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Crystal Structure and Chemical Bonding of a Palladium Thiosulphate Complex: Bisethylenediaminepalladium(II) *cis*-Dithiosulphate-ethylenediaminepalladite

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The crystal structure of $[Pd(en)_2]\,[Pd(S_2O_3)_2(en)]$ (en = ethylenediamine) has been determined. It forms crystals of space group $P\bar{1}$ with $a=8{\cdot}76\pm0{\cdot}03$, $b=13{\cdot}30\pm0{\cdot}05$, $c=8{\cdot}66\pm0{\cdot}03$ Å, $\alpha=108{\cdot}3\pm0{\cdot}3$, $\beta=98{\cdot}5\pm0{\cdot}3$, $\gamma=103{\cdot}1\pm0{\cdot}3°$, the calculated density for two formula weights in the unit cell is 2·26 g.cm^{-3}, D measured is 2·24 g.cm^{-3}. The structure was solved by Patterson methods and refined by full-matrix least-squares to a convention R value of 9·8% for the 3575 reflections measured on films. This study established that the title compound consists of discrete $[Pd(en)_2]^{2+}$ cations and *cis*-$[Pd(S_2O_3)_2(en)]^{2-}$ anions. There is the expected square planar coordination around the Pd atoms, the S_2O_3 groups being bonded through S. The average bond distances are: Pd–N 2·071 ± 0·007, Pd–S 2·297 ± 0·004, C–N 1·48 ± 0·01, C–C 1·53 ± 0·02, S–S 2·066 ± 0·006, and S–O 1·457 ± 0·007 Å; the average O–S–O angle of the S_2O_3 groups is 111·8 ± 0·7°. The S–S bonds of the coordinated S_2O_3 groups are significantly longer than in the thiosulphate ion.

Introduction

Chemical and spectroscopic studies of coordination compounds having the S_2O_3 group as a ligand indicate that the latter can be a bridging ligand, a bidentate ligand or a monodentate ligand (Livingstone, 1965). The structure determinations of two thiosulphates have been reported so far; that of $Ni[SC(NH_2)_2]_4S_2O_3.H_2O$ (Fava Gasparri, Mangia, Musatti & Nardelli, 1969) where the S_2O_3 is bonded through S and O to Ni, and that of $Na_{4n}[Cu(NH_3)_4]_n[Cu_n(S_2O_3)_{2n}]_2$ (Ferrari, Braibanti & Tiripicchio, 1966) in which the S_2O_3 ligand bridges two Cu atoms through one S atom. In this last compound there is a significant lengthening of the S–S bond of the coordinated S_2O_3 group as compared with the dimensions of the $S_2O_3^{2-}$ ion determined in $S_2O_3Mg.6H_2O$ (Baggio, Amzel & Becka, 1969). We undertook the present study in order to establish whether this lengthening would also occur when the group behaves as a monodendate S bonded ligand. Since transition metals of the platinum group are likely to bind $S_2O_3^{2-}$ through a metal–S bond we selected a palladium thiosulphate complex that crystallized forming suitable crystal for a X-ray diffraction study. We chose the title compound, of minimum formula $Pd_2(S_2O_3)_2(en)_3$ (en = ethylenediamine, $NH_2C_2H_4NH_2$), which also presented the problem of assigning it the correct structural formula. This compound has a stoichiometry similar to that of compounds formulated as $[Pt(NH_3)_4]_3Pt(S_2O_3)_4$ and $[Pt(NH_3)_4]_3Pd(S_2O_3)_4$ (Costamagna & Levitus, 1966), which are not plausible formulae since they imply the existence of anions of charge −6.

Experimental

The crystals were kindly supplied to us by Dr Levitus. Their chemical formula was established by quantitative chemical analysis (spectroscopic analysis of Pd and microanalysis of C, H, N, and S) and the presence of coordinated S_2O_3 was confirmed by the fact that the infrared spectrum of the complex showed S–O stretching modes at higher frequencies than in the ionic thiosulphates. The appearance of the crystals is that of orange coloured plates having the c unit-cell axis perpendicular to the prominent face. Long exposures to X-ray radiation in contact with air did not damage the crystals.

Crystal data

Bisethylenediaminepalladium(II)-*cis*-dithiosulphate-ethylenediaminepalladite,
$Pd(NH_2C_2H_4NH_2)_2.Pd(S_2O_3)_2(NH_2C_2H_4NH_2)$.
F.W. 617·3. Triclinic, $a=8{\cdot}76\pm0{\cdot}03$, $b=13{\cdot}30\pm0{\cdot}05$,
$c=8{\cdot}66\pm0{\cdot}03$ Å, $\alpha=108{\cdot}3\pm0{\cdot}3°$, $\beta=98{\cdot}5\pm0{\cdot}3°$,
$\gamma=103{\cdot}1\pm0{\cdot}3°$ at 295°K, $V=907\pm5$ Å^3,
$D_m=2{\cdot}24$ g.cm^{-3} (by flotation), $D_c(Z=2)=2{\cdot}26$ g.cm^3,
$F(000)=612$, $\mu=18$cm^{-1} for Mo$K\alpha$ radiation. Space group $P\bar{1}$.

The cell constants were refined using the Cu $K\alpha_I-K\alpha_{II}$ splitting of 104 independent high angle reflections, measured on Weissenberg equi-inclination photographs of levels $5kl$, $6kl$, and $7kl$, in the least-squares procedure proposed by Alcock & Sheldrick (1967). A Deldundy reduction did not indicate that there was higher symmetry. The data used to solve and refine the structure were collected with Zr-filtered Mo K radiation with crystals cut to the shape of a parallelepiped and mounted with their a axis parallel to the spindle axis. The dimensions of the cut crystals were of less than 0·03 cm along **a** and with cross sections

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of 0·02 × 0·02 cm. We recorded reflections from layers $0kl$ to $6kl$ with the equi-inclination Weissenberg method using multiple films, and from layers $hk0$ to $hk2$ with an integrating precession camera. The intensities on the Weissenberg photographs were estimated by visual comparison with calibrated scales and on the precession photographs with a microdensitometer. Within a ratio of 2000:1 of maximum to minimum observable intensity we were able to measure the intensity of 3575 independent reflections, representing ~95% of the reflections that could be recorded on the films at $\sin\theta/\lambda < 0{\cdot}65$ Å^{-1}. The intensities were corrected for Lorentz, polarization and applying the Philips spot-shape factor to the reflections of the extended half of the equi inclination Weissenberg photographs (the complete set of measurable reflections of a layer was recorded on two photographs). The data of the different Weissenberg and precession photographs were put on the same scale by correlating the intensities of equivalent reflections and applying the least-squares refinement proposed by Hamilton, Rollett & Sparks (1965). The discrepancy factor of the equivalent reflections after scaling is 8%. We did not apply an absorption correction to the data since the change of absorption factor in any one layer is less than 2% in the region of observable intensity, and the different layer lines obtained from the Weissenberg photographs have been put on the same scale using the intensities measured on the precession photographs, which have negligible difference of absorption correction within each and between layer lines.

Structure determination and refinement

A three-dimensional Patterson synthesis gave a map showing clearly the Pd–Pd and Pd–S vectors. Next we computed a three-dimensional Fourier synthesis using the signs given by the heavy atoms, which in turn allowed us to locate unequivocally the positions of the remaining non-hydrogen atoms. A structure factor calculation including only the heavy atoms gave a conventional R value of 0·30, when the light atoms were included R dropped to 0·19. We initiated a full-matrix least-squares refinement [minimizing $\sum\omega(F_o-F_c)^2$] allowing for the simultaneous variation of coordinates, individual isotropic temperature factors and a different scale factor for each set of reflections registered on the $0kl$ to $6kl$ equi inclination Weissenberg photographs, and after 4 cycles the indicated shift of the parameters was less than $\frac{1}{5}$ one-fifth of their estimated standard

Table 1. *Final positional parameters* (× 10⁴), *and coefficients B in the temperature factor expression* $\exp[-B(\sin\theta/\lambda)^2]$, *all with their e.s.d.'s in parentheses*

	x	y	z	B (Å^2)
Pd(1)	5000	0	0	*
Pd(2)	0	5000	5000	*
Pd(3)	1795 (2)	2154 (1)	4523 (1)	*
S(1)	3292 (4)	−3458 (3)	−1806 (3)	*
S(2)	1244 (5)	2475 (3)	2086 (4)	*
S(3)	−0809 (4)	0973 (2)	3547 (4)	*
S(4)	−2148 (4)	1769 (3)	5017 (4)	*
O(1)	2734 (17)	3622 (11)	0260 (16)	3·13 (24)
O(2)	3850 (15)	4483 (10)	3227 (14)	2·48 (20)
O(3)	4507 (15)	2883 (10)	1752 (14)	2·34 (19)
O(4)	−1069 (13)	2422 (8)	6663 (12)	1·75 (16)
O(5)	−3510 (15)	0878 (9)	5022 (14)	2·28 (18)
O(6)	−2802 (15)	2451 (10)	5778 (14)	2·29 (18)
C(1)	3801 (20)	1917 (12)	7252 (18)	1·95 (23)
C(2)	4828 (20)	3031 (12)	7186 (18)	1·95 (23)
C(3)	1082 (21)	5273 (13)	8436 (19)	2·18 (25)
C(4)	2392 (23)	4992 (13)	7630 (21)	2·48 (27)
C(5)	2267 (21)	−0308 (13)	1554 (20)	2·20 (25)
C(6)	2556 (19)	−1437 (11)	0897 (17)	1·67 (17)
N(1)	2082 (17)	1770 (10)	6667 (15)	1·95 (16)
N(2)	4245 (15)	3021 (10)	5473 (14)	1·44 (17)
N(3)	−0489 (18)	4792 (11)	7171 (16)	2·13 (21)
N(4)	2412 (16)	5333 (10)	6170 (14)	1·66 (18)
N(5)	3881 (17)	0543 (10)	1883 (15)	1·93 (20)
N(6)	3297 (17)	−1488 (10)	−0524 (15)	1·90 (20)

* These atoms have been refined with anisotropic temperature factor; the following are the final values of the coefficients B_{ii} (× 10⁴) in the expressions $\exp-(B_{11}h^2+B_{22}k^2+B_{33}l^2+2B_{12}hk+2B_{13}hl+2B_{23}kl)$

	B_{11}	B_{22}	B_{33}	B_{12}	B_{13}	B_{23}
Pd(1)	69 (2)	23 (1)	40 (1)	14 (1)	21 (2)	07 (1)
Pd(2)	58 (2)	24 (1)	76 (2)	11 (1)	34 (2)	15 (1)
Pd(3)	47 (1)	23 (1)	50 (1)	08 (1)	16 (1)	12 (1)
S(1)	59 (5)	21 (2)	46 (4)	11 (2)	21 (4)	14 (2)
S(2)	56 (5)	37 (2)	62 (4)	03 (3)	07 (4)	24 (3)
S(3)	43 (5)	24 (2)	62 (4)	04 (3)	17 (4)	04 (2)
S(4)	48 (5)	29 (2)	40 (3)	10 (2)	18 (3)	13 (2)

Table 2. *Observed and calculated structure factors*

The columns give l, F_o (×10) and F_c (×10). The parameters used in the calculation are those given in Table 1. The letter U following l indicates an unobserved reflexion.

H=	0 K=	0
1	1231	-1294
2	1033	1035
3	832	-730
4	1050	572
5U	58	58
6	874	836
7U	73	-80
8U	66	6
9	153	172
10U	91	67
11	194	289
H=	0 K=	1
-12	121	171
-11	152	211
-10U	84	-73
-9	396	377
-8	56	-122
-7	692	644
-6	104	96
-5	855	804
-4	1135	-1057
-3	1210	1089
-2	842	808
-1	1169	1177
1	194	-256
2	486	516
3	104	-59
4	1127	1030
5	45	-76
6	110	134
7	281	-265
8	300	312
9	274	291
10U	42	21
11	145	205
13	166	214
H=	0 K=	2
-13	113	-145
-12	292	356
-11	150	-168
-10	414	392
-8	558	513
-5	396	308
-3	1061	1012
-2	521	-500
-1	707	724
0	424	-421
1	1040	988
2	621	616
3	431	341
4	304	269
5	304	-268
6	536	602
7U	73	-57
8	824	781
9	367	-355
10	265	298
12	163	234
H=	0 K=	3
-13	194	219
-11	171	191
-10	74	117
-9	170	198
-8	86	114
-7	398	338
-6	560	527
-5	311	317
-4	834	709
-3	302	310
-2	672	-726
-1	1700	1911
0	1038	-533
1	1533	1593
2	407	-348
3	903	814
4	576	-909
5	1427	1415
6	234	-208
7	583	559
8	99	-165
9U	93	98
10	102	164
11	122	216
H=	0 K=	4
-11	69	113
-10	78	61
-9	163	206
-8	243	241
-7	161	122
-6	856	747
-5	516	-471
-4	1291	1187
-3	1125	-1032
-2	1031	983
-1	496	-493
0	1434	1433
1	704	-698
2	170	154
3	188	143
4	433	326
5	629	603
6	227	-228
7	434	373
8U	86	-12
9	381	352
H=	0 K=	5
-11	243	333
-10U	66	-16
-9	509	518
-8	499	-476
-7	613	635
-6	263	-265
-5	559	637
-4	125	-91
-3	538	515
-2	359	344
-1	629	555
0	196	211
1	161	-112
2	1018	919
3U	49	-2
4	616	610
5	64	49
6	57	95
7	450	415
8U	83	89
9	303	345
11	98	170
H=	0 K=	6
-12	446	487
-11U	90	17
-10	286	314
-9	287	-303
-8	176	228
-7	473	456
-6U	64	45
-5	634	618
-4	101	68
-3	288	286
-2	495	478
-1	471	420
0	599	576
1	383	281
2	674	674
3	484	-403
4	1093	1021
5	630	-635
6	801	728
7	158	-175
8	462	434
9	128	-191
10	214	299
H=	0 K=	7
-12	73	147
-10	176	218
-8	447	400
-7	273	272
-6U	64	-38
-5	91	144
-4	250	-220
-3	1043	1033
-2	121	-129
-1	983	948
0	805	-747
1	1290	1251
2	459	-414
3	845	790
4	134	-166
5	305	307
7U	89	144
10	98	207
H=	0 K=	8
-10	385	378
-9U	3	0
-8	556	547
-7	515	-489
-6	566	533
-5	116	135
-4	984	951
-3	444	-384
-2	679	695
-1	133	-137
0	456	449
1	808	724
2U	67	-66
3	552	531
4	141	-156
5	342	351
6U	44	4
7	204	262
H=	0 K=	9
-12	222	-200
-11	583	636
-10	147	-261
-9	495	552
-8	120	-168
-7	543	535
-6	244	231
-5	614	609
-4	225	-200
-3	213	204
-2	512	439
-1	223	176
0	393	430
1U	68	-82
2U	6	1
3	481	428
4U	40	28
5	408	363
6U	91	-127
7	216	327
8	170	-236
9	370	407
H=	0 K=	10
-12	148	152
-11	70	170
-10	141	-79
-9	136	186
-8	147	223
-7	457	426
-6	54	-90
-5	104	142
-4	231	250
-3	206	177
-2	534	455
-1	455	-393
0	505	474
1	591	-510
2	862	801
4	581	568
5	403	-393
6	364	385
8	271	320
H=	0 K=	11
-12	168	222
-11U	46	-78
-10	198	237
-9	162	210
-8U	82	105
-7	142	127
-6	55	62
-5	455	355
-4	112	-120
-3	978	967
-2	477	-452
-1	302	319
0U	59	-18
1	508	475
2	128	155
3	134	163
4U	65	-9
5U	93	104
6	363	373
8	214	291
H=	0 K=	12
-12	389	409
-11	162	-230
-10	444	464
-9	144	-225
-8	519	503
-7	453	-440
-6	677	678
-5	152	-213
-4	552	558
-3U	74	-65
-2	85	115
-1	253	245
0	251	260
1U	73	97
2U	84	35
3	143	177
4U	92	77
5	179	218
6	103	184
8	187	227
H=	0 K=	13
-11	165	241
-10U	94	-38
-9	191	244
-8U	84	26
-7U	77	-11
-6	255	256
-5U	73	-81
-4	167	146
-3	145	191
-2	296	306
-1	96	135
0U	82	70
1	136	145
2U	88	54
3	454	425
5	228	347
6	153	-260
7	262	339
H=	0 K=	14
-11	226	255
-9	215	257
-8	67	-110
-7	140	195
-6	63	92
-5U	83	75
-4	541	529
-3	153	-160
-2	405	393
-1U	83	-90
0	384	375
1U	86	-55
2	536	539
H=	0 K=	15
-9	222	303
-8	130	-158
-7	621	650
-6	292	-344
-5	466	478
-4	244	-263
-3	491	438
-2U	87	-145
-1	427	447
0U	91	-102
1	174	204
H=	0 K=	16
-11	223	-263
-10	390	415
-8	216	305
-7U	93	-40
-6U	78	21
-5U	90	67
-4	264	310
-3	117	190
-2U	92	-57
-1	145	155
H=	0 K=	17
-10	248	297
-8	105	130
-6	303	338
-5	191	151
-4U	94	135
1	381	366
2	174	-194
3	183	233
H=	0 K=	18
-8	163	235
-6	293	341
-5	105	-166
-4	149	242
-3	206	-294
-2	484	498
-1	77	-145
0	236	220
H=	0 K=	19
-9	227	324
-7	215	304
-6	206	-313
-5	220	300
-3	157	243
H=	0 K=	20
-5	141	255
H=	0 K=	21
-1	235	290
H=	0 K=	22
-6	182	265
-5	148	-196
H=	1 K=	-19
10	105	-141
H=	1 K=	-18
-2	211	-203
1	176	-195
3	266	-325
5	221	-256
6	182	170
7	392	-375
9	234	-292
H=	1 K=	-17
-2	272	-318
0	403	-415
1	188	180
2	362	-335
3	198	220
4	245	-308
5U	93	28
6	232	-285
7	159	-157
9	150	-166
H=	1 K=	-16
-5	240	-306
-3	266	-361
-1	231	-254
0U	94	-42
1	144	-165
2U	87	-39
3	204	-211
4	195	-226
5U	90	-16
6	205	-220
7U	93	-60
8U	82	-15
9	221	-211
11	236	-317
H=	1 K=	-15
-3	236	-338
-1	148	-184
0U	49	-82
1	310	-332
2U	85	-71
3	177	-184
4U	84	-81
5U	7	0
6	394	-401
7	234	272
8	387	-392
9	157	155
10	222	-273
H=	1 K=	-14
-6	234	-252
-5	162	-111
-2U	91	15
-1	616	-355
0	165	-163
1	491	-435
2	374	393
3	548	-580
4	231	280
5	555	-575
6U	75	34
7	177	-185
8U	84	25
9	120	-101
12	193	-195
H=	1 K=	-13
-6	430	-437
-5	273	212
-4	285	-348
-3	193	182
-2	635	-772
-1	296	285
0	592	-685
1	541	555
2	512	-520
3U	74	5
4	446	-470
5	193	-190
6U	83	61
7U	82	-117
8U	96	-107
9	145	-173
10U	94	-85
12	190	-105
H=	1 K=	-12
-7	247	-282
-5	226	-251
-4U	90	-170
-3	205	-239
-2	179	-117
-1U	73	-17
0U	72	-48
1U	74	67
2	799	-745
3	153	155
4U	73	-126
5U	72	-24
6	151	-130
7	363	-361
8U	84	-120
9	193	-196
10	202	194
11	380	-371
H=	1 K=	-11
-7	197	-192
-5	244	-264
-4U	86	-61
-3	242	-255
-2	281	296
-1	491	-507
0	356	-314
1U	70	-65
2	415	-391
3	484	483
4	534	-566
5	113	-74
6	830	-945
7	265	315
8	429	-444
9	195	235
10	444	-481
11	153	-106
H=	1 K=	-10
-7	252	-272
-6U	91	-38
-5	227	-225
-4U	82	74
-3	428	-429
-2	290	275
-1	1132	-1210
0	565	545
1	845	-715
2	617	648
3	949	-1040
4U	65	81
5	576	-593
6	286	275
7	239	-238
8	221	-157
9	242	-288
10	209	-243
11U	95	68
H=	1 K=	-9
-8	237	-292
-6	368	-414
-5	359	333
-4	430	-490
-3	157	191
-2	706	-771
-1	305	308
0	296	-330
1	452	-411
2U	60	-45
3	693	-522
4U	63	-48
5	270	-272
6	306	294
7	677	-729
8U	79	-84
9	123	-119
10U	87	-81
11	122	108
12	549	-161
H=	1 K=	-8
-7	100	-107
-6U	85	28
-5	241	-285
-4	263	-257
-3	149	135
-2	605	-660
-1	165	-175
0	242	-266
1	538	-550
2	651	-645
3	106	-138
4	241	-196
5	421	-348
6U	60	-18
7	688	-685
8	434	415
9	410	-470
10U	87	153
11	360	-375
H=	1 K=	-7
-8	222	-240
-7	294	-309
-6U	82	71
-5	353	-345
-4	243	-205
-3	157	-140
-2	630	-655
-1	294	254
0	942	-1007
1	174	139
2	1248	-1074
3	858	909
4	1237	-1267
5	651	655
6	947	-865
7	457	415
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6	376	343
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4	197	-145
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-7	551	506
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1	509	-533
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1	252	285
2	235	-295
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2	223	-265
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-1	262	-270
0	220	-266
1U	123	70
2	221	-285
H= 7 K= -7		
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-1	271	-267
0	257	-236
1	289	301
2	631	-678
H= 7 K= -6		
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-1	657	-630
0	101	165
1	924	-852
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0	383	-325
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-1	81	-155
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1	175	-212
2	155	-151
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-1	361	324
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2	631	-660
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-1	695	-665
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1	661	-659
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2	197	180
3	538	-580
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7	229	-247
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1	242	-207
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1	249	232
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2	190	-204
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0	159	181
1	238	257
2	246	-252
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1	277	345
2U	124	-45
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2	661	813
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-7	63	-116
-6	203	213
-5	118	138
-4	400	390
-3	300	305
-2	307	-237
-1	507	443
0U	116	54
1	268	260
2	126	107
4	129	130
6	173	179
H= 8 K= 1		
-2	245	-241
-1	701	649
0	295	-363
1	822	703
2	470	-414
H= 8 K= 2		
-2	604	804
-1U	131	-125
0	227	265
1	110	-149
2	310	310
H= 8 K= 3		
-2	79	134
-1	93	-125
0U	113	60
1	123	162
2	422	370
H= 8 K= 4		
-2U	125	65
-1U	114	15
0	441	425
1U	135	81
2U	130	105
H= 8 K= 5		
-2U	117	31
-1	557	545
0	257	-258
1	375	333
2	243	-264
H= 8 K= 6		
-2	530	442
-1U	135	-152
0U	111	106
1U	135	100
H= 8 K= 7		
-2U	32	-3
-1U	32	0
0	205	226
H= 8 K= 8		
-2	114	170
-1U	134	-107
0	368	379
1	183	-202
2	311	325
H= 8 K= 9		
-2	283	-270
-1	277	346
H= 8 K= 10		
-2	165	151
-1	84	153
H= 8 K= 11		
-2	89	132
H= 8 K= 12		
-2	220	230
-1	124	-169
H= 9 K=-16		
-1	203	-266
1	377	337
H= 9 K=-15		
0	560	-411
1	251	256
2	216	-259
H= 9 K=-14		
1U	135	22
2	293	-305
H= 9 K=-13		
-1U	135	-192
0U	108	-93
1U	135	-67
2	170	-214
H= 9 K=-12		
-2U	130	82
-1	508	-483
0	392	436
1	587	-564
2	242	277
H= 9 K=-11		
-2	382	-321
-1U	134	85
0	157	-180
1	169	-195
2U	130	-78
H= 9 K=-10		
-2	240	-273
-1U	134	-14
0	388	-358
1U	134	-122
2U	117	22
H= 9 K= -9		
-2	513	-439
-1U	133	121
0	499	-469
1	367	344
2	428	-433
H= 9 K= -8		
-2	194	230
-1	282	-301
0	185	158
1	486	-443
2	173	-179
H= 9 K= -7		
-2U	125	63
-1	140	-152
0	161	-197
1	269	-230
2U	125	86
H= 9 K= -6		
-2U	107	-24
-1	244	-248
0U	14	0
1	245	-204
2	139	120
H= 9 K= -5		
-2	544	-463
-1	302	288
0	505	-492
1	269	271
2	631	-666
H= 9 K= -4		
-2	167	169
-1	432	-380
0U	116	35
1	127	-93
2	414	-359
H= 9 K= -3		
-2U	127	-105
-1	268	-252
0U	116	-24
1	221	-246
2	199	-213
H= 9 K= -2		
-2U	123	10
-1	292	-318
0U	116	74
1	639	-576
2	244	282
H= 9 K= -1		
-2	892	-833
-1	168	215
0	438	-405
1U	135	107
2U	130	-110
H= 9 K= 0		
-9	494	-506
-7	605	-642
-6	512	531
-5	494	-492
-4	209	195
-3	543	-534
-2U	125	-104
-1U	130	-25
0U	92	16
1	91	-108
2	216	-218
H= 9 K= 1		
-2	242	246
-1U	116	-23
0	199	-214
1	224	-245
2	152	-185
H= 9 K= 2		
-2	222	153
-1	576	-517
0U	105	41
1	257	-280
2	379	348
H= 9 K= 3		
-2	632	-677
-1	129	147
0	236	-248
1	91	147
2	171	-160
H= 9 K= 4		
-2U	131	-115
-1U	136	61
0	228	-241
1U	135	30
2	225	-231
H= 9 K= 5		
-2U	43	-3
-1U	135	-158
0	293	-314
2	307	-296
H= 9 K= 6		
-2	292	301
-1	394	-554
0	254	297
1	235	-234
H= 9 K= 7		
-2	156	-167
1	173	-159
H= 9 K= 8		
-2	446	-363
H= 9 K= 9		
-2	352	-364
-1	249	284
1	114	144
H= 9 K= 10		
-2	151	165
-1	201	-256
H= 10 K=-15		
-1	251	271
1	388	384
H= 10 K=-13		
0	214	256
H= 10 K=-12		
0	291	337
1	219	-231
2U	129	232
H= 10 K=-11		
-2U	129	-100
-1	415	373
0	205	-227
1	237	246
2U	128	33
H= 10 K=-10		
-2	170	176
-1U	133	34
0	150	156
1	202	193
2U	131	141
H= 10 K= -9		
-2U	129	86
-1U	135	166
0	177	175
1U	134	51
2U	131	-65
H= 10 K= -8		
-2	410	362
-1U	132	-58
0	236	231
1	385	-321
2	444	511
H= 10 K= -7		
-2	268	-238
-1	424	363
0	298	-282
1	301	335
2U	131	145
H= 10 K= -6		
-2U	130	146
-1	128	203
0U	114	-90
1	273	290
2U	132	-36
H= 10 K= -5		
-2	303	262
-1U	132	61
0U	114	40
1	387	334
2U	131	-127
H= 10 K= -4		
-2	806	716
-1	421	-372
0	634	667
1	250	-255
2	258	295
H= 10 K= -3		
-2U	131	93
-1	144	143
0U	113	82
1U	101	0
2U	131	130
H= 10 K= -2		
-2	100	-135
-1	90	124
0U	113	-41
1	274	305
2U	132	177
H= 10 K= -1		
-2	548	-428
-1	519	458
0U	111	-90
1	356	372
2	362	-256
H= 10 K= 0		
-8	253	305
-7	154	-182
-6	956	657
-5	417	-441
-4	432	426
-3	297	-316
-2	563	461
-1U	134	-62
0	308	336
1	212	-240
2	119	120
3	190	207
H= 10 K= 1		
-2	173	184
-1U	136	-73
0	208	214
1U	135	-103
2	355	334
H= 10 K= 2		
-2	141	105
-1U	136	38
0	175	227
2	373	356
H= 10 K= 3		
-2	281	-248
-1	669	670
2	207	-198
H= 10 K= 4		
-2	220	186
-1	140	163
H= 10 K= 5		
-2	215	243
H= 10 K= 6		
-2	547	454
-1	228	-306
2	190	313
H= 11 K=-14		
-1	282	-409
1	207	-199
H= 11 K=-12		
1	84	-158
H= 11 K=-11		
-2	504	-394
0	227	-289
1	273	279
H= 11 K=-10		
-2	166	170
0	167	183
1	239	-211
H= 11 K= -9		
0U	99	52
H= 11 K= -8		
-2	226	-170
-1U	132	-61
0U	102	10
1	266	-243
H= 11 K= -7		
-2	547	-574
-1	311	313
0	474	-495
1	308	267
H= 11 K= -6		
-2U	122	-8
-1U	134	-143
0	269	-296
1U	134	147
H= 11 K= -5		
-2U	131	-15
-1	270	-258
0U	104	7
1U	132	-45
H= 11 K= -4		
-2	287	274
-1	270	-352
0	139	153
1	483	-478
H= 11 K= -3		
-2	285	-268
-1U	131	-45
0	229	-236
H= 11 K= -2		
-2	198	-191
-1U	135	-22
0U	100	-73
H= 11 K= -1		
-2	99	-107
-1U	132	25
2	295	-475
H= 11 K= 0		
-3	224	-216
-2	240	178
-1	386	-355
0	123	155
H= 11 K= 1		
-2	275	-273
H= 11 K= 2		
-2	235	-212
H= 11 K= 3		
-2	297	-250
H= 12 K=-13		
-1	153	210
H= 12 K= -8		
-1	250	258
H= 12 K= -7		
-1	157	181
1	305	296
H= 12 K= -5		
-1	255	235

deviation (hereafter, e.s.d.) and R was 0·12. The scale factors for the different layer lines were within one e.s.d. of their initial values. At this stage we continued the refinement allowing for anisotropic temperature factors of the Pd and S atoms and reducing the number of variable scale factors to one overall scale factor and taking the scale factors obtained from the interscaling procedure before refining. This new series of refinement cycles converged after three cycles, the R value having decreased to 0·098 with an $R'(=\sum\omega\Delta^2/\sum F_o^2)$ value of 0·111. With the structure factors calculated in the last cycle we computed a three-dimensional difference Fourier synthesis. The resulting map was featureless, and had an electron density varying between +2 and −2 e.Å^{-3}, whereas in the original Fourier synthesis the light atoms were located at peaks of at least 4·5 e.Å^{-3}. Table 1 gives the coordinates and temperature factors used in the last structure factor calculation, Table 2 contains the values of the observed and calculated structure factors. Throughout the refinement we used atomic scattering factors for neutral atoms taken from a paper by Cromer & Waber (1965), without making corrections for anomalous dispersion. The effect of neglecting anomalous dispersion was tested by a least-squares calculation using corrected scattering factors for Pd. This calculation gave the same R value of 0·098 and shifts less than one-fifth of their e.s.d. for all parameters except the overall scale factor. We looked for evidence of extinction effects analyzing the values of $\sum|F_o|/\sum|F_c|$ for the reflections at $\sin\theta/\lambda<0{\cdot}35$ Å, and found that this ratio was $\sim 1{\cdot}01$, indicating that this effect can be neglected here. We did not include hydrogen atoms in our structure factor calculations since this would lengthen our computation time and would have negligible effect on the values of the distances we feel are worth analyzing in detail in this structure determination, namely those involving the S atoms. We used Hughes's weighing scheme considering it was suitable because with this scheme the average value of $\omega\Delta^2$ is relatively constant in ranges of F_o and of $\sin\theta/\lambda$.

Description of the structure

There are two crystallographically independent $[Pd(en)_2]^{2+}$ ions in the unit cell, each of them located on a center of symmetry, and one crystallographically independent anion, $[Pd(S_2O_3)_2(en)]^{2-}$ in a general position. Fig. 1 shows a projection of the contents of the unit cell on the (001) plane of the set of orthogonal coordinates obtained by the transformation:

$$X'=(8{\cdot}663)x+(-3{\cdot}674)y$$

$$Y'=(12{\cdot}096)y$$

$$Z'=(-1{\cdot}300)x+(-4{\cdot}154)y+(8{\cdot}660)z$$

The packing of the ions is such that each cation is at the center of a distorted octahedron having the center S atom of the S_2O_3 groups at the vertices. The average distance between these S atoms and the Pd atom of the nearest cation is 4·8 Å. Each of the S atoms defining these octahedra is shared by three octahedra, so that the cation of charge +2 is at the center of an octahedron having formal charges $-\frac{1}{3}$ at the vertices. In turn, the anions are at the centers of distorted tetrahedra determined by the Pd atoms of the cations. Each cation is shared by four of these tetrahedra, contributing a charge of $+\frac{1}{2}$ to each. The distance between nearest anions is shorter than between nearest cations; the shortest distance between anions is of 5·3 Å, being that between a Pd atom and a S atom of two anions related by the symmetry center.

The coordination around the Pd atoms has no unusual features. The three crystallographically independent ions have the expected square planar coordination around Pd(II), although in the anion there is significant departure from coplanarity and the angles around the Pd differ significantly from 90°. The S_2O_3

groups coordinate through their terminal S atom and form nearly tetrahedral Pd–S–SO_3 angles. The nonlinearity of this bond angle can account for the splitting of the asymmetric S–O stretching modes observed in the infrared spectrum of this and other complex thiosulphates (Costamagna & Levitus, 1966). The (en) ligands take the *gauche* conformation without a significant change in bond distances and angles with respect to the free ligand, the coordinated S_2O_3 group maintains the pyramidal shape of the free ion but there is a significant lengthening of the S–S bond. Values of the bond distances and angles are shown in Table 3, together with their e.s.d.'s. The latter were calculated taking into account correlation and unit cell errors, and for the C–N, S–O, and Pd–N distances these values are almost equal to the values of the e.s.d. of a single observation as calculated using the corresponding sets of chemically equivalent distances (for the six C–N distances we calculate a e.s.d. in a single observation of 0·024 Å, and similarly we calculate e.s.d.'s of 0·013 Å and 0·016 Å for the S–O and Pd–N distances, respectively).

The mean value of the Pd–N bond length is 2·071 ± 0·007 Å, nearly equal to the value determined in other Pd(II) complexes having N and S atoms in their coordination sites, *i.e.* 2·060 in $Na_2[Pd(SO_3)_2(NH_3)_2].6H_2O$ (Capparelli & Becka, 1969). The average dimensions of the (en) ligands are C–N 1·484 ± 0·010 Å, C–C 1·527 ± 0·018 Å, N–C–N 108·6 ± 1·0°, practically the same as those of the free ligand and the C–N and C–C distances are within one e.s.d. of very accurately determined values published recently [*i.e.* $T_{\mathrm{C-C}} = 1\cdot519$ (2) $T_{\mathrm{C-N}} = 1\cdot483$ (2) (O'Connell, 1969)]. None of the six crystallographically independent S–O bond lengths or their mean value, 1·457 ± 0·007 Å, differs significantly from the S–O bond length determined in $MgS_2O_3.6H_2O$ (Baggio, Amzel & Becka, 1969) at the 5% level of a '*t*' distribution. Thus with the accuracy of our results we cannot state that there is a correlation between a longer S–O bond and the shift to higher frequencies of the S–O stretching modes in the S bonded thiosulphate complexes.

There is a significant lengthening, even at the 0·1% level of a '*t*' distribution, of the S–S bonds of the coordinated S_2O_3, 2·061 and 2·072 Å with a mean of 2·066 ± 0·006 Å as compared with the length of 2·013 ± 0·003 determined for this bond in $MgS_2O_3.6H_2O$. Thus, it is clear that the S–S bond is weaker and in consequence the S–O bond should be stronger, in agreement with the interpretation of the infrared spectrum of the complexes. The Pd–S bond distances of the two S_2O_3 groups are 2·282 ± 0·006 and 2·312 ± 0·006 Å, both smaller than the sum of covalent radii of Pd and S [2·36 Å, according to Pauling (1960)]. The difference between the two Pd–S distances is significant at the 5% level of a '*t*' distribution, and the ratio of the difference between these two distances and their e.s.d.'s, $t=(r_1-r_2)/(\sigma_1^2+\sigma_2^2)^{1/2}$ is larger than any such ratio calculated for the other pairs of chemically equivalent bonds. In recently published fairly

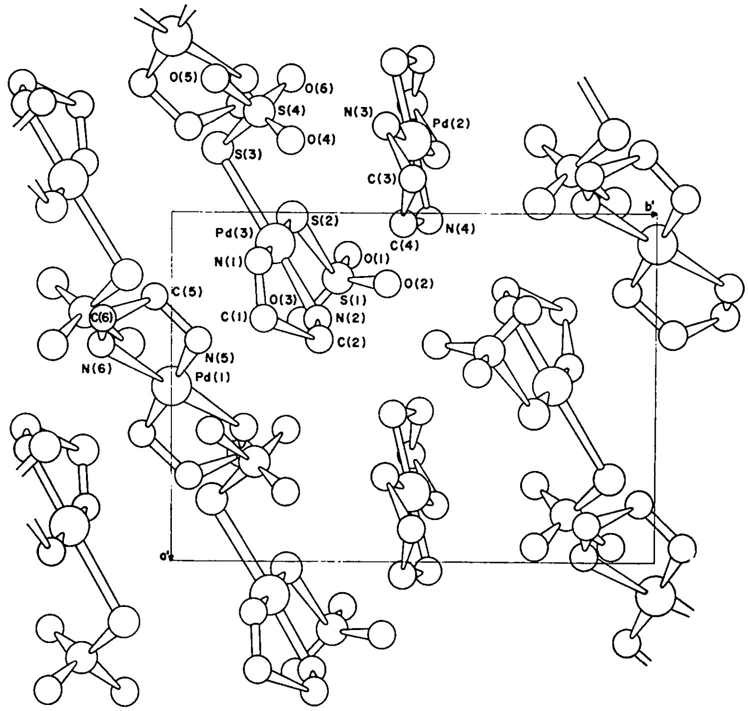

Fig. 1. Projection down the *c* axis of the unit cell. The rectangle outlined by *a'*, *b'* corresponds to the cartesian coordinates defined in the text. Atoms are numbered as in Table 1.

accurate structure determinations of coordination compounds involving non-chelating S bonding ligands, *i.e.* $NiCl_2(SCNH[CH_2]_3NH)_4$ (Luth & Truter, 1968), $Ni(NCS)_2(SC[NHC_2H_5]_2)_4$ (Amzel, Baggio & Becka, 1969), $Ni(SC[NH_2]_2)_4\,S_2O_3\,.\,H_2O$ (Fava Gasparri, Mangia, Musatti & Nardelli, 1969), $Ni(SC[NH_2]_2)_4Br_2$ (Weininger, O'Connor & Amma, 1969), there also are significant differences at the 5% level between crystallographically independent metal–S bonds and the *t* values are also larger for these bonds than for the other chemically equivalent bonds. Finally, we think that the correct structural formula for the compounds previously formulated as $[Pt(NH_3)_4]_3[M(S_2O_3)_4]$ (M = Pt or Pd) is the same as for the compound studied by us, namely $[Pt(NH_3)_4]\,[M(S_2O_3)_2(NH_3)_2]$.

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Table 3. *Bond distances* (Å) *and angles*(°)

The e.s.d.'s are given in parentheses in units of the last decimal place.

Distances		Angles	
Pd(1)–N(6)	2·059 (14)	N(S) –Pd(1)–N(6)	83·1 (1·0)
Pd(1)–N(5)	2·048 (14)		
Pd(2)–N(4)	2·085 (15)	N(4) –Pd(2)–N(3)	94·7 (1·0)
Pd(2)–N(3)	2·074 (13)		
Pd(3)–N(2)	2·091 (15)	N(2) –Pd(3)–N(1)	83·3 (1·0)
Pd(3)–N(1)	2·070 (13)	N(2) –Pd(3)–S(2)	101·9 (0·8)
Mean Pd——N	2·071 (07)	N(2) –Pd(3)–S(3)	171·3 (0·8)
Pd(3)–S(2)	2·282 (06)	N(1) –Pd(3)–S(2)	174·8 (0·8)
Pd(3)–S(3)	2·312 (06)	N(1) –Pd(3)–S(3)	90·9 (0·8)
Mean Pd——S	2·297 (06)	S(2)—Pd(3)–S(3)	83·9 (0·5)
S(1)—S(2)	2·061 (06)	Pd(3)–S(2)—S(1)	110·1 (0·5)
S(3)—S(4)	2·072 (06)	Pd(3)–S(3)—S(4)	105·3 (0·5)
Mean S——S	2·066 (06)		
S(1)—O(2)	1·441 (12)	O(1)—S(1)—O(2)	112·0 (1·0)
S(1)—O(3)	1·445 (14)	O(2)—S(1)—O(3)	109·4 (1·0)
S(1)—O(1)	1·456 (12)	O(3)—S(1)—O(1)	114·4 (1·0)
S(4)—O(6)	1·460 (13)	O(5)—S(4)—O(6)	112·6 (1·0)
S(4)—O(5)	1·478 (14)	O(6)—S(4)—O(1)	108·6 (1·0)
S(4)—O(4)	1·461 (14)		
Mean S——O	1·457 (07)	Mean O——S——O	111·4 (0·6)
N(2) –C(2)	1·490 (20)	N(2) –C(2)—C(1)	106·5 (1·5)
N(1) –C(1)	1·463 (24)	N(1) –C(1)—C(2)	109·9 (1·4)
N(6) –C(6)	1·465 (22)	N(6) –C(6)—C(5)	108·2 (1·4)
N(5) –C(5)	1·522 (24)	N(5) –C(5)—C(6)	106·1 (1·4)
N(3) –C(3)	1·489 (22)	N(3) –C(3)—C(4)	109·9 (1·5)
N(4) –C(4)	1·474 (21)	N(4) –C(4)—C(3)	110·9 (1·5)
Mean N——C	1·484 (10)	Mean N——C——N	108·6 (1·0)
C(1)—C(2)	1·571 (25)	Pd(1)–N(6) –C(6)	109·6 (1·1)
C(3)—C(4)	1·487 (27)	Pd(1)–N(5) –C(5)	109·3 (1·1)
C(5)—C(6)	1·523 (25)	Pd(2)–N(3) –C(3)	105·9 (1·1)
Mean C——C	1·527 (19)	Pd(2)–N(4) –C(4)	105·8 (1·1)
		Pd(3)–N(1) –C(1)	107·6 (1·1)
		Pd(3)–N(2) –C(2)	110·9 (1·1)

Dihedral angles (°) for (en) groups

Pd(1) N(5) N(6)	Pd(1) C(5) C(6)	40 (5)
Pd(2) N(3) N(4)	Pd(2) C(3) C(4)	41 (5)
Pd(3) N(1) N(2)	Pd(3) C(1) C(2)	29 (5)

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The Crystal Structure of Acetohydroxamic Acid Hemihydrate

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The structure of acetohydroxamic acid hemihydrate, $CH_3.CO.NHOH.\frac{1}{2}H_2O$, has been determined using three-dimensional X-ray intensities measured with a proportional counter. Atomic positions and anisotropic vibrational parameters have been refined using full-matrix least squares, and a vibrational analysis carried out. The final R value is 0·052, the principal interatomic distances (Å) within the molecule are C–C, 1·505 (6); C–O, 1·245 (6); C–N, 1·333 (6); N–O, 1·400 (5).

Introduction

This study of acetohydroxamic acid,

$$CH_3.CO.NHOH.\tfrac{1}{2}H_2O,$$

is part of a series of investigations of the crystal structures of simple amides. The authors' interest lay in comparing the geometries of the amidic and hydroxamic groups, and in a comparison of the hydrogen bonding arrangement with those found in typical amides.

Crystal data were gathered, and the approximate structure determined, at the University of Birmingham. Three-dimensional refinement was started, using Rollett's block-diagonal least-squares program, on the MERCURY computer at the University of Oxford. Refinement was later recommenced and completed by one of the authors (BHB) at the Atomic Energy Research Establishment, Harwell. The second refinement used a full-matrix least-squares program, and the results of this second refinement are reported here.

Experimental

Acetohydroxamic acid was prepared by heating equivalent proportions of acetamide and hydroxylamine hydrochloride to 100°C. Ethyl acetate was used as a solvent for extracting and recrystallizing the product. During preliminary crystallographic work, it became evident that the acid obtained in this way is hydrated, and the composition was checked by quantitative analysis with the following results:

	%C	%H	%N
Expected for $CH_3CONHOH$	32·0	6·7	18·7
Expected for $CH_3CONHOH.\frac{1}{2}H_2O$	28·6	7·1	16·7
Found	27·4	7·1	16·9

Weissenberg photographs showed that the crystal is orthorhombic. The systematic absences, $h0l$ when $(h+l)=2n+1$ and $0kl$ when $(k+l)=2n+1$, indicated the space group to be either $Pnnm$ or $Pnn2$. A statistical analysis of X-ray intensities in the $0kl$ zone (Howells, Phillips & Rogers, 1950) indicated that this zone is acentric, and the space group is therefore, $Pnn2$. The cell dimensions were obtained from a least-squares fit to 19 interplanar spacings, measured on the three-circle diffractometer using Cu $K\alpha$ radiation ($\lambda=1·5418$ Å). The density was measured by flotation in a mixture of chlorobenzene and bromobenzene. Crystal data are given in Table 1.

Table 1. *Crystal data*

Acetohydroxamic acid hemihydrate $C_2NO_2H_5.\frac{1}{2}H_2O$

$a = 8·406 \pm 0·002$ Å
$b = 12·046 \pm 0·004$
$c = 4·037 \pm 0·001$
$V = 408·8 \pm 0·02$ Å^3
$Z = 4C_2NO_2H_5 + 2H_2O$
$D_{\rm obs} = 1·36$ g.cm^{-3}
$D_{\rm calc} = 1·36$ g.cm^{-3}

Initially, two-dimensional intensity data were measured, using photographic and counter methods, for the $hk0$ and $0kl$ zones. After determination of the ap-