# Crystal structure of 4-hexyl-4′-hydroxyazobenzene isopropanol hemisolvate, $C_{18}H_{22}N_2O \cdot \frac{1}{2}C_3H_8O$

H. R. Nasiri[I], M. Bolte[II], M. Lorch[III] and H. Schwalbe*,[I]

[I] Johann-Wolfgang-Goethe-Universität Frankfurt, Institut für Organische Chemie und Chemische Biologie, Center for Biomolecular Magnetic Resonance, Max-von-Laue-Str. 7, 60438 Frankfurt/Main, Germany
[II] Johann-Wolfgang-Goethe-Universität Frankfurt, Institut für Anorganische Chemie, Max-von-Laue-Str. 7, 60438 Frankfurt/Main, Germany
[III] University of Hull, Department of Chemistry, Cottingham Road, Kingston upon Hull, HU67RX, U. K.

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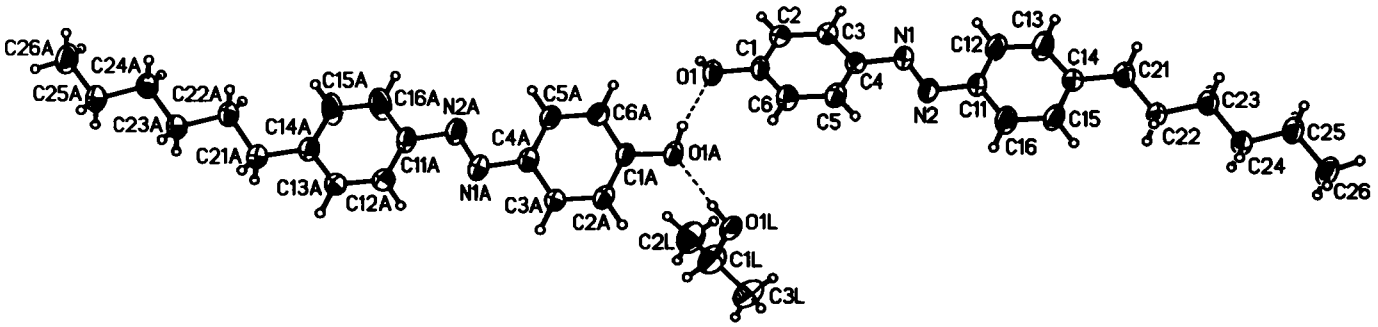


## Abstract

$C_{19.5}H_{26}N_2O_{1.5}$, triclinic, $P\bar{1}$ (no. 2), $a$ = 6.1744(8) Å, $b$ = 14.915(2) Å, $c$ = 19.453(3) Å, $\alpha$ = 87.55(1)°, $\beta$ = 84.37(1)°, $\gamma$ = 88.94(1)°, $V$ = 1781.0 Å$^3$, $Z$ = 4, $R_{gt}(F)$ = 0.059, $wR_{ref}(F^2)$ = 0.160, $T$ = 173 K.

## Source of material

The title compound was synthesized according to the literature [1]. Our procedure differed from the original by filtration and washing of the precipitate with dichloromethane and subsequent purification by silica chromatography (dichloromethane, $R_f$ = 0.53), rather than recrystallization from benzene/hexane mixture. Single crystals suitable for X-ray structure analysis were grown by evaporation of an isopropanol solution.

## Discussion

As a molecular switch, the title compound exists in two conformational states. Irradiation at 254 nm wavelength converts the shown thermodynamic stable *trans*-form into the *cis*-form by a reversible *trans-cis* photoisomerization of the azobenzene system. This molecule serves as precursor for the synthesis of azobenzene phospholipids. In *para*-position to the N=N group, a hydrophobic hexyl-sidechain and a hydrophilic hydroxy group were introduced. Two molecules associate together through the hydroxy group by intermolecular hydrogen bond O1A–H1A···O1 ($d$(H1A···O1) = 1.838 Å). One isopropanol molecule coordinates via an additional intermolecular hydrogen bond to the dimer O1L–H1L···O1A ($d$(H1L···O1A) = 1.920 Å). The aliphatic chain has an all-*trans* conformation leading the compound to associate into linear tapes. The bond lengths of the N=N bonds are 1.265 Å for N1=N2 and 1.257 Å for N1A=N2A. The respective torsion angles are ∠C11A–N2A=N1A–C4A = –179.9° and ∠C4–N1=N2–C11 = –179.3°.

* Correspondence author (e-mail: schwalbe@nmr.uni-frankfurt.de)

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

| | |
|---|---|
| Crystal: | orange needle, size 0.10 × 0.14 × 0.37 mm |
| Wavelength: | Mo $K_\alpha$ radiation (0.71073 Å) |
| $\mu$: | 0.74 cm$^{-1}$ |
| Diffractometer, scan mode: | Stoe IPDS II, $\omega$ |
| $2\theta_{max}$: | 51.06° |
| $N(hkl)_{measured}$, $N(hkl)_{unique}$: | 15874, 6561 |
| Criterion for $I_{obs}$, $N(hkl)_{gt}$: | $I_{obs} > 2\,\sigma(I_{obs})$, 2681 |
| $N(param)_{refined}$: | 419 |
| Programs: | SHELXS-97 [2], SHELXL-97 [3] |

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

| Atom | Site | $x$ | $y$ | $z$ | $U_{iso}$ |
|---|---|---|---|---|---|
| H(1) | 2*i* | –0.0427 | 0.3648 | 0.4859 | 0.054 |
| H(2) | 2*i* | –0.1582 | 0.4078 | 0.3783 | 0.049 |
| H(3) | 2*i* | –0.0422 | 0.4647 | 0.2672 | 0.050 |
| H(5) | 2*i* | 0.5479 | 0.5186 | 0.3293 | 0.048 |
| H(6) | 2*i* | 0.4276 | 0.4662 | 0.4413 | 0.049 |
| H(12) | 2*i* | 0.2808 | 0.5823 | 0.0994 | 0.160 |
| H(13) | 2*i* | 0.3969 | 0.6298 | –0.0118 | 0.179 |
| H(15) | 2*i* | 1.0107 | 0.6156 | 0.0297 | 0.058 |
| H(16) | 2*i* | 0.8982 | 0.5679 | 0.1428 | 0.062 |
| H(21A) | 2*i* | 0.7130 | 0.6200 | –0.1086 | 0.069 |
| H(21B) | 2*i* | 0.7088 | 0.7207 | –0.0841 | 0.069 |
| H(22A) | 2*i* | 1.0838 | 0.6083 | –0.1015 | 0.050 |
| H(22B) | 2*i* | 1.0860 | 0.7051 | –0.0692 | 0.050 |
| H(23A) | 2*i* | 0.9713 | 0.6795 | –0.2059 | 0.051 |
| H(23B) | 2*i* | 0.9993 | 0.7747 | –0.1734 | 0.051 |
| H(24A) | 2*i* | 1.3463 | 0.6505 | –0.2071 | 0.053 |
| H(24B) | 2*i* | 1.3776 | 0.7432 | –0.1712 | 0.053 |
| H(25A) | 2*i* | 1.2742 | 0.8231 | –0.2710 | 0.060 |
| H(25B) | 2*i* | 1.2445 | 0.7303 | –0.3069 | 0.060 |
| H(26A) | 2*i* | 1.5801 | 0.7935 | –0.3489 | 0.088 |
| H(26B) | 2*i* | 1.6227 | 0.7004 | –0.3081 | 0.088 |
| H(26C) | 2*i* | 1.6524 | 0.7933 | –0.2721 | 0.088 |
| H(1A) | 2*i* | 0.2599 | 0.3615 | 0.5470 | 0.058 |
| H(2A) | 2*i* | 0.5874 | 0.2690 | 0.6572 | 0.051 |

**Table 2.** Continued.

| Atom | Site | x | y | z | $U_{iso}$ |
|---|---|---|---|---|---|
| H(3A) | 2*i* | 0.4715 | 0.2149 | 0.7695 | 0.050 |
| H(5A) | 2*i* | −0.1468 | 0.2906 | 0.7415 | 0.049 |
| H(6A) | 2*i* | −0.0310 | 0.3468 | 0.6303 | 0.046 |
| H(12A) | 2*i* | 0.1453 | 0.1531 | 0.9519 | 0.067 |
| H(13A) | 2*i* | 0.0339 | 0.0879 | 1.0589 | 0.071 |
| H(15A) | 2*i* | −0.5846 | 0.1137 | 1.0218 | 0.111 |
| H(16A) | 2*i* | −0.4771 | 0.1779 | 0.9128 | 0.114 |
| H(21C) | 2*i* | −0.2657 | 0.0806 | 1.1567 | 0.054 |
| H(21D) | 2*i* | −0.2981 | −0.0126 | 1.1223 | 0.054 |
| H(22C) | 2*i* | −0.6724 | 0.0214 | 1.1183 | 0.055 |
| H(22D) | 2*i* | −0.6401 | 0.1154 | 1.1524 | 0.055 |
| H(23C) | 2*i* | −0.5682 | −0.0556 | 1.2204 | 0.055 |
| H(23D) | 2*i* | −0.5412 | 0.0387 | 1.2545 | 0.055 |
| H(24C) | 2*i* | −0.9226 | 0.0674 | 1.2517 | 0.055 |

**Table 2.** Continued.

| Atom | Site | x | y | z | $U_{iso}$ |
|---|---|---|---|---|---|
| H(24D) | 2*i* | −0.9468 | −0.0290 | 1.2204 | 0.055 |
| H(25C) | 2*i* | −0.8152 | −0.0017 | 1.3546 | 0.061 |
| H(25D) | 2*i* | −0.8341 | −0.0984 | 1.3234 | 0.061 |
| H(26D) | 2*i* | −1.1445 | −0.0691 | 1.3995 | 0.093 |
| H(26E) | 2*i* | −1.2154 | −0.0761 | 1.3230 | 0.093 |
| H(26F) | 2*i* | −1.1958 | 0.0202 | 1.3550 | 0.093 |
| H(1L) | 2*i* | 0.6235 | 0.2969 | 0.5160 | 0.067 |
| H(1LA) | 2*i* | 0.8516 | 0.1890 | 0.5537 | 0.080 |
| H(2L1) | 2*i* | 0.4990 | 0.1497 | 0.5405 | 0.107 |
| H(2L2) | 2*i* | 0.6660 | 0.0703 | 0.5184 | 0.107 |
| H(2L3) | 2*i* | 0.5650 | 0.1329 | 0.4604 | 0.107 |
| H(3L1) | 2*i* | 1.1115 | 0.2142 | 0.4605 | 0.107 |
| H(3L2) | 2*i* | 0.9532 | 0.1746 | 0.4092 | 0.107 |
| H(3L3) | 2*i* | 1.0480 | 0.1105 | 0.4675 | 0.107 |

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

| Atom | Site | x | y | z | $U_{11}$ | $U_{22}$ | $U_{33}$ | $U_{12}$ | $U_{13}$ | $U_{23}$ |
|---|---|---|---|---|---|---|---|---|---|---|
| O(1) | 2*i* | 0.0647(3) | 0.3986(1) | 0.48574(9) | 0.043(1) | 0.056(2) | 0.035(1) | −0.008(1) | 0.0042(9) | 0.008(1) |
| N(1) | 2*i* | 0.3229(4) | 0.5246(2) | 0.2168(1) | 0.043(2) | 0.053(2) | 0.038(1) | 0.004(1) | 0.007(1) | 0.011(1) |
| N(2) | 2*i* | 0.5240(4) | 0.5387(2) | 0.2025(1) | 0.044(2) | 0.039(2) | 0.037(1) | −0.000(1) | 0.009(1) | 0.003(1) |
| C(1) | 2*i* | 0.1249(4) | 0.4306(2) | 0.4190(1) | 0.043(2) | 0.036(2) | 0.028(2) | −0.003(1) | 0.006(1) | 0.004(1) |
| C(2) | 2*i* | −0.0152(4) | 0.4304(2) | 0.3683(1) | 0.035(2) | 0.045(2) | 0.042(2) | −0.000(1) | 0.002(1) | 0.007(1) |
| C(3) | 2*i* | 0.0547(4) | 0.4635(2) | 0.3023(1) | 0.036(2) | 0.048(2) | 0.040(2) | 0.001(1) | 0.000(1) | 0.004(1) |
| C(4) | 2*i* | 0.2654(4) | 0.4949(2) | 0.2870(1) | 0.038(2) | 0.035(2) | 0.039(2) | 0.004(1) | 0.002(1) | 0.005(1) |
| C(5) | 2*i* | 0.4047(4) | 0.4962(2) | 0.3392(1) | 0.039(2) | 0.040(2) | 0.039(2) | −0.004(1) | 0.005(1) | −0.000(1) |
| C(6) | 2*i* | 0.3341(5) | 0.4647(2) | 0.4054(1) | 0.044(2) | 0.043(2) | 0.035(2) | −0.008(1) | −0.002(1) | 0.000(1) |
| C(11) | 2*i* | 0.5784(5) | 0.5693(2) | 0.1322(1) | 0.041(2) | 0.047(2) | 0.036(2) | 0.002(2) | 0.007(1) | 0.010(1) |
| C(12) | 2*i* | 0.4321(6) | 0.5885(4) | 0.0860(2) | 0.030(2) | 0.273(7) | 0.085(3) | 0.018(3) | 0.009(2) | 0.106(4) |
| C(13) | 2*i* | 0.5029(6) | 0.6169(4) | 0.0196(2) | 0.034(2) | 0.324(8) | 0.079(3) | 0.012(3) | −0.001(2) | 0.117(4) |
| C(14) | 2*i* | 0.7158(5) | 0.6275(2) | −0.0036(2) | 0.038(2) | 0.060(2) | 0.040(2) | 0.004(2) | 0.000(1) | 0.011(2) |
| C(15) | 2*i* | 0.8596(5) | 0.6089(2) | 0.0433(1) | 0.033(2) | 0.074(3) | 0.038(2) | −0.014(2) | 0.001(1) | 0.007(2) |
| C(16) | 2*i* | 0.7927(5) | 0.5801(2) | 0.1111(2) | 0.042(2) | 0.074(3) | 0.038(2) | −0.019(2) | −0.007(1) | 0.008(2) |
| C(21) | 2*i* | 0.7779(5) | 0.6609(2) | −0.0777(2) | 0.043(2) | 0.086(3) | 0.041(2) | −0.001(2) | −0.002(1) | 0.022(2) |
| C(22) | 2*i* | 1.0154(4) | 0.6689(2) | −0.1019(1) | 0.040(2) | 0.050(2) | 0.034(2) | −0.001(1) | 0.000(1) | 0.006(1) |
| C(23) | 2*i* | 1.0556(4) | 0.7121(2) | −0.1744(1) | 0.044(2) | 0.050(2) | 0.031(2) | −0.002(2) | 0.001(1) | 0.002(1) |
| C(24) | 2*i* | 1.2918(4) | 0.7132(2) | −0.2037(1) | 0.044(2) | 0.046(2) | 0.040(2) | 0.000(1) | 0.005(1) | 0.005(1) |
| C(25) | 2*i* | 1.3295(5) | 0.7605(2) | −0.2743(1) | 0.049(2) | 0.063(2) | 0.035(2) | −0.003(2) | 0.006(1) | 0.005(2) |
| C(26) | 2*i* | 1.5673(5) | 0.7621(2) | −0.3035(2) | 0.054(2) | 0.063(2) | 0.053(2) | −0.001(2) | 0.021(2) | 0.001(2) |
| O(1A) | 2*i* | 0.3644(3) | 0.3405(2) | 0.56748(9) | 0.034(1) | 0.073(2) | 0.036(1) | −0.006(1) | 0.0052(9) | 0.010(1) |
| N(1A) | 2*i* | 0.0975(4) | 0.2089(2) | 0.8338(1) | 0.042(2) | 0.043(2) | 0.037(1) | −0.007(1) | 0.007(1) | −0.002(1) |
| N(2A) | 2*i* | −0.1033(4) | 0.2084(2) | 0.8523(1) | 0.043(2) | 0.058(2) | 0.036(1) | −0.012(1) | 0.003(1) | 0.003(1) |
| C(1A) | 2*i* | 0.2883(4) | 0.3092(2) | 0.6330(1) | 0.035(2) | 0.045(2) | 0.029(2) | −0.006(1) | 0.004(1) | 0.003(1) |
| C(2A) | 2*i* | 0.4379(4) | 0.2726(2) | 0.6742(1) | 0.034(2) | 0.053(2) | 0.040(2) | −0.004(1) | 0.006(1) | 0.002(2) |
| C(3A) | 2*i* | 0.3691(4) | 0.2408(2) | 0.7408(1) | 0.035(2) | 0.050(2) | 0.039(2) | −0.001(1) | 0.000(1) | 0.007(1) |
| C(4A) | 2*i* | 0.1504(4) | 0.2470(2) | 0.7655(1) | 0.040(2) | 0.035(2) | 0.035(2) | −0.006(1) | 0.004(1) | −0.001(1) |
| C(5A) | 2*i* | 0.0023(4) | 0.2860(2) | 0.7244(1) | 0.036(2) | 0.046(2) | 0.039(2) | −0.005(1) | 0.004(1) | 0.002(1) |
| C(6A) | 2*i* | 0.0701(4) | 0.3185(2) | 0.6582(1) | 0.036(2) | 0.044(2) | 0.035(2) | 0.000(1) | 0.001(1) | −0.003(1) |
| C(11A) | 2*i* | −0.1542(5) | 0.1700(2) | 0.9212(1) | 0.042(2) | 0.048(2) | 0.030(2) | −0.008(1) | 0.003(1) | 0.002(1) |
| C(12A) | 2*i* | −0.0054(5) | 0.1446(2) | 0.9652(2) | 0.035(2) | 0.079(3) | 0.050(2) | 0.006(2) | 0.004(2) | 0.022(2) |
| C(13A) | 2*i* | −0.0735(5) | 0.1065(2) | 1.0293(2) | 0.035(2) | 0.093(3) | 0.047(2) | 0.009(2) | 0.003(1) | 0.025(2) |
| C(14A) | 2*i* | −0.2861(4) | 0.0941(2) | 1.0523(1) | 0.039(2) | 0.044(2) | 0.035(2) | −0.004(1) | 0.002(1) | 0.000(1) |
| C(15A) | 2*i* | −0.4340(5) | 0.1211(3) | 1.0079(2) | 0.036(2) | 0.191(5) | 0.047(2) | −0.032(2) | −0.005(2) | 0.047(3) |
| C(16A) | 2*i* | −0.3702(5) | 0.1593(3) | 0.9426(2) | 0.041(2) | 0.189(5) | 0.053(2) | −0.032(2) | −0.010(2) | 0.051(3) |
| C(21A) | 2*i* | −0.3479(4) | 0.0508(2) | 1.1231(1) | 0.043(2) | 0.055(2) | 0.036(2) | 0.002(2) | 0.003(1) | 0.005(1) |
| C(22A) | 2*i* | −0.5873(4) | 0.0524(2) | 1.1505(1) | 0.042(2) | 0.059(2) | 0.035(2) | −0.003(2) | 0.002(1) | 0.008(2) |
| C(23A) | 2*i* | −0.6250(4) | 0.0068(2) | 1.2224(1) | 0.044(2) | 0.052(2) | 0.038(2) | −0.000(2) | 0.003(1) | 0.014(2) |
| C(24A) | 2*i* | −0.8637(4) | 0.0052(2) | 1.2513(1) | 0.044(2) | 0.049(2) | 0.042(2) | −0.005(2) | 0.001(1) | 0.009(2) |
| C(25A) | 2*i* | −0.8963(5) | −0.0367(2) | 1.3237(1) | 0.053(2) | 0.059(2) | 0.038(2) | −0.004(2) | 0.007(1) | 0.010(2) |
| C(26A) | 2*i* | −1.1342(5) | −0.0408(2) | 1.3529(2) | 0.058(2) | 0.077(3) | 0.047(2) | −0.011(2) | 0.012(2) | 0.006(2) |
| O(1L) | 2*i* | 0.7435(3) | 0.2855(2) | 0.4937(1) | 0.039(1) | 0.061(2) | 0.062(1) | 0.001(1) | 0.015(1) | 0.020(1) |
| C(1L) | 2*i* | 0.8016(5) | 0.1938(3) | 0.5063(2) | 0.060(2) | 0.063(3) | 0.074(2) | 0.000(2) | 0.013(2) | −0.004(2) |
| C(2L) | 2*i* | 0.6173(6) | 0.1314(2) | 0.5064(2) | 0.066(2) | 0.063(3) | 0.082(3) | −0.017(2) | 0.010(2) | −0.001(2) |
| C(3L) | 2*i* | 0.9955(5) | 0.1713(3) | 0.4565(2) | 0.052(2) | 0.075(3) | 0.083(3) | 0.003(2) | 0.013(2) | −0.014(2) |

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