Supporting information

The ethylacetate, molecule of solvation.

Nomenclature and refinement details:

Described in the asymmetric unit the overlapping atoms are the two closest to the inversion centre (O99 in the atoms list, which really consists of an oxygen atom from one molecule and a CH_2 group from an inverted molecule, and C99, consisting of a trigonal carbon atom from one molecule and a methyl group of an inverted molecule). O99 and C99 will thus in the mean description be best described to have total occupancy 1.0 since these atom positions are always filled no matter which orientation the ethylacetate molecule takes in the void, while the two positions further away (O98 and C98) have occupancy 0.5 since they are only occupied in half of the voids.

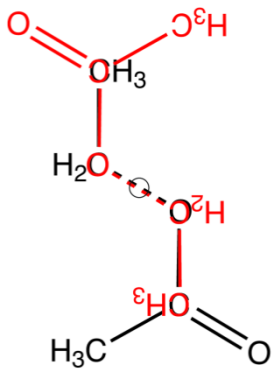

Figure S1 Ethylacetate disorder around a centre of symmetry.

Individual isotropic thermal parameters refine to pretty much identical, moderate to substantial Ueq values for the presumed ethylacetate atomic positions. F.inst. for Cr_7Co (100 K): 0.143(2) $Å^2$ (O99), 0.169(4) $Å^2$ (C99), 0.166(5) $Å^2$ (O98), 0.135(6) $Å^2$ (C98), and for Cr_7Zn (100 K): 0.186(4) $Å^2$ (O99), 0.207(8) $Å^2$ (C99), 0.226(11) $Å^2$ (O98), 0.173(11) $Å^2$ (C98). All other complexes have Ueq values, which hardly deviate significantly from those of the Cr_7Co complex.

It is difficult to calculate the expected hydrogen positions of the ethylacetate molecule because of the disorder. The ethylacetate group should accommodate a total of 8 hydrogen atoms. Only C98, which is considered to be a methyl group was supplied with calculated hydrogen positions in the atoms list. However, because of the half occupancy of this atom they only account for 1.5 hydrogens, so 6.5 hydrogen atoms are missing in the total count of atoms. O99 should really be half oxygen and half carbon. In the total atom count there is therefore half an oxygen too many and half a carbon too few in the way the disordered ethylacetate molecule is described.

The bridging pivalate groups

Nomenclature:

The 8 metal atoms are pairwise connected by one fluorine atom and two bridging pivalate groups. The planes of 8 of the 16 pivalate groups approximately coincide with the general plane of the metal hetero atoms - as well as that of the fluorine atoms. They are termed axial groups, and their corresponding carboxylate α-carbon atoms are numbered C10, C20,---- C80.

Of the remaining 8 pivalate groups 4 extend out from each side of the ring, every second group is termed up, with their α-carbon atoms numbered C15, C35, C55 and C75, and the other 4 are termed down with their α-carbon atoms numbered C25, C45, C65 and C85.

Half the pivalate groups appear to have substantially disordered tert-butyl groups at 100 K. Two on each side of the ring - namely those attached to C15 and C35 for the molecular up side and C45 and C85 for the down side - and four of the axially positioned pivalate groups with α -carbon atoms C10, C40, C50 and C70.

The disorder was described as two approximately staggered orientations of the methyl groups, as located in difference Fourier maps and subsequently refined, except for one which was modelled as a “full” torus consisting of 4 times 3 methyl groups for the tert-butyl group connected to the C50 α-carbon atom.

Definitions of free variables in the refinements

Below is shown the occupation factor for the major component of the individual disordered groups, namely FVAR 11-26, except FVAR 19, which is the Cα-Cβ distance in the rotor, C50, which is modeled with 4 *tert*-butyl groups 52, 52A, 52B and 52C).

FVAR definitions. - Constrained Parameters

1	Overall Scale Factor	OSF
2	Average pivalic Cα-Cβ distance	FVAR 2
3	Cr occupancy at wheel position 1	FVAR 3
4	Cr occupancy at wheel position 2	FVAR 4
5	Cr occupancy at wheel position 3	FVAR 5
6	Cr occupancy at wheel position 4	FVAR 6
7	Cr occupancy at wheel position 5	FVAR 7
8	Cr occupancy at wheel position 6	FVAR 8
9	Cr occupancy at wheel position 7	FVAR 9
10	Cr occupancy at wheel position 8	FVAR 10
11	C11, tert-butyl occupancy disorder	FVAR 11
12	C16, tert-butyl occupancy disorder	FVAR 12
13	C21, tert-butyl occupancy disorder	FVAR 13
14	C26, tert-butyl occupancy disorder	FVAR 14
15	C31, tert-butyl occupancy disorder	FVAR 15
16	C36, tert-butyl occupancy disorder	FVAR 16
17	C41, tert-butyl occupancy disorder	FVAR 17
18	C46, tert-butyl occupancy disorder	FVAR 18
19	Cα-Cβ distance in C51 "rotor",	FVAR 19
20	C56, tert-butyl occupancy disorder	FVAR 20
21	C61, tert-butyl occupancy disorder	FVAR 21
22	C66, tert-butyl occupancy disorder	FVAR 22
23	C71, tert-butyl occupancy disorder	FVAR 23
24	C76, tert-butyl occupancy disorder	FVAR 24
25	C81, tert-butyl occupancy disorder	FVAR 25
26	C86, tert-butyl occupancy disorder	FVAR 26
27	Average C-C distance in diethylamin	FVAR 27
28	Average N-C distance in diethylamin	FVAR 28
29	Occupation of N1-diethylamin	FVAR 29
30	Occupation of N1A-diethylamin	FVAR 30
31	Occupation of N1B-diethylamin	FVAR 31
32	$<u^2>$ of diethylamin-N1	FVAR 32
33	$<u^2>$ of diethylamin-C1	FVAR 33
34	$<u^2>$ of diethylamin-C2	FVAR 34
35	$<u^2>$ of "rotor" C52	FVAR 35
36	Occupation of "rotor" C52	FVAR 36
37	Occupation of "rotor" C52A	FVAR 37
38	Occupation of "rotor" C52B	FVAR 38
39	Occupation of "rotor" C52C	FVAR 39

Cr7Zn 100K Apex

1	0.13560	0.00017	-0.001	OSF
2	1.52393	0.00117	0.000	FVAR 2
3	0.77746	0.00937	0.000	FVAR 3
4	0.91462	0.00923	0.000	FVAR 4
5	0.99964	9.99999	0.000	FVAR 5
6	0.99945	9.99999	0.000	FVAR 6
7	0.77759	0.00978	0.000	FVAR 7
8	0.72997	0.00987	0.000	FVAR 8
9	0.99841	9.99999	0.000	FVAR 9
10	0.87539	0.00949	0.000	FVAR 10
11	0.64993	0.00579	0.000	FVAR 11
12	0.80643	0.00936	0.000	FVAR 12
13	1.00000	9.99999	0.000	FVAR 13
14	1.00000	9.99999	0.000	FVAR 14
15	1.00000	9.99999	0.000	FVAR 15
16	0.75520	0.01096	0.000	FVAR 16
17	0.90162	0.01095	0.000	FVAR 17
18	0.88838	0.00981	0.000	FVAR 18
19	1.52397	0.00133	0.000	FVAR 19
20	1.00000	9.99999	0.000	FVAR 20
21	1.00000	9.99999	0.000	FVAR 21
22	1.00000	9.99999	0.000	FVAR 22
23	0.52062	0.00903	0.001	FVAR 23
24	1.00000	9.99999	0.000	FVAR 24
25	1.00000	9.99999	0.000	FVAR 25
26	0.80124	0.01250	0.000	FVAR 26
27	1.46788	0.00988	-0.003	FVAR 27
28	1.44023	0.00794	-0.002	FVAR 28
29	0.36781	0.00644	0.001	FVAR 29
30	0.21389	0.00878	0.003	FVAR 30
31	0.41789	0.00885	-0.003	FVAR 31
32	0.04921	0.00211	-0.001	FVAR 32
33	0.05015	0.00197	0.004	FVAR 33
34	0.05513	0.00187	-0.001	FVAR 34
35	0.07004	0.00232	0.000	FVAR 35
36	0.19010	0.00980	0.000	FVAR 36
37	0.35132	0.00994	0.000	FVAR 37
38	0.15019	0.00995	0.000	FVAR 38
39	0.30824	0.00980	0.000	FVAR 39

Cr7Zn 120K SMART

1	0.04778	0.00005	0.001	OSF
2	1.52247	0.00107	0.000	FVAR 2
3	0.77129	0.00826	0.000	FVAR 3
4	0.90587	0.00811	0.000	FVAR 4
5	0.99892	0.00102	0.000	FVAR 5
6	0.99888	0.00102	0.000	FVAR 6
7	0.81856	0.00843	0.000	FVAR 7
8	0.75397	0.00850	0.000	FVAR 8
9	0.99810	0.00102	0.000	FVAR 9
10	0.85417	0.00826	0.001	FVAR 10
11	0.67799	0.00540	0.000	FVAR 11
12	0.83554	0.00886	0.000	FVAR 12
13	1.00000	9.99999	0.000	FVAR 13
14	1.00000	9.99999	0.000	FVAR 14
15	1.00000	9.99999	0.000	FVAR 15
16	0.76968	0.01052	0.000	FVAR 16
17	0.90305	0.00953	0.000	FVAR 17
18	0.91344	0.00931	0.000	FVAR 18
19	1.52254	0.00125	0.000	FVAR 19
20	1.00000	9.99999	0.000	FVAR 20
21	1.00000	9.99999	0.000	FVAR 21
22	1.00000	9.99999	0.000	FVAR 22
23	0.54311	0.00793	0.000	FVAR 23
24	1.00000	9.99999	0.000	FVAR 24
25	1.00000	9.99999	0.000	FVAR 25
26	0.77164	0.01243	0.000	FVAR 26
27	1.46829	0.00847	0.000	FVAR 27
28	1.44568	0.00676	0.000	FVAR 28
29	0.41997	0.00566	0.000	FVAR 29
30	0.19609	0.00759	0.000	FVAR 30
31	0.38319	0.00757	0.000	FVAR 31
32	0.05412	0.00189	0.000	FVAR 32
33	0.05050	0.00171	0.000	FVAR 33
34	0.06285	0.00177	0.000	FVAR 34
35	0.06685	0.00188	0.000	FVAR 35
36	0.20332	0.00805	0.000	FVAR 36
37	0.29582	0.00804	0.000	FVAR 37
38	0.16828	0.00814	0.000	FVAR 38
39	0.33253	0.00815	0.000	FVAR 39

Cr7Zn ESRF

1	0.89622	0.00315	-0.003	OSF
2	1.50278	0.00327	-0.002	FVAR 2
3	0.77548	0.03577	-0.001	FVAR 3
4	0.92041	0.03701	0.007	FVAR 4
5	0.99998	9.99999	0.000	FVAR 5
6	1.00001	9.99999	0.000	FVAR 6
7	0.70760	0.04118	0.007	FVAR 7
8	0.74181	0.03869	-0.013	FVAR 8
9	0.99992	9.99999	0.000	FVAR 9
10	0.85753	0.03714	0.000	FVAR 10
11	0.59789	0.01470	0.002	FVAR 11
12	0.78216	0.02999	0.011	FVAR 12
13	1.00000	9.99999	0.000	FVAR 13
14	1.00000	9.99999	0.000	FVAR 14
15	1.00000	9.99999	0.000	FVAR 15
16	0.73055	0.02939	0.002	FVAR 16
17	0.87522	0.03350	0.001	FVAR 17
18	0.83014	0.03981	-0.011	FVAR 18
19	1.50278	0.00334	-0.002	FVAR 19
20	1.00000	9.99999	0.000	FVAR 20
21	1.00000	9.99999	0.000	FVAR 21
22	1.00000	9.99999	0.000	FVAR 22
23	0.53024	0.02058	0.011	FVAR 23
24	1.00000	9.99999	0.000	FVAR 24
25	1.00000	9.99999	0.000	FVAR 25
26	0.79060	0.03016	-0.006	FVAR 26
27	1.40333	0.01803	0.005	FVAR 27
28	1.36275	0.01493	0.006	FVAR 28
29	0.38051	0.02463	0.002	FVAR 29
30	0.36142	0.02493	0.000	FVAR 30
31	0.25832	0.02918	-0.002	FVAR 31
32	0.08684	0.01184	0.003	FVAR 32
33	0.07682	0.00662	-0.009	FVAR 33
34	0.09613	0.01090	-0.002	FVAR 34
35	0.13743	0.00980	-0.001	FVAR 35
36	0.19352	0.03410	0.000	FVAR 36
37	0.28299	0.03397	0.005	FVAR 37
38	0.10960	0.03452	-0.003	FVAR 38
39	0.41384	0.03466	-0.001	FVAR 39

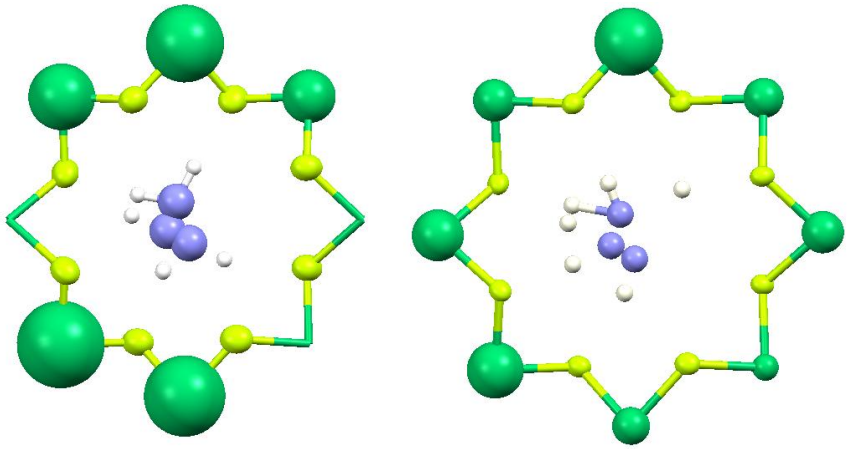

Figure S2. Occupation of Ni atoms in the Cr7Ni structure from (left) conventional X-ray data and (right) from ILL.

Cr7Ni

1	0.10367	0.00011	0.000	OSF
2	1.52204	0.00098	0.000	FVAR 2
3	0.79569	0.01231	0.000	FVAR 3
4	0.90383	0.01217	0.001	FVAR 4
5	0.99963	9.99999	0.000	FVAR 5
6	0.99940	9.99999	0.000	FVAR 6
7	0.77665	0.01253	0.000	FVAR 7
8	0.74076	0.01249	0.000	FVAR 8
9	0.99903	9.99999	0.000	FVAR 9
10	0.84969	0.01244	0.000	FVAR 10
11	0.74569	0.00528	0.000	FVAR 11
12	0.76538	0.00853	0.000	FVAR 12
13	1.00000	9.99999	0.000	FVAR 13
14	1.00000	9.99999	0.000	FVAR 14
15	1.00000	9.99999	0.000	FVAR 15
16	0.75265	0.01061	0.000	FVAR 16
17	0.96104	0.00847	0.000	FVAR 17
18	0.93713	0.00780	0.000	FVAR 18
19	1.52211	0.00116	0.000	FVAR 19
20	1.00000	9.99999	0.000	FVAR 20
21	1.00000	9.99999	0.000	FVAR 21
22	1.00000	9.99999	0.000	FVAR 22
23	0.33815	0.00800	0.000	FVAR 23
24	1.00000	9.99999	0.000	FVAR 24
25	1.00000	9.99999	0.000	FVAR 25
26	0.83223	0.00951	0.000	FVAR 26
27	1.46260	0.00850	0.000	FVAR 27
28	1.44542	0.00668	0.000	FVAR 28
29	0.37632	0.00588	0.000	FVAR 29
30	0.20561	0.00751	0.000	FVAR 30
31	0.41741	0.00766	0.000	FVAR 31
32	0.04510	0.00188	0.000	FVAR 32
33	0.04476	0.00178	0.000	FVAR 33
34	0.05212	0.00170	0.000	FVAR 34
35	0.05575	0.00169	0.000	FVAR 35
36	0.14021	0.00837	0.000	FVAR 36
37	0.50585	0.00863	0.000	FVAR 37
38	0.15512	0.00833	0.000	FVAR 38
39	0.19869	0.00757	0.000	FVAR 39

Cr7Ni ILL

1	1.91670	0.02368	0.065	OSF
2	1.52325	0.00052	0.000	FVAR 2
3	0.74009	0.07849	-0.029	FVAR 3
4	0.90510	0.07561	0.021	FVAR 4
5	0.90358	0.07853	-0.012	FVAR 5
6	0.96204	0.07691	-0.020	FVAR 6
7	0.91697	0.08147	0.011	FVAR 7
8	0.81600	0.07929	-0.017	FVAR 8
9	0.85465	0.07805	-0.013	FVAR 9
10	0.90241	0.07990	0.080	FVAR 10
11	0.67835	0.03707	-0.012	FVAR 11
12	0.86012	0.03837	0.072	FVAR 12
13	1.00000	9.99999	0.000	FVAR 13
14	1.00000	9.99999	0.000	FVAR 14
15	1.00000	9.99999	0.000	FVAR 15
16	0.78617	0.05259	0.117	FVAR 16
17	0.88697	0.03167	0.024	FVAR 17
18	0.83347	0.03751	-0.016	FVAR 18
19	1.52216	0.00129	0.000	FVAR 19
20	1.00000	9.99999	0.000	FVAR 20
21	1.00000	9.99999	0.000	FVAR 21
22	1.00000	9.99999	0.000	FVAR 22
23	0.50205	0.04435	0.120	FVAR 23
24	1.00000	9.99999	0.000	FVAR 24
25	1.00000	9.99999	0.000	FVAR 25
26	0.70636	0.04330	0.011	FVAR 26
27	1.46325	0.00237	0.000	FVAR 27
28	1.44590	0.00237	0.000	FVAR 28
29	0.42113	0.02879	-0.001	FVAR 29
30	0.24272	0.03203	-0.017	FVAR 30
31	0.33629	0.03482	0.009	FVAR 31
32	0.04098	0.01356	0.006	FVAR 32
33	0.04989	0.00925	0.005	FVAR 33
34	0.08405	0.02251	-0.015	FVAR 34
35	0.05171	0.01543	0.097	FVAR 35
36	0.24969	0.05807	-0.006	FVAR 36
37	0.30225	0.05506	-0.113	FVAR 37
38	0.19208	0.05014	0.053	FVAR 38
39	0.25585	0.05193	0.084	FVAR 39
40	1.09374	0.00868	0.049	FVAR 40
41	0.07297	0.00234	0.047	FVAR 41
42	0.10067	0.00375	0.017	FVAR 42

Cr7Co

1	0.05082	0.00006	0.000	OSF
2	1.52530	0.00094	0.000	FVAR 2
3	0.79839	0.01431	0.000	FVAR 3
4	0.91540	0.01406	0.000	FVAR 4
5	0.99969	9.99999	0.000	FVAR 5
6	0.99974	9.99999	0.000	FVAR 6
7	0.78083	0.01458	0.000	FVAR 7
8	0.68691	0.01464	0.000	FVAR 8
9	0.99936	9.99999	0.000	FVAR 9
10	0.87291	0.01439	0.000	FVAR 10
11	0.70044	0.00464	0.000	FVAR 11
12	0.83558	0.00769	0.000	FVAR 12
13	1.00000	9.99999	0.000	FVAR 13
14	1.00000	9.99999	0.000	FVAR 14
15	1.00000	9.99999	0.000	FVAR 15
16	0.76325	0.00974	0.000	FVAR 16
17	0.92054	0.00851	0.000	FVAR 17
18	0.93524	0.00782	0.000	FVAR 18
19	1.52545	0.00113	0.000	FVAR 19
20	1.00000	9.99999	0.000	FVAR 20
21	1.00000	9.99999	0.000	FVAR 21
22	1.00000	9.99999	0.000	FVAR 22
23	0.46627	0.00660	0.000	FVAR 23
24	1.00000	9.99999	0.000	FVAR 24
25	1.00000	9.99999	0.000	FVAR 25
26	0.79473	0.01064	0.001	FVAR 26
27	1.46478	0.00785	0.002	FVAR 27
28	1.43818	0.00632	0.001	FVAR 28
29	0.35765	0.00498	-0.002	FVAR 29
30	0.27309	0.01049	0.009	FVAR 30
31	0.36749	0.01048	-0.009	FVAR 31
32	0.04860	0.00161	-0.001	FVAR 32
33	0.05193	0.00125	-0.001	FVAR 33
34	0.06178	0.00162	0.001	FVAR 34
35	0.06499	0.00161	0.000	FVAR 35
36	0.19047	0.00689	0.000	FVAR 36
37	0.29458	0.00683	0.000	FVAR 37
38	0.17974	0.00690	0.000	FVAR 38
39	0.33533	0.00694	0.000	FVAR 39

Cr7Fe

1	0.11546	0.00014	0.000	OSF
2	1.52256	0.00109	0.000	FVAR 2
3	0.71188	0.02561	0.000	FVAR 3
4	0.88877	0.02500	0.000	FVAR 4
5	0.99994	9.99999	0.000	FVAR 5
6	0.99999	9.99999	0.000	FVAR 6
7	0.83001	0.02637	0.000	FVAR 7
8	0.67274	0.02660	0.000	FVAR 8
9	0.99990	9.99999	0.000	FVAR 9
10	0.91807	0.02561	0.000	FVAR 10
11	0.65752	0.00504	0.000	FVAR 11
12	0.85584	0.00861	0.000	FVAR 12
13	1.00000	9.99999	0.000	FVAR 13
14	1.00000	9.99999	0.000	FVAR 14
15	1.00000	9.99999	0.000	FVAR 15
16	0.78644	0.00952	0.000	FVAR 16
17	0.90174	0.00988	0.000	FVAR 17
18	0.92216	0.00963	0.000	FVAR 18
19	1.52264	0.00126	0.000	FVAR 19
20	1.00000	9.99999	0.000	FVAR 20
21	1.00000	9.99999	0.000	FVAR 21
22	1.00000	9.99999	0.000	FVAR 22
23	0.55557	0.00757	0.000	FVAR 23
24	1.00000	9.99999	0.000	FVAR 24
25	1.00000	9.99999	0.000	FVAR 25
26	0.81021	0.01217	0.000	FVAR 26
27	1.45352	0.00932	-0.002	FVAR 27
28	1.43536	0.00761	-0.002	FVAR 28
29	0.40138	0.00556	-0.001	FVAR 29
30	0.27393	0.01050	0.004	FVAR 30
31	0.32314	0.01052	-0.003	FVAR 31
32	0.05540	0.00200	0.000	FVAR 32
33	0.05553	0.00145	0.000	FVAR 33
34	0.06880	0.00202	0.000	FVAR 34
35	0.07746	0.00211	0.000	FVAR 35
36	0.20905	0.00855	0.000	FVAR 36
37	0.29266	0.00855	0.000	FVAR 37
38	0.15957	0.00866	0.000	FVAR 38
39	0.33922	0.00870	0.000	FVAR 39

Cr7Mn single M model

1	0.10360	0.00014	0.000	OSF
2	1.51827	0.00132	0.000	FVAR 2
3	0.53982	0.07135	0.000	FVAR 3
4	0.83088	0.06919	0.000	FVAR 4
5	1.00003	9.99999	0.000	FVAR 5
6	1.00000	9.99999	0.000	FVAR 6
7	0.96689	0.07345	0.000	FVAR 7
8	0.66794	0.07600	0.000	FVAR 8
9	1.00000	9.99999	0.000	FVAR 9
10	0.99999	9.99999	0.000	FVAR 10
11	0.63725	0.00626	0.000	FVAR 11
12	0.85431	0.01080	0.000	FVAR 12
13	1.00000	9.99999	0.000	FVAR 13
14	1.00000	9.99999	0.000	FVAR 14
15	1.00000	9.99999	0.000	FVAR 15
16	0.76922	0.01051	0.000	FVAR 16
17	0.88841	0.01284	0.000	FVAR 17
18	0.86786	0.01148	0.000	FVAR 18
19	1.51831	0.00147	0.000	FVAR 19
20	1.00000	9.99999	0.000	FVAR 20
21	1.00000	9.99999	0.000	FVAR 21
22	1.00000	9.99999	0.000	FVAR 22
23	0.54935	0.00981	0.000	FVAR 23
24	1.00000	9.99999	0.000	FVAR 24
25	1.00000	9.99999	0.000	FVAR 25
26	0.78800	0.01454	0.000	FVAR 26
27	1.43516	0.01145	-0.002	FVAR 27
28	1.41116	0.00951	-0.002	FVAR 28
29	0.40806	0.00621	0.000	FVAR 29
30	0.21849	0.01002	0.001	FVAR 30
31	0.37282	0.01000	-0.001	FVAR 31
32	0.05047	0.00222	0.000	FVAR 32
33	0.05315	0.00162	0.001	FVAR 33
34	0.05879	0.00208	0.000	FVAR 34
35	0.08830	0.00301	0.000	FVAR 35
36	0.21694	0.01195	0.000	FVAR 36
37	0.30190	0.01206	0.000	FVAR 37
38	0.14801	0.01217	0.000	FVAR 38
39	0.33330	0.01221	0.000	FVAR 39

Cr7Cd singleMmodel

1	0.10379	0.00016	0.000	OSF
2	1.52125	0.00172	0.000	FVAR 2
3	0.45372	0.00480	0.000	FVAR 3
4	0.99529	0.00452	0.000	FVAR 4
5	0.98523	0.00471	0.000	FVAR 5
6	0.98693	0.00466	0.000	FVAR 6
7	0.81623	0.00503	0.000	FVAR 7
8	0.90007	0.00507	0.000	FVAR 8
9	0.96049	0.00484	0.000	FVAR 9
10	0.97497	0.00469	0.000	FVAR 10
11	0.70606	0.00870	0.000	FVAR 11
12	0.95036	0.01389	0.001	FVAR 12
13	0.70818	0.01235	0.000	FVAR 13
14	0.90383	0.01779	0.000	FVAR 14
15	0.86723	0.01449	0.000	FVAR 15
16	1.52128	0.00183	0.000	FVAR 16
17	0.55462	0.01127	0.000	FVAR 17
18	0.69307	0.02039	0.000	FVAR 18
19	1.43421	0.01105	0.000	FVAR 19
20	1.41719	0.00918	0.000	FVAR 20
21	0.63780	0.00829	0.000	FVAR 21
22	0.17733	0.01190	0.000	FVAR 22
23	0.18469	0.01194	0.000	FVAR 23
24	0.03695	0.00207	0.000	FVAR 24
25	0.04315	0.00208	0.000	FVAR 25
26	0.05308	0.00231	0.000	FVAR 26
27	0.08257	0.00390	0.000	FVAR 27
28	0.24210	0.01541	0.000	FVAR 28
29	0.36642	0.01560	0.000	FVAR 29
30	0.14236	0.01560	0.000	FVAR 30
31	0.24944	0.01534	0.000	FVAR 31

Cr7Cd doubleMmodel

1	0.10472	0.00015	0.000	OSF
2	1.52087	0.00159	0.000	FVAR 2
3	0.44671	0.00483	0.000	FVAR 3
4	0.99963	9.99999	0.000	FVAR 4
5	0.99951	9.99999	0.000	FVAR 5
6	0.99950	9.99999	0.000	FVAR 6
7	0.79601	0.00542	0.000	FVAR 7
8	0.88366	0.00484	0.000	FVAR 8
9	0.99836	9.99999	0.000	FVAR 9
10	0.97094	0.00272	0.000	FVAR 10
11	0.70101	0.00792	0.000	FVAR 11
12	0.94465	0.01233	-0.001	FVAR 12
13	1.00000	9.99999	0.000	FVAR 13
14	1.00000	9.99999	0.000	FVAR 14
15	1.00000	9.99999	0.000	FVAR 15
16	0.70104	0.01119	0.000	FVAR 16
17	0.89199	0.01651	0.000	FVAR 17
18	0.87237	0.01309	0.000	FVAR 18
19	1.52089	0.00171	0.000	FVAR 19
20	1.00000	9.99999	0.000	FVAR 20
21	1.00000	9.99999	0.000	FVAR 21
22	1.00000	9.99999	0.000	FVAR 22
23	0.54969	0.01039	0.000	FVAR 23
24	1.00000	9.99999	0.000	FVAR 24
25	1.00000	9.99999	0.000	FVAR 25
26	0.75065	0.01989	0.000	FVAR 26
27	1.42947	0.00989	0.000	FVAR 27
28	1.40892	0.00799	0.000	FVAR 28
29	0.63575	0.00739	0.000	FVAR 29
30	0.17166	0.01053	0.000	FVAR 30
31	0.19175	0.01052	0.000	FVAR 31
32	0.03734	0.00190	0.000	FVAR 32
33	0.04237	0.00199	0.000	FVAR 33
34	0.05344	0.00211	0.000	FVAR 34
35	0.08325	0.00356	0.000	FVAR 35
36	0.25031	0.01424	0.000	FVAR 36
37	0.36171	0.01452	0.000	FVAR 37
38	0.14686	0.01452	0.000	FVAR 38
39	0.24119	0.01420	0.000	FVAR 39

Neutron diffraction for Cr_7Ni at VIVALDI, ILL.

Experimental details and data treatment:

Diffraction data from a Cr_7Ni crystal was collected in 11 settings with an angular separation of 20° with respect to the incoming neutron beam, from -105° to -45° and from -15° to 105°. Every pattern was collected for 80 min except for the data at 85° and 105°, which were collected for 60 min.

The patterns were indexed using the program NEWLAUEGEN of the Daresbury Laboratory Laue Suite of program (Campbell, 1995; Campbell et al., 1998), and the reflections integrated using the local program ARGONNE_BOXES, which uses a two-dimensional version of the $\sigma(I)/I$ algorithm (Wilkinson et al., 1988).

The integrated reflections were normalized to a common incident wavelength, using a curve derived by comparing equivalent reflections and multiple observations, via the program LAUENORM (Campbell et al., 1986).

Reflections were observed with wavelengths between 0.8 and 5.2 Å, but only reflections collected with neutrons in the wavelength interval 0.86 to 3.2 Å were accepted for scaling, as reflections at longer wavelength had too few equivalents to be able to determine the normalization curve with confidence. In all, 81219 reflections were observed, of which 19655 were single well-resolved reflections with $I/\sigma(I) > 3$. A set of 8861 pseudo-monochromated reflections were selected for the modelling process with 1405 least squares parameters and 1089 restraints.

We chose to use a deuterated sample in order to lower the inelastic background and thereby improve the number of significant reflections. It was observed that lowering the data collection temperature did not decrease the magnitude of mean-square amplitudes linearly with decreasing temperature. This indicates that packing disorder may account for some of the magnitude of the mean-square amplitudes. Deuteration in this case will not be as beneficial as for well-ordered compounds. The crystal did not scatter to high scattering angles, also because of the thermal strain in the big sample crystal, and the widespread disorder in many tert-butyl groups even at low temperature all meant that the experiment disappointingly allowed only a relatively limited set of significant reflections to be collected.

References

Campbell, J. W. (1995). *J. Appl. Cryst.*, **28**, 228-236.

Campbell, J. W., Hao, Q., Harding, M. M., Nguti, N. D. & Wilkinson, C. (1998). *J. Appl. Cryst.* **31**, 23-31.

Campbell, J. W., Habash, J., Helliwell, J. R. & Moffat, K. (1986). *Inf. Q. Protein Crystallogr.* **18,** 23-31.

Wilkinson, C., Khamis, H. W., Stansfield, R. F. D. & McIntyre, G. J. (1988). *J. Appl. Cryst.* **21**, 471-478.