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Crystal structures of *cis*- and *trans*-2-azido-indan-1-ol, $C_9H_9N_3O$

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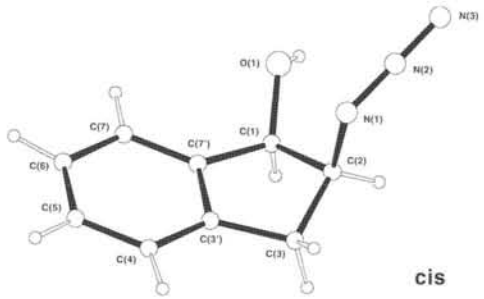
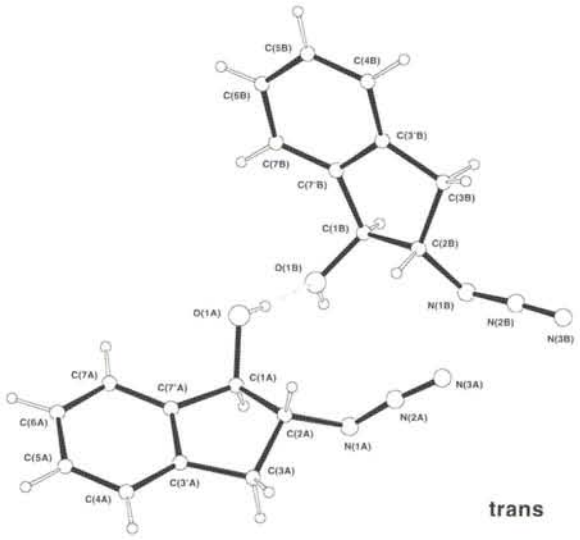


cis



trans

Abstract

$C_9H_9N_3O$, orthorhombic, $P2_12_12_1$ (No. 19), $a = 4.698(1)$ Å, $b = 12.653(1)$ Å, $c = 14.808(2)$ Å, $V = 880.2$ Å^3, $Z = 4$, $R_{gt}(F) = 0.050$, $wR_{ref}(F^2) = 0.099$, $T = 293$ K.

$C_9H_9N_3O$, monoclinic, $P12_1/n1$ (No. 14), $a = 12.763(2)$ Å, $b = 4.774(1)$ Å, $c = 29.048(5)$ Å, $\beta = 97.35(2)°$, $V = 1755.4$ Å^3, $Z = 8$, $R_{gt}(F) = 0.063$, $wR_{ref}(F^2) = 0.138$, $T = 293$ K.

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Source of material

The title compounds were prepared via photoinduced electron transfer oxidation of azide anions [1] in the presence of indene and molecular oxygen [2] and subsequent reduction of the corresponding β-azidohydroperoxides with sodium sulfite. A 65:35 mixture of *cis*- and *trans*-diastereoisomers was isolated which could be separated by column chromatography on silica gel. Recrystallization from methanol resulted in single crystals of pure *cis*- and t*rans*-2-azido-indane-1-ols [3].

The *cis*-compound was crystallized from methanol, mp = 402 K – 403 K [4], the *trans* isomer was crystallized from methanol, mp = 374 K – 375 K.

Discussion

Both compounds are connected in the crystal by consecutive hydrogen bonds. These bonds have identical bond lengths of 1.88 Å (H···O) for the cid-diastereomer, whereas the trans-distereomer has two independent molecules in th eunit cell with two different hydrogen bonds with bond lengths of 1.93 Å and 2.02 Å.

1. *cis*-2-Azido-indan-1-ol, $C_9H_9N_3O$

Table 1. Data collection and handling.

Crystal:	colourless prism, size 0.08 × 0.10 × 0.15 mm
Wavelength:	Mo K_α radiation (0.71069 Å)
μ:	0.91 cm^{-1}
Diffractometer, scan mode:	Nonius CAD4, ω/2θ
$2\theta_{max}$:	49.92°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	942, 942
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 653
$N(param)_{refined}$:	156
Programs:	PLATON [5], SHELXS-97 [6], SHELXL-97 [7], XTAL [8]

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

Atom	Site	x	y	z	U_{iso}
H(1)	4*e*	–0.082(8)	0.073(3)	0.495(2)	0.04(1)
H(2)	4*e*	–0.005(9)	0.067(3)	0.343(2)	0.05(1)
H(3A)	4*e*	0.37(1)	–0.084(3)	0.336(3)	0.07(2)
H(3B)	4*e*	0.06(1)	–0.102(3)	0.388(3)	0.08(2)
H(4)	4*e*	0.61(1)	–0.194(3)	0.475(3)	0.09(2)
H(5)	4*e*	0.84(1)	–0.165(3)	0.617(3)	0.09(2)
H(6)	4*e*	0.73(1)	–0.009(4)	0.695(3)	0.08(2)
H(7)	4*e*	0.41(1)	0.109(3)	0.640(2)	0.04(1)
HO(1)	4*e*	0.01(1)	0.237(4)	0.487(4)	0.07(2)

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}
C(1)	4*e*	0.1142(9)	0.0938(3)	0.4731(3)	0.030(2)	0.042(2)	0.059(3)	–0.005(2)	0.003(3)	–0.005(2)
C(2)	4*e*	0.157(1)	0.0554(4)	0.3764(3)	0.039(3)	0.063(3)	0.057(3)	0.007(3)	–0.001(3)	–0.001(2)
C(3)	4*e*	0.242(1)	–0.0589(4)	0.3857(4)	0.049(3)	0.054(3)	0.068(3)	–0.004(3)	–0.001(3)	–0.019(3)
C(3')	4*e*	0.3958(9)	–0.0612(3)	0.4749(3)	0.036(2)	0.039(2)	0.065(3)	–0.006(2)	0.004(3)	–0.001(2)
C(4)	4*e*	0.584(1)	–0.1335(4)	0.5109(4)	0.054(3)	0.043(3)	0.103(4)	0.003(3)	0.007(3)	0.009(3)
C(5)	4*e*	0.702(1)	–0.1154(5)	0.5944(4)	0.061(4)	0.074(4)	0.098(5)	0.009(3)	0.002(4)	0.040(4)
C(6)	4*e*	0.631(1)	–0.0271(5)	0.6425(4)	0.060(4)	0.096(4)	0.059(3)	–0.007(4)	–0.005(3)	0.024(3)
C(7)	4*e*	0.440(1)	0.0454(4)	0.6084(3)	0.056(3)	0.066(3)	0.051(3)	–0.008(3)	0.012(3)	0.000(3)
C(7')	4*e*	0.3214(8)	0.0283(3)	0.5245(3)	0.031(2)	0.048(2)	0.045(2)	–0.011(2)	0.006(2)	0.001(2)
N(1)	4*e*	0.398(1)	0.1107(3)	0.3331(3)	0.061(3)	0.075(3)	0.064(2)	0.008(3)	0.021(3)	0.012(2)
N(2)	4*e*	0.343(1)	0.1948(4)	0.2966(3)	0.090(4)	0.075(3)	0.069(3)	0.008(3)	0.025(3)	0.007(2)
N(3)	4*e*	0.315(2)	0.2714(4)	0.2592(4)	0.158(6)	0.085(3)	0.121(4)	0.025(5)	0.053(4)	0.039(3)
O(1)	4*e*	0.1540(7)	0.2037(2)	0.4838(2)	0.032(2)	0.042(2)	0.094(2)	0.002(2)	0.003(2)	–0.011(2)

2. *trans*-2-Azido-indan-1-ol, $C_9H_9N_3O$

Table 4. Data collection and handling.

Crystal:	colourless prism, size 0.10 × 0.15 × 0.20 mm
Wavelength:	Mo K_α radiation (0.71069 Å)
μ:	0.91 cm^{-1}
Diffractometer, scan mode:	Nonius CAD4, ω/2θ
$2\theta_{max}$:	49.94°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	2854, 2854
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 1537
$N(param)_{refined}$:	309
Programs:	PLATON [5], SHELXS-97 [6], SHELXL-97 [7] , Xtal [8]

Table 5. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	*x*	*y*	*z*	U_{iso}
H(1A)	4*e*	0.477(2)	–0.449(7)	0.359(1)	0.05(1)
H(2A)	4*e*	0.562(3)	–0.050(9)	0.427(1)	0.07(1)
H(3AA)	4*e*	0.352(3)	–0.27(1)	0.431(2)	0.11(2)
H(3BA)	4*e*	0.405(4)	0.02(1)	0.454(2)	0.12(2)
H(4A)	4*e*	0.241(3)	0.286(9)	0.394(1)	0.09(1)
H(5A)	4*e*	0.201(3)	0.463(9)	0.319(1)	0.09(2)
H(6A)	4*e*	0.301(2)	0.298(8)	0.261(1)	0.06(1)
H(7A)	4*e*	0.442(3)	–0.023(8)	0.280(1)	0.06(1)
HO(1A)	4*e*	0.634(4)	–0.35(1)	0.350(2)	0.13(2)
H(1B)	4*e*	0.828(2)	–0.912(6)	0.3499(9)	0.024(8)
H(2B)	4*e*	0.792(3)	–0.524(8)	0.417(1)	0.07(1)
H(3AB)	4*e*	0.981(3)	–0.471(8)	0.440(1)	0.07(1)
H(3BB)	4*e*	1.001(3)	–0.742(8)	0.414(1)	0.06(1)
H(4B)	4*e*	1.093(3)	–0.217(8)	0.375(1)	0.06(1)
H(5B)	4*e*	1.063(3)	–0.013(9)	0.300(1)	0.08(1)
H(6B)	4*e*	0.914(2)	–0.143(7)	0.250(1)	0.05(1)
H(7B)	4*e*	0.795(3)	–0.468(7)	0.269(1)	0.06(1)
HO(1B)	4*e*	0.672(4)	–0.84(1)	0.345(2)	0.09(2)

Table 6. 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}
C(1A)	4*e*	0.5048(3)	–0.2565(8)	0.3655(1)	0.056(3)	0.031(2)	0.064(3)	–0.007(2)	0.013(2)	0.000(2)
C(2A)	4*e*	0.5134(4)	–0.1992(9)	0.4175(2)	0.101(4)	0.038(3)	0.065(3)	–0.007(3)	–0.004(3)	0.007(2)
C(3A)	4*e*	0.4039(5)	–0.106(1)	0.4258(2)	0.102(4)	0.063(3)	0.070(3)	–0.003(3)	0.029(3)	–0.002(3)
C(3'A)	4*e*	0.3619(3)	0.0321(8)	0.3807(1)	0.057(3)	0.042(2)	0.066(3)	–0.008(2)	0.023(2)	–0.003(2)
C(4A)	4*e*	0.2797(4)	0.219(1)	0.3700(2)	0.057(3)	0.054(3)	0.110(4)	0.001(2)	0.036(3)	–0.018(3)
C(5A)	4*e*	0.2597(4)	0.321(1)	0.3253(2)	0.050(3)	0.056(3)	0.117(5)	0.001(2)	0.004(3)	0.001(3)
C(6A)	4*e*	0.3197(4)	0.238(1)	0.2922(2)	0.063(3)	0.063(3)	0.071(3)	0.000(3)	–0.011(3)	0.001(3)
C(7A)	4*e*	0.4005(3)	0.0482(9)	0.3020(2)	0.051(3)	0.045(2)	0.064(3)	0.003(2)	0.005(2)	–0.008(2)
C(7'A)	4*e*	0.4212(3)	–0.0518(7)	0.3464(1)	0.042(2)	0.027(2)	0.056(2)	–0.007(2)	0.006(2)	0.001(2)
N(1A)	4*e*	0.5499(4)	–0.4482(9)	0.4451(2)	0.154(4)	0.051(3)	0.088(3)	–0.013(3)	–0.030(3)	0.021(2)
N(2A)	4*e*	0.6297(4)	–0.4180(8)	0.4720(1)	0.104(4)	0.051(2)	0.053(3)	0.019(3)	0.025(2)	0.015(2)
N(3A)	4*e*	0.7020(4)	–0.418(1)	0.4987(2)	0.105(4)	0.087(3)	0.080(3)	0.014(3)	0.011(2)	0.026(3)
O(1A)	4*e*	0.6007(2)	–0.2166(6)	0.3466(1)	0.053(2)	0.032(2)	0.113(3)	0.001(1)	0.020(2)	0.008(2)
C(1B)	4*e*	0.8071(3)	–0.7399(8)	0.3579(1)	0.049(3)	0.027(2)	0.057(2)	0.006(2)	0.007(2)	–0.002(2)
C(2B)	4*e*	0.8395(3)	–0.6814(8)	0.4091(1)	0.071(3)	0.033(2)	0.054(3)	–0.002(2)	0.011(2)	0.003(2)
C(3B)	4*e*	0.9537(4)	–0.587(1)	0.4127(2)	0.064(3)	0.046(3)	0.064(3)	0.002(2)	–0.010(2)	–0.008(2)
C(3'B)	4*e*	0.9569(3)	–0.4438(8)	0.3668(1)	0.044(2)	0.034(2)	0.055(2)	0.002(2)	0.003(2)	–0.003(2)
C(4B)	4*e*	1.0283(3)	–0.2478(9)	0.3536(2)	0.041(3)	0.051(3)	0.067(3)	0.002(2)	0.005(2)	–0.012(2)
C(5B)	4*e*	1.0121(3)	–0.1416(9)	0.3094(2)	0.051(3)	0.056(3)	0.079(3)	–0.005(2)	0.023(2)	–0.003(3)
C(6B)	4*e*	0.9264(3)	–0.2254(9)	0.2788(2)	0.071(3)	0.047(3)	0.061(3)	0.007(2)	0.022(2)	0.006(2)
C(7B)	4*e*	0.8553(3)	–0.4220(9)	0.2914(1)	0.052(3)	0.046(2)	0.050(2)	0.007(2)	0.005(2)	–0.006(2)
C(7'B)	4*e*	0.8713(3)	–0.5278(7)	0.3358(1)	0.043(2)	0.029(2)	0.053(2)	0.004(2)	0.006(2)	–0.006(2)
N(1B)	4*e*	0.8184(3)	–0.9306(8)	0.4364(1)	0.123(3)	0.050(2)	0.053(2)	–0.014(2)	0.004(2)	0.010(2)
N(2B)	4*e*	0.8505(3)	–0.9171(8)	0.4779(2)	0.082(3)	0.054(2)	0.076(3)	–0.003(2)	0.001(2)	0.017(2)
N(3B)	4*e*	0.8757(4)	–0.927(1)	0.5166(2)	0.134(4)	0.101(4)	0.077(3)	–0.008(3)	–0.020(3)	0.028(3)
O(1B)	4*e*	0.6975(2)	–0.7119(6)	0.3429(1)	0.046(2)	0.035(2)	0.089(2)	0.002(2)	0.004(1)	0.005(2)

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