 0.3078(1) | 0.1465(4) | 0.3818(2) | 0.042(3) | 0.049(3) | 0.058(3) | −0.005(3) | 0.025(3) | −0.008(3) |
| N(2) | 8*f* | | 0.0659(2) | 0.1675(5) | 0.5478(3) | 0.066(4) | 0.065(4) | 0.091(4) | 0.015(3) | 0.055(3) | 0.029(3) |
| C(1) | 8*f* | | 0.3391(2) | 0.1553(6) | 0.3550(3) | 0.055(4) | 0.080(5) | 0.084(5) | −0.010(4) | 0.043(4) | −0.021(4) |
| C(2) | 8*f* | | 0.2748(2) | 0.2139(6) | 0.3584(3) | 0.056(4) | 0.060(5) | 0.069(4) | 0.007(4) | 0.027(4) | 0.012(4) |
| C(3) | 8*f* | | 0.2454(2) | 0.2077(5) | 0.3816(3) | 0.048(4) | 0.054(4) | 0.074(4) | 0.010(3) | 0.034(4) | 0.004(4) |
| C(4) | 8*f* | | 0.3118(2) | 0.0696(6) | 0.4286(3) | 0.051(4) | 0.053(4) | 0.069(4) | 0.012(4) | 0.027(4) | −0.005(4) |
| C(5) | 8*f* | | 0.2834(2) | 0.0592(5) | 0.4547(3) | 0.053(4) | 0.048(4) | 0.058(4) | 0.009(4) | 0.026(3) | 0.008(3) |
| C(6) | 8*f* | | 0.2486(2) | 0.1284(5) | 0.4313(3) | 0.045(4) | 0.040(4) | 0.050(4) | −0.001(3) | 0.023(3) | −0.004(3) |
| C(7) | 8*f* | | 0.2184(2) | 0.1188(5) | 0.4581(3) | 0.051(4) | 0.044(4) | 0.056(4) | −0.005(3) | 0.027(3) | 0.003(3) |
| C(8) | 8*f* | | 0.1851(2) | 0.1842(5) | 0.4428(3) | 0.046(4) | 0.049(4) | 0.051(4) | −0.005(3) | 0.024(3) | 0.000(3) |
| C(9) | 8*f* | | 0.1544(2) | 0.1766(5) | 0.4682(3) | 0.046(4) | 0.045(4) | 0.042(3) | −0.003(3) | 0.018(3) | 0.001(3) |
| C(10) | 8*f* | | 0.1212(2) | 0.2528(5) | 0.4471(3) | 0.053(4) | 0.047(4) | 0.048(4) | −0.006(4) | 0.021(3) | 0.004(3) |
| C(11) | 8*f* | | 0.0919(2) | 0.2504(5) | 0.4724(3) | 0.039(4) | 0.056(4) | 0.063(4) | 0.008(3) | 0.027(3) | 0.014(4) |
| C(12) | 8*f* | | 0.1556(2) | 0.0959(5) | 0.5162(3) | 0.054(4) | 0.043(4) | 0.068(4) | 0.013(3) | 0.027(4) | 0.010(3) |
| C(13) | 8*f* | | 0.1271(2) | 0.0913(5) | 0.5423(3) | 0.056(4) | 0.049(4) | 0.062(4) | 0.009(4) | 0.032(4) | 0.019(3) |
| C(14) | 8*f* | | 0.0943(2) | 0.1685(5) | 0.5210(3) | 0.065(4) | 0.048(4) | 0.062(4) | −0.004(4) | 0.042(4) | 0.001(4) |
| C(15) | 8*f* | | 0.0296(2) | 0.2395(6) | 0.5228(3) | 0.060(5) | 0.087(6) | 0.093(5) | −0.006(5) | 0.044(4) | 0.010(5) |
| C(16) | 8*f* | | 0.0352(2) | 0.3521(6) | 0.5572(4) | 0.087(6) | 0.075(6) | 0.154(8) | 0.017(5) | 0.052(6) | 0.004(6) |
| C(17) | 8*f* | | 0.0723(2) | 0.0959(6) | 0.6078(4) | 0.070(5) | 0.073(5) | 0.098(6) | 0.001(5) | 0.048(5) | 0.008(5) |
| C(18) | 8*f* | | 0.0565(2) | −0.0231(7) | 0.5879(4) | 0.111(7) | 0.095(7) | 0.130(7) | −0.018(6) | 0.074(6) | −0.006(6) |
| O(2) | 8*f* | | 0.3141(1) | 0.5733(4) | 0.3205(2) | 0.046(3) | 0.073(4) | 0.100(4) | −0.008(3) | 0.035(3) | 0.000(3) |
| N(3) | 8*f* | | 0.4278(2) | 0.4444(5) | 0.1300(3) | 0.047(3) | 0.056(4) | 0.061(3) | 0.002(3) | 0.030(3) | 0.002(3) |
| N(4) | 8*f* | | 0.1791(1) | 0.4455(4) | 0.2824(2) | 0.041(3) | 0.064(4) | 0.067(3) | −0.013(3) | 0.029(3) | −0.020(3) |
| C(19) | 8*f* | | 0.4614(2) | 0.4371(7) | 0.1077(3) | 0.071(5) | 0.130(7) | 0.101(6) | 0.014(5) | 0.072(5) | 0.012(5) |

**Table 3.** Continued.

| Atom | Site | Occ. | $x$ | $y$ | $z$ | $U_{11}$ | $U_{22}$ | $U_{33}$ | $U_{12}$ | $U_{13}$ | $U_{23}$ |
|---|---|---|---|---|---|---|---|---|---|---|---|
| C(20) | 8*f* | | 0.3938(2) | 0.3863(6) | 0.0984(3) | 0.079(6) | 0.058(5) | 0.075(5) | −0.008(4) | 0.032(5) | −0.008(4) |
| C(21) | 8*f* | | 0.3630(2) | 0.3897(7) | 0.1198(5) | 0.054(5) | 0.068(6) | 0.134(8) | −0.006(4) | 0.032(6) | 0.029(6) |
| C(22) | 8*f* | | 0.4326(2) | 0.5065(6) | 0.1834(3) | 0.069(5) | 0.072(5) | 0.065(5) | −0.008(4) | 0.024(4) | −0.019(4) |
| C(23) | 8*f* | | 0.4026(3) | 0.5095(7) | 0.2057(4) | 0.115(7) | 0.095(7) | 0.080(6) | 0.038(6) | 0.070(6) | 0.008(5) |
| C(24) | 8*f* | | 0.3664(3) | 0.4520(8) | 0.1744(5) | 0.078(7) | 0.087(7) | 0.131(8) | 0.038(6) | 0.077(7) | 0.053(6) |
| C(25A) | 8*f* | 0.54(3) | 0.3434(6) | 0.479(1) | 0.2182(7) | 0.05(1) | 0.036(7) | 0.042(9) | 0.007(7) | 0.023(7) | 0.007(6) |
| C(26A) | 8*f* | 0.54 | 0.3069(6) | 0.431(1) | 0.1976(9) | 0.04(1) | 0.038(8) | 0.05(1) | 0.002(7) | 0.025(8) | 0.006(7) |
| C(25B) | 8*f* | 0.46 | 0.3240(6) | 0.424(2) | 0.173(1) | 0.04(1) | 0.05(1) | 0.05(1) | 0.005(8) | 0.019(9) | 0.005(9) |
| C(26B) | 8*f* | 0.46 | 0.3207(8) | 0.467(2) | 0.226(1) | 0.04(1) | 0.045(9) | 0.047(9) | 0.006(7) | 0.023(7) | 0.005(7) |
| C(27) | 8*f* | | 0.2798(2) | 0.4458(6) | 0.2309(3) | 0.060(5) | 0.053(4) | 0.074(5) | 0.016(4) | 0.045(4) | 0.018(4) |
| C(28) | 8*f* | | 0.2442(2) | 0.3846(5) | 0.1955(3) | 0.081(5) | 0.058(5) | 0.059(4) | 0.010(4) | 0.045(4) | 0.004(4) |
| C(29) | 8*f* | | 0.2116(2) | 0.3832(5) | 0.2121(3) | 0.061(4) | 0.047(4) | 0.064(4) | −0.003(3) | 0.033(4) | −0.001(3) |
| C(30) | 8*f* | | 0.2123(2) | 0.4466(5) | 0.2666(3) | 0.049(4) | 0.041(4) | 0.046(4) | 0.006(3) | 0.022(3) | 0.002(3) |
| C(31) | 8*f* | | 0.2803(2) | 0.5112(5) | 0.2847(3) | 0.051(4) | 0.044(4) | 0.069(4) | 0.004(3) | 0.029(4) | 0.024(4) |
| C(32) | 8*f* | | 0.2474(2) | 0.5110(5) | 0.3029(3) | 0.045(4) | 0.048(4) | 0.053(4) | −0.002(3) | 0.025(3) | 0.006(3) |
| C(33) | 8*f* | | 0.1391(2) | 0.3990(6) | 0.2361(3) | 0.044(4) | 0.074(5) | 0.074(5) | −0.007(4) | 0.026(4) | −0.010(4) |
| C(34) | 8*f* | | 0.1342(2) | 0.2744(6) | 0.2467(4) | 0.092(6) | 0.073(6) | 0.094(6) | −0.034(5) | 0.038(5) | −0.011(5) |
| C(35) | 8*f* | | 0.1796(2) | 0.5018(6) | 0.3418(3) | 0.057(4) | 0.075(5) | 0.077(5) | −0.016(4) | 0.042(4) | −0.013(4) |
| C(36) | 8*f* | | 0.1629(3) | 0.6214(7) | 0.3297(4) | 0.141(9) | 0.080(7) | 0.127(7) | 0.016(6) | 0.049(7) | −0.024(6) |
| N(5) | 8*f* | | 0.0145(3) | 0.1931(8) | 0.1841(5) | 0.137(8) | 0.123(7) | 0.126(7) | −0.017(7) | 0.062(6) | 0.007(7) |
| C(37) | 8*f* | | 0.0185(3) | 0.283(1) | 0.1766(6) | 0.068(6) | 0.105(8) | 0.130(8) | 0.000(7) | 0.028(6) | −0.014(8) |
| C(38) | 8*f* | | 0.0249(3) | 0.4015(9) | 0.1599(7) | 0.11(1) | 0.103(9) | 0.38(2) | −0.018(8) | 0.06(1) | −0.04(1) |
| O(1W) | 8*f* | | 0.1821(1) | 0.1837(4) | 0.0721(2) | 0.072(3) | 0.088(4) | 0.104(4) | 0.013(3) | 0.037(3) | 0.007(3) |
| O(2W) | 8*f* | | 0.0689(2) | 0.5120(5) | 0.3787(2) | 0.117(5) | 0.098(4) | 0.111(4) | 0.035(4) | 0.026(4) | 0.002(3) |

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