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Technetium–Phosphine Complexes. Diethylphenylphosphonite Complexes of Technetium(III) and Mixed Ligand Complexes of Technetium(I) with Carbonyls and Diethylphenylphosphonite, and Crystal and Molecular Structure of cis-Dicarbonyltetrakis(diethylphenylphosphonite)technetium(I) Perchlorate

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By reaction of trans-$[TcX_2(P(OEt)_2Ph)_4]ClO_4$ *(X = Cl, Br, I) with carbon monoxide at room pressure, the cis- and trans-*$[Tc(CO)_2(P(OEt)_2Ph)_4]ClO_4$ *isomers were obtained. To this purpose the complexes trans-*$[TcX_2(P(OEt)_2Ph)_4]Y$ *(X = Br, I and Y =* ClO_4^-, I^-*) were therefore synthesized. The formulation of all complexes is substantiated by elemental analysis, magnetic and conductivity measurements, i.r. and* ^1H *n.m.r. spectra and for the cis-isomer also by X-ray analysis. In the latter complex the technetium atom presents an octahedral coordination with some degree of distortion. The four Tc–P distances are 2.38(1), 2.41(1), 2.44(1), 2.44(1) Å, and the two Tc–CO distances are both 1.90(2) Å.*

Introduction

Mixed carbonyl phosphine complexes of technetium are not yet reported in literature. Our only reference marks are the rhenium complexes. The synthesis of rhenium complexes follows essentially two methods: the substitution of carbonyl with phosphine in the carbonyl complexes[1–6], or the substitution of phosphines with carbon monoxide in the phosphine complexes[7,8]. In the first case the reactions are often performed under drastic conditions of temperature and pressure, only in one case[6] the reaction is carried out photochemically in mild conditions. In the second case the operative conditions are normal. We adopted the second method in order to study also the reactivity of the $[TcX_2L_4]Y$ (X = Cl, Br, I; Y = Cl^-, I^-, ClO_4^-, BPh_4^- and L = diethylphenylphosphonite) complexes, in part already synthesized by us[9], to the substitution of phosphonite with carbon monoxide. Rhenium complexes having the same formulation to those here described are the $[Re(CO)_2(DFE)_2]X$ (DFE = 1,2-bis(diphenylphosphino)ethane, and X = Cl^-, I^-, $SnCl_3^-$ and ClO_4^-) derivatives, synthesized from carbonyl complexes.

To study the change of reactivity as a function of halogen the complexes $[TcX_2(P(OEt)_2Ph)_4]Y$ (X = Br, I and Y = ClO_4^-, I^-) were prepared and characterized. Crystal structure of the complex *cis*-$[Tc(CO)_2(P(OEt)_2Ph)_4]ClO_4$ was also carried out to confirm the structural data obtained from spectroscopy studies and to achieve informations about the coordination geometry of technetium atom.

Experimental

Materials

Solid samples of NH_4TcO_4 were prepared by concentration and filtration of ammonia solutions obtained from “Radiochemical Centre” Ltd., Amersham (England). The $(NH_4)_2TcCl_6$ was prepared by electrochemical reduction of NH_4TcO_4 in HCl 6N on a platinum electrode[10]. The $(NH_4)_2TcBr_6$ and the $(NH_4)_2TcI_6$ were prepared by refluxing NH_4TcO_4 in HBr or HI conc., respectively. The diethylphenylphosphonite was synthesized by the method of Rabinoviz and Pellon[11]. All the solvents used are purified and dried by standard methods.

Apparatus

Magnetic susceptibilities of dichloromethane solutions were measured by the Evans method[12] and corrected for diamagnetism of the ligands from tables given by Figgis and Lewis[13]. ^1H n.m.r. spectra were recorded on a Bruker HFX-10 instrument with TMS as internal standard. Conductivities of 10^{-3} molar solutions in nitroethane at 25° C have been measured with an 3116B model L.K.B. conductivity bridge. Infrared spectra of nujol samples and CH_2Cl_2 solutions with

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CsI windows were recorded on a Perkin–Elmer 621 instrument in the 4000–400 cm^{-1} range, and on a Beckman I.R.11 spectrophotometer in the far infrared region (400–50 cm^{-1}).

Analytical Methods

The elemental analyses for the radioactive technetium was carried out by a Intertechnique Liquid Scintillation spectrometer model SL20 with Insta-gel as scintillator. The efficienty for technetium was about 97%. The halides were analyzed by potentiometrical titration with standard $AgNO_3$ of oxidized solutions of the complexes with the Shöniger method. The perchlorate ion of carbonyl complexes was determined by conductometric titration in ethanol solutions with $AsPh_4Cl$ as titrant.

Syntheses of Complexes

All the preparative work was carried out under nitrogen and with the normal health-precautions[9] used for radioactive technetium 99.

Dichlorotetrakis(diethylphenylphosphonite) technetium(III) perchlorate

The complex was synthesized following our published method[9].

Dibromotetrakis(diethylphenylphosphonite) technetium(III) perchlorate

Diammonium hexabromotechnetate(IV) (0.5 g), diethylphenylphosphonite (6 ml) and anhydrous ethanol (40 ml) were placed in two necked roundbottomed flask equipped with condenser and inlet–outlet tube. The apparatus was fushed out with nitrogen and the reaction mixture was heated under reflux for two hours. During which time the solution became deep redviolet, but the starting solid was still present. By heating for a longer time the complex began to decompose. Then the solution was decanted and diethylphenylphosphonite (3 ml) and ethanol (20 ml) was again added to the residue. The mixture was refluxed for other two hours and the solid disappeared. A lithium perchlorate saturated solution was added to the joined together solutions to obtain a powder precipitate of $[TcBr_2(P(OEt)_2Ph)_4]ClO_4$ which was recrystallized from ethanol (yield 75%).

Diiodotetrakis(diethylphenylphosphonite) technetium(III) iodide and perchlorate

Diammonium hexaiodotechnetate(IV) (0.5 g), diethylphenylphosphonite (4 ml) and anhydrous ethanol (30 ml) were placed in two necked roundbottomed flask equipped with inlet–outlet tube. The starting mixture was stirred at room temperature for one hour. The solution became deep green but the starting solid did not completely disappear. The residual solid was filtered away, treated with diethylphenylphosphonite (2 ml) and ethanol (20 ml) and the mixture stirred for one hour. Addition of pentane to the joined solutions caused precipitation of a blue–green powder of $[TcI_2(P(OEt)_2Ph)_4]I$. The perchlorate salt was obtained by adding a concentrated ethanol solution of $LiClO_4$ to a concentrated ethanol solution of the iodine salt (yield 70%).

Trans- and cis-dicarbonyltetrakis(diethylphenylphosphonite)technetium(I) perchlorate

These two isomers were prepared treating the halides derivatives above described in ethanol solution and in the presence of the phosphonite (0.5*M*), with carbon monoxide at room pressure. We describe two cases since the complex with iodine anion requires different reaction conditions:

a) $[TcCl_2(P(OEt)_2Ph)_4]ClO_4$, $[TcBr_2(P(OEt)_2Ph)_4]ClO_4$ or $[TcI_2(P(OEt)_2Ph)_4]ClO_4$ (0.5 g) reacts with CO fluxed on, if it is stirred in ethanol (20 ml) and diethylphenylphosphonite (2 ml) at 50° C for 30 or 8 hours for the chloro- or the bromo-derivative respectively. For the iodo-derivative the reaction was carried out at room temperature for 5 hours. The reaction was stopped when the red or the red–violet color of the starting solutions disappears to give a clear solution. By cooling, white crystals of *trans*-$[Tc(CO)_2(P(OEt)_2Ph)_4]ClO_4$ precipitated (yield 45%).

The mother liquor, treated with pentane, separated a white powder of crude *cis*-$[Tc(CO)_2(P(OEt)_2Ph_4]ClO_4$ impure with about the 2% of *trans*-isomer. This product was purified by dissolving it in a small amount of boiling ethanol and by adding pentane to the cooled and filtered solution. The impurities of *trans*-isomer so reduced to about 0.5%. The yield of crude product was about 36%, whilst the yield of final purified product was of 10%.

b) $[TcI_2(P(OEt)_2Ph)_4]I$ (0.5 g) reacts with CO fluxed on, stirred in ethanol (20 ml) and diethylphenylphosphonite (2 ml) at room temperature for 5 hours. The final clear solution, treated with pentane gave a white powder of mixed isomers. The separation of the two isomers was not possible, and the relative yield in the two isomers was determined by ^1H n.m.r. measurements (yield: *cis*-, 30%; *trans*-, 70%).

Crystal Data

The *cis*-$[Tc(CO)_2(P(OEt)_2Ph)_4]ClO_4$ complex was recrystallized from ethanol.

Triclinic, space group $P\bar{1}$; $a = 17.708(15)$, $b = 13.977(12)$, $c = 10.185(10)$ Å; $\alpha = 93.22(8)$, $\beta = 90.48(9)$, $\gamma = 96.13(11)°$, $U = 2502.2$ Å^3; m.w. = 1047.3; $D_m = 1.37$, $D_c(Z = 2) = 1.39$ g cm^{-3}, λ(Mo-Kα) = 0.7107 Å; μ(Mo-Kα) = 5.26 cm^{-1}. 3570 Independent observed reflections, four-circle diffractometer.

Accurate unit cell and orientation parameters were obtained by least-squares fit[14] for 27 values of Θ,χ,Φ accurately measured.

X-ray Intensity Measurements

Data were collected at room temperature for a crystal protected from the air (against the possible oxidation of Tc(I)) and mounted on the Siemens automatic single-crystal diffractometer, with the c axis nearly coincident with the Φ-axis of the goniostat. Intensities were measured with a Na(Tl)I scintillation counter using Mo-Kα radiation at a take-off angle of 4.5°. The Θ–2Θ scan technique was emploied, using a "five-value" measuring procedure[15]. A general reflection was remeasured every twenty reflections as a monitor of crystal stability and to normalize the intensities to a common basis. The monitor reflection was essentially constant with time. In the reciprocal space corresponding to $2\Theta_{max}$ of 44° there were 3570 independent reflections with $I > 2\sigma(I)$. Lorentz and polarisation corrections were applied but not absorption corrections. The data were put on an approximately absolute scale with a Wilson plot.

The atomic scattering factors were taken from ref. 16 and values of $\Delta f'$ and $\Delta f''$ used to correct for anomalous dispersion in the scattering factor of technetium, phosphorus and chlorine were taken from Cromer's tables[17].

Solution and Refinement of the Structure

The structure was solved using conventional heavy-atom technique in space group $P\bar{1}$. The position of the technetium, chlorine and phosphorus atoms were determined from a three-dimensional unsharpened Patterson function and confirmed by least-squares refinement to R 0.31. A difference Fourier synthesis phased on these atoms revealed the position for all 62 non-hydrogen atoms. Four cycles of block-diagonal least-squares refinement of the positional parameters and their isotropic temperature factors lowered the index to $R = 0.12$. From this point all atoms were allowed to vibrate anisotropically except for the perchlorate ion since might be disordered.

Two cycles of block-diagonal least-squares refinement, using unit weight for all reflections, decreased the R index to 0.092. At this point the weighting scheme was $\omega^{-1} = \Sigma_i a_i |F_o|^i$. The number and value of the a_i parameters were re-evaluated several time during the refinement so as to give approximately constant averages of $\omega(K|F_o|-|F_c|)^2$ for equally populated ranges of $|F_o|$. Four final cycles of block-diagonal least-squares refinement converged to a residual index $R = 0.086$. A difference-Fourier map computed at this stage showed some peaks up to a maximum of 0.58 eÅ^{-3} in the vicinity of the technetium atom position, while the highest peaks (0.84–0.88 eÅ^{-3}) are in the vicinity of the perchlorate ion.

Positional and thermal parameters (only for perchlorate ion) are given in Table IV; anisotropic temperature factors for the cation are given in Table V. The atom numbering system used and the configuration of the molecule are illustrated in Figure 1, while bond lengths and bond angles with their estimated standard deviations are listed in Table VI and VII respectively.

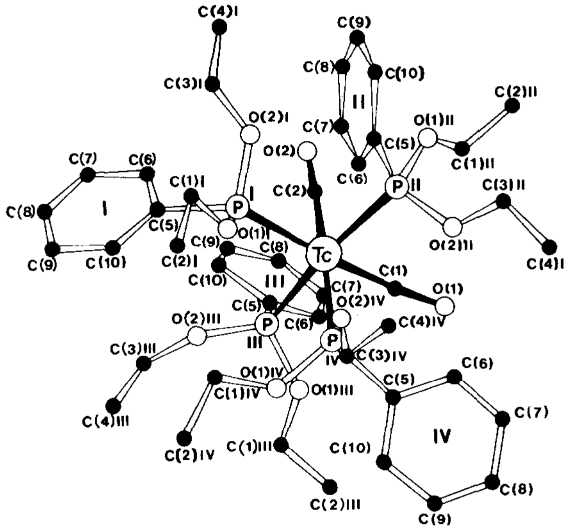

Figure 1. Three-dimensional view of the cation *cis*-[Tc(CO)$_2$(P(OEt)$_2$Ph)$_4$]$^+$.

Equations of the principal planes and the deviations of atoms therefrom are given in Table VIII; Table IX shows interligand (phosphine) contacts within the same molecule; the observed and calculated structure factors are given in Table X.

The solution and refinement of the structure were carried out by use of the Crystal Structure Calculations System 'X-Ray '70[18]. Calculations were done on the Consorzio Interuniversitario dell'Italia Nord-Orientale, Casalecchio (Bologna), CDC 6600 computer.

Result and Discussion

Haloderivatives

By reaction of $(NH_4)_2TcX_6$ (X = Cl, Br, and I) with an excess of diethylphenylphosphonite in ethanol, the complexes of technetium(III) $[TcX_2(P(OEt)_2Ph)_4]Y$ (X = Cl, Br, I, and Y = Cl$^-$, I$^-$, BPh$_4^-$, ClO$_4^-$) were formed. The chloro complexes are reported elsewhere[9]; the bromo derivative was obtained as solid only with perchlorate anion. These complexes are air stable in the solid state, but in polar solvent solutions their stability might be hold by means of free ligand (about 0.5M). The conductivity measurements (Table I) of 10^{-3}M nitromethane solutions at 25°C indicate that these compounds are 1:1 electrolytes. The magnetic moment data are in good agreement with two unpaired electrons in the technetium(III) octahedral complexes. A lower value than the expected 3.2 B.M. can be explained with a deviation from the octahedral

TABLE I. General Properties of Complexes.[a]

Compound	Colour	X%		Tc%		Λ_{eq}	μ_{eff}
		Calc.	Found	Calc.	Found		
$[TcCl_2L_4]ClO_4$	red	10.02	9.82	9.32	9.35	75.9	2.6
$[TcBr_2L_4]ClO_4$	red–violet	13.89[b]	13.34	8.60	8.42	70.4	2.5
$[TcI_2L_4]I$	green	29.93	29.15	7.78	7.69	68.5	2.3
$[TcI_2L_4]ClO_4$	green	20.39[b]	20.02	7.95	7.80	71.6	2.4
trans-$[Tc(CO)_2L_4]ClO_4$	white	9.50[c]	9.42	9.46	9.41	93.8	
cis-$[Tc(CO)_2L_4]ClO_4$	white	9.50[c]	9.48	9.46	9.39	80.2	

[a] L = $P(OEt)_2Ph$. [b] Excluding perchlorate anion. [c] X = ClO_4^- %.

geometry, expecially for the iodine derivatives. Far i.r. spectra data confirm the presence of two halogens in *trans*-position (Table II).

Carbonyl Complexes

Ethanol solutions of the dihalogenotetrakis(diethylphenylphosphonite)technetium(III) complexes $[TcX_2L_4]ClO_4$ (X = Cl, Br, I and L = $P(OEt)_2Ph$) take up CO at 1 atm with the disappearence of deep colours to give clear solutions of the two isomers *cis*- and *trans*-$[Tc(CO)_2L_4]ClO_4$. The starting solutions are 0.5*M* in the diethylphenylphosphonite because of the decomposition of halogenocomplexes. The CO substitution reactivity of the halogenoderivatives increases in the series $Cl<Br<I$. The relative yield in the two isomers is *ca.* 40% for *cis*- and 60% for *trans*- and seems do not drastically change with the halogeno complexes.

The reaction rate and the yield in the two isomers change with the ligand free concentration and precisely, increasing the concentration, the reaction rate and the yield in the *cis*-isomer decrease.

These qualitative informations will be substantiated by further studies and will be the subject of another comunication. The two dicarbonyl complexes are air stable in the solid state and in solution of organic polar solvents such as acetone, 1,2-dichloroethane, dichloromethane, nitromethane et so on. Their are diamagnetic and this is consistent with a d^6 spin-paired configuration in octahedral environment and 1:1 electrolytes in nitromethane solutions. The infrared and ^1H n.m.r. spectra are in agreement with the formulation based on elemental analysis (Table I) and indicate the presence of the phosphine ligands, ClO_4^- anion and two terminal carbonyl groups.

TABLE II. I.r. Spectral Data of the Complexes.

Compound	ν(Tc–X)	ν(C–O)	
	Solid	Solid	CH_2Cl_2 Solution
$[TcCl_2L_4]ClO_4$	346		
$[TcBr_2L_4]ClO_4$	275		
$[TcI_2L_4]I$	237–222		
$[TcI_2L_4]ClO_4$	239–223		
trans-$[Tc(CO)_2L_4]ClO_4$		2028(w)	
		1937(vs)	1946(s)
cis-$[Tc(CO)_2L_4]ClO_4$		1933(s)	2009(s)
		1939(s)	1954(s)

Infrared and ^1H n.m.r. Spectra

The structure of $[TcCl_2L_4]$ complex has been shown by single crystal X-ray analysis[9] to be lightly distorted octahedral with the two chlorine in *trans*-position, also the far i.r. measurements are in agreement with X-ray result. By analogy one can assume that the bromo and iodo derivatives has a *trans*-configuration. The i.r. spectra in the region of stretching (Tc–X) show only a band for $[TcBr_2L_4]ClO_4$ and two near bands for the iodo complexes (Table II). We can assume also for the $[TcI_2L_4]Y$ (Y = I^- or ClO_4^-) complexes a *trans*-configuration but in more distorted octahedron.

The structure of carbonyl complexes are substantiated by i.r., ^1H n.m.r. and for the *cis*-isomer by X-ray analysis. The $[Tc(CO)_2L_4]ClO_4$ complex, obtained by cooling the clear final solution, shows two bands in the i.r. spectra but the first one should not be fundamental and the explanation could be the same given by us[19] about cobalt carbonyl complexes. So, we can assume the *trans*-configuration for this isomer. Further support is given by the ^1H n.m.r. spectra (Table III).

TABLE III. ^1H n.m.r. Spectral Data for the Two Carbonyl Isomers[a].

Compound	Phenyl Protons	Methylene Protons	Methyl Protons
trans-$[Tc(CO)_2L_4]ClO_4$	7.4 (m)	3.6 (q)	1.22 (t)
cis-$[Tc(CO)_2L_4]ClO_4$	7.5 (m)	3.7 (m)	1.24 (t) 1.26 (t)

[a] The spectra were measured in $CDCl_3$ unless otherwise stated. m = multiplet, q = quartet, t = triplet.

In fact this complex exhibits a 1:2:1 triplet (J = 7 Hz), centered at 1.22 δ, due to the resonance of methyl protons of diethylphenylphosphonite split by methylene protons, and suggesting that the four phosphine ligands are magnetically and hence stereochemically equivalent. This indicates that the four phosphine ligands are in *trans* position, and that the "virtual" coupling between the four phosphorus nuclei is so small that it cannot be observed. The $[Tc(CO)_2L_4]ClO_4$ complex, instead, obtained by adding pentane, shows in the i.r. spectra two strong bands with the same intensity in the carbonyl region (Table II). From the Rëickziegel and Bigorgne[20] equation:

$$I_{antisym.}/I_{sym.} = 1-\cos\delta/(1+\cos\delta)$$

the CO–Tc–CO bond angle (δ) is found to be about 90°; these results suggest a structure *cis-* for this complex.

Also the ^1H n.m.r. spectra of this complex support this structure. The resonance lines of phosphonite methyl protons appear as two 1:2:1 triplet (J = 7 Hz) at 1.24 and 1.26 δ with the same intensity. This is in accordance with the phosphine ligands two to two equivalent.

X-ray Structural Analysis

The main result of the structural analysis is to have demonstrated unequivocally that the nature of the isomerism is of the type *cis–trans,* thus allowing the use of much faster methods (like i.r. and n.m.r.) for predicting and extending the geometrical structure of technetium complexes.

The structure consists of discrete monomeric octahedral cations of *cis*-$[Tc(CO)_2(P(OEt)_2Ph)_4]^+$, the least Tc–Tc separation being 9.90 Å, and perchlorate anions. The technetium atom presents an octahedral coordination with some degree of distortion (Figure 1, Table VII, VIII); the angles between the planes (1)–(2)–(3) are not very different from 90° as required by ideal octahedron. The PII–Tc–PIII angle is the *trans*-angle which differs more significantly from the appropriate rectilinear value; moreover, since the plane (3) is the only plane with significant deviations, practically the major part of the distortion can be ascribed

TABLE IV. Final Fractional Co-ordinates (× 10^4) with Standard Deviations in Parentheses and Isotropic Temperature Factor (Å^2 × 10^4) for Perchlorate Ion.

Atom	x/a	y/b	z/c	U
Tc	2197(1)	2235(1)	–1271(2)	
PI*	3351(3)	3355(4)	–980(5)	
PII	1423(3)	3538(3)	–861(5)	
PIII	2781(3)	783(4)	–1551(5)	
PIV	2173(3)	2310(4)	–3657(5)	
C(1)	1282(12)	1384(14)	–1348(19)	
O(1)	712(8)	890(10)	–1381(15)	
C(2)	2276(12)	2152(12)	583(21)	
O(2)	2323(8)	2087(11)	1740(13)	
O(1)I	3540(7)	3979(9)	–2189(13)	
C(1)I	4072(13)	4861(15)	–2220(22)	
C(2)I	4283(16)	5018(18)	–3651(27)	
O(2)I	3194(7)	4036(8)	308(13)	
C(3)I	3726(12)	4831(15)	890(22)	
C(4)I	3583(17)	4874(19)	2368(24)	
C(5)I	4266(11)	2955(14)	–520(20)	
C(6)I	4351(13)	2653(15)	780(22)	
C(7)I	5043(14)	2350(17)	1148(23)	
C(8)I	5628(14)	2290(18)	254(25)	
C(9)I	5539(12)	2583(16)	–1092(25)	
C(10)I	4848(11)	2902(15)	–1462(21)	
O(1)II	995(8)	3550(9)	519(14)	
C(1)II	514(16)	2717(16)	955(27)	
C(2)II	–103(17)	3044(24)	1786(34)	
O(2)II	758(8)	3480(10)	–1965(13)	
C(3)II	171(13)	4145(16)	–1946(24)	
C(4)II	–347(19)	3886(31)	–3009(34)	
C(5)II	1787(11)	4811(14)	–785(22)	
C(6)II	2045(13)	5216(14)	–1961(25)	
C(7)II	2322(13)	6173(15)	–1918(26)	
C(8)II	2336(13)	6758(15)	–766(31)	
C(9)II	2086(15)	6320(17)	423(31)	
C(10)II	1792(12)	5351(13)	426(24)	
O(1)III	2357(8)	135(9)	–2753(13)	
C(1)III	2569(16)	–787(16)	–3236(25)	
C(2)III	1909(15)	–1325(20)	–3916(30)	
O(2)III	3676(8)	898(8)	–1897(15)	
C(3)III	4234(15)	159(18)	–1832(35)	
C(4)III	4697(16)	208(21)	–2999(35)	
C(5)III	2687(12)	35(13)	–185(19)	
C(6)III	2005(14)	–551(15)	–61(23)	
C(7)III	1869(16)	–1073(16)	1045(28)	
C(8)III	2426(16)	–1064(17)	2074(24)	
C(9)III	3108(16)	–493(17)	1899(25)	
C(10)III	3252(16)	115(15)	826(24)	
O(1)IV	2844(9)	1927(11)	–4513(15)	
C(1)IV	3623(13)	2265(19)	–4478(24)	
C(2)IV	3967(15)	1765(25)	–5598(28)	
O(2)IV	2153(8)	3391(9)	–4094(13)	
C(3)IV	2117(17)	3615(19)	–5468(22)	
C(4)IV	1751(21)	4501(28)	–5588(30)	
C(5)IV	1389(12)	1643(16)	–4605(21)	
C(6)IV	668(13)	1855(17)	–4300(24)	
C(7)IV	38(16)	1439(21)	–5085(28)	
C(8)IV	164(16)	816(22)	–6155(28)	
C(9)IV	886(16)	618(19)	–6469(24)	
C(10)IV	1515(14)	1011(17)	–5697(21)	
Cl	3709(3)	–2591(4)	4854(6)	739
O(3)	4347(10)	–3039(13)	4368(18)	1089
O(4)	3732(13)	–1679(16)	4350(22)	1400
O(5)	3064(16)	–3082(20)	4524(27)	1819
O(6)	3717(12)	–2464(15)	6214(20)	1269

* In all the work roman numerals refer to the four phosphine ligands.

TABLE V. Anisotropic Temperature Factors ($Å^2 \times 10^4$) for the Cation *cis*-$[Tc(CO)_2(P(OEt)_2Ph)_4]^+$.

	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Tc	420	323	375	97	12	−2
PI	477	467	454	104	39	25
PII	504	348	623	92	71	−5
PIII	571	419	570	166	−17	−40
PIV	581	518	449	170	33	−7
C(1)	890	476	569	296	151	60
O(1)	697	660	940	−164	24	66
C(2)	863	197	736	44	176	−14
O(2)	1023	905	406	104	135	140
O(1)I	606	480	635	92	95	86
C(1)I	736	573	835	11	256	170
C(2)I	1247	748	1015	83	112	338
O(2)I	621	417	749	102	32	−130
C(3)I	624	605	832	−35	−24	−302
C(4)I	1290	1039	671	9	148	−297
C(5)I	454	620	665	150	−135	−42
C(6)I	875	507	805	186	−302	−10
C(7)I	797	809	832	150	−86	155
C(8)I	846	874	889	89	−177	85
C(9)I	533	650	1169	212	−12	−51
C(10)I	513	700	673	93	27	126
O(1)II	876	473	724	191	192	−31
C(1)II	1241	542	1233	174	714	169
C(2)II	1061	1465	1712	84	873	641
O(2)II	703	773	597	342	13	−67
C(3)II	815	755	1015	532	−191	−121
C(4)II	1211	2568	1367	1216	−324	−337
C(5)II	436	514	910	186	−1	145
C(6)II	744	339	1266	220	317	143
C(7)II	815	395	1350	152	425	286
C(8)II	660	364	1978	111	89	147
C(9)II	965	544	1658	256	70	−182
C(10)II	688	254	1148	147	−25	−117
O(1)III	809	510	680	214	−149	−164
C(1)III	1348	537	1044	455	−345	−572
C(2)III	819	1026	1370	181	−327	−569
O(2)III	726	317	1090	145	110	−31
C(3)III	796	750	2328	526	183	160
C(4)III	907	1083	1968	423	585	−222
C(5)III	841	441	552	380	97	95
C(6)III	1114	559	776	230	122	241
C(7)III	1225	457	1243	63	141	−71
C(8)III	1405	740	721	319	47	−52
C(9)III	1316	609	970	400	−142	43
C(10)III	1335	470	895	409	−273	−78
O(1)IV	865	925	677	197	119	−65
C(1)IV	621	1202	774	72	82	−130
C(2)IV	724	2003	978	299	277	−621
O(2)IV	666	682	588	79	4	24
C(3)IV	1482	1042	522	352	91	327
C(4)IV	1774	2163	937	1174	162	728
C(5)IV	606	803	653	128	−112	−199
C(6)IV	680	865	878	165	−192	−152
C(7)IV	1005	1184	1033	−26	−232	−316
C(8)IV	1018	1399	966	147	−373	−171
C(9)IV	1168	1025	699	−5	−117	−214
C(10)IV	1086	823	473	26	−13	38

Cœfficients in the temperature factor expression: $\exp[-2\pi^2 (U_{11}h^2a^{*2} + U_{22}K^2b^{*2} + \cdots\cdots + 2U_{12}h\,K\,a^*b^* + \cdots)]$.

TABLE VI. Bond Lengths (Å) for the Cation *cis*-$[Tc(CO)_2(P(OEt)_2Ph)_4]^+$, with Standard Deviations in Parentheses.

(a) Distances in the inner coordination sphere of the technetium atom:

Tc–PI	2.44(1)	Tc–PIII	2.38(1)	Tc–C(1)	1.90(2)	C(1)–O(1)	1.16(2)
Tc–PII	2.41(1)	Tc–PIV	2.44(1)	Tc–C(2)	1.90(2)	C(2)–O(2)	1.19(3)

(b) Distances in the diethylphenylphosphonite ligands:

	Ligand I	Ligand II	Ligand III	Ligand IV
P–O(1)	1.57(2)	1.60(2)	1.61(1)	1.60(2)
P–O(2)	1.62(1)	1.61(2)	1.62(2)	1.60(1)
P–C(5)	1.84(2)	1.82(2)	1.79(2)	1.82(2)
O(1)–C(1)	1.47(2)	1.46(2)	1.44(3)	1.41(3)
O(2)–C(3)	1.47(2)	1.47(3)	1.51(3)	1.45(3)
C(1)–C(2)	1.53(4)	1.48(4)	1.47(4)	1.47(4)
C(3)–C(4)	1.53(3)	1.42(4)	1.45(5)	1.47(5)

(c) Phenyl rings:

	Ligand I	Ligand II	Ligand III	Ligand IV
C(5)–C(6)	1.42(3)	1.41(3)	1.40(3)	1.38(3)
C(6)–C(7)	1.39(4)	1.37(3)	1.39(4)	1.42(4)
C(7)–C(8)	1.39(4)	1.39(4)	1.43(4)	1.39(4)
C(8)–C(9)	1.46(4)	1.44(4)	1.39(4)	1.37(4)
C(9)–C(10)	1.40(3)	1.40(3)	1.43(4)	1.40(3)
C(10)–C(5)	1.42(3)	1.41(3)	1.42(3)	1.41(3)

TABLE VII. Bond Angles (deg.) for the Cation *cis*-$[Tc(CO)_2(P(OEt_2)Ph)_4]^+$, with Standard Deviations in Parentheses.

(a) Angles about technetium atom:

PI–Tc–PII	90.6(2)	PII–Tc–PIII	170.4(2)	PIII–Tc–C(1)	83.5(7)
PI–Tc–PIII	98.1(2)	PII–Tc–PIV	95.1(2)	PIII–Tc–C(2)	89.0(6)
PI–Tc–PIV	93.9(2)	PII–Tc–C(1)	87.5(7)	PIV–Tc–C(1)	90.6(6)
PI–Tc–C(1)	175.3(6)	PII–Tc–C(2)	87.8(6)	PIV–Tc–C(2)	176.5(7)
PI–Tc–C(2)	84.2(6)	PIII–Tc–PIV	88.4(2)	C(1)–Tc–C(2)	91.4(8)

(b) Angles about phosphorus atoms:

	Ligand I	Ligand II	Ligand III	Ligand IV
Tc–P–O(1)	113.9(5)	115.7(6)	108.3(6)	120.0(6)
Tc–P–O(2)	104.6(5)	109.8(6)	116.3(5)	111.9(5)
Tc–P–C(5)	122.1(7)	124.2(7)	114.5(7)	119.2(7)
O(1)–P–O(2)	110.6(7)	105.2(8)	106.3(8)	103.3(8)
O(1)–P–C(5)	104.1(8)	97.0(9)	104.6(8)	96.8(9)
O(2)–P–C(5)	100.7(8)	102.6(9)	106.0(9)	103.0(9)

(c) Other angles in diethylphenylphosphonite ligands:

	Ligand I	Ligand II	Ligand III	Ligand IV
P–O(1)–C(1)	127.2(1.2)	122.6(1.3)	124.2(1.4)	128.6(1.4)
P–O(2)–C(3)	125.6(1.2)	122.9(1.3)	128.4(1.3)	122.0(1.3)
P–C(5)–C(6)	117.8(1.5)	117.9(1.5)	118.0(1.6)	117.0(1.6)
P–C(5)–C(10)	120.4(1.5)	119.3(1.6)	120.9(1.5)	121.8(1.7)
O(1)–C(1)–C(2)	108.4(1.7)	109.8(2.0)	108.4(2.2)	106.4(1.9)
O(2)–C(3)–C(4)	106.5(1.7)	109.5(2.3)	108.2(2.4)	109.8(2.1)

(d) Phenyl rings:

	Ligand I	Ligand II	Ligand III	Ligand IV
C(6)–C(5)–C(10)	121.8(1.9)	122.8(1.8)	120.7(1.9)	120.8(2.0)
C(5)–C(6)–C(7)	118.5(2.0)	118.6(2.1)	120.6(2.2)	120.0(2.2)
C(6)–C(7)–C(8)	121.3(2.2)	122.2(2.4)	121.8(2.3)	118.9(2.5)
C(7)–C(8)–C(9)	120.3(2.2)	118.0(2.0)	116.0(2.3)	120.9(2.5)
C(8)–C(9)–C(10)	118.6(2.1)	121.9(2.4)	124.2(2.5)	121.1(2.3)
C(5)–C(10)–C(9)	119.4(2.0)	116.6(2.2)	116.4(2.1)	118.2(2.2)

TABLE VIII. Learst-squares Planes with the Deviations (Å) of Relevant Atoms in Square Brackets. The equation of a plane in direct space is given by $Px + Qy + Rz = S$, where x, y and z are in fractional unit cell coordinates.

Plane (1):	P	Q	R	S
Tc, PI, PII, PIII, C(1)	0.414	–2.333	10.120	–1.652
[Tc –0.06, PI 0.02, PII 0.01, PIII 0.02, C(1) 0.02, O(1) 0.08]				
Plane (2):				
Tc, PI, C(1), C(2), PIV	–11.212	11.603	0.672	0.035
[Tc 0.01, PI 0.03, C(1) 0.04, C(2) –0.05, PIV –0.04, O(1) 0.11, O(2) –0.10]				
Plane (3):				
Tc, PII, C(2), PIII, PIV	14.239	7.042	–0.007	4.643
[Tc 0.06, PII –0.12, C(2) 0.11, PIII –0.13, PIV 0.08, O(2) 0.13]				
Plane (4):				
C(5)I–C(10)I	4.596	12.428	2.355	5.497
[C(5)I 0.01, C(6)I –0.02, C(7)I 0.01, C(8)I 0.0, C(9)I 0.01, C(10)I 0.0, PI –0.02]				

TABLE VIII. (Cont.)

	P	Q	R	S
Plane (5):				
C(5)II–C(10)II	16.816	–4.848	2.028	0.513
[C(5)II 0.0, C(6)II 0.0, C(7)II 0.01, C(8)II –0.02, C(9)II 0.02, C(10)II –0.01, PII –0.01]				
Plane (6):				
C(5)III–C(10)III	–8.587	10.951	4.415	–2.338
[C(5)III –0.01, C(6)III –0.01, C(7)III 0.02, C(8)III 0.0, C(9)III –0.03, C(10)III 0.03, PIII 0.12]				
Plane (7):				
C(5)IV–C(10)IV	0.559	11.247	–6.394	4.870
[C(5)IV 0.0, C(6)IV 0.0, C(7)IV 0.0, C(8)IV –0.01, C(9)IV 0.01, C(10)IV –0.01, PIV 0.19]				

Angles (deg.) between the mean planes:

(1)–(2)	87.8	(2)–(4)	55.0	(3)–(7)	61.0
(1)–(3)	87.7	(2)–(5)	39.6	(4)–(5)	85.7
(1)–(4)	82.2	(2)–(6)	22.9	(4)–(6)	46.5
(1)–(5)	75.3	(2)–(7)	57.2	(4)–(7)	53.8
(1)–(6)	70.6	(3)–(4)	39.9	(5)–(6)	56.6
(1)–(7)	45.0	(3)–(5)	51.3	(5)–(7)	73.6
(2)–(3)	87.1	(3)–(6)	86.2	(6)–(7)	72.1

TABLE IX. Interligand (Phosphine) Contacts within the Same Molecule.

I–II	
PI–PII	3.45(1)
O1I–C6II	3.32(3)
O_2I–PII	3.33(1)
O2I–C5II	3.05(2)
O2I–C10II	3.24(3)

I–III	
C5I–O2III	3.20(2)
C10I–O2III	3.31(2)

I–IV	
O1I–PIV	3.45(1)
O1I–C1IV	3.26(3)
O1I–O2IV	3.13(2)

II–IV	
O2II–O2IV	3.31(2)
O2II–C6IV	3.19(3)
C6II–O2IV	3.28(3)

III–IV	
PIII–PIV	3.36(1)
PIII–O1IV	3.49(2)
O1III–PIV	3.27(1)
O1III–O1IV	3.21(2)
O1III–C5IV	3.48(3)
O2III–O1IV	3.49(2)
O2III–C1IV	3.34(3)

to the displacement of PI and PIII atoms away from PI atom, but remaining still contained in plane (1). At first this distortion appears to result only from non-bonded repulsions between the bulky ligands as suggested also by the average value of the P–Tc–P angles (93.2°), while the six P–Tc–C angles have a mean value of 87.1°. Nevertheless, an examination of Table IX suggests that the larger number of contacts are between ligand III and IV and the angle PIII–Tc–PIV is the only P–Tc–P angle less than 90°. The distortion in the cation *cis*-$[Tc(CO)_2(P(OEt)_2Ph)_4]^+$ is less pronounced than that of the *trans*-$[TcCl_2(P(OEt)_2Ph)_4]$ compound[9], and this difference may be due to electronic effects; in fact the Tc(II) compound has a d^5 configuration with one unpaired electron, while the compound of Tc(I) has a d^6 spin-paired configuration. Altough the technetium atom is formally Tc(I), two Tc–P (Tc–PII and Tc–PIII) distances parallel those of the just mentioned compound of Tc(II), while the remaining Tc–P bonds (*trans*- to carbonyl groups) are little longer.

The difference indicates a *trans*-influence lightly greater for carbonyl group in respect to phosphine ligand, as is normal for ligands with π-acceptor properties (carbon monoxide) that have in *trans*-position a ligand like diethylphenylphosphonite capable of significant π-interaction with transition metals[21]. No Tc–P distances are known in literature, so it is difficult to discuss about and to have an indication of π back-donation and *trans*-influence. We wish to return on this subject when other technetium–phosphine structures are be completed.

The average P–C (1.82 Å), P–O (1.60 Å) and C–O (1.47 Å) distances in the diethylphenylphosphonite ligands are in accordance with the same distances found for $[H_2Fe(P(OEt)_2Ph)_4]$[22], $[TcCl_2(P(OEt)_2Ph)_4]$[9] and $[Ni(CN)_2(P(OEt)_2Ph)_3]$[23]; the same is true for the angles; the M–P–C (M = Fe, Tc, Ni) angles are always larger than regular tetrahedral values while a decrease is evident in the tetrahedral C–P–O or O–P–O angles about phosphorus atoms. The C–C distances in the ethoxy groups average 1.48 Å. A similar situation is observed in the iron (1.46 Å), nickel (1.44 Å) and technetium (1.48 Å) compounds before mentioned.

We have no intention to analyse in detail this situation because it is beyond our aims, but slight disordering of the ethoxy groups was invoked by the other authors.

TABLE X. Observed and calculated structure factors. The columns are, h, $10F_o$ and $10F_c$ in electrons. Reflexions marked by asterisks are unobserved and were not inclued in the refinement.

H,0,0		
2	2001	-2047
4	425	420
5	1644	1492
6	232	-318
7	1172	-1156
8	212*	155
9	713	781
10	202*	15
11	327	-305
12	317	-336
13	269	270
14	632	662
15	199*	-72
16	295	-245
17	199*	-112
18	176	211
H,1,0		
-18	180*	14
-17	208	-277
-16	185*	53
-15	578	589
-14	168*	49
-13	300	-262
-12	621	-659
-11	164	169
-10	686	747
-9	311	-327
-8	460	-464
-7	148*	25
-6	187	-193
-5	140*	42
-4	701	711
-3	386*	-428
-2	746*	820
1	1888*	-1779
2	1655*	-1609
3	391	580
4	774	732
5	863	-812
6	948	-905
7	284	259
8	979	934
9	138	-102
10	550	-628
11	157*	44
12	355	377
13	315	356
14	176*	133
15	461	-512
H,2,0		
-18	189*	-198
-17	180*	-8
-16	176*	103
-15	199	264
-14	330	-347
-13	579	-602
-12	334	339
-11	799	797
-10	162*	-47
-9	638	-691
-8	152*	-50
-7	520	529
-6	600	529
-5	238	183
-4	447	-421
-3	124*	8
-2	250	350
-1	241*	404
0	1377*	-1391
1	234	243
2	124*	22
3	658	672
4	462	-301
5	1421	-1448
6	262	253
7	1170	1198
8	319	-405
9	728	-729
10	161*	37
11	285	276
12	434	446
13	169*	-68
14	514	-559
15	178*	58
H,3,0		
-18	184*	-38
-17	170	154
-16	181*	117
-15	198*	-106
-14	180*	-77
-13	172*	55
-12	788	756
-11	275	-296
-10	981	-1032
-9	603	640
-8	483	513
-7	291	294
-6	817	-772
-5	2047	-1951
-4	1260	1197
-3	2305	2112
-2	713	-693
-1	444	-409
0	215	284
1	292	186
2	1248	1178
3	1224	-1250
4	948	-993
5	138	80
6	821	808
7	154*	179
8	740	-744
9	161*	-115
10	337	408
11	170	154
12	166	-117
13	175	-189
14	170*	-55
15	236	264
H,4,0		
-18	171	167
-17	151	113
-16	194*	-6
-15	338	-327
-14	175*	-42
-13	305	302
-12	174*	-31
-11	406	-406
-10	252	-269
-9	377	363
-8	151	191
-7	554	-477
-6	1035	-1043
-5	706	674
-4	956	1015
-3	672	575
-2	162	-225
-1	2100	-1863
0	924	747
1	708	-642
2	167	-242
3	1126	-1105
4	185*	77
5	1093	1108
6	150*	-16
7	630	-672
8	288	-259
9	246	230
10	493	498
11	166*	74
12	422	-478
13	175*	-40
14	336	339
15	190	-214
H,5,0		
-18	191*	93
-17	194*	-213
-16	396	-435
-15	161	125
-14	241	295
-13	169	179
-12	269	-321
-11	303	-300
-10	334	313
-9	381	435
-8	164*	-73
-7	634	-658
-6	391	381
-5	1137	1114
-4	159	179
-3	1026	-1043
-2	605	-674
-1	346	356
0	175*	93
1	152*	-49
2	760	-729
3	597	596
4	884	892
5	601	-607
6	679	-667
7	349	-355
8	369	388
9	645	641
10	151	-116
11	575	-598
12	175*	-81
13	352	314
14	177*	28
15	207	-213
H,6,0		
-17	171	-174
-16	181*	139
-15	522	545
-14	148	-43
-13	369	-389
-12	175*	109
-11	161*	10
-10	270	272
-9	173*	-66
-8	764	-795
-7	150	178
-6	549	581
-5	152*	17
-4	751	-794
-3	881	-843
-2	481	496
-1	1238	1253
0	175*	-260
1	955	-1020
2	212	-127
3	439	465
4	136	-102
5	433	-491
6	225	-255
7	491	501
8	377	381
9	165*	27
10	349	-369
11	218	-216
12	427	441
13	180*	137
14	292	-289
15	187*	-55
H,7,0		
-17	198*	173
-16	376	399
-15	192*	-170
-14	510	-516
-13	177*	-51
-12	371	367
-11	168*	111
-10	372	-374
-9	198	-227
-8	163*	94
-7	667	656
-6	194	240
-5	431	-453
-4	275	-284
-3	752	767
-2	843	840
-1	400	-387
0	1257	-1244
1	331	-279
2	1088	1155
3	138	100
4	278	-305
5	257	-154
6	442	424
7	547	585
8	166*	-134
9	348	-386
10	172*	62
11	285	323
12	181*	120
13	286	-249
14	250	-233
15	198*	79
H,8,0		
-16	199*	-79
-15	429	-462
-14	185*	-69
-13	505	482
-12	175*	109
-11	307	-315
-10	275	-288
-9	155	239
-8	648	639
-7	166*	-70
-6	623	-609
-5	368	369
-4	177*	-19
-3	161*	53
-2	401	-349
-1	965	-1009
0	478	483
1	812	818
2	161*	115
3	523	-582
4	268	-308
5	409	421
6	414	458
7	347	-427
8	208	-256
9	156	150
10	192	170
11	175*	39
12	396	-313
13	160	-186
14	191*	158
H,9,0		
-15	189*	15
-14	169	236
-13	270	268
-12	270	-276
-11	534	-539
-10	363	336
-9	529	593
-8	168*	-53
-7	615	-651
-6	328	-305
-5	344	339
-4	196	237
-3	329	-364
-2	338	-367
-1	164*	176
0	606	591
1	264	267
2	913	-952
3	367	-431
4	544	577
5	318	333
6	171*	-18
7	329	-369
8	175*	47
9	369	358
10	152	-183
11	223	-251
12	189*	32
13	189*	111
H,10,0		
-14	191*	86
-13	194*	-205
-12	185	-202
-11	185*	181
-10	519	574
-9	179*	-54
-8	669	-682
-7	174*	12
-6	395	426
-5	179*	233
-4	166*	-85
-3	329	-324
-2	163*	26
-1	314	323
0	201*	25
1	593	-636
2	172*	-175
3	542	621
4	526	560
5	176*	-78
6	526	-567
7	175*	5
8	494	419
9	181*	-10
10	361	-332
11	185*	-86
12	198	209
H,11,0		
-13	194*	-156
-12	180*	72
-11	377	411
-10	204	-217
-9	485	-517
-8	175*	-32
-7	301	340
-6	226	162
-5	264	-312
-4	438	-459
-3	171*	139
-2	174*	74
-1	166*	29
0	199*	-175
1	149*	57
2	437	500
3	156	113
4	1092	-190
5	391	-372
6	184*	-117
7	367	397
8	149	104
9	377	-406
10	191*	-105
H,12,0		
-12	208	158
-11	185*	-34
-10	272	-310
-9	189*	-102
-8	401	384
-7	223	218
-6	314	-330
-5	301	-295
-4	178*	21
-3	650	694
-2	179*	105
-1	333	-361
0	198*	177
1	177*	73
2	177*	-32
3	180*	10
4	276	-297
5	184*	-46
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-6	211	203
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1	178*	-75
2	177	-147
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4	175*	31
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6	176*	-43
7	184*	154
8	346	385
9	187*	-55
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12	156	129
13	189*	-34
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-11	194*	61
-10	159	128
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-6	181*	-165
-5	364	-383
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2	155	-67
3	169*	92
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5	410	-446
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8	213	-193
9	437	-470
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11	362	346
12	269	279
13	196*	-147
14	199*	-120
H,-9,2		
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-11	188*	124
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3	168*	128
4	175	147
5	169*	-168
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7	173*	23
8	178*	-115
9	172*	-29
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12	198*	-128
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7	166*	-156
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9	167*	-74
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14	208	108
15	191*	59
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-8	167*	-51
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6	371	-382
7	166*	-146
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12	188*	96
13	195	189
14	177	113
15	192	-184
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2	361	-333
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9	222	-226
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14	285	-282
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17	196*	131
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-13	172*	-28
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-11	173*	-184
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-7	238	226
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8	165*	-128
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16	184*	55
17	175	-184
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-13	172*	-19
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-8	304	312
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2	414	444
3	669	-597
4	531	-517
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6	189	158
7	380	-346
8	297	-289
9	163*	-163
10	311	334
11	335	337
12	257	-247
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15	232	-184
16	188*	-78
17	186*	-31
18	181*	-6
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6	573	-537
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9	922	926
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11	755	-783
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13	154	172
14	173*	-6
15	168	-134
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18	190*	64
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-4	488	-517
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2	363	361
3	191	-145
4	171	233
5	325	-238
6	679	624
7	152*	-8
8	367	367
9	181	-118
10	674	-648
11	275	-235
12	622	614
13	168*	-16
14	182*	-202
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16	183*	26
17	196*	120
18	193*	76
H,-1,2		
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-16	185*	145
-15	181	-145
-14	178*	-95
-13	173*	-87
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-11	139	160
-10	161*	-32
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-8	165	-164
-7	456	431
-6	171	218
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-4	175	-141
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3	1008	825
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8	299	343
9	*466	-452
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11	245	346
12	165*	27
13	178*	-185
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16	193*	218
17	184*	-32
18	199*	-174
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-14	188*	38
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-12	164*	101
-11	162*	44
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-8	227	-146
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-6	172	199
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-3	341	-338
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-1	989	-1000
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3	146*	-40
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5	219	155
6	498	486
7	272	191
8	272	-268
9	235	136
10	280	276
11	168*	66
12	264	-271
13	171*	-115
14	179*	14
15	326	362
16	218	174
17	189	-245
18	195*	-7
H,1,2		
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-17	185*	-57
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-13	262	283
-12	293	-275
-11	332	-314
-10	419	430
-9	182	197
-8	197	224
-7	249	269
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-4	315	-315
-3	615	-487
-2	694	645
-1	585	508
0	550	518
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2	765	-688
3	540	537
4	259	-191
5	648	563
6	578	578
7	564	-604
8	157*	-4
9	171*	138
10	348	-362
11	169*	110
12	165*	108
13	188	-185
14	373	385
15	172	185
16	278	-289
17	185*	-56
H,2,2		
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-17	177*	-33
-16	151	-85
-15	180*	121
-14	176*	-24
-13	166*	-6
-12	328	-373
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-10	680	674
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-6	154*	-87
-5	149*	25
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-3	843	-728
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4	834	900
5	154*	-26
6	248	-295
7	156*	-16
8	167*	104
9	157	-132
10	167*	82
11	161*	23
12	176*	210
13	173*	68
14	152	-169
15	182*	-145
16	183*	-7
17	161	93
H,3,2		
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-17	172	-181
-16	181*	34
-15	169	11
-14	250	256
-13	276	-308
-12	140	-125
-11	525	564

TABLE X. (Cont.)

H,3,2		
-10	158*	86
-9	275	-314
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-3	177	-209
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1	226	176
2	377	-350
3	611	528
4	149*	51
5	418	-378
6	155*	-159
7	473	493
8	168*	139
9	198	-172
10	280	-263
11	169*	-87
12	232	286
13	198	-172
14	189*	-167
15	192*	166
16	194*	108
17	193*	12
H,4,2		
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-17	189*	29
-16	266	261
-15	179*	-62
-14	174*	18
-13	177*	-159
-12	171*	174
-11	170*	157
-10	325	-328
-9	152	-130
-8	391	361
-7	156*	132
-6	192	-266
-5	1083	-1032
-4	144	109
-3	1640	1585
-2	242	-195
-1	426	-407
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1	284	-314
2	498	459
3	157*	100
4	646	-728
5	195	-157
6	179	242
7	166*	5
8	166*	-134
9	804	-848
10	278	-281
11	669	704
12	176*	-33
13	161	-209
14	180*	78
15	189*	62
16	186*	43
H,5,2		
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-16	188*	-98
-15	192*	-177
-14	180*	-48
-13	239	262
-12	172*	-75
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-10	166*	25
-9	355	-322
-8	229	220
-7	268	221
-6	917	-994
-5	155*	-100
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-3	317	241
-2	587	-601
-1	233	-271
0	302	274
1	333	307
2	218	-257
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4	198	-237
5	152	-114
6	168*	-17
7	164	-276
8	278	-179
9	164*	74
10	437	450
11	223	207
12	189*	-242
13	184*	-85
14	222	182
15	196*	45
16	199*	-100
H,6,2		
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-16	322	-350
-15	236	209
-14	420	444
-13	181*	-58
-12	171*	-188
-11	171*	127
-10	174	-161
-9	249	248
-8	164*	192
-7	162*	-145
-6	138	-64
-5	170	215
-4	462	481
-3	156*	-44
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1	131	42
2	468	-509
3	264	249
4	147	-76
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6	171	-169
7	166*	134
8	162*	129
9	179	233
10	177*	122
11	305	-361
12	179*	-79
13	226	215
14	183*	118
15	189*	-38
H,7,2		
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-15	305	301
-14	156	-123
-13	485	-491
-12	177*	113
-11	173*	74
-10	172*	-21
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-8	353	-368
-7	164	244
-6	344	351
-5	480	-453
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-3	278	271
-2	367	325
-1	363	353
0	268	-243
1	830	-855
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3	423	435
4	161	-210
5	165*	-68
6	142	-135
7	135	112
8	167*	49
9	171*	-132
10	172*	-44
11	177*	-7
12	255	220
13	185*	131
14	189*	-95
H,8,2		
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-14	180	-167
-13	187*	-82
-12	184*	131
-11	306	303
-10	175*	51
-9	455	-457
-8	232	243
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-5	160*	-7
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-3	222	246
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3	159*	-15
4	230	-238
5	274	-284
6	162*	151
7	171*	48
8	175*	80
9	170*	-10
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11	199*	248
12	187*	-111
13	194*	-125
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-11	181*	-33
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-7	166	147
-6	167*	21
-5	173*	-157
-4	178	-89
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-2	164*	-3
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2	171*	-139
3	180	-237
4	244	-225
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6	340	347
7	180*	130
8	250	-258
9	179*	61
10	185*	137
11	186*	-122
12	189*	-110
H,10,2		
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-12	188*	-66
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-10	175*	14
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-8	215	-222
-7	185	-157
-6	140	-144
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2	169*	-12
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4	172*	-59
5	307	357
6	178*	-3
7	173	-187
8	194*	-191
9	173	148
10	191*	95
11	194*	-143
H,11,2		
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5	185*	-9
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9	196*	71
10	194*	-53
H,12,2		
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3	185*	60
4	184*	-30
5	192	-207
6	194*	176
7	229	149
8	193*	94
H,13,2		
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-7	271	296
-6	193*	-78
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-3	260	235
-2	229	237
-1	249	-256
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2	168	136
3	184*	40
4	194*	-138
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H,14,2		
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-4	237	244
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1	283	284
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-1	194*	-97
0	191	-150
1	190*	-53
2	201*	238
3	197*	142
4	319	-335
5	214	-198
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-3	199*	188
-2	180*	-62
-1	179	-208
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2	194*	181
3	288	-244
4	277	-311
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6	233	264
7	187*	-42
8	175	-176
9	193*	-65
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-6	222	-207
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2	199	-223
3	407	-369
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5	232	235
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7	290	-328
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10	193*	139
11	213	-232
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-9	191*	132
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2	184	-191
3	180*	115
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7	182*	-42
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9	333	347
10	327	-348
11	406	-366
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-6	232	269
-5	404	-419
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-1	152	204
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3	173*	138
4	169*	13
5	171*	-60
6	147	-118
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8	316	288
9	489	-471
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12	322	376
13	188*	41
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3	169*	-48
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7	169*	48
8	365	-380
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12	183*	11
13	407	-392
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2	592	-569
3	189	-209
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6	290	336
7	218	-190
8	338	-292
9	278	261
10	171*	37
11	146	-152
12	208	-225
13	193	-178
14	297	261
15	166	242
H,-7,3		
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9	544	569
10	160	-167
11	669	-629
12	239	-237
13	218	222
14	226	230
15	189*	-59
16	260	-266
17	189*	6
H,-1,4		
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-16	180*	126
-15	232	228
-14	180*	17
-13	426	-430
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-11	241	215
-10	256	276
-9	115	-93
-8	263	-324
-7	143	-91
-6	140	162
-5	156*	-156
-4	354	-374
-3	292	-256
-2	398	387
-1	491	638
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2	143	-133
3	621	626
4	432	382
5	338	-316
6	619	-594
7	171*	18
8	522	526
9	166*	-36
10	452	-449
11	171*	-5
12	231	225
13	442	438
14	182*	8
15	379	-414
16	189*	47
17	264	242
H,0,4		
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-15	179	21
-14	382	-320
-13	250	-235
-12	368	353
-11	659	648
-10	310	-281
-9	658	-644
-8	157*	33
-7	492	476
-6	305	374
-5	153*	-12
-4	157*	-68
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-1	212	-276
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1	386	-339
2	347	343
3	437	454
4	1016	-1024
5	489	-452
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7	466	470
8	145	201
9	245	-254
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11	264	242
12	172*	58
13	172*	-30
14	307	-297
15	186*	-108
16	306	294
17	200*	75
H,1,4		
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-16	184*	-3
-15	185*	-141
-14	252	-277
-13	269	244
-12	558	569
-11	545	-549
-10	641	-667
-9	163*	91
-8	840	850
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-6	518	-525
-5	199	-216
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-2	280	-295
-1	1046	-1028
0	826	-854
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2	288	274
3	1024	-1016
4	314	-329
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6	1132	1112
7	161*	49
8	562	-584
9	203	191
10	250	310
11	171*	24
12	189	-190
13	212	-221
14	182*	42
15	292	327
16	189*	2
H,2,4		
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-16	175	-172
-15	186*	-131
-14	181*	16
-13	343	305
-12	171*	-48
-11	622	-583
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-9	755	755
-8	160*	73
-7	766	-733
-6	219	-214
-5	157*	-9
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-3	336	-356
-2	925	-847
-1	155*	-3
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1	157*	117
2	157*	72
3	859	-832
4	805	806
5	1065	1055
6	200	-158
7	873	-869
8	251	-228
9	564	579
10	221	228
11	351	-372
12	181*	-150
13	180*	102
14	182	196
15	187*	-17
16	288	-254
H,3,4		
-17	194*	-138
-16	192*	-169
-15	184*	-12
-14	311	312
-13	180*	15
-12	447	-466
-11	177	115
-10	358	389
-9	157*	-67
-8	751	-780
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-6	756	759
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1	183	-219
2	290	-263
3	346	-324
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5	253	230
6	900	-932
7	194	-170
8	609	581
9	258	216
10	199	-210
11	329	-363
12	177*	-12
13	238	223
14	191*	-92
15	244	-236
16	190*	94
H,4,4		
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-15	282	272
-14	186*	-24
-13	313	-312
-12	190*	-79
-11	226	255
-10	174*	50
-9	323	-350
-8	166*	47
-7	451	428
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-5	262	-250
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-3	161*	54
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2	147	137
3	575	618
4	172*	-65
5	433	-408
6	167*	163
7	526	527
8	177	225
9	277	-318
10	591	-570
11	182*	102
12	308	345
13	183*	-11
14	240	-206
15	193*	59
H,5,4		
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-15	173	-123
-14	371	-346
-13	179*	120
-12	293	314
-11	170*	-8
-10	175*	149
-9	182	-147
-8	161*	93
-7	484	536
-6	582	-578
-5	887	-893
-4	291	351
-3	377	390
-2	159	81
-1	168*	-154
0	177	-191
1	463	469
2	550	575
3	170	-226
4	419	-437
5	164*	-17
6	431	458
7	166*	-38
8	316	-331
9	302	-336
10	181*	-124
11	502	550
12	185*	113
13	252	-312
14	171	-55
15	190	149
H,6,4		
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-15	248	-271
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-13	360	372
-12	173*	-83
-11	305	-354
-10	391	-381
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-8	409	459
-7	175	-208
-6	739	-798
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-4	954	1002
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-2	195	-189
-1	476	-481
0	235	271
1	726	764
2	143	-156
3	677	-706
4	248	217
5	288	347
6	156	-102
7	175*	-43
8	174*	-85
9	173*	65
10	347	382
11	189*	-109
12	249	-222
13	191*	-39
14	199*	171
H,7,4		
-15	194*	127
-14	254	305
-13	176*	10
-12	296	-346
-11	180*	-125
-10	270	285
-9	522	512
-8	226	221
-7	420	-394
-6	169*	75
-5	420	406
-4	138	78
-3	162*	-137
-2	399	-402
-1	163*	72
0	864	865
1	169*	-95
2	749	-742
3	209	162
4	470	482
5	278	229
6	181*	-103
7	246	-258
8	185*	134
9	172	156
10	199	-193
11	193	-165
12	187*	74
13	168	174
H,8,4		
-14	185*	6
-13	181	-181
-12	162	-168
-11	301	346
-10	582	557
-9	330	-351
-8	278	-285
-7	175*	-37
-6	173*	-32
-5	175*	23
-4	186	-127
-3	166*	-19
-2	149	83
-1	540	575
0	177*	-62
1	718	-758
2	171*	32
3	660	651
4	344	376
5	263	-291
6	608	-621
7	189*	-84
8	459	499
9	195*	-38
10	327	-333
11	197*	156
12	214	148
H,9,4		
-13	186*	-78
-12	195	215
-11	491	415
-10	363	-403
-9	616	-701
-8	179*	99
-7	468	480
-6	183*	10
-5	200	-197
-4	175*	82
-3	179*	128
-2	204	204
-1	195	-211
0	272	-258
1	172	193
2	208	227
3	181*	94
4	185*	-74
5	522	-494
6	189*	14
7	413	433
8	197*	-34
9	260	-226
10	200*	25
11	196	176
H,10,4		
-12	167	188
-11	176*	-63
-10	552	-542
-9	221	148
-8	475	546
-7	199	232
-6	255	-279
-5	502	-563
-4	313	311
-3	345	408
-2	255	-305
-1	215	-273
0	217	172
1	184*	71
2	178*	103
3	185*	-108
4	338	-361
5	182*	98
6	261	212
7	194*	-24
8	295	-269
9	191*	-97

TABLE X. (Cont.)

H,11,4		
-10	184*	76
-9	192*	150
-8	156*	56
-7	328	-350
-6	260	-323
-5	332	324
-4	456	499
-3	250	-247
-2	520	-518
-1	246	238
0	377	359
1	181*	-28
2	199	-223
3	266	-296
4	182*	128
5	289	287
6	197*	-146
7	283	-318
8	189*	13
H,12,4		
-9	193*	-74
-8	191*	-11
-7	180*	-85
-6	197*	185
-5	372	355
-4	180	-180
-3	356	-349
-2	183*	5
-1	181	225
0	173	82
1	197	-202
2	194	-200
3	196*	95
4	198*	142
5	199*	-137
H,13,4		
-5	261	-225
-4	246	-276
-3	167	-118
-2	302	294
-1	199*	219
0	227	-244
1	176	-204
H,-13,5		
-2	200*	165
-1	184*	64
0	199*	-86
1	188*	-160
2	184	89
3	320	332
4	200	-201
5	208	-224
H,-12,5		
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-4	187*	124
-3	195*	199
-2	166	100
-1	190*	-130
0	269	-218
1	161	142
2	425	390
3	199*	-156
4	322	-343
5	182*	51
6	261	273
7	188*	133
8	252	-269
H,-11,5		
-8	199*	144
-7	213	-237
-6	190*	-22
-5	194*	123
-4	194	181
-3	189*	20
-2	225	-183
-1	185*	-118
0	189*	13
1	186*	153
2	185*	-43
3	192*	-182
4	187*	14
5	188*	42
6	186*	30
7	232	-244
8	188*	-33
9	197*	151
10	350	337
H,-10,5		
-9	223	232
-8	191*	-15
-7	184*	-98
-6	186*	51
-5	224	226
-4	180*	-36
-3	168	-160
-2	265	-295
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1	176*	-140
2	177*	-87
3	173*	-42
4	185*	-92
5	208	277
6	172*	10
7	183*	-178
8	283	283
9	211	233
10	231	-242
11	255	-216
12	187*	56
H,-9,5		
-11	181	171
-10	235	203
-9	186*	-75
-8	157	-190
-7	223	119
-6	180*	75
-5	184*	-4
-4	177	-243
-3	267	-221
-2	579	577
-1	439	446
0	328	-316
1	333	-370
2	177*	8
3	235	236
4	185*	160
5	179*	10
6	307	-421
7	184*	108
8	182*	179
9	183*	-46
10	288	-302
11	188*	70
12	213	170
13	185*	71
H,-8,5		
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-11	189*	95
-10	183*	-105
-9	264	-219
-8	175*	-70
-7	204	177
-6	197	217
-5	176*	-123
-4	211	-196
-3	215	215
-2	327	354
-1	209	-261
0	567	-565
1	171*	-10
2	268	234
3	171*	69
4	180	-169
5	428	-448
6	182*	136
7	386	392
8	176*	-14
9	183*	-83
10	186*	-55
11	175*	45
12	285	287
13	200*	-152
14	284	-296
H,-7,5		
-13	198	164
-12	207	182
-11	193*	-130
-10	274	-261
-9	180	195
-8	349	359
-7	271	246
-6	175*	-47
-5	672	-649
-4	174*	124
-3	461	490
-2	293	-339
-1	216	-259
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2	364	370
3	522	-502
4	423	-402
5	625	635
6	487	497
7	404	-398
8	179*	-69
9	190	-211
10	169	136
11	181*	88
12	160	-116
13	274	-275
14	161	31
H,-6,5		
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-13	198*	138
-12	222	-167
-11	190*	-104
-10	344	347
-9	205	220
-8	222	-206
-7	316	-299
-6	521	-486
-5	309	287
-4	255	265
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-2	176*	-228
-1	232	-226
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1	232	235
2	189	-131
3	566	-508
4	503	506
5	714	711
6	513	-516
7	428	-429
8	171*	-103
9	233	225
10	185*	157
11	180*	-123
12	194*	-220
13	180*	52
14	193*	151
15	188*	-72
H,-5,5		
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-13	194*	-65
-12	297	-248
-11	181*	122
-10	224	205
-9	306	-290
-8	544	-523
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-4	272	-323
-3	325	-376
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-1	161*	-24
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1	187	-86
2	568	-610
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4	525	550
5	399	-408
6	506	-537
7	162*	19
8	333	355
9	518	486
10	506	-496
11	264	-245
12	241	217
13	182*	76
14	190*	-36
15	189*	-46
H,-4,5		
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-14	192*	-133
-13	227	-260
-12	184*	61
-11	180*	158
-10	175	174
-9	288	-293
-8	236	-227
-7	719	733
-6	306	311
-5	653	-655
-4	316	-354
-3	167*	121
-2	166*	107
-1	229	241
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1	165*	-100
2	378	392
3	169*	177
4	259	-197
5	404	-422
6	161*	-2
7	353	368
8	175	212
9	520	-559
10	411	-398
11	232	254
12	305	318
13	211	-192
14	175	-109
15	188*	100
16	183*	27
H,-3,5		
-15	229	-213
-14	179*	-79
-13	183*	92
-12	185*	136
-11	314	340
-10	179	-170
-9	341	-393
-8	213	209
-7	286	305
-6	457	-436
-5	381	-401
-4	158	185
-3	476	544
-2	172	139
-1	243	-298
0	139	-139
1	452	406
2	188	288
3	182	127
4	329	-389
5	165*	-158
6	325	373
7	269	233
8	213	-219
9	382	-411
10	480	489
11	412	426
12	182	-107
13	246	-214
14	185*	69
15	208	192
16	187*	110
H,-2,5		
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-15	187*	-105
-14	241	241
-13	152	61
-12	175*	21
-11	249	-307
-10	278	-333
-9	159*	54
-8	265	309
-7	165*	-52
-6	309	-303
-5	369	368
-4	429	414
-3	163*	119
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2	136	-289
3	306	-277
4	311	-268
5	269	302
6	313	308
7	200	-175
8	210	-272
9	287	317
10	253	287
11	171*	-58
12	419	-409
13	179*	-55
14	292	335
15	172	124
16	197*	-159
H,-1,5		
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-15	195*	200
-14	177*	-8
-13	180*	-65
-12	346	-332
-11	308	-275
-10	444	409
-9	303	269
-8	162*	-44
-7	226	-234
-6	175	-153
-5	164*	169
-4	169*	94
-3	550	-603
-2	336	-309
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0	341	325
1	157*	-112
2	512	-538
3	136	-114
4	543	516
5	193	169
6	157*	-24
7	204	-204
8	161*	-9
9	222	228
10	177*	-176
11	179*	-152
12	175*	-24
13	187	214
14	212	210
15	186	-179
16	181	-239
H,0,5		
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-15	196*	170
-14	199*	40
-13	333	-260
-12	177*	-45
-11	490	517
-10	330	338
-9	302	-268
-8	520	-490
-7	161*	51
-6	219	232
-5	301	281
-4	185	-221
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-2	157*	102
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0	225	-212
1	841	-810
2	159*	108
3	484	487
4	283	312
5	202*	-203
6	573	-560
7	197	204
8	210	206
9	168*	-87
10	172*	12
11	175*	-44
12	201	173
13	185*	92
14	156	-146
15	169	-148
16	189*	69
H,1,5		
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-15	189*	-17
-14	187*	-127
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-12	279	268
-11	336	349
-10	663	-709
-9	434	-440
-8	484	389
-7	330	349
-6	342	387
-5	332	-311
-4	481	-456
-3	681	629
-2	129	132
-1	476	-469
0	324	-304
1	154*	-21
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3	283	321
4	830	-852
5	262	-299
6	286	319
7	566	588
8	148	-137
9	514	-533
10	160	111
11	200	227
12	166*	4
13	145*	-129
14	188*	-128
15	189*	73
16	175	216
H,2,5		
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-15	192*	-226
-14	231	-190
-13	186*	130
-12	234	242
-11	407	-439
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-9	330	285
-8	655	631
-7	160*	-23
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-5	563	-539
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-3	503	477
-2	213	-235
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1	269	304
2	246	227
3	292	-267
4	336	-400
5	431	448
6	706	776
7	349	-343
8	473	-488
9	168*	-2
10	290	315
11	249	258
12	199	-222
13	192*	-115
14	207	171
15	194*	96
H,3,5		
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-15	183	-93
-14	161	192
-13	222	267
-12	182*	-51
-11	256	-217
-10	257	276
-9	281	327
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-7	329	-339
-6	471	-447
-5	817	781
-4	968	932
-3	587	-487
-2	316	-308
-1	273	228
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1	428	392
2	199	-182
3	578	-614
4	336	357
5	387	385
6	170*	-129
7	559	-559
8	173*	-64
9	404	404
10	241	216
11	206	-202
12	231	-248
13	189*	130
14	202	164
15	199*	-145
H,4,5		
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-14	188	185
-13	353	-381
-12	188*	-112
-11	171*	-75
-10	191*	181
-9	301	298
-8	461	-457
-7	171*	85
-6	651	658
-5	342	359
-4	629	-637
-3	484	-508
-2	189	197
-1	358	411
0	265	-239
1	157	-156
2	165*	-90
3	208	176
4	434	449
5	163*	-45
6	432	-475
7	298	354
8	240	252
9	224	219
10	186*	-171
11	445	-452
12	247	231
13	259	267
14	179	-165
H,5,5		
-15	194*	38
-14	302	-303
-13	159	-140
-12	186*	168
-11	316	355
-10	254	282
-9	189	-244
-8	171*	-119
-7	281	188
-6	262	265
-5	646	-683
-4	688	-711
-3	423	427
-2	484	473
-1	162*	-60
0	428	-408
1	141	-115
2	476	504
3	324	384
4	297	-306
5	171*	-11
6	171*	24
7	180*	93
8	180*	131
9	251	-310
10	363	-336
11	236	248
12	297	285
13	188	-156
14	202	-239
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3	186	143
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7	236	210
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3	208	174
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13	156	-45
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13	307	-358
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12	164	-172
13	166	-220
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15	194*	-55
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8	392	411
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9	171	-220
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12	207	161
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3	168*	-115
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6	229	228
7	227	259
8	293	-292
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13	157	-119
14	187*	-50
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11	199*	7
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H,10,6		
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9	170	152
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11	186*	105
12	194*	43
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1	173*	-97
2	178	-164
3	167*	110
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13	193*	-124
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2	366	390
3	173*	-36
4	213	-235
5	306	252
6	181*	-72
7	180*	121
8	248	225
9	434	-425
10	182*	-86
11	191*	135
12	175	185
13	195*	-101
H,-2,7		
-13	224	181
-12	192*	115
-11	155	71
-10	265	-214
-9	281	-275
-8	236	237
-7	190*	194
-6	318	-327
-5	174*	-12
-4	175*	64
-3	202	195
-2	167*	75
-1	489	-618
0	291	-285
1	742	774
2	206	212
3	194	-235
4	177*	26
5	179*	103
6	172*	38
7	181*	-71
8	166	-166
9	182*	-11
10	183*	117
11	157	176
12	172	-116
13	198*	-182
14	185*	-11
H,-1,7		
-14	175	153
-13	199	170
-12	185*	20
-11	357	-357
-10	180*	22
-9	416	460
-8	175*	67
-7	189	-158
-6	185	-178
-5	229	199
-4	187	192
-3	178*	27
-2	451	-494
-1	227	-267
0	668	701
1	533	536
2	539	-533
3	518	-552
4	143	134
5	229	227
6	178*	-19
7	279	-265
8	182*	-61
9	340	358
10	189*	-89
11	187*	-103
12	185*	-128
13	189*	-3
14	171	126
H,0,7		
-14	203*	117
-13	196*	-2
-12	325	-345
-11	188*	-51
-10	479	455
-9	185*	-12
-8	447	-432
-7	182*	-61
-6	180*	147
-5	174*	-66
-4	209	220
-3	447	-480
-2	218	-145
-1	557	528
0	179*	85
1	373	-409
2	397	-392
3	173*	28
4	641	622
5	144	88
6	517	-520
7	186*	-87
8	354	354
9	182*	43
10	168	-189
11	220	-181
12	193*	108
13	190*	98
H,1,7		
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-13	236	-250
-12	217	-173
-11	554	457
-10	185*	-56
-9	444	-469
-8	180*	-121
-7	176*	39
-6	324	324
-5	171*	2
-4	442	-441
-3	162*	-58
-2	271	299
-1	174*	-38
0	170*	74
1	152	-187
2	175*	154
3	409	443
4	174*	-104
5	404	-386
6	310	-331
7	431	423
8	265	294
9	413	-417
10	178*	-58
11	189*	73
12	162	165
13	192*	28
H,2,7		
-14	192*	-136
-13	189*	-42
-12	172	159
-11	190*	-30
-10	251	-275
-9	166	-126
-8	436	408
-7	283	338
-6	286	-198
-5	377	-377
-4	172*	7
-3	541	567
-2	162*	50
-1	174*	-147
0	174*	30
1	171*	61
2	171*	-120
3	175*	-90
4	394	-401
5	202	-226
6	523	576
7	189*	175
8	199	-234
9	168	-164
10	187*	-21
11	223	288
12	189*	-3
13	196*	-126
H,3,7		
5	346	401
6	304	265
7	160	-168
8	199*	-221
9	186*	91
10	197	162
11	204*	156
12	200*	-165
H,4,7		
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-12	193*	-68
-11	188*	-61
-10	184*	-84
-9	295	275
-8	185*	44
-7	355	-349
-6	177*	-48
-5	302	315
-4	292	268
-3	443	-459
-2	249	-231
-1	175*	89
0	204	206
1	179*	87
2	198	-190
3	180*	106
4	365	391
5	180*	-16
6	278	-296
7	180*	-22
8	171*	16
9	194*	147
10	189*	168
11	292	-266
12	193*	-50
H,5,7		
-13	164*	-194
-12	189*	-111
-11	189*	71
-10	187*	64
-9	185*	77
-8	185*	-114
-7	189*	-162
-6	297	312
-5	147*	150
-4	301	-256
-3	179*	-30
-2	177*	37
-1	322	319
0	273	257
1	536	-546
2	182*	-45
3	397	391
4	180*	-77
5	180*	-25
6	181*	-106
7	189*	56
8	165*	223
9	193*	-49
10	198*	-133
11	192*	-75
H,6,7		
-12	194*	76
-11	198*	177
-10	187*	57
-9	298	-270
-8	191*	46
-7	190	167
-6	194*	64
-5	182*	7
-4	197*	-137
-3	185*	92
-2	180	197
-1	198*	-14
0	411	-422
1	196*	-178
2	417	401
3	311	296
4	191*	-64
5	214	-243
6	196*	27
7	198*	74
8	199*	21
9	203*	-128
10	199*	-92
H,7,7		
-11	188*	-34
-10	343	-310
-9	196*	-3
-8	197*	227
-7	199*	142
-6	189*	-144
-5	193*	-134
-4	190*	33
-3	185*	-47
-2	190*	-69
-1	270	-248
0	204*	-23
1	378	350
2	196*	193
3	165	-224
4	283	-273
5	195*	-101
6	190*	144
7	194*	62
8	200*	-185
9	196*	-34
H,8,7		
-10	195*	-91
-9	410	409
-8	196*	89
-7	175	-226
-6	190*	-149
-5	180*	-5
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-2	195*	-130
-1	172	101
0	188*	135
1	190*	120
2	210	-227

TABLE X. (Cont.)

H,8,7		
3	265	-304
4	160	98
5	174	244
6	192*	27
7	199*	-118
H,9,7		
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-7	241	-245
-6	175	188
-5	209	234
-4	190*	-119
-3	173	-188
-2	187*	-66
-1	194*	120
0	194*	97
1	221	-207
2	191*	-114
3	189*	118
4	203*	195
5	200*	110
H,10,7		
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-5	194*	-40
-4	291	-244
-3	198*	-86
-2	306	273
-1	192*	46
0	194*	-113
1	194*	67
2	198*	59
3	189*	57
H,-10,8		
-2	204*	-135
-1	211	-242
0	189*	37
1	279	274
2	188*	98
3	163	-137
4	195*	-88
H,-9,8		
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-4	210	208
-3	189*	6
-2	285	-232
-1	190*	42
0	306	269
1	189*	17
2	201	-181
3	188	-229
4	193*	84
5	201*	199
6	197*	-67
7	198*	-176
H,-8,8		
-7	204*	-83
-6	187*	85
-5	218	237
-4	189*	-61
-3	191*	-141
-2	157*	7
-1	198*	128
0	197*	75
1	223	-237
2	204*	-184
3	209*	237
4	317	278
5	203*	-78
6	242*	-275
7	190*	-38
8	201	203
9	193*	47
H,-7,8		
-8	189*	-62
-7	175*	162
-6	224	162
-5	189*	-76
-4	196*	-217
-3	187*	-112
-2	228	277
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0	208	-187
1	179*	-7
2	186*	32
3	363	357
4	193*	-120
5	578	-520
6	189*	-10
7	316	281
8	180*	95
9	199*	-108
10	163*	-114
H,-6,8		
-9	189*	-38
-8	189*	210
-7	212*	232
-6	184*	-150
-5	291	-304
-4	188*	70
-3	298	357
-2	181*	86
-1	180*	-112
0	194*	-192
1	175*	15
2	232	246
3	199	-182
4	339	-373
5	189*	-21
6	404	-22
7	232	226
8	215	-216
9	194	-141
10	187*	91
H,-5,8		
-10	192*	-56
-9	273	277
-8	164	157
-7	175	-164
-6	243	-233
-5	199	208
-4	298	359
-3	180*	-11
-2	359	-387
-1	156	-112
0	182*	39
1	179*	98
2	177*	51
3	382	-390
4	177*	63
5	531	516
6	177*	5
7	255	-257
8	239	-214
9	188*	81
10	331	327
11	190*	-69
H,-4,8		
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-10	193*	97
-9	191*	78
-8	188*	-46
-7	373	-362
-6	212	205
-5	343	396
-4	247	-287
-3	256	-365
-2	180*	-6
-1	182*	104
0	311	335
1	181*	-5
2	340	-380
3	149	103
4	243	267
5	180*	60
6	172	-210
7	214	-244
8	243	225
9	361	357
10	194*	-82
11	333	-339
H,-3,8		
-11	181*	41
-10	194*	175
-9	189*	-140
-8	292	-286
-7	189	131
-6	369	336
-5	204	-182
-4	251	-260
-3	179*	-88
-2	287	350
-1	159	193
0	202	-259
1	283	-285
2	148	-69
3	205	220
4	171*	16
5	175*	-37
6	179*	6
7	180*	14
8	280	267
9	186*	-93
10	311	-316
11	192*	-28
12	310	281
H,-2,8		
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-11	198*	152
-10	192*	-81
-9	185	-211
-8	191*	67
-7	349	359
-6	185*	-71
-5	198*	-147
-4	190*	-89
-3	177	184
-2	173	142
-1	171	-235
0	482	-494
1	258	220
2	527	509
3	189*	-162
4	185*	36
5	181*	8
6	184*	-71
7	215	194
8	195*	-151
9	198	-198
10	188*	49
11	155	174
12	199*	48
H,-1,8		
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-11	193*	-90
-10	322	-325
-9	228	211
-8	264	271
-7	185*	-82
-6	180*	-68
-5	183*	-148
-4	180*	-6
-3	324	355
-2	260	-261
-1	264	-319
0	322	326
1	546	568
2	175*	18
3	430	-422
4	177	-199
5	386	278
6	175	254
7	178*	-61
8	251	-255
9	186*	47
10	255	219
11	186*	59
12	191	-139
H,0,8		
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-11	285	-273
-10	262	252
-9	315	272
-8	185*	-16
-7	274	-250
-6	166	-184
-5	181*	11
-4	254	273
-3	172*	-53
-2	173	-211
-1	179*	-2
0	222	214
1	185*	-1
2	308	-314
3	227	-193
4	199*	200
5	526	509
6	187*	-23
7	473	-466
8	188*	90
9	218	207
10	198*	-3
11	189*	-73
12	199*	-94
H,1,8		
-12	175	-229
-11	231	168
-10	457	444
-9	183*	-91
-8	395	-389
-7	179*	-46
-6	185*	157
-5	344	304
-4	173*	-24
-3	588	-553
-2	221	206
-1	178*	171
0	180*	-54
1	214	203
2	184*	-100
3	185*	149
4	532	558
5	154	-120
6	487	-461
7	186*	18
8	247	241
9	198*	-28
10	197*	-118
11	201*	-153
H,2,8		
-12	215	179
-11	190	236
-10	188*	-92
-9	251	-288
-8	183*	-89
-7	411	373
-6	229	186
-5	414	-389
-4	457	-472
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-2	350	375
-1	175*	-44
0	181*	-116
1	178*	-18
2	181*	167
3	183	221
4	149	-151
5	475	-520
6	185*	-53
7	365	362
8	185*	17
9	166	-163
10	260	-219
11	200*	102
H,3,8		
-12	201*	128
-11	190*	-4
-10	194*	-171
-9	162	-98
-8	269	251
-7	185*	160
-6	260	-287
-5	416	-393
-4	353	368
-3	553	570
-2	311	-280
-1	283	-316
0	180*	-49
1	189*	120
2	229	198
3	176	-206
4	316	-355
5	198*	145
6	215	171
7	185*	15
8	195	-125
9	396	-300
10	194*	127
H,4,8		
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-10	193*	-62
-9	188*	6
-8	334	311
-7	253	-236
-6	455	-417
-5	316	286
-4	343	377
-3	194*	-183
-2	346	-346
-1	241	-253
0	470	462
1	274	283
2	333	-309
3	197*	-143
4	190*	130
5	196*	59
6	187*	-100
7	212	-215
8	192*	-98
9	171	154
10	250	228
H,5,8		
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-10	182*	75
-9	182*	88
-8	220	-162
-7	187*	-153
-6	181*	97
-5	182	195
-4	181*	37
-3	403	-370
-2	186*	-90
-1	346	384
0	335	316
1	282	-274
2	446	-456
3	183*	20
4	189*	164
5	192*	-120
6	202*	-148
7	193*	-6
8	189*	106
9	199	184
H,6,8		
-10	185*	82
-9	190*	-150
-8	194*	-106
-7	213	173
-6	162	176
-5	182*	-35
-4	184*	-108
-3	198*	-8
-2	182	142
-1	198	238
0	316	-312
1	302	-328
2	194*	46
3	236	237
4	189	123
5	197*	-146
6	191*	-51
7	197*	144
8	190*	103
H,7,8		
-8	189	204
-7	194*	152
-6	194*	-38
-5	173	-222
-4	185*	-73
-3	194*	160
-2	189*	-19
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0	192*	-107
1	200*	146
2	317	284
3	192*	7
4	245	-263
5	189*	-36
6	198*	127
H,8,8		
-7	190*	-38
-6	327	-306
-5	190*	-59
-4	216	170
-3	169	184
-2	189*	-7
-1	228	-249
0	197	189
1	219	225
2	192*	-76
3	202	-238
4	173	-141
H,9,8		
-3	189*	-94
-2	214	-230
-1	192*	50
0	197*	137
1	194*	-29
H,-8,9		
-3	188*	-109
-2	163	-187
-1	189*	101
0	187*	116
1	188*	-18
2	166	-212
3	166	-77
4	338	323
5	196*	185
H,-7,9		
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-3	200*	-179
-2	194*	-33
-1	161	182
0	195*	59
1	187*	-115
2	187*	-54
3	251	219
4	197*	162
5	204	-217
6	271	-293
H,-6,9		
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-5	198*	-188
-4	221	-280
-3	194*	120
-2	290	315
-1	191*	60
0	190*	-51
1	241	-216
2	187*	72
3	343	271
4	273	-218
5	269	-265
6	200*	116
7	224	218
8	194*	33
H,-5,9		
-8	240	206
-7	212	215
-6	279	-261
-5	191*	-151
-4	184	166
-3	163	208
-2	185*	79
-1	199*	-222
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1	187	147
2	200*	209
3	188*	-104
4	178	-175
5	185*	-29
6	321	304
7	189*	49
8	299	-233
H,-4,9		
-8	187	159
-7	259	-201
-6	202*	-213
-5	192*	114
-4	197*	200
-3	185*	-75
-2	222	-250
-1	185*	-60
0	193	215
1	245	247
2	228	-177
3	226	-250
4	190*	101
5	164	161
6	186*	29
7	193*	-141
8	243	-220
9	179	198
H,-3,9		
-9	209	214
-8	190*	-171
-7	279	-303
-6	232	209
-5	189*	200
-4	175*	13
-3	228	-184
-2	182*	-99
-1	316	383
0	222	245
1	377	-373
2	243	-260
3	185*	153
4	151	142
5	158	111
6	158	-130
7	192*	-112
8	160	163
9	175	170
H,-2,9		
-9	164	-157
-8	192*	-161
-7	179*	89
-6	161	211
-5	184*	144
-4	239	-266
-3	189*	-146
-2	231	310
-1	192*	145
0	372	-390
1	404	-354
2	199	229
3	330	323
4	186*	44
5	192*	-136
6	230	-193
7	182*	76
8	227	202
9	205	-160
10	209	-154
H,-1,9		
-10	196*	-172
-9	199*	-160
-8	193*	63
-7	219	167
-6	188*	-37
-5	186*	-99
-4	241	-226
-3	198*	188
-2	217	221
-1	325	-366
0	267	-274
1	449	449
2	283	310
3	186*	-85
4	164	-212
5	300	-307
6	246	256
7	194	204
8	216	-217
9	197*	-96
10	187*	46
H,0,9		
-10	200*	-152
-9	163	160
-8	190*	62
-7	180	-38
-6	188*	-74
-5	259	-224
-4	195*	187
-3	227	228
-2	188*	-199
-1	262	-222
0	194*	181
1	263	199
2	186*	-38
3	354	-332
4	206	-234
5	324	322
6	163	144
7	241	-226
8	202*	-154
9	194*	63
H,1,9		
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-9	200*	159
-8	191*	-39
-7	259	-217
-6	189*	-4
-5	202*	180
-4	248	205
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-2	302	-286
-1	193*	-7
0	199*	167
1	195*	112
2	194*	-152
3	348	-288
4	247	219
5	305	281
6	310	-333
7	201*	-173
8	180	149
9	192*	101
H,2,9		
-10	208	159
-9	198	-104
-8	208*	-181
-7	200*	-17
-6	247	235
-5	175	179
-4	470	-445
-3	190*	-142
-2	194*	183
-1	185	181
0	191*	149
1	191*	-77
2	263	-241
3	192*	127
4	198*	176
5	194	-225
6	164	-131
7	192*	94
8	215	222
9	189*	76
H,3,9		
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-8	173	-158
-7	338	302
-6	189*	114
-5	348	-372
-4	218	-204
-3	204	240
-2	327	311
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2	196*	128
3	197*	52
4	195*	-60
5	196*	-150
6	172	143
7	181	215
8	199*	3
H,4,9		
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-7	232	197
-6	220	-209
-5	345	-304
-4	283	278
-3	310	297
-2	196*	46
-1	426	-417
0	159	-153
1	395	377
2	205	206
3	173	-162
4	189*	-90
5	179	144
6	197*	116
7	191*	56
H,5,9		
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-7	208	-215
-6	196*	-150
-5	198*	126
-4	215	190
-3	199*	85
-2	301	-262
-1	194*	-135
0	382	416
1	199*	101
2	278	-242
3	192	-211
4	196*	81
5	188	188
6	202*	-8
H,6,9		
-6	198*	96
-5	194	221
-4	201*	-32
-3	201*	-86
-2	195*	-73
-1	172	199
0	205*	118
1	283	-313
2	248	-261
3	222	226
4	247	215
H,7,9		
-4	193*	-105
-3	202*	-108
-2	203*	127
-1	161	101
0	246	-274
1	218	-108
H,-6,10		
0	194*	-20
1	197*	-132
2	195*	31
H,-5,10		
-3	204*	152
-2	199*	56
-1	194*	-78
0	201*	-104
1	188*	-19
2	195	194
3	190*	-26
4	199*	-135
H,-4,10		
-5	195*	43
-4	192*	103
-3	189*	60
-2	187*	-113
-1	208*	-213
0	165	88
1	284	232
2	198*	-80
3	191*	-185
4	193*	-41
5	195*	11
H,-3,10		
-5	202*	146
-4	201*	83
-3	180	-131
-2	202*	-153
-1	194*	132
0	339	296
1	194*	-17
2	339	-339
3	194*	4
4	200*	75
5	197*	-10
6	194*	32
H,-2,10		
-6	201*	98
-5	193	112
-4	190*	-9
-3	179	-196
-2	183*	58
-1	263	303
0	194*	-56
1	394	-384
2	191*	-48
3	264*	130
4	198*	138
5	198*	-41
6	222	-158
H,-1,10		
-6	199*	156
-5	198*	-15
-4	236	-233
-3	190*	-6
-2	194*	163
-1	191*	-19
0	262	-222
1	199*	-101
2	213	193
3	196*	108
4	201*	-82
5	197	-166
6	195*	82
H,0,10		
-6	198*	-79
-5	181	-140
-4	193*	-15
-3	236	216
-2	195*	60
-1	199*	-169
0	192*	-28
1	196*	132
2	195*	60
3	195*	-25
4	203*	-175
5	178	144
6	305	304
H,1,10		
-6	196*	-145
-5	179*	95
-4	269	273
-3	191*	-43
-2	196*	-166
-1	195*	-187
0	196*	45
1	203*	160
2	202*	-45
3	216	-188
4	167	123
5	282	273
H,2,10		
-6	205*	139
-5	216*	216
-4	196*	-81
-3	213	-215
-2	189*	-43
-1	194*	88
0	198*	102
1	198*	40
2	195*	-96
3	194*	10
4	189	242
5	194*	-27
H,3,10		
-4	311	-305
-3	188*	95
-2	173	129
-1	201*	73
0	193*	-20
1	200*	-148
2	199*	50
3	197*	155
4	200*	-49

The bond distances in perchlorate anion are Cl–O(3) = 1.43 Å, Cl–O(4) = 1.40 Å, Cl–O(5) = 1.30 Å and 1.39 Å, the mean dimension of O–Cl–O angle is 109.5°. Only one distance Cl–O(5) differs significantly from the expected Cl–O value (1.40 Å)[24], but the O(5) atom possesses the highest thermal motion ($U = 0.1819$ Å^2) in the perchlorate group and many peaks in the difference Fourier synthesis are near this atom, moreover the O(5) atom is not involved in the closest intramolecular contacts of ClO_4^- anion: O(3)–C(9)I $(1-x, -y, -z)$ = 3.43 Å, O(3)–C(2)IV$(1-x, -y, -z)$ = 3.49 Å, O(3)–C(10)I$(1-x, -y, -z)$ = 3.30 Å, O(3)–C(2)I$(x, -1+y, 1+z)$ = 3.47 Å, O(4)–C(1)III$(x, y, 1+z)$ = 3.48 Å, O(4)–C(8)III(x, y, z) = 3.47 Å, O(4)–C(9) III(x, y, z) = 3.32 Å, O(6)–C(1)III$(x, y, 1+z)$ = 3.29 Å, O(6)–C(7)I$(1-x, -y, 1-z)$ = 3.44 Å. No cation–cation approaches exist below 3.34 Å, the closest intercation separation being O(2)–C(10)IV$(x, y, 1+z)$ = 3.34 Å; moreover there exists some contacts of the order of 3.5 Å between the C(5)IV–C(10)IV benzene ring with its centrosymmetrically $(0,0,-{}^{1}/_{2})$ related benzene ring, moreover the benzene rings are not superimposed.

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