

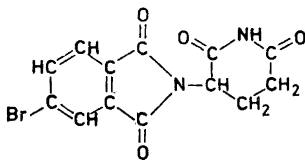
The Crystal Structure of *N*-(α -Glutarimido)-4-bromophthalimide

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The crystal structure of *N*-(α -glutarimido)-4-bromophthalimide has been determined by three-dimensional X-ray analysis. The crystals are triclinic with space group $P\bar{1}$ and unit cell constants $a=7.111 \text{ \AA}$, $b=7.916 \text{ \AA}$, $c=11.455 \text{ \AA}$, $\alpha=81.87^\circ$, $\beta=82.55^\circ$, $\gamma=88.13^\circ$. The structure was refined by full-matrix least-squares calculations of positional and thermal parameters to a final R index of 9.9%. All the atoms in the phthalimide part of the molecule, except the two oxygens, define a plane where distances to all atoms are within the standard deviations. The α -carbon atom of the glutarimide part lies also in the phthalimide plane, which is bisecting the C—C—C angle at this atom. The glutarimide ring is roughly in a half-chair conformation and the N—C bond connecting the two parts of the molecule is in an equatorial position. The molecules in the crystal are hydrogen bonded across the centre of symmetry to dimers.

In view of the pharmacological activities of thalidomide and the possible significance of *N*-substituted phthalimide structures in biological systems,¹ the X-ray investigation of *N*-(α -glutarimido)-4-bromophthalimide (I) was undertaken. A preliminary note on the structure² and a discussion of its structural relationship to nucleosides³ have been published earlier.



(I). *N*-(α -Glutarimido)-4-bromophthalimide.

Structurally the molecule consists of two ring systems, the phthalimide and the glutarimide ring, connected through a single N—C bond. The carbon atom in glutarimide involved in this bond is in the α -position. This differs

from the glutarimide antibiotics,⁴ of which (–)-cycloheximide is the best known member, where the substitution is in the β -position. Precise structural information on the phthalimide- and glutarimide ring, their steric relationship around the central N–C bond and the conformation at the asymmetric carbon atom in glutarimide do not appear to be reported in the literature.

EXPERIMENTAL

The compound was synthesized by Dr. Else Kloster-Jensen.⁵ Crystals were grown by sublimation at 180°C/0.1 mm Hg, but were, however, not true single crystals. More satisfactory crystals were obtained by cleaving the samples parallel to the b -axis. Weissenberg photographs showed the crystal to be triclinic with possible space groups $P1$ or $P\bar{1}$. The centrosymmetric space group $P\bar{1}$ was assumed, a choice supported by the synthetic procedure. To determine the unit cell dimensions 51 lines on a Guinier powder diagram were indexed using KCl as calibrating substance.

The crystal data are:

$a = 7.111 \pm 0.003$ Å, $b = 7.916 \pm 0.002$ Å, $c = 11.455 \pm 0.005$ Å, $\alpha = 81.87 \pm 0.04^\circ$,
 $\beta = 82.55 \pm 0.07^\circ$, $\gamma = 88.13 \pm 0.05^\circ$, $\lambda = 1.542$ Å (CuK α).

The density was found to be 1.75 g cm⁻³ and there are two molecules (calc. 1.98) in the unit cell.

Three crystals of rather irregular shape were used. Their cross sections were approximately 0.04 mm \times 0.02 mm. The intensity data were collected from integrated equi-inclination Weissenberg diagrams with the multiple film technique using Ni-filtered CuK α radiation. Some splitting of the reflections occurred, but this was partly compensated by integration.

Layers from $k=0$ to $k=4$, $h=0$, $l=0$ and $l=1$ were recorded. The data for least-squares refinement consisted of 1443 unique reflections out of which 404 were unobserved. Intensities were measured on a Hilger and Watts photometer and the weaker ones visually.

No correction for extinction was made and correction for absorption was considered unnecessary on account of the minute size of the crystals. Lorentz and polarization correction factors were applied and the structure amplitudes derived. Individual scale factors for the levels were found by comparing structure amplitudes of reflections common to these levels and two intersecting zero level films. In the later stages of the structure refinement the data were rescaled by making $k \sum |F_o| = \sum |F_c|$ for each layer.

STRUCTURE DETERMINATION

The structure was solved by the standard heavy atom procedure in the a - and b -projections. The (010) Patterson projection which was based on 164 observed reflections, had two maxima of equal height. One of the possible heavy atom positions was after a while rejected because of the difficulties in interpreting the corresponding Fourier syntheses. This maximum was later explained by the overlap of atoms at $x \approx 0.25$ and $z \approx 0.7$. Preliminary scale and temperature factors were derived from a Wilson plot. The first set of structure factor calculations in the $h00$ and $0k0$ zones yielded values for the conventional R factor of about 0.40. The atom parameters were refined by difference syntheses. A common B value of 4.0 Å² was assigned to the isotropic temperature factor. The refinement using zonal data was stopped when the R factor did not drop below 0.16 and 0.18, respectively.

Table 1. Fractional coordinates and thermal parameters. Estimated standard deviations in parentheses refer to the last decimal position. The expression for the temperature factor is $\exp[-(\beta_{11}h^2 + \beta_{22}k^2 + \beta_{33}l^2 + \beta_{12}hk + \beta_{13}hl + \beta_{23}kl)]$.

	<i>x</i>	<i>y</i>	<i>z</i>	β_{11}	β_{22}	β_{33}	β_{12}	β_{13}	β_{23}	
Br	-0.2518 (2)	-0.2545 (2)	0.3225 (2)	0.0169 (4)	0.0131 (3)	0.0082 (2)	0.0006 (5)	0.0020 (4)	0.0048 (4)	
C(1)	-0.2524 (17)	-0.4755 (19)	0.4168 (11)	0.009 (3)	0.011 (3)	0.003 (1)	-0.001 (5)	0.001 (3)	0.007 (3)	
C(2)	-0.2613 (23)	-0.6157 (26)	0.3470 (17)	0.022 (5)	0.016 (4)	0.009 (2)	-0.001 (7)	-0.001 (5)	0.002 (6)	
C(3)	-0.2661 (29)	-0.7762 (27)	0.4180 (19)	0.028 (5)	0.017 (5)	0.011 (2)	-0.003 (8)	0.002 (6)	0.007 (6)	
C(4)	-0.2632 (22)	-0.7891 (21)	0.5404 (16)	0.020 (4)	0.008 (3)	0.011 (2)	0.008 (6)	0.003 (4)	-0.008 (5)	
C(5)	-0.2523 (18)	-0.6539 (19)	0.5945 (12)	0.013 (3)	0.008 (3)	0.004 (1)	-0.001 (5)	0.002 (3)	0.003 (3)	
C(6)	-0.2464 (24)	-0.4877 (24)	0.5340 (15)	0.020 (4)	0.016 (4)	0.006 (2)	-0.001 (7)	0.002 (4)	0.004 (5)	
C(7)	-0.2641 (20)	-0.9516 (20)	0.6296 (12)	0.014 (3)	0.009 (3)	0.005 (1)	0.001 (5)	0.003 (3)	0.007 (4)	
C(8)	-0.2521 (20)	-0.7157 (20)	0.7239 (14)	0.012 (3)	0.011 (3)	0.008 (1)	0.000 (5)	-0.005 (3)	-0.007 (4)	
C(9)	-0.2608 (20)	-0.0064 (19)	0.8529 (13)	0.017 (3)	0.009 (3)	0.006 (1)	0.001 (5)	-0.004 (4)	0.007 (4)	
C(10)	-0.0797 (20)	-0.1225 (21)	0.8524 (14)	0.011 (3)	0.013 (3)	0.008 (2)	0.000 (5)	0.000 (3)	-0.002 (4)	
C(11)	-0.0923 (21)	-0.2369 (21)	0.9668 (14)	0.017 (3)	0.012 (3)	0.007 (1)	0.002 (5)	-0.006 (4)	-0.002 (4)	
C(12)	-0.2695 (18)	-0.3468 (19)	0.9887 (13)	0.010 (3)	0.008 (3)	0.006 (1)	-0.002 (5)	-0.007 (3)	-0.008 (4)	
C(13)	-0.4395 (20)	-0.1142 (21)	0.8772 (13)	0.013 (3)	0.012 (3)	0.006 (1)	0.001 (5)	0.003 (3)	0.005 (4)	
O(1)	-0.2827 (19)	-0.0978 (14)	0.6176 (11)	0.038 (4)	0.007 (2)	0.010 (1)	-0.007 (5)	-0.009 (4)	-0.001 (3)	
O(2)	-0.2533 (16)	-0.6408 (15)	0.8106 (10)	0.025 (3)	0.012 (2)	0.008 (1)	0.002 (4)	-0.004 (3)	-0.002 (3)	
O(3)	-0.2834 (14)	-0.4827 (14)	0.0515 (10)	0.014 (2)	0.011 (2)	0.011 (1)	-0.001 (4)	-0.005 (3)	0.006 (3)	
O(4)	-0.5886 (13)	-0.0619 (15)	0.8449 (10)	0.013 (2)	0.015 (2)	0.010 (1)	-0.002 (4)	0.010 (3)	0.008 (3)	
N(1)	-0.2590 (16)	-0.8957 (16)	0.7367 (10)	0.016 (3)	0.007 (2)	0.006 (1)	-0.003 (4)	0.001 (3)	0.000 (3)	
N(2)	-0.4255 (15)	-0.2698 (17)	0.9408 (11)	0.011 (2)	0.013 (3)	0.008 (1)	-0.005 (4)	-0.009 (3)	0.004 (3)	
H(1)	-0.2671	-0.5948	0.2357	<i>B</i> (Å ²)						
H(2)	-0.2701	-0.8820	0.3803	3.3						
H(3)	-0.2367	-0.3683	0.5824	3.3						
H(4)	-0.2604	0.0920	0.9069	3.3						
H(5)	0.0561	-0.0560	0.8250	3.3						
H(6)	-0.0439	-0.1856	0.7724	3.3						
H(7)	-0.0725	-0.1761	0.0376	3.3						
H(8)	0.0300	-0.3058	0.9794	3.3						
H(9)	-0.5159	-0.3228	0.9521	3.3						

Table 2. Observed and calculated structure factors. The columns are h , k , l , F_o and F_c . The half value of the minimum observable $|F_o|$ is listed for reflexions too weak to be observed, and they are marked by an asterisk.

0	0	1	12.13	14.82	-4	0	-1	47.14	41.92	-1	1	-4	24.81	26.86	2	1	9	16.61	15.91	
0	0	2	15.43	10.45	-4	0	-2	21.39	17.84	-1	1	-5	32.71	27.13	2	1	-1	91.47	67.82	
0	0	3	57.59	57.21	-4	0	-3	46.43	46.51	-1	1	-6	30.33	29.92	2	1	-2	11.12	7.57	
0	0	4	31.36	34.97	-4	0	-4	14.02	7.56	-1	1	-7	13.52	4.88	2	1	-3	27.75	14.87	
0	0	5	14.20	6.19	-4	0	-5	6.39	2.79	-1	1	-8	28.90	26.15	2	1	-4	34.96	35.15	
0	0	6	25.83	32.96	-4	0	-6	14.11	16.87	-1	1	-9	14.97	15.26	2	1	-5	14.81	21.34	
0	0	7	16.33	12.66	-4	0	-7	5.90	3.86	-2	1	0	36.35	35.41	2	1	-6	24.97	21.57	
0	0	8	15.30	18.77	-4	0	-8	15.39	14.62	-2	1	1	64.08	63.69	2	-7	0	30.27	37.38	
0	0	9	7.48	4.21	-4	0	-11	5.26	5.74	-2	1	2	34.52	44.73	2	1	-8	15.80	14.75	
0	0	10	11.40	4.73	-5	0	0	18.52	14.75	-2	1	3	12.52	8.23	3	1	0	5.34	1.06	
0	0	11	11.64	14.47	-5	0	1	5.83	9.15	-2	1	4	34.31	69.45	3	1	2	37.81	36.74	
0	0	12	48.07	6.91	-5	0	2	21.14	22.68	-2	1	5	19.14	14.88	3	1	3	41.92	46.97	
-1	0	0	35.21	24.96	-5	0	4	29.12	27.43	-2	1	6	21.21	22.72	3	1	5	16.26	21.88	
-1	0	1	13.57	13.82	-5	0	5	10.18	14.53	-2	1	7	19.77	17.51	3	1	6	38.56	39.25	
-1	0	2	46.33	45.21	-5	0	7	17.23	14.95	-2	1	8	14.38	13.89	3	1	9	20.94	20.79	
-1	0	3	22.94	14.36	-5	0	8	15.33	7.89	-2	1	-1	82.80	34.96	3	1	-1	54.86	48.15	
-1	0	4	49.83	47.92	-5	0	9	14.41	14.68	-2	1	-2	19.18	17.76	3	1	-2	27.76	25.87	
-1	0	5	31.20	37.10	-5	0	10	11.87	13.54	-2	1	-3	22.68	23.81	3	1	-3	33.61	31.42	
-1	0	6	16.85	15.03	-5	0	-1	15.41	10.19	-2	1	-4	59.09	32.88	3	1	-4	22.80	23.49	
-1	0	7	27.65	21.25	-5	0	-2	31.95	32.88	-2	1	-5	32.87	38.74	3	1	-5	21.22	20.82	
-1	0	8	17.93	16.81	-5	0	-3	22.42	17.91	-2	1	-6	18.41	19.97	3	1	-6	27.75	29.46	
-1	0	9	21.35	24.29	-5	0	-4	24.98	22.59	-3	1	0	5.69	.62	3	1	-8	21.45	19.46	
-1	0	10	25.36	26.51	-5	0	-5	20.22	22.48	-3	1	1	9.98	8.17	3	1	-9	16.08	15.21	
-1	0	11	14.59	12.12	-5	0	-6	14.99	4.75	-3	1	2	29.15	28.00	4	1	0	16.54	15.71	
-1	0	12	7.76	9.17	-5	0	-7	15.55	14.93	-3	1	3	46.91	49.61	4	1	1	31.13	29.54	
-1	0	13	6.75	6.48	-5	0	-8	12.84	12.43	-3	1	4	25.72	29.98	4	1	2	29.87	31.94	
-1	0	-1	9.92	1.18	-5	0	-9	11.85	13.24	-3	1	5	40.20	41.40	4	1	3	19.93	16.51	
-1	0	-2	25.40	20.40	-5	0	-10	12.84	13.03	-3	1	6	16.14	12.31	4	1	4	21.22	20.82	
-1	0	-3	11.84	9.56	-5	0	-11	6.81	4.19	-3	1	7	22.95	21.25	4	1	5	19.62	14.36	
-1	0	-4	73.08	76.53	-6	0	0	65.36	61.75	-3	1	11	16.90	16.08	4	1	6	23.86	27.91	
-1	0	-5	18.36	25.67	-6	0	1	5.85	5.91	-3	1	-1	38.37	27.79	4	1	-1	39.19	36.66	
-1	0	-6	19.25	9.84	-6	0	2	7.21	7.85	-3	1	-2	14.30	17.87	4	1	-2	18.43	17.60	
-1	0	-7	30.17	34.32	-6	0	3	18.19	14.74	-3	1	-3	34.59	34.34	4	1	-3	15.84	15.88	
-1	0	-8	15.47	16.40	-6	0	4	9.71	11.08	-3	1	-4	12.01	9.93	4	1	-4	31.49	30.34	
-1	0	-9	17.37	20.81	-6	0	5	9.47	10.12	-3	1	-5	23.31	17.04	4	1	-7	20.85	23.63	
-1	0	-10	27.88	28.10	-6	0	6	9.01	11.60	-3	1	-6	24.74	25.79	4	1	-8	15.16	14.66	
-1	0	-11	14.64	12.15	-6	0	7	11.83	9.77	-3	1	-7	24.05	23.88	4	1	-10	15.84	14.66	
-1	0	-12	6.08	8.22	-6	0	9	4.18	7.61	-3	1	-9	16.21	17.50	5	1	0	48.03	14.12	
-1	0	-13	8.43	7.24	-6	0	-1	13.60	9.81	-4	1	0	15.89	14.73	5	1	3	30.46	32.11	
-2	0	0	172.31	158.78	-6	0	-2	15.36	11.87	-4	1	1	14.58	55.25	5	1	5	19.45	19.18	
-2	0	1	46.54	42.21	-6	0	-3	17.94	14.92	-4	1	2	27.94	31.82	5	1	6	15.84	15.84	
-2	0	2	43.52	36.96	-6	0	-4	4.87	6.05	-4	1	4	25.51	25.05	5	1	-1	21.19	14.58	
-2	0	3	65.61	63.87	-6	0	-5	7.80	5.00	-4	1	5	16.47	13.96	5	1	-3	22.35	23.06	
-2	0	4	10.50	6.04	-6	0	-6	15.57	14.69	-4	1	7	11.85	12.90	5	1	-4	16.01	14.69	
-2	0	5	3.40	7.87	-6	0	-7	11.83	9.77	-4	1	-1	55.64	57.00	5	1	-6	14.84	14.84	
-2	0	6	17.93	22.14	-6	0	-8	8.35	10.55	-4	1	-4	30.56	26.87	5	1	-8	19.04	23.24	
-2	0	7	7.46	4.30	-6	0	-11	6.67	6.88	-4	1	-5	14.62	13.12	6	1	1	14.63	15.41	
-2	0	8	20.94	24.51	-7	0	0	10.77	14.08	-4	1	-6	17.16	15.12	6	1	2	32.06	34.29	
-2	0	9	10.59	9.21	-7	0	-4	15.12	12.16	-4	1	-7	23.52	15.93	6	1	-3	27.48	27.48	
-2	0	10	5.85	2.53	-7	0	2	10.46	8.53	-5	1	0	23.44	24.68	6	1	5	17.28	11.28	
-2	0	11	5.48	5.27	-7	0	4	12.89	11.42	-5	1	1	20.76	14.62	6	1	-1	15.32	14.19	
-2	0	12	10.39	10.46	-7	0	5	11.92	11.93	-5	1	3	37.21	31.98	6	1	-2	17.85	16.51	
-2	0	-1	78.07	71.08	-7	0	-1	8.75	8.90	-5	1	5	15.84	22.48	6	1	-4	17.20	17.20	
-2	0	-2	20.78	16.35	-7	0	-7	5.40	4.67	-5	1	6	25.36	23.26	7	1	2	14.96	13.69	
-2	0	-3	63.08	63.05	-7	0	-2	17.66	14.75	-5	1	9	14.47	16.44	7	1	0	40.47	44.40	
-2	0	-4	17.73	16.05	-7	0	-3	6.15	4.08	-5	1	-3	23.76	20.90	7	1	1	16.92	12.79	
-2	0	-5	13.45	11.46	-7	0	-4	17.10	13.87	-5	1	-4	17.20	13.53	7	1	-2	11.63	11.63	
-2	0	-6	13.80	18.03	-7	0	-5	17.74	13.09	-5	1	-6	17.24	20.19	7	1	-3	63.78	85.62	
-2	0	-7	15.17	11.56	-7	0	-7	6.74	6.45	-5	1	-8	19.71	16.70	7	1	-4	18.00	13.26	
-2	0	-8	7.08	10.70	-7	0	-10	8.23	9.85	-6	1	1	36.83	37.24	7	1	5	53.51	52.78	
-2	0	-9	13.08	13.53	-7	0	0	15.74	14.20	-6	1	2	26.02	26.81	7	1	-6	26.52	24.72	
-2	0	-10	6.53	7.77	-7	0	1	15.24	14.22	-6	1	5	15.74	12.41	7	1	8	19.55	14.62	
-2	0	-11	6.49	8.96	-8	0	2	10.33	9.29	-6	1	-1	15.22	14.72	7	1	9	11.84	9.16	
-2	0	-12	8.91	7.70	-8	0	3	8.11	11.14	-6	1	-2	19.80	14.75	7	1	-10	17.40	4.16	
-3	0	0	23.54	21.58	-8	0	-1	17.19	10.61	-6	1	-4	22.06	21.24	7	1	11	11.66	8.29	
-3	0	1	15.76	16.15	-8	0	-3	14.47	13.34	-6	1	5	15.74	12.41	7	1	12	55.37	49.64	
-3	0	2	20.62	22.48	-8	0	1	7.02	8.76	-7	1	3	18.48	21.42	7	1	-2	34.72	43.17	
-3	0	3	17.23	15.29	-8	0	1	54.54	65.48	-7	1	6	13.60	14.75	7	1	-3	44.39	44.19	
-3	0	4	36.95	55.89	-8	0	2	93.87	104.81	-7	1	-3	18.19	16.40	7	1	-4	37.51	36.22	
-3	0	5	11.04	12.94	-8	0	1	42.22	73.77	-8	1	1	15.64	17.81	7	1	-5	11.82	9.03	
-3	0	6	7.51	4.50	-8	0	1	4	13.93	1.79	-8	1	2	11.34	11.55	7	1	-6	24.37	26.12
-3	0	7	23.95	25.36	-8	0	5	24.80	25.33	-8	1	-1	13.18	13.09	7	1	-7	40.10	36.97	
-3	0	8	10.97	11.05	-8	0	6	16.06	20.00	-8	1	0	37.55	35.33	7	1	-8	29.23	27.10	
-3	0	9	17.13	16.91	-8	0	7	38.00	38.53	-8	1	1	54.24	40.92	7	1	-9	16.99	14.33	
-3	0	10	22.02	25.21	-8	0	1	20.75	25.82	-8	1	2	32.29	31.40	7	1	-10	11.52	12.43	
-3	0	11	6.65	7.11	-8	0	-1	40.84	42.73	-8	1	3	58.13	67.40	-1	2	0	5.19	4.79	
-3	0	12	7.04	7.25	-8	0	-2	37.86	32.24	-8	1	4	9.95	7.25	-1	2	1	28.12	33.93	
-3	0	-1	21.91	20.93	-8	0	-3	22.27	24.08	-8	1	5	29.72	37.70	-1	2</				

Table 2. Continued.

-2	2	5	21.86	22.68	2	2	-11	11.92	13.41	-3	3	V	17.39	17.86	5	3	-2	16.01	13.70
-2	2	6	42.96	43.96	3	2	1	41.00	42.83	-3	3	-2	31.94	33.07	5	3	-3	28.20	31.85
-2	2	7	15.36	11.95	3	2	2	27.11	26.63	-3	3	-3	35.55	42.45	5	3	-6	23.05	27.21
-2	2	8	18.11	15.63	3	2	3	10.63	10.00	-3	3	-4	24.54	24.82	5	3	-7	13.44	15.33
-2	2	-1	27.97	25.08	3	2	4	14.03	16.77	-3	3	-5	31.26	32.82	6	3	1	17.27	16.82
-2	2	-2	43.11	45.08	3	2	5	18.91	16.17	-3	3	-6	37.01	37.08	6	3	3	15.65	9.88
-2	2	-3	15.65	16.85	3	2	7	15.44	13.50	-3	3	-7	25.30	19.16	6	3	4	14.87	11.36
-2	2	-4	31.74	31.08	3	2	8	16.50	17.12	-3	3	-8	18.34	19.35	6	3	5	16.36	18.80
-2	2	-5	26.04	24.15	3	2	-1	44.73	44.39	-4	3	1	17.17	12.63	6	3	6	16.12	14.42
-2	2	-6	10.56	7.28	3	2	-2	15.89	15.39	-4	3	2	24.67	22.74	6	3	7	14.34	13.74
-2	2	-7	26.84	26.23	3	2	-4	40.67	43.24	-4	3	3	36.10	37.60	6	3	-1	23.17	21.94
-2	2	-8	31.74	30.99	3	2	-5	40.70	37.68	-4	3	4	18.55	18.06	6	3	-2	16.43	15.29
-2	2	-9	15.26	15.69	3	2	-6	11.13	12.08	-4	3	5	20.27	17.31	6	3	-3	12.82	13.16
-3	2	0	15.56	10.92	3	2	-7	30.63	35.76	-4	3	6	20.45	19.58	7	3	0	13.48	14.40
-3	2	1	27.05	30.41	3	2	-10	11.02	9.72	-4	3	-1	26.41	29.37	7	3	-3	15.30	17.85
-3	2	2	35.62	37.60	4	2	0	23.57	29.52	-4	3	-2	35.56	37.52	0	4	0	39.76	40.00
-3	2	3	9.16	5.44	4	2	1	15.11	13.25	-4	3	-4	20.45	23.62	0	4	1	9.23	9.79
-3	2	4	18.46	23.58	4	2	2	34.38	35.92	-4	3	-7	24.43	23.77	0	4	2	15.52	14.29
-3	2	5	21.16	19.29	4	2	3	24.26	26.89	-4	3	-8	14.97	12.53	0	4	3	19.67	19.52
-3	2	6	11.82	18.77	4	2	4	17.94	16.53	-5	3	0	18.73	19.05	0	4	4	13.11	9.42
-3	2	7	15.85	15.18	4	2	5	22.00	23.46	-5	3	1	14.11	17.57	0	4	5	29.55	31.01
-3	2	8	15.72	13.92	4	2	6	26.62	27.10	-5	3	2	18.35	17.13	0	4	6	15.34	14.46
-3	2	-1	57.07	60.90	4	2	-1	26.97	24.48	-5	3	3	27.84	22.49	0	4	8	24.21	27.41
-3	2	-2	10.85	10.85	4	2	-2	29.14	26.04	-5	3	4	14.46	16.62	0	4	-1	19.83	21.62
-3	2	-3	17.57	10.97	4	2	3	18.49	20.40	-5	3	5	14.17	13.35	0	4	-2	23.50	19.00
-3	2	-4	36.03	37.44	4	2	-5	15.88	12.56	-5	3	6	17.67	12.52	0	4	-3	54.02	53.64
-3	2	-5	41.72	38.45	4	2	-6	23.28	22.31	-5	3	-2	24.57	24.70	0	4	-5	27.59	29.41
-3	2	-6	12.50	12.50	4	2	-8	20.95	17.71	-5	3	-3	26.26	31.92	0	4	-6	10.46	8.41
-4	2	0	18.30	22.36	4	2	-9	19.53	19.83	-5	3	-4	18.55	12.31	0	4	-8	24.14	23.22
-4	2	1	21.98	21.92	5	2	1	23.77	24.43	-6	3	1	14.00	12.89	0	4	-9	15.32	13.72
-4	2	2	24.46	24.61	5	2	2	23.48	22.06	-6	3	2	14.70	14.70	0	4	-11	12.58	17.01
-4	2	3	11.82	18.77	5	2	3	18.47	17.71	-6	3	3	14.04	17.53	0	4	-12	10.46	8.41
-4	2	4	19.35	18.11	5	2	4	23.04	21.76	-6	3	4	14.73	14.73	0	4	-13	17.56	16.01
-4	2	5	27.90	27.16	5	2	5	17.12	17.05	-6	3	5	27.83	20.09	-1	4	2	46.80	44.14
-4	2	6	17.32	10.56	5	2	-1	18.24	18.49	-6	3	-2	14.57	14.49	-1	4	3	14.44	13.39
-4	2	7	10.56	8.79	5	2	-2	18.56	19.14	-6	3	-3	14.85	14.49	-1	4	4	21.16	20.49
-4	2	-1	15.94	13.17	5	2	-4	20.26	20.00	-6	3	-4	14.56	12.81	-1	4	5	16.13	13.13
-4	2	-2	19.34	21.83	5	2	-5	21.36	22.10	-6	3	-5	12.84	12.01	-1	4	6	24.34	24.56
-4	2	-3	27.89	27.91	5	2	-7	17.66	19.44	-7	3	0	16.64	17.94	-1	4	10	15.02	11.03
-4	2	-4	11.55	11.55	5	2	-8	11.84	12.72	-7	3	1	12.32	11.24	-1	4	-1	11.86	11.86
-4	2	-5	14.43	12.99	6	2	0	14.44	14.44	-7	3	2	17.41	11.30	-1	4	-2	30.43	32.68
-4	2	-6	15.18	13.91	6	2	1	21.88	22.11	-7	3	-3	16.06	14.14	-1	4	-4	25.45	27.24
-4	2	-7	21.13	26.97	6	2	2	25.52	26.93	-7	3	-4	11.86	14.33	-1	4	-5	12.64	14.43
-4	2	-8	10.30	9.71	6	2	-1	15.90	14.12	1	3	0	37.00	39.58	-1	4	-7	18.07	19.55
-5	2	1	17.35	19.21	6	2	-2	14.91	13.16	1	3	1	13.16	13.16	-1	4	-9	18.23	9.42
-5	2	2	24.22	26.73	6	2	-3	18.47	19.69	1	3	2	49.86	51.05	-1	4	-10	20.78	21.30
-5	2	3	14.40	12.41	6	2	-4	17.16	15.67	1	3	3	33.16	33.20	-2	4	0	38.14	37.77
-5	2	4	18.02	18.18	6	2	-6	12.47	14.70	1	3	4	9.12	4.59	-2	4	2	9.09	6.41
-5	2	5	17.03	17.08	7	2	1	11.43	11.87	1	3	5	35.93	38.44	-2	4	3	16.71	16.21
-5	2	6	10.15	9.30	7	2	2	16.98	13.65	1	3	6	31.95	30.33	-2	4	5	36.19	37.85
-5	2	-1	20.11	21.62	7	2	-1	11.15	19.31	1	3	7	23.54	23.96	-2	4	6	14.92	13.44
-5	2	-2	17.97	16.85	7	2	-4	14.06	16.83	1	3	-2	47.34	39.89	-2	4	8	14.94	14.76
-5	2	-3	24.65	24.77	7	2	-5	17.49	17.49	1	3	-3	35.63	60.65	-2	4	11	18.04	16.51
-5	2	-4	29.65	23.42	8	2	2	13.99	15.47	1	3	-4	12.06	8.49	-2	4	-1	32.88	35.08
-5	2	-5	13.62	14.55	0	3	0	6.61	7.33	1	3	-5	17.54	19.88	-2	4	-2	9.64	4.12
-5	2	-6	13.77	8.09	0	3	1	38.92	41.72	1	3	-6	48.15	49.22	-2	4	-3	30.76	37.39
-5	2	-7	11.78	11.78	0	3	2	16.36	15.62	1	3	-7	14.62	13.98	-2	4	-4	22.85	22.85
-6	2	0	18.73	19.91	0	3	3	12.72	17.75	1	3	-11	12.93	12.01	-2	4	-6	21.18	22.49
-6	2	1	27.10	30.61	0	3	4	33.51	30.83	2	3	0	13.16	14.34	-2	4	-8	18.29	17.99
-6	2	2	11.90	10.69	0	3	5	56.77	57.99	2	3	1	12.97	17.53	-2	4	-9	17.43	16.46
-6	2	3	11.19	11.19	0	3	6	45.81	42.44	2	3	2	12.41	12.47	-3	4	-10	13.25	13.11
-6	2	-1	17.39	8.70	0	3	7	21.86	22.91	2	3	3	11.07	4.73	-3	4	2	30.34	29.34
-6	2	-2	17.55	15.92	0	3	8	15.46	13.44	2	3	4	61.31	61.97	-3	4	4	25.15	24.15
-6	2	-3	20.11	21.46	0	3	-1	64.14	63.79	2	3	5	15.33	13.85	-3	4	5	19.39	19.39
-6	2	-4	11.75	11.69	0	3	-2	52.72	57.90	2	3	6	22.92	20.11	-3	4	6	18.70	18.70
-6	2	-5	12.37	13.39	0	3	-3	8.35	2.98	2	3	7	27.74	23.27	-3	4	-1	32.43	31.30
-6	2	-6	11.50	8.89	0	3	-4	15.23	19.77	2	3	8	14.25	13.45	-3	4	-2	26.04	27.92
-6	2	-7	16.37	18.50	0	3	-5	44.37	40.26	2	3	-1	45.11	45.73	-3	4	-3	30.11	30.66
-6	2	-8	16.85	17.76	0	3	-6	17.65	13.65	2	3	-2	13.45	6.66	-3	4	-4	26.33	26.88
-7	2	-1	14.68	15.24	0	3	-7	27.27	27.63	2	3	-3	11.31	7.74	-3	4	-7	19.47	21.71
-7	2	-2	11.27	8.72	0	3	-8	17.40	13.27	2	3	-4	29.46	33.27	-4	4	0	46.20	26.26
-7	2	-3	11.53	9.68	0	3	-10	25.57	20.04	2	3	-5	14.59	12.15	-4	4	1	15.17	11.62
-7	2	-4	18.46	12.24	0	3	-11	50.93	55.32	2	3	-6	17.16	10.38	-4	4	2	16.44	14.90
-8	2	0	11.83	13.24	-1	3	1	21.06	17.48	2	3	-7	35.38	36.80	-4	4	3	24.71	24.10
1	2	0	14.14	13.63	-1	3	2	54.86	54.76	2	3	-8	17.11	10.64	-4	4	4	18.44	12.85
1	2	1	33.00	39.31	-1	3	3	33.05	28.72	2	3	-10	15.47	13.74	-4	4	-1	24.94	26.79
1	2	2	36.85	74.82	-1	3	4	20.65	22.50	2	3	-11	23.07	25.43	-4	4	-2	13.37	9.33
1	2	3	24.72	29.27	-1	3	5	20.45	26.13	3	3	1	39.42	34.53	-4	4	-3		

Table 2. Continued.

2	4	-1	32.46	34.13	-3	6	1	26.40	28.88	-7	0	-6.4	2.74	5.77	-4	2	-7.6	5.40	3.73	
2	4	-2	16.04	11.45	-3	6	-1	10.11	11.73	-7	0	-9.4	8.06	0.49	-4	2	-10.6	5.47	5.44	
2	4	-3	17.45	17.07	-4	6	0	9.04	9.96	-8	0	4.8	1.34	1.78	-5	2	0.8	5.41	2.33	
2	4	-4	18.95	20.88	-4	6	1	8.93	12.23	-8	0	5.4	1.19	1.96	-5	2	4.6	5.41	12.39	
2	4	-5	20.66	27.21	-4	6	-1	21.76	22.25	-8	0	-2.8	2.11	4.91	-5	2	0.8	5.47	3.32	
2	4	-6	23.78	29.90	-5	6	0	12.66	12.83	-8	0	0	0	0	-5	2	6.8	3.34	8.53	
2	4	-8	20.87	18.94	-5	6	-1	11.34	12.94	-8	0	-5.4	1.93	4.63	-5	2	9.0	4.77	1.98	
2	4	-9	14.06	15.99	-5	6	1	8.35	12.94	-5	6	1	6.6	6.79	2.49	-5	2	-3.8	5.34	4.30
2	4	-11	12.98	16.70	-6	6	0	10.74	14.32	0	1	10.8	7.67	1.50	-5	2	-9.4	5.46	6.84	
3	4	1	27.77	27.54	1	6	0	31.30	31.26	0	1	11.4	7.64	1.92	-6	2	-1.8	5.48	1.60	
3	4	2	29.40	30.67	1	6	-1	14.95	11.68	-2	1	0.8	7.34	4.05	-6	2	4.6	5.80	2.14	
3	4	3	11.26	28.78	2	6	0	8.12	6.92	-2	1	10.6	7.66	10.25	-6	2	-7.8	5.34	4.73	
3	4	4	20.56	30.42	2	6	0	10.73	14.94	-2	1	11.4	7.24	3.31	-7	2	0.8	5.47	3.77	
3	4	5	13.30	14.11	2	6	1	10.73	14.94	-2	1	11.4	7.24	3.31	-7	2	1.8	5.57	7.93	
3	4	6	15.15	16.90	2	6	-1	29.07	29.90	-2	1	-6.0	5.47	3.26	-7	2	-3.8	5.57	3.24	
3	4	7	38.41	35.49	3	6	0	36.13	36.65	-2	1	-9.4	7.76	9.70	1	2	-9.4	5.68	3.35	
3	4	8	24.47	25.96	3	6	-1	9.48	10.12	-3	1	4.4	6.29	1.44	2	2	7.8	4.90	4.18	
3	4	9	29.27	27.89	4	6	0	12.70	12.24	-3	1	7.8	7.75	7.23	2	2	-6.6	5.96	7.34	
3	4	10	19.47	16.84	4	6	-1	20.94	22.75	-3	1	10.4	7.76	3.21	2	2	-4.0	4.29	1.98	
3	4	11	22.72	23.04	5	6	0	17.70	12.24	-3	1	-7.4	7.00	7.42	2	2	-7.8	5.36	3.13	
4	4	0	13.71	12.45	5	6	1	6.41	8.04	-4	1	3.6	6.40	4.22	2	2	-11.8	5.69	7.40	
4	4	1	25.03	24.10	6	6	0	6.41	8.04	-4	1	3.6	6.40	4.22	2	2	0.6	2.87	1.08	
4	4	2	14.08	11.32	7	6	0	9.66	12.84	-4	1	0.8	7.66	5.74	3	2	6.0	6.82	10.16	
4	4	3	18.24	18.72	7	6	1	11.86	11.14	-4	1	9.4	7.31	9.34	3	2	0.8	8.16	9.67	
4	4	4	21.10	24.08	7	6	-1	8.96	7.49	-4	1	10.4	6.62	3.33	3	2	0.8	8.16	9.67	
4	4	5	25.43	27.76	7	6	0	16.96	20.48	-4	1	1.6	5.65	2.20	3	2	-8.0	5.89	6.33	
4	4	6	21.65	21.40	7	6	1	15.58	15.74	-4	1	-2.6	5.97	7.67	3	2	-4.0	5.71	6.36	
4	4	7	15.83	11.02	7	6	-1	11.57	11.74	-4	1	-3.8	6.16	7.08	4	2	7.0	7.07	4.27	
4	4	8	20.56	20.44	7	6	0	11.28	10.28	-4	1	-6.6	7.66	8.90	4	2	6.0	8.26	4.69	
4	4	9	14.91	11.87	7	6	-1	16.58	17.03	-5	1	2.4	7.78	9.25	4	2	-4.0	5.45	1.02	
4	4	10	17.01	17.63	7	6	0	17.01	21.78	-5	1	8.0	7.98	3.03	4	2	-7.8	5.80	7.23	
4	4	11	11.32	11.51	7	6	-1	17.01	21.78	-5	1	8.0	7.98	3.03	4	2	0.8	8.16	9.67	
4	4	12	18.61	18.61	7	6	0	18.01	17.00	-5	1	8.0	7.98	3.03	4	2	-7.8	5.80	7.23	
5	4	2	23.60	23.76	7	6	-1	17.05	16.27	-5	1	1.6	6.40	8.52	5	2	0.8	7.72	5.84	
5	4	3	27.03	23.09	7	6	-1	17.75	22.43	-5	1	1.6	6.40	8.52	5	2	7.8	6.07	5.42	
5	4	4	15.10	17.45	7	6	0	11.34	9.06	-5	1	-2.0	6.94	7.73	5	2	-3.0	5.19	3.40	
5	4	5	17.42	16.20	7	6	-1	14.93	17.45	-5	1	-2.0	6.94	7.73	5	2	7.8	6.07	5.42	
5	4	6	14.46	12.79	7	6	0	9.13	9.13	-5	1	-4.4	7.23	8.70	5	2	-1.8	5.46	3.86	
5	4	7	17.06	15.67	-1	7	0	18.78	16.68	-5	1	-1.8	7.70	4.70	6	2	1.0	8.05	1.94	
5	4	8	18.21	12.88	-2	7	0	14.51	12.32	-6	1	0.8	7.33	3.91	6	2	-1.8	5.05	1.90	
5	4	9	14.06	14.06	-2	7	1	11.95	10.49	-6	1	3.8	7.71	7.22	6	2	2.8	5.84	9.60	
5	4	10	15.83	11.02	-2	7	1	9.88	8.87	-6	1	4.8	7.64	10.79	6	2	4.0	6.24	2.41	
5	4	11	15.83	11.02	-2	7	-1	19.02	16.76	-6	1	6.0	7.00	2.72	6	2	-4.0	5.74	4.58	
5	4	12	25.91	26.18	-3	7	0	18.80	16.14	-6	1	-3.8	7.66	3.42	6	2	-5.0	5.40	4.48	
5	4	13	20.50	27.00	-3	7	-1	21.19	21.81	-6	1	-5.8	7.71	5.27	6	2	-7.8	5.47	1.65	
5	4	14	29.41	29.94	-4	7	1	16.74	19.46	-7	1	0.8	7.50	9.59	6	2	-7.8	5.47	1.65	
5	4	15	17.46	15.66	-5	7	0	7.90	7.12	-7	1	1.4	7.72	2.67	7	2	-4.0	5.62	11.78	
5	4	16	34.76	34.76	-6	7	-1	7.00	6.82	-7	1	4.8	7.28	3.52	7	2	-4.0	5.47	1.65	
5	4	17	11.41	11.15	-6	7	0	8.01	9.75	-7	1	4.4	6.71	2.51	7	2	-3.0	5.19	3.40	
5	4	18	11.42	9.08	-6	7	1	19.69	19.17	-7	1	5.8	6.21	9.45	8	2	0.8	6.68	9.01	
5	4	19	19.06	12.51	-7	7	0	11.46	10.60	-7	1	-1.8	7.70	4.70	8	2	0.8	6.68	9.01	
5	4	20	30.54	28.79	-7	7	-1	15.98	14.06	-7	1	-1.8	7.70	4.70	8	2	0.8	6.68	9.01	
5	4	21	35.42	36.89	-7	7	0	10.49	9.48	-7	1	-4.4	7.35	6.61	0	3	1.0	7.43	6.34	
5	4	22	37.82	35.60	-7	7	1	16.14	15.52	-8	1	0.8	6.16	5.52	0	3	-9.0	7.23	6.53	
5	4	23	23.18	25.39	-7	7	-1	20.88	18.35	-8	1	3.8	5.23	3.98	-1	3	-4.0	6.16	11.12	
5	4	24	15.83	11.02	-7	7	0	8.51	8.70	-8	1	-2.8	6.24	8.89	-1	3	7.4	6.73	4.47	
5	4	25	19.18	18.84	-7	7	-1	10.53	8.17	1	1	0.6	6.76	7.63	-1	3	-6.0	7.00	3.88	
5	4	26	25.96	26.66	-7	7	0	20.48	20.46	1	1	10.6	7.65	4.86	-1	3	-6.0	7.23	7.45	
5	4	27	17.38	18.84	-7	7	-1	7.81	6.89	2	1	8.4	7.12	3.33	-1	3	-6.0	7.23	7.45	
-1	5	0	44.11	45.23	4	7	0	10.64	10.44	2	1	8.4	7.12	3.33	-1	3	-6.0	7.23	7.45	
-1	5	1	17.10	10.49	4	7	-1	10.33	9.14	2	1	11.8	6.15	2.61	-2	3	-9.0	4.73	10.09	
-1	5	2	9.74	7.45	4	7	0	11.04	13.29	3	1	1.4	4.77	8.05	-2	3	-6.0	6.34	2.81	
-2	5	0	17.76	10.12	0	8	0	27.66	26.08	3	1	4.8	6.07	3.21	-2	3	-6.0	6.34	2.81	
-2	5	1	27.90	30.35	0	8	1	12.00	12.46	3	1	7.8	7.24	1.21	-2	3	-6.0	7.22	3.44	
-2	5	2	21.89	21.17	0	8	-1	17.59	14.40	3	1	6.0	7.74	4.98	-3	3	4.6	5.93	5.85	
-3	5	0	33.00	33.00	0	8	0	9.69	8.86	3	1	-7.8	7.47	1.04	-3	3	7.4	7.15	4.45	
-3	5	1	18.93	12.46	0	8	-1	12.53	12.06	4	1	7.8	7.76	10.66	-3	3	7.8	6.33	3.33	
-4	5	0	24.03	23.25	0	8	-6	12.53	12.06	4	1	7.8	7.76	10.66	-3	3	-1.8	5.08	4.49	
-4	5	1	17.44	17.39	-1	8	-1	10.92	7.76	4	1	-5.0	7.24	7.44	-3	3	-7.8	7.05	3.06	
-5	5	0	15.81	19.31	-2	8	0	19.29	19.30	4	1	-6.0	7.57	7.11	-3	3	-6.0	7.22	9.45	
-5	5	1	18.07	18.32	-2	8	-1	5.11	5.40	4	1	-4.4	7.12	3.33	-3	3	-6.0	7.22	9.45	
-5	5	2	30.66	29.62	-2	8	0	13.66	15.81	4	1	-4.4	6.73	4.20	4	3	6.0	7.00	11.42	
-5	5	3	25.34	29.36	-3	8	-1	12.57	10.95	5	1	4.0	6.85	9.06	-4	3	6.0	6.85	1.05	
-5	5	4	17.22	11.07	-4	8	0	11.16	12.04	5	1	4.8	7.63	7.47	-4	3	-3.4	6.36	9.13	
-5	5	5	7.57	7.56	-4	8	1	17.76	13.32	5	1	7.4	7.11	6.13	-4	3	-5.8	6.86	9.41	
-5	5	6	34.26	35.63	-4	8	-1	9.76	6.61	5	1	-2.4	7.19	11.28	-4	3	-6.0	7.00	1.12	
-2	5	-1	20.35	19.61	1	8	0	19.67	19.76	5	1	-7.4	7.41	2.47	-5	3	4.8	7.14	2.18	
-3	5	0	48.98	29.43	2	8	-1	9.22	7.03	5	1	-8.8	6.88	10.41	-5	3	0.8	7.13	1	

Table 2. Continued.

4	3	0	5.94	.18	-4	4	6	7.57	13.52	6	4	-2	6.89	2.12	0	9	0	7.31	2.17
4	3	9	7.43	8.80	-4	4	7	7.51	.43	0	4	-4	6.30	6.63	0	9	2	5.76	7.32
4	3	-3	6.65	6.57	-4	4	-4	7.28	8.88	0	5	6	5.50	5.99	0	9	-3	4.60	6.14
4	3	-6	7.23	7.28	-4	4	-5	7.97	10.21	0	5	-3	5.26	2.97	-1	4	1	3.32	6.68
4	3	-8	6.85	5.40	-4	4	-8	7.23	11.47	0	5	-8	11.25	12.56	-2	9	0	2.96	.45
4	3	-9	5.90	2.65	-5	4	0	7.44	4.44	-3	5	1	4.22	12.60	1	9	1	3.98	4.78
5	3	1	6.75	6.11	-5	4	3	7.12	1.64	-4	5	0	4.15	4.96	2	9	0	3.06	.79
5	3	4	7.09	4.66	-5	4	5	7.06	9.95	-5	5	-1	4.46	5.33	4	9	0	1.88	.06
5	3	6	7.36	5.66	-5	4	-2	7.05	11.99	-6	5	0	3.81	5.57					
5	3	7	7.43	7.70	-5	4	-3	7.10	2.25	-6	5	1	4.05	11.30					
5	3	-1	6.93	1.89	-6	4	1	7.45	7.57	-6	5	-1	4.07	9.47					
5	3	-4	7.23	9.81	-6	4	2	7.38	8.54	-7	5	0	4.89	15.86					
5	3	-5	7.16	11.75	-6	4	3	7.25	9.32	-7	5	-1	3.78	2.77					
6	3	0	7.23	3.67	-6	4	-1	7.04	9.20	7	5	1	4.09	9.74					
6	3	4	7.22	1.82	-6	4	-2	7.01	4.23	0	6	4	5.61	3.72					
6	3	-2	7.05	4.43	-6	4	-4	7.22	3.21	0	6	7	5.66	3.13					
6	3	-4	6.81	13.35	-6	4	-5	6.98	9.98	0	6	-7	5.45	7.37					
7	3	1	7.02	6.07	-7	4	0	6.12	5.98	-6	6	1	3.48	1.54					
7	3	2	7.05	9.75	-7	4	1	6.03	9.95	-6	6	-1	3.49	6.57					
7	3	3	7.02	8.15	1	4	3	3.61	2.59	3	6	-1	4.49	1.66					
7	3	-1	4.99	2.89	1	4	8	6.04	7.76	4	6	1	5.32	4.90					
7	3	-2	4.34	9.67	1	4	11	6.90	.26	5	6	-1	4.16	4.17					
8	3	0	5.16	6.05	1	4	-3	5.20	7.39	6	6	1	4.43	.29					
8	3	1	5.49	5.20	1	4	-6	7.05	3.04	6	6	-1	3.49	3.76					
8	3	2	6.57	.06	1	4	-8	7.57	6.51	0	7	2	4.58	1.61					
8	3	4	6.15	7.11	1	4	-9	7.34	13.23	0	7	5	5.65	7.83					
8	3	9	7.03	5.21	2	4	1	4.02	1.05	-1	7	1	4.49	6.82					
0	4	-4	5.60	4.05	2	4	3	4.36	4.10	-3	7	0	4.10	2.82					
0	4	-7	6.34	4.35	2	4	6	4.82	7.62	-4	7	1	3.94	3.84					
0	4	-10	6.92	2.11	2	4	9	7.06	8.38	-4	7	1	4.12	5.97					
-1	4	6	5.94	7.44	2	4	-7	7.56	8.96	-6	7	0	2.39	3.45					
-1	4	7	6.39	11.17	2	4	-10	6.20	2.96	5	7	1	4.47	3.61					
-1	4	8	6.44	2.54	3	4	0	5.76	1.76	5	7	-1	3.28	9.84					
-1	4	-3	5.04	4.50	3	4	5	5.92	4.76	6	7	0	2.61	1.98					
-1	4	-6	6.89	4.36	3	4	6	6.30	1.46	6	7	-1	3.26	7.29					
-1	4	-8	7.57	1.11	3	4	8	6.98	4.77	0	8	1	4.34	.42					
-2	4	4	4.01	4.01	3	4	-3	6.22	1.76	0	8	3	6.62	10.68					
-2	4	4	5.36	7.43	3	4	-5	7.36	4.22	0	8	4	6.73	2.16					
-2	4	7	6.76	2.27	3	4	-8	7.15	3.12	0	8	-1	4.23	3.47					
-2	4	9	7.12	10.35	3	4	-9	6.43	17.30	0	8	-2	5.39	1.97					
-2	4	10	6.87	6.37	4	4	1	6.13	3.36	0	8	-5	9.35	3.95					
-2	4	-4	6.10	6.17	4	4	6	6.37	.47	-1	4	0	3.98	1.38					
-2	4	-7	7.45	4.28	4	4	8	6.62	9.81	-2	4	1	4.15	4.33					
-3	4	0	5.37	1.90	4	4	-2	6.63	4.84	-3	6	0	3.57	1.55					
-3	4	3	5.83	3.51	4	4	-7	7.07	1.69	-4	6	1	3.34	7.25					
-3	4	5	6.52	4.20	5	4	0	6.96	2.99	-4	6	-1	3.26	9.14					
-3	4	6	6.85	2.97	5	4	3	6.92	4.43	-5	6	0	2.27	1.90					
-3	4	7	7.07	12.95	5	4	4	6.99	9.57	-5	6	-1	2.32	1.59					
-3	4	8	7.12	.28	5	4	5	7.07	3.21	1	6	0	4.00	.83					
-3	4	-3	5.95	2.71	5	4	6	7.12	9.46	2	6	1	4.22	6.53					
-3	4	-5	7.07	2.63	5	4	-3	7.13	1.82	3	6	0	3.65	1.62					
-3	4	-8	7.51	.28	5	4	-6	6.77	.60	4	6	-1	3.27	3.93					
-4	4	3	6.46	5.99	5	4	-7	6.05	6.09	5	6	0	2.49	4.57					
-4	4	4	6.86	8.13	6	4	1	7.10	2.46	5	6	-1	2.39	5.34					

REFINEMENT

The three-dimensional refinement was then performed by using a full-matrix least-squares program, originally written by P. K. Gantzel, R. A. Sparks and K. N. Trueblood,⁶ revised and adapted to UNIVAC 1107 by Chr. Rømming at this institute. Positional and thermal isotropic parameters for all atoms except hydrogen were refined giving an $R=0.15$ for all observed reflections. Anisotropic temperature factors for bromine were introduced and by two more cycles the R factor was reduced to 0.12.

To the intensity data set was now added the 404 nonobservable reflections with half their minimum observable F value. At this stage of the refinement the contribution of the nine hydrogen atoms was included, assuming calculated positions with C—H and N—H lengths equal to 1.0 Å. A common isotropic temperature factor of $B=3.3$ Å² was assigned to the hydrogen atoms, this being the mean value for the atoms to which they were attached. The introduction of anisotropic temperature factors for all non-hydrogen atoms had to be carried through in three steps, because of limitations in the least-squares program (maximum 100 variable parameters). The scale factors for the individual layers obtained from the last isotropic refinement were held constant during the anisotropic refinement. The final value of R was 0.099. The average shift in the atomic coordinates in terms of their standard deviations was less than 0.3 σ .

The final atomic and thermal parameters are given in Table 1. In Table 2 are listed observed and calculated structure factors. The atomic form factors used in the calculations were those given by Stewart *et al.*⁷ for hydrogen atoms and for the other atoms those given by Hanson *et al.*⁸

Table 3. Bond lengths (including the hydrogen bond) and bond angles. Estimated standard deviations in parentheses refer to the last decimal positions of respective values.

<i>i</i>	<i>i</i>	D(<i>ij</i>) Å	<i>i</i>	<i>j</i>	<i>k</i>	∠ <i>ijc</i> (°)
Br	C(1)	1.92 (2)	Br	C(1)	C(2)	113 (1)
C(1)	C(2)	1.46 (3)	Br	C(1)	C(6)	120 (1)
C(2)	C(3)	1.41 (3)	C(6)	C(1)	C(2)	127 (2)
C(3)	C(4)	1.39 (3)	C(1)	C(2)	C(3)	112 (2)
C(4)	C(5)	1.32 (2)	C(2)	C(3)	C(4)	121 (2)
C(5)	C(6)	1.40 (3)	C(3)	C(4)	C(5)	122 (2)
C(6)	C(1)	1.34 (2)	C(4)	C(5)	C(6)	123 (1)
C(4)	C(7)	1.53 (2)	C(5)	C(6)	C(1)	115 (2)
C(5)	C(8)	1.49 (2)	C(3)	C(4)	C(7)	128 (2)
C(7)	N(1)	1.36 (2)	C(6)	C(5)	C(8)	130 (2)
C(8)	N(1)	1.41 (2)	C(5)	C(4)	C(7)	110 (1)
C(7)	O(1)	1.20 (2)	C(4)	C(5)	C(8)	107 (2)
C(8)	O(2)	1.23 (2)	C(4)	C(7)	N(1)	105 (1)
N(1)	C(9)	1.49 (2)	C(5)	C(8)	N(1)	106 (1)
C(9)	C(10)	1.56 (2)	C(7)	N(1)	C(8)	111 (1)
C(10)	C(11)	1.48 (2)	C(4)	C(7)	O(1)	131 (1)
C(11)	C(12)	1.53 (2)	C(5)	C(8)	O(2)	132 (2)
C(9)	C(13)	1.53 (2)	O(1)	C(7)	N(1)	124 (1)
C(12)	N(2)	1.39 (2)	O(2)	C(8)	N(1)	121 (1)
N(2)	C(13)	1.35 (2)	C(7)	N(1)	C(9)	125 (1)
C(12)	O(3)	1.21 (2)	C(8)	N(1)	C(9)	123 (1)
C(13)	O(4)	1.21 (2)	N(1)	C(9)	C(10)	110 (1)
N(2)	O(3')	2.88 (2)	N(1)	C(9)	C(13)	109 (1)
			C(13)	C(9)	C(10)	111 (1)
			C(9)	C(10)	C(11)	108 (1)
			C(10)	C(11)	C(12)	112 (1)
			C(11)	C(12)	N(2)	114 (1)
			C(12)	N(2)	C(13)	129 (1)
			N(2)	C(13)	C(9)	116 (1)
			C(11)	C(12)	O(3)	124 (1)
			N(2)	C(12)	O(3)	121 (1)
			N(2)	C(13)	O(4)	121 (2)
			O(4)	C(13)	C(9)	122 (2)
			C(13)	N(2)	O(3')	120 (1)
			C(12)	N(2)	O(3')	110 (1)

RESULTS AND DISCUSSION

Interatomic distances and angles and their estimated standard deviations are given in Table 3. The numbering of atoms is referred to Fig. 1, which is a schematical drawing of the structure viewed along the *a*-axis. The principal axes of the thermal vibration ellipsoids for the atoms were calculated from the temperature parameters given in Table 1. Root mean square amplitudes

along the principal axes as well as the components of these along the crystal axes are given in Table 4. No attempt to interpret these values has been carried out because of their limited accuracy.

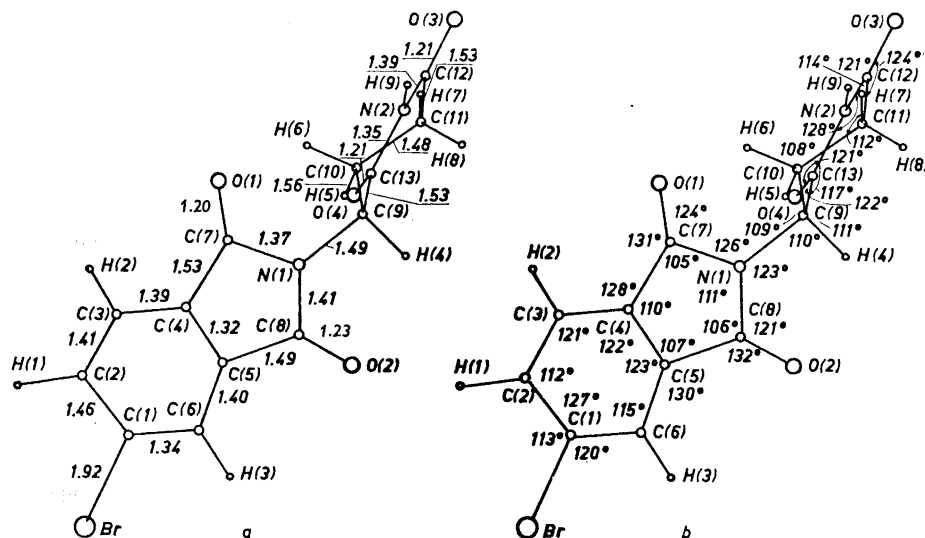


Fig. 1. Bond lengths and angles in *N*-(α -glutarimido)-4-bromophthalimide.

The distances from least-square planes are listed in Table 5. Plane I was defined by all non-hydrogen atoms of the phthalimide part. The calculation showed that this part is essentially planar. The two oxygen atoms showed the largest atom-to-plane distances, of the order 2σ , σ taken to be 0.023 Å, the mean value of e.s.d. for all distances. Plane II was calculated with O(1) and O(2) omitted and C(9) included among the atoms defining the plane. The displacements of the oxygen atoms were 0.08 Å and 0.06 Å to the same side and might possibly be significant. This distance is of the same order of magnitude as that found for the oxygen atom in the structure of meso-3,3'-di-(*p*-bromophenyl)bi-3-phthalidyl⁹ which has a planar system in some ways similar to that of phthalimide.

The atom C(9) in the glutarimide ring lies in Planes I and II well within the limits of error. C(10) and C(13) are displaced from the phthalimide plane (Plane II) by 1.29 Å and 1.24 Å, respectively, and the phthalimide plane approximately bisects the C(13)—C(9)—C(10) angle. This implies that the glutarimide ring is nearly at right angles to the phthalimide plane, a geometry also found in 5-halogeno-2-phthalimide-benzoic acid monohydrate.¹⁰ There the two planes have twisted through an angle of 88° with respect to each other about the N—C bond. The O(4) and O(1) atoms, which are at a distance of 3.20 Å apart, will represent a steric hindrance of a rotation of the glutarimide ring in one direction around the N—C bond. A limiting factor of rotation in

Table 4. R.m.s. amplitudes along the principal axes of the thermal vibrational ellipsoids and the components of these along the crystal axes.

	Axis	R.m.s. amplitude	Components of the amplitudes (Å) along the crystal axes		
	i		a_i	b_i	c_i
Br	1	0.2691	-0.0723	-0.1256	-0.2000
	2	0.2011	-0.1857	0.0830	0.0266
	3	0.1720	-0.0522	-0.1368	0.1176
C(1)	1	0.2246	-0.0508	-0.1580	-0.1235
	2	0.1658	-0.1485	0.0747	-0.0166
	3	0.0955	-0.0386	-0.0542	0.0813
C(2)	1	0.2656	-0.0669	-0.1349	-0.1915
	2	0.2359	-0.2250	0.0774	0.0408
	3	0.1995	-0.0419	-0.1614	0.1393
C(3)	1	0.3142	-0.1003	-0.1255	-0.2395
	2	0.2655	-0.2428	0.1094	0.0559
	3	0.1958	-0.0552	-0.1628	0.1257
C(4)	1	0.2591	0.0590	-0.0471	0.2473
	2	0.2357	0.2008	0.1222	-0.0674
	3	0.1263	0.0618	-0.1071	
C(5)	1	0.2122	-0.1622	-0.0449	-0.1030
	2	0.1709	-0.0948	0.1260	0.0662
	3	0.1104	-0.0391	-0.0726	0.0891
C(6)	1	0.2570	-0.1084	-0.1522	-0.1414
	2	0.2240	-0.1837	0.1344	0.0032
	3	0.1692	-0.0691	-0.0941	0.1450
C(7)	1	0.2436	-0.0971	-0.1265	-0.1540
	2	0.1818	-0.1648	0.0738	0.0477
	3	0.1050	-0.0200	-0.0804	0.0792
C(8)	1	0.2310	0.0629	0.1043	-0.2195
	2	0.1762	0.1197	-0.1298	-0.0267
	3	0.1575	0.1093	0.0824	0.0526
C(9)	1	0.2381	0.0304	0.1306	0.1750
	2	0.2014	0.2010	-0.0049	-0.0545
	3	0.1300	0.0093	-0.1102	0.0846
C(10)	1	0.2296	0.0319	-0.0163	0.2251
	2	0.2001	0.0109	0.2013	-0.0176
	3	0.1645	0.1641	-0.0098	-0.0431
C(11)	1	0.2266	-0.1373	-0.0282	0.2005
	2	0.2004	-0.1173	-0.1210	-0.0766
	3	0.1816	-0.1007	0.1454	-0.0597
C(12)	1	0.2059	0.0131	0.1349	-0.1768
	2	0.1775	-0.1631	0.0647	0.0514
	3	0.0959	-0.0394	-0.0695	-0.0446

Table 4. Continued.

C(13)	1	0.2457	-0.0750	-0.1459	-0.1532
	2	0.1790	-0.1464	0.1056	-0.0183
	3	0.1419	-0.0717	-0.0803	0.1133
O(1)	1	0.3139	0.3141	-0.0394	-0.0534
	2	0.2461	0.0138	-0.0061	0.2447
	3	0.1529	0.0171	0.1532	-0.0212
O(2)	1	0.2586	0.2519	0.0350	0.0172
	2	0.2253	0.0433	0.0327	-0.2290
	3	0.1861	0.0328	-0.1843	0.0007
O(3)	1	0.2807	0.0087	-0.0973	-0.2509
	2	0.1882	-0.1893	0.0133	0.0127
	3	0.1643	-0.0116	-0.1554	0.0793
O(4)	1	0.2912	-0.0785	0.1513	0.2268
	2	0.1920	-0.1181	-0.1431	0.0868
	3	0.1604	-0.1207	0.0709	-0.0763
N(1)	1	0.2153	-0.1546	0.0030	-0.1316
	2	0.1857	-0.1199	0.0784	0.1253
	3	0.1424	-0.0415	-0.1307	0.0650
N(2)	1	0.2621	-0.1024	0.1353	0.1962
	2	0.1794	-0.0495	-0.1554	0.1051
	3	0.1403	-0.1245	0.0083	-0.0512

Table 5. Departures from the least-square planes, calculated for the phthalimide and glutarimide parts of the molecule.

Plane I		Plane II		Plane III		Plane IV	
Å		Å		Å		Å	
Distances to atoms defining the plane.							
Br	0.037	Br	0.026	C(9)	0.004	C(9)	-0.008
C(1)	-0.007	C(1)	-0.008	C(11)	0.014	C(11)	0.009
C(2)	-0.006	C(2)	-0.005	C(12)	-0.039	C(12)	-0.009
C(3)	-0.007	C(3)	0.001	C(13)	-0.015	C(13)	0.007
C(4)	-0.003	C(4)	0.010	N(2)	-0.038	N(2)	0.001
C(5)	-0.025	C(5)	-0.013	O(3)	0.041		
C(6)	-0.025	C(6)	-0.022	O(4)	0.033		
C(7)	-0.027	C(7)	-0.007				
C(8)	-0.014	C(8)	0.002				
N(1)	-0.019	N(1)	0.002				
O(1)	0.054	C(9)	0.014				
O(2)	0.040						
Distances to atoms not defining the plane.							
	Å		Å		Å		Å
C(9)	-0.015	O(1)	0.078	C(10)	-0.67	C(10)	-0.69
		O(2)	0.056			O(3)	0.088
		C(10)	-1.29			O(4)	0.067
		C(13)	1.24				

the opposite direction will be the axial hydrogen H(6) at C(10). This rotation involves a decrease in the distance between H(6) and O(1), which in the present conformation is about 2.6 Å.

Least-square plane III was defined by all atoms in the glutarimide part, except C(10). Plane IV was defined by five ring atoms, also excluding C(10). The calculation showed corresponding displacements for the oxygen atoms as for the phthalimide part and the carbon atom C(10) was distinctly out of both planes. With only one ring atom out of the plane, the conformation could be described as a distorted half chair. C(10) has a distance of 0.69 Å from the mean plane (Plane IV) of the other atoms. This puckering is in the range of what is found for five-membered rings, as the furanoses. Bond lengths and angles in the glutarimide part agree within the experimental errors with accepted values (Sutton).¹¹ The nearly planar CO—NH—CO group is very similar both in bond lengths and angles to the corresponding planar system in dihydrothymine.¹² The angles at C(9) are all very close to tetrahedral values. A further discussion of details in the glutarimide part of the molecule is postponed until the completion of the structure analysis of glutarimide which is now in progress in our laboratory.

The central N(1)—C(9) bond is in equatorial position with respect to the glutarimide ring. Its bond length of 1.49 Å is in good agreement with the glycosidic N—C bond found in nucleosides. Bond lengths and angles in the benzene ring are normal with a possible exception for the angle C(2)—C(1)—C(6). The significance of the deviation from a normal value of 7°, which is $\approx 3.5 \sigma$, is somewhat questionable.

The mean value of the C—N bonds and C=O bonds in the phthalimide part is 1.39 Å and 1.22 Å. These values are normal and the same as for *N*-chloro-succinimide,¹³ which has the same CO—N—CO group and is in this

Table 6. Intermolecular distances less than 3.6 Å. Molecules marked with ' are translated +1 along the *x*-axis.

Atom <i>i</i>	Molecule No.	Atom <i>j</i>	Molecule No.	Distance (Å) <i>ij</i>
O(3)	I	C(2)	II	3.42
O(1)	I	C(6)	III	3.35
C(12)	I	O(2)	III	3.30
N(2)	I	O(2)	III	3.59
O(3)	I	O(2)	III	3.17
C(10)	I	O(4)	I'	3.53
C(6)	I	C(1)	IV	3.55
O(1)	I	C(3)	V	3.48
N(2)	I	O(3)	VI	2.88
C(8)	I	Br	IV	3.56
C(6)	I	C(6)	IV	3.50
C(11)	I	O(3)	VI'	3.42
Br	I	O(4)	IV	3.20
N(2)	I	O(2)	VII	3.42
O(4)	I	C(9)	VII	3.60
C(13)	I	C(13)	VII	3.55

aspect comparable to the five-membered ring in phthalimide. The C(4)—C(7) and C(5)—C(8) distances have a mean value of 1.51 Å. This bond length is significantly longer than an aromatic C—C bond and indicates that the π -electron system is limited to the aromatic ring and the CO—N—CO group. One interesting feature is the significant differences of 7° and 11° in the outer angles at C(7) and C(8). This point has also been observed in several of the structures containing the γ -lactone ring system.^{9,14}

MOLECULAR PACKING

Intermolecular distances less than 3.6 Å are listed in Table 6. There is only one intermolecular distance shorter than normal van der Waals separation, namely O(3)'—N(2) of length 2.88 Å. This clearly corresponds to a hydrogen bond. The bond is directed to the molecule situated across the center of symmetry; Fig. 2. The molecules in the crystals are consequently formed as centrosymmetrical dimers through a symmetrical pair of N—H \cdots O hydrogen bonds. A corresponding arrangement is found in the crystal structure of succinimide.¹⁵

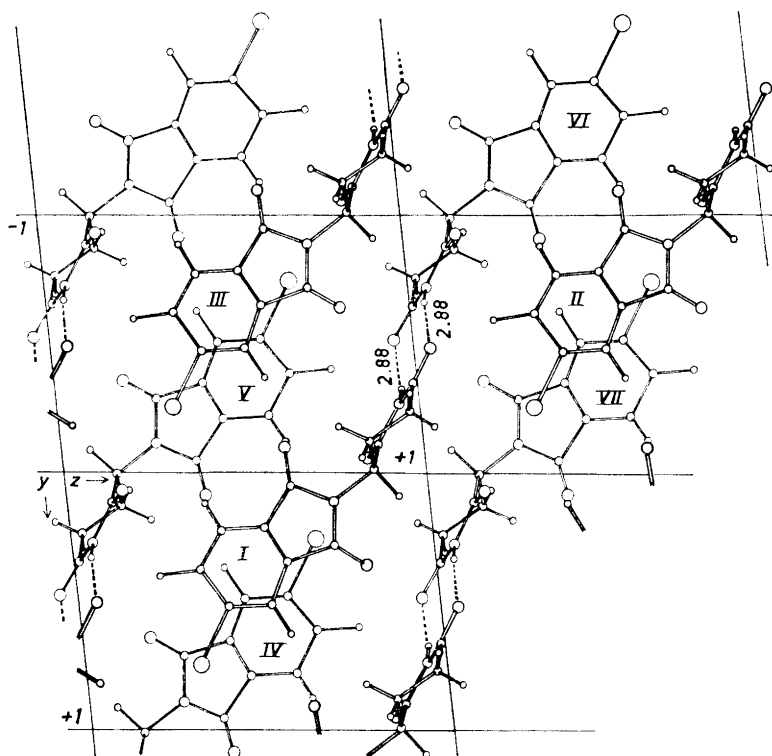


Fig. 2. The structure viewed along the a -axis. Broken lines indicate hydrogen bonds.

This system of hydrogen bonding is likely to occur in the crystals of D,L-thalidomide and might possibly be the explanation of the lower solubility of this compound compared to its optical isomers.¹⁶

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