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The Crystal and Molecular Structure of Diammine-(*o*-phthalato)copper (II)

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Diammine-(*o*-phthalato)copper(II), $Cu(NH_3)_2C_8H_4O_4$, is monoclinic, $P2_1/c$: $a=6{\cdot}64$ (1), $b=19{\cdot}91$ (1), $c=7{\cdot}55$ (1) Å, $\beta=109{\cdot}9°$ (·1°), $Z=4$. The crystal structure has been determined from three-dimensional X-ray photographic data and refined by differential methods with anisotropic parameters to a final R value of 7·7%. The coordination around the metal atom is nearly square planar and involves two oxygen atoms from different phthalate ions and two nitrogen atoms from ammonia molecules [Cu–O 1·988 (5), 1·971 (5); Cu–N 1·992 (6), 1·997 (8) Å]. Two more oxygen atoms, *trans* with respect to the coordination plane, make two long contacts [Cu–O 2·313 (5), 2·799 (5) Å] so the coordination polyhedron can be considered also a distorted bipyramid. Two of these bipyramids are coupled along an O···O edge and the couples are linked in chains by phthalate bridges. Both carboxyl groups are slightly rotated with respect to the benzene ring in the same direction. Packing is determined by a set of hydrogen bonds involving the nitrogen atoms from ammonia molecules and the oxygen atoms from the carboxyl groups.

Introduction

The X-ray structural analysis of diammine-(*o*-phthalato)copper(II) was carried out as a part of a research programme concerning the coordination of aromatic carboxyl ligands (Biagini Cingi, Guastini, Musatti & Nardelli, 1969).

Experimental

Crystals of diammine-(*o*-phthalato)copper(II) were obtained by slow evaporation, at room temperature, from an aqueous solution of copper(II) phthalate to which concentrated ammonia was added. Their habit was that of flattened dark blue monoclinic prisms. Cell constants, determined from rotation and Weissenberg photographs, are as follows:

$[Cu(NH_3)_2C_8H_4O_4]$, $M=261{\cdot}7$

$a=6{\cdot}64$ (1), $b=19{\cdot}91$ (1), $c=7{\cdot}55$ (1) Å

$\beta=109{\cdot}9°$ (0·1°), $V=938{\cdot}5$ Å^3, $Z=4$, $D_m=1{\cdot}83$, $D_x=1{\cdot}85$ g.cm^{-3}

$\mu=33{\cdot}6$ cm^{-1} (Cu $K\alpha$), $F(000)=492$

Space group $P2_1/c$ (from systematic absences).

Two series of equi-inclination Weissenberg photographs (Ni-filtered Cu-radiation; multiple film technique) were collected at room temperature around [100] and [001] up to the sixth layer. Of the 2133 possible independent reflexions within the Cu $K\alpha$ sphere, 1470 were collected; the remainder 663 were too weak to be estimated or were screened in the blind region of the camera. The intensities were measured photometrically and corrected for Lorentz, polarization and spot-shape effects. The sample used to take the photographs around [100] was a prism elongated along this axis and for correcting the absorption was considered to be a cylinder with a mean radius of 0·008 cm; the photographs around [001] were taken with a nearly spherical fragment with a mean radius of 0·01 cm and for this set of data the absorption correction for spherical specimens was used. The data of both sets were correlated and put on a common scale using the least-squares procedure of Rollett & Sparks (1960). The absolute scale was determined, first by Wilson's method, then by comparison with the calculated values.

Structure determination and refinement

The structure was solved by the heavy-atom method starting from a three-dimensional Patterson map and refined by several cycles of Booth's differential synthesis with anisotropic thermal parameters determined using the second derivatives of the electron density from differential synthesis (Nardelli & Fava, 1960). The final residual error indices (R, for observed reflexions only; R' assuming $F_o=\frac{1}{2}F_{min}$ when $F_c \geq F_{min}$ for unobserved reflexions, multiplicities not considered) were $R=7{\cdot}7\%$ and $R'=9{\cdot}4\%$ including the four benzene hydrogen atoms localized from a final F_o-F_c synthesis. The coordinates of these atoms are reported in Table 1. Attempts to locate the ammonia hydrogen atoms directly were unsuccessful. The final parameters for non-hydrogen atoms with their estimated standard deviations (e.s.d.'s) (Cruickshank, 1949, 1950, 1956) and the ratios between the e.s.d.'s and coordinate shifts are quoted in Table 2. In Table 3 observed atomic peak shapes are compared with the calculated ones. The structure factors reported in Table 4 are calculated with the final parameters of Table 2, including the benzene hydrogen atoms contributions, using the atomic scattering factors of Cromer & Mann (1968) for Cu, N, O, C and Stewart, Davidson & Simpson (1965) for H.

Table 1. *Observed fractional coordinates for hydrogen atoms in the benzene ring*

	x/a	*y/b*	*z/c*	ϱ_0
H(1)	0·2167	0·2361	0·3717	0·5 e.Å^{-3}
H(2)	0·5000	0·3208	0·4753	0·7
H(3)	0·8667	0·2901	0·5253	0·6
H(4)	0·9479	0·1811	0·4477	0·7

Discussion

Bond distances and angles in the coordination polyhedron and in the organic ion are quoted in Table 5 and in Fig. 1 which shows a clinographic projection of the structure. The coordination around each copper atom is nearly square planar and concerns two oxygen atoms from two different phthalate ions and two nitrogen

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

	x/a	*y/b*	*z/c*	B_{11}	B_{22}	B_{33}	B_{12}	B_{13}	B_{23}	$\|r(x)\|$	$\|r(y)\|$	$\|r(z)\|$
Cu	542 (2)	−47 (1)	3053 (1)	25 (1)	26 (0)	26 (1)	0 (1)	9 (1)	−1 (1)	3	3	6
O(1)	5691 (9)	550 (2)	1531 (7)	22 (4)	27 (1)	20 (0)	4 (3)	0 (5)	−6 (3)	4	9	6
O(2)	8728 (8)	541 (2)	4020 (7)	17 (4)	22 (1)	15 (4)	5 (3)	4 (5)	2 (3)	6	3	5
O(3)	985 (9)	1162 (2)	1329 (8)	22 (4)	32 (1)	22 (4)	0 (3)	2 (5)	−2 (4)	4	10	7
O(4)	2801 (8)	637 (1)	4010 (7)	22 (4)	23 (1)	24 (5)	−3 (3)	8 (6)	2 (4)	4	8	24
N(1)	8170 (11)	−708 (2)	2057 (8)	24 (5)	24 (0)	26 (5)	−5 (3)	6 (6)	−9 (4)	4	2	7
N(2)	2555 (13)	−554 (2)	2094 (11)	30 (7)	29 (1)	36 (7)	1 (4)	18 (9)	−4 (5)	10	2	6
C(1)	6386 (12)	1452 (3)	3646 (10)	18 (5)	18 (1)	18 (5)	1 (4)	7 (7)	−1 (4)	6	3	14
C(2)	4262 (12)	1633 (2)	3378 (10)	22 (5)	19 (1)	20 (6)	0 (4)	6 (7)	0 (4)	24	3	33
C(3)	3782 (15)	2277 (3)	3743 (13)	32 (8)	23 (1)	26 (7)	4 (5)	14 (9)	−1 (5)	9	8	6
C(4)	5373 (15)	2752 (2)	4383 (12)	34 (8)	26 (1)	32 (8)	1 (5)	13 (10)	−4 (6)	17	8	10
C(5)	7521 (14)	2579 (3)	4720 (11)	38 (8)	24 (0)	33 (7)	−5 (4)	10 (10)	−6 (5)	28	4	22
C(6)	7990 (12)	1933 (3)	4332 (10)	25 (6)	21 (0)	27 (6)	−6 (3)	9 (8)	−5 (4)	4	8	19
C(7)	6939 (10)	800 (2)	3003 (9)	15 (5)	18 (1)	18 (5)	2 (3)	5 (6)	−2 (4)	3	5	14
C(8)	2547 (13)	1114 (2)	2825 (10)	18 (5)	21 (1)	20 (6)	2 (4)	7 (7)	0 (5)	3	4	5

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}
Cu	obs.	66·9	653	696	605	−39	184	−1
	calc.	68·3	649	691	603	−38	184	0
O(1)	obs.	13·9	113	123	119	−10	33	7
	calc.	13·9	113	123	118	−9	33	6
O(2)	obs.	14·1	139	143	105	−23	37	18
	calc.	14·0	137	143	105	−22	37	16
O(3)	obs.	13·1	119	113	104	−7	33	−1
	calc.	13·3	122	115	107	−6	39	−1
O(4)	obs.	13·3	120	145	94	−4	19	−16
	calc.	13·3	119	143	93	−4	19	−14
N(1)	obs.	10·6	96	108	90	−9	21	−13
	calc.	10·4	94	108	90	−8	22	−12
N(2)	obs.	9·4	86	99	71	−23	28	4
	calc.	9·3	86	98	72	−22	28	3
C(1)	obs.	9·6	91	88	89	0	28	−2
	calc.	9·3	89	86	87	1	27	−2
C(2)	obs.	9·8	92	92	89	−5	31	−2
	calc.	9·5	90	91	87	−5	30	−3
C(3)	obs.	8·8	75	91	69	2	22	4
	calc.	8·6	74	89	68	2	22	3
C(4)	obs.	8·5	75	96	70	−6	25	3
	calc.	8·4	74	94	69	−5	24	2
C(5)	obs.	8·1	74	76	71	−4	20	−4
	calc.	7·8	72	74	70	−4	19	−3
C(6)	obs.	8·8	74	93	72	2	19	−3
	calc.	8·5	73	91	71	2	18	−3
C(7)	obs.	10·8	102	106	96	1	27	0
	calc.	10·7	100	105	95	1	27	−1

Table 4. *Observed and calculated structure factors*

A minus sign with F_o means 'less than'.

h	k	l	$10F_o$	$10F_c$	h	k	l	$10F_o$	$10F_c$	h	k	l	$10F_o$	$10F_c$	h	k	l	$10F_o$	$10F_c$	h	k	l	$10F_o$	$10F_c$	h	k	l	$10F_o$	$10F_c$
1	0	0	410	523	2	13	0	144	160	2	3	-1	120	177	1	10	1	56-	11	4	17	1	289	-274	7	1	2	39-	-14	3	6	-2	333	-440
2	0	0	71	63	3	13	0	96	-88	3	3	1	273	-334	1	10	-1	48	46	4	17	-1	329	317	7	1	-2	64-	-11	4	8	2	308	213
3	0	0	160	203	4	13	0	264	250	3	3	-1	176	262	2	10	1	152	155	5	17	1	144	-139	8	1	-2	39-	-48	4	8	-2	289	-329
4	0	0	79	105	5	13	0	79	78	4	3	1	587	-612	2	10	-1	264	-258	5	17	-1	257	239	0	2	2	1433	-1355	5	8	2	152	147
5	0	0	120	-118	6	13	0	112	105	4	3	-1	588	641	3	10	1	56-	78	6	17	-1	128	121	1	2	2	241	-278	5	8	-2	71	-65
6	0	0	365	-314	0	14	0	644	631	5	3	1	128	-112	3	10	-1	208	-226	0	18	1	185	181	1	2	-2	740	-836	6	8	2	206	204
7	0	0	112	-71	1	14	0	281	298	5	3	-1	499	584	4	10	1	48-	-34	1	18	1	193	186	2	2	2	71	-82	6	8	-2	112	-106
1	1	0	160	192	2	14	0	273	272	6	3	1	281	-256	4	10	-1	200	-208	1	18	-1	79	-97	2	2	-2	361	-479	7	8	-2	64-	47
2	1	0	39	-9	3	14	0	273	270	6	3	-1	401	414	5	10	-1	128	-116	2	18	1	71-	14	3	2	-2	64	61	8	8	-2	31-	34
3	1	0	112	-101	4	14	0	48-	7	7	3	1	144	-131	5	10	-1	39-	19	2	18	-1	264	-226	3	2	-2	329	-437	0	9	2	168	-212
4	1	0	39	-51	5	14	0	39-	-24	8	3	-1	39-	72	6	10	1	39-	-22	3	18	1	79	-79	4	2	2	39-	-7	1	9	2	241	-237
5	1	0	31-	19	6	14	0	31-	11	0	4	1	144	52	6	10	-1	48	30	3	18	-1	96	-94	4	2	-2	547	-567	1	9	2	249	240
6	1	0	39-	4	1	15	0	79-	22	1	4	1	249	258	7	10	1	31-	62	4	18	1	39-	-12	5	2	2	351	322	2	9	2	168	-165
7	1	0	87	71	2	15	0	71-	39	1	4	-1	64	-94	7	10	-1	48-	-17	4	18	-1	56	-34	5	2	-2	466	-459	2	9	-2	264	-252
8	1	0	39-	-41	3	15	0	120	110	2	4	1	307	-288	0	11	1	273	-279	5	18	1	53-	28	6	2	2	257	238	3	9	2	87	-105
0	2	0	877	1207	4	15	0	297	303	2	4	-1	87	-157	1	11	1	152	-184	5	18	-1	71	-68	6	2	-2	39	-51	3	9	-2	193	-183
1	2	0	1071	1287	5	15	0	96	93	3	4	1	313	-286	1	11	-1	56-	-48	6	18	-1	15-	-20	7	2	2	208	221	4	9	2	48-	-52
2	2	0	305	426	6	15	0	68	45	3	4	-1	216	-248	2	11	-1	442	-486	0	19	1	112	-118	7	2	-2	64-	-29	4	9	-2	152	144
3	2	0	322	375	0	16	0	692	654	4	4	1	39-	17	2	11	-1	160	161	1	19	1	305	-285	0	3	2	48-	-7	5	9	2	39-	41
4	2	0	87	107	1	16	0	538	509	4	4	-1	64	-38	3	11	1	474	-500	1	19	-1	79	72	1	3	2	64	-70	5	9	-2	87	-85
5	2	0	216	-209	2	16	0	71	68	5	4	1	281	260	3	11	-1	523	565	2	19	1	168	-161	1	3	-2	515	-489	6	9	2	79	-79
6	2	0	176	-166	3	16	0	87	70	5	4	-1	96	-113	4	11	1	394	-409	2	19	-1	104	87	2	3	2	233	-236	6	9	-2	152	-148
7	2	0	71-	-36	4	16	0	120	113	6	4	1	71	63	4	11	-1	104	102	3	19	1	79	-69	2	3	-2	885	895	7	9	-2	64-	-48
1	3	0	216	-208	5	16	0	71	-57	6	4	-1	31-	36	5	11	1	349	-235	3	19	-1	56-	54	3	3	2	120	105	8	9	-2	7-	-77
2	3	0	426	-406	6	16	0	112	-117	7	4	1	104	-101	5	11	-1	273	257	4	19	1	193	-188	3	3	-2	56	5	0	10	2	434	-459
3	3	0	378	388	1	17	0	144	119	8	4	-1	39-	20	6	11	1	104	-95	4	19	-1	193	194	4	3	2	48-	-17	1	10	2	386	-408
4	3	0	64	60	2	17	0	71-	16	0	5	1	434	-416	6	11	-1	297	274	5	19	1	104	-113	4	3	-2	176	-215	1	10	-2	538	-517
5	3	0	120	-114	3	17	0	104	104	1	5	1	652	-723	0	12	1	233	229	5	19	-1	216	207	5	3	2	233	-201	2	10	2	71	58
6	3	0	39-	6	4	17	0	152	148	1	5	-1	241	-249	1	12	1	249	-222	0	10	1	87-	80	5	3	-2	39	-39	2	10	-2	579	-638
7	3	0	71-	9	5	17	0	112	104	2	5	1	401	-450	1	12	-1	193	-204	1	20	1	176	159	6	3	2	120	104	3	10	2	56-	-26
8	3	0	23-	31	6	17	0	96	117	2	5	-1	71	0	2	12	1	313	314	2	20	-1	128	-115	6	3	-2	56	-60	3	10	-2	442	-480
0	4	0	716	854	0	18	0	603	561	3	5	1	595	-641	2	12	-1	183	-203	2	20	1	64-	-43	7	3	2	39-	34	4	10	2	220	211
1	4	0	580	674	1	18	0	216	213	3	5	-1	588	632	3	12	1	185	186	2	20	-1	193	-181	8	3	-2	39-	-48	4	10	-2	434	-450
2	4	0	628	696	2	18	0	305	296	4	5	1	281	-296	3	12	-1	241	-238	3	20	1	48-	12	0	4	2	144	-190	5	10	2	152	141
3	4	0	241	260	3	18	0	64	58	4	5	-1	443	525	4	12	1	128	-113	3	20	-1	152	-145	1	4	2	337	-349	5	10	-2	39-	43
4	4	0	216	226	4	18	0	39-	26	5	5	1	329	-320	4	12	-1	144	147	4	20	1	56	-41	1	4	-2	893	-1053	6	10	2	193	187
5	4	0	31-	33	5	18	0	71	-62	5	5	-1	193	229	5	12	1	176	-170	4	20	-1	79	-74	2	4	2	313	-303	6	10	-2	71	-73
6	4	0	120	-103	1	19	0	193	-185	6	5	1	193	-165	5	12	-1	168	-160	5	20	-1	23-	-25	2	4	-2	861	-979	7	10	-2	56-	52
7	4	0	168	-146	2	19	0	112	110	6	5	-1	345	339	6	12	1	39-	-16	0	21	1	112	-99	3	4	2	241	232	0	11	2	538	-326
1	5	0	329	357	3	19	0	168	159	7	5	1	112	-110	6	12	-1	39-	9	1	21	1	160	-136	3	4	-2	918	-981	1	11	2	152	-165
2	5	0	185	196	4	19	0	104	95	0	6	1	297	289	7	12	-1	39-	-18	1	21	-1	112	-98	4	4	2	401	391	1	11	-2	264	277
3	5	0	322	337	5	19	0	96	98	1	6	1	418	441	0	13	1	224	-222	2	21	1	200	-189	4	4	-2	674	-526	2	11	2	120	-140
4	5	0	64	-47	0	20	0	281	261	1	6	-1	79	-114	1	13	1	128	-136	2	21	-1	79	80	5	4	2	144	119	2	11	-2	200	-207
5	5	0	264	272	1	20	0	273	242	2	6	1	401	-388	1	13	-1	96	-88	3	21	1	39-	-34	5	4	-2	120	-136	3	11	2	216	-231
6	5	0	168	-174	2	20	0	128	123	2	6	-1	273	-304	2	13	1	305	-327	3	21	-1	87	71	6	4	2	233	207	3	11	-2	128	-122
7	5	0	71-	-46	3	20	0	87	74	3	6	1	160	133	2	13	-1	64-	14	4	21	1	64	-51	6	4	-2	152	-183	4	11	2	64	67
0	6	0	233	-66	4	20	0	48	31	3	6	-1	39-	23	3	13	1	563	-572	4	21	-1	200	210	7	4	2	128	141	4	11	-2	56	-68
1	6	0	676	785	5	20	0	15-	15	4	6	1	128	129	3	13	-1	193	190	0	22	1	87	84	7	4	-2	64-	80	5	11	2	96	98
2	6	0	901	1068	1	21	0	64-	49	4	6	-1	79	-93	4	13	1	112	-92	1	22	1	120	101	0	5	2	402	344	5	11	-2	160	-166
3	6	0	329	375	2	21	0	112	106	5	6	1	39-	21	4	13	-1	289	280	1	22	-1	144	-132	1	5	2	241	208	6	11	2	31-	-27
4	6	0	39-	-18	3	21	0	168	161	5	6	-1	128	130	5	13	1	200	-184	2	22	1	48-	0	1	5	-2	466	-462	6	11	-2	168	-147
5	6	0	87	87	4	21	0	48	44	6	6	1	104	-100	5	13	-1	370	340	2	22	-1	144	-132	2	5	2	233	-219	7	11	-2	56-	-50
6	6	0	87	-75	0	22	0	136	114	6	6	-1	39-	19	6	13	1	104	-94	3	22	1	31-	13	2	5	-2	675	653	0	12	2	418	-435
7	6	0	233	-217	1	22	0	120	108	7	6	1	104	-105	6	13	-1	264	247	3	22	-1	176	-178	3	5	2	120	-105	1	12	2	152	-155
1	7	0	426	452	2	22	0	224	211	7	6	-1	64-	61	0	14	1	257	262	4	22	-1	56	-61	3	5	-2	112	129	1	12	-2	555	-609
2	7	0	394	408	3	22	0	96	89	8	6	-1	31-	32	1	14	1	361	353	0	23	1	64-	-17	4	5	2	87	-90	2	12	2	71-	29
3	7	0	257	274	4	22	0	23-	-29	0	7	1	224	-223	1	14	-1	176	-174	1	23	1	96	-78	4	5	-2	160	-185	2	12	-2	659	-705
4	7	0	56	75	1	23	0	144	135	1	7	1	264	-315	2	14	1	273	269	1	23	-1	56	-54	5	5	2	297	-270	3	12	2	168	162
5	7	0	56	53	2	23	0	112	112	1	7	-1	386	406	2	14	-1	104	-108	2	23	1	128	-132	5	5	-2	64	58	3	12	-2	682	-498
6	7	0	64	50	3	23	0	160	177	2	7	1	901	-1009	3	14	1	64-	5	2	23	-1	48	42	6	5	2	39-	40	4	12	2	152	152
7	7	0	54-	-58	0	24	0	64-	23	2	7	-1	241	260	3	14	-1	104	91	3	23	1	135	-156	6	5	-2	31-	-11	4	12	-2	152	-143
0	8	0	121	210	1	24	0	160	152	3	7	1	611	-658	4	14	1	112	-109	3	23	-1	120	126	7	5	2	31-	35	5	12	2	152	141
1	8	0	675	805	2	24	0	120	133	3	7	-1	547	634	4	14	-1	120	-119	0	24	1	136	137	7	5	-2	64-	-64	5	12	-2	168	-145
2	8	0	740	767	1	25	0	96	116	4	7	1	322	-349	5	14	1	112	-105	1	24	-1	79	75	0	6	2	442	-470	6	12	2	71	72
3	8	0	401	458	0	1	1	723	-652	4	7	-1	216	257	5	14	-1	39-	-6	1	24	-1	96	-105	1	6	2	401	-430	6	12	-2	120	-112
4	8	0	48	46	1	1	1	603	-676	5	7	1	378	-375	6	14	1	64	-59	2	24	1	31-	6	1	6	-2	418	-515	7	12	-2	39-	40
5	8	0	79	-75	1	1	-1	48	34	5	7	-1	241	260	6	14	-1	39-	30	2	24	-1	71	-75	2	6	2	144	-130	0	13	2	257	-271
6	8	0	168	-152	2	1	1	394	-463	6	7	1	120	-104	7	14	-1	23-	-14	0	23	1	39-	-13	2	6	-2	1034	-1109	1	13	2	152	-162
7	8	0	264	-257	2	1	-1	491	627	6	7	-1	233	228	0	15	1	273	-257	1	25	1	31-	-12	3	6	2	144	144	1	13	-2	305	294
1	9	0	152	140	3	1	1	490	-539	7	7	1	87	-100	1	15	1	144	-146	1	25	-1	31-	21	4	6	-2	636	-764	2	13	2	87	-84
2	9	0	48-	76	3	1	-1	31	91	0	8	1	112	165	1	15	-1	193	-176	2	25	-1	23-	2	4	6	2	224	226	3	13	-2	249	-254
3	9	0	48-	70	4	1	1	636	-658	1	8	1	144	164	2	15	1	241	-242	0	0	2	1151	-967	4	6	-2	353	-441	3	13	2	216	-202
4	9	0	71	79	4	1	-1	668	741	1	8	-1	394	-436	2	15	-1	273	268	1	0	2	434	-458	5	6	2	281	257	3	13	-2	128	-118
5	9	0	87	80	5	1	1	490	-450	2	8	1	48-	-60	3	15	1	208	-194	1	0	-2	387	-656	5	6	-2	224	-254	4	13	2	79	-66
6	9	0	87	82	5	1	-1	717	738	2	8	-1	152	145	3	15	-1	120	113	2	0	2	682	-480	6	6	2	168	149	4	13	-2	112	-103
7	9	0	64-	10	6	1	1	233	-213	3	8	1	136	118	4	15	1	410	-407	2	0	-2	120	0	6	6	-2	96	101	5	13	2	104	88
0	10	0	71	148	6	1	-1	281	284	3	8	-1	264	-280	4	15	-1	289	276	3	0	2	71	-68	7	6	-2	64-	23	5	13	-2	71	-74
1	10	0	796	888	7	1	1	48-	-5	4	8	1	104	58	5	15	1	128	-119	3	0	-2	748	-765	8	6	-2	39-	48	6	13	2	64	-65
2	10	0	482	523	7	1	-1	64-	52	4	8	-1	144	-156	5	15	-1	337	303	4	0	-2	48	30	0	7	2	185	140	6	13	-2	39-	-33
3	10	0	216	252	8	1	-1	39-	82	5	8	1	87	-79	6	15	1	71	-76	4	0	-2	250	-302	1	7	2	56-	7	0	14	2	336	-393
4	10	0	128	145	0	2	1	949	-955	5	8	-1	87	114	6	15	-1	144	129	5	0	2	401	363	1	7	-2	466	-473	1	14	2	176	-179
5	10	0	56	-52	1	2	1	144	146	6	8	1	79	-75	0	16	1	305	294	5	0	-2	507	-473	2	7	2	104	-102	1	14	-2	499	-515
6	10	0	56	-53	1	2	-1	418	-432	6	8	-1	39-	26	1	16	1	87	67	6	0	2	289	266	2	7	-2	128	129	2	14	2	87	102
7	10	0	193	-202	2	2	1	434	-386	7	8	1	39-	-38	1	16	-1	345	-340	6	0	-2	13-	-66	3	7	2	79	-77	2	14	-2	466	-467
1	11	0	120	-72	2	2	-1	410	-499	7	8	-1	48-	55	2	16	1	305	304	7	0	2	160	153	3	7	-2	233	246	3	14	2	64-	38
2	11	0	64-	82	3	2	1	273	-261	0	9	1	160	118	2	16	-1	71-	-68	7	0	-2	64-	-36	4	7	2	176	-177	3	14	-2	322	-305
3	11	0	56-	45	3	2	-1	23-	-17	1	9	1	466	-515	3	16	1	64-	-58	8	0	-2	39-	-15	4	7	-2	39	33	4	14	2	136	119
4	11	0	48-	-32	4	2	1	39-	45	1	9	-1	370	393	3	16	-1	64-	35	0	1	2	998	839	5	7	2	224	-207	4	14	-2	233	-214
5	11	0	87	79	4	2	-1	168	-232	2	9	1	595	-649	4	16	1	48-	-15	1	1	2	64	-67	5	7	-2	112	-125	5	14	2	120	109
6	11	0	128	124	5	2	1	257	242	2	9	-1	323	339	4	16	-1	56	46	1	1	-2	764	796	6	7	2	39-	-9	5	14	-2	193	-186
7	11	0	136	138	5	2	-1	64	63	3	9	1	700	-773	5	16	1	64	-50	2	1	2	128	-116	6	7	-2	39-	-21	6	14	-2	104	-90
0	12	0	482	484	6	2	1	71	53	3	9	-1	482	525	5	16	-1	120	-117	2	1	-2	459	455	7	7	2	23-	50	7	14	-2	31-	43
1	12	0	386	420	6	2	-1	132	-135	4	9	1	353	-371	6	16	-1	31-	26	3	1	2	48-	-1	7	7	-2	64-	16	0	15	2	87-	-5
2	12	0	303	322	7	2	1	64	-55	4	9	-1	257	282	0	17	1	87-	-35	3	1	-2	79	-84	8	7	-2	31-	-41	1	15	2	386	-372
3	12	0	305	316	7	2	-1	64-	67	5	9	1	361	-355	1	17	1	434	-422	4	1	2	120	-109	0	8	2	370	-414	1	15	-2	152	142
4	12	0	144	130	8	2	-1	39-	80	5	9	-1	120	121	1	17	-1	120	123	4	1	-2	233	-221	1	8	2	323	-349	2	15	2	71-	-33
5	12	0	39-	9	0	3	1	185	-197	6	9	1	168	-153	2	17	1	152	-158	5	1	2	144	130	1	8	-2	611	-693	2	15	-2	200	-169
6	12	0	64	-44	1	3	1	675	-716	6	9	-1	264	246	2	17	-1	71-	58	5	1	-2	15-	30	2	8	2	64-	-17	3	15	2	64-	3
7	12	0	71	-83	1	3	-1	361	-372	7	9	1	31-	-4	3	17	1	168	-158	6	1	2	39-	33	2	8	-2	853	-936	3	15	-2	64-	-47
1	13	0	64-	-8	2	3	1	636	-711	0	10	1	96	129	3	17	-1	37	70	6	1	-2	13-	-14	3	8	2	56-	-48	4	15	2	225	-218

atoms from the ammonia molecules. The Cu–O(1), Cu–O(2) and Cu–N(1^i), Cu–N(2) distances are in good agreement with those generally found in the planar part of other copper(II)-complexes as shown in Table 6.

There are two further coordination sites, *trans* with respect to the coordination plane, which correspond to longer interactions, one, Cu–O(3) 2·799 (5) Å, being much longer than the other, Cu–O(2^{ii}) 2·313 (5) Å, so the coordination polyhedron is an unsymmetrically elongated bipyramid. This situation is quite similar to that in glycylglycylglycinocopper(II) chloride sesquihydrate [Cu–O 2·304 (7), 2·817 (7) Å] (Freeman, Robinson & Schoone, 1964). The Cu–O(2^{ii}) bond is nearly perpendicular to the main coordination plane, the angle it makes with this plane being 80·2°. The Cu–O(3) bond is much more tilted with respect to that plane as it makes an angle of 52·2° with it; the distortion is probably due to steric requirements connected with the orientation of the organic ion. The Cu atom is displaced by only 0·03 Å out of the main coordination plane in the Cu–O(2^{ii}) direction. The O(2) atom makes two coordinative bonds with two adjacent atoms so the coordination polyhedra are joined in centrosymmetrical dimers in which a weak intermetallic interaction

Table 4 (*cont.*)

h	k	l	10F$_o$	10F$_c$	h	k	l	10F$_o$	10F$_c$	h	k	l	10F$_o$	10F$_c$	h	k	l	10F$_o$	10F$_c$	h	k	l	10F$_o$	10F$_c$	h	k	l	10F$_o$	10F$_c$	h	k	l	10F$_o$	10F$_c$
4	15	-2	79	-58	0	2	3	627	-649	1	9	3	291	318	5	16	-3	1n0	154	4	2	4	273	-246	4	9	4	48-	-24	6	16	4	104	-95
5	15	2	31	-33	1	2	3	120	-115	1	9	-3	281	325	6	16	-3	56	54	4	2	-4	401	443	4	9	-4	104	-89	1	18	4	64-	-56
5	15	-2	96	-84	1	2	-3	273	255	2	9	3	305	297	0	17	3	160	158	5	2	4	104	-90	5	9	4	31-	-7	1	18	-4	152	143
6	15	-2	87	-73	2	2	3	313	283	2	9	-3	152	190	1	17	3	249	267	5	2	-4	378	374	5	9	-4	136	127	2	18	4	48-	-43
0	16	2	273	-255	2	2	-3	200	189	3	9	3	112	82	1	17	-3	216	208	6	2	4	104	-124	6	9	-4	39-	26	2	18	-4	64-	48
1	16	2	160	-151	3	2	3	160	137	3	9	-3	281	268	2	17	3	273	269	6	2	-4	337	333	7	9	-4	48-	-6	3	18	4	136	-139
1	16	-2	305	-301	3	2	-3	31-	13	4	9	3	313	320	2	17	-3	71-	12	7	2	-4	64-	45	8	9	-4	23-	58	3	18	-4	337	337
2	16	2	208	-199	4	2	3	273	261	4	9	-3	56	-61	3	17	3	160	149	8	2	-4	39-	50	0	10	4	297	286	4	18	-4	241	238
2	16	-2	241	-243	4	2	-3	64	54	5	9	3	193	204	3	17	-3	56-	-4	0	3	4	168	-156	1	10	4	193	-189	5	18	-4	185	183
3	16	2	56-	-13	5	2	3	144	-127	5	9	-3	168	-160	4	17	3	128	137	1	3	4	64-	-7	1	10	-4	128	146	0	19	4	112	96
3	16	-2	200	-190	5	2	-3	200	-202	6	9	3	23-	28	4	17	-3	48-	-7	1	3	-4	241	-231	2	10	4	160	-146	1	19	4	144	135
4	16	2	104	81	6	2	3	39-	22	6	9	-3	297	-291	5	17	-3	128	-116	2	3	4	281	270	2	10	-4	361	368	1	19	-4	64-	-11
4	16	-2	233	-224	6	2	-3	23-	7	7	9	-3	273	-244	6	17	-3	96	-83	2	3	-4	208	-211	3	10	4	71	-47	2	19	4	87	92
5	16	2	136	134	7	2	-3	71-	62	0	10	3	136	123	0	18	3	136	-118	3	3	4	64-	-41	3	10	-4	208	220	2	19	-4	208	-201
5	16	-2	152	-154	8	2	-3	48-	38	1	10	3	64-	-39	1	18	3	79-	-6	3	3	-4	193	-190	4	10	4	289	-299	3	19	-4	48-	-35
6	16	-2	31-	-13	0	3	3	329	351	1	10	-3	144	149	1	18	-3	183	170	4	3	4	48-	18	4	10	-4	329	331	4	19	-4	48	51
0	17	2	112	-107	1	3	3	949	972	2	10	3	71	-65	2	18	3	87	84	4	3	-4	418	421	5	10	4	152	-160	5	19	-4	64	62
1	17	2	79	-68	1	3	-3	941	995	2	10	-3	273	285	2	18	-3	160	148	5	3	4	31-	0	5	10	-4	353	346	0	20	4	64-	2
1	17	-2	79-	27	2	3	3	619	620	3	10	3	71	61	3	18	3	56	52	5	3	-4	112	122	6	10	4	261	265	1	20	4	56-	-48
2	17	2	160	-153	2	3	-3	152	157	3	10	-3	386	408	3	18	-3	104	107	6	3	4	56	-64	7	10	-4	144	141	1	20	-4	144	123
2	17	-2	193	186	3	3	3	515	530	4	10	3	152	141	4	18	3	39	40	6	3	-4	39	-32	0	11	4	185	204	2	20	-4	71	-68
3	17	2	152	-146	3	3	-3	264	-241	4	10	-3	87	-84	4	18	-3	96	85	7	3	-4	64-	-3	1	11	4	79	72	2	20	-4	144	130
3	17	-2	104	-96	4	3	3	264	241	5	10	3	136	134	5	18	-3	144	123	8	3	-4	39-	-9	1	11	-4	185	-175	3	20	-4	168	173
4	17	2	104	-98	4	3	-3	168	-185	5	10	-3	176	172	6	18	-3	15-	-3	0	4	4	233	209	2	11	4	200	185	4	20	-4	193	205
4	17	-2	200	-183	5	3	3	39-	15	6	10	3	15-	8	0	19	3	136	135	1	4	4	87	-65	2	11	-4	71-	24	5	20	-4	87	110
5	17	2	64	-73	5	3	-3	361	-403	6	10	-3	39	38	1	19	3	370	350	1	4	-4	466	464	3	11	4	193	193	0	21	4	112	124
5	17	-2	104	-99	6	3	3	31-	-17	7	10	-3	79	-72	1	19	-3	79-	69	2	4	4	193	-173	3	11	-4	249	247	1	21	4	96	98
6	17	-2	39	-42	6	3	-3	136	-152	0	11	3	410	425	2	19	3	208	204	2	4	-4	523	522	4	11	4	39-	-13	1	21	-4	160	-147
0	18	2	297	-279	0	4	3	490	-421	1	11	3	386	387	2	19	-3	87	76	3	4	4	329	-302	4	11	-4	71	12	2	21	4	31-	77
1	18	2	79-	-29	1	4	3	56-	-38	1	11	-3	297	314	3	19	3	87	84	3	4	-4	555	614	5	11	4	31	-36	2	21	-4	48-	-11
1	18	-2	394	-373	1	4	-3	176	166	2	11	3	345	364	3	19	-3	56-	-28	4	4	4	337	-318	5	11	-4	200	-185	3	21	-4	128	-127
2	18	2	185	-182	2	4	3	233	217	2	11	-3	249	275	4	19	-3	96	-89	4	4	-4	426	464	6	11	-4	39-	10	4	21	-4	31	47
2	18	-2	71-	31	2	4	-3	208	196	3	11	3	249	250	5	19	-3	64	-68	5	4	4	241	-227	7	11	-4	96	93	0	22	4	48-	7
3	18	2	56-	20	3	4	3	176	154	3	11	-3	56-	13	0	20	3	120	-132	5	4	-4	322	322	0	12	4	200	214	1	22	4	31-	28
3	18	-2	176	-175	3	4	-3	39-	-24	4	11	3	193	187	1	20	3	96	-99	6	4	4	112	-142	1	12	4	79-	46	1	22	-4	64	59
4	18	2	31-	-27	4	4	3	48-	38	4	11	-3	42	12	1	20	-3	64-	42	6	4	-4	200	192	1	12	-4	249	247	2	22	-4	120	127
4	18	-2	208	-196	4	4	-3	87	99	5	11	3	112	110	2	20	3	152	141	8	4	-4	39-	5	2	12	4	160	-170	3	22	-4	128	144
5	18	-2	185	-188	5	4	3	39-	21	5	11	-3	305	-301	2	20	-3	208	213	0	5	4	322	292	2	12	-4	305	324	1	23	-4	56	-73
6	18	-2	15-	-1	5	4	-3	249	-246	6	11	-3	337	-310	3	20	3	56	54	1	5	4	64-	34	3	12	4	216	-233	2	23	-4	79	-89
0	19	2	87-	-61	6	4	3	64	60	7	11	-3	136	-132	3	20	-3	168	156	1	5	-4	273	245	3	12	-4	289	304	0	1	5	322	-291
1	19	2	193	-180	6	4	-3	31-	-14	0	12	3	168	158	4	20	-3	112	110	2	5	4	71-	-10	4	12	4	176	-184	1	1	5	370	-369
1	19	-2	144	124	8	4	-3	48-	47	1	12	3	79	-69	5	20	-3	64	-64	2	5	-4	547	-499	4	12	-4	216	212	1	1	-5	418	-445
2	19	2	152	-151	0	5	3	829	808	1	12	-3	112	125	0	21	3	168	152	3	5	4	208	-200	5	12	4	160	-177	2	1	5	208	-200
2	19	-2	71-	12	1	5	3	756	772	2	12	3	160	-159	1	21	3	313	303	3	5	-4	353	-331	5	12	-4	233	231	2	1	-5	821	-832
3	19	2	79	-69	1	5	-3	361	378	2	12	-3	241	258	1	21	-3	79	73	4	5	4	112	93	6	12	-4	257	239	3	1	5	185	-176
3	19	-2	56-	11	2	5	3	845	826	3	12	3	216	205	2	21	3	152	156	4	5	-4	233	220	7	12	-4	64	54	3	1	-5	507	-490
4	19	2	48	-41	2	5	-3	208	231	3	12	-3	176	171	2	21	-3	64-	-4	5	5	4	31-	-7	0	13	4	87-	44	4	1	5	48-	-9
4	19	-2	168	-147	3	5	3	281	276	4	12	3	48-	34	3	21	-3	39-	-21	5	5	-4	64	65	1	13	4	233	234	4	1	-5	233	-222
5	19	-2	64	-66	3	5	-3	79	-70	4	12	-3	160	157	4	21	-3	71	-77	6	5	4	15-	-10	1	13	-4	160	-164	5	1	5	39	39
0	20	2	136	-136	4	5	3	168	169	5	12	3	87	94	0	22	3	160	-164	6	5	-4	208	202	2	13	4	71-	25	5	1	-5	64	57
1	20	2	160	-134	4	5	-3	313	-336	5	12	-3	185	182	1	22	3	56-	-28	7	5	-4	64-	-84	2	13	-4	71-	-21	6	1	-5	112	93
1	20	-2	87	-61	5	5	3	104	81	6	12	-3	71	58	1	22	-3	56-	38	8	5	-4	39-	4	3	13	4	176	171	0	2	5	281	241
2	20	2	129	-119	5	5	-3	160	-171	7	12	-3	79	-81	2	22	3	48	48	0	6	4	98	104	3	13	-4	87	78	1	2	5	96	-77
2	20	-2	273	-251	6	5	3	31-	-17	0	13	3	627	615	2	22	-3	71	64	1	6	-4	79	-74	4	13	4	39	-15	1	2	-5	185	175
3	20	2	39-	23	6	5	-3	313	-327	1	13	3	79-	58	3	22	-3	112	135	1	6	-4	386	376	4	13	-4	160	-145	2	2	5	168	171
3	20	-2	208	-206	0	6	3	459	-428	1	13	-3	394	415	4	22	-3	56	73	2	6	4	71	-46	5	13	-4	39-	-49	2	2	-5	68	-47
4	20	2	39	49	1	6	3	249	-223	2	13	3	361	375	0	23	3	96	111	2	6	-4	434	443	6	13	-4	39-	20	3	2	5	71	68
4	20	-2	160	-159	1	6	-3	56	12	2	13	-3	152	154	1	23	3	120	120	3	6	4	257	-230	0	14	4	87-	-28	3	2	-5	104	77
5	20	-2	136	-136	2	6	3	401	407	3	13	3	401	408	1	23	-3	120	117	3	6	-4	401	436	1	14	4	120	122	4	2	5	128	-126
0	21	2	71-	-70	2	6	-3	128	-108	3	13	-3	56-	-46	2	23	3	39	43	4	6	4	401	-355	1	14	-4	176	175	4	2	-5	96	83
1	21	2	64-	-6	3	6	3	56-	-53	4	13	3	152	155	3	23	-3	31-	-20	4	6	-4	466	514	2	14	4	120	-119	5	2	5	23-	8
1	21	-2	64-	27	3	6	-3	128	-117	4	13	-3	257	-246	0	24	3	48	-65	5	6	4	249	-244	2	14	-4	401	409	5	2	-5	39-	13
2	21	2	168	-174	4	6	3	48-	-3	5	13	3	31-	1	1	24	-3	31-	30	5	6	-4	337	340	3	14	4	281	-285	6	2	-5	56	45
2	21	-2	120	106	4	6	-3	112	129	5	13	-3	168	-142	2	24	-3	31-	58	6	6	-4	200	184	3	14	-4	313	296	8	2	-5	39-	-39
3	21	2	128	-143	5	6	3	39-	4	6	13	-3	257	235	0	0	4	410	-343	8	6	-4	39-	32	4	14	4	120	-120	0	3	5	345	-339
3	21	-2	64	-54	5	6	-3	71	61	7	13	-3	128	-132	1	0	4	176	-158	0	7	4	200	198	4	14	-4	168	160	1	3	5	426	-405
4	21	-2	79	-74	6	6	3	39	47	0	14	3	200	197	1	0	-4	619	618	1	7	4	64-	49	5	14	-4	160	162	1	3	-5	394	-402
5	21	-2	7-	-57	6	6	-3	48	-36	1	14	3	112	101	2	0	4	507	-490	1	7	-4	264	-262	6	14	-4	208	193	2	3	5	313	-291
0	22	2	71-	-73	7	6	-3	136	123	1	14	-3	128	150	2	0	-4	418	436	2	7	4	87	-66	0	15	4	87-	10	2	3	-5	466	-481
1	22	2	152	-138	8	6	-3	39-	62	2	14	3	120	-114	3	0	4	466	-427	2	7	-4	128	-128	1	15	4	96	93	3	3	5	233	-225
1	22	-2	128	-111	0	7	3	692	b76	2	14	-3	152	166	3	0	-4	756	777	3	7	4	64-	-107	1	15	-4	144	-141	3	3	-5	281	-293
2	22	2	48-	-12	1	7	3	587	609	3	14	3	120	-114	4	0	4	193	193	3	7	-4	249	-229	2	15	4	87	78	4	3	5	71	66
2	22	-2	185	-169	1	7	-3	128	159	3	14	-3	112	106	4	0	-4	466	500	4	7	4	48-	6	2	15	-4	128	-140	4	3	-5	233	-227
3	22	2	23-	15	2	7	3	322	321	4	14	3	96	89	5	0	4	249	-239	4	7	-4	39-	24	3	15	4	79	84	5	3	5	23-	-35
3	22	-2	208	-216	2	7	-3	538	557	4	14	-3	96	94	5	0	-4	515	516	5	7	4	31-	28	3	15	-4	64-	-29	5	3	-5	39-	-24
4	22	-2	120	-126	3	7	3	224	212	5	14	3	56	56	6	0	4	104	-119	5	7	-4	104	97	4	15	4	31-	43	6	3	-5	39-	0
0	23	2	64-	-15	3	7	-3	48-	33	5	14	-3	264	252	6	0	-4	378	351	6	7	-4	39-	16	4	15	-4	168	-159	0	4	5	71-	30
1	23	2	87	-84	4	7	3	249	239	6	14	-3	39-	35	8	0	-4	48-	-11	7	7	-4	64-	45	5	15	-4	39-	30	1	4	5	79	-56
1	23	-2	56-	-33	4	7	-3	208	-220	7	14	-3	39-	-3	0	1	4	136	118	8	7	-4	31-	75	6	15	-4	39	46	1	4	-5	193	-164
2	23	2	79	-108	5	7	3	224	221	0	15	3	144	134	1	1	4	152	130	0	8	4	200	190	0	16	4	87-	24	2	4	5	71-	34
2	23	-2	48-	23	5	7	-3	64	67	1	15	3	394	396	1	1	-4	112	98	1	8	4	160	-157	1	16	4	120	-123	2	4	-5	144	116
3	23	-2	31-	-17	6	7	3	31-	-5	1	15	-3	305	287	2	1	4	64-	53	1	8	-4	208	216	1	16	-4	128	128	3	4	5	56-	14
0	24	2	120	-129	6	7	-3	289	-286	2	15	3	264	261	2	1	-4	104	-89	2	8	4	120	-112	2	16	4	193	-185	3	4	-5	193	182
1	24	2	64	-64	7	7	-3	329	-293	2	15	-3	128	134	3	1	4	96	-55	2	8	-4	152	173	2	16	-4	281	270	4	4	5	39-	-25
1	24	-2	64	-71	0	8	3	233	-218	3	15	3	345	337	3	1	-4	168	153	3	8	4	112	-97	3	16	4	96	-91	4	4	-5	104	-103
2	24	-2	112	-144	1	8	3	64-	-12	3	15	-3	64-	-23	4	1	4	71	58	3	8	-4	136	158	3	16	-4	264	263	5	4	5	23-	-5
1	25	-2	31-	-69	1	8	-3	87	-69	4	15	3	71	62	4	1	-4	31-	3	4	8	4	289	-288	4	16	4	71	-78	5	4	-5	64	-51
0	1	3	595	596	2	8	3	64-	27	4	15	-3	64	-41	5	1	4	39-	35	4	8	-4	684	739	4	16	-4	216	207	6	4	-5	48	-41
1	1	3	603	646	2	8	-3	104	107	5	15	3	23-	-20	5	1	-4	104	86	5	8	4	224	-239	5	16	-4	200	193	8	4	-5	39-	-1
1	1	-3	361	404	3	8	3	56-	-4	5	15	-3	249	-224	6	1	4	23-	-21	5	8	-4	442	442	6	16	-4	193	184	0	5	5	394	-386
2	1	3	571	579	3	8	-3	48-	86	6	15	-3	136	-127	6	1	-4	31-	-26	6	8	4	96	89	0	17	4	136	124	1	5	5	329	-309
2	1	-3	87	76	4	8	3	79	64	0	16	3	96	-80	7	1	-4	64-	-9	7	8	-4	185	173	1	17	4	152	141	1	5	-5	555	-560
3	1	3	636	633	4	8	-3	224	240	1	16	3	144	127	8	1	-4	39-	17	8	8	-4	31-	73	1	17	-4	128	111	2	5	5	378	-345
3	1	-3	168	163	5	8	3	39-	22	1	16	-3	193	207	0	2	4	64-	8	0	9	4	168	173	2	17	4	79	73	2	5	-5	401	-397
4	1	3	216	189	5	8	-3	112	-122	2	16	3	71-	-66	1	2	4	64-	-22	1	9	4	112	111	2	17	-4	71-	4	3	5	5	120	-108
4	1	-3	71	-81	6	8	3	104	112	2	16	-3	71-	-75	1	2	-4	120	90	1	9	-4	120	-118	3	17	4	39-	11	3	5	-5	160	-182
5	1	3	64	40	6	8	-3	144	145	3	16	3	96	82	2	2	4	365	-329	2	9	4	144	138	3	17	-4	152	-137	4	5	5	39-	-44
5	1	-3	313	-359	7	8	-3	64-	9	3	16	-3	128	124	2	2	-4	867	643	2	9	-4	64	35	4	17	-4	48	-25	4	5	-5	87	-96
6	1	3	56	58	8	8	-3	31-	-6	4	16	3	31-	-35	3	2	4	370	-342	3	9	4	79	60	5	17	-4	31-	13	5	5	5	23-	25
6	1	-3	224	-261	0	9	3	619	627	4	16	-3	185	185	3	2	-4	853	888	3	9	-4	64	-53	6	17	-4	71	64	5	5	-5	56	-44

[Cu$\cdots$Cu = 3·260 (2) Å is also present]. The dimers are joined in chains, running along [100], by phthalate bridges.

The benzene ring is planar, its least-squares plane being:

$$0{\cdot}2176X + 0{\cdot}2641Y - 0{\cdot}9396Z = -0{\cdot}9582.$$

Bond lengths and angles in it are quoted in Table 5.

The carboxyl groups, C(1)C(7)O(1)O(2) and C(2)C(8)O(3)O(4), are both planar, their least-squares planes being:

$$-0{\cdot}6943X - 0{\cdot}4803Y + 0{\cdot}5360Z = -2{\cdot}2905$$
$$-0{\cdot}7129X + 0{\cdot}5150Y + 0{\cdot}4759Z = \quad 1{\cdot}4166\ .$$

They are slightly rotated around their C–C bonds in the same direction, the dihedral angles they form with the benzene plane being 38·9 and 27·9° respectively. As observed in other cases (Nardelli, Fava & Giraldi, 1962) the two C–O distances in each carboxyl group are not equal, the longer one involves the oxygen atom lying in the main coordination plane.

The packing is determined by hydrogen bonds which the free oxygen atoms from the carboxyl groups form with the nitrogen atoms from the ammonia molecules [O(1)–N(1^{iii}) = 3·04(1), O(1)–N(2^{iii}) = 3·32(1), O(3)–N(1^{iii}) = 2·94 (1) and O(3)–N(2^{iv}) = 3·08 (1) Å] as shown in Fig. 2. Coordinates for derived positions are as follows:

Table 4 (*cont.*)

h	k	l	10F$_o$	10F$_c$	h	k	l	10F$_o$	10F$_c$	h	k	l	10F$_o$	10F$_c$	h	k	l	10F$_o$	10F$_c$	h	k	l	10F$_o$	10F$_c$	h	k	l	10F$_o$	10F$_c$
6	5	-5	39-	-14	1	13	-5	241	-227	3	1	-6	36-	33	0	9	6	160	-170	3	18	-6	36	-52	2	9	-7	128	115	1	4	8	152	-155
0	6	5	87-	19	2	13	5	224	-235	4	1	6	104	122	1	9	6	79-	7	4	18	-6	144	-153	3	9	-7	144	148	3	4	-8	56-	-18
1	6	5	264	-249	2	13	-5	241	-251	4	1	-6	71	-58	1	9	-6	64	67	0	19	6	39-	-15	4	9	-7	264	268	4	4	-8	39-	31
1	6	-5	120	125	3	13	5	79	-70	5	1	-6	36	59	2	9	6	64-	37	1	19	-6	87	84	5	9	-7	200	194	0	5	8	144	-184
2	6	5	87	69	3	13	-5	176	-183	6	1	-6	71	61	2	9	-6	64	60	2	19	-6	71	81	6	9	-7	31-	-1	1	5	8	39-	12
2	6	-5	64-	50	4	13	5	23-	-32	7	1	-6	39-	-50	3	9	6	39-	-24	3	19	-6	39	39	0	10	7	112	117	2	5	-8	64-	53
3	6	5	56-	-30	4	13	-5	79	-69	8	1	-6	23-	58	3	9	-6	96	95	4	19	-6	64	57	1	10	7	56-	-78	3	5	-8	56-	-55
3	6	-5	56-	-38	5	13	-5	39-	13	0	2	6	160	163	4	9	-6	48-	-52	1	20	-6	31	-31	4	10	-7	112	-94	4	5	-8	112	-113
4	6	5	39-	-11	6	13	-5	39-	15	1	2	6	264	243	5	9	-6	71	74	2	20	-6	48	52	2	10	7	39-	42	5	5	-8	31-	-39
4	6	-5	224	-232	7	13	-5	23-	68	1	2	-6	56	-57	6	9	-6	128	138	3	20	-6	15-	-93	3	10	-7	64-	-71	6	5	-8	31-	-50
5	6	5	23-	-27	0	14	5	87-	67	2	2	6	401	379	7	9	-6	31-	-34	0	1	7	281	280	3	10	-7	64-	-56	0	6	8	185	-171
5	6	-5	128	-118	1	14	5	193	-192	2	2	-6	87	-74	0	10	6	79	72	1	1	7	152	142	4	10	-7	79	94	1	6	8	144	-147
6	6	-5	39-	-25	1	14	-5	257	-85	3	2	6	241	226	1	10	6	313	319	1	1	-7	264	247	5	10	-7	31-	-4	1	6	-8	216	-218
7	6	-5	120	-109	2	14	5	160	-162	3	2	-6	329	-313	1	10	-6	128	108	2	1	7	64-	-37	6	10	-7	104	104	2	6	-8	128	-135
8	6	-5	31-	-20	2	14	-5	79	-73	4	2	6	112	125	2	10	6	152	164	2	1	-7	401	405	0	11	7	200	199	3	6	-8	56-	31
0	7	5	675	-690	3	14	5	96	-106	4	2	-6	160	-165	2	10	-6	64	-55	3	1	7	79	-92	1	11	7	56-	60	4	6	-8	96	95
1	7	5	434	-431	3	14	-5	112	-100	5	2	-6	216	-191	3	10	6	128	140	4	1	-7	233	218	1	11	-7	128	112	5	6	-8	31-	-10
1	7	-5	555	-548	4	14	-5	120	-117	6	2	-6	345	-300	3	10	-6	64	-63	5	1	-7	39-	36	2	11	7	31-	2	0	7	8	64-	-12
2	7	5	144	-133	5	14	-5	128	-120	0	3	6	112	123	4	10	-6	264	-256	6	1	-7	39-	51	2	11	-7	120	110	1	7	8	39-	9
2	7	-5	241	-227	6	14	-5	56	-55	1	3	6	79-	36	5	10	-6	499	-501	0	2	7	87-	85	3	11	-7	289	301	1	7	-8	64-	4
3	7	5	56-	48	0	15	5	120	-110	1	3	-6	96	93	6	10	-6	120	-118	1	2	7	64-	63	4	11	-7	185	189	2	7	-8	64-	-54
3	7	-5	249	-248	1	15	5	185	-171	2	3	6	160	-172	7	10	-6	48	-52	1	2	-7	120	-99	5	11	-7	144	149	3	7	-8	56-	13
4	7	5	48	-48	1	15	-5	233	-218	2	3	-6	71	-65	0	11	6	87-	-36	2	2	7	64-	-39	6	11	-7	96	112	4	7	-8	39-	-31
4	7	-5	87	-85	2	15	5	112	-113	3	3	6	56-	-12	1	11	6	64-	-60	2	2	-7	71-	41	0	12	7	64-	-50	5	7	-8	31-	-65
5	7	5	79	109	2	15	-5	394	-402	3	3	-6	48	-48	1	11	-6	79-	0	3	2	7	31-	15	1	12	7	56-	27	6	7	-8	23-	-42
5	7	-5	185	-182	3	15	-5	152	-171	4	3	6	31-	-23	2	11	6	64-	19	3	2	-7	64-	62	1	12	-7	64-	-58	0	8	8	71	-69
6	7	5	297	278	3	15	-5	128	-114	4	3	-6	48-	19	2	11	-6	104	105	4	2	-7	87	-87	2	12	7	31-	70	1	8	8	176	-207
7	7	-5	96	76	4	15	-5	48-	-34	5	3	-6	104	92	3	11	-6	48	53	5	2	-7	39-	25	2	12	-7	112	-113	1	8	-8	56-	-40
8	7	-5	23-	58	5	15	-5	31-	-28	6	3	-6	39-	32	3	11	-6	128	134	6	2	-7	39-	14	3	12	-7	56	-59	2	8	-8	144	-153
0	8	5	87-	1	6	15	-5	31-	32	7	3	-6	39-	38	4	11	-6	71	68	0	3	7	208	215	4	12	-7	39-	36	3	8	-8	43-	13
1	8	5	79-	-12	0	16	5	120	-116	8	3	-6	23-	26	5	11	-6	71	63	1	3	7	64-	-9	5	12	-7	31-	31	4	8	-8	87	-22
1	8	-5	193	171	1	16	5	152	-139	0	4	6	144	127	6	11	-6	48	-50	1	3	-7	144	140	6	12	-7	64	73	5	8	-8	136	151
2	8	5	128	-125	1	16	-5	185	-185	1	4	6	120	112	7	11	-6	23-	-17	2	3	7	64-	-6	0	13	7	64-	-37	6	8	-8	23-	80
2	8	-5	120	-113	2	16	5	152	-155	1	4	-6	281	280	0	12	6	200	193	2	3	-7	538	516	1	13	7	56	73	0	9	8	87	97
3	8	5	56-	19	2	16	-5	64-	-54	2	4	6	241	215	1	12	6	96	89	3	3	7	31-	-14	1	13	-7	176	163	1	9	8	31-	29
3	8	-5	56-	-41	3	16	5	31-	1	2	4	-6	185	-160	1	12	-6	96	97	4	3	-7	216	203	2	13	-7	185	196	1	9	-8	56-	41
4	8	5	56	-56	3	16	-5	79	-73	3	4	6	216	194	2	12	6	128	136	5	3	-7	168	168	3	13	-7	281	291	2	9	-8	48-	-11
4	8	-5	87	-83	4	16	-5	112	-106	3	4	-6	152	-141	2	12	-6	71	-58	6	3	-7	120	111	4	13	-7	152	153	4	9	-8	39-	-43
5	8	-5	200	-182	5	16	-5	128	-119	4	4	6	168	187	3	12	6	120	129	0	4	7	87	111	5	13	-7	87	85	5	9	-8	56	-57
6	8	-5	64	-63	6	16	-5	39	-36	4	4	-6	208	-211	3	12	-6	144	-144	1	4	7	96	94	6	13	-7	15-	78	6	9	-8	15-	-20
7	8	-5	48-	-30	0	17	5	112	-103	5	4	-6	257	-244	4	12	-6	112	-109	1	4	-7	79-	-102	0	14	7	48-	0	0	10	8	168	-189
0	9	5	667	-658	1	17	5	176	-162	6	4	-6	264	-233	5	12	-6	185	-185	2	4	7	48-	-31	1	14	7	31-	44	1	10	-8	79	-76
1	9	5	345	-355	1	17	-5	193	-191	7	4	-6	241	-279	6	12	-6	241	-237	2	4	-7	71-	24	1	14	-7	56-	34	2	10	-8	48-	-33
1	9	-5	547	-556	2	17	5	144	-143	0	5	6	56	38	0	13	6	71-	-36	3	4	7	31-	28	2	14	-7	71	-76	3	10	-8	39-	20
2	9	5	87	-89	2	17	-5	273	-263	1	5	6	96	-78	1	13	5	64-	-32	4	4	-7	48-	-1	3	14	-7	48-	-46	4	10	-8	31-	-7
2	9	-5	200	-199	3	17	-5	200	-186	1	5	-6	241	229	1	13	-6	87	76	5	4	-7	39-	-20	4	14	-7	71	79	5	10	-8	23-	93
3	9	5	56-	51	4	17	-5	104	-104	2	5	6	144	-147	2	13	6	48-	32	6	4	-7	39-	9	5	14	-7	23-	6	0	11	8	48-	23
3	9	-5	257	-267	5	17	-5	31-	41	2	5	-6	71-	-34	2	13	-6	241	264	0	5	7	87	71	0	15	7	112	142	1	11	-8	87	-90
4	9	5	31-	-1	0	18	5	64-	-8	3	5	6	48-	12	3	13	6	23-	10	1	5	7	64-	74	1	15	7	23-	50	2	11	-8	48-	-60
4	9	-5	241	-242	1	18	5	120	-108	3	5	-6	48	23	3	13	-6	56-	10	1	5	-7	370	354	1	15	-7	56-	39	4	11	-8	31-	5
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6	9	-5	112	98	2	18	5	64	-52	4	5	-6	87	-82	5	13	-6	31-	27	2	5	-7	281	272	3	15	-7	193	197	1	12	-8	96	-105
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2	10	5	71-	-37	0	19	5	136	-118	1	6	6	273	263	2	14	6	152	150	6	5	-7	104	92	2	16	-7	39-	9	1	13	-8	31-	-22
2	10	-5	71-	-37	1	19	5	144	-134	1	6	-6	71	-59	2	14	-6	79	-71	0	6	7	96	106	3	16	-7	31-	89	2	13	-8	39-	-40
3	10	5	136	-149	1	19	-5	128	-124	2	6	6	120	101	3	14	-6	112	-104	1	6	7	160	171	4	16	-7	48	67	3	13	-8	31-	-33
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4	10	-5	48	30	4	19	-5	87	-92	3	6	-6	144	-151	6	14	-6	185	-192	2	6	-7	71-	67	2	18	-7	23-	-30	3	14	-8	23-	32
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1	11	5	176	-160	2	20	-5	71	-78	7	6	-6	136	-127	2	15	-6	152	153	6	6	-7	79	77	4	0	-8	48-	-22	1	2	-9	39-	-4
1	11	-5	418	-424	3	20	-5	39-	-2	0	7	6	160	-171	3	15	-6	128	114	0	7	7	120	112	0	1	8	71-	-43	2	2	-9	48-	60
2	11	5	144	-145	4	20	-5	31	-43	1	7	6	152	-149	4	15	-6	112	109	1	7	7	64-	72	1	1	8	56-	-74	4	2	-9	31-	-13
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3	11	5	96	-92	1	21	-5	112	-110	2	7	6	64-	-25	6	15	-6	48	-62	2	7	7	87	98	2	1	-8	31-	16	0	3	9	39-	-45
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0	12	5	257	-246	2	0	6	555	529	6	7	-6	56	-43	4	16	-6	112	-112	0	8	7	71-	7	1	2	8	152	-140	0	5	9	23-	-25
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4	12	-5	87	-84	0	1	6	120	115	3	8	-6	136	-135	3	17	-6	23-	10	6	8	-7	71	-66	2	3	8	31-	45	3	7	-9	7-	-86
5	12	-5	39-	43	1	1	6	64	46	4	8	6	120	173	0	18	6	48-	61	0	9	7	200	174	3	3	-8	56-	8	1	8	-9	23-	-31
6	12	-5	87	-78	1	1	-6	56	-55	4	8	-6	345	-344	1	18	6	56	87	1	9	7	144	140	4	3	-8	48-	-74	2	8	-9	31-	17
7	12	-5	31-	-27	2	1	6	112	-94	5	8	-6	410	-405	1	18	-5	56-	-28	1	9	-7	297	284	6	3	-8	31-	-19	3	8	-9	31-	34
0	13	5	233	-228	2	1	-6	31	23	6	8	-6	185	-167	2	18	-5	71	-62	2	9	7	39-	-20	0	4	8	233	-221	4	8	-9	23-	62
1	13	5	249	-237	3	1	6	128	-124	7	8	-6	144	-134																				

Table 5. *Bond distances and angles*

(*a*) In the coordination polyhedron

Cu–N(1^{i})	1·992 (6) Å	N(2)–Cu–N(1^{i})	94·0 (0·3)°
Cu–N(2)	1·997 (8)	N(1^{i})–Cu–O(2^{i})	91·6 (0·2)
Cu–O(2^{i})	1·988 (5)	O(2^{i})–Cu–O(4)	86·5 (0·2)
Cu–O(4)	1·971 (5)	O(4)–Cu–N(2)	87·9 (0·2)
Cu–O(2^{ii})	2·313 (5)		
Cu–O(3)	2·799 (5)		

(*b*) In the phthalate ion

C(7)–O(1)	1·241 (8) Å	C(3)–C(4)	1·376 (12) Å
C(7)–O(2)	1·282 (8)	C(4)–C(5)	1·403 (14)
C(7)–C(1)	1·475 (8)	C(5)–C(6)	1·378 (8)
C(8)–O(3)	1·249 (10)	C(6)–C(1)	1·394 (10)
C(8)–O(4)	1·275 (7)	C(3)–H(1)	1·08 (1)
C(8)–C(2)	1·488 (10)	C(4)–H(2)	1·01 (1)
C(1)–C(2)	1·401 (12)	C(5)–H(3)	0·97 (1)
C(2)–C(3)	1·371 (9)	C(6)–H(4)	0·99 (1)

Table 5 (*cont.*)

O(1)-C(7)-O(2)	125·0 (0·5)°	C(5)-C(4)-C(3)	120·3 (0·6)°
O(1)-C(7)-C(1)	118·8 (0·6)	C(4)-C(3)-C(2)	120·7 (0·9)
O(2)-C(7)-C(1)	116·1 (0·5)	C(3)-C(2)-C(1)	120·2 (0·7)
C(7)-C(1)-C(6)	118·9 (0·7)	Cu—O(4)-C(8)	110·8 (0·6)
C(7)-C(1)-C(2)	122·0 (0·6)	Cu—O(2)-C(7)	126·3 (0·4)
O(3)-C(8)-O(4)	124·1 (0·6)	C(2)-C(3)-H(1)	116·0 (0·7)
O(3)-C(8)-C(2)	121·3 (0·5)	C(4)-C(3)-H(1)	122·6 (0·7)
O(4)-C(8)-C(2)	114·5 (0·6)	C(3)-C(4)-H(2)	119·1 (1·0)
C(8)-C(2)-C(1)	119·8 (0·5)	C(5)-C(4)-H(2)	120·2 (0·8)
C(8)-C(2)-C(3)	119·9 (0·8)	C(4)-C(5)-H(3)	121·1 (0·6)
C(2)-C(1)-C(6)	118·7 (0·6)	C(6)-C(5)-H(3)	120·2 (0·9)
C(1)-C(6)-C(5)	121·4 (0·8)	C(5)-C(6)-H(4)	119·8 (0·7)
C(6)-C(5)-C(4)	118·7 (0·7)	C(1)-C(6)-H(4)	118·8 (0·6)

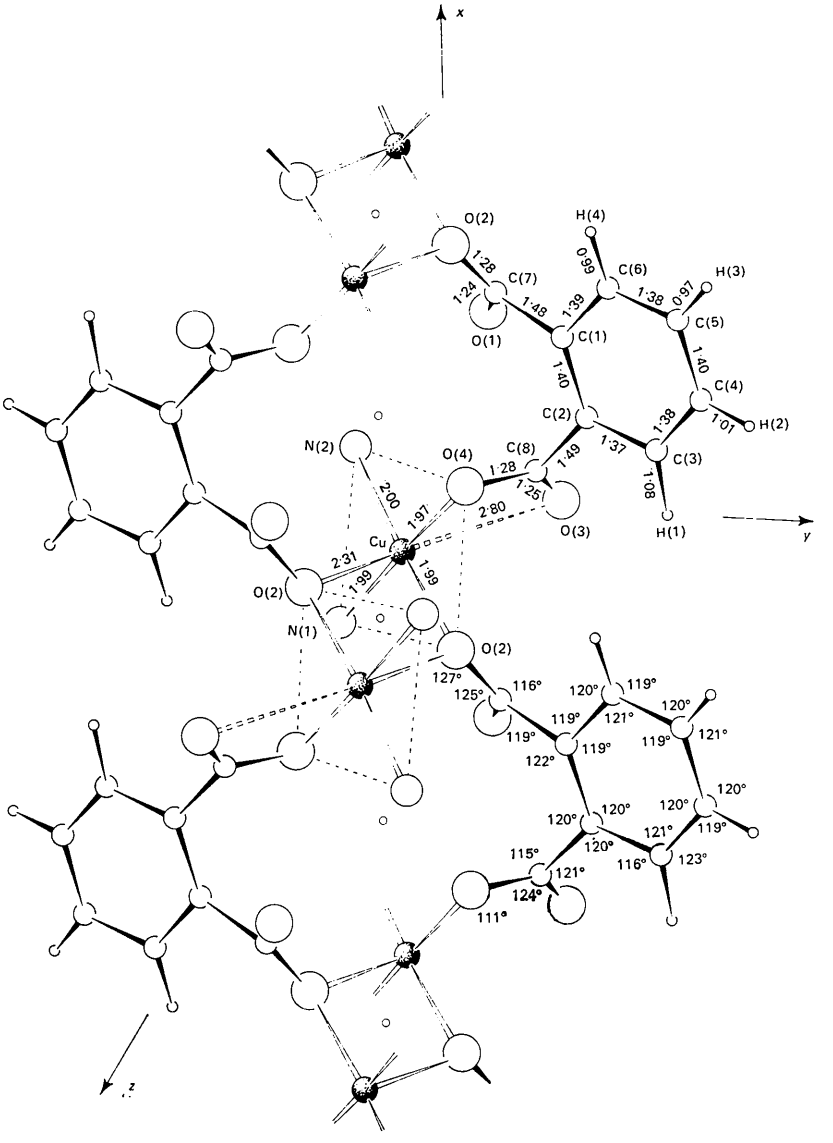

Fig. 1. Clinographic projection of the structure.

Table 6. Cu–O *and* Cu–N *distances in the main coordination plane of* Cu(II)-*complexes*

Compound	Cu–O	Cu–N	Literature
Bis(hydrogen *o*-phthalato)diaquocopper(II)	1·930 (8) Å 1·967 (8)		Biagini Cingi, Guastini, Musatti & Nardelli (1969)
Copper glutamate dihydrate	1·967 (4) 1·981 (4) 1·991 (4)	1·998 (4) Å	Gramaccioli & Marsh (1966)
Cu(II) succinate dihydrate	1·990 (7) 1·975 (6) 1·962 (7) 1·975 (7)		O'Connor & Maslen (1966)
Di-μ-hydroxobis(dimethylamine)copper(II) sulphate monohydrate	1·941 (15) 2·000 (15) 1·938 (15) 1·983 (15)	2·007 (19) 2·020 (19) 2·024 (19) 2·011 (19)	Iitaka, Shimizu & Kwan (1966)
Glycylglycylglycinocopper(II) chloride sesquihydrate	1·988 (7) 1·930 (7)	1·990 (8)	Freeman, Robinson & Schoone (1964)
Bis(ethylenediamine)copper(II) thiocyanate		2·01 (1) 1·99 (1)	Brown & Lingafelter (1964)
Bis(ethylenediamine)copper(II) nitrate		2·044 (13) 2·012 (13)	Komiyama & Lingafelter (1964)
Dipotassium bis(glycylglycinato)cuprate(II) hexahydrate		2·04 (1) 1·97 (1)	Sugihara, Tamaichi, Sasada & Kakudo (1968)
Bis(ethylenediamine)copper(II) fluoroborate		2·02 (1) 2·03 (1)	Brown, Lee & Melsom (1968)
Bis(diethylenetriamine)copper(II) bromide monohydrate		2·040 (23) 2·131 (24) 2·027 (26) 2·066 (25)	Stephens (1969)
Present paper	1·988 (5) 1·971 (5)	1·997 (8) 1·992 (6)	(1969)

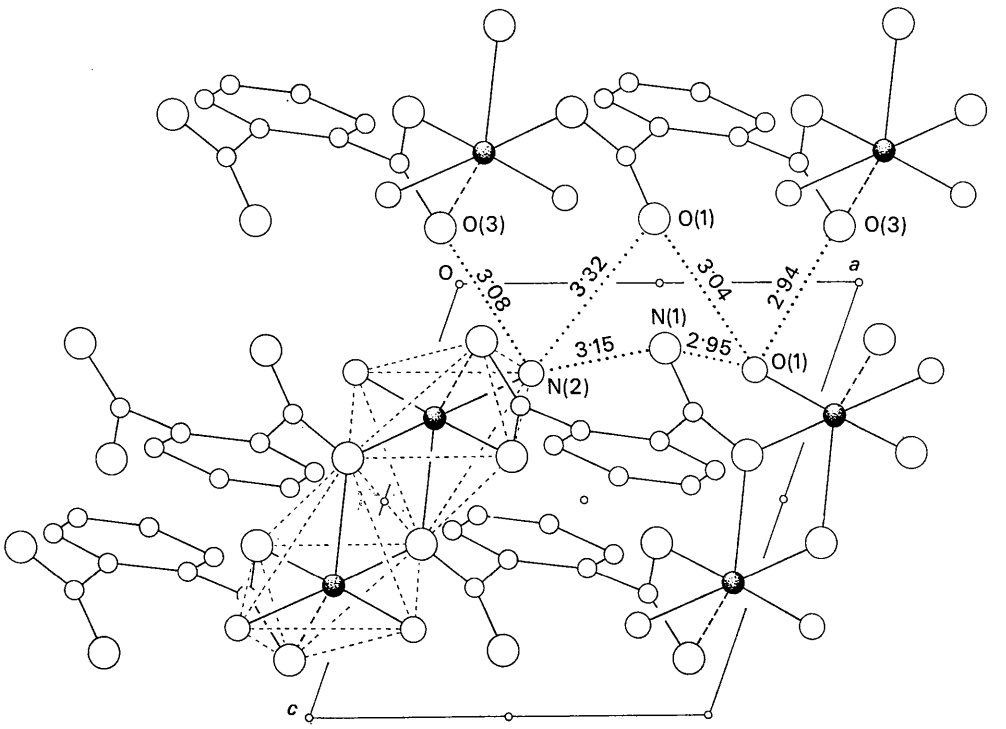

Fig. 2. Diagrammatic projection of the structure on (010).

i $x-1,y,z$
ii $1-x,\bar{y},1-z$
iii $1-x,\bar{y},\bar{z}$
iv $\bar{x},\bar{y},\bar{z}$

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Crystal Structure of the Praseodymium β-Diketonate of 2,2,6,6-Tetramethyl-3,5-heptanedione, $Pr_2(thd)_6$

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The crystal structure of the β-diketonate with empirical formula $Pr[(CH_3)_3C.CO.CH.CO.C.(CH_3)_3]_3$ has been determined from 5373 intensities measured visually using the multiple-film Weissenberg technique at room temperature. The crystals are monoclinic with space group $P2_1/n$ and cell constants $a=22{\cdot}28$ (6), $b=28{\cdot}51$ (7), $c=12{\cdot}56$ (5) Å, and $\beta=105°\pm30'$. Observed and calculated densities are 1·20 and 1·19 g.cm^{-3} respectively, for $Z=8$, *i.e.* with two crystallographically independent formula units. Refinement by full-matrix least-squares including 9 layer-line scale factors and individual isotropic temperature factors (329 parameters) converged to a conventional R of 0·133. The asymmetric unit consists of a dimer $Pr_2(thd)_6$ in which each of the Pr atoms is surrounded by 7 oxygen atoms in positions consistent with maximum repulsion. Two of the oxygen atoms are shared equally between Pr atoms. Only the methyl groups of neighbouring dimers are in loose van der Waals contact as evidenced by exceptionally high temperature factors for the methyl carbon atoms. This is interpreted as symptomatic of the known volatility of the thd lanthanides.

Introduction

The β-diketone $[(CH_3)_3.C.CO]_2CH_2$, H(thd), forms volatile lanthanide complexes with the general empirical formula $Ln(thd)_3$ (Eisentraut & Sievers, 1965). These complexes have been the subject of several crystallographic studies (*e.g.* Chen, 1967; Mode & Smith, 1969) which established that the complexes of the lighter lanthanides (La to Dy) are monoclinic and that those of Ho to Lu are orthorhombic. Work in this laboratory has been confined to the study of the crystallography of the lighter series and the detailed analysis of two of the structure types encountered. Part of the work is described in this paper, a preliminary report having been published previously (Erasmus & Boeyens, 1969).

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Crystallographic relationships

Sublimed samples of each of the La^{III} to Dy^{III} thd complexes were kindly supplied by Dr R. E. Sievers of Aerospace Research Laboratories, Dayton, Ohio. These were recrystallized from n-hexane, exposed to the atmosphere and found to constitute an isomorphous series with space group $P2_1/n$. The cell constants as determined from suitable oscillation, Weissenberg