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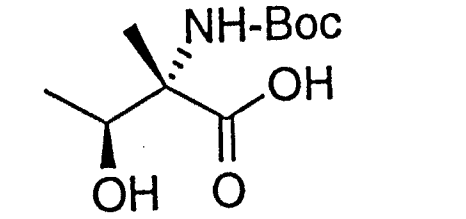
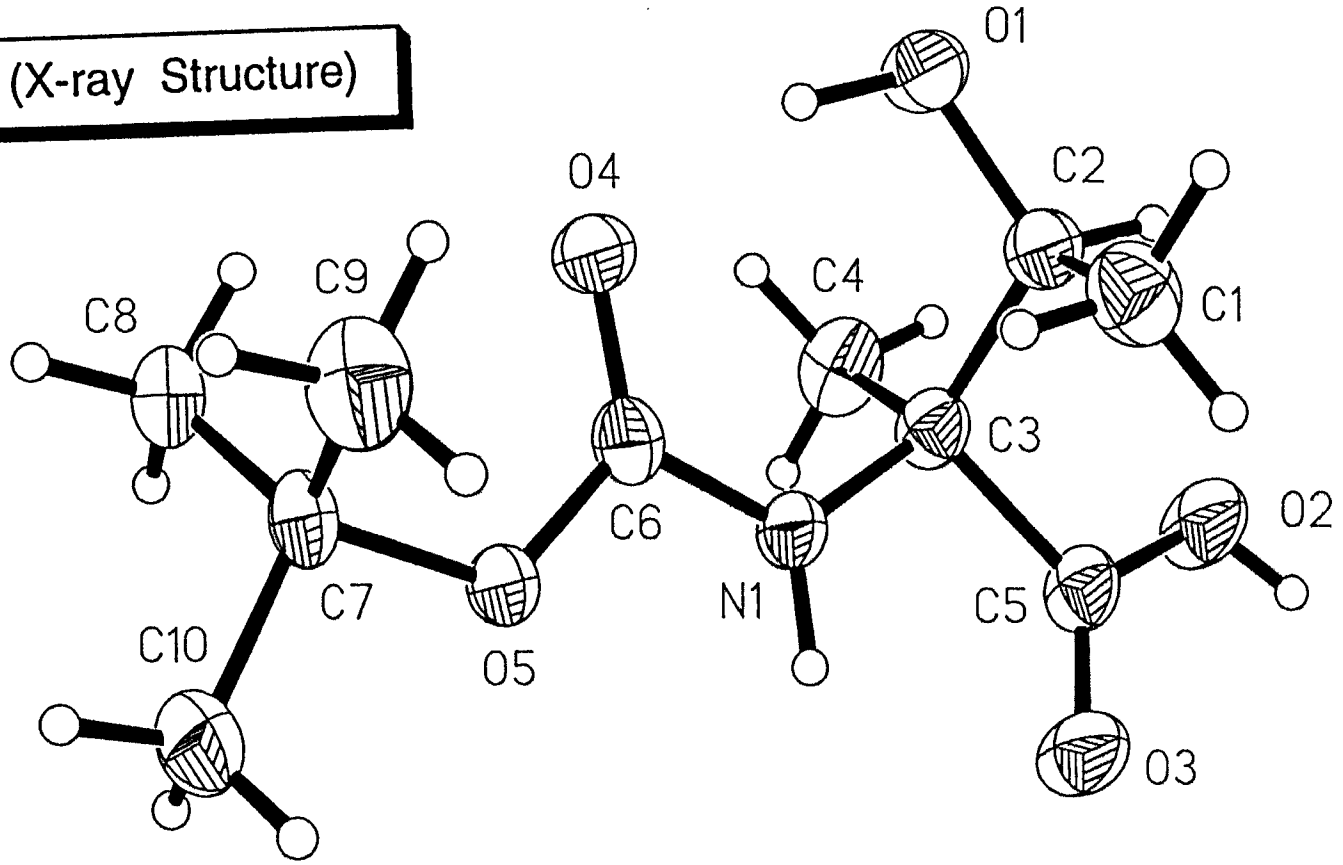
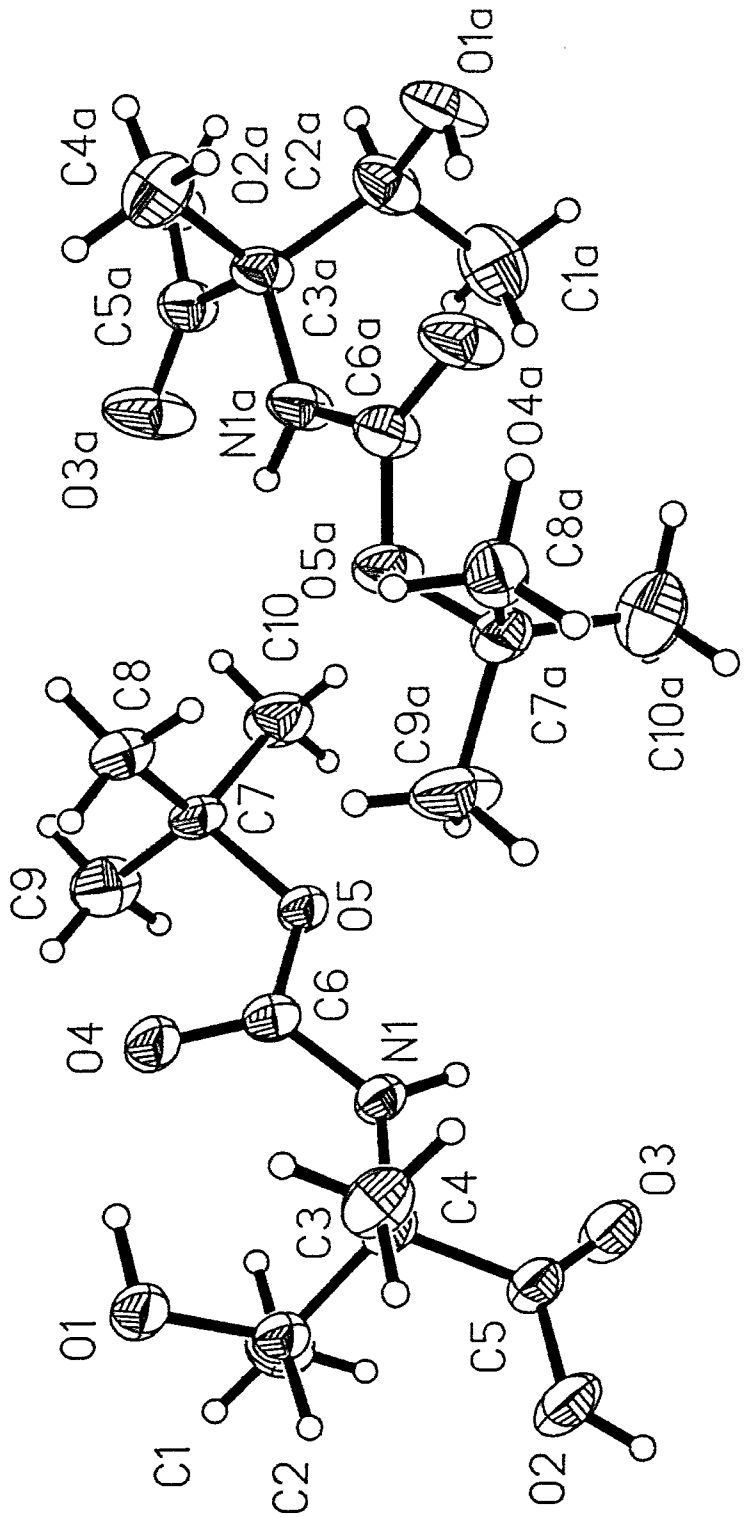


(X-ray Structure)




O1a
C4a
O2a
C2a
C5a
C3a
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O3a
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C6a
O4a
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O5a
C10
C9a
C7a
C10a
C8
C7
C9
O5
C6
O4
N1
C3
C4
O3
O1
C5
C1
C2
O2

STRUCTURE DETERMINATION SUMMARY

Crystal Data

Empirical Formula	$C_{20}\ H_{38}\ N_2\ O_{10}$
Color; Habit	colorless block
Crystal size (mm)	0.3 x 0.5 x 0.8
Crystal System	Orthorhombic
Space Group	$P2_12_12$
Unit Cell Dimensions	a = 10.211(2) Å
	b = 10.960(3) Å
	c = 22.792(6) Å
Volume	2550.6(11) $Å^3$
Z	4
Formula weight	466.5
Density(calc.)	1.215 Mg/m^3
Absorption Coefficient	0.097 mm^{-1}
F(000)	1008

Data Collection

Diffractometer Used	Siemens R3m/V
Radiation	MoKα (λ = 0.71073 Å)
Temperature (K)	176
Monochromator	Highly oriented graphite crystal
2θ Range	3.0 to 55.0°
Scan Type	Wyckoff
Scan Speed	Constant; 4.99°/min. in ω
Scan Range (ω)	0.60°
Background Measurement	Stationary crystal and stationary counter at beginning and end of scan, each for 0.5% of total scan time
Standard Reflections	0 measured every 197 reflections
Index Ranges	$0 \leq h \leq 10$, $-1 \leq k \leq 14$ $0 \leq l \leq 29$
Reflections Collected	3280
Independent Reflections	3270 (R_{int} = 0.74%)
Observed Reflections	2814 ($F > 4.0\sigma(F)$)
Absorption Correction	N/A

Solution and Refinement

System Used	Siemens SHELXTL PLUS (PC Version)
Solution	Direct Methods
Refinement Method	Full-Matrix Least-Squares
Quantity Minimized	$\sum w(F_o-F_c)^2$
Absolute Structure	N/A
Extinction Correction	χ = 0.00038(13), where $F^* = F\,[\,1 + 0.002\chi F^2/\sin(2\theta)\,]^{-1/4}$
Hydrogen Atoms	Riding model, fixed isotropic U
Weighting Scheme	$w^{-1} = \sigma^2(F) + 0.0010F^2$
Number of Parameters Refined	300
Final R Indices (obs. data)	R = 4.31 %, wR = 6.07 %
R Indices (all data)	R = 5.11 %, wR = 6.27 %
Goodness-of-Fit	1.46
Largest and Mean Δ/σ	0.002, 0.001
Data-to-Parameter Ratio	9.4:1
Largest Difference Peak	0.36 eÅ^{-3}
Largest Difference Hole	-0.27 eÅ^{-3}

Table 1. Atomic coordinates ($x10^4$) and equivalent isotropic displacement coefficients ($Å^2 x10^3$)

	x	y	z	U(eq)
O(1)	8858(2)	6675(2)	5809(1)	38(1)
O(2)	5985(2)	8259(3)	4812(1)	45(1)
O(3)	4713(2)	8493(3)	5595(1)	44(1)
O(4)	8508(2)	7786(2)	6811(1)	41(1)
O(5)	6761(2)	8549(2)	7306(1)	29(1)
N(1)	6613(2)	8321(2)	6347(1)	29(1)
C(1)	6663(4)	5859(3)	5848(1)	43(1)
C(2)	7553(3)	6843(3)	5609(1)	33(1)
C(3)	7035(3)	8159(3)	5738(1)	30(1)
C(4)	8038(3)	9117(3)	5557(1)	41(1)
C(5)	5765(3)	8326(3)	5381(1)	32(1)
C(6)	7381(3)	8191(3)	6822(1)	28(1)
C(7)	7361(3)	8360(3)	7893(1)	28(1)
C(8)	8638(3)	9050(3)	7941(1)	37(1)
C(9)	7507(4)	7003(3)	8008(1)	48(1)
C(10)	6340(3)	8913(4)	8292(1)	47(1)
O(1A)	7499(3)	14849(2)	9209(1)	49(1)
O(2A)	7782(2)	11772(2)	10162(1)	38(1)
O(3A)	7951(3)	10533(2)	9402(1)	59(1)
O(4A)	7906(3)	14090(2)	8148(1)	48(1)
O(5A)	7979(2)	12298(2)	7662(1)	39(1)
N(1A)	8020(3)	12269(2)	8623(1)	36(1)
C(1A)	5767(4)	13315(4)	9201(2)	57(1)
C(2A)	7120(3)	13649(3)	9405(1)	39(1)
C(3A)	8163(3)	12694(3)	9232(1)	32(1)
C(4A)	9555(3)	13168(4)	9348(1)	49(1)
C(5A)	7933(3)	11537(3)	9603(1)	35(1)
C(6A)	7972(3)	12986(3)	8143(1)	35(1)
C(7A)	7689(3)	12842(3)	7079(1)	33(1)
C(8A)	8661(3)	13824(3)	6930(1)	36(1)
C(9A)	7851(4)	11740(3)	6675(1)	55(1)
C(10A)	6281(3)	13306(4)	7078(2)	52(1)

* Equivalent isotropic U defined as one third of the trace of the orthogonalized U_{ij} tensor

Table 2. Bond lengths (Å)

O(1)-C(2)	1.420	(4)	O(2)-C(5)	1.318	(3)
O(3)-C(5)	1.194	(4)	O(4)-C(6)	1.234	(4)
O(5)-C(6)	1.332	(3)	O(5)-C(7)	1.485	(3)
N(1)-C(3)	1.465	(3)	N(1)-C(6)	1.343	(3)
C(1)-C(2)	1.511	(5)	C(2)-C(3)	1.564	(4)
C(3)-C(4)	1.523	(5)	C(3)-C(5)	1.542	(4)
C(7)-C(8)	1.511	(4)	C(7)-C(9)	1.518	(4)
C(7)-C(10)	1.511	(4)	O(1A)-C(2A)	1.442	(4)
O(2A)-C(5A)	1.308	(3)	O(3A)-C(5A)	1.192	(4)
O(4A)-C(6A)	1.212	(3)	O(5A)-C(6A)	1.330	(3)
O(5A)-C(7A)	1.486	(3)	N(1A)-C(3A)	1.470	(3)
N(1A)-C(6A)	1.349	(3)	C(1A)-C(2A)	1.503	(5)
C(2A)-C(3A)	1.544	(5)	C(3A)-C(4A)	1.537	(5)
C(3A)-C(5A)	1.543	(4)	C(7A)-C(8A)	1.503	(5)
C(7A)-C(9A)	1.528	(4)	C(7A)-C(10A)	1.525	(5)

Table 3. Bond angles (°)

C(6)-O(5)-C(7)	120.6(2)	C(3)-N(1)-C(6)	125.4(2)
O(1)-C(2)-C(1)	110.8(3)	O(1)-C(2)-C(3)	112.1(2)
C(1)-C(2)-C(3)	112.8(3)	N(1)-C(3)-C(2)	112.9(2)
N(1)-C(3)-C(4)	111.8(2)	C(2)-C(3)-C(4)	110.9(2)
N(1)-C(3)-C(5)	103.8(2)	C(2)-C(3)-C(5)	107.1(2)
C(4)-C(3)-C(5)	109.9(2)	O(2)-C(5)-O(3)	124.3(3)
O(2)-C(5)-C(3)	111.6(2)	O(3)-C(5)-C(3)	124.0(2)
O(4)-C(6)-O(5)	124.4(2)	O(4)-C(6)-N(1)	124.5(2)
O(5)-C(6)-N(1)	111.1(2)	O(5)-C(7)-C(8)	110.6(2)
O(5)-C(7)-C(9)	109.4(2)	C(8)-C(7)-C(9)	113.1(3)
O(5)-C(7)-C(10)	101.6(2)	C(8)-C(7)-C(10)	110.6(2)
C(9)-C(7)-C(10)	110.9(3)	C(6A)-O(5A)-C(7A)	120.5(2)
C(3A)-N(1A)-C(6A)	125.8(2)	O(1A)-C(2A)-C(1A)	111.9(3)
O(1A)-C(2A)-C(3A)	110.8(3)	C(1A)-C(2A)-C(3A)	113.0(3)
N(1A)-C(3A)-C(2A)	112.8(2)	N(1A)-C(3A)-C(4A)	111.2(2)
C(2A)-C(3A)-C(4A)	111.4(3)	N(1A)-C(3A)-C(5A)	104.0(2)
C(2A)-C(3A)-C(5A)	108.2(2)	C(4A)-C(3A)-C(5A)	108.9(3)
O(2A)-C(5A)-O(3A)	124.0(3)	O(2A)-C(5A)-C(3A)	113.0(2)
O(3A)-C(5A)-C(3A)	123.0(2)	O(4A)-C(6A)-O(5A)	125.0(2)
O(4A)-C(6A)-N(1A)	125.2(2)	O(5A)-C(6A)-N(1A)	109.8(2)
O(5A)-C(7A)-C(8A)	110.9(2)	O(5A)-C(7A)-C(9A)	101.5(2)
C(8A)-C(7A)-C(9A)	111.0(3)	O(5A)-C(7A)-C(10A)	108.8(2)
C(8A)-C(7A)-C(10A)	112.6(3)	C(9A)-C(7A)-C(10A)	111.5(3)

Table 4. Anisotropic displacement coefficients ($Å^2 x 10^3$)

	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
O(1)	31(1)	61(1)	22(1)	9(1)	1(1)	-2(1)
O(2)	36(1)	79(2)	20(1)	4(1)	-5(1)	0(1)
O(3)	33(1)	72(2)	27(1)	7(1)	-3(1)	0(1)
O(4)	34(1)	64(1)	25(1)	14(1)	-2(1)	-7(1)
O(5)	28(1)	38(1)	21(1)	4(1)	-1(1)	-3(1)
N(1)	27(1)	39(1)	21(1)	3(1)	-2(1)	-2(1)
C(1)	47(2)	34(2)	47(2)	-4(2)	2(2)	-3(1)
C(2)	32(2)	45(2)	22(1)	4(2)	0(1)	-4(1)
C(3)	29(2)	38(1)	22(1)	-2(1)	-1(1)	2(1)
C(4)	43(2)	47(2)	32(1)	-8(2)	0(1)	10(1)
C(5)	34(2)	38(2)	23(1)	0(2)	-4(1)	3(1)
C(6)	32(2)	31(1)	22(1)	-2(1)	1(1)	-1(1)
C(7)	37(2)	29(1)	19(1)	-3(1)	-2(1)	-1(1)
C(8)	42(2)	39(2)	30(1)	-3(2)	-5(1)	-6(1)
C(9)	70(2)	36(2)	38(2)	-2(2)	-7(2)	6(1)
C(10)	44(2)	65(2)	33(1)	1(2)	4(1)	-11(2)
O(1A)	96(2)	22(1)	28(1)	3(1)	13(1)	-1(1)
O(2A)	62(1)	27(1)	24(1)	0(1)	2(1)	1(1)
O(3A)	122(3)	23(1)	30(1)	-3(1)	0(1)	1(1)
O(4A)	91(2)	25(1)	27(1)	4(1)	4(1)	1(1)
O(5A)	70(2)	28(1)	19(1)	3(1)	-4(1)	0(1)
N(1A)	66(2)	22(1)	19(1)	6(1)	-2(1)	0(1)
C(1A)	45(2)	56(2)	70(2)	8(2)	12(2)	5(2)
C(2A)	56(2)	26(1)	34(1)	4(2)	8(1)	4(1)
C(3A)	48(2)	25(1)	22(1)	1(1)	0(1)	-1(1)
C(4A)	44(2)	58(2)	44(2)	-7(2)	-6(1)	7(2)
C(5A)	50(2)	27(1)	28(1)	2(2)	-4(1)	1(1)
C(6A)	53(2)	25(1)	26(1)	2(1)	1(1)	-1(1)
C(7A)	47(2)	34(1)	20(1)	3(1)	-6(1)	1(1)
C(8A)	37(2)	41(2)	31(1)	6(1)	3(1)	4(1)
C(9A)	97(3)	45(2)	23(1)	-7(2)	-6(2)	-6(1)
C(10A)	41(2)	65(3)	51(2)	-6(2)	-4(2)	12(2)

The anisotropic displacement exponent takes the form:

$-2\pi^2(h^2a^{*2}U_{11} + \ldots + 2hka^*b^*U_{12})$

Table 5. H-Atom coordinates ($x10^4$) and isotropic displacement coefficients ($Å^2 x10^3$)

	x	y	z	U
H(1)	8883	6892	6220	50
H(2)	5353	8273	4580	50
H(1A)	5775	8543	6407	50
H(1B)	7017	5070	5757	44(6)
H(1C)	5804	5932	5681	44(6)
H(1D)	6611	5954	6266	44(6)
H(2A)	7564	6744	5191	50
H(4A)	8832	9003	5775	56(6)
H(4B)	7685	9911	5638	56(6)
H(4C)	8221	9047	5145	56(6)
H(8A)	8469	9892	7853	62(7)
H(8B)	9260	8731	7665	62(7)
H(8C)	8987	8982	8331	62(7)
H(9A)	8146	6644	7752	69(7)
H(9B)	6672	6624	7944	69(7)
H(9C)	7769	6884	8409	69(7)
H(10A)	6278	9771	8211	62(7)
H(10B)	6600	8796	8693	62(7)
H(10C)	5503	8536	8228	62(7)
H(1E)	7691	14809	8847	50
H(2B)	7577	12534	10317	50
H(1F)	7928	11461	8567	50
H(1G)	5142	13873	9365	86(8)
H(1H)	5562	12499	9323	86(8)
H(1I)	5727	13347	8780	86(8)
H(2B)	7075	13676	9825	50
H(4D)	10179	12556	9237	76(8)
H(4E)	9699	13893	9121	76(8)
H(4F)	9657	13353	9758	76(8)
H(8D)	9540	13513	6938	68(7)
H(8E)	8465	14123	6544	68(7)
H(8F)	8580	14476	7209	68(7)
H(9D)	7246	11106	6779	70(8)
H(9E)	7695	11991	6278	70(8)
H(9F)	8731	11439	6711	70(8)
H(10D)	5678	12667	7178	60(7)
H(10E)	6201	13959	7357	60(7)
H(10F)	6086	13606	6692	60(7)