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The Crystal Structure of a Colored Complex, $C_7H_7OHg \cdot C_7H_6O_2N$, Obtained in the Millon Reaction of *p*-Cresol

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The crystal structure of the complex of 2-mercuri-4-methylphenol and 2-nitroso-4-methylphenol obtained in the modified Millon reaction was determined using three-dimensional X-ray diffraction data. The complex crystallizes in triclinic system, space group $P\bar{1}$, with a unit cell of dimensions $a=10.26$ Å, $b=22.71$ Å, $c=5.61$ Å, $\alpha=97.2°$, $\beta=93.1°$, $\gamma=91.1°$, containing four complex molecules. Each of the two crystallographycally independent complex molecules both having essentially the same structure, forms a dimer with its nearest neighbor related by a center of symmetry through a pair of hydrogen bonds. The mercury atoms adopt a planar four-fold coordination with nitrogen, carbon and two oxygen atoms.

In the modified Millon reaction of *p*-cresol with Hopkins-Cole reagent, three kinds of pigment (pigment 1, 2 and 3) were produced depending upon the reaction condition. Pigment 1 is known as a complex of 2-mercuri-4-methylphenol (MMP) and 2-nitroso-4-methylphenol (NMP).[1,2] In order to determine the structure of the complex, two-dimensional X-ray analysis of pigment 1 was already carried out by Tamura, Iitaka and Kido[2] using the crystal grown from chloroform solution. However, this crystal contained chloroform as a solvent of crystallization and was so small and deteriorative against X-ray irradiation that it was not possible to determine the structure in three dimensions. Later, more stable and larger crystals were obtained from ethanol solutions. The present investigation was therefore undertaken to elucidate the detailed spacial structure of the complex molecule by X-ray diffraction using the new crystals.

Experimental

The crystals of pigment 1 were grown from ethanol solutions as massive aggregates of triclinic prisms elongated along the *c*-axis. They were dark red in color. The lattice constants and space group were determined from the *a*-, *b*- and *c*-axis equatorial precession photographs taken with Cu$K\alpha$ radiation. The density was measured by the floatation method using the mixed solutions of 1,1,2,2-tetrabromoethane and carbon tetrachloride. Unlike the crystals grown from chloroform solutions, the present crystal does not contain the solvent of crystallization.

Crystal Data. 2-Mercuri-4-methylphenol·2-nitroso-4-methylphenol complex $C_7H_7OHg \cdot C_7H_6O_2N$.

Mol wt: 444

Space group: triclinic $P\bar{1}$

Lattice constants:

$a=10.26\pm0.02$, $b=22.71\pm0.03$, $c=5.61\pm0.01$ Å,

$\alpha=97.2\pm0.2$, $\beta=93.1\pm0.2$, $\gamma=91.1\pm0.2°$

Volume of the unit cell: 1297 Å^3

Four structure units in the unit cell

Density measured: 2.25 g·cm^{-3}

Density calculated: 2.27 g·cm^{-3}

Linear absorption coefficient for Cu$K\alpha$ radiation: $\mu=237.6$ cm^{-1}

Three-dimensional intensity data were collected from multiple-film equi-inclination Weissenberg photographs taken with Cu$K\alpha$ radiation of the layers zero to fourth about the *c*-axis and zero to third about [101] axis. The intensities were measured visually with the aid of a calibrated intensity scale and they were corrected for Lorentz and polarization factors. The size of the specimens used for the intensity measurement was about 0.065×0.065 mm in cross section corresponding to the μR value of 1.53, but no absorption correction was applied. The structure factors obtained for the two axes were correlated and scaled to a common base. A total of 2418 independent structure factors was finally derived.

Determination of the Crystal Structure

The three-dimensional Patterson function yielded the positions of two mercury atoms involved in the asymmetric unit. A three-dimensional Fourier map was then calculated on the basis of the contribution of these two atoms which gave the *R* factor of 0.33. Although many spurious peaks were present on the Fourier map especially around the mercury atoms, the locations of the thirty six light atoms forming the two crystallographically independent complex molecules were determined by succesive use of Fourier and difference Fourier syntheses. The

1) Z. Tamura and Y. Kido, *Chem. Pharm. Bull.* (Tokyo), **16**, 1808 (1968).

2) Z. Tamura, Y. Iitaka and Y. Kido, *ibid.*, **17**, 1767 (1969).

refinement of the atomic positional and thermal parameters was first carried out by the block-matrix least-squares method, using the program HBLS.[3] Seven cycles of calculations including isotropic temperature factors for each atom reduced the R factor from 0.30 to 0.15. This factor was further reduced to 0.11 by three cycles of the least-squares calculations including individual anisotropic temperature factor parameters. The weighting system adopted in the calculations was:

$$\sqrt{w}=75/F_o, \text{ when } 75<F_o,$$

$$\sqrt{w}=1, \text{ when } 12<F_o\leq 75,$$

$$\sqrt{w}=0, \text{ when } F_o\leq 12.$$

The final atomic parameters and their standard deviations are listed in Table 1. In Table 2 the observed structure factors are listed. The atomic scattering factors used in the present structure determination were taken from *International Tables for X-ray Crystallography*.[4]

Figure 1 depicts the superposed three-dimensional electron density distribution function calculated on the basis of the atomic parameters listed in Table 1.

Discussion of the Structure

The present crystal contains two crystallographically independent complex molecules I and II. Each of these molecules forms a dimer with its nearest neighbor related by a center of symmetry through a pair of hydrogen bonds.

The bond lengths and angles of the complex molecules calculated from the coordinates given in Table 1 are shown in Fig. 2 along with their standard deviations. It is seen that they resemble each other considering their rather large standard deviations. Furthermore, the c-axis projection of the dimer molecule found in the crystal grown from chloroform solutions (Fig. 1 of the previous paper[2]) closely resembles those found in the present crystal, although the sturucture of the former was solved only in a two-dimensional projection and no detailed spacial arrangement of the atoms was given.

In the previous paper, two resonance forms of the complex were suggested as shown in Fig. 3 of the present paper, but it was not possible to decide which is more predominant. In Table 3, the length of the C–N and N–O bonds reported for various oximes, nitroso compounds and N-oxides are shown. The length of the C–N bonds found in the molecules I and II are both 1.25 Å indicating a strong double bond character. The N–O bond lengths in the molecules I and II differ considerably. In view of the rather large standard deviations, it can not be said that the difference is really significant, but the mean value of the two lengths, 1.27 Å, is nearly of the same order of magnitude as those found in nitroso compounds[12–16] or in N-oxides[17–20] and is clearly shorter than those found in oximes.[5–11]

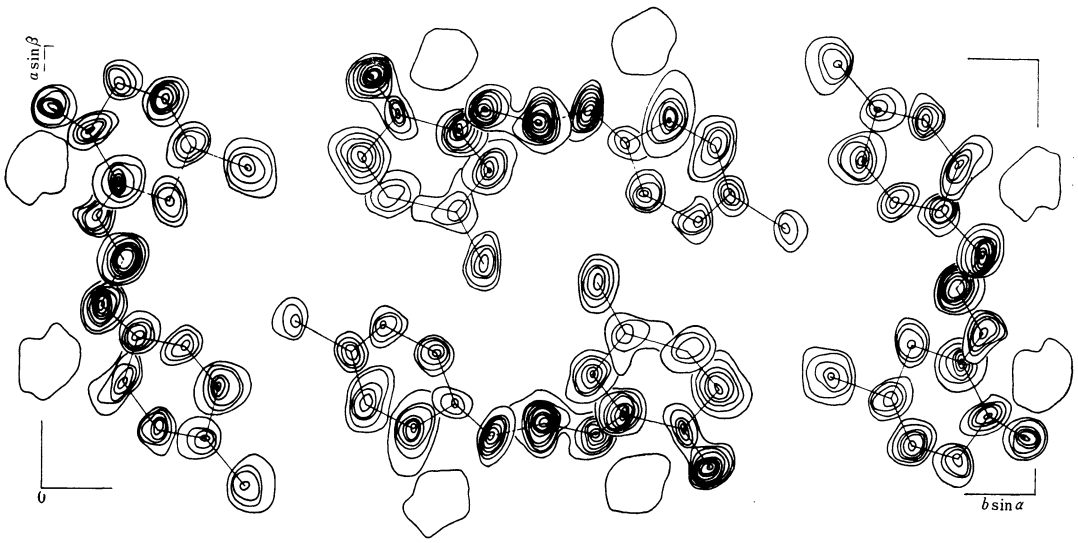

Fig. 1. Composite drawing of the electron density map viewed along the c-axis. Contours are drawn at intervals of 1 e. Å^{-3} starting at 2 e. Å^{-3}. Those for mercury atoms are not shown.

3) Y. Okaya and T. Ashida, HBLS IV, *The Universal Crystallographic Computing System* (I), p. 65, Japanese Crystallographic Association.

4) *International Tables for X-ray Crystallography*, Vol. III, Kynoch Press, Birmingham (1962).

TABLE 1. FINAL ATOMIC PARAMETERS AND THEIR STANDARD DEVIATIONS (e.s.d)

E.s.d.'s are given in parentheses denoting the least significant digits of the corresponding values. x, y, z are the fractional coordinates. The temperature factors are of the form

$$T=\exp\{-(\beta_{11}h^2+\beta_{22}k^2+\beta_{33}l^2+2\beta_{12}hk+2\beta_{13}hl+2\beta_{23}kl)\}$$

ATOM	x	y	z	β_{11}	β_{22}	β_{33}	β_{12}	β_{13}	β_{23}
Hg(1)	0.2914	0.0115	0.1128	0.0093	0.0014	0.0242	0.0007	-0.0006	-0.0013
	(2)	(1)	(3)	(2)	(0)	(8)	(1)	(3)	(1)
Hg(2)	0.0036	0.4983	0.0971	0.0090	0.0015	0.0242	0.0006	-0.0035	-0.0002
	(2)	(1)	(4)	(2)	(0)	(9)	(1)	(3)	(1)
O(1)	0.1243	-0.0155	-0.2448	-0.0014	0.0004	0.0582	-0.0010	-0.0011	0.0011
	(24)	(10)	(56)	(25)	(5)	(158)	(8)	(45)	(20)
O(2)	0.4631	-0.0876	-0.0428	0.0124	0.0023	0.0031	0.0011	-0.0073	-0.0036
	(30)	(15)	(51)	(43)	(6)	(120)	(14)	(51)	(22)
O(3)	0.4129	0.0528	0.0158	0.0140	0.0013	0.0291	0.0017	-0.0114	0.0010
	(30)	(13)	(62)	(47)	(6)	(152)	(14)	(364)	(24)
O(4)	-0.0920	0.5288	-0.2472	0.0114	0.0006	0.0411	0.0002	-0.0147	-0.0060
	(33)	(12)	(62)	(41)	(5)	(159)	(11)	(62)	(22)
O(5)	-0.1800	0.4927	-0.0405	0.0144	0.0012	-0.0002	-0.0015	-0.0077	-0.0040
	(33)	(12)	(49)	(45)	(6)	(113)	(13)	(351)	(19)
O(6)	0.1022	0.4508	0.5900	0.0030	0.0017	0.0361	0.0005	-0.0034	-0.0017
	(27)	(13)	(54)	(29)	(7)	(144)	(11)	(49)	(24)
N(1)	0.3566	-0.0611	-0.1095	0.0011	0.0008	0.0478	0.0006	0.0008	0.0006
	(36)	(13)	(74)	(51)	(6)	(190)	(11)	(58)	(26)
N(2)	-0.1492	0.4430	-0.1108	0.0125	0.0004	0.0101	-0.0000	0.0154	-0.0052
	(41)	(13)	(65)	(56)	(6)	(156)	(14)	(72)	(23)
C(1)	0.1726	-0.0528	-0.3851	0.0097	0.0016	-0.0091	0.0009	-0.0120	-0.0018
	(42)	(16)	(68)	(52)	(7)	(155)	(15)	(63)	(24)
C(2)	0.1141	-0.0723	-0.5886	0.0072	0.0031	0.0248	0.0035	-0.0027	0.0057
	(46)	(24)	(95)	(54)	(14)	(224)	(22)	(82)	(44)
C(3)	0.1535	-0.1226	-0.7334	0.0050	0.0025	0.0312	0.0002	-0.0019	-0.0007
	(42)	(22)	(92)	(46)	(12)	(222)	(18)	(74)	(38)
C(4)	0.2619	-0.1497	-0.6736	0.0116	0.0014	0.0075	0.0022	-0.0008	-0.0045
	(47)	(19)	(78)	(59)	(9)	(178)	(18)	(72)	(29)
C(5)	0.3510	-0.1341	-0.4629	0.0060	0.0017	0.0045	-0.0004	-0.0001	-0.0034
	(40)	(18)	(72)	(46)	(9)	(163)	(16)	(61)	(28)
C(6)	0.2960	-0.0829	-0.3017	0.0098	0.0012	0.0226	0.0001	-0.0164	-0.0050
	(45)	(18)	(83)	(59)	(9)	(214)	(18)	(86)	(34)
C(7)	0.3134	-0.2072	-0.8300	0.0029	-0.0002	0.0970	-0.0028	0.0166	-0.0038
	(41)	(16)	(110)	(45)	(6)	(333)	(14)	(97)	(34)
C(8)	0.3202	0.0940	0.5531	0.0061	0.0015	0.0359	0.0016	0.0034	-0.0055
	(45)	(20)	(93)	(49)	(9)	(227)	(17)	(79)	(35)
C(9)	0.2984	0.1382	0.7155	0.0097	0.0039	-0.0491	0.0006	-0.0002	-0.0018
	(65)	(25)	(72)	(107)	(15)	(172)	(30)	(77)	(31)
C(10)	0.2005	0.1822	0.6437	0.0117	0.0011	0.0395	0.0010	0.0017	-0.0056
	(53)	(20)	(99)	(65)	(9)	(251)	(19)	(95)	(36)
C(11)	0.1232	0.1666	0.4456	-0.0120	0.0003	0.0244	-0.0010	-0.0004	-0.0005
	(28)	(12)	(64)	(30)	(5)	(149)	(9)	(44)	(20)
C(12)	0.1503	0.1197	0.2832	0.0060	0.0015	0.0054	-0.0009	0.0043	-0.0057
	(42)	(18)	(72)	(51)	(9)	(165)	(16)	(65)	(28)
C(13)	0.2456	0.0840	0.3526	0.0019	0.0017	-0.0199	-0.0013	-0.0173	-0.0016
	(35)	(16)	(66)	(37)	(8)	(144)	(13)	(57)	(24)
C(14)	0.0238	0.2106	0.3620	0.0183	0.0024	0.0025	0.0028	-0.0061	-0.0052
	(59)	(23)	(82)	(83)	(12)	(190)	(25)	(88)	(35)
C(15)	-0.1745	0.3522	-0.3634	0.0085	-0.0001	0.0207	-0.0004	0.0039	-0.0030
	(41)	(14)	(74)	(49)	(6)	(175)	(13)	(68)	(23)
C(16)	-0.2257	0.3242	-0.5880	0.0066	0.0011	0.0479	-0.0001	0.0018	0.0025
	(47)	(19)	(104)	(53)	(9)	(261)	(17)	(90)	(37)
C(17)	-0.3095	0.3525	-0.7194	0.0050	0.0042	0.0035	-0.0019	-0.0043	-0.0058
	(44)	(26)	(79)	(50)	(17)	(180)	(22)	(66)	(41)
C(18)	-0.3470	0.4144	-0.6443	-0.0174	0.0027	0.0526	0.0031	-0.0089	0.0059
	(32)	(19)	(85)	(33)	(10)	(215)	(12)	(55)	(36)
C(19)	-0.2918	0.4446	-0.4475	0.0054	0.0015	0.0228	-0.0016	-0.0033	0.0007
	(42)	(19)	(85)	(46)	(9)	(201)	(16)	(71)	(32)
C(20)	-0.2022	0.4147	-0.3035	0.0137	0.0012	-0.0045	0.0000	-0.0078	0.0014
	(47)	(17)	(89)	(68)	(9)	(168)	(19)	(73)	(27)
C(21)	-0.4615	0.4421	-0.7823	0.0024	0.0045	-0.0051	0.0014	-0.0287	0.0187
	(41)	(22)	(88)	(47)	(13)	(208)	(19)	(79)	(47)
C(22)	0.1815	0.4056	0.5250	0.0105	0.0017	-0.0101	-0.0039	-0.0064	0.0021
	(43)	(18)	(67)	(54)	(9)	(149)	(18)	(62)	(26)
C(23)	0.2790	0.3904	0.6654	0.0055	0.0014	0.0284	-0.0012	-0.0100	-0.0064
	(42)	(19)	(88)	(47)	(9)	(215)	(16)	(76)	(35)
C(24)	0.3636	0.5441	0.5864	0.0134	0.0026	0.0567	-0.0024	0.0065	-0.0118
	(63)	(27)	(121)	(83)	(15)	(330)	(28)	(127)	(57)
C(25)	0.3339	0.5129	0.3750	0.0054	0.0016	0.0369	-0.0006	0.0090	0.0078
	(43)	(20)	(94)	(79)	(8)	(169)	(19)	(77)	(27)
C(26)	0.2270	0.5262	0.2247	0.0268	0.0008	-0.0092	0.0003	-0.0036	-0.0059
	(54)	(17)	(75)	(48)	(9)	(234)	(16)	(82)	(39)
C(27)	0.1535	0.3752	0.2952	0.0104	0.0017	0.0190	0.0001	0.0042	0.0055
	(50)	(20)	(88)	(62)	(10)	(208)	(20)	(84)	(36)
C(28)	0.4137	0.2563	0.2818	0.0072	0.0030	0.0656	-0.0022	-0.0058	0.0006
	(56)	(29)	(131)	(65)	(17)	(352)	(26)	(117)	(59)

TABLE 2. OBSERVED STRUCTURE FACTORS

H	K	L	F(OBS)
-10	2	0	40.25
-10	3	0	40.25
-10	5	0	56.44
-10	7	0	34.02
-10	8	0	19.44
-10	9	0	19.06
-10	10	0	45.98
-10	11	0	25.92
-10	14	0	28.28
-10	16	0	20.19
-10	17	0	26.04
-9	1	0	70.40
-9	2	0	35.26
-9	3	0	28.78
-9	4	0	70.52
-9	6	0	67.66
-9	8	0	49.96
-9	9	0	64.17
-9	11	0	63.17
-9	14	0	56.19
-9	16	0	48.84
-8	1	0	51.09
-8	4	0	58.56
-8	5	0	58.94
-8	6	0	34.26
-8	8	0	34.89
-8	9	0	45.23
-8	10	0	57.57
-8	12	0	28.91
-8	13	0	49.72
-8	15	0	59.06
-8	17	0	37.01
-8	18	0	30.53
-8	20	0	40.74
-7	1	0	24.92
-7	2	0	90.33
-7	3	0	64.29
-7	5	0	122.11
-7	7	0	93.57
-7	8	0	105.91
-7	10	0	116.13
-7	11	0	34.51
-7	12	0	57.07
-7	13	0	73.39
-7	15	0	67.53
-7	17	0	34.39
-7	18	0	27.29
-7	20	0	49.59
-7	21	0	23.18
-7	22	0	21.18
-6	1	0	59.06
-6	2	0	27.41
-6	4	0	99.80
-6	5	0	65.79
-6	6	0	94.95
-6	7	0	24.17
-6	8	0	35.01
-6	9	0	107.78
-6	10	0	45.11
-6	11	0	119.37
-6	12	0	27.29
-6	13	0	59.18
-6	14	0	75.01
-6	16	0	84.23
-6	18	0	49.34
-6	19	0	62.18
-6	21	0	65.17
-6	22	0	23.92
-6	23	0	26.91
-6	24	0	36.63
-5	1	0	171.57
-5	2	0	77.75
-5	3	0	40.00
-5	4	0	136.44
-5	5	0	54.95
-5	6	0	143.79
-5	7	0	53.70
-5	8	0	31.90
-5	9	0	146.65
-5	10	0	56.57
-5	11	0	156.62
-5	13	0	62.18
-5	14	0	90.46
-5	15	0	59.31
-5	16	0	60.43
-5	17	0	53.83
-5	20	0	59.56
-5	22	0	31.65
-5	25	0	27.79
-4	1	0	63.55
-4	2	0	41.99
-4	3	0	131.70
-4	4	0	21.93
-4	5	0	227.15
-4	6	0	42.49
-4	7	0	123.98
-4	8	0	122.23
-4	10	0	224.65
-4	12	0	147.78
-4	13	0	105.04
-4	14	0	48.72
-4	15	0	145.03
-4	17	0	72.27
-4	18	0	49.84
-4	20	0	81.11
-4	22	0	57.57
-4	23	0	36.38
-4	25	0	43.49
-3	1	0	13.96
-3	2	0	82.98
-3	3	0	109.15
-3	4	0	97.94
-3	5	0	214.06
-3	6	0	96.56
-3	7	0	86.85
-3	8	0	52.95
-3	9	0	48.97
-3	10	0	110.02
-3	11	0	118.62
-3	12	0	34.89
-3	13	0	17.19
-3	14	0	88.47
-3	15	0	65.17
-3	16	0	135.07
-3	18	0	59.43
-3	19	0	78.25
-3	20	0	37.38
-3	21	0	76.63
-3	23	0	39.00
-3	24	0	56.82
-3	26	0	44.11
-2	0	0	119.37
-2	1	0	243.34
-2	2	0	90.33
-2	3	0	93.33

H	K	L	F(OBS)
-2	4	0	282.59
-2	6	0	309.01
-2	7	0	112.64
-2	8	0	120.74
-2	9	0	215.06
-2	10	0	46.60
-2	11	0	189.77
-2	12	0	65.29
-2	13	0	102.05
-2	14	0	140.30
-2	16	0	147.40
-2	17	0	20.43
-2	18	0	43.24
-2	19	0	76.50
-2	21	0	95.57
-2	23	0	29.90
-2	24	0	34.14
-1	1	0	196.12
-1	2	0	41.12
-1	3	0	20.06
-1	4	0	114.76
-1	5	0	165.22
-1	6	0	50.21
-1	7	0	81.24
-1	8	0	94.07
-1	9	0	23.30
-1	10	0	158.99
-1	11	0	21.68
-1	12	0	102.79
-1	13	0	98.93
-1	14	0	24.17
-1	15	0	117.37
-1	17	0	93.33
-1	18	0	72.77
-1	20	0	99.68
-1	22	0	85.97
-1	23	0	30.28
-1	24	0	39.37
-1	25	0	68.41
0	2	0	220.17
0	3	0	182.54
0	4	0	71.89
0	5	0	238.98
0	6	0	14.33
0	7	0	152.63
0	8	0	78.75
0	9	0	73.02
0	10	0	185.65
0	11	0	43.49
0	12	0	161.61
0	13	0	65.79
0	15	0	142.79
0	16	0	29.28
0	17	0	55.45
0	18	0	25.79
0	19	0	32.40
0	20	0	58.56
0	21	0	55.45
0	22	0	39.87
0	26	0	41.99
1	0	0	152.51
1	1	0	194.25
1	4	0	144.91
1	5	0	98.43
1	6	0	140.55
1	8	0	72.39
1	9	0	137.56
1	10	0	51.71
1	11	0	183.66
1	12	0	38.50
1	13	0	102.79
1	14	0	150.77
1	16	0	154.75
1	18	0	60.18
1	19	0	87.59
1	21	0	91.46
1	23	0	48.22
1	24	0	54.45
1	26	0	49.59
2	0	0	103.54
2	1	0	146.90
2	2	0	76.63
2	3	0	79.49
2	4	0	42.74
2	5	0	61.55
2	6	0	178.55
2	8	0	47.22
2	9	0	84.98
2	10	0	78.75
2	11	0	104.54
2	12	0	20.56
2	14	0	64.42
2	15	0	72.14
2	16	0	65.79
2	17	0	63.80
2	20	0	64.04
2	22	0	53.45
2	23	0	23.80
3	0	0	191.76
3	2	0	104.17
3	3	0	144.79
3	4	0	40.12
3	5	0	212.82
3	7	0	136.31
3	8	0	92.45
3	9	0	18.94
3	10	0	174.56
3	12	0	117.87
3	13	0	112.02
3	14	0	50.46
3	15	0	150.77
3	17	0	98.68
3	18	0	39.87
3	19	0	36.26
3	20	0	75.51
3	22	0	53.08
3	23	0	20.31
4	0	0	120.86
4	1	0	40.00
4	2	0	66.91
4	3	0	30.53
4	4	0	47.85
4	5	0	96.07
4	6	0	69.65
4	7	0	49.22
4	9	0	72.14
4	10	0	78.50
4	11	0	86.72
4	12	0	34.76
4	13	0	25.54
4	14	0	87.72
4	16	0	108.53
4	18	0	49.96
4	19	0	57.69
4	21	0	61.93

H	K	L	F(OBS)
4	23	0	35.26
4	24	0	32.52
5	0	0	19.56
5	1	0	170.45
5	2	0	70.27
5	3	0	71.40
5	4	0	133.45
5	6	0	178.55
5	7	0	47.35
5	8	0	65.04
5	9	0	147.53
5	11	0	157.49
5	13	0	63.30
5	14	0	64.92
5	16	0	78.50
5	17	0	35.39
5	18	0	35.26
5	20	0	51.58
6	0	0	104.66
6	1	0	79.12
6	2	0	45.11
6	3	0	22.80
6	4	0	54.33
6	5	0	89.84
6	6	0	41.74
6	7	0	60.31
6	8	0	61.80
6	10	0	100.93
6	11	0	57.57
6	12	0	27.66
6	13	0	19.94
6	14	0	67.16
6	16	0	70.65
6	17	0	28.66
6	19	0	66.66
6	21	0	38.63
7	0	0	127.34
7	2	0	94.20
7	3	0	62.18
7	5	0	119.12
7	6	0	26.42
7	7	0	75.88
7	8	0	38.50
7	10	0	65.91
7	11	0	28.41
7	12	0	49.72
7	15	0	35.01
7	16	0	28.16
8	0	0	27.29
8	1	0	69.78
8	3	0	33.77
8	4	0	58.94
8	6	0	82.24
8	8	0	49.59
8	9	0	57.57
8	11	0	76.38
8	13	0	34.76
8	14	0	39.50
8	16	0	52.46
8	18	0	23.67
9	0	0	28.78
9	1	0	60.93
9	4	0	49.96
9	5	0	28.91
9	6	0	45.60
9	9	0	20.19
9	10	0	34.51
9	11	0	34.02
9	15	0	24.42
10	0	0	60.43
10	1	0	40.25
10	2	0	28.41
10	4	0	59.81
10	6	0	39.25
10	7	0	33.52
10	9	0	52.83
10	11	0	43.24
10	12	0	24.05
10	15	0	24.42
10	17	0	15.82
10	18	0	17.32
-9	-16	1	21.06
-9	-14	1	32.65
-9	-11	1	42.99
-9	-9	1	56.57
-9	-7	1	25.42
-9	-6	1	67.41
-9	-4	1	84.23
-9	-2	1	50.71
-9	-1	1	50.59
-9	1	1	87.72
-9	3	1	56.69
-9	4	1	80.37
-9	6	1	76.50
-9	8	1	46.72
-9	9	1	53.70
-9	11	1	58.81
-9	14	1	34.64
-9	16	1	44.73
-8	-19	1	28.53
-8	-16	1	33.52
-8	-14	1	49.84
-8	-11	1	36.01
-8	-9	1	50.59
-8	-6	1	42.86
-8	-5	1	42.49
-8	-4	1	68.78
-8	-3	1	34.26
-8	0	1	67.91
-8	1	1	53.70
-8	2	1	63.67
-8	5	1	82.36
-8	6	1	37.13
-8	7	1	83.73
-8	9	1	38.00
-8	10	1	60.43
-8	12	1	71.40
-8	14	1	45.73
-8	15	1	57.69
-8	17	1	63.55
-8	20	1	43.61
-7	-20	1	30.40
-7	-18	1	23.80
-7	-17	1	34.76
-7	-15	1	67.16
-7	-13	1	62.30
-7	-12	1	43.73
-7	-10	1	91.95
-7	-8	1	78.87
-7	-7	1	61.93
-7	-6	1	39.87
-7	-5	1	122.23
-7	-3	1	99.31
-7	-2	1	62.30
-7	0	1	116.00

H	K	L	F(OBS)
-7	1	1	38.00
-7	2	1	112.51
-7	3	1	54.45
-7	4	1	31.77
-7	5	1	108.15
-7	6	1	34.64
-7	7	1	105.79
-7	8	1	35.88
-7	9	1	44.61
-7	10	1	86.85
-7	11	1	59.31
-7	12	1	46.48
-7	13	1	38.13
-7	16	1	53.08
-7	21	1	41.49
-6	-20	1	40.62
-6	-18	1	35.39
-6	-15	1	50.69
-6	-14	1	35.51
-6	-13	1	34.89
-6	-11	1	23.67
-6	-10	1	56.44
-6	-9	1	59.56
-6	-8	1	38.00
-6	-7	1	21.43
-6	-6	1	55.45
-6	-5	1	45.85
-6	-4	1	78.12
-6	-3	1	28.16
-6	-2	1	44.11
-6	-1	1	95.94
-6	0	1	33.89
-6	1	1	152.01
-6	2	1	27.91
-6	3	1	97.94
-6	4	1	99.43
-6	6	1	162.98
-6	8	1	99.56
-6	9	1	96.32
-6	11	1	131.45
-6	13	1	69.78
-6	14	1	59.81
-6	16	1	89.59
-6	18	1	69.78
-6	19	1	62.67
-6	21	1	56.57
-6	23	1	38.63
-5	-19	1	61.80
-5	-17	1	36.01
-5	-16	1	56.32
-5	-14	1	90.33
-5	-12	1	50.96
-5	-11	1	101.05
-5	-9	1	132.08
-5	-8	1	34.51
-5	-7	1	88.59
-5	-6	1	124.48
-5	-4	1	136.06
-5	-2	1	57.44
-5	-1	1	95.69
-5	0	1	41.99
-5	1	1	166.22
-5	2	1	46.10
-5	3	1	70.77
-5	4	1	59.81
-5	5	1	48.10
-5	6	1	92.58
-5	7	1	85.85
-5	10	1	69.03
-5	11	1	58.19
-5	12	1	115.13
-5	15	1	70.03
-5	16	1	38.00
-5	17	1	81.11
-5	19	1	37.50
-5	20	1	63.17
-5	22	1	57.07
-5	24	1	33.52
-4	-24	1	34.39
-4	-21	1	37.38
-4	-20	1	34.89
-4	-19	1	40.49
-4	-18	1	19.19
-4	-17	1	24.30
-4	-16	1	28.16
-4	-15	1	54.95
-4	-14	1	50.34
-4	-13	1	55.95
-4	-12	1	36.26
-4	-11	1	21.56
-4	-10	1	111.64
-4	-9	1	50.46
-4	-8	1	33.89
-4	-7	1	72.02
-4	-6	1	38.00
-4	-5	1	152.26
-4	-4	1	38.88
-4	-3	1	129.46
-4	-2	1	54.20
-4	-1	1	69.90
-4	0	1	214.31
-4	1	1	21.68
-4	2	1	233.62
-4	3	1	125.97
-4	4	1	73.89
-4	5	1	210.20
-4	6	1	31.77
-4	7	1	158.74
-4	8	1	92.70
-4	9	1	103.54
-4	10	1	152.51
-4	12	1	156.25
-4	13	1	51.34
-4	14	1	52.46
-4	15	1	102.92
-4	16	1	32.89
-4	17	1	113.88
-4	18	1	26.17
-4	19	1	40.25
-4	20	1	51.46
-4	22	1	51.96
-4	24	1	29.16
-3	-25	1	60.06
-3	-23	1	55.95
-3	-22	1	42.49
-3	-20	1	81.86
-3	-18	1	77.50
-3	-17	1	75.76
-3	-15	1	122.11
-3	-14	1	25.29
-3	-13	1	87.22
-3	-12	1	63.67
-3	-11	1	31.15
-3	-10	1	136.81
-3	-9	1	62.05

H	K	L	F(OBS)
-3	-8	1	105.79
-3	-7	1	56.82
-3	-5	1	171.32
-3	-4	1	90.83
-3	-3	1	134.94
-3	-2	1	81.49
-3	-1	1	12.96
-3	0	1	94.20
-3	1	1	158.74
-3	2	1	66.91
-3	3	1	69.78
-3	4	1	94.32
-3	5	1	66.16
-3	6	1	148.90
-3	8	1	77.87
-3	9	1	101.05
-3	11	1	128.84
-3	13	1	100.30
-3	14	1	93.82
-3	16	1	122.98
-3	18	1	97.56
-3	19	1	55.82
-3	20	1	48.72
-3	21	1	93.45
-3	23	1	63.67
-3	24	1	38.75
-2	-25	1	34.39
-2	-24	1	26.04
-2	-21	1	37.26
-2	-19	1	68.16
-2	-17	1	23.92
-2	-16	1	77.87
-2	-15	1	29.28
-2	-14	1	96.19
-2	-13	1	35.26
-2	-12	1	35.76
-2	-11	1	108.40
-2	-9	1	137.31
-2	-8	1	39.37
-2	-7	1	127.71
-2	-6	1	201.35
-2	-5	1	45.11
-2	-4	1	352.49
-2	-3	1	13.46
-2	-2	1	117.12
-2	-1	1	171.95
-2	0	1	97.81
-2	1	1	262.03
-2	2	1	16.45
-2	3	1	145.28
-2	4	1	134.44
-2	5	1	54.70
-2	6	1	221.66
-2	7	1	25.29
-2	8	1	188.89
-2	9	1	113.76
-2	11	1	165.72
-2	12	1	45.98
-2	13	1	78.12
-2	14	1	49.22
-2	15	1	26.91
-2	16	1	91.08
-2	17	1	53.70
-2	18	1	47.10
-2	20	1	33.77
-2	21	1	35.64
-2	22	1	73.89
-2	23	1	31.40
-2	24	1	32.65
-1	-26	1	34.76
-1	-25	1	50.21
-1	-24	1	46.10
-1	-22	1	32.77
-1	-21	1	58.81
-1	-19	1	78.12
-1	-17	1	58.31
-1	-16	1	86.97
-1	-15	1	26.17
-1	-14	1	116.63
-1	-12	1	60.18
-1	-11	1	78.00
-1	-10	1	60.18
-1	-9	1	109.27
-1	-8	1	90.46
-1	-7	1	75.88
-1	-6	1	21.43
-1	-5	1	80.62
-1	-4	1	105.66
-1	-3	1	97.44
-1	-2	1	68.53
-1	-1	1	35.01
-1	0	1	148.90
-1	1	1	118.37
-1	2	1	119.87
-1	3	1	42.11
-1	4	1	69.03
-1	5	1	168.09
-1	6	1	58.81
-1	7	1	220.92
-1	8	1	80.37
-1	9	1	113.26
-1	10	1	191.51
-1	11	1	68.41
-1	12	1	172.70
-1	13	1	36.26
-1	14	1	77.00
-1	15	1	125.60
-1	16	1	31.65
-1	17	1	131.20
-1	18	1	42.11
-1	19	1	66.66
-1	20	1	80.87
-1	22	1	83.48
-1	24	1	33.14
-1	25	1	42.99
0	-25	1	43.73
0	-23	1	33.27
0	-22	1	29.53
0	-20	1	90.58
0	-18	1	79.49
0	-17	1	66.79
0	-16	1	38.13
0	-15	1	126.59
0	-14	1	27.79
0	-13	1	130.46
0	-12	1	94.45
0	-11	1	55.45
0	-10	1	198.49
0	-8	1	187.27
0	-7	1	75.38
1	-20	1	80.87
0	-6	1	82.98
0	-5	1	198.49
0	-4	1	89.09
0	-3	1	161.36

H	K	L	F(OBS)
0	-2	1	84.73
0	-1	1	70.90
0	0	1	32.89
0	1	1	53.33
0	2	1	186.40
0	3	1	29.28
0	4	1	38.75
0	5	1	115.75
0	6	1	81.36
0	7	1	151.64
0	9	1	37.50
0	10	1	109.27
0	11	1	98.06
0	12	1	81.11
0	13	1	75.38
0	14	1	31.77
0	16	1	84.85
0	18	1	65.54
0	19	1	35.64
0	21	1	72.69
0	23	1	61.43
0	24	1	28.91
1	-25	1	38.13
1	-23	1	44.73
1	-22	1	48.22
1	-19	1	37.01
1	-18	1	54.82
1	-17	1	32.65
1	-16	1	35.39
1	-15	1	95.32
1	-14	1	79.87
1	-13	1	30.26
1	-12	1	27.41
1	-11	1	66.29
1	-10	1	83.73
1	-9	1	138.18
1	-8	1	77.63
1	-7	1	75.13
1	-6	1	88.22
1	-5	1	22.18
1	-4	1	157.49
1	-2	1	81.74
1	-1	1	206.96
1	0	1	40.00
1	1	1	199.11
1	3	1	116.38
1	4	1	149.77
1	5	1	18.81
1	6	1	226.90
1	8	1	166.22
1	9	1	166.96
1	10	1	63.05
1	11	1	213.94
1	13	1	124.85
1	14	1	76.13
1	15	1	35.64
1	16	1	131.33
1	18	1	86.97
1	19	1	55.57
1	21	1	60.79
1	23	1	42.36
2	-26	1	33.89
2	-24	1	46.10
2	-22	1	29.78
2	-21	1	62.67
2	-19	1	114.51
2	-18	1	56.69
2	-17	1	79.62
2	-16	1	133.70
2	-14	1	189.64
2	-13	1	31.15
2	-12	1	94.95
2	-11	1	121.61
2	-9	1	206.34
2	-8	1	20.93
2	-7	1	102.67
2	-6	1	122.73
2	-5	1	68.65
2	-4	1	175.31
2	-3	1	34.76
2	-2	1	85.48
2	-1	1	70.15
2	0	1	43.61
2	1	1	165.59
2	2	1	49.22
2	3	1	55.32
2	4	1	9.84
2	5	1	90.46
2	6	1	133.32
2	7	1	152.51
2	8	1	47.22
2	9	1	36.51
2	10	1	117.62
2	11	1	32.77
2	12	1	131.95
2	13	1	33.89
2	14	1	52.83
2	15	1	83.48
2	16	1	20.93
2	17	1	102.55
2	19	1	69.65
2	20	1	73.02
2	22	1	72.89
2	24	1	35.26
2	25	1	40.37
3	-26	1	32.15
3	-24	1	38.63
3	-21	1	35.64
3	-20	1	50.96
3	-19	1	43.98
3	-18	1	61.30
3	-17	1	48.97
3	-16	1	41.12
3	-15	1	119.24
3	-14	1	58.31
3	-13	1	105.66
3	-12	1	28.53
3	-11	1	27.29
3	-10	1	140.30
3	-9	1	86.10
3	-8	1	152.63
3	-7	1	51.96
3	-6	1	72.52
3	-5	1	264.90
3	-3	1	251.69
3	-2	1	121.61
3	-1	1	95.32
3	0	1	204.72
3	1	1	32.27
3	2	1	250.70
3	3	1	102.30
3	4	1	106.28
3	5	1	215.43
3	7	1	225.40
3	9	1	79.62

H	K	L	F(OBS)
3	10	1	116.13
3	12	1	129.09
3	14	1	60.43
3	15	1	72.27
3	17	1	96.44
4	-25	1	43.11
4	-23	1	45.48
4	-22	1	33.77
4	-20	1	61.93
4	-18	1	67.16
4	-17	1	61.30
4	-15	1	109.40
4	-13	1	102.42
4	-12	1	48.22
4	-10	1	116.38
4	-9	1	79.12
4	-8	1	129.96
4	-7	1	28.91
4	-6	1	59.81
4	-5	1	125.97
4	-4	1	160.24
4	-3	1	153.51
4	-2	1	37.63
4	-1	1	50.21
4	0	1	103.42
4	1	1	160.61
4	2	1	70.90
4	3	1	97.94
4	4	1	98.93
4	6	1	144.29
4	7	1	28.28
4	8	1	41.49
4	9	1	52.83
4	11	1	111.64
4	13	1	111.64
4	14	1	62.92
4	16	1	119.74
4	18	1	85.35
4	19	1	53.20
4	21	1	65.66
4	23	1	43.11
5	-24	1	27.54
5	-21	1	33.77
5	-19	1	66.79
5	-17	1	56.82
5	-16	1	43.61
5	-14	1	116.38
5	-12	1	105.41
5	-11	1	102.30
5	-9	1	157.37
5	-7	1	87.10
5	-6	1	131.83
5	-5	1	49.34
5	-4	1	214.93
5	-3	1	55.20
5	-2	1	135.44
5	-1	1	117.25
5	1	1	223.41
5	2	1	39.37
5	3	1	134.07
5	4	1	79.00
5	6	1	117.50
5	7	1	22.05
5	8	1	68.16
5	9	1	33.14
5	11	1	70.27
5	12	1	44.23
5	13	1	45.23
5	16	1	46.85
5	17	1	46.60
6	-24	1	34.26
6	-21	1	44.73
6	-19	1	48.84
6	-17	1	35.88
6	-16	1	30.01
6	-14	1	66.54
6	-12	1	42.24
6	-11	1	41.37
6	-10	1	57.32
6	-9	1	91.58
6	-8	1	58.31
6	-5	1	74.64
6	-4	1	38.50
6	-3	1	100.93
6	-1	1	43.61
6	0	1	100.18
6	1	1	38.00
6	2	1	125.35
6	4	1	55.82
6	5	1	95.57
6	7	1	113.26
6	9	1	50.46
6	10	1	89.09
6	12	1	112.89
6	14	1	46.85
6	15	1	71.40
6	17	1	78.37
6	19	1	34.14
6	20	1	39.25
6	22	1	26.66
7	-20	1	43.24
7	-18	1	58.31
7	-15	1	76.01
7	-13	1	80.49
7	-12	1	75.88
7	-11	1	35.51
7	-10	1	110.89
7	-8	1	111.27
7	-7	1	48.59
7	-6	1	45.23
7	-5	1	92.70
7	-3	1	94.45
7	0	1	84.10
7	1	1	34.39
7	2	1	84.85
7	5	1	61.93
7	6	1	62.67
7	7	1	63.55
7	8	1	45.48
7	11	1	46.72
7	12	1	38.25
7	13	1	46.60
7	16	1	50.84
8	-15	1	34.26
8	-14	1	42.86
8	-11	1	50.96
8	-9	1	95.07
8	-7	1	56.32
8	-6	1	53.70
8	-4	1	87.34
8	-3	1	26.29
8	-2	1	58.56
8	-1	1	45.35
8	1	1	98.18
8	3	1	64.79

H	K	L	F(OBS)
8	4	1	59.43
8	6	1	96.81
8	8	1	76.50
8	9	1	46.85
8	11	1	70.65
8	13	1	51.58
8	16	1	51.34
8	18	1	39.87
9	-16	1	41.87
9	-14	1	60.43
9	-12	1	41.74
9	-11	1	42.49
9	-8	1	66.41
9	-7	1	35.88
9	-6	1	43.98
9	-5	1	54.08
9	-4	1	38.25
9	-3	1	38.25
9	1	1	54.08
9	2	1	54.08
9	4	1	53.95
9	7	1	46.10
9	10	1	36.13
9	12	1	54.08
9	15	1	36.01
9	17	1	39.00
10	-5	1	51.71
10	-3	1	52.21
10	0	1	45.23
10	2	1	51.96
10	5	1	43.86
-10	3	2	57.69
-10	6	2	50.46
-9	-15	2	40.87
-9	-13	2	38.13
-9	-12	2	39.12
-9	-10	2	65.91
-9	-7	2	47.60
-9	-6	2	33.64
-9	-4	2	71.52
-9	-2	2	62.92
-9	-1	2	41.24
-9	1	2	75.26
-9	3	2	65.04
-9	4	2	37.50
-9	6	2	45.85
-9	7	2	37.38
-8	-10	2	33.64
-8	-9	2	33.64
-8	-8	2	40.99
-8	-5	2	56.82
-8	-3	2	85.97
-8	-1	2	51.09
-8	0	2	68.53
-8	2	2	99.18
-8	4	2	51.83
-8	5	2	58.31
-8	7	2	83.36
-8	9	2	45.98
-8	10	2	45.98
-8	12	2	73.89
-8	14	2	50.21
-8	15	2	42.11
-8	17	2	69.65
-8	19	2	37.50
-7	-18	2	44.11
-7	-15	2	52.83
-7	-13	2	67.28
-7	-12	2	33.52
-7	-11	2	40.62
-7	-10	2	77.00
-7	-8	2	84.35
-7	-6	2	38.00
-7	-5	2	71.89
-7	-3	2	56.19
-7	-1	2	36.51
-7	0	2	47.22
-7	1	2	36.63
-7	2	2	71.27
-7	3	2	47.85
-7	4	2	24.17
-7	5	2	42.49
-7	6	2	55.57
-7	7	2	43.73
-7	8	2	76.75
-7	11	2	64.79
-7	12	2	45.98
-7	13	2	59.31
-7	16	2	56.32
-7	18	2	46.23
-7	21	2	47.35
-6	-19	2	32.02
-6	-18	2	32.89
-6	-14	2	62.42
-6	-13	2	40.37
-6	-11	2	44.86
-6	-9	2	90.83
-6	-7	2	81.86
-6	-6	2	44.73
-6	-5	2	43.98
-6	-4	2	117.87
-6	-2	2	115.50
-6	-1	2	73.26
-6	0	2	32.77
-6	1	2	141.17
-6	3	2	112.76
-6	4	2	76.38
-6	5	2	55.07
-6	6	2	119.37
-6	8	2	115.38
-6	9	2	55.07
-6	10	2	35.64
-6	11	2	99.43
-6	13	2	105.54
-6	15	2	37.50
-6	16	2	52.46
-6	18	2	49.72
-6	22	2	37.01
-5	-22	2	37.38
-5	-21	2	42.74
-5	-19	2	58.69
-5	-17	2	47.35
-5	-16	2	47.10
-5	-14	2	88.09
-5	-13	2	32.40
-5	-12	2	66.79
-5	-11	2	56.69
-5	-9	2	109.02
-5	-8	2	52.58
-5	-7	2	72.27
-5	-6	2	32.15
-5	-4	2	74.01
-5	-3	2	101.18
-5	-2	2	73.89
-5	-1	2	34.89

H	K	L	F(OBS)
-5	0	2	55.20
-5	1	2	73.64
-5	2	2	110.89
-5	4	2	46.10
-5	5	2	86.35
-5	7	2	122.11
-5	9	2	92.08
-5	10	2	93.33
-5	12	2	118.25
-5	14	2	90.09
-5	15	2	66.16
-5	16	2	40.00
-5	17	2	109.77
-5	19	2	83.73
-5	20	2	40.37
-5	21	2	28.41
-5	22	2	68.65
-4	-22	2	38.00
-4	-20	2	40.74
-4	-19	2	48.47
-4	-18	2	55.07
-4	-15	2	70.52
-4	-13	2	87.22
-4	-11	2	76.63
-4	-10	2	133.07
-4	-9	2	28.28
-4	-8	2	208.95
-4	-7	2	21.06
-4	-6	2	56.32
-4	-5	2	116.63
-4	-4	2	61.18
-4	-3	2	196.49
-4	-1	2	104.04
-4	0	2	138.68
-4	1	2	25.17
-4	2	2	193.63
-4	3	2	26.42
-4	4	2	148.27
-4	5	2	122.73
-4	7	2	164.85
-4	8	2	46.60
-4	9	2	104.41
-4	10	2	56.94
-4	12	2	97.69
-4	13	2	40.74
-4	14	2	43.24
-4	15	2	30.40
-4	16	2	37.50
-4	17	2	44.73
-4	18	2	68.03
-4	21	2	41.12
-4	23	2	53.58
-3	-25	2	34.26
-3	-23	2	43.24
-3	-21	2	32.89
-3	-20	2	62.30
-3	-18	2	63.80
-3	-15	2	53.70
-3	-14	2	40.87
-3	-13	2	56.94
-3	-12	2	46.10
-3	-9	2	36.13
-3	-8	2	77.75
-3	-7	2	54.57
-3	-5	2	37.26
-3	-4	2	102.17
-3	-3	2	75.63
-3	-2	2	89.59
-3	-1	2	51.58
-3	0	2	29.90
-3	1	2	147.40
-3	2	2	30.28
-3	3	2	166.22
-3	4	2	82.49
-3	5	2	78.37
-3	6	2	175.06
-3	7	2	69.03
-3	8	2	175.06
-3	9	2	51.21
-3	10	2	76.38
-3	11	2	138.56
-3	12	2	30.78
-3	13	2	140.17
-3	14	2	50.84
-3	15	2	60.31
-3	16	2	93.70
-3	18	2	97.31
-3	20	2	43.98
-3	21	2	73.02
-3	23	2	67.78
-3	25	2	23.42
-2	-24	2	59.68
-2	-22	2	40.99
-2	-21	2	45.60
-2	-19	2	84.35
-2	-17	2	75.26
-2	-16	2	63.30
-2	-15	2	22.68
-2	-14	2	125.22
-2	-12	2	115.25
-2	-11	2	76.75
-2	-10	2	61.93
-2	-9	2	158.74
-2	-8	2	55.57
-2	-7	2	125.97
-2	-6	2	70.90
-2	-5	2	36.38
-2	-4	2	128.46
-2	-3	2	26.66
-2	-2	2	140.92
-2	-1	2	63.55
-2	0	2	22.93
-2	1	2	145.41
-2	2	2	70.40
-2	3	2	162.98
-2	4	2	41.49
-2	5	2	42.86
-2	6	2	125.47
-2	7	2	106.91
-2	8	2	85.23
-2	9	2	66.04
-2	10	2	35.51
-2	11	2	48.34
-2	12	2	94.70
-2	13	2	35.02
-2	14	2	60.80
-2	15	2	36.88
-2	17	2	78.75
-2	19	2	67.41
-2	20	2	44.86
-2	22	2	75.13
-2	24	2	63.42
-1	-26	2	31.77
-1	-24	2	55.3
-1	-22	2	23.[illegible]

TABLE 2. (Continued)

-1-21	2	24.17
-1-20	2	24.30
-1-19	2	57.19
-1-18	2	51.58
-1-17	2	28.53
-1-15	2	36.88
-1-14	2	61.68
-1-13	2	71.02
-1-12	2	32.15
-1-11	2	37.01
-1-10	2	77.75
-1 -9	2	43.49
-1 -8	2	118.25
-1 -7	2	18.32
-1 -6	2	49.96
-1 -5	2	119.37
-1 -4	2	126.97
-1 -3	2	158.24
-1 -1	2	69.90
-1 0	2	149.77
-1 1	2	28.78
-1 2	2	208.70
-1 3	2	17.94
-1 4	2	173.32
-1 5	2	162.23
-1 6	2	53.83
-1 7	2	202.35
-1 9	2	114.88
-1 10	2	101.05
-1 12	2	152.63
-1 14	2	92.70
-1 15	2	69.90
-1 17	2	91.83
-1 19	2	58.31
-1 20	2	42.24
-1 22	2	60.43
-1 24	2	40.74
0-25	2	44.48
0-23	2	62.30
0-22	2	31.52
0-21	2	37.26
0-20	2	80.12
0-18	2	125.22
0-17	2	22.43
0-16	2	56.94
0-15	2	91.95
0-13	2	132.82
0-12	2	28.53
0-11	2	61.30
0-10	2	107.53
0 -9	2	30.78
0 -8	2	140.67
0 -7	2	21.31
0 -6	2	63.42
0 -5	2	93.20
0 -4	2	27.04
0 -3	2	146.03
0 -2	2	33.52
0 -1	2	48.97
0 0	2	25.54
0 1	2	89.09
0 2	2	145.91
0 3	2	166.71
0 4	2	40.87
0 5	2	27.16
0 6	2	139.55
0 7	2	55.57
0 8	2	144.04
0 9	2	39.12
0 10	2	28.03
0 11	2	94.20
0 12	2	30.90
0 13	2	123.60
0 15	2	72.27
0 16	2	92.70
0 18	2	108.65
0 19	2	29.16
0 20	2	43.98
0 21	2	76.26
0 23	2	80.49
0 25	2	39.87
1-25	2	34.51
1-24	2	21.43
1-23	2	23.55
1-22	2	29.28
1-21	2	33.64
1-20	2	35.39
1-19	2	74.51
1-18	2	39.62
1-17	2	56.44
1-16	2	39.75
1-14	2	95.07
1-13	2	61.80
1-12	2	89.21
1-11	2	48.59
1-10	2	31.03
1 -9	2	191.88
1 -8	2	30.65
1 -7	2	161.11
1 -6	2	96.81
1 -5	2	74.88
1 -4	2	208.46
1 -1	2	116.00
1 0	2	116.87
1 1	2	203.22
1 2	2	22.18
1 3	2	190.51
1 4	2	41.74
1 5	2	60.93
1 6	2	170.95
1 7	2	38.38
1 8	2	164.60
1 9	2	46.85
1 10	2	72.14
1 11	2	108.90
1 12	2	23.42
1 13	2	120.99
1 14	2	17.82
1 15	2	60.18
1 16	2	61.18
1 17	2	48.22
1 18	2	80.49
1 19	2	52.21
1 21	2	26.29
1 22	2	34.02
1 23	2	20.31
2-24	2	41.87
2-22	2	51.46
2-21	2	40.87
2-19	2	78.75
2-17	2	64.79
2-16	2	58.81
2-14	2	106.78
2-13	2	48.10
2-12	2	97.31

2-11	2	39.87
2-10	2	47.85
2 -9	2	151.89
2 -8	2	138.93
2 -7	2	132.95
2 -5	2	117.87
2 -4	2	167.34
2 -3	2	195.12
2 -2	2	98.56
2 -1	2	141.42
2 0	2	108.78
2 2	2	130.71
2 3	2	38.50
2 4	2	26.66
2 5	2	87.47
2 7	2	126.97
2 9	2	115.75
2 10	2	84.73
2 11	2	44.36
2 12	2	159.11
2 14	2	127.47
2 15	2	89.09
2 17	2	115.63
2 19	2	86.72
2 20	2	36.01
2 21	2	34.39
3-23	2	42.99
3-21	2	32.89
3-20	2	40.99
3-18	2	85.48
3-16	2	72.14
3-15	2	85.85
3-13	2	153.01
3-11	2	96.94
3-10	2	166.84
3 -9	2	65.17
3 -8	2	232.01
3 -7	2	84.73
3 -6	2	155.63
3 -5	2	168.33
3 -4	2	14.45
3 -3	2	259.79
3 -2	2	26.91
3 -1	2	162.60
3 0	2	136.06
3 1	2	22.43
3 2	2	162.85
3 3	2	23.80
3 4	2	79.99
3 5	2	72.64
3 7	2	98.81
3 8	2	46.23
3 9	2	68.16
3 10	2	31.77
3 11	2	23.30
3 12	2	87.34
3 13	2	75.01
3 14	2	25.67
3 16	2	37.50
3 17	2	45.98
3 18	2	58.69
3 20	2	34.89
3 21	2	40.49
4-23	2	40.87
4-20	2	40.37
4-18	2	58.31
4-16	2	33.14
4-15	2	51.34
4-14	2	50.09
4-13	2	92.33
4-12	2	63.30
4-10	2	27.91
4 -9	2	122.11
4 -8	2	71.52
4 -7	2	123.98
4 -5	2	53.70
4 -4	2	133.20
4 -3	2	66.04
4 -2	2	166.34
4 0	2	57.57
4 1	2	164.72
4 2	2	32.52
4 3	2	143.41
4 4	2	19.69
4 5	2	53.58
4 6	2	127.34
4 8	2	159.86
4 9	2	40.12
4 10	2	63.30
4 11	2	125.60
4 13	2	136.94
4 14	2	37.13
4 15	2	59.31
4 16	2	59.31
4 18	2	57.32
4 20	2	33.14
4 21	2	37.88
5-24	2	38.00
5-22	2	40.62
5-21	2	30.53
5-19	2	76.88
5-17	2	79.00
5-16	2	78.87
5-15	2	33.39
5-14	2	118.74
5-12	2	118.49
5-11	2	69.15
5-10	2	30.15
5 -9	2	145.66
5 -8	2	40.62
5 -7	2	145.91
5 -6	2	38.88
5 -5	2	42.74
5 -4	2	153.26
5 -3	2	36.01
5 -2	2	120.74
5 -1	2	35.88
5 1	2	106.78
5 2	2	84.85
5 3	2	88.84
5 4	2	38.00
5 6	2	60.80
5 7	2	78.12
5 8	2	63.92
5 9	2	55.32
5 10	2	25.29
5 12	2	94.45
5 14	2	79.62
5 15	2	37.38
5 17	2	56.82
5 19	2	51.83
5 20	2	30.53
5 21	2	52.21
6-19	2	38.25
6-18	2	45.60

6-17	2	33.02
6-15	2	58.31
6-14	2	23.80
6-13	2	91.58
6-11	2	40.00
6-10	2	68.28
6 -9	2	31.65
6 -8	2	132.33
6 -7	2	43.49
6 -6	2	88.34
6 -5	2	89.84
6 -3	2	167.46
6 -1	2	89.59
6 0	2	95.69
6 2	2	154.38
6 4	2	107.90
6 5	2	81.24
6 7	2	133.82
6 9	2	93.82
6 10	2	58.81
6 12	2	91.95
6 14	2	58.44
6 17	2	47.60
6 19	2	41.74
7-20	2	42.24
7-18	2	63.67
7-16	2	39.37
7-15	2	46.48
7-13	2	82.36
7-11	2	47.60
7-10	2	58.06
7 -8	2	90.58
7 -7	2	40.12
7 -6	2	56.44
7 -5	2	51.09
7 -4	2	39.37
7 -3	2	87.47
7 -2	2	75.63
7 1	2	84.10
7 2	2	62.42
7 3	2	72.52
7 6	2	58.69
7 7	2	45.73
7 8	2	102.67
7 10	2	37.50
7 11	2	64.42
7 13	2	76.38
7 15	2	41.24
7 16	2	45.35
7 18	2	51.83
7 19	2	24.80
8-19	2	42.49
8-17	2	34.39
8-14	2	70.52
8-12	2	77.00
8-10	2	40.99
8 -9	2	78.87
8 -7	2	89.09
8 -5	2	33.64
8 -4	2	91.95
8 -2	2	95.32
8 -1	2	45.73
8 0	2	45.85
8 1	2	91.71
8 3	2	87.97
8 6	2	52.95
8 8	2	58.56
8 11	2	35.26
8 12	2	34.14
8 13	2	40.25
9-14	2	34.39
9-12	2	36.88
9 -9	2	39.25
9 -8	2	45.85
9 -7	2	46.23
9 -4	2	46.97
9 -3	2	52.46
9 -1	2	52.46
9 0	2	37.01
9 2	2	63.80
9 4	2	44.61
9 7	2	60.80
9 9	2	40.99
9 10	2	32.40
9 12	2	41.99
9 14	2	36.51
10 -3	2	63.80
10 -1	2	41.87
10 0	2	34.02
10 2	2	47.47
10 3	2	33.27
10 4	2	32.77
-9-12	3	41.74
-9 -9	3	38.38
-9 -7	3	39.12
-9 -4	3	39.62
-9 -2	3	45.85
-8 -8	3	51.34
-8 -6	3	51.21
-8 -5	3	39.62
-8 -3	3	75.38
-8 -1	3	68.03
-8 0	3	50.71
-8 2	3	71.27
-8 4	3	60.56
-8 5	3	38.38
-8 7	3	60.68
-8 9	3	71.02
-8 12	3	56.82
-7-18	3	40.62
-7-16	3	30.90
-7-15	3	38.75
-7-13	3	60.68
-7-12	3	22.93
-7-11	3	32.40
-7 -8	3	54.95
-7 -7	3	49.72
-7 -6	3	49.22
-7 -4	3	21.68
-7 -3	3	48.10
-7 -2	3	64.29
-7 0	3	21.43
-7 1	3	50.96
-7 3	3	73.02
-7 5	3	52.58
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5 7	4	57.19
5 9	4	73.64
5 11	4	57.81
5 12	4	39.50
5 14	4	75.88
5 16	4	56.19
6-14	4	41.49
6-13	4	67.28
6-11	4	93.20
6 -9	4	54.57
6 -8	4	89.46
6 -6	4	114.26
6 -4	4	63.30
6 -3	4	43.24
6 -2	4	30.53
6 -1	4	80.87
6 0	4	37.50
6 1	4	37.50
6 2	4	37.50
6 3	4	48.34
6 4	4	48.10
6 5	4	47.85
6 8	4	35.64
6 9	4	28.41
6 10	4	58.56
6 12	4	25.17
6 13	4	50.09
6 15	4	50.84
7-12	4	56.57
7-10	4	59.18
7 -7	4	86.72
7 -5	4	75.88
7 -2	4	76.50
7 0	4	76.26
7 2	4	29.65
7 3	4	71.89
7 5	4	83.36
7 8	4	56.19
7 9	4	54.08
7 11	4	64.29
7 14	4	26.29
-5 -9	5	41.62
-5 -8	5	29.78
-5 -7	5	24.42
-5 -6	5	52.08
-5 -4	5	46.10
-5 -3	5	34.89
-5 -1	5	57.81
-5 1	5	49.09
-5 4	5	48.34
-5 6	5	64.54
-4 0	5	81.86
-4 2	5	40.74
-4 3	5	52.58
-4 5	5	73.39
-4 7	5	50.71
-4 8	5	52.33
-4 10	5	68.53
-4 12	5	41.37
-4 13	5	39.00
-4 15	5	50.71

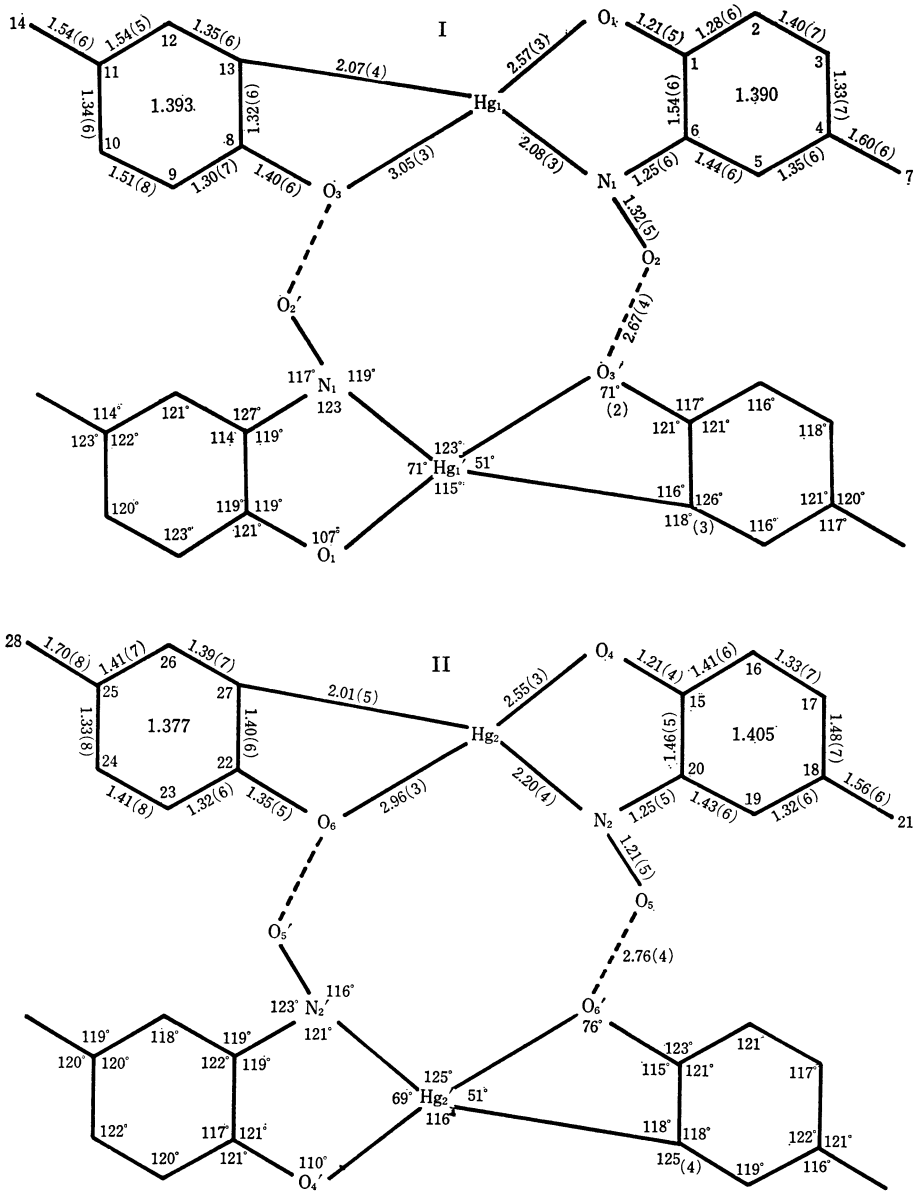

Fig. 2. Bond lengths (Å) and angles (°) found in molecule I and molecule II. E.s.d.'s are shown in parentheses denoting the least significant digits of the corresponding values.

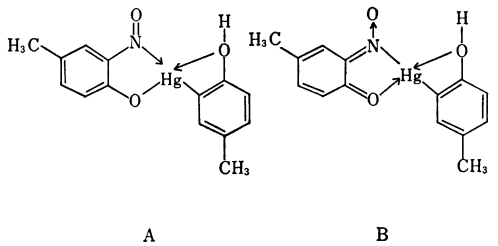

Fig. 3. The structures of the present complex molecule proposed in the previous paper.[2)]

Although the location of hydrogen atoms are not shown in the present structure determination, a comparison of the N–O bond lengths in NMP-group which have double bond character and the C–O bond lengths in MMP-group which correspond to the carbon to phenolic oxygen bond lengths (C–OH), indicates that the latter oxygen atoms exist in the form of hydroxyl groups and act as hydrogen donors in the hydrogen bond systems.

From the discussions on the C–N and N–O bond lengths it may be concluded that the structure of the

TABLE 3. COMPARISON OF THE N-O AND C-N BOND LENGTHS AND C-N-O ANGLES OF THE OXIMES, NITROSO AND *N*-OXIDE COMPOUNDS

Compounds	N-O	C-N	C-N-O	Ref.
Oximes				
Bis(salicylaldoximato)copper(II)	1.45 Å	1.25 Å	115°	5
Nickel dimethylglyoxime	1.38 1.37	1.20 1.25	121 121	6
Nickel glyoxime	1.350 1.343	1.277 1.303	121.8 121.1	7
5-Methoxy-2-quinone-2-oxime	1.36	1.22	117	8
N-Methyl-*p*-chlorobenzaldoxime	1.284	1.309	125.2	9
β-5-*n*-Propoxy-*o*-quinone-2-oxime	1.353	1.319	116.8	10
α-5-(2′-Chloroethoxy)-*o*-quinone-2-oxime	1.365	1.306	112.5	11
Nitroso compounds				
(*trans*)	1.25	1.57	125	12
Dinitrosomethane	1.30	1.48	119	13
(*cis*)	1.32	1.45	120	
1-(4-Chlorobenzyl)-1-nitroso-2-(4,5-dihydro-2-imidazolyl) hydrazine monohydrate	1.25			14
p-Methoxyindophenol *N*-oxide	1.266	1.357 1.450	122.7 116.1	15
Nitrosobenzene (dimer)	1.35			16
N-Oxides				
anti-2,5-Dimethyl-4-chloro-*N*-methylbenzene aldoxime	1.298	1.29	124	17
anti-4-Chloro-*N*-methylbenzenealdoxime	1.28	1.30	125	17
Bis-(pyridine *N*-oxide)Copper(II) nitrate	1.362 1.361	1.352 1.337 1.325 1.369	116.8 119.1 120.1 116.9	18
Di-*μ*-(pyridine oxide)-bis(dichloro-copper(II))	1.346	1.341	118.2	19
Pyridine oxide hydrochloride	1.37	1.31 1.36	116.1 117.1	20
Present Compound (mean of the two complexes)	1.27	1.25	120	

present complex is represented as shown in Fig. 3, B.

The mercury atoms adopt planar four-fold coordination if one takes the intramolecular short distances of O(3)–Hg(1), 3.05 Å and O(6)–Hg(2), 2.96 Å as the fourth coordination bonds. Table 4 shows the deviations of atoms from the plane formed by each coordination group.

Figures 4 and 5 show the crystal structure projected along the *c*- and *a*-axis, respectively. Each molecule takes a nearly planar conformation and is inclined to the *c*-axis at an angle of 56° in I and 54° in II. As seen in Fig. 5, the neighboring dimers in the *c* direction overlap strongly and the dimers of each kind, I-I or II-II, are piled up along the *c*-axis forming a columnar unit. The columns are

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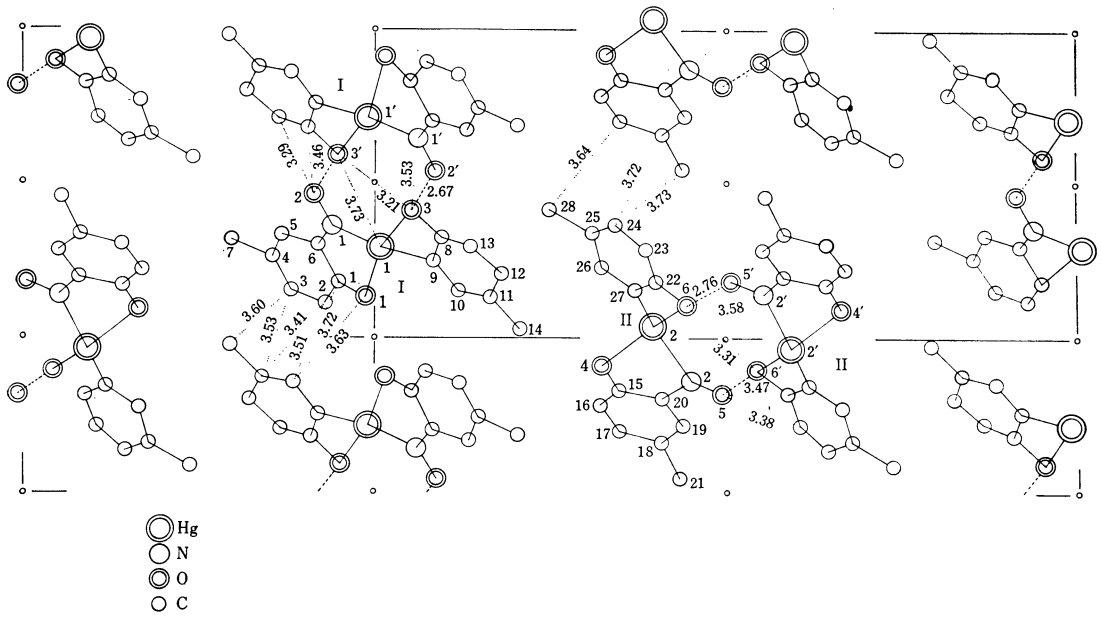

Fig. 4. The *c*-axis projection of the crystal structuere. Hydrogen bonds are shown by broken lines and intermolecular short distances less than 3.75Å are shown by dotted lines.

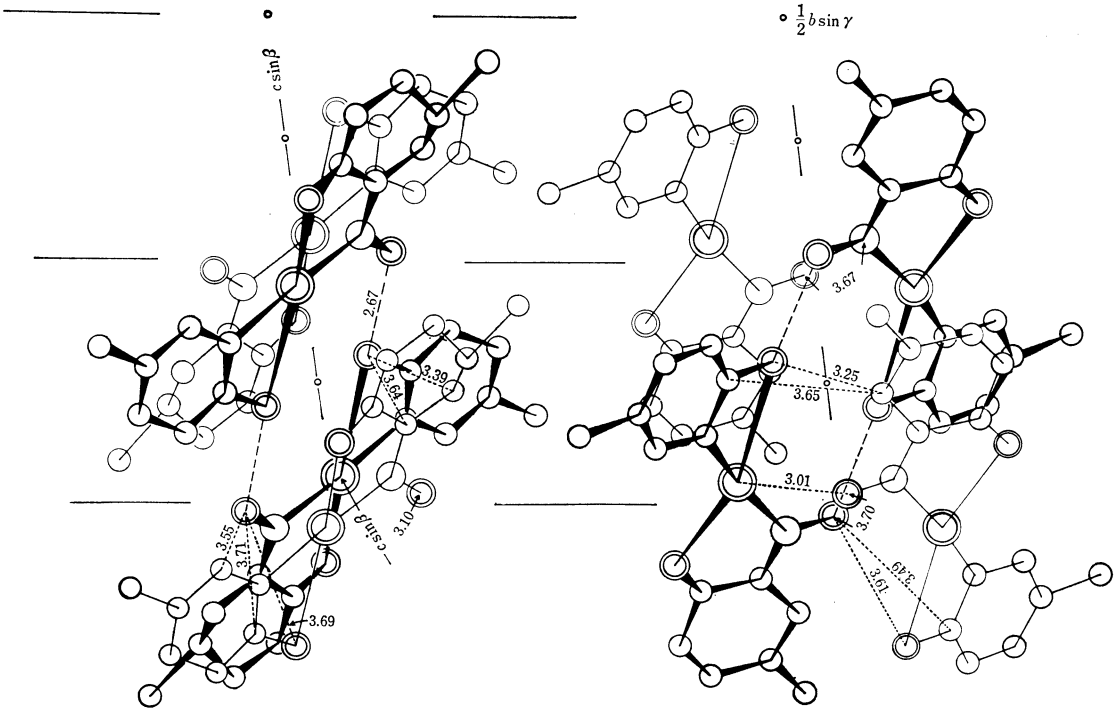

Fig. 5. The *a*-axis projection of the crystal structure showing the stacking of the complex molecules.

Table 4. The deviations of the atoms from the least-squares planes

Molecule I		Molecule II	
Atom	Distances	Atom	Distances
Coordinated group around Hg			
Hg(1)	−0.09 Å	Hg(2)	−0.13 Å
O(1)	0.16	O(4)	0.26
O(3)	0.17	O(6)	0.27
N(1)	−0.09	N(2)	−0.14
C(13)	−0.16	C(17)	−0.26
2-Nitroso-4-methylphenol group			
C(1)	0.01	C(15)	0.05
C(2)	−0.02	C(16)	−0.03
C(3)	0.02	C(17)	−0.01
C(4)	−0.02	C(18)	0.04
C(5)	0.02	C(19)	−0.02
C(6)	0.01	C(20)	−0.03
distance to the plane			
C(7)	0.01	C(21)	0.29
O(1)	0.20	O(4)	−0.04
N(1)	−0.07	N(2)	−0.08
C(2)	−0.09	O(5)	0.01
2-Mercuri-4-Methylphenol group			
C(8)	−0.02	C(22)	0.01
C(9)	0.05	C(23)	0.03
C(10)	−0.07	C(24)	−0.03
C(11)	0.05	C(25)	0.00
C(12)	−0.02	C(26)	0.03
C(13)	0.00	C(27)	−0.04
distance to the plane			
Hg(1)	−0.10	Hg(2)	−0.10
O(3)	0.07	O(6)	0.01
C(14)	−0.09	C(28)	0.17

packed together in the ***a*** direction mainly through the van der Waals interaction between the phenolic groups forming the layers of the dimer molecules. The two kinds of layers each consisting of the dimers I-I or II-II are packed together alternately along the *b*-axis. The packing of the layers in the ***b***-direction is rather loose and no close contact shorter than 3.75 Å is found between the layers.

In Figures 4 and 5 are shown intermolecular short distances less than 3.75 Å. It is to be noted that the dimers I-I and II-II are situated approximately in such positions that they are related by a rotation about the axis parallel to ***c*** followed by a mirror operation with the mirror plane parallel to ***c***. Comparison of Fig. 4 with Fig. 1 of the previous paper[2] indicates that the structure of the present crystal resembles, in some respect, that found in the crystal grown from chloroform solutions. The latter crystal contains chloroform molecules as the solvent of crystallization filling up the spaces between the II-II dimers. These spaces are filled by the dimers I-I in the present crystal.

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