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The Crystal and Molecular Structure of *anti*-7-Norbornenyl *p*-Bromobenzoate

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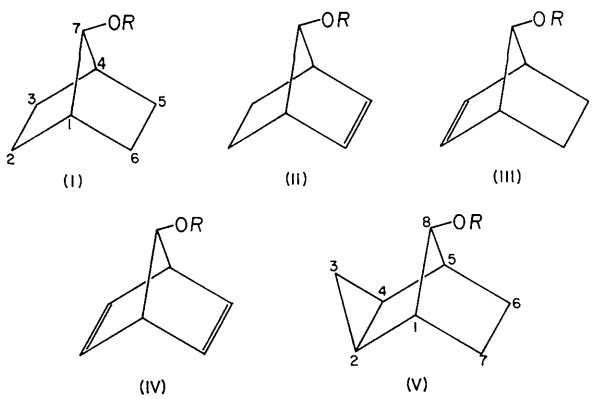
The crystal structure of *anti*-7-norbornenyl *p*-bromobenzoate, $C_{14}H_{13}O_2Br$, has been determined to provide further quantitative molecular data to assist in the interpretation of rates of solvolysis of norbornane derivatives. The crystals are monoclinic, $a=8{\cdot}81$, $b=10{\cdot}17$, $c=14{\cdot}10$ Å, $\beta=99°\ 51'$, $Z=4$, space group $P2_1/a$. The intensities of 2506 reflexions were measured with a scintillation counter using Cu $K\alpha$ radiation; 2059 reflexions had an intensity greater than background. The structure was determined by heavy-atom Patterson and Fourier methods, and the positional and anisotropic thermal parameters were refined by differential syntheses and least squares. The final R was 0·18.

The bond distances in the molecule are all normal, but the valency angles in the norbornene nucleus are all less than the tetrahedral value. The angles at the double bond are 107°; the bridgehead angle is 96°, not significantly different from the corresponding angle in the *anti*-8-tricyclo-octane nucleus. All the intermolecular distances correspond to van der Waals interactions.

Introduction

The rates of solvolysis of the compounds (I), (II), (III), (IV) (R=*p*-bromobenzenesulphonyl) are in the ratio 1: 10^4: 10^{11}: 10^{14}. It has been postulated as one possible reason for this remarkable variation in solvolytic reactivity that, since the transition state involves sp^2 hybridization at atom 7, an increase in this bridgehead angle would facilitate formation of the transition state and hence increase the solvolysis rate. No real quantitative assessment of this factor is possible owing to a lack of molecular structure data, and the angle has generally been estimated from models, or derived from empirical relations involving infrared stretching frequencies (see *e.g.* Schleyer & Nicholas, 1961).



Recently we have determined the structure of *anti*-8-tricyclo[3,2,1,$0^{2,4}$]-octyl *p*-bromobenzenesulphonate (V, R=*p*-bromobenzenesulphonyl), and found that the bridgehead angle, C(1)–C(8)–C(5), is 97° (Macdonald & Trotter, 1965); this compound has a solvolysis rate comparable to that of (I), so that an angle of about 97° would appear to be characteristic of a lack of solvolytic reactivity. We have now investigated the structure of *anti*-7-norbornenyl *p*-bromobenzoate (III, R=*p*-bromobenzoyl); if the variation of bridgehead angle controls the solvolysis rate, then the angle should be considerably greater than 97°. We find that the angle is 96°.

Experimental

Crystals of *anti*-7-norbornenyl *p*-bromobenzoate are colourless plates elongated along a with (001) developed. The density was measured by flotation in aqueous potassium iodide, and the unit-cell dimensions and space group were determined from various rotation and Weissenberg photographs, and on the General Electric Spectrogoniometer.

Crystal data (λ, Cu $K\alpha=1{\cdot}5418$ Å).

anti-7-Norbornenyl *p*-bromobenzoate, $C_{14}H_{13}O_2Br$; M.W. 293·2; m.p.70–73 °C.

Monoclinic, $a=8{\cdot}81\pm0{\cdot}02$, $b=10{\cdot}17\pm0{\cdot}02$, $c=14{\cdot}10\pm0{\cdot}03$ Å, $\beta=99°\ 51'\pm5'$.

$U=1245$ Å^3.

Table 1. *Measured and calculated structure factors*

The values listed are $\frac{1}{2}|F_o|$ and $\frac{1}{2}F_c$; unobserved reflexions, for which $\frac{1}{2}|F_o|$ is listed as 0·0, have threshold values in the range $\frac{1}{2}|F_o|=2-4$.

h	k	l	F_{obs}	F_{calc}
0	0	1	10.0	-18.3
0	0	2	35.0	-48.3
0	0	3	23.9	-24.5
0	0	4	21.4	22.2
0	0	5	19.9	18.8
0	0	6	4.1	-5.4
0	0	7	20.8	-19.4
0	0	8	30.5	29.7
0	0	9	28.0	23.6
0	0	10	38.7	-34.7
0	0	11	22.8	-19.4
0	0	12	10.9	-10.4
0	0	13	18.2	16.1
0	0	14	4.3	2.2
0	0	15	12.8	-12.0
0	0	16	4.8	-2.7
0	0	17	8.7	7.4
0	1	1	2.5	-0.8
0	1	2	5.5	1.6
0	1	3	25.6	25.9
0	1	4	18.4	22.7
0	1	5	21.5	-26.3
0	1	6	10.5	-13.0
0	1	7	26.1	25.5
0	1	8	12.7	12.0
0	1	9	7.1	-6.1
0	1	10	4.1	-3.1
0	1	11	2.5	2.2
0	1	12	12.2	11.4
0	1	13	4.8	-4.4
0	1	14	11.0	-9.3
0	1	15	2.9	1.4
0	1	16	5.4	5.0
0	1	17	1.9	-0.3
0	2	0	66.2	64.3
0	2	0	70.3	65.0
0	2	1	26.5	20.5
0	2	2	35.6	-30.1
0	2	3	36.5	-31.5
0	2	4	41.2	39.3
0	2	5	11.8	9.8
0	2	6	22.7	-24.2
0	2	7	5.6	4.6
0	2	8	7.7	8.1
0	2	9	7.4	7.2
0	2	10	6.4	-7.1
0	2	11	18.6	-16.4
0	2	12	1.0	0.2
0	2	13	9.7	8.8
0	2	14	2.1	0.6
0	2	15	9.6	-9.5
0	2	16	2.6	0.5
0	2	17	3.9	3.2
0	3	1	52.6	-45.1
0	3	2	8.5	-4.4
0	3	3	46.3	39.7
0	3	4	11.2	9.2
0	3	5	8.3	-10.6
0	3	6	28.1	-28.7
0	3	7	31.5	35.6
0	3	8	20.6	25.8
0	3	9	20.2	-21.2
0	3	10	26.0	-26.1
0	3	11	24.1	22.5
0	3	12	14.3	14.0
0	3	13	3.4	-4.3
0	3	14	12.1	-10.8
0	3	15	3.0	-1.7
0	3	16	8.6	8.3
0	4	0	18.2	-13.1
0	4	1	12.9	-8.0
0	4	2	24.2	16.5
0	4	3	9.2	5.7
0	4	4	7.0	-6.3
0	4	5	13.5	-12.1
0	4	6	6.8	6.4
0	4	7	10.0	7.2
0	4	8	4.8	-6.3
0	4	9	1.7	-2.8
0	4	10	0.0	2.0
0	4	11	0.0	-0.2
0	4	12	5.8	4.0
0	4	13	4.9	-4.9
0	4	14	1.9	-1.4
0	4	15	3.9	3.3
0	4	16	1.7	0.0
0	5	1	32.5	-25.9
0	5	2	24.5	-20.7
0	5	3	11.3	10.4
0	5	4	21.9	19.4
0	5	5	18.7	-16.3
0	5	6	7.9	-8.1
0	5	7	18.5	19.2
0	5	8	5.9	4.7
0	5	9	14.8	-18.5
0	5	10	14.2	-15.1
0	5	11	10.4	11.7
0	5	12	10.0	9.2
0	5	13	2.0	0.9
0	5	14	8.2	-8.5
0	5	15	2.5	1.9
0	6	0	35.4	-28.9
0	6	1	3.6	-3.7
0	6	2	52.0	42.8
0	6	3	6.9	4.9
0	6	4	24.9	-21.1
0	6	5	8.6	-9.0
0	6	6	10.4	11.1
0	6	7	13.4	13.7
0	6	8	6.0	-8.5
0	6	9	9.8	-11.9
0	6	10	2.8	2.6
0	6	11	9.3	8.4
0	6	12	6.2	-5.6
0	6	13	8.0	-8.2
0	6	14	2.1	-1.3
0	6	15	4.8	5.3
0	7	1	6.0	-6.4
0	7	2	0.9	1.2
0	7	3	6.5	-6.9
0	7	4	6.3	5.3
0	7	5	4.8	4.9
0	7	6	3.4	3.0
0	7	7	0.0	-0.9
0	7	8	2.1	-2.7
0	7	9	0.0	-1.8
0	7	10	2.6	1.0
0	7	11	3.8	2.7
0	7	12	2.3	-2.0
0	7	13	0.0	0.5
0	7	14	2.1	-0.6
0	8	0	20.4	-18.1
0	8	1	2.1	-2.7
0	8	2	22.8	20.3
0	8	3	8.1	8.9
0	8	4	11.4	-11.3
0	8	5	3.8	-2.3
0	8	6	8.3	8.6
0	8	7	5.0	2.9
0	8	8	7.9	-10.6
0	8	9	0.0	-1.8
0	8	10	2.2	1.0
0	8	11	5.9	6.9
0	8	12	2.4	-0.4
0	8	13	8.3	-8.7
0	9	1	11.5	10.6
0	9	2	7.8	-7.4
0	9	3	5.1	-4.2
0	9	4	2.1	-1.6
0	9	5	9.5	10.7
0	9	6	9.4	9.2
0	9	7	0.0	-2.0
0	9	8	8.6	-12.0
0	9	9	8.6	10.8
0	9	10	1.9	0.4
0	9	11	2.1	-0.1
0	9	12	3.5	-4.4
0	10	0	0.0	-3.2
0	10	1	2.4	-0.6
0	10	2	2.8	2.6
0	10	3	0.0	-2.7
0	10	4	0.0	0.6
0	10	5	3.7	2.8
0	10	6	2.2	-1.1
0	10	7	1.6	-0.7
0	10	8	1.9	-2.3
0	10	9	2.2	0.3
0	10	10	1.8	2.4
0	11	1	2.1	2.1
0	11	2	3.4	3.7
0	11	3	2.3	-6.2
0	11	4	2.2	-3.2
0	11	5	5.4	6.4
0	11	6	3.1	3.1
0	11	7	1.8	-3.6
0	11	8	0.0	-2.2
0	12	0	2.6	2.9
0	12	1	1.5	2.3
0	12	2	5.5	-7.1
0	12	3	1.7	1.7
0	12	4	0.0	0.1
0	12	5	2.1	3.6
1	1	-17	2.1	1.3
1	1	-16	12.1	-10.4
1	1	-15	3.7	-3.3
1	1	-14	15.1	14.5
1	1	-13	6.3	5.6
1	1	-12	17.1	-17.0
1	1	-11	13.5	-13.7
1	1	-10	23.2	21.8
1	1	-9	7.9	7.9
1	1	-8	36.3	-39.0
1	1	-7	51.9	-53.1
1	1	-6	37.3	37.5
1	1	-5	31.2	39.1
1	1	-4	12.8	-16.5
1	1	-3	40.0	-38.6
1	1	-2	33.3	-24.0
1	1	-1	5.2	5.6
1	1	0	25.6	21.6
1	1	1	38.5	-36.6
1	1	2	35.5	27.7
1	1	3	66.3	66.5
1	1	4	21.8	26.8
1	1	5	29.1	-34.8
1	1	6	26.3	-30.9
1	1	7	34.8	33.8
1	1	8	27.0	27.6
1	1	9	10.3	-8.4
1	1	10	16.8	-15.4
1	1	11	25.1	22.7
1	1	12	4.5	2.2
1	1	13	7.5	-7.6
1	1	14	15.1	-14.6
1	1	15	2.1	1.7
1	1	16	8.4	8.8
1	2	-17	7.2	-6.5
1	2	-16	1.9	1.4
1	2	-15	11.8	11.4
1	2	-14	0.0	0.5
1	2	-13	11.8	-10.2
1	2	-12	3.2	1.2
1	2	-11	11.9	17.0
1	2	-10	14.9	14.7
1	2	-9	12.5	-13.0
1	2	-8	13.8	-15.4
1	2	-6	14.1	17.5
1	2	-5	26.3	-24.9
1	2	-4	45.4	-39.1
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1	2	-1	2.3	1.3
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1	2	1	20.3	15.8
1	2	2	37.0	30.5
1	2	3	8.1	-7.8
1	2	4	36.0	-31.4
1	2	5	9.7	-13.0
1	2	6	16.1	20.3
1	2	7	24.2	25.4
1	2	8	17.8	-17.5
1	2	9	18.6	-19.9
1	2	10	14.2	14.0
1	2	11	11.8	9.2
1	2	12	9.8	8.6
1	2	13	10.7	-11.1
1	2	14	2.5	0.6
1	2	15	9.1	9.3
1	2	16	0.8	0.1
1	3	-16	1.7	-0.2
1	3	-15	2.3	-2.2
1	3	-14	3.0	3.4
1	3	-13	4.7	-2.8
1	3	-12	4.9	3.4
1	3	-11	15.1	-14.3
1	3	-10	13.0	12.6
1	3	-9	0.0	-0.0
1	3	-8	5.1	-5.8
1	3	-7	0.0	-0.1
1	3	-6	0.0	-0.1
1	3	-5	6.2	6.3
1	3	-4	0.7	-2.7
1	3	-3	5.9	-4.3
1	3	-2	5.8	-5.6
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1	3	2	7.0	-5.4
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1	3	9	1.7	-3.9
1	3	10	1.7	-2.9
1	3	11	4.2	2.0
1	3	12	4.5	-0.3
1	3	13	2.4	-1.4
1	3	14	0.0	-2.3
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1	4	-14	3.1	1.5
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1	4	-12	6.2	-3.0
1	4	-11	6.8	5.0
1	4	-10	7.2	7.6
1	4	-9	12.2	-16.2
1	4	-8	6.0	-10.1
1	4	-7	17.6	19.3
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1	4	2	50.6	40.2
1	4	3	1.1	2.1
1	4	4	59.7	-53.6
1	4	5	3.5	-5.0
1	4	6	23.7	24.2
1	4	7	20.2	21.6
1	4	8	5.7	-7.0
1	4	9	12.9	-16.0
1	4	10	3.1	-0.9
1	4	11	15.3	14.8
1	4	12	7.2	-6.4
1	4	13	9.1	-9.2
1	4	14	4.5	3.6
1	4	15	8.2	9.3
1	5	-15	1.7	-1.3
1	5	-14	5.5	-5.2
1	5	-13	0.0	-0.0
1	5	-12	11.0	10.9
1	5	-11	0.0	3.2
1	5	-10	14.0	-16.8
1	5	-9	11.1	-14.8
1	5	-8	14.9	17.3
1	5	-7	15.0	14.7
1	5	-6	7.1	-10.8
1	5	-5	13.5	-12.7
1	5	-4	5.0	-4.4
1	5	-3	24.6	20.4
1	5	-2	5.2	-6.1
1	5	-1	23.2	-20.0
1	5	0	10.0	8.0
1	5	1	20.7	16.5
1	5	2	15.1	13.9
1	5	3	19.9	-19.4
1	5	4	5.2	-4.5
1	5	5	8.3	7.3
1	5	6	17.7	17.5
1	5	7	15.8	-15.2
1	5	8	4.8	-7.5
1	5	9	10.7	12.0
1	5	10	8.7	8.2
1	5	11	6.2	-6.4
1	5	12	10.6	-10.0
1	5	13	0.0	3.7
1	5	14	1.4	1.9
1	5	15	1.4	2.6
1	6	-15	2.4	1.2
1	6	-14	1.4	3.4
1	6	-13	1.9	-3.9
1	6	-12	0.0	-2.2
1	6	-11	4.8	5.8
1	6	-10	1.7	-5.8
1	6	-9	3.4	-0.3
1	6	-8	6.4	-6.9
1	6	-7	5.9	5.8
1	6	-6	10.0	10.7
1	6	-5	3.1	4.3
1	6	-4	27.5	-22.8
1	6	-3	6.6	5.5
1	6	-2	3.4	4.1
1	6	-1	7.0	6.1
1	6	0	9.9	-9.7
1	6	1	12.4	11.7
1	6	2	15.8	12.1
1	6	3	6.5	-5.2
1	6	4	12.9	-11.1
1	6	5	3.0	-3.3
1	6	6	10.0	10.0
1	6	7	4.0	3.9
1	6	8	2.9	0.9
1	6	9	5.8	-5.3
1	6	10	3.9	2.9
1	6	11	3.1	-0.2
1	6	12	2.6	-2.9
1	6	13	2.6	-3.1
1	6	14	3.3	1.9
1	7	-14	5.2	-6.3
1	7	-13	0.0	-2.6
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1	7	-11	2.0	3.9
1	7	-10	11.1	-13.2
1	7	-9	12.7	-16.4
1	7	-8	15.8	15.5
1	7	-7	10.2	13.1
1	7	-6	17.9	-17.0
1	7	-5	17.5	-16.7
1	7	-4	0.0	2.6
1	7	-3	12.4	12.0
1	7	-2	0.0	-0.8
1	7	-1	20.1	-15.4
1	7	0	1.4	-1.7
1	7	1	26.0	21.7
1	7	2	5.2	4.3
1	7	3	13.4	-14.6
1	7	4	6.4	-7.1
1	7	5	21.6	19.3
1	7	6	14.9	14.3
1	7	7	10.6	-10.8
1	7	8	9.1	-9.9
1	7	9	11.1	14.7
1	7	10	3.6	5.1
1	7	11	2.1	-4.5
1	7	12	7.4	-6.5
1	7	13	1.7	3.2
1	7	14	4.3	6.0
1	8	-13	3.9	4.5
1	8	-12	0.0	1.3
1	8	-11	0.0	-2.7
1	8	-10	0.0	0.7
1	8	-9	0.0	1.2
1	8	-8	0.0	3.7
1	8	-7	4.4	-4.4
1	8	-6	1.8	-2.3
1	8	-5	6.1	5.3
1	8	-4	5.4	4.8
1	8	-3	6.2	-7.8
1	8	-2	16.2	-15.8
1	8	-1	1.8	4.4
1	8	0	8.0	7.2
1	8	1	4.7	5.6
1	8	2	17.8	-15.2
1	8	3	0.0	-1.5
1	8	4	2.1	2.3
1	8	5	1.7	5.5
1	8	6	5.3	-5.5
1	8	7	4.5	-3.8
1	8	8	4.6	4.4
1	8	9	6.8	7.3
1	8	10	0.0	-0.6
1	8	11	1.0	-3.6
1	8	12	0.0	-0.5
1	9	-11	0.0	1.4
1	9	-10	0.0	-3.2
1	9	-9	0.0	-0.6
1	9	-8	0.0	2.9
1	9	-7	3.1	4.6
1	9	-6	5.9	-6.2
1	9	-5	8.6	-8.2
1	9	-4	1.7	2.7
1	9	-3	3.6	5.9
1	9	-2	0.0	0.1
1	9	-1	0.0	-2.0
1	9	0	0.0	0.8
1	9	1	0.0	1.6
1	9	2	2.2	1.3
1	9	3	6.6	-8.4
1	9	4	4.5	-0.7
1	9	5	6.2	8.3
1	9	6	3.2	7.1
1	9	7	3.7	-5.5
1	9	8	4.0	-4.0
1	9	9	1.8	2.9
1	9	10	1.7	0.3
1	9	11	3.6	-1.7
1	10	-9	0.0	1.1
1	10	-8	4.5	7.7
1	10	-7	2.3	-4.5
1	10	-6	3.2	-5.2
1	10	-5	4.8	4.0
1	10	-4	0.0	4.1
1	10	-3	2.6	-5.0
1	10	-2	12.2	-12.7
1	10	-1	3.8	3.8
1	10	0	7.3	9.3
1	10	1	4.2	5.6
1	10	2	16.3	-18.0
1	10	3	0.0	1.7
1	10	4	1.7	3.1
1	10	5	1.6	2.8
1	10	6	1.5	-4.9
1	10	7	1.4	-3.0
1	10	8	3.4	5.3
1	10	9	3.1	5.0
1	10	10	2.4	-1.2
1	11	-8	0.0	-2.4
1	11	-7	0.0	-0.5
1	11	-6	0.0	-0.4
1	11	-5	0.0	4.0
1	11	-4	0.0	-0.8
1	11	-3	0.0	0.3
1	11	-2	1.7	-2.3
1	11	-1	4.3	3.8
1	11	0	1.7	-0.4
1	11	1	4.4	-3.3
1	11	2	1.9	2.1
1	11	3	1.9	-2.2
1	11	4	1.6	2.7
1	11	5	0.0	-1.1
1	11	7	0.0	-1.4
1	12	-4	0.0	3.8
1	12	-3	0.0	2.9
1	12	-2	0.0	-6.5
1	12	-1	2.3	4.5
1	12	0	1.6	0.9
1	12	1	1.9	-1.2
1	12	2	2.1	-3.0
1	12	3	1.4	0.9
1	12	4	2.0	2.8
2	0	-17	7.6	-6.3
2	0	-16	3.2	2.3
2	0	-15	19.9	17.7
2	0	-14	5.4	2.1
2	0	-13	16.2	-12.2
2	0	-12	5.9	-5.2
2	0	-11	28.0	23.9
2	0	-10	16.2	14.1
2	0	-9	11.1	-11.8
2	0	-8	13.2	-19.5
2	0	-7	7.7	-1.2
2	0	-6	36.9	35.4
2	0	-5	43.0	-42.5
2	0	-4	54.7	-66.3
2	0	-3	27.7	35.4
2	0	-2	77.6	52.0
2	0	-1	23.1	-37.1
2	0	0	101.2	-98.3
2	0	1	4.1	-2.9
2	0	2	19.7	27.0
2	0	3	11.4	14.5
2	0	4	41.3	-41.5
2	0	5	0.8	-1.2
2	0	6	36.0	33.9
2	0	7	13.6	19.0
2	0	8	21.8	-20.1
2	0	9	21.9	-22.0
2	0	9	21.9	-22.0
2	0	10	18.1	17.2
2	0	11	24.6	22.9
2	0	12	9.0	6.9
2	0	13	13.0	-13.2
2	0	14	3.2	-0.9
2	0	15	9.7	10.4
2	0	16	3.1	-2.2
2	1	-17	1.4	0.1
2	1	-15	2.4	-0.9
2	1	-14	5.8	-5.9
2	1	-13	2.4	-2.8
2	1	-12	11.4	10.6
2	1	-11	2.6	4.4
2	1	-10	10.4	-9.6
2	1	-9	1.0	-6.7
2	1	-8	21.1	20.3
2	1	-7	27.0	25.8
2	1	-6	24.1	-25.2
2	1	-5	1.9	-2.9
2	1	-4	3.8	5.3
2	1	-3	15.6	15.2
2	1	-2	6.6	7.8
2	1	-1	2.0	-3.4
2	1	0	49.1	-36.9
2	1	1	60.5	52.5
2	1	2	14.6	-12.3
2	1	3	37.2	-35.0
2	1	4	3.1	3.2
2	1	5	2.8	-3.8
2	1	6	11.4	10.5
2	1	7	5.7	-6.2
2	1	8	14.9	-14.5
2	1	9	5.0	5.3
2	1	10	12.9	12.5
2	1	11	20.1	-18.6
2	1	12	5.9	3.3
2	1	13	1.2	0.4
2	1	14	4.5	4.9
2	1	15	2.0	2.0
2	1	16	3.0	-3.5
2	2	-17	7.8	-6.4
2	2	-16	3.0	2.4
2	2	-15	10.5	10.6
2	2	-14	0.0	1.5
2	2	-13	6.9	-6.1
2	2	-12	3.0	-1.5
2	2	-11	3.7	2.3
2	2	-10	15.1	14.3
2	2	-9	14.3	-14.5
2	2	-8	13.1	-14.7
2	2	-7	12.2	14.7
2	2	-6	13.1	12.8
2	2	-5	22.3	-23.2
2	2	-4	29.1	-26.6
2	2	-3	0.0	1.2
2	2	-2	29.8	24.1
2	2	-1	19.3	11.0
2	2	0	88.2	-74.8
2	2	1	44.0	30.5
2	2	2	17.4	16.3
2	2	3	1.0	2.7
2	2	4	25.6	-25.0
2	2	5	8.9	7.4
2	2	6	6.9	8.5
2	2	7	16.2	16.7
2	2	8	6.8	-7.2
2	2	9	16.1	-14.6
2	2	10	10.4	10.9
2	2	11	13.0	13.0
2	2	12	4.3	-2.0
2	2	13	6.6	-7.6
2	2	14	4.2	2.2
2	2	15	4.5	3.5
2	2	16	1.0	0.6
2	3	-16	12.1	8.4
2	3	-15	0.0	1.8
2	3	-14	16.6	-15.8
2	3	-13	2.6	0.7
2	3	-12	26.4	23.8
2	3	-11	7.9	7.5
2	3	-10	24.9	-23.6
2	3	-9	15.8	-17.0
2	3	-8	23.1	26.5
2	3	-7	29.8	33.2
2	3	-6	19.3	-20.7
2	3	-5	28.6	-27.2
2	3	-4	27.7	22.3
2	3	-4	29.6	22.5
2	3	-3	37.6	30.9

Table 1 (*cont.*)

continued:—

h	k	l	F_{obs}	F_{calc}
2	3	-2	16.7	-11.7
2	3	-1	54.2	-39.9
2	3	0	17.5	-12.1
2	3	1	56.4	41.6
2	3	2	26.1	22.5
2	3	3	40.4	-34.4
2	3	4	36.6	-29.4
2	3	5	23.5	23.4
2	3	6	12.0	13.6
2	3	7	23.2	-29.8
2	3	8	6.6	-9.0
2	3	9	18.6	19.4
2	3	10	11.0	11.5
2	3	11	15.2	-13.9
2	3	12	9.2	-8.6
2	3	13	4.5	1.7
2	3	14	6.8	9.2
2	3	15	1.3	0.6
2	4	-16	2.1	-1.1
2	4	-15	3.8	-3.4
2	4	-14	0.0	1.5
2	4	-13	2.8	1.5
2	4	-12	4.7	2.6
2	4	-11	3.2	-0.7
2	4	-10	7.2	-9.0
2	4	-9	5.2	5.7
2	4	-8	0.0	0.0
2	4	-7	9.2	-10.9
2	4	-6	0.0	-0.9
2	4	-5	9.3	10.0
2	4	-4	1.6	-2.3
2	4	-3	10.8	8.9
2	4	-2	27.3	-22.3
2	4	-1	0.0	1.1
2	4	0	14.5	14.1
2	4	1	1.3	2.9
2	4	2	13.8	-12.3
2	4	3	12.8	10.3
2	4	4	12.8	10.4
2	4	5	1.4	1.0
2	4	6	1.5	1.0
2	4	7	12.2	-13.4
2	4	8	5.1	7.7
2	4	9	8.1	6.9
2	4	10	4.2	1.6
2	4	11	5.5	-4.4
2	4	12	0.0	-0.9
2	4	13	3.3	1.8
2	4	14	3.1	-0.6
2	4	15	2.2	-0.1
2	5	-15	0.0	0.8
2	5	-14	7.0	-7.9
2	5	-13	0.0	-0.0
2	5	-12	8.6	8.7
2	5	-11	13.7	14.2
2	5	-10	18.7	-21.4
2	5	-9	9.7	-12.2
2	5	-8	12.7	13.3
2	5	-7	10.4	10.5
2	5	-6	17.3	-16.8
2	5	-5	5.2	-5.3
2	5	-4	6.6	5.3
2	5	-3	14.1	12.6
2	5	-2	0.0	-0.4
2	5	-1	49.2	-38.7
2	5	0	2.2	0.4
2	5	1	29.0	24.0
2	5	2	16.6	13.5
2	5	3	14.8	-14.1
2	5	4	8.6	-8.7
2	5	5	14.5	13.9
2	5	6	7.9	7.4
2	5	7	17.1	-17.5
2	5	8	0.0	-3.2
2	5	9	12.9	16.1
2	5	10	9.9	8.7
2	5	11	6.3	-6.5
2	5	12	7.0	-6.1
2	5	13	0.0	1.0
2	5	14	1.3	4.1
2	6	-15	10.1	-8.9
2	6	-14	0.0	-1.3
2	6	-13	12.1	11.4
2	6	-12	3.1	2.3
2	6	-11	0.0	-1.2
2	6	-10	2.7	-4.0
2	6	-9	2.6	4.5
2	6	-8	9.7	10.2
2	6	-7	16.2	-15.6
2	6	-6	20.1	-18.6
2	6	-5	13.8	13.7
2	6	-4	26.4	24.3
2	6	-3	14.0	-12.0
2	6	-2	39.2	-31.5
2	6	-1	1.3	2.8
2	6	0	20.2	17.7
2	6	1	7.8	-5.2
2	6	2	30.7	-24.8
2	6	3	4.0	1.8
2	6	4	21.5	22.5
2	6	5	9.7	8.3
2	6	6	14.5	-15.9
2	6	7	11.8	-13.3
2	6	8	9.5	11.0
2	6	9	10.0	11.9
2	6	10	0.0	0.5
2	6	11	2.6	-8.1
2	6	12	2.7	1.8
2	6	13	8.3	8.4
2	6	14	3.2	-0.3
2	7	-14	0.0	-1.1
2	7	-13	0.0	-2.9
2	7	-12	0.0	-2.2
2	7	-11	0.0	3.5
2	7	-10	1.7	-0.7
2	7	-9	6.9	9.4
2	7	-8	9.7	-8.7
2	7	-7	0.0	1.6
2	7	-6	0.0	-0.3
2	7	-5	0.0	-1.6
2	7	-4	3.1	3.3
2	7	-3	0.0	-0.4
2	7	-2	4.8	-4.0
2	7	-1	8.0	6.1
2	7	0	2.0	-0.5
2	7	1	9.3	-8.0
2	7	2	2.7	2.3
2	7	3	3.4	-3.6
2	7	4	0.0	1.6
2	7	5	0.0	3.9
2	7	6	4.1	-2.5
2	7	7	5.9	-3.6
2	7	8	5.8	3.7
2	7	9	1.8	-1.8
2	7	10	0.0	1.3
2	7	11	0.0	-1.4
2	7	12	0.0	3.9
2	7	13	0.7	-1.6
2	8	-13	4.1	5.9
2	8	-12	2.4	3.0
2	8	-11	4.7	-6.2
2	8	-10	0.0	-2.0
2	8	-9	0.0	1.1
2	8	-8	8.8	10.6
2	8	-7	7.9	-7.8
2	8	-6	5.9	-7.4
2	8	-5	7.2	3.5
2	8	-4	12.8	11.7
2	8	-3	10.9	-11.2
2	8	-2	19.1	-16.9
2	8	-1	1.5	1.5
2	8	0	16.8	15.1
2	8	1	2.4	2.7
2	8	2	23.9	-22.3
2	8	3	6.9	6.5
2	8	4	3.5	3.5
2	8	5	1.2	1.3
2	8	6	4.6	-5.8
2	8	7	1.7	-2.9
2	8	8	4.5	6.0
2	8	9	7.5	9.9
2	8	10	0.0	-5.2
2	8	11	3.4	-5.9
2	8	12	3.1	2.9
2	9	-12	5.7	-6.7
2	9	-11	0.0	0.3
2	9	-10	0.0	7.8
2	9	-9	1.6	4.4
2	9	-8	2.1	-5.7
2	9	-7	2.5	-5.0
2	9	-6	8.5	6.1
2	9	-5	9.3	11.3
2	9	-4	0.0	-2.0
2	9	-3	11.0	-10.8
2	9	-2	0.0	3.1
2	9	-1	1.1	5.9
2	9	0	4.1	1.0
2	9	1	9.7	-7.5
2	9	2	2.9	-6.0
2	9	3	7.8	7.5
2	9	4	4.2	4.0
2	9	5	5.2	-7.9
2	9	6	4.3	-7.1
2	9	7	3.0	3.9
2	9	8	4.1	3.2
2	9	9	4.8	-5.4
2	9	10	2.5	-0.4
2	9	11	2.7	1.2
2	10	-10	0.0	-1.5
2	10	-9	0.0	0.9
2	10	-8	0.0	0.1
2	10	-7	0.0	1.9
2	10	-6	0.0	-0.8
2	10	-5	4.5	-3.3
2	10	-4	2.1	4.3
2	10	-3	0.0	-2.5
2	10	-2	3.2	-3.0
2	10	-1	4.6	4.0
2	10	0	0.0	2.5
2	10	1	2.1	-2.4
2	10	2	5.7	4.9
2	10	3	2.6	-4.8
2	10	4	1.1	1.2
2	10	5	0.0	-1.5
2	10	6	0.0	0.1
2	10	7	1.0	2.3
2	10	8	2.0	3.5
2	10	9	1.9	-4.2
2	11	-8	0.0	-5.7
2	11	-7	0.0	-1.4
2	11	-6	0.0	4.3
2	11	-5	0.0	4.9
2	11	-4	0.0	-4.0
2	11	-3	0.0	-2.9
2	11	-2	1.7	-2.9
2	11	-1	4.8	5.4
2	11	0	2.1	1.3
2	11	1	4.8	-4.5
2	11	2	3.1	-2.0
2	11	3	5.8	6.4
2	11	4	1.2	-0.1
2	11	5	0.9	-4.9
2	11	7	0.6	1.8
2	12	-4	0.0	-2.6
2	12	-3	0.0	2.1
2	12	-2	1.3	3.8
2	12	-1	1.5	-1.9
2	12	0	2.5	-2.0
2	12	1	0.0	-0.4
2	12	2	2.7	3.9
2	12	3	1.1	-0.2
2	12	4	0.0	-1.8
3	1	-17	1.1	-0.5
3	1	-16	10.5	9.5
3	1	-15	1.5	1.1
3	1	-14	15.9	-15.2
3	1	-13	4.0	3.0
3	1	-12	24.5	22.6
3	1	-11	11.4	14.0
3	1	-10	24.5	-22.5
3	1	-9	6.9	-7.8
3	1	-8	26.3	27.0
3	1	-7	27.9	28.7
3	1	-6	25.3	-26.1
3	1	-5	13.3	-18.6
3	1	-4	19.8	21.5
3	1	-3	27.7	27.1
3	1	-2	6.5	-1.6
3	1	-1	69.5	-53.1
3	1	0	31.2	-23.9
3	1	1	50.1	46.1
3	1	2	7.5	4.9
3	1	3	42.5	-44.4
3	1	4	17.1	-20.1
3	1	5	18.2	20.7
3	1	6	8.0	10.4
3	1	7	20.9	-21.6
3	1	8	12.3	-11.8
3	1	9	14.2	12.6
3	1	10	13.6	12.3
3	1	11	16.3	-15.3
3	1	12	5.2	2.0
3	1	13	3.6	1.1
3	1	14	9.9	9.4
3	1	15	0.0	0.4
3	2	-17	5.5	5.2
3	2	-16	2.3	-1.9
3	2	-15	12.7	-11.6
3	2	-14	1.1	-0.2
3	2	-13	9.5	8.1
3	2	-12	6.6	6.0
3	2	-11	16.5	-16.8
3	2	-10	11.4	-12.2
3	2	-9	12.8	13.4
3	2	-8	14.5	16.5
3	2	-7	3.9	-6.0
3	2	-6	26.5	-29.6
3	2	-5	27.9	25.2
3	2	-4	35.2	31.5
3	2	-3	10.7	-10.0
3	2	-2	34.4	-30.4
3	2	-1	35.8	27.7
3	2	0	38.5	30.8
3	2	1	2.5	1.6
3	2	2	31.8	-28.5
3	2	3	0.9	-1.7
3	2	4	34.9	35.9
3	2	5	5.0	-1.4
3	2	6	11.8	-13.6
3	2	7	16.6	-18.0
3	2	8	10.7	10.5
3	2	9	14.5	13.6
3	2	10	5.9	-5.1
3	2	11	19.7	-18.1
3	2	12	3.8	4.7
3	2	13	6.2	6.2
3	2	14	3.4	-1.6
3	2	15	5.5	-5.5
3	3	-16	2.3	1.1
3	3	-15	2.3	1.4
3	3	-14	1.6	-2.6
3	3	-13	0.0	2.0
3	3	-12	9.7	6.4
3	3	-11	7.9	6.9
3	3	-10	7.1	-7.6
3	3	-9	1.7	-1.1
3	3	-8	1.6	-3.0
3	3	-7	3.2	4.4
3	3	-6	5.8	-6.0
3	3	-5	0.0	-1.4
3	3	-4	2.8	3.7
3	3	-3	3.7	-2.2
3	3	-2	0.0	-3.8
3	3	-1	28.0	-20.1
3	3	0	3.2	-0.6
3	3	1	1.9	1.6
3	3	2	4.0	3.4
3	3	3	4.8	-3.1
3	3	4	5.5	-6.4
3	3	5	12.4	12.9
3	3	6	4.3	-2.6
3	3	7	6.3	-7.0
3	3	8	2.4	3.0
3	3	9	5.1	5.1
3	3	10	3.4	1.6
3	3	11	4.6	2.3
3	3	12	1.1	-1.1
3	3	13	3.4	0.5
3	3	14	2.5	3.5
3	3	15	2.8	-3.3
3	4	-16	1.4	-0.9
3	4	-15	12.8	-11.2
3	4	-14	1.7	-2.5
3	4	-13	13.8	12.6
3	4	-12	5.7	3.2
3	4	-11	7.2	-6.1
3	4	-10	8.2	-9.5
3	4	-9	9.6	11.9
3	4	-8	18.3	22.5
3	4	-7	20.8	-21.3
3	4	-6	26.3	-24.1
3	4	-5	19.8	16.8
3	4	-4	38.1	32.7
3	4	-3	13.8	-12.0
3	4	-2	26.9	-23.6
3	4	-1	2.1	-1.5
3	4	0	54.0	43.5
3	4	1	37.8	-30.2
3	4	2	27.0	-23.3
3	4	3	6.5	3.4
3	4	4	26.2	24.6
3	4	5	9.6	9.5
3	4	6	14.9	-15.5
3	4	7	16.9	-20.3
3	4	8	9.1	10.2
3	4	9	14.2	15.4
3	4	10	10.6	-10.6
3	4	11	6.4	-5.5
3	4	12	2.4	-2.1
3	4	13	10.6	11.4
3	4	14	3.5	-3.8
3	5	-15	2.3	1.9
3	5	-14	7.9	8.0
3	5	-13	1.2	-1.0
3	5	-12	13.5	-13.3
3	5	-11	2.1	4.2
3	5	-10	6.0	6.3
3	5	-9	13.7	18.5
3	5	-8	16.6	-16.5
3	5	-7	8.1	-9.9
3	5	-6	12.1	11.6
3	5	-5	8.4	7.9
3	5	-4	16.4	-12.3
3	5	-3	14.2	-13.4
3	5	-2	1.7	2.2
3	5	-1	27.5	19.2
3	5	0	14.5	11.5
3	5	1	46.4	-35.6
3	5	2	8.3	-6.9
3	5	3	10.4	7.7
3	5	4	10.4	11.1
3	5	5	9.5	-8.5
3	5	6	4.3	-4.8
3	5	7	3.6	7.2
3	5	8	7.2	7.8
3	5	9	9.3	-10.4
3	5	10	4.7	-2.5
3	5	11	4.7	3.3
3	5	12	7.3	6.5
3	5	13	0.0	0.2
3	5	14	0.7	-2.6
3	6	-15	1.6	-0.4
3	6	-14	2.8	0.8
3	6	-13	2.1	4.1
3	6	-12	0.0	1.6
3	6	-11	0.0	-2.5
3	6	-10	2.4	-4.2
3	6	-9	3.1	3.1
3	6	-8	7.4	7.6
3	6	-7	1.2	-1.8
3	6	-6	5.0	-6.4
3	6	-5	3.8	5.1
3	6	-4	11.7	8.6
3	6	-3	12.1	-12.3
3	6	-2	7.9	-6.7
3	6	-1	1.4	-0.4
3	6	0	13.3	11.8
3	6	1	6.8	-5.1
3	6	2	6.7	-7.6
3	6	3	1.1	-2.5
3	6	4	3.1	4.8
3	6	5	0.0	-2.4
3	6	6	5.5	-4.0
3	6	7	4.1	-0.7
3	6	8	2.6	1.7
3	6	9	0.0	4.6
3	6	10	2.4	-4.2
3	6	11	3.9	-2.4
3	6	12	2.7	3.8
3	6	13	1.9	1.9
3	7	-14	8.6	7.9
3	7	-13	0.0	2.3
3	7	-12	3.5	-11.1
3	7	-11	0.0	-2.9
3	7	-10	14.4	17.8
3	7	-9	3.6	6.1
3	7	-8	6.4	-7.7
3	7	-7	7.6	-8.4
3	7	-6	11.9	10.9
3	7	-5	12.7	12.1
3	7	-4	4.8	-4.0
3	7	-3	23.7	-19.1
3	7	-2	8.7	6.9
3	7	-1	21.0	18.2
3	7	0	0.0	-1.5
3	7	0	0.0	-1.5
3	7	1	21.5	-17.2
3	7	2	18.0	-15.1
3	7	3	19.1	18.2
3	7	4	4.1	3.1
3	7	5	9.7	-11.0
3	7	6	10.6	-10.6
3	7	7	9.9	12.1
3	7	8	2.7	1.2
3	7	9	5.4	-7.6
3	7	10	4.8	-3.8
3	7	11	0.0	4.0
3	7	12	3.3	5.1
3	8	-13	0.0	-2.3
3	8	-12	0.0	-0.1
3	8	-11	1.5	-1.5
3	8	-10	0.0	1.9
3	8	-9	0.0	-3.0
3	8	-8	4.4	-5.6
3	8	-7	8.3	8.8
3	8	-6	4.4	8.3
3	8	-5	4.6	-5.5
3	8	-4	2.5	-0.6
3	8	-3	4.7	-3.0
3	8	-2	6.6	6.2
3	8	-1	0.0	0.6
3	8	0	4.1	-2.0
3	8	1	1.9	-1.0
3	8	2	18.7	15.9
3	8	3	8.9	-7.5
3	8	4	5.3	-5.2
3	8	5	2.7	-4.9
3	8	6	0.0	3.0
3	8	7	5.8	6.3
3	8	8	1.6	-2.4
3	8	9	5.5	-5.5
3	8	10	0.0	0.0
3	8	11	1.5	2.7
3	9	-11	0.0	-2.0
3	9	-10	2.0	4.5
3	9	-9	0.0	1.5
3	9	-8	1.6	-4.0
3	9	-7	0.0	-3.8
3	9	-6	5.7	5.5
3	9	-5	3.5	3.3
3	9	-4	0.0	-0.5
3	9	-3	0.0	-3.6
3	9	-2	0.0	-0.8
3	9	-1	0.0	1.6
3	9	0	0.0	-0.3
3	9	1	4.8	-5.2
3	9	2	0.0	-1.9
3	9	3	11.4	11.2
3	9	4	2.9	-1.8
3	9	5	1.7	-6.3
3	9	6	0.0	-2.6
3	9	7	3.1	3.6
3	9	8	1.4	0.9
3	9	9	1.6	-1.8
3	9	10	1.7	0.1
3	10	-9	0.0	-3.0
3	10	-8	1.4	-4.2
3	10	-7	0.0	2.6
3	10	-6	3.8	5.5
3	10	-5	0.0	-0.1
3	10	-4	4.8	-7.3
3	10	-3	6.5	6.0
3	10	-2	8.3	9.0
3	10	-1	4.3	-5.9
3	10	0	5.8	-5.2
3	10	1	1.8	-0.2
3	10	2	8.8	10.0
3	10	3	2.4	-1.5
3	10	4	2.8	-2.7
3	10	5	1.4	-4.2
3	10	6	1.0	5.3
3	10	7	0.0	1.6
3	10	8	1.4	-3.6
3	11	-7	0.0	1.2
3	11	-6	0.0	-1.9
3	11	-5	0.0	-4.1
3	11	-4	0.0	2.3
3	11	-3	0.0	1.2
3	11	-2	0.0	0.4
3	11	-1	3.0	-2.3
3	11	0	2.0	-1.2
3	11	1	1.7	0.6
3	11	2	3.5	4.1
3	11	3	3.1	-2.7
3	11	4	0.7	-0.7
3	11	5	0.0	0.8
3	12	-4	0.0	-2.7
3	12	-3	0.0	2.0
3	12	-2	0.0	2.5
3	12	-1	2.2	-3.0
3	12	0	1.5	-2.2
3	12	1	0.9	-0.6
4	0	-17	6.0	8.0
4	0	-16	4.4	-2.2
4	0	-15	15.7	-13.9
4	0	-14	4.1	-1.6
4	0	-13	9.6	8.6
4	0	-12	4.0	5.0
4	0	-11	19.2	-17.0
4	0	-10	13.5	-13.6
4	0	-9	28.0	26.5
4	0	-8	30.2	30.7
4	0	-7	14.9	-15.6
4	0	-6	36.1	-36.5
4	0	-5	28.0	30.4
4	0	-4	26.7	32.9
4	0	-3	1.7	-3.6
4	0	-2	11.6	-16.4
4	0	-1	13.6	18.8
4	0	0	41.2	35.9
4	0	1	12.1	-13.6
4	0	2	26.7	-31.5
4	0	3	7.6	-8.3
4	0	4	37.4	39.5
4	0	5	8.3	-7.8
4	0	6	12.5	-9.6
4	0	7	23.2	-21.8
4	0	8	8.3	8.0
4	0	9	16.2	14.6
4	0	10	11.6	-10.3
4	0	11	21.2	-20.4
4	0	12	11.8	11.2
4	0	13	7.4	6.3
4	0	14	4.3	-3.6
4	1	-17	1.0	-0.7
4	1	-16	5.4	-5.4
4	1	-15	2.7	-1.7
4	1	-14	8.5	7.9
4	1	-13	2.4	-0.6
4	1	-12	8.0	-5.8
4	1	-11	4.9	-3.6
4	1	-10	9.3	8.1
4	1	-9	2.2	-0.7
4	1	-8	15.8	-17.2
4	1	-7	4.9	-4.8
4	1	-6	3.6	5.2
4	1	-5	15.6	18.6
4	1	-4	7.1	-9.1
4	1	-3	20.2	-20.8
4	1	-2	3.3	-2.0
4	1	-1	24.8	22.9
4	1	0	7.2	-5.4
4	1	1	5.5	-5.2
4	1	2	0.0	-4.1
4	1	3	14.5	16.8
4	1	4	8.5	13.0
4	1	5	12.6	-14.9
4	1	6	4.7	-5.6
4	1	7	10.3	10.2
4	1	8	5.9	5.4
4	1	9	5.2	-4.0
4	1	10	4.8	-0.4
4	1	11	4.5	1.3
4	1	12	1.6	1.8
4	1	13	2.3	-1.4
4	1	14	2.8	-2.4
4	2	-17	6.4	4.9
4	2	-16	1.5	0.3
4	2	-15	8.6	-7.9
4	2	-14	4.5	-4.3
4	2	-13	7.6	6.8
4	2	-12	3.4	3.5
4	2	-11	10.0	-7.6
4	2	-10	1.1	1.6
4	2	-9	11.4	12.6
4	2	-8	17.1	17.9
4	2	-7	9.4	-10.6
4	2	-6	22.5	-24.0
4	2	-5	12.1	13.2
4	2	-4	30.9	29.8
4	2	-3	9.2	-8.4
4	2	-2	4.1	-5.3
4	2	-1	4.6	5.5
4	2	0	18.7	17.7
4	2	1	28.4	-23.9
4	2	2	16.5	-12.7
4	2	3	4.5	-5.3
4	2	4	15.9	15.5
4	2	5	6.2	8.4
4	2	6	11.1	-14.3
4	2	7	12.4	-12.1
4	2	8	5.3	4.7
4	2	9	7.2	7.4
4	2	10	9.6	-10.6
4	2	11	0.0	-0.9
4	2	12	1.1	1.3
4	2	13	7.2	7.5
4	2	14	3.2	-1.7
4	3	-16	13.4	-9.8

Table 1 (*cont.*)

continued :–

h	k	l	F_{obs}	F_{calc}
4	3	-15	13.1	-1.3
4	3	-14	13.1	12.8
4	3	-13	3.1	-1.1
4	3	-12	17.7	-15.2
4	3	-11	6.2	-4.5
4	3	-10	16.3	15.4
4	3	-9	7.9	8.9
4	3	-8	21.6	-25.3
4	3	-7	15.2	-18.0
4	3	-6	28.4	28.6
4	3	-5	17.1	16.7
4	3	-4	18.2	-14.6
4	3	-3	25.9	-24.4
4	3	-2	8.2	-5.7
4	3	-1	47.0	38.1
4	3	0	21.9	17.7
4	3	1	55.0	-46.0
4	3	2	1.0	1.7
4	3	3	35.0	30.7
4	3	4	7.1	6.2
4	3	5	18.3	-19.6
4	3	6	5.7	-7.4
4	3	7	11.7	13.1
4	3	8	10.1	12.4
4	3	9	9.3	-9.3
4	3	10	10.7	-9.3
4	3	11	8.3	8.3
4	3	12	1.6	0.1
4	3	13	0.0	1.0
4	3	14	3.9	-5.8
4	4	-16	1.1	-0.7
4	4	-15	4.4	3.6
4	4	-14	0.0	0.1
4	4	-13	5.6	-4.4
4	4	-12	1.8	0.2
4	4	-11	9.4	9.7
4	4	-10	1.8	1.3
4	4	-9	0.0	2.3
4	4	-8	0.0	-4.7
4	4	-7	0.0	1.0
4	4	-6	9.8	9.6
4	4	-5	1.5	-0.8
4	4	-4	8.5	-7.6
4	4	-3	0.0	3.9
4	4	-2	9.4	8.3
4	4	-1	17.7	-13.6
4	4	0	5.6	3.3
4	4	1	17.5	-14.4
4	4	2	9.6	9.8
4	4	3	4.2	-4.2
4	4	4	7.5	-6.0
4	4	5	4.9	-5.6
4	4	6	2.5	4.1
4	4	7	3.2	1.8
4	4	8	7.0	-4.3
4	4	9	6.4	-2.3
4	4	10	1.7	-1.8
4	4	11	7.6	6.9
4	4	12	0.0	-1.2
4	4	13	0.0	-0.3
4	4	14	2.3	-0.5
4	5	-15	4.2	-3.6
4	5	-14	8.4	7.7
4	5	-13	0.0	1.4
4	5	-12	11.4	-11.7
4	5	-11	0.0	-0.0
4	5	-10	13.9	16.1
4	5	-9	2.7	-1.7
4	5	-8	2.4	-3.3
4	5	-7	12.3	-13.2
4	5	-6	13.8	13.8
4	5	-5	17.8	16.8
4	5	-4	6.5	-4.1
4	5	-3	19.0	-16.8
4	5	-2	9.0	8.1
4	5	-1	22.4	15.3
4	5	0	0.0	0.2
4	5	0	0.0	0.2
4	5	1	20.1	-15.0
4	5	2	9.3	-8.7
4	5	3	21.8	21.6
4	5	4	2.6	1.3
4	5	5	15.3	-16.1
4	5	6	6.1	-6.6
4	5	7	10.7	12.2
4	5	8	5.8	-0.9
4	5	9	5.9	-4.7
4	5	10	5.9	-5.0
4	5	11	3.8	3.5
4	5	12	4.5	1.0
4	5	13	0.0	-0.1
4	6	-14	1.2	0.7
4	6	-13	6.6	-7.0
4	6	-12	0.0	-2.1
4	6	-11	3.5	5.2
4	6	-10	4.6	7.4
4	6	-9	5.0	-8.7
4	6	-8	11.1	-15.3
4	6	-7	19.4	15.5
4	6	-6	17.6	17.4
4	6	-5	9.3	-8.5
4	6	-4	10.7	-10.5
4	6	-3	3.7	2.6
4	6	-2	15.5	13.0
4	6	-1	2.1	0.6
4	6	0	17.2	-15.6
4	6	1	7.2	5.6
4	6	2	26.2	22.8
4	6	3	11.3	-11.0
4	6	4	10.2	-10.3
4	6	5	7.4	-7.7
4	6	6	6.9	5.4
4	6	7	7.2	11.6
4	6	8	6.9	-7.3
4	6	9	8.4	-10.3
4	6	10	4.9	5.6
4	6	11	3.4	4.1
4	6	12	2.5	-3.6
4	7	-13	0.0	0.6
4	7	-12	4.3	5.6
4	7	-11	0.0	-3.2
4	7	-10	1.7	4.4
4	7	-9	2.8	-4.9
4	7	-8	0.0	-1.8
4	7	-7	6.2	-2.2
4	7	-6	0.0	1.6
4	7	-5	0.0	-0.7
4	7	-4	10.2	9.8
4	7	-3	3.5	4.2
4	7	-2	5.5	-4.5
4	7	-1	1.5	-2.8
4	7	0	9.4	-8.3
4	7	1	7.3	5.8
4	7	2	4.7	3.6
4	7	3	7.9	7.7
4	7	4	9.2	-4.2
4	7	5	0.0	-1.4
4	7	6	4.8	-0.7
4	7	7	3.4	-0.5
4	7	8	4.4	-3.9
4	7	9	3.8	2.1
4	7	10	1.4	0.9
4	7	11	0.0	1.6
4	8	-12	0.0	-0.8
4	8	-11	2.7	3.4
4	8	-10	2.7	5.9
4	8	-9	4.1	-6.8
4	8	-8	0.0	-4.2
4	8	-7	5.0	5.3
4	8	-6	6.2	9.0
4	8	-5	0.0	-1.8
4	8	-4	11.8	-12.2
4	8	-3	9.8	9.8
4	8	-2	11.4	11.3
4	8	-1	6.3	-5.7
4	8	0	8.3	-7.0
4	8	1	5.4	5.4
4	8	2	3.5	3.3
4	8	3	0.0	2.9
4	8	4	3.4	-8.2
4	8	5	1.2	-2.7
4	8	6	4.8	6.8
4	8	7	2.2	3.2
4	8	8	2.1	-6.6
4	8	9	4.4	-3.6
4	8	10	3.6	1.5
4	9	-11	0.0	0.0
4	9	-10	2.4	-4.1
4	9	-9	0.0	-4.2
4	9	-8	0.0	5.3
4	9	-7	0.0	0.7
4	9	-6	7.1	-6.1
4	9	-5	5.4	-6.3
4	9	-4	6.5	6.5
4	9	-3	8.4	8.2
4	9	-2	0.0	-0.2
4	9	-1	9.5	-9.1
4	9	0	3.7	-3.3
4	9	1	9.0	7.9
4	9	2	4.8	7.7
4	9	3	9.3	-8.8
4	9	4	1.6	1.2
4	9	5	2.4	4.8
4	9	6	1.8	2.3
4	9	7	1.4	-2.2
4	9	8	3.1	-4.0
4	9	9	1.0	2.2
4	10	-9	0.0	1.7
4	10	-8	0.0	-1.5
4	10	-7	0.0	3.3
4	10	-6	0.0	-0.2
4	10	-5	0.0	-1.1
4	10	-4	0.0	-3.2
4	10	-3	0.0	3.6
4	10	-2	0.0	-1.0
4	10	-1	2.5	0.2
4	10	0	1.7	-0.6
4	10	1	0.0	-0.8
4	10	2	2.1	0.8
4	10	3	1.8	-2.0
4	10	4	1.0	-0.3
4	10	5	0.0	0.3
4	10	6	0.8	0.8
4	10	7	0.0	-0.9
4	11	-6	0.0	-2.1
4	11	-5	0.0	-3.2
4	11	-4	0.0	0.1
4	11	-3	0.0	6.4
4	11	-2	0.0	-0.8
4	11	-1	3.5	-3.4
4	11	0	1.8	-0.2
4	11	1	4.8	4.8
4	11	2	1.4	-0.6
4	11	3	3.6	-3.5
4	11	4	0.0	-1.2
5	1	-16	11.1	-10.2
5	1	-15	2.0	-1.3
5	1	-14	14.5	13.1
5	1	-13	3.1	-2.7
5	1	-12	17.3	-14.8
5	1	-11	11.6	-10.4
5	1	-10	13.7	12.7
5	1	-9	1.6	2.5
5	1	-8	12.8	-13.2
5	1	-7	7.6	-8.4
5	1	-6	17.0	20.9
5	1	-5	12.8	16.6
5	1	-4	15.9	-16.8
5	1	-3	23.9	-24.5
5	1	-2	0.0	3.6
5	1	-1	56.4	46.8
5	1	0	7.6	6.2
5	1	1	26.4	-24.6
5	1	2	5.5	-2.7
5	1	3	19.7	23.2
5	1	4	4.2	5.7
5	1	5	18.3	-21.5
5	1	6	1.1	-0.4
5	1	7	13.2	12.8
5	1	8	6.8	7.5
5	1	9	10.4	-8.9
5	1	10	9.3	-8.7
5	1	11	3.4	1.3
5	1	12	3.1	1.9
5	1	13	3.2	1.1
5	2	-16	3.0	2.3
5	2	-15	9.8	8.9
5	2	-14	2.4	-1.0
5	2	-13	13.8	-13.0
5	2	-12	0.0	-2.0
5	2	-11	15.5	15.7
5	2	-10	11.6	12.0
5	2	-9	9.5	-8.7
5	2	-8	21.7	-23.0
5	2	-7	7.0	10.2
5	2	-6	12.8	14.0
5	2	-5	18.7	-16.9
5	2	-4	17.0	-16.2
5	2	-3	17.4	15.3
5	2	-2	18.2	15.0
5	2	-1	6.6	-8.4
5	2	0	29.1	-25.4
5	2	1	6.6	-5.2
5	2	2	18.3	15.7
5	2	3	4.5	5.0
5	2	4	11.2	-10.9
5	2	5	0.0	0.5
5	2	6	4.8	7.0
5	2	7	8.8	7.1
5	2	8	9.7	-9.3
5	2	9	8.1	-9.4
5	2	10	7.6	8.7
5	2	11	11.1	11.3
5	2	12	5.1	-5.1
5	2	13	4.0	-3.6
5	3	-16	2.7	-1.2
5	3	-15	1.3	-0.1
5	3	-14	3.1	1.9
5	3	-13	0.0	-1.2
5	3	-12	8.4	-8.3
5	3	-11	5.2	-3.2
5	3	-10	7.8	6.8
5	3	-9	3.1	-1.2
5	3	-8	9.6	11.7
5	3	-7	4.4	-4.8
5	3	-6	9.0	8.3
5	3	-5	0.0	-0.3
5	3	-4	3.4	-3.0
5	3	-3	3.9	-2.6
5	3	-2	13.6	10.9
5	3	-1	11.4	9.8
5	3	0	9.6	8.3
5	3	1	8.2	-7.2
5	3	2	6.4	-5.7
5	3	3	5.9	5.7
5	3	4	10.1	-10.0
5	3	5	0.0	0.7
5	3	6	4.4	0.7
5	3	7	5.5	4.4
5	3	8	3.6	-3.0
5	3	9	3.6	-2.4
5	3	10	5.5	-8.2
5	3	11	11.0	4.3
5	3	12	4.0	-3.9
5	3	13	2.7	3.0
5	4	-15	8.9	7.4
5	4	-14	2.4	0.2
5	4	-13	13.1	-12.0
5	4	-12	7.1	-7.3
5	4	-11	14.1	13.1
5	4	-10	9.2	8.6
5	4	-9	9.3	-11.0
5	4	-8	15.5	-17.2
5	4	-7	11.3	11.1
5	4	-6	18.0	18.2
5	4	-5	19.4	-17.8
5	4	-4	18.5	-15.6
5	4	-3	17.4	16.2
5	4	-2	19.6	17.1
5	4	-1	3.5	-4.5
5	4	0	23.9	-18.3
5	4	1	1.0	0.2
5	4	2	20.1	17.8
5	4	3	2.0	2.5
5	4	4	12.1	-10.5
5	4	5	2.7	-1.6
5	4	6	5.5	8.0
5	4	7	8.9	8.0
5	4	8	11.7	-10.6
5	4	9	9.8	-9.3
5	4	10	10.0	9.6
5	4	11	6.2	6.2
5	4	12	4.5	-4.9
5	4	13	4.1	-4.3
5	5	-14	7.9	-7.6
5	5	-13	0.0	-1.8
5	5	-12	9.0	7.1
5	5	-11	0.0	-1.0
5	5	-10	0.0	-5.1
5	5	-9	2.1	-2.2
5	5	-8	6.9	8.2
5	5	-7	6.2	5.9
5	5	-6	7.3	-7.7
5	5	-5	9.2	-9.9
5	5	-4	12.5	11.1
5	5	-3	17.8	15.6
5	5	-2	0.0	-1.4
5	5	-1	12.8	-10.7
5	5	0	7.2	-5.4
5	5	1	21.4	18.7
5	5	2	4.5	3.1
5	5	3	15.4	-13.5
5	5	4	2.1	-1.4
5	5	5	7.5	5.9
5	5	6	5.8	5.4
5	5	7	7.8	-6.2
5	5	8	4.9	-6.8
5	5	9	3.4	1.9
5	5	10	5.6	3.9
5	5	11	1.9	-3.1
5	5	12	3.1	-1.2
5	6	-13	5.5	-5.2
5	6	-12	2.1	-3.8
5	6	-11	0.0	0.8
5	6	-10	4.8	6.8
5	6	-9	3.2	-5.2
5	6	-8	0.0	-0.1
5	6	-7	1.8	1.7
5	6	-6	3.4	-1.3
5	6	-5	4.3	-4.1
5	6	-4	7.4	-6.3
5	6	-3	10.4	11.0
5	6	-2	11.1	9.5
5	6	-1	0.0	1.0
5	6	0	12.6	-11.4
5	6	1	10.4	10.3
5	6	2	10.7	-8.8
5	6	3	8.8	9.9
5	6	4	0.0	-4.5
5	6	5	3.3	3.4
5	6	6	5.3	4.5
5	6	7	1.2	1.5
5	6	8	6.6	-6.9
5	6	9	1.9	-2.3
5	6	10	0.0	3.2
5	6	11	0.0	-0.3
5	7	-13	0.0	-1.5
5	7	-12	5.2	7.8
5	7	-11	0.0	-1.7
5	7	-10	4.8	-5.5
5	7	-9	1.7	-5.6
5	7	-8	5.0	6.3
5	7	-7	2.5	5.1
5	7	-6	10.3	-10.3
5	7	-5	7.5	-8.4
5	7	-4	11.0	9.0
5	7	-3	14.4	12.6
5	7	-2	0.0	-3.2
5	7	-1	6.9	-11.7
5	7	0	5.0	-4.1
5	7	1	21.7	18.4
5	7	2	5.6	5.1
5	7	3	14.8	-14.2
5	7	4	1.2	1.7
5	7	5	4.7	8.7
5	7	6	3.4	1.9
5	7	7	4.4	-5.5
5	7	8	3.1	-3.2
5	7	9	3.1	3.4
5	7	10	3.8	3.8
5	8	-12	0.0	-0.0
5	8	-11	2.2	-3.6
5	8	-10	0.0	-1.6
5	8	-9	3.1	6.1
5	8	-8	0.0	3.2
5	8	-7	2.7	-3.2
5	8	-6	6.7	-7.8
5	8	-5	0.0	-2.2
5	8	-4	3.9	4.0
5	8	-3	0.0	2.6
5	8	-2	0.0	-4.3
5	8	-1	0.0	3.6
5	8	0	0.0	1.6
5	8	0	0.0	1.6
5	8	1	0.0	-1.8
5	8	2	7.9	-6.7
5	8	3	0.0	1.0
5	8	4	3.3	2.3
5	8	5	3.0	6.1
5	8	6	1.8	-0.8
5	8	7	3.0	-2.9
5	8	8	3.4	1.7
5	8	9	1.0	0.5
5	9	-9	0.0	-0.0
5	9	-8	0.0	2.2
5	9	-7	0.0	0.6
5	9	-6	0.0	-1.4
5	9	-5	0.0	0.1
5	9	-4	0.0	-2.0
5	9	-3	9.1	8.1
5	9	-2	0.0	-3.1
5	9	-1	4.5	-5.3
5	9	0	3.4	3.0
5	9	1	4.8	4.3
5	9	2	0.0	-0.1
5	9	3	3.7	-2.9
5	9	4	1.7	0.4
5	9	5	1.0	1.3
5	9	6	0.0	2.0
5	9	7	4.1	-6.3
5	10	-8	2.6	4.3
5	10	-7	0.0	-3.6
5	10	-6	3.0	-4.9
5	10	-5	0.0	2.7
5	10	-4	0.0	3.1
5	10	-3	2.8	-3.3
5	10	-2	3.1	-4.1
5	10	-1	2.3	2.0
5	10	0	5.4	4.1
5	10	1	0.0	0.2
5	10	2	4.2	-4.9
5	10	3	2.3	-0.3
5	10	4	0.0	2.5
5	10	5	0.0	2.4
5	11	-5	0.0	3.4
5	11	-4	0.0	0.2
5	11	-3	0.0	-1.6
5	11	-2	0.0	-0.9
5	11	-1	1.2	0.8
5	11	0	1.2	-0.4
5	11	1	2.0	-1.3
5	11	2	1.0	0.8
6	0	-16	3.1	0.7
6	0	-15	7.6	8.4
6	0	-14	3.7	0.0
6	0	-13	16.6	-15.0
6	0	-12	2.4	-1.3
6	0	-11	19.7	15.7
6	0	-10	7.2	5.2
6	0	-9	16.9	-15.7
6	0	-8	28.4	-27.4
6	0	-7	17.5	17.7
6	0	-6	10.3	16.7
6	0	-5	13.7	-15.6
6	0	-4	15.1	-19.9
6	0	-3	9.2	12.8
6	0	-2	2.9	9.0
6	0	-1	0.0	-1.9
6	0	0	29.7	-23.6
6	0	1	5.8	7.7
6	0	2	16.8	16.9
6	0	3	8.8	7.3
6	0	4	10.6	-11.0
6	0	5	7.9	-7.3
6	0	6	12.4	10.6
6	0	7	8.5	8.8
6	0	8	7.3	-6.4
6	0	9	7.6	-7.7
6	0	10	14.2	12.4
6	0	11	4.1	1.8
6	0	12	3.8	-4.2
6	1	-16	5.7	5.0
6	1	-15	1.5	1.8
6	1	-14	4.8	-4.4
6	1	-13	1.9	0.5
6	1	-12	2.8	2.5
6	1	-11	3.6	0.2
6	1	-10	2.7	-3.7
6	1	-9	5.4	-1.3
6	1	-8	12.4	11.6
6	1	-7	5.9	8.2
6	1	-6	6.3	-8.3
6	1	-5	10.1	-8.3
6	1	-4	0.0	1.8
6	1	-3	0.0	6.1
6	1	-2	1.5	3.7
6	1	-1	12.5	-9.9
6	1	0	2.0	2.2
6	1	1	13.4	17.5
6	1	2	8.0	-6.2
6	1	3	10.7	-12.4
6	1	4	2.1	-5.4
6	1	5	9.4	10.7
6	1	6	3.7	1.5
6	1	7	3.5	-0.9
6	1	8	6.7	-4.4
6	1	9	0.0	-0.7
6	1	10	2.4	0.2
6	1	11	1.7	0.4
6	1	12	2.7	-2.6
6	2	-15	3.8	3.4
6	2	-14	0.0	0.3
6	2	-13	5.9	-5.7
6	2	-12	4.1	-4.5
6	2	-11	7.2	6.6
6	2	-10	1.7	1.3
6	2	-9	15.0	-15.4
6	2	-8	9.3	-11.1
6	2	-7	7.7	9.2
6	2	-6	12.9	15.2
6	2	-5	9.0	-10.7
6	2	-4	8.5	-8.3
6	2	-3	2.7	4.2
6	2	-2	5.4	4.2
6	2	-1	1.1	-0.9
6	2	0	6.9	-6.5
6	2	1	8.6	7.5
6	2	2	15.2	13.4
6	2	3	5.9	7.1
6	2	4	12.7	-14.8
6	2	5	1.7	1.2
6	2	6	4.1	1.5
6	2	7	8.2	7.4
6	2	8	4.5	-2.8
6	2	9	3.7	-2.6
6	2	10	2.6	4.6
6	2	11	2.7	2.1
6	2	12	6.7	-6.7
6	3	-15	4.4	3.7
6	3	-14	10.3	-8.7
6	3	-13	2.2	0.5
6	3	-12	11.0	9.0
6	3	-11	0.0	0.5
6	3	-10	15.6	-14.3
6	3	-9	5.1	1.6
6	3	-8	9.0	11.2
6	3	-7	11.4	14.0
6	3	-6	10.7	-10.1
6	3	-5	21.0	-21.0
6	3	-4	7.9	5.9
6	3	-3	17.6	17.1
6	3	-2	6.9	-5.1
6	3	-1	23.7	-18.4
6	3	0	8.6	8.5
6	3	1	19.6	17.0
6	3	2	1.1	0.0
6	3	3	27.5	-26.8
6	3	4	3.4	-2.8
6	3	5	12.8	15.4
6	3	6	6.8	7.1
6	3	7	6.6	-6.2
6	3	8	4.8	-5.2
6	3	9	0.0	1.7
6	3	10	4.7	3.5
6	3	11	2.5	-2.4
6	3	12	3.1	-2.2
6	4	-15	4.4	-3.5
6	4	-14	2.4	1.1
6	4	-13	3.5	3.4
6	4	-12	2.3	-2.8
6	4	-11	3.7	-4.9
6	4	-10	5.6	-6.2
6	4	-9	1.7	-2.7
6	4	-8	6.0	7.8
6	4	-7	5.3	-5.5
6	4	-6	3.0	-1.3
6	4	-5	5.9	5.9
6	4	-4	0.0	1.1
6	4	-3	0.0	-1.4
6	4	-2	0.0	-3.0
6	4	-1	3.6	-1.1
6	4	0	8.0	7.6
6	4	1	13.6	13.6
6	4	2	7.9	-9.2
6	4	3	6.8	8.3
6	4	4	5.0	-3.7
6	4	5	0.0	1.2
6	4	6	0.0	-0.2
6	4	7	2.4	-0.6
6	4	8	3.8	1.1
6	4	9	5.7	4.2
6	4	10	2.7	-1.6
6	4	11	4.1	-5.2
6	5	-14	4.5	-4.1
6	5	-13	0.0	1.1
6	5	-12	5.2	4.9
6	5	-11	0.0	-1.5
6	5	-10	7.4	-8.8
6	5	-9	2.4	-3.8
6	5	-8	3.1	5.5
6	5	-7	8.4	9.6
6	5	-6	12.1	-11.5
6	5	-5	7.6	-8.0
6	5	-4	0.0	0.7
6	5	-3	6.7	6.3
6	5	-2	0.0	-3.0
6	5	-1	11.4	-8.6
6	5	0	2.7	1.3
6	5	1	18.0	17.9
6	5	2	2.1	-2.8
6	5	3	16.0	-16.3
6	5	4	4.5	4.6
6	5	5	6.6	6.8
6	5	6	3.7	3.9
6	5	7	0.0	-1.3
6	5	8	4.9	-1.4
6	5	9	3.1	0.9
6	5	10	3.1	4.2
6	5	11	2.4	-5.1
6	6	-13	7.0	7.1
6	6	-12	0.0	2.0
6	6	-11	9.1	-10.9
6	6	-10	2.9	-4.3
6	6	-9	5.4	8.7
6	6	-8	4.3	5.5
6	6	-7	4.5	-6.2
6	6	-6	11.0	-12.7
6	6	-5	4.9	4.1
6	6	-4	14.5	12.5
6	6	-3	9.9	-7.2
6	6	-2	10.6	-10.2
6	6	-1	3.0	6.7
6	6	0	6.2	4.5
6	6	1	5.5	3.3
6	6	2	8.3	-7.9
6	6	3	3.0	-2.1
6	6	4	5.9	7.5
6	6	5	0.0	4.9

Table 1 (*cont.*)

continued :–

h	k	l	F_{obs}	F_{calc}
6	6	6	4.5	-5.0
6	6	7	3.3	-3.5
6	6	8	5.5	5.2
6	6	9	1.3	2.6
6	6	10	1.6	-2.2
6	7	-12	0.0	-0.4
6	7	-11	0.0	-1.6
6	7	-10	0.0	-2.6
6	7	-9	0.0	2.4
6	7	-8	0.0	-0.5
6	7	-7	0.0	1.7
6	7	-6	0.0	-0.5
6	7	-5	0.0	0.8
6	7	-4	9.6	-7.7
6	7	-3	0.0	1.4
6	7	-2	0.0	-2.3
6	7	-1	0.0	1.8
6	7	0	8.3	5.8
6	7	1	0.0	-1.5
6	7	2	6.3	-4.6
6	7	3	1.7	2.0
6	7	4	1.6	-1.6
6	7	5	0.0	-0.0
6	7	6	2.6	2.3
6	7	7	2.7	-1.3
6	7	8	4.1	5.4
6	7	9	0.0	-0.7
6	8	-10	2.0	-5.0
6	8	-9	2.6	5.7
6	8	-8	2.4	5.4
6	8	-7	6.4	-7.5
6	8	-6	2.8	-5.4
6	8	-5	0.0	3.5
6	8	-4	1.7	1.7
6	8	-3	3.4	-4.2
6	8	-2	0.0	-2.9
6	8	-1	1.8	0.6
6	8	0	7.9	6.2
6	8	1	3.1	-2.9
6	8	2	3.8	-4.0
6	8	3	1.4	-0.8
6	8	4	1.4	5.4
6	8	5	0.0	2.5
6	8	6	0.9	-3.5
6	8	7	2.4	-1.9
6	9	-8	0.0	-4.3
6	9	-7	0.0	-0.3
6	9	-6	0.0	2.4
6	9	-5	3.1	4.0
6	9	-4	1.7	-3.1
6	9	-3	5.8	-5.8
6	9	-2	0.0	1.4
6	9	-1	4.4	4.3
6	9	0	3.0	2.0
6	9	1	9.7	-8.7
6	9	2	0.0	2.3
6	9	3	2.7	1.7
6	9	4	0.8	2.2
6	9	5	1.4	-4.7
6	10	-6	0.0	-0.6
6	10	-5	0.0	-0.4
6	10	-4	0.0	0.9
6	10	-3	0.0	-0.2
6	10	-2	0.0	2.0
6	10	-1	2.6	2.0
6	10	0	1.5	-0.4
6	10	1	1.9	-2.6
6	10	2	1.4	-0.7
6	10	3	1.3	1.0
7	1	-15	1.0	2.2
7	1	-14	9.1	-7.9
7	1	-13	0.0	0.5
7	1	-12	10.9	8.7
7	1	-11	2.8	2.8
7	1	-10	11.4	-10.3
7	1	-9	0.0	-0.6
7	1	-8	3.8	1.8
7	1	-7	11.4	11.2
7	1	-6	10.8	-13.1
7	1	-5	4.1	-13.0
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7	1	-3	12.4	12.8
7	1	-2	6.9	-7.5
7	1	-1	21.8	-18.1
7	1	0	4.6	2.7
7	1	1	14.5	12.9
7	1	2	5.3	1.2
7	1	3	13.1	-15.9
7	1	4	5.2	3.8
7	1	5	10.3	10.1
7	1	6	0.0	1.0
7	1	7	0.0	-5.3
7	1	8	4.9	-3.4
7	1	9	3.6	3.0
7	1	10	5.6	7.0
7	1	11	2.1	-1.3
7	2	-14	3.1	-0.1
7	2	-13	11.5	10.4
7	2	-12	2.1	1.4
7	2	-11	11.3	-8.6
7	2	-10	5.5	-5.7
7	2	-9	5.5	5.1
7	2	-8	12.0	11.2
7	2	-7	10.5	-12.7
7	2	-6	4.1	-4.8
7	2	-5	13.0	14.4
7	2	-4	4.5	11.2
7	2	-3	4.5	-8.8
7	2	-2	11.4	-10.3
7	2	-1	4.6	-4.8
7	2	0	10.4	15.2
7	2	1	7.6	5.8
7	2	2	10.3	-8.2
7	2	3	5.5	1.2
7	2	4	3.0	1.3
7	2	5	3.6	2.1
7	2	6	7.2	-7.4
7	2	7	4.0	-3.3
7	2	8	5.5	5.7
7	2	9	6.6	6.5
7	2	10	5.5	-0.4
7	2	11	1.6	-1.9
7	3	-14	1.7	-0.9
7	3	-13	2.2	1.2
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7	3	-11	2.7	2.5
7	3	-10	3.5	-3.4
7	3	-9	4.6	-3.6
7	3	-8	4.1	-1.9
7	3	-7	4.5	-3.4
7	3	-6	5.3	-5.3
7	3	-5	3.5	-0.8
7	3	-4	2.2	1.1
7	3	-3	1.7	3.1
7	3	-2	4.4	-2.6
7	3	-1	9.5	-8.7
7	3	0	2.7	0.8
7	3	1	2.7	1.9
7	3	2	1.2	1.2
7	3	3	5.1	1.9
7	3	4	2.6	4.2
7	3	5	4.5	2.7
7	3	6	4.6	-1.4
7	3	7	3.9	-1.2
7	3	8	0.0	-0.1
7	3	9	0.0	2.8
7	3	10	2.4	2.6
7	4	-13	11.4	9.4
7	4	-12	4.3	4.8
7	4	-11	9.6	-8.9
7	4	-10	3.8	-3.0
7	4	-9	8.8	10.4
7	4	-8	0.0	3.8
7	4	-7	8.4	-9.3
7	4	-6	9.0	-8.8
7	4	-5	13.5	10.5
7	4	-4	14.4	13.5
7	4	-3	10.7	-10.7
7	4	-2	13.1	-12.4
7	4	-1	0.0	-0.0
7	4	0	8.6	7.9
7	4	1	3.8	1.5
7	4	2	5.2	-3.2
7	4	3	7.6	-7.8
7	4	4	10.3	11.7
7	4	5	1.7	-2.9
7	4	6	3.4	-5.4
7	4	7	2.8	-3.4
7	4	8	5.2	7.0
7	4	9	5.6	3.9
7	4	10	2.7	-2.6
7	5	-13	0.0	-0.1
7	5	-12	4.2	-3.5
7	5	-11	0.0	-1.1
7	5	-10	1.9	3.4
7	5	-9	1.1	2.5
7	5	-8	4.1	-6.4
7	5	-7	4.1	-5.0
7	5	-6	0.0	-0.0
7	5	-5	6.6	[illegible]
7	5	-4	6.2	-6.6
7	5	-3	5.9	-4.8
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7	5	-1	0.0	6.1
7	5	0	3.2	-2.2
7	5	1	8.3	-7.0
7	5	2	5.4	-2.4
7	5	3	11.8	11.9
7	5	4	2.9	1.0
7	5	5	4.2	-5.2
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7	5	7	0.0	0.7
7	5	8	4.0	4.5
7	5	9	2.9	-1.0
7	6	-12	0.0	3.0
7	6	-11	1.2	-2.6
7	6	-10	0.0	-0.6
7	6	-9	0.0	1.6
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7	6	-7	2.1	-3.1
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7	6	-5	5.1	4.8
7	6	-4	0.0	0.2
7	6	-3	3.1	-4.9
7	6	-2	2.3	-3.4
7	6	-1	0.0	-1.0
7	6	0	4.1	2.1
7	6	1	0.0	-1.0
7	6	2	2.6	-0.4
7	6	3	0.0	-2.1
7	6	4	1.1	3.8
7	6	5	1.8	-2.9
7	6	6	3.5	-2.5
7	6	7	2.9	-0.4
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7	7	-8	1.4	-5.0
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7	7	-2	0.0	3.2
7	7	-1	7.3	7.2
7	7	0	3.4	-0.3
7	7	1	11.8	-11.5
7	7	2	4.3	4.0
7	7	3	4.0	4.1
7	7	4	0.0	-0.3
7	7	5	2.3	-3.8
7	7	6	3.7	-2.5
7	7	7	4.7	4.9
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7	8	-8	0.0	-2.3
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7	8	-3	0.0	0.9
7	8	-2	0.0	2.4
7	8	-1	1.9	-1.0
7	8	0	2.3	0.9
7	8	1	1.5	-4.9
7	8	2	4.4	5.2
7	8	3	0.0	-0.3
7	8	4	0.8	-1.0
7	8	5	0.7	-3.0
7	9	-6	0.0	2.7
7	9	-5	0.0	1.4
7	9	-4	0.0	-2.5
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7	9	0	1.1	0.8
7	9	0	1.0	0.8
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7	9	3	1.4	1.8
8	0	-14	2.1	0.0
8	0	-13	11.4	10.4
8	0	-12	3.3	2.6
8	0	-11	11.8	-8.5
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8	0	-5	10.8	11.6
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8	0	-2	8.3	-8.3
8	0	-1	2.8	-3.3
8	0	0	16.5	11.3
8	0	1	4.7	0.1
8	0	2	9.6	-10.2
8	0	3	5.3	-3.7
8	0	4	8.6	8.7
8	0	5	4.7	1.3
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8	0	7	5.8	-4.1
8	0	8	3.5	4.1
8	0	9	4.8	3.0
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8	1	-10	5.3	4.4
8	1	-9	0.0	-1.3
8	1	-8	0.0	0.9
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8	1	-6	3.2	2.5
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8	1	2	4.2	-1.9
8	1	3	7.2	10.0
8	1	4	3.0	-2.5
8	1	5	0.0	0.1
8	1	6	1.5	-2.3
8	1	7	0.0	1.6
8	1	8	3.1	0.4
8	1	9	1.9	0.4
8	2	-13	6.5	6.3
8	2	-12	2.1	2.4
8	2	-11	5.7	-4.6
8	2	-10	1.8	0.3
8	2	-9	7.8	7.4
8	2	-8	1.2	3.2
8	2	-7	3.1	-6.7
8	2	-6	6.1	-5.0
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8	2	-3	5.7	-5.8
8	2	-2	6.6	-5.2
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8	2	0	6.3	4.9
8	2	1	3.9	-4.8
8	2	2	3.1	-2.8
8	2	3	4.2	-6.7
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8	2	6	0.0	-2.3
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8	2	8	1.8	2.3
8	2	9	1.5	1.0
8	3	-13	1.9	-1.1
8	3	-12	8.3	-6.8
8	3	-11	1.4	-1.9
8	3	-10	8.1	7.5
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8	3	-8	1.2	-3.0
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8	3	-1	3.3	8.9
8	3	0	4.2	-4.0
8	3	1	11.4	-11.0
8	3	2	1.2	-0.8
8	3	3	12.7	12.5
8	3	4	0.0	-1.0
8	3	5	3.1	-5.2
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8	3	8	2.4	2.3
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8	4	-11	0.0	2.0
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8	4	-9	1.0	-1.6
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8	4	-6	0.0	0.3
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8	4	0	4.0	-3.8
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8	4	3	2.0	-0.8
8	4	4	2.7	1.3
8	4	5	3.7	-3.7
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8	4	7	1.2	0.3
8	4	8	0.0	0.3
8	5	-11	0.0	-1.5
8	5	-10	4.8	6.0
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8	5	-5	1.7	3.5
8	5	-4	0.0	-0.6
8	5	-3	8.5	-7.3
8	5	-2	0.0	4.9
8	5	-1	0.0	7.4
8	5	0	3.8	-6.2
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8	5	2	1.9	2.3
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8	6	-9	3.8	-4.7
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8	6	-5	4.7	-5.3
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8	6	-1	2.8	-0.1
8	6	0	5.2	-3.9
8	6	1	3.3	-3.9
8	6	2	6.6	6.3
8	6	3	0.0	1.0
8	6	4	1.8	-5.0
8	6	5	0.8	-2.0
8	6	6	3.8	3.4
8	7	-8	0.0	1.1
8	7	-7	0.0	-1.0
8	7	-6	0.0	3.5
8	7	-5	0.0	-0.4
8	7	-4	0.0	0.2
8	7	-3	0.0	0.1
8	7	-2	0.0	0.5
8	7	-1	1.7	-1.9
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8	7	0	2.4	-0.1
8	7	1	2.5	-0.1
8	7	2	1.8	0.8
8	7	3	0.0	0.8
8	7	4	0.0	-2.5
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8	8	-5	1.9	-2.7
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8	8	0	2.2	-2.1
8	8	1	1.4	-1.5
8	8	2	2.7	4.4
8	5	4	1.0	0.9
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9	1	-11	0.0	-0.3
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9	1	2	3.1	-1.6
9	1	3	4.6	7.9
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9	1	5	1.4	-2.3
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9	1	7	0.0	1.5
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9	2	-9	8.3	-7.4
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9	2	-3	3.9	3.9
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9	2	2	3.8	2.9
9	2	3	0.0	4.0
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9	2	5	2.5	-0.3
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9	2	7	1.4	1.7
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10	0	-1	2.0	-0.3
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10	0	2	0.0	0.6
10	0	3	3.4	5.9
10	0	4	10.0	-9.3
10	1	-9	0.0	-1.3
10	1	-8	0.8	1.5
10	1	-7	2.3	1.9
10	1	-6	1.3	-2.1
10	1	-5	0.9	-2.0
10	1	-4	0.0	2.1
10	1	-3	2.1	2.4
10	1	-2	0.0	-1.0
10	1	-1	0.0	-1.5
10	1	0	1.7	-0.7
10	1	1	2.4	1.5
10	1	2	0.0	0.3
10	1	3	0.0	-2.1
10	1	4	1.8	1.1
10	2	-9	0.0	-1.0
10	2	-8	0.0	-0.6
10	2	-7	1.3	4.5
10	2	-6	0.0	1.7
10	2	-5	1.7	-2.6
10	2	-4	0.0	-0.8
10	2	-3	0.0	2.8
10	2	-2	0.0	3.3
10	2	-1	0.0	1.8
10	2	0	2.6	-3.3
10	2	1	0.0	0.2
10	2	2	1.4	2.3
10	2	3	0.0	-0.0
10	2	4	2.7	-3.0
10	3	-8	0.7	0.7
10	3	-7	1.2	3.5
10	3	-6	2.4	-4.9
10	3	-5	0.0	-2.8
10	3	-4	0.0	3.4
10	3	-3	5.8	6.8
10	3	-2	3.7	-3.2
10	3	-1	0.0	-3.6
10	3	0	2.0	-0.2
10	3	1	1.7	2.6
10	3	2	0.0	0.1
10	3	3	1.6	-1.9
10	4	-7	0.0	1.2
10	4	-6	0.0	-1.8
10	4	-5	0.0	1.2
10	4	-4	0.0	2.3
10	4	-3	0.0	-0.3
10	4	-2	0.0	1.1
10	4	-1	0.0	1.8
10	4	0	2.3	0.5
10	4	1	1.1	1.5
10	5	-4	0.0	3.5
10	5	-3	2.9	3.8
10	5	-2	0.0	-2.0
10	5	-1	0.0	-2.7

$D_m = 1{\cdot}54$ g.cm^{-3}, $Z = 4$, $D_x = 1{\cdot}56$ g.cm^{-3}.
$F(000) = 592$.
Absorption coefficient for Cu $K\alpha$ radiation, $\mu = 48$ cm^{-1}.
Absent reflexions: $h0l$ when h is odd, $0k0$ when k is odd. Space group is $P2_1/a$.

The intensities of the reflexions were measured on a General Electric Spectrogoniometer, with Single Crystal Orienter, scintillation counter, approximately monochromatic Cu $K\alpha$ radiation (nickel filter and pulse height analyser), and the moving-crystal moving-counter method (Furnas, 1957). All 2506 reflexions in the range $0 < 2\theta \leq 145°$ (corresponding to a minimum interplanar spacing $d = 0{\cdot}81$ Å) were examined, and 2059 (82%) had an intensity above background, although a large number were of low intensity. The intensities were corrected for background, Lorentz and polarization factors were applied, and the structure amplitudes were derived. The crystals were all plates, that used in recording the intensities being mounted with **a*** parallel to the φ axis of the goniostat, and having cross-section 0·6 mm × 0·1 mm; no absorption corrections were applied, so that there are some inaccuracies in the measured structure amplitudes as a result of absorption.

Structure analysis

The position of the bromine atom was determined from the three axial Patterson projections as (0·25, 0·07, 0·23). A three-dimensional Fourier series was summed with signs of the structure factors based on bromine only; as a result of the pseudo-special coordinates of the bromine atom a large number of planes had systematically small calculated structure factors, and the corresponding F_o terms were omitted from the summation. However, on the resulting electron-density distribution all the Br, C and O atoms were well resolved, with very little spurious detail due to false symmetry. Structure factors were calculated using the scattering factors of *International Tables for X-ray Crystallography* (1962), with $B = 4{\cdot}0$ Å^2 for all the atoms; the discrepancy index R was 0·39 for the observed reflexions.

Refinement of the positional and thermal parameters was carried out initially by computing differential syntheses, correcting for series termination errors by the 'backshift' method. Three cycles reduced R to 0·22. Further refinement was by (block-diagonal) least-squares; the function minimized was $\Sigma w(F_o - F_c)^2$, with $\sqrt{w} = |F_o|/20$ when $|F_o| < 20$, and $\sqrt{w} = 20/|F_o|$ when $|F_o| \geq 20$. Two cycles with isotropic thermal parameters, and two further cycles with anisotropic thermal parameters reduced R to 0·18 for the 2059 observed reflexions. Measured and calculated structure factors are listed in Table 1. A final set of observed and calculated differential syntheses was computed, and these indicated small changes in positional parameters. The shifts were applied but structure factors were not recalculated.

A final three-dimensional Fourier series was summed, and superimposed sections of the resulting elec-

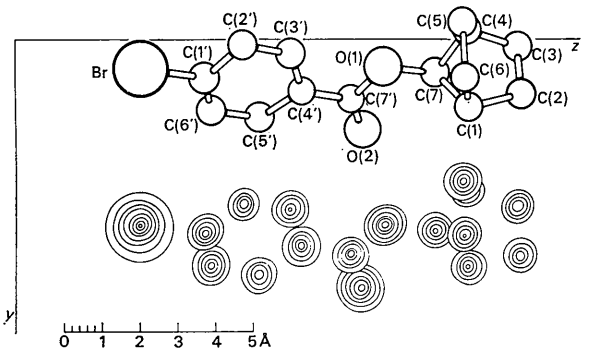

Fig. 1. Superimposed sections of the final three-dimensional electron-density distribution, through the atomic centres parallel to (100). Contours start at 2 e.Å^{-3}, and are at intervals of 1 e.Å^{-3} for C and O, and 5 e.Å^{-3} for Br. A perspective drawing of the molecule is also shown.

Table 2. *Final positional parameters (fractional) with standard deviations* (Å), *and anisotropic thermal parameters* ($b_{ij} \times 10^4$)

Atom	x	y	z	$\sigma(x)$	$\sigma(y)$	$\sigma(z)$	b_{11}	b_{22}	b_{33}	b_{23}	b_{13}	b_{12}
Br	0·2540	0·0733	0·2343	0·0021	0·0023	0·0016	193	135	48	−19	55	−36
O(1)	0·1719	0·0652	0·7065	0·012	0·011	0·009	155	93	31	25	6	55
O(2)	0·0241	0·2393	0·6569	0·016	0·013	0·012	239	88	55	−15	24	126
C(1′)	0·2085	0·0945	0·3600	0·012	0·014	0·009	113	146	32	−45	6	10
C(2′)	0·2817	0·0171	0·4352	0·018	0·018	0·015	170	73	48	−12	−11	45
C(3′)	0·2472	0·0349	0·5252	0·014	0·015	0·015	96	47	44	10	−12	11
C(4′)	0·1400	0·1264	0·5450	0·016	0·014	0·014	131	77	34	5	−8	−63
C(5′)	0·0589	0·1976	0·4673	0·016	0·016	0·014	145	74	37	6	−62	28
C(6′)	0·0977	0·1841	0·3761	0·016	0·016	0·013	139	93	37	11	5	13
C(7′)	0·1050	0·1501	0·6399	0·015	0·015	0·014	98	93	42	50	−11	10
C(1)	0·2679	0·1716	0·8630	0·023	0·017	0·015	248	90	33	7	51	−8
C(2)	0·2373	0·1383	0·9628	0·022	0·020	0·018	237	132	42	−28	62	1
C(3)	0·1848	0·0149	0·9609	0·019	0·018	0·013	127	179	31	3	62	−34
C(4)	0·1912	−0·0389	0·8620	0·020	0·017	0·015	145	100	44	29	17	−6
C(5)	0·3645	−0·0501	0·8532	0·015	0·017	0·014	102	95	47	23	30	26
C(6)	0·4216	0·0951	0·8561	0·018	0·024	0·017	159	99	60	9	68	−19
C(7)	0·1483	0·0861	0·8038	0·016	0·016	0·013	174	79	35	32	29	−18

tron-density distribution are shown, together with a perspective drawing of the molecule, in Fig. 1.

Atomic parameters and molecular dimensions

The final positional (fractional) and anisotropic thermal ($\exp\{-\Sigma b_{ij}h_ih_j\}$) parameters are given in Table 2, together with the positional parameter standard deviations (in Å) calculated from the least-squares residuals.

The bond lengths and valency angles, and their standard deviations, are given in Table 3.

Table 3. *Bond distances and standard deviations* (Å), *and bond angles* (°)

The angles have standard deviations in the range 0·9°–1·6°

Bond	l	σ	Angle	θ
Br–C(1′)	1·90	$0{\cdot}02_1$	Br–C(1′)–C(2′)	120·3
			Br–C(1′)–C(6′)	119·8
C(1′)–C(2′)	1·39	$0{\cdot}02_0$	C(6′)–C(1′)–C(2′)	119·8
C(2′)–C(3′)	1·37	$0{\cdot}02_2$	C(1′)–C(2′)–C(3′)	119·1
C(3′)–C(4′)	1·39	$0{\cdot}02_1$	C(2′)–C(3′)–C(4′)	122·9
C(4′)–C(5′)	1·40	$0{\cdot}02_1$	C(3′)–C(4′)–C(5′)	117·5
C(5′)–C(6′)	1·39	$0{\cdot}02_1$	C(4′)–C(5′)–C(6′)	119·8
C(6′)–C(1′)	1·38	$0{\cdot}01_9$	C(5′)–C(6′)–C(1′)	120·5
Mean C_{ar}–C_{ar}	1·39	$0{\cdot}00_8$	C(3′)–C(4′)–C(7′)	124·0
			C(5′)–C(4′)–C(7′)	118·5
C(4′)–C(7′)	1·44	$0{\cdot}02_1$		
C(7′)–O(2)	1·20	$0{\cdot}01_7$	C(4′)–C(7′)–O(2)	122·6
C(7′)–O(1)	1·34	$0{\cdot}01_8$	C(4′)–C(7′)–O(1)	113·9
O(1)–C(7)	1·44	$0{\cdot}01_8$	O(1)–C(7′)–O(2)	123·5
C(1)–C(2)	1·52	$0{\cdot}02_7$	C(7′)–O(1)–C(7)	117·2
C(3)–C(4)	1·51	$0{\cdot}02_4$	O(1)–C(7)–C(1)	114·4
C(4)–C(5)	1·56	$0{\cdot}02_3$	O(1)–C(7)–C(4)	109·1
C(5)–C(6)	1·56	$0{\cdot}02_5$		
C(6)–C(1)	1·58	$0{\cdot}02_7$	C(2)–C(1)–C(6)	103·7
C(1)–C(7)	1·50	$0{\cdot}02_3$	C(2)–C(1)–C(7)	99·6
C(4)–C(7)	1·52	$0{\cdot}02_3$	C(6)–C(1)–C(7)	101·9
Mean C–C	1·54	$0{\cdot}00_9$	C(1)–C(2)–C(3)	107·9
			C(2)–C(3)–C(4)	107·0
C(2)–C(3)	1·34	$0{\cdot}02_6$	C(3)–C(4)–C(5)	107·0
			C(3)–C(4)–C(7)	98·6
			C(5)–C(4)–C(7)	100·0
			C(4)–C(5)–C(6)	104·2
			C(5)–C(6)–C(1)	101·1
			C(1)–C(7)–C(4)	95·6

Table 4. *Equations of various planes, deviations of the atoms from the planes* (Å), *and angles between the normals*

Equations

A: C(1), C(4), C(5), C(6)
$-0{\cdot}064X'+0{\cdot}034Y-0{\cdot}997Z'+11{\cdot}924=0$

B: C(1), C(2), C(3), C(4)
$-0{\cdot}908X'+0{\cdot}300Y-0{\cdot}290Z'+3{\cdot}288=0$

D: C(1), C(4), C(7)
$0{\cdot}766X'-0{\cdot}237Y-0{\cdot}598Z'+7{\cdot}366=0$

Deviations

Atom	Δ_A	Δ_B	Δ_D
C(1)	0·007	0·016	0
C(2)		−0·025	
C(3)		0·021	
C(4)	−0·008	−0·012	0
C(5)	0·011		
C(6)	−0·011		
C(7)			0

Table 4 (*cont.*)

Angles	A⌒B	111°
	A⌒D	123°
	B⌒D	126°

Equations of the planes through the regions of the molecule which are of particular interest are given in Table 4 (X', Y, Z' in Å referred to orthogonal axes a, b, c^*), together with the deviations of the atoms from the planes, and the interplanar angles.

All the intermolecular separations ≤4·0 Å were calculated, and those less than 3·8 Å are listed in Table 5. The packing of the molecules in the unit cell is shown in Fig. 2.

Table 5. *Shorter intermolecular distances*

All intermolecular distances ≤3·8 Å between a standard molecule (1) and its neighbours are given

Atom (molecule 1)	to	Atom	in	Molecule	d
O(1)		C(1′)		2	3·70 Å
O(1)		C(6′)		2	3·53
O(2)		C(6)		3	3·52
C(1′)		C(7′)		2	3·72
C(2′)		C(5′)		3	3·77
C(2′)		C(7′)		2	3·79
C(3′)		O(2)		3	3·62
C(3′)		C(4′)		2	3·76
C(3′)		C(5′)		2	3·60
C(4′)		O(2)		3	3·74
C(4′)		C(4′)		2	3·64
C(4′)		C(5′)		2	3·72
C(3)		C(3)		5	3·62
Br		C(5)		4	3·78
Br		C(6)		4	3·74

Molecule		x	y	z
	1			
	2	$-x$	$-y$	$1-z$
	3	$\frac{1}{2}+x$	$\frac{1}{2}-y$	z
	4	$1-x$	$-y$	$1-z$
	5	$-x$	$-y$	$2-z$

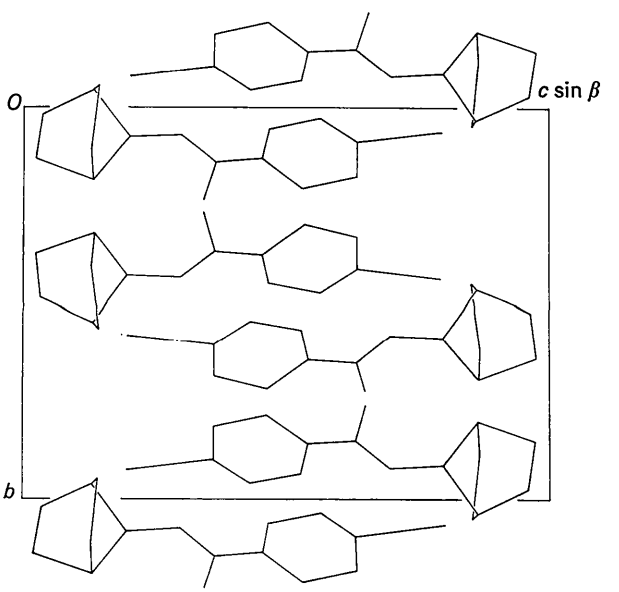

Fig. 2. Projection of the structure along [100], illustrating the packing of the molecules in the crystal.

Discussion

The present analysis has established the complete structure of *anti*-7-norbornenyl *p*-bromobenzoate. The interesting part of the molecule is the norbornene nucleus. The carbon–carbon single bonds are all of normal length, the mean distance being 1·54 Å; the C=C bond length is 1·34 Å. The valency angles are given in Table 3 and are illustrated in Fig. 3; the norbornene nucleus has symmetry *m* within experimental error. All the angles are less than the tetrahedral value, indicating considerable strain in the skeleton; the angles at the double bond are only 107°. The bridgehead angle is 95·6°.

The corresponding features in the tricyclo-octane nucleus (Macdonald & Trotter, 1965) are included in Fig. 3 for comparison. The skeletons are obviously very similar. The angles between the various planes in the norbornenyl nucleus, illustrated in Fig. 3, are given in Table 4, $A\widehat{\ }B=111°$, $A\widehat{\ }D=123°$, $B\widehat{\ }D=126°$, identical with the corresponding angles in the tricyclo-octyl skeleton. The C(1)...C(4) distances are equal. The bridgehead angles (97° in the tricyclo-octane nucleus, 96° in the norbornene nucleus) are identical within experimental error.

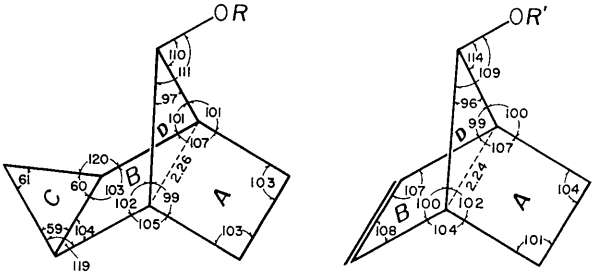

Fig. 3. Bond angles in the tricyclo-octane and norbornene nuclei.

The rate of solvolysis of the *p*-bromobenzenesulphonyl derivative of *anti*-7-norbornenol (III, $R=p$-bromobenzenesulphonyl) is about 10^{11} faster than that of the corresponding derivative of *anti*-8-tricyclo-octanol (V, $R=p$-bromobenzenesulphonyl), and since the bridgehead angles are the same in the two skeletons, variation in bridgehead angle cannot be the explanation for variation in solvolysis rate.

The bond distances and valency angles in the *p*-bromobenzoyl group are all normal; Br–C=1·90 Å, mean C_{ar}–C_{ar}=1·39 Å, C_{ar}–$C_{carboxyl}$=1·44 Å, C=O=1·20Å, C–OR=1·34 Å, O–R=1·44 Å, mean angle at C_{ar}=120°, C–C=O=123°, C–C–O=114°, O=C–O=123°, C–O–C=117°. The intermolecular distances (Table 5) all correspond to van der Waals interactions.

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On the Cementite Structure

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The preparation of single crystals of $(Fe_{2\cdot7}Mn_{0\cdot3})C$ has permitted a complete three-dimensional anisotropic refinement of the cementite structure with this composition. The atomic coordinates of the metal and carbon atoms have been determined to an estimated standard deviation of 0·001 Å and 0·01 Å respectively.

The structure can be rationally described in terms of a pleated-layer hexagonal arrangement of the iron atoms and as such is related to the structures of ε-iron and the ε-carbide.

Introduction

Cementite, Fe_3C, has been the subject of a large number of publications bearing upon its phase and structural relationships to iron and the other iron carbides, of which recent examples are those of Andrews (1963, 1964). This and closely related structures also occur in certain binary compounds especially borides, of the