these crystals and determined their unit cell and space group, to Dr B. C. Pratt of the Central Research Department, E. I. du Pont de Nemours & Company, for the sample, to the U.S. Air Force for their partial support of this work, to Maryellin Reinecke for the illustrations, and to the staff of the UCLA Computing Facility for their cooperation in the machine calculations.

References

ALMENNINGEN, A. & BASTIANSEN, O. (1958). *K. Norske Videnskab Selsk. Skr.* no. 4, 1.

ALMENNINGEN, A., BASTIANSEN, O. & TRAETTEBERG, M. (1958). *Acta Chem. Scand.* **12**, 1221.

BENSON, R. E. (1960). *J. Amer. Chem. Soc.* **82**, 5948.

BRASEN, W. R., HOLMQUIST, H. E. & BENSON, R. E. (1960). *J. Amer. Chem. Soc.* **82**, 995.

BRASEN, W. R., HOLMQUIST, H. E. & BENSON, R. E. (1961). *J. Amer. Chem. Soc.* **83**, 3125.

COX, E. G., CRUICKSHANK, D. W. J. & SMITH, J. A. S. (1958). *Proc. Roy. Soc.* A, **247**, 1.

CRUICKSHANK, D. W. J. (1956*a*). *Acta Cryst.* **9**, 754.

CRUICKSHANK, D. W. J. (1956*b*). *Acta Cryst.* **9**, 757.

EATON, D. R., PHILLIPS, W. D. & CALDWELL, D. J. (1963). *J. Amer. Chem. Soc.* **85**, 397.

HAMILTON, W. (1955). *Acta Cryst.* **8**, 185.

HOERNI, J. A. & IBERS, J. A. (1954). *Acta Cryst.* **7**, 744.

HUGHES, E. W. (1941). *J. Amer. Chem. Soc.* **63**, 1737.

International Tables for X-Ray Crystallography (1962). Vol. III. p. 202. Birmingham: Kynoch Press.

JACOBSON, R. A., WUNDERLICH, J. A. & LIPSCOMB, W. N. (1961). *Acta Cryst.* **14**, 598.

LEUNG, Y. C. & MARSH, R. E. (1958). *Acta Cryst.* **11**, 17.

MARSH, R. E., BIERSTEDT, R. & EICHHORN, E. L. (1962). *Acta Cryst.* **15**, 310.

REIN, R. & HARRIS, F. E. (1965). *J. Chem. Phys.* **42**, 2177.

ROBERTSON, J. M., SHEARER, H. M. M., SIM, G. A. & WATSON, D. G. (1962). *Acta Cryst.* **15**, 1.

ROBERTSON, G. B. (1961). *Nature, Lond.* **191**, 593.

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

ROMERS, C. (1964). *Acta Cryst.* **17**, 1287.

SASADA, Y. & NITTA, I. (1956). *Acta Cryst.* **9**, 205.

SASADA, Y. & NITTA, I. (1957). *Bull. Chem. Soc. Japan*, **30**, 62.

SCHOMAKER, V. & TRUEBLOOD, K. N. (1966). *Acta Cryst.* **21**, A 247.

SCHOMAKER, V. & TRUEBLOOD, K. N (1967). To be published.

SCHOMAKER, V., WASER, J., MARSH, R. E., & BERGMAN, G. (1959). *Acta Cryst.* **12**, 600.

STOICHEFF, B. P. (1954). *Canad. J. Phys.* **32**, 339.

TEMPLETON, D. H. (1960). *Z. Kristallogr.* **113**, 234.

WEBB, L. E. & FLEISCHER, E. B. (1965). *J. Chem. Phys.* **43**, 3100.

Acta Cryst. (1967). **23**, 156

The Crystal Structure of Phenylethynyl(isopropylamine)gold(I).

BY P. W. R. CORFIELD* AND H. M. M. SHEARER

Chemistry Department, The University, Durham, England

(*Received* 29 *October* 1966)

Phenylethynyl(isopropylamine)gold(I), i-$C_3H_7NH_2AuC{\equiv}CC_6H_5$, crystallizes in an orthorhombic cell with $a=17{\cdot}92$, $b=17{\cdot}15$, $c=7{\cdot}22$ Å, $Z=8$, space group *Pccn*. The structure was solved by the heavy atom method and refined by the method of least squares to an R value of 0·064, calculated on the 1032 observed reflexions. The gold atoms lie in infinite zigzag chains, extending along the direction of the c axis, with gold–gold distances of 3·72 Å along the chains and angles of 153°. The chains are related in pairs by the twofold axes through $\frac{1}{4}\frac{1}{4}0$ and $\frac{3}{4}\frac{3}{4}0$, the gold–gold distances between the chains being 3·27 Å. Each gold atom is bonded, in an almost linear manner, to a nitrogen and to an ethynyl carbon atom with these atoms lying nearly in the plane of the zigzag. The gold–nitrogen and gold–carbon distances are 2·03 and 1·94 Å respectively. The amino hydrogen atoms are directed approximately towards neighbouring ethynyl groups and may be engaged in hydrogen bond formation.

Introduction

The complexes formed by phenylethynylgold(I) with primary and secondary amines tend to be sparingly soluble in inert solvents. Molecular-weight measurements (cryoscopically in benzene) show the presence of associated species, the extent of association increasing with concentration. The n-octylamine complex has degrees of association in the range 3·1–4·1 for the concentration range 1·3–5·7 wt.%; the n-nonylamine complex in the range 2·6–4·1 (0·7–5·2 wt.%). The isopropylamine complex was not sufficiently soluble to permit such measurements.

The ethynyl stretching frequencies for these complexes lie in the range 2122–2125 cm^{-1} (in a potassium bromide disc) and those of bisphenylethynylmercury at 2149 cm^{-1} with a weaker band at 2117 cm^{-1}. The very slight lowering in the stretching frequencies of the gold complexes suggests that π-bonding between the

---

* Present address: Chemistry Department, Northwestern University, Evanston, Illinois, U.S.A.

metal and the acetylenes is not significantly greater than in the mercury compound and that the association does not come about in this way. The parent compound, phenylethynylgold(I), which is regarded as a coordination polymer with strong metal–ethynyl interactions, has a weak band at 1973 cm^{-1}, about 150 cm^{-1} less than in the amine complexes (Coates & Parkin, 1962).

The molecular association could be most simply examined in the case of the isopropylamine complex. Confirmation that this compound possesses the same structural arrangement as the more soluble higher amine complexes was obtained by comparison of the unit-cell dimensions of three of the complexes, and from similarities in their diffraction patterns. The unit-cell dimensions and space groups are shown in Table 1. The *b* and *c* axes have almost the same lengths and the increase in the value of *a* suggests that the hydrocarbon chains extend along this direction.

Table 1. *Unit-cell dimensions of complexes* $C_6H_5C{\equiv}CAuL$

L	*a*	*b*	*c*	*β*	*Z*	Space group
i-$C_3H_7NH_2$	17·9	17·2	7·2		8	*Pccn*
n-$C_5H_{11}NH_2$	22·0	17·0	7·2	98°	8	*P*2_1/*c*
n-$C_9H_{19}NH_2$	29·2	17·0	7·2		8	*Pccn*

Experimental

The compound crystallized from acetone solution as white needles elongated along **c**. The crystals are sensitive to light and deteriorate rapidly on exposure to X-radiation so that a freshly prepared specimen was used for each set of photographs.

The unit cell lengths *a* and *b* were obtained by a least-squares procedure from a Weissenberg photograph of the *hk*0 layer, calibrated with sodium chloride powder lines. The value of *c* was determined from a calibrated rotation photograph.

Crystal data

i-$C_3H_7NH_2AuC{\equiv}CC_6H_5$, $C_{11}H_{14}AuN$ M.W. 357·2
Orthorhombic
$a=17{\cdot}92\pm0{\cdot}01$, $b=17{\cdot}15\pm0{\cdot}02$, $c=7{\cdot}22\pm0{\cdot}01$ Å,
$V=2220$ Å^3, $D_m=2{\cdot}08$, $Z=8$, $D_x=2{\cdot}14$ g.cm^{-3}
Absorption coefficient for Cu *Kα* radiation, $\mu=245$ cm^{-1}.
The *hk*0 reflexions were observed when $h+k=2n$, the 0*kl* when $l=2n$ and the *h*0*l* when $l=2n$.
Space group *Pccn*.

The layers *hk*0–*hk*5 were recorded photographically using the equi-inclination Weissenberg technique and unfiltered copper radiation. The intensities of the *Kα* reflexions were estimated visually by comparison with a calibrated scale. The usual Lorentz and polarization factors were applied, together with empirical corrections for spot extension. The cross-sections of the crystals varied from 0·015 × 0·025 to 0·025 × 0·035 mm^2 and corrections for absorption were not applied. The intensities were placed on the same relative scale by recording portions of several levels on the same set of films. 1032 independent reflexions were observed.

The 0*kl* and *h*0*l* layers were recorded by the precession method. The reflexions were very distorted and were used to calculate the Patterson function in projection along **a** and **b** but, with the exception of the 002 and 004 reflexions, were not included in the subsequent refinement.

Structure determination

The position of the gold atom was found from the Patterson function in projection along the three crystal axes. In these projections, the function is equally satisfied with gold atoms in the positions x,y,z and $x,y,\frac{1}{4}-z$ but this ambiguity was removed when the three-dimensional data became available. Indeed, estimates of the coordinates of the gold atom, in good agreement with those obtained from the Patterson function and with the final coordinates, were obtained by direct inspection of the intensities. These fall below the minimum observable value when the contributions of the gold atoms to the structure factors become very small, *i.e.* when one of the trigonometrical functions in the structure factor equation approaches zero.

After one cycle of least-squares refinement of the coordinates of the gold atom, the *R* value was 0·23. An F_o–F_c synthesis then showed clearly the positions of the nitrogen and carbon atoms, resulting in an improved value of *R* of 0·125.

The atomic parameters were refined by least-squares methods, first with isotropic and then with anisotropic temperature parameters for all the atoms, to an *R* value of 0·073. At this stage, a comparison of the observed and calculated structure factors suggested that the scaling in two of the layers was in error by more than 5%. Rescaling of the layers and two further cycles of refinement gave rise only to small changes in the atomic parameters so that the effect of the rescaling on these was slight. During the final cycle, the mean shift in coordinates was 0·0025 Å with a maximum shift of 0·0072 Å, and only two parameter shifts were more than one-half of the corresponding estimated standard deviation. The final *R* value, based on the observed reflexions, was 0·064.

A final F_o–F_c synthesis showed that, near the gold and nitrogen atoms, there were ridges of electron density of height 1–2 e.Å^{-3} extending in the direction of **c**. Over the rest of the unit cell, the electron density was always between −0·6 and 0·7 e.Å^{-3}. Of the 13 sites, were the value was greater than 0·4 e.Å^{-3}, 10 were near positions which might be expected for hydrogen atoms. No allowance was made, however, for the hydrogen atoms.

The least-squares calculations, using the block-diagonal approximation, were carried out with the program written by Cruickshank, Pilling, Bujosa, Lovell & Truter (1961). In the final cycles the weighting was of the form

$$w=1/\{A+K|F_o|+B(K|F_o|)^2\} \text{ when } K|F_o|\geq 32$$

$$\text{and } w=C+D(K|F_o|) \qquad \text{when } K|F_o|<32$$

with $A=33\cdot6$, $B=0\cdot003$, $C=0\cdot0164$, $D=0\cdot00134$, $K=0\cdot99$.

The final positional parameters are quoted in Table 2 and the thermal parameters in Table 3. The observed and calculated structure factors are listed in Table 4. The unobserved reflexions were given zero weight in the refinement but few calculate at more than the minimum observable values. The scattering factors for nitrogen and carbon are those due to Berghuis, Haanappel, Potters, Loopstra, MacGillavry & Veenendaal (1955) and for gold those given by Thomas & Umeda (1957). In the case of gold, the form factors were modified (James, 1948) to make an approximate correction for dispersion.

Description

A perspective view of the structure is given in Fig. 1. The bond lengths and angles are shown in Fig. 2 and listed in Table 5. The non-bonding contacts are given in Table 6.

Table 2. *Atomic coordinates* (Å) *and their standard deviations* (Å × 10^3)

	x	*y*	*z*
Au	4·031 (1)	2·714 (1)	0·344 (1)
N	6·052 (13)	2·582 (14)	0·261 (16)
C(1)	2·102 (19)	2·757 (20)	0·496 (20)
C(2)	0·908 (21)	2·697 (20)	0·679 (20)
C(3)	−0·558 (19)	2·640 (21)	0·870 (22)
C(4)	−1·298 (22)	1·657 (21)	0·234 (23)
C(5)	−2·698 (21)	1·638 (24)	0·418 (26)
C(6)	−3·332 (21)	2·538 (25)	1·243 (29)
C(7)	−2·581 (20)	3·481 (22)	1·864 (28)
C(8)	−1·205 (21)	3·572 (23)	1·718 (25)
C(9)	6·695 (22)	1·236 (20)	0·677 (24)
C(10)	8·212 (22)	1·423 (26)	0·798 (24)
C(11)	6·314 (19)	0·199 (21)	−0·320 (24)

Table 3. *Thermal vibration tensor components*, U_{ij} (Å²)

The standard deviations are 0·0004–0·0006 for the gold and 0·010–0·023 for the carbon atoms.

	U_{11}	U_{22}	U_{33}	U_{12}	U_{23}	U_{13}
Au	0·0622	0·0767	0·0863	0·0048	−0·0015	−0·0007
N	0·060	0·062	0·052	−0·002	−0·001	0·009
C(1)	0·074	0·082	0·054	0·011	−0·006	0·005
C(2)	0·089	0·069	0·053	0·003	0·000	0·007
C(3)	0·072	0·076	0·064	0·009	0·012	0·008
C(4)	0·088	0·082	0·062	−0·006	0·005	−0·006
C(5)	0·076	0·102	0·101	−0·015	0·009	0·012
C(6)	0·074	0·120	0·111	0·025	0·023	0·022
C(7)	0·081	0·076	0·108	−0·005	−0·003	0·015
C(8)	0·083	0·095	0·069	0·018	−0·002	0·013
C(9)	0·082	0·067	0·103	0·016	0·001	−0·006
C(10)	0·074	0·115	0·088	0·020	−0·004	−0·016
C(11)	0·072	0·075	0·106	−0·008	−0·002	0·007

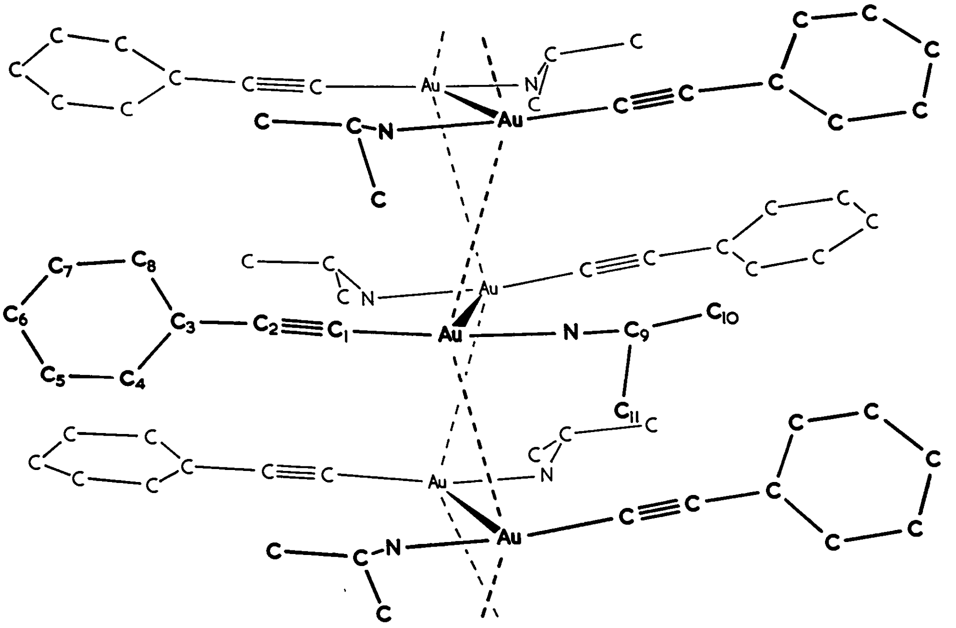

Fig. 1. A perspective view of the crystal structure.

Table 4. *Observed and calculated structure factors*

Successive columns give values of h, k, $|F_c|$, F_c.

L= 0

0	0	---	1328
0	2	282	-263
0	4	534	-490
0	6	517	456
0	8	51	-51
0	10	256	-230
0	12	207	189
0	14	54	57
0	16	120	-112
0	18	31	34
0	20	29	27
1	1	---	-455
1	3	134	-115
1	5	507	448
1	7	301	-269
1	9	168	-164
1	11	269	251
1	13	65	-61
1	15	86	-87
1	17	74	66
1	19	<17	-7
1	21	32	-28
2	0	422	-461
2	2	216	193
2	4	301	278
2	6	436	-387
2	8	17	14
2	10	227	211
2	12	145	-133
2	14	22	-26
2	16	79	77
2	18	33	-32
2	20	15	-17
3	1	402	410
3	3	14	20
3	5	369	-351
3	7	200	175
3	9	90	91
3	11	165	-184
3	13	45	46
3	15	61	63
3	17	67	-63
3	19	<16	-3
3	21	20	23
4	0	260	266
4	2	190	-180
4	4	264	-239
4	6	302	284
4	8	16	-15
4	10	184	-181
4	12	108	107
4	14	<22	11
4	16	71	-67
4	18	27	29
5	1	268	-265
5	3	43	-44
5	5	258	252
5	7	128	-127
5	9	55	-52
5	11	135	130
5	13	43	-40
5	15	37	-39
5	17	47	46
6	0	176	-194
6	2	118	113
6	4	138	131
6	6	154	-148
6	8	35	38
6	10	91	91
6	12	73	-72
6	14	<22	-1
6	16	43	42
6	18	22	-21
7	1	181	177
7	3	19	21
7	5	154	-159
7	7	87	90
7	9	40	41
7	11	84	-84
7	13	22	23
7	15	22	26
7	17	29	-27
8	0	87	87
8	2	21	-25
8	4	37	-35
8	6	80	80
8	8	31	-27
8	10	38	-39
8	12	38	42
9	1	59	-59
9	3	32	-33
9	5	33	34
9	7	29	-28
9	9	<20	-7
9	11	27	27
10	0	80	-74
10	2	<17	-3
10	4	<18	-2
10	6	<19	-4
10	8	20	12
11	1	44	-49
11	3	19	-18
11	5	19	16
12	0	88	-88
12	2	22	23
12	4	47	54
12	6	47	-50
12	8	<22	7
12	10	46	44
12	12	22	-27
12	14	<21	-15
12	16	18	14
13	1	74	77
13	3	25	28
13	5	79	-83
13	7	43	45
13	9	35	38
13	11	53	-55
13	13	<21	8
13	15	22	20
14	0	105	113
14	2	32	-35
14	4	49	-50
14	6	64	68
14	8	<22	-3
14	10	29	-33
14	12	26	25
14	14	<18	11
14	16	16	-19
15	1	78	-78
15	3	<22	-11
15	5	87	93
15	7	45	-49
15	9	49	-49
15	11	52	48
15	13	<19	-7
15	15	21	-22
16	0	108	-111
16	2	35	38
16	4	65	70
16	6	81	-86
16	8	<22	-3
16	10	55	53
16	12	39	-35
17	1	96	99
17	3	19	21
17	5	90	-94
17	7	48	47
17	9	26	33
17	11	54	-50
18	0	98	91
18	2	33	-39
18	4	59	-60
18	6	68	72
18	8	<19	-5
18	10	56	-50
18	12	34	32
19	1	74	-71
19	3	<20	-13
19	5	73	73
19	7	43	-38
19	9	24	-25
19	11	45	42
20	0	69	-66
20	2	25	29
20	4	45	44
20	6	58	-52
20	8	<15	7
20	10	36	34
21	1	49	44
21	3	<16	9
21	5	40	-40
21	7	26	23
22	0	41	41
22	2	15	-16
22	4	25	-24
22	6	26	32

L= 1

1	1	41	62
1	2	93	-100
1	3	197	-174
1	4	131	118
1	5	<16	3
1	6	<15	-6
1	7	60	65
1	8	28	-30
1	9	45	-46
1	10	<18	-4
1	11	<20	-2
1	12	<21	6
1	13	22	23
2	1	39	39
2	2	18	-29
2	3	22	-20
2	4	185	180
2	5	<16	-5
2	6	25	25
2	7	72	-72
2	8	116	-117
2	9	33	31
2	10	31	32
2	11	<20	2
2	12	31	35
2	13	<22	-13
2	14	23	-26
3	1	74	-79
3	2	85	84
3	3	222	198
3	4	46	-50
3	5	<17	-1
3	6	<17	-9
3	7	72	76
3	8	27	26
3	9	85	88
3	10	<20	-9
3	11	<22	-1
3	12	<23	-3
3	13	42	-45
3	14	<23	13
3	15	33	36
3	16	<23	1
3	17	<22	10
3	18	<21	-7
3	19	19	-17
4	1	49	47
4	2	217	214
4	3	49	-49
4	4	128	-129
4	5	23	19
4	6	54	-55
4	7	<19	14
4	8	115	119
4	9	24	-24
4	10	65	-66
4	11	<22	2
4	12	49	-48
4	13	<22	21
4	14	62	60
4	15	<23	-10
4	16	<23	-9
4	17	<22	-5
4	18	25	-25
5	1	168	159
5	2	94	-94
5	3	281	-262
5	4	52	50
5	5	63	65
5	6	39	41
5	7	166	162
5	8	72	-73
5	9	153	-147
5	10	23	23
5	11	<23	-17
5	12	34	30
5	13	84	89
5	14	29	-25
5	15	42	-36
5	16	<22	1
5	17	<22	-16
5	18	<20	8
5	19	18	26
6	1	35	-39
6	2	294	-275
6	3	75	78
6	4	208	190
6	5	<19	-21
6	6	65	68
6	7	30	-38
6	8	205	-199
6	9	44	48
6	10	75	73
6	11	<23	1
6	12	69	72
6	13	25	-24
6	14	77	-81
6	15	<25	11
6	16	<25	6
6	17	<24	5
6	18	28	28
6	19	<18	-8
6	20	21	-22
7	1	238	-224
7	2	106	99
7	3	327	307
7	4	62	-68
7	5	74	-76
7	6	28	-31
7	7	198	-200
7	8	62	66
7	9	164	161
7	10	28	-23
7	11	<23	12
7	12	35	-32
7	13	105	-107
7	14	31	26
7	15	44	46
7	16	<23	-2
7	17	<22	22
7	18	<19	-11
7	19	34	-33
8	1	78	74
8	2	369	339
8	3	115	-119
8	4	219	-212
8	5	<19	18
8	6	91	-90
8	7	50	51
8	8	226	220
8	9	52	-54
8	10	73	-79
8	11	<23	-5
8	12	78	-79
8	13	33	34
8	14	94	95
8	15	<24	-15
8	16	<25	-5
8	17	<23	-10
8	18	33	-35
8	19	<16	12
8	20	21	23
9	1	160	168
9	2	67	-69
9	3	300	-287
9	4	45	48
9	5	80	83
9	6	31	33
9	7	190	188
9	8	61	-65
9	9	161	-162
9	10	31	26
9	11	<21	-18
9	12	27	22
9	13	96	97
9	14	23	-24
9	15	49	-47
9	16	<21	5
9	17	<19	-19
9	18	<17	8
9	19	31	32
10	1	42	-45
10	2	260	-261
10	3	86	90
10	4	198	201
10	5	30	-28
10	6	74	78
10	7	49	-54
10	8	189	-185
10	9	37	40
10	10	64	66
10	11	<23	6
10	12	63	61
10	13	33	-25
10	14	74	-78
10	15	<22	15
10	16	<20	13
10	17	<18	6
10	18	32	33
11	1	116	-120
11	2	58	65
11	3	248	245
11	4	61	-66
11	5	55	-56
11	6	<23	-12
11	7	156	-145
11	8	47	49
11	9	131	134
11	10	<27	-14
11	11	<28	9
11	12	<27	-15
11	13	71	-76
11	14	22	21
11	15	44	46
12	1	34	35
12	2	186	173
12	3	53	-56
12	4	147	-152
12	5	<27	7
12	6	50	-50
12	7	53	57
12	8	136	140
12	9	40	-41
12	10	53	-56
12	11	<28	-3
12	12	51	-51
12	13	31	25
12	14	57	55
12	15	<22	-14
12	16	<20	-11
12	17	<18	-2
12	18	17	-22
13	1	87	87
13	2	44	-44
13	3	138	-142
13	4	<22	26
13	5	<26	16
13	6	<25	-1
13	7	63	63
13	8	<27	-15
13	9	78	-79
13	10	<27	14
13	11	<28	9
13	12	<26	4
13	13	45	47
13	14	<24	-12
13	15	32	-31
14	1	<22	-24
14	2	106	-102
14	3	25	30
14	4	74	76
14	5	<29	-7
14	6	<25	22
14	7	<27	-15
14	8	70	-75
14	9	<27	18
14	10	43	40
14	11	<27	1
14	12	32	28
14	13	<24	-13
14	14	38	-33
15	1	53	-49
15	2	<25	23
15	3	91	87
15	4	<23	-16
15	5	33	-30
15	6	<16	-10
15	7	59	-53
15	8	33	26
15	9	64	57
15	10	<27	-12
15	11	<27	-1
15	12	<27	-9
15	13	27	-31
16	1	<25	13
16	2	71	67
16	3	27	-27
16	4	47	-49
16	5	<25	10
16	6	<25	-13
16	7	<25	9
16	8	47	47
16	9	<25	-12
16	10	25	-23
16	11	<24	1
16	12	<24	-11
16	13	<24	5
16	14	28	24
17	1	29	26
17	2	<25	-11
17	3	44	-42
17	4	<25	12
17	5	<25	21
17	6	<25	4
17	7	32	28
17	8	<25	-11
17	9	32	-27
18	1	<25	-3
18	2	31	-26

L= 2

0	0	452	478
0	1	404	-374
0	2	64	-69
0	3	159	-141
0	4	329	-296
0	5	316	272
0	6	305	278
0	7	121	-114
0	8	<15	0
0	9	64	-66
0	10	170	-171
0	11	145	148
0	12	136	142
0	13	29	-29
0	14	21	20
0	15	53	-56
0	16	69	-72
0	17	37	40
0	18	27	27
0	19	<17	2
0	20	15	15
0	21	14	-16
1	0	262	-263
1	1	416	-390
1	2	121	111
1	3	27	-33
1	4	171	160
1	5	373	331
1	6	193	-181
1	7	208	-203
1	8	35	35
1	9	104	-107
1	10	114	120
1	11	150	161
1	12	69	-77
1	13	35	-32
1	14	<21	-20
1	15	53	-66
1	16	37	43
1	17	38	48
1	18	17	-16
1	19	<17	-2
1	20	<15	-9
1	21	20	-22
2	0	348	-355
2	1	165	160
2	2	154	148
2	3	12	12
2	4	186	185
2	5	188	-180
2	6	285	-262
2	7	92	94
2	8	26	22
2	9	61	64
2	10	134	144
2	11	83	-88
2	12	92	-97
2	13	21	19
2	14	21	-21
2	15	26	30
2	16	49	59
2	17	25	-28
2	18	19	-24
3	0	168	172
3	1	228	227
3	2	86	-85
3	3	94	64
3	4	109	-104
3	5	259	-249
3	6	163	152
3	7	112	108
3	8	<15	3
3	9	76	79
3	10	73	-76
3	11	120	-132
3	12	46	52
3	13	44	44
3	14	<21	7
3	15	37	43
3	16	30	-37
3	17	40	-44
4	0	227	229
4	1	147	-144
4	2	145	-148
4	3	11	-7
4	4	137	-141
4	5	188	156
4	6	200	193
4	7	84	-87
	8	33	-34
4	9	59	-59
4	10	101	-107
4	11	65	70
4	12	60	65
4	13	21	-21
4	14	<21	11
4	15	34	-29
4	16	42	-45
4	17	24	26
4	18	18	18
5	0	165	-164
5	1	157	-161
5	2	36	38
5	3	21	-18
5	4	84	85
5	5	153	162
5	6	101	-104
5	7	85	-86
5	8	<16	4
5	9	41	-43
5	10	49	52
5	11	88	93
5	12	48	-50
5	13	26	-27
5	14	<21	-4
5	15	26	-27
5	16	27	28
5	17	37	34
6	0	157	-155
6	1	92	86
6	2	76	77
6	3	<13	7
6	4	108	110
6	5	80	-77
6	6	112	-116
6	7	50	56
6	8	27	24
6	9	22	23
6	10	63	70
6	11	46	-44
6	12	49	-54
6	13	21	18
6	14	<21	-1
6	15	<21	17
6	16	29	30
6	17	19	-16
7	0	103	106
7	1	86	87
7	2	40	-42
7	3	20	16
7	4	64	-58
7	5	85	-86
7	6	80	79
7	7	56	55
7	8	<18	-6
7	9	19	16
7	10	40	-44
7	11	48	-52
7	12	26	29
7	13	21	21
7	14	<21	6
7	15	<21	12
7	16	<20	-16
7	17	19	-17
8	0	86	91
8	1	32	-30
8	2	36	-36
8	3	<15	-5
8	4	37	-33
8	5	26	27
8	6	62	67
8	7	25	-26
8	8	<19	-14
8	9	<20	-2
8	10	32	-33
8	11	25	26
8	12	26	28
9	0	41	-40
9	1	38	-40
9	2	<20	12
9	3	<16	-8
9	4	20	23
9	5	24	23
9	6	<18	-7
9	7	19	-24
10	0	17	-18
10	1	17	14
10	2	21	-20
11	0	31	-29
11	1	18	-16
11	2	<18	-4
11	3	26	-28
11	4	<19	10
11	5	19	22
11	6	<20	0
11	7	<20	5
11	8	<20	0
11	9	<21	-6
11	10	<21	-1
11	11	21	17
12	0	50	-49
12	1	40	42
12	2	23	21
12	3	19	21
12	4	31	29
12	5	32	-32
12	6	41	-39
12	7	<21	17
12	8	<21	5
12	9	<21	12
12	10	26	27
12	11	26	-27
13	0	57	59
13	1	58	57
13	2	20	-20
13	3	<20	4
13	4	35	-36
13	5	51	-48
13	6	39	38
13	7	26	30
13	8	<21	-6
13	9	21	16
13	10	26	-30
13	11	25	-25
13	12	27	28
14	0	73	74
14	1	36	-34
14	2	<21	-13
14	3	<21	-7
14	4	52	-50
14	5	37	34
14	6	52	52
14	7	<21	-16
14	8	<21	6
14	9	<21	-12
14	10	36	-36
14	11	20	19
14	12	27	28
14	13	<18	-3
14	14	<16	6
14	15	<15	-7
14	16	19	-16
15	0	40	-38
15	1	60	-61
15	2	26	25
15	3	<21	-6
15	4	34	31
15	5	63	62
15	6	40	-42
15	7	40	-38
15	8	<21	-2
15	9	25	-26
15	10	28	29
15	11	33	32
15	12	<18	-10
15	13	<16	-5
15	14	<15	-5
15	15	17	-16
16	0	83	-86
16	1	41	44
16	2	34	30
16	3	<21	9
16	4	50	50
16	5	50	-47
16	6	61	-62
16	7	21	20
16	8	<20	-1
16	9	20	17
16	10	40	38
16	11	25	-24
16	12	26	-24
17	0	51	57
17	1	56	62
17	2	21	-21
17	3	<21	16
17	4	39	-37
17	5	67	-64
17	6	40	39
17	7	31	32
17	8	<22	0
17	9	22	25
17	10	27	-26
17	11	43	-38
17	12	17	17
18	0	70	70
18	1	38	-37
18	2	32	-30
18	3	<20	-6
18	4	48	-45
18	5	41	38
18	6	56	55
18	7	25	-22
18	8	<17	-4
18	9	<16	-14
18	10	41	-35
18	11	25	22
18	12	34	22
19	0	42	-43
19	1	49	-50
19	2	<19	15
19	3	<22	-9
19	4	29	26
19	5	53	50
19	6	40	-33
19	7	32	-27
19	8	<15	1
19	9	17	-17
19	10	25	20
19	11	33	31
20	0	46	-47
20	1	27	28
20	2	21	20
20	3	<16	5
20	4	34	30
20	5	31	-27
20	6	40	-35
20	7	14	17
20	8	<12	5
20	9	13	10
21	0	32	26
21	1	35	32
21	2	<15	-8
21	3	<14	7
21	4	16	-14
21	5	39	-32
21	6	14	17
21	7	20	18
22	0	32	27
22	1	17	-16
22	2	11	-12
22	3	<10	-4
22	4	15	-15

L= 3

1	1	22	20
1	2	59	-58
1	3	<16	-13
1	4	41	42
1	5	23	-24
1	6	48	49
1	7	<22	-13
1	8	66	-64
1	9	<24	-7
1	10	<25	11
1	11	<27	11
1	12	<27	10
1	13	<28	4
1	14	28	-25
2	1	34	-31
2	2	51	-47
2	3	106	100
2	4	50	55
2	5	85	-91
2	6	<21	10
2	7	70	-62
2	8	46	-46
2	9	46	40
2	10	<25	20
2	11	<27	7
2	12	27	22
2	13	28	-20
3	1	28	-31
3	2	89	88
3	3	87	85
3	4	44	-47
3	5	19	-17
3	6	29	-30
3	7	35	-33
3	8	64	59
3	9	51	49
3	10	26	-21
3	11	<26	-1
3	12	<26	-13
3	13	28	-23
3	14	41	34
4	1	89	87
4	2	98	102
4	3	109	-109
4	4	70	-73
4	5	<20	15
4	6	45	-36
4	7	85	76
4	8	73	69
4	9	68	-67
4	10	32	-28
4	11	<27	2
4	12	27	-23
4	13	50	44
4	14	28	29
4	15	28	-21
5	1	48	51
5	2	142	-147
5	3	97	-99
5	4	107	110
5	5	36	35
5	6	31	31
5	7	61	63
5	8	104	-101
5	9	62	-61
5	10	41	38
5	11	<27	-3
5	12	45	43
5	13	41	35
5	14	45	-39
5	15	28	-19
5	16	<27	0
5	17	<25	-5
5	18	22	16
6	1	93	-93
6	2	138	-148
6	3	157	166
6	4	99	102
6	5	26	-27
6	6	43	39
6	7	94	-94
6	8	98	-100
6	9	92	94
6	10	33	34
6	11	<26	12
6	12	38	37
6	13	62	-59
6	14	41	-41
6	15	27	27
6	16	<26	2
6	17	<24	12
6	18	21	16
7	1	88	-96
7	2	169	180
7	3	134	136
7	4	120	-127
7	5	28	-30
7	6	55	-55
7	7	86	-93
7	8	126	126
7	9	71	75
7	10	48	-50
7	11	<28	0
7	12	50	-53
7	13	50	-50
7	14	53	49
7	15	27	27
7	16	<25	-4
7	17	<25	10
7	18	25	-22
8	1	106	114
8	2	133	137
8	3	175	-190
8	4	92	-104
8	5	24	18
8	6	35	-36
8	7	109	109
8	8	96	102
8	9	96	-104
8	10	35	-34
8	11	<28	-9
8	12	41	-35
8	13	65	65
8	14	40	42

Table 4 (*cont.*)

8	15	32	-32
8	16	<25	-3
8	17	<25	-15
8	18	19	-16
9	1	72	81
9	2	148	-163
9	3	131	-147
9	4	125	134
9	5	44	45
9	6	37	39
9	7	91	96
9	8	115	-117
9	9	74	-82
9	10	40	42
9	11	<28	-10
9	12	45	47
9	13	49	50
9	14	51	-47
9	15	25	-24
9	16	<25	5
9	17	<25	-10
9	18	24	22
10	1	76	-83
10	2	125	-131
10	3	140	153
10	4	70	84
10	5	32	-36
10	6	33	39
10	7	101	-103
10	8	81	-86
10	9	79	83
10	10	33	39
10	11	<28	5
10	12	31	30
10	13	51	-49
10	14	42	-42
10	15	30	31
11	1	61	-66
11	2	123	130
11	3	102	115
11	4	96	-105
11	5	24	-22
11	6	44	-45
11	7	59	-60
11	8	105	109
11	9	61	65
11	10	36	-36
11	11	<28	3
11	12	36	-34
11	13	37	-38
11	14	53	46
11	15	23	19
12	1	60	67
12	2	66	69
12	3	106	-116
12	4	52	-62
12	5	45	45
12	6	<28	-15
12	7	69	67
12	8	59	58
12	9	64	-60
12	10	28	-27
12	11	<27	-3
12	12	26	-21
12	13	44	39
12	14	23	24
13	1	44	44
13	2	76	-82
13	3	72	-79
13	4	40	41
13	5	<28	10
13	6	<28	23
13	7	45	44
13	8	53	-52
13	9	45	-43
13	10	34	29
13	11	<27	0
13	12	<24	16
13	13	24	25
13	14	34	-31
14	1	35	-39
14	2	41	-47
14	3	54	57
14	4	45	46
14	5	<28	-16
14	6	<28	9
14	7	35	-34
14	8	45	-40
14	9	38	40
14	10	<24	20
14	11	<24	-2
14	12	<24	11
14	13	27	-23
15	1	<28	-22
15	2	50	52
15	3	36	39
15	4	43	-45
15	5	<28	-14
15	6	<28	-6
15	7	30	-24
15	8	35	38
15	9	26	23
15	10	<25	-17
15	11	<25	-2
15	12	<24	-14
15	13	<24	-13
15	14	20	15
16	1	28	23
16	2	35	31
16	3	49	-43
16	4	<27	-20
16	5	<27	7
16	6	<26	-5
16	7	22	26
16	8	25	20
16	9	30	-27
17	1	<28	14
17	2	27	-23
17	3	<27	-20
17	4	26	24
17	5	<26	9
17	6	<26	4
17	7	<26	13
17	8	26	-20

L= 4

0	0	110	99
0	1	302	-248
0	2	57	-54
0	3	79	-74
0	4	33	-32
0	5	260	221
0	6	74	70
0	7	108	-104
0	8	<29	-7
0	9	69	-59
0	10	19	-19
0	11	135	126
0	12	20	19
0	13	38	-32
0	14	<24	3
0	15	48	-47
0	16	<19	-18
0	17	38	41
1	0	232	-262
1	1	72	-69
1	2	127	117
1	3	24	-13
1	4	190	167
1	5	104	93
1	6	213	-198
1	7	51	-46
1	8	25	21
1	9	42	-42
1	10	133	129
1	11	45	46
1	12	73	-78
1	13	<28	-14
1	14	26	-21
1	15	<24	-15
1	16	50	48
1	17	17	15
1	18	17	-18
2	0	111	-115
2	1	175	179
2	2	37	37
2	3	29	23
2	4	72	71
2	5	204	-178
2	6	81	-83
2	7	102	101
2	8	<26	9
2	9	61	58
2	10	47	52
2	11	95	-98
2	12	36	-39
2	13	28	23
2	14	<24	-7
2	15	34	36
2	16	21	20
2	17	30	-33
3	0	184	196
3	1	95	99
3	2	70	-69
3	3	25	22
3	4	128	-120
3	5	62	-60
3	6	143	141
3	7	33	36
3	8	<20	-4
3	9	<18	5
3	10	79	-80
3	11	35	-36
3	12	60	60
3	13	<28	11
3	14	<24	9
3	15	23	16
3	16	36	-38
4	0	40	41
4	1	123	-129
4	2	22	-19
4	3	<18	-12
4	4	36	-35
4	5	133	136
4	6	37	39
4	7	75	-82
4	8	<18	-9
4	9	51	-52
4	10	33	-32
4	11	69	71
4	12	28	23
4	13	19	-21
4	14	<24	0
4	15	23	-28
4	16	<18	-10
4	17	20	25
5	0	139	-150
5	1	38	-38
5	2	52	50
5	3	<23	3
5	4	72	72
5	5	43	46
5	6	103	-108
5	7	31	-33
5	8	<19	9
5	9	<19	-13
5	10	53	53
5	11	27	27
5	12	42	-47
5	13	<24	-5
5	14	<24	-6
5	15	<19	-5
5	16	25	28
6	0	43	-45
6	1	87	95
6	2	26	20
6	3	24	18
6	4	30	27
6	5	86	-94
6	6	28	-26
6	7	52	57
6	8	<18	7
6	9	31	31
6	10	<27	14
6	11	49	-52
6	12	<24	-14
6	13	<24	20
6	14	<23	1
6	15	<19	18
6	16	<16	9
6	17	16	-18
7	0	69	77
7	1	22	22
7	2	26	-28
7	3	<23	2
7	4	39	-40
7	5	<20	-13
7	6	54	57
7	7	<18	11
7	8	<19	-12
7	9	<19	-3
7	10	34	-31
7	11	<24	-10
7	12	24	28
7	13	<24	8
7	14	<19	1
7	15	<18	2
7	16	15	-14
8	0	<27	16
8	1	47	-53
8	2	<23	-8
8	3	<23	-5
8	4	<23	-12
8	5	39	43
8	6	25	25
8	7	31	-31
8	8	<23	-1
8	9	<27	-8
8	10	<27	-12
8	11	31	28
11	0	31	-33
12	0	<30	11
12	1	37	36
12	2	<30	5
12	3	<30	7
12	4	<30	-5
12	5	30	-31
13	0	44	42
13	1	<31	0
13	2	<31	-18
13	3	<31	1
13	4	31	-29
13	5	<31	-11
13	6	32	33
13	7	<30	3
13	8	<30	-1
13	9	<30	7
13	10	28	-25
14	0	37	35
14	1	43	-44
14	2	<31	-10
14	3	<31	-13
14	4	<31	-15
14	5	43	44
14	6	<30	16
14	7	<30	-18
14	8	<29	-2
14	9	<28	-15
14	10	<27	-6
14	11	26	25
15	0	42	-43
15	1	<30	-20
15	2	29	34
15	3	<30	-7
15	4	32	31
15	5	<30	19
15	6	41	-40
15	7	<28	-11
15	8	<27	2
15	9	<26	-8
15	10	31	28
16	0	<30	-22
16	1	47	49
16	2	<29	7
16	3	<29	11
16	4	<29	14
16	5	53	-50
16	6	<27	-18
16	7	<27	21
16	8	<25	1
16	9	24	20
16	10	<22	12
16	11	33	-27
17	0	58	55
17	1	<27	19
17	2	<27	-21
17	3	<27	2
17	4	35	-35
17	5	<32	-18
17	6	43	41
17	7	<24	11
17	8	<23	-2
17	9	<21	6
17	10	31	-27
18	0	<26	21
18	1	46	-42
18	2	<25	-9
18	3	<25	-8
18	4	<24	-15
18	5	52	44
18	6	<22	16
18	7	<27	-24
18	8	<18	0
18	9	19	-17
19	0	52	-46
19	1	<22	-13
19	2	<22	16
19	3	<22	-2
19	4	29	26
19	5	<19	13
19	6	35	-34
20	0	<18	-14
20	1	31	28
20	2	<18	6
20	3	<17	6
20	4	<15	9
20	5	24	-28

L= 5

1	1	<11	1
1	2	34	-37
1	3	15	18
1	4	19	17
1	5	<19	-12
1	6	<18	-2
1	7	23	-24
2	1	19	-25
2	2	<15	7
2	3	43	38
2	4	<17	-15
2	5	19	-18
2	6	<18	-6
2	7	46	-47
2	8	<17	9
2	9	31	36
2	10	<20	3
2	11	<21	3
2	12	<23	-1
2	13	25	-21
3	1	<13	-3
3	2	66	68
3	3	16	9
3	4	51	-50
3	5	<17	-4
3	6	19	-12
3	7	<16	-7
3	8	44	47
3	9	<20	10
3	10	21	-20
3	11	<21	2
3	12	19	-11
3	13	<20	-4
3	14	25	23
4	1	51	52
4	2	<16	4
4	3	91	-81
4	4	<22	-8
4	5	23	22
4	6	<18	2
4	7	50	48
4	8	<19	1
4	9	41	-39
4	10	<21	-2
4	11	<21	-2
4	12	<21	2
4	13	34	27
4	14	<19	2
4	15	14	-14
5	1	<17	1
5	2	79	-79
5	3	<19	-8
5	4	65	63
5	5	<19	5
5	6	24	25
5	7	<19	7
5	8	65	-68
5	9	<20	-6
5	10	25	26
5	11	<21	1
5	12	29	26
5	13	<20	1
5	14	29	-28
6	1	71	-68
6	2	<19	-6
6	3	126	119
6	4	<20	7
6	5	23	-18
6	6	<19	4
6	7	67	-70
6	8	<20	-8
6	9	61	67
6	10	<21	2
6	11	<21	7
6	12	<21	1
6	13	51	-41
6	14	<19	-3
6	15	19	19
7	1	<23	-9
7	2	117	117
7	3	<24	13
7	4	86	-84
7	5	<21	-4
7	6	32	-35
7	7	<20	-3
7	8	79	81
7	9	<21	1
7	10	30	-33
7	11	<21	-3
7	12	40	-35
7	13	<19	-2
7	14	37	36
8	1	66	67
8	2	<20	3
8	3	124	-125
8	4	<21	-11
8	5	25	24
8	6	<20	-2
8	7	65	71
8	8	<21	12
8	9	65	-67
8	10	<21	-3
8	11	<21	-8
8	12	<20	-2
8	13	47	42
8	14	<16	3
8	15	21	-21
9	1	<21	5
9	2	105	-107
9	3	<22	-12
9	4	84	87
9	5	<22	5
9	6	37	37
9	7	<21	10
9	8	87	-92
9	9	<21	-9
9	10	29	33
9	11	<20	-1
9	12	39	35
9	13	<18	5
9	14	37	-36
10	1	62	-68
10	2	26	-22
10	3	103	114
10	4	<26	6
10	5	28	-25
10	6	<21	2
10	7	62	-67
10	8	<21	-3
10	9	56	60
10	10	<20	6
10	11	<19	2
10	12	<19	4
10	13	41	-39
10	14	<14	-5
10	15	21	22
11	1	<21	-2
11	2	83	92
11	3	<23	-2
11	4	60	-67
11	5	<21	3
11	6	25	-22
11	7	<21	4
11	8	66	65
11	9	<20	3
11	10	22	-26
11	11	<19	-2
11	12	22	-24
11	13	<14	-2
11	14	31	30
12	1	34	38
12	2	<21	3
12	3	56	-62
12	4	<21	-4
12	5	21	19
12	6	<21	0
12	7	46	48
12	8	<20	5
12	9	45	-44
12	10	<19	-3
12	11	<18	0
12	12	<14	-1
12	13	29	26
13	1	<21	3
13	2	61	-63
13	3	<21	-11
13	4	43	43
13	5	<25	6
13	6	<20	13
13	7	<20	6
13	8	42	-39
13	9	<19	-5
13	10	22	18
14	1	26	-24
14	2	<20	-5
14	3	46	44
14	4	<20	8
14	5	22	-16
14	6	<19	3
14	7	35	-27
14	8	<18	-7
14	9	36	28
15	1	<20	0
15	2	30	31
15	3	<20	1
15	4	26	-25
15	5	<19	-2
15	6	<18	-10
15	7	<18	0
15	8	30	28
16	1	20	17
16	2	<17	0
16	3	32	-30
16	4	<16	2
16	5	<17	7
16	6	<17	2
16	7	17	12
16	8	<15	-2
16	9	20	-17
17	1	<18	2
17	2	20	-18
17	3	<14	-1
17	4	23	17
17	5	<15	0
17	6	<16	4
17	7	<16	0
17	8	13	-15
18	1	<17	-5
18	2	<15	2
18	3	12	13

The gold atoms lie in infinite zigzag chains, extending along the direction of the *c* axis, with the plane of the zigzag parallel to (010). The gold–gold distances along the chains are 3·72 Å with angles of 153°. The chains are grouped in pairs around the twofold axes through $\frac{1}{4}\frac{1}{4}0$ and $\frac{3}{4}\frac{3}{4}0$ with gold–gold distances of 3·27 Å between the chains. Each gold atom is almost linearly coordinated to a nitrogen and to an ethynyl carbon atom which are situated nearly in the plane of the chain of gold atoms.

Thus each gold atom has a nitrogen and a carbon atom as near neighbours and three gold atoms as more distant ones. The five atoms lie at five of the corners of an irregular octahedron, the angles being distorted by the zigzag of the chain. In addition, there are also three nitrogen atoms at distances similar to the gold–gold ones. The arrangement results in the gold atoms being closer to nitrogen atoms of adjacent molecules than to ethynyl carbons.

The gold–gold distances are much greater than in the metal (2·884 Å). Rundle (1954) postulated weak gold–gold bonding in Au(III)(DMG)$_2$Au(I)Cl$_2$ (where HDMG represents dimethylglyoxime) at distances of 3·26 Å, so completing a square planar arrangement around Au(I). In the present work a simple description of the bonding in such terms does not seem possible, though it is noticed that the gold–gold and the gold–nitrogen contacts between the molecules are amongst the shortest observed. The tendency shown by Au(I)

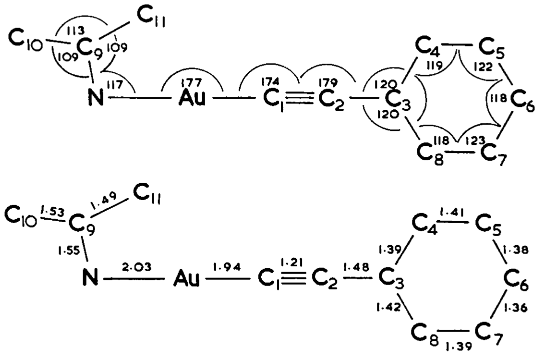

Fig. 2. Bond lengths and angles.

towards twofold coordination is discussed by Orgel (1960).

The Au–C(1) distance of 1·94 ± 0·02 Å can be compared with the value of 2·12 ± 0·14 Å found in KAu(CN)$_2$ (Rosenzweig & Cromer, 1959). It is shorter than the Ag–C distance of 2·040 ± 0·013 Å in phenylethynyl(trimethylphosphine)silver(I) (Corfield & Shearer, 1966*a*) possibly owing to greater end-on π-bonding between the metal and the acetylene in the gold complex, where the other attached atom is nitrogen. The bond Au–N has a length of 2·03 Å. A covalent radius of 1·33 Å for linearly coordinated gold(I) may be deduced from the Au(I)–Cl bond lengths of 2·31 Å in Cs$_2$Au(I)Au(III)Cl$_6$ (Elliott & Pauling, 1938) and the length of 2·33 Å in AuCl.PCl$_3$ (Arai, 1962). The covalent radius of nitrogen is 0·70 Å (Pauling, 1960) so that the Au–N distance is as expected. The angle N–Au–C(1) is 176·8 ± 0·7° and the coordination at Au departs slightly from linearity.

The carbon–carbon bond lengths are normal, the C(1)–C(2) distance of 1·21 Å being as expected for a triple bond whilst the C(2)–C(3) distance of 1·48 Å is very similar to the corresponding values in the trimethylphosphine complexes of phenylethynylsilver(I) and phenylethynylcopper(I) (Corfield & Shearer, 1966*a*,*b*). The angle Au–C(1)–C(2) is 174·2° and the distortion from linearity is in a direction almost perpendicular to the plane of the carbon atoms, as this plane makes an angle of 84° with the one through Au, C(1) and C(2). The angle C(1)–C(2)–C(3) does not differ significantly from 180°.

The bond lengths in the benzene ring have a mean value of 1·389 Å with a mean value of 120·0° in ring angle and none of the individual values differs significantly from its mean. If the ring is assumed to be regular, the statistical standard deviations in bond lengths and angles are 0·023 Å and 2·0°, somewhat less than the mean values of 0·032 Å and 2·1° obtained from the least-squares estimates of the coordinate standard deviations. The latter values are preferred since they are based on the experimental errors in a more satisfactory manner. The phenyl carbon atoms are accurately coplanar, the greatest deviation (0·011 Å) from the mean plane being found for C(4). C(1) and C(2) are at distances of 0·040 and 0·010 Å respectively but Au and N are considerably displaced, with deviations of −0·106 and −0·371 Å.

In the isopropyl group, the bonds C(9)–C(10) and C(9)–C(11) are normal. The distance N–C(9) is 1·55 ± 0·026 Å and the difference between this and the expected value of 1·48 Å (Sutton, 1965) is significant at the 1% probability level. The valence angles at C(9) are close to the tetrahedral value but, as might be expected, the angle Au–N–C(9) is considerably greater, being 117·2°.

Discussion

The distances from gold atoms to ethynyl carbons of neighbouring molecules are all greater than 4·2 Å, and rule out the possibility of π-bonding between them. Apart from the contacts involving the gold atoms, the closest approach distances between molecules are between nitrogen and ethynyl carbon atoms. The distance N–C(1′), between the chains, is 3·34 Å and N–C(1‴), along the chain, is 3·48 Å, using the same notation as

Table 6. *Non-bonding contacts*

′	$\frac{1}{2}-x$	$\frac{1}{2}-y$	z
″	$\frac{1}{2}-x$	y	$\frac{1}{2}+z$
‴	$\frac{1}{2}-x$	y	$-\frac{1}{2}+z$
iv	x	$\frac{1}{2}-y$	$\frac{1}{2}+z$
v	x	$\frac{1}{2}-y$	$-\frac{1}{2}+z$
vi	$\frac{1}{2}+x$	$-y$	$\frac{1}{2}-z$
vii	$-\frac{1}{2}+x$	$-y$	$\frac{1}{2}-z$
viii	$1-x$	$-y$	$-z$

Au—Au′	3·274 Å	C(2)—C(10″)	3·94 Å
Au—Au″	3·722	C(2)—C(10‴)	3·72
Au—C(11)	3·46	C(3)—C(8$^{\text{v}}$)	3·69
Au—N′	3·47	C(3)—C(10″)	3·96
Au—N″	3·70	C(4)—C(8$^{\text{v}}$)	3·97
Au—N‴	3·86	C(5)—C(9$^{\text{vi}}$)	3·84
N—C(1′)	3·34	C(5)—C(11$^{\text{vi}}$)	3·97
N—C(1″)	3·93	C(6)—C(11$^{\text{vii}}$)	3·90
N—C(1‴)	3·48	C(7)—C(7$^{\text{iv}}$)	3·95
N—C(2′)	3·88	C(8)—C(8$^{\text{iv}}$)	3·88
N—C(2‴)	3·77	C(8)—C(10″)	3·96
C(1)–C(9‴)	3·75	C(10)–C(10$^{\text{viii}}$)	3·59
C(1)–C(10‴)	3·82	C(10)–C(11$^{\text{viii}}$)	3·79
C(1)–C(11″)	3·83		

Table 5. *Interatomic distances and angles and their standard deviations*

	Length	σ		Angle	σ
Au—C(1)	1·935 Å	0·019 Å	C(1)—Au—N	176·8°	0·7°
Au—N	2·028	0·013	Au—C(1)–C(2)	174·2	1·8
C(1)–C(2)	1·210	0·028	C(1)—C(2)–C(3)	178·6	2·1
C(2)–C(3)	1·479	0·028	C(2)—C(3)–C(4)	119·8	1·9
C(3)–C(4)	1·385	0·030	C(2)—C(3)–C(8)	120·3	1·9
C(4)–C(5)	1·413	0·030	C(3)—C(4)–C(5)	118·7	2·0
C(5)–C(6)	1·375	0·035	C(4)—C(5)–C(6)	121·7	2·2
C(6)–C(7)	1·356	0·034	C(5)—C(6)–C(7)	118·3	2·3
C(7)–C(8)	1·387	0·029	C(6)—C(7)–C(8)	123·1	2·2
C(8)–C(3)	1·416	0·031	C(7)—C(8)–C(3)	118·3	2·0
N—C(9)	1·549	0·026	C(8)—C(3)–C(4)	119·8	1·9
C(9)–C(10)	1·533	0·031	Au—N—C(9)	117·2	1·1
C(9)–C(11)	1·488	0·033	N—C(9)–C(10)	109·0	1·7
			N—C(9)–C(11)	108·6	1·7
			C(10)–C(9)–C(11)	112·9	1·8

in Table 6. The angles Au–N–C(1′) and Au–N–C(1‴) are 100 and 106° respectively, whilst C(9)–N–C(1′), C(9)–N–C(1‴) and C(1′)–N–C(1‴) are 136, 102 and 88°. The corresponding angles to the mid-points of the ethynyl groups are 109, 115, 127, 97 and 84°. These values indicate that the amino hydrogen atoms are directed approximately towards neighbouring ethynyl groups. Calculation of the positions of these two atoms, assuming N–H bond lengths of 1·0 Å and H–N–H angles cf 108°, leads to H···C distances of 2·58 and 2·49 Å to C(1′) and C(1‴). The N–H···C angles are 133 and 167° so that the angle to C(1′) departs considerably from 180°.

Tertiary phosphine complexes of phenylethynylgold(I) are monomeric in benzene solution and have large dipole moments of about 6·5D. In contrast, the amine complexes are associated in solution and their dielectric constants show the presence of much less polar aggregates (Coates & Parkin, 1962). The Au–N bonds are expected to be at least as polar as the Au–P bonds, so some factor, other than dipole interactions, must be responsible for the association of the amine complexes.

Hydrogen bonds are formed by acidic hydroxyl groups with ethylenes (West, 1959), with acetylenes and also between NH groups and olefins (Schleyer, Trifan & Bacskai, 1958). In the present case, hydrogen bonds may be formed between N and the ethynyl groups C(1′)–C(2′) and C(1‴)–C(2‴), although spectroscopic evidence for this is lacking. The N–H···C distances are about the same as the N–H···N distances of 3·38 Å in solid ammonia (Olovsson & Templeton, 1959). The interaction may be linked with the angle Au–N–C(9) being greater than tetrahedral and with the distortion of the angle Au–C(1)–C(2) to 174°.

The authors wish to express their thanks to Dr C. Parkin for crystal samples, to Professor D. W. J. Cruickshank for the use of his program and to Professor G. E. Coates for suggesting the problem and for his interest throughout the work.

References

Arai, G. J. (1962). *Rec. Trav. chim. Pays-Bas*, **81**, 307.

Berghuis, J., Haanappel, IJ. M., Potters, M., Loopstra, B. O., MacGillavry, C. H. & Veenendaal, A. L. (1955). *Acta Cryst.* **8**, 478.

Coates, G. E. & Parkin, C. (1962). *J. Chem. Soc.* p. 3220.

Corfield, P. W. R. & Shearer, H. M. M. (1966*a*). *Acta Cryst.* **20**, 502.

Corfield, P. W. R. & Shearer, H. M. M. (1966*b*). *Acta Cryst.* **21**, 957.

Cruickshank, D. W. J., Pilling, D. E., Bujosa, A., Lovell, F. M. & Truter, M. R. (1961). In *Computing Methods and the Phase Problem in X-ray Crystal Analysis*, p. 32. Oxford: Pergamon Press.

Elliott, N. & Pauling, L. (1938). *J. Amer. Chem. Soc.* **60**, 1846.

James, R. W. (1948). *The Crystalline State*, Vol. II, p. 149. London: Bell.

Olovsson, I. & Templeton, D. H. (1959). *Acta Cryst.* **12**, 832.

Orgel, L. E. (1960). *An Introduction to Transition-Metal Chemistry*. London: Methuen.

Pauling, L. (1960). *The Nature of the Chemical Bond*. Ithaca: Cornell Univ. Press.

Rosenzweig, A. & Cromer, D. T. (1959). *Acta Cryst.* **12**, 709.

Rundle, R. E. (1954). *J. Amer. Chem. Soc.* **76**, 3101.

Schleyer, P. von R., Trifan, D. S. & Bacskai, R. (1958). *J. Amer. Chem. Soc.* **80**, 6691.

Sutton, L. E. (1965). *Tables of Interatomic Distances, Supplement*. London: The Chemical Society.

Thomas, L. H. & Umeda, K. (1957). *J. Chem. Phys.* **26**, 293.

West, R. (1959). *J. Amer. Chem. Soc.* **81**, 1614.

Acta Cryst. (1967). **23**, 162

The Crystal Structure of Thomsenolite

By G. Cocco, P. C. Castiglione and G. Vagliasindi
Centro Nazionale di Cristallografia del C.N.R., Istituto di Mineralogia dell'Università di Perugia, Italy

(*Received* 3 *November* 1966 *and in revised form* 14 *December* 1966)

The determination of the crystal structure of thomsenolite, $NaCaAlF_6.H_2O$, has been carried out with integrating Weissenberg data. The mineral is monoclinic: space group $P2_1/c$; $a_0 = 5·583 \pm 0·004$, $b_0 = 5·508 \pm 0·005$, $c_0 = 16·127 \pm 0·006$ Å and $\beta = 96°26' \pm 3'$; $Z = 4$. The final R value is 9·0 %. Fundamentally, the structure is made up by sheets subparallel to {001}, resulting from Ca^{2+} bonded to four $[AlF_6]^{3-}$ octahedra and by chains parallel to [001] of alternating Ca polyhedra and $[AlF_6]^{3-}$ octahedra. Sodium is surrounded by the unusual number of eight neighbouring fluorine atoms. The proposed structure is in good agreement with the physical properties of the mineral.

Introduction

Thomsenolite is a monoclinic hydrated aluminofluoride of sodium and calcium: $NaCaAlF_6.H_2O$, dimorphous with pachnolite for which the structure was recently announced but has not yet been published (Gerhard, 1966).

The unit cell of thomsenolite has been determined by Ferguson (1946); a refinement by the least-squares method on the diffractometric data gives the values