Title
AN ANTITUMOR AGENT, 2-(3,3-DIMETHYL-1-TRIAZEN0) PHENYL-1-CARBOXAMIDE

Author
Edwards, Steven L.

Publication Date
1976-07-01

Submitted to Acta Crystallographica

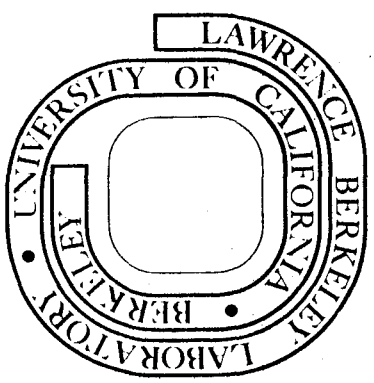
LBL-5408
Preprint c. 1

AN ANTITUMOR AGENT, 2-(3,3-DIMETHYL-1-TRIAZENO)PHENYL-1-CARBOXAMIDE

Steven L. Edwards, Gervais Chapuis, David H. Templeton, and Allan Zalkin

July 1976

Prepared for the U. S. Energy Research and Development Administration under Contract W-7405-ENG-48



DISCLAIMER

LBL-5408

AN ANTITUMOR AGENT, 2-(3,3-DIMETHYL-1-TRIAZENO)PHENYL-1-CARBOXAMIDE*

By Steven L. Edwards, Gervais Chapuis, David H. Templeton

and Allan Zalkin

Materials and Molecular Research Division
Lawrence Berkeley Laboratory

and

Department of Chemistry
University of California
Berkeley, California 94720, U.S.A.

(Received)

Abstract. $C_9H_{12}ON_4$, monoclinic, $P2_1/c$, a = 9.444 (5), b = 7.085 (4), c = 16.005 (7) Å, β = 108.08 (4)°, Z = 4, D_x = 1.257 (1), D_m = 1.25 (1) g cm^{-3}, at 23°C. Its structure is similar to that of $C_6H_{11}ON_6$, a related antitumor agent reported earlier, both having an internal hydrogen bond.

*Work done in part with support from the U.S. Energy Research and Development Administration.

Introduction. The sample was provided by Dr. Corwin Hansch of Pomona College. Two distinct crystalline forms grew from the pure sample dissolved in toluene. The predominant form, which was orthorhombic, appeared to include a molecule of toluene in the asymmetric unit according to its density, cell volume and mass spectroscopic information. Investigation of the other (monoclinic) form suggested that it did not include the solvent molecule, and it was used for the structure determination.

Photographic x-ray diffraction data revealed systematic absences, h0l, l≠2n and 0k0, k≠2n, indicating the space group as $P2_1/c$. A single crystal, .07 x .08 x .21 mm in size, was mounted on a full circle Picker Nuclear FACS-I automated diffractometer. Data were collected in the hemisphere with k non-negative and $\sin\theta/\lambda < 0.54$, using MoK$\alpha$ (λ = 0.70926 Å for α_1) and the θ-2θ scan technique. The data were averaged to give 1339 independent reflections of which 108 were recorded as zero. The absorption parameter, μ, is 0.53 cm^{-1} which was considered small enough to be ignored. The intensity decay of three regularly monitored reflections was less than 2% during the experiment, and no correction was made for this effect.

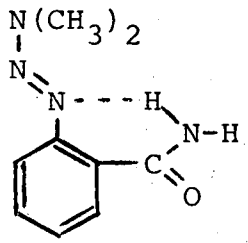
The crystal structure was solved both by interpretation of the Patterson function and using the MULTAN program (Germain, Main & Woolfson, 1971), with essentially the same result.

After preliminary refinement of the heavy atoms, eleven of the hydrogen atoms were found on a difference Fourier map. The position of the remaining hydrogen atom was calculated. Tentative refinement of the hydrogen parameters was unsatisfactory. The two amide hydrogen

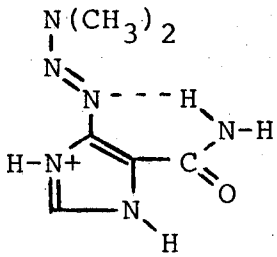
atoms were then constrained to the positions found in the Fourier maps, while the others were held at positions calculated for C-H = 0.97 Å. The methyl hydrogens were constrained to tetrahedral geometry, rotated for greatest overlap with the electron density map. An overall isotropic temperature parameter for all hydrogen atoms was refined to a value of $B = 9.4\ (7)\ Å^2$. For the non-hydrogen atoms, the positional and anisotropic thermal parameters were refined. When refinement was concluded the shift of no parameter was more than 3% of the corresponding standard deviation. Weights were derived from $\sigma^2(F^2) = s^2 + (pF^2)^2$, where s^2 is the variance according to counting statistics and $p = 0.05$ was chosen to equalize $\langle w(\Delta F)^2 \rangle$ for strong and weak reflections. The final residual $R_w = (\Sigma w(\Delta F)^2/\Sigma w F_o^2)^{1/2}$ was 0.053 using 563 reflections with $F^2 > 2\sigma$. The unweighted residual $R = \Sigma|\Delta F|/\Sigma|F_o|$ was 0.059 for the same data. For all the data R was 0.166. The refinement was calculated by our full-matrix least-squares program using the atomic scattering factors of Doyle and Turner (1968) with anomalous scattering correction of Cromer and Liberman (1970) for neutral carbon, nitrogen and oxygen and those of Stewart, Davidson and Simpson (1965) for spherical hydrogen. Final parameters are given in Table 1 and 2.*

*A list of structure factors has been deposited with the British Library Lending Division.

Discussion. This study was done to determine the structural similarity between 2-(3,3-dimethyl-1-triazeno)phenyl-1-carboxamide (DTPC) and 5-(3,3-dimethyl-1-triazeno)imidazole-4-carboxamide (DTIC, Edwards, Sherfinski and Marsh, 1974). Both of these compounds belong to a class of related antitumor agents currently being studied for potency and effectiveness.



DTPC



DTIC cation

The DTPC molecule (Fig. 1) is approximately planar due in part to bond conjugation through the phenyl ring and triazene group. The carboxamide group is held coplanar by the internal hydrogen bond, N-H...N, with N-N = 2.71 (1) Å.

There is great similarity between DTPC and DTIC which can be seen from a comparison of bond lengths given in Table 3. An important similarity between the two structures is the internal hydrogen bond. In DTIC, the N-N distance of this hydrogen bond is 2.97 Å, while in DTPC the more favorable bond angles in the phenyl ring allow N-N = 2.71 Å. The existence of this internal hydrogen bond lends credence to the mechanism of deactivation of bis(chloroethyl)triazenoimidazolecarboxamide proposed earlier by Edwards, Sherfinski and Marsh (1974). The internal hydrogen bond may be important in explaining the success of similar antitumor agents.

The second amide hydrogen atom, H(12), provides a hydrogen bond to the oxygen atom of a neighboring molecule, with distance N(14)- O(13) = 2.91 (1) Å.

We thank Dr. Corwin Hansch for providing this sample.

REFERENCES

Cromer, D. T. & Liberman, D. (1970). J. Chem. Phys. 53, 1891 - 1898.

Doyle, P. A. & Turner, P. S. (1968). Acta Cryst. A24, 390 - 397.

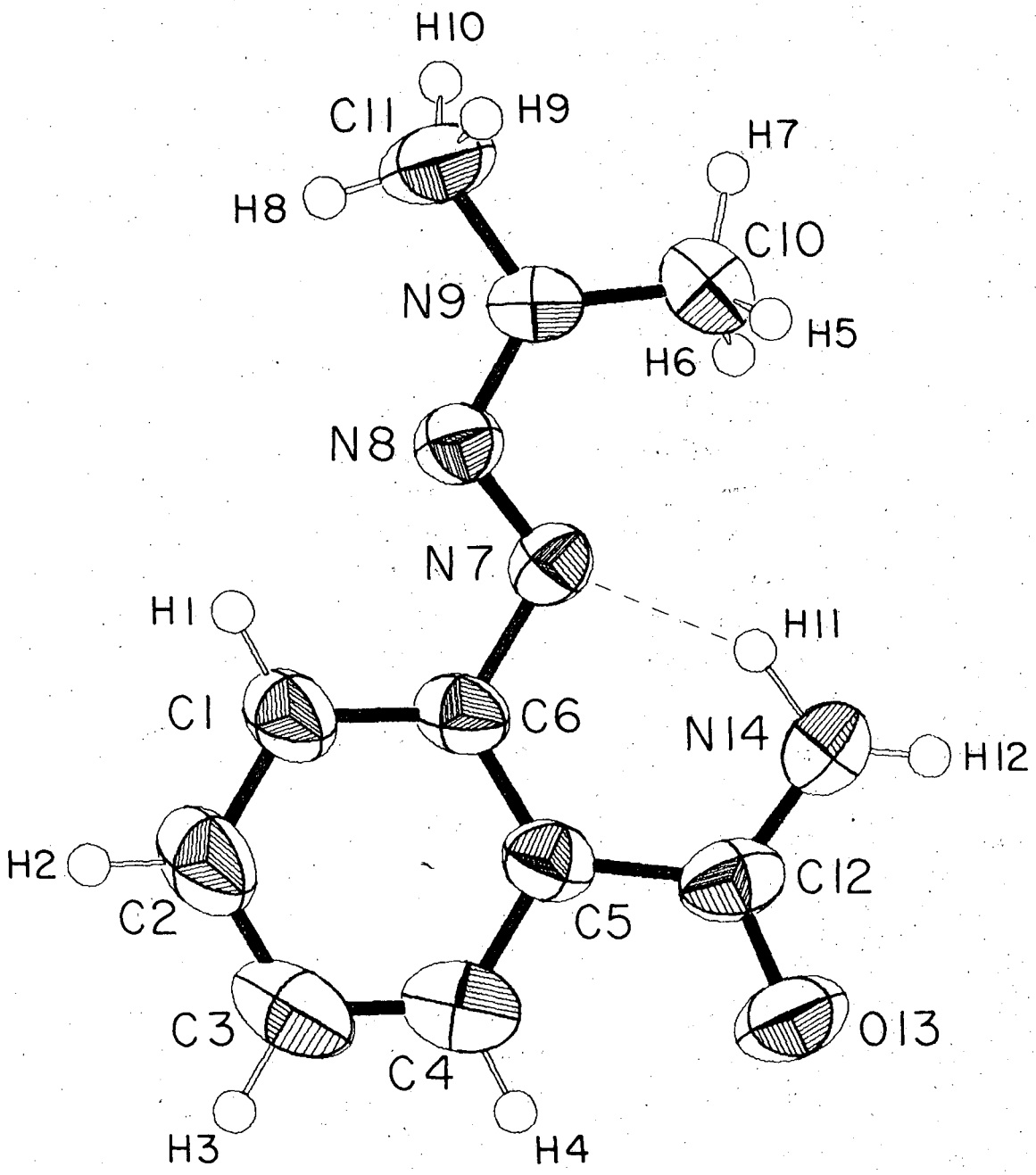
Edwards, S. L., Sherfinski, J. S. & Marsh, R. E. (1974). J. Amer. Chem. Soc. 96, 2593 - 2597.

Germain, G., Main, P. & Woolfson, M. M. (1971). Acta Cryst. A27, 368 - 376.

Stewart, R. F., Davidson, E. R. & Simpson, W. T. (1965). J. Chem. Phys. 42, 3175 - 3187.

FIGURES

Figure 1. Molecular structure and numbering scheme for the DTPC molecule.



XBL 757-1809

Table 1. Positional parameters.

	x	y	z
C(1)	.2514(8)	.350(1)	.2876(5)
C(2)	.1001(9)	.349(1)	.2478(5)
C(3)	.0414(8)	.376(1)	.1569(6)
C(4)	.1356(9)	.397(1)	.1081(5)
C(5)	.2897(7)	.400(1)	.1467(4)
C(6)	.3475(7)	.380(1)	.2376(4)
N(7)	.5054(5)	.3871(8)	.2778(3)
N(8)	.5403(6)	.4115(9)	.3611(4)
N(9)	.6843(6)	.4146(8)	.4003(3)
C(10)	.7939(7)	.392(1)	.3550(5)
C(11)	.7303(7)	.450(1)	.4946(4)
C(12)	.3771(8)	.432(1)	.0839(5)
O(13)	.3100(5)	.4624(8)	.0052(3)
N(14)	.5255(7)	.4269(8)	.1135(3)
H(1)	.295	.334	.351
H(2)	.031	.333	.281
H(3)	-.066	.379	.129
H(4)	.090	.407	.045
H(5)	.803	.510	.327
H(6)	.761	.293	.311
H(7)	.888	.358	.397
H(8)	.643	.458	.514
H(9)	.785	.567	.507
H(10)	.793	.347	.525
H(11)	.556	.424	.179
H(12)	.578	.418	.078

Table 2. Anisotropic thermal parameters ($Å^2$)

The temperature factor expression is $\exp[-(h^2a^{*2}B_{11}+2hka^*b^*B_{12}+\ldots)/4]$.

	B_{11}	B_{22}	B_{33}	B_{12}	B_{13}	B_{23}
C(1)	3.8(4)	7.3(5)	5.4(4)	.2(4)	1.5(4)	-1.9(4)
C(2)	4.5(4)	9.2(6)	7.3(5)	.4(4)	2.5(4)	-3.2(5)
C(3)	3.8(4)	6.9(5)	8.2(5)	.4(4)	.5(4)	-2.6(5)
C(4)	5.0(4)	3.5(4)	6.1(4)	.6(4)	.3(3)	-1.0(4)
C(5)	3.8(4)	4.4(4)	4.5(3)	-.4(3)	.7(3)	.1(4)
C(6)	3.6(3)	4.5(4)	4.4(4)	1.0(3)	.7(3)	-1.0(4)
N(7)	4.4(3)	3.9(3)	3.5(3)	.2(3)	1.2(2)	.4(3)
N(8)	4.1(3)	4.7(3)	3.9(3)	-.3(3)	.8(2)	.6(3)
N(9)	4.0(3)	5.7(4)	3.8(3)	.1(3)	.3(2)	.2(3)
C(10)	4.0(3)	5.7(5)	6.0(4)	.1(3)	1.6(3)	-.0(4)
C(11)	6.3(4)	7.8(6)	3.8(3)	.1(4)	-.1(3)	.9(4)
C(12)	5.6(4)	4.2(4)	4.0(4)	-.6(4)	.3(3)	.5(4)
O(13)	6.9(3)	9.4(4)	3.9(2)	-2.0(3)	.0(2)	1.7(3)
N(14)	5.5(3)	7.7(4)	3.9(3)	-.2(3)	1.7(2)	-.4(3)

Table 3. Bond lengths of DTPC and corresponding values for DTIC (Å).

	DTPC	DTIC
C(1) -C(2)	1.37 (1)	
C(2) -C(3)	1.40 (1)	
C(3) -C(4)	1.36 (1)	
C(4) -C(5)	1.39 (1)	
C(5) -C(6)	1.39 (1)	
C(6) -C(1)	1.40 (1)	
C(6) -N(7)	1.429 (8)	1.378 (3)
N(7) -N(8)	1.281 (7)	1.288 (3)
N(8) -N(9)	1.309 (7)	1.305 (3)
N(9) -C(10)	1.45 (1)	1.446 (3)
N(9) -C(11)	1.46 (1)	1.449 (3)
C(5) -C(12)	1.50 (1)	1.463 (3)
C(12)-O(13)	1.24 (1)	1.226 (3)
C(12)-N(14)	1.33 (1)	1.328 (3)
N(14)-H(11)	1.00	
N(14)-H(12)	0.87	

Table 4. Bond angles (°).

C(6)—C(1)—C(2)	120.1 (7)
C(1)—C(2)—C(3)	120.1 (7)
C(2)—C(3)—C(4)	119.5 (7)
C(3)—C(4)—C(5)	121.7 (7)
C(4)—C(5)—C(6)	118.6 (6)
C(5)—C(6)—C(1)	119.9 (6)
C(5)—C(6)—N(7)	118.9 (6)
C(1)—C(6)—N(7)	121.3 (6)
C(6)—N(7)—N(8)	111.7 (5)
N(7)—N(8)—N(9)	113.3 (5)
N(8)—N(9)—C(10)	123.8 (5)
N(8)—N(9)—C(11)	115.6 (5)
C(10)-N(9)—C(11)	120.6 (5)
C(4)—C(5)—C(12)	114.9 (6)
C(6)—C(5)—C(12)	126.5 (6)
C(5)—C(12)-O(13)	119.4 (6)
C(5)—C(12)-N(14)	119.6 (6)
O(13)-C(12)-N(14)	121.0 (6)

OBSERVED STRJCTURE FACTORS, STANDARD DEVIATIONS, AND DIFFERENCES (X 6.7) FOR C(9)H(12)ON(4) F(0,0,0) = 2735

FOB AND FCA ARE THE OBSERVED AND CALCULATED STRUCTURE FACTORS.
SG = ESTIMATED STANDARD DEVIATION OF FOB. DEL = /FOB/ - /FCA/.
* INDICATES ZERO WEIGHTED DATA.

H	FOB	SG	DEL	H	FOB	SG	DEL	H	FOB	SG	DEL	H	FOB	SG	DEL	H	FOB	SG	DEL
K,L=	0,	0		-8	105	10	2	-9	13	25	7*	-2	225	7	-5	-10	26	37	28*
1	447	12	-26	-7	71	5	-7	-8	29	27	8*	-1	158	4	-6	-9	11	21	6*
2	187	5	-7	-6	33	5	1	-7	42	7	2	0	158	5	6	-8	129	5	-7
3	65	4	1	-5	0	26	-7*	-6	22	32	16*	1	182	5	-1	-7	51	4	4
4	201	5	-3	-4	267	7	1	-5	58	5	-9	2	70	18	-11	-6	18	15	15*
5	314	8	15	-3	173	5	5	-4	0	24	-27*	3	75	3	-0	-5	13	24	10*
6	83	4	-5	-2	279	7	-0	-3	103	3	0	4	319	8	9	-4	79	3	2
7	31	18	-3*	-1	138	5	-3	-2	64	6	-5	5	44	13	13*	-3	233	6	7
8	26	15	15*	0	0	28	-8*	-1	30	7	5	6	9	31	-5*	-2	56	12	-5
9	17	21	17*	1	288	7	11	0	62	8	-4	7	84	4	-1	-1	266	7	-1
K,L=	8,	2		2	119	4	5	1	28	29	15*	8	20	40	-9*	0	142	7	-5
-9	0	21	-26*	3	13	22	11*	2	90	8	1	9	28	33	-13*	1	51	13	4
-8	37	6	0	4	155	4	-5	3	131	5	-3	K,L=	1,	2		2	52	12	-4
-7	166	5	-8	5	45	8	3	4	79	4	0	-9	19	26	1*	3	98	3	6
-6	13	17	12*	6	66	4	2	K,L=	8,	14		-8	14	32	9*	4	25	9	-2*
-5	34	5	8	7	52	6	-3	-8	20	26	14*	-7	0	28	-5*	5	15	21	-1*
-4	0	23	-23*	K,L=	0,	8		-7	0	23	-20*	-6	25	25	21*	6	0	33	-23*
-3	271	7	-4	-10	18	32	17*	-6	24	26	8*	-5	21	28	1*	7	16	35	12*
-2	340	9	-2	-9	46	7	4	-5	14	34	8*	-4	42	6	8	8	18	24	-12*
-1	529	13	-30	-8	17	25	11*	-4	127	4	-2	-3	60	11	2	K,L=	1,	5	
0	167	7	-13	-7	28	8	13	-3	7	28	-1*	-2	171	5	1	-10	0	26	-6*
1	609	15	-38	-6	56	5	10	-2	100	4	3	-1	340	9	-3	-9	61	8	3
2	257	7	8	-5	36	18	4*	-1	22	26	-10*	0	27	4	0	-8	191	6	-8
3	146	5	2	-4	21	26	8*	0	78	4	4	1	93	5	-4	-7	31	6	3
4	163	5	10	-3	115	4	4	1	102	5	-8	2	91	8	-3	-6	57	4	2
5	50	4	5	-2	29	17	5*	2	23	31	9*	3	72	6	2	-5	38	11	-0*
6	30	8	10	-1	95	5	8	3	23	20	14*	4	182	5	11	-4	232	6	6
7	33	20	20*	0	445	11	14	K,L=	0,	16		5	36	10	4	-3	372	9	4
8	49	6	-6	1	134	4	-2	-6	39	12	4*	6	17	22	14*	-2	185	5	-3
9	26	28	26*	2	99	4	3	-5	41	6	3	7	46	10	0	-1	221	6	-8
K,L=	0,	4		3	72	5	-1	-4	56	10	3	8	22	14	-0*	0	161	5	3
-10	21	35	13*	4	65	4	-0	-3	9	25	-3*	9	31	10	-5*	1	78	5	-3
-9	19	20	14*	5	230	6	-6	-2	28	10	-5*	K,L=	1,	3		2	45	5	-3
-8	92	4	1	6	19	28	-26*	-1	29	38	2*	-10	0	29	-7*	3	125	4	-2
-7	126	4	-2	7	13	35	11*	0	29	19	14*	-9	50	5	10	4	18	17	-10*
-6	66	9	2	K,L=	0,	10		K,L=	1,	0		-8	53	6	5	5	7	20	-8*
-5	169	5	-2	-9	34	9	-1	1	224	6	1	-7	84	3	-3	6	19	26	-11*
-4	46	5	-3	-8	25	25	12*	2	90	9	-0	-6	102	3	2	7	23	31	18*
-3	159	5	2	-7	43	7	-22	3	68	5	3	-5	75	3	1	8	32	14	20*
-2	321	9	1	-6	24	9	14*	4	225	6	7	-4	17	10	15*	K,L=	1,	6	
-1	154	5	-1	-5	89	4	-4	5	65	6	6	-3	62	6	5	-10	0	27	-0*
0	124	8	-9	-4	33	5	-5	6	21	22	-9*	-2	31	47	-12*	-9	30	13	-14*
1	53	14	2	-3	61	5	8	7	60	5	-6	-1	327	8	5	-8	100	6	-5
2	136	4	8	-2	106	4	1	8	43	13	8*	0	63	6	8	-7	65	4	-5
3	184	5	-0	-1	56	4	-0	9	30	13	6*	1	40	41	9*	-6	32	7	0
4	78	4	7	0	85	4	-4	K,L=	1,	1		2	124	6	1	-5	35	4	2
5	27	8	19*	1	100	3	1	-9	15	34	11*	3	78	5	3	-4	190	5	9
6	22	30	6*	2	67	6	-11	-8	61	9	-3	4	47	9	7	-3	133	4	8
7	19	33	-2*	3	35	15	-12*	-7	64	5	-5	5	47	6	3	-2	20	30	1*
8	39	10	4	4	0	30	-2*	-6	81	4	1	6	20	26	18*	-1	73	3	1
K,L=	0,	6		5	46	8	12	-5	118	3	-2	7	14	24	0*	0	109	3	3
-10	38	13	15*	6	50	6	5	-4	101	4	2	8	63	10	-15	1	60	3	3
-9	19	32	-2*	K,L=	0,	12		-3	117	6	1	K,L=	1,	4		2	53	4	4

STRUCTURE FACTORS CONTINUED FOR
C(9)H(12)ON(4)

H	FOB	SG	DEL	H	FOB	SG	DEL	H	FOB	SG	DEL	H	FOB	SG	DEL	H	FOB	SG	DEL
3	0	18	-2*	5	35	36	-2*	-1	27	28	8*	-3	49	6	-2	-4	58	3	4
4	20	21	15*	6	42	7	5	0	1	21	1*	-2	134	6	-2	-3	140	4	-2
5	66	5	1	K,L=	1,	10		1	28	38	17*	-1	608	15	-6	-2	202	5	-6
6	36	12	34*	-9	0	25	-0*	2	36	8	-5	8	1004	25	-70	-1	68	3	2
7	40	24	14*	-8	12	26	-6*	3	41	8	-3	1	244	7	2	0	71	3	-3
K,L=	1,	7		-7	2	25	-24*	K,L=	1,	14		2	17	28	3*	1	0	26	-4*
-10	0	28	-10*	-6	31	21	15*	-8	21	23	-4*	3	50	3	1	2	94	4	3
-9	30	30	-17*	-5	26	8	11*	-7	0	30	-1*	4	190	5	-5	3	79	3	-0
-8	0	36	0*	-4	39	5	4	-6	0	29	-7*	5	162	4	1	4	9	29	-7*
-7	111	4	-5	-3	60	5	5	-5	14	26	2*	6	11	21	-7*	5	32	8	8
-6	11	21	8*	-2	53	7	4	-4	47	25	-3*	7	24	27	19*	6	25	11	7*
-5	65	4	-2	-1	60	4	7	-3	66	5	1	8	22	31	-10*	7	14	37	1*
-4	141	4	6	0	42	6	-8	-2	37	11	16*	9	23	13	21*	8	28	9	21*
-3	43	4	-2	1	7	25	-3*	-1	0	21	-1*	K,L=	2,	2		K,L=	2,	5	
-2	99	3	4	2	0	36	-2*	0	0	22	-3*	-9	28	30	26*	-9	0	26	-9*
-1	14	20	6*	3	29	9	18	1	30	17	13*	-8	14	30	12*	-8	90	11	-4
0	50	7	5	4	89	6	-4	2	13	24	-15*	-7	24	33	-3*	-7	92	4	-0
1	8	29	-8*	5	14	21	12*	K,L=	1,	15		-6	33	6	12	-6	0	19	-5*
2	49	7	-9	K,L=	1,	11		-7	25	33	15*	-5	93	3	0	-5	92	4	5
3	90	3	-2	-9	23	26	15*	-6	18	22	5*	-4	44	8	-14	-4	144	4	1
4	37	9	0	-8	34	11	1*	-5	19	26	9*	-3	117	4	-2	-3	37	11	4*
5	140	4	-2	-7	46	6	-8	-4	24	12	5*	-2	230	6	-5	-2	33	9	-10
6	65	8	3	-6	34	8	-8	-3	23	37	-7*	-1	179	6	6	-1	80	3	0
7	27	12	-4*	-5	28	23	28*	-2	47	12	-9	0	310	8	-2	0	80	3	-1
K,L=	1,	8		-4	10	24	-2*	-1	15	29	1*	1	213	6	-3	1	137	4	5
-9	20	23	6*	-3	131	4	-3	0	13	36	8*	2	101	4	-2	2	29	5	-1
-8	27	32	14*	-2	132	4	2	1	37	12	19*	3	134	4	-1	3	46	4	-0
-7	32	16	-3*	-1	94	5	-8	K,L=	1,	16		4	38	16	19*	4	85	4	3
-6	17	29	9*	0	66	3	0	-6	29	34	24*	5	39	5	-2	5	0	20	-8*
-5	17	26	7*	1	44	9	3	-5	9	33	-7*	6	19	30	18*	6	17	37	1*
-4	22	27	4*	2	9	25	-5*	-4	32	12	15*	7	23	27	9*	7	9	23	-9*
-3	34	14	9*	3	39	23	2*	-3	24	24	14*	8	3	27	-3*	8	28	30	-5*
-2	43	7	1	4	36	28	1*	-2	19	21	16*	K,L=	2,	3		K,L=	2,	6	
-1	17	31	12*	5	0	34	-20*	-1	20	21	20*	-9	14	21	4*	-9	16	21	1*
0	55	7	4	K,L=	1,	12		0	0	29	-21*	-8	59	9	4	-8	34	16	4*
1	7	26	-25*	-9	19	28	16*	K,L=	1,	17		-7	141	4	4	-7	32	14	25*
2	31	12	16*	-8	26	22	-19*	-4	16	42	-10*	-6	36	12	-8*	-6	26	9	2*
3	55	7	1	-7	38	17	-5*	-3	23	28	10*	-5	45	9	-4	-5	30	22	-8*
4	104	4	-4	-6	27	9	19*	-2	26	23	16*	-4	65	4	-5	-4	62	3	4
5	91	4	2	-5	0	24	-18*	K,L=	2,	0		-3	84	4	-6	-3	16	23	-8*
6	21	24	19*	-4	32	7	3	0	557	14	-5	-2	32	16	-9*	-2	99	4	2
7	26	31	25*	-3	163	5	-8	1	296	8	-9	-1	43	12	-8	-1	5	22	-16*
K,L=	1,	9		-2	77	9	-2	2	80	6	0	0	147	5	-5	0	64	4	-13
-9	0	28	-5*	-1	49	6	11	3	64	5	0	1	130	5	-1	1	30	17	5*
-8	21	28	12*	0	30	32	10*	4	38	13	-8*	2	81	3	-3	2	84	4	-2
-7	21	24	21*	1	20	30	7*	5	92	3	2	3	68	4	7	3	44	4	9
-5	0	20	-3*	2	33	41	4*	6	60	5	2	4	11	19	-6*	4	49	5	3
-5	48	7	13	3	37	7	5	7	22	30	6*	5	31	21	22*	5	54	5	5
-4	61	3	2	4	16	22	-4*	8	17	24	3*	6	10	27	-2*	6	29	11	11*
-3	63	6	1	K,L=	1,	13		9	17	25	5*	7	18	27	16*	7	22	34	-12*
-2	30	6	14	-8	52	6	2	K,L=	2,	1		8	21	22	-16*	K,L=	2,	7	
-1	29	12	5*	-7	17	28	-6*	-9	10	31	-3*	K,L=	2,	4		-9	0	34	-27*
0	10	17	6*	-6	21	29	16*	-8	23	13	13*	-9	30	9	13*	-8	46	17	2*
1	40	8	10	-5	0	33	-0*	-7	95	4	3	-8	19	30	8*	-7	0	23	-6*
2	18	26	2*	-4	112	4	-1	-6	69	5	-7	-7	15	22	-4*	-6	27	14	-6*
3	22	18	14*	-3	185	6	-4	-5	112	3	-0	-6	10	27	4*	-5	35	5	11
4	155	4	-9	-2	40	12	-13*	-4	17	22	13*	-5	65	3	7	-4	88	3	1

STRUCTURE FACTORS CONTINUED FOR
C(9)H(12)ON(4)

H	FOB	SG	DEL	H	FOB	SG	DEL	H	FOB	SG	DEL	H	FOB	SG	DEL	H	FOB	SG	DEL
-3	159	4	3	2	29	11	-13*	2	0	27	-3*	3	85	3	3	4	0	20	-14*
-2	64	9	2	3	46	10	-5	K,L=	2,	15		4	128	4	5	5	56	11	12
-1	40	12	-13*	4	35	7	17	-6	25	13	15*	5	11	29	-11*	6	6	21	2*
0	260	7	9	5	44	30	-1*	-5	36	21	10*	6	32	32	20*	7	22	25	16*
1	232	6	7	K,L=	2,	11		-4	82	4	6	7	29	10	-4*	K,L=	3,	6	
2	19	24	18*	-9	16	24	6*	-3	0	36	-13*	8	39	15	7*	-9	83	12	6
3	61	4	2	-8	0	38	-9*	-2	22	14	6*	K,L=	3,	3		-8	72	4	0
4	63	5	2	-7	48	12	-1	-1	25	31	8*	-9	25	25	24*	-7	60	25	-14*
5	144	4	-4	-6	26	16	13*	0	51	5	4	-8	60	5	3	-6	42	9	10
6	76	6	4	-5	17	24	13*	1	14	32	-17*	-7	23	13	20*	-5	45	10	5
7	36	8	8	-4	20	23	16*	K,L=	2,	16		-6	35	9	-13	-4	173	5	-1
K,L=	2,	8		-3	27	18	-2*	-5	13	25	-3*	-5	31	8	1	-3	120	3	2
-9	0	30	-10*	-2	29	8	5	-4	23	24	11*	-4	19	24	15*	-2	41	7	5
-8	13	29	4*	-1	65	3	3	-3	21	28	19*	-3	105	3	1	-1	33	10	-2*
-7	45	5	2	0	14	26	7*	-2	36	10	8	-2	20	25	14*	0	47	4	3
-6	11	28	2*	1	36	14	27*	-1	23	27	4*	-1	176	5	1	1	15	22	-7*
-5	4	20	-7*	2	38	8	25	K,L=	3,	0		0	21	9	18*	2	58	7	-7
-4	61	3	-1	3	68	11	4	1	80	5	-3	1	52	4	0	3	17	20	11*
-3	16	26	12*	4	51	6	-7	2	31	9	-13	2	21	22	-14*	4	40	7	4
-2	48	8	2	5	43	7	-0	3	34	9	-3	3	76	4	3	5	61	8	6
-1	89	4	3	K,L=	2,	12		4	155	5	-1	4	82	3	1	6	30	33	-5*
0	157	5	-1	-8	0	25	-20*	5	122	6	0	5	23	15	10*	7	31	14	1*
1	41	6	-7	-7	16	22	10*	6	63	7	4	6	9	21	0*	K,L=	3,	7	
2	47	18	-10	-6	18	27	14*	7	67	6	2	7	36	9	3	-9	21	27	2*
3	35	6	-4	-5	32	19	6*	8	53	5	-4	8	0	25	-1*	-8	74	4	3
4	62	11	-5	-4	18	28	-11*	K,L=	3,	1		K,L=	3,	4		-7	29	24	19*
5	66	5	-7	-3	38	8	5	-9	0	38	-12*	-9	29	34	27*	-6	16	21	7*
6	41	6	17	-2	87	4	3	-8	15	21	12*	-8	133	4	2	-5	28	13	10*
K,L=	2,	9		-1	25	30	18*	-7	25	26	21*	-7	102	5	-3	-4	44	13	6*
-9	37	9	-0	0	46	16	-0*	-6	18	22	-9*	-6	9	22	-19*	-3	51	9	2
-8	29	12	15*	1	41	8	14	-5	31	8	4	-5	19	28	1*	-2	16	19	-4*
-7	41	7	16	2	47	9	-5	-4	72	4	-1	-4	55	4	4	-1	40	7	3
-6	48	8	10	3	68	6	5	-3	7	26	-11*	-3	177	5	3	0	45	5	-9
-5	73	4	1	4	32	36	9*	-2	75	4	1	-2	219	6	-4	1	31	20	-6*
-4	29	16	4*	K,L=	2,	13		-1	225	6	10	-1	180	5	2	2	28	11	8*
-3	56	4	-1	-8	27	15	11*	0	59	3	-1	0	66	3	-4	3	25	25	23*
-2	68	7	-4	-7	37	8	10	1	54	3	-1	1	22	26	6*	4	36	38	-14*
-1	57	8	-5	-6	16	27	4*	2	8	22	4*	2	36	5	9	5	36	7	2
0	119	4	6	-5	23	36	-11*	3	36	10	11	3	94	5	-1	6	20	39	-15*
1	60	4	2	-4	98	6	4	4	87	4	-2	4	46	10	15	K,L=	3,	8	
2	40	13	25*	-3	54	8	4	5	18	20	8*	5	30	9	-0	-9	0	33	-24*
3	36	8	16	-2	38	12	2*	6	23	27	19*	6	18	29	11*	-8	35	9	-2
4	55	8	-14	-1	42	20	8*	7	21	30	-6*	7	28	35	-6*	-7	38	6	-4
5	81	7	-4	0	10	35	6*	8	33	11	26*	K,L=	3,	5		-6	10	33	1*
6	37	7	13	1	83	6	-1	K,L=	3,	2		-9	0	27	-3*	-5	14	25	11*
K,L=	2,	10		2	63	8	-4	-9	28	39	-4*	-8	59	6	-2	-4	26	18	24*
-9	20	29	19*	3	42	9	-6	-8	43	10	-4	-7	48	8	7	-3	86	4	-2
-8	0	31	-3*	K,L=	2,	14		-7	22	31	10*	-6	0	20	-9*	-2	40	8	-8
-7	5	29	-3*	-7	15	23	9*	-6	15	21	-5*	-5	34	6	4	-1	10	19	8*
-6	33	14	25*	-6	0	27	-13*	-5	6	28	1*	-4	59	3	-3	0	18	16	1*
-5	31	8	-8	-5	30	32	6*	-4	11	19	3*	-3	41	8	10	1	27	8	14*
-4	32	7	27	-4	28	17	4*	-3	19	21	13*	-2	60	10	-6	2	26	18	22*
-3	42	8	11	-3	23	31	-0*	-2	44	5	4	-1	55	3	-2	3	46	12	2
-2	25	9	-2*	-2	24	37	-18*	-1	127	4	-5	0	65	3	3	4	106	6	-4
-1	9	19	-1*	-1	17	24	-9*	0	85	5	-3	1	25	26	-1*	5	97	7	-2
0	90	6	-6	0	37	8	1	1	24	5	8	2	12	19	-3*	6	0	23	-19*
1	47	17	-6*	1	29	36	-10*	2	59	9	-2	3	14	26	1*	K,L=	3,	9	

STRUCTURE FACTORS CONTINUED FOR
C(9)H(12)ON(4)

H	FOB	SG	DEL	H	FOB	SG	DEL	H	FOB	SG	DEL	H	FOB	SG	DEL	H	FOB	SG	DEL
-8	35	10	14	-7	19	25	8*	-6	44	15	8*	0	70	3	3	-5	22	24	2*
-7	33	33	32*	-6	0	26	-4*	-5	68	4	-1	1	12	19	1*	-4	0	29	-23*
-6	37	11	24	-5	0	22	-3*	-4	47	6	9	2	30	15	14*	-3	21	31	8*
-5	20	28	18*	-4	0	31	-5*	-3	13	21	-2*	3	6	34	-3*	-2	25	26	10*
-4	26	21	4*	-3	46	9	-9	-2	22	13	2*	4	30	19	5*	-1	74	4	-1
-3	46	8	1	-2	25	14	13*	-1	199	5	-1	5	62	12	-2	0	21	29	7*
-2	19	25	1*	-1	0	21	-1*	0	206	6	-7	6	15	34	5*	1	62	5	3
-1	48	24	8*	0	18	21	11*	1	65	3	0	K,L=	4,	6		2	20	39	3*
0	53	7	-6	1	24	30	7*	2	19	21	5*	-8	48	6	1	3	41	7	1
1	26	34	13*	2	35	9	14	3	41	9	-0	-7	32	36	25*	4	83	4	0
2	19	31	6*	K,L=	3,	14		4	67	6	2	-6	21	21	18*	K,L=	4,	10	
3	33	39	-4*	-6	17	24	14*	5	53	9	3	-5	31	21	-3*	-7	23	29	1*
4	60	4	4	-5	25	45	-1*	6	0	22	-1*	-4	49	5	-2	-6	18	29	12*
5	46	5	12	-4	93	5	-1	7	16	35	9*	-3	28	21	-1*	-5	25	42	4*
K,L=	3,	10		-3	34	12	-12*	K,L=	4,	3		-2	18	19	13*	-4	37	8	15
-8	25	27	21*	-2	45	12	6	-8	26	28	18*	-1	47	6	2	-3	19	27	17*
-7	24	25	1*	-1	0	25	-8*	-7	0	22	-9*	0	75	3	-1	-2	14	38	-1*
-6	0	39	-19*	0	35	14	32*	-6	24	31	23*	1	126	4	2	-1	0	42	-14*
-5	22	17	19*	1	33	13	14*	-5	10	26	-6*	2	49	5	9	0	66	7	7
-4	43	8	6	K,L=	3,	15		-4	43	5	-5	3	44	11	2	1	23	27	19*
-3	6	21	6*	-5	0	30	-12*	-3	14	20	-6*	4	23	19	-12*	2	23	15	15*
-2	61	8	6	-4	7	21	1*	-2	0	23	-13*	5	44	13	-14*	3	51	11	13
-1	76	8	3	-3	40	11	0	-1	36	12	1*	6	46	9	8	4	22	25	14*
0	32	53	-7*	-2	2	25	-14*	0	217	6	-2	K,L=	4,	7		K,L=	4,	11	
1	28	36	4*	-1	30	36	10*	1	71	3	5	-8	11	24	-2*	-7	12	39	7*
2	16	35	4*	0	28	39	20*	2	18	20	-14*	-7	31	11	-2*	-6	20	32	1*
3	52	5	5	K,L=	4,	0		3	11	24	9*	-6	8	32	3*	-5	28	8	28*
4	92	5	3	8	392	10	-11	4	26	10	7*	-5	13	35	-12*	-4	50	15	15
5	11	30	-9*	1	55	9	8	5	46	8	6	-4	0	20	-19*	-3	24	29	23*
K,L=	3,	11		2	44	5	-6	6	0	25	-11*	-3	51	7	10	-2	46	7	-12
-8	5	27	-17*	3	37	5	-10	7	7	23	5*	-2	8	24	-9*	-1	22	36	-28*
-7	43	8	19	4	31	11	2*	K,L=	4,	4		-1	33	13	6*	0	40	7	15
-6	17	23	8*	5	62	6	5	-8	54	17	2*	0	84	4	-3	1	29	37	3*
-5	19	22	-9*	6	32	14	-5*	-7	93	4	3	1	87	6	5	2	27	34	19*
-4	21	39	15*	7	36	8	5	-6	29	34	24*	2	24	32	3*	3	17	34	-11*
-3	80	4	-2	8	20	28	8*	-5	24	30	1*	3	38	6	8	K,L=	4,	12	
-2	22	25	-4*	K,L=	4,	1		-4	43	13	11*	4	31	7	20	-6	26	27	24*
-1	18	21	11*	-8	0	24	-15*	-3	24	21	-6*	5	29	10	-9*	-5	22	16	21*
0	12	22	7*	-7	0	36	-11*	-2	66	4	2	K,L=	4,	8		-4	30	11	-12*
1	19	35	8*	-6	71	5	1	-1	34	9	-9	-8	0	27	-17*	-3	11	30	-12*
2	22	29	18*	-5	21	27	17*	0	98	4	-2	-7	21	24	7*	-2	29	21	-1*
3	4	28	-15*	-4	51	10	1	1	18	20	-6*	-6	26	40	-13*	-1	12	25	4*
4	51	13	-6	-3	25	13	20*	2	41	6	-0	-5	54	11	2	0	22	39	5*
K,L=	5,	12		-2	118	5	-14	3	7	24	1*	-4	53	16	9	1	28	31	-14*
-8	34	13	-11*	-1	321	8	0	4	33	22	21*	-3	64	4	0	2	23	28	7*
-7	36	8	-8	0	159	4	4	5	25	22	1*	-2	25	11	-7*	K,L=	4,	13	
-5	0	29	-13*	1	57	3	3	6	16	27	2*	-1	14	19	9*	-6	18	34	7*
-5	0	24	-14*	2	49	7	4	7	27	29	6*	0	85	6	4	-5	21	27	19*
-4	54	8	-3	3	52	6	3	K,L=	4,	5		1	29	9	14*	-4	25	13	7*
-3	169	5	-4	4	78	3	5	-8	45	12	15	2	8	43	-1*	-3	15	31	-3*
-2	112	5	-3	5	32	16	-10*	-7	0	30	-1*	3	25	41	23*	-2	22	30	18*
-1	63	15	2	6	27	10	10*	-6	40	11	10	4	27	14	24*	-1	46	10	6
0	20	29	3*	7	0	25	-6*	-5	33	15	17*	5	56	6	-8	0	18	25	11*
1	12	21	-1*	8	17	22	15*	-4	35	11	-11*	K,L=	4,	9		1	51	11	14
2	28	32	5*	K,L=	4,	2		-3	94	3	3	-8	20	31	7*	K,L=	4,	14	
3	54	11	1	-8	31	9	16	-2	23	25	5*	-7	25	32	11*	-5	19	22	15*
K,L=	3,	13		-7	64	5	-6	-1	22	23	17*	-5	0	22	-1*	-4	52	7	-3

STRUCTURE FACTORS CONTINUED FOR
C(9)H(12)ON(4)

H	FOB	SG	DEL	H	FOB	SG	DEL	H	FOB	SG	DEL	H	FOB	SG	DEL	H	FOB	SG	DEL
-3	8	22	-3*	K,L=	5,	4		-7	19	27	16*	5	21	34	0*	-3	21	23	-7*
-2	24	38	20*	-7	46	25	-1*	-6	30	34	20*	6	32	11	0*	-2	22	23	-6*
-1	0	28	-9*	-6	35	10	1	-5	21	21	14*	K,L=	6,	1		-1	16	33	10*
K,L=	5,	0		-5	0	29	-6*	-4	37	28	8*	-5	23	26	1*	0	19	28	11*
1	52	9	7	-4	23	29	-1*	-3	41	12	-2	-5	36	13	6*	1	55	7	1
2	28	11	13*	-3	13	29	7*	-2	36	19	4*	-4	36	13	7*	2	7	21	6*
3	6	22	6*	-2	51	5	3	-1	50	17	9*	-3	24	14	1*	3	30	33	15*
4	7	33	-17*	-1	30	27	2*	0	42	11	-4	-2	6	34	-25*	4	30	18	22*
5	29	15	-16*	0	24	11	16*	1	0	22	-2*	-1	25	21	15*	K,L=	6,	6	
6	37	12	6*	1	22	11	-5*	2	13	22	1*	0	93	8	-8	-6	6	23	5*
7	8	22	8*	2	18	20	-3*	3	19	22	13*	1	34	13	24*	-5	24	32	20*
K,L=	5,	1		3	21	35	-10*	4	9	32	-4*	2	39	10	1	-4	25	28	6*
-7	16	33	4*	4	27	30	-7*	K,L=	5,	9		3	0	39	-7*	-3	0	26	-16*
-6	18	28	13*	5	31	22	10*	-6	0	32	-9*	4	0	25	-2*	-2	0	33	-3*
-5	44	9	3	6	36	12	24*	-5	0	22	-11*	5	32	8	13	-1	23	15	14*
-4	31	8	11	K,L=	5,	5		-4	16	27	9*	K,L=	6,	2		0	0	24	-19*
-3	18	21	9*	-7	13	23	13*	-3	35	9	-5	-6	33	9	3	1	39	6	4
-2	24	29	1*	-6	19	37	6*	-2	21	22	12*	-5	9	29	7*	2	21	23	19*
-1	44	12	-17	-5	3	38	-6*	-1	35	14	4*	-4	18	27	6*	3	19	24	12*
0	24	11	7*	-4	65	12	-7	0	22	30	13*	-3	20	30	2*	K,L=	6,	7	
1	0	28	-12*	-3	84	6	-2	1	35	6	12	-2	46	10	8	-5	41	13	38*
2	0	25	-6*	-2	79	4	9	2	23	19	19*	-1	97	6	-3	-4	22	28	-8*
3	22	27	18*	-1	31	29	7*	3	15	24	14*	0	31	13	13*	-3	4	30	-3*
4	83	4	-6	0	21	29	-13*	K,L=	5,	10		1	57	13	-4	-2	0	21	-1*
5	33	14	-10*	1	0	20	0*	-6	15	26	10*	2	0	33	-5*	-1	27	31	20*
6	10	23	-11*	2	14	22	3*	-5	28	34	9*	3	25	35	13*	0	33	13	7*
7	30	19	-3*	3	30	33	-1*	-4	37	12	20*	4	25	12	5*	1	29	8	20
K,L=	5,	2		4	27	11	-17*	-3	50	14	-1	5	26	26	20*	2	14	34	-6*
-7	28	40	15*	5	16	25	1*	-2	9	21	-9*	K,L=	6,	3		3	21	24	16*
-6	37	14	23*	K,L=	5,	6		-1	21	24	19*	-6	44	6	17	K,L=	6,	8	
-5	5	22	-24*	-7	0	34	-5*	0	0	26	-2*	-5	12	31	-11*	-5	22	26	0*
-4	41	6	8	-6	25	13	6*	1	0	29	-6*	-4	23	30	12*	-4	27	34	17*
-3	42	13	2*	-5	14	43	7*	2	13	26	13*	-3	13	26	0*	-3	22	24	-1*
-2	35	15	28*	-4	0	23	-6*	K,L=	5,	11		-2	0	30	-21*	-2	24	44	11*
-1	91	5	-5	-3	0	24	-7*	-5	21	23	14*	-1	108	4	-5	-1	33	8	-4
0	24	9	1*	-2	43	20	38*	-4	8	26	4*	0	23	28	-19*	0	37	8	-1
1	25	12	7*	-1	21	28	-5*	-3	48	8	-10	1	15	21	10*	1	7	31	8*
2	11	20	-18*	0	25	13	1*	-2	67	14	-11	2	24	24	9*	2	2	33	-1*
3	15	22	14*	1	15	40	4*	-1	50	5	-1	3	23	24	16*	K,L=	6,	9	
4	29	34	1*	2	19	20	14*	0	37	10	24	4	24	16	1*	-4	16	24	15*
5	12	24	12*	3	21	15	10*	1	24	13	22*	5	12	31	10*	-3	26	12	12*
6	4	29	1*	4	28	14	1*	K,L=	5,	12		K,L=	6,	4		-2	26	11	9*
K,L=	5,	3		5	28	30	2*	-5	32	17	29*	-6	26	21	17*	-1	22	20	18*
-7	64	18	4	K,L=	5,	7		-4	0	26	-5*	-5	32	18	29*	0	40	7	-2
-5	0	22	-10*	-7	43	9	7	-3	16	28	18*	-4	33	35	31*	1	25	25	5*
-5	0	43	-19*	-6	10	23	4*	-2	47	8	15	-3	22	31	-8*	K,L=	6,	10	
-4	16	21	6*	-5	16	28	-1*	-1	20	25	7*	-2	0	40	-5*	-3	14	35	11*
-3	45	5	5	-4	36	16	-13*	0	16	26	15*	-1	17	25	1*	-2	20	32	3*
-2	102	3	2	-3	18	23	15*	K,L=	5,	13		0	95	4	-2	-1	23	27	15*
-1	65	7	-9	-2	16	30	14*	-3	69	5	-6	1	20	30	4*	0	34	11	25*
0	24	30	9*	-1	14	24	-2*	-2	31	15	22*	2	18	29	5*	K,L=	7,	0	
1	41	5	10	0	33	13	27*	K,L=	6,	0		3	24	12	15*	1	0	31	-13*
2	18	28	-4*	1	27	28	4*	0	129	8	-2	4	21	26	9*	2	31	12	18*
3	68	4	-5	2	34	9	5	1	71	8	-3	K,L=	6,	5		3	9	25	3*
4	25	17	-1*	3	20	21	4*	2	34	7	18	-6	31	18	20*	K,L=	7,	1	
5	32	26	6*	4	0	33	-22*	3	17	38	2*	-5	37	7	26	-4	18	44	-4*
6	21	35	9*	K,L=	5,	8		4	0	23	-1*	-4	42	12	39*	-3	0	38	-7*

STRUCTURE FACTORS CONTINUED FOR
C(9)H(12)ON(4)

H	FOB	SG	DEL	H	FOB	SG	DEL	H	FOB	SG	DEL	H	FOB	SG	DEL	H	FOB	SG	DEL
-2	15	23	10*																
-1	33	12	9*																
0	0	21	-6*																
1	25	30	17*																
2	18	39	16*																
3	18	23	17*																
K,L=	7,	2																	
-4	0	26	-3*																
-3	28	35	16*																
-2	33	16	-0*																
-1	22	22	-17*																
0	0	22	-7*																
1	12	35	-12*																
2	0	25	-9*																
3	33	22	23*																
K,L=	7,	3																	
-4	0	27	-3*																
-3	32	11	23*																
-2	20	36	3*																
-1	29	12	9*																
0	0	22	-1*																
1	22	25	6*																
2	18	21	-2*																
K,L=	7,	4																	
-3	27	35	-6*																
-2	39	10	-9																
-1	15	33	7*																
0	11	25	8*																
1	18	34	12*																
2	8	24	2*																
K,L=	7,	5																	
-3	0	33	-21*																
-2	39	9	27																
-1	23	31	12*																
0	23	44	15*																
1	28	19	21*																
K,L=	7,	6																	
-3	23	33	1*																
-2	0	36	-11*																
-1	0	34	-3*																
0	28	34	18*																

TECHNICAL INFORMATION DIVISION
LAWRENCE BERKELEY LABORATORY
UNIVERSITY OF CALIFORNIA
BERKELEY, CALIFORNIA 94720