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Research Article

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XRD Data analysis of 1-(2,5-dimethoxyphenylazo)-2-naphtol

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ABSTRACT

The present paper reports the synthesis and XRD characterization of 1- (2, 5- diméthoxyphénylazo)-2-naphtol which are prepared by diazotization and copulation reaction using 2, 5- diméthoxyaniline and β-naphtol as a coupling component. After recrystallization in pentane,the azo-compound was isolated as brown reddish crystal plates. XRD data analysis shows that our compound of empirical formula $C_{18}H_{16}N_2O_3$ crystallizes in space group Pbca orthorhombic system with:

a = 12.09390 Å; b = 15.61670 Å; c = 16.50370 Å; α =90.00°; β = 90.00°;γ =90.00°; Z = 8.

Keywords: X-Ray Diffraction, azo-dyes, diazotization, copulation, chromophore.

INTRODUCTION

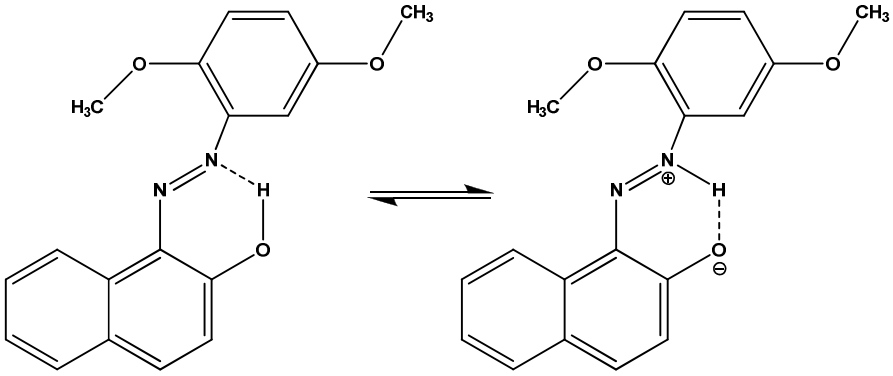
Azo compounds, which were developed in the mid 1800s, are one of the most common dye materials and are useful synthetic intermediates [1, 2]. They have been widely utilized as dyes and analytic reagents; theyare also used in substrates such as textile fibres, leather, plastics, papers, hair, mineral oils, waxes, foodstuffs and cosmetics [3-5]and considered as the most important class of synthetic dyes and pigments, representing 60 - 80% of all organic colorants. Hence azo colorants are part of our everyday colourful life, they are all around us and we could not do without them [6-9].They contain the basic structure of Ar-N=N-Ar'. Their color is due to the high level of conjugation that extends through N-N double bond to the aryl unit [10-11].

RESULTS AND DISCUSSION

The product concerned was prepared according to the classical method of synthesis of an azo dye where an aryl diazonium-cation attacks via electrophilic substitution reaction another aromatic aryl ring ; the reaction is conducted near ice temperatures (0-5°C) at pH mildly acidic or neutral[12-17].

The synthetized azo-compound (scheme 1) is in the form of a red-orange powder; it is not water-soluble, but readily soluble in many organic solvents with a melting point of 156 °C. After recrystallization from pentane, reddish brown crystals, stable in air, were obtained.

XRD recording data of the title compound provides not only the size of the mesh, but also nature and distance of the chemical bonds that allow us to know the geometry and material properties.



Scheme 1:1- (2, 5- diméthoxyphénylazo)-2-naphtol

The monomer crystal unit, that empirical formula $C_{18}H_{16}N_2O_3$,was then analyzed by X-ray diffraction[18-26];the results of the data analysis are presented below:

Table1. ***Crystal data***

$C_{18}H_{16}N_2O_3$	
M_r = 308.33	D_x = 1.314 Mg m^{-3}
Orthorhombic, *Pbca*	Melting point: 429° K
Hall symbol: -P 2ac 2ab	Mo *K*α radiation, λ = 0.71073 Å
a = 12.094 (5) Å	Cellparametersfrom2729reflections
b = 15.617 (5) Å	θ = 2.5–25.5°
c = 16.504 (5) Å	μ = 0.09 mm^{-1}
V = 3117.1 (19) Å^3	*T* = 293 K
Z = 8	Needle
F(000) = 1296	0.09 × 0.04 × 0.02 mm

Table2. ***Data collection***

Kappa CCD diffractometer	1506 reflections with $I > 2\sigma(I)$
Radiation source: fine-focus sealed tube	R_{int} = 0.039
graphite	θ_{max} = 25.5°, θ_{min} = 2.5°
Detector resolution: ? pixels mm^{-1}	*h* = −13 13
φ scans	*k* = −18 18
5124measuredreflections	*l* = −19 19
2729independentreflections	

Table 3.*Refinement*

Refinement onF^2	Secondary atom site location:difference Fourier map
Least-squares matrix:full	Hydrogen site location:inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.055	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2)$ = 0.179	$w = 1/[\sigma^2(F_o^2) + (0.0876P)^2 + 0.613P]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.00	$(\Delta/\sigma)_{max}$ = 0.036
2729reflections	$\Delta\rho_{max}$ = 0.18 e Å^{-3}
212parameters	$\Delta\rho_{min}$ = −0.11 e Å^{-3}
1restraint	Extinction correction:none
?constraints	Extinction coefficient:?
Primary atom site location:structure-invariant direct methods	

Table 4.*Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)*

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	−0.0827 (2)	0.16525 (17)	0.03680 (13)	0.0958 (10)
O2	0.13249 (18)	0.03742 (16)	0.12579 (16)	0.0953 (10)
O3	0.0923 (2)	0.06998 (15)	0.45663 (15)	0.0887 (9)
N1	−0.03311 (18)	0.12696 (15)	0.18240 (16)	0.0630 (9)
N2	−0.11602 (16)	0.17362 (14)	0.20761 (13)	0.0565 (8)
C1	0.0381 (2)	0.08927 (16)	0.23904 (19)	0.0600 (10)
C2	0.1272 (2)	0.04151 (19)	0.2085 (2)	0.0709 (11)
C3	0.1999 (2)	0.00431 (19)	0.2628 (3)	0.0786 (13)
C4	0.1856 (2)	0.0146 (2)	0.3445 (2)	0.0814 (14)
C5	0.0985 (2)	0.06239 (18)	0.3748 (2)	0.0676 (11)
C6	0.0238 (2)	0.09948 (17)	0.32143 (18)	0.0606 (10)
C7	−0.1799 (2)	0.21258 (17)	0.15324 (16)	0.0578 (9)
C8	−0.1611 (3)	0.2097 (2)	0.06693 (19)	0.0750 (12)
C9	−0.2344 (3)	0.2576 (3)	0.0165 (2)	0.0907 (16)
C10	−0.3191 (3)	0.3023 (2)	0.0478 (3)	0.0913 (16)
C11	−0.3426 (2)	0.30649 (19)	0.1322 (2)	0.0729 (13)
C12	−0.2725 (2)	0.26125 (17)	0.18591 (19)	0.0599 (10)
C13	−0.2957 (2)	0.26399 (18)	0.2683 (2)	0.0696 (11)
C14	−0.3862 (3)	0.3087 (2)	0.2971 (2)	0.0867 (14)
C15	−0.4546 (3)	0.3524 (2)	0.2448 (3)	0.1017 (18)
C16	−0.4329 (3)	0.3515 (2)	0.1635 (3)	0.0967 (16)
C17	0.2144 (3)	−0.0156 (3)	0.0910 (3)	0.1183 (19)
C18	0.0011 (3)	0.1166 (2)	0.4890 (2)	0.0917 (16)
H1	−0.025 (3)	0.117 (2)	0.1303 (5)	0.104 (13)*
H3	0.25904	−0.02794	0.24359	0.0941*
H4	0.23511	−0.01096	0.38022	0.0975*
H6	−0.03557	0.13106	0.34111	0.0728*
H9	−0.22325	0.25788	−0.03923	0.1090*
H10	−0.36494	0.33209	0.01237	0.1096*
H13	−0.24975	0.23541	0.30449	0.0834*
H14	−0.40096	0.30931	0.35238	0.1038*
H15	−0.51534	0.38234	0.26460	0.1218*
H16	−0.47902	0.38135	0.12832	0.1159*
H17A	0.20923	−0.01289	0.03305	0.1771*
H17B	0.20350	−0.07355	0.10861	0.1771*
H17C	0.28622	0.00373	0.10783	0.1771*
H18A	0.00597	0.11728	0.54709	0.1374*
H18B	−0.06672	0.08952	0.47296	0.1374*
H18C	0.00267	0.17420	0.46888	0.1374*

Table 5.*Atomic displacement parameters (Å^2)*

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0984 (17)	0.123 (2)	0.0659 (15)	0.0002 (15)	0.0164 (12)	−0.0039 (13)
O2	0.0896 (16)	0.0975 (18)	0.0987 (19)	0.0222 (12)	0.0280 (13)	−0.0069 (14)
O3	0.0953 (16)	0.0878 (16)	0.0831 (17)	0.0083 (12)	−0.0237 (12)	−0.0001 (13)
N1	0.0575 (14)	0.0650 (15)	0.0666 (17)	−0.0001 (11)	0.0066 (11)	−0.0008 (13)
N2	0.0483 (12)	0.0542 (13)	0.0669 (15)	−0.0013 (10)	0.0026 (10)	−0.0018 (11)
C1	0.0482 (15)	0.0508 (16)	0.081 (2)	−0.0027 (12)	0.0012 (13)	0.0001 (14)
C2	0.0586 (17)	0.0611 (19)	0.093 (2)	−0.0022 (13)	0.0143 (16)	−0.0060 (17)
C3	0.0549 (17)	0.061 (2)	0.120 (3)	0.0066 (13)	−0.0009 (17)	−0.0032 (19)
C4	0.0621 (19)	0.065 (2)	0.117 (3)	0.0054 (15)	−0.0202 (18)	0.0041 (19)
C5	0.0628 (18)	0.0570 (18)	0.083 (2)	−0.0038 (14)	−0.0108 (14)	−0.0002 (16)
C6	0.0520 (15)	0.0527 (16)	0.077 (2)	0.0018 (12)	−0.0023 (13)	−0.0016 (14)
C7	0.0540 (15)	0.0608 (17)	0.0585 (17)	−0.0090 (12)	−0.0050 (12)	0.0049 (14)
C8	0.071 (2)	0.085 (2)	0.069 (2)	−0.0171 (16)	−0.0018 (16)	0.0036 (18)
C9	0.094 (3)	0.110 (3)	0.068 (2)	−0.022 (2)	−0.0201 (18)	0.018 (2)
C10	0.082 (2)	0.088 (3)	0.104 (3)	−0.0185 (19)	−0.036 (2)	0.029 (2)
C11	0.0604 (18)	0.0603 (19)	0.098 (3)	−0.0129 (14)	−0.0196 (16)	0.0142 (17)
C12	0.0481 (14)	0.0515 (16)	0.080 (2)	−0.0084 (11)	−0.0090 (12)	0.0052 (14)
C13	0.0608 (17)	0.064 (2)	0.084 (2)	0.0031 (14)	0.0034 (14)	0.0011 (15)
C14	0.069 (2)	0.070 (2)	0.121 (3)	0.0083 (16)	0.0150 (19)	−0.007 (2)
C15	0.064 (2)	0.074 (3)	0.167 (4)	0.0113 (16)	0.013 (2)	0.001 (3)
C16	0.060 (2)	0.072 (2)	0.158 (4)	0.0012 (16)	−0.021 (2)	0.022 (2)
C17	0.098 (3)	0.116 (3)	0.141 (4)	0.022 (2)	0.048 (2)	−0.025 (3)
C18	0.105 (3)	0.093 (3)	0.077 (2)	0.004 (2)	−0.012 (2)	−0.0060 (19)

Table 6.*Geometricparameters (Å, °)*

O1—C8	1.276 (4)	C11—C12	1.416 (4)
O2—C2	1.368 (4)	C12—C13	1.389 (5)
O2—C17	1.413 (5)	C13—C14	1.383 (4)
O3—C5	1.358 (4)	C14—C15	1.377 (5)
O3—C18	1.426 (4)	C15—C16	1.367 (7)
N1—N2	1.308 (3)	C3—H3	0.9300
N1—C1	1.401 (4)	C4—H4	0.9300
N2—C7	1.331 (3)	C6—H6	0.9300
N1—H1	0.879 (11)	C9—H9	0.9300
C1—C2	1.404 (4)	C10—H10	0.9300
C1—C6	1.380 (4)	C13—H13	0.9300
C2—C3	1.383 (5)	C14—H14	0.9300
C3—C4	1.369 (6)	C15—H15	0.9300
C4—C5	1.385 (4)	C16—H16	0.9300
C5—C6	1.388 (4)	C17—H17A	0.9600
C7—C8	1.443 (4)	C17—H17B	0.9600
C7—C12	1.457 (4)	C17—H17C	0.9600
C8—C9	1.428 (5)	C18—H18A	0.9600
C9—C10	1.343 (5)	C18—H18B	0.9600
C10—C11	1.423 (6)	C18—H18C	0.9600
C11—C16	1.398 (5)		
O1···N1	2.548 (4)	C10···H18C^{v}	2.9500
O1···N2	2.851 (3)	C14···H3^{i}	3.0500
O2···N1	2.615 (3)	C15···H3^{i}	3.0200
O3···C18	3.251 (4)	C17···H3	2.5800
O3···C17^{ii}	3.332 (5)	C18···H6	2.4900
O1···H1	1.85 (2)	C18···H14^{xi}	3.1000
O1···H10^{iii}	2.7600	H1···O1	1.85 (2)
O2···H1	2.28 (3)	H1···O2	2.28 (3)
O3···H17A^{ii}	2.8500	H1···C8	2.43 (3)
O3···H18B	2.7700	H3···C17	2.5800
N1···O1	2.548 (4)	H3···H17B	2.4300
N1···O2	2.615 (3)	H3···H17C	2.3200
N1···C14^{iv}	3.366 (4)	H3···C14^{xii}	3.0500
N2···O1	2.851 (3)	H3···C15^{xii}	3.0200
N2···H6	2.5000	H6···N2	2.5000
N2···H13	2.4700	H6···C18	2.4900
C1···C13^{iv}	3.391 (4)	H6···H18B	2.3000
C1···C14^{iv}	3.597 (4)	H6···H18C	2.2600
C4···C7^{v}	3.494 (4)	H10···H16	2.4800
C5···C7^{v}	3.591 (4)	H10···O1^{xiii}	2.7600
C6···C12^{iv}	3.531 (4)	H13···N2	2.4700
C7···C5^{v}	3.591 (4)	H14···C18^{xiv}	3.1000
C7···C4^{v}	3.494 (4)	H14···H18A^{xiv}	2.3100
C8···C18^{i}	3.586 (5)	H15···C2^{xv}	2.9800
C9···C18^{i}	3.490 (5)	H15···C3^{xv}	2.9300
C12···C6^{v}	3.531 (4)	H16···H10	2.4800
C13···C1^{v}	3.391 (4)	H17A···O3^{ii}	2.8500
C14···N1^{v}	3.366 (4)	H17B···C3	2.8200
C14···C1^{v}	3.597 (4)	H17B···H3	2.4300
C17···O3^{ii}	3.332 (5)	H17C···C3	2.7600
C18···C8^{iii}	3.586 (5)	H17C···H3	2.3200
C18···O3^{i}	3.251 (4)	H17C···H18B^{iv}	2.6000
C18···C9^{iii}	3.490 (5)	H18A···H14^{xi}	2.3100
C2···H15^{ix}	2.9800	H18B···C6	2.7300
C3···H17C	2.7600	H18B···H6	2.3000
C3···H17B	2.8200	H18B···O3^{i}	2.7700
C3···H15^{ix}	2.9300	H18B···H17C^{v}	2.6000
C6···H18B	2.7300	H18C···C6	2.7100
C6···H18C	2.7100	H18C···H6	2.2600
C8···H1	2.43 (3)	H18C···C10^{iv}	2.9500
C2—O2—C17	117.8 (3)	C13—C14—C15	120.7 (3)
C5—O3—C18	117.4 (2)	C14—C15—C16	119.7 (3)
N2—N1—C1	119.6 (2)	C11—C16—C15	121.2 (4)
N1—N2—C7	119.0 (2)	C2—C3—H3	120.00
C1—N1—H1	121 (2)	C4—C3—H3	120.00
N2—N1—H1	120 (2)	C3—C4—H4	120.00
C2—C1—C6	120.8 (3)	C5—C4—H4	119.00
N1—C1—C2	117.1 (3)	C1—C6—H6	120.00

N1—C1—C6	122.1 (2)	C5—C6—H6	120.00
O2—C2—C3	126.7 (3)	C8—C9—H9	119.00
O2—C2—C1	114.8 (3)	C10—C9—H9	119.00
C1—C2—C3	118.6 (3)	C9—C10—H10	118.00
C2—C3—C4	120.6 (3)	C11—C10—H10	118.00
C3—C4—C5	121.0 (3)	C12—C13—H13	120.00
C4—C5—C6	119.4 (3)	C14—C13—H13	120.00
O3—C5—C4	116.6 (3)	C13—C14—H14	120.00
O3—C5—C6	124.0 (2)	C15—C14—H14	120.00
C1—C6—C5	119.7 (2)	C14—C15—H15	120.00
C8—C7—C12	120.2 (3)	C16—C15—H15	120.00
N2—C7—C8	124.0 (2)	C11—C16—H16	119.00
N2—C7—C12	115.8 (2)	C15—C16—H16	119.00
C7—C8—C9	117.5 (3)	O2—C17—H17A	109.00
O1—C8—C9	121.3 (3)	O2—C17—H17B	109.00
O1—C8—C7	121.2 (3)	O2—C17—H17C	109.00
C8—C9—C10	121.5 (3)	H17A—C17—H17B	110.00
C9—C10—C11	123.6 (4)	H17A—C17—H17C	109.00
C12—C11—C16	119.2 (3)	H17B—C17—H17C	109.00
C10—C11—C12	118.1 (3)	O3—C18—H18A	109.00
C10—C11—C16	122.8 (3)	O3—C18—H18B	109.00
C11—C12—C13	118.5 (2)	O3—C18—H18C	110.00
C7—C12—C11	119.3 (3)	H18A—C18—H18B	109.00
C7—C12—C13	122.3 (2)	H18A—C18—H18C	110.00
C12—C13—C14	120.8 (3)	H18B—C18—H18C	110.00
C17—O2—C2—C1	−174.7 (3)	N2—C7—C8—C9	−177.6 (3)
C17—O2—C2—C3	5.7 (4)	C12—C7—C8—O1	−177.7 (3)
C18—O3—C5—C4	−178.0 (3)	C12—C7—C8—C9	1.5 (4)
C18—O3—C5—C6	2.2 (4)	N2—C7—C12—C11	178.3 (2)
C1—N1—N2—C7	176.7 (2)	N2—C7—C12—C13	−2.6 (4)
N2—N1—C1—C2	−178.6 (2)	C8—C7—C12—C11	−0.9 (4)
N2—N1—C1—C6	0.1 (4)	C8—C7—C12—C13	178.2 (3)
N1—N2—C7—C8	−2.7 (4)	O1—C8—C9—C10	177.9 (4)
N1—N2—C7—C12	178.2 (2)	C7—C8—C9—C10	−1.3 (5)
N1—C1—C2—O2	−0.5 (4)	C8—C9—C10—C11	0.5 (6)
N1—C1—C2—C3	179.2 (2)	C9—C10—C11—C12	0.2 (5)
C6—C1—C2—O2	−179.2 (2)	C9—C10—C11—C16	−178.2 (4)
C6—C1—C2—C3	0.5 (4)	C10—C11—C12—C7	0.0 (4)
N1—C1—C6—C5	−178.4 (2)	C10—C11—C12—C13	−179.1 (3)
C2—C1—C6—C5	0.2 (4)	C16—C11—C12—C7	178.5 (3)
O2—C2—C3—C4	179.1 (3)	C16—C11—C12—C13	−0.6 (4)
C1—C2—C3—C4	−0.6 (4)	C10—C11—C16—C15	178.3 (3)
C2—C3—C4—C5	−0.1 (4)	C12—C11—C16—C15	−0.1 (5)
C3—C4—C5—O3	−179.0 (3)	C7—C12—C13—C14	−178.0 (3)
C3—C4—C5—C6	0.8 (4)	C11—C12—C13—C14	1.1 (4)
O3—C5—C6—C1	178.9 (3)	C12—C13—C14—C15	−0.8 (5)
C4—C5—C6—C1	−0.9 (4)	C13—C14—C15—C16	0.1 (5)
N2—C7—C8—O1	3.2 (5)	C14—C15—C16—C11	0.4 (5)

Symmetry codes: (i) −*x*, −*y*, −*z*+1; (ii) −*x*+1/2, −*y*, *z*+1/2; (iii) *x*+1/2, −*y*+1/2, −*z*; (iv) *x*+1/2, *y*, −*z*+1/2; (v) *x*−1/2, *y*, −*z*+1/2; (vi) *x*, −*y*+1/2, *z*−1/2; (vii) −*x*+1/2, −*y*, *z*−1/2; (viii) *x*, −*y*+1/2, *z*+1/2; (ix) −*x*−1/2, *y*−1/2, *z*; (x) −*x*, *y*+1/2, −*z*+1/2; (xi) *x*+1/2, −*y*+1/2, −*z*+1; (xii) −*x*, *y*−1/2, −*z*+1/2; (xiii) *x*−1/2, −*y*+1/2, −*z*; (xiv) *x*−1/2, −*y*+1/2, −*z*+1; (xv) −*x*−1/2, *y*+1/2, *z*.

Table 7.*Hydrogen-bond geometry (Å, °)*

D*—H···*A	***D*—H**	**H···*A***	***D*···*A***	***D*—H···*A***
N1—H1···O1	0.879 (11)	1.85 (2)	2.548 (4)	134 (3)
N1—H1···O2	0.879 (11)	2.28 (3)	2.615 (3)	103 (2)

Bond distances, angles etc. have been calculated using the rounded fractional coordinates. All su's are estimated from the variances of the (full) variance-covariance matrix. The cell esds are taken into account in the estimation of distances, angles and torsion angles. The structure of the title compound in the single crystal is given in **figure 1**.

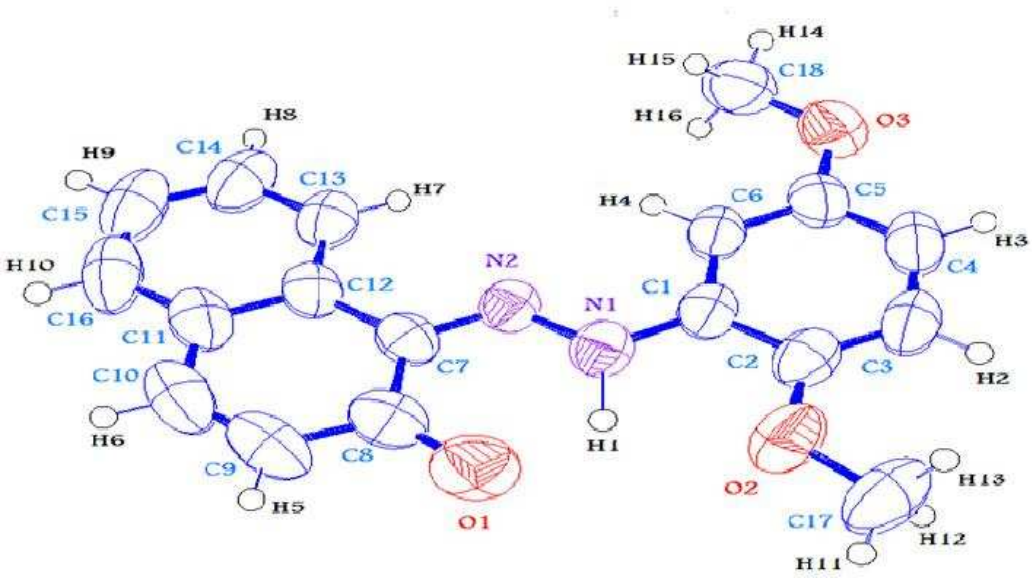


Figure 1:*Structure of (1-(2,5-diméthoxyphénylazo)-2-naphtol)*

CONCLUSION

XRD data analysis of the structure shows a crystal monomer unit of empirical formula $C_{18}H_{16}N_2O_3$ witch crystallizes in space group P_{bca}orthorhombic system.The packing diagram of the compound is given in **figure 2**.

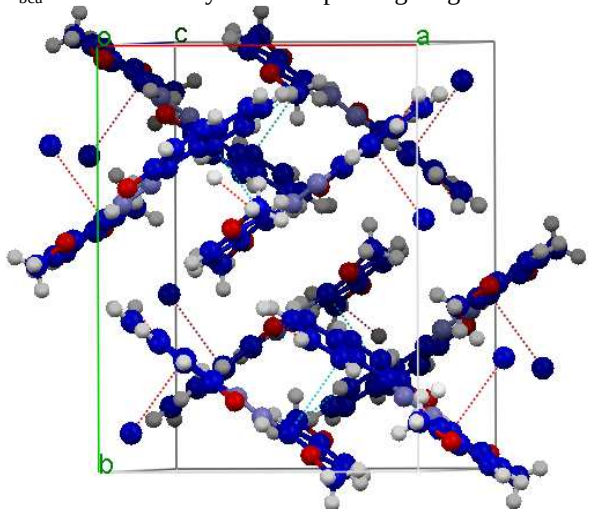


Figure 2:*The packing diagram of thecompound withinteratomic interactions bonds.*

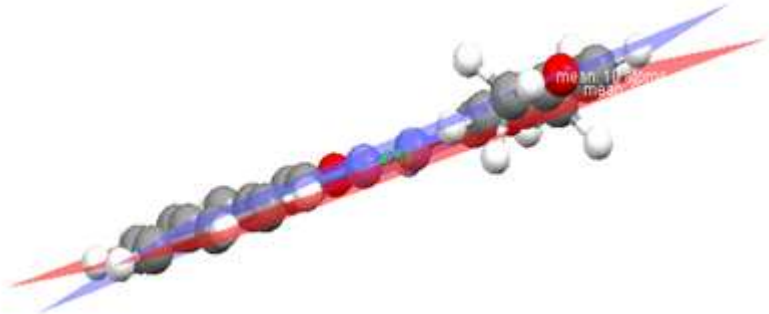


Figure 3:*Angle between naphthalene and benzene ring systems*

The angle between the naphthalene and the benzene ring systems is 6.69 °. Therefore this deviation shows that the structure is not completely planar.

Computing details
Data collection: Collect (Bruker AXS BV, 1997-2004); **cell refinement**: *HKLDENZO* and *SCALEPACK* (Otwinowski& Minor 1997); **data reduction**: *HKLDENZO* and *SCALEPACK* (Otwinowski& Minor 1997); **program(s) used to solve structure:***SHELXL97* (Sheldrick, 1997); **program(s) used to refine structur**e: *SHELXL97* (Sheldrick, 1997); **molecular graphics**: *ORTEP-3 for Windows* (Farrugia, 1997) and *PLATON* (Spek, 2010); **software used to prepare material for publicatio**n: *PLATON* (Spek, 2003).

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