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3,4,5-Triiodotoluene, C₇H₅I₃

BY YOU-XI LIU, CAROLYN B. KNOBLER, KENNETH N. TRUEBLOOD* AND ROGER HELGESON

J. D. McCullough X-ray Crystallography Laboratory, Department of Chemistry and Biochemistry, University of California, Los Angeles, CA 90024, USA

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Abstract. $M_r = 469.83$, tetragonal, $P\bar{4}2_1m$, $a = 15.407(4)$, $c = 4.348(1)$ Å, $V = 1032.3(4)$ Å³, $Z = 4$, $D_x = 3.02$ g cm⁻³, Mo $K\alpha$, $\lambda = 0.7107$ Å, $\mu = 84.4$ cm⁻¹, $F(000) = 824$, $T = 298$ K, $R = 0.033$ for 572 unique nonzero reflections. A crystallographic mirror plane, normal to the molecular plane, bisects the molecule. With the exception of the H atoms of the methyl group, the molecule is planar. A short intermolecular I...I contact of 3.746(1) Å involves I atoms only in the 3 and 5 positions and links molecules in sheets perpendicular to the c axis.

Introduction. Although 3,4,5-triiodotoluene has been known for some time (Wheeler & Liddle, 1909), its crystal structure has not been reported. Short intermolecular contacts are well known for halogen compounds, including simple organic halides such as *p*-iodotoluene (Ahn, Soled & Carpenter, 1972). Compounds such as hexaiodobenzene have been found to be planar, with short I...I intramolecular and intermolecular contacts (Steer, Watkins & Woodward, 1970).

Experimental. 2,6-Diiodo-4-methylaniline was treated with isoamyl nitrite and dimethyl disulfide at 353 K to produce small amounts of 3,4,5-triiodotoluene (1), as well as the main product 2,6-diiodo-4-methylthioanisole. Colorless parallelepiped of (1) 0.15 ×

0.2 × 0.42 mm; c axis approximately coaxial with fiber mount; Syntex $P\bar{1}$ diffractometer; orientation matrix and unit-cell dimensions from 15 carefully centered reflections with $2\theta < 27^\circ$; intensities measured for $2\theta < 50^\circ$, $k \geq h$ ($h_{\max} = 12$, $k_{\max} = 18$, $l_{\max} = 5$), no indices negative; θ - 2θ scan from 1.0° below Mo $K\alpha_1$ to 1.0° above Mo $K\alpha_2$; scan rate 12.0° min⁻¹; intensities of three standard reflections measured every 97 reflections showed no significant change during data collection; total of 581 independent reflections; 572 independent nonzero reflections used in structure solution and refinement; absorption corrections were made, range of transmission factors 0.515–0.546, ave. 0.532 (applied to F); correction for extinction [$F^* = F_c(1 - 0.0001kF_c^2/\sin 2\theta)$, $k = 0.0020(1)$ (Sheldrick, 1976)]; structure determined by heavy-atom methods and refined by least squares on F (Busing, Martin & Levy, 1962); scattering factors and anomalous-dispersion terms from *International Tables for X-ray Crystallography* (1974); all H atoms were located approximately and fixed in position (C–H = 1.0 Å; for H on ring C 360° – $\angle C-C-C = 2$ $\angle C-C-H$; for H on methyl group $\angle H-C-H = 109.5^\circ$, but methyl not constrained to be in ordered position bisected by the mirror plane through the molecule); refinement completed with anisotropic displacement parameters† for nonhydrogen atoms, isotropic displacement parameters for H atoms (for H of

* To whom correspondence should be addressed.

† More commonly, but misleadingly, called 'vibration parameters' or 'temperature factor parameters'.

the methyl group, a single displacement parameter was refined); all positional parameters refined for non-hydrogen atoms; in all, 58 parameters were refined, $wR = 0.043$, weighted on $1/\sigma^2(F_o)$, $GOF = 1.38$; largest positional-parameter and displacement-parameter shift/error ratios in final cycle of refinement for non-hydrogen atoms were 0.27 and 0.12, respectively; largest peak in final difference map $0.6 \text{ e } \text{Å}^{-3}$ [within 1.0 Å of I(1)]. All calculations performed on a VAX 11/780 using the *UCLA Crystallographic Package* [locally edited versions of *CARESS*, *PROFILE*, *MULTAN*, *ORFLS*, *ORFFE*, *ABSORB* and *ORTEP*] and *SHELX76* (Sheldrick, 1976).

Discussion. The entire molecule, with the exception of the H atoms of the methyl group, is essentially planar. I(1) and I(2) are situated 0.005 (1) and 0.023 (2) Å from the least-squares plane through the six ring C atoms; they deviate 0.001 (1) and 0.018 (2) Å respectively from the unweighted least-squares plane through all 10 non-hydrogen atoms. Within the molecule the distance between I atoms is 3.613 (1) Å. Between molecules, the closest I...I contact is 3.746 (1) Å, noticeably smaller than the van der Waals separation of about 4.0 Å (Bondi, 1964) but almost the same as that found in hexaiodobenzene (3.76 Å) (Steer, Watkins & Woodward, 1970). There are no intermolecular C...C contacts below 3.80 Å, no C...I contacts below 3.80 Å, and no I...H contacts below 3.3 Å. In (1), only I atoms in the positions *meta* to CH_3 are involved in these contacts and each molecule is linked to four other molecules to form sheets perpendicular to the *c* axis. The angles C—I...I of $178.1 (3)$ and $92.1 (3)^\circ$ are a further indication of secondary bonding about I. By use of the relationship between bond order and bond distance proposed by Pauling (Pauling, 1960; McCullough & Knobler, 1976) we find that the above distance corresponds to a bond order of only 0.03. Final atomic parameters for all refined atoms are given in Table 1.* The atom-numbering scheme is illustrated in Fig. 1 (Johnson, 1965). A view of the molecule down *c* is shown in Fig. 2. Bond distances and angles and their estimated standard deviations (Busing, Martin & Levy, 1964) are given in Table 2. The intramolecular geometry is quite normal.

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* Lists of structure factors and anisotropic displacement parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 42091 (6 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. Atomic coordinates and isotropic displacement parameters

$$B_{\text{eq}} = \frac{1}{3} \pi^2 \sum_i \sum_j U_{ij} a_i^* a_j^* a_i a_j$$

	x	y	z	B or $B_{\text{eq}}(\text{Å}^2)$
I(1)	0.8454 (1)	0.0628 (1)	-0.1041 (2)	4.49 (2)
I(2)	0.6283 (1)	0.1283 (1)	0.1076 (4)	5.13 (3)
C(1)	0.7120 (6)	0.2120 (6)	-0.1266 (46)	3.8 (3)
C(2)	0.7959 (7)	0.1857 (6)	-0.2063 (25)	3.4 (3)
C(3)	0.8499 (6)	0.2406 (7)	-0.3635 (28)	3.9 (3)
C(4)	0.8248 (7)	0.3248 (7)	-0.4458 (38)	3.7 (3)
C(5)	0.8848 (8)	0.3848 (8)	-0.6265 (56)	5.4 (3)
H(3)	0.9092	0.2199	-0.4218	5. (3)
H(5A)	0.8568	0.4416	-0.6766	0. (2)
H(5B)	0.9109	0.3596	-0.8171	0. (2)
H(5C)	0.9314	0.3942	-0.4695	0. (2)

Table 2. Interatomic distances (Å) and bond angles ($^\circ$)

I(1)—C(2)	2.089 (9)	I(2)—C(1)—C(2)	120.9 (7)
I(2)—C(1)	2.088 (12)	C(2)—C(1)—C(2')	118.3 (11)
C(1)—C(2)	1.398 (14)	I(1)—C(2)—C(1)	123.2 (7)
C(2)—C(3)	1.370 (14)	C(1)—C(2)—C(3)	120.5 (9)
C(3)—C(4)	1.400 (14)	I(1)—C(2)—C(3)	116.3 (7)
C(4)—C(5)	1.527 (20)	C(2)—C(3)—C(4)	122.1 (9)
		C(3)—C(4)—C(3')	116.6 (10)
		C(3)—C(4)—C(5)	121.7 (9)

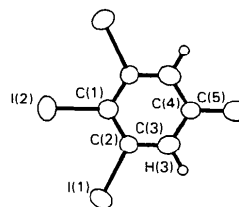


Fig. 1. A view of the molecule, illustrating the numbering system employed. Thermal ellipsoids, except for H, are at the 50% probability level. H atoms have been included with *B* set at 1.0 Å^2 . H atoms (disordered) of the methyl group have been omitted.

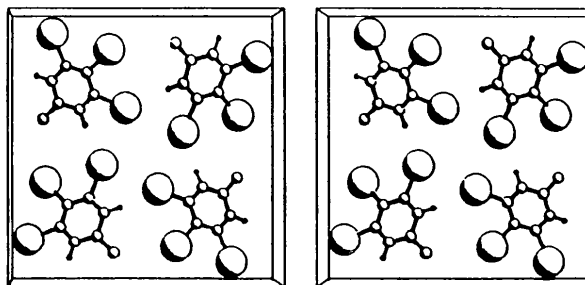


Fig. 2. A view of the structure down the *c* axis, illustrating the short I...I intermolecular contacts around $\frac{1}{2}, \frac{1}{2}, 0$.

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Structure of Dinaphtho[1,2-*b*:2',1'-*d*]furan, C₂₀H₁₂O

BY ROGER E. GERKIN AND WILLIAM J. REPPART

Department of Chemistry, The Ohio State University, Columbus, Ohio 43210, USA

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Abstract. $M_r = 268.32$, monoclinic, $P2_1/c$, $a = 14.309$ (3), $b = 4.665$ (1), $c = 20.827$ (5) Å, $\beta = 109.30$ (2)°, $V = 1312.1$ (9) Å³, $Z = 4$, $D_x = 1.36$ g cm⁻³, $\lambda(\text{Mo } K\alpha) = 0.71069$ Å, $\mu = 0.766$ cm⁻¹, $F(000) = 560$, $T = 294$ K, $R(F) = 0.072$ for 2672 reflections. The dinaphthofuran molecule is non-planar, but displays near twofold symmetry with respect to an axis passing through the O atom and the center of the C(10)–C(20) bond. The C(10)–C(20) bond has a length of 1.438 (3) Å, identical to that found for the corresponding bond in dibenzofuran.

Introduction. The study of dilutely substituted aromatic molecular triplet states by low-field electron paramagnetic resonance spectroscopy is facilitated by knowledge of the crystal structures of the compounds of interest. The crystal structure of dinaphtho[1,2-*b*:2',1'-*d*]furan was determined in part in anticipation of the use of this compound as a host material for such experiments.

Experimental. Dinaphthofuran was synthesized from α -naphthol by the method of Clemo & Spence (1928). The product was a white solid, melting between 457 and 458 K. The identity of the compound was confirmed by ¹H and ¹³C NMR. Clear, acicular crystals grown from acetone featured prominent {100}, {102}, and {10 $\bar{2}}$ faces and easily fractured along [010].

Crystal 0.12 × 0.25 × 0.65 mm mounted along the needle axis (*b*) on a Syntex P $\bar{1}$ diffractometer. Unit-cell parameters and the orientation matrix obtained from a least-squares fit of the setting angles for 22 reflections with 20° < 2 θ < 30°. Intensity data collected for 3815

reflections with +*h*, +*k*, ±*l* and 4° < 2 θ < 55°, using graphite-monochromated Mo *K* α radiation. Six standard reflections measured after every 94 reflections, maximum intensity variation ±4.5%. Space group established by systematic absences. Multiplicative absorption correction factors applied to the F_o 's using the program *DIFABS* (Walker & Stuart, 1984); max. and min. correction factors 1.13 and 0.72, respectively. Averaging of equivalent reflections gave 2791 independent reflections, $R_{\text{int}} = 0.029$. Structure solved using *MULTAN80* (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980). Refinement by full-matrix least squares, using *SHELX76* (Sheldrick, 1976) to minimize $\sum \sigma_F^{-2}(|F_o| - |F_c|)^2$ for 2672 reflections with $F > 0$. Neutral-atom scattering factors for C and O from Cromer & Mann (1968), and for H from Stewart, Davidson & Simpson (1965). H-atom positions determined from an electron density difference map, and refined isotropically. Final refinement cycle: 238 variables, $R = 0.072$, $wR = 0.043$, $w = 1/\sigma^2(F)$, $S = 1.4$, $(\Delta/\sigma)_{\text{max}} = 0.2$. Max. and min. peak heights in final $F_o - F_c$ map 0.19 and -0.20 e Å⁻³.

Discussion. The final atomic coordinates and temperature factors for dinaphthofuran are given in Table 1.* Bond lengths and angles, calculated using *XRAY72* (Stewart, Kruger, Ammon, Dickinson & Hall, 1972), are shown in Figs. 1(*a*) and 1(*b*), respectively. There are

* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 42037 (14 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.