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# α-(4′-Iodo-1′-diazocyclohexane)-β-naphthol

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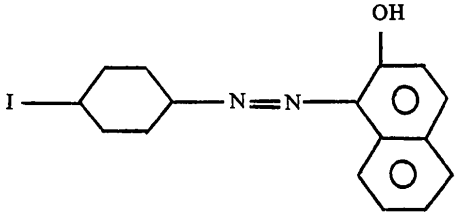
**Abstract.** $C_{16}H_{17}N_2OI$, $M=380{\cdot}06$. Monoclinic, $P2_1/c$; $a=12{\cdot}69$, $b=4{\cdot}198$, $c=27{\cdot}48$ Å, $\beta=111°$; $Z=4$, $D_m=1{\cdot}782$, $D_c=1{\cdot}846$ g cm$^{-3}$. Cu $K\alpha$ radiation, $\lambda=1{\cdot}5418$ Å, $\mu=187{\cdot}0$ cm$^{-1}$. The refinement of the structure converged at $R=0{\cdot}124$; the structural formula of the molecule is :




**Introduction.** Bright red crystals (elongated along the *b* axis) of the title compound were obtained from the Organic Chemistry Department of UMIST, Manchester, England, and the investigation of its crystal and molecular structure was carried out as part of a study of potential carcinogens. The crystals grow in two different forms in the monoclinic system and the present investigation is directed towards the determination of the structure of the frequently occurring variety. The X-ray data were collected by oscillation and Weissenberg techniques and a crystal of approximate dimensions $1\times0{\cdot}50\times0{\cdot}05$ mm was used. The intensities of X-ray reflexions were measured by visual comparison with an exposure chart which yielded 373 independent reflexions. Lorentz and polarization corrections were applied (but none for absorption). The layer scales and the initial temperature factor ($B=3{\cdot}5$ Å$^2$) for all the atoms were obtained from a Wilson (1942) plot.

Systematic absences $h0l$, $l=2n+1$ and $0k0$, $k=2n+1$ confirmed the space group as $P2_1/c$. The structure was solved by the standard heavy-atom method. The position of the heavy atom was deduced from a Patterson synthesis and the remaining non-hydrogen atoms were located from an iodine-phased Fourier synthesis and a difference synthesis which was computed with the iodine contributions of the calculated structure factors being withdrawn from their observed counterparts (Bryan & Tomita, 1962; Sobell & Tomita, 1964). This meant a Fourier synthesis with $(F_o-{}^IF_c)$ as Fourier coefficients. Block-diagonal least-squares refinement reduced $R$ to 0·124, assuming isotropic thermal motions for all the atoms. The weighting scheme was $\sqrt{w}=1/\{1+[(KF_o-b)/a]^2\}^{1/2}$ with $b=5F_{\min}$, and $a=8F_{\min}$, $K$ being a scale factor. Scattering factors were taken from *International Tables for X-ray Crystallography* (1968). As the data were not corrected for absorption, which was presumably high and which also affected the overall isotropic temperature factor $B$, anisotropic refinement was not helpful in refining the structure further. The final atomic parameters are listed in Table 1. The molecular geometry (viewed down the *b* axis) and the numbering of the molecule is shown in Fig. 1. Bond lengths, bond angles and mean plane calculations are listed in Tables 2, 3 and 4, respectively. The orders of magnitude of standard deviations for the positional parameters and bond lengths and angles are as follows: 0·004 Å for positional parameters for iodine; 0·03 Å for the positional parameters of lighter atoms; 0·05 Å for interatomic bond distances; and 3° for valency angles. However, this limited accuracy is somewhat compensated by the overall agreement between chemically equivalent (Wyckoff, 1965) bond lengths and angles (Tables 2 and 3) which suggests that the molecular dimensions have been determined with better accuracy than is estimated by their respective standard deviations.*

* A list of structure factors has been deposited with the British Library Lending Division as Supplementary Publication No. SUP 31708 (10 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.

Table 1. *Final positional parameters* ($\times10^3$) *and isotropic thermal parameters* ($\times10^2$) *of non-hydrogen atoms*

| | $x$ | $y$ | $z$ | $B$ |
|---|---|---|---|---|
| I | 42 | 305 | 185 | 363 |
| C(1) | 334 | 186 | 326 | 418 |
| C(2) | 231 | 311 | 288 | 421 |
| C(3) | 205 | 169 | 235 | 329 |
| C(4) | 280 | −76 | 222 | 406 |
| C(5) | 394 | −185 | 264 | 413 |
| C(6) | 423 | −14 | 316 | 416 |
| C(7) | 647 | −167 | 423 | 316 |
| C(8) | 711 | −253 | 393 | 385 |
| C(9) | 826 | −296 | 417 | 402 |
| C(10) | 863 | −200 | 468 | 365 |
| C(11) | 801 | −133 | 500 | 369 |
| C(12) | 843 | −18 | 550 | 364 |
| C(13) | 778 | 52 | 579 | 318 |
| C(14) | 669 | 125 | 554 | 373 |
| C(15) | 630 | 55 | 502 | 353 |
| C(16) | 690 | −67 | 474 | 356 |
| N(1) | 516 | −160 | 356 | 403 |
| N(2) | 531 | −72 | 400 | 382 |
| O | 634 | −97 | 348 | 384 |

**Discussion.** The structure consists of discrete molecules with no intermolecular contact distances shorter than the sum of the van der Waals radii (Pauling, 1960).

Table 2. *Bond lengths* (Å)

| | | | |
|---|---|---|---|
| I——C(3) | 2·12 | C(1)—C(2) | 1·45 |
| C(1)—C(6) | 1·51 | C(2)—C(3) | 1·50 |
| C(3)—C(4) | 1·52 | C(4)—C(5) | 1·54 |
| C(5)—C(6) | 1·54 | C(7)—C(8) | 1·38 |
| C(7)—C(16) | 1·38 | C(8)—C(9) | 1·38 |
| C(9)—C(10) | 1·37 | C(10)–C(11) | 1·38 |
| C(11)–C(12) | 1·38 | C(11)–C(16) | 1·36 |
| C(12)–C(13) | 1·38 | C(13)–C(14) | 1·35 |
| C(14)–C(15) | 1·36 | C(15)–C(16) | 1·36 |
| C(6)—N(1) | 1·43 | C(7)—N(2) | 1·44 |
| C(8)—O | 1·45 | N(1)—N(2) | 1·21 |

Table 3. *Bond angles* (°)

| | | | |
|---|---|---|---|
| I——C(3)—C(2) | 110·4 | C(9)—C(10)–C(11) | 129·4 |
| I——C(3)—C(4) | 125·5 | C(9)—C(8)—O | 138·1 |
| C(1)—C(2)—C(3) | 112·6 | C(10)–C(11)–C(16) | 115·7 |
| C(2)—C(3)—C(4) | 123·5 | C(11)–C(16)–C(7) | 114·7 |
| C(3)—C(4)—C(5) | 121·7 | C(11)–C(16)–C(15) | 117·6 |
| C(4)—C(5)—C(6) | 113·7 | C(11)–C(12)–C(13) | 124·3 |
| C(5)—C(6)—C(1) | 118·5 | C(12)–C(13)–C(14) | 117·9 |
| C(6)—C(1)—C(2) | 128·2 | C(13)–C(14)–C(15) | 114·2 |
| C(5)—C(6)—N(1) | 112·1 | C(14)–C(15)–C(16) | 127·9 |
| C(6)—N(1)—N(2) | 114·8 | C(16)–C(11)–C(12) | 114·0 |
| N(1)—N(2)—C(7) | 96·6 | C(16)–C(7)—C(8) | 125·5 |
| C(7)—C(8)—C(9) | 119·8 | C(1)—C(6)—N(1) | 125·1 |
| C(7)—C(8)—O | 92·0 | C(16)–C(7)—N(2) | 108·6 |
| C(8)—C(9)—C(10) | 111·5 | C(8)—C(7)—N(2) | 123·4 |

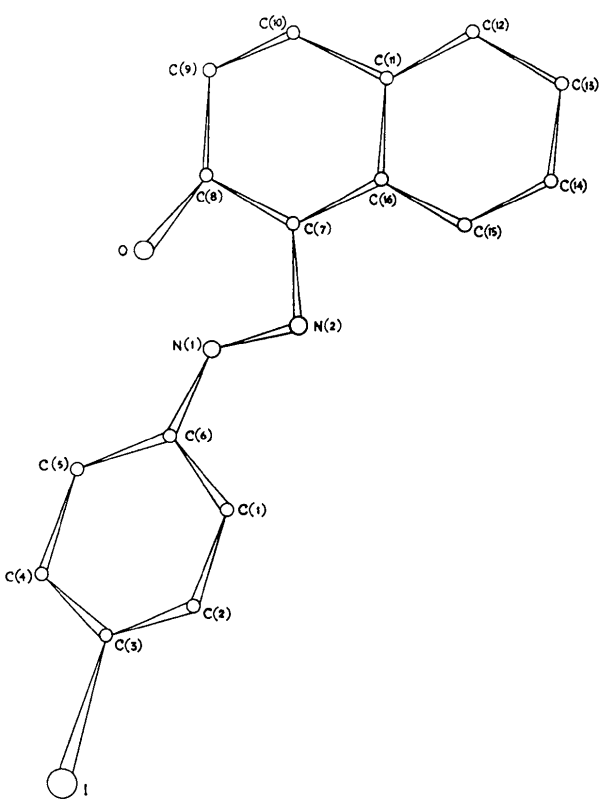



Fig. 1. The molecular geometry (viewed down the *b* axis) and the numbering of the molecule.

It is observed in Table 4 that while the naphthalene part of the molecule has a slightly strained planar configuration, the cyclohexane part exhibits a chair conformation. The estimated value of the dihedral angle between the mean planes of these two parts of the molecule is 20·1° and Fig. 1 reveals that the N=N bond is *trans*.

Table 4. *Parameters of mean planes through sets of atoms and (in square brackets) distances of atoms from the planes*

The equation of a plane is $lx+my+nz=p$, with coordinates and distances (Å) referred to orthogonal axes.

Plane (I) ($l$=0·247, $m$=0·942, $n$=−0·227, $p$=−2·027)
C(7), C(8), C(9), C(10), C(11), C(12), C(13), C(14), C(15), C(16).
[C(7) −0·08, C(8) 0·01, C(9) 0·004, C(10) 0·08, C(11) −0·10, C(12) 0·06, C(13) −0·10, C(14) 0·05, C(15) 0·08, C(16) 0·01, N(2) 0·10, O 0·76]

Plane (II) ($l$=0·570, $m$=0·782, $n$=−0·252, $p$=−0·852)
C(1), C(3), C(4), C(6).
[C(1) −0·06, C(3) 0·05, C(4) −0·05, C(6) 0·05, C(2) 0·07, C(5) −0·09, N(1) −0·24]

Plane (III) ($l$=0·409, $m$=0·900, $n$=−0·150, $p$=−0·306)
C(1), C(2), C(3), C(4), C(5), C(6), C(7), C(8), C(9), C(10), C(11), C(12), C(13), C(14), C(15), C(16), N(1), N(2), O, I.
[C(1) 0·17, C(2) 0·41, C(3) 0·16, C(4) −0·28, C(5) −043, C(6) −0·04, C(7) −0·29, C(8) −0·06, C(9) 0·19, C(10) 0·34, C(11) 0·03, C(12) 0·28, C(13) −0·02, C(14) −0·11, C(15) −0·17, C(16) −0·10, N(1) −0·43, N(2) −0·36, O 0·49, I 0·22]

The calculations were performed on the computers of TIFR, Bombay, I.I.T., Kharagpur, CMERI, Durgapur and the University of Calcutta. The programs used were those of Ahmed, Busing & Levy and Ahmed & Vickery.

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