BENEDETTI, E., CORRADINI, P., PEDONE, C. & POST, B. (1969). *J. Amer. Chem. Soc.* **91**, 4072.
BENEDETTI, E., PEDONE, C. & ALLEGRA, G. (1970). *J. Phys. Chem.* **74**, 512
CORRADINI, P. (1960). *Acc. Naz. Lincei*, **24**, 8.
CRUICKSHANK, D. W. J. & PILLING, D. E. (1961). In *Computing Methods and the Phase Problem in X-ray Crystal Analysis*. Ed. R. PEPINSKY, J. M. ROBERTSON & J. C. SPEAKMAN, p. 32. New York: Pergamon Press.
DUNITZ, J. D. (1949). *Acta Cryst.* **2**, 1.
DUNITZ, J. D. & STRICKLER, P. (1968). In *Structural Chemistry and Molecular Biology*. Ed. by A. Rich & N. Davidson, p. 595. San Francisco: W. H. Freeman and Co.
GREENBERG, B. & POST, B. (1968). *Acta Cryst.* B**24**, 918.
KARLE, I. L., KARLE, J. & BRITTS, K. (1966). *J. Amer. Chem. Soc.* **88**, 2918.
KRAINOV, E. P., PROKOFIEV, N. I. & SVERDLOV, L. M. (1964). *Opt. i Spektr.* **16**(4), 566.
LORD, R. C. & NAKAGAWA, J. (1963). *J. Chem. Phys.* **39**, 2951.
MARGULIS, T. N. & FISHER, M. (1967). *J. Amer. Chem. Soc.* **89**, 223.
OWEN, T. B. & HOARD, J. L. (1951). *Acta Cryst.* **4**, 172.
RATHJENS, W. D., FREEMAN, J., GWINN, W. D. & PITZER, K. S. (1953). *J. Amer. Chem. Soc.* **75**, 5634.
SCHAFFER, K. (1961). *Z. Elektrochem.* **65**, 2.
Abstr. Amer. Cryst. Assoc. Meeting, Minneapolis.
VAN DER HELM, D., SIMS, J. J. & SEIGLER, D. S. (1967).
WILSON, T. (1943). *J. Chem. Phys.* **11**, 369.

Acta Cryst. (1970). B**26**, 498

The Crystal and Molecular Structure of *p*-Methylaminophenol Sulphate (Metol)

BY LUIGI CAVALCA, GIOVANNA FAVA GASPARRI, ALESSANDRO MANGIA AND GIANCARLO PELIZZI
Istituto di Strutturistica Chimica, Università degli Studi, Parma, Italy

(*Received* 13 *May* 1969)

A three-dimensional X-ray study of *p*-methylaminophenol sulphate (metol) has been accomplished by Patterson and Fourier methods. The crystals are monoclinic (space group $C2/c$ or Cc) with four molecules in the unit cell of dimensions: $a=23{\cdot}16$ (6), $b=5{\cdot}87$ (1), $c=14{\cdot}71$ (4) Å. Without considering the hydrogen atoms, two models of the structure, based on the two possible space groups, can be postulated: both give $R=11{\cdot}5\%$. The location of the hydrogen atoms is possible only with the model based on the $C2/c$ space group, in which there is a disordered arrangement of the SO_4^{2-} group; the introduction of the hydrogen atoms improves the R value to $10{\cdot}5\%$. Packing and hydrogen bonding are discussed.

Introduction

p-Methylaminophenol sulphate (metol) is a compound largely used in photographic work as a reducing agent. Although it is very common in X-ray laboratories, the available information on its crystal structure concerns only the morphological and optical properties and preliminary X-ray powder diffraction data (McCrone, Cook, Whitney, Corvin & Tull, 1948). The X-ray structural analysis of this compound was started in our laboratory as long ago as 1955, in order to study the hydrogen bonding system formed by the protonated methylamino and the hydroxyl phenolic groups with the oxygen atoms of the anion. This work was interrupted owing to the difficulties found in the solution of the two-dimensional structure. The three-dimensional analysis taken up recently showed that the trouble was due to the existence of two models, which are equivalent apart from the SO_4^{2-} situation, both giving nearly equal R values.

Experimental

Single crystals suitable for X-ray work were grown by slow cooling from aqueous solutions of the commercial product. In this way monoclinic prisms elongated along the b axis were obtained. Cell constants, determined from Weissenberg and rotation photographs, are as follows:

$(C_7H_9ON)_2 \cdot H_2SO_4$; $M=344{\cdot}4$
$a=23{\cdot}16\pm0{\cdot}06$, $b=5{\cdot}87\pm0{\cdot}01$, $c=14{\cdot}71\pm0{\cdot}04$ Å
$\beta=126°42'\pm7'$
$V=1603$ Å^3, $Z=4$, $D_x=1{\cdot}422$, $D_m=1{\cdot}416$ g.cm^{-3},
$\mu=22{\cdot}3$ cm^{-1} (Cu $K\alpha$).

Space group $C2/c$ or Cc (from systematic absences). These data agree well with those previously given in the literature if the crystal axes are changed according to the trasformation matrix: $\bar{1}0\bar{1}/010/001$.

A set of intensity data was obtained up to the 5th layer around [010] and up to the 13th layer around [001] on an integrating Weissenberg camera by means of the multiple-film technique and Ni-filtered Cu $K\alpha$ radiation. 1764 independent reflexions were collected out of the 1976 possible ones contained in the limiting sphere; 333 were too weak to be measured. For the photographs taken around [010] the crystal used was a prism with nearly rectangular cross-section ($0{\cdot}014\times 0{\cdot}021$ cm), while for the data taken around [001] a

Table 1. *Final atomic fractional coordinates* ($\times 10^4$) *with e.s.d.'s, thermal parameters* ($\times 10$ Å^2*) *and ratios (e.s.d.)/(coordinate shift)*

	x/a	y/b	z/c	B_{11}	B_{22}	B_{33}	B_{23}	B_{13}	B_{12}	$r(x)$	$r(y)$	$r(z)$
S	0	2367±2	2500	18	19	31	0	12	0	—	6	—
O(1)	−407±23	1356±49	1334±28	79	86	34	−35	18	−37	58	4	2
O(2)	699±11	1401±13	3199±18	16	36	67	7	12	12	11	5	14
O(3)	−425±12	1778±13	2968±24	60	32	97	9	69	9	6	7	59
O(4)	0	4853±8	2500	29	27	58	−7	28	1	—	7	—
O(5)	1169±4	8287±10	4985±8	19	66	48	5	14	−3	9	14	7
N	4154±4	7491±6	7801±7	21	29	34	−2	16	−1	11	6	18
C(1)	3097±5	6849±8	4341±7	20	40	27	−4	13	−1	∞	7	12
C(2)	2684±6	5104±9	4316±9	28	32	31	1	17	−2	11	5	43
C(3)	1933±5	5341±7	3603±7	24	27	32	1	15	−2	6	4	6
C(4)	1627±4	7268±6	2954±6	22	23	29	−5	16	−2	42	21	7
C(5)	2037±5	9008±9	2971±8	27	30	31	2	17	−4	13	29	∞
C(6)	2787±6	8786±11	3680±9	27	42	39	1	20	−7	15	12	4
C(7)	531±7	7112±18	979±11	27	92	38	−14	9	3	24	∞	28

* The B_{ij}'s refer to the thermal factor in the form:

$$\exp\left[-\tfrac{1}{4}(B_{11}h^2a^{*2}+B_{22}k^2b^{*2}+B_{33}l^2c^{*2}+2B_{12}hka^*b^*+2B_{13}hla^*c^*+2B_{23}klb^*c^*)\right].$$

Table 2. *Atomic fractional coordinates* ($\times 10^4$) *and isotropic thermal parameters of the hydrogen atoms*

	x/a	y/b	z/c	B
H(1)	1083	9333	4667	3·1 Å^2
H(2)	2917	3500	4751	2·9
H(3)	1583	4067	3417	3·6
H(4)	4250	6500	7333	5·0
H(5)	1834	167	2500	5·0
H(6)	3083	10000	3667	2·7
H(7)	4333	8667	7667	2·8
H(8)	667	5333	1000	2·8
H(9)	84	7000	500	4·0
H(10)	750	8333	667	5·0

rectangular fragment (cross-section: 0·018 × 0·051 cm) was selected. Discontinuous absorption effects were corrected graphically using Albrecht's (1939) method and the shape of the spots of non-equatorial layers was taken into account following Phillips (1956). The structure amplitudes, derived by the usual formulae, were put on the same relative scale by the least-squares cross-correlation method of Rollett & Sparks (1960). The absolute scale factor was determined first by Wilson's method, then by correlating the observed with the calculated values.

Table 3. *Atomic peak heights* (e.Å^{-3}) *and curvatures* (e.Å^{-5})

		ϱ	$-A_{hh}$	$-A_{kk}$	$-A_{ll}$	A_{kl}	A_{hl}	A_{hk}
S	Obs	38·0	387	387	328	−3	208	0
	Calc	38·0	387	387	328	−4	208	0
O(1)	Obs	3·6	17	20	19	2	9	−7
	Calc	3·7	18	20	19	3	9	−7
O(2)	Obs	5·8	41	48	36	−1	24	−1
	Calc	5·8	41	48	36	−1	24	0
O(3)	Obs	5·3	33	54	24	−1	16	4
	Calc	5·4	33	53	24	−1	15	4
O(4)	Obs	11·6	103	86	73	−2	53	1
	Calc	11·4	103	85	73	−1	53	1
O(5)	Obs	10·5	92	68	67	1	43	−3
	Calc	10·3	91	68	67	0	43	−2
N	Obs	11·4	106	104	95	−4	63	2
	Calc	11·3	106	104	95	−4	73	2
C(1)	Obs	9·4	87	77	87	−5	52	1
	Calc	9·4	88	75	86	−5	52	2
C(2)	Obs	8·9	74	79	76	−1	45	3
	Calc	8·9	75	79	75	0	45	2
C(3)	Obs	9·7	89	86	84	−1	52	−7
	Calc	9·6	89	86	84	−1	52	−8
C(4)	Obs	10·6	100	98	101	−6	62	3
	Calc	10·6	100	98	102	−6	62	3
C(5)	Obs	9·3	86	83	82	4	53	4
	Calc	9·3	86	84	83	4	53	5
C(6)	Obs	8·6	80	68	73	4	48	−10
	Calc	8·6	80	67	73	4	47	−10
C(7)	Obs	6·4	55	32	49	−6	29	5
	Calc	6·3	55	32	48	−4	28	3
	E.s.d.	0·2	2	2	2	1	1	1

Table 4. *Observed and calculated structure factors*

A minus sign after an F_o means 'less than'.

h	k	l	$10F_o$	$10F_c$
0	2	0	1626	-1014
0	4	0	499	441
0	6	0	82	-73
0	2	1	410	-395
0	4	1	438	378
0	6	1	262	-229
0	0	2	1165	-1303
0	2	2	495	531
0	4	2	414	-387
0	6	2	298	291
0	2	3	182	-172
0	4	3	191	-151
0	6	3	41-	-4
0	0	4	663	668
0	2	4	718	-762
0	4	4	371	360
0	6	4	177	-168
0	2	5	89	-27
0	4	5	196	193
0	6	5	22-	-31
0	0	6	468	-433
0	2	6	252	262
0	4	6	434	-449
0	6	6	68	79
0	2	7	28-	4
0	4	7	34-	-26
0	6	7	55	46
0	0	8	311	310
0	2	8	140	-115
0	4	8	126	93
0	6	8	70	-71
0	2	9	37-	14
0	4	9	82	-106
0	6	9	16-	-18
0	0	10	34	-43
0	2	10	112	-126
0	4	10	94	-123
0	2	11	126	118
0	4	11	32	24
0	0	12	32-	4
0	2	12	49	-60
0	4	12	71	104
0	2	13	13-	-12
0	0	14	145	148
0	2	14	66	63
1	1	0	222	287
1	3	0	688	-760
1	5	0	64-	17
1	7	0	60	-45
1	1	1	920	-955
1	1	$\bar{1}$	429	-575
1	3	1	211	-190
1	3	$\bar{1}$	108	-130
1	5	1	54-	22
1	5	$\bar{1}$	54-	-27
1	7	1	63	-41
1	7	$\bar{1}$	58	-50
1	1	2	29-	29
1	1	$\bar{2}$	458	426
1	3	2	117	-99
1	3	$\bar{2}$	540	527
1	5	2	332	-226
1	5	$\bar{2}$	334	319
1	7	1	94	80
1	7	$\bar{2}$	71	54
1	1	3	340	348
1	1	$\bar{3}$	349	-316
1	3	3	73	52
1	3	$\bar{3}$	399	386
1	5	3	241	224
1	5	$\bar{3}$	51	-42
1	7	3	47	-48
1	7	$\bar{3}$	24-	-9
1	1	4	383	-340
1	1	$\bar{4}$	684	695
1	3	4	215	-201
1	3	$\bar{4}$	418	-316
1	5	4	113	106
1	5	$\bar{4}$	49	34
1	7	4	125	-126
1	7	$\bar{4}$	16-	-17
1	1	5	284	-288
1	1	$\bar{5}$	359	-255
1	3	5	241	212
1	3	$\bar{5}$	162	-172
1	5	5	166	-171
1	5	$\bar{5}$	203	154
1	7	$\bar{5}$	54	24
1	1	6	218	210
1	1	$\bar{6}$	325	294
1	3	6	112	121
1	3	$\bar{6}$	284	-240
1	5	6	141	-154
1	5	$\bar{6}$	67	72
1	1	7	141	142
1	1	$\bar{7}$	71	-103
1	3	7	212	-195
1	3	$\bar{7}$	140	121
1	5	7	145	130
1	5	$\bar{7}$	174	-157
1	1	8	167	-143
1	1	$\bar{8}$	100	-53
1	3	8	147	-174
1	3	$\bar{8}$	72	-78
1	5	8	85	81
1	5	$\bar{8}$	31-	-18
1	1	9	343	-306
1	1	$\bar{9}$	337	296
1	3	9	145	109
1	3	$\bar{9}$	253	-263
1	5	9	82	-97
1	5	$\bar{9}$	149	159
1	1	10	144	151
1	1	$\overline{10}$	60	-72
1	3	10	24-	9
1	3	$\overline{10}$	113	125
1	5	10	20	-17
1	5	$\overline{10}$	49	-65
1	1	11	268	244
1	1	$\overline{11}$	289	-267
1	3	11	80	-76
1	3	$\overline{11}$	224	233
1	5	$\overline{11}$	110	-117
1	1	12	137	126
1	1	$\overline{12}$	122	-123
1	3	12	26-	1
1	3	$\overline{12}$	85	-87
1	1	13	76	-86
1	1	$\overline{13}$	113	100
1	3	13	93	97
1	3	$\overline{13}$	103	-111
1	1	14	29-	16
1	1	$\overline{14}$	37-	40
1	3	$\overline{14}$	22-	-22
1	1	$\overline{15}$	203	-254
2	0	0	370	270
2	2	0	899	-1170
2	4	0	300	270
2	6	0	125	-117
2	2	1	928	-989
2	2	$\bar{1}$	433	394
2	4	1	403	347
2	4	$\bar{1}$	59	1
2	6	1	88	75
2	6	$\bar{1}$	115	-103
2	0	2	1017	-1132
2	0	$\bar{2}$	1016	-1165
2	2	2	569	623
2	2	$\bar{2}$	39	51
2	4	2	410	-430
2	4	$\bar{2}$	430	-374
2	6	2	59-	-13
2	6	$\bar{2}$	61-	29
2	2	3	346	-335
2	2	$\bar{3}$	649	-654
2	4	3	73	73
2	4	$\bar{3}$	271	221
2	6	3	63	39
2	6	$\bar{3}$	178	-155
2	0	4	191	198
2	0	$\bar{4}$	1573	1885
2	2	4	18-	-40
2	2	$\bar{4}$	37	-75
2	4	4	353	363
2	4	$\bar{4}$	338	324
2	6	4	88	-90
2	6	$\bar{4}$	46	-62
2	2	5	118	50
2	2	$\bar{5}$	543	-532
2	4	5	31	-42
2	4	$\bar{5}$	37	-51
2	6	5	45	-36
2	6	$\bar{5}$	144	149
2	0	6	453	-403
2	0	$\bar{6}$	434	-417
2	2	6	286	268
2	2	$\bar{6}$	311	302
2	4	6	284	-319
2	4	$\bar{6}$	154	-153
2	6	6	106	101
2	6	$\bar{6}$	193	210
2	2	7	111	131
2	2	$\bar{7}$	74	57
2	4	7	66	-48
2	4	$\bar{7}$	238	237
2	6	7	69	48
2	6	$\bar{7}$	63	-72
2	0	8	658	615
2	0	$\bar{8}$	291	280
2	2	8	263	-237
2	2	$\bar{8}$	161	-138
2	4	8	260	274
2	4	$\bar{8}$	180	176
2	6	8	41	-36
2	6	$\bar{8}$	69	-75
2	2	9	119	-127
2	2	$\bar{9}$	34-	-46
2	4	9	98	111
2	4	$\bar{9}$	126	-131
2	6	$\bar{9}$	54	55
2	0	10	24-	-26
2	0	$\overline{10}$	128	-146
2	2	10	158	152
2	2	$\overline{10}$	97	76
2	4	10	56	-51
2	4	$\overline{10}$	82	-87
2	2	11	139	-163
2	2	$\overline{11}$	14-	35
2	4	11	10-	-23
2	4	$\overline{11}$	15	29
2	0	12	266	258
2	0	$\overline{12}$	170	153
2	2	12	137	-149
2	2	$\overline{12}$	32-	-7
2	4	$\overline{12}$	44	52
2	2	13	11-	-21
2	2	$\overline{13}$	53	-51
2	4	$\overline{13}$	10-	12
2	0	14	217	-256
2	0	$\overline{14}$	214	-177
2	2	$\overline{14}$	31-	-5
2	2	$\overline{15}$	22-	-22
3	1	0	213	-179
3	3	0	135	-78
3	5	0	81	57
3	7	0	113	-109
3	1	1	594	-613
3	1	$\bar{1}$	1249	1593
3	3	1	360	319
3	3	$\bar{1}$	168	-154
3	5	1	292	-269
3	5	$\bar{1}$	411	382
3	7	1	100	100
3	7	$\bar{1}$	100	-106
3	1	2	257	224
3	1	$\bar{2}$	637	-512
3	3	2	599	568
3	3	$\bar{2}$	492	477
3	5	2	69	-72
3	5	$\bar{2}$	426	-395
3	7	2	158	132
3	7	$\bar{2}$	82	75
3	1	3	253	262
3	1	$\bar{3}$	1742	-2479
3	3	3	533	-532
3	3	$\bar{3}$	253	252
3	5	3	234	206
3	5	$\bar{3}$	179	-172
3	7	3	67	-59
3	7	$\bar{3}$	162	159
3	1	4	318	-319
3	1	$\bar{4}$	1200	-1315
3	3	4	111	-106
3	3	$\bar{4}$	195	-204
3	5	4	155	147
3	5	$\bar{4}$	63	-26
3	7	$\bar{4}$	111	-109
3	1	5	421	-385
3	1	$\bar{5}$	204	-131
3	3	5	277	256
3	3	$\bar{5}$	301	-298
3	5	5	79	-63
3	5	$\bar{5}$	246	242
3	7	$\bar{5}$	237	-240
3	1	6	266	232
3	1	$\bar{6}$	429	364
3	3	6	146	156
3	3	$\bar{6}$	134	142
3	5	6	30-	39
3	5	$\bar{6}$	61	62
3	7	$\bar{6}$	45	34
3	1	7	199	-140
3	1	$\bar{7}$	513	-483
3	3	7	50	-30
3	3	$\bar{7}$	399	403
3	5	7	62	57
3	5	$\bar{7}$	233	-219
3	1	8	195	175
3	1	$\bar{8}$	143	115
3	3	8	36-	-43
3	3	$\bar{8}$	48	-26
3	5	8	27-	-26
3	5	$\bar{8}$	34-	31
3	1	9	39	-95
3	1	$\bar{9}$	254	235
3	3	9	51	47
3	3	$\bar{9}$	254	-234
3	5	9	53	-52
3	5	$\bar{9}$	189	205
3	1	10	183	-169
3	1	$\overline{10}$	21-	-4
3	3	10	21-	19
3	3	$\overline{10}$	33	35
3	5	10	10-	30
3	5	$\overline{10}$	57	59
3	1	11	101	-105
3	1	$\overline{11}$	103	-109
3	3	11	49	-65
3	3	$\overline{11}$	75	63
3	5	$\overline{11}$	81	-97
3	1	12	94	-104
3	1	$\overline{12}$	31-	8
3	3	12	41	-46
3	3	$\overline{12}$	60	76
3	5	$\overline{12}$	17-	-16
3	1	13	40	-42
3	1	$\overline{13}$	65	51
3	3	$\overline{13}$	125	-136
3	1	14	42-	-2
3	3	$\overline{14}$	83	87
3	1	15	35	-36
3	3	$\overline{15}$	104	145
4	0	0	866	970
4	2	0	467	416
4	4	0	234	225
4	6	0	60-	-32
4	2	1	103	80
4	2	$\bar{1}$	47	-56
4	4	1	59	65
4	4	$\bar{1}$	230	-193
4	6	1	140	-117
4	6	$\bar{1}$	58	52
4	0	2	997	-1015
4	0	$\bar{2}$	433	-543
4	2	2	291	264
4	2	$\bar{2}$	151	182
4	4	2	280	-303
4	4	$\bar{2}$	253	223
4	6	2	150	145
4	6	$\bar{2}$	63	-40
4	2	3	146	-104
4	2	$\bar{3}$	95	-10
4	4	3	115	-152
4	4	$\bar{3}$	112	67
4	6	3	132	128
4	6	$\bar{3}$	150	136
4	0	4	448	383
4	0	$\bar{4}$	1258	-1428
4	2	4	144	-120
4	2	$\bar{4}$	334	262
4	4	4	376	419
4	4	$\bar{4}$	160	126
4	6	4	126	-113
4	6	$\bar{4}$	29	-56
4	2	5	21-	-1
4	2	$\bar{5}$	301	253
4	4	5	198	242
4	4	$\bar{5}$	111	116
4	6	5	111	-113
4	6	$\bar{5}$	56	-58
4	0	6	835	-800
4	0	$\bar{6}$	591	-539
4	2	6	443	405
4	2	$\bar{6}$	244	-206
4	4	6	212	-196
4	4	$\bar{6}$	367	-353
4	6	6	140	128
4	6	$\bar{6}$	129	-128
4	2	7	140	-126
4	2	$\bar{7}$	306	-263
4	4	7	139	-148
4	4	$\bar{7}$	32-	-9
4	6	7	45	38
4	6	$\bar{7}$	28	-59
4	0	8	457	432
4	0	$\bar{8}$	356	-372
4	2	8	271	-396
4	2	$\bar{8}$	179	136
4	4	8	104	106
4	4	$\bar{8}$	153	151
4	6	$\bar{8}$	27	-37
4	2	9	131	-137
4	2	$\bar{9}$	165	-134
4	4	9	128	118
4	4	$\bar{9}$	57	62
4	6	$\bar{9}$	24-	24
4	0	10	490	-400
4	0	10	225	-239
4	2	10	166	169
4	2	$\overline{10}$	168	147
4	4	10	209	-217
4	4	$\overline{10}$	105	-83
4	6	$\overline{10}$	49	51
4	2	11	58	61
4	2	$\overline{11}$	58	-48
4	4	11	59	40
4	4	$\overline{11}$	97	96
4	0	12	26-	6
4	0	$\overline{12}$	280	226
4	2	12	50	-54
4	2	$\overline{12}$	137	-175
4	4	$\overline{12}$	159	169
4	2	$\overline{13}$	77	45
4	4	$\overline{14}$	93	-95
4	0	$\overline{14}$	95-	-78
4	2	$\overline{14}$	137	142
4	4	$\overline{14}$	81	-99
4	2	$\overline{15}$	29-	39
4	0	$\overline{16}$	130	163
5	1	0	459	-394
5	3	0	193	-109
5	5	0	65-	-14
5	7	0	119	-105
5	1	1	97	-117
5	1	$\bar{1}$	1288	1618
5	3	1	377	393
5	3	$\bar{1}$	745	-757
5	5	1	262	-212
5	5	$\bar{1}$	215	195
5	7	1	117	104
5	7	$\bar{1}$	110	-106
5	1	2	386	327
5	1	$\bar{2}$	344	-340
5	3	2	58	-12
5	3	$\bar{2}$	741	698
5	5	2	72	36
5	5	$\bar{2}$	151	146
5	7	2	76	68
5	7	$\bar{2}$	136	126
5	1	3	83	95
5	1	$\bar{3}$	420	-424
5	3	3	255	-310
5	3	$\bar{3}$	590	595
5	5	3	232	202
5	5	$\bar{3}$	475	-454
5	7	$\bar{3}$	110	111
5	1	4	161	-151
5	1	$\bar{4}$	638	563
5	3	4	72	-63
5	3	$\bar{4}$	136	154
5	5	4	39	-34
5	5	$\bar{4}$	31	-33
5	7	$\bar{4}$	61	-52
5	1	5	366	-343
5	1	$\bar{5}$	963	964
5	3	5	235	234
5	3	$\bar{5}$	375	-402
5	5	5	166	-165
5	5	$\bar{5}$	403	331
5	7	$\bar{5}$	55	-69
5	1	6	369	-347
5	1	$\bar{6}$	24	-8
5	3	6	179	185
5	3	$\bar{6}$	251	249
5	5	6	130	-123
5	5	$\bar{6}$	167	-152
5	7	$\bar{6}$	55	59
5	1	7	230	312
5	1	$\bar{7}$	779	-724
5	3	7	34-	15
5	3	$\bar{7}$	355	356
5	5	7	54	51
5	5	$\bar{7}$	257	-250
5	1	8	217	-218
5	1	$\bar{8}$	675	-611
5	3	8	25-	25
5	3	$\bar{8}$	61	-89
5	5	8	22-	-31
5	5	$\bar{8}$	25-	25
5	1	9	58	-73
5	1	$\bar{9}$	562	528
5	3	9	35-	-29
5	3	$\bar{9}$	234	-221
5	5	9	16-	6
5	5	$\bar{9}$	133	147
5	1	10	110	121
5	1	$\overline{10}$	62	-25
5	3	10	121	126
5	3	$\overline{10}$	135	138
5	5	$\overline{10}$	117	-123
5	1	11	189	204
5	1	$\overline{11}$	245	-219
5	3	11	31	-61
5	3	$\overline{11}$	105	102
5	5	$\overline{11}$	95	-124
5	1	12	23-	13
5	1	$\overline{12}$	20-	-34
5	3	$\overline{12}$	32-	-23
5	5	$\overline{12}$	38	26
5	1	$\overline{13}$	93	78
5	3	$\overline{13}$	53	-34
5	1	$\overline{14}$	45-	29
5	3	$\overline{14}$	34-	-42
5	1	$\overline{15}$	29-	-41
5	3	$\overline{15}$	25-	-2
5	1	$\overline{16}$	38-	-3
6	0	0	213	202
6	2	0	95	-87
6	4	0	369	361
6	6	0	132	-120
6	2	1	651	217
6	2	$\bar{1}$	442	381
6	4	1	152	161
6	4	$\bar{1}$	209	-119
6	6	1	162	-163
6	6	$\bar{1}$	90	100
6	0	2	930	-866
6	0	$\bar{2}$	457	-372
6	2	2	258	234
6	2	$\bar{2}$	537	-459
6	4	2	373	-380
6	4	$\bar{2}$	398	-350
6	6	2	54-	26
6	6	$\bar{2}$	141	139
6	2	3	34-	-17
6	2	$\bar{3}$	284	308
6	4	3	204	-205
6	4	$\bar{3}$	78	-53
6	6	3	88	80
6	6	$\bar{3}$	100	-99
6	0	4	274	-233
6	0	$\bar{4}$	1490	1517
6	2	4	249	-229
6	2	$\bar{4}$	55	-17
6	4	4	182	170
6	4	$\bar{4}$	232	209
6	6	4	77	-80
6	6	$\bar{4}$	82	-87
6	2	5	66	-50
6	2	$\bar{5}$	18	16
6	4	5	60	60
6	4	$\bar{5}$	405	-397
6	6	5	16-	4
6	6	$\bar{5}$	73	70
6	0	6	265	-275
6	0	$\bar{6}$	269	-225
6	2	6	217	203
6	2	$\bar{6}$	311	277
6	4	6	174	-167
6	4	$\bar{6}$	118	109
6	6	6	15-	-20
6	6	$\bar{6}$	158	172
6	2	7	367	394
6	2	$\bar{7}$	685	670
6	4	7	30-	-36
6	4	$\bar{7}$	57	-49
6	6	$\bar{7}$	47	31
6	0	8	256	240
6	0	$\bar{8}$	60	62
6	2	8	36-	27
6	2	$\bar{8}$	252	216
6	4	8	220	227
6	4	$\bar{8}$	158	160
6	6	$\bar{8}$	76	-82
6	2	9	37-	-9
6	2	$\bar{9}$	181	182
6	4	9	80	-66
6	4	$\bar{9}$	36-	-19
6	6	$\bar{9}$	26-	25
6	0	10	68	-72
6	0	$\overline{10}$	555	-482
6	2	10	96	100
6	2	$\overline{10}$	112	101
6	4	10	11-	-29
6	4	$\overline{10}$	201	-240
6	6	$\overline{10}$	14-	5
6	2	11	10-	-13
6	2	$\overline{11}$	114	-113
6	4	$\overline{11}$	15	-18
6	0	12	60	57
6	0	$\overline{12}$	70	78
6	2	$\overline{12}$	139	-126
6	4	$\overline{12}$	89	115
6	2	$\overline{13}$	16-	-4
6	4	$\overline{13}$	46	30
6	0	$\overline{14}$	100-	-78
6	2	$\overline{14}$	72	63
6	4	$\overline{14}$	103	-108
6	2	$\overline{15}$	33-	13
6	0	$\overline{16}$	94-	71
6	2	$\overline{15}$	159	-169
7	1	0	402	-335
7	3	0	123	-71
7	5	0	65-	-11
7	7	0	35-	-74
7	1	1	241	-225
7	1	$\bar{1}$	799	787
7	3	1	321	326
7	3	$\bar{1}$	191	-221
7	5	1	414	-391
7	5	$\bar{1}$	321	296
7	7	1	92	81
7	7	$\bar{1}$	137	-129
7	1	2	245	209
7	1	$\bar{2}$	433	404
7	3	2	56-	-16
7	3	$\bar{2}$	173	-138
7	5	2	139	-153
7	5	$\bar{2}$	63-	-7
7	7	$\bar{2}$	37-	19
7	1	3	830	767
7	1	$\bar{3}$	776	-175
7	3	3	269	-245
7	3	$\bar{3}$	84	65
7	5	3	262	238
7	5	$\bar{3}$	119	-110
7	7	$\bar{3}$	155	148
7	1	4	199	-180
7	1	$\bar{4}$	753	-716
7	3	4	34	-30
7	3	$\bar{4}$	352	-284
7	5	4	170	159
7	5	$\bar{4}$	46	-75
7	7	$\bar{4}$	98	-84
7	1	5	546	-514
7	1	$\bar{5}$	335	361
7	3	5	294	276
7	3	$\bar{5}$	97	-88
7	5	5	158	-154
7	5	$\bar{5}$	49	24
7	7	$\bar{5}$	94	-95
7	1	6	181	174
7	1	$\bar{6}$	261	220
7	3	6	308	321
7	3	$\bar{6}$	81	-112
7	5	6	45	-35
7	5	$\bar{6}$	179	163
7	7	$\bar{6}$	63	62
7	1	7	590	586
7	1	$\bar{7}$	299	201
7	3	7	298	-282
7	3	$\bar{7}$	195	192
7	5	7	64	74
7	5	$\bar{7}$	269	-248
7	1	8	149	140
7	1	$\bar{8}$	668	576
7	3	8	144	-151
7	3	$\bar{8}$	173	-159
7	5	8	27	41
7	5	$\bar{8}$	42	45
7	1	9	62	-42
7	1	$\bar{9}$	335	327
7	3	9	139	150
7	3	$\bar{9}$	243	-240
7	5	$\bar{9}$	71	79
7	1	10	75	-70
7	1	$\overline{10}$	27	44
7	3	10	47	-54
7	3	$\overline{10}$	228	236
7	5	$\overline{10}$	21-	33
7	1	11	105	121
7	1	$\overline{11}$	32	16
7	3	11	138	129
7	5	$\overline{11}$	45	-57
7	1	$\overline{12}$	78	-64
7	3	$\overline{12}$	58	-69
7	5	$\overline{12}$	23-	14
7	1	$\overline{13}$	236	205
7	3	$\overline{13}$	117	-115
7	5	$\overline{13}$	45	40
7	1	$\overline{14}$	47-	-19
7	3	$\overline{14}$	76	72
7	1	$\overline{15}$	113	-109
7	3	$\overline{15}$	85	102
7	1	$\overline{16}$	45-	-12
7	3	$\overline{16}$	36	-49
7	1	$\overline{17}$	30-	26
8	0	0	501	-413
8	2	0	76	24
8	4	0	316	277
8	6	0	55-	-65
8	2	1	247	231
8	2	$\bar{1}$	298	-258
8	4	1	53-	-17
8	4	1	51-	10
8	6	1	53	33
8	6	$\bar{1}$	46-	18
8	0	2	600	477
8	0	$\bar{2}$	159	-164
8	2	2	96	125
8	2	$\bar{2}$	167	161
8	4	2	230	-222
8	4	$\bar{2}$	393	-385
8	6	2	49-	53
8	6	$\bar{2}$	133	108
8	2	3	293	280
8	2	$\bar{3}$	91	-76
8	4	3	145	-155
8	4	$\bar{3}$	85	71
8	6	3	65	58
8	6	$\bar{3}$	58	-92
8	0	4	666	662
8	0	$\bar{4}$	1161	1232
8	2	4	58	70
8	2	$\bar{4}$	682	-661
8	4	4	131	113
8	4	$\bar{4}$	538	476
8	6	4	69	73
8	6	$\bar{4}$	144	-143
8	2	5	83	61
8	2	$\bar{5}$	348	352
8	4	5	44	-35
8	4	$\bar{5}$	70	72
8	6	$\bar{5}$	58	61
8	0	6	23-	-65
8	0	$\bar{6}$	235	-179
8	2	6	220	-220
8	2	$\bar{6}$	63	-35
8	4	6	59	74
8	4	$\bar{6}$	715	-705

Structure analysis and refinement

The first step in solving the structure was a two-dimensional analysis based on the assumption of the $C2/c$ space group. This analysis carried out on the (010) projection gave the location of the organic molecule, but was unsuccessful in determining the orientation of the SO_4^{2-} group. A 0·22 value for the $R(h0l)$ index was obtained by putting the SO_4^{2-} tetrahedron on the twofold axis, and it was impossible to improve this value by changing the coordinates of the oxygen atoms.

The analysis resumed with a three-dimensional Patterson synthesis showed that the SO_4^{2-} tetrahedron must have an S–O direction parallel to [010]. Therefore there were two possibilities connected with the space group choice:

(i) There are no symmetry restrictions on the SO_4^{2-} orientation and the space group is Cc.

(ii) The SO_4^{2-} tetrahedron is oriented in such a way that an S–O bond is on a twofold axis in the $C2/c$ space group, with a disordered distribution around that axis of the tetrahedron which degenerates into an hexagonal pyramid. This last hypothesis was suggested by the presence in the two-dimensional projections of a ring-

like region of electron density, around the sulphur peak, having a radius consistent with the projection of the S–O bond.

The structural analysis was then carried out assuming both hypotheses independently. Fourier and Booth's differential syntheses with anisotropic thermal parameters improved the R index to 11·5% in both cases (not considering the hydrogen atoms). At the end of the two refinements the differences between the two models concerned only the orientation of the SO_4^{2-} groups. The centrosymmetrical hypothesis was preferred on the atoms which was achieved in the final $F_o - F_c$ synthesis only for the centrosymmetrical model,

(ii) A better hydrogen-bonding scheme,

(iii) The well known fact (see *e.g.* Wilson, 1950; Phillips, Rogers & Wilson, 1950; Hamilton, 1965) that, the R value being equal, the centrosymmetrical model is always more reliable. The introduction of the hydrogen atom contributions in the last structure factor calculation improved the $R(hkl)$ value to 10·4% (for observed reflexions only: $R' = 13{\cdot}1\%$ assuming $F_o = \frac{1}{2}F_{\min}$ when $F_c > F_{\min}$ for unobserved reflexions, multiplicities

Table 4 (*cont.*)

h	k	l	$10F_o$	$10F_c$
18	4	$\bar{7}$	117	157
18	0	$\bar{8}$	442	384
18	2	$\bar{8}$	355	−318
18	4	$\bar{8}$	34−	9
18	2	$\bar{9}$	153	−142
18	4	$\bar{9}$	118	−100
18	0	$\bar{10}$	131	−110
18	2	$\bar{10}$	24−	−27
18	4	$\bar{10}$	97	−144
18	2	$\bar{11}$	108	117
18	4	$\bar{11}$	53	−31
18	0	$\bar{12}$	163	152
18	2	$\bar{12}$	84	−72
18	4	$\bar{12}$	156	139
18	2	$\bar{13}$	16−	−2
18	4	$\bar{13}$	13−	−15
18	0	$\bar{14}$	356	−305
18	2	$\bar{14}$	113	104
18	4	$\bar{14}$	174	−140
18	2	$\bar{15}$	38−	−8
18	4	$\bar{15}$	57	53
18	0	$\bar{16}$	263	234
18	2	$\bar{16}$	43−	−50
18	2	$\bar{17}$	66	−100
18	0	$\bar{18}$	102	−99
18	2	$\bar{18}$	132	153
19	1	0	51−	−69
19	3	0	51−	6
19	1	1	148	−136
19	1	$\bar{1}$	53−	36
19	3	1	113	132
19	3	$\bar{1}$	144	−127
19	1	2	51−	35
19	1	$\bar{2}$	130	−136
19	3	2	35−	−5
19	3	$\bar{2}$	119	117
19	5	$\bar{2}$	31−	−9
19	1	3	152	158
19	1	$\bar{3}$	411	−405
19	3	$\bar{3}$	154	133
19	5	$\bar{3}$	88	−110
19	1	4	13−	−11
19	1	$\bar{4}$	50	45
19	3	$\bar{4}$	125	−122
19	5	$\bar{4}$	18−	20
19	1	$\bar{5}$	69	61
19	3	$\bar{5}$	25−	−19
19	5	$\bar{5}$	53	57
19	1	$\bar{6}$	348	330
19	3	$\bar{6}$	207	202
19	5	$\bar{6}$	23−	−14
19	1	$\bar{7}$	495	−487
19	3	$\bar{7}$	34−	29
19	5	$\bar{7}$	42	35
19	1	$\bar{8}$	87	−72
19	3	$\bar{8}$	103	−113
19	5	$\bar{8}$	116	143
19	1	$\bar{9}$	173	151
19	3	$\bar{9}$	68	−66
19	5	$\bar{9}$	110	147
19	1	$\bar{10}$	23−	11
19	3	$\bar{10}$	24−	−18
19	5	$\bar{10}$	51	−55
19	1	$\bar{11}$	158	−134
19	3	$\bar{11}$	90	97
19	5	$\bar{11}$	88	−130
19	1	$\bar{12}$	39	−47
19	3	$\bar{12}$	112	−111
19	5	$\bar{12}$	35	34
19	1	$\bar{13}$	99	66
19	3	$\bar{13}$	99	−101
19	5	$\bar{13}$	66	82
19	1	$\bar{14}$	47−	19
19	3	$\bar{14}$	28−	6
19	1	$\bar{15}$	95	−92
19	3	$\bar{15}$	85	94
19	1	$\bar{16}$	64	−65
19	3	$\bar{16}$	36−	−22
19	1	$\bar{17}$	45−	24
19	3	$\bar{17}$	85	−88
19	1	$\bar{18}$	40	−47
20	0	0	150	159
20	2	0	125	−59
20	4	0	147	140
20	2	1	40−	−12
20	2	$\bar{1}$	48−	9
20	4	$\bar{1}$	127	−59
20	0	2	111	−104
20	0	$\bar{2}$	402	−399
20	2	2	37−	−24
20	2	$\bar{2}$	118	132
20	4	$\bar{2}$	180	−163
20	2	$\bar{3}$	42−	18
20	4	$\bar{3}$	178	148
20	0	$\bar{4}$	262	284
20	2	$\bar{4}$	242	−231
20	4	$\bar{4}$	90	79
20	2	$\bar{5}$	78	−77
20	4	$\bar{5}$	97	−128
20	0	$\bar{6}$	673	−675
20	2	$\bar{6}$	460	450
20	4	$\bar{5}$	150	−185
20	2	$\bar{7}$	316	−294
20	4	$\bar{7}$	105	140
20	0	$\bar{8}$	519	459
20	2	$\bar{8}$	162	−150
20	4	$\bar{8}$	375	378
20	2	$\bar{9}$	60	27
20	4	$\bar{9}$	97	58
20	0	$\bar{10}$	99	−73
20	2	$\bar{10}$	258	257
20	4	$\bar{10}$	265	−265
20	2	$\bar{11}$	74	−69
20	4	$\bar{11}$	51	64
20	0	$\bar{12}$	193	154
20	2	$\bar{12}$	248	−218
20	4	$\bar{12}$	235	200
20	2	$\bar{13}$	68	−58
20	4	$\bar{13}$	13−	7
20	0	$\bar{14}$	105	−52
20	2	$\bar{14}$	65	67
20	4	$\bar{14}$	118	−115
20	2	$\bar{15}$	35−	16
20	4	$\bar{15}$	58	−58
20	0	$\bar{16}$	112−	−32
20	2	$\bar{16}$	41−	−20
20	2	$\bar{17}$	45	51
20	0	$\bar{18}$	103	−102
21	1	0	51−	−60
21	3	0	40	−53
21	1	1	123	−130
21	1	$\bar{1}$	178	151
21	3	1	122	165
21	3	$\bar{1}$	153	−176
21	1	2	36−	39
21	1	$\bar{2}$	59−	32
21	3	$\bar{2}$	119	110
21	1	$\bar{3}$	156	−169
21	3	$\bar{3}$	90	−82
21	1	$\bar{4}$	50	−50
21	3	$\bar{4}$	24−	−28
21	1	$\bar{5}$	126	125
21	3	$\bar{5}$	47	−44
21	1	$\bar{6}$	313	−304
21	3	$\bar{6}$	151	143
21	5	$\bar{6}$	17−	−20
21	1	$\bar{7}$	54	47
21	3	$\bar{7}$	54	69
21	5	$\bar{7}$	33	−43
21	1	$\bar{8}$	74	−74
21	3	$\bar{8}$	76	−59
21	5	$\bar{8}$	104	−129
21	1	$\bar{9}$	65	−90
21	3	$\bar{9}$	82	−79
21	5	9	21−	8
21	1	$\bar{10}$	99	113
21	3	$\bar{10}$	180	180
21	5	$\bar{10}$	12−	20
21	1	$\bar{11}$	81	−63
21	3	$\bar{11}$	13−	−16
21	1	$\bar{12}$	59	−93
21	3	$\bar{12}$	111	122
21	1	$\bar{13}$	43	−29
21	3	$\bar{13}$	14−	32
21	1	$\bar{14}$	49	59
21	3	$\bar{14}$	34−	−21
21	1	$\bar{15}$	87	−100
21	3	$\bar{15}$	52	56
21	1	$\bar{16}$	49−	10
21	3	$\bar{16}$	44	−51
21	1	$\bar{17}$	128	148
21	3	$\bar{17}$	61	−80
21	1	$\bar{18}$	33−	31
22	0	0	128	112
22	2	0	56	−53
22	2	$\bar{1}$	38−	−34
22	0	2	82	−106
22	0	$\bar{2}$	148	−137
22	2	$\bar{2}$	76	73
22	4	$\bar{2}$	97	−119
22	2	$\bar{3}$	38−	23
22	4	$\bar{3}$	63	−61
22	0	$\bar{4}$	235	238
22	2	$\bar{4}$	107	−110
22	4	$\bar{4}$	257	246
22	2	$\bar{5}$	180	178
22	4	$\bar{5}$	126	−107
22	0	$\bar{6}$	406	−395
22	2	$\bar{6}$	250	235
22	4	$\bar{6}$	203	−186
22	2	$\bar{7}$	58	66
22	4	$\bar{7}$	108	74
22	0	$\bar{8}$	177	153
22	2	$\bar{8}$	185	−173
22	4	$\bar{8}$	80	83
22	2	$\bar{9}$	37−	−36
22	4	$\bar{9}$	146	−120
22	0	$\bar{10}$	573	−658
22	2	$\bar{10}$	140	144
22	4	$\bar{10}$	173	−159
22	2	$\bar{11}$	75	76
22	4	$\bar{11}$	15	16
22	0	$\bar{12}$	266	206
22	2	$\bar{12}$	85	−69
22	4	$\bar{12}$	216	201
22	2	$\bar{13}$	15−	−11
22	4	$\bar{13}$	23	−23
22	0	$\bar{14}$	102	−100
22	2	$\bar{14}$	147	148
22	4	$\bar{14}$	111	−107
22	2	$\bar{15}$	33−	−26
22	0	$\bar{16}$	86	91
22	2	$\bar{16}$	67	−66
22	2	$\bar{17}$	30−	−14
22	0	$\bar{18}$	71−	−17
23	1	0	53	−79
23	1	1	132	134
23	3	$\bar{1}$	41	−59
23	1	$\bar{2}$	49−	55
23	3	$\bar{2}$	132	92
23	1	$\bar{3}$	96	−100
23	3	$\bar{3}$	117	131
23	1	$\bar{4}$	31	−46
23	3	$\bar{4}$	106	−101
23	1	$\bar{5}$	155	180
23	3	$\bar{5}$	58	−87
23	1	$\bar{6}$	153	162
23	3	$\bar{6}$	81	83
23	1	$\bar{7}$	179	−174
23	3	$\bar{7}$	27−	32
23	1	$\bar{8}$	35−	4
23	3	$\bar{8}$	29−	−21
23	1	$\bar{9}$	389	395
23	3	$\bar{9}$	94	−95
23	1	$\bar{10}$	94	−80
23	3	$\bar{10}$	20−	6
23	1	$\bar{11}$	73	75
23	3	$\bar{11}$	147	150
23	1	$\bar{12}$	99	−108
23	3	$\bar{12}$	45	−41
23	1	$\bar{13}$	48	42
23	3	$\bar{13}$	194	−176
23	1	$\bar{14}$	42−	−17
23	3	$\bar{14}$	50	59
23	1	$\bar{15}$	69	−80
23	3	$\bar{15}$	94	102
23	1	$\bar{16}$	45−	−7
23	3	$\bar{16}$	32	28
23	1	$\bar{17}$	38−	50
24	0	0	106	134
24	0	$\bar{2}$	154	−152
24	2	$\bar{2}$	129	157
24	2	$\bar{3}$	29−	23
24	0	$\bar{4}$	227	224
24	2	$\bar{4}$	131	−146
24	2	$\bar{5}$	20−	11
24	0	$\bar{6}$	153	−139
24	2	$\bar{6}$	27−	25
24	4	$\bar{6}$	96	−105
24	2	$\bar{7}$	29−	−1
24	4	$\bar{7}$	54	−64
24	0	$\bar{8}$	165	164
24	2	$\bar{8}$	31−	−12
24	4	$\bar{8}$	135	129
24	2	$\bar{9}$	117	108
24	4	$\bar{9}$	22−	−2
24	0	$\bar{10}$	244	255
24	2	$\bar{10}$	30	32
24	4	$\bar{10}$	82	−77
24	2	$\bar{11}$	12−	27
24	4	$\bar{11}$	9−	−23
24	0	$\bar{12}$	30−	15
24	2	$\bar{12}$	45	−22
24	4	$\bar{12}$	47	48
24	2	$\bar{13}$	52	55
24	4	$\bar{13}$	56	−53
24	0	$\bar{14}$	184	−165
24	2	$\bar{14}$	60	−70
24	2	$\bar{15}$	46	68
24	0	$\bar{16}$	94−	65
24	2	$\bar{16}$	32−	−22
24	0	$\bar{18}$	103	−121
25	1	$\bar{2}$	31−	−32
25	1	$\bar{3}$	31	15
25	1	$\bar{4}$	36	−69
25	3	$\bar{4}$	10−	16
25	1	$\bar{5}$	45	55
25	3	$\bar{5}$	63	−74
25	1	$\bar{6}$	27−	40
25	3	$\bar{6}$	20−	−11
25	1	$\bar{7}$	160	−173
25	3	$\bar{7}$	170	208
25	1	$\bar{8}$	30−	−7
25	3	$\bar{8}$	176	−170
25	1	$\bar{9}$	164	161
25	3	$\bar{9}$	156	−163
25	1	$\bar{10}$	116	136
25	3	$\bar{10}$	17−	22
25	1	$\bar{11}$	248	−255
25	3	$\bar{11}$	81	84
25	1	$\bar{12}$	38	37
25	3	$\bar{12}$	22−	−11
25	1	$\bar{13}$	349	340
25	3	$\bar{13}$	117	−115
25	1	$\bar{14}$	86	89
25	3	$\bar{14}$	23−	−8
25	1	$\bar{15}$	126	−145
25	3	$\bar{15}$	120	142
25	1	$\bar{16}$	39−	−29
26	0	$\bar{4}$	194	218
26	2	$\bar{4}$	134	−171
26	2	$\bar{5}$	14−	−14
26	0	$\bar{6}$	99	−103
26	2	$\bar{6}$	58	63
26	2	$\bar{7}$	22−	2
26	0	$\bar{8}$	112	110
26	2	$\bar{8}$	25−	−17
26	2	$\bar{9}$	72	−75
26	0	$\bar{10}$	221	−216
26	2	$\bar{10}$	32	−58
26	2	$\bar{11}$	11−	−20
26	0	$\bar{12}$	160	154
26	2	$\bar{12}$	50	−45
26	2	$\bar{13}$	12−	−21
26	0	$\bar{14}$	250	214
26	2	$\bar{14}$	78−	22
26	2	$\bar{15}$	58	−54
26	0	$\bar{16}$	78−	73
27	1	$\bar{5}$	56	75
27	1	$\bar{6}$	19−	21
27	1	$\bar{7}$	105	−122
27	1	$\bar{8}$	24−	−25
27	3	$\bar{8}$	31	25
27	1	$\bar{9}$	60	61
27	3	$\bar{9}$	73	−77
27	1	$\bar{10}$	16−	3
27	3	$\bar{10}$	11−	6
27	1	$\bar{11}$	90	−109
27	3	$\bar{11}$	142	157
27	1	$\bar{12}$	34	−33
27	3	$\bar{12}$	56	−99
27	1	$\bar{13}$	12−	5
27	3	$\bar{13}$	61	−123
27	1	$\bar{14}$	47	−56
27	1	$\bar{15}$	136	−172
28	0	$\bar{6}$	48	65
28	0	$\bar{8}$	53	71
28	2	$\bar{9}$	19−	−2
28	0	$\bar{10}$	134	−145
28	2	$\bar{10}$	137	141
28	2	$\bar{11}$	69	−76
28	0	$\bar{12}$	124	132
28	2	$\bar{12}$	33	−41
28	0	$\bar{14}$	330	−358
29	1	$\bar{9}$	69	79
29	1	$\bar{10}$	12−	25
29	1	$\bar{11}$	43	−65
29	1	$\bar{12}$	30	−22
29	1	$\bar{13}$	103	111

Table 5. *Bond distances and angles for the* SO_4^{2-} *tetrahedron*

S–O(1)	1·49 ± 3Å	O(1)–S–O(2)	109·0 ± 2·1°
S–O(2)	1·43 ± 2	O(1)–S–O(3)	107·4 ± 2·3
S–O(3)	1·52 ± 3	O(1)–S–O(4)	116·6 ± 1·2
S–O(4)	1·46 ± 1	O(2)–S–O(3)	110·3 ± 1·4
		O(2)–S–O(4)	112·0 ± 0·7
		O(3)–S–O(4)	101·1 ± 0·6

tions (Cruickshank, 1949) are given in Tables 1 and 2 and the comparison between observed and calculated peak shapes is shown in Table 3. The F_c values reported in Table 4 are calculated with the final parameters of Tables 1 and 2 using the scattering factors of Dawson (1960) for S, of Berghuis, Haanappel, Potters, Loopstra, MacGillavry & Veenendaal (1955) for O, N and C and McWeeny (1951) for H. For the hydrogen atoms the isotropic *B* values used were derived from those of the attached atoms.

The standard deviations, quoted in the next section, are calculated from the formulae of Ahmed & Cruickshank (1953) for bond lengths and of Darlow (1960) for angles.

Discussion

Even if the centrosymmetrical model is more reliable for the reasons already given, it is interesting to compare the results of the two independent analyses. From the two projections on (010) shown in Fig. 1(*a*) and (*b*) it is possible to see the way in which the organic cations are packed together which is practically the same for both models; the only appreciable differences concern their interactions with the SO_4^{2-} groups.

Bond distances and angles in the organic cations quoted in Fig. 2 show that the differences are not significant, but the values in the centrosymmetrical model are more uniform and have a better correspondence with those reported in the literature for similar compounds. The benzene rings are strictly planar in both cases and the hydroxyl oxygen atom and the amine nitrogen atom lie in the benzene plane.

Fig. 3 shows the location of the hydrogen atoms of the organic cation for the centrosymmetrical model; the bond distances concerning the hydrogen atoms agree satisfactorily with the values generally found in X-ray determinations with the exception of the hydroxyl hydrogen atom, which is too near to the oxygen atom. It is interesting to observe that this hydrogen atom participates in a strong hydrogen bond [O(5)–H(1)···O(1′) = 2·46 Å].

The environment of the nitrogen atom is tetrahedral as expected; the angles around N and C(7) are as follows:

C(4)–N–C(7) = 113·2°	N(1)–C(7)–H(8) = 100·6°
C(4)–N–H(4) = 96·1	N(1)–C(7)–H(9) = 119·5
C(4)–N–H(7) = 115·7	N(1)–C(7)–H(10) = 112·3
C(7)–N–H(4) = 125·3	H(8)–C(7)–H(9) = 98·1
C(7)–N–H(7) = 115·2	H(8)–C(7)–H(10) = 115·6
H(4)–N–H(7) = 88·6	H(9)–C(7)–H(10) = 109·9

Bond distances and angles for the SO_4^{2-} tetrahedron in the centrosymmetrical model are quoted in Table 5; the e.s.d.'s are such that the differences in bond distances are not significant. Fig. 4 (*a*) and (*b*) shows two clinographic projections of the centrosymmetrical structure, drawn from two different points of view, showing the packing of the molecules and hydrogen bonding. The angles formed in the these bonds are as follows:

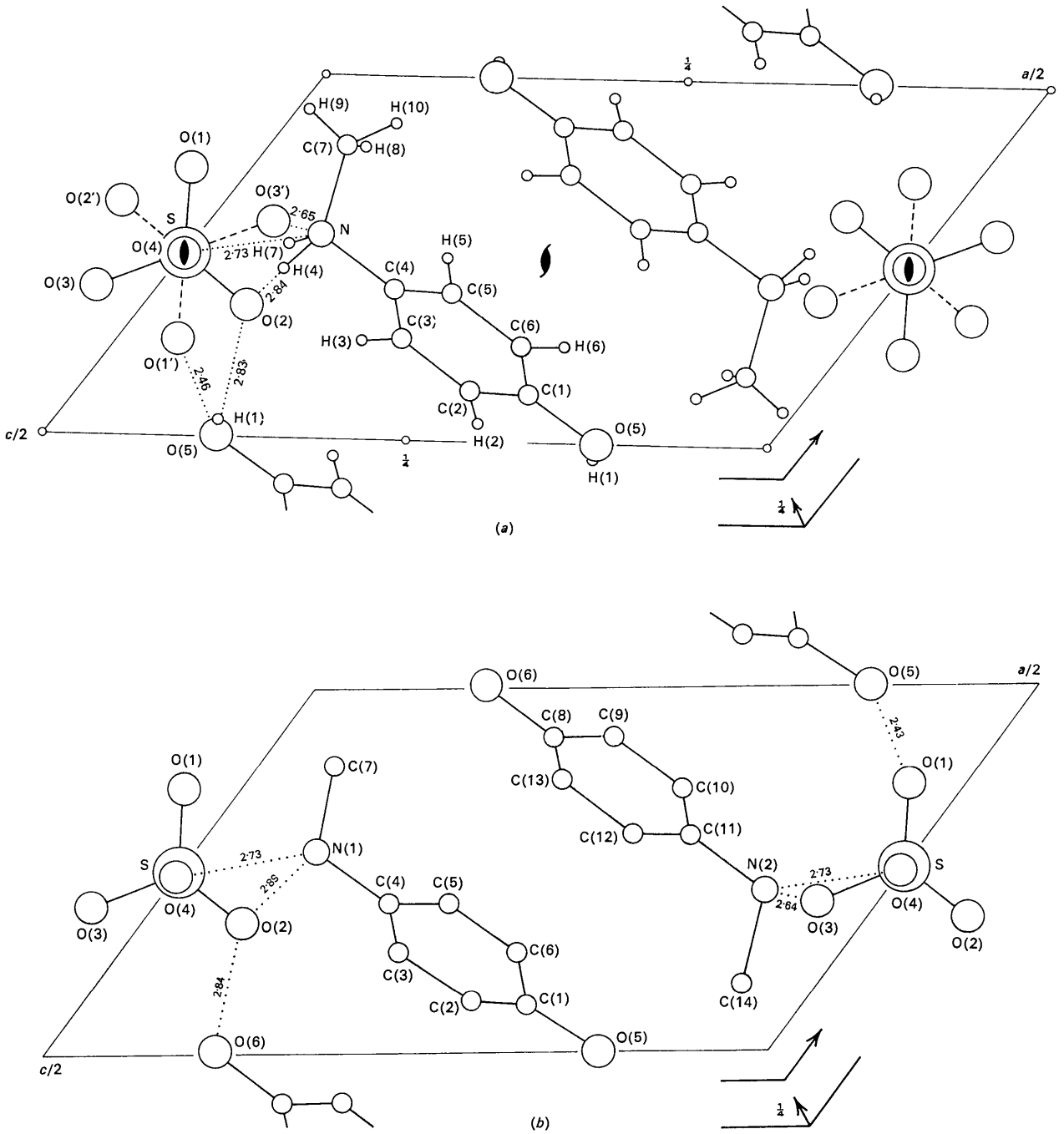

Fig. 1. (*a*) Projection of the centrosymmetrical model on (010). (*b*) Projection of the non-centrosymmetrical model on (010).

O(4)–H(4)–N = 142·1°
O(2″)–H(7)–N = 155·8 O(5)–H(1)–O(2″) = 146·1°
O(3′)–H(7)–N = 144·7 O(5)–H(1)–O(1′) = 143·6
′ $\bar{x}, y+1, \frac{1}{2}-z$
″ $x, y+1, z$

It appears that the H(1) and H(7) hydrogen atoms lie between two oxygen atoms of two disordered tetrahedra. All other packing distances are longer than 3·5 Å.

All the calculations were performed on the Olivetti Elea 6001/S computer of the Centro di Calcolo Elettronico della Università di Parma with the programs of Nardelli, Musatti, Domiano & Andreetti (1964, 1965).

This work was done with the financial support of the Consiglio Nazionale delle Ricerche (Roma).

References

ALBRECHT, G. (1939). *Rev. Sci. Instrum.* **10**, 221.
AHMED, F. R. & CRUICKSHANK, D. W. J. (1953). *Acta Cryst.* **6**, 385.
BERGHUIS, J., HAANAPPEL, IJ. M., POTTERS, M., LOOPSTRA, B. O., MACGILLAVRY, C. H. & VEENENDAAL, A. L. (1955). *Acta Cryst.* **8**, 478.
CRUICKSHANK, D. W. J. (1949). *Acta Cryst.* **2**, 65.
CRUICKSHANK, D. W. J. (1956). *Acta Cryst.* **9**, 754.
DARLOW, S. F. (1960). *Acta Cryst.* **13**, 683.

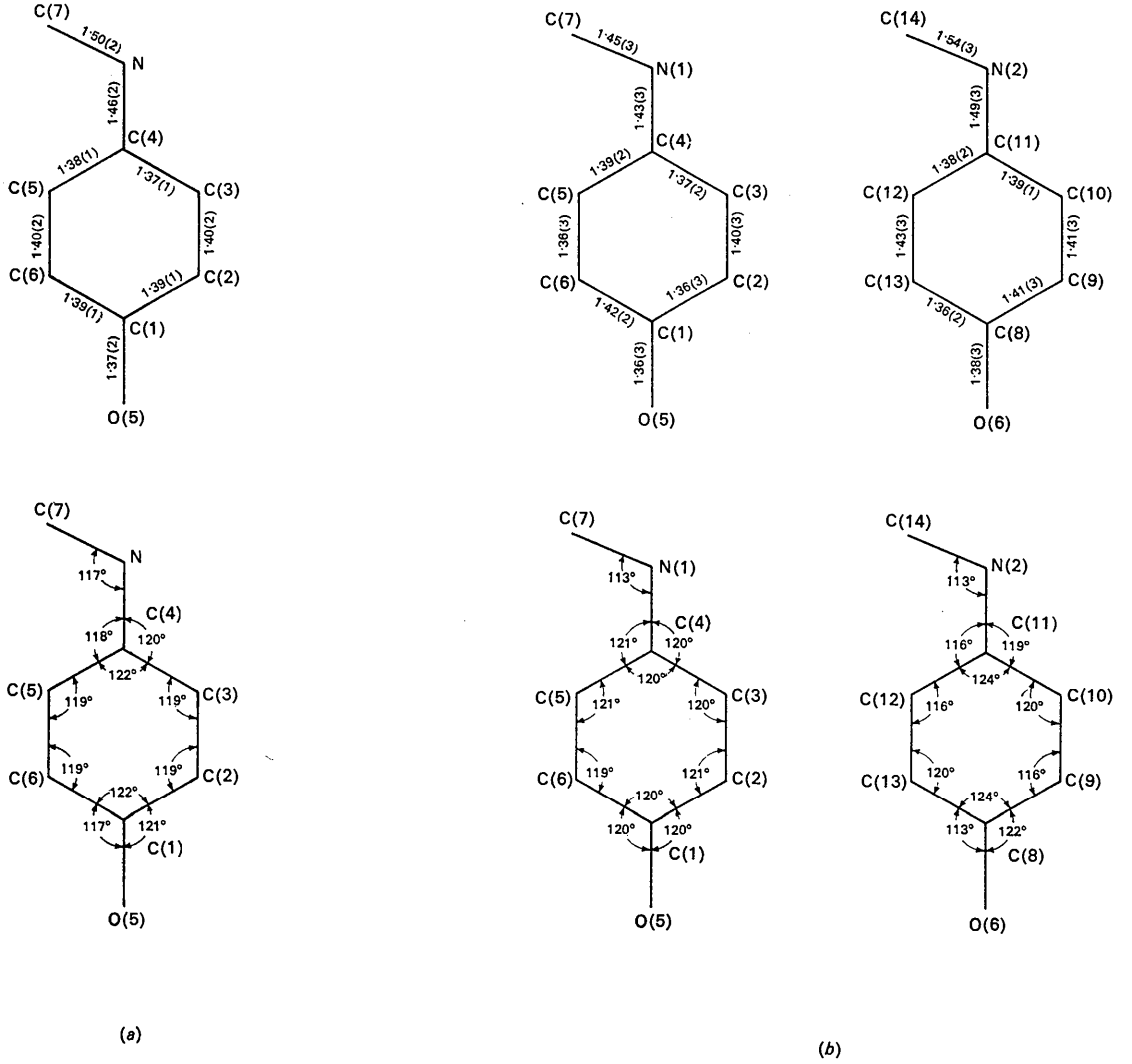

Fig. 2. Distances and angles in the organic cations for the two models of the structure: (*a*) centrosymmetrical, (*b*) non-centrosymmetrical.

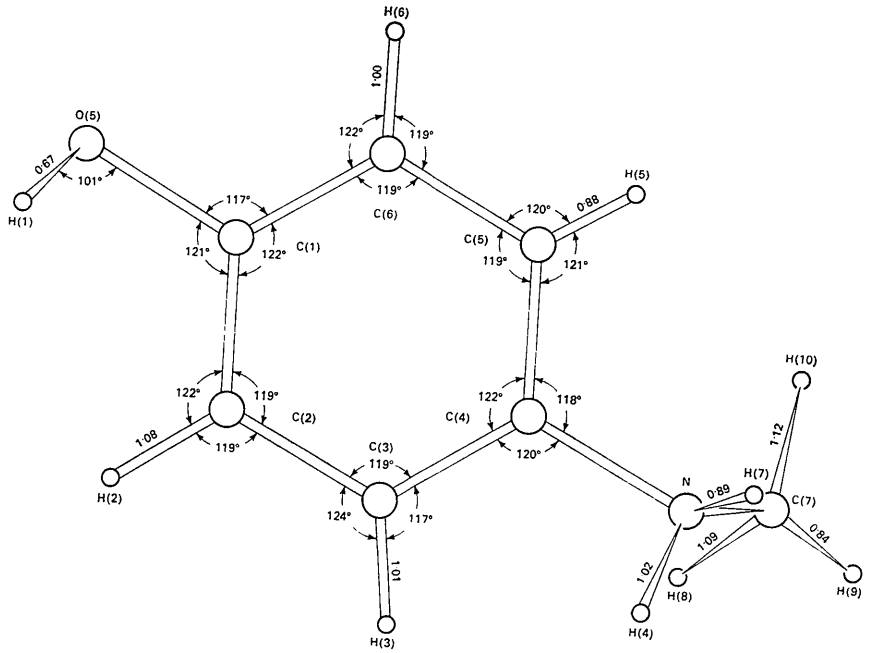

Fig. 3. The bond distances involving the hydrogen atoms of the organic cation for the centrosymmetrical model.

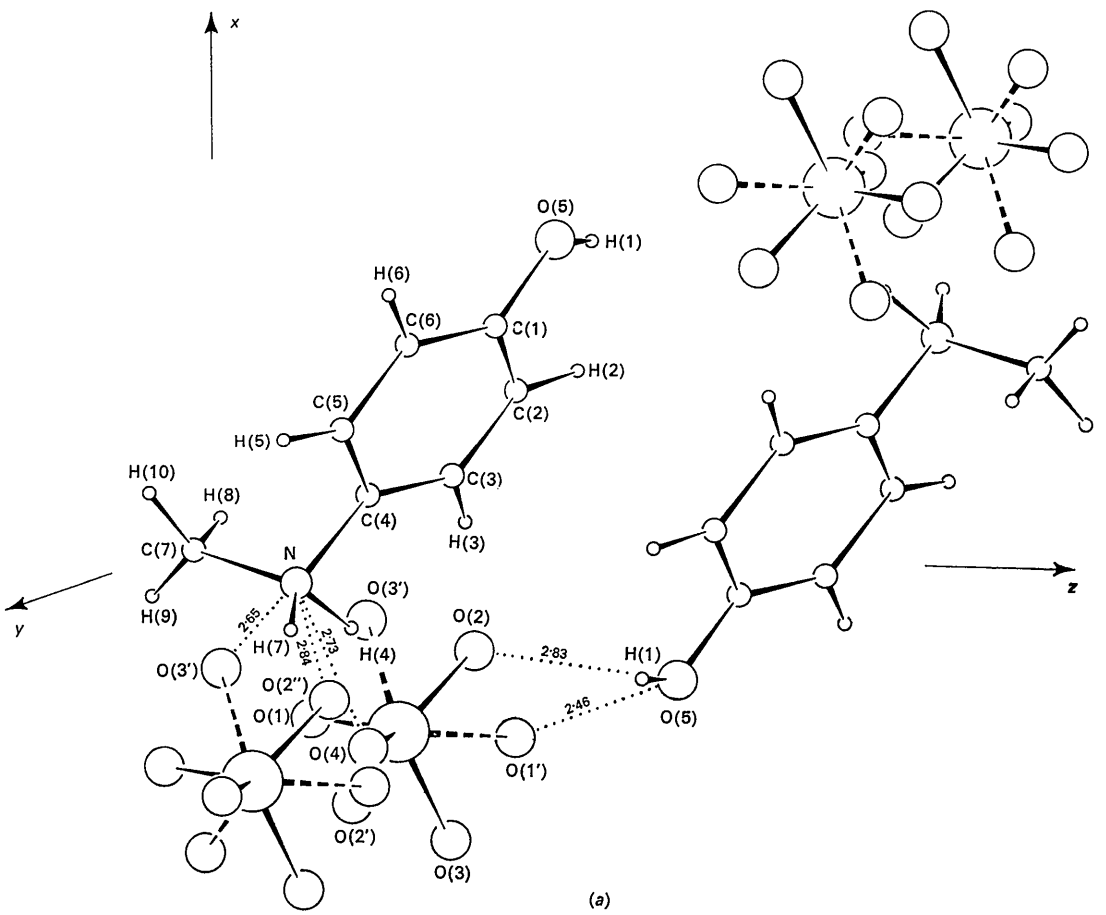

Fig. 4. Clinographic projections of the centrosymmetrical structure drawn from two different points of view.

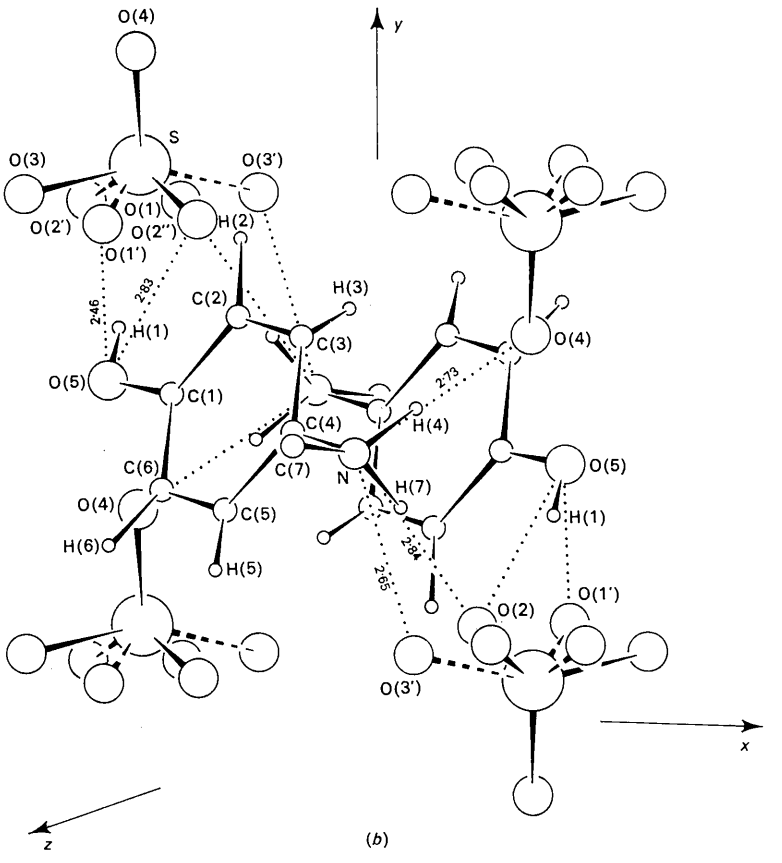

Fig. 4 (*cont.*)

DAWSON, B. (1960). *Acta Cryst.* **13**, 403.

HAMILTON, W. C. (1965). *Acta Cryst.* **18**, 502.

MCCRONE, W. C., COOK, J. W., WHITNEY, J. F., CORVIN, I. & TULL, M. (1948). *Anal. Chem.* **20**, 593.

MCWEENY, R. (1951). *Acta Cryst.* **4**, 513.

NARDELLI, M., MUSATTI, A., DOMIANO, P. & ANDREETTI, G. (1964). *Ric. Sci.* **34**, II-A,711.

NARDELLI, M., MUSATTI, A., DOMIANO, P. & ANDREETTI, G• (1965). *Ric. Sci.* **35**, II-A,469,477,807.

PHILLIPS, D. C. (1956). *Acta Cryst.* **9**, 819.

PHILLIPS, D. C., ROGERS, D. & WILSON, A. J. C. (1950). *Acta Cryst.* **3**, 398.

ROLLETT, J. S. & SPARKS, R. A. (1960). *Acta Cryst.* **13**, 273.

WILSON, A. J. C. (1950). *Acta Cryst.* **3**, 397.