JOC The Journal of Organic Chemistry

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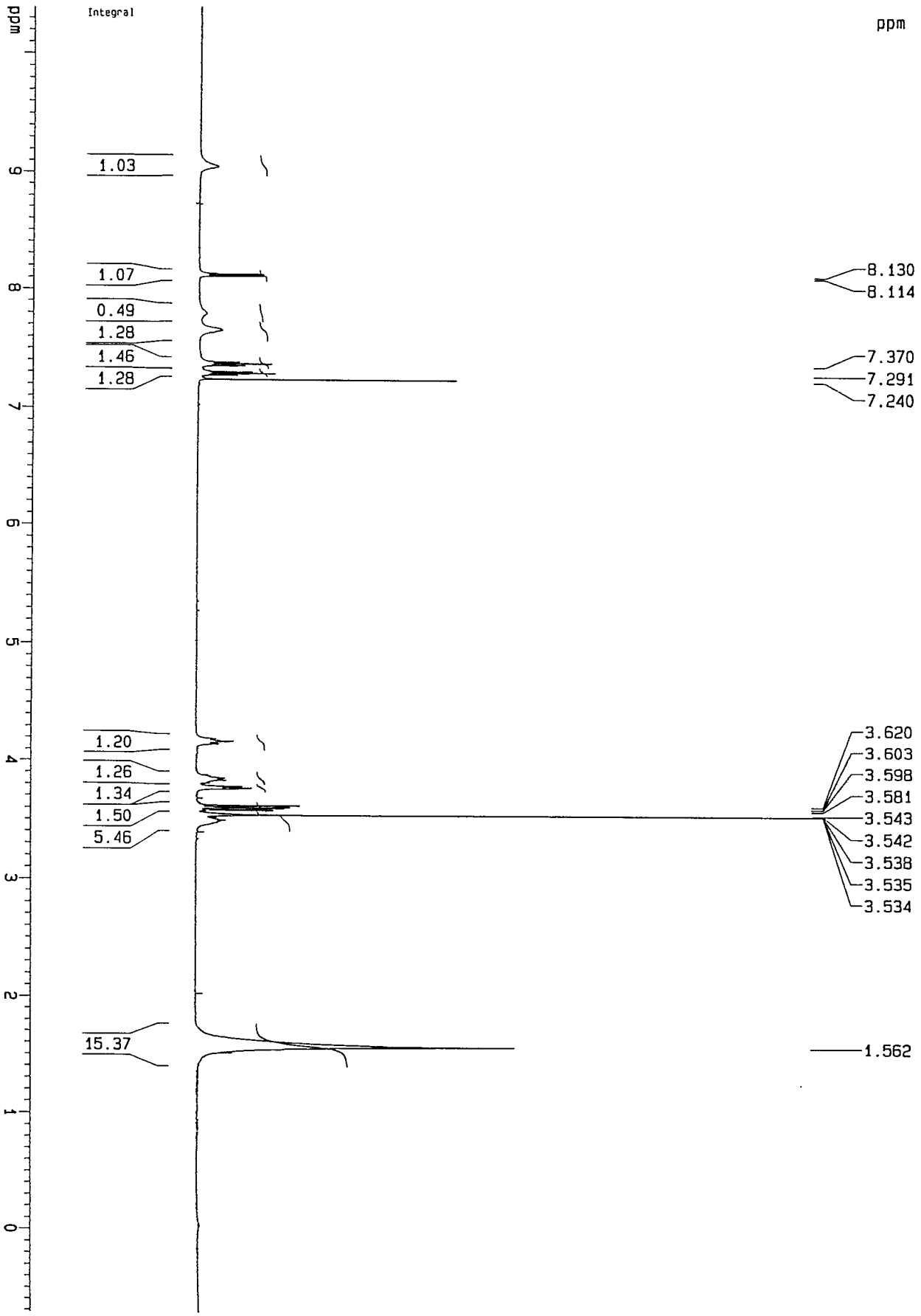
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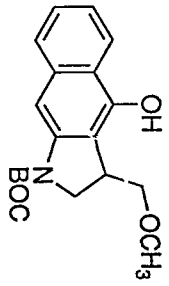
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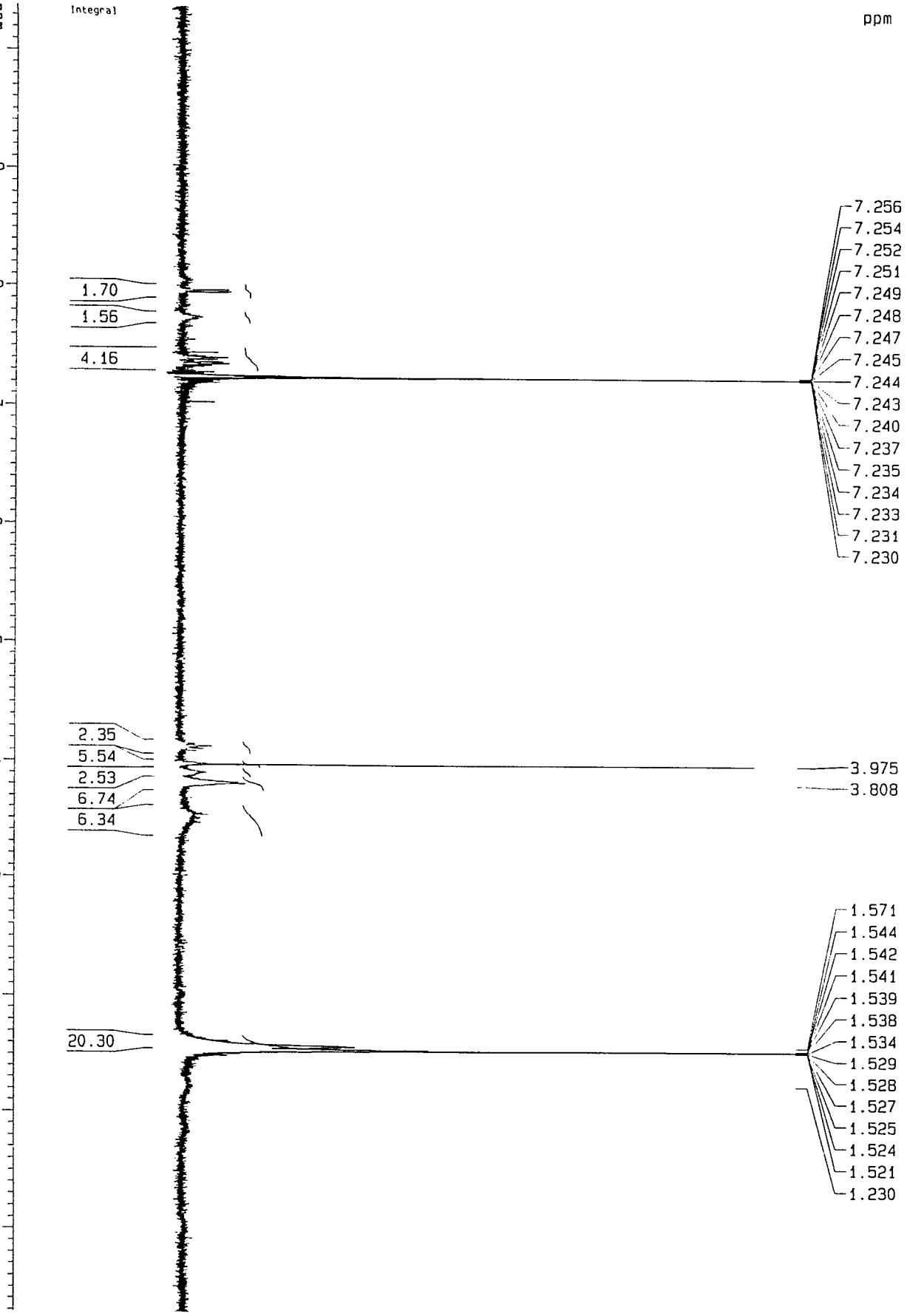
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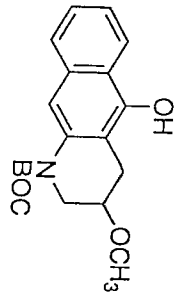
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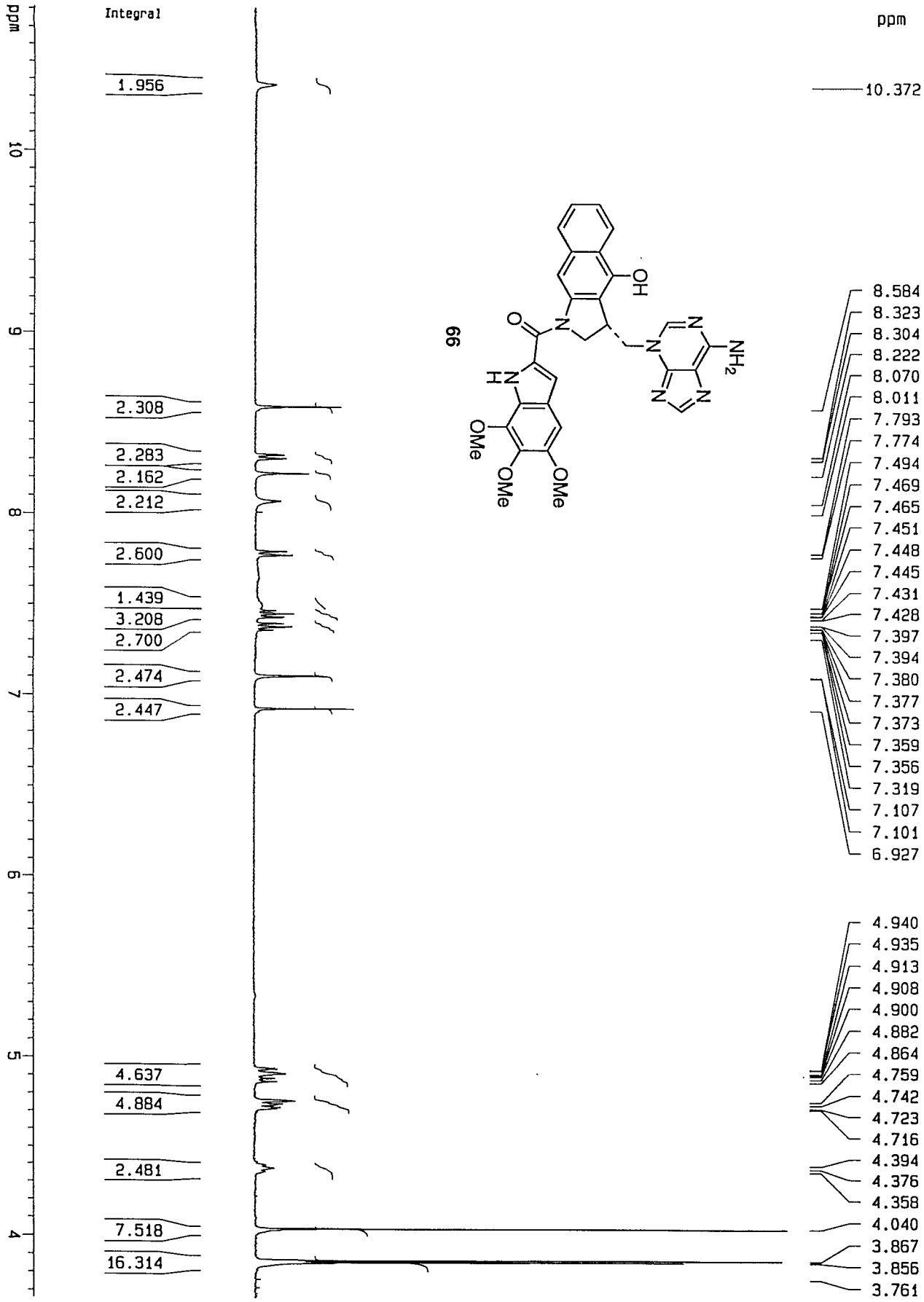
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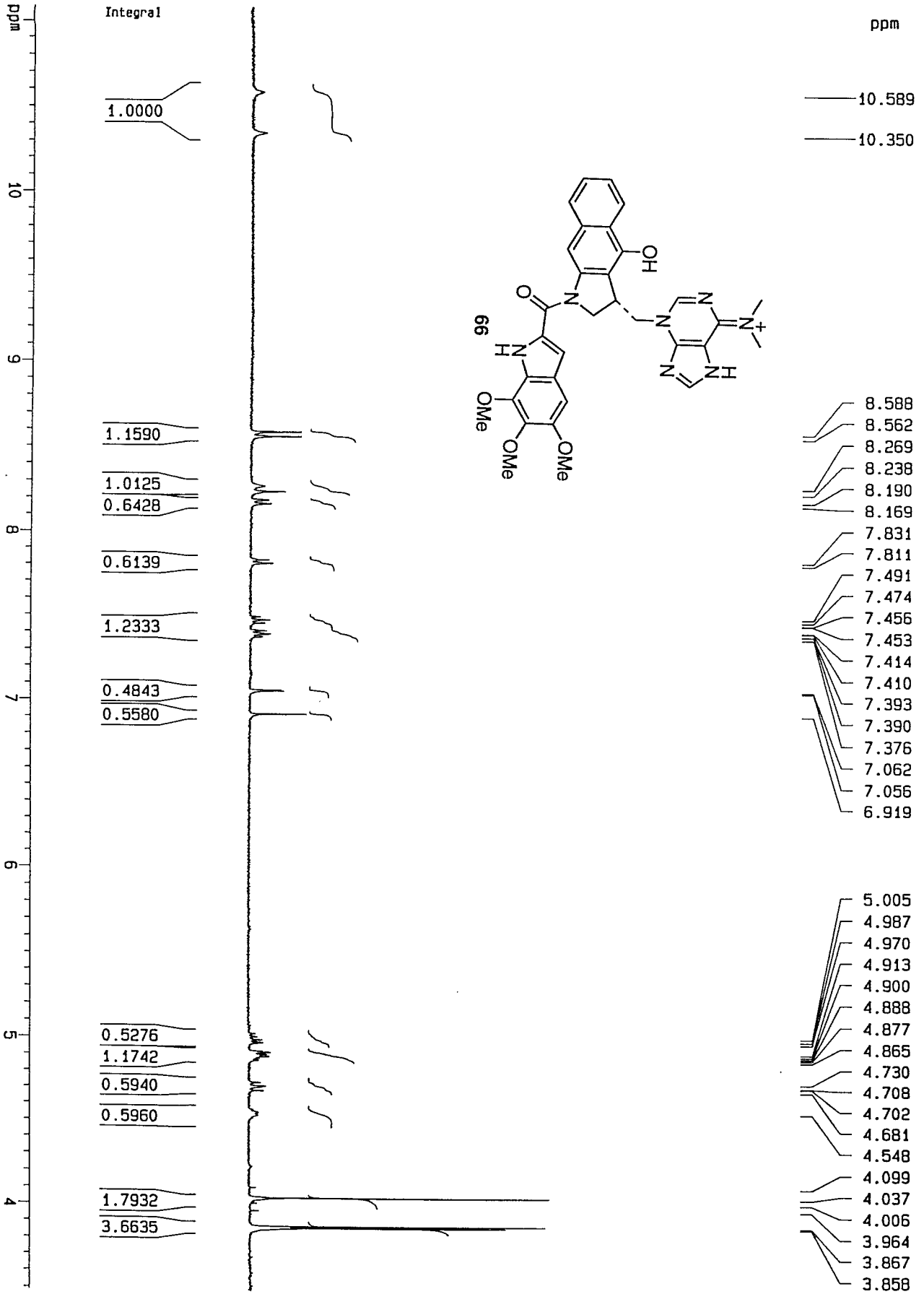
Acetone-D_6



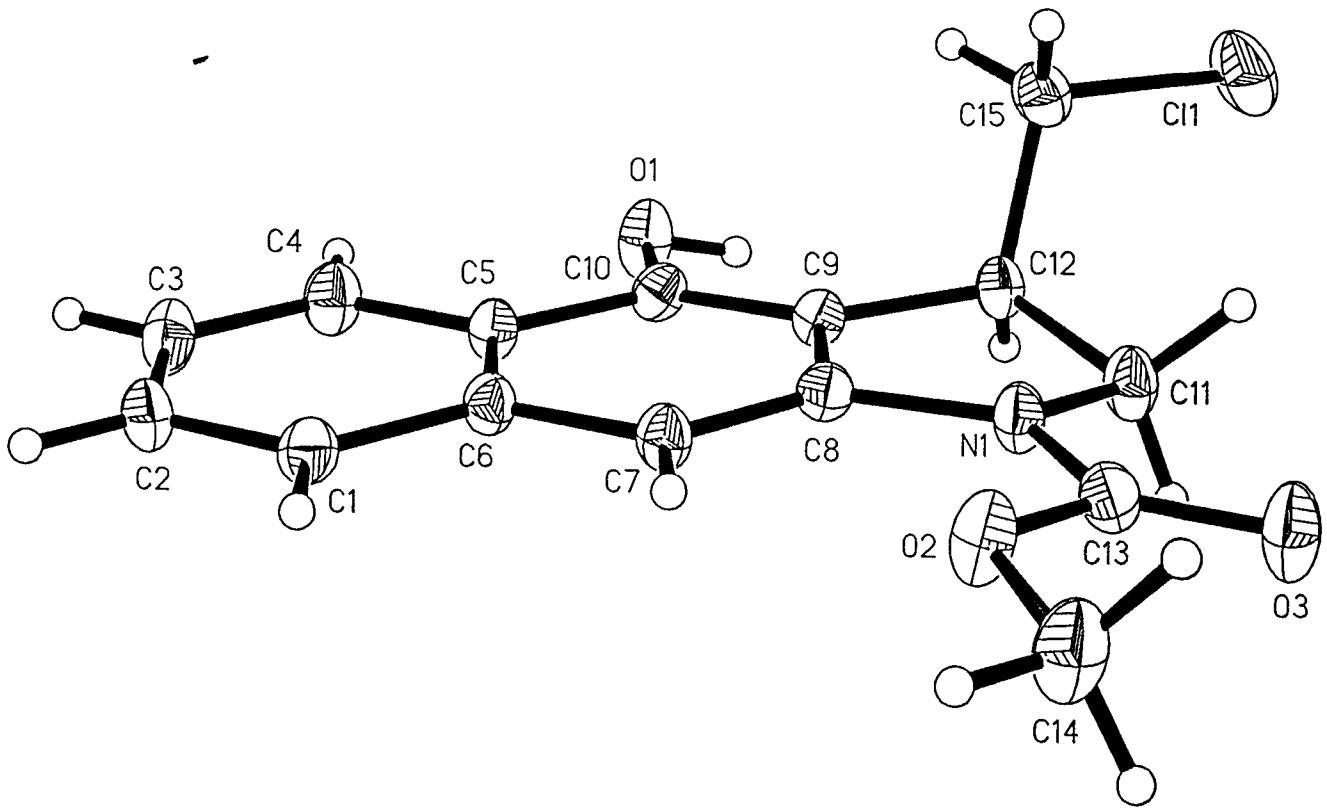
Acetone-d_6 + TFA

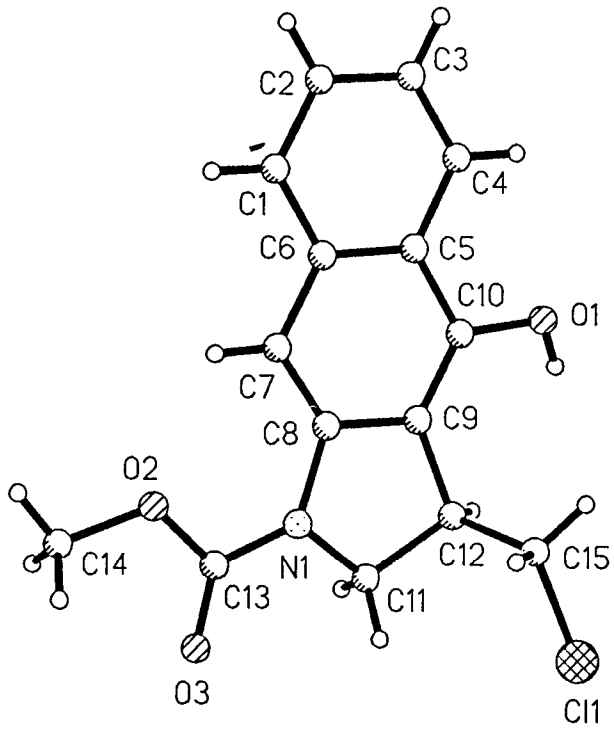
Captions to the figures

FIG. 1 ORTEP view of the "IV165" structure. The atoms are drawn with the 30% probability ellipsoids.

Fig. 2 Unit cell packing diagram of the "IV165" . Hydrogen atoms are omitted for clarity.

Fig. 3 Stereoview projection of the "IV165" structure.

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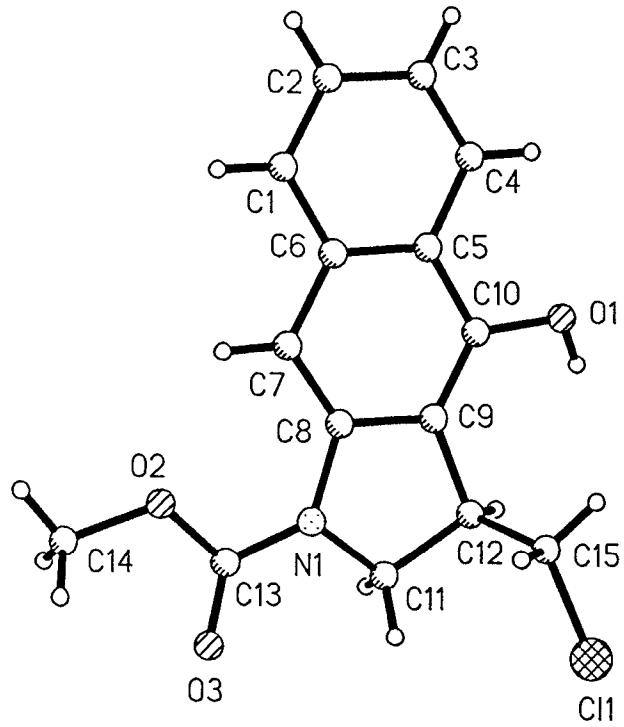
O3
Cl1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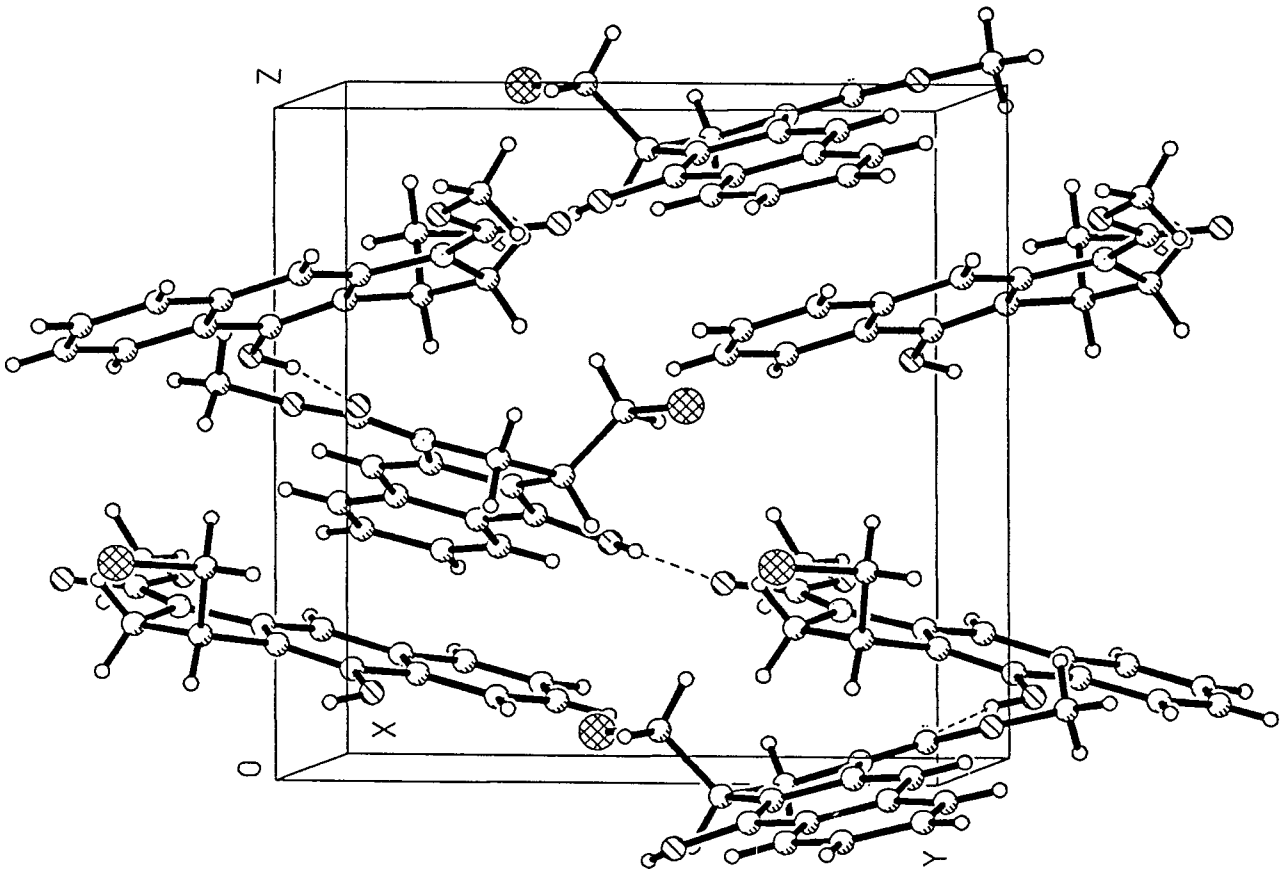
C12
C15
O3
Cl1


Z
O
X
Y

Experimental

A colorless, parallelepiped shaped crystal was mounted alongwith the largest dimension and data were collected with a Rigaku AFC6R diffractometer equipped with a copper rotating anode and a highly oriented graphite monochromator. A constant scan speed of 4°/min in ω was used and the weak reflections [$I<5\sigma(I)$] were rescanned to a maximum of 6 times and the counts accumulated to assure good counting statistics. Bijvoet pairs for data upto 80° were also collected with a view to determine absolute configuration of the compound. The intensities of three monitor reflections measured after every 200 reflections did not change significantly during 30 hrs of X-ray exposure. Unit cell dimensions and standard deviations were obtained by least squares fit to 25 reflections ($50<2\theta<80^{o}$). The data were corrected for Lorentz and polarization effects and an absorption correction based on a psi-scan was also applied. See Table 1 for cell parameters and other relevant data.

The systematic absences (00l, $l\neq 4n$) indicated the space group $P4_1$). The structure was solved by direct methods using SHELXS86. The function minimized was $\sum w(\|Fo\|-\|Fc\|^2)$. Hydrogen atoms were included in the ideal positions with a fixed isotropic U values of 0.08Å^2. A weighting scheme of the form $w=1/[\sigma^2(Fo^2)+(aP)^2)+bP]$ with a=0.0335 and b=0.74 was used.(P is defined as $Max(Fo^2,0)+2F_c^2)/3$.) There was no evidence of secondary extinction; therefore it was not applied. The refinement converged to the R indices given in the Table 1 which also includes the the largest difference peak and the hole in the last cycles of refinement. The Flack's parameter x being equal to 0.00(3) confirms the compound is an R-enatiomer. The final difference map was devoid of significant features.

All calculations were done on a Silicon graphics Personal Iris 4D/35 and an IBM compatible PC using programs TEXSAN (data reduction), Shake and Bake, and DIRDIF (structure solution) and SHELXL-93 (refinement) and SHELXTL-PC (plotting). Final atomic coordinates are listed in Table 2 and selected bond lengths and bond angles in Table 3.

References

TEXSAN. Structure Analysis Package. Molecular Structure Corporation. The Woodlands, TX 77381. 1992.

SHELXL-93. G.M.Sheldrick, J Appl. Cryst., (1993) in preparation.

SHELXTL-PC. G.M.Sheldrick, Siemens Analytical X-ray Instruments Inc. Madison, WI. (1990).

SHELXS86. G.M.Sheldrick, Acta Crystallographica A46, 467-473(1990).

H.D.Flack. Acta Crystallographica, A39, 876-881(1983).

Table 1. Crystal data and structure refinement for IV165.

Empirical formula	$C_{15}H_{14}ClNO_3$
Formula weight	291.72
Temperature	296(2) K
Wavelength	1.54178 Å
Crystal system	Tetrgonal
Space group	$P4_1$ (No. 76, C_4^2)
Unit cell dimensions	a = 10.946(1) Å α = 90°
	b = 10.946(1) Å β = 90°
	c = 11.345(1) Å γ = 90°
Volume	1359.4(2) Å^3
Z and F(000)	4 and 608
Density (calculated)	1.425 Mg/m^3
Absorption coefficient	2.555 mm^{-1}
Absorption Correction	Transmission factors: 0.76-1.00
Crystal size	0.17 x 0.12 x 0.07 mm
θ range for data collection	4.04 to 59.96°
Scan Type	2θ-θ
Scan width	1.155+0.140tanθ
Scan time/ background time	2:1
Index ranges	$-9 \le h \le 12$, $-9 \le k \le 12$, $-11 \le \ell \le 12$
Reflections collected	1769
Independent reflections	1453 (R_{int} = 0.0155)
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	1453 / 0 / 186
Goodness-of-fit on F^2, (S)	1.106
Final R indices [I>2σ(I)]	R1 = 0.0331, wR2 = 0.0757
R indices (all data)	R1 = 0.0452, wR2 = 0.0889
Flack's Absol. struct. parameter	0.00(3)
Largest diff. peak and hole	0.181 and -0.186 eÅ^{-3}

$R1=(\sum \| \|F\|-\|F\| \| / \sum \|F\|)$, $wR2=\sum w(F^2-F^2)^2/\sum w((F^2)^2)^{1/2}$, $S=[\sum w(F^2-F^2)^2/(n-p)]^{1/2}$

Table 2. Atomic coordinates [x 10^4] and equivalent isotropic displacement parameters [$Å^2$ x 10^3] for IV165.

U(eq) is defined as

$$U_{eq}=1/3\sum_i\sum_j U_{ij}a_i^*a_j^*a_i\bullet a_j$$

	x	y	z	U(eq)
Cl(1)	6951(1)	4108(1)	470	85(1)
O(1)	10890(2)	4831(2)	-1401(3)	61(1)
O(2)	8588(3)	9884(2)	520(3)	62(1)
O(3)	6683(3)	9099(3)	426(4)	71(1)
N(1)	8319(3)	7935(3)	-6(3)	47(1)
C(1)	12725(4)	8734(4)	-769(5)	50(1)
C(2)	13834(4)	8274(4)	-1110(5)	56(1)
C(3)	13930(4)	7044(5)	-1411(4)	61(1)
C(4)	12940(4)	6298(4)	-1385(4)	56(1)
C(5)	11789(3)	6747(4)	-1038(4)	43(1)
C(6)	11680(3)	7988(3)	-700(4)	42(1)
C(7)	10532(3)	8463(4)	-317(4)	44(1)
C(8)	9562(3)	7691(3)	-296(4)	41(1)
C(9)	9638(3)	6447(3)	-624(4)	40(1)
C(10)	10724(3)	5986(3)	-1008(4)	43(1)
C(11)	7528(4)	6885(3)	-317(5)	54(1)
C(12)	8412(3)	5820(4)	-522(4)	44(1)
C(13)	7789(4)	8987(4)	317(4)	51(1)
C(14)	8095(4)	11066(4)	799(6)	71(2)
C(15)	8385(4)	4887(4)	442(5)	58(1)

Table 3. Bond lengths [Å] and angles [°] for IV165.

Cl(1)-C(15)	1.787(4)	O(1)-C(10)	1.353(5)
O(2)-C(13)	1.335(5)	O(2)-C(14)	1.437(5)
O(3)-C(13)	1.223(5)	N(1)-C(13)	1.341(5)
N(1)-C(8)	1.426(5)	N(1)-C(11)	1.481(5)
C(1)-C(2)	1.370(6)	C(1)-C(6)	1.407(5)
C(2)-C(3)	1.393(6)	C(3)-C(4)	1.357(6)
C(4)-C(5)	1.409(6)	C(5)-C(6)	1.417(5)
C(5)-C(10)	1.433(5)	C(6)-C(7)	1.428(5)
C(7)-C(8)	1.357(5)	C(8)-C(9)	1.414(5)
C(9)-C(10)	1.363(5)	C(9)-C(12)	1.512(5)
C(11)-C(12)	1.533(5)	C(12)-C(15)	1.497(6)
C(13)-O(2)-C(14)	117.0(3)	C(13)-N(1)-C(8)	129.6(3)
C(13)-N(1)-C(11)	118.7(3)	C(8)-N(1)-C(11)	110.9(3)
C(2)-C(1)-C(6)	121.5(4)	C(1)-C(2)-C(3)	119.5(4)
C(4)-C(3)-C(2)	121.0(4)	C(3)-C(4)-C(5)	120.7(4)
C(4)-C(5)-C(6)	119.1(3)	C(4)-C(5)-C(10)	122.1(4)
C(6)-C(5)-C(10)	118.8(3)	C(1)-C(6)-C(5)	118.2(4)
C(1)-C(6)-C(7)	121.4(4)	C(5)-C(6)-C(7)	120.4(3)
C(8)-C(7)-C(6)	117.9(4)	C(7)-C(8)-C(9)	123.3(4)
C(7)-C(8)-N(1)	129.4(4)	C(9)-C(8)-N(1)	107.3(3)
C(10)-C(9)-C(8)	119.4(3)	C(10)-C(9)-C(12)	129.1(4)
C(8)-C(9)-C(12)	111.4(3)	O(1)-C(10)-C(9)	124.6(4)
O(1)-C(10)-C(5)	115.2(3)	C(9)-C(10)-C(5)	120.1(4)
N(1)-C(11)-C(12)	104.9(3)	C(15)-C(12)-C(9)	112.5(3)
C(15)-C(12)-C(11)	113.3(4)	C(9)-C(12)-C(11)	103.1(3)
O(3)-C(13)-O(2)	123.9(4)	O(3)-C(13)-N(1)	122.8(4)
O(2)-C(13)-N(1)	113.3(4)	C(12)-C(15)-Cl(1)	110.8(3)

Supplementary material to be deposited

Table 4. Anisotropic displacement coefficients.

Table 5. H-atom coordinates.

Table 6. Observed and Calculated structure factors. (4 pp)

<u>You may also include the following in the supplementary material.</u>

1. Unit cell packing diagram.
2. Stereview diagram.
3. Torsion angles and the mean plane equations.

Table 4. Anisotropic displacement parameters [$Å^2 \times 10^3$] for IV165.

The anisotropic displacement factor exponent takes the form:

$-2\pi^2(h^2a^{*2}U_{11}+k^2b^{*2}U_{22}+l^2c^{*2}U_{33}+2hka^*b^*U_{12}+2hla^*c^*U_{13}+2klb^*c^*U_{23})$

	U11	U22	U33	U23	U13	U12
Cl(1)	79(1)	65(1)	111(1)	7(1)	28(1)	-27(1)
O(1)	43(2)	42(2)	97(2)	-20(2)	4(2)	-1(1)
O(2)	44(2)	47(2)	94(3)	-21(2)	-1(2)	0(1)
O(3)	43(2)	52(2)	116(3)	-15(2)	19(2)	0(1)
N(1)	34(2)	41(2)	66(2)	-2(2)	9(2)	1(2)
C(1)	46(3)	49(2)	54(3)	-2(2)	-11(2)	-12(2)
C(2)	39(2)	63(3)	66(3)	4(3)	0(2)	-15(2)
C(3)	38(3)	78(3)	68(3)	-5(3)	4(2)	-2(2)
C(4)	47(3)	49(3)	73(3)	-11(2)	4(2)	1(2)
C(5)	34(2)	42(2)	54(3)	-2(2)	2(2)	-1(2)
C(6)	36(2)	40(2)	50(3)	-1(2)	-2(2)	-5(2)
C(7)	37(2)	41(2)	54(3)	-4(2)	-1(2)	-8(2)
C(8)	41(2)	37(2)	46(2)	-1(2)	0(2)	0(2)
C(9)	38(2)	36(2)	45(2)	-3(2)	-1(2)	0(2)
C(10)	41(2)	37(2)	50(2)	-4(2)	-4(2)	4(2)
C(11)	41(2)	41(2)	80(3)	-4(2)	8(2)	-7(2)
C(12)	34(2)	45(2)	51(3)	-5(2)	3(2)	-6(2)
C(13)	46(3)	43(2)	62(3)	2(2)	10(2)	0(2)
C(14)	55(3)	43(3)	116(5)	-18(3)	7(3)	3(2)
C(15)	53(3)	57(3)	63(3)	7(2)	3(3)	-13(2)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$ x 10^3) for IV165.

	x	y	z	U(eq)
H(1A)	10225(4)	4526(17)	-1558(45)	80
H(1B)	12661(4)	9558(4)	-578(5)	80
H(2A)	14516(4)	8779(4)	-1140(5)	80
H(3A)	14685(4)	6729(5)	-1633(4)	80
H(4A)	13023(4)	5482(4)	-1599(4)	80
H(7A)	10451(3)	9275(4)	-90(4)	80
H(11A)	7061(4)	7057(3)	-1025(5)	80
H(11B)	6967(4)	6700(3)	320(5)	80
H(12A)	8223(31)	5404(33)	-1264(34)	40(11)
H(14A)	8749(5)	11645(7)	872(27)	80
H(14B)	7655(23)	11021(7)	1530(14)	80
H(14C)	7551(21)	11320(12)	182(13)	80
H(15A)	8521(4)	5285(4)	1194(5)	80
H(15B)	9037(4)	4300(4)	320(5)	80

Selected torsion angles

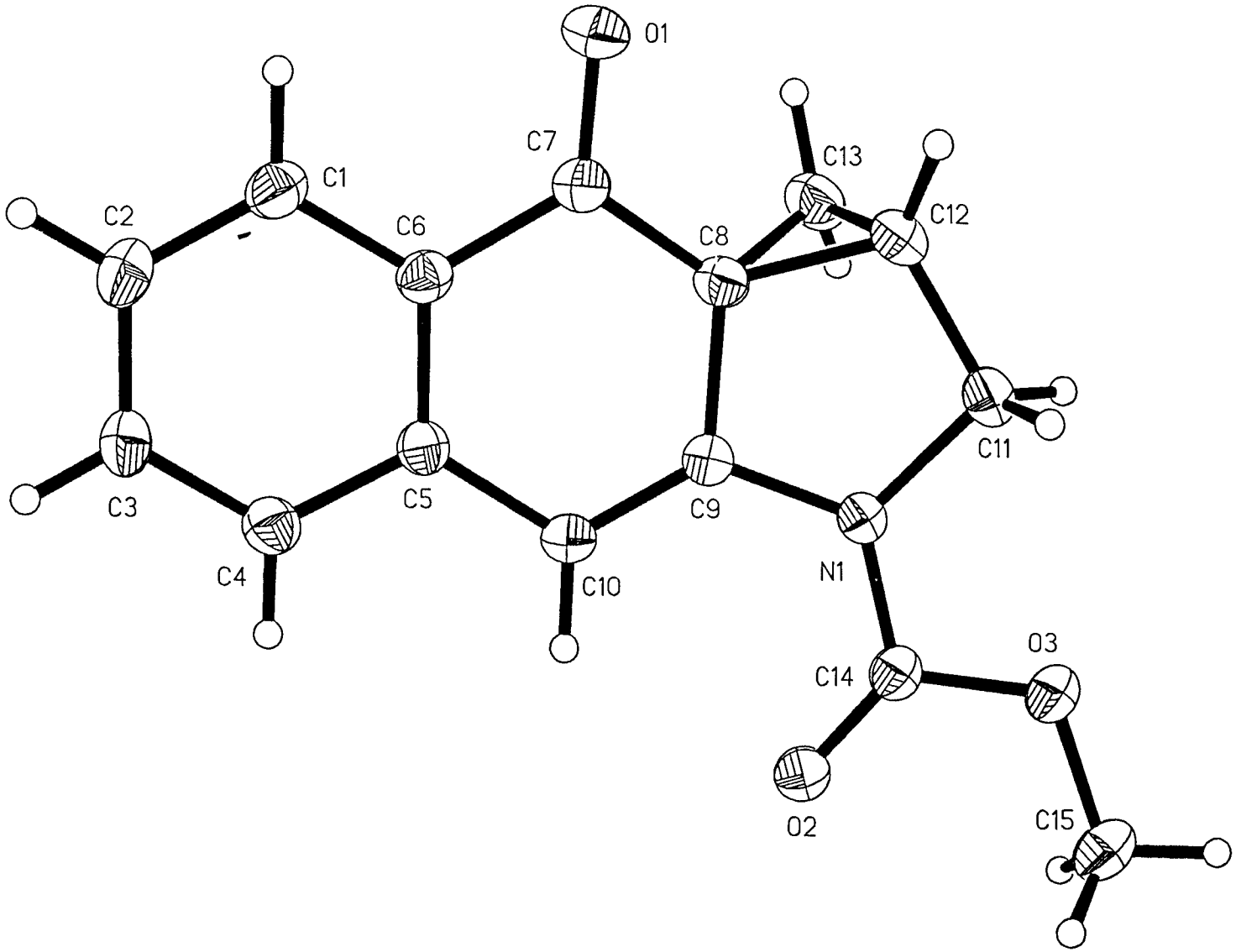
0.83	(0.67)	C6 - C1 - C2 - C3
0.80	(0.71)	C1 - C2 - C3 - C4
-0.79	(0.75)	C2 - C3 - C4 - C5
-0.81	(0.72)	C3 - C4 - C5 - C6
179.63	(0.42)	C3 - C4 - C5 - C10
-2.38	(0.61)	C2 - C1 - C6 - C5
178.46	(0.44)	C2 - C1 - C6 - C7
2.34	(0.62)	C4 - C5 - C6 - C1
-178.09	(0.40)	C10 - C5 - C6 - C1
-178.49	(0.43)	C4 - C5 - C6 - C7
1.08	(0.60)	C10 - C5 - C6 - C7
178.83	(0.41)	C1 - C6 - C7 - C8
-0.32	(0.61)	C5 - C6 - C7 - C8
0.42	(0.63)	C6 - C7 - C8 - C9
-176.88	(0.41)	C6 - C7 - C8 - N1
0.06	(0.77)	C13 - N1 - C8 - C7
169.13	(0.43)	C11 - N1 - C8 - C7
-177.58	(0.41)	C13 - N1 - C8 - C9
-8.51	(0.46)	C11 - N1 - C8 - C9
-1.35	(0.63)	C7 - C8 - C9 - C10
176.47	(0.38)	N1 - C8 - C9 - C10
-179.43	(0.40)	C7 - C8 - C9 - C12
-1.61	(0.46)	N1 - C8 - C9 - C12
-176.47	(0.39)	C8 - C9 - C10 - O1
1.23	(0.73)	C12 - C9 - C10 - O1
2.10	(0.61)	C8 - C9 - C10 - C5
179.81	(0.40)	C12 - C9 - C10 - C5
-3.74	(0.61)	C4 - C5 - C10 - O1
176.70	(0.39)	C6 - C5 - C10 - O1
177.56	(0.44)	C4 - C5 - C10 - C9
-2.00	(0.62)	C6 - C5 - C10 - C9
-174.76	(0.40)	C13 - N1 - C11 - C12
14.82	(0.48)	C8 - N1 - C11 - C12
70.08	(0.56)	C10 - C9 - C12 - C15
-112.07	(0.41)	C8 - C9 - C12 - C15
-167.48	(0.44)	C10 - C9 - C12 - C11
10.37	(0.48)	C8 - C9 - C12 - C11
107.36	(0.40)	N1 - C11 - C12 - C15
-14.55	(0.46)	N1 - C11 - C12 - C9
-4.63	(0.73)	C14 - O2 - C13 - O3
176.22	(0.43)	C14 - O2 - C13 - N1
172.34	(0.46)	C8 - N1 - C13 - O3
3.98	(0.69)	C11 - N1 - C13 - O3
-8.50	(0.68)	C8 - N1 - C13 - O2
-176.86	(0.38)	C11 - N1 - C13 - O2
-176.52	(0.31)	C9 - C12 - C15 - C11
67.01	(0.43)	C11 - C12 - C15 - C11

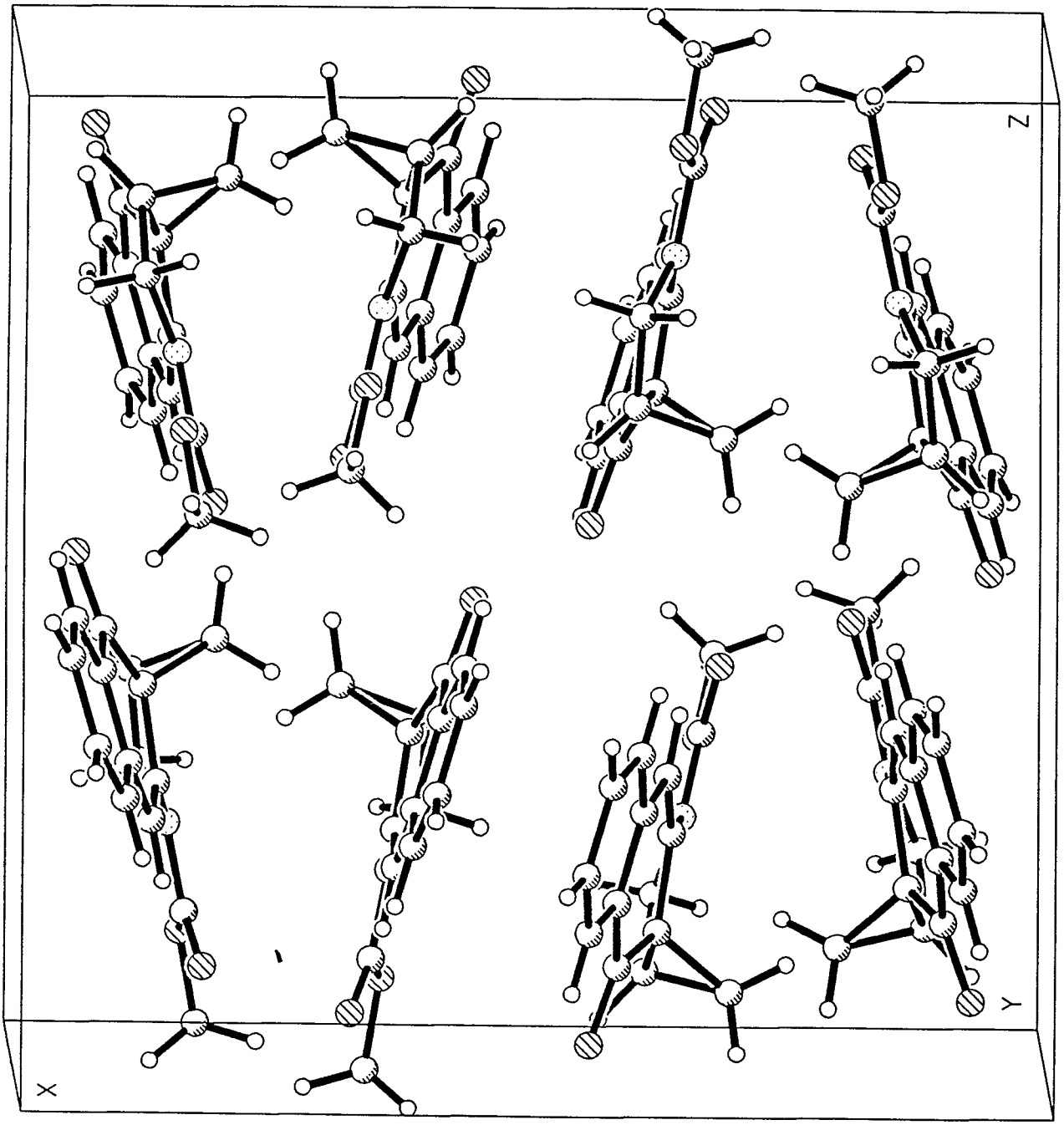
Captions to the figures

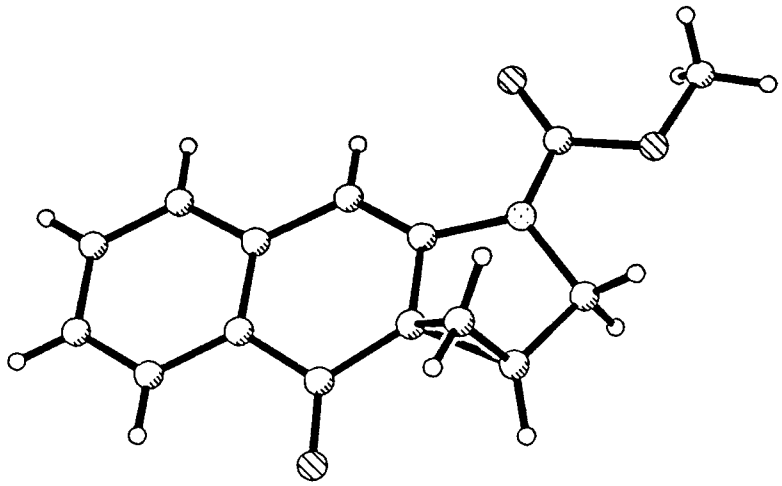
FIG. 1 ORTEP view of the "GARBA" structure. The atoms are drawn with the 30% probability ellipsoids.

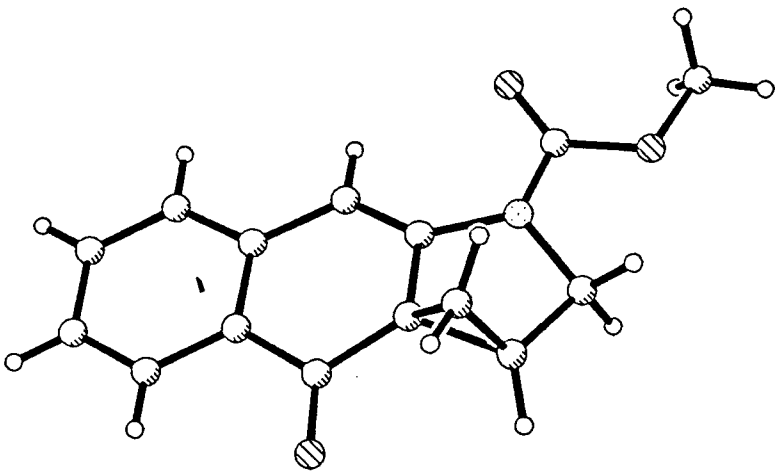
Fig. 2 Unit cell packing diagram of the "GARBA" . Hydrogen atoms are omitted for clarity.

Fig. 3 Stereoview projection of the "GARBA" structure.




X
Y
Z





Experimental

A colorless, plate like crystal was mounted alongwith the largest dimension and data were collected with a Rigaku AFC6R diffractometer equipped with a copper rotating anode and a highly oriented graphite monochromator. A constant scan speed of 8°/min in ω was used and the weak reflections [$I<5\sigma(I)$] were rescanned to a maximum of 6 times and the counts accumulated to assure good counting statistics. The intensities of three monitor reflections measured after every 200 reflections did not change significantly during 32 hrs of X-ray exposure. Unit cell dimensions and standard deviations were obtained by least squares fit to 25 reflections ($50<2\theta<80^{o}$). The data were corrected for Lorentz and polarization effects and not for absorption because of low value of μ. See Table 1 for cell parameters and other relevant data.

The systematic absences (0kl, k=2n+1; h0l, l=2n+1; and hk0, h=2n+1) indicated the space group Pbca. The structure was solved by direct methods using SHELXS86. All non-hydrogen atoms were refined anisotropically by the full matrix least-squares method. The function minimized was $\sum w(\|Fo\|-\|Fc\|^2)$. Hydrogen atoms were included in the ideal positions with a fixed isotropic U values of 0.08Å^2. A weighting scheme of the form $w=1/[\sigma^2(Fo^2)+(aP)^2)+bP]$ with a=0.0598 and b=1.97 was used.(P is defined as $Max(Fo^2,0)+2F_c^2)/3$.) There was no evidence of secondary extinction; therefore it was not applied. The refinement converged to the R indices given in the Table 1 which also includes the the largest difference peak and the hole in the last cycles of refinement. The final difference map was devoid of significant features.

All calculations were done on a Silicon graphics Personal Iris 4D/35 and an IBM compatible PC using programs TEXSAN (data reduction), SHELXL-93 (refinement) and SHELXTL-PC (plotting). Final atomic coordinates are listed in Table 2 and selected bond lengths and bond angles in Table 3.

References

TEXSAN. Structure Analysis Package. Molecular Structure Corporation. The Woodlands, TX 77381. 1992.

SHELXL-93. G.M.Sheldrick, J Appl. Cryst., (1993) in preparation.

SHELXTL-PC. G.M.Sheldrick, Siemens Analytical X-ray Instruments Inc. Madison, WI. (1990).

SHELXS86. G.M.Sheldrick, Acta Crystallographica A46, 467-473(1990).

Table 1. Crystal data and structure refinement for GARBA.

Empirical formula	$C_{15}H_{13}NO_3$
Formula weight	255.26
Temperature	296(2) K
Wavelength	1.54178 Å
Crystal system	Orthorhombic
Space group	Pbca (No.61, C_{2h}^{15})
Unit cell dimensions	a = 14.960(6) Å $\alpha = 90^{\circ}$
	b = 10.755(2) Å $\beta = 90^{\circ}$
	c = 15.149(4) Å $\gamma = 90^{\circ}$
Volume	2437(2) $Å^3$
Z and F(000)	8 and 1072
Density (calculated)	1.391 Mg/m^3
Absorption coefficient	0.802 mm^{-1}
Crystal size	0.27 x 0.18 x 0.14 mm
θ range for data collection	5.84 to 60.03°
Scan Type	2θ-θ
Scan width	$1.837+0.140\tan\theta$
Scan time/ background time	2:1
Index ranges	$-6 \leq h \leq 16$, $-12 \leq k \leq 12$, $-17 \leq \ell \leq 5$
Reflections collected	2124
Independent reflections	1800 (R_{int} = 0.0000)
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	1800 / 0 / 173
Goodness-of-fit on F^2, (S)	1.080
Final R indices [$I>2\sigma(I)$]	R1 = 0.0488, wR2 = 0.1228
R indices (all data)	R1 = 0.0818, wR2 = 0.1549
Largest diff. peak and hole	0.219 and -0.209 $eÅ^{-3}$

$R1=(\sum \| \|F_o\|-\|F_c\| \| / \sum \|F_o\|)$, $wR2=\sum w(F_o^2-F_c^2)^2/\sum w[(F_o^2)^2]^{1/2}$, $S=[\sum w(F_o^2-F_c^2)^2/(n-p)]^{1/2}$

Table 2. Atomic coordinates [x 10^4] and equivalent isotropic displacement parameters [$Å^2$ x 10^3] for GARBA.

U(eq) is defined as

$$U_{eq}=1/3\sum_i\sum_j U_{ij}a_i^*a_j^*a_i\bullet a_j$$

	x	y	z	U(eq)
O(1)	9385(2)	-1885(2)	9905(1)	82(1)
O(2)	8174(2)	25(2)	6010(1)	70(1)
O(3)	8432(2)	1957(2)	6534(1)	68(1)
N(1)	8536(2)	366(2)	7454(1)	52(1)
C(1)	9347(2)	-4212(3)	9021(2)	58(1)
C(2)	9354(2)	-5307(3)	8553(2)	65(1)
C(3)	9103(2)	-5304(3)	7674(2)	64(1)
C(4)	8851(2)	-4216(3)	7270(2)	57(1)
C(5)	8842(2)	-3080(2)	7724(2)	48(1)
C(6)	9099(2)	-3106(3)	8624(2)	49(1)
C(7)	9118(2)	-1944(3)	9140(2)	57(1)
C(8)	8751(2)	-838(3)	8694(2)	52(1)
C(9)	8613(2)	-870(3)	7732(2)	48(1)
C(10)	8638(2)	-1931(3)	7271(2)	52(1)
C(11)	8827(2)	1273(3)	8139(2)	61(1)
C(12)	8894(2)	508(3)	8964(2)	60(1)
C(13)	8065(2)	-85(3)	9220(2)	69(1)
C(14)	8364(2)	716(3)	6604(2)	55(1)
C(15)	8277(3)	2467(4)	5671(2)	83(1)

Table 3. Bond lengths [Å] and angles [°] for GARBA.

O(1)-C(7)	1.226(4)	O(2)-C(14)	1.201(4)
O(3)-C(14)	1.343(4)	O(3)-C(15)	1.437(4)
N(1)-C(14)	1.365(4)	N(1)-C(9)	1.400(3)
N(1)-C(11)	1.489(4)	C(1)-C(2)	1.374(5)
C(1)-C(6)	1.384(4)	C(2)-C(3)	1.384(5)
C(3)-C(4)	1.373(4)	C(4)-C(5)	1.403(4)
C(5)-C(6)	1.416(4)	C(5)-C(10)	1.447(4)
C(6)-C(7)	1.475(4)	C(7)-C(8)	1.475(4)
C(8)-C(9)	1.473(4)	C(8)-C(12)	1.519(4)
C(8)-C(13)	1.531(4)	C(9)-C(10)	1.338(4)
C(11)-C(12)	1.499(4)	C(12)-C(13)	1.448(5)

C(14)-O(3)-C(15)	116.0(3)	C(14)-N(1)-C(9)	124.1(2)
C(14)-N(1)-C(11)	122.2(2)	C(9)-N(1)-C(11)	112.8(2)
C(2)-C(1)-C(6)	121.0(3)	C(1)-C(2)-C(3)	119.4(3)
C(4)-C(3)-C(2)	120.4(3)	C(3)-C(4)-C(5)	121.8(3)
C(4)-C(5)-C(6)	116.9(3)	C(4)-C(5)-C(10)	120.9(3)
C(6)-C(5)-C(10)	122.1(2)	C(1)-C(6)-C(5)	120.6(3)
C(1)-C(6)-C(7)	119.5(3)	C(5)-C(6)-C(7)	119.9(3)
O(1)-C(7)-C(6)	123.5(3)	O(1)-C(7)-C(8)	120.8(3)
C(6)-C(7)-C(8)	115.7(2)	C(9)-C(8)-C(7)	119.1(2)
C(9)-C(8)-C(12)	107.9(2)	C(7)-C(8)-C(12)	126.4(3)
C(9)-C(8)-C(13)	115.7(3)	C(7)-C(8)-C(13)	116.0(3)
C(12)-C(8)-C(13)	56.7(2)	C(10)-C(9)-N(1)	130.9(3)
C(10)-C(9)-C(8)	122.2(3)	N(1)-C(9)-C(8)	106.7(2)
C(9)-C(10)-C(5)	119.1(3)	N(1)-C(11)-C(12)	104.0(2)
C(13)-C(12)-C(11)	114.0(3)	C(13)-C(12)-C(8)	62.1(2)
C(11)-C(12)-C(8)	106.8(2)	C(12)-C(13)-C(8)	61.3(2)
O(2)-C(14)-O(3)	125.0(3)	O(2)-C(14)-N(1)	125.5(3)
O(3)-C(14)-N(1)	109.5(3)		

Supplementary material to be deposited

Table 4. Anisotropic displacement coefficients.

Table 5. H-atom coordinates.

Table 6. Observed and Calculated structure factors. (4 pp)

<u>You may also include the following in the supplementary material.</u>

1. Unit cell packing diagram.
2. Stereview diagram.
3. Torsion angles and the mean plane equations.

Table 4. Anisotropic displacement parameters [$Å^2 \times 10^3$] for GARBA.

The anisotropic displacement factor exponent takes the form:

$-2\pi^2(h^2a^{*2}U_{11}+k^2b^{*2}U_{22}+l^2c^{*2}U_{33}+2hka^*b^*U_{12}+2hla^*c^*U_{13}+2klb^*c^*U_{23})$

	U11	U22	U33	U23	U13	U12
O(1)	123(2)	74(2)	49(1)	-4(1)	-24(1)	-10(1)
O(2)	103(2)	58(1)	50(1)	3(1)	-14(1)	3(1)
O(3)	95(2)	50(1)	58(1)	9(1)	3(1)	6(1)
N(1)	68(2)	44(1)	45(1)	-2(1)	-1(1)	4(1)
C(1)	62(2)	58(2)	55(2)	9(2)	-10(1)	-6(2)
C(2)	71(2)	49(2)	75(2)	11(2)	-3(2)	-1(2)
C(3)	78(2)	42(2)	72(2)	-1(2)	1(2)	-3(2)
C(4)	62(2)	55(2)	55(2)	-4(2)	-3(2)	-6(2)
C(5)	47(2)	46(2)	49(2)	-1(1)	-2(1)	-6(1)
C(6)	48(2)	52(2)	46(2)	3(1)	-3(1)	-4(1)
C(7)	67(2)	57(2)	47(2)	0(1)	-3(2)	-4(2)
C(8)	59(2)	54(2)	43(2)	-3(1)	2(1)	-2(1)
C(9)	53(2)	47(2)	45(2)	0(1)	1(1)	0(1)
C(10)	61(2)	54(2)	40(2)	0(1)	-5(1)	0(1)
C(11)	75(2)	48(2)	59(2)	-8(2)	-4(2)	1(2)
C(12)	71(2)	60(2)	50(2)	-13(2)	-3(2)	-1(2)
C(13)	80(2)	71(2)	56(2)	-14(2)	10(2)	1(2)
C(14)	63(2)	51(2)	52(2)	4(2)	-2(2)	5(1)
C(15)	115(3)	68(2)	67(2)	23(2)	0(2)	4(2)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$ x 10^3) for GARBA.

	x	y	z	U(eq)
H(1A)	9512(2)	-4215(3)	9613(2)	80
H(2A)	9526(2)	-6045(3)	8826(2)	80
H(3A)	9104(2)	-6042(3)	7355(2)	80
H(4A)	8682(2)	-4234(3)	6679(2)	80
H(10A)	8525(2)	-1929(3)	6667(2)	80
H(11A)	8390(2)	1933(3)	8208(2)	80
H(11B)	9400(2)	1636(3)	7989(2)	80
H(12A)	9341(2)	715(3)	9413(2)	80
H(13A)	7524(2)	171(3)	8917(2)	80
H(13B)	7985(2)	-291(3)	9838(2)	80
H(15A)	8333(13)	3355(4)	5693(4)	80
H(15B)	7687(5)	2249(16)	5477(6)	80
H(15C)	8709(9)	2136(15)	5265(4)	80

Selected torsion angles

-0.47	(0.50)	C6 - C1 - C2 - C3
0.18	(0.52)	C1 - C2 - C3 - C4
0.28	(0.52)	C2 - C3 - C4 - C5
-0.43	(0.45)	C3 - C4 - C5 - C6
175.42	(0.30)	C3 - C4 - C5 - C10
0.30	(0.47)	C2 - C1 - C6 - C5
-178.71	(0.30)	C2 - C1 - C6 - C7
0.15	(0.42)	C4 - C5 - C6 - C1
-175.65	(0.28)	C10 - C5 - C6 - C1
179.15	(0.27)	C4 - C5 - C6 - C7
3.36	(0.43)	C10 - C5 - C6 - C7
3.61	(0.49)	C1 - C6 - C7 - O1
-175.41	(0.31)	C5 - C6 - C7 - O1
-173.60	(0.28)	C1 - C6 - C7 - C8
7.38	(0.42)	C5 - C6 - C7 - C8
167.28	(0.31)	O1 - C7 - C8 - C9
-15.43	(0.41)	C6 - C7 - C8 - C9
19.21	(0.51)	O1 - C7 - C8 - C12
-163.50	(0.29)	C6 - C7 - C8 - C12
-47.22	(0.44)	O1 - C7 - C8 - C13
130.07	(0.29)	C6 - C7 - C8 - C13
7.90	(0.52)	C14 - N1 - C9 - C10
-161.40	(0.32)	C11 - N1 - C9 - C10
-177.02	(0.27)	C14 - N1 - C9 - C8
13.68	(0.34)	C11 - N1 - C9 - C8
13.65	(0.45)	C7 - C8 - C9 - C10
167.06	(0.28)	C12 - C8 - C9 - C10
-131.96	(0.31)	C13 - C8 - C9 - C10
-161.95	(0.27)	C7 - C8 - C9 - N1
-8.54	(0.33)	C12 - C8 - C9 - N1
52.44	(0.34)	C13 - C8 - C9 - N1
171.85	(0.30)	N1 - C9 - C10 - C5
-2.57	(0.44)	C8 - C9 - C10 - C5
178.29	(0.29)	C4 - C5 - C10 - C9
-6.08	(0.44)	C6 - C5 - C10 - C9
177.42	(0.28)	C14 - N1 - C11 - C12
-13.04	(0.35)	C9 - N1 - C11 - C12
-59.49	(0.34)	N1 - C11 - C12 - C13
6.87	(0.35)	N1 - C11 - C12 - C8
109.42	(0.29)	C9 - C8 - C12 - C13
-99.63	(0.35)	C7 - C8 - C12 - C13
0.00		C13 - C8 - C12 - C13
0.68	(0.36)	C9 - C8 - C12 - C11
151.63	(0.31)	C7 - C8 - C12 - C11
-108.73	(0.31)	C13 - C8 - C12 - C11
97.02	(0.28)	C11 - C12 - C13 - C8
0.00		C8 - C12 - C13 - C8
-95.33	(0.29)	C9 - C8 - C13 - C12
117.97	(0.30)	C7 - C8 - C13 - C12
0.00		C12 - C8 - C13 - C12
-1.03	(0.50)	C15 - O3 - C14 - O2
179.36	(0.28)	C15 - O3 - C14 - N1
7.42	(0.53)	C9 - N1 - C14 - O2
175.76	(0.32)	C11 - N1 - C14 - O2
-172.98	(0.27)	C9 - N1 - C14 - O3
-4.64	(0.42)	C11 - N1 - C14 - O3

Least-squares planes (x,y,z in crystal coordinates) and deviations from them
(* indicates atom used to define plane)