is widely distributed throughout the anion rather than concentrated on any particular oxygen atom.

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References

BAK, B., CHRISTENSEN, D., DIXON, W. B., HANSEN-NYGAARD, L., ANDERSEN, J. R. & SCHOTTLÄNDER, M. (1962). *J. Mol. Spectr.* **9**, 124.

COCKER, W., DAVIS, W. J., MCMURRY, T. B. H. & START, P. A. (1959). *Tetrahedron*, **7**, 299.

CRUICKSHANK, D. W. J. (1960). *Acta Cryst.* **13**, 774.

DARLOW, S. F. (1961). *Acta Cryst.* **14**, 1257.

ELLISON, R. D. & LEVY, H. A. (1965). *Acta Cryst.* **19**, 260.

FERGUSON, G. & SIM, G. A. (1962). *Acta Cryst.* **15**, 346.

FREEMAN, A. J. (1959). *Acta Cryst.* **12**, 261.

GANTZEL, P. K., SPARKS, R. A. & TRUEBLOOD, K. N. (1962). *I.U.C. World List of Crystallographic Computer Programs*, No.384.

HUDSON, P. (1962). *Acta Cryst.* **15**, 919.

HUDSON, P. & ROBERTSON, J. H. (1964). *Acta Cryst.* **17**, 1497.

KENNARD, O. (1962). In *International Tables for X-ray Crystallography*, Vol.III, p.275. Birmingham: Kynoch Press.

MILLS, H. H. & SPEAKMAN, J. C. (1963). *J. Chem. Soc.* p.4355.

NARDELLI, M., FAVA, G. & GIRALDI, G. (1962). *Acta Cryst.* **15**, 737.

PETERSON, S. W. & LEVY, H. A. (1958). *J. Chem. Phys.* **29**, 948.

PIMENTEL, G. C. & McClellan, A. L. (1960). *The Hydrogen Bond*, pp.258–269. San Francisco: W. H. Freeman.

ROBERTSON, J. H. (1965). *Acta Cryst.* **18**, 410.

ROLLETT, J. S. (1961). In *Computing Methods and the Phase Problem in X-ray Crystal Analysis* (Ed. by R. Pepinsky, J. M. Robertson, and J. C. Speakman). p.87. New York: Pergamon Press.

WATSON, R. E. & FREEMAN, A. J. (1961). *Phys. Rev.* **123**, 521.

WILLIAMS, D. E. & RUNDLE, R. E. (1964). *J. Amer. Chem. Soc.* **86**, 1660.

Acta Cryst. (1967). **22**, 567

The Structure of the Twinned Crystal of D,L-6-Thioctic Acid, $C_8H_{14}O_2S_2$

BY I. L. KARLE, J. A. ESTLIN AND K. BRITTS

U.S. Naval Research Laboratory, Washington, D.C., 20390, *U.S.A.*

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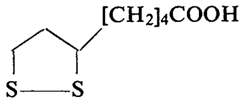
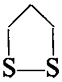
Crystals of 5-[3-(1,2-dithiolanyl)]pentanoic acid (D,L-6-thioctic acid, α-lipoic acid),



occur as twins with the twinning occurring along the c axis. The twinned crystals form an orthorhombic supercell with $a=133\cdot2$, $b=9\cdot89$, $c=9\cdot22$ Å. The real cell is monoclinic, space group $P2_1/c$, with $a=11\cdot75$, $b=9\cdot89$, $c=9\cdot22$ Å and $\beta=109°05'$. The molecules occur as dimers with the acid groups hydrogen-bonded across centers of symmetry. No four atoms in the five-membered ring are coplanar. Phases for the structure determination were obtained by the symbolic addition procedure.

Introduction

The naturally occurring (+) α-lipoic acid (Reed, De Busk, Gunsalus & Hornberger, 1951) is an essential factor for the enzymatic decarboxylation of pyruvate to acetate and is also required in the subsequent introduction of acetate into the citric acid cycle. The synthetic product, called D, L-6-thioctic acid (Bullock, Brockman, Patterson, Pierce, von Saltza, Sanders & Stokstad, 1954), has biological properties similar to those of α-lipoic acid. The initial interest in this material concerned the configuration of the  ring; however, since all the crystals which were examined were twinned, the nature of the twinning became equally interesting.

Experimental

D, L-6-Thioctic acid, obtained from the Aldrich Chemical Company, was recrystallized from ethyl alcohol. The crystals were light yellow rectangular plates elongated along the *c* axis. A crystal was mounted on the *c* axis and precession and Weissenberg photographs were taken with Cu *Kα* radiation.

The zero layer Weissenberg photograph appeared quite normal, whereas the first through the fifth layers

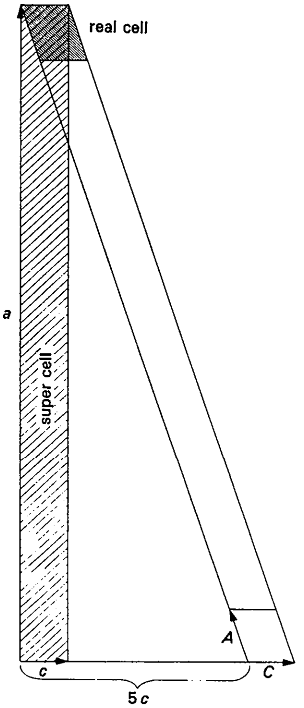


Fig. 1. Relationship between orthorhombic supercell and monoclinic real cell.

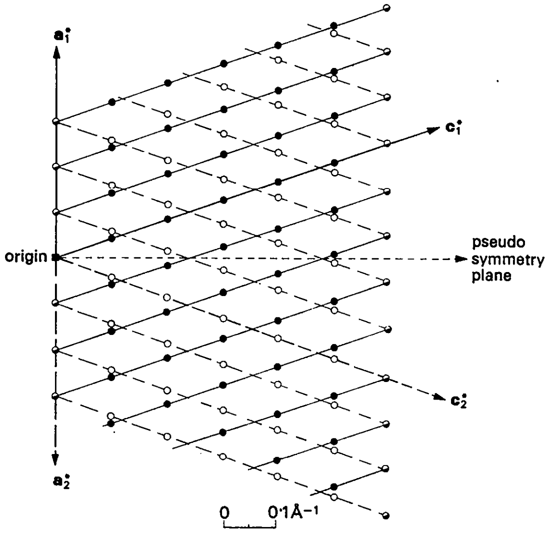


Fig. 2. The superimposed *a*c** nets of the twinned individuals. The closed circles belong to one individual whereas the open circles belong to the other. The half-closed circles belong to both. No attempt was made to indicate intensities.

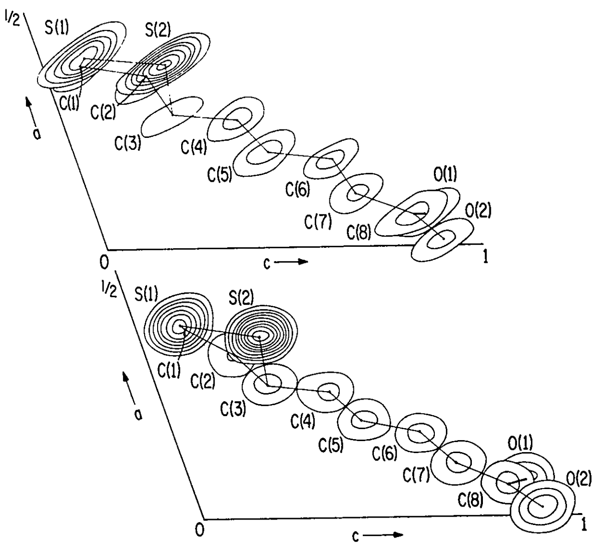


Fig. 3. The upper figure represents sections from a three-dimensional *E* map projected along [010]. This map was computed from the 200 terms with $|E| > 1{\cdot}5$ for which phases had been determined by the symbolic addition procedure. The equally spaced contours are on an arbitrary scale. The lower figure represents sections from the final electron density map computed with 1149 terms. The contours are spaced at 2 e.Å^{-3} beginning with the 2 e.Å^{-3} level.

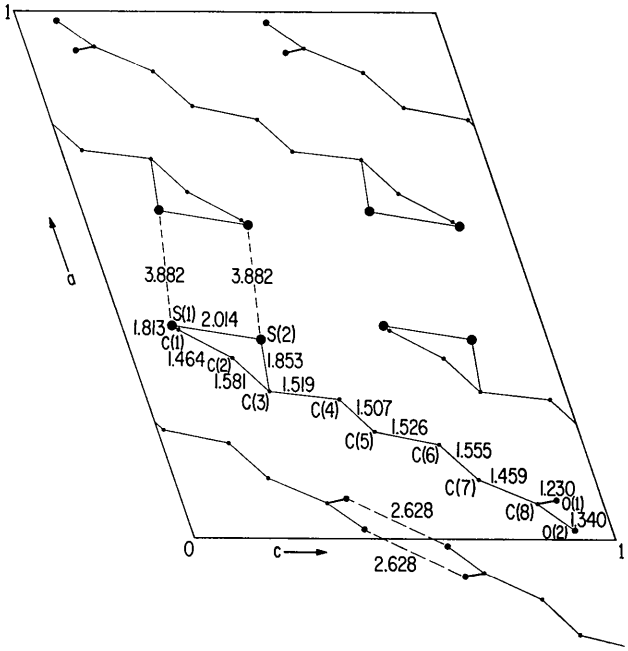


Fig. 4. The contents of a unit cell and the bond lengths in the molecule. The standard deviations for the bond lengths range from 0·022 Å for the S–S bond to 0·035 Å for the C–C bonds.

Table 1. *Observed and calculated structure factors*

Successive columns contain $l, |F_{\text{obs}}|, |F_{\text{cal}}|$, and α_{cal} (radians).

-11 4 L			
2	6.7	7.2	0.00
4	4.9	8.8	3.14
-11 5 L			
2	4.2	3.2	0.00
-11 6 L			
1	3.7	4.1	0.00
2	10.9	10.4	3.14
3	10.9	12.2	3.14
4	4.3	8.2	3.14
-11 7 L			
2	2.7	2.8	0.00
5	5.2	9.1	3.14
-11 8 L			
2	9.8	13.1	3.14
-10 1 L			
5	18.9	27.5	3.14
-10 2 L			
2	8.3	9.6	0.00
3	23.4	22.1	3.14
-10 3 L			
1	9.9	8.9	0.00
2	4.6	2.3	0.00
3	6.9	6.5	0.00
5	6.7	7.9	3.14
-10 4 L			
2	9.1	5.6	3.14
3	6.4	8.3	3.14
4	20.9	26.7	0.00
-10 5 L			
2	8.2	9.8	3.14
-10 6 L			
1	4.2	3.2	3.14
2	7.0	6.5	3.14
3	10.4	10.5	0.00
4	0.0	1.2	0.00
5	6.8	14.4	3.14
-10 7 L			
1	7.6	8.2	3.14
2	0.0	0.1	3.14
5	5.8	9.5	0.00
-10 8 L			
1	3.2	3.7	0.00
2	0.0	3.2	3.14
3	2.7	5.0	0.00
4	5.2	11.7	3.14
-10 9 L			
1	3.3	5.4	3.14
2	4.4	5.8	0.00
3	2.1	4.0	0.00
-9 1 L			
1	8.7	8.2	0.00
2	8.6	5.7	3.14
3	13.0	11.2	3.14
4	17.9	13.9	0.00
5	18.7	19.7	0.00
-9 2 L			
1	24.6	21.3	0.00
2	7.2	4.5	0.00
3	18.9	18.5	0.00
4	9.9	10.3	0.00
5	7.7	10.9	3.14
-9 3 L			
1	5.8	2.9	3.14
2	11.8	7.8	3.14
3	18.5	17.8	0.00
4	17.3	17.2	0.00
5	0.0	2.4	0.00
-9 4 L			
2	16.0	12.8	0.00
3	9.8	12.2	0.00
4	16.8	17.1	3.14
5	4.4	5.8	0.00
-9 5 L			
1	5.9	4.4	0.00
2	5.3	5.8	0.00
4	4.5	5.8	3.14
5	7.9	12.5	3.14
-9 6 L			
1	10.4	9.4	3.14
2	4.5	2.3	0.00
3	0.0	2.7	0.00
4	4.4	6.6	3.14
5	5.4	9.9	0.00
-9 7 L			
1	12.0	11.4	0.00
2	7.4	7.9	0.00
3	18.1	21.9	3.14
4	8.3	14.4	3.14
5	0.0	4.0	3.14
-9 8 L			
1	0.0	0.7	0.00
2	0.0	2.1	3.14
3	0.0	0.2	0.00
4	6.4	10.7	0.00
-9 9 L			
1	0.0	2.5	0.00
2	3.2	3.7	3.14
3	7.3	11.0	3.14
-8 1 L			
1	11.1	9.1	0.00
2	18.3	14.7	0.00
3	42.8	38.5	0.00
4	27.2	21.8	3.14
5	18.9	20.6	3.14
-8 2 L			
1	0.0	3.3	0.00
2	14.3	10.0	0.00
3	8.1	7.0	0.00
4	19.3	16.0	0.00
5	2.3	5.4	0.00
-8 3 L			
1	6.5	4.3	0.00
2	9.7	8.8	0.00
3	10.7	8.8	3.14
4	30.0	29.0	3.14
5	26.7	30.9	0.00
-8 4 L			
1	10.8	9.7	0.00
2	12.4	11.5	0.00
3	0.0	0.7	3.14
4	10.0	8.3	0.00
5	0.0	0.8	0.00
-8 5 L			
1	4.5	3.8	3.14
2	13.3	11.1	3.14
3	29.7	31.2	3.14
4	0.0	1.9	0.00
5	15.5	23.4	0.00
-8 6 L			
1	15.0	14.6	0.00
2	12.1	11.0	3.14
3	5.3	2.4	0.00
4	4.5	5.6	3.14
5	2.5	2.9	3.14
-8 7 L			
1	5.8	4.5	3.14
2	0.0	1.6	3.14
3	8.9	10.6	3.14
4	7.5	11.0	0.00
5	6.8	10.3	3.14
-8 8 L			
1	0.0	1.7	0.00
2	11.6	12.4	3.14
3	6.4	5.7	0.00
4	0.0	0.6	0.00
-8 9 L			
1	3.7	3.4	0.00
2	6.8	6.5	0.00
3	0.0	1.7	3.14
-8 10 L			
1	6.8	8.0	3.14
2	2.9	2.3	0.00
3	0.0	0.4	3.14
-7 1 L			
1	31.7	28.2	3.14
2	18.0	15.6	0.00
3	28.7	24.8	3.14
4	23.2	19.3	0.00
5	27.2	24.0	0.00
-7 2 L			
1	15.9	13.1	0.00
2	8.1	5.7	3.14
3	20.9	17.9	3.14
4	8.1	6.3	0.00
5	17.1	-16.1	0.00
-7 3 L			
1	12.9	11.1	3.14
2	7.1	8.1	0.00
3	36.5	36.1	0.00
4	28.6	28.0	0.00
5	12.0	10.8	3.14
-7 4 L			
1	19.2	15.1	3.14
2	8.9	5.9	3.14
3	10.1	8.2	3.14
4	22.5	22.3	0.00
5	15.0	15.5	0.00
-7 5 L			
1	6.2	4.9	0.00
2	9.4	9.3	3.14
3	28.6	28.2	0.00
4	0.0	5.1	0.00
5	16.1	18.1	3.14
-7 6 L			
1	14.8	14.7	3.14
2	16.8	14.3	0.00
3	10.4	13.3	0.00
4	7.7	8.7	3.14
5	0.0	6.3	3.14
-7 7 L			
1	0.0	0.8	0.00
2	0.0	1.0	0.00
3	2.6	4.6	3.14
4	3.6	4.3	3.14
5	0.0	7.0	0.00
-7 8 L			
1	8.9	8.3	0.00
2	14.6	14.1	0.00
3	4.4	5.1	0.00
4	11.3	15.1	3.14
-7 9 L			
1	4.0	3.6	3.14
2	0.0	3.9	0.00
3	6.1	6.3	3.14
4	2.2	1.8	0.00
-7 10 L			
1	9.2	9.6	0.00
2	5.5	4.4	3.14
3	5.4	7.8	3.14
-6 1 L			
1	10.0	5.9	3.14
2	24.8	19.5	0.00
3	22.5	17.8	0.00
4	4.6	1.8	3.14
5	18.0	13.7	0.00
-6 2 L			
1	16.6	11.9	0.00
2	10.0	8.5	0.00
3	39.1	38.3	0.00
4	24.5	18.4	0.00
5	33.8	31.8	3.14
-6 3 L			
1	44.5	41.0	3.14
2	29.6	24.6	0.00
3	28.6	28.0	0.00
4	8.9	7.4	0.00
5	19.2	15.3	0.00
-6 4 L			
1	10.1	6.8	3.14
2	21.1	17.6	0.00
3	3.2	2.6	0.00
4	42.2	43.7	3.14
5	14.3	13.9	3.14
-6 5 L			
1	20.1	17.1	3.14
2	14.5	14.5	0.00
3	6.3	4.4	3.14
4	22.0	21.2	0.00
5	3.4	5.3	3.14
-6 6 L			
1	12.4	9.1	3.14
2	7.6	5.8	3.14
3	17.8	16.5	3.14
4	31.7	35.2	3.14
5	7.5	6.8	0.00
-6 7 L			
1	21.1	17.9	0.00
2	4.6	6.6	0.00
3	15.9	18.9	3.14
4	13.7	13.6	0.00
5	0.0	10.8	3.14
-6 8 L			
1	7.9	6.3	3.14
2	7.4	5.4	3.14
3	0.0	1.0	0.00
4	0.0	0.7	3.14
-6 9 L			
1	12.1	10.6	0.00
2	9.3	8.4	3.14
3	0.0	0.1	3.14
4	0.0	3.1	3.14
-6 10 L			
1	5.3	5.6	3.14
2	8.6	8.5	0.00
3	7.2	8.2	0.00
-6 11 L			
1	4.6	5.2	3.14
2	6.1	9.1	3.14
3	6.2	9.4	0.00
4	3.6	6.4	3.14
-5 1 L			
1	15.0	12.6	3.14
2	30.1	24.1	0.00
3	9.5	9.7	3.14
4	16.4	11.8	3.14
5	22.5	16.9	3.14
-5 2 L			
1	22.9	19.0	3.14
2	22.6	17.5	3.14
3	5.3	5.6	3.14
4	5.4	4.4	0.00
5	31.8	29.7	0.00
-5 3 L			
1	15.2	11.8	0.00
2	6.0	5.2	0.00
3	12.3	12.9	3.14
4	21.1	19.2	3.14
5	21.1	21.5	0.00
-5 4 L			
1	14.3	12.2	3.14
2	16.7	12.2	3.14
3	13.0	11.6	3.14
4	47.1	50.4	0.00
5	15.2	13.3	0.00
-5 5 L			
1	7.8	7.4	0.00
2	29.7	28.4	3.14
3	0.0	1.0	0.00
4	0.0	0.8	0.00
5	13.3	12.5	0.00
-5 6 L			
1	15.1	13.3	0.00
2	0.0	1.3	3.14
3	9.2	8.1	0.00
4	9.2	9.0	0.00
5	9.8	10.3	3.14
-5 7 L			
1	12.7	8.4	3.14
2	3.7	2.9	3.14
3	16.3	18.4	0.00
4	11.2	12.4	0.00
5	6.2	5.9	3.14
-5 8 L			
1	11.2	9.7	0.00
2	5.3	2.7	0.00
3	10.5	11.7	0.00
4	12.4	14.5	3.14
-5 9 L			
1	0.0	2.9	0.00
2	17.8	17.6	0.00
3	5.0	5.6	0.00
4	0.0	0.3	0.00
5	3.9	4.7	3.14
-5 10 L			
1	6.9	6.0	3.14
2	0.0	1.0	0.00
3	0.0	0.5	3.14
4	5.1	5.6	3.14
-5 11 L			
1	13.7	13.8	0.00
2	6.9	8.2	0.00
3	7.5	9.5	3.14
4	4.1	6.5	3.14
-4 1 L			
1	47.9	44.6	3.14
2	4.1	2.1	0.00
3	66.8	58.7	3.14
4	25.6	27.4	0.00
5	48.4	44.3	0.00
-4 2 L			
1	46.7	42.2	0.00
2	64.3	49.7	3.14
3	62.8	59.6	0.00
4	41.6	34.8	0.00
5	5.8	5.3	3.14
-4 3 L			
1	23.1	20.3	3.14
2	23.7	15.1	3.14
3	32.0	29.4	0.00
4	25.8	27.9	0.00
5	16.7	16.2	3.14
-4 4 L			
1	5.7	3.9	0.00
2	40.6	37.0	3.14
3	22.0	22.4	0.00
4	16.6	15.1	0.00
5	29.8	28.7	0.00
-4 5 L			
1	32.8	33.2	0.00
2	16.0	13.5	3.14
3	19.0	20.6	0.00
4	11.7	11.7	3.14
5	45.2	44.1	3.14
-4 6 L			
1	10.6	8.6	3.14
2	13.3	11.9	0.00
3	0.0	1.0	3.14
4	12.7	13.1	3.14
5	30.6	31.6	0.00
-4 7 L			
1	37.9	38.6	0.00
2	0.0	0.3	0.00
3	12.1	12.2	3.14
4	15.0	15.0	3.14
5	15.9	13.8	3.14
-4 8 L			
1	11.2	8.9	0.00
2	22.7	20.7	0.00
3	4.6	5.3	3.14
4	0.0	2.0	3.14
-4 9 L			
1	10.5	7.8	0.00
2	6.9	6.8	3.14
3	0.0	0.7	3.14
4	0.0	2.9	0.00
5	5.1	4.7	0.00
-4 10 L			
1	7.6	8.7	0.00
2	0.0	2.6	3.14
3	6.9	9.1	3.14
4	6.5	8.0	0.00
-4 11 L			
1	5.3	4.2	3.14
2	2.8	3.6	3.14
3	8.1	9.9	0.00
4	4.5	5.5	0.00
-4 12 L			
2	8.0	10.0	3.14
-3 1 L			
1	46.8	37.4	0.00
2	14.6	11.1	0.00
3	15.6	12.6	0.00
4	22.4	23.2	3.14
5	37.6	35.5	3.14
-3 2 L			
1	14.5	11.6	3.14
2	10.2	3.8	0.00
3	32.2	32.4	0.00
4	0.0	0.8	3.14
5	15.5	13.1	3.14
-3 3 L			
1	33.2	32.4	3.14
2	10.0	9.7	3.14
3	19.2	17.2	3.14
4	27.3	29.7	3.14
5	28.9	27.6	0.00
-3 4 L			
1	12.4	11.1	3.14
2	11.7	9.3	0.00
3	15.8	15.7	3.14
4	5.5	2.6	3.14
5	8.0	6.6	3.14
-3 5 L			
1	29.4	26.9	3.14
2	21.7	18.9	3.14
3	24.4	25.8	3.14
4	8.1	7.2	0.00
5	25.5	21.9	0.00
-3 6 L			
1	13.1	10.2	0.00
2	18.0	17.8	3.14
3	21.4	21.6	3.14
4	0.0	2.8	0.00
5	7.6	7.5	0.00
-3 7 L			
1	2.5	2.1	0.00
2	0.0	3.4	0.00
3	0.0	0.1	0.00
4	19.0	19.8	0.00
5	12.1	10.7	3.14
-3 8 L			
1	0.0	1.7	0.00
2	12.0	10.0	3.14
3	7.9	8.4	0.00
4	9.0	8.5	0.08
5	8.6	8.5	0.00
-3 9 L			
1	9.1	8.7	0.00
2	0.0	1.7	0.00
3	11.8	12.6	0.00
4	0.0	2.6	0.00
5	10.4	10.3	3.14
-3 10 L			
1	12.2	10.9	3.14
2	16.6	15.9	0.00
3	10.8	12.0	0.00
4	0.0	0.4	3.14
-3 11 L			
1	6.0	5.0	0.00
2	5.1	5.7	3.14
3	0.0	3.0	0.00
4	0.0	2.6	3.14
-3 12 L			
2	10.7	13.4	0.00
-2 1 L			
1	57.4	55.7	3.14
2	17.6	15.9	0.00
3	45.6	55.8	3.14
4	39.3	40.3	0.00
5	23.4	19.4	0.00
-2 2 L			
1	19.3	18.8	3.14
2	34.7	36.0	3.14
3	27.2	29.4	3.14
4	28.6	26.5	3.14
5	23.8	24.5	0.00
-2 3 L			
1	42.9	39.3	0.00
2	7.2	1.2	0.00
3	33.6	31.3	3.14
4	39.8	40.7	0.00
5	21.8	19.6	0.00
-2 4 L			
1	47.9	49.5	3.14
2	10.9	6.9	0.00
3	28.7	27.4	3.14
4	34.8	34.3	0.00
5	7.9	5.6	0.00
-2 5 L			
1	39.1	40.1	0.00
2	16.0	14.6	3.14
3	5.0	7.2	0.00
4	6.0	5.2	3.14
5	0.0	1.4	3.14
-2 6 L			
1	32.8	25.1	3.14
2	48.0	49.2	0.00
3	6.4	4.5	0.00
4	6.2	5.6	0.00
5	17.4	16.0	3.14
-2 7 L			
1	15.0	14.9	3.14
2	6.9	5.6	0.00
3	18.2	22.0	0.00
4	8.7	9.2	3.14
5	0.0	1.7	3.14
-2 8 L			
1	6.3	5.5	0.00
2	35.6	35.1	0.00
3	5.2	3.6	0.00
4	9.0	8.6	3.14
5	7.9	8.0	3.14
-2 9 L			
1	25.0	22.3	3.14
2	12.0	11.8	0.00
3	5.8	3.7	0.00
4	0.0	1.5	3.14
5	6.6	5.9	0.00
-2 10 L			
1	8.0	5.2	0.00
2	5.0	1.8	0.00
3	5.9	4.7	3.14
4	0.0	2.6	0.00
5	0.0	2.7	0.00
-2 11 L			
1	3.1	0.4	0.00
2	0.0	2.2	0.00
3	8.1	10.5	3.14
4	0.0	2.9	3.14
-2 12 L			
2	5.8	6.1	3.14
3	3.3	4.5	0.00
-1 0 L			
4	17.7	20.6	0.00
-1 1 L			
1	45.6	47.2	0.00
2	3.8	1.4	3.14
3	9.7	13.9	3.14
4	19.6	16.4	0.00
5	7.2	7.4	0.00
-1 2 L			
1	52.9	57.9	0.00
2	0.0	3.7	3.14
3	30.2	39.0	0.00
4	0.0	2.2	0.00
5	38.3	38.5	3.14
-1 3 L			
1	45.8	43.6	3.14
2	11.3	8.7	3.14
3	7.7	8.0	0.00
4	9.6	8.5	0.00
5	11.3	10.7	0.00
-1 4 L			
1	12.0	10.4	0.00
2	18.6	15.9	3.14
3	19.1	19.2	3.14
4	28.3	29.0	3.14
5	9.7	9.2	3.14
-1 5 L			
1	14.4	11.0	3.14
2	12.3	9.3	0.00
3	14.3	16.1	3.14
4	17.3	15.3	3.14
5	3.8	0.4	3.14
-1 6 L			
1	19.6	17.8	3.14
2	0.0	0.6	3.14
3	24.1	25.7	3.14
4	18.0	19.1	3.14
5	21.2	18.2	0.00
-1 7 L			
1	25.1	20.8	0.00
2	16.9	15.3	0.00
3	20.4	22.4	3.14
4	7.1	6.9	3.14
5	0.0	2.0	3.14
-1 8 L			
1	10.9	9.7	3.14
2	7.8	5.7	0.00
3	0.0	1.1	0.00
4	10.3	11.1	0.00
5	7.1	6.7	0.00
-1 9 L			
1	4.6	0.1	3.14
2	11.0	9.4	3.14
3	4.5	5.7	0.00
4	5.0	4.9	0.00
5	0.0	2.2	0.00
-1 10 L			
1	0.0	0.2	3.14
2	6.1	5.9	0.00
3	2.4	2.8	0.00
4	9.0	9.9	0.00
5	4.0	3.4	3.14
-1 11 L			
1	12.6	11.5	3.14
2	9.2	8.3	3.14
3	13.6	15.5	0.00
4	0.0	2.4	0.00
-1 12 L			
2	3.2	4.4	0.00
3	1.4	2.0	3.14
0 0 L			
2	46.9	44.3	3.14
4	42.2	42.6	3.14
0 1 L			
1	11.1	16.4	0.00
2	19.2	17.8	0.00
3	20.0	17.6	3.14
4	21.1	19.8	3.14
5	47.8	52.0	3.14
0 2 L			
1	6.3	7.6	3.14
2	0.0	1.6	0.00
3	15.1	18.2	3.14
4	21.0	17.1	3.14
5	36.2	30.7	0.00
0 3 L			
1	26.0	23.8	3.14
2	21.6	20.8	3.14
3	18.3	17.6	3.14
4	31.2	31.4	3.14
5	9.7	8.1	0.00
0 4 L			
0	69.6	72.1	3.14
1	20.0	10.9	3.14
2	24.2	26.1	0.00
3	26.3	26.1	3.14
4	14.1	12.2	0.00
5	8.3	5.9	0.00
0 5 L			
1	22.3	20.8	3.14
2	49.3	49.0	3.14
3	13.3	12.7	0.00
4	6.1	4.5	3.14
5	17.9	17.3	0.00
0 6 L			
0	26.2	22.6	3.14
1	17.1	15.9	0.00
2	10.8	9.7	3.14
3	3.2	1.6	3.14
4	14.0	14.0	0.00
5	12.9	12.4	3.14
0 7 L			
1	15.8	14.6	3.14
2	11.1	6.4	3.14
3	35.2	37.6	0.00
4	10.1	8.8	0.00
5	0.0	1.4	3.14
0 8 L			
0	12.9	9.4	0.00
1	14.1	14.1	0.00
2	27.6	25.4	3.14
3	12.8	10.9	0.00
4	6.8	4.1	0.00
5	0.0	1.4	3.14
0 9 L			
1	6.5	5.2	3.14
2	15.0	15.5	0.00
3	18.5	20.5	0.00
4	0.0	0.7	3.14
5	0.0	2.2	3.14
0 10 L			
0	2.5	2.2	3.14
1	0.0	2.5	3.14
2	7.9	6.2	3.14
3	2.4	1.6	0.00
4	3.8	3.9	3.14
5	2.7	4.0	0.00
0 11 L			
1	6.7	6.1	0.00
2	0.0	2.9	0.00
3	0.0	3.1	3.14
4	0.0	0.2	0.00
0 12 L			
1	5.1	0.4	3.14
2	4.1	4.1	0.00
3	2.3	2.2	3.14
1 0 L			
0	16.1	15.4	3.14
2	32.3	29.6	0.00
4	10.3	8.3	3.14
1 1 L			
0	26.5	31.2	3.14
1	36.5	34.0	3.14
2	19.2	14.7	0.00
3	12.2	15.2	3.14
4	27.9	29.1	0.00
5	5.2	4.5	0.00
1 2 L			
0	8.3	8.4	0.00
1	66.6	64.7	0.00
2	10.5	8.6	3.14
3	0.0	2.0	3.14
4	0.0	1.9	0.00
5	7.4	7.0	0.00
1 3 L			
0	29.5	30.3	3.14
1	17.2	11.7	0.00
2	14.0	9.9	0.00
3	11.0	8.6	0.00
4	22.2	19.2	0.00
5	16.6	13.5	3.14
1 4 L			
0	37.6	40.1	0.00
1	0.0	1.0	0.00
2	21.7	17.8	3.14
3	0.0	2.0	0.00
4	2.9	2.5	3.14
5	0.0	1.1	3.14
1 5 L			
0	18.0	14.9	0.00
1	33.4	30.5	0.00
2	6.4	5.0	3.14
3	15.6	15.9	0.00
4	26.2	22.7	3.14
5	22.8	21.5	3.14
1 6 L			
0	22.4	20.1	0.00
1	31.7	28.6	3.14
2	18.1	14.2	0.00
3	15.1	15.1	0.00
4	16.2	13.9	3.14
5	6.0	6.2	3.14
1 7 L			
0	22.7	21.7	0.00
1	0.0	2.9	0.00
2	7.0	6.3	3.14
3	7.6	6.5	0.00
4	16.7	13.2	3.14
5	0.0	1.3	3.14
1 8 L			
0	19.0	16.6	3.14
1	0.0	3.3	3.14
2	12.9	8.6	0.00
3	3.7	0.7	0.00
4	0.0	2.6	3.14
5	0.0	2.6	3.14

Table 1 (*cont.*)

	1	9	L
0	2.6	2.3	0.00
1	16.5	14.3	3.14
2	11.0	8.9	3.14
3	3.6	2.5	0.00
4	6.0	6.0	0.00
5	0.0	9.1	0.00
	1	10	L
0	12.9	12.0	3.14
1	10.8	9.0	0.00
2	13.4	12.9	3.14
3	9.4	10.1	3.14
4	7.4	7.5	0.00
5	0.0	0.1	3.14
	1	11	L
0	3.8	3.3	3.14
1	4.9	5.1	3.14
2	4.2	3.7	3.14
3	3.3	4.5	0.00
4	2.9	4.1	0.00
	1	12	L
0	2.4	1.7	0.00
2	7.6	10.8	3.14
3	2.1	0.4	0.00
	2	0	L
0	6.0	10.9	0.00
2	26.1	24.5	0.00
4	0.0	0.3	0.00
	2	1	L
0	11.8	20.4	0.00
1	175.0	188.4	0.00
2	17.3	10.5	0.00
3	4.7	4.0	0.00
4	12.0	13.4	3.14
5	18.4	17.3	3.14
	2	2	L
0	27.7	25.7	3.14
1	2.7	0.8	0.00
2	13.6	11.9	0.00
3	21.1	22.0	0.00
4	7.9	6.8	3.14
5	20.7	20.1	3.14
	2	3	L
0	29.0	29.6	0.00
1	21.9	15.1	3.14
2	5.1	1.2	0.00
3	6.9	7.1	0.00
4	27.3	24.7	3.14
5	11.4	9.0	0.00
	2	4	L
0	38.0	35.8	3.14
1	13.9	9.3	0.00
2	5.3	2.4	3.14
3	26.4	25.0	3.14
4	12.8	11.4	3.14
5	13.1	11.5	3.14
	2	5	L
0	7.6	4.5	3.14
1	52.6	43.2	3.14
2	0.0	0.0	3.14
3	10.9	10.7	3.14
4	9.5	7.6	3.14
5	14.3	11.3	0.00
	2	6	L
0	14.0	11.0	3.14
1	10.1	9.7	0.00
2	17.4	13.1	3.14
3	23.7	22.3	3.14
4	8.6	6.0	0.00
5	3.5	3.0	0.00
	2	7	L
0	22.3	19.5	3.14
1	0.0	0.8	0.00
2	11.5	11.3	0.00
3	17.6	18.5	3.14
4	10.3	7.5	0.00
5	0.0	0.1	0.00
	2	8	L
0	6.6	5.1	3.14
1	7.6	5.1	3.14
2	15.4	13.7	3.14
3	10.5	11.6	0.00
4	17.9	17.9	0.00
5	0.0	1.9	0.00
	2	9	L
0	3.7	2.5	3.14
1	0.0	2.1	0.00
2	0.0	1.7	0.00
3	7.6	6.8	3.14
4	0.0	2.0	0.00
5	0.0	2.0	3.14
	2	10	L
0	0.0	0.6	3.14
1	10.7	10.0	3.14
2	3.4	2.2	0.00
3	9.1	10.1	0.00
4	5.4	6.1	0.00
5	0.0	0.4	3.14
	2	11	L
0	3.8	3.3	0.00
1	6.4	5.1	3.14
2	0.0	2.6	3.14
3	0.0	1.3	0.00
4	3.0	2.5	3.14
	2	12	L
0	2.8	2.4	0.00
2	6.2	8.6	0.00
	3	0	L
0	58.5	66.8	0.00
2	10.1	13.3	3.14
4	24.2	26.6	3.14
	3	1	L
0	51.7	52.7	3.14
1	29.6	24.5	0.00
2	38.8	37.9	0.00
3	0.0	3.6	3.14
4	0.0	0.1	0.00
5	0.0	1.8	0.00
	3	2	L
0	17.9	12.5	3.14
1	8.4	8.8	3.14
2	6.8	7.2	3.14
3	7.0	5.7	3.14
4	3.5	3.5	3.14
5	19.4	20.4	0.00
	3	3	L
0	55.9	53.2	3.14
1	32.6	27.9	0.00
2	31.7	26.8	0.00
3	9.1	9.2	3.14
4	0.0	0.5	3.14
5	0.0	1.6	0.00
	3	4	L
0	50.6	46.9	3.14
1	6.5	7.4	3.14
2	10.9	6.3	0.00
3	6.1	3.1	3.14
4	22.3	19.4	0.00
5	0.0	0.6	3.14
	3	5	L
0	12.3	11.4	0.00
1	25.6	23.8	0.00
2	23.6	21.7	3.14
3	4.7	1.1	0.00
4	0.0	3.5	0.00
5	3.5	3.9	3.14
	3	6	L
0	0.0	2.1	0.00
1	13.5	10.9	0.00
2	13.6	11.8	0.00
3	7.0	5.9	0.00,
4	11.7	10.6	0.00
5	15.9	13.8	3.14
	3	7	L
0	20.1	18.7	0.00
1	7.5	6.2	3.14
2	9.6	8.4	3.14
3	7.6	6.6	0.00
4	0.0	2.5	0.00
5	0.0	1.3	3.14
	3	8	L
0	13.5	9.3	0.00
1	14.9	12.8	0.00
2	0.0	0.1	0.00
3	0.0	2.3	3.14
4	0.0	0.4	0.00
5	0.0	0.5	3.14
	3	9	L
0	3.7	2.7	3.14
1	16.9	15.1	3.14
2	13.5	12.3	0.00
3	6.5	6.4	3.14
4	4.0	4.0	3.14
5	0.0	3.4	0.00
	3	10	L
0	4.9	5.3	3.14
1	0.0	1.0	0.00
2	4.7	5.0	3.14
3	6.8	8.4	3.14
4	0.0	1.7	0.00
5	0.0	5.8	0.00
	3	11	L
0	5.1	6.1	3.14
1	4.1	0.3	3.14
2	5.0	5.8	0.00
3	7.5	11.9	3.14
4	1.3	3.0	3.14
	3	12	L
0	3.3	4.0	3.14
	4	0	L
0	51.9	57.2	3.14
2	79.5	85.6	0.00
4	12.6	12.6	0.00
	4	1	L
0	18.0	16.5	0.00
1	5.2	1.6	0.00
2	2.6	0.9	0.00
3	4.4	6.0	0.00
4	11.1	12.1	0.00
5	0.0	1.5	0.00
	4	2	L
0	7.8	6.5	3.14
1	37.5	34.4	0.00
2	35.1	29.7	0.00
3	18.4	15.5	0.00
4	6.3	6.4	0.00
5	14.8	14.2	3.14
	4	3	L
0	37.7	32.6	0.00
1	34.9	29.0	3.14
2	3.5	6.6	0.00
3	10.3	12.9	0.00
4	6.9	5.5	0.00
5	5.4	5.4	3.14
	4	4	L
0	50.7	49.1	0.00
1	7.6	10.7	0.00
2	23.6	21.7	3.14
3	6.5	2.0	0.00
4	9.1	7.1	3.14
5	7.8	8.1	3.14
	4	5	L
0	26.6	19.2	0.00
1	19.0	13.4	3.14
2	15.7	14.8	0.00
3	0.0	2.0	0.00
4	11.2	12.1	3.14
5	6.2	5.6	3.14
	4	6	L
0	27.5	22.0	0.00
1	19.0	12.2	3.14
2	13.2	9.0	3.14
3	0.0	0.8	3.14
4	9.7	9.7	3.14
5	7.0	3.8	0.00
	4	7	L
0	4.3	1.5	0.00
1	0.0	3.6	0.00
2	4.5	4.5	0.00
3	11.6	12.7	3.14
4	0.0	1.0	3.14
5	4.6	4.2	0.00
	4	8	L
0	20.7	17.7	3.14
1	4.6	4.9	3.14
2	0.0	0.1	0.00
3	0.0	0.4	0.00
4	0.0	1.5	3.14
5	0.0	2.7	0.00
	4	9	L
0	0.0	1.1	3.14
1	0.0	0.1	0.00
2	12.3	11.9	3.14
3	4.1	5.7	3.14
4	6.4	7.9	0.00
5	6.2	6.4	0.00
	4	10	L
0	10.2	8.8	3.14
1	3.3	3.1	0.00
2	3.8	4.3	3.14
3	0.0	2.1	3.14
4	0.0	1.0	0.00
5	1.2	1.5	3.14
	4	11	L
0	0.0	0.5	0.00
1	10.7	2.8	3.14
2	5.4	7.8	3.14
3	3.2	5.5	0.00
	4	12	L
0	3.1	4.3	0.00
	5	0	L
0	48.0	49.7	0.00
2	22.7	21.6	0.00
4	5.7	6.3	3.14
	5	1	L
0	8.2	7.3	0.00
1	15.9	13.2	0.00
2	6.4	6.7	0.00
3	5.4	6.0	0.00
4	7.0	6.0	3.14
5	10.9	12.8	3.14
	5	2	L
0	3.1	1.8	0.00
1	47.6	46.4	3.14
2	22.9	18.8	0.00
3	20.6	21.7	0.00
4	0.0	3.6	3.14
5	0.0	0.5	0.00
	5	3	L
0	16.1	15.9	0.00
1	34.0	29.5	3.14
2	2.2	0.9	0.00
3	4.1	2.2	3.14
4	8.5	7.0	3.14
5	7.9	6.3	0.00
	5	4	L
0	36.2	32.4	3.14
1	16.9	13.4	3.14
2	16.6	14.4	0.00
3	0.0	4.2	3.14
4	0.0	0.2	3.14
5	0.0	0.5	3.14
	5	5	L
0	6.4	5.3	0.00
1	24.0	18.1	3.14
2	4.9	4.9	3.14
3	0.0	1.1	0.00
4	5.2	5.4	0.00
5	10.1	9.2	0.00
	5	6	L
0	6.9	6.6	3.14
1	20.6	18.0	0.00
2	0.0	0.3	3.14
3	8.3	10.2	3.14
4	0.0	0.7	0.00
5	0.0	0.8	3.14
	5	7	L
0	2.6	2.3	3.14
1	0.0	0.9	0.00
2	4.6	5.1	0.00
3	4.5	7.3	0.00
4	7.0	6.4	0.00
5	0.0	1.4	0.00
	5	8	L
0	10.3	9.8	0.00
1	3.7	3.7	0.00
2	11.4	11.6	3.14
3	5.5	6.7	0.00
4	2.3	2.8	3.14
5	0.0	1.2	3.14
	5	9	L
0	5.7	5.0	0.00
1	0.0	1.3	3.14
2	5.3	5.1	0.00
3	0.0	2.4	0.00
4	2.7	3.1	3.14
5	1.5	0.1	3.14
	5	10	L
0	3.9	5.3	0.00
1	8.1	8.1	3.14
2	0.0	3.4	3.14
3	4.3	7.7	0.00
4	3.4	5.5	3.14
	5	11	L
0	4.4	4.9	0.00
1	3.7	1.3	3.14
	6	0	L
0	34.9	32.7	3.14
2	10.9	13.4	0.00
4	6.1	4.6	3.14
	6	1	L
0	28.4	27.4	3.14
1	20.6	20.8	3.14
2	11.0	13.4	0.00
3	12.5	15.5	0.00
4	7.9	8.0	0.00
5	8.7	6.6	0.00
	6	2	L
0	27.1	26.1	3.14
1	22.7	18.3	0.00
2	7.3	5.5	3.14
3	0.0	1.4	0.00
4	0.0	3.3	0.00
5	6.2	5.9	0.00
	6	3	L
0	28.5	28.6	3.14
1	15.6	11.9	0.00
2	11.2	13.5	0.00
3	0.0	1.4	0.00
4	8.9	8.7	0.00
	6	4	L
0	9.0	5.9	0.00
1	8.9	7.1	0.00
2	17.5	15.3	3.14
3	6.3	8.6	0.00
4	7.8	7.1	0.00
5	0.0	0.6	3.14
	6	5	L
0	7.5	5.4	0.00
1	28.9	24.9	0.00
2	7.3	3.1	3.14
3	0.0	3.0	3.14
4	0.0	1.6	3.14
5	6.3	5.1	3.14
	6	6	L
0	29.2	25.0	0.00
1	2.6	3.4	3.14
2	0.0	2.2	0.00
3	7.4	10.1	0.00
4	0.0	2.8	3.14
5	10.4	2.0	3.14
	6	7	L
0	3.7	4.1	0.00
1	0.0	1.5	0.00
2	0.0	2.8	3.14
3	0.0	2.2	3.14
4	2.3	2.6	3.14
5	0.0	0.1	0.00
	6	8	L
0	10.7	8.8	0.00
1	0.0	0.6	0.00
2	5.6	6.3	0.00
3	3.3	2.5	3.14
4	3.5	5.9	3.14
5	1.6	2.1	0.00
	6	9	L
0	7.8	7.6	3.14
1	9.6	8.4	3.14
2	2.2	2.0	0.00
3	2.0	3.5	3.14
4	0.0	0.9	3.14
	6	10	L
0	0.0	1.7	3.14
1	2.8	2.5	0.00
2	0.0	2.0	3.14
3	3.6	8.2	3.14
	6	11	L
0	2.2	3.5	3.14
	7	0	L
0	13.5	11.3	3.14
2	5.9	2.6	0.00
4	8.2	8.9	0.00
	7	1	L
0	16.9	15.0	0.00
1	19.7	21.5	0.00
2	12.3	14.2	3.14
3	12.5	9.9	0.00
4	4.5	4.4	0.00
5	4.3	3.1	3.14
	7	2	L
0	5.0	3.9	3.14
1	8.1	7.7	0.00
2	7.4	6.4	3.14
3	8.9	10.6	0.00
4	0.0	2.7	0.00
5	6.1	5.0	3.14
	7	3	L
0	20.0	16.7	0.00
1	16.9	12.6	3.14
2	16.4	17.2	3.14
3	8.2	7.4	0.00
4	2.6	2.1	0.00
	7	4	L
0	9.3	7.6	0.00
1	8.7	7.7	0.00
2	10.4	10.8	3.14
4	2.6	3.3	3.14
5	4.0	0.5	0.00
	7	5	L
0	15.6	12.0	3.14
1	11.2	10.1	3.14
2	4.6	4.7	0.00
3	0.0	0.1	0.00
4	0.0	3.7	3.14
	7	6	L
0	3.7	3.9	0.00
1	4.6	3.4	0.00
2	5.3	5.1	3.14
3	3.6	4.5	3.14
4	0.0	1.5	0.00
5	5.3	5.2	0.00
	7	7	L
0	19.2	18.1	3.14
1	6.9	7.6	0.00
2	6.2	7.3	0.00
3	5.6	6.2	3.14
4	0.0	0.8	0.00
5	1.7	1.3	3.14
	7	8	L
0	0.0	0.8	3.14
1	0.0	1.6	0.00
2	3.3	3.2	3.14
3	0.0	2.0	0.00
4	3.0	4.6	0.00
	7	9	L
0	0.0	0.2	0.00
1	4.8	6.1	0.00
2	3.9	3.9	3.14
	7	10	L
0	5.2	4.9	0.00
	7	11	L
0	3.3	5.5	0.00
	8	0	L
0	16.1	13.9	0.00
2	7.3	9.7	3.14
4	0.0	0.0	0.00
	8	1	L
0	11.6	9.9	3.14
1	16.8	17.0	3.14
2	7.3	7.1	0.00
3	5.3	7.0	0.00
4	0.0	0.3	3.14
5	0.0	2.0	3.14
	8	2	L
0	10.7	9.6	3.14
1	18.9	20.2	3.14
3	0.0	4.5	0.00
4	0.0	2.2	0.00
5	5.6	5.5	0.00
	8	3	L
0	8.0	8.4	3.14
1	0.0	2.7	3.14
2	5.3	4.9	0.00
3	5.9	6.9	3.14
4	0.0	1.7	0.00
	8	4	L
0	24.6	22.4	3.14
1	7.4	6.7	3.14
2	7.0	6.2	0.00
4	2.4	3.3	0.00
	8	5	L
0	9.3	6.2	0.00
1	12.1	11.0	0.00
2	6.4	6.4	3.14
3	2.5	4.1	3.14
4	3.9	4.3	0.00
	8	6	L
0	0.0	2.2	0.00
1	5.2	6.0	0.00
2	3.6	5.1	0.00
3	0.0	0.2	3.14
4	6.5	0.2	0.00
5	1.6	2.2	3.14
	8	7	L
0	8.2	7.2	0.00
1	3.5	5.2	0.00
2	0.0	2.5	3.14
3	3.0	4.7	0.00
4	0.0	2.3	0.00
	8	8	L
0	10.7	11.5	0.00
1	0.0	0.4	3.14
2	0.0	1.9	3.14
3	1.6	1.5	0.00
	8	9	L
0	2.6	3.2	0.00
1	0.0	1.6	3.14
2	2.3	3.2	0.00
	8	10	L
0	2.1	2.2	0.00
	9	0	L
0	24.3	24.3	3.14
2	10.6	13.7	0.00
4	4.2	3.6	0.00
	9	1	L
0	5.1	4.4	3.14
1	5.2	5.2	3.14
2	0.0	0.3	0.00
3	3.7	2.8	3.14
4	3.4	4.6	0.00
5	2.9	3.1	0.00
	9	2	L
0	14.9	13.8	3.14
1	12.9	13.0	0.00
3	5.1	6.6	3.14
4	2.3	2.6	0.00
5	0.0	0.5	0.00
	9	3	L
0	3.7	2.1	0.00
1	0.0	2.9	3.14
2	0.0	1.6	0.00
4	3.2	4.3	0.00
	9	4	L
0	3.7	4.1	0.00
1	2.6	1.7	3.14
2	4.5	6.6	3.14
4	0.0	0.2	3.14
	9	5	L
0	11.9	11.2	0.00
1	2.6	2.3	0.00
2	3.5	3.5	0.00
4	1.9	2.3	3.14
	9	6	L
0	3.6	4.6	0.00
1	0.0	13.6	3.14
3	2.1	3.1	0.00
4	4.1	2.7	3.14
	9	7	L
0	2.4	1.9	0.00
1	2.3	2.5	0.00
2	2.1	2.7	0.00
3	1.7	3.4	3.14
	9	8	L
0	3.6	3.5	3.14
1	3.9	4.9	3.14
2	3.4	5.9	0.00
	9	9	L
0	3.4	4.7	3.14
1	2.9	1.1	0.00
	10	0	L
0	12.0	10.3	0.00
2	7.7	9.1	3.14
4	3.6	4.0	0.00
	10	1	L
0	5.3	3.6	0.00
1	5.3	4.8	0.00
2	2.6	4.4	3.14
	10	2	L
0	2.6	1.9	0.00
1	7.4	8.7	3.14
5	4.8	0.2	0.00
	10	3	L
0	3.7	2.0	0.00
1	7.6	8.8	3.14
2	4.3	5.5	3.14
	10	4	L
0	6.3	6.7	3.14
1	0.0	1.5	3.14
2	4.1	4.7	0.00
4	0.0	3.0	3.14
	10	5	L
0	8.0	7.7	3.14
1	2.4	4.1	3.14
2	0.0	0.2	0.00
	10	6	L
0	2.3	2.8	3.14
1	4.9	4.4	0.00
3	2.6	4.4	3.14
	10	7	L
0	5.1	6.2	3.14
1	3.3	5.2	0.00
	10	8	L
0	2.4	1.8	0.00
	10	9	L
0	1.5	2.4	0.00
	11	0	L
0	8.3	8.0	0.00
4	1.6	1.8	3.14
	11	1	L
0	10.3	10.3	3.14
1	8.6	10.0	3.14
2	2.3	3.3	0.00
	11	2	L
0	3.6	2.9	0.00
1	0.0	0.8	0.00
	11	3	L
0	9.5	9.9	3.14
1	0.0	0.6	3.14
	11	4	L
0	0.0	1.1	0.00
1	3.2	3.6	0.00
	11	5	L
0	4.5	4.4	0.00
1	2.1	4.3	0.00
2	4.4	5.8	3.14
	11	6	L
0	4.0	5.2	0.00
1	0.0	2.5	0.00
	11	7	L
0	3.4	4.1	0.00
	11	8	L
0	1.6	3.2	0.00
	12	0	L
0	5.3	2.5	3.14
	12	1	L
0	0.0	0.8	0.00
	12	2	L
1	3.7	2.9	0.00
	12	3	L
0	4.4	3.4	0.00
1	3.5	4.3	3.14
	12	4	L
0	4.6	5.6	0.00
	12	5	L
0	1.6	2.5	0.00
1	1.6	4.0	3.14
	13	0	L
0	2.6	3.7	0.00
	13	1	L
0	1.9	0.4	0.00
	13	2	L
1	3.6	6.2	3.14
	13	3	L
1	2.6	0.8	0.00
	13	4	L
0	3.4	4.7	3.14

had a double lattice character. The sixth layer also appeared quite normal. The Weissenberg photographs and the $h0l$ precession photograph had the symmetry of an orthorhombic system but with numerous systematic absences. It was apparent that the crystal was twinned and that there were nearly equal amounts of each twin. Many other crystals were photographed and each gave similar results.

It was possible to index the photographs on the basis of an orthorhombic supercell with the following cell parameters:

$$a=133.2,\ b=9.89,\ c=9.22\ \text{Å}.$$

From an analysis of the systematic absences it was possible to choose the real cell. The relationship between the real monoclinic cell and the orthorhombic super cell is shown in Fig. 1, where the new A axis is related to the old a axis by $\mathbf{A}=(\mathbf{a}-5\mathbf{c})/12$. The parameters for the real cell are

$$\begin{aligned} a &= 11.75 \pm 0.02\ \text{Å}, \\ b &= \ \ 9.89 \pm 0.02, \\ c &= \ \ 9.22 \pm 0.02, \\ \beta &= 109^\circ 05' \pm 20'. \end{aligned}$$

The density computed for four molecules in the unit cell is 1.351 g.cm^{-3}. A density determination by flotation in mixed solvents was not possible since the material dissolved both in organic solvents and in aqueous solutions.

The superimposed a^*c^* nets of the two twinned individuals are illustrated in Fig. 2 in the manner used by Herbstein (*e.g.* Herbstein, 1965). The open circles belong to one individual, the closed circles belong to the other individual, and the half-closed circles belong to both. The transformation of indices from the orthorhombic cell to the twinned monoclinic cells is accomplished by $H=(h-5l)/12$, $K=k$, and $L=l$ for one individual and $\mathscr{H}=-(h+5l)/12$, $\mathscr{K}=k$, and $\mathscr{L}=l$ for the other. The $hk0$ reflections of both individuals superimpose, and therefore the intensity for one individual was taken to be one-half the total value read since the twins appeared to be present in equal amounts. The intensities for the reflections from each twin could be readily estimated for layers $l=1$ to 5 since there were no actual overlaps. For the $hk6$ reflections, there was perfect superposition again. However, in this case different reflections from each twin combined to form each spot, *e.g.* the $0k6$ from one individual overlaps the $\bar{5}k6$ from the other and therefore the intensities were not usable. Accordingly, only the intensities for $l=0$ to 5 were used. After the transformation of indices, it was obvious from the remaining systematic absences that the space group was $P2_1/c$.

The intensities were estimated visually by comparison with a calibrated film strip. The multiple-film equi-inclination Weissenberg technique was used for recording the intensities. Corrections were made for Lorentz and polarization factors and spot size. Structure factor amplitudes $|F|$ and normalized structure factor amplitudes $|E|$ were computed for 1149 reflections. The values of the statistical averages $\langle|E|\rangle$ and $\langle|E^2-1|\rangle$, 0.79 and 0.95 respectively, and the distribution of $|E|$ values are consistent with a centrosymmetric crystal.

The phases for D, L-6-thioctic acid were obtained directly from the structure factor magnitudes by means of the symbolic addition procedure (*e.g.* Karle & Karle, 1963, 1966). In the course of determining two hundred phases for reflections with $|E|>1.5$ using the Σ_2 formula, it became apparent that one set of phases was much more probable than any other. An E map computed with these 200 phases, illustrated in the upper portion of Fig. 3, revealed the location of all the atoms in the asymmetric unit. There were no spurious peaks as large as any peaks representing atoms. A final elec-

Table 2. *Fractional coordinates* for* D, L-6-*thioctic acid*

The thermal parameters are of the form $T=\exp[-(\beta_{11}h^2+\beta_{22}k^2+\beta_{33}l^2+2\beta_{12}hk+2\beta_{13}hl+2\beta_{23}kl)]$. Each thermal parameter is multiplied by 10^4.

Atom	x	y	z	β_{11}	β_{22}	β_{33}	β_{12}	β_{13}	β_{23}
S(1)	0·4028	0·0891	0·1172	160	115	236	−15	136	20
S(2)	0·3790	0·1248	0·3204	88	82	195	−12	85	−2
C(1)	0·3964	0·9064	0·1279	280	111	436	17	295	44
C(2)	0·3381	0·8607	0·2373	197	91	236	−5	176	−33
C(3)	0·2754	0·9792	0·2982	90	80	230	−12	112	−20
C(4)	0·2625	0·9450	0·4526	112	92	174	5	108	13
C(5)	0·2016	0·0545	0·5141	117	102	150	−7	120	13
C(6)	0·1767	0·0037	0·6571	104	96	205	10	93	−15
C(7)†	0·1116	0·1126	0·7228	117	111	147	−17	129	9
C(8)	0·0673	0·0603	0·8424	77	107	141	−11	68	−28
O(1)	0·0730	0·9429	0·8881	104	79	249	−2	120	1
O(2)	0·0170	0·1539	0·9071	106	93	233	−12	114	−13
Standard deviations									
S	4	4	6	4	4	11	3	5	5
C	8	8	13	18	14	44	12	22	18
O	14	13	21	10	9	26	7	12	11

* Coordinates are so chosen that they may be substituted directly into the equations representing the least-squares planes.

† Assumed thermal parameters.

tron density map computed with all 1149 F values using the phases from the least-squares refinement is also illustrated in Fig. 3.

The coordinates, as obtained from the E map, were refined by least squares with a modification of the ORFLS program (Busing, Martin & Levy, 1962). The function which was minimized was $\Sigma(F_o-F_c)^2$. The atomic scattering factors used were those listed in *International Tables for X-ray Crystallography*. Since meaningful intensity data were obtained from only one axis for this twinned crystal, the individual layer photographs were made with controlled exposures and development. Accordingly, when the scale factors for each layer were allowed to vary during the isotropic refinement, they did not change much from unity. For the anisotropic refinement, the individual scale factors were kept constant. The refinement was terminated at $R=13.7\%$ for the following reasons. The temperature factor for C(7) persisted in becoming negative and the hydrogen atoms could not be located in a difference map. These two difficulties probably reflect limitations in data obtained from an intimately twinned crystal.

The observed and calculated structure factors are listed in Table 1. Parameters for the heavy atoms are shown in Table 2 and bond distances and angles are shown in Figs. 4 and 5.

Structure description

Like many other organic acids crystallizing in the centrosymmetric space groups, D, L-6-thioctic acid occurs as a dimer formed by hydrogen bonding across a center of symmetry. An inspection of the contents of a unit cell (Fig. 4) shows that the dimers pack in bands along $a=0$. At $a=\frac{1}{2}$, the only attraction between molecules is between the sulfur atoms at an intermolecular distance of 3.88 Å. It is in this region that the twinning probably occurs. If the cell is rotated by 180° at $a=\frac{1}{2}$ about an axis parallel to the c axis and then shifted by $\frac{1}{2}c$ (see Fig. 6) the environment of the sulfur atoms and hence the intermolecular forces are nearly the same as in the original cell. Hence the twinning can occur easily, without disturbing the packing of the dimers, a fact which is consistent with the observation that untwinned crystals could not be found.

The bond lengths and angles in the chain and carboxyl group are similar to those found in other carboxylic acids. The equations for the least-squares planes are, for the C(3)C(4)C(5)C(6)C(7) chain,

$$8.8545\,x+4.0203\,y+2.2303\,z=7.0967 \qquad (1)$$

and, for the C(7)C(8)O(1)O(2) group,

$$8.2829\,x+1.8512\,y+3.8367\,z=5.7557, \qquad (2)$$

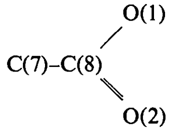
where the right-hand sides of the equations represent the origin-to-plane distance. Deviations of atoms from these planes are shown in Table 3. The dihedral angle between the plane of the chain and the plane of the carboxyl group is approximately 16°.

Table 3. *Distances of atoms from least-squares planes*

C(3)–C(4)–C(5)–C(6)–C(7)

Equation (1)

	Δ
C(3)	−0·056 Å
C(4)	0·036
C(5)	0·074
C(6)	−0·031
C(7)	−0·024



$$\text{C(7)–C(8)}\begin{matrix}\nearrow \text{O(1)}\\ \searrow \text{O(2)}\end{matrix}$$

Equation (2)

	Δ
C(7)	0·001 Å
C(8)	−0·005
O(1)	0·002
O(2)	0·001

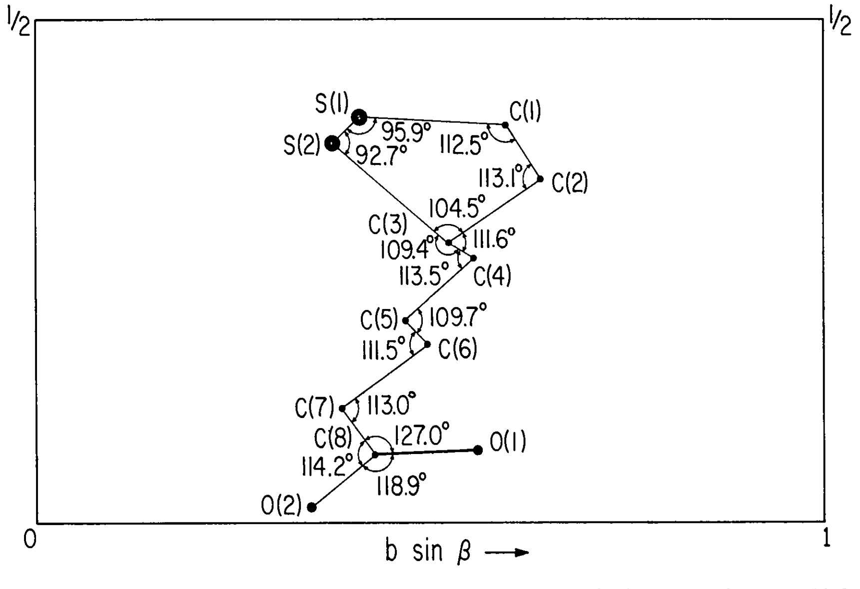


Fig. 5. Bond angles. The standard deviations range from 0·7° for those angles including sulfur to 1·4° for those angles where sulfur is not included.

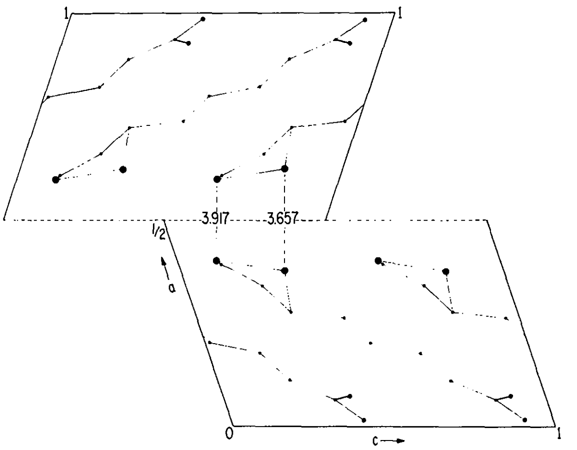
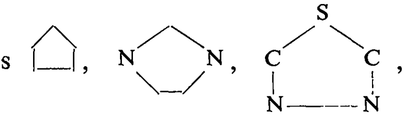
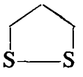


Fig. 6. The probable configuration along the twin boundary.

In many derivatives in which saturated five-membered rings such as , for example, occur, the five-membered rings have four coplanar atoms and one about 0.6 Å out of the plane. In the  ring in D, L-6-thioctic acid, however, no four atoms form a plane. The –CSSC– atoms attempt to arrange themselves in the hydrogen peroxide configuration but are constrained by the ring closure. The dihedral angle in the –CSSC– group is near 96° in tetraethylthiuram disulfide (Karle, Estlin & Britts, 1967) and t-butyl-*N*,*N*-dimethyltrithiopercarbamate (Mitchell, 1966) whereas in D, L-6-thioctic acid the dihedral angle is only 35°.

The S–S bond length, 2.01 Å, and the C–S bond lengths, 1.81 and 1.85 Å, are near those found in tetraethylthiuram disulfide, t-butyl-*N*,*N*-dimethyltrithiopercarbamate and *trans*-2,5-dibromo-1,4-dithiane for example. The large difference between the two C–C bond lengths in the ring, 1.46 and 1.58 Å, is unexplained.

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References

BULLOCK, M. W., BROCKMAN, J. A., PATTERSON, E. L., PIERCE, J. V., VON SALTZA, M.H., SANDERS, F. & STOKSTAD, E. L. R. (1954). *J. Amer. Chem. Soc.* **76**, 1828.

BUSING, W. R., MARTIN, K. O. & LEVY, H. A. (1962). ORFLS. Oak Ridge National Laboratory, Oak Ridge, Tennessee, U.S.A.

HERBSTEIN, F. H. (1965). *Acta Cryst.* **19**, 592.

KALFF, H. T. & ROMERS, C. (1966). *Rec. Trav. chim.Pays-Bas*, **85**, no.2, 198.

KARLE, I. L., ESTLIN, J. A. & BRITTS, K. (1967). *Acta Cryst.* **22**, 273.

KARLE, I. L. & KARLE, J. (1963). *Acta Cryst.* **16**, 969.

KARLE, J. & KARLE, I. L. (1966). *Acta Cryst.* **21**, 849.

MITCHELL, D. (1966). Abstr., ACA, Austin, Texas.

REED, L. J., DE BUSK, B. G., GUNSALUS, I. C. & HORNBERGER, C. S. (1951). *Science*, **114**, 93.

Acta Cryst. (1967). **22**, 573

First-Found Rhombohedral Polytypes of Cadmium Iodide Two Unique Cases of Structure-Transformation during Growth

BY G. K. CHADHA* AND G. C. TRIGUNAYAT

Department of Physics and Astrophysics, University of Delhi, Delhi 7, India

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Although a large number (over 80) of hexagonal polytypes of cadmium iodide have been reported earlier, two rhombohedral polytypes of this substance have been discovered for the first time during an investigation of a batch of crystals grown from the vapour phase. These are 30*R* and 42*R*, respectively. Their complete structures have also been worked out and are found to be $(221212)_3$ and $(22221212)_3$ respectively. A unique characteristic is that each of these polytypes occurs in syntactic coalescence with a hexagonal polytype having the same cell dimensions as the rhombohedral one, showing that the lattice was transformed from hexagonal to rhombohedral, or *vice versa*, during crystal growth. The results are discussed.

Introduction

There seems to be no upper limit to the number of polytypic modifications of cadmium iodide. Experience shows that investigation of a group of cadmium iodide crystals chosen at random is certain to yield one or more new polytypes. A total of over 80 modifications has so far been discovered by different workers (Mitchell, 1956; Trigunayat & Verma, 1962; Srivastava & Verma, 1965; Chadha & Trigunayat, 1966), with

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* C.S.I.R. research fellow.