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The Crystal and Molecular Structure of Tetrakis(biuret)strontium(II) Perchlorate

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Crystals of tetrakis(biuret)strontium(II) perchlorate, $[Sr((NH_2CO)_2NH)_4](ClO_4)_2$, are monoclinic with $a = 11.21(1)$, $b = 7.30(1)$, $c = 14.52(1)$ A, $\beta = 98.2(2)°$, $D_{obs} = 1.976$, $D_{calcd}(Z = 2) = 1.973$ g/cm^3, space group P2/c. 1526 independent reflections recorded photographically by the equi-inclination Weissenberg technique using CuKα, the intensities of which were estimated visually, were used in the structure determination. The structure was solved by Patterson and Fourier methods and refined anisotropically by diagonal least squares to an R factor of 0.094. The strontium atoms are located at special positions related by inversion. The perchlorate moieties are ionic although extensive hydrogen bonding exists between them and the primary and secondary amines of biuret. The two biuret ligands in the asymmetric unit are bonded to strontium as bidentates via the carbonyl oxygens with a small twist (10.9, 12.0°) about the oxygen–oxygen line. The six membered rings formed by the chelate ligands with strontium are folded (49.6, 1.8°) about the oxygen–oxygen line. This is attributed to packing considerations. The co-ordination polyhedron is best described as approximately D_2–222 distorted square antiprism with the bidentate ligands spanning opposite edges of the rectangular faces. To our knowledge this complex presents the first case of an eight co-ordinate non-transition metal complex, characterized by x-rays, where all of the ligands are non-ionic chemically identical oxygen donor bidentates.

Introduction

Biuret, $NH_2CONHCONH_2$, has been reported to complex with transition metals as a neutral bidentate via the oxygens, a dianionic bidentate via the nitrogens, or as a monodentate ligand via one of the two oxygens.[1–5] The x-ray structural determination of the α-form of biuret hydrate[6], potassium bis (biureto)cuprate(II) tetrahydrate[7], bis(biuret)copper (II) chloride[8], bis(biuret)zinc(II) chloride[9], and bis (biuret)cadium(II) chloride[10], have been carried out. Diacetamide, $CH_3CONHCOCH_3$, like biuret can behave as a neutral bidentate ligand forming complexes of high co-ordination number with the alkaline earth perchlorates[11]. The structure of one, pentakis(diacetamide)barium(II) perchlorate[12] shows ten co-ordinate barium(II) with the ligands bonded through the oxygens as bidentates. The perchlorate moieties were not co-ordinated although the infrared showed significant deviation towards C_{3v} symmetry[13].

On the other hand, the alkaline earth perchlorate complexes with strongly basic neutral nitrogen donor ligands such as diethylenetriamine suggest a possible co-ordination number of 9[14] and with weakly basic 1,10-phenanthroline and 2,2-bipyridine a co-ordination number of 8[15]. However with ligands of smaller bite such as 1,8-naphthyridine and 2,7-dimethyl-1,8-naphthyridine the co-ordination number of the alkaline earth perchlorates rarely exceeds 4[16] while with the lanthanide perchlorates it reaches 12[17].

We have recently undertaken the study of biuret complexes with the alkaline earths, the lanthanides and actinides which is a natural extension of the work on diacetamide with the alkaline earth. The infrared of most of the perchlorate complexes obtained show biuret to be the only ligand and to be bonded as a neutral bidentate via the oxygens[18]. However, significant variations in the CO stretching region and the NH_2 deformation region would indicate deviations in the biuret configuration from the α-form[2]. Systematic distortion of the perchlorate moiety from T_d symmetry with changes in cationic size were also observed. The crystal structure of $Sr(biuret)_4(ClO_4)_2$ was carried out in order to establish "standards" to be used in the interpretation of the infrared spectra and the study of the effect of cationic size and charge on the co-ordination number and the disposition of the neutral bidentate ligands.

Experimental

Preparation

Reagent grade hydrated strontium perchlorate and biuret in the molar ratio 1:6 were refluxed for six hours in minimum acetone then absolute ethanol was added to salt out the complex. Large crystals suitable for x-ray work developed as a second crop from the mother liquor upon leaving it to evaporate slowly at room temperature. The air stable crystals were collected and washed with dry acetone. M.p. 205–6 °C. *Anal.* Calcd. for $Sr(NH_2CONHCONH_2)_4(ClO_4)_2$: Sr, 12.53; C, 13.71; N, 24.04; H, 2.86. Found: Sr, 12.50; C, 13.79; N, 23.96; H, 2.83.

X-Ray

Data collection and reduction

Crystal data are listed in Table I. The unit cell translations were determined from rotation photographs about each axis by extrapolation to a Bragg angle of 90°. The error reported is the standard deviation of the intercept. The monoclinic angle, β, was determined from a Weissenberg photograph of the h0l zone. The error reported is the standard deviation of the mean. The density was measured by flotation. Oscillation and Weissenberg photographs showed systematic absences in the general set h0l when $l = 2n + 1$. In this light two space groups were possible *Pc*(No. 7), and *P*2/*c*(No. 13 if the heavy atoms are located at centers of inversion related by translation *c*/2. The solution and consequent refinement of the structure established unequivocally the space group as *P*2/*c*. No attempts were made to choose the space group on the basis of morphology, etch figures, piezoelectric or other effects, however, stoichiometry and infrared spectroscopy would indicate that such a proposed chelate would have higher symmetry than that present in the space group *Pc*.

The external morphology showed elongation along the *c*-axis. The single crystal used for the collection of intensity data was cut from a larger crystal to have the dimensions 0.06 × 0.09 × 0.2 mm^3 ($c \times a \times b$), and was mounted about the *b*-axis. Equiinclination Weissenberg levels, h0l–h6l were recorded on multiple films using nickel filtered copper radiation. The intensity of 1526 observable independent reflections were estimated visually and corrected for Lorentz, polarization and cylindrical absorption effects[19,20]. Interlayer scales were tentatively set at unity. Final values for the interlayer scales were obtained by comparing ΣF_o and ΣF_c within each level (h0l–h6l) after all of the atoms were located and the structure refined isotropically to R of 0.14[21]. The approximate absolute scale factor and the overall isotropic thermal parameter ($\bar{B} = 3.16$ A^2) were determined by a Wilson plot[22]. Scattering curves for hydrogen, carbon, nitrogen, oxygen and chlorine were obtained from self-consistent wave functions[23], while that for strontium from the Thomas–Fermi–Dirac statistical model[24]. Scattering factors for strontium and chlorine were corrected for real, $\Delta f'$, and immaginary, $\Delta f''$, dispersion[25].

TABLE I. Crystallographic Data for $[Sr(biuret)_4](ClO_4)_2$.

M. W. = 698.85		
Crystallographic system: monoclinic		
Space group: *P*2/*c*		
Unit cell parameters:	$a = 11.21(1)$ A[a]	
	$b = 7.30(1)$ A	
	$c = 14.52(1)$ A	
	$\beta = 98.2\ (2)°$	
	V = 1176.1 A^3	
$d_{calcd} = 1.973$ g/cm^3	Z = 2	$\mu_{CuK\alpha} = 63.3$ cm^{-1}
$d_{obs} = 1.976$ g/cm^3		$\lambda = 1.5418$ A
$F_{000} = 704$		

[a] Estimated standard deviations, shown in parentheses, are right-adjusted to the least significant digit in the preceeding number.

Solution and refinement of structure

The structure was determined by the heavy atom method. The space group was assumed to be *P*2/*c* rather than *Pc*. A three dimensional Patterson summation[26] reavealed a major peak at the Harker line 0, *v*, $^1/_2$ that corresponds to the fractional co-ordinates for the heavy atoms 0, 0.1517, $^1/_4$ and 0, –0.1517, $^3/_4$. A three dimensional Fourier summation[26,27] phased on the strontium atom revealed the approximate positions of the other nineteen non-hydrogen atoms in the asymmetric unit. Few other peaks were also observed. The R-factor at this point was 0.44. The inclusion in the Fourier summation of the nineteen non-hydrogen atoms followed by five cycles of diagonal matrix least squares refinement of the co-ordinates and the individual isotropic temperature factors reduced R to 0.116. Subsequent allowance for the anisotropic vibration of all of the atoms followed by five cycles of further least squares refinement converged to a final R value of 0.094. The observed and calculated structure amplitudes are reported in Table II. The maximum shift in the last cycle of refinement was less than $.03\sigma$ for the co-ordinates (Table III) and less than $.1\sigma$ for the anisotropic thermal parameters (Table IV). A difference electron density map run at this point revealed several residual peaks of intensity less than .4e/A^3 some of which corresponds to likely hydrogen positions, the peak height of an average carbon atom being 8e/A^3. No attempts however were made to deduce the hydrogen atom positions. The least squares refinement was carried out on F with the function minimized being $\Sigma w(|F_o|-|F_c|)^2$. The residual, $R = \Sigma||F_o|-|F_c||/\Sigma|F_o|$ and the weighted residual, $wR = (\Sigma w||F_o|-|F_c||^2/\Sigma w F_o{}^2)^{1/2}$. Empirical weighting schemes outlined in the structural factor least squares program[27] were tried but all gave worse values of wR than simply weighing all observations equally. All observations were therefore given unit weight. Reflections were not excluded from refinement because of extinction effects. The unobserved planes were not included in the calculation of the residual. Interatomic distances (Table V) and bond angles (Table VI) were

TABLE II. Observed and Calculated Structure Amplitudes × 10 (F_{000} = 704).

L	FO	FC
H= 0, K= 0		
2	558	-643
4	882	-989
6	604	465
8	293	322
10	582	-530
12	401	417
14	412	-411
18	61	-65
H= 1, K= 0		
0	436	479
2	593	-677
4	608	-517
6	543	-534
8	237	244
10	1140	-1120
12	237	238
14	112	55
16	133	117
18	231	-234
H= 1, K= 0		
-2	406	-415
-4	1077	2334
-6	523	-496
-8	157	159
-10	356	-372
-12	405	368
-16	198	197
-18	238	-245
H= 2, K= 0		
0	583	633
2	311	-292
4	813	-769
6	1140	-1218
8	857	743
10	150	-159
12	451	484
14	157	-141
H= 2, K= 0		
-2	608	-727
-4	375	444
-6	879	-870
-8	737	683
-10	547	-512
-14	295	-294
-16	237	222
-18	94	-113
H= 3, K= 0		
0	277	278
2	1602	-1804
4	899	981
8	415	464
10	303	-270
12	163	186
14	186	-191
16	247	227
H= 3, K= 0		
-2	669	619
-4	765	723
-6	366	-323
-8	375	397
-10	461	-417
-12	523	570
-14	352	-345
-18	155	-171
H= 4, K= 0		
0	92	71
2	673	-592
4	700	697
6	708	-721
8	145	128
10	160	-169
12	390	374
14	204	-217
H= 4, K= 0		
-2	725	-804
-4	792	909
-8	439	446
-10	434	-425
-12	428	403
-16	284	275
-18	199	-193
H= 5, K= 0		
0	907	907
2	733	-677
6	138	-132
8	583	595
10	167	-170
12	112	133
14	244	-223
16	211	219
H= 5, K= 0		
-2	952	-954
-4	134	127
-6	459	-461
-8	303	279
-10	371	-272
-12	374	362
-14	288	-263
-16	160	151
H= 6, K= 0		
2	656	-587
4	888	829
6	191	-183
10	211	-231
12	184	217
14	166	-140
H= 6, K= 0		
-2	275	-300
-4	882	857
-6	352	-319
-10	710	-696
-12	282	249
-16	240	219
H= 7, K= 0		
0	802	718
2	311	-288
6	318	-298
8	330	290
10	158	-139
12	194	203
14	287	-267
H= 7, K= 0		
-2	509	-529
-6	348	-355
-8	808	823
-10	297	-266
-12	227	-182
-14	363	-331
-16	278	296
H= 8, K= 0		
2	735	-705
4	583	498
8	316	315
10	410	-382
12	118	129
H= 8, K= 0		
-2	207	198
-4	620	628
-6	571	-526
-8	478	502
-10	288	-257
-14	95	-97
-16	67	91
H= 9, K= 0		
0	450	432
2	353	-379
4	622	555
6	317	-303
10	92	-111
12	178	176
H= 9, K= 0		
-2	328	-296
-4	331	-351
-8	921	974
-10	366	-370
-12	103	135
-16	125	130
H= 10, K= 0		
0	392	375
2	582	-566
8	165	145
10	228	-267
H= 10, K= 0		
-2	340	-338
-4	115	135
-6	340	-356
-12	485	416
-14	256	-250
H= 11, K= 0		
0	195	187
4	309	344
6	163	-165
H= 11, K= 0		
-2	573	-542
-4	252	272
-6	221	-224
-10	189	-190
-12	155	173
H= 12, K= 0		
0	334	328
6	192	-205
H= 12, K= 0		
-6	345	-351
-8	311	297
-10	135	-144
-12	107	142
H= 13, K= 0		
2	178	-166
4	172	139
H= 13, K= 0		
-2	174	-139
-4	224	238
-6	142	-197
-10	222	-232
H= 14, K= 0		
0	244	253
H= 14, K= 0		
-2	182	-236
H= 0, K= 1		
0	103	39
1	257	-271
2	70	-88
3	929	1136
4	543	476
6	355	-343
8	761	245
9	611	-546
10	472	-455
11	590	549
12	767	213
17	248	-231
18	120	-110
H= 1, K= 1		
0	650	1045
1	323	-391
2	356	-310
3	511	-495
4	222	169
5	231	-185
6	719	-661
7	921	930
8	455	406
9	557	-550
11	109	86
13	304	-291
14	111	-139
H= 1, K= 1		
-1	985	1283
-2	92	82
-3	593	-631
-4	56	-35
-5	294	331
-7	736	-673
-8	441	435
-9	187	173
-10	381	-298
-13	305	248
-14	276	-246
-15	452	-435
-17	142	164
H= 2, K= 1		
0	143	149
1	362	-338
2	1525	-1628
3	1803	1817
4	508	524
5	613	-687
9	463	-446
10	211	-201
11	187	132
12	191	133
14	189	-186
15	173	169
16	123	136
17	218	-243
H= 2, K= 1		
-1	556	-687
-2	299	331
-3	554	-636
-4	472	506
-5	447	464
-6	282	-259
-7	540	-502
-8	423	348
-9	211	159
-10	101	-36
-11	347	-326
-12	289	247
-13	315	289
-14	113	-132
-15	107	-144
-18	145	-158
H= 3, K= 1		
0	303	287
1	575	461
2	633	-603
3	682	595
5	750	-679
7	444	401
11	196	208
12	369	382
13	299	-293
14	187	-184
15	94	156
H= 3, K= 1		
-1	1069	1136
-2	1029	-1042
-3	736	-755
-5	587	529
-6	171	-169
-7	453	-464
-8	474	431
-9	147	150
-10	145	-163
-11	109	-79
-13	198	196
-15	364	-337
-16	166	163
-17	142	126
-19	123	-127
H= 4, K= 1		
0	551	557
1	94	-86
2	750	-754
3	306	285
4	469	382
5	341	-325
6	407	339
8	339	327
9	533	-456
10	233	-239
11	161	121
12	190	163
13	153	-167
14	100	-115
15	201	222
16	70	59
H= 4, K= 1		
-1	790	875
-2	120	97
-3	576	-522
-4	369	-341
-5	795	846
-6	592	-571
-7	308	-315
-8	555	504
-10	231	-229
-12	310	243
-13	512	527
-15	159	-153
-19	115	-129
H= 5, K= 1		
0	508	452
1	109	112
3	83	-74
4	207	165
5	1072	-1009
7	307	255
8	152	161
9	158	-169
10	360	-376
11	331	277
12	110	136
H= 5, K= 1		
-2	748	-738
-3	825	-769
-4	300	236
-5	428	419
-7	414	-315
-9	277	266
-10	406	-395
-11	522	-511
-12	315	281
-13	114	138
-16	200	173
-17	231	206
-18	96	-127
H= 6, K= 1		
0	616	538
1	189	-189
2	185	-191
5	428	-433
6	313	-259
7	706	729
8	270	280
H= 6, K= 1		
-1	553	651
-2	218	-216
-4	219	-184
-5	235	208
-6	359	-372
-7	347	-570
-8	215	206
-9	108	113
-10	414	-374
-11	254	-197
-13	273	245
-15	311	-303
H= 7, K= 1		
0	253	200
1	611	-539
2	315	-241
3	565	539
4	486	435
8	114	116
9	196	-188
10	110	-158
11	202	206
13	82	-117
14	65	-19
H= 7, K= 1		
-1	405	358
-3	186	-211
-4	432	400
-5	616	573
-6	102	-159
-7	193	195
-9	158	140
-10	414	-415
-11	424	-371
-13	211	209
-14	100	-98
-15	90	-109
-17	213	209
H= 8, K= 1		
0	511	496
1	271	-252
2	151	-165
3	109	-118
5	382	-408
6	161	-189
7	342	356
13	191	-188
H= 8, K= 1		
-1	364	375
-2	294	-261
-3	268	-247
-4	149	161
-5	235	220
-6	109	127
-7	589	-573
-8	387	303
-9	415	368
-10	161	-163
-14	244	-223
-15	282	-241
-16	110	126
H= 9, K= 1		
0	111	87
1	513	-500
2	426	-373
3	444	400
9	238	-243
10	219	-190
11	110	130
12	61	89
H= 9, K= 1		
-2	110	-105
-3	169	-139
-4	298	272
-5	485	442
-6	226	-234
-7	322	-269
-8	198	177
-11	363	-339
-13	207	199
-14	81	-128
H= 10, K= 1		
5	282	-244
6	182	-202
8	93	97
10	72	-105
11	105	139
H= 10, K= 1		
-1	415	444
-2	378	-354
-3	315	-281
-4	287	248
-6	114	-152
-8	157	171
-9	279	246
-11	309	-247
-13	160	154
-15	155	-190
H= 11, K= 1		
1	229	-240
2	198	-190
4	145	181
6	191	-197
7	247	253
9	173	-208
H= 11, K= 1		
-3	158	-156
-5	416	425
-6	367	-316
-8	214	226
-12	106	101
-13	209	219
H= 12, K= 1		
1	98	-92
2	165	-191
3	158	187
4	223	214
5	160	-158
H= 12, K= 1		
-1	101	104
-3	330	-331
-5	99	130
-9	192	188
-10	169	-148
-11	203	-227
-12	70	84
H= 13, K= 1		
0	185	203
1	178	-180
H= 13, K= 1		
-1	169	177
-3	156	-175
-4	119	-9
-6	79	-118
-7	105	-127
-8	125	162
-9	186	195
-10	45	-95
H= 14, K= 1		
0	90	85
H= 14, K= 1		
-3	200	-225
-4	81	98
-5	118	147
H= 0, K= 2		
0	898	-950
1	815	-980
2	107	-69
3	86	-50
4	83	63
5	784	-737
6	428	416
7	628	609
8	349	-341
9	395	-396
10	181	-176
13	279	-246
15	262	264
17	149	-154
H= 1, K= 2		
0	287	276
1	527	-456
2	704	699
3	685	615
4	421	-421
5	711	-676
7	267	236
9	358	-360
10	260	235
11	415	381
12	114	-17
15	187	167
17	346	-305
H= 1, K= 2		
-1	357	-344
-2	112	-107
-3	1447	-1423
-4	286	-222
-5	965	1002
-6	330	305
-7	282	-235
-8	252	-212
-10	180	180
-11	379	-430
-12	196	-146
-13	198	182
-14	111	-93
-15	265	-250
-17	172	196
H= 2, K= 2		
0	310	-327
1	147	-134
2	401	402
3	751	768
4	149	-138
5	732	-667
6	198	187
7	591	539
9	145	183
11	196	203
12	257	-224
13	421	-435
14	185	185
15	255	240
16	116	-9
H= 2, K= 2		
-1	406	479
-2	181	-213
-3	208	-250
-4	262	320
-5	635	636
-6	715	739
-7	684	-751
-8	180	-225
-9	430	499
-10	105	144
-15	454	-518
-16	132	-123
-17	110	119
H= 3, K= 2		
0	493	-454
1	198	-173
2	395	346
3	654	680
4	328	-268
5	549	-478
6	337	-314
7	264	246
9	629	-593
10	223	233
11	229	213
13	110	-151
15	181	180
H= 3, K= 2		
-1	237	-182
-3	284	-318
-4	364	-355
-5	1130	1233
-7	551	-431
-8	278	248
-9	314	309
-10	211	191
-12	228	-232
-13	403	406
-14	193	235
-15	207	-212
H= 4, K= 2		
0	136	-108
1	523	-498
3	527	537
4	259	-245
5	963	-947
6	415	335
7	200	160
11	404	377
13	184	-197
H= 4, K= 2		
-1	346	348
-2	161	-156
-3	526	-532
-4	554	-521
-5	112	-99
-7	300	-261
-8	97	-105
-9	655	615
-10	187	157
-11	335	-293
-13	279	229
-15	227	-199
-17	172	183
-18	90	104
H= 5, K= 2		
1	819	-805
5	135	-123
6	340	288
7	589	551
8	233	-216
9	253	-224
15	166	196
H= 5, K= 2		
-1	880	944
-2	350	344
-3	448	-438
-4	463	-439
-5	184	201
-7	318	-312
-8	174	-159
-11	197	-198
-13	464	447
-14	107	109
-15	196	-220
H= 6, K= 2		
2	617	512
4	141	-111
5	371	-346
7	375	291
9	213	-208
11	351	275
H= 6, K= 2		
-1	381	350
-3	950	-1035
-5	686	643
-6	220	-169
-8	238	-176
-9	220	208
-10	399	386
-11	323	-319
-12	197	-181
-13	246	188
-14	145	-132
-17	215	205
H= 7, K= 2		
0	182	-156
1	368	-332
5	191	-183
6	160	158
7	638	630
8	162	-204
13	209	-207
H= 7, K= 2		
-1	543	490
-2	452	415
-3	347	-329
-4	100	119
-5	103	108
-7	585	-512
-8	313	-314
-9	349	350
-11	114	-136
-13	324	289
-14	137	108
-15	233	-225
H= 8, K= 2		
1	201	-137
2	231	195
3	639	534
6	114	-148
7	300	294
8	157	-172
9	281	-305
10	140	147
H= 8, K= 2		
-2	150	134
-3	397	-371
-4	273	-238
-5	477	480
-7	160	-192
-11	302	-325
-12	212	-217
-13	317	330
-15	106	-110
H= 9, K= 2		
1	160	-158
3	162	150
4	242	-222
5	310	-321
6	158	164
7	187	157
H= 9, K= 2		
-1	490	520
-2	112	158
-3	372	-363
-7	199	-239
-8	213	-231
-9	226	184
-11	181	-148
-13	176	180
-15	213	-230
H= 10, K= 2		
1	241	-226
3	159	146
7	255	220
9	276	-289
H= 10, K= 2		
-1	229	223
-3	242	-265
-4	198	-203
-5	440	455
-6	227	271
-7	224	-216
-11	200	-274
-13	278	279
-14	149	114
H= 11, K= 2		
3	252	220
4	242	-203
5	326	-347
H= 11, K= 2		
-3	300	-282
-5	305	361
-7	104	-88
-9	164	192
-11	217	-206
H= 12, K= 2		
0	97	-89
1	347	-320
H= 12, K= 2		
-1	98	179
-5	166	188
-7	200	-173
-8	119	-129
-9	156	157
-11	99	-98
H= 13, K= 2		
1	105	-116
3	182	214
H= 13, K= 2		
-3	227	-245
-5	154	162
-9	154	165
H= 14, K= 2		
-1	182	251
-2	82	94
-3	83	-103
H= 0, K= 3		
0	693	-689
1	704	-645
2	936	895
3	154	147
4	165	-197
5	235	272
6	138	-114
8	251	-276
10	669	634
11	210	224
12	451	-563
H= 1, K= 3		
0	897	-827
1	723	583
2	388	356
3	100	91
4	286	-275
6	710	756
7	289	321
8	862	-723
9	138	-171
12	281	-281
14	257	252
H= 1, K= 3		
-1	344	309
-2	307	287
-3	724	-619
-4	125	128
-5	310	323
-6	589	623
-7	115	171
-8	622	-635
-10	101	140
-11	149	-157
-12	151	-153
-14	316	311
-16	268	-275
H= 2, K= 3		
0	409	-389
1	373	266
2	1425	1156
3	255	-317
4	423	-377
5	248	-260
6	81	-127
7	426	447
8	418	-428
9	211	-243
10	464	515
11	107	-116
12	106	-128
14	94	127
16	96	-86
H= 2, K= 3		
-1	272	274
-2	580	466
-3	334	-365
-4	757	-685
-5	444	372
-6	521	513
-10	175	168
-12	398	-411
-14	100	137
-16	112	-122
H= 3, K= 3		
0	460	-469
1	112	130
2	482	405
3	226	220
4	590	-618
5	337	-381
6	291	306
8	138	-150
12	346	-362
14	260	287
15	109	126
H= 3, K= 3		
-1	172	133
-2	863	786
-3	417	-351
-4	135	-127
-6	523	469
-8	516	-462
-10	477	469
-15	129	-125
-16	207	-186
H= 4, K= 3		
0	543	-541
1	70	-102
2	284	315
4	539	-493
5	184	-191
7	98	105
8	556	-544
10	238	268
14	185	225
15	118	125
H= 4, K= 3		
-4	577	-531
-5	313	298
-6	722	704
-7	253	-202
-8	313	-333
-10	264	206
-12	360	-394
-14	248	227
H= 5, K= 3		
0	514	-420
2	347	299
3	121	-132
4	488	-434
5	94	-131
6	219	256
9	107	-132
10	381	419
11	177	155
12	191	-229
14	127	168
H= 5, K= 3		
-1	262	250
-2	640	581
-3	275	-253
-4	520	-469
-5	171	198
-6	156	-123
-8	261	-237
-10	648	567
-12	471	-397
-13	143	162
-15	85	-84
-16	224	-220
H= 6, K= 3		
0	589	-500
2	462	395
3	94	106
5	274	-260
6	525	451
7	224	210
8	368	-329
10	103	122
12	88	-98
14	167	207
H= 6, K= 3		
-1	304	-265
-2	388	339
-3	152	-159
-5	359	366
-6	543	491
-7	100	-81
-8	363	-364
-9	105	-130
-10	185	248
-12	103	-140
-13	170	180
-14	191	236
-15	79	-104
-16	205	-212
H= 7, K= 3		
0	270	-273
2	591	459
3	188	177
4	364	-403
5	149	-151
8	210	-229
10	324	356
12	108	-175
H= 7, K= 3		
-2	191	239
-3	180	-177
-4	552	-514
-5	142	166
-6	176	152
-7	180	-212
-10	437	445
-12	173	-197
-16	193	-158
H= 8, K= 3		
0	397	-409
2	105	112
3	150	173
4	238	-240
6	326	365
12	85	-99
H= 8, K= 3		
-2	339	358
-3	145	-135
-4	147	-153
-6	184	212
-8	539	-567
-10	103	130
-14	166	169
H= 9, K= 3		
0	151	-149
2	582	545
4	211	-246
10	193	225
H= 9, K= 3		
-1	195	208
-4	213	-218
-6	397	418
-8	181	-216
-9	143	193
-10	169	213
-11	130	-158
-12	272	-220
-14	105	109
H= 10, K= 3		
0	182	-136
4	269	-303
6	201	207
9	109	-115
H= 10, K= 3		
-1	196	203
-2	364	374
-4	149	-148
-6	103	130
-8	258	-254
-10	297	307
H= 11, K= 3		
0	275	-299
2	198	216
6	127	181
8	139	-139
H= 11, K= 3		
-1	99	159
-4	138	-140
-6	209	253
-8	150	-151
-12	167	-189
H= 12, K= 3		
2	135	159
4	184	-205
6	107	133
H= 12, K= 3		
-2	171	189
-4	222	-258
-8	99	-103
-10	166	180
H= 13, K= 3		
0	205	-209
1	58	-22
H= 13, K= 3		
-2	131	160
-4	90	-96
-6	133	174
H= 0, K= 4		
0	940	-950
1	836	857
2	326	352
3	165	169
4	191	-157
5	162	167
6	282	277
7	186	-225
8	212	-229
9	375	366
10	302	269
12	207	-201
15	170	-211
16	94	-103
H= 1, K= 4		
0	549	-615
1	403	509
2	291	267
3	176	-198
4	207	174
5	112	106
6	284	283
7	252	224
8	279	-274
9	416	425
10	324	351
11	388	-434
12	234	-255
16	87	-119
H= 1, K= 4		
-2	531	508
-3	773	769
-4	577	-614
-5	483	-457
-6	247	267
-9	149	-137
-10	381	370
-11	158	172
-12	343	-310
-15	86	96
H= 2, K= 4		
0	284	-315
1	302	313
2	356	361
4	408	-362
5	331	277
6	390	425
7	386	-386
8	277	-264
11	237	-262
12	230	-264
14	156	180
15	107	-104
H= 2, K= 4		
-1	308	-392
-2	248	276
-3	112	120
-4	101	-88
-6	122	114
-7	397	413
-8	426	-431
-9	562	-507
-10	583	552
-12	111	-113
-14	139	146
-15	174	175
-16	122	-99
H= 3, K= 4		
0	516	-575
1	189	165
2	486	450
3	401	-352
6	137	107
7	263	-247
8	458	-428
9	315	320
10	224	245
11	234	-212
14	157	163
H= 3, K= 4		
-2	188	163
-3	309	340
-4	468	-414
-5	518	-491
-6	207	179
-7	96	137
-10	192	212
-11	158	142
-12	304	-282
-13	351	-377
-14	258	239
H= 4, K= 4		
0	350	-364
1	113	-113
2	472	457
3	151	-148
4	321	-324
5	536	549
6	145	126
7	252	-264
10	260	266
11	185	-170
12	265	-244
13	126	164
14	129	141
H= 4, K= 4		
-1	324	-321
-2	500	477
-3	611	704
-4	171	-157
-5	421	-418
-9	154	-102
-10	250	275
-11	158	189
-13	180	-181
-14	94	63
-15	116	157
-16	205	-192
H= 5, K= 4		
0	397	-421
1	256	254
2	373	334
3	256	-240
5	267	251
6	216	177
7	376	-344
9	388	-393
10	187	202
11	102	80
12	92	-81
H= 5, K= 4		
-1	176	-174
-2	178	159
-4	257	-226
-5	308	-259
-6	453	466
-7	494	493
-8	243	-233
-10	158	147
-11	156	24
-12	185	-147
-13	200	-166
-14	156	131
-15	154	136
H= 6, K= 4		
0	139	-86
1	100	79
2	322	310
3	363	-323
4	430	-441
5	219	218
6	193	172
10	144	162
11	161	-181
12	116	-127
13	113	152
H= 6, K= 4		
-1	195	-160
-2	138	115
-3	372	352
-4	418	-359
-5	275	-297
-6	185	169
-8	315	-308
-9	158	-161
-10	316	307
-11	243	261
-12	103	-130
-16	98	-96
H= 7, K= 4		
0	291	-248
1	364	342
2	462	460
4	157	-138
6	158	161
7	220	-227
12	114	-152
H= 7, K= 4		
-1	520	-530
-2	106	121
-4	152	-156
-6	207	197
-7	317	302
-8	327	-320
-9	306	-296
-10	153	137
-12	138	-115
-13	88	-105
-14	151	157
-15	140	164
H= 8, K= 4		
1	193	164
2	317	325
3	263	-251
4	224	-234
5	110	100
9	200	231
10	136	168
11	89	-99
H= 8, K= 4		
-1	156	205
-2	192	224
-3	111	103
-4	158	-161
-5	405	-449
-6	210	207
-7	158	167
-8	191	-233
-10	179	181
-11	97	91
-12	126	-140
-13	135	-128
-14	88	104
H= 9, K= 4		
0	223	-188
1	249	209
2	156	163
3	109	-87
4	262	-279
5	243	247
7	92	-132
10	79	105
H= 9, K= 4		
-1	158	-147
-2	193	215
-7	241	244
-8	297	-301
-9	275	-235
-10	251	216
H= 10, K= 4		
0	319	-301
1	364	338
2	145	123
3	310	-276
4	96	-79
8	109	-117
H= 10, K= 4		
-2	214	242
-5	147	-143
-6	177	166
-7	172	144
-8	134	-115
-11	161	157
-12	188	-171
H= 11, K= 4		
3	120	-114
4	158	-203
5	72	77
6	107	118
H= 11, K= 4		
-1	203	-202
-2	136	128
-3	262	259
-4	189	-157
-6	190	196
-8	138	-154
-9	103	-104
-10	165	177
-11	137	141
H= 12, K= 4		
0	222	-220
1	126	113
2	68	72
H= 12, K= 4		
-1	199	-161
-3	78	88
-6	186	198
-7	90	118
-8	156	-182
H= 13, K= 4		
-3	101	144
-4	87	-124
H= 0, K= 5		
0	129	-112
1	133	114
3	654	-585
4	167	168
5	669	549
6	72	74
7	162	-200
9	244	259
11	325	-320
13	151	156
15	127	-148
H= 1, K= 5		
0	196	189
1	227	216
3	299	-311
4	135	-121
5	638	601
6	154	-119
7	511	-527
8	101	106
9	211	213
13	193	234
15	169	-204
H= 1, K= 5		
-1	380	-334
-2	70	97
-3	310	334
-4	275	-248
-5	247	-258
-6	149	-136
-7	429	415
-8	264	266
-9	420	-462
-10	106	107
-13	200	-189
-15	205	253
H= 2, K= 5		
0	179	-190
1	679	604
2	296	-221
3	748	-733
4	159	147
5	274	331
6	54	36
7	133	-128
9	252	273
11	216	-203
13	68	98
15	151	-196
H= 2, K= 5		
-2	130	105
-3	382	374
-5	409	-379
-6	194	-195
-7	295	323
-8	116	-102
-9	167	-195
-11	205	228
-12	115	124
-13	308	-311
-15	54	81
H= 3, K= 5		
0	255	251
1	59	66
2	93	74
3	365	-330
5	424	388
7	230	-271
10	103	110
11	205	-229
12	52	86
13	195	216
14	50	-67
H= 3, K= 5		
-1	813	-754
-2	279	-265
-3	778	754
-6	142	147
-7	326	294
-8	315	-312
-9	323	-291
-11	133	140
-12	114	105
-13	176	-175
-14	80	-126
-15	213	233
H= 4, K= 5		
0	151	-159
1	330	345
2	218	226
3	283	-318
4	55	-78
5	81	72
6	59	-79
7	260	-247
8	172	161
9	346	371
11	125	-130
13	97	119
H= 4, K= 5		
-1	383	-388
-3	114	111
-4	527	532
-5	518	-521
-6	184	-154
-7	576	620
-8	217	-188
-10	219	214
-11	118	134
-13	273	-269
-14	76	-106
-15	90	121
H= 5, K= 5		
0	446	-504
1	53	-93
2	206	204
3	220	-196
4	162	128
5	503	503
6	251	-258
7	122	-139
8	60	68
9	190	222
11	284	-278
13	110	126
H= 5, K= 5		
-1	386	-404
-3	479	451
-4	450	409
-5	244	-266
-6	83	-114
-7	264	282
-9	271	-250
-11	336	342
-13	68	-76
-15	99	117
H= 6, K= 5		
1	406	421
2	213	-186
3	133	-143
4	105	112
5	266	289
7	398	-395
9	156	158
10	70	116
12	58	-79
H= 6, K= 5		
-1	344	-391
-3	100	-145
-4	390	-367
-5	343	-298
-7	329	379
-8	159	182
-9	134	-158
-10	135	-133
-11	234	243
-13	190	-197
H= 7, K= 5		
1	328	293
3	330	-310
5	234	258
6	118	134
9	148	167
10	60	-80
11	147	-173
H= 7, K= 5		
-1	293	-276
-3	294	290
-4	105	92
-5	390	-388
-8	103	-105
-9	130	-149
-11	289	295
-13	188	-206
H= 8, K= 5		
0	190	199
1	240	246
2	135	133
3	84	88
5	229	247
7	248	-268
9	70	75
H= 8, K= 5		
-1	383	-338
-2	105	-149
-3	405	378
-5	279	-268
-7	131	141
-9	174	-185
-10	50	72
-13	147	-155
H= 9, K= 5		
0	58	-72
1	219	250
2	99	-93
3	222	-200
5	72	74
7	178	-163
H= 9, K= 5		
-2	131	161
-3	197	222
-4	58	80
-5	372	-340
-6	112	-136
-7	211	215
-9	97	-82
-11	221	154
H= 10, K= 5		
3	96	-86
5	183	208
7	140	-180
H= 10, K= 5		
-1	201	-174
-2	76	-90
-3	286	319
-5	135	-153
-7	67	82
-9	195	-211
H= 11, K= 5		
1	207	238
3	106	-112
H= 11, K= 5		
-1	78	-89
-3	63	50
-5	179	-179
-7	208	246
-9	68	-74
H= 12, K= 5		
-1	52	-75
-3	176	275
-5	96	-120
H= 0, K= 6		
0	606	589
1	147	162
2	231	-244
3	180	-144
4	200	195
5	319	300
6	332	-342
8	229	262
11	156	-170
12	116	144
14	200	-234
H= 1, K= 6		
1	211	158
2	436	-444
3	138	-130
4	399	404
5	390	389
6	182	-160
7	273	-273
10	211	-235
12	222	186
14	90	-128

TABLE II. (Cont.)

L	FO	FC
H= 1, K= 6		
-3	200	160
-4	545	518
-5	377	-327
-6	499	-482
-10	96	-146
-11	129	130
-12	187	201
-13	102	-136
H= 2, K= 6		
0	440	401
1	110	128
2	150	-124
4	144	137
5	89	112
6	313	-316
7	229	-219
8	98	150
9	96	91
12	247	274
13	120	110
H= 2, K= 6		
-1	218	-209
-2	337	-360
-3	430	476
-4	78	115
-5	209	-193
-6	282	-256
-8	367	341
-10	246	-284
-11	125	-119
-14	77	-124
H= 3, K= 6		
0	337	281
1	107	114
2	450	-438
3	84	-65
4	178	153
5	132	131
6	96	-133
7	296	-287
8	310	339
9	133	144
10	262	-266
H= 3, K= 6		
-2	169	160
-3	266	278
-4	333	318
-5	177	-186
-6	424	-395
-7	234	259
-8	98	149
-9	97	-123
-10	211	-222
-12	194	238
-13	70	-67
-14	114	-124
H= 4, K= 6		
0	118	140
2	152	-172
3	351	-312
4	368	366
5	235	211
6	381	-390
7	168	-207
12	131	151
H= 4, K= 6		
-2	321	-333
-4	352	302
-7	310	309
-8	195	230
-9	136	-173
-10	297	-322
-14	59	-104
H= 5, K= 6		
0	470	477
1	229	221
2	509	-517
3	165	-171
8	215	233
9	119	99
H= 5, K= 6		
-1	363	-356
-2	92	-94
-3	93	97
-4	94	113
-5	96	-135
-6	168	-210
-8	193	193
-11	117	133
-12	127	163
-13	118	-128
H= 6, K= 6		
2	295	-257
4	240	218
5	135	163
9	105	118
10	204	-207
H= 6, K= 6		
-1	136	-124
-2	441	-376
-3	218	198
-4	381	390
-5	333	-333
-10	218	-246
-12	92	121
-13	45	-101
H= 7, K= 6		
3	319	296
5	202	170
6	149	-145
7	114	-137
8	240	275
H= 7, K= 6		
-2	381	-346
-3	168	176
-5	215	-231
-7	92	101
-8	211	266
-9	84	-107
-10	78	-124
-11	67	85
-12	72	118
H= 8, K= 6		
2	129	-143
3	89	-119
4	268	247
8	176	208
H= 8, K= 6		
-1	94	-108
-2	163	-147
-3	162	173
-4	230	292
-6	125	-180
H= 9, K= 6		
0	85	94
4	243	247
6	211	-235
H= 9, K= 6		
-1	121	-134
-2	214	-256
-3	185	159
-8	194	179
H= 10, K= 6		
0	142	150
2	196	-203
4	82	124
H= 10, K= 6		
-1	125	-143
-2	73	-69
-3	125	133
-6	149	-238
H= 11, K= 6		
-1	64	-84
-2	164	-191
-4	100	152

TABLE III. Fractional Atomic Coordinates.[a]

Atom	x/a	y/b	z/c
Sr	0.0000	–0.1517(3)	–0.2500
O_1	0.0227(8)	0.1235(14)	–0.3478(6)
O_2	0.1407(10)	–0.2058(14)	–0.3668(7)
C_1	0.0753(11)	0.1672(22)	–0.4106(9)
C_2	0.1800(12)	–0.1241(21)	–0.4311(9)
N_1	0.0714(12)	0.3463(20)	–0.4412(9)
N_2	0.1421(11)	0.0527(17)	–0.4585(8)
N_3	0.2602(13)	–0.1943(21)	–0.4814(10)
O_3	–0.2207(9)	–0.0917(13)	–0.3445(7)
O_4	–0.0986(9)	–0.4179(14)	–0.3490(7)
C_3	–0.3053(14)	–0.1977(24)	–0.3329(10)
C_4	–0.1888(12)	–0.4814(20)	–0.3249(8)
N_4	–0.4212(11)	–0.1371(21)	–0.3385(11)
N_5	–0.2932(10)	–0.3844(15)	–0.3170(8)
N_6	–0.1976(11)	–0.6648(17)	–0.3037(9)
Cl	0.4202(3)	0.3331(6)	–0.3896(2)
O_5	0.3394(11)	0.3510(22)	–0.4763(8)
O_6	0.5375(11)	0.2725(18)	–0.4029(10)
O_7	0.4302(12)	0.5153(19)	–0.3438(10)
O_8	0.3695(11)	0.2054(18)	–0.3277(9)

[a] Estimated standard deviations, shown in parentheses, are right-adjusted to the least significant digit in the preceeding number.

calculated without correction for thermal motion using NRC-12 computer program[28]. Pertinent least squares planes and deviations therefrom and dihedral angles between[29] are reported in Table, VII. Figure 1 shows the molecular unit, [Sr (biuret)$_4$](ClO$_4$)$_2$. Figure 2 is a packing diagram showing the contents of the unit cell. Figure 3 shows distances in the strontium(II) co-ordination polyhedron.

Discussion

The crystal structure consists of well separated monomeric units. The molecular unit, [Sr(NH$_2$CONHCONH$_2$)$_4$](ClO$_4$)$_2$, shows C_2 symmetry (Figure 1, 2). The four bonded biuret oxygens in the asymmetric unit, Sr/2(NH$_2$CONHCONH$_2$)$_2$(ClO$_4$), will complete the co-ordination polyhedron upon π rotation (Figure 3).

The co-ordination polyhedron is best described as being closer to square antiprismatic (D_{4d}) than cubic (O_h). Such co-ordination is expected for this tetrakis biuret complex with strontium(II) considering that the ligand is a neutral bidentate[30,31]. The $\bar{8}$ axis of the idealized D_{4d}–$\bar{8}2m$ square antiprism should be taken

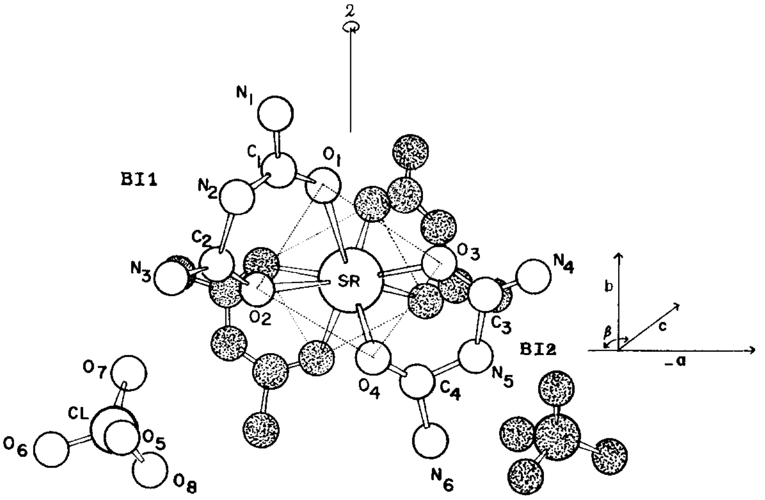

Figure 1. Molecular unit. Atoms at x, y, z and x̄, y, 1/2–z viewed perpendicular to the *ab*-face. The shaded atoms at x̄, y, 1/2–z are related to the unshaded atoms at x, y, z by the two-fold rotation axis. Strontium at fractional co-ordinates 0.0, –0.1517, –0.25.

TABLE IV. Anisotropic Thermal Parameters as Mean Square Amplitude (A^2).[a]

Atom	$U_{11} \times 10^3$	$U_{22} \times 10^3$	$U_{33} \times 10^3$	$U_{23} \times 10^3$	$U_{13} \times 10^3$	$U_{12} \times 10^3$
Sr	26(1)[b]	20(1)	26(1)	–00(1)	08(1)	06(1)
O_1	41(5)	33(6)	29(5)	03(5)	09(4)	–03(5)
O_2	51(8)	33(7)	39(5)	05(4)	20(5)	11(5)
C_1	26(6)	45(9)	27(6)	–02(7)	–05(5)	–09(7)
C_2	29(7)	38(9)	34(7)	–08(7)	06(5)	–04(6)
N_1	55(8)	38(9)	48(7)	18(7)	13(6)	15(7)
N_2	38(7)	33(8)	31(6)	10(5)	14(5)	08(5)
N_3	51(8)	62(11)	57(9)	–07(8)	25(7)	05(7)
O_3	38(5)	24(6)	51(6)	09(5)	04(4)	–04(4)
O_4	31(5)	30(6)	53(6)	–04(5)	10(4)	–01(4)
C_3	39(8)	56(12)	36(8)	–07(7)	01(6)	00(7)
C_4	31(7)	31(9)	21(6)	00(5)	07(5)	–03(6)
N_4	27(6)	44(9)	84(10)	00(8)	03(6)	05(6)
N_5	38(6)	15(7)	41(6)	03(5)	13(5)	00(5)
N_6	42(7)	17(7)	60(8)	03(6)	07(6)	–04(6)
Cl	30(2)	39(2)	35(2)	–01(2)	03(1)	–03(2)
O_5	57(7)	114(12)	36(6)	20(8)	–18(5)	–33(9)
O_6	42(7)	55(9)	103(11)	–01(8)	34(7)	12(6)
O_7	69(9)	49(9)	93(10)	–27(8)	23(8)	02(7)
O_8	54(8)	62(9)	67(8)	17(7)	10(6)	–13(6)

[a] The form of the anisotropic thermal parameter is $\exp[-2\pi^2(U_{11}h^2a^{*2} + U_{22}k^2b^{*2} + U_{33}l^2c^{*2} + 2U_{23}klb^*c^*\ 2U_{13}hla^*c^* + U_{12}hka^*b^*)]$. [b] Estimated standard deviation, shown in parentheses, are right-adjusted to the least significant digit in the preceeding number.

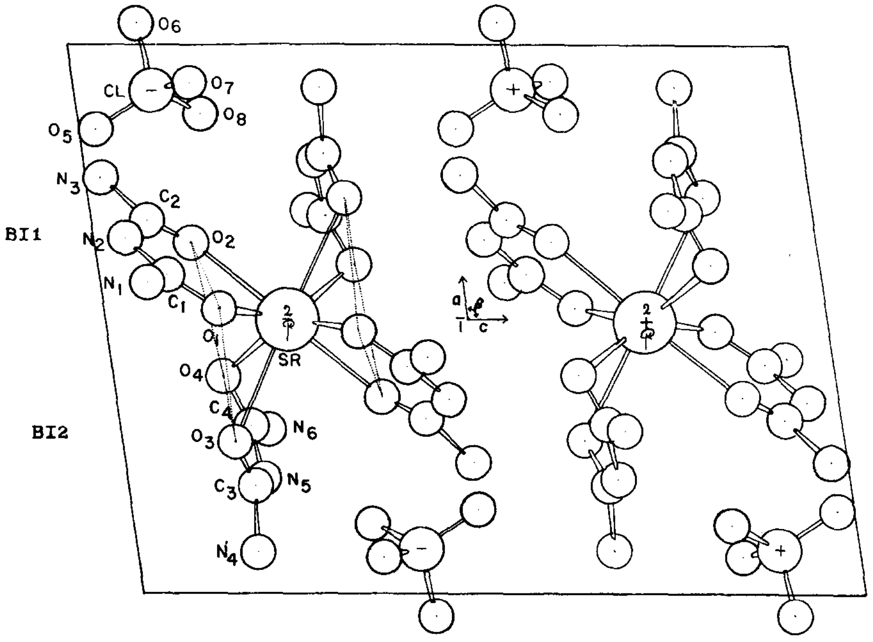

Figure 2. Packing diagram showing contents of the unit cell viewed down *b*-axis.

TABLE V. Interatomic Distances (A).

Bond distances in co-ordination sphere			
Sr—O$_1$	2.494(10)[a]	Sr—O$_3$	2.689(10)
Sr—O$_2$	2.506(10)	Sr—O$_4$	2.571(10)
Distances within ligands			
C$_1$—O$_1$	1.198(16)	C$_3$—O$_3$	1.253(19)
C$_2$—O$_2$	1.240(17)	C$_4$—O$_4$	1.209(17)
C$_1$—N$_2$	1.375(18)	C$_3$—N$_5$	1.386(21)
C$_2$—N$_2$	1.399(20)	C$_4$—N$_5$	1.386(18)
C$_1$—N$_1$	1.380(22)	C$_3$—N$_4$	1.364(20)
C$_2$—N$_3$	1.339(20)	C$_2$—N$_6$	1.380(19)
Pertinent non-bonded approaches			
Sr···N$_2$	3.913(11)	Sr···N$_5$	3.704(12)
O$_1$···O$_2$	2.776(14)	O$_3$···O$_4$	2.752(14)
Distances within anion			
	Cl—O$_5$	1.450(12)	
	Cl—O$_6$	1.426(13)	
	Cl—O$_7$	1.484(14)	
	Cl—O$_8$	1.465(13)	
Hydrogen-bonded atoms			
N$_1$···O$_4$(I)[2]	3.019(17)	N$_4$···O$_7$(III)	3.030(20)
N$_3$···O$_6$(II)	3.062(19)	N$_5$···O$_5$(IV)	2.981(16)
		N$_5$···O$_7$(V)	3.061(18)
		N$_6$···O$_1$(VI)	3.058(16)
		N$_6$···O$_8$(V)	3.050(18)

[a] Estimated standard deviations, shown in parentheses, are right-adjusted to the least significant digit in the preceeding number. [b] Roman numerals refer to the following transformations of the atoms, relative to the assymetric unit at x,y,z

I	x, 1 + y, z	IV	$\bar{x}$, $\bar{y}$, $\bar{z}$ – 1
II	1 – x, $\bar{y}$, $\bar{z}$ – 1	V	$\bar{x}$, y – 1, $\bar{z}$ – 1/2
III	x – 1, y – 1, z	VI	x, y – 1, z

TABLE VI. Bond Angles (deg.).

Angles involving cation cluster			
Sr—O$_1$—C$_1$	138.5(9)[a]	Sr—O$_3$—C$_3$	119.3(9)
Sr—O$_2$—C$_2$	139.4(9)	Sr—O$_4$—C$_4$	116.0(9)
O$_1$—C$_1$—N$_2$	126(1)	O$_3$—C$_3$—N$_5$	125(1)
O$_2$—C$_2$—N$_2$	122(1)	O$_4$—C$_4$—N$_5$	125(1)
C$_1$—N$_2$—C$_2$	125(1)	C$_3$—N$_5$—C$_4$	123(1)
O$_1$—C$_1$—N$_1$	120(1)	O$_3$—C$_3$—N$_4$	122(1)
O$_2$—C$_2$—N$_3$	124(1)	O$_4$—C$_4$—N$_6$	122(1)
N$_1$—C$_1$—N$_2$	114(1)	N$_4$—C$_3$—N$_5$	114(1)
N$_3$—C$_2$—N$_2$	113(1)	N$_6$—C$_4$—N$_5$	113(1)
O$_1$—Sr—O$_2$	67.4(3)	O$_3$—Sr—O$_4$	63.1(3)
Angles about chlorine in anion			
	O$_5$—Cl—O$_6$	112.6(8)	
	O$_5$—Cl—O$_7$	107.8(8)	
	O$_5$—Cl—O$_8$	110.0(8)	
	O$_6$—Cl—O$_7$	109.1(8)	
	O$_6$—Cl—O$_8$	109.4(8)	
	O$_7$—Cl—O$_8$	107.9(8)	

[a] Estimated standard deviations, shown in parentheses, are right-adjusted to the least significant digit in the preceeding number.

TABLE VII. Planes of Best Fit and Deviations Therefrom.[a]

Plane (A): O$_1$, O$_2$, C$_1$, C$_2$, N$_1$, N$_2$, N$_3$
$-0.687X - 0.295Y - 0.664Z = 2.373$
Deviation (A) from plane
O$_1$ 0.010, O$_2$ –0.037, C$_1$ 0.20, C$_2$ 0.006, N$_1$ –0.087 N$_2$ 0.140, N$_3$ –0.052
Distance (A) of other atoms from plane
Sr –0.017

Plane (B): O$_1$, C$_1$, N$_1$, N$_2$
$-0.730X - 0.206Y - 0.652Z = 2.362$
Deviation (A) from plane
O$_1$ 0.001, C$_1$ –0.002, N$_1$ 0.001, N$_2$ 0.001

Plane (C): O$_2$, C$_2$, N$_3$, N$_2$
$-0.658X - 0.382Y - 0.649Z = 2.458$
Deviation (A) from plane
O$_2$ 0.002, C$_2$ –0.004, N$_3$ 0.001, N$_2$ 0.001

Plane (D): O$_1$, Sr, O$_2$
$-0.680X - 0.310Y - 0.664Z = 2.377$

Plane (E): O$_3$, O$_4$, C$_3$, C$_4$, N$_4$, N$_5$, N$_6$
$-0.126X - 0.158Y - 0.979Z = 5.299$
Deviation (A) from plane
O$_3$ –0.123, O$_4$ 0.143, C$_3$ –0.041, C$_4$ 0.012, N$_4$ 0.130, N$_5$ –0.063, N$_6$ –0.058
Distance (A) of other atoms from plane
Sr –1.671

Plane (F): O$_3$, C$_3$, N$_4$, N$_5$
$-0.011X - 0.177Y - 0.984Z = 5.006$
Deviation (A) from plane
O$_3$ 0.004, C$_3$ –0.012, N$_4$ 0.004, N$_5$ 0.004

Plane (G): O$_4$, C$_4$, N$_6$, N$_5$
$-0.217X - 0.200Y - 0.956Z = 5.483$
Deviation (A) from plane
O$_4$ 0.002, C$_4$ –0.005, N$_6$ 0.002, N$_5$ 0.002

Plane (H): O$_3$, Sr, O$_4$
$0.529X + 0.328Y - 0.783Z = 2.722$

Plane (I): O$_5$, Cl, O$_6$
$0.302X + 0.950Y - 0.076Z = 4.400$

Plane (J): O$_7$, Cl, O$_8$
$-0.868X + 0.229Y - 0.441Z = -1.765$

Dihedral angles between planes (deg.)

Plane (A) and Plane (D)	1.8
Plane (B) and Plane (C)	10.9
Plane (E) and Plane (H)	49.6
Plane (F) and Plane (G)	12.0
Plane (A) and Plane (E)	38.4
Plane (I) and Plane (J)	90.6

[a] Equations are expressed in the cosine form $lX + mY + nZ = p$ where X, Y and Z are cartesian co-ordinates related to the cell co-ordinates by $X = xa$, $Y = yb$, $Z = zc$.

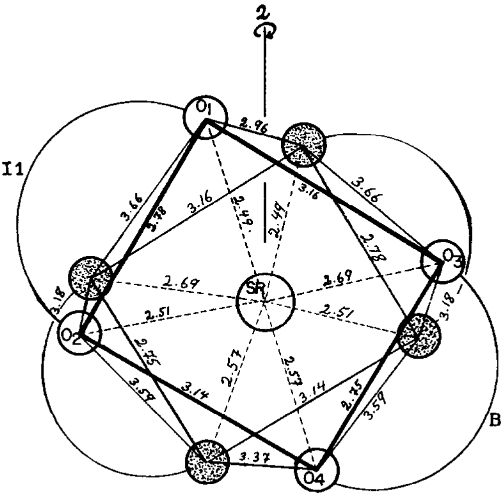

Figure 3. Antiprismatic co-ordination polyhedron viewed perpendicular to the *ab*-face showing distances (A). The shaded atoms are related to the unshaded atoms by the two-fold rotation axis.

as perpendicular to the *ab*-face and the 2 axis parallel to the *b*-axis. The bidentate ligands span opposite edges of the square faces of the antiprism (Figure 3). Deviation from idealized square antiprismatic geometry is towards the quasi-symmetry D_2–222. Such a quasi-symmetry has been achieved in the tetrakis complex $[Zr(CH_3COCHCOCH_3)_4]$[32] where, with an average zirconium–oxygen distance of 2.198 A, the edge length (2.675 A) of an equilateral antiprism is just the average intra-ring oxygen–oxygen distance (2.674 A). In $[Sr(NH_2CONHCONH_2)_4]^{++}$, the Sr(II) antiprismatic polyhedron exhibits significant deviation from D_2 symmetry. This deviation is attributed to a smaller average intra-ring O–O distance (2.76 A) than is required to span the edge of the equilateral antiprism with an average Sr–O bond distance of 2.55 A. The co-ordination polyhedron in $[Ce(CH_3COCHCOCH_3)_4]$[33] is also approximately square antiprismatic with symmetry approximating D_2 but with more deviation from D_2 than in the Zr(IV) case and less deviation than in the Sr(II) case. Since in the three cases the chelate bite is nearly the same, deviation from D_2 symmetry will be greatest with the largest cation, namely Sr(II) (ionis radius[34] for Sr(II) = 1.13 A, Ce(IV) = 1.01 A, and Zr(IV) = 0.80 A). The intra-ring O–O separation is 2.81 A along square face edges, while the inter-ring O–O separation is 2.97 A along square face edges, and 2.95 A along triangular faces in the Ce(IV) complex. However, the intra-ring O–O distance is 2.76 A along square face edges, while the inter-ring O–O distance is 3.15 A along square face edges, and 3.45 A along triangular faces in the Sr(II) complex. In contrast, the co-ordination polyhedron in the charged complex anion $[Y(CF_3COCHCOCF_3)_4]^-$[35] is, as expected[30], best described as D_2 dodecahedral rather than antiprismatic.

The strontium–oxygen bond distances (2.49, 2.51, 2.69, 2.57 A) average 0.03 A longer than the sum of the ionic radii (Sr^{++} = 1.13 A, 0^{2-} = 1.40 A)[34], indicating weak metal–oxygen bonding. This was previously found to be the case in the similar alkaline earth complex $[Ba(CH_3CONHCOCH_3)_5](ClO_4)_2$[12], where the metal–oxygen bond distance average 0.06 A longer than the sum of the ionic radii.

Octaco-ordination for strontium perchlorate complexes with neutral ligands is not unusual. It was suggested for the similar oxygen chelate with diacetamide, $Sr(CH_3CONHCOCH_3)_4(ClO_4)_2$[11], and for complexes with the weakly basic nitrogen bidentates 1,10-phenanthroline and 2,2′-bipyridine[14]. It seems however that the bidentate bite is a critical factor in determining the co-ordination number. The weakly basic nitrogen bidentates 1,8-naphthyridine, and 2,7-dimethyl-1,8-naphthyridine form four membered rings with the metal and exhibit an unexpected co-ordination number of four[16]. Since the biuret bite (2.75, 2.78 A) is shown to be equal to that of diacetamide in $[Ba(CH_3CONHCOCH_3)_5](ClO_4)_2$[12] (2.72–2.79 A), both neutral ligands are expected to yield complexes of similar co-ordination numbers with the alkaline earth perchlorates. This was found to be generally true except for barium where the biuret complex was found to be $1.5Ba(ClO_4)_2(biuret)_3 \cdot 1solvent$[18] and its infrared spectrum indicated bridging ligands in the α-form[2]. The x-ray structure of $1.5Ba(ClO_4)_2(biuret)_3 \cdot 1butanol$ awaits determination.

The two biuret ligands (BI1 and BI2 Figure 2, 3) in the asymmetric unit span opposit edges of the square face of the antiprism with BI2 folded in such a way as to make it approximately coplanar with the square antiprismatic face (Figure 2). BI1 makes with strontium an overall flat six membered ring except for some puckering. The flatness of the ring is exhibited as a 1.8° dihedral angle (Table VII) between the ligand best fit plane (O_1, O_2, C_1, C_2, N_1, N_2, N_3) and the (O_1, Sr, O_2) plane, and is supported by a long Sr–N_2 separation (Table V) and a short Sr to ligand plane distance (0.017 A, Table VII). BI2, on the other hand, makes with strontium a folded and puckered six membered ring. The folding about the O–O line is exhibited as a 49.6° dihedral angle between the ligands best fit plane (O_3, O_4, C_3, C_4, N_4, N_5, N_6) and the (O_3, Sr, O_4) plane, and is supported by a short Sr–N_5 separation (Table V) and a long Sr to ligand plane distance (1.671 A, Table VII). Ring puckering is exhibited mainly by twisting about the O–O line, the magnitude of which is indicated by the dihedral angle between the ligands two (N, O, C, N) best fit planes (10.9° for BI1 and 12.0° for BI2). Some twisting about the O–O lines has been anticipated from the infrared spectrum since a remnant band in the NH_2 deformation region suggests deviation from the β-form of biuret towards the α-form[2]. The difference in the disposition of the two ligands has been suspected from the numerous carbonyl stretching bands in the infrared spectrum.

Variations in intra-ligand dimensions, which add to the complexity of the infrared spectrum, are obvious from Tables V and VI. The carbonyl entry angles in BI1 are significantly larger than in BI1 (138.5, 139.4° in BI1 compared to 119.3, 116.0° in BI2, Table VI). This is expected considering the planarity of the first chelate ring. This large variation in the entry angle would tend to support an electrostatic metal–oxygen bonding model over a covalent hybrid orbitals model. The larger bite and bite angle of BI1 over BI2 (2.776 A, 67.4° in BI1 compared to 2.752 A, 63.1° in BI2, Table V, VI) are again consistant with the planarity of the first chelate ring.

The perchlorate moiety is ionic but with obvious deviation from T_d symmetry (Table V, VI). This deviation, which is apparent from the infrared, is caused by packing considerations as well as extensive hydrogen bonding, in particular with the primary and secondary amines of BI2 (Table V). In fact this hydrogen bonding might have been the driving force behind BI2 chelate ring's folding (Figure 2). The amine stretching region in the infrared shows weaker amine–perchlorate hydrogen bonding than amine–carbonyl hydrogen bonding in crystalline biuret.

The thermal vibrations of the atoms (Table IV) are not highly anisotropic. Allowance for anisotropy reduced the R-factor by only 0.022. The thermal parameters are generally low in agreement with the air stability and non-hygroscopy of this perchlorate complex.

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