

Hospital, 1973). Cependant on peut noter une ouverture des angles N(9)–C(12)–C(11) et N(9)–C(13)–C(14) (127 et 125 au lieu de 120 et 121° pour la quinacrine) accompagnée d'une fermeture des angles C(12)–N(9)–C(13) et C(11)–C(10)–C(14) (115 au lieu de 122 et 118° pour la quinacrine) comme nous l'avons déjà observé pour différents dimères de l'acridine non chargé (Courseille, Geoffre, Hospital & Barbet, 1977; Courseille, Leroy, Busetta & Barbet, 1977; Courseille, Leroy, Hospital & Barbet, 1977).

Le noyau acridine est légèrement déformé au niveau du cycle pyridinique central; les deux cycles phényles latéraux qui restent plans font un angle dièdre de 2° alors qu'il est de 8° pour la quinacrine.

La chaîne amino-aliphatique présente une chélation intramoléculaire entre N(30) et N(34): N(30)···N(34) 2,92 Å, N(30)–H(130)···N(34) 136°.

Cette chélation entraîne un repliement de la chaîne amino-aliphatique et la place dans un plan assez proche de celui du noyau acridine (Fig. 3).

La cohésion cristalline est assurée dans la direction *c* par des liaisons de superposition entre les noyaux acridine: deux noyaux acridine homologues par un centre de symétrie sont distants de 3,56 Å avec une surface de recouvrement importante et forment ainsi des dimères de superposition (Fig. 4). La cohésion entre deux dimères différents n'est assurée que par de faibles liaisons de van der Waals.

Références

- COURSEILLE, C., BUSETTA, B. & HOSPITAL, M. (1973). *Acta Cryst.* B29, 2349–2355.
 COURSEILLE, C., GEOFFRE, S., HOSPITAL, M. & BARBET, J. (1977). *Acta Cryst.* B33, 1573–1576.
 COURSEILLE, C., LEROY, F., BUSETTA, B. & BARBET, J. (1977). *Acta Cryst.* B33, 1570–1572.
 COURSEILLE, C., LEROY, F., HOSPITAL, M. & BARBET, J. (1977). *Acta Cryst.* B33, 1565–1569.
 GERMAIN, G., MAIN, P. & WOOLFSON, M. M. (1970). *Acta Cryst.* B26, 274–285.

Acta Cryst. (1977). B33, 1579–1581

Yttrium–Gold–Antimony Y₃Au₃Sb₄

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Abstract. Y₃Au₃Sb₄, cubic, $I\bar{4}3d$, $cI40$, $a = 9.818$ (1) Å, $Z = 4$, $D_m = 9.35$, $D_x = 9.42$ g cm⁻³. Y in 12(*a*), Au in 12(*b*), Sb in 16(*c*), $x = 0.088 \pm 0.002$. Y has four close Au neighbors and four close Sb neighbors.

Introduction. Debye–Scherrer diffraction patterns (Cu $K\alpha$ radiation, Ni filter) of an equiatomic YAuSb alloy contained lines of a b.c. cube with faint extra lines. Other alloys of closely related compositions were prepared, from which the single phase composition was found to be Y₃Au₃Sb₄. It was established that the unit-cell constant was invariant (*e.g.* no substitution of Au for Sb is possible). Examination of the extinctions revealed *hhl* reflections only when $2h + l = 4n$. These conditions are met only by space groups $I\bar{4}3d$ and $Ia3d$. The latter is eliminated by the requirement $0kl: k, l = 2n$ (the 310 line is present).

The measured density of the alloy indicated four formula weights (Y₁₂Au₁₂Sb₁₆) to the unit cell, which fits the 12(*a*), 12(*b*) and 16(*c*) sites of $I\bar{4}3d$. Assignment of elements to sites was made by trial intensity calculations (Yvon, Jeitschko & Parthé, 1969) with atomic

scattering factors (Moore, 1963). Intensity calculations were also made with the assumed Pu₂C₃-type structure (Zachariasen, 1952) in which Sb was placed in 16(*c*) sites, and Y and Au in 24(*d*) sites. Intensities for the four trial structures are given in Table 1. A reasonable fit between observed and calculated intensities was found only with the placement of Y in 12(*a*), Au in 12(*b*) and Sb in 16(*c*).

The only variable atomic positional parameter, x , of the 16(*c*) sites, was determined by a graphical method in which calculated intensities (Yvon, Jeitschko & Parthé, 1969) were plotted on a log scale against x . For a group of adjacent reflections, $x = 0.088 \pm 0.002$ gave best agreement with observed intensities. The (001) projection of the unit cell is shown in Fig. 1. Interatomic distances and the number of neighbors are given in Table 2.

Discussion. The lanthanides from Nd through Lu (except Pm, Eu and Yb) form compounds isotypic with Y₃Au₃Sb₄. Sc failed to form this structure. Unit-cell constants are given in Table 3.

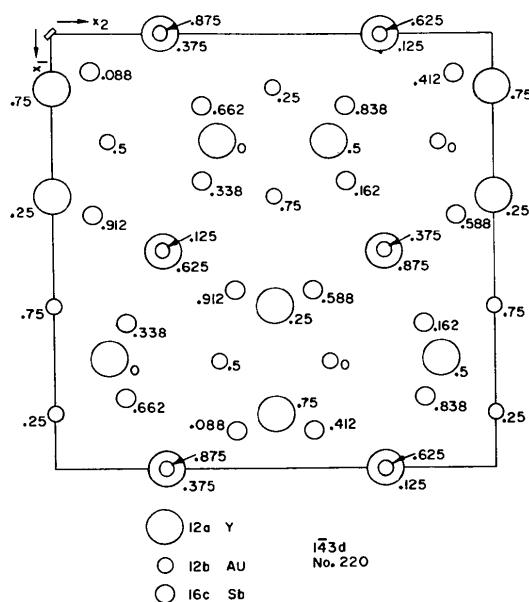
Table 1. *d* spacings and intensities of $Y_3Au_3Sb_4$
(Cu $K\alpha_1$ radiation)

<i>hkl</i>	d_o	d_c	I_o	$I_c^{(1)}$	$I_c^{(2)}$	$I_c^{(3)}$	$I_c^{(4)}$
211	3.98	4.009	<i>s</i>	640	63	111	413
220	3.45	3.472	<i>w</i>	4	77	77	77
310	3.09	2.106	<i>vw</i>	1000	380	126	9
321	2.61	2.625	<i>vs</i>	860	1000	1000	1000
400	—	2.455	—	439	0	0	0
420	2.19	2.196	<i>s</i>	76	407	409	407
332	2.085	2.094	<i>m</i>	180	97	67	85
422	1.996	2.005	<i>ms</i>	37	140	141	141
510, 431	1.92	1.926	<i>s</i>	382	44	95	259
521	1.788	1.793	<i>ms</i>	41	141	141	141
440	—	1.736	—	121	4	4	4
530	1.68	1.684	<i>w</i>	85	29	29	29
611, 532	1.584	1.593	<i>w</i>	138	154	65	42
620	1.545	1.553	<i>m</i>	107	104	104	104
541	1.51	1.515	<i>vw</i>	58	75	29	20
631	1.442	1.448	<i>ms</i>	181	100	99	100
444,	1.412	1.417	<i>ms</i>	40	102	101	102
543, 710	1.384	1.389	<i>m</i>	180	46	46	46
640	1.358	1.362	<i>ms</i>	16	89	89	89
633, 721, 552	1.332	1.336	<i>s</i>	239	114	101	139
642	1.31	1.312	<i>m</i>	60	84	84	84
730	1.287	1.290	<i>vw</i>	55	0	8	28
732, 651	1.244	1.247	<i>mw</i>	152	46	46	46
800	1.225	1.228	<i>vw</i>	0	25	25	25
741	1.207	1.209	<i>vw</i>	114	20	20	20
820	—	1.191	—	74	0	0	0
653	1.171	1.174	<i>m</i>	23	22	36	68
822, 660	1.155	1.157	<i>mw</i>	10	41	41	41
743, 750, 831	1.14	1.142	<i>mw</i>	70	46	25	43
752	1.11	1.112	<i>m</i>	31	71	71	71
840	1.096	1.098	<i>w</i>	0	20	20	20
910	—	1.084	—	4	7	7	7
842	1.07	1.071	<i>ms</i>	44	90	90	90
921, 761, 655	1.057	1.059	<i>ms</i>	151	124	90	90
664	—	1.047	—	36	0	0	0
754, 851, 930	1.033	1.035	<i>w</i>	78	41	12	17
932, 763	1.011	1.013	<i>m</i>	87	48	50	66
844	1.00	1.002	<i>w</i>	14	25	25	25
941, 853	0.99	0.9918	<i>w</i>	31	26	26	26
860	—	0.9818	—	51	0	0	0
772, 1011	0.97	0.9721	<i>m</i>	112	62	57	66
1020, 862	0.961	0.9628	<i>vw</i>	135	16	16	16
950, 943	—	0.9536	—	11	19	6	12
1031, 952, 765	0.935	0.9361	<i>m</i>	122	51	52	51
871	0.919	0.9196	<i>vw</i>	30	13	13	13
1040, 864	0.911	0.9116	<i>s</i>	48	123	124	123
1033, 961	0.903	0.9038	<i>m</i>	49	42	45	66
1042	0.895	0.8963	<i>w</i>	17	23	24	23
1110, 954, 873	0.889	0.8889	<i>mw</i>	41	25	20	39
1051, 1121, 963	0.8745	0.8747	<i>m</i>	146	49	50	49
880	0.8672	0.8678	<i>ms</i>	2	56	56	56
1130, 970	0.861	0.8611	<i>vw</i>	12	10	10	10
1053, 972, 1132	0.8476	0.8482	<i>vs</i>	182	183	173	215
866, 1060	0.8415	0.8419	<i>ms</i>	30	88	89	88
1141, 875	0.8356	0.8358	<i>vw</i>	144	19	4	24
1062	—	0.8298	—	0	0	0	0
965	0.8238	0.8239	<i>m</i>	131	40	41	40
1200, 884	0.818	0.8182	<i>m</i>	30	61	61	61

Table 1 (cont.)

<i>hkl</i>	d_o	d_c	I_o	$I_c^{(1)}$	$I_c^{(2)}$	$I_c^{(3)}$	$I_c^{(4)}$
974, 1150, 981, and 1143	0.812	0.8182	<i>m</i>	142	51	51	51
1220	0.8068	0.8071	<i>ms</i>	89	69	70	69
1055, 1152, 1071	0.8015	0.8017	<i>s</i>	175	104	85	126
1064, 1222	0.796	0.7964	<i>s</i>	5	159	161	159
1231; 983	0.792	0.7912	<i>vw</i>	26	82	24	25
1073, 1161	0.7812	0.7811	<i>s</i>	162	201	203	201
1240	0.7762	0.7762	<i>ms</i>	1	155	157	155

Assumptions: (1) Pu_2C_3 -type; Y Au in 24(*d*), $x = 0.3$; Sb in 16(*c*), $x = 0.05$. (2) $Y_3Au_3Sb_4$ -type; Y in 12(*b*); Au in 12(*a*); Sb in 16(*c*); $x = 0.088$. (3) $Y_3Au_3Sb_4$ -type; Y and Au in 12(*a*) and 12(*b*); Sb in 16(*c*); $x = 0.088$. (4) $Y_3Au_3Sb_4$ -type; Y in 12(*a*); Sb in 16(*c*); $x = 0.088$.

Fig. 1. Projection of the $Y_3Au_3Sb_4$ structure onto the face of a cube.Table 2. Interatomic distances (Å) in $Y_3Au_3Sb_4$

Y-8Y*	4.592 ± 0.001	Au-8Au	4.592 ± 0.001
Y-4Y	5.489 ± 0.001	Au-4Au	5.489 ± 0.001
Y-8Y	6.722 ± 0.001	Au-8Au	6.722 ± 0.001
Y-4Au	3.006 ± 0.001	Au-4Sb	2.766 ± 0.010
Y-2Au	4.909 ± 0.001	Au-4Sb	4.362 ± 0.027
Y-4Au	5.489 ± 0.001	Au-4Sb	5.694 ± 0.030
Y-4Au	5.756 ± 0.001	Au-4Sb	5.943 ± 0.012
Y-4Sb	3.349 ± 0.021	Sb-3Sb	3.620 ± 0.016
Y-4Sb	3.448 ± 0.022	Sb-2Sb	4.251 ± 0.001
Y-4Sb	5.635 ± 0.005	Sb-6Sb	4.904 ± 0.028
Y-4Sb	6.290 ± 0.003	Sb-6Sb	6.100 ± 0.009

* The distance from a Y atom to eight other Y atoms.

Table 3. Unit-cell constants (\AA) ($\pm 0.002 \text{\AA}$)

$Nd_3Au_3Sb_4$	9.961	$Dy_3Au_3Sb_4$	9.811
$Sm_3Au_3Sb_4$	9.909	$Ho_3Au_3Sb_4$	9.788
$Gd_3Au_3Sb_4$	9.864	$Er_3Au_3Sb_4$	9.768
$Tb_3Au_3Sb_4$	9.834	$Tm_3Au_3Sb_4$	9.752
$Y_3Au_3Sb_4$	9.821	$Lu_3Au_3Sb_4$	9.723

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References

- MOORE, F. H. (1963). *Acta Cryst.* **16**, 1169–1175.
 YVON, K., JEITSCHKO, W. & PARTHÉ, E. (1969). *A Fortran IV Program for the Intensity Calculation of Powder Patterns*. Univ. of Pennsylvania.
 ZACHARIASEN, W. H. (1952). *Acta Cryst.* **5**, 17–19.

Acta Cryst. (1977). B33, 1581–1583

Structural Studies of Precursor and Partially Oxidized Conducting Complexes.

V. A Neutron Diffraction Study of Dianilinium Tetracyanoplatinate(II)*

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Abstract. $(C_6H_5NH_3)_2[Pt(CN)_4]$, triclinic, $P\bar{1}$ (C^1), $a = 7.933$ (5), $b = 10.654$ (7), $c = 5.497$ (4) \AA , $\alpha = 103.24$ (3), $\beta = 91.19$ (3), $\gamma = 110.34$ (3) $^\circ$, $Z = 1$, $D_m = 1.90$ (2), $D_c = 1.92$ g cm^{-3} . A room-temperature single-crystal neutron diffraction study was performed. The structural solution was obtained by direct methods, and refinement by full-matrix least-squares techniques gave $R(F_o^2) = 0.061$ for 1304 observed reflections $> \sigma(F_o^2)$. The structure consists of isolated tetracyanoplatinate groups interacting with the anilinium groups *via* $N-H \cdots N \equiv C$ hydrogen bonds. No Pt–Pt chains are formed *via* overlapping d_{z^2} orbitals of the Pt atoms.

Introduction. As part of a continuing study of partially oxidized tetracyanoplatinate conducting salts and their starting materials, we have performed a neutron diffraction analysis of $(C_6H_5NH_3)_2[Pt(CN)_4]$. Utilization of the compound $K_2[Pt(CN)_4] \cdot 3H_2O$ is a standard method in preparing the well known one-dimensional conductor $K_{1.75}[Pt(CN)_4] \cdot 1.5H_2O$ (Williams, Keefer, Washecheck & Enright, 1976), and therefore $(C_6H_5NH_3)_2[Pt(CN)_4]$ might be expected to be the precursor of a corresponding partially oxidized, aniline-deficient compound.

The compound $Ba[Pt(CN)_4] \cdot 4H_2O$ (preparation described by Koch, Abys & Williams, 1976) was used to prepare single crystals of $(C_6H_5NH_3)_2[Pt(CN)_4]$. Aniline sulfate was added to a solution of $Ba[Pt(CN)_4] \cdot 4H_2O$ and $BaSO_4$ was removed by filtration. The pH of the filtrate was adjusted to ~ 2 and

Table 1. Positional parameters for $(C_6H_5NH_3)_2[Pt(CN)_4]$ ($\times 10^4$)

Here and elsewhere in this paper the e.s.d.'s in parentheses refer to the least-significant figure.

	<i>x</i>	<i>y</i>	<i>z</i>
Pt	0	0	0
C(1)	–2493 (2)	–783 (2)	–1698 (4)
C(2)	929 (2)	–905 (2)	–2942 (4)
N(1)	–3974 (2)	–1207 (2)	–2600 (4)
N(2)	1540 (2)	–1403 (2)	–4609 (3)
C(3)	–3341 (2)	–6598 (2)	–1170 (3)
C(4)	–2340 (3)	–6087 (2)	1147 (4)
C(5)	–1600 (3)	–4662 (2)	2136 (4)
C(6)	–1871 (3)	–3781 (2)	809 (4)
C(7)	–2874 (3)	–4314 (2)	–1507 (5)
C(8)	–3627 (3)	–5736 (2)	–2523 (4)
N(3)	–4075 (2)	–8092 (2)	–2265 (4)
H(1)	5583 (8)	3842 (5)	5659 (10)
H(2)	6915 (6)	1596 (4)	6847 (10)
H(3)	4930 (7)	1641 (5)	6294 (12)
H(4)	5436 (8)	1394 (5)	9028 (11)
H(5)	1280 (8)	2670 (5)	8418 (12)
H(6)	–3098 (10)	–3630 (6)	–2533 (12)
H(7)	805 (8)	4238 (6)	6050 (11)
H(8)	7880 (8)	3202 (6)	2154 (11)

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