Crystal Structure $C_{22}H_{24}N_2O_8.HCl$

(STRUCTURE DETERMINATION BY POWDER DIFFRACTOMETRY ROUND ROBIN)

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Step 1.

The extraction of individual profiles of reflexes with the help of the program "Profan (CSD for DOS)" was carried out.
The determination of cell parameters with the help of the program "Cell (CSD for WINDOWS)" was carried out.

a = 10.9980(4); b = 12.8789(5); c = 15.7511(5) A; Sp.gr.$P2_12_12_1$

The experimental set from 158 reflexes is received.

Table 1: F(obs).

0	1	1	45.	7.
1	0	1	55.	5.
1	1	0	51.	6.
0	0	2	129.	9.
1	1	1	44.	3.
0	1	2	58.	5.
0	2	0	44.	3.
1	0	2	90.	5.
0	2	1	41.	3.
1	1	2	62.	4.
1	2	0	143.	7.
2	0	0	39.	4.
1	2	1	66.	4.
2	0	1	11.	3.
2	1	0	16.	4.
0	2	2	28.	4.
0	1	3	12.	1.
2	1	1	69.	3.
1	0	3	22.	6.
1	2	2	66.	3.
2	0	2	2.	2.
1	1	3	91.	5.
2	1	2	84.	6.
2	2	0	64.	4.
0	3	1	98.	5.
0	2	3	75.	2.
2	2	1	76.	3.
1	3	0	24.	5.
0	0	4	80.	4.
1	3	1	39.	4.
1	2	3	75.	3.
2	0	3	25.	2.
0	3	2	25.	2.
0	1	4	31.	2.
1	0	4	28.	2.
2	2	2	79.	4.
2	1	3	91.	4.
3	0	1	51.	5.
1	3	2	36.	4.
1	1	4	36.	4.
3	1	0	2.	2.
3	1	1	2.	2.
2	3	0	24.	5.
0	2	4	9.	2.
3	0	2	62.	4.
0	3	3	62.	4.
2	3	1	34.	2.
1	3	3	23.	2.
0	4	1	38.	5.
3	2	1	86.	3.
2	3	2	71.	4.
2	1	4	71.	4.
1	4	0	57.	2.
0	1	5	55.	4.
1	4	1	26.	2.
1	0	5	26.	2.
3	0	3	2.	2.
0	4	2	51.	6.
3	2	2	44.	3.
1	1	5	72.	6.
3	1	3	24.	2.
0	3	4	38.	4.
1	4	2	40.	4.
2	2	4	42.	4.
2	3	3	62.	3.
0	2	5	3.	1.
1	3	4	59.	3.
3	3	0	46.	2.
2	4	0	28.	2.
4	0	0	2.	2.
3	3	1	25.	4.
0	4	3	49.	5.
1	2	5	56.	4.
2	4	1	56.	4.
2	0	5	111.	5.
3	2	3	46.	3.
4	0	1	77.	4.
4	1	0	49.	2.
3	0	4	45.	2.
2	1	5	63.	4.
1	4	3	20.	3.
4	1	1	38.	3.
3	3	2	35.	4.
3	1	4	35.	4.
0	0	6	71.	5.
2	4	2	34.	2.
4	0	2	27.	3.
0	1	6	56.	5.
2	3	4	65.	5.
1	0	6	31.	2.
4	1	2	54.	5.
0	5	1	76.	5.
0	3	5	76.	5.
4	2	0	35.	3.
1	5	1	46.	5.
1	3	5	46.	5.
3	3	3	8.	1.
2	4	3	24.	3.
0	5	2	45.	7.
4	0	3	47.	4.
1	4	4	43.	5.
0	2	6	61.	5.
3	4	0	17.	2.
4	2	2	32.	3.
4	1	3	30.	4.
3	4	1	30.	4.
1	5	2	30.	4.
3	0	5	43.	4.
1	2	6	59.	6.
2	0	6	16.	2.
3	1	5	37.	5.
2	5	0	22.	2.
2	1	6	16.	2.
4	3	0	22.	2.
3	4	2	16.	2.
0	5	3	22.	2.
2	5	1	36.	4.
2	3	5	36.	4.
4	3	1	53.	4.
4	2	3	38.	2.
3	3	4	38.	4.
2	4	4	27.	4.
1	5	3	34.	2.
4	0	4	33.	7.
0	4	5	33.	7.
3	2	5	42.	5.
0	3	6	58.	5.
2	5	2	35.	4.
2	2	6	35.	4.
4	3	2	35.	4.
4	1	4	35.	4.
0	1	7	58.	4.
1	4	5	21.	2.
1	0	7	30.	2.
3	4	3	66.	7.
1	3	6	66.	7.
5	0	1	32.	2.
1	1	7	59.	8.
5	1	0	83.	8.
0	5	4	52.	4.

2	2	3	92.	4.	2	2	5	28.	3.	5	1	1	20.	3.
0	4	0	54.	4.	1	5	0	44.	7.	0	6	0	38.	3.
3	1	2	54.	4.	1	1	6	31.	7.	3	0	6	2.	2.
1	2	4	67.	2.	4	2	1	17.	4.	2	5	3	39.	3.
2	0	4	59.	2.	0	4	4	17.	4.	4	2	4	39.	3.
3	2	0	23.	2.	3	2	4	46.	5.	0	6	1	55.	3.

Step 2.

Calculation of Patterson function. Program CSDmain (CSD for WINDOWS); (E*F "Sharp Patterson")

Table 2: Distribution of Patterson function and definition of heavy atom.

N	x	y	z	H(c)	H(o)	Vector
1	.0000	.0000	.0000	999.9	999.9	
2*	.1345	.5000	.5000	151.8	154.1	2x; 1/2; 2z-1/2
3	.2030	.0000	.1549	125.3	126.7	
4*	.3576	.1587	.5000	92.3	92.5	1/2-2x; 2y; 1/2
5	.5000	.2408	.1982	87.8	88.3	
6	.0826	.4953	.0860	85.3	87.0	
7*	.5000	.3460	.0000	79.0	79.3	1/2; 1/2-2y; -2z
8	.0000	.1168	.4144	77.8	80.6	
9	.1852	.0708	.3246	66.8	69.3	
10	.1993	.1866	.0771	65.3	66.1	
11	.2667	.3288	.0699	61.0	61.6	
12	.3166	.4974	.0000	60.7	61.8	
13	.0000	.3524	.1961	54.5	55.9	
14	.3333	.2771	.3480	53.5	54.8	
15	.5000	.0000	.0360	53.2	53.1	
16	.0814	.4967	.3496	52.8	53.6	
17	.4228	.2302	.0000	46.3	46.6	
18	.1956	.0000	.5000	45.7	45.6	
19	.5000	.3581	.4017	44.5	45.9	
20	.5000	.4919	.3529	43.3	43.4	
21	.2893	.4988	.3107	38.7	40.6	
22	.5000	.4991	.1633	35.3	36.6	
23	.0729	.4941	.2139	33.2	33.9	
24	.3349	.3759	.5000	28.8	29.2	
25	.4757	.1254	.3391	23.2	24.5	
26	.0000	.1859	.1134	19.5	21.2	
27	.3205	.3678	.2237	18.5	18.9	
28	.0000	.0929	.1918	11.8	12.4	

Atom Cl ---> x = 0.069; y = 0.078; z = 0.0

Step 3.

Structure factors calculation and scale parameter refiniment.
R_f=0.57; Program CSDmain (CSD for WINDOWS).
The Fourier differences syntheses. (Number 1) Program CSDmain (CSD for WINDOWS).

x	y	z	H(c)	H(o)	
.7286	.2096	.4911	949.0	964.0	
.4023	.0633	.9424	937.0	948.0	
.8811	.1947	.7957	576.0	577.0	Atom 3 (c6)
.6042	.1444	.0138	574.0	576.0	
.8362	.0599	.8765	516.0	521.0	Atom 1 (O3)
.2497	.0483	.8287	514.0	519.0	(N1)
.9766	.1358	.4669	510.0	521.0	
.8250	.1106	.1499	485.0	494.0	(O4)
.2505	.1450	.5917	483.0	485.0	
.9920	.1350	.5799	449.0	453.0	(O1)
.8821	.1503	.6974	418.0	424.0	Atom 4 (c5)
.7970	.1878	.8624	377.0	378.0	Atom 2 (c7)
.8385	.2194	.0293	370.0	371.0	
.0542	.2002	.7954	283.0	287.0	
.4634	.1139	.6199	277.0	280.0	(N2)
.6898	.0500	.9054	274.0	285.0	
.0601	.0353	.7958	243.0	244.0	
.4644	.2008	.7799	240.0	240.0	
.2429	.1454	.2740	235.0	239.0	
.8315	.0358	.7344	224.0	229.0	Atom 5 (c8)
.2527	.0610	.1660	166.0	177.0	
.6748	.0177	.7610	146.0	149.0	Atom 6 (c9)
.4462	.1281	.3819	142.0	147.0	
.3997	.1256	.1272	93.0	93.0	
.3096	.1736	.0974	78.0	80.0	
.3554	.1455	.1137	74.0	74.0	

On this synthesis we searched for the connected chain of atoms:

1 - 2 - 3 - 4 - 5 - 6

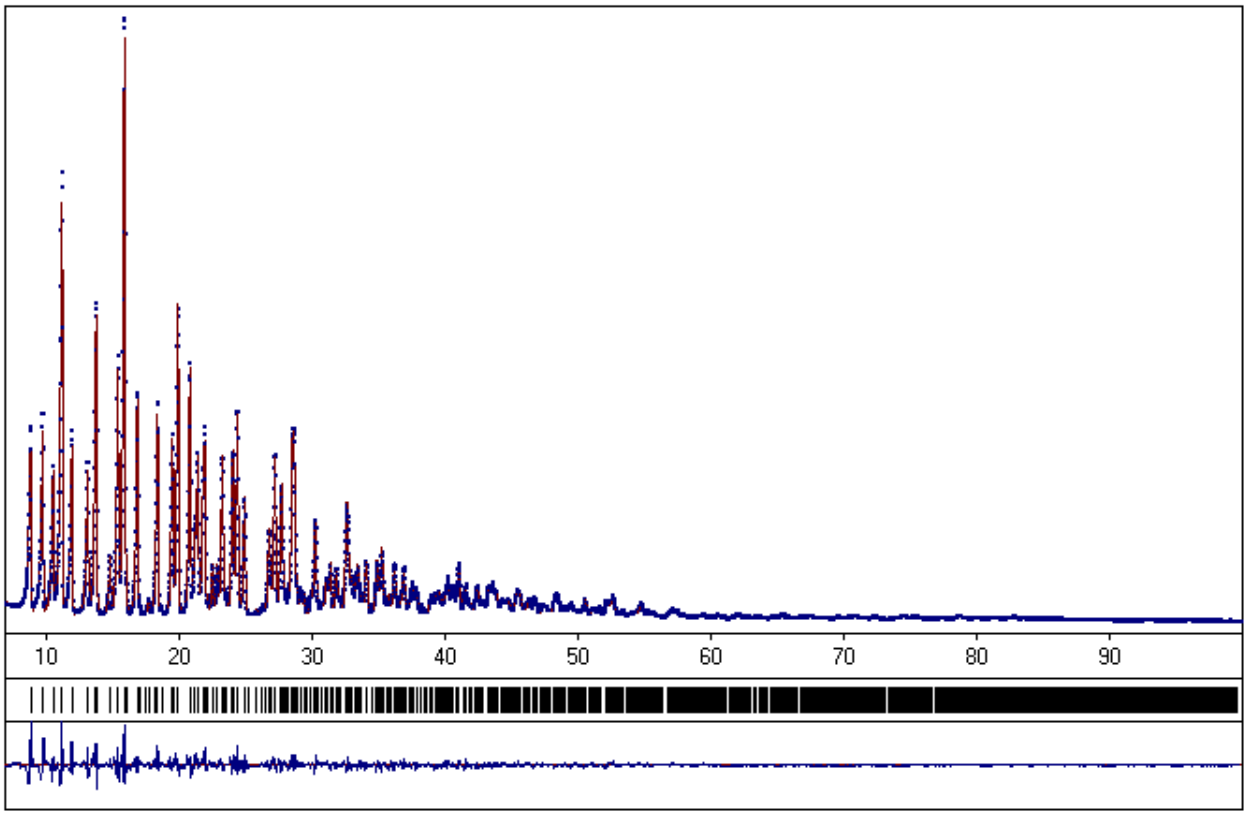
These atoms were added in structural model.

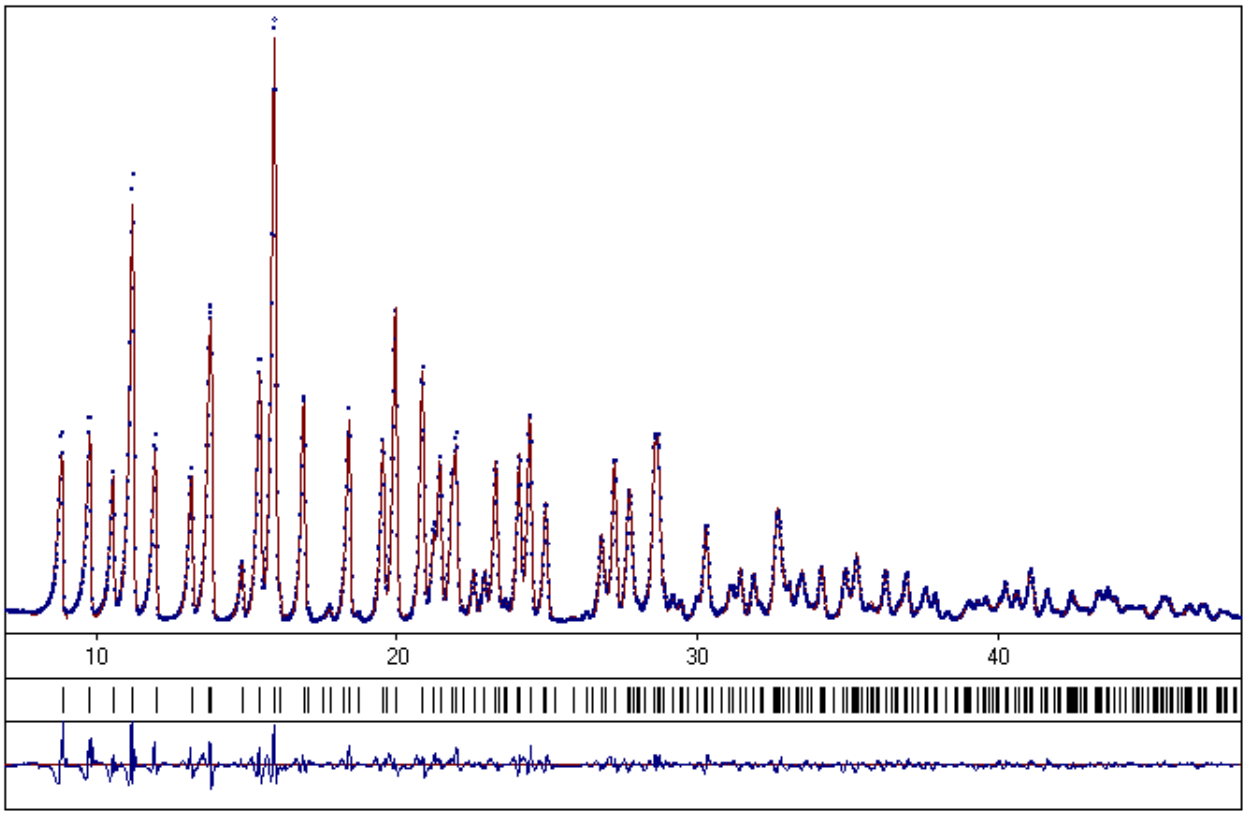
(As was found out, after the end of calculations, on this synthesis there were four correct positions: N1, N2, O1, O4)

Structure factors calculation and scale parameter refiniment.
R_f=0.44;

Step 4. Rietveld refinements.

We repeated Rietveld refinements and Fourier syntheses, the not final model (without atoms of hydrogen) was not received yet. (Program CSDmain (CSD for WINDOWS))
All atoms were put gradually, however for a correct arrangement of atoms C,O,N we have taken advantage of rules of chemistry.





Crystallographic data for $C_{22}H_{24}N_2O_8HCl$

Space group	:	P 2_1 2_1 2_1
a (A)	:	11.0045(3)
b	:	12.8852(3)
c	:	15.7538(3)
Cell volume (A**3)	:	2233.8(2)
Radiation and wavelenght	:	Cu
Mode of refinement	:	Full profile
Number of atom sites	:	33
Number of free parameters	:	140
Two-theta and sinT/l (max)	:	99.73 0.496
Texture axis and parameter	:	[0 0 1] 0.920(6)
R(f)	:	0.042
R(profile)	:	0.064
R(profile - background)	:	0.076

Atomic parameters for $C_{22}H_{24}N_2O_8HCl$

Atom	x/a	y/b	z/c	B(is/eq)	N
Cl	0.0689(2)	0.0793(1)	0.0011(1)	5.77(11)E	4
N1	0.2325(8)	0.0594(6)	0.8534(5)	5.6(3)	4
N2	0.4523(9)	0.1368(5)	0.5893(5)	4.9(2)	4
O1	0.9868(5)	0.1245(4)	0.5627(4)	2.7(2)	4
O2	0.6713(5)	0.0047(4)	0.1192(3)	1.64(12)	4
O3	0.8536(5)	0.0471(5)	0.8726(4)	4.6(2)	4
O4	0.8468(5)	0.1118(5)	0.1441(4)	4.5(2)	4
O5	0.0877(5)	0.1013(5)	0.1962(4)	2.93(14)	4
O6	0.8672(6)	0.2177(5)	0.3101(4)	3.1(2)	4
O7	0.2552(6)	0.1432(5)	0.4302(5)	3.0(2)	4

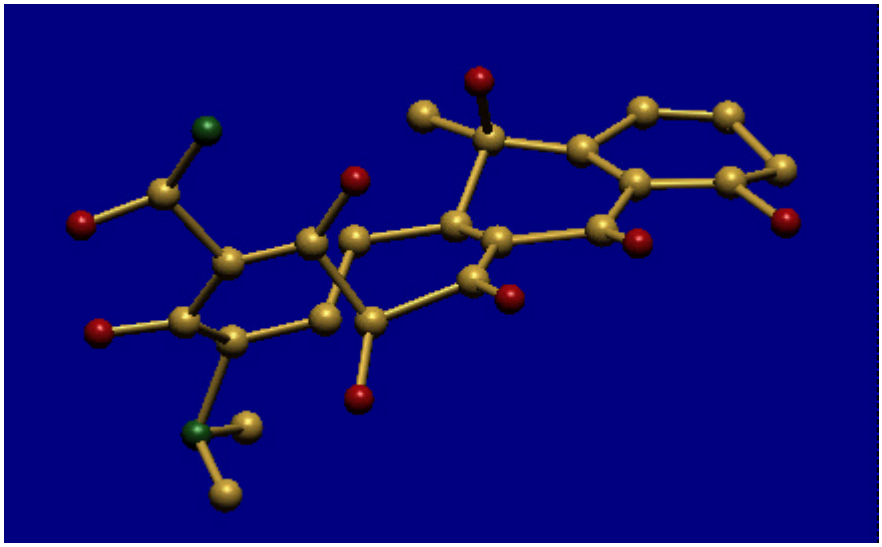
O8	0.6629(4)	0.1896(3)	0.5386(2)	5.5(2)	4
C1	0.4746(12)	0.2295(8)	0.2002(8)	5.9(4)	4
C2	0.5475(11)	0.1930(9)	0.2574(9)	6.5(4)	4
C3	0.5396(12)	0.2289(10)	0.3474(7)	7.2(5)	4
C4	0.9792(10)	0.1636(7)	0.6470(6)	2.5(3)	4
C5	0.8908(10)	0.1451(8)	0.7131(8)	4.4(4)	4
C6	0.8784(11)	0.1925(10)	0.7939(8)	5.2(4)	4
C7	0.8050(9)	0.1480(8)	0.8593(7)	1.8(3)	4
C8	0.8109(11)	0.0537(9)	0.6765(7)	2.8(3)	4
C9	0.7045(8)	0.0420(7)	0.7459(5)	2.2(3)	4
C10	0.6843(8)	0.1102(8)	0.8128(7)	2.1(3)	4
C11	0.5763(9)	0.0741(7)	0.8686(6)	2.4(3)	4
C12	0.8605(8)	0.0453(7)	0.2077(5)	1.4(3)	4
C13	0.9956(8)	0.0725(7)	0.2500(6)	1.3(3)	4
C14	0.4675(9)	0.0262(8)	0.8215(6)	3.0(3)	4
C15	0.1399(9)	-0.0106(8)	0.3734(6)	3.7(3)	4
C16	0.9702(11)	0.1654(9)	0.3186(8)	6.8(4)	4
C17	0.0662(11)	0.1816(10)	0.3645(7)	3.7(3)	4
C18	0.1654(12)	0.1167(10)	0.3804(7)	6.7(4)	4
C19	0.5558(10)	0.2080(7)	0.5748(7)	4.0(3)	4
C20	0.1953(9)	0.0222(7)	0.7764(6)	3.4(3)	4
C21	0.2874(9)	0.1710(7)	0.8575(5)	1.9(3)	4
C22	0.7703(7)	0.1963(7)	0.9428(5)	2.4(3)	4

* B(eq) = 1/3[B11 a*2 a2 + ... 2B23 b* c* b c cos(alpha)]

Anisotropic parameters for $C_{22}H_{24}N_2O_8HCl$

Atom	B11	B22	B33	B12	B13	B23
Cl	4.9(2)*	8.0(2)*	4.4(2)*	-1.2(2)*	-1.6(2)*	0.6(2)*

* T = exp[-1/4(B11 a*2 h2 + ... 2B23 b* c* k l)]



(My wife speaks, that it tetracycline hydrochloride ...)