X-RAY ANALYSIS OF THE COMPOUND

$C_{24}H_{23}NO_2$: Mr = 357.46, monoclinic, $P2_1$, a = 10.106(1), b=11.173(2), c = 8.710(2) Å, β = 99.93(2)°, V = 968.7(3) $Å^{-3}$, Z = 2 , D_x = 1.225 $Mg.m^{-3}$, $\lambda(MoK\alpha)$ = 0.70926Å , μ = 0.72 cm^{-1} , F(000) = 380 , T = 294 K , final R = 0.036 for 1471 observations.

The sample (0.40*0.40*0.55 mm) is studied on an automatic diffractometer CAD4 ENRAF-NONIUS with graphite monochromatized MoKα radiation.The cell parameters are obtained by fitting a set of 25 high-theta reflections . The data collection ($2\theta_{max}$ = 50°, scan $\omega/2\theta$ = 1, t_{max} = 60 s, range HKL : H -12,12 K 0,13 L 0,10 , intensity controls without appreciable decay (0.3%) gives 1927 reflections from which 1471 independent (Rint = 0.011) with $I>2\sigma(I)$.

After Lorenz and polarization corrections the structure was solved with Direct Methods which reveal all the non hydrogen atoms of the molecule. After isotropic (R = 0.11), then anisotropic refinement (R = 0.083), the hydrogene atoms are found with a Fourier Difference (between 0.45 and 0.15 $eÅ^{-3}$). The whole structure was refined by the full-matrix least-square techniques (use of F magnitude ; x , y, z, β_{ij} for N, O and C atoms and x, y, z for H atoms ; 313 variables and 1471 observations ; $w = 1/\sigma(F_O)^2 = [\sigma^2(I) + (0.04F_O^2)^2]^{-1/2}$) with the resulting R = 0.050, R_w = 0.036 and S_w= 2.02 (residual $\Delta\rho \leq 0.12$ $eÅ^{-3}$).

Atomic scattering factors from International Tables for X-ray Crystallography (1974). All the calculations were performed on a Digital MicroVAX 3100 computer with the MOLEN package (Fair , 1990) .

References

FAIR, C.K. (1990). MolEN. An Interactive Intelligent System for Crystal Structure Analysis. Enraf-Nonius, Delft, The Netherlands.

International Tables for X-ray Crystallography (1974). Vol.IV.Birmingham : Kynoch Press. (Present distributeur D.Reidel, Dordrecht.)

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Table of Positional Parameters and Their Estimated Standard Deviations

Atom	x	y	z	B(A2)
O2	0.613	0.7217	0.6538(3)	6.50(6)
O1	0.5755(2)	0.9435(2)	0.5513(3)	6.36(5)
N	0.7229(2)	0.5498(2)	0.7066(2)	3.99(5)
C1	0.7213(3)	0.6682(3)	0.6867(3)	4.57(6)
C2	0.8528(3)	0.7361(3)	0.7039(3)	5.01(7)
C3	0.9708(3)	0.6597(3)	0.6839(3)	4.54(6)
C4	1.0857(3)	0.7085(3)	0.6401(3)	5.92(7)
C5	1.1919(3)	0.6377(4)	0.6222(4)	6.67(8)
C6	1.1884(3)	0.5163(4)	0.6471(4)	6.37(9)
C7	1.0760(3)	0.4656(3)	0.6895(3)	5.00(6)
C8	0.9673(3)	0.5370(3)	0.7081(3)	4.05(5)
C9	0.8470(3)	0.4810(3)	0.7562(3)	4.39(6)
C10	0.8764(4)	0.8093(3)	0.8574(4)	5.69(8)
C11	0.8910(3)	0.7348(3)	1.0019(3)	4.70(6)
C12	1.0152(3)	0.6915(3)	1.0704(3)	5.24(7)
C13	1.0312(4)	0.6240(3)	1.2029(4)	6.33(9)
C14	0.9218(5)	0.5971(4)	1.2694(4)	7.5(1)
C15	0.7976(4)	0.6382(4)	1.2039(4)	7.7(1)
C16	0.7814(3)	0.7076(4)	1.0715(4)	6.09(8)
C17	0.5942(2)	0.4856(3)	0.6745(3)	4.06(5)
C18	0.4389(3)	0.9625(3)	0.5012(3)	4.87(6)
C19	0.5934(2)	0.3796(3)	0.7821(3)	4.17(6)
C20	0.6007(3)	0.4003(3)	0.9406(4)	5.43(7)
C21	0.6075(4)	0.3057(4)	1.0441(4)	6.59(9)
C22	0.6044(4)	0.1917(4)	0.9918(4)	6.93(8)
C23	0.5943(3)	0.1693(3)	0.8375(4)	6.10(8)
C24	0.5895(3)	0.2622(3)	0.7326(3)	4.91(6)

Table of Bond Angles in Degrees

Atom 1	Atom 2	Atom 3	Angle
C18	O1	H1	115.(1)
C1	N	C9	123.1(2)
C1	N	C17	117.9(2)
C9	N	C17	118.9(2)
O2	C1	N	120.1(3)
O2	C1	C2	120.3(3)
N	C1	C2	119.6(2)
C1	C2	C3	113.8(3)
C1	C2	C10	110.6(3)
C1	C2	H2	102.(2)
C3	C2	C10	113.1(2)
C3	C2	H2	113.(2)
C10	C2	H2	104.(2)
C2	C3	C4	121.7(3)
C2	C3	C8	120.2(3)
C4	C3	C8	118.2(3)
C3	C4	C5	120.9(3)
C3	C4	H4	114.(1)
C5	C4	H4	125.(1)
C4	C5	C6	120.8(3)
C4	C5	H5	122.(2)
C6	C5	H5	117.(2)
C5	C6	C7	119.6(3)
C5	C6	H6	128.(2)
C7	C6	H6	113.(2)
C6	C7	C8	120.0(3)
C6	C7	H7	119.(1)
C8	C7	H7	121.(1)
C3	C8	C7	120.5(3)
C3	C8	C9	120.1(3)
C7	C8	C9	119.4(3)
N	C9	C8	113.2(2)
N	C9	H9a	107.(2)
N	C9	H9b	106.(1)
C8	C9	H9a	110.(1)
C8	C9	H9b	105.(2)
H9a	C9	H9b	117.(2)
C2	C10	C11	114.2(3)
C2	C10	H10a	106.(1)
C2	C10	H10b	113.(1)
C11	C10	H10a	108.(1)
C11	C10	H10b	101.(2)
H10a	C10	H10b	116.(2)
C10	C11	C12	120.6(3)
C10	C11	C16	121.4(3)
C12	C11	C16	118.0(3)
C11	C12	C13	121.7(3)
C11	C12	H12	122.(1)
C13	C12	H12	116.(1)
C12	C13	C14	119.5(3)
C12	C13	H13	117.(2)
C14	C13	H13	123.(1)
C13	C14	C15	120.1(3)
C13	C14	H14	117.(1)
C15	C14	H14	123.(1)
C14	C15	C16	120.5(4)
C14	C15	H15	122.(2)

Bond Angles (cont.)

Atom 1	Atom 2	Atom 3	Angle
C16	C15	H15	117.(2)
C11	C16	C15	120.2(3)
C11	C16	H16	115.(2)
C15	C16	H16	124.(2)
N	C17	C19	111.3(2)
N	C17	H17	105.(2)
C19	C17	H17	109.(2)
O1	C18	H18a	106.(1)
O1	C18	H18b	106.(1)
H18a	C18	H18b	107.(2)
C17	C19	C20	118.7(3)
C17	C19	C24	123.7(2)
C20	C19	C24	117.6(3)
C19	C20	C21	120.5(3)
C19	C20	H20	119.(1)
C21	C20	H20	120.(1)
C20	C21	C22	120.4(3)
C20	C21	H21	120.(2)
C22	C21	H21	119.(2)
C21	C22	C23	120.1(4)
C21	C22	H22	119.(2)
C23	C22	H22	121.(2)
C22	C23	C24	120.5(3)
C22	C23	H23	120.(2)
C24	C23	H23	119.(2)
C19	C24	C23	120.8(3)
C19	C24	H24	119.(2)
C23	C24	H24	120.(2)

Numbers in parentheses are estimated standard deviations in the least significant digits.

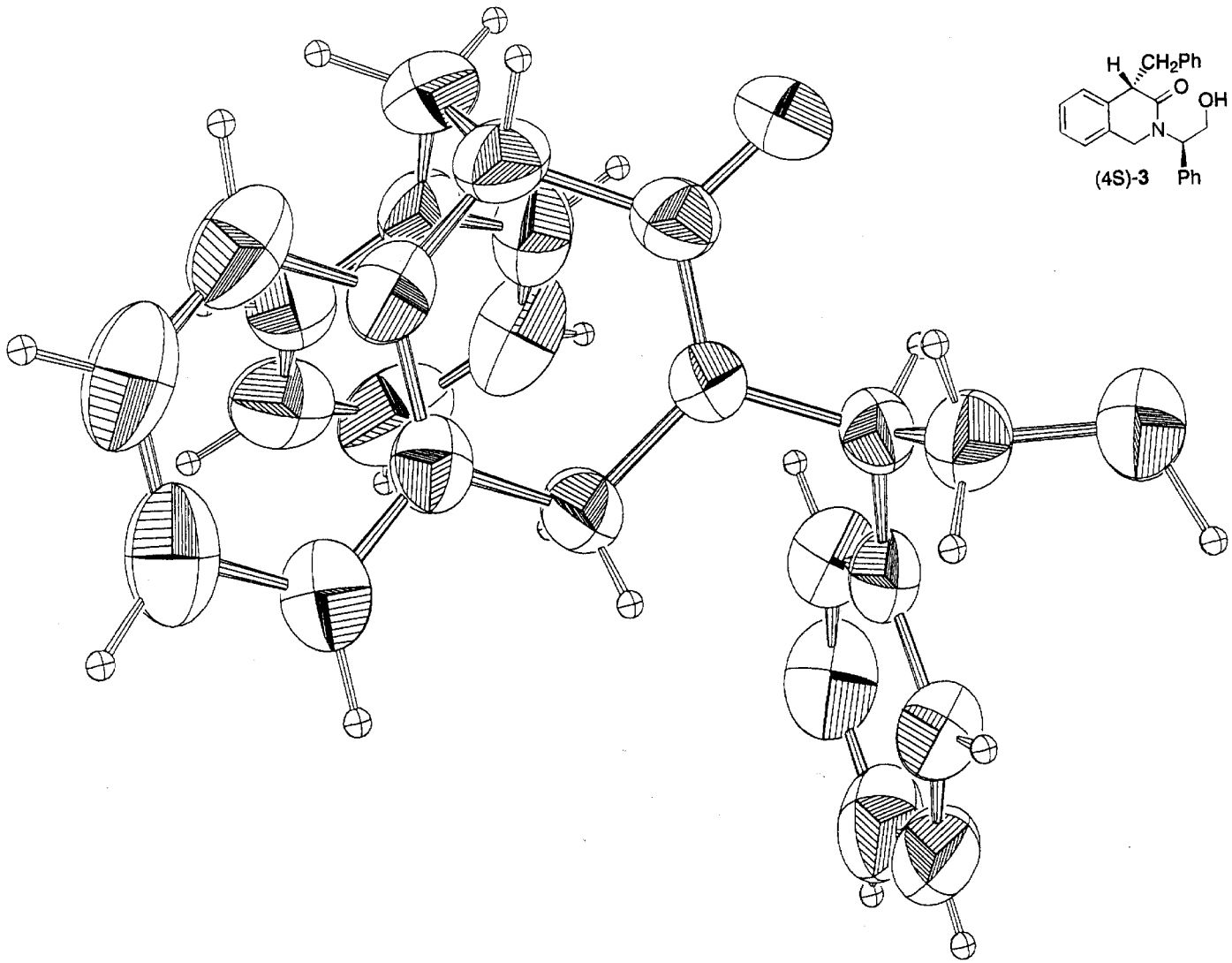
Table of Positional Parameters and Their Estimated Standard Deviations

Atom	x	y	z	B(A2)
H1	0.602(3)	0.853(2)	0.584	4.0*
H2	0.833(3)	0.798(2)	0.622	4.0*
H4	1.081(2)	0.791(3)	0.622	4.0*
H5	1.267(2)	0.668(3)	0.595	4.0*
H6	1.260(2)	0.457(3)	0.637	4.0*
H7	1.073(2)	0.377(2)	0.703	4.0*
H9a	0.860(2)	0.475(3)	0.868	4.0*
H9b	0.835(2)	0.408(3)	0.700	4.0*
H10a	0.966(3)	0.854(2)	0.859	4.0*
H10b	0.804(3)	0.856(2)	0.869	4.0*
H12	1.094(2)	0.703(2)	1.024	4.0*
H13	1.124(2)	0.595(3)	1.246	4.0*
H14	0.937(2)	0.546(2)	1.361	4.0*
H15	0.727(2)	0.628(2)	1.247	4.0*
H16	0.697(2)	0.739(2)	1.020	4.0*
H17	0.532(2)	0.539(2)	0.694	4.0*
H18a	0.392(2)	0.893(3)	0.542	4.0*
H18b	0.417(2)	1.032(2)	0.553	4.0*
H20	0.606(2)	0.479(2)	0.977	4.0*
H21	0.610(2)	0.320(3)	1.153	4.0*
H22	0.615(3)	0.135(2)	1.057	4.0*
H23	0.596(2)	0.103(2)	0.808	4.0*
H24	0.579(2)	0.248(3)	0.633	4.0*

Starred atoms were refined isotropically.
Anisotropically refined atoms are given in the form of the isotropic equivalent displacement parameter defined as:
(4/3) * [a2*B(1,1) + b2*B(2,2) + c2*B(3,3) + ab(cos gamma)*B(1,2) + ac(cos beta)*B(1,3) + bc(cos alpha)*B(2,3)]

CRYSTAL DATA

Formula	:	$C_{24}H_{23}NO_2$
Mol.Wt	:	357.46
Cryst. Syst	:	monoclinic
Space Group	:	$P2_1$
a	:	10.106(1)
b	:	11.173(2)
c	:	8.710(2)
α	:	----------
β	:	99.93(2)
γ	:	----------
V	:	968.7(3)
Z	:	2
ρcalcg.cm^{-3}	:	1.225
F(000)	:	380
μ(MoKα) cm^{-1}	:	0.72
T (° K)	:	294
Crystal size (mm)	:	0.40*0.40*0.55
Radiation	:	Mo Kα
Max 2Θ (°)	:	50°
Scan	:	$\omega/2\theta = 1$
t_{max} (for one measure), s	:	60
Variance of standards	:	0.3%
Range of HKL	:	-12,12 ; 0,13; 0,10
Reflections measured	:	1927
Reflections observed (I> σ(I))	:	1471 (2σ)
R_{int}(from merging equiv refl)	:	0.011
R(isotropic)	:	0.11
R(anisotropic)	:	0.083
Fourier Difference	:	0.45 - 0.15
N(obs)/N(var)	:	1471/313
Final R	:	0.050
Rw	:	0.036
$w = 1/\sigma(F_o)^2=[\sigma^2(I) + (0.04F_o^2)^2]^{-1/2}$		
Sw	:	2.02
Max residual e.Å^{-3}, Δ/σ	:	0.12, 0.9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OH
N
(4S)-3
Ph