ACTA CHEMICA SCANDINAVICA 24 (1970) 2785–2795

Crystal Structure of Bisethylene-1,2-bis(methylphenylphosphine)palladium(II) Chloride

P. GROTH

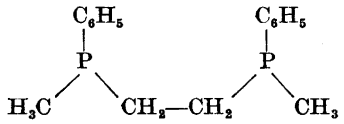
Department of Chemistry, University of Oslo, Oslo 3, Norway

The crystals belong to the orthorhombic system with space group *Pccn* and cell dimensions

$$a = 19.09_9 \text{ Å}, b = 15.71_7 \text{ Å}, c = 11.84_3 \text{ Å}$$

The cell contains four formula units. 1509 reflections were registrated on an automatic four circle diffractometer. The structure was solved by the heavy atom method and refined by full-matrix least squares technique to $R = 7.9$ % ($R_w = 6.0$ %). The complex crystallizes with half a molecule of ethanol per asymmetric unit. The Pd–P distances are 2.33_3 Å and the mean value of the six P–C distances 1.81_5 Å. The complex may be described in terms of a MESO-*anti-trans* form, and the Cl^- ions are situated at distances 4.09_0 Å from Pd.

The two stereoisomeric complexes formed by ruthenium(III) chloride with *meso*-ethylene-1,2-bis-(methyl-phenylphosphine):



have been studied by Horner *et al.*[1] A series of analogous complexes with other metals (Co, Pt, Pd, Ni) have been synthesized by Müller.[2,3]

Since the structural information, that can be obtained by conventional methods (IR, PMR, *etc.*), is somewhat limited, an X-ray analysis of the Pd-complex:

$$[Pd(C_6H_5CH_3PCH_2CH_2PCH_3C_6H_5)_2Cl_2]\cdot C_2H_5OH$$

has been undertaken in order to clarify some of the stereochemical problems concerning these complexes.

The crystals belong to the orthorhombic system, the systematic absences leading to the space group *Pccn*. Cell dimensions, determined by a manual four circle diffractometer, with estimated standard deviations [4]* are:

$$a = 19.099(6)\ \text{Å}, b = 15.717(5)\ \text{Å}, c = 11.843(4)\ \text{Å}$$

With four molecules in the unit cell the calculated density ($\varrho_c = 1.356$ gcm^{-3}) is somewhat smaller than the observed value ($\varrho_o = 1.44$ gcm^{-3}). The corresponding difference in molecular weight may be accounted for by assuming four molecules of crystal ethanol per unit cell.

With $2\theta_{max} = 55°$ and Mo$K\alpha$-radiation, about 4000 reflections were registered by an automatic four circle diffractometer. Only 1509 were recorded as observed using an observed-unobserved cutoff at 2.5 $\sigma(I)$. The intensities were corrected for absorption effects.

With the space group *Pccn* and $Z = 4$ the complex must be centrosymmetric and thus Pd situated at the center of symmetry. The structure was solved by the heavy atom method and refined by fullmatrix least squares technique. Anisotropic temperature factors were introduced for Pd, P, and Cl. The weights in least squares were calculated from the standard deviations in intensities, $\sigma(I)$, taken as

$$\sigma(I) = (C_T + (0.02\ C_N)^2)^{\frac{1}{2}}$$

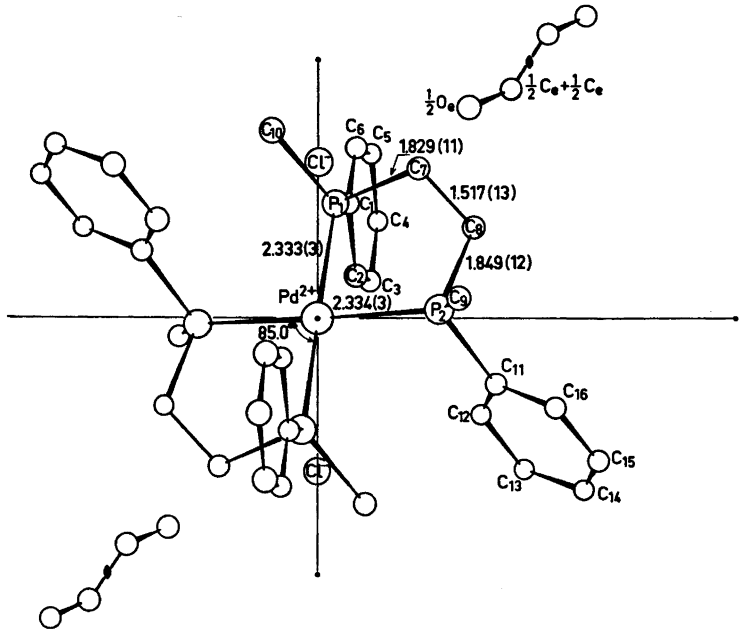


Fig. 1. Schematical drawing of the complex viewed along [001].

* All programs used are included in this reference.

Table 1. Fractional atomic coordinates with estimated standard deviations (multiplied by 10^5).[a] Isotopic temperature factors for oxygen, carbon and hydrogen.[b]

Atom	x	y	z	B (Å^2)
Pd	50000	0	50000	
	0	0	0	
Cl	35090	−00134	74789	
	14	24	28	
P_1	38933	01896	42028	
	15	17	29	
P_2	49377	14430	54105	
	18	17	25	
C_1	39624	03624	27041	2.79
	63	63	97	25
C_2	45993	04287	22215	2.81
	59	66	102	26
C_3	46523	06054	10937	3.38
	61	70	110	27
C_4	40712	07075	04169	4.33
	69	75	110	31
C_5	34132	06273	08933	4.12
	67	75	113	31
C_6	33516	04568	20615	3.19
	61	68	108	28
C_7	35564	11825	47978	2.96
	54	65	95	25
C_8	41332	18439	47221	3.31
	58	69	101	28
C_9	48192	16651	68974	4.07
	58	73	105	31
C_{10}	31911	−05521	44199	3.60
	59	70	106	29
C_{11}	56313	21189	49054	2.35
	50	62	104	23
C_{12}	59364	19468	38706	2.98
	58	66	103	27
C_{13}	64712	24741	34448	4.14
	61	82	104	30
C_{14}	66787	31701	40869	4.20
	62	76	112	31
C_{15}	63864	33448	51107	4.18
	60	73	120	28
C_{16}	58655	28200	55060	3.34
	60	69	103	27
C_e	28605	22850	75328	4.89
	59	75	113	34
O_e	29532	17651	84111	4.41
	77	97	142	39
H_2	51072	04924	25844	4.0
H_3	51168	07085	05166	3.4
H_4	40716	07812	−03161	4.0
H_5	30391	07406	04921	5.6
H_6	29210	03554	23922	2.6
H_{12}	58054	14549	34637	2.2
H_{13}	66169	23167	29249	3.8
H_{14}	69389	34376	37938	4.3
H_{15}	64872	37860	55868	6.4
H_{16}	57022	29056	61876	3.0
$H_{7,1}$	32357	13599	44491	3.2

Table 1. Continued.

Atom	x	y	z	B (Å²)
$H_{7,2}$	35412	09910	54823	3.0
$H_{8,1}$	40703	22484	51809	4.7
$H_{8,2}$	43224	19716	39254	2.5
$H_{9,1}$	46148	12972	71656	3.3
$H_{9,2}$	46577	22469	69083	2.4
$H_{9,3}$	53135	15683	72544	2.8
$H_{10,1}$	31698	−06694	51060	1.0
$H_{10,2}$	32952	−10622	40561	1.4
$H_{10,3}$	27821	−03030	41047	1.1

[a] For numbering of the atoms, see Fig. 1.
[b] The hydrogen atoms H_m (or $H_{m,n}$) are bonded to C_m. Estimated standard deviations are about 10 times those of the carbon atoms.

Table 2. (a) Anisotropic thermal vibration parameters for Pd, P, and Cl with estimated standard deviations (multiplied by 10^5).

Atom	B_{11}	B_{22}	B_{33}	B_{12}	B_{13}	B_{23}
Pd	151	264	671	3	−123	−36
	3	5	11	11	16	17
Cl	221	450	773	100	−9	100
	9	14	30	29	31	47
P_1	109	286	668	1	−72	34
	9	16	32	20	30	37
P_2	174	250	612	2	−138	−80
	10	13	32	24	43	30

(b) The principal axes of the thermal vibration ellipsoids given by the components of a unit vector in fractional coordinates e_x, e_y, e_z; the corresponding r.m.s. amplitudes, and the B-values.

Atom	e_x	e_y	e_z	$(\overline{u^2})^{\frac{1}{2}}$ (Å)	B (Å²)
	.016	.006	−.080	.244	3.96
Pd	−.003	.063	.007	.181	2.60
	.050	.002	.026	.160	2.02
	.015	.051	.044	.249	4.89
Cl	.016	.028	−.071	.230	4.16
	.048	−.025	.011	.194	2.96
	−.008	.008	.083	.220	3.81
P_1	.002	.063	−.010	.189	2.81
	.052	−.001	.013	.140	1.54
	.023	.013	−.074	.220	3.81
P_2	.025	−.056	.003	.177	2.48
	.040	.028	.040	.165	2.16

Table 3. Observed and calculated structure factors on ten times absolute scale. The columns are h, k, l, F_o and F_c.

0	0	2	2973	3112	1	2	2	618	634	2	1	6	560	- 577	2	12	5	413	- 412
0	0	4	3581	3504	1	2	3	273	232	2	1	7	211	229	2	14	0	990	989
0	0	6	384	- 345	1	2	4	492	483	2	1	8	273	- 278	2	14	1	392	- 423
0	0	8	894	927	1	2	6	275	- 272	2	1	9	569	- 760	2	14	2	1084	1100
0	0	10	551	489	1	2	7	540	- 517	2	1	13	346	- 129	2	14	4	538	540
0	1	2	189	166	1	2	10	260	- 271	2	2	0	1320	1254	2	14	5	264	263
0	1	4	618	- 546	1	3	0	1516	-1595	2	2	1	509	- 512	2	14	6	520	569
0	1	6	1108	-1097	1	3	1	635	652	2	2	2	808	859	2	14	7	302	257
0	2	0	3061	3083	1	3	2	480	- 414	2	2	3	840	- 839	2	14	8	331	223
0	2	2	235	142	1	3	3	1221	1230	2	2	4	907	896	2	15	1	294	- 276
0	2	4	2117	2056	1	3	4	195	169	2	2	5	306	- 282	2	15	2	399	295
0	2	6	959	- 874	1	3	5	285	240	2	2	6	833	840	2	16	2	401	395
0	2	8	779	742	1	3	7	644	610	2	2	8	800	846	2	16	3	307	122
0	2	10	544	444	1	3	9	693	616	2	2	10	579	565	2	16	4	333	252
0	2	12	857	616	1	3	11	800	691	2	2	12	353	421	2	18	4	334	197
0	2	14	455	306	1	3	13	670	672	2	3	1	551	538	2	18	6	463	349
0	3	2	1628	-1645	1	4	1	1432	1436	2	3	3	1554	1465	2	20	0	498	376
0	3	4	313	- 295	1	4	4	633	600	2	3	4	283	- 232	3	0	2	208	- 208
0	3	6	216	- 227	1	4	5	507	497	2	3	6	170	- 115	3	0	4	879	879
0	3	10	293	- 176	1	4	8	367	- 311	2	3	7	245	280	3	0	6	339	319
0	4	0	3323	3358	1	4	9	267	- 230	2	3	9	637	- 630	3	0	8	217	- 191
0	4	2	768	- 691	1	4	10	292	- 251	2	4	0	588	624	3	1	0	651	565
0	4	4	1584	1599	1	5	0	788	- 778	2	4	1	1275	1265	3	1	1	1788	1728
0	4	6	335	- 370	1	5	1	2203	2258	2	4	2	1829	1791	3	1	2	285	- 164
0	4	8	1219	1153	1	5	2	364	353	2	4	3	542	561	3	1	3	1400	1421
0	4	10	532	407	1	5	3	1239	1225	2	4	4	665	671	3	1	4	210	- 138
0	4	12	1047	865	1	5	4	189	- 142	2	4	5	254	242	3	1	5	1572	1656
0	5	2	323	- 303	1	5	5	897	861	2	4	6	1190	1136	3	1	7	939	1053
0	5	6	388	366	1	5	6	635	607	2	4	8	864	798	3	1	8	218	263
0	5	8	312	343	1	5	7	663	670	2	4	10	925	873	3	1	9	323	461
0	6	0	3014	3132	1	5	9	836	782	2	4	12	530	481	3	1	11	337	406
0	6	2	1050	1060	1	5	10	286	- 251	2	5	1	844	- 833	3	2	1	441	- 461
0	6	4	1691	1746	1	5	11	678	586	2	5	2	624	602	3	2	2	554	523
0	6	6	358	335	1	5	13	490	342	2	5	3	862	864	3	2	3	306	- 286
0	6	8	818	785	1	6	1	1336	1313	2	5	4	541	500	3	2	5	840	- 825
0	6	10	349	332	1	6	2	532	541	2	5	5	306	- 359	3	2	6	237	- 253
0	6	12	606	496	1	6	3	760	739	2	5	6	666	670	3	2	7	601	- 746
0	7	2	272	308	1	6	4	107	114	2	5	8	354	314	3	2	8	331	- 340
0	7	4	653	688	1	6	5	988	940	2	5	9	718	- 696	3	2	10	281	- 256
0	7	6	282	- 264	1	7	0	384	- 394	2	6	0	1837	1919	3	3	0	2520	-2496
0	7	8	270	- 228	1	7	1	2401	2499	2	6	1	333	319	3	3	1	451	- 401
0	8	0	1021	1075	1	7	2	484	493	2	6	2	1598	1656	3	3	2	468	- 435
0	8	2	489	554	1	7	3	1279	1358	2	6	4	1358	1344	3	3	3	1149	1145
0	8	4	1567	1608	1	7	4	244	232	2	6	5	429	450	3	3	4	360	- 295
0	8	6	433	418	1	7	5	804	753	2	6	6	1026	967	3	3	5	977	1026
0	8	8	728	687	1	7	9	292	200	2	6	7	202	277	3	3	6	381	374
0	8	10	585	500	1	7	11	310	113	2	6	8	414	349	3	3	7	1009	1245
0	9	6	232	- 280	1	8	1	194	197	2	6	10	386	390	3	3	9	720	775
0	9	8	371	- 337	1	8	2	353	339	2	7	1	621	- 601	3	4	1	143	- 118
0	9	10	341	- 338	1	8	3	458	- 469	2	7	2	820	810	3	4	2	620	564
0	10	0	558	595	1	8	4	331	422	2	7	4	290	311	3	4	4	581	543
0	10	2	655	681	1	8	7	601	- 610	2	7	5	419	- 389	3	4	6	184	130
0	10	4	811	801	1	8	8	302	- 360	2	7	7	268	222	3	4	7	391	419
0	10	6	369	325	1	8	9	359	- 320	2	7	9	374	- 322	3	4	8	267	- 255
0	10	8	970	933	1	9	0	419	- 423	2	8	0	964	1011	3	4	9	246	258
0	10	10	425	432	1	9	1	1392	1440	2	8	1	611	- 595	3	4	10	257	- 298
0	10	12	723	582	1	9	2	906	932	2	8	2	1526	1615	3	5	0	782	- 786
0	11	2	769	781	1	9	5	645	661	2	8	3	250	- 241	3	5	1	1654	1701
0	11	4	735	791	1	9	6	438	396	2	8	4	777	788	3	5	3	1336	1347
0	11	6	409	421	1	9	7	618	629	2	8	5	211	220	3	5	4	218	213
0	11	8	265	- 116	1	9	9	704	743	2	8	6	831	819	3	5	5	1298	1359
0	12	0	800	809	1	9	11	584	507	2	8	8	307	190	3	5	6	438	401
0	12	2	402	430	1	9	13	371	365	2	8	10	410	313	3	5	7	1138	1131
0	12	4	674	617	1	10	1	352	340	2	9	1	870	- 906	3	5	9	708	657
0	12	6	465	481	1	10	9	265	- 239	2	9	2	172	- 171	3	5	11	509	508
0	12	8	550	519	1	11	0	282	- 270	2	9	3	491	491	3	6	2	434	414
0	12	12	474	406	1	11	1	611	618	2	9	4	281	- 299	3	6	3	476	456
0	13	2	781	823	1	11	3	346	382	2	9	5	633	- 660	3	6	4	526	535
0	13	4	657	640	1	11	5	518	510	2	9	6	424	- 410	3	6	5	506	518
0	13	6	281	312	1	11	7	529	414	2	9	7	284	296	3	6	6	206	153
0	14	0	1012	993	1	11	9	553	549	2	9	8	244	- 298	3	6	7	357	392
0	14	2	873	932	1	11	11	444	374	2	9	9	298	- 265	3	6	9	403	420
0	14	4	854	865	1	12	2	435	399	2	10	0	425	421	3	7	0	854	- 869
0	14	6	349	250	1	12	5	544	519	2	10	1	562	- 603	3	7	1	1652	1709
0	14	8	358	383	1	12	6	209	- 195	2	10	2	864	910	3	7	3	1367	1406
0	15	1	358	- 438	1	13	1	681	684	2	10	4	501	478	3	7	5	1160	1152
0	16	0	620	605	1	13	3	617	577	2	10	5	428	411	3	7	6	489	495
0	16	2	382	336	1	13	5	662	679	2	10	6	810	773	3	7	7	838	779
0	16	4	344	299	1	13	7	310	310	2	10	7	386	366	3	7	9	332	273
0	16	6	479	425	1	14	1	622	656	2	10	8	558	527	3	8	1	426	- 455
0	17	2	301	320	1	14	3	685	677	2	10	10	635	553	3	8	2	493	510
0	18	0	356	382	1	14	5	472	530	2	10	12	468	384	3	8	3	245	- 228
0	18	4	505	- 497	1	15	2	517	408	2	11	1	506	- 545	3	8	4	322	281
0	20	0	369	383	1	17	2	306	- 292	2	11	2	347	365	3	8	5	552	- 587
1	0	2	195	- 172	1	17	3	302	256	2	11	3	470	444	3	8	7	338	- 264
1	0	4	897	882	1	17	5	405	395	2	11	7	309	298	3	8	8	251	- 239
1	0	6	383	375	1	17	7	454	345	2	12	0	521	447	3	9	0	625	- 640
1	1	1	2968	2786	1	18	5	417	346	2	12	1	595	- 632	3	9	1	411	443
1	1	2	820	676	2	0	0	3247	3398	2	12	2	741	775	3	9	3	906	921
1	1	3	2175	2123	2	0	2	3138	3058	2	12	4	537	513	3	9	5	598	590
1	1	4	622	- 600	2	0	4	1836	1753	2	12	5	281	206	3	9	6	392	395
1	1	5	1274	1269	2	0	6	1329	1358	2	12	6	645	620	3	9	7	731	671
1	1	7	598	624	2	0	8	489	478	2	12	7	306	290	3	9	9	587	455
1	1	9	420	384	2	0	10	598	552	2	12	8	304	244	3	9	11	354	209
1	1	11	419	416	2	1	1	561	623	2	12	10	448	374	3	9	13	361	226
1	1	13	451	345	2	1	2	1109	-1071	2	13	1	474	- 513	3	10	1	195	- 244
1	2	1	396	390	2	1	3	2138	2098	2	13	3	404	409	3	10	2	218	272
					2	1	5	444	406	2	13	4	366	363	3	10	3	417	- 448

Table 3. Continued.

3	10	5	265	- 231
3	10	6	315	155
3	11	1	632	620
3	11	3	541	496
3	11	5	581	546
3	11	7	629	613
3	11	8	280	114
3	11	9	585	504
3	11	11	421	395
3	12	7	315	348
3	13	0	345	- 338
3	13	1	1024	1041
3	13	2	284	- 254
3	13	3	935	917
3	13	5	742	700
3	13	7	426	423
3	14	2	298	15
3	15	1	565	617
3	15	3	622	606
3	15	6	268	294
3	15	8	446	366
3	15	7	332	254
3	15	9	327	191
3	16	3	278	- 301
3	16	5	338	- 295
3	19	1	379	344
3	19	2	339	- 109
4	0	0	1149	-1128
4	0	2	1103	1092
4	0	4	951	996
4	0	6	1942	1889
4	0	8	471	376
4	0	10	488	424
4	1	1	736	654
4	1	2	1121	-1095
4	1	3	895	- 899
4	1	4	187	- 191
4	1	5	349	349
4	1	6	933	- 342
4	1	8	442	- 513
4	1	9	253	- 203
4	2	0	109	- 86
4	2	2	2536	2435
4	2	3	313	241
4	2	4	380	399
4	2	5	104	- 138
4	2	6	2018	2112
4	2	8	702	809
4	2	10	484	736
4	3	1	1088	1038
4	3	4	462	- 450
4	3	5	604	571
4	3	6	377	- 371
4	3	7	407	- 519
4	4	0	788	- 747
4	4	1	351	- 339
4	4	2	1746	1713
4	4	4	926	918
4	4	5	341	319
4	4	6	1645	1751
4	4	8	898	919
4	4	10	785	903
4	5	1	618	618
4	5	3	493	- 667
4	5	4	558	511
4	5	6	712	683
4	5	8	483	527
4	5	10	294	189
4	6	0	810	817
4	6	1	363	- 373
4	6	2	1716	1766
4	6	4	781	777
4	6	6	1722	1722
4	6	7	256	296
4	6	8	246	138
4	6	10	348	340
4	7	1	376	389
4	7	2	557	- 513
4	7	4	391	- 373
4	7	5	301	342
4	7	6	211	- 123
4	7	7	353	- 232
4	7	8	433	461
4	8	0	693	733
4	8	1	370	- 339
4	8	2	1772	1821
4	8	4	871	854
4	8	6	1059	1021
4	8	8	406	338
4	8	10	430	442
4	9	1	354	370
4	9	2	658	- 668
4	9	3	260	- 278
4	9	4	600	- 563
4	9	5	268	228
4	9	6	589	- 559
4	9	7	288	- 334
4	10	1	181	- 137
4	10	2	660	665
4	10	3	229	84
4	10	4	308	352
4	10	5	223	297
4	10	6	1067	1062
4	10	8	511	416
4	10	10	627	559
4	11	2	241	- 223
4	11	4	253	- 306
4	11	5	304	290
4	11	8	281	337
4	12	0	455	461
4	12	2	784	815
4	12	4	423	446
4	12	6	748	727
4	12	8	392	248
4	12	10	500	446
4	13	7	298	- 201
4	14	0	873	864
4	14	2	1047	1028
4	14	4	555	517
4	14	6	567	576
4	14	10	184	225
4	15	4	295	- 292
4	16	2	574	490
4	16	6	355	338
4	17	4	326	- 247
4	18	6	351	249
5	0	2	157	- 68
5	0	4	459	- 484
5	0	6	457	- 416
5	1	0	851	- 874
5	1	1	1395	1398
5	1	2	423	417
5	1	3	2264	2251
5	1	4	210	198
5	1	5	1578	1700
5	1	7	1111	1204
5	2	1	1216	-1132
5	2	2	697	- 693
5	2	3	1158	-1119
5	2	4	326	- 323
5	2	5	765	- 829
5	2	7	548	- 623
5	3	0	314	326
5	3	1	639	642
5	3	2	322	- 306
5	3	3	1047	1037
5	3	4	200	- 161
5	3	5	934	975
5	3	7	1475	1602
5	3	9	478	791
5	4	1	899	854
5	4	2	308	309
5	4	3	273	219
5	4	4	185	- 173
5	4	6	215	206
5	4	7	501	507
5	5	1	1045	1081
5	5	7	877	987
5	5	9	411	474
5	5	10	263	177
5	6	1	448	- 443
5	6	2	242	- 268
5	6	3	443	- 419
5	6	5	224	- 172
5	6	6	226	- 128
5	6	7	355	342
5	6	11	300	178
5	7	0	265	- 268
5	7	1	1302	1347
5	7	2	396	- 368
5	7	3	1164	1177
5	7	4	358	- 347
5	7	5	1288	1230
5	7	7	843	891
5	7	9	468	602
5	8	1	337	- 341
5	8	2	168	- 110
5	8	3	590	- 619
5	8	4	383	- 386
5	8	5	393	- 370
5	8	11	294	70
5	9	1	595	635
5	9	2	174	165
5	9	3	578	545
5	9	5	716	701
5	9	7	720	652
5	9	9	559	533
5	9	11	384	233
5	10	1	181	- 199
5	10	3	346	- 350
5	10	5	406	- 433
5	11	0	624	620
5	11	2	400	446
5	11	3	376	363
5	11	5	803	796
5	11	7	581	563
5	11	9	665	653
5	11	11	341	372
5	12	5	284	291
5	12	7	440	449
5	12	9	368	366
5	13	1	1122	1135
5	13	3	739	756
5	13	5	676	695
5	13	7	396	321
5	13	9	340	309
5	14	1	269	- 210
5	15	1	663	651
5	15	3	580	596
5	15	5	461	479
5	16	3	361	- 401
5	17	1	305	255
5	19	1	520	445
6	0	0	3267	3300
6	0	2	693	709
6	0	4	1833	1885
6	0	6	711	752
6	0	8	723	690
6	1	1	317	307
6	1	2	459	- 422
6	1	3	1031	- 991
6	1	4	344	- 326
6	1	5	426	- 459
6	1	7	411	- 457
6	2	0	2355	2404
6	2	1	706	674
6	2	2	1037	1014
6	2	3	266	242
6	2	4	1944	1980
6	2	5	647	639
6	2	8	1058	1154
6	2	10	327	370
6	3	1	794	731
6	3	2	991	- 922
6	3	3	1133	-1082
6	3	4	292	- 300
6	3	5	244	- 307
6	3	9	284	441
6	4	0	421	455
6	4	1	396	357
6	4	2	593	- 488
6	4	3	478	- 420
6	4	4	1166	1142
6	4	5	351	- 382
6	4	6	722	765
6	4	7	456	- 513
6	4	8	864	1084
6	4	10	273	370
6	5	3	661	- 678
6	5	4	232	197
6	5	6	377	415
6	5	8	607	751
6	5	9	332	345
6	6	0	1713	1778
6	6	1	592	667
6	6	2	857	869
6	6	4	1912	1881
6	6	6	673	683
6	6	7	335	- 385
6	6	8	680	736
6	6	13	343	141
6	7	2	641	- 624
6	7	3	974	- 940
6	7	6	334	- 350
6	7	7	220	- 216
6	8	0	1598	1645
6	8	1	297	298
6	8	2	962	978
6	8	4	1559	1543
6	8	5	230	- 232
6	8	6	781	799
6	8	7	282	- 296
6	8	8	813	883
6	9	2	280	- 285
6	9	3	480	- 465
6	9	4	409	- 435
6	9	6	498	- 478
6	10	1	373	341
6	10	4	860	860
6	10	6	389	355
6	10	7	324	- 300
6	10	8	740	786
6	10	10	505	461
6	11	3	360	- 329
6	11	5	289	264
6	12	0	1088	1063
6	12	1	304	335
6	12	2	345	381
6	12	4	778	755
6	12	6	371	393
6	12	8	632	632
6	12	10	392	221
6	14	0	810	853
6	14	2	529	533
6	14	4	791	740
6	14	6	312	169
6	14	8	342	370
6	15	2	291	- 133
6	16	0	658	647
6	16	2	322	296
6	16	4	435	444
6	17	4	373	- 230
6	18	0	453	466
7	0	2	1449	-1412
7	0	4	356	- 340
7	0	6	605	- 615
7	0	8	548	574
7	1	0	212	- 217
7	1	1	1861	1841
7	1	2	359	320
7	1	3	1588	1573
7	1	4	202	194
7	1	5	1242	1272
7	1	7	603	689
7	1	9	313	308
7	2	2	803	- 743
7	2	3	540	- 513
7	2	5	513	- 493
7	2	7	339	- 407
7	2	9	531	685
7	3	1	1041	1058
7	3	2	343	378
7	3	3	1341	1319
7	3	5	818	820
7	3	7	787	938
7	3	9	604	939
7	3	10	279	- 244
7	3	11	319	605
7	4	1	374	363
7	4	2	665	- 616
7	4	3	735	702
7	4	4	283	- 286
7	4	5	340	387
7	4	8	249	335
7	4	11	279	188
7	5	0	568	556
7	5	1	565	624
7	5	2	494	477
7	5	3	440	461
7	5	4	213	192
7	5	5	666	690
7	5	7	406	422
7	5	9	409	332
7	5	11	339	303
7	6	1	221	244
7	6	2	441	- 432
7	6	4	577	- 585
7	6	5	201	188
7	6	6	403	- 414
7	7	0	383	307
7	7	1	1791	1790
7	7	2	497	491
7	7	3	1303	1292
7	7	5	971	950
7	7	6	419	- 429
7	7	7	528	564
7	7	9	389	470
7	8	4	426	- 374
7	8	5	244	- 223
7	8	8	310	262
7	9	1	1080	1077
7	9	3	904	915
7	9	4	370	- 368
7	9	5	689	619
7	9	6	323	- 380
7	9	7	518	442
7	9	9	420	494
7	9	11	342	298
7	10	4	300	- 335
7	11	0	496	512
7	11	1	236	321
7	11	3	310	293
7	11	5	603	566
7	11	6	293	- 301
7	11	7	472	482
7	11	9	497	479
7	11	11	542	488
7	12	3	279	264
7	12	5	308	297
7	12	7	344	373
7	13	0	537	565
7	13	1	893	874
7	13	3	629	609
7	13	5	590	589
7	13	6	301	- 309
7	13	7	351	263
7	14	2	253	13
7	15	1	621	555
7	15	3	661	617
7	15	5	385	463
7	17	3	332	221
8	0	0	2047	2165
8	0	2	1567	1570
8	0	4	1520	1495
8	0	6	547	540
8	0	8	375	462
8	1	1	502	- 608
8	1	2	308	- 330
8	1	3	421	467
8	1	4	297	- 310
8	1	5	977	- 985
8	1	6	355	- 364
8	1	8	309	- 322
8	2	0	2040	2075
8	2	1	422	- 414
8	2	2	1278	1289
8	2	3	324	- 264
8	2	4	1221	1149
8	2	5	229	- 208
8	2	6	327	340
8	2	7	288	- 252
8	2	8	625	777
8	2	10	329	484
8	2	12	388	592
8	3	1	990	- 956
8	3	2	239	219
8	3	5	610	- 591
8	3	7	593	754
8	4	0	981	1039
8	4	1	623	611
8	4	2	382	419
8	4	3	831	- 811
8	4	4	499	475
8	4	6	295	- 273

Table 3. Continued.

8	4	4	219	233
8	4	7	372	- 390
8	4	8	574	649
8	4	9	318	360
8	4	10	308	486
8	5	1	500	- 592
8	5	2	1008	1005
8	5	3	354	341
8	5	4	547	564
8	5	5	567	- 585
8	5	6	590	648
8	6	0	2059	2126
8	6	1	799	796
8	6	2	1380	1404
8	6	4	1394	1356
8	6	5	250	215
8	6	6	396	379
8	6	7	385	- 439
8	6	8	367	434
8	6	10	292	304
8	7	1	1085	-1082
8	7	4	232	200
8	7	5	725	- 723
8	7	7	424	457
8	7	8	292	- 322
8	8	0	1975	2055
8	8	2	1292	1286
8	8	4	1012	956
8	8	6	387	393
8	8	7	280	- 285
8	8	8	339	407
8	9	1	524	- 581
8	9	7	420	507
8	9	8	326	- 336
8	10	0	473	584
8	10	1	273	292
8	10	2	315	289
8	10	4	517	518
8	10	6	496	449
8	10	10	360	373
8	11	1	470	- 455
8	11	2	292	322
8	11	3	315	314
8	11	4	255	305
8	11	5	359	- 447
8	12	0	993	977
8	12	2	578	581
8	12	3	264	212
8	12	4	734	715
8	12	6	536	464
8	12	7	302	- 146
8	12	8	423	424
8	12	10	397	317
8	13	1	328	- 339
8	13	3	288	85
8	13	4	307	322
8	13	5	275	- 192
8	14	0	755	721
8	14	2	620	544
8	14	4	560	545
8	14	6	397	268
8	14	8	353	217
8	15	5	319	- 222
8	16	0	395	313
8	16	4	452	419
8	16	6	428	359
8	17	3	332	[illegible]
9	0	2	864	- 825
9	0	6	382	- 419
9	1	0	272	- 305
9	1	1	2273	2234
9	1	2	243	- 227
9	1	3	882	906
9	1	5	638	584
9	1	6	202	220
9	1	8	516	614
9	1	13	407	320
9	2	1	304	- 259
9	2	5	249	- 247
9	2	7	341	- 449
9	2	8	260	- 247
9	2	9	468	- 473
9	3	1	1205	1239
9	3	3	711	693
9	3	5	676	695
9	3	7	317	394
9	3	8	482	741
9	4	2	196	- 213
9	4	3	184	195
9	4	5	442	421
9	4	7	264	352
9	5	1	1543	1556
9	5	2	218	- 171
9	5	3	876	864
9	5	5	399	370
9	5	7	279	232
9	5	8	287	487
9	6	3	832	815
9	6	5	529	520
9	6	6	322	- 365
9	6	11	406	- 103
9	7	1	1751	1806
9	7	2	374	340
9	7	3	1525	1473
9	7	4	322	333
9	7	5	844	814
9	7	7	283	410
9	7	8	276	143
9	8	7	348	- 384
9	8	1	608	634
9	8	2	216	- 226
9	8	3	772	766
9	8	4	336	- 251
9	8	5	288	307
9	8	7	530	494
9	10	2	280	291
9	10	8	322	- 289
9	11	1	594	570
9	11	2	240	- 167
9	11	3	585	595
9	11	5	320	278
9	11	7	501	533
9	11	8	387	357
9	12	1	402	388
9	12	3	523	585
9	12	5	404	427
9	12	8	470	466
9	13	1	637	670
9	13	3	757	741
9	13	5	421	427
9	13	7	388	459
9	15	1	537	489
9	15	3	473	461
9	15	7	502	359
10	0	0	1518	1574
10	0	2	2342	2354
10	0	4	563	531
10	0	6	656	673
10	1	4	340	- 364
10	1	5	485	- 441
10	1	7	339	- 393
10	1	8	424	- 427
10	2	0	681	705
10	2	1	503	- 506
10	2	2	2017	1976
10	2	3	208	- 231
10	2	4	371	348
10	2	5	227	- 252
10	2	6	970	1023
10	2	10	502	660
10	3	1	463	427
10	3	2	355	317
10	3	3	416	422
10	3	4	242	- 236
10	3	5	202	258
10	4	0	496	540
10	4	1	354	- 359
10	4	2	1624	1587
10	4	4	299	322
10	4	6	613	625
10	4	8	308	332
10	4	10	549	793
10	5	1	196	19
10	5	2	459	412
10	5	3	381	376
10	5	4	371	391
10	5	6	445	433
10	5	8	335	- 242
10	6	0	1318	1353
10	6	2	1628	1631
10	6	4	245	121
10	6	6	801	894
10	7	2	589	595
10	7	8	298	- 226
10	8	0	613	591
10	8	1	406	- 355
10	8	2	1332	1347
10	8	4	323	347
10	8	6	525	541
10	8	7	313	285
10	8	10	422	568
10	9	3	440	381
10	9	6	224	- 64
10	9	8	245	- 428
10	10	0	326	337
10	10	1	281	- 248
10	10	2	707	694
10	10	4	638	615
10	10	10	545	600
10	11	2	363	346
10	11	4	349	238
10	11	6	303	143
10	12	8	403	398
10	12	2	839	843
10	12	4	424	382
10	12	6	694	689
10	12	10	360	457
10	13	4	349	462
10	13	6	304	277
10	14	0	557	540
10	14	1	265	- 133
10	14	2	865	876
10	14	4	392	379
10	14	6	490	524
10	14	8	380	- 7
10	16	2	447	479
10	16	6	485	473
11	0	2	552	520
11	0	8	515	- 523
11	0	10	306	- 330
11	1	0	927	- 940
11	1	1	1606	1585
11	1	2	1076	1032
11	1	6	798	765
11	1	8	220	- 39
11	1	7	419	465
11	1	9	367	508
11	2	2	465	459
11	2	3	366	- 331
11	2	4	385	396
11	2	6	374	- 319
11	2	7	264	- 304
11	2	8	328	- 433
11	2	10	316	- 244
11	3	0	183	125
11	3	1	609	607
11	3	3	613	571
11	3	6	588	542
11	3	8	250	277
11	3	7	449	439
11	3	9	437	577
11	4	2	612	617
11	4	4	377	378
11	4	6	215	126
11	4	8	272	279
11	4	7	239	212
11	5	0	245	- 219
11	5	1	1089	1124
11	5	3	940	918
11	5	5	836	801
11	5	7	661	663
11	5	9	434	566
11	6	1	286	217
11	6	3	316	330
11	6	6	386	417
11	6	7	293	268
11	7	0	580	- 561
11	7	1	1313	1338
11	7	3	1203	1206
11	7	5	855	844
11	7	8	282	266
11	7	7	496	459
11	8	3	256	- 238
11	8	5	279	- 325
11	8	8	247	- 144
11	9	0	557	- 577
11	9	1	238	266
11	9	3	444	420
11	9	5	412	329
11	9	8	372	380
11	9	7	567	445
11	9	11	405	349
11	11	0	312	- 277
11	11	1	485	458
11	11	3	528	498
11	11	5	419	458
11	11	8	362	349
11	11	7	600	571
11	11	9	394	361
11	12	3	298	318
11	13	0	307	- 305
11	13	1	654	604
11	13	3	602	642
11	13	5	533	467
11	13	7	454	408
11	15	0	428	- 395
11	15	1	473	499
11	15	3	478	395
12	0	0	1729	1783
12	0	2	1585	1641
12	0	4	1150	1158
12	0	6	716	728
12	0	8	434	347
12	1	1	841	826
12	1	3	198	- 167
12	1	4	259	- 247
12	1	5	687	650
12	1	8	325	- 382
12	1	7	590	- 610
12	2	0	561	600
12	2	2	415	392
12	2	4	1081	1082
12	2	6	864	825
12	2	8	632	654
12	2	10	341	316
12	3	1	1011	1010
12	3	2	270	- 256
12	3	4	269	- 295
12	3	5	698	695
12	3	6	236	- 189
12	3	7	282	- 348
12	4	0	505	456
12	4	2	487	469
12	4	3	256	255
12	4	4	800	734
12	4	6	806	787
12	4	8	727	682
12	4	10	389	448
12	5	1	630	589
12	5	5	613	626
12	5	7	316	- 338
12	6	0	977	1016
12	6	1	232	- 240
12	6	2	778	741
12	6	4	1007	936
12	6	6	592	567
12	6	8	470	619
12	6	10	395	259
12	7	1	354	375
12	7	3	272	- 277
12	7	7	577	- 587
12	8	0	646	607
12	8	1	326	- 339
12	8	2	560	549
12	8	4	786	766
12	8	6	638	651
12	8	7	396	377
12	8	8	522	501
12	9	1	422	411
12	9	3	399	- 331
12	9	5	280	- 246
12	9	6	416	- 433
12	10	0	499	488
12	10	1	416	- 431
12	10	2	391	308
12	10	4	533	470
12	10	6	421	372
12	10	8	568	608
12	10	10	377	355
12	11	5	281	280
12	12	0	496	483
12	12	2	324	376
12	12	4	525	535
12	12	6	374	336
12	12	8	333	389
12	14	0	711	727
12	14	1	357	- 290
12	14	2	603	589
12	14	4	536	551
12	14	6	362	297
12	14	8	463	428
12	16	4	443	311
13	0	2	332	- 292
13	1	1	712	736
13	1	2	248	- 301
13	1	3	1009	931
13	1	4	349	- 300
13	1	5	1161	1134
13	1	6	335	- 303
13	1	7	959	950
13	1	9	433	512
13	2	2	256	248
13	2	3	236	- 200
13	2	5	299	- 261
13	3	0	310	- 331
13	3	3	505	451
13	3	5	822	782
13	3	6	265	317
13	3	7	776	758
13	3	9	562	615
13	4	2	342	345
13	4	4	316	252
13	4	6	269	234
13	4	9	297	45
13	5	1	426	441
13	5	2	209	168
13	5	3	676	654
13	5	4	326	265
13	5	5	879	914
13	5	7	724	774
13	5	9	415	484
13	6	7	406	376
13	7	0	271	- 299
13	7	1	876	907
13	7	3	915	926
13	7	5	874	836
13	7	7	600	476
13	8	1	232	- 260
13	8	2	244	250
13	8	3	485	- 445
13	8	5	376	- 432
13	9	0	408	- 363
13	9	1	487	481
13	9	3	650	621
13	9	5	663	657
13	9	7	531	513
13	10	2	272	- 297
13	10	5	290	- 231
13	11	1	276	312
13	11	3	409	362
13	11	5	404	461
13	11	7	413	381
13	13	1	505	500
13	13	3	435	438
13	13	5	329	385
13	14	5	321	- 137
13	16	1	506	500
13	16	3	494	413
14	0	0	1084	1133
14	0	2	827	812
14	0	4	1607	1576
14	0	6	1104	1118
14	0	8	831	874
14	1	4	365	- 367
14	1	5	400	- 340
14	1	10	335	- 16
14	1	12	380	- 8
14	2	1	219	- 237
14	2	3	233	- 173
14	2	4	963	912
14	2	6	682	649
14	2	7	266	23
14	2	8	709	731
14	3	5	239	- 195

Table 3. Continued.

14	3	8	299	- 81	15	5	7	541	539	16	12	6	394	308	19	1	1	820	845
14	4	0	333	359	15	5	9	379	341	16	14	0	509	496	19	1	3	560	612
14	4	1	530	526	15	6	11	378	257	16	14	2	483	521	19	1	5	373	305
14	4	4	850	838	15	6	1	362	- 342	16	14	4	346	379	19	1	9	354	301
14	4	6	621	575	15	6	3	277	- 236	17	0	4	315	- 287	19	3	1	531	546
14	4	8	771	828	15	6	4	302	- 246	17	1	1	1111	1198	19	3	9	389	328
14	5	1	299	- 315	15	6	7	330	297	17	1	3	764	777	19	4	1	384	245
14	5	2	262	169	15	7	1	560	558	17	1	5	364	312	19	4	3	296	182
14	5	6	308	255	15	7	3	721	703	17	2	3	300	- 342	19	4	5	426	301
14	5	8	385	262	15	7	5	645	619	17	2	4	312	- 268	19	5	1	692	695
14	6	0	913	955	15	7	7	402	467	17	2	5	288	- 320	19	5	3	422	497
14	6	2	568	581	15	8	1	305	- 304	17	3	0	283	254	19	6	5	336	251
14	6	4	1170	1125	15	8	3	439	450	17	3	1	759	747	19	7	1	784	778
14	6	6	246	- 144	15	8	5	708	645	17	3	3	445	429	19	7	3	537	568
14	6	8	602	602	15	8	8	644	638	17	3	5	283	170	19	7	5	331	318
14	6	9	518	619	15	9	7	584	578	17	3	9	413	378	19	8	1	499	547
14	7	1	349	- 321	15	10	8	351	169	17	5	0	759	750	19	8	3	350	348
14	7	5	353	- 396	15	11	0	423	397	17	5	1	736	760	19	8	5	406	262
14	8	0	1195	1204	15	11	6	413	- 307	17	5	3	734	666	20	0	0	1053	1117
14	8	2	430	398	15	11	7	372	326	17	5	5	514	472	20	0	2	487	427
14	8	4	929	914	15	12	0	382	335	17	5	7	342	336	20	0	4	765	736
14	8	6	395	348	15	12	1	499	536	17	6	4	297	- 247	20	0	8	416	470
14	8	8	499	556	15	12	3	460	458	17	6	6	328	- 286	20	2	1	738	770
14	9	1	284	- 244	15	12	5	422	292	17	7	0	279	277	20	2	4	709	683
14	9	2	278	- 290	15	13	1	477	492	17	7	1	700	690	20	2	8	415	424
14	9	4	382	- 445	15	13	3	384	398	17	7	3	571	549	20	4	0	602	623
14	9	5	303	- 238	16	0	2	1406	1467	17	7	5	341	334	20	4	4	397	444
14	9	6	385	- 232	16	0	4	614	586	17	7	7	389	269	20	4	9	377	421
14	10	0	559	610	16	0	6	855	905	17	8	2	322	- 348	20	6	0	780	752
14	10	4	756	743	16	1	1	814	- 823	17	9	4	334	- 296	20	6	2	345	344
14	10	8	568	536	16	1	3	292	- 258	17	9	1	484	449	20	6	4	587	588
14	11	2	274	- 286	16	1	5	653	- 650	17	9	3	456	436	20	8	0	728	729
14	11	3	277	248	16	1	7	310	165	17	11	3	311	228	20	8	1	413	- 364
14	12	0	644	700	16	2	0	406	383	17	11	7	362	315	20	8	2	445	351
14	12	3	280	81	16	2	1	305	- 347	17	12	1	441	588	20	8	4	525	533
14	12	4	598	556	16	2	2	958	929	17	12	3	487	487	20	10	0	475	392
14	12	8	418	378	16	2	3	325	- 266	18	0	0	970	966	20	10	4	411	332
14	14	0	806	759	16	2	4	328	239	18	0	2	804	869	21	1	1	538	479
14	14	2	378	332	16	2	5	335	- 267	18	0	4	515	494	21	1	3	434	444
14	14	4	616	626	16	2	6	427	439	18	2	0	884	956	21	1	5	503	487
15	0	2	653	- 630	16	2	7	378	- 386	18	2	2	816	787	21	1	7	490	369
15	0	4	390	- 408	16	3	2	302	161	18	3	5	361	301	21	2	7	356	- 73
15	0	6	304	- 182	16	3	4	345	- 109	18	4	0	696	718	21	3	5	391	491
15	1	0	265	- 263	16	3	5	394	- 392	18	4	2	641	661	21	3	7	409	358
15	1	1	924	924	16	4	2	889	881	18	4	4	403	339	21	5	1	320	294
15	1	2	260	- 165	16	4	4	334	255	18	4	5	291	- 51	21	7	1	349	452
15	1	3	837	833	16	4	6	685	703	18	4	6	306	287	21	7	3	616	526
15	1	4	351	- 290	16	4	10	380	513	18	5	2	300	249	21	7	5	472	466
15	1	5	878	836	16	5	1	468	- 518	18	5	4	459	441	21	9	0	378	- 318
15	1	7	500	534	16	5	3	340	292	18	5	6	382	210	21	9	1	357	264
15	1	9	458	390	16	5	5	308	- 392	18	6	0	763	785	22	0	0	415	354
15	2	1	309	- 313	16	6	0	686	697	18	6	2	872	854	22	0	2	596	569
15	2	2	367	- 347	16	6	1	318	326	18	6	3	299	215	22	0	4	554	553
15	2	3	242	- 229	16	6	2	987	980	18	6	4	319	266	22	0	6	481	531
15	2	4	546	- 560	16	6	4	485	417	18	7	3	314	- 329	22	2	2	362	262
15	3	1	398	338	16	6	6	618	572	18	7	6	361	194	22	2	4	534	458
15	3	2	357	- 278	16	7	1	285	- 282	18	8	0	594	652	22	2	6	569	540
15	3	3	348	318	16	7	5	362	- 360	18	8	1	312	- 174	22	4	0	402	241
15	3	5	730	741	16	8	0	706	714	18	8	2	735	752	22	4	2	383	396
15	3	7	640	643	16	8	2	844	861	18	8	4	390	323	22	4	4	415	420
15	3	9	518	501	16	8	4	387	276	18	8	6	456	296	22	8	0	357	217
15	4	1	276	- 292	16	8	5	712	- 285	18	9	6	381	- 94	22	8	2	438	291
15	4	2	227	- 237	16	8	6	431	418	18	10	2	429	429	23	1	3	439	422
15	4	7	249	217	16	10	2	315	340	18	10	4	357	313	23	1	5	464	412
15	5	0	562	512	16	10	6	517	454	18	11	2	316	214	23	3	3	394	314
15	5	1	411	407	16	12	0	381	302	18	12	0	337	219	24	0	2	490	421
15	5	3	647	673	16	12	1	309	373	18	12	2	386	447					
15	5	5	722	706	16	12	2	372	389	19	0	8	337	- 361					

where C_T is the total number of counts and C_N the net count (peak minus background).

After six cycles the R-value converged at 12.8 %, and the corresponding Fourier map contained additional peaks with heights up to 4.8 e.Å^{-3}. The two largest were situated very close to the two-fold axis of symmetry and could only be interpreted in terms of statistically distributed atoms, which indeed is required by the space group symmetry with four molecules of crystal ethanol per unit cell. The distance between the two symmetry related peaks closest to the rotation axis (Fig. 1) were about 1.54 Å. This peak was therefore interpreted as corresponding to half a methylene carbon atom of one ethanol molecule and half a methyl carbon of another molecule. In the least squares refinement it was treated as a normal carbon atom. Since the final B-value was reasonable (4.89 Å^2), refinement of individual half-atoms at this position

Table 4. Bond distances and angles with estimated standard deviations.

Distance	(Å)	Distance	(Å)[a]
Pd—P_1	2.334(3)	C_2—H_2	1.07
Pd—P_2	2.333(3)	C_3—H_3	1.13
P_1—C_7	1.829(11)	C_4—H_4	0.88
P_2—C_8	1.849(12)	C_5—H_5	0.89
P_2—C_9	1.809(13)	C_6—H_6	0.92
P_1—C_{10}	1.800(12)	C_{12}—H_{12}	0.94
P_1—C_1	1.800(12)	C_{13}—H_{13}	0.72
P_2—C_{11}	1.800(10)	C_{14}—H_{14}	0.74
C_7—C_8	1.517(13)	C_{15}—H_{15}	0.91
C_1—C_2	1.348(14)	C_{16}—H_{16}	0.88
C_2—C_3	1.368(15)	C_7—$H_{7,1}$	0.79
C_3—C_4	1.378(15)	C_7—$H_{7,2}$	0.87
C_4—C_5	1.383(15)	C_8—$H_{8,1}$	0.85
C_5—C_6	1.414(16)	C_8—$H_{8,2}$	1.03
C_6—C_1	1.401(14)	C_9—$H_{9,1}$	0.77
C_{11}—C_{12}	1.384(14)	C_9—$H_{9,2}$	0.97
C_{12}—C_{13}	1.409(14)	C_9—$H_{9,3}$	1.05
C_{13}—C_{14}	1.390(15)	C_{10}—$H_{10,1}$	0.83
C_{14}—C_{15}	1.363(16)	C_{10}—$H_{10,2}$	0.93
C_{15}—C_{16}	1.375(14)	C_{10}—$H_{10,3}$	0.95
C_{16}—C_{11}	1.386(13)		
C_e—C_e'	1.534(18)		
C_e—O_e	1.335(17)		

Angle	(°)	Angle	(°)[b]
P_1—Pd—P_2	85.0(1)	C_1—C_2—H_2	131
Pd—P_1—C_1	110.6(4)	H_2—C_2—C_3	108
Pd—P_1—C_7	105.8(4)	C_2—C_3—H_3	132
Pd—P_1—C_{10}	122.4(4)	H_3—C_3—C_4	105
Pd—P_2—C_{11}	118.0(4)	C_3—C_4—H_4	126
Pd—P_2—C_8	106.4(4)	H_4—C_4—C_5	115
Pd—P_2—C_9	113.5(4)	C_4—C_5—H_5	120
P_1—C_7—C_8	107.8(7)	H_5—C_5—C_6	120
P_2—C_8—C_7	110.1(7)	C_5—C_6—H_6	121
P_1—C_1—C_2	119.7(1.0)	H_6—C_6—C_1	120
P_1—C_1—C_6	119.4(9)	C_{11}—C_{12}—H_{12}	120
C_1—C_2—C_3	119.8(1.1)	H_{12}—C_{12}—C_{13}	119
C_2—C_3—C_4	122.1(1.2)	C_{12}—C_{13}—H_{13}	113
C_3—C_4—C_5	118.9(1.2)	H_{13}—C_{13}—C_{14}	129
C_4—C_5—C_6	119.5(1.2)	C_{13}—H_{14}—C_{14}	113
C_5—C_6—C_1	118.8(1.2)	H_{14}—C_{14}—C_{15}	126
C_6—C_1—C_2	120.9(1.1)	C_{14}—C_{15}—H_{15}	128
P_2—C_{11}—C_{12}	119.3(8)	H_{15}—C_{15}—C_{16}	113
P_2—C_{11}—C_{13}	122.4(9)	C_{15}—C_{16}—H_{16}	119
C_{11}—C_{12}—C_{13}	120.5(1.1)	H_{16}—C_{16}—C_{11}	119
C_{12}—C_{13}—C_{14}	118.3(1.2)	P_1—C_7—$H_{7,1}$	112
C_{13}—C_{14}—C_{15}	121.9(1.2)	P_1—C_7—$H_{7,2}$	94
C_{14}—C_{15}—C_{16}	118.6(1.2)	$H_{7,1}$—C_7—$H_{7,2}$	126
C_{15}—C_{16}—C_{11}	122.4(1.2)	P_2—C_8—$H_{8,1}$	95
C_{16}—C_{11}—C_{12}	118.3(1.0)	P_2—C_8—$H_{8,2}$	100
C_e—C_e'—O_e	112.9(1.3)		

Angle	(°)	Angle	(°)
$H_{8,1}$—C_8—$H_{8,2}$	120	P_2—C_9—$H_{9,1}$	109
P_1—C_{10}—$H_{10,1}$	109	P_2—C_9—$H_{9,2}$	104
P_1—C_{10}—$H_{10,2}$	109	P_2—C_9—$H_{9,3}$	105
P_1—C_{19}—$H_{10,3}$	107	$H_{9,1}$—C_9—$H_{9,2}$	123
$H_{10,1}$—C_{10}—$H_{10,2}$	106	$H_{9,1}$—C_9—$H_{9,3}$	101
$H_{10,1}$—C_{10}—$H_{10,3}$	116	$H_{9,2}$—C_9—$H_{9,3}$	115
$H_{10,2}$—C_{10}—$H_{10,3}$	110		

[a] e.s.d.'s range from 0.1 to 0.2 Å.
[b] e.s.d.'s of about 4°.

was not attempted. The other peak was interpreted as a half oxygen atom and refined with multiplicity factor 0.5.

Methylene and phenyl hydrogen positions were calculated by assuming C–H bonds of length 1.03 Å. Three methyl hydrogen atoms could be localized in the difference Fourier map while positional parameters of the remaining three were calculated. Hydrogens of the statistically distributed ethanol molecules were not included in the calculations. Coordinates and isotropic thermal parameters were refined for all H-atoms. The final conventional R-value was 7.9 % and the weighted value $R_w = 6.0$ % for 1509 observed reflections.

Final positional and thermal parameters are given in Tables 1 and 2. A comparison between observed and calculated structure factors is presented in Table 3.

Fig. 1 shows a schematical drawing of the complex viewed along [001]. It may be described[1] in terms of a MESO-*anti-trans* form, where MESO indicates a centre of symmetry or a mirror plane, and *anti* means that pairs of equal ligands are situated at opposite sides of the plane through the phosphorus atoms (planarity is required by the center of symmetry).

Bond distances and angles may be found in Table 4. The two Pd–P distances (2.334 Å and 2.333 Å) are equal and somewhat shorter than the sum of covalent radii (2.42 Å). The bond angle P_1-Pd-P_2 is 85.0°. The two Pd–P–C angles as well as the two P–C–C angles of the five-membered ring are equal within probable limits of error, with values (105.8°; 106.4°) and (107.8°; 110.1°), respectively. The distances from the P_1-Pd-P_2 plane to C_7 and C_8 are +0.56 Å and −0.20 Å, respectively. The best least squares plane through four ring atoms is:

Atoms defining the plane	Distance from the plane
Pd	−0.013
P_1	0.032
P_2	0.050
C_8	−0.107

C_7 is at a distance of 0.65 Å from this plane. The geometry of the five-membered ring is thus somewhere between an envelope and the conformation with symmetry C_2.

Table 5. Least squares planes of the phenyl rings.

Atoms defining the plane	Distance (Å)	Atoms defining the plane	Distance (Å)
C_1	.005	C_{11}	−.001
C_2	−.007	C_{12}	.000
C_3	.003	C_{13}	.003
C_4	.004	C_{14}	−.004
C_5	−.007	C_{15}	.003
C_6	.002	C_{16}	.000

The six P–C distances are equal within error limits. Their mean value, 1.815 Å, is somewhat smaller than the microwave result (1.841±0.003 Å) reported for trimethylphosphine.[5] According to the large standard deviations in the present results, the difference is probably not significant.

As shown in Table 5, the phenyl groups are planar, the largest deviation being 0.007 Å.

The larger distance (4.09 Å) between the chlorine ions and palladium shows that there is no bonds between these atoms. On the other hand, several chlorine-hydrogen contacts are somewhat (but not significantly) shorter than van der Waals distance. Also the distance $Cl^-...O_e$ of 3.19 Å corresponds to van der Waals contact. The angles $P_1-Pd...Cl^-$ and $P_2-Pd...Cl^-$ are 70.2° and 79.6°, respectively.

The distance C_e-C_e' (1.534 Å) and the angle $C_e-C_e'-O_e$ (112.9°) of the ethanol molecule are normal. The C_e-O_e distance (1.335 Å) indicates, however, a slight disorder of C_e and C_e' with the methylene carbon situated about 0.08 Å further away from the oxygen and the methyl group slightly displaced in the opposite direction. The separation should be no more than 0.15–0.20 Å (in agreement with the thermal parameter value arrived at) and, as mentioned before, refinement under these assumptions was not attempted.

REFERENCES

1. Bercz, J. P., Horner, L. and Bercz, C. V. *Ann.* **703** (1967) 17.
2. Horner, L. *Private communication.*
3. Müller, E. *Dissertation*, Mainz 1969.
4. Dahl, T., Gram, F., Groth, P., Klewe, B. and Rømming, C. *To be published in Acta Chem. Scand.*
5. Lide, D. R. and Mann, D. E. *J. Chem. Phys.* **29** (1958) 914.

Received February 26, 1970.