QUESTIONNAIRE FOR
STRUCTURE DETERMINATION BY POWDER DIFFRACTOMETRY ROUND ROBIN

Sample number 2: C22 H24 N2 O8 HCl

0.2 Is the second sample structure solvable with this quality of data ? **Yes [x]** No [ ]

1. Preliminary work

1.1 Did you obtained additional informations from the chemical formula ? If yes, how and what information ? (for instance from CSD or ICSD databanks)

>> Answer: Molecular topology obtained from CSD.

1.2 Did you obtained additional informations from the powder pattern ? If yes, how and what information ? (for instance using the JCPDS-ICDD database)

>> Answer: No additional information from the powder pattern

1.3 Did you extract the structure factors ? **Yes [x]** No [ ]

1.3.1 If yes, which program(s) did you use ?

SR15LS - Rietveld & Pawley synchrotron X-ray refinement program based upon the Cambridge Crystallography Subroutine Library

1.3.2 Give the angular range:

3-30 degrees - aldsync

1.3.3 Give the number of extracted structure factors:

594 extracted structure factors

1.3.4 Give the Rp and Rwp (background subtracted):

Pawley refinement:
Rp (background subtracted) = 7.89%
Rwp (background subtracted) = 8.78%

1.3.5 Give the Rp and Rwp (background not subtracted):

Pawley refinement:
Rp (background not subtracted) = 1.41%
Rwp (background not subtracted) = 2.32%

1.3.6 If not, did you use the whole pattern ?

1.3.7 Or a partial pattern (if yes, give the angular range):

2- Structure solution

2.1 Did you use direct methods ? Yes [ ] **No [x]**

2.1.1 If yes, was it on the whole dataset ?
2.1.2 Or on a partial dataset ?
2.1.3 Give the number of reflections:

2.1.4 Which program(s) did you use ?
2.1.5 Did you modified intensities of closely neighbouring reflections ? If yes, explain how.

2.2 Did you use Patterson methods ? Yes [ ] **No [x]**

2.2.1 If yes, was it on the whole dataset ?
2.2.2 Or on a partial dataset ?
2.2.3 Give the number of reflections:
2.2.4 Which program(s) did you use ?
2.2.5 Did you modified intensities of closely neighbouring reflections ? If yes, explain how.

2.3 Did you use another method ? **Yes [x]** No [ ]

2.3.1 If yes, which method(s) (give details) ?

Global optimisation by simulated annealing

2.3.2 Which program(s) did you use ?

MYSTIC - processing of correlated integrated intensities
DRUID - simulated annealing structure solution

2.4 Did you first locate the whole structure ? **Yes [x]** No [ ]

2.4.1 If not, how many atoms did you locate ?
2.4.2 Give their name and initial atomic coordinates

Atom	x	y	z
.....			
.....			
.....			

2.4.3 Was the initial model derived from the molecular formula? **Yes [x]** No [ ]
2.4.4 Were the initial atomic coordinates taken from a known structure ? Yes [ ] **No [x]**

The initial atomic coordinates were randomly located within the unit cell subject to the molecular bond length, angle and torsion angle constraints imposed by the molecular z-matrix.

2.4.5 If yes, which one (give reference) ?

3- Structure completion

>> N.B. this section is not applicable to the global optimisation method as the entire structure was used throughout the optimisation process.

3.1 Did you perform Fourier difference syntheses before refining the structure by the Rietveld method ? Yes [ ] No [ ]
3.2 If yes, with what program ?
3.3 If yes, how many atoms did you obtained from Fourier difference syntheses ?
3.4 Give their name and atomic coordinates as they were obtained

Atom	x	y	z
.....			
.....			

3.5 Did you make first Rietveld refinements without preliminary Fourier difference syntheses ? Yes [ ] No [ ]

3.5.1 If yes, with what program ?
3.5.2 What were the Rp and Rwp (background subtracted AND not subtracted) and RB and RF that you obtained at the first Rietveld application ?
3.5.3 Did you get the structure factors from this result and performed a Fourier difference synthesis ?
3.5.4 Did you locate additional atoms at this stage ?
3.5.5 And which one ?

Atom	x	y	z
.....			
.....			
.....			

3.5.6 If you repeated Rietveld refinements and Fourier syntheses several times before completing the model, give the number of times and which atoms you locate and the Rp, Rwp RB, RF at each times.

Atom	x	y	z
.....			
.....			
.....			

4- Final refinement

The structure was solved several times (See discussion in 5). The output from the first simulated annealing global optimisation is given in the following table:

atom	x	y	z
O1	0.15006	0.04048	-0.11491
C2	0.20227	0.13385	-0.15158
C3	0.32042	0.10186	-0.19863
C4	0.29318	0.02806	-0.27166
C5	0.18877	0.04918	-0.32428
O6	0.16742	-0.00307	-0.39117
C7	0.10490	0.13325	-0.29874
C8	0.01708	0.16952	-0.35719
O9	0.00760	0.12993	-0.43709
H10	0.05101	0.07567	-0.43581
C11	-0.06163	0.24935	-0.33433
C12	-0.05541	0.29237	-0.25395
C13	0.03123	0.25661	-0.19497
C14	0.11150	0.17747	-0.21656
H15	0.03200	0.28817	-0.14141
H16	-0.10831	0.34145	-0.23389
H17	-0.11412	0.27483	-0.37326
C18	0.36911	-0.05224	-0.29352
O19	0.35129	-0.11156	-0.36197
H20	0.29316	-0.08742	-0.38595
C21	0.48267	-0.08255	-0.24382
O22	0.57696	-0.11497	-0.30075
H23	0.59239	-0.06503	-0.33057
C24	0.45289	-0.18091	-0.18953
O25	0.35728	-0.22855	-0.20431
C26	0.54345	-0.20866	-0.12785
C27	0.53269	-0.30539	-0.07897
O28	0.61542	-0.33946	-0.03065
N29	0.43041	-0.36133	-0.08849
H30	0.42669	-0.42495	-0.06073
H31	0.37883	-0.34233	-0.11376
C32	0.64631	-0.14116	-0.11547
O33	0.74713	-0.16722	-0.08751
C34	0.62439	-0.02387	-0.12546
N35	0.74432	0.03008	-0.13616
C36	0.81924	-0.00639	-0.20960
H37	0.90338	0.02173	-0.19672
H38	0.77834	0.05031	-0.27020
H39	0.82656	-0.08299	-0.21055
C40	0.73076	0.14569	-0.13776
H41	0.81500	0.17995	-0.13233
H42	0.68728	0.16417	-0.18900
H43	0.68443	0.16656	-0.08659
H44	0.79712	0.01844	-0.08780
C45	0.52424	0.00972	-0.18814
C46	0.41616	0.05557	-0.13915
H47	0.45167	0.10619	-0.09816
H48	0.38231	-0.00227	-0.10789
H49	0.55590	0.05880	-0.22710
H50	0.59953	-0.00475	-0.06897
H51	0.35582	0.16080	-0.21500
C52	0.23158	0.21034	-0.08067
H53	0.16286	0.22431	-0.04921
H54	0.26908	0.27013	-0.10313
H55	0.29248	0.17974	-0.04181
H56	0.10287	0.05857	-0.08509
Cl57	0.07638	0.57157	0.49660

Output from final Rietveld analysis:

a = 10.98029(7)A b = 12.85242(7)A c = 15.73303(8)A

atom	x	y	z	B
O1	0.14120	0.03882	-0.12385	3.24960
C2	0.19663	0.13364	-0.15500	2.51348
C3	0.31865	0.10673	-0.19950	2.51348
C4	0.29608	0.03414	-0.27392	2.51348
C5	0.18817	0.05234	-0.32415	2.51348
O6	0.16127	-0.00717	-0.38565	3.65630
C7	0.10654	0.13931	-0.30220	2.51348
C8	0.02352	0.17772	-0.36236	2.51348
O9	0.01210	0.13052	-0.43893	3.24960
H10	-0.00945	0.06887	-0.42672	14.48616
C11	-0.04880	0.26366	-0.34279	2.51348
C12	-0.03965	0.31058	-0.26311	2.51348
C13	0.03705	0.26742	-0.20066	2.51348
C14	0.11166	0.18365	-0.22070	2.51348
H15	0.04003	0.29721	-0.14544	14.48616
H16	-0.08648	0.36719	-0.25024	14.48616
H17	-0.10946	0.28620	-0.38172	14.48616
C18	0.36511	-0.05364	-0.28828	2.51348
O19	0.35154	-0.11278	-0.35730	3.24960
H20	0.28666	-0.09711	-0.37728	14.48616
C21	0.48222	-0.07898	-0.23964	2.51348
O22	0.57582	-0.10741	-0.29877	3.24960
H23	0.54419	-0.10176	-0.34695	14.48616
C24	0.45931	-0.17241	-0.17854	1.95393
O25	0.36600	-0.22487	-0.18858	3.65630
C26	0.55918	-0.19753	-0.12339	2.51348
C27	0.56838	-0.29830	-0.08249	2.38784
O28	0.65920	-0.32149	-0.03742	3.65630
N29	0.46668	-0.35615	-0.07732	5.04263
H30	0.46642	-0.41641	-0.04487	14.48616
H31	0.41258	-0.34176	-0.10206	14.48616
C32	0.65701	-0.12611	-0.11160	2.51348
O33	0.75081	-0.14553	-0.07051	3.65630
C34	0.62752	-0.01027	-0.12489	2.51348
N35	0.74529	0.04798	-0.13787	5.04263
C36	0.80307	0.01648	-0.22022	2.38784
H37	0.89143	0.03890	-0.22006	14.48616
H38	0.74758	0.06149	-0.28178	14.48616
H39	0.79876	-0.05990	-0.22662	14.48616
C40	0.72174	0.16260	-0.13747	2.38784
H41	0.80329	0.20185	-0.13667	14.48616
H42	0.67624	0.18147	-0.18771	14.48616
H43	0.67429	0.18117	-0.08619	14.48616
H44	0.80066	0.03152	-0.09188	14.48616
C45	0.52324	0.01779	-0.18814	2.51348
C46	0.41376	0.06179	-0.13847	2.51348
H47	0.44280	0.11751	-0.09947	14.48616
H48	0.37728	0.00664	-0.10551	14.48616
H49	0.55164	0.06893	-0.22652	14.48616
H50	0.60316	0.00997	-0.06854	14.48616
H51	0.35201	0.16722	-0.21402	14.48616
C52	0.21821	0.20768	-0.08094	2.38784
H53	0.15454	0.20279	-0.04309	14.48616
H54	0.22404	0.27636	-0.10139	14.48616
H55	0.29487	0.18887	-0.05189	14.48616
H56	0.09417	0.05525	-0.09323	14.48616
Cl57	0.07320	0.57892	0.49654	2.46124

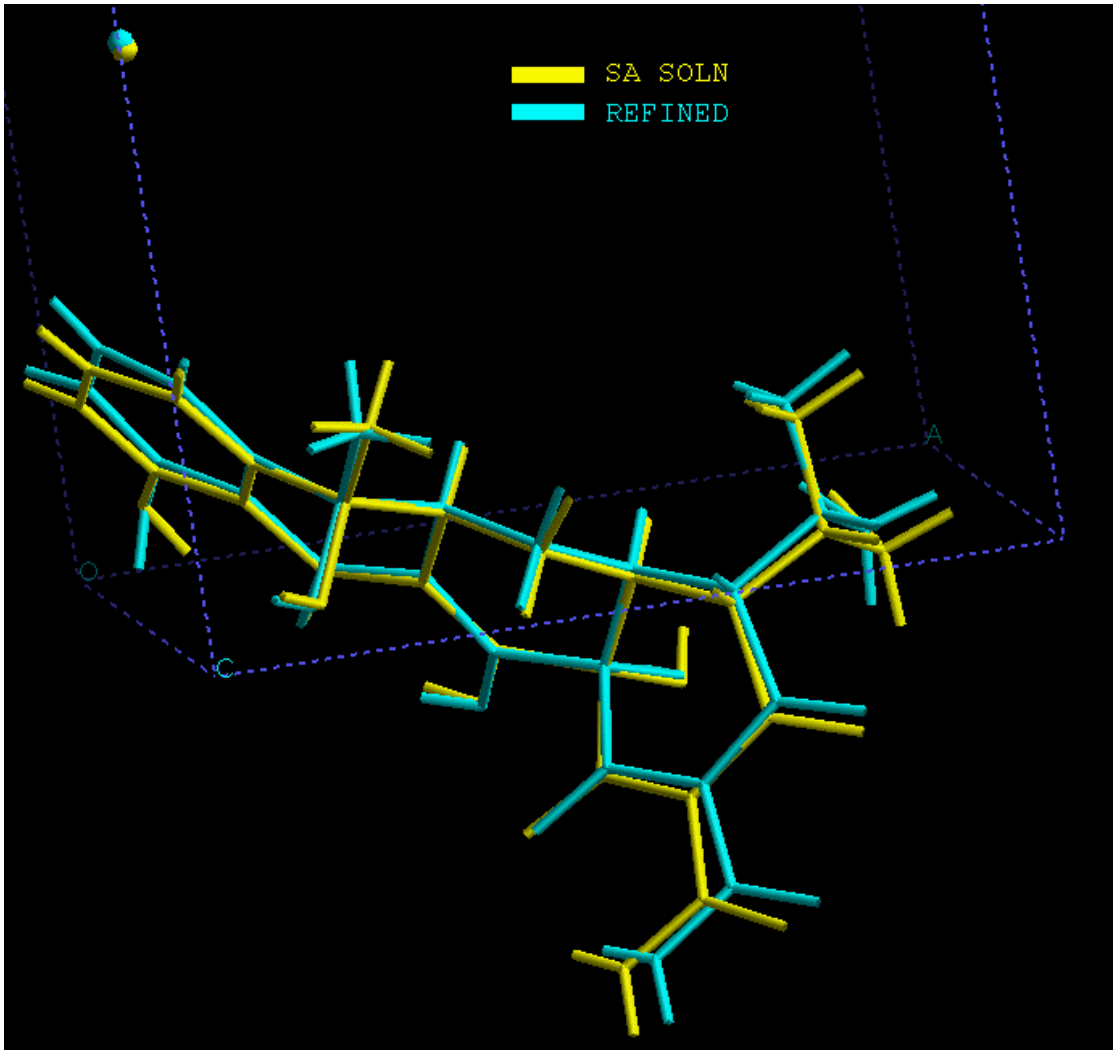
Final Rietveld refinement:
Rp (background subtracted) = 12.10%
Rwp (background subtracted) = 11.16%

Final Rietveld refinement:
Rp (background not subtracted) = 2.11%
Rwp (background not subtracted) = 2.92%

The average separation between the positions of the non-hydrogen atoms at the end of the global optimisation and the end of the Rietveld refinement was 0.19 Angstroms. Given that the global optimisation was performed with 2.5 Angstrom resolution data, this agreement is very good.

Average deviation for non-H atoms = 0.191A
Minimum deviation for non-H atoms = 0.041A
Maximum deviation for non-H atoms = 0.544A

Slack constraints were used in the Rietveld refinement to maintain sensible hydrogen positions and to allow a small amount of molecular relaxation from the bond-lengths, angles and torsion angles given in the molecular z-matrix.



Comparison of the molecular structures obtained from global optimisation and from the final Rietveld refinement.

5- Intermediate results (list of extracted structure factors, software decisive input and output data...) or comments you might consider as essential (details on hardware, time for solving the structure, number of moves by Monte Carlo or molecule position trial...).

MYSTIC:

Although 594 structure factors were extracted, the small number of internal torsion angles suggested that the structure could solved using a more limited set of data. Accordingly, only the first 100 reflections were used. The last reflection (4 2 0) is at 15.757 degrees and corresponds to a d-spacing of 2.52 Angstroms.

The first 100 reflections with their correlations are listed below:

Integrated Intensities used for structure solution

h	k	l	intensity	esd	clump	<	percentage integrated intensity correlations										>
0	1	1	2525.397	17.6510	1	5	3	1	0	-1	-1	0	-1	0	0	0	1	1	1
1	0	1	3634.060	22.9500	2	6	4	2	-1	-1	-1	-2	-1	0	0	0	0	1	1
1	1	0	2456.114	22.4956	3	5	4	1	0	0	-1	-1	-1	-1	-1	-1	0	0	0
0	0	2	16683.375	72.6729	4	4	3	1	0	0	-1	-1	-1	-1	-1	-1	-1	-1	0
1	1	1	1935.280	15.4605	5	5	2	1	2	0	0	-1	-1	-1	-2	-2	-1	-1	-1
0	1	2	3575.600	34.8567	6	2	2	4	3	1	1	0	0	-1	-1	-1	-1	-1	-1
0	2	0	1829.053	105.7208	7	-63	2	2	1	1	0	0	0	0	0	0	0	0	0
1	0	2	8692.088	64.0547	8	2	2	1	1	1	1	1	0	0	0	0	0	0	0
0	2	1	1661.140	37.2779	9	5	4	5	3	4	4	3	2	1	2	0	0	0	-1
1	1	2	4500.994	28.9618	10	2	4	4	4	5	4	3	2	3	1	1	0	-1	-1
1	2	0	20351.135	81.8309	11	-16	3	3	4	3	3	2	2	1	1	1	0	0	-1
2	0	0	2842.955	97.2369	12	4	5	6	5	4	3	4	2	1	1	0	0	-1	-1
1	2	1	4315.415	33.5482	13	-17	5	5	5	3	4	2	2	2	1	0	0	0	0
2	0	1	306.647	49.7878	14	6	7	6	4	6	3	3	3	1	1	0	0	0	-1
2	1	0	-47.190	48.1838	15	3	7	6	8	4	4	4	3	2	1	1	0	0	0
0	2	2	558.871	52.0194	16	6	6	8	4	4	4	3	2	2	1	1	1	0	0
0	1	3	-259.622	54.5412	17	-8	7	4	4	4	3	3	2	2	1	2	1	1	0
2	1	1	5541.909	41.3858	18	1	3	3	4	3	3	2	2	1	2	1	1	0	0
1	0	3	382.661	58.4749	19	5	5	6	5	4	4	3	2	3	3	2	1	1	1
1	2	2	4654.250	45.0868	20	-25	4	4	4	3	3	2	3	3	3	1	1	2	1
2	0	2	-39.216	71.7773	21	0	4	4	4	3	3	4	4	3	2	1	2	1	1
1	1	3	8992.146	54.3492	22	5	5	5	4	3	5	5	4	2	2	3	2	1	2
2	1	2	8381.264	57.1040	23	4	5	5	4	6	6	6	3	3	5	3	2	3	1
2	2	0	4944.725	94.7245	24	-4	5	5	7	7	7	4	3	5	3	3	4	1	1
0	3	1	10264.974	108.0404	25	3	4	7	7	6	4	3	5	3	3	4	1	1	3
0	2	3	5752.476	105.6094	26	-17	6	6	6	4	3	5	3	3	4	1	1	3	2
2	2	1	7049.933	62.1501	27	-2	5	6	3	3	5	3	2	4	1	1	3	2	1
1	3	0	1240.665	93.8587	28	6	8	5	4	7	5	4	6	2	1	5	4	3	2
0	0	4	7727.656	208.1714	29	7	5	5	8	5	4	6	2	1	6	5	3	3	2
1	3	1	1849.617	53.7468	30	3	5	8	5	4	7	2	2	7	6	4	3	2	2
1	2	3	6484.474	71.2959	31	-37	6	3	3	4	2	1	4	4	3	2	2	1	1
2	0	3	1244.871	123.3373	32	-8	3	3	4	1	1	4	4	3	2	2	2	2	1
0	3	2	1050.041	55.0610	33	3	4	7	3	2	7	7	5	4	3	3	3	1	-1
0	1	4	1050.041	55.0610	33	3	4	7	3	2	7	7	5	4	3	3	3	1	-1
1	0	4	850.170	133.8499	34	-43	5	2	1	5	5	4	3	2	2	2	1	-1	1
2	2	2	7088.535	79.1795	35	1	1	1	5	4	3	3	2	2	2	1	-1	1	0
2	1	3	10009.218	78.2814	36	2	2	8	8	6	6	4	4	4	2	-1	2	0	1
3	0	1	133.765	249.4457	37	-83	5	3	2	2	2	2	2	1	0	1	0	1	1
1	3	2	2707.762	64.7549	38	-2	2	2	2	1	1	2	1	0	1	0	1	1	1
1	1	4	2707.762	64.7549	38	-2	2	2	2	1	1	2	1	0	1	0	1	1	1
3	1	0	-154.722	119.7908	39	10	9	8	6	6	7	4	-2	4	0	3	6	4	3
3	1	1	-49.043	62.8910	40	8	9	7	7	9	5	-2	5	0	3	9	5	4	7
2	3	0	445.292	131.0449	41	-6	6	7	9	5	-2	5	0	3	8	5	4	7	7
0	2	4	-205.085	131.2091	42	2	7	8	5	-2	5	0	3	9	5	4	7	8	7
3	0	2	4455.336	82.6313	43	-27	6	4	-2	4	0	3	7	4	3	5	6	6	6
0	3	3	4455.336	82.6313	43	-27	6	4	-2	4	0	3	7	4	3	5	6	6	6
2	3	1	1320.778	77.5931	44	3	4	-2	5	0	3	7	5	4	6	7	7	7	6
2	2	3	10148.692	94.1966	45	4	-2	5	0	4	10	6	5	8	9	9	9	8	2
3	1	2	2280.915	158.0921	46	-88	45	-10	9	6	4	3	5	6	6	6	6	2	1
0	4	0	2280.915	158.0921	46	-88	45	-10	9	6	4	3	5	6	6	6	6	2	1
1	2	4	5311.829	226.2054	47	-61	14	-11	-2	-2	-1	-2	-2	-2	-2	-2	-1	0	-2
2	0	4	4410.974	230.6248	48	-30	23	5	4	3	6	7	7	7	6	2	1	6	6
3	2	0	717.606	332.3557	49	-88	3	0	0	0	0	0	0	0	0	0	0	0	0
1	3	3	821.546	164.1521	50	-1	3	2	4	5	5	5	4	1	1	4	4	2	2
0	4	1	1309.325	153.4370	51	3	6	10	12	12	12	12	3	2	11	11	6	5	7
3	2	1	8512.062	104.6434	52	-27	8	8	8	8	8	2	2	7	7	4	4	5	5
2	3	2	5844.777	55.8667	53	-10	6	6	6	6	2	1	6	6	3	3	4	4	4
2	1	4	5844.777	55.8667	53	-10	6	6	6	6	2	1	6	6	3	3	4	4	4
1	4	0	4164.511	171.7079	54	5	11	11	11	3	2	10	11	6	5	7	8	8	5
0	1	5	3696.524	167.8808	55	9	13	13	4	3	12	13	7	6	9	9	10	6	7
1	4	1	1004.083	56.1045	56	7	13	4	3	13	13	7	7	9	10	11	7	8	5
1	0	5	1004.083	56.1045	56	7	13	4	3	13	13	7	7	9	10	11	7	8	5
3	0	3	261.229	164.0454	57	9	4	3	14	14	8	7	10	11	11	8	8	6	-2
0	4	2	2632.935	171.4105	58	-1	5	13	14	8	7	10	11	12	8	9	6	-2	2
3	2	2	797.018	185.9977	59	-84	9	4	2	2	3	3	4	2	3	2	-1	1	1
1	1	5	7361.737	192.2722	60	-6	3	2	2	2	3	3	2	2	2	0	0	0	2
3	1	3	1022.431	87.7246	61	10	8	8	11	12	13	9	10	7	-2	2	2	8	10
0	3	4	1746.718	175.6341	62	5	9	12	13	14	9	11	8	-2	2	2	9	11	7
1	4	2	1367.385	108.3302	63	-50	8	7	8	5	6	5	-1	1	1	5	7	4	0
2	2	4	2147.864	110.3555	64	1	7	8	5	6	4	-1	1	1	5	7	4	0	4
2	3	3	4206.330	100.0254	65	-13	11	7	9	7	-2	2	2	8	10	6	0	7	7
0	2	5	166.251	181.1861	66	8	8	10	7	-2	2	2	9	11	7	0	8	8	11
1	3	4	4070.528	101.7890	67	0	12	8	-2	3	3	10	13	8	0	9	10	12	8

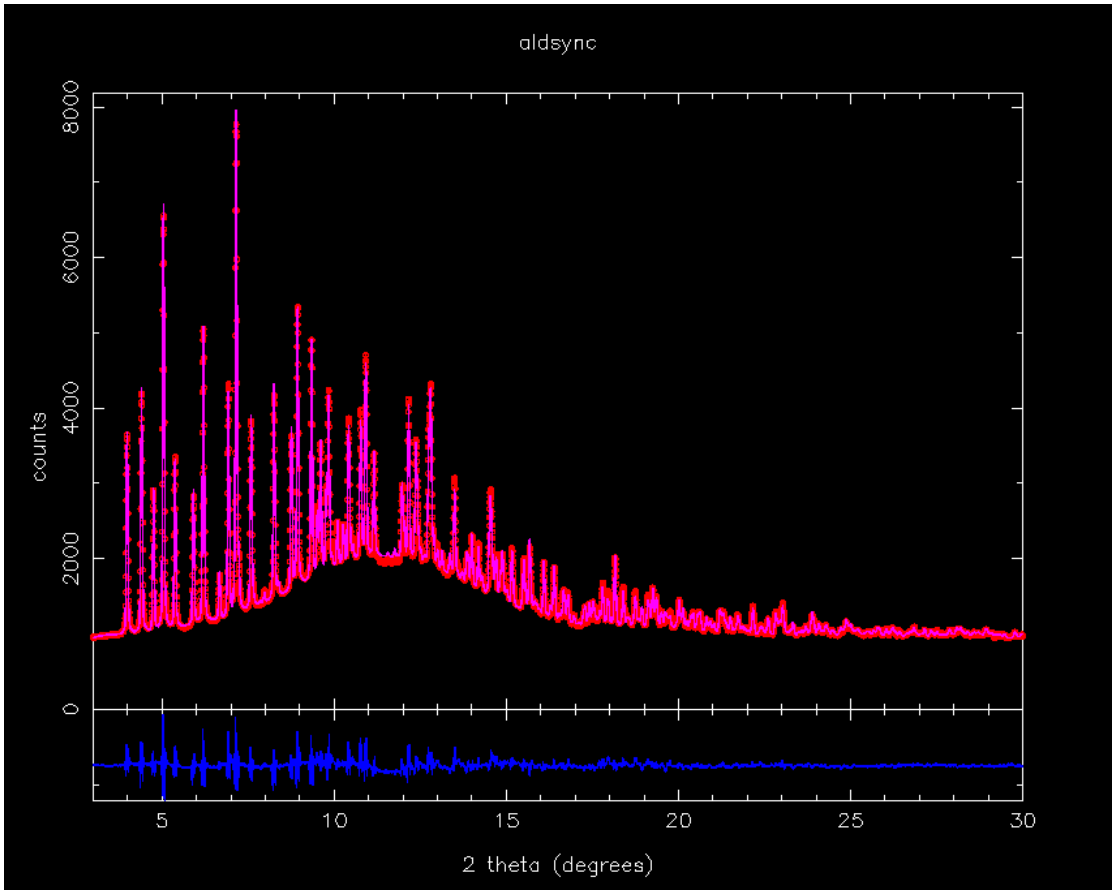
3	3	0	2331.788	199.1066	68	-33	6	-2	2	2	7	9	6	0	6	7	9	6	6
2	4	0	952.205	197.2373	69	4	-1	1	3	8	11	7	0	7	8	10	7	7	12
4	0	0	-590.852	840.3317	70	-82	64	-33	14	7	5	0	6	6	8	6	6	9	6
3	3	1	1957.447	336.3828	71	-92	58	-17	-1	-2	0	-2	-2	-2	-2	-2	-3	-2	-1
0	4	3	1957.447	336.3828	71	-92	58	-17	-1	-2	0	-2	-2	-2	-2	-2	-3	-2	-1
1	2	5	4074.963	488.4318	72	-76	23	1	2	0	2	2	3	2	2	3	2	1	2
2	4	1	4301.207	141.1959	73	-35	6	1	0	2	2	3	2	2	3	2	1	2	1
2	0	5	4301.207	141.1959	73	-35	6	1	0	2	2	3	2	2	3	2	1	2	1
3	2	3	2362.742	112.4926	74	-5	6	0	7	8	10	7	8	12	7	4	8	2	3
4	0	1	7859.427	211.6215	75	2	3	8	10	14	10	10	16	10	5	10	3	5	11
4	1	0	2463.972	309.2284	76	-75	33	1	10	6	7	11	7	4	7	2	3	7	7
3	0	4	3024.325	345.1788	77	-47	8	-1	0	0	1	0	0	0	0	0	0	0	0
2	1	5	4284.830	126.0894	78	-17	12	7	7	12	7	4	8	2	4	8	7	7	2
1	4	3	1024.328	102.8356	79	-12	8	8	14	8	5	9	2	4	9	8	8	2	6
4	1	1	1498.488	102.9823	80	7	11	18	11	6	12	3	6	12	12	11	2	8	3
3	3	2	2381.760	55.7140	81	-38	13	8	5	9	2	4	9	8	8	2	6	2	1
3	1	4	2381.760	55.7140	81	-38	13	8	5	9	2	4	9	8	8	2	6	2	1
0	0	6	2381.760	55.7140	81	-38	13	8	5	9	2	4	9	8	8	2	6	2	1
2	4	2	1344.925	117.3194	82	11	8	5	9	3	4	10	9	9	2	6	2	1	8
4	0	2	798.651	204.5659	83	11	9	16	4	7	17	16	15	3	11	4	2	13	6
0	1	6	3462.311	258.2057	84	-48	17	0	6	10	10	10	2	7	3	1	8	4	9
2	3	4	4787.189	134.0268	85	-17	7	-1	6	6	5	1	4	2	1	5	2	5	3
1	0	6	854.169	229.3437	86	-34	26	8	11	11	2	8	3	2	10	5	10	5	6
4	1	2	612.417	186.5483	87	-76	15	1	3	0	2	1	0	2	1	3	1	1	3
0	5	1	6790.884	189.9230	88	-16	7	4	1	3	1	1	5	2	5	2	3	5	5
0	3	5	6790.884	189.9230	88	-16	7	4	1	3	1	1	5	2	5	2	3	5	5
4	2	0	2187.711	219.6176	89	-7	14	1	9	3	2	11	5	11	6	6	13	12	8

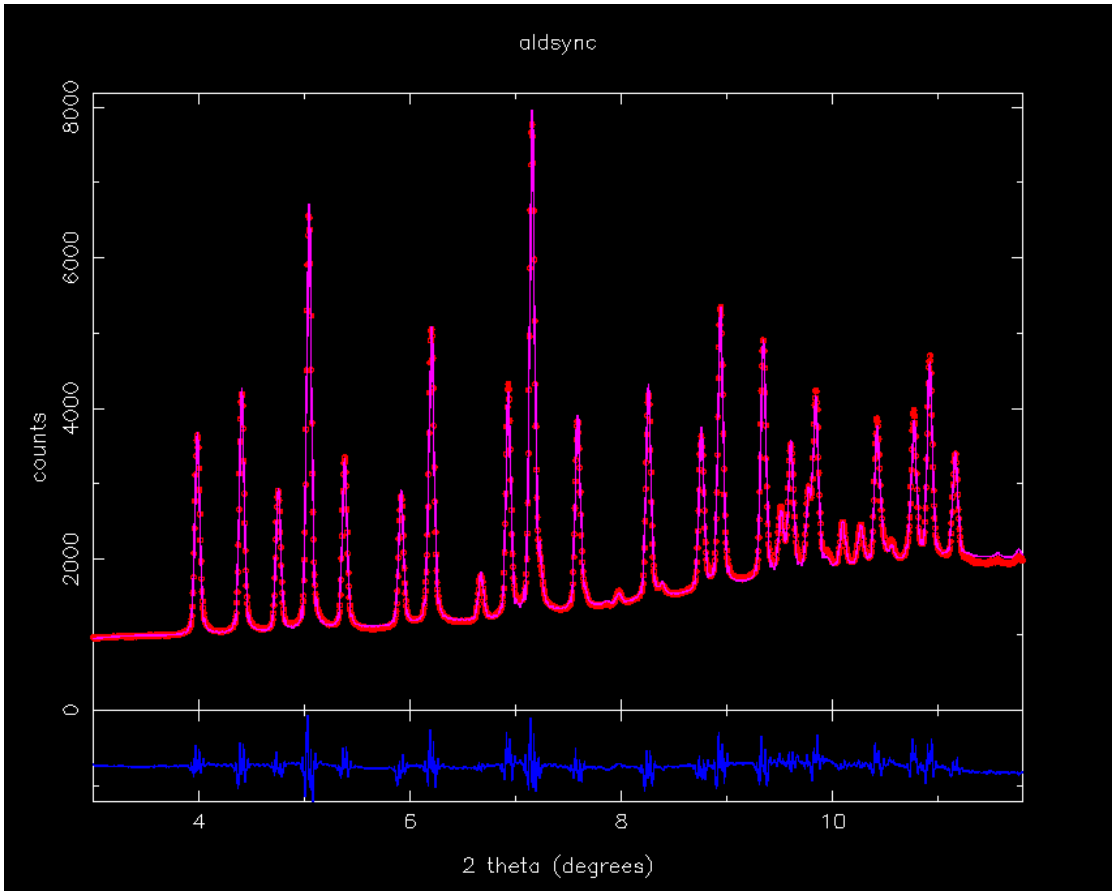
The 100 reflections are contained in 89 distinct clumps. The criterion for two reflections being too close together to be considered independent is approximately 0.1 FWHM. The kth correlation column gives the percentage correlation between the ith and (i+k)th clump. For example, the integrated intensities for clumps 87 and 88 are -76% correlated. Note that the correlations between neigbouring clumps are nearly always negative. This implies that there is less error in the sum of these two clump intensities than in the individual intensities themselves.

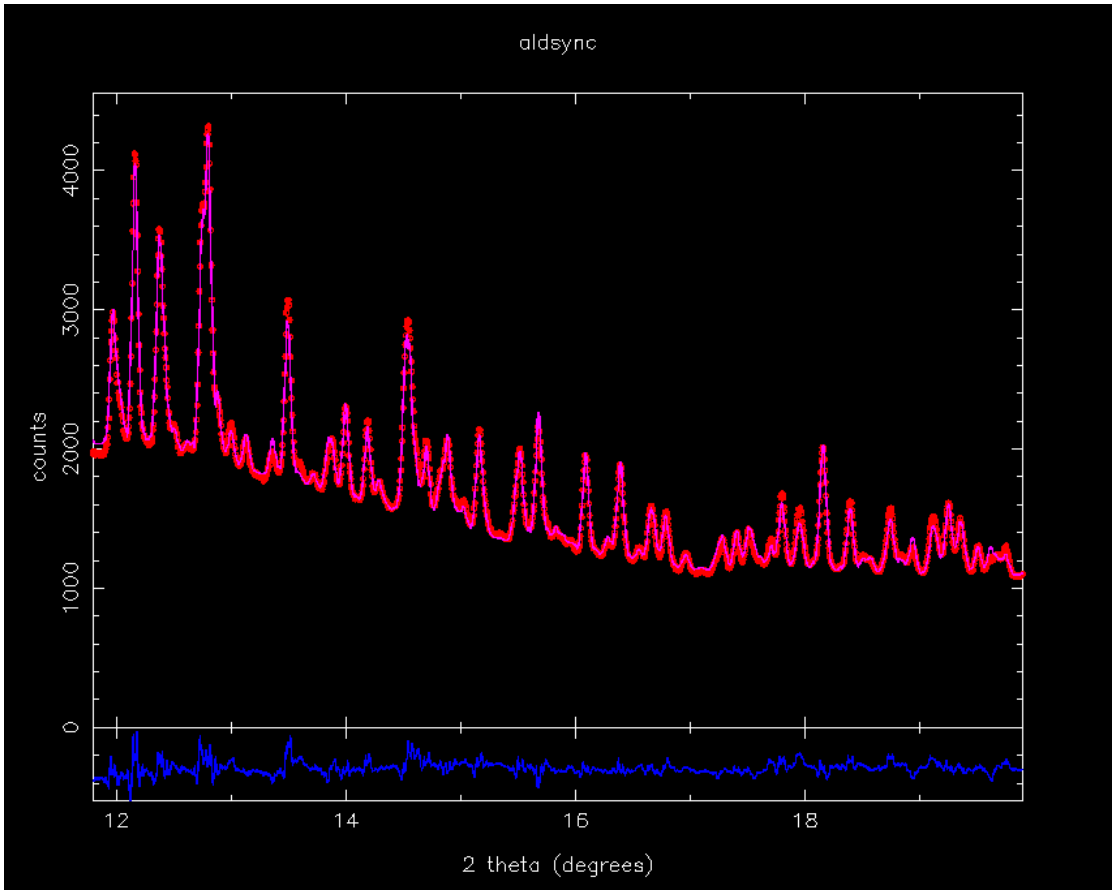
DRUID:

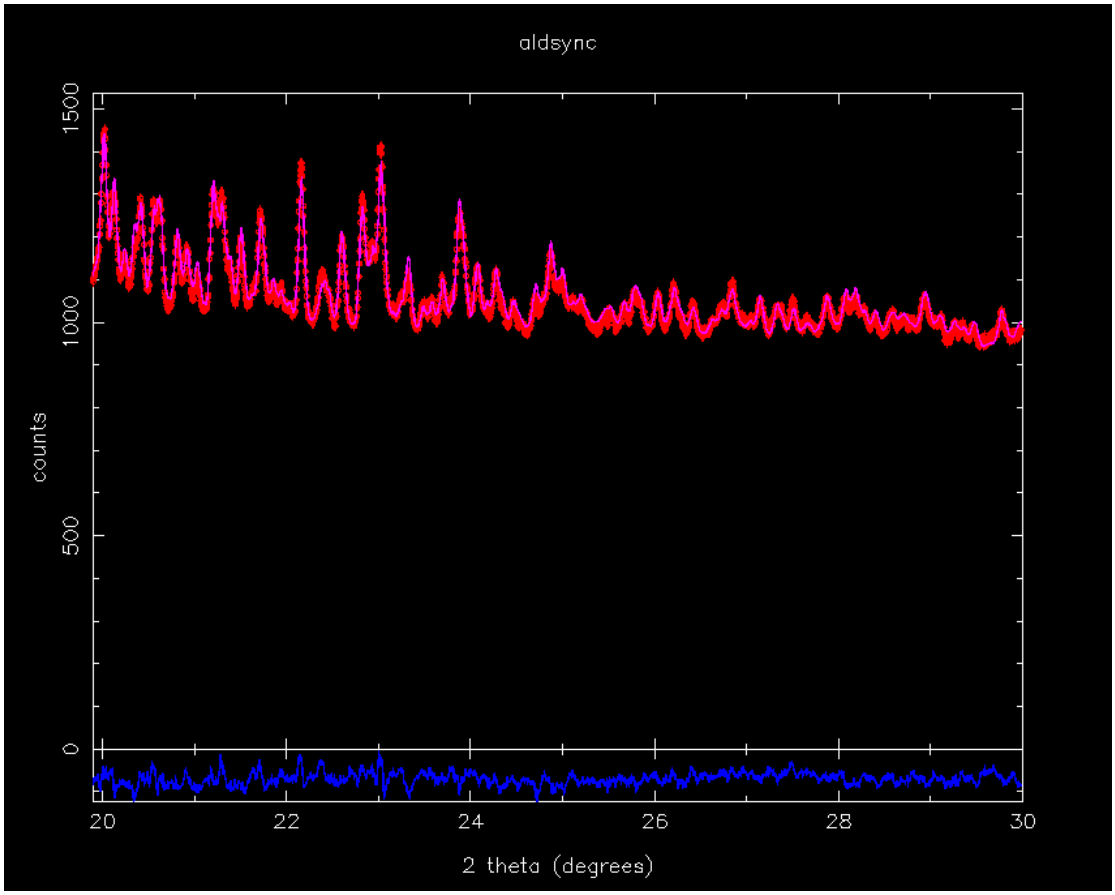
Global optimisation was performed using a simulated annealing technique on a DEC-Alpha 433Mhz Personal Workstation. Several structure solutions were performed and solution times varied from 26 seconds (see below) to 600 seconds. Logged output from the fastest structure solution process is given below. Note that there are 12 variables - the first three correspond to the centre of the molecule, the next four are quaternions that define the orientation of the molecule within the unit cell (the four quaternions represent three independent variables). The next two variables (#8 and #9) are the two undetermined internal molecular torsion angles. The final three coordinates are the fractional coordinates for the chlorine ion. The initial cost function (chi-squared) is very high at 6951 reflecting the fact that the initial coordinates are wrong. The initial temperature was set at 200 (in chi-squared units) and 8400 moves were performed at each temperature. At the end of the first temperature iteration, 58 new minima had been found and the chi-squared had reduced to 779.3. A total of four temperatures were sampled before a simplex semi-global optimisation was invoked - this reduced the chi-squared rapidly in 8 cycles from 465 to 285. Various approximate CPU timings are given in the log file. The CPU time taken for 3 cycles of (3*8400) trial pattern fits is (18.04-0.53) = 17.51 seconds corresponding to around 1440 trial structures generated and tested against the diffraction pattern per second.

The final profile refinement is illustrated in the figures below.


aldsync
counts
0
2000
4000
6000
8000
5
10
15
20
25
30
2 theta (degrees)


aldsync
counts
0
2000
4000
6000
8000
4
6
8
10
2 theta (degrees)


aldsync
counts
0
1000
2000
3000
4000
12
14
16
18
2 theta (degrees)


aldsync
counts
0
500
1000
1500
20
22
24
26
28
30
2 theta (degrees)

```
          >> PUNCH Alpha VMS V7.1-1H1  on Cluster Node JARAK <<
Computer support: mobile 0370 858090 (3029 internal), office number (x5414)
Username: wifd
Password:
   Welcome to OpenVMS (TM) Alpha Operating System, Version V7.1-1H1 on node JARAK
    Last interactive login on Wednesday,  3-JUN-1998 08:58:31.76
    Last non-interactive login on Tuesday,  2-JUN-1998 10:52:31.23

 Process: [7m08:59:59.78WIFD[0m  on Wednesday  3-JUN-1998 at 08:59:59.78

$  run [WIFD.SA.ALD.QUICKER]sa5torx
Give SA input file: alds.inp
 The information file contains:
 151 contributing terms from   100 reflections
 Fragment file number  1 is ALDS.zmatrix
 Fragment file number  2 is CL.zmatrix
 Initial parameter listing:
   #    Initial value      Lower and upper bounds    Initial step
   1        0.42337         -0.50000     0.50000       0.05000
   2       -0.03288         -0.25000     0.25000       0.02500
   3        0.47298         -0.50000     0.50000       0.05000
   4       -0.43520         -1.00000     1.00000       1.00000
   5        0.25293         -1.00000     1.00000       1.00000
   6       -0.86372         -1.00000     1.00000       1.00000
   7        0.16801          0.00000     1.00000       1.00000
   8      331.19318          0.00000   360.00000      12.00000
   9      339.95935          0.00000   360.00000      12.00000
  10        0.05451          0.00000     1.00000       0.05000
  11        0.48755          0.00000     1.00000       0.02500
  12        0.00683          0.00000     1.00000       0.05000
 Initial cost function value:      6951.37012
JARAK::08:59:59.78WIFD 09:00:21 SA5TORX   CPU=00:00:00.53 PF=713 IO=364 MEM=221

 Intermediate results before next temperature reduction
 Current temperature:          200.000
 Minimum cost function so far:     779.290 <<<<
 Average for this temperature:    4173.170
  +/- standard deviation     :    1452.917
    Total moves:                     8400
       downhill:                     1723
       accepted uphill:              1733
       rejected uphill:              4944
    Out of bounds trials:            1307
    New minima this temperature:       58
 Fragment number  1 contains  56 atoms
  and is positioned at    0.10068  -0.03582   0.31012
  with orientation 4-vector    0.33306   0.77287  -0.54013   0.00147
  torsion number 28 is    177.221
  torsion number 36 is     63.463
 Fragment number  2 is a single atom
  and is positioned at    0.57970   0.92507   0.52216
 Current parameter listing:
   #    Current value      Average +/- sigma      Weighted av +/- sigma       Lower and upper bounds    Current step
   1        0.10068       0.12944     0.03808       0.11531     0.00000         -0.50000     0.50000       0.01000
   2       -0.03582      -0.01681     0.04379      -0.05691     0.06047         -0.25000     0.25000       0.00625
   3        0.31012      -0.01478     0.37334       0.27111     0.42904         -0.50000     0.50000       0.01000
   4        0.33306       0.25258     0.13649      -0.04620     0.28650         -1.00000     1.00000       0.02000
   5        0.77287       0.71563     0.09577       0.63473     0.00000         -1.00000     1.00000       0.02000
   6       -0.54013      -0.62427     0.12070      -0.71134     0.35185         -1.00000     1.00000       0.02000
   7        0.00147       0.05626     0.04745       0.11534     0.11256          0.00000     1.00000       0.01000
   8      177.22150     184.76863   108.30930      67.67538     0.00000          0.00000   360.00000     360.00000
   9       63.46288     177.42136    96.16237     176.75487   122.34173          0.00000   360.00000      36.00000
  10        0.57970       0.31865     0.23775       0.18024     0.00000          0.00000     1.00000       0.01000
  11        0.92507       0.58915     0.29754       0.16988     0.00000          0.00000     1.00000       0.01000
  12        0.52216       0.30451     0.15788       0.31876     0.17284          0.00000     1.00000       0.01000
JARAK::08:59:59.78WIFD 09:00:28 SA5TORX   CPU=00:00:06.99 PF=795 IO=425 MEM=303

 Intermediate results before next temperature reduction
 Current temperature:          199.817
 Minimum cost function so far:     628.091 <<<<
 Average for this temperature:    2774.232
  +/- standard deviation     :    1307.026
    Total moves:                     8400
       downhill:                     1558
       accepted uphill:              1559
       rejected uphill:              5283
    Out of bounds trials:            1320
    New minima this temperature:        2
 Fragment number  1 contains  56 atoms
  and is positioned at    0.10243  -0.02899   0.31001
  with orientation 4-vector    0.34431   0.75552  -0.55730   0.00724
  torsion number 28 is    351.701
  torsion number 36 is     77.309
 Fragment number  2 is a single atom
  and is positioned at    0.59496   0.95883   0.50816
 Current parameter listing:
   #    Current value      Average +/- sigma      Weighted av +/- sigma       Lower and upper bounds    Current step
   1        0.10243       0.10828     0.02203       0.07499     0.00000         -0.50000     0.50000       0.01000
   2       -0.02899      -0.02061     0.01878       0.02393     0.01561         -0.25000     0.25000       0.00600
   3        0.31001       0.30796     0.00942       0.28428     0.00000         -0.50000     0.50000       0.01000
   4        0.34431       0.34838     0.02419       0.38749     0.17701         -1.00000     1.00000       0.02000
   5        0.75552       0.76488     0.02250       0.75275     0.00000         -1.00000     1.00000       0.02000
   6       -0.55730      -0.54368     0.03753      -0.64235     0.34782         -1.00000     1.00000       0.02000
   7        0.00724       0.01987     0.01395       0.03103     0.02597          0.00000     1.00000       0.01000
   8      351.70139     165.61230   109.85207     198.25246   154.42096          0.00000   360.00000     360.00000
   9       77.30882     167.54103    98.05720     222.43893   151.88013          0.00000   360.00000      98.43750
  10        0.59496       0.57477     0.03592       0.59185     0.14425          0.00000     1.00000       0.01875
  11        0.95883       0.92863     0.03403       0.97712     0.30743          0.00000     1.00000       0.01200
  12        0.50816       0.51510     0.02803       0.57421     0.25610          0.00000     1.00000       0.01000
JARAK::08:59:59.78WIFD 09:00:34 SA5TORX   CPU=00:00:12.45 PF=795 IO=468 MEM=303

 Intermediate results before next temperature reduction
 Current temperature:          199.613
 Minimum cost function so far:     628.091 <<<<
 Average for this temperature:    2698.727
  +/- standard deviation     :    1357.031
    Total moves:                     8400
       downhill:                     1513
       accepted uphill:              1512
       rejected uphill:              5375
    Out of bounds trials:            1258
    New minima this temperature:        0
 Fragment number  1 contains  56 atoms
  and is positioned at    0.10243  -0.02899   0.31001
  with orientation 4-vector    0.34431   0.75552  -0.55730   0.00724
  torsion number 28 is    351.701
```

```
  torsion number 36 is     77.309
 Fragment number  2 is a single atom
  and is positioned at    0.59496   0.95883   0.50816
 Current parameter listing:
   #    Current value      Average +/- sigma      Weighted av +/- sigma       Lower and upper bounds    Current step
   1        0.10243       0.10282     0.01858       0.11664     0.06010         -0.50000     0.50000       0.01000
   2       -0.02899      -0.01924     0.01888      -0.01599     0.02825         -0.25000     0.25000       0.00500
   3        0.31001       0.31128     0.00964       0.31340     0.03874         -0.50000     0.50000       0.01000
   4        0.34431       0.35028     0.02201       0.31990     0.00000         -1.00000     1.00000       0.02000
   5        0.75552       0.76607     0.02054       0.77073     0.08680         -1.00000     1.00000       0.02000
   6       -0.55730      -0.54132     0.03028      -0.51544     0.00000         -1.00000     1.00000       0.02000
   7        0.00724       0.01903     0.01228       0.02101     0.01159          0.00000     1.00000       0.01000
   8      351.70139     176.38170   112.97839      79.99482     0.00000          0.00000   360.00000     360.00000
   9       77.30882     154.44429    93.06140     211.67654   154.84767          0.00000   360.00000      37.33333
  10        0.59496       0.57365     0.03547       0.53405     0.00000          0.00000     1.00000       0.01500
  11        0.95883       0.92992     0.03441       0.83715     0.00000          0.00000     1.00000       0.01750
  12        0.50816       0.51218     0.02622       0.56572     0.24366          0.00000     1.00000       0.01000
JARAK::08:59:59.78WIFD 09:00:40 SA5TORX   CPU=00:00:18.04 PF=795 IO=511 MEM=303

 Intermediate results before next temperature reduction
 Current temperature:          199.418
 Minimum cost function so far:     464.826 <<<<
 Average for this temperature:    2769.419
  +/- standard deviation     :    1386.185
    Total moves:                     8400
       downhill:                     1503
       accepted uphill:              1483
       rejected uphill:              5414
    Out of bounds trials:            1139
    New minima this temperature:        2
 Fragment number  1 contains  56 atoms
  and is positioned at    0.09446  -0.02936   0.31309
  with orientation 4-vector    0.34612   0.75673  -0.55454   0.00757
  torsion number 28 is    350.558
  torsion number 36 is     44.649
 Fragment number  2 is a single atom
  and is positioned at    0.57067   0.93491   0.51184
 Current parameter listing:
   #    Current value      Average +/- sigma      Weighted av +/- sigma       Lower and upper bounds    Current step
   1        0.09446       0.10427     0.02128       0.15503     0.11493         -0.50000     0.50000       0.01000
   2       -0.02936      -0.02334     0.01615      -0.00872     0.00000         -0.25000     0.25000       0.00500
   3        0.31309       0.30645     0.01602       0.27733     0.00000         -0.50000     0.50000       0.01000
   4        0.34612       0.34214     0.03705       0.21755     0.00000         -1.00000     1.00000       0.02000
   5        0.75673       0.77037     0.02429       0.82281     0.28942         -1.00000     1.00000       0.02000
   6       -0.55454      -0.53738     0.03073      -0.50709     0.00000         -1.00000     1.00000       0.02000
   7        0.00757       0.01983     0.01767       0.07826     0.07770          0.00000     1.00000       0.01000
   8      350.55801     162.86594   102.03476     217.13586   144.79462          0.00000   360.00000      38.69992
   9       44.64865     156.15518    94.43134     286.86652   246.16415          0.00000   360.00000      26.33143
  10        0.57067       0.56877     0.03679       0.56054     0.00000          0.00000     1.00000       0.01458
  11        0.93491       0.92597     0.03325       0.94883     0.20821          0.00000     1.00000       0.01000
  12        0.51184       0.52461     0.04003       0.64593     0.37686          0.00000     1.00000       0.01000

Chi-squared =   4.6483E+02
 9.7302228E-02 -2.7880201E-02  0.3096238      0.3510732      0.7619110
-0.5501963     -3.5772775E-03   353.8829       56.78468      0.5810159
 0.9329863      0.5055604
Chi-squared =   2.8846E+02
 9.7601779E-02 -2.7789406E-02  0.3096109      0.3512039      0.7615417
-0.5504315     -4.1349619E-03   354.2409       57.66206      0.5812972
 0.9322408      0.5055087
Chi-squared =   2.8788E+02
 9.7566023E-02 -2.7887261E-02  0.3096344      0.3509044      0.7628151
-0.5514075     -3.4533280E-03   353.1144       56.15508      0.5798918
 0.9314442      0.5066160
Chi-squared =   2.8652E+02
 9.7411819E-02 -2.8129332E-02  0.3096532      0.3511983      0.7634436
-0.5521745     -3.8068504E-03   352.8967       55.75591      0.5790533
 0.9305474      0.5063740
Chi-squared =   2.8639E+02
 9.8012872E-02 -2.8001869E-02  0.3090901      0.3550573      0.7720915
-0.5582050     -4.6355729E-03   351.5725       56.98579      0.5799305
 0.9309301      0.5055078
Chi-squared =   2.8546E+02
JARAK::08:59:59.78WIFD 09:00:47 SA5TORX   CPU=00:00:23.97 PF=795 IO=567 MEM=303
JARAK::08:59:59.78WIFD 09:00:47 SA5TORX   CPU=00:00:23.97 PF=795 IO=568 MEM=303
JARAK::08:59:59.78WIFD 09:00:47 SA5TORX   CPU=00:00:23.97 PF=795 IO=569 MEM=303
 9.7617500E-02 -2.8136203E-02  0.3092462      0.3532997      0.7691700
-0.5563671     -4.5130588E-03   352.8362       56.45619      0.5794866
 0.9301219      0.5057124
Chi-squared =   2.8525E+02
 9.8015197E-02 -2.8180838E-02  0.3091633      0.3539108      0.7702206
-0.5576432     -4.4868719E-03   352.8619       56.36272      0.5794346
 0.9307655      0.5057389
Chi-squared =   2.8504E+02
 9.7968042E-02 -2.8415114E-02  0.3091053      0.3547501      0.7713645
-0.5580869     -4.5659821E-03   352.9192       56.76941      0.5789888
 0.9305471      0.5055303
Chi-squared =   2.8501E+02
JARAK::08:59:59.78WIFD 09:00:49 SA5TORX   CPU=00:00:26.19 PF=795 IO=591 MEM=303
$ log
  WIFD         logged out at  3-JUN-1998 09:00:52.10
```