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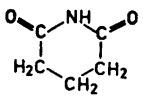
The Crystal Structure of Glutarimide

C. S. PETERSEN

Department of Chemistry, University of Oslo, Blindern, Oslo, Norway

The crystal structure of glutarimide has been determined by three-dimensional X-ray analysis. The refinement has been carried out separately on intensity data collected from Weissenberg diagrams and from an automatic four circle diffractometer. The crystals are monoclinic with space group $P2_1/c$ and unit cell parameters $a = 10.226$ Å, $b = 7.416$ Å, $c = 7.300$ Å, $\beta = 102.28°$. The phase problem was solved by direct methods and refined by full-matrix least squares to $R = 6.2$ %. The conformation of the molecule may be described as a half-chair. The atom C3 is out of the plane of the other ring atoms by 0.59 Å. In the crystal the molecules are hydrogen bonded such as to form chains running in the direction of the b-axis.

Glutarimide is an important part of many compounds displaying biological activity. One of the α-monosubstituted derivatives, thalidomide, is known for its teratogenic effects [1] and its central nervous system activity, the latter a property common to many of the substituted glutarimides.[2]



In the discussion of the stereochemistry of α- and β-substituted [3,4] glutarimides, very little is reported about the possible conformations of the glutarimide ring, which may be either a chair or a flattened form. This has, however, been considered for glutaric anhydrides,[5] a ring system comparable to glutarimide. Experimental data have led to the suggestion of a half-chair conformation for glutaric anhydrides.[6] In the crystal structure of *N*-(α-glutarimido)-4-bromophthalimide [7] the glutarimide ring was found to have a half-chair conformation.

A description of the structure of glutarimide itself is presented in this paper.

EXPERIMENTAL

Glutarimide crystals of suitable size were obtained by recrystallization from water. They were elongated along *b*, with (001) as the predominant face. Weissenberg photographs showed the crystals to be monoclinic, and the systematic absences lead to the space group $P2_1/c$.

The cell constants were determined by diffractometer measurements of ten moderately high-angle reflections, using Cu$K\alpha$ and Cu$K\beta$ radiation. The unit cell dimensions are $a = 10.226(2)$ Å, $b = 7.416(2)$ Å, $c = 7.300(2)$ Å, $\beta = 102.280(9)°$. The standard deviations given in parentheses refer to the last figure.

Integrated equi-inclination Weissenberg diagrams *h*0*l*···*h*6*l* and *hk*0 were taken, using Ni-filtered Cu$K\alpha$ radiation. In all, 937 independent reflections were strong enough to be measured, while 137 were given 1/4 I_{min}-values. Most of the intensities were measured on a Hilger and Watts photometer, but the weaker ones were estimated visually. The different layers were put on the same scale by means of common reflections, obtained by rotation about the *b* and *c* axes. No corrections have been made for absorption or secondary extinction effects.

When a Picker four-circle automatic diffractometer became available, a new set of intensity data was collected with Mo$K\alpha$ radiation. A crystal of size 0.25 × 0.28 × 0.25 mm was used. Reflections out to $2\theta = 65°$ were measured by $\theta/2\theta$ scan. Of the 1678 reflections recorded, 904 were unobserved, using a cut-off limit of 2.5δ. The data were corrected for secondary extinction.

The atomic form factors used were those of Hanson *et al.*[8] All programs applied are written or revised for CDC 3300 by T. Dahl, F. Gram, P. Groth, B. Klewe and C. Rømming.[9]

DETERMINATION OF THE STRUCTURE

The intensities were statistically adjusted to an absolute scale, and a preliminary temperature factor, $B = 1.7$ Å^2, was used in the calculation of unitary structure factors. The distribution of $|U|$ values was favourable, as 26 reflections had $|U| > 0.40$ and six $|U| > 0.50$. Three origin-fixing signs and two signs for structure invariants gave 13 more signs by Harker-Kasper inequalities. These 18 signs were used in a sign-determination program, and 158 additional signs were determined. The corresponding three dimensional Fourier map had eight peaks in the asymmetric unit. Two of these were higher than the rest and were considered to correspond to the oxygen atoms.

REFINEMENT

The coordinates obtained from the Fourier map and the preliminary scale and temperature factor were used as starting parameters in a full-matrix least squares refinement based on the film data. The weighting scheme

$$W = \mathrm{A1}(F_o)\mathrm{B1} \text{ for } F_o/ \leq /\mathrm{FB}$$
$$W = \mathrm{A2}(F_o)\mathrm{B2} \text{ for } F_o/ > /\mathrm{FB}$$

was used, with A1 = 10.0, A2 = 31.65, B1 = −0.5, and FB = 10.0. After three cycles of isotropic refinement, anisotropic thermal parameters were introduced. A three-dimensional difference Fourier map then yielded the position of the *N*-hydrogen atom and, less clearly, of four of the others. The coordinates of the remaining two hydrogens were calculated assuming tetrahedral C−H

bonds of length 1.03 Å. Additional refinements including positional parameters and isotropic temperature factors for the hydrogen atoms were then carried out. In the last two cycles of refinement, the 11 strongest reflections, probably suffering from extinction, were excluded, while the 137 non-observed were included. These were given constant weight $W = A1/3$ ($W = 1/\sigma$). The final R-factor was 8.2 %.

The parameters from the final refinement above were used in a further refinement, based on the diffractometer data. Only the 774 observed reflections were included in the calculations. The final R-value was 6.2 % (R_w = 5.2 %). A check of the 176 signs originally determined showed no discrepancies.

The fractional atomic coordinates and anisotropic thermal parameters are given in Table 1, and observed and calculated structure factors in Table 2.

Table 1. Positional and thermal parameters ($B_{ij} \times 10^5$) with estimated standard deviations.[a]

Atom	x	y	z	B_{11}	B_{22}	B_{33}	B_{12}	B_{13}	B_{23}
C1	0.11108	0.55051	0.21615	842	1245	1938	24	590	253
	28	35	37	32	59	68	71	73	109
C2	0.20165	0.39458	0.21087	1031	1198	3063	218	1031	159
	34	42	58	39	65	108	83	100	131
C3	0.30997	0.43151	0.10515	959	1553	4380	427	1418	−564
	33	47	65	40	75	132	90	113	160
C4	0.37944	0.60384	0.16333	802	1948	2852	155	1014	160
	30	43	58	34	78	89	81	95	138
C5	0.28745	0.76012	0.16371	733	1690	1584	−200	543	−268
	25	41	35	32	70	62	70	68	109
O1	−0.00187	0.53665	0.24409	854	1386	3933	−203	1915	139
	20	25	32	24	42	61	52	60	95
O2	0.31843	0.91548	0.14523	1052	1523	2883	−805	1195	29
	19	27	29	26	51	59	54	57	82
N1	0.16058	0.71933	0.18956	705	1054	2293	118	862	147
	22	31	30	25	45	60	57	62	83
H1	0.1060	0.8075	0.2017	3.9					
	27	39	37	0.7					
H2	0.1469	0.3005	0.1629	4.6					
	29	45	41	0.8					
H3	0.2473	0.3789	0.3409	8.4					
	38	56	58	1.2					
H4	0.2560	0.4619	−0.0381	12.8					
	50	68	72	1.5					
H5	0.3688	0.3317	0.1288	5.2					
	29	46	40	0.8					
H6	0.4254	0.5871	0.3016	5.9					
	31	40	46	0.9					
H7	0.4434	0.6467	0.0900	6.9					
	35	49	49	0.9					

[a] The B_{ij} are the thermal parameters in the expression: $\exp[-(B_{11}h^2+B_{22}k^2+B_{33}l^2+B_{12}hk+B_{13}hl+B_{23}kl)]$. For numbering of atoms, see Fig. 1.

Table 2. Observed and calculated structure factors. The columns are h, k, l, F_o and F_c.

1	0	0	24.73	22.11	-1	1	5	10.95	11.18	7	2	0	2.68	1.74	3	2	6	2.22	1.96
2	0	0	44.37	46.57	0	1	5	8.17	8.18	8	2	0	5.73	5.64	4	2	7	2.84	3.37
3	0	0	41.04	38.40	3	1	5	2.88	2.29	9	2	0	15.37	15.42	0	2	7	2.56	1.17
4	0	0	9.11	8.10	4	1	5	5.97	6.88	10	2	0	8.33	8.34	-5	2	7	2.38	1.33
6	0	0	30.85	29.72	5	1	5	3.22	1.33	11	2	1	3.87	2.98	-6	2	7	4.54	4.74
7	0	0	18.98	18.95	6	1	5	2.23	2.60	10	2	1	3.72	4.32	-7	2	8	3.68	4.25
9	0	0	3.29	2.85	7	1	5	2.26	1.85	9	2	1	2.11	1.82	-6	2	8	6.44	5.87
10	0	0	11.85	11.99	8	1	5	3.35	2.69	8	2	1	14.26	14.39	0	2	8	3.24	2.86
-12	0	2	3.48	4.54	9	1	4	4.33	4.34	7	2	1	2.66	2.81	1	2	8	3.37	3.88
-10	0	2	9.42	9.68	7	1	4	6.36	6.29	5	2	1	11.68	11.88	-1	3	8	3.80	3.71
-9	0	2	12.10	12.17	6	1	4	3.47	3.14	4	2	1	22.36	23.17	-5	3	8	3.97	4.23
-8	0	2	3.00	3.02	5	1	4	4.28	4.11	3	2	1	8.06	8.76	-6	3	8	4.86	3.25
-6	0	2	32.06	30.93	4	1	4	1.74	2.31	2	2	1	44.98	44.73	-5	3	7	3.73	3.05
-5	0	2	41.38	40.99	3	1	4	1.65	1.74	1	2	1	2.84	3.33	-1	3	7	2.66	2.17
-4	0	2	22.41	23.60	2	1	4	13.61	13.88	0	2	1	4.25	3.51	5	3	6	3.90	3.57
-3	0	2	4.94	4.23	1	1	4	8.32	8.24	-1	2	1	31.13	30.40	3	3	6	2.82	2.95
-2	0	2	28.23	25.27	0	1	4	21.15	21.15	-2	2	1	31.46	30.00	0	3	6	2.45	2.90
-1	0	2	45.93	44.67	-1	1	4	7.33	6.69	-3	2	1	18.80	17.72	-2	3	6	9.95	9.49
0	0	2	84.24	87.82	-2	1	4	1.50	1.81	-4	2	1	4.39	4.03	-4	3	6	2.64	1.61
1	0	2	110.52	113.43	-3	1	4	7.67	8.12	-5	2	1	11.43	11.52	-5	3	6	2.19	2.29
2	0	2	17.82	18.89	-4	1	4	11.58	11.00	-6	2	1	11.24	11.34	-6	3	6	8.73	8.91
3	0	2	3.43	2.91	-5	1	4	7.44	7.49	-7	2	1	15.81	15.85	-8	3	6	6.96	6.88
4	0	2	14.18	13.59	-6	1	4	5.66	5.40	-8	2	1	14.84	14.62	-9	3	6	2.65	2.82
5	0	2	8.21	7.64	-7	1	4	6.47	6.52	-9	2	1	8.41	8.56	-9	3	5	3.29	3.47
6	0	2	2.16	1.00	-9	1	4	4.75	5.17	-10	2	1	2.58	2.35	-8	3	5	3.56	3.52
7	0	2	14.08	14.30	-11	1	3	4.57	4.34	-11	2	1	4.56	4.85	-6	3	5	3.76	3.93
8	0	2	8.50	8.73	-10	1	3	3.53	3.33	-9	2	2	15.56	15.84	-5	3	5	5.19	5.48
9	0	2	2.43	2.39	-9	1	3	2.60	2.86	-8	2	2	18.83	19.23	-3	3	5	4.61	5.11
10	0	2	3.08	3.39	-8	1	3	3.48	3.19	-5	2	2	12.17	12.37	-2	3	5	5.03	5.20
11	0	2	6.82	6.31	-7	1	3	6.24	6.55	-4	2	2	2.58	2.80	-1	3	5	3.19	2.78
-12	0	4	3.69	3.21	-6	1	3	2.90	3.14	-3	2	2	15.21	14.26	2	3	5	2.21	1.79
-11	0	4	2.31	3.11	-5	1	3	9.10	9.43	-2	2	2	41.78	41.39	6	3	5	2.23	2.80
-9	0	4	9.86	9.22	-4	1	3	14.98	15.48	-1	2	2	19.43	19.38	9	3	4	3.92	3.48
-8	0	4	7.23	7.51	-3	1	3	8.99	8.35	0	2	2	11.68	11.14	8	3	4	2.37	1.71
-6	0	4	3.66	2.76	-2	1	3	19.15	19.59	1	2	2	13.63	13.43	6	3	4	3.87	3.18
-5	0	4	32.84	32.73	-1	1	3	18.18	18.17	2	2	2	11.83	10.68	5	3	4	4.92	4.88
-4	0	4	11.12	11.49	0	1	3	11.25	11.68	3	2	2	16.43	16.06	4	3	4	7.42	7.33
-3	0	4	16.68	17.11	1	1	3	1.80	1.44	4	2	2	13.96	14.54	3	3	4	8.63	8.41
-2	0	4	12.87	12.89	2	1	3	6.22	7.20	5	2	2	4.68	4.53	2	3	4	2.70	2.29
-1	0	4	6.18	5.97	3	1	3	11.04	10.87	6	2	2	1.82	1.83	1	3	4	2.17	1.82
0	0	4	5.69	6.14	4	1	3	3.58	4.04	9	2	2	6.19	6.03	0	3	4	2.83	2.10
1	0	4	50.16	51.64	5	1	3	8.96	8.63	10	2	2	8.02	7.80	-1	3	4	7.49	7.22
2	0	4	23.19	23.74	8	1	3	7.19	6.53	9	2	3	5.32	5.37	-2	3	4	13.05	12.57
3	0	4	10.57	10.47	10	1	2	2.73	2.07	8	2	3	2.83	3.80	-3	3	4	12.68	12.54
4	0	4	2.09	2.30	9	1	2	5.50	5.42	7	2	3	2.74	2.89	-4	3	4	4.07	3.59
6	0	4	2.99	3.62	8	1	2	4.20	4.67	6	2	3	5.84	6.18	-6	3	4	10.48	10.72
8	0	4	6.50	6.52	7	1	2	6.13	5.91	5	2	3	8.16	8.22	-7	3	4	7.60	7.79
9	0	4	3.38	2.66	6	1	2	11.03	11.16	4	2	3	8.81	8.21	-8	3	4	7.02	6.56
-10	0	6	3.87	3.72	4	1	2	9.38	9.73	3	2	3	20.85	21.51	-9	3	4	5.88	4.99
-9	0	6	7.13	7.18	2	1	2	14.97	14.91	2	2	3	7.87	7.42	-10	3	3	2.32	1.76
-8	0	6	3.31	3.96	1	1	2	22.63	22.94	1	2	3	3.02	3.71	-9	3	3	5.60	5.49
-7	0	6	2.87	2.84	0	1	2	37.26	37.86	0	2	3	15.30	15.40	-7	3	3	4.85	5.00
-5	0	6	6.35	6.80	-1	1	2	16.03	17.14	-1	2	3	13.07	13.36	-6	3	3	6.70	6.29
-4	0	6	14.53	14.51	-2	1	2	13.66	13.27	-2	2	3	2.35	2.99	-5	3	3	5.35	5.41
-3	0	6	3.54	4.35	-3	1	2	13.94	14.11	-3	2	3	1.34	2.16	-4	3	3	5.55	5.92
-2	0	6	4.51	3.45	-4	1	2	2.12	1.74	-4	2	3	8.70	8.88	-3	3	3	10.97	11.33
-1	0	6	3.01	3.28	-5	1	2	18.20	18.04	-5	2	3	6.93	7.24	-2	3	3	5.19	5.32
0	0	6	6.83	7.91	-6	1	2	4.66	4.43	-6	2	3	2.67	2.73	0	3	3	4.12	4.35
1	0	6	6.96	7.79	-7	1	2	8.01	8.48	-7	2	3	21.39	21.44	1	3	3	3.47	3.34
2	0	6	15.71	16.99	-8	1	2	7.52	7.36	-9	2	3	3.96	3.61	2	3	3	1.79	1.96
4	0	6	2.63	2.46	-9	1	2	4.16	4.96	-10	2	3	8.27	8.32	4	3	3	1.81	.78
7	0	6	2.62	2.75	-10	1	2	3.09	3.48	-11	2	4	2.67	2.89	5	3	3	5.96	5.94
-8	0	8	3.74	3.56	-11	1	1	5.82	5.45	-9	2	4	3.42	3.62	7	3	3	3.89	3.33
-4	0	8	4.41	5.28	-9	1	1	2.00	1.80	-8	2	4	17.83	18.88	9	3	3	2.70	2.87
-3	0	8	2.50	1.47	-7	1	1	8.96	9.79	-7	2	4	13.19	13.37	9	3	2	3.41	2.96
-2	0	8	3.49	3.60	-6	1	1	4.03	3.66	-6	2	4	4.16	3.92	8	3	2	8.16	8.20
2	0	8	3.72	5.54	-5	1	1	1.99	1.12	-5	2	4	3.63	3.90	6	3	2	1.83	1.78
3	0	8	3.21	3.60	-4	1	1	22.48	22.28	-4	2	4	5.89	5.67	4	3	2	18.88	18.69
2	1	8	3.45	3.09	-3	1	1	13.69	13.90	-3	2	4	4.51	4.53	2	3	2	18.63	19.34
1	1	8	3.44	3.39	-2	1	1	16.83	16.83	-2	2	4	11.42	11.65	1	3	2	2.87	2.39
0	1	8	3.51	2.99	-1	1	1	32.23	33.47	-1	2	4	20.57	20.83	-1	3	2	14.67	14.03
-3	1	8	3.10	2.91	0	1	1	19.55	21.02	0	2	4	8.11	8.56	-2	3	2	1.34	.48
-5	1	8	2.73	2.54	1	1	1	31.95	31.71	1	2	4	3.27	3.18	-3	3	2	27.45	26.83
-8	1	7	2.66	2.10	2	1	1	17.46	18.50	2	2	4	4.47	4.21	-4	3	2	1.61	1.98
-7	1	7	2.79	2.18	3	1	1	16.34	16.02	3	2	4	5.14	5.21	-6	3	2	6.40	6.36
-5	1	7	3.24	2.65	4	1	1	10.07	10.07	4	2	4	4.80	4.85	-7	3	2	15.73	16.05
-4	1	7	2.20	1.39	5	1	1	8.92	8.58	5	2	4	3.32	2.87	-9	3	2	6.24	6.14
-2	1	7	3.22	2.19	6	1	1	4.65	4.72	6	2	4	2.14	2.17	-10	3	1	3.89	3.70
-1	1	7	3.18	2.47	7	1	1	7.40	7.48	8	2	4	3.13	2.54	-8	3	1	4.76	5.01
0	1	7	4.30	5.48	8	1	1	4.41	4.16	5	2	5	7.83	7.16	-7	3	1	4.20	4.27
7	1	6	3.23	2.10	10	1	1	3.48	2.88	4	2	5	6.19	4.93	-6	3	1	7.58	7.76
5	1	6	2.11	1.49	11	1	1	3.85	3.23	3	2	5	6.61	7.13	-5	3	1	1.88	1.68
3	1	6	4.39	4.62	10	1	0	4.77	4.91	2	2	5	2.24	1.79	-4	3	1	17.20	16.23
1	1	6	11.07	10.73	9	1	0	2.53	1.62	1	2	5	5.51	4.96	-3	3	1	8.76	8.51
0	1	6	2.20	1.55	8	1	0	9.36	9.71	0	2	5	4.48	5.27	-2	3	1	1.72	1.86
-1	1	6	6.27	5.92	6	1	0	13.86	13.25	-3	2	5	2.49	3.25	-1	3	1	7.07	6.64
-2	1	6	4.54	4.45	5	1	0	13.84	14.21	-6	2	5	9.59	10.02	0	3	1	2.37	1.61
-4	1	6	6.98	6.73	4	1	0	8.06	8.09	-7	2	5	6.31	6.06	1	3	1	9.30	8.75
-5	1	6	1.91	2.05	3	1	0	11.40	11.71	-8	2	5	4.39	4.41	3	3	1	2.34	2.20
-6	1	6	4.66	4.34	2	1	0	5.99	5.73	-9	2	5	4.53	4.88	4	3	1	8.83	8.70
-8	1	6	2.95	3.35	1	1	0	47.17	42.40	-8	2	6	4.60	4.76	5	3	1	7.69	7.65
-11	1	5	2.34	1.96	0	2	0	30.45	28.78	-7	2	6	13.06	12.78	6	3	1	5.10	5.37
-10	1	5	6.29	5.88	1	2	0	12.81	12.82	-6	2	6	4.19	3.93	7	3	1	3.97	3.94
-7	1	5	2.82	2.39	2	2	0	25.23	24.41	-5	2	6	3.74	3.23	8	3	1	3.63	3.44
-6	1	5	6.64	6.50	3	2	0	39.30	37.26	-4	2	6	2.90	2.84	9	3	1	2.91	2.60
-5	1	5	2.21	2.21	4	2	0	10.47	10.05	-2	2	6	3.87	3.52	10	3	0	3.44	3.49
-3	1	5	8.96	8.71	5	2	0	3.39	3.51	-1	2	6	6.09	6.08	9	3	0	3.79	3.72
-2	1	5	2.31	1.53	6	2	0	5.25	5.28	0	2	6	8.67	8.21	8	3	0	9.25	9.26

Table 2. Continued.

7	3	0	10,93	11,07	1	5	5	3,43	3,04	-6	6	4	3,04	2,99	7	5	5	4·89	3·74
6	3	0	1,93	2,03	2	5	5	2,57	2,38	-5	6	4	6,38	6,43	-10	5	4	4·47	3·49
5	3	0	5,79	5,94	3	5	5	2,99	1,81	-3	6	4	6,81	6,91	-11	5	4	2·98	·37
4	3	0	12,01	12,01	7	5	4	5,02	4,24	-2	6	4	3,41	3,52	-12	5	3	4·07	3·76
3	3	0	28,40	27,37	5	5	4	2,49	2,37	-1	6	4	4,18	4,00	-11	5	3	4·13	3·04
2	3	0	20,85	20,85	3	5	4	2,81	3,66	0	6	4	4,98	5,25	-11	5	2	3·03	2·74
1	3	0	15,05	14,72	2	5	4	7,74	7,62	1	6	4	9,64	9,74	-11	5	1	6·73	6·32
0	4	0	1,88	2,69	1	5	4	2,87	3,00	2	6	4	3,18	2,34	10	5	1	2·92	3·09
1	4	0	22,54	21,56	0	5	4	11,77	11,71	3	6	4	6,81	7,10	11	5	1	3·77	3·25
2	4	0	8,02	8,20	-1	5	4	3,11	3,15	3	6	5	2,74	2,70	12	5	1	3·53	2·94
3	4	0	2,61	2,19	-2	5	4	2,17	1,28	2	6	5	5,65	5,55	10	6	0	4·13	3·30
4	4	0	8,22	8,32	-3	5	4	3,93	3·59	1	6	5	6,29	6,10	9	6	1	4·75	4·12
5	4	0	4,76	4,56	-4	5	4	3,15	3·13	0	6	5	4,81	4,38	10	6	3	3·21	2·31
6	4	0	4,10	3,89	-5	5	4	7,84	7·61	-1	6	5	3,45	3,17	-10	6	3	2·96	3·12
7	4	0	14,42	14,54	-6	5	4	2,38	2·77	-2	6	5	3,57	3,80	-11	6	3	4·17	4·00
8	4	0	3,42	3,37	-7	5	4	3·21	3·23	-3	6	5	3,08	2,50	-8	6	5	4·27	3·10
9	4	1	0,09	0,31	-8	5	4	4,65	4·27	-4	6	5	3,58	4,12	-9	6	5	4·47	2·46
8	4	1	10,94	10,75	-10	5	3	5,21	4·85	-5	6	5	4,58	4,72	4	6	6	3·73	2·13
7	4	1	4,99	5,28	-9	5	3	3,62	3·15	-4	6	6	3,08	3,23	3	6	7	2·99	2·09
6	4	1	3,18	2,70	-7	5	3	4·15	4·14	-3	6	6	3,24	3,27	-2	7	7	3·51	2·49
5	4	1	7,37	7,69	-6	5	3	4,30	4·12	0	6	6	3,30	3,53	0	7	6	3·13	2·96
4	4	1	3,93	4,18	-5	5	3	5,78	5·38	2	6	6	4,59	4,35	-1	7	6	4·14	3·73
3	4	1	11,55	11,56	-2	5	3	2·12	·52	-3	7	5	5,00	4,79	4	7	5	4·16	3·60
2	4	1	29,25	29,01	1	5	3	4,47	4·44	-1	7	5	4,73	4,12	-7	7	4	3·16	1·41
1	4	1	3,15	2,96	5	5	3	2·29	2·47	0	7	5	2,52	2,30	-10	7	3	3·47	2·25
-1	4	1	27,15	26,80	6	5	3	7·07	6·46	3	7	4	3,33	3,19	-10	7	2	3·14	2·20
-2	4	1	21,01	20,36	7	5	3	2·88	2·83	-1	7	4	8,38	8,27	9	7	0	3·05	1·11
-4	4	1	8,01	8,50	8	5	3	4,67	3·85	-3	7	4	3,57	3,32	8	7	0	4·38	3·32
-5	4	1	3,65	3,21	7	5	2	3,95	3·83	-4	7	3	5,77	5,55	9	8	0	3·78	2·87
-6	4	1	12,20	11,84	6	5	2	6,23	5·91	-3	7	3	4,45	4,52	-9	8	1	6·95	6·89
-7	4	1	11,04	11,16	4	5	2	2,09	1·62	-2	7	3	4,18	3,98	-8	8	2	4·55	2·98
-8	4	1	15,40	15,35	3	5	2	1·97	2·15	-1	7	3	5,47	5,15	5	8	3	3·86	3·58
-10	4	1	6,59	5·90	2	5	2	10·00	10·01	2	7	3	2,94	2,45	4	8	3	4·62	4·72
-11	4	1	4,65	4,70	1	5	2	7·16	7·25	3	7	3	6,39	6,10	-8	8	3	5·86	5·54
-11	4	2	3,51	3·61	0	5	2	9,96	9·94	5	7	3	2,85	2,88	-9	8	3	6·68	5·69
-7	4	2	11,21	11·49	-1	5	2	14·95	15·22	4	7	2	2,54	3,31	-8	8	4	4·95	3·87
-6	4	2	13,71	14·19	-4	5	2	2,08	1,44	3	7	2	5,27	5,66	-7	8	4	2·88	3·15
-5	4	2	5,08	5·49	-5	5	2	6,26	7,41	1	7	2	3,50	3,01	-6	8	5	2·96	2·54
-4	4	2	5,04	5,35	-6	5	2	5,34	5,50	0	7	2	4,34	4,38	-5	8	6	3·25	·58
-3	4	2	7,76	7·77	-7	5	2	3,74	4,24	-1	7	2	6,28	6,47	-8	8	6	3·61	1·84
-2	4	2	4,69	4,29	-9	5	2	4,00	3,97	-2	7	2	8,24	8,08	-6	9	3	3·85	3·34
-1	4	2	13,35	13·11	-9	5	1	3,76	3,44	-3	7	2	5,75	5,96	-5	9	3	4·14	2·92
0	4	2	7,09	7·06	-7	5	1	7,33	7,02	-8	7	2	3,59	2,80	-3	9	3	5·32	4·77
1	4	2	10,20	9·64	-5	5	1	8,68	8·35	-5	7	1	4,14	3,81	-2	9	3	5·51	3·27
2	4	2	15,78	16·29	-4	5	1	2,40	1·98	-4	7	1	5,77	6,10	-6	9	1	4·65	3·85
4	4	2	2,79	2·14	-2	5	1	3,39	3·49	-3	7	1	2,23	1,04	-4	9	1	6·48	5·71
5	4	2	3,63	3·54	1	5	1	3,02	3·29	-2	7	1	6,00	6,48	-3	9	1	3·93	3·42
7	4	2	7,81	7·74	2	5	1	4·38	3·95	-1	7	1	4,68	4,94	4	9	1	4·43	4·40
8	4	2	6·24	6·38	4	5	1	4·22	4·15	2	7	1	6,99	6,99	5	9	1	3·22	3·29
9	4	3	4,66	5·04	5	5	1	4,11	3·66	3	7	1	4,61	4,88	6	9	1	3·82	3·05
7	4	3	3,37	3·67	6	5	1	8·81	9·08	5	7	1	4,09	4,35	1	10	1	3·88	3·05
6	4	3	2·90	2·68	7	5	1	4,38	3·69	4	7	0	4,61	5,34	0	10	1	4,00	3·87
4	4	3	1·77	·88	8	5	1	2·98	3·22	2	7	0	10,16	10,62					
3	4	3	15·11	15·14	6	5	0	7,09	7·52	1	7	0	2,26	2,52					
2	4	3	5·93	5·85	5	5	0	2,99	3·50	0	8	0	2,55	2,69					
0	4	3	13·72	14·08	2	5	0	9,48	9·50	3	8	0	2,52	2,40					
-1	4	3	13·88	13·84	1	5	0	14·19	14·43	5	8	0	4,70	4,60					
-2	4	3	3·71	4·10	0	6	0	23·83	23·65	4	8	1	7,96	7,68					
-3	4	3	6·35	6·62	1	6	0	5·01	4·92	3	8	1	2,99	3,64					
-4	4	3	3·41	3·64	2	6	0	15·36	15·18	1	8	1	4,78	4,62					
-5	4	3	10·25	10·23	3	6	0	6,18	6,04	0	8	1	6,69	6,07					
-6	4	3	3·15	2·86	5	6	0	6,57	6,46	-3	8	1	7,77	7,76					
-7	4	3	18·77	18·52	6	6	0	3,84	4,69	-4	8	1	3·10	2·87					
-8	4	3	4·20	4·54	8	6	0	4,69	4,99	-4	8	2	4·34	4·19					
-9	4	3	4·38	3·93	7	6	1	3,03	3·34	-2	8	2	4·26	3·29					
-10	4	3	8·50	8·15	6	6	1	7,10	6,80	1	8	2	3·02	3·23					
-11	4	3	2·75	2·34	5	6	1	3,05	3,78	2	8	2	3,35	2·80					
-10	4	4	2·45	2·72	4	6	1	2,87	3,08	3	8	3	2,65	1·54					
-8	4	4	2·73	2·12	3	6	1	4,49	4,89	1	8	3	5,00	4·54					
-6	4	4	13·57	13·97	2	6	1	8,67	9,34	0	8	3	2·82	2·18					
-4	4	4	3·54	3·32	1	6	1	15,14	15·33	-2	8	3	4,51	4·74					
-3	4	4	8·54	9·03	0	6	1	14,77	15,19	-3	8	3	4·00	3·51					
0	4	4	5·24	5·32	-1	6	1	16,33	16,63	-1	8	4	3·78	3·14					
1	4	4	3·89	3·25	-2	6	1	2,22	1·80	-1	9	1	3·01	2·79					
2	4	4	8·51	8·78	-3	6	1	6,33	7·16	13	0	0	4·47	4·49					
3	4	4	2·14	3·32	-4	6	1	4,74	4·67	12	0	2	3·19	1·77					
6	4	4	4·45	3·75	-5	6	1	8,21	8·17	-15	0	4	3·73	2·08					
8	4	4	4·95	4·70	-6	6	1	6,78	7·07	11	0	4	3,47	2·84					
5	4	5	2,25	1·51	-8	6	1	3·41	3·36	9	0	6	4,02	3,04					
4	4	5	5,00	4,31	-9	6	1	4·43	3·62	-9	0	8	3,48	3,58					
3	4	5	3,68	3,70	-9	6	2	2·67	2·82	1	1	10	3,04	·32					
1	4	5	5,26	4,85	-8	6	2	3·41	3·16	-9	1	9	3,08	2,51					
0	4	5	5,91	6,07	-7	6	2	5·23	5·58	0	1	9	3,24	1,46					
-2	4	5	3,99	3,62	-6	6	2	4,80	4·73	-10	1	7	5,04	3,95					
-4	4	5	6,83	5,95	-5	6	2	3·04	4·10	8	1	6	2,88	2,95					
-5	4	5	2,82	2,55	-4	6	2	10·06	10·25	-12	1	6	3,10	2,39					
-6	4	5	10,57	10,35	-1	6	2	15·38	15·54	-14	1	6	3,30	2,03					
-7	4	5	7,41	7,51	0	6	2	8,75	8·42	10	1	5	2,99	·39					
-8	4	5	2,67	2,43	1	6	2	14·08	14·26	-13	1	4	4,33	3,87					
-9	4	5	7,05	6,51	2	6	2	10,82	11·38	12	1	1	2,70	2,28					
-7	4	6	3,73	3,39	3	6	2	5,87	5·95	-13	2	1	3,22	2,15					
-6	4	6	4,17	3,77	4	6	2	4,10	4·32	12	2	2	3,67	·79					
-5	4	6	7,37	7,05	5	6	2	2,62	3·38	-14	2	4	5,26	4,65					
-4	4	6	3,16	3,16	6	6	2	2,98	2·67	10	2	4	3,65	3,77					
-2	4	6	3,83	3,99	7	6	2	3·23	3·02	-12	2	5	4,64	4,19					
2	4	6	3,98	3,60	7	6	3	3,77	3,65	6	2	7	3,07	2,35					
3	4	6	2,98	3,15	6	6	3	2,70	2,06	-8	3	9	3,06	1,39					
-3	4	7	4,43	3,09	4	6	3	3,19	3,31	-7	3	8	4,16	4,45					
-4	4	7	2,39	2,78	2	6	3	9,20	9,00	-8	3	8	3,20	2,79					
-5	4	7	3,55	2,73	1	6	3	10,10	10,32	-12	3	5	2,95	1,29					
-6	4	7	4,82	5,20	0	6	3	12,53	12,64	11	4	1	5,05	4,42					
-7	4	7	2,99	2,43	-1	6	3	4,18	4,17	-12	4	1	3,95	3,21					
3	5	6	4,68	4,39	-2	6	3	7,42	7,62	-11	4	5	3,94	3,27					
1	5	6	6,91	6,18	-4	6	3	6,50	6,58	-8	4	7	3,88	3,02					
0	5	6	4,23	3,53	-5	6	3	6·41	6·80	-5	4	8	3,21	3,47					
-1	5	6	2,68	·88	-6	6	3	5,89	6·01	2	5	8	3,22	1,97					
-4	5	6	5,67	5,13	-8	6	3	3·76	3·09	1	5	8	3·41	2,53					
-6	5	6	3,94	3,51	-9	6	3	3,95	3,25	0	5	6	3,03	1,38					
-6	5	5	4,44	4,26	-8	6	4	2,74	1·01	-9	5	6	3,72	2,70					
-4	5	5	2,74	1,63	-7	6	4	3,88	4,00	-10	5	5	5·04	4·60					

THERMAL MOTIONS

The principal axes of the thermal vibration ellipsoids for the carbon, nitrogen, and oxygen atoms were calculated from the temperature parameters given in Table 1. Root mean square amplitudes and the corresponding B-values for the atomic anisotropic thermal vibrations along the principal axes are given in Table 3, as well as the components of the amplitudes along the crystal axes.

The anisotropic thermal vibration parameters of Table 1 were used in an

Table 3. R.m.s. amplitudes and the corresponding B-values along the principal axes of the thermal vibrational ellipsoids and the components of these along the crystal axes.

Atom	R.m.s. amplitude Å	B Å²	Components of the amplitudes along the crystal axes Å		
C1	0.225	4.00	0.0476	0.0466	0.2254
	0.205	3.33	0.2057	−0.0065	0.0017
	0.184	2.68	0.0022	−0.1802	0.0390
C2	0.281	6.25	0.0810	0.0146	0.2869
	0.225	4.00	0.2159	0.0452	−0.0184
	0.180	2.57	0.0382	−0.1764	0.0067
C3	0.337	8.98	0.0758	−0.0356	0.3432
	0.232	4.23	0.1760	0.1534	0.0151
	0.183	2.65	−0.1186	0.1359	0.0158
C4	0.272	5.84	0.0812	0.0321	0.2755
	0.233	4.29	0.0233	0.2292	−0.0306
	0.193	2.94	−0.1880	0.0265	0.0132
C5	0.223	3.91	0.0728	−0.1971	0.0905
	0.198	3.10	0.0284	0.0784	0.1858
	0.189	2.81	0.1809	0.0458	−0.0088
O1	0.322	8.21	0.1295	0.0051	0.3240
	0.202	3.23	−0.0886	0.1798	0.0150
	0.172	2.35	0.1437	0.0791	−0.0307
O2	0.276	6.00	0.1353	−0.0331	0.2685
	0.246	4.79	−0.1624	0.1537	0.0743
	0.172	2.35	0.1051	0.1331	−0.0159
N	0.244	4.71	0.0819	0.0175	0.2474
	0.183	2.64	0.1641	0.0639	−0.0258
	0.169	2.26	0.0610	−0.1580	0.0043

analysis of the rigid body motion of the molecule, where the S-tensor was included to account for correlations of libration and translation.[10] The results are presented in Table 4. It is seen that translational vibrations of the molecule are essentially isotropic, whereas its librations are rather anisotropic. The maximum librational motion is approximately a 9° oscillation about an axis lying in the *ab*-plane. The r.m.s. discrepancy between observed and calculated atomic vibration tensor U_{ij} is 0.0029 (Table 4) and supports the assumption of regarding the molecule as an oscillating rigid body. Corrections of fractional coordinates are listed in Table 5.

Table 4. Results of the rigid body analysis.

In the orthogonal system, where the axes J_1, J_2, J_3 are defined by: $J_1 \| a$, $J_2 \| b$, $J_3 \| c^*$. E.s.d. of component of L are given in parentheses in units of last digit shown.

$$L = \begin{Bmatrix} 152(13) & 89(12) & -20(9) \\ & 134(11) & -9(8) \\ & & 38(6) \end{Bmatrix} \times 10^{-4} \text{ rad}^2$$

	R.m.s. amplitude	Direction cosines J_1	J_2	J_3
	8.77°	−0.7406	−0.6633	0.1077
L	4.27°	−0.6080	0.7296	0.3131
	3.33°	−0.2862	0.1664	−0.9436
	0.196 Å	−0.3779	0.2287	0.8971
T^a	0.184 Å	−0.8772	−0.3984	−0.2680
	0.174 Å	0.2961	−0.8882	0.3512

In the orthogonal system L_1, L_2, L_3, defined by the principal axes of the libration tensor L.

Displacement of libration axes from intersecting

Parallel to L_1	−0.618 Å
Parallel to L_2	0.785 Å
Parallel to L_3	−0.096 Å

Effective screw translation

Parallel to L_1	0.005 Å
Parallel to L_2	0.015 Å
Parallel to L_3	−0.033 Å

$(\overline{\Delta U_{ij}^2})^{\frac{1}{2}} = 0.0029$ Å

[a] T is the reduced translation tensor.

Table 5. Corrections in fractional atomic coordinates due to libration compared with e.s.d. of the coordinates. (The Δ's and σ's are multiplied by 10^5.)

Atom	Δx	$\sigma(x)$	Δy	$\sigma(y)$	Δz	$\sigma(z)$
C1	−57	28	2	35	27	37
C2	44	34	−149	42	23	58
C3	74	33	−141	47	−57	65
C4	105	30	−82	43	1	58
C5	17	25	−75	41	7	35
O1	−103	20	34	25	34	32
O2	−2	19	144	27	−18	29
N	−64	22	105	31	7	30

DISCUSSION

The following discussion of the molecular structure is based on the results from the refinement of diffractometer data, since they have the lowest estimated standard deviations. Interatomic distances and angles are given in Table

Table 6. Distances and angles with e.s.d. in parentheses.

Distance	(Å)	Angle	(°)
C1 – C2	1.487 (4)	C2 – C1 – N	116.9 (2)
C2 – C3	1.504 (5)	C1 – C2 – C3	113.7 (3)
C3 – C4	1.480 (4)	C2 – C3 – C4	111.8 (3)
C4 – C5	1.493 (4)	C3 – C4 – C5	114.0 (3)
C5 – N	1.384 (3)	C4 – C5 – N	116.1 (3)
C1 – N	1.380 (3)	C5 – N – C1	126.9 (2)
C1 – O1	1.219 (3)	N – C1 – O1	119.3 (2)
C5 – O2	1.210 (3)	O1 – C1 – C2	123.8 (3)
N1 – H1	0.88 (3)	C4 – C5 – O2	124.0 (3)
C2 – H2	0.92 (3)	O2 – C5 – N	119.9 (3)
C2 – H3	0.97 (4)		
C3 – H4	1.10 (5)		
C3 – H5	0.95 (3)		
C4 – H6	1.03 (3)		
C4 – H7	0.98 (4)		
N – O1′	2.957 (3)		

6 and those corrected for librational effects in Fig. 1. The standard deviations have been calculated from the last full-matrix least squares cycle, not taking the e.s.d. of the cell parameters into account.

The conformation of the molecule might be described as a half-chair, with C3 0.58 Å out of the essentially coplanar system, formed by the five other

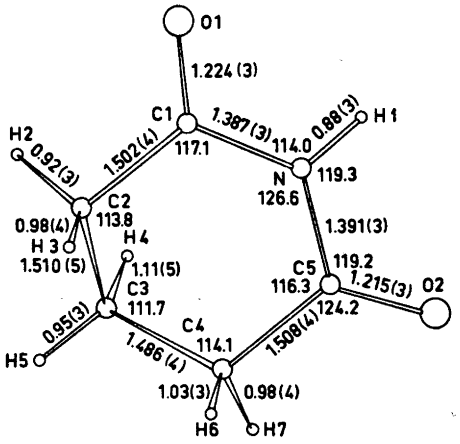


Fig. 1. Bond lengths and angles in glutarimide corrected for libration.

atoms of the glutarimide ring. Deviations from least squares planes are given in Table 7. The atom O1 is significantly out of plane 1, while O2 is not. The distances to least squares plane 2 indicate a slight bending of the C1 = O1 bond, possibly associated with the formation of the hydrogen bond O1 ··· H – N.

Table 7. Departures from least-squares planes.

Plane 1		Plane 2	
Distances to atoms defining the plane			
C1	0.013 Å	C4	−0.001 Å
C2	−0.012 Å	C5	0.004 Å
C4	0.007 Å	O2	−0.001 Å
C5	−0.005 Å	N	−0.001 Å
N	−0.005 Å		
Distances to atoms not defining the plane			
O1	0.060 Å	C1	−0.022 Å
O2	0.002 Å	C2	0.008 Å
C3	−0.588 Å	C3	0.593 Å
H1	0.067 Å	O1	−0.075 Å
		H1	−0.077 Å

The chemically equivalent bonds, N−C1, N−C5 and C1−C2, C4−C5, have within each pair a difference in bond length of less than 1.5σ. Their mean values, 1.389 Å and 1.505 Å, differ a little more than 3σ from the corresponding distances in 5-ethyl-5-(1-methylbutenyl)-barbituric acid;[11] *i.e.*, the N−C bond is 0.019 Å longer, and the C(sp^2)−C(sp^3) bond 0.019 Å shorter in glutarimide. They are, however, consistent with other observations of bonds which involve trigonal carbon.[12,13]

The two C=O bonds have normal lengths, with C1−O1 0.009 Å longer than C5−O2.

The two C(sp^3)−C(sp^3) bonds have a mean value of 1.498 Å. The apparent shortening of these bonds is not considered to be real and is probably due to an independent thermal vibration of the C3 atom nearly perpendicular to the plane of the molecule, the main component of the greatest r.m.s. amplitude for C3 being 0.34 Å in the direction of the *c*-axis. This may be taken as an indication of a certain conformational lability, and the assumption of a rigid body is possibly not valid for this molecule, in spite of the satisfactory value of the r.m.s. discrepancy between atomic vibration tensor components.

There are no significant deviations between equivalent angles. The ring angles at C2 and C4 are slightly greater than the tetrahedral angle, while at C1 and C5 they are less than 120°. At −C1−NH−C5− the ring angles are much the same as those found for a similar group in dihydrothymine.[14] There is a significant difference between the angles at the C=O bonds, a feature observed earlier.[7]

There is one intermolecular distance shorter than normal van der Waals separation, namely N···O1 of length 2.94 Å. This corresponds to a hydrogen bond N−H···O1, which is nearly linear, the angle at the hydrogen atom being 171°.

The carbonyl group C5=O2 is neither taking part in hydrogen bonding nor involved in dipole-dipole [15] interactions. The shortest distances from O2 to hydrogen atoms in neighboring molecules are 2.66 Å (H3) and 2.67 Å (H4).

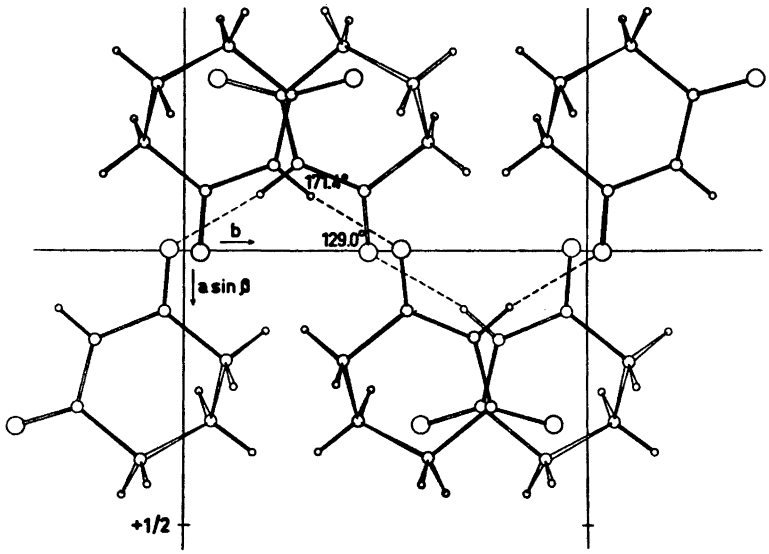


Fig. 2. The structure viewed along the *c*-axis. Broken lines indicate hydrogen bonds.

In the crystal structures of substituted glutarimides,[3,7] dimers are formed by N–H···O bonds around centers of symmetry. This arrangement is also observed for other molecules possessing the –CO–NH group.[16] The hydrogen bonding in glutarimide (Fig. 2), however, links the molecules in zigzag chains running parallel to the *b*-axis.

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