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The Crystal Structure of an Adduct of *o*-Nitrobenzeneselenenyl Thiocyanate with Thiourea

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o-Nitrobenzeneselenenyl thiocyanate has been found to react with thiourea to give an adduct. The crystal structure of the adduct has been determined by X-ray methods, and refined by full-matrix least squares analysis. The crystals are monoclinic, space group $C2/c$ (No. 15) with $a = 34.360(18)$ Å, $b = 5.095(2)$ Å, $c = 14.589(6)$ Å, $\beta = 98.44(5)°$, and eight formula units per unit cell.

The structure shows that the thiourea group has replaced the thiocyanate group, and a salt is formed. The cation has the benzene ring, the nitro group, the selenium atom, and the sulphur atom of the thiourea group in nearly the same plane. The angle between this plane and a plane through the thiourea group is 92.8°. A loose five-membered ring is formed by the selenium atom, one oxygen atom and the nitrogen atom of the nitro group, and two carbon atoms of the benzene ring. The distance between the selenium atom and the oxygen atom in this system is 2.505(8) Å. The selenium-sulphur bond length in the cation is 2.189(3) Å.

o-Nitrobenzeneselenenyl compounds, like *o*-nitrobenzenesulphenyl compounds, are usually found to be more stable than the unsubstituted compounds. This feature of the *ortho*-substituted compounds is probably, judging from the crystal structures of *o*-nitrobenzenesulphenic acid methyl ester [1] and of bis(*o*-nitrophenyl) disulphide,[2] due to the formation of a five-membered ring system, comprised of the sulphur atom, one oxygen atom and the nitrogen atom of the nitro group, and two carbon atoms of the benzene ring, and involving a close S···O approach.

The present work on the adduct of *o*-nitrobenzeneselenenyl thiocyanate with thiourea, has been carried out mainly to study the influence of a short Se···O distance on the Se–S bond length.

Preparative and crystallographic data on the adduct have been reported earlier.[3]

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CRYSTAL DATA

The adduct of *o*-nitrobenzeneselenenyl thiocyanate with thiourea forms long, yellow, monoclinic prisms elongated along the *b* axis, with $a = 34.360(18)$ Å, $b = 5.095(2)$ Å, $c = 14.589(6)$ Å and $\beta = 98.44(5)°$. The unit cell dimensions were determined from 67 reflections on zero-layer Weissenberg photographs around the *b* and *c* axes, and evaluated by means of a least squares program.

There are eight formula units per unit cell; density, calc. 1.76, found 1.77 g/cm^3. The space group, from systematic absences and subsequent structure analysis, is $C2/c$ (No. 15).

Intensities were estimated visually from integrated Weissenberg photographs around the *b* and *c* axes, taken with Cu$K\alpha$ radiation using the multi-film technique.

Three different crystals were used. Crystal No. 1, used for the collection of the $h0l - h2l$ data, had the following dimensions, from an arbitrarily chosen origin to the crystal faces: to (100) and ($\bar{1}00$), 0.021 mm; to (010) and (0$\bar{1}$0), 0.066 mm; to (101) and ($\bar{1}0\bar{1}$), 0.031 mm. The corresponding dimensions of crystal No. 2, used for the $h3l$ data, were 0.012 mm, 0.047 mm, and 0.024 mm, respectively, and the dimensions of crystal No. 3, used for the $hk0$ and $hk1$ data, were: to (100) and ($\bar{1}00$), 0.031 mm; to (001) and (00$\bar{1}$), 0.053 mm; to ($\bar{1}12$) and ($\bar{1}\bar{1}2$), 0.077 mm; to ($\bar{1}10$) and (012), 0.126 mm; to (11$\bar{2}$), 0.125 mm. The linear absorption coefficient, $\mu = 74.5$ cm^{-1}.

The structure was solved from the $h0l$ and $hk0$ data. The three-dimensional refinement was based on 255 out of 307 $h0l$, 441 out of 613 $h1l$, 410 out of 563 $h2l$, 296 out of 480 $h3l$, 82 out of 125 $hk0$, 134 out of 199 $hk1$ reflections, in all 1618 observed $h0l - h3l$ and $hk0 - hk1$ reflections out of 2287 accessible with Cu$K\alpha$ radiation.

THE STRUCTURE ANALYSIS

The approximate position of the selenium atom in the *b*-axis projection was found from the Patterson map. A structure factor calculation based on the selenium position gave signs to 81 of 255 $h0l$ reflections. The positions of the sulphur atoms were found from the subsequent Fourier map. A new structure factor calculation, based on the positions of the selenium and sulphur atoms, gave signs to 165 reflections. The carbon, nitrogen, and oxygen atoms were placed partly on the basis of subsequent Fourier maps, and partly from the known dimensions of the benzene and thiourea groups. Least squares refinement, with isotropic temperature factors, brought the reliability index, *R*, down to 0.091.

The approximate *y* coordinates of the atoms in the *c*-axis projection were found from Patterson and Fourier maps, and from considerations of the packing of the molecules in the unit cell.

The three-dimensional refinement was carried out on an IBM 360/50H computer using a full-matrix least squares program minimizing the function, $r = \sum W(|F_o| - {}^{\iota}K|F_c|)^2$, where K is the scale factor, and $W = 1/[(Ka_1)^2 + (a_2F_o)^2/4W_o]$. The weight, W_o, is based on the estimated reliability of the film readings. The constants a_1 and a_2 were both given the value one. Unob-

served reflections with calculated structure factors, $|F_c|$, greater than the threshold value, F_t, were included in the refinement with F_o equal to F_t.

Refinement with isotropic temperature factors brought the reliability index, R, to 0.13. The intensities were then corrected for absorption by the method of Coppens *et al.*[4] A sub-division of 4, 12, and 8 Gaussian points along the a, b, and c axes, respectively, was used for crystals Nos. 1 and 2, and 4, 14,

Table 1. Atomic coordinates in fractions of monoclinic cell edges. Standard deviations from least squares are given in parentheses.

	x	y	z
Se	0.136790(29)	0.57130(23)	0.00220(7)
S_1	0.10049(8)	0.7719(6)	0.09184(18)
S_2	−0.08373(10)	0.0121(8)	0.16643(26)
O_1	0.22542(21)	0.0147(15)	−0.0477(5)
O_2	0.18415(21)	0.3247(17)	−0.0809(5)
N_1	0.03952(27)	0.6065(20)	0.1606(7)
N_2	0.05215(24)	0.3559(16)	0.0352(6)
N_3	0.20161(24)	0.1684(19)	−0.0258(6)
N_4	−0.01562(30)	0.1674(30)	0.1137(10)
C_1	0.16627(25)	0.3433(17)	0.0937(6)
C_2	0.19464(26)	0.1757(19)	0.0704(6)
C_3	0.21614(27)	0.0022(19)	0.1301(7)
C_4	0.20907(29)	−0.0050(20)	0.2224(7)
C_5	0.18126(29)	0.1733(20)	0.2481(6)
C_6	0.15992(28)	0.3470(20)	0.1869(6)
C_7	0.06002(28)	0.5486(23)	0.0935(8)
C_8	−0.0439(4)	0.1013(25)	0.1393(7)

Table 2. Anisotropic thermal parameters (Å²) in the form $\exp[-2\pi^2(h^2a^{-2}U_{11}+\cdots\cdots+2hka^{-1}b^{-1}U_{12}+\cdots\cdots)]$. The values have been multiplied by 10^3. Standard deviations are given in parentheses.

	U_{11}	U_{22}	U_{33}	U_{12}	U_{23}	U_{13}
Se	58.4(6)	56.8(8)	50.5(5)	−5.4(6)	5.7(6)	6.1(4)
S_1	69.4(17)	49.3(19)	82.8(19)	3.8(16)	−9.7(17)	10.6(15)
S_2	90.6(23)	169(4)	111.9(28)	14.0(26)	−53.8(29)	22.8(21)
O_1	80(5)	93(7)	61(4)	−5(5)	17(4)	18(4)
O_2	78(5)	100(7)	51(4)	−6(5)	−6(5)	11(4)
N_1	84(7)	115(9)	104(8)	−1(7)	−30(7)	40(6)
N_2	82(6)	31(6)	81(6)	−2(5)	−11(5)	3(5)
N_3	47(5)	64(8)	56(5)	11(5)	22(5)	13(4)
N_4	67(7)	216(15)	196(14)	23(9)	−102(11)	2(9)
C_1	47(5)	27(6)	48(5)	−14(5)	−3(5)	5(4)
C_2	48(5)	48(8)	44(5)	−16(5)	−8(5)	15(4)
C_3	49(5)	60(8)	61(6)	−5(5)	11(6)	3(5)
C_4	69(6)	59(8)	57(6)	1(6)	3(6)	14(5)
C_5	69(6)	60(8)	51(6)	9(6)	17(6)	13(5)
C_6	66(6)	68(8)	45(5)	6(6)	13(6)	12(5)
C_7	56(6)	60(9)	77(7)	19(6)	21(7)	20(6)
C_8	86(8)	80(9)	64(7)	6(8)	−24(7)	−4(6)

Table 3. Observed and calculated structure factors. Unobserved reflections are indicated by a minus sign on $F(O)$.

h	k	l	F(O)	F(C)	h	k	l	F(O)	F(C)	h	k	l	F(O)	F(C)	h	k	l	F(O)	F(C)	h	k	l	F(O)	F(C)
2	0	0	928	-975	30	0	8	181	177	40	0	-4	167	-193	8	0-16	235	232	31	1	3	261	206
4	0	0	2112	-2253	32	0	8	115	-123	2	0	-6	838	659	10	0-16	264	-204	33	1	3	185	117
6	0	0	435	353	34	0	8	-97	-139	4	0	-6	889	-839	12	0-16	332	-296	35	1	3	298	-279
8	0	0	1087	-948	36	0	8	114	143	6	0	-6	-76	14	14	0-16	394	364	37	1	3	-129	-5
10	0	0	151	58	0	0	10	1018	997	8	0	-6	2656	2556	16	0-16	258	269	39	1	3	243	269
12	0	0	1299	-1216	2	0	10	208	-170	10	0	-6	1928	-2074	18	0-16	260	-286	41	1	3	-77	-96
14	0	0	1041	1005	4	0	10	1307	-1346	12	0	-6	677	-990	20	0-16	175	-172	1	1	4	1315	-1313
16	0	0	336	-312	6	0	10	529	519	14	0	-6	1415	1552	22	0-16	221	255	3	1	4	124	-70
18	0	0	1111	-1115	8	0	10	500	979	16	0	-6	180	160	24	0-16	-101	-35	5	1	4	1406	1430
20	0	0	297	323	10	0	10	401	-364	18	0	-6	1483	-1575	26	0-16	88	-111	7	1	4	1359	-1358
22	0	0	1467	1438	12	0	10	-125	-21	20	0	-6	367	-390	2	0-18	-93	37	9	1	4	142	180
24	0	0	202	-182	14	0	10	482	493	22	0	-6	961	977	4	0-18	267	-287	11	1	4	1477	1449
26	0	0	930	-937	16	0	10	-132	-26	24	0	-6	703	675	6	0-18	-96	8	13	1	4	1070	-1087
28	0	0	123	157	18	0	10	592	-583	26	0	-6	-125	-5	8	0-18	280	289	15	1	4	1006	-983
30	0	0	477	471	20	0	10	259	225	28	0	-6	182	196	10	0-18	94	-90	17	1	4	476	473
32	0	0	418	-378	22	0	10	472	454	30	0	-6	190	225	12	0-18	203	-227	19	1	4	1130	1159
34	0	0	425	-423	24	0	10	353	-343	32	0	-6	573	-578	14	0-18	237	236	21	1	4	418	-404
36	0	0	344	310	26	0	10	367	-355	34	0	-6	-140	-113	16	0-18	137	126	23	1	4	442	-430
38	0	0	194	127	28	0	10	-118	71	36	0	-6	273	291	1	1	0	785	-686	25	1	4	531	568
40	0	0	240	-233	30	0	10	242	275	38	0	-6	-125	91	3	1	0	892	945	27	1	4	-143	-54
42	0	0	-84	-48	32	0	10	-82	1	40	0	-6	273	-264	5	1	0	1295	1225	29	1	4	420	-412
0	0	2	3100	3238	0	0	12	1045	1113	2	0	-8	952	-871	7	1	0	3496	-3557	31	1	4	-151	89
2	0	2	1845	-1813	2	0	12	276	-313	4	0	-8	1143	-1160	9	1	0	1820	1825	33	1	4	362	322
4	0	2	1565	-1471	4	0	12	1188	-1230	6	0	-8	597	633	11	1	0	2031	2054	35	1	4	-138	2
6	0	2	1078	1087	6	0	12	589	577	8	0	-8	2322	2498	13	1	0	435	345	37	1	4	254	-236
8	0	2	1594	1537	8	0	12	186	145	10	0	-8	500	-544	15	1	0	1241	-1159	39	1	4	97	116
10	0	2	1365	-1176	10	0	12	233	-185	12	0	-8	1274	-1327	17	1	0	311	-291	1	1	5	400	369
12	0	2	1613	-1574	12	0	12	195	55	14	0	-8	420	408	19	1	0	603	610	3	1	5	657	-647
14	0	2	1466	1552	14	0	12	-141	44	16	0	-8	366	381	21	1	0	742	-776	5	1	5	1057	-986
16	0	2	1738	1666	16	0	12	414	412	18	0	-8	324	-330	23	1	0	145	-146	7	1	5	664	624
18	0	2	-99	20	18	0	12	462	-472	20	0	-8	298	-350	25	1	0	777	724	9	1	5	1030	1025
20	0	2	370	-349	20	0	12	-135	12	22	0	-8	610	605	27	1	0	299	293	11	1	5	330	-304
22	0	2	610	661	22	0	12	361	349	24	0	-8	-126	-30	29	1	0	628	-636	13	1	5	673	-663
24	0	2	668	-656	24	0	12	230	-221	26	0	-8	903	-980	31	1	0	229	-205	15	1	5	183	-79
26	0	2	570	-557	26	0	12	198	-213	28	0	-8	736	716	33	1	0	378	339	17	1	5	661	631
28	0	2	638	661	28	0	12	138	140	30	0	-8	326	385	35	1	0	-151	-66	19	1	5	-128	61
30	0	2	694	728	0	0	14	421	427	32	0	-8	413	-407	37	1	0	340	-269	21	1	5	704	-669
32	0	2	566	-632	2	0	14	257	-273	34	0	-8	-139	-34	39	1	0	198	141	23	1	5	-136	-21
34	0	2	491	-630	4	0	14	458	-473	36	0	-8	-130	74	41	1	0	145	148	25	1	5	-140	73
36	0	2	423	445	6	0	14	411	356	38	0	-8	-115	129	43	1	0	122	-152	27	1	5	-146	-129
38	0	2	-119	-23	8	0	14	343	332	40	0	-8	211	-229	1	1	1	129	-132	29	1	5	-151	125
40	0	2	-100	-57	10	0	14	352	-349	2	0-10		-108	8	3	1	1	2005	2134	31	1	5	-151	106
0	0	4	453	415	12	0	14	345	-321	4	0-10		1383	-1448	5	1	1	1280	-1318	33	1	5	-144	-106
2	0	4	2494	-2737	14	0	14	284	289	6	0-10		510	509	7	1	1	2330	-2320	35	1	5	206	-171
4	0	4	3422	-3550	16	0	14	267	231	8	0-10		824	823	9	1	1	2022	2112	37	1	5	1110	92
6	0	4	2114	2271	18	0	14	311	-309	10	0-10		605	-563	11	1	1	478	510	39	1	5	116	129
8	0	4	1879	1956	20	0	14	-102	6	12	0-10		504	-544	13	1	1	308	-296	1	1	6	161	-162
10	0	4	455	-477	22	0	14	160	195	14	0-10		558	596	15	1	1	420	392	3	1	6	802	821
12	0	4	1727	-1670	0	0	16	213	237	16	0-10		522	532	17	1	1	161	-160	5	1	6	93	104
14	0	4	1481	1450	2	0	16	230	-157	18	0-10		281	-245	19	1	1	286	-221	7	1	6	1898	-1904
16	0	4	1217	954	4	0	16	291	-263	20	0-10		-124	6	21	1	1	-119	9	9	1	6	313	-281
18	0	4	1011	-951	6	0	16	331	330	22	0-10		-128	-11	23	1	1	588	564	11	1	6	1340	1383
20	0	4	-112	52	8	0	16	315	316	24	0-10		-134	42	25	1	1	-131	12	13	1	6	116	-138
22	0	4	529	515	10	0	16	305	-308	26	0-10		813	-820	27	1	1	568	-524	15	1	6	566	-559
24	0	4	256	-293	12	0	16	-103	-83	28	0-10		397	315	29	1	1	-141	148	17	1	6	157	179
26	0	4	573	-613	14	0	16	168	189	30	0-10		574	620	31	1	1	451	355	19	1	6	582	561
28	0	4	366	415	16	0	16	-74	31	32	0-10		461	-448	33	1	1	222	165	21	1	6	639	-601
30	0	4	412	406	0	0	18	-89	107	34	0-10		206	-133	35	1	1	-150	-122	23	1	6	139	-132
32	0	4	460	-421	2	0	-2	394	256	36	0-10		114	101	37	1	1	140	-116	25	1	6	537	516
34	0	4	-133	52	4	0	-2	2173	-2149	38	0-10		183	185	39	1	1	150	118	27	1	6	-150	72
36	0	4	358	287	6	0	-2	370	367	2	0-12		295	330	41	1	1	-98	-15	29	1	6	495	-436
38	0	4	-105	36	8	0	-2	1879	1901	4	0-12		851	-935	1	1	2	1236	-1332	31	1	6	-147	-81
40	0	4	-92	-106	10	0	-2	690	-757	6	0-12		-122	17	3	1	2	3057	3290	33	1	6	321	310
0	0	6	1011	991	12	0	-2	1895	-1925	8	0-12		511	565	5	1	2	2061	2272	35	1	6	-120	-43
2	0	6	588	521	14	0	-2	894	826	10	0-12		235	-269	7	1	2	772	-908	37	1	6	97	-117
4	0	6	1755	-1706	16	0	-2	-87	-80	12	0-12		199	-238	9	1	2	704	633	1	1	7	457	426
6	0	6	268	221	18	0	-2	1901	-1965	14	0-12		386	407	11	1	2	307	331	3	1	7	170	-139
8	0	6	341	385	20	0	-2	1049	1131	16	0-12		-129	32	13	1	2	-94	-68	5	1	7	-102	58
10	0	6	1360	-1483	22	0	-2	1342	1460	18	0-12		732	-739	15	1	2	1160	-1181	7	1	7	813	816
12	0	6	937	-872	24	0	-2	652	-673	20	0-12		-136	6	17	1	2	441	462	9	1	7	583	565
14	0	6	1917	1859	26	0	-2	995	-1134	22	0-12		634	650	19	1	2	822	824	11	1	7	161	-210
16	0	6	-111	-27	28	0	-2	407	-426	24	0-12		-143	43	21	1	2	773	-843	13	1	7	506	-492
18	0	6	1212	-1305	30	0	-2	657	701	26	0-12		427	-425	23	1	2	691	-730	15	1	7	203	204
20	0	6	121	-130	32	0	-2	-133	-50	28	0-12		139	103	25	1	2	792	788	17	1	7	493	442
22	0	6	553	605	34	0	-2	169	-183	30	0-12		163	213	27	1	2	513	555	19	1	7	377	-399
24	0	6	333	367	36	0	-2	261	270	32	0-12		121	-63	29	1	2	468	-539	21	1	7	515	-512
26	0	6	424	-428	38	0	-2	-130	47	34	0-12		131	-149	31	1	2	211	182	23	1	7	289	240
28	0	6	-136	66	40	0	-2	169	-187	36	0-12		87	85	33	1	2	151	148	25	1	7	258	219
30	0	6	-137	107	2	0	-4	737	-671	2	0-14		-138	48	35	1	2	147	-106	27	1	7	-151	-50
32	0	6	209	-266	4	0	-4	677	738	4	0-14		371	-308	37	1	2	233	-216	29	1	7	-149	12
34	0	6	-121	-50	6	0	-4	503	457	6	0-14		-137	140	39	1	2	142	134	31	1	7	-141	22
36	0	6	285	303	8	0	-4	728	727	8	0-14		463	445	41	1	2	127	166	33	1	7	-126	-31
0	0	8	971	953	10	0	-4	2353	-2468	10	0-14		-138	-60	1	1	3	902	920	35	1	7	-106	-52
2	0	8	779	742	12	0	-4	1390	-1363	12	0-14		545	-550	3	1	3	923	-874	1	1	8	-105	-12
4	0	8	805	-821	14	0	-4	1947	1931	14	0-14		495	407	5	1	3	2225	-2382	3	1	8	974	1011
6	0	8	735	726	16	0	-4	1314	1286	16	0-14		-141	88	7	1	3	1178	-1138	5	1	8	1039	1016
8	0	8	135	97	18	0	-4	1524	-1544	18	0-14		578	-564	9	1	3	1337	1279	7	1	8	861	-905
10	0	8	1617	-567	20	0	-4	391	-381	20	0-14		-141	11	11	1	3	257	204	9	1	8	567	-539
12	0	8	687	-691	22	0	-4	1125	1112	22	0-14		440	423	13	1	3	713	-709	11	1	8	888	972
14	0	8	1144	1189	24	0	-4	-116	57	24	0-14		-136	68	15	1	3	-105	3	13	1	8	418	-454
16	0	8	309	322	26	0	-4	446	-463	26	0-14		325	-331	17	1	3	448	439	15	1	8	622	-599
18	0	8	942	-1023	28	0	-4	126	82	28	0-14		-118	6	19	1	3	-118	44	17	1	8	371	366
20	0	8	-129	70	30	0	-4	362	350	30	0-14		182	197	21	1	3	-125	-81	19	1	8	391	367
22	0	8	595	624	32	0	-4	423	-403	32	0-14		-88	7	23	1	3	-130	72	21	1	8	598	-602
24	0	8	-136	-125	34	0	-4	197	-184	2	0-16		136	-52	25	1	3	269	-268	23	1	8	259	-192
26	0	8	468	-456	36	0	-4	276	234	4	0-16		271	-297	27	1	3	171	-147	25	1	8	402	376
28	0	8	136	165	38	0	-4	183	209	6	0-16		201	174	29	1	3	178	155	27	1	8	257	174

Table 3. Continued.

H	K	L	F(O)	F(C)	H	K	L	F(O)	F(C)	H	K	L	F(O)	F(C)	H	K	L	F(O)	F(C)	H	K	L	F(O)	F(C)
29	1	8	228	-215	13	1	16	121	-108	25	1	-5	245	236	29	1	-10	166	-119	5	1	-17	-135	-101
31	1	8	-130	-8	15	1	16	191	-201	27	1	-5	-147	58	31	1	-10	162	-182	7	1	-17	-139	-18
33	1	8	193	207	1	1	17	-121	71	29	1	-5	-153	-18	33	1	-10	265	229	9	1	-17	-134	78
35	1	8	87	-119	3	1	17	-116	7	31	1	-5	-159	52	35	1	-10	-137	-75	11	1	-17	-132	91
1	1	9	361	321	5	1	17	-109	-126	33	1	-5	-163	-59	37	1	-10	202	-213	13	1	-17	128	-159
3	1	9	306	247	7	1	17	-101	80	35	1	-5	-161	-144	39	1	-10	175	173	15	1	-17	-124	-24
5	1	9	-120	-124	9	1	17	-88	80	37	1	-5	282	216	1	1	-11	344	256	17	1	-17	202	144
7	1	9	-124	-35	1	1	18	135	-130	39	1	-5	135	103	3	1	-11	720	722	19	1	-17	-109	3
9	1	9	220	167	3	1	18	149	174	41	1	-5	-112	-111	5	1	-11	274	-212	21	1	-17	-95	-78
11	1	9	162	-113	1	1	-1	163	-176	1	1	-6	1109	-1048	7	1	-11	555	-521	1	1	-18	-97	-92
13	1	9	-136	-24	3	1	-1	1476	1702	3	1	-6	1399	1487	9	1	-11	-138	69	3	1	-18	160	89
15	1	9	410	356	5	1	-1	777	796	5	1	-6	116	-58	11	1	-11	240	176	5	1	-18	177	149
17	1	9	-143	52	7	1	-1	1151	-1226	7	1	-6	1380	-1512	13	1	-11	141	-108	7	1	-18	207	-175
19	1	9	296	-252	9	1	-1	736	744	9	1	-6	583	-526	15	1	-11	-144	-108	9	1	-18	-102	-90
21	1	9	313	-256	11	1	-1	422	383	11	1	-6	1681	1739	17	1	-11	252	216	11	1	-18	230	205
23	1	9	274	242	13	1	-1	272	228	13	1	-6	935	913	19	1	-11	299	-249	13	1	-18	-94	46
25	1	9	340	303	15	1	-1	663	671	15	1	-6	1077	-1089	21	1	-11	-156	-166	15	1	-18	205	-188
27	1	9	249	-233	17	1	-1	202	186	17	1	-6	581	558	23	1	-11	485	438	0	2	0	969	1014
29	1	9	-132	-75	19	1	-1	450	-464	19	1	-6	328	308	25	1	-11	-167	127	2	2	0	256	-232
31	1	9	-116	132	21	1	-1	567	-483	21	1	-6	479	-467	27	1	-11	287	-222	4	2	0	129	-105
33	1	9	-93	-36	23	1	-1	686	657	23	1	-6	411	-412	29	1	-11	-163	-145	6	2	0	433	-400
1	1	10	790	-790	25	1	-1	382	351	25	1	-6	713	709	31	1	-11	190	118	8	2	0	796	687
3	1	10	863	888	27	1	-1	708	-705	27	1	-6	-150	90	33	1	-11	-142	52	10	2	0	1311	-1228
5	1	10	-128	77	29	1	-1	254	-232	29	1	-6	598	-603	35	1	-11	152	-119	12	2	0	834	-802
7	1	10	444	-463	31	1	-1	276	225	31	1	-6	-162	-71	37	1	-11	-100	-6	14	2	0	1884	1858
9	1	10	185	169	33	1	-1	-160	146	33	1	-6	491	390	1	1	-12	636	-652	16	2	0	-93	-25
11	1	10	780	777	35	1	-1	-161	-92	35	1	-6	160	153	3	1	-12	645	620	18	2	0	657	-630
13	1	10	343	-334	37	1	-1	-153	-58	37	1	-6	237	-165	5	1	-12	-144	119	20	2	0	548	-568
15	1	10	709	-693	39	1	-1	-138	8	39	1	-6	-131	89	7	1	-12	714	-685	22	2	0	638	584
17	1	10	284	255	41	1	-1	-114	-57	41	1	-6	-107	62	9	1	-12	-145	-64	24	2	0	347	330
19	1	10	305	278	43	1	-1	-85	-11	1	1	-7	1043	961	11	1	-12	889	893	26	2	0	252	-232
21	1	10	265	-225	1	1	-2	2135	-2410	3	1	-7	-99	28	13	1	-12	-149	86	28	2	0	148	178
23	1	10	-151	-126	3	1	-2	209	-150	5	1	-7	1421	-1401	15	1	-12	526	-527	30	2	0	-131	-90
25	1	10	245	216	5	1	-2	127	119	7	1	-7	568	544	17	1	-12	-155	28	32	2	0	255	-186
27	1	10	-133	64	7	1	-2	1518	-1643	9	1	-7	645	845	19	1	-12	294	267	34	2	0	-131	67
29	1	10	216	-228	9	1	-2	145	169	11	1	-7	132	94	21	1	-12	380	-324	36	2	0	308	274
31	1	10	-96	41	11	1	-2	1857	1968	13	1	-7	500	-540	23	1	-12	203	-205	38	2	0	-106	28
1	1	11	347	271	13	1	-2	484	-453	15	1	-7	577	-552	25	1	-12	371	298	40	2	0	238	-233
3	1	11	235	250	15	1	-2	1700	-1816	17	1	-7	657	648	27	1	-12	-163	114	0	2	1	339	-345
5	1	11	-135	-28	17	1	-2	674	666	19	1	-7	206	223	29	1	-12	308	-236	2	2	1	1183	-1209
7	1	11	457	-468	19	1	-2	886	1012	21	1	-7	216	-237	31	1	-12	-143	-97	4	2	1	190	174
9	1	11	253	223	21	1	-2	383	-383	23	1	-7	-143	65	33	1	-12	268	211	6	2	1	827	802
11	1	11	177	172	23	1	-2	662	-657	25	1	-7	-148	-122	35	1	-12	-106	14	8	2	1	275	217
13	1	11	389	-335	25	1	-2	506	508	27	1	-7	-153	-20	1	1	-13	-154	10	10	2	1	1117	-1069
15	1	11	282	237	27	1	-2	-144	-59	29	1	-7	-159	-27	3	1	-13	306	277	12	2	1	1227	1256
17	1	11	-156	62	29	1	-2	449	-451	31	1	-7	282	266	5	1	-13	-153	30	14	2	1	1004	1020
19	1	11	254	-232	31	1	-2	154	141	33	1	-7	-164	89	7	1	-13	-153	-39	16	2	1	1545	-1621
21	1	11	-151	28	33	1	-2	433	363	35	1	-7	341	-297	9	1	-13	244	177	18	2	1	-101	13
23	1	11	203	158	35	1	-2	-161	-63	37	1	-7	249	145	11	1	-13	-156	-13	20	2	1	879	865
25	1	11	-132	52	37	1	-2	467	-434	39	1	-7	177	149	13	1	-13	-158	-104	22	2	1	181	-194
27	1	11	230	-157	39	1	-2	-139	74	41	1	-7	-99	-79	15	1	-13	-160	12	24	2	1	259	-194
29	1	11	-96	-8	41	1	-2	216	168	1	1	-8	1237	-1330	17	1	-13	-163	104	26	2	1	171	110
1	1	12	-138	-124	1	1	-3	382	-371	3	1	-8	707	676	19	1	-13	261	-189	28	2	1	371	319
3	1	12	611	574	3	1	-3	1164	1157	5	1	-8	508	489	21	1	-13	203	-216	30	2	1	-132	-108
5	1	12	143	-169	5	1	-3	952	-933	7	1	-8	870	-851	23	1	-13	203	163	32	2	1	263	-236
7	1	12	424	-358	7	1	-3	290	-226	9	1	-8	-113	-64	25	1	-13	257	204	34	2	1	252	184
9	1	12	204	-137	9	1	-3	302	262	11	1	-8	604	640	27	1	-13	-154	-108	36	2	1	-119	44
11	1	12	656	615	11	1	-3	1604	-1592	13	1	-8	396	-318	29	1	-13	226	-157	38	2	1	179	-111
13	1	12	-155	-188	13	1	-3	112	-64	15	1	-8	982	-1054	31	1	-13	-127	91	40	2	1	-79	-27
15	1	12	362	-304	15	1	-3	560	955	17	1	-8	528	446	33	1	-13	-108	73	0	2	2	346	347
17	1	12	190	189	17	1	-3	544	567	19	1	-8	1252	1232	1	1	-14	401	-303	2	2	2	566	578
19	1	12	211	186	19	1	-3	-117	-24	21	1	-8	260	-215	3	1	-14	301	242	4	2	2	78	86
21	1	12	-141	-88	21	1	-3	841	-894	23	1	-8	341	-323	5	1	-14	279	239	6	2	2	-63	20
23	1	12	221	-202	23	1	-3	210	195	25	1	-8	319	350	7	1	-14	494	-434	8	2	2	408	375
25	1	12	285	280	25	1	-3	523	562	27	1	-8	-157	43	9	1	-14	-162	-107	10	2	2	1568	-1556
27	1	12	-92	9	27	1	-3	428	-431	29	1	-8	470	-452	11	1	-14	330	381	12	2	2	-85	-52
1	1	13	330	326	29	1	-3	320	-253	31	1	-8	-165	-132	13	1	-14	-164	-42	14	2	2	1400	1372
3	1	13	149	155	31	1	-3	-155	30	33	1	-8	518	455	15	1	-14	493	-359	16	2	2	-97	118
5	1	13	328	-299	33	1	-3	-161	-79	35	1	-8	243	-162	17	1	-14	-165	35	18	2	2	701	-684
7	1	13	266	-172	35	1	-3	-162	-23	37	1	-8	138	-167	19	1	-14	547	495	20	2	2	557	-571
9	1	13	376	465	37	1	-3	-154	89	39	1	-8	-117	85	21	1	-14	303	-196	22	2	2	660	687
11	1	13	-156	-24	39	1	-3	-139	11	41	1	-8	-86	67	23	1	-14	-158	-164	24	2	2	205	-228
13	1	13	406	-232	41	1	-3	-117	-34	1	1	-9	843	817	25	1	-14	238	169	26	2	2	198	-219
15	1	13	-153	122	1	1	-4	1437	-1573	3	1	-9	644	562	27	1	-14	-138	43	28	2	2	129	130
17	1	13	-146	-82	3	1	-4	1763	1914	5	1	-9	671	-663	29	1	-14	213	-155	30	2	2	-133	119
19	1	13	-136	-62	5	1	-4	1031	1126	7	1	-9	447	-451	31	1	-14	-105	-9	32	2	2	-133	53
21	1	13	122	94	7	1	-4	1421	-1555	9	1	-9	378	349	1	1	-15	-165	-60	34	2	2	127	-135
23	1	13	-107	21	9	1	-4	934	-995	11	1	-9	380	392	3	1	-15	326	225	36	2	2	140	135
1	1	14	267	-158	11	1	-4	1120	1144	13	1	-9	611	-599	5	1	-15	201	-126	38	2	2	-96	-40
3	1	14	558	457	13	1	-4	371	322	15	1	-9	225	-207	7	1	-15	-184	23	40	2	2	-70	-126
5	1	14	157	180	15	1	-4	1012	-1031	17	1	-9	630	656	9	1	-15	282	230	0	2	3	-54	51
7	1	14	455	-404	17	1	-4	1025	1001	19	1	-9	171	-145	11	1	-15	281	-163	2	2	3	1446	-1408
9	1	14	-155	-57	19	1	-4	277	284	21	1	-9	287	191	13	1	-15	230	-183	4	2	3	864	729
11	1	14	282	232	21	1	-4	1055	-1027	23	1	-9	369	368	15	1	-15	-161	74	6	2	3	884	809
13	1	14	148	-166	23	1	-4	832	-905	25	1	-9	412	-382	17	1	-15	224	179	8	2	3	636	-546
15	1	14	242	-211	25	1	-4	607	560	27	1	-9	-162	-58	19	1	-15	-155	18	10	2	3	853	-786
17	1	14	180	155	27	1	-4	436	417	29	1	-9	-166	-82	21	1	-15	257	-198	12	2	3	834	800
19	1	14	266	252	29	1	-4	239	-228	31	1	-9	-165	103	23	1	-15	-141	-16	14	2	3	223	200
21	1	14	175	-169	31	1	-4	-157	45	33	1	-9	159	174	25	1	-15	240	192	16	2	3	1077	-1132
23	1	14	88	-110	33	1	-4	467	432	35	1	-9	357	-319	27	1	-15	-116	-75	18	2	3	199	174
1	1	15	328	246	35	1	-4	-162	-55	37	1	-9	-129	-34	29	1	-15	-96	-37	20	2	3	550	560
3	1	15	-154	-105	37	1	-4	345	-341	39	1	-9	166	163	1	1	-16	189	-157	22	2	3	-116	-38
5	1	15	186	-152	39	1	-4	239	176	1	1	-10	691	-720	3	1	-16	311	235	24	2	3	304	-286
7	1	15	-149	48	41	1	-4	-116	88	3	1	-10	1100	1038	5	1	-16	329	247	26	2	3	-126	-62
9	1	15	293	193	1	1	-5	364	353	5	1	-10	449	451	7	1	-16	384	-333	28	2	3	358	405
11	1	15	-137	13	3	1	-5	291	-264	7	1	-10	689	-683	9	1	-16	245	-204	30	2	3	-134	-121
13	1	15	222	-187	5	1	-5	582	-984	9	1	-10	275	-247	11	1	-16	322	254	32	2	3	279	-322
15	1	15	-116	33	7	1	-5	372	362	11	1	-10	658	664	13	1	-16	-151	-8	34	2	3	213	210
17	1	15	-101	67	9	1	-5	189	166	13	1	-10	299	-319	15	1	-16	253	-230	36	2	3	-108	93
19	1	15	-82	-42	11	1	-5	486	-467	15	1	-10	237	-237	17	1	-16	225	157	38	2	3	-88	-92
1	1	16	248	-188	13	1	-5	893	-910	17	1	-10	-141	51	19	1	-16	269	214	0	2	4	1106	1076
3	1	16	280	256	15	1	-5	-106	-65	19	1	-10	630	643	21	1	-16	221	-176	2	2	4	-66	-77
5	1	16	-136	71	17	1	-5	1111	1105	21	1	-10	477	-421	23	1	-16	-117	-96	4	2	4	1328	-1291
7	1	16	298	-243	19	1	-5	256	-217	23	1	-10	605	-614	25	1	-16	-101	60	6	2	4	514	453
9	1	16	-122	14	21	1	-5	809	-838	25	1	-10	550	542	1	1	-17	-132	114	8	2	4	743	703
11	1	16	213	209	23	1	-5	194	166	27	1	-10	166	159	3	1	-17	-134	3	10	2	4	295	334

Table 3. Continued.

H	K	L	F(O)	F(C)	H	K	L	F(O)	F(C)	H	K	L	F(O)	F(C)	H	K	L	F(O)	F(C)	H	K	L	F(O)	F(C)
12	2	4	133	-126	2	2	10	376	-373	2	2	-2	1133	-1158	16	2	-7	785	-838	16	2-13		220	-237
14	2	4	359	383	4	2	10	244	-228	4	2	-2	1483	-1337	18	2	-7	206	-191	18	2-13		-137	-134
16	2	4	342	-357	6	2	10	417	420	6	2	-2	1503	1432	20	2	-7	479	522	20	2-13		218	169
18	2	4	602	-669	8	2	10	482	527	8	2	-2	1899	1915	22	2	-7	209	230	22	2-13		189	199
20	2	4	315	290	10	2	10	445	-476	10	2	-2	1013	-937	24	2	-7	374	-371	24	2-13		287	-259
22	2	4	563	583	12	2	10	407	-434	12	2	-2	737	-911	26	2	-7	-130	72	26	2-13		-120	-77
24	2	4	214	-163	14	2	10	335	289	14	2	-2	-85	70	28	2	-7	407	443	28	2-13		173	188
26	2	4	502	-550	16	2	10	135	177	16	2	-2	458	629	30	2	-7	168	-117	30	2-13		135	-109
28	2	4	-133	65	18	2	10	194	-216	18	2	-2	-99	26	32	2	-7	191	-209	2	2-14		168	-139
30	2	4	327	349	20	2	10	-135	-61	20	2	-2	-106	-13	34	2	-7	-127	75	4	2-14		406	-399
32	2	4	-128	-34	22	2	10	160	179	22	2	-2	444	450	36	2	-7	161	186	6	2-14		335	310
34	2	4	143	-118	24	2	10	-122	-42	24	2	-2	324	-313	38	2	-7	168	-166	8	2-14		355	344
36	2	4	-100	26	26	2	10	110	-142	26	2	-2	331	-346	2	2	-8	153	-165	10	2-14		-136	-96
38	2	4	-78	-19	28	2	10	95	92	28	2	-2	235	233	4	2	-8	867	-810	12	2-14		330	-325
0	2	5	-72	149	30	2	10	-74	37	30	2	-2	227	190	6	2	-8	97	39	14	2-14		-135	41
2	2	5	759	-741	0	2	11	-119	145	32	2	-2	351	-332	8	2	-8	416	424	16	2-14		-134	48
4	2	5	1034	537	2	2	11	449	-464	34	2	-2	-132	-30	10	2	-8	277	-246	18	2-14		-132	-50
6	2	5	572	566	4	2	11	-123	84	36	2	-2	216	210	12	2	-8	-104	-105	20	2-14		-128	45
8	2	5	556	-570	6	2	11	464	477	38	2	-2	-110	70	14	2	-8	464	472	22	2-14		151	121
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26	2	5	131	104	24	2	11	188	-200	16	2	-3	628	-654	32	2	-8	-132	149	12	2-15		-129	86
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32	2	5	244	-276	0	2	12	-127	156	22	2	-3	-112	-97	38	2	-8	-89	-43	18	2-15		-119	-94
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6	2	7	889	934	0	2	14	333	315	32	2	-4	432	-452	14	2	-10	584	638	7	3	0	441	-487
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20	2	7	457	485	14	2	14	257	262	6	2	-5	1159	1213	28	2	-10	136	52	21	3	0	376	-417
22	2	7	-130	-18	16	2	14	-102	-4	8	2	-5	609	-569	30	2	-10	130	92	23	3	0	351	347
24	2	7	365	-400	18	2	14	154	-159	10	2	-5	514	-477	32	2	-10	147	-129	25	3	0	279	212
26	2	7	-133	119	20	2	14	-71	50	12	2	-5	708	694	34	2	-10	-106	-23	27	3	0	-172	30
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30	2	7	239	-230	2	2	15	224	-244	16	2	-5	356	-380	4	2	-11	268	-226	31	3	0	-170	-31
32	2	7	105	-123	4	2	15	126	133	18	2	-5	490	-492	6	2	-11	145	164	33	3	0	195	108
34	2	7	148	164	6	2	15	122	161	20	2	-5	378	395	8	2	-11	-119	39	35	3	0	141	-55
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4	2	8	637	-619	12	2	15	175	206	26	2	-5	176	176	14	2	-11	702	752	1	3	1	474	499
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14	2	8	320	337	4	2	16	151	-176	36	2	-5	172	194	24	2	-11	365	-350	11	3	1	352	-361
16	2	8	284	263	6	2	16	-101	25	38	2	-5	-106	-69	26	2	-11	-137	-5	13	3	1	1064	-1117
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22	2	8	-134	75	12	2	16	-68	-42	4	2	-6	1248	-1303	32	2	-11	110	-111	19	3	1	512	-512
24	2	8	134	-75	0	2	17	110	-110	6	2	-6	363	-318	34	2	-11	-94	106	21	3	1	645	-684
26	2	8	130	-143	2	2	17	120	-174	8	2	-6	485	460	2	2	-12	178	93	23	3	1	323	328
28	2	8	226	243	4	2	17	78	102	10	2	-6	-87	8	4	2	-12	385	-387	25	3	1	300	268
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6	2	9	400	432	14	2	-1	578	539	24	2	-6	222	-233	18	2	-12	344	-339	1	3	2	166	-183
8	2	9	600	-617	16	2	-1	1054	-1045	26	2	-6	289	-247	20	2	-12	137	-131	3	3	2	624	609
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14	2	9	383	437	22	2	-1	310	307	32	2	-6	218	-144	26	2	-12	-131	-98	9	3	2	740	-740
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18	2	9	134	-224	26	2	-1	208	159	36	2	-6	168	164	30	2	-12	111	12	13	3	2	238	160
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22	2	9	135	-52	30	2	-1	352	-294	2	2	-7	1137	-1158	2	2	-13	420	-439	17	3	2	679	760
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26	2	9	122	147	34	2	-1	279	269	6	2	-7	753	762	6	2	-13	283	291	21	3	2	268	-267
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30	2	9	149	-149	38	2	-1	267	-206	10	2	-7	454	-453	10	2	-13	267	-307	25	3	2	170	-233
32	2	9	-71	-64	40	2	-1	109	-66	12	2	-7	423	427	12	2	-13	296	319	27	3	2	246	120
0	2	10	174	183	42	2	-1	-64	85	14	2	-7	-101	122	14	2	-13	238	228	29	3	2	173	-177

Table 3. Continued.

H	K	L	F(O)	F(C)	H	K	L	F(O)	F(C)	H	K	L	F(O)	F(C)	H	K	L	F(O)	F(C)	H	K	L	F(O)	F(C)
31	3	2	-165	-36	5	3	9	759	-737	33	3	-2	-162	94	21	3	-8	-173	85	11	3-15	-163	82
33	3	2	-150	75	7	3	9	-159	-34	35	3	-2	-145	-70	23	3	-8	-180	91	13	3-15	259	-212
35	3	2	-129	-37	9	3	9	774	787	37	3	-2	-121	-28	25	3	-8	-180	-1	15	3-15	-133	-40
37	3	2	-98	-13	11	3	9	-170	60	1	3	-3	1067	1150	27	3	-8	217	-78	17	3-15	218	216
1	3	3	973	541	13	3	9	556	-515	3	3	-3	426	440	29	3	-8	-169	-33	19	3-15	113	-77
2	3	3	654	622	15	3	9	-179	69	5	3	-3	1464	-1542	31	3	-8	-156	109	0	4	0	-115	3
5	3	3	923	-929	17	3	9	386	313	7	3	-3	558	-578	33	3	-8	-137	71	2	4	0	-115	-67
7	3	3	501	-435	19	3	9	176	-134	9	3	-3	1038	1094	35	3	-8	-113	-35	4	4	0	-116	7
9	3	3	513	504	21	3	9	239	-219	11	3	-3	317	310	1	3	-9	274	216	6	4	0	537	-572
11	3	3	295	-317	23	3	9	192	154	13	3	-3	901	-929	3	3	-9	548	502	8	4	0	-120	118
13	3	3	428	-414	25	3	9	-141	94	15	3	-3	-131	21	5	3	-9	373	-278	10	4	0	608	606
15	3	3	792	737	27	3	9	309	-209	17	3	-3	695	699	7	3	-9	255	-235	12	4	0	127	172
17	3	3	795	816	1	3	10	518	-471	19	3	-3	-146	-31	9	3	-9	413	444	14	4	0	-132	-43
19	3	3	611	-555	3	3	10	-161	-119	21	3	-3	247	-212	11	3	-9	464	-376	16	4	0	407	-352
21	3	3	580	-551	5	3	10	-165	67	23	3	-3	400	390	13	3	-9	731	-772	18	4	0	-146	106
23	3	3	434	430	7	3	10	-169	-5	25	3	-3	167	100	15	3	-9	436	356	20	4	0	-154	89
25	3	3	323	272	9	3	10	-174	63	27	3	-3	521	-469	17	3	-9	795	726	22	4	0	-158	6
27	3	3	416	-345	11	3	10	-178	26	29	3	-3	-176	-6	19	3	-9	-173	-109	24	4	0	-149	-190
29	3	3	-171	-151	13	3	10	-180	55	31	3	-3	403	364	21	3	-9	479	-440	26	4	0	-139	-59
31	3	3	300	230	15	3	10	-181	-110	33	3	-3	-162	-63	23	3	-9	286	237	28	4	0	-126	123
33	3	3	-143	46	17	3	10	-176	18	35	3	-3	369	-380	25	3	-9	253	262	30	4	0	-110	-65
35	3	3	168	-122	19	3	10	168	118	37	3	-3	-148	24	27	3	-9	173	-199	32	4	0	-92	86
1	3	4	232	165	21	3	10	-155	-150	1	3	-4	465	-457	29	3	-9	162	-122	34	4	0	-67	30
3	3	4	254	314	23	3	10	-139	-54	3	3	-4	852	891	31	3	-9	-145	153	1	5	0	403	369
5	3	4	235	-249	25	3	10	-120	62	5	3	-4	156	-111	33	3	-9	-125	-13	3	5	0	432	-396
7	3	4	-113	-191	1	3	11	296	264	7	3	-4	217	-241	35	3	-9	-96	-119	5	5	0	170	-132
9	3	4	-122	66	3	3	11	-172	116	9	3	-4	252	219	1	3-10		249	187	7	5	0	-141	67
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13	3	4	-139	-74	7	3	11	-177	88	13	3	-4	-124	-94	5	3-10		391	369	11	5	0	178	-158
15	3	4	237	-216	9	3	11	509	465	15	3	-4	-133	-28	7	3-10		-157	60	13	5	0	-147	-40
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21	3	4	-164	67	15	3	11	213	201	21	3	-4	155	-137	13	3-10		309	-228	19	5	0	-145	-68
23	3	4	-171	-13	17	3	11	430	376	23	3	-4	380	-359	15	3-10		-170	-53	21	5	0	130	141
25	3	4	174	-123	19	3	11	151	-57	25	3	-4	317	228	17	3-10		213	248	23	5	0	-114	54
27	3	4	-174	-80	21	3	11	253	-233	27	3	-4	-175	11	19	3-10		177	-157	25	5	0	204	-156
29	3	4	-167	-103	23	3	11	142	170	29	3	-4	-176	-83	21	3-10		-180	-106	27	5	0	-76	-40
31	3	4	-153	9	1	3	12	397	-259	31	3	-4	-172	75	23	3-10		-180	13	0	6	0	123	-72
33	3	4	-133	136	3	3	12	-177	144	33	3	-4	-160	-115	25	3-10		303	214	2	6	0	-123	-135
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5	3	5	792	-803	11	3	12	173	213	3	3	-5	659	930	33	3-10		-108	2	10	6	0	140	81
7	3	5	595	-582	13	3	12	-168	4	5	3	-5	1178	-1066	1	3-11		443	349	12	6	0	-110	82
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11	3	5	-137	50	17	3	12	-144	-22	9	3	-5	739	715	5	3-11		455	-393	16	6	0	-80	-28
13	3	5	628	-612	19	3	12	-128	-23	11	3	-5	396	422	7	3-11		206	-165	0	4	1	-151	122
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17	3	5	336	357	1	3	13	303	229	15	3	-5	291	242	11	3-11		-171	-30	4	4	1	-154	117
19	3	5	544	-538	3	3	13	175	163	17	3	-5	251	275	13	3-11		592	-548	6	4	1	821	832
21	3	5	317	-274	5	3	13	274	-255	19	3	-5	508	-515	15	3-11		-176	23	8	4	1	432	-444
23	3	5	478	456	7	3	13	-170	40	21	3	-5	398	-392	17	3-11		531	524	10	4	1	317	-274
25	3	5	264	164	9	3	13	308	271	23	3	-5	527	493	19	3-11		-178	-79	12	4	1	519	504
27	3	5	422	-363	11	3	13	193	-172	25	3	-5	280	279	21	3-11		379	-353	14	4	1	-177	136
29	3	5	156	-109	13	3	13	310	-280	27	3	-5	668	-690	23	3-11		303	208	16	4	1	-185	-114
31	3	5	327	304	15	3	13	133	142	29	3	-5	177	-142	25	3-11		260	222	18	4	1	275	-244
33	3	5	-121	50	17	3	13	164	175	31	3	-5	381	370	27	3-11		214	-116	20	4	1	249	221
1	3	6	207	209	1	3	14	164	-120	33	3	-5	-158	61	29	3-11		-135	-74	22	4	1	-204	7
3	3	6	-122	68	3	3	14	-161	123	35	3	-5	283	-243	31	3-11		139	112	24	4	1	524	-498
5	3	6	252	-217	5	3	14	-157	113	37	3	-5	-114	-24	1	3-12		-175	30	26	4	1	356	299
7	3	6	377	-408	7	3	14	-151	-114	1	3	-6	204	162	3	3-12		-175	63	28	4	1	366	311
9	3	6	-118	81	9	3	14	-163	-4	3	3	-6	389	428	5	3-12		-175	-174	30	4	1	278	-231
11	3	6	357	351	11	3	14	132	74	5	3	-6	407	-365	7	3-12		175	131	32	4	1	139	119
13	3	6	194	89	13	3	14	-117	-8	7	3	-6	276	-290	9	3-12		-175	-121	1	5	1	667	720
15	3	6	222	-82	1	3	15	290	234	9	3	-6	-125	19	11	3-12		279	198	3	5	1	293	256
17	3	6	-164	9	3	3	15	218	154	11	3	-6	353	359	13	3-12		-177	74	5	5	1	420	-342
19	3	6	-170	208	5	3	15	220	-221	13	3	-6	165	-177	15	3-12		-177	-89	7	5	1	-197	-46
21	3	6	-175	-126	7	3	15	-122	-39	15	3	-6	351	-329	17	3-12		-176	49	9	5	1	-190	132
23	3	6	-176	-53	9	3	15	171	178	17	3	-6	-147	44	19	3-12		-174	-73	11	5	1	-193	77
25	3	6	-173	-36	1	3	16	-183	-53	19	3	-6	-155	96	21	3-12		-169	-69	13	5	1	195	-214
27	3	6	-162	27	1	3	-1	672	765	21	3	-6	317	257	23	3-12		-161	9	15	5	1	-195	-84
29	3	6	-160	-69	3	3	-1	162	183	23	3	-6	-170	-144	25	3-12		211	183	17	5	1	-193	99
31	3	6	-130	16	5	3	-1	1090	-1203	25	3	-6	-176	145	27	3-12		-134	39	19	5	1	-186	-101
1	3	7	558	610	7	3	-1	273	235	27	3	-6	-178	4	29	3-12		179	-168	21	5	1	-165	34
3	3	7	333	350	9	3	-1	1404	1434	29	3	-6	278	-242	31	3-12		-79	22	23	5	1	205	184
5	3	7	516	-519	11	3	-1	-139	31	31	3	-6	-168	89	1	3-13		306	237	25	5	1	122	85
7	3	7	263	-159	13	3	-1	1303	-1300	33	3	-6	-131	-6	3	3-13		-175	155	27	5	1	246	-208
9	3	7	1007	995	15	3	-1	-128	-122	35	3	-6	-113	-9	5	3-13		458	-401	0	6	1	-162	22
11	3	7	240	131	17	3	-1	953	953	1	3	-7	500	490	7	3-13		-175	-123	2	6	1	-161	-72
13	3	7	561	-569	19	3	-1	-144	-90	3	3	-7	689	704	9	3-13		624	583	4	6	1	-160	22
15	3	7	-165	-52	21	3	-1	463	-477	5	3	-7	564	-539	11	3-13		-174	173	6	6	1	-158	-84
17	3	7	-172	122	23	3	-1	355	258	7	3	-7	333	-393	13	3-13		457	-362	8	6	1	-154	-133
19	3	7	375	-283	25	3	-1	-165	154	9	3	-7	627	604	15	3-13		170	-127	10	6	1	-148	43
21	3	7	217	-167	27	3	-1	409	-363	11	3	-7	-137	22	17	3-13		264	230	12	6	1	140	179
23	3	7	424	393	29	3	-1	-175	40	13	3	-7	564	-493	19	3-13		-162	-49	2	4	-1	848	-871
25	3	7	-159	76	31	3	-1	352	304	15	3	-7	309	317	21	3-13		-155	-157	4	4	-1	-153	-101
27	3	7	330	-280	33	3	-1	-161	-93	17	3	-7	377	379	23	3-13		143	139	6	4	-1	689	644
29	3	7	-136	14	35	3	-1	281	-246	19	3	-7	489	-449	25	3-13		-128	65	8	4	-1	305	-330
31	3	7	263	276	37	3	-1	173	143	21	3	-7	439	-479	27	3-13		202	-173	10	4	-1	466	-483
1	3	8	308	-321	39	3	-1	157	190	23	3	-7	486	408	1	3-14		-166	-94	12	4	-1	493	504
3	3	8	-141	61	1	3	-2	296	-312	25	3	-7	312	273	3	3-14		-166	-39	14	4	-1	-173	87
5	3	8	-145	-47	3	3	-2	77	128	27	3	-7	485	-415	5	3-14		-167	75	16	4	-1	517	-393
7	3	8	255	-220	5	3	-2	-177	100	29	3	-7	-174	-237	7	3-14		-166	-26	18	4	-1	-190	28
9	3	8	-154	155	7	3	-2	367	327	31	3	-7	357	302	9	3-14		-165	124	20	4	-1	561	450
11	3	8	-160	-63	9	3	-2	154	165	33	3	-7	146	160	11	3-14		-163	121	22	4	-1	-206	-119
13	3	8	-167	-21	11	3	-2	-109	-57	35	3	-7	-125	-106	13	3-14		278	-189	24	4	-1	557	-458
15	3	8	-173	207	13	3	-2	326	-283	1	3	-8	138	-146	15	3-14		-155	-15	26	4	-1	228	135
17	3	8	-178	-21	15	3	-2	250	-264	3	3	-8	422	403	17	3-14		150	56	28	4	-1	379	296
19	3	8	282	268	17	3	-2	400	364	5	3	-8	-138	76	19	3-14		-143	20	30	4	-1	-149	-72
21	3	8	176	-151	19	3	-2	177	134	7	3	-8	-139	-74	21	3-14		-132	-80	32	4	-1	-126	-138
23	3	8	169	-194	21	3	-2	-151	-3	9	3	-8	-141	146	23	3-14		-165	-47	34	4	-1	133	103
25	3	8	-197	90	23	3	-2	276	-143	11	3	-8	251	225	1	3-15		230	192	1	5	-1	314	350
27	3	8	-140	16	25	3	-2	166	125	13	3	-8	402	-341	3	3-15		208	238	3	5	-1	429	506
29	3	8	-118	-8	27	3	-2	-172	101	15	3	-8	338	-370	5	3-15		233	-217	5	5	-1	293	-258
1	3	9	672	629	29	3	-2	-175	-143	17	3	-8	-159	36	7	3-15		147	-152	7	5	-1	-185	26
3	3	9	-151	46	31	3	-2	-172	46	19	3	-8	-166	23	9	3-15		272	231	9	5	-1	-188	227

Table 3. Continued.

H	K	L	F(O)	F(C)	H	K	L	F(O)	F(C)	H	K	L	F(O)	F(C)	H	K	L	F(O)	F(C)	H	K	L	F(O)	F(C)
11	5	-1	-195	-156	17	5	-1	-193	44	23	5	-1	243	227	4	6	-1	-160	75	12	6	-1	-144	210
13	5	-1	236	-241	19	5	-1	301	-294	25	5	-1	-132	149	6	6	-1	-158	37	14	6	-1	-130	126
15	5	-1	-164	129	21	5	-1	-173	6	27	5	-1	145	-115	8	6	-1	190	-225	16	6	-1	-108	-95
										2	6	-1	-161	-111	10	6	-1	150	-89	18	6	-1	-74	-33

and 8, respectively, for crystal No. 3. This brought R down to 0.11. The observed structure factors were next corrected for secondary extinction using the method of Zachariasen,[5] neglecting the absorption term: $F_{corr} = K\ F_o (1 + \beta C I_o)$, where $\beta = 2(1 + \cos^4 2\theta)/(1 + \cos^2 2\theta)^2$, and C was found to be 1.5×10^{-6}. This brought R down to 0.10. The intensities of reflections that occurred more than once in the data set were averaged, and such reflections thereafter included only once.

The final refinement based on the corrected observed structure factors, and with anisotropic temperature factors, brought the reliability index, R, down to 0.074, with unobserved reflections included if $|F_c|$ exceeds the observable limit.

The programs used for the least squares refinement and for the absorption correction were made available by the Chemical Department of X-Ray Crystallography, Weizmann Institute of Science, Rehovoth, Israel, and modified for use on the IBM 360/50H computer by Dr. D. Rabinovich. The program used for extinction correction was written by K. Maartmann-Moe of this institute.

The calculated structure factors were based on the atomic scattering factors given in the *International Tables* (Ref. 6, Table 3.3.1A). The scattering factors for selenium were corrected for anomalous dispersion, real and imaginary parts (Ref. 6, Table 3.3.2A), by taking the amplitude of f as the corrected value.

The final atomic coordinates are listed in Table 1, the temperature parameters in Table 2, and structure factors in Table 3.

RESULTS

Bond lengths and angles in the adduct of *o*-nitrobenzeneselenenyl thiocyanate with thiourea, based on the atomic coordinates in Table 1, are listed in Table 4. The uncertainties in the cell dimensions are taken into account in the given standard deviations. Drawings of the molecule are reproduced in Figs. 1 and 2. As seen from the drawings, the thiourea group has replaced the thiocyanate group, in accordance with the greater nucleophilic reactivity of thiourea, as compared with thiocyanate ion, toward divalent selenium. A salt is thus formed.

The selenium atom, the sulphur atom of the thiourea group, the atoms of the benzene ring, and the atoms of the nitro group, are nearly co-planar. The largest deviation of an atom from a least squares plane is 0.067 Å (*cf.* Table 5). The atoms of the thiourea group are also co-planar. These two planes make an angle of 92.8° with each other.

The cation is a mixed selenide-sulphide. The Se – S bond length is 2.189(3) Å, which is about 0.02 Å longer than what has been found for the Se – S

Table 4. Bond lengths (Å) and angles (°) in the adduct of *o*-nitrobenzeneselenenyl thiocyanate with thiourea. Standard deviations are given in parentheses.

Distances and angles involving selenium and neighbouring atoms.	
$Se-C_1 = 1.939(8)$	$\angle C_1-Se-S_1 = 98.88(28)$
$Se-S_1 = 2.1886(30)$	$\angle C_1-Se\cdots O_2 = 73.87(32)$
$Se\cdots O_2 = 2.505(8)$	$\angle S_1-Se\cdots O_2 = 172.27(17)$
Benzene ring	
$C_1-C_2 = 1.375(13)$	$\angle C_6-C_1-Se = 121.1(6)$
$C_2-C_3 = 1.377(13)$	$\angle C_2-C_1-Se = 121.3(6)$
$C_3-C_4 = 1.404(15)$	$\angle C_6-C_1-C_2 = 117.5(7)$
$C_4-C_5 = 1.408(15)$	$\angle C_1-C_2-N_3 = 118.4(7)$
$C_5-C_6 = 1.388(13)$	$\angle C_3-C_2-N_3 = 116.3(8)$
$C_1-C_6 = 1.408(13)$	$\angle C_1-C_2-C_3 = 125.2(8)$
$C_2-N_3 = 1.460(12)$	$\angle C_2-C_3-C_4 = 118.1(8)$
	$\angle C_3-C_4-C_5 = 117.3(8)$
	$\angle C_4-C_5-C_6 = 123.7(8)$
	$\angle C_1-C_6-C_5 = 118.1(8)$
Nitro group	
$N_3-O_1 = 1.208(12)$	$\angle C_2-N_3-O_1 = 118.9(7)$
$N_3-O_2 = 1.225(11)$	$\angle C_2-N_3-O_2 = 118.8(8)$
	$\angle O_1-N_3-O_2 = 122.2(8)$
	$\angle N_3-O_2-Se = 107.4(6)$
Thiourea group	
$S_1-C_7 = 1.799(11)$	$\angle Se-S_1-C_7 = 102.8(4)$
$C_7-N_1 = 1.320(16)$	$\angle S_1-C_7-N_1 = 111.5(7)$
$C_7-N_2 = 1.301(14)$	$\angle S_1-C_7-N_2 = 123.7(7)$
	$\angle N_1-C_7-N_2 = 124.8(9)$
Thiocyanate ion	
$S_2-C_8 = 1.588(13)$	$\angle S_2-C_8-N_4 = 174.1(10)$
$C_8-N_4 = 1.142(17)$	
Hydrogen bonding	
$N_2\cdots N_4 = 2.904(15)$	$\angle C_7-N_2\cdots N_4 = 95.4(7)$
$N_1\cdots N_4 = 2.947(16)$	$\angle C_7-N_1\cdots N_4 = 93.0(7)$
$N_1\cdots S_2' = 3.365(10)$	$\angle C_7-N_1\cdots S_2' = 117.0(6)$
	$\angle N_4\cdots N_1\cdots S_2' = 144.7(4)$

S_2' denotes a sulphur atom at $(x, 1-y, \frac{1}{2}-z)$, where x, y, z are the S_2 coordinates of Table 1.

bonds in selenopentathionates.[7,8] The Se–C bond is 1.939(8) Å, the same as found in the formamidinium diselenide ion [9] and in the triselenourea ion.[10] The angles C_1-Se-S_1 and $Se-S_1-C_7$ are 98.88(28)° and 102.8(4)°, respectively, and the dihedral angle C_1SeS_1/SeS_1C_7 is 95.7°. Thus, the valency angles of sulphur and selenium and the dihedral angle are in accordance with what has earlier been found in disulphides and diselenides.[9,11,12]

A loose five-membered ring is formed by the selenium atom, one oxygen atom and the nitrogen atom of the nitro group, and two carbon atoms of the benzene ring. The Se···O distance is 2.505(8) Å. This value is about midway

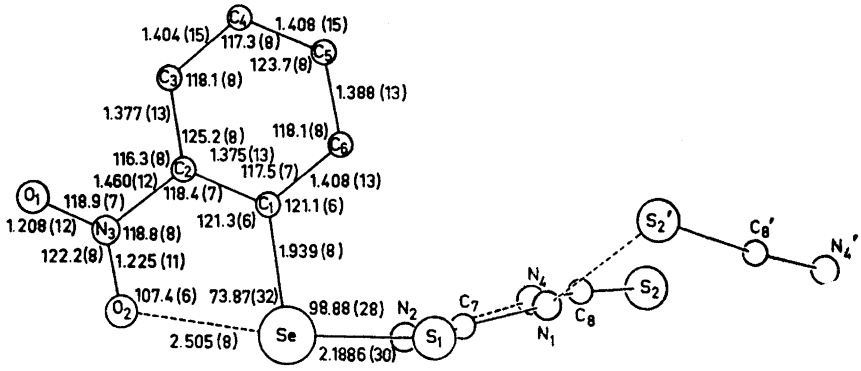

Fig. 1. The adduct of *o*-nitrobenzeneselenenyl thiocyanate with thiourea as seen normal to a plane through Se, S_1, and C_1.

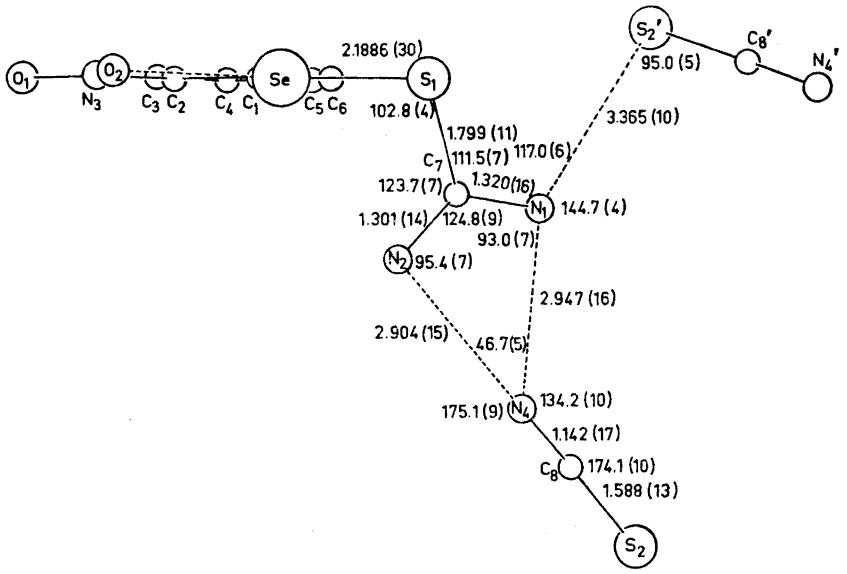

Fig. 2. The adduct of *o*-nitrobenzeneselenenyl thiocyanate with thiourea as seen along a plane through Se, S_1, and C, normal to the bond $Se-S_1$.

between the sum of the van der Waals radii and the sum of the covalent radii of selenium and oxygen, and indicates some Se···O bonding interaction. The S–Se···O angle is 172.37(17)° and is, from a geometrical point of view, favourable for the use of selenium *p* and *d* orbitals in the partial bonding.

The dimensions of the nitrophenyl group are, within the experimental error, the same as found in the crystals of nitrobenzene.[13]

Table 5. Distances from least squares planes. The equations of the planes were calculated with the selenium coordinates given nine times and the sulphur coordinates given three times the weight of the carbon, nitrogen, and oxygen coordinates, and refer to the monoclinic axes, with coordinates X, Y, and Z in Å.

Plane through Se, S_1, and the atoms of the benzene ring and the nitro group:
$0.67690\ X + 0.70393\ Y + 0.11354\ Z - 5.24634 = 0$

Se	−0.012 Å	N_3	0.004 Å	C_1	0.007 Å
S_1	0.011	O_1	−0.030	C_2	0.027
		O_2	0.067	C_3	0.004
				C_4	−0.033
				C_5	0.002
				C_6	0.027

Plane through the atoms of the thiourea group:
$-0.50147\ X + 0.60299\ Y - 0.54008\ Z - 0.08361 = 0$

S_1	0.000 Å	C_7	−0.002 Å	N_1	0.001 Å
				N_2	0.001

Plane through C_1, Se, S_1:
$0.68022\ X + 0.70533\ Y + 0.09757\ Z - 5.25331 = 0$

Plane through Se, S_1, C_7:
$-0.38966\ X + 0.46061\ Y - 0.73169\ Z + 0.51413 = 0$

The dihedral angle C_1SeS_1/SeS_1C_7 is 95.7°.

Thus, the formation of the five-membered ring system does not seem to influence the structure of the nitrophenyl group, nor does the Se···O interaction influence the structure of the −Se−S− group.

In the crystals of the *o*-nitrobenzenesulphenic acid methyl ester,[1] all atoms in the molecule, with the exception of the methyl group, are coplanar. The five-membered ring there is established in the same way as in the present structure. The O−S···O angle is 177°, the S−OCH_3 bond length is 1.65 Å, which is in the range given for single S−O bonds, and the nitro O···S distance is 2.44 Å. In the crystals of bis(*o*-nitrophenyl) disulphide,[2] there are two crystallographically independent five-membered ring systems, with nitro O···S distances of 2.636(8) and 2.588(7) Å. By taking the difference in the van der Waals radii of selenium and sulphur to be 0.15 Å,[14] the Se···O distance of 2.505(8) Å indicates a rather stronger Se···O interaction in the present structure than the S···O interactions in the structures mentioned above.

As mentioned earlier, the thiourea group is planar, the largest deviation of an atom from a least squares plane being 0.002 Å (*cf.* Table 5). The two C−N bonds of the group, 1.320(16) and 1.301(14) Å, are equal within the error. The S−C bond length is 1.799(11) Å, and the angles $S_1-C_7-N_1$, $S_1-C_7-N_2$, and $N_1-C_7-N_2$ are 111.5(7)°, 123.7(7)°, and 124.8(9)°, respectively.

The sulphur atom of the thiourea group in the present structure is covalently bonded to a selenium atom, and accordingly, the S−C bond length is expected to be longer than in the crystals of thiourea itself,[15] where the S−C bond length is 1.720(9) Å.

The dimensions of the thiocyanate ion, S–C = 1.588(13) Å, C–N = 1.142(17) Å, and the angle S–C–N = 174.1(10)°, are well in accordance with values recently published for other thiocyanates.[16,17]

HYDROGEN BONDING

The amino nitrogen atoms of the thiourea group may be assumed to have a trigonal-planar bonding system, *i.e.*, the hydrogen atoms lie in or close to the plane through the thiourea group. The nitrogen atom, N_1, of the thiourea group forms hydrogen bonds to the nitrogen atom, N_4, of the reference thiocyanate group, and to the sulphur atom, $S_2'(\bar{x},1-y,\frac{1}{2}-z)$, of another thiocyanate group. N_4 is 0.03 Å and S_2' is 0.25 Å out of the thiourea plane. The $N_1\cdots N_4$ distance is 2.947(16) Å, the $N_1\cdots S_2'$ distance is 3.365(10) Å, and the $C_7-N_1\cdots N_4$, $C_7-N_1\cdots S_2'$, and $N_4\cdots N_1\cdots S_2'$ angles are 93.0(7)°, 117.0(6)°, and 144.7(4)°, respectively. The nitrogen atom, N_2, also forms a hydrogen bond to N_4. The distance $N_2\cdots N_4$ is 2.904(15) Å and the angle $C_7-N_2\cdots N_4$ is 95.4(7)°.

Hydrogen bonding to selenium, N–H···Se, has been found in crystals of selenourea [18] and related compounds.[19–21] In the crystal structure reported here, such hydrogen bonding probably occurs between N_2 and Se. The selenium atom is 0.535 Å out of the thiourea plane. By assuming a trigonal-planar bonding system at N_2, there will be a close contact H···Se of about 2.6 Å. Such a position of the hydrogen atom is in accordance with a peak found in the difference map.

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