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SYNTHESIS AND CRYSTAL AND MOLECULAR STRUCTURE OF μ-OXO-BIS[TRIFLUOROACETATO(p-TOLYL)IODINE]

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Crystal and molecular structure of μ-oxo-bis[trifluoroacetato(p-tolyl)iodine] (I) synthesized by a new procedure was determined by X-ray diffraction analysis. Crystals I are orthorhombic, unstable, space group Pbcn, $a = 17.684(3)$, $b = 8.453(3)$, $c = 30.560(4)$ Å, $Z = 8$. The structure of I was solved by direct and Fourier methods and refined by the full-matrix least-squares procedure in an anisotropic-isotropic approximation to $R = 0.098$ (CAD-4 automatic diffractometer, λCuK_α, 1200 observed reflections with $I \geq 2\sigma$). In molecule I, two iodine atoms have T-configuration of valence bonds with the average bond angles O–I–O 169(1) and O–I–C 86(2)°, average bond lengths I–O_μ 2.009(9), I–O_{acet} 2.269(9), and I–C_{aryl} 2.11(1) Å, and the bond angle I–O–I 118.1(5)°. In molecule I, two p-Tol substituents are directed to approximately the same side of the medium plane of the central O–I–O–I–O fragment. Crystal structure I has I···O type intra- and intermolecular nonvalent interactions (secondary bonds).

INTRODUCTION

Oxo-bridged organic compounds of trivalent iodine [1-3] are of considerable interest for organic chemistry as useful reagents and for structural chemistry as substances with poorly defined structure. This paper presents the results of an X-ray diffraction (XRD) analysis and briefly describes a new method for the synthesis of one of such compounds – μ-oxo-bis[trifluoroacetato(p-tolyl)iodine] (I). According to our knowledge, the crystal and molecular structure was investigated by XRD analysis only for two O-bridged iodoorganic compounds: μ-oxo-bis[trifluoroacetato(phenyl)iodine] (II) [2] and μ-oxo-bis[nitrato(phenyl)iodine] (III) [3]; the latter structure was determined with a low accuracy. A comparison of structural data for O-bridged binuclear compounds I, II, and III and also for the related mononuclear compounds bis(trifluoroacetato)phenyliodine (IV) [4] and bis(trifluoroacetato)pentafluorophenyliodine (V) [5] allowed us to make more reliable conclusions about their common structural features.

EXPERIMENTAL

Compound I and other compounds (with other Ar substituents) were synthesized by the reaction of aryl iodide with bis(trifluoroacetato)xenon [6] formed from XeF_2 and CF_3COOH *in situ*; the reaction was performed in dried CH_2Cl_2:

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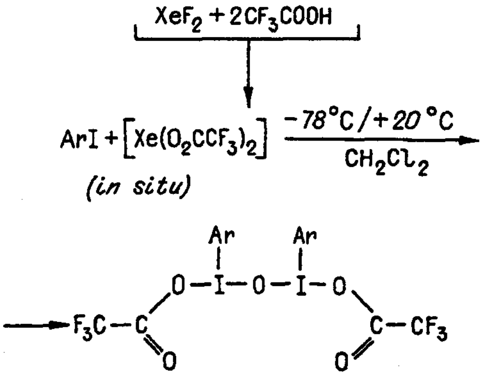


(I) et al.

where Ar = *p*-Tol (I), Ph (II), *o*-Tol, *p*-NO_2–C_6H_4–, *p*-Ph–C_6H_4–, and *p*-MeC(O)O–C_6H_4–. The new technique of synthesis and the properties of the compounds (yields 62-96%) will be published separately. Crystals I for XRD analysis were grown by two recrystallizations from the chloroform–hexane solution. Crystals I are light yellow, cloudy, and unstable, mp 84-86°C.

The XRD analysis of compound I was performed on an "Enraf–Nonius CAD4-LSI11/02-PDP11/23" automatic diffractometry system (CuK_α radiation, graphite monochromator). Crystals I are orthorhombic: $C_{18}H_{14}F_6I_2O_5$, $M = 678.11$; $a = 17.684(3)$, $b = 8.453(3)$, $c = 30.560(4)$ Å, $V = 4568(3)$ Å^3, $Z = 8$, $d_{calc} = 1.972$ g/cm^3, $\mu(CuK_\alpha) = 226$ cm^{-1}, space group *Pbcn*.

The intensities of 3020 reflections were measured in an octant of reciprocal space ($2\theta \le 105°$) using the $\omega/2\theta$ scan mode for single crystal I (0.11×0.28×0.90 mm). During the exposure time (57.9 h), the intensities of three standard reflections decreased, on the average, by 55% (the exposed single crystal decomposed by more than one half). Then the intensities of all measured reflections were corrected for crystal decay in an anisotropic approximation and also for the absorption effect by Gaussian numerical integration. After systematically extinct and incorrectly measured reflections had been discarded, the number of independent reflections became 2612; 1200 observed reflections with $I \ge 2\sigma(I)$ were used in the final calculations, and the other reflections proved to be weak (unobservable).

In the crystal structure of I, the positions of two heavy iodine atoms were determined by direct methods, and the other nonhydrogen atoms were located by *F*-syntheses on electron density maps. The positional and thermal coordinates of all nonhydrogen atoms were refined by the full-matrix least-squares procedure in an anisotropic approximation for I, F, and O atoms and in an isotropic approximation for C atoms. The H atoms were not localized on difference Fourier maps and thus were not taken into account in the calculations. In the final cycle of the refinement procedure, $|\Delta|/\sigma < 0.02$ for the 190 varied structural parameters. The final parameters of the nonhydrogen atoms of structure I are given in Table 1.

The final *R* calculated for 1200 observed reflections are: $R = 0.098$ and $R_w = 0.108$; the goodness of fit is $S = 1.74$. The high values of *R* are explained by the poor quality and instability of crystals I. In the final difference Fourier synthesis, $\Delta\rho < 1.05$ eÅ^{-3}. The weighting scheme for reflections in the least-squares procedure (weighting scheme coefficient $p = 0.10$), the *f*-curves used in the procedure, and the anomalous dispersion corrections to them ($\Delta f'$ and $\Delta f''$) are indicated, e.g., in [7]. All X-ray diffraction calculations were performed on a PDP11/23 minicomputer using the "Enraf–Nonius" SDP-PLUS program package.

DISCUSSION OF RESULTS

The molecular structure of I in a crystal is shown in Fig. 1; the bond lengths and the bond and torsion angles are given in Tables 2, 3, and 4. Below we discuss and compare the geometrical parameters and the general structure of molecule I with the molecular geometry of similar and related compounds II-V [2-5] listed in the introduction.

In molecule I, the I_1 and I_2 atoms have a *T*-configuration of valence bonds, which is characteristic for compounds of trivalent iodine [1-5]; in this case, the O–I–O bond angles are slightly smaller than 180°, and the O–I–C bond angles

TABLE 1. Coordinates ($\times 10^4$) and Isotropic Thermal Parameters of Atoms in Crystal Structure I

Atom	x	y	z	B_{eq}, Å^2
I_1	2997.1(6)	7153(1)	1273.6(3)	5.87(3)
I_2	3597.2(7)	4566(2)	474.6(4)	6.44(4)
F_1	1787(7)	9606(12)	2581(4)	11.4(4)
F_2	1918(7)	7165(17)	2744(3)	11.7(4)
F_3	875(5)	7972(19)	2495(4)	12.8(5)
F_4	4227(6)	-1148(15)	-59(4)	10.2(4)
F_5	3656(8)	279(17)	-534(3)	11.6(5)
F_6	3066(7)	-1394(17)	-155(4)	12.2(4)
O_1	3565(6)	6782(13)	715(3)	5.3(3)
O_2	2610(6)	7760(17)	1954(3)	8.5(4)
O_3	1444(6)	7653(17)	1693(4)	8.7(4)
O_4	3794(7)	2200(13)	129(3)	6.5(3)
O_5	3064(7)	642(12)	528(4)	7.0(3)
C_1	4046(8)	7696(19)	1566(5)	4.8(4)
C_2	4477(8)	6433(19)	1707(4)	4.2(3)
C_3	5194(11)	6688(26)	1881(6)	7.9(5)
C_4	5464(11)	8218(26)	1920(6)	7.7(5)
C_5	5014(13)	9499(26)	1788(6)	8.8(5)
C_6	4298(10)	9242(22)	1613(5)	6.3(4)
C_7	6248(13)	8456(30)	2135(7)	10.0(9)
C_8	4734(9)	4378(22)	685(5)	5.8(4)
C_9	4969(9)	3119(22)	943(4)	5.1(4)
C_{10}	5707(13)	3043(27)	1096(6)	9.0(6)
C_{11}	6193(10)	4292(24)	1005(6)	7.5(5)
C_{12}	5939(9)	5579(22)	762(5)	5.8(4)
C_{13}	5210(9)	5624(20)	592(4)	4.6(4)
C_{14}	7005(9)	4233(28)	1195(5)	10.0(8)
C_{15}	1888(8)	7799(19)	1988(4)	3.7(3)
C_{16}	1621(10)	8130(22)	2461(5)	6.4(4)
C_{17}	3459(11)	897(28)	217(6)	9.5(6)
C_{18}	3604(9)	-319(24)	-143(5)	6.7(5)

Note. For I, F, and O atoms, equivalent isotropic thermal parameters B_{eq} calculated from anisotropic thermal parameters b_{ij} are given.

are close to 90° or slightly smaller. With the *T*-configuration of the bonds, the iodine atom usually lies nearly exactly in the plane of the three valence-bonded atoms, as it occurs, e.g., in molecules II [2] and V [5]. For steric reasons in molecule I, which are due to the effects of crystal packing and intermolecular interactions, the I_1 atom slightly deviates from the plane of O_1, O_2, and C_1 atoms by 0.033(1) Å, and the I_2 atom significantly deviates from the plane of O_1, O_4, and C_8 atoms by 0.161(1) Å. Pronounced deviations (for the same reasons) of two iodine atoms from the planes of the three valence-bonded atoms (by 0.140(3) and 0.105(3) Å) are also observed in molecule III [3].

In molecule I, the lengths of the I_1–O_1 and I_2–O_1 "bridging" bonds (average 2.009(9) Å), of the I_1–O_2 and

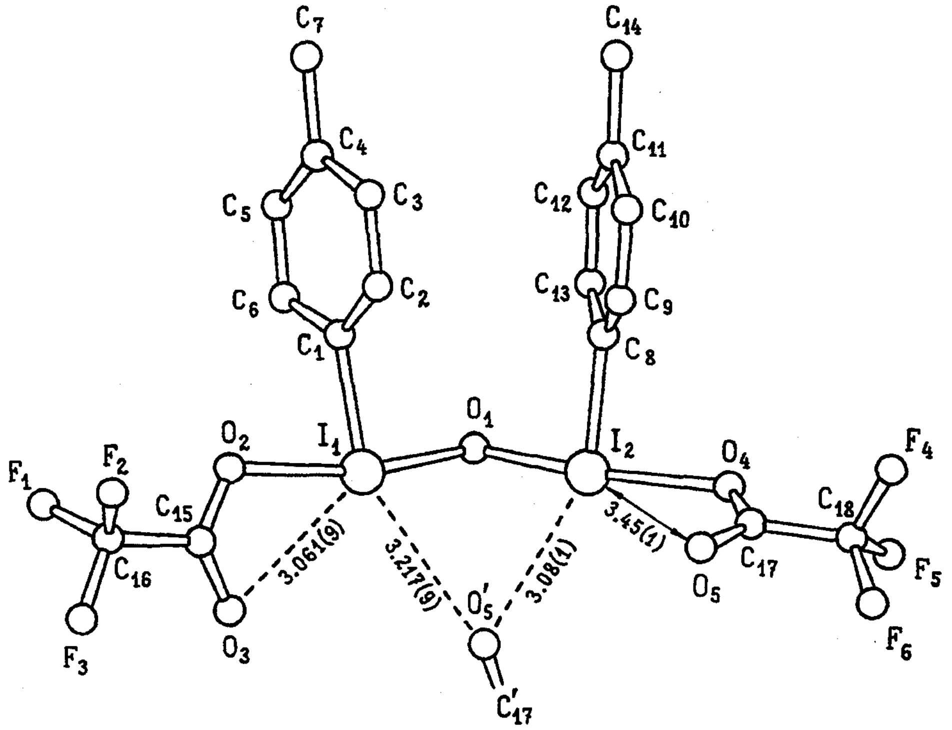


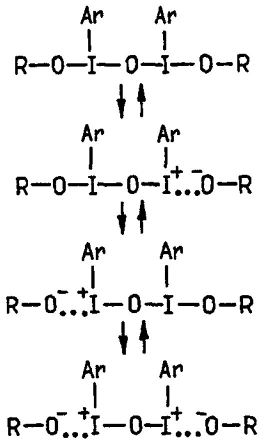
Fig. 1. Molecular structure of compound I in a crystal. The dashed lines denote the $I\cdots O$ nonvalent interactions (secondary bonds) with indications of their lengths in Å.

I_2–O_4 "outer" bonds (average 2.269(9) Å), and of the I_1–C_1 and I_2–C_8 bonds (average 2.11(1) Å) are close, within experimental error, to the average lengths of similar bonds in molecules II (2.017(7), 2.272(8), and 2.067(8) Å) [2] and III (2.00(5), 2.32(3), and 2.06(2) Å) [3]. For molecules I, II, and III, the lengths of the bridging I–O bonds and the I–C bond lengths are close to the standard lengths of covalent bonds (I–O 2.00 and I–C(Ar) 2.08 Å) calculated from the covalent atomic radii with corrections for bond polarity included [8]; the lengths of the outer I–O bonds are

TABLE 2. Bond Lengths *d* (Å) in Molecule I

Bond	*d*	Bond	*d*
I_1–O_1	2.005(9)	I_2–O_1	2.013(10)
I_1–O_2	2.250(8)	I_2–O_4	2.288(10)
I_1–C_1	2.109(13)	I_2–C_8	2.117(15)
O_2–C_{15}	1.28(1)	O_4–C_{17}	1.28(2)
$O_3=C_{15}$	1.20(1)	$O_5=C_{17}$	1.20(2)
C_1–C_2	1.38(2)	C_8–C_9	1.39(2)
C_1–C_6	1.39(2)	C_8–C_{13}	1.38(2)
C_2–C_3	1.39(2)	C_9–C_{10}	1.39(2)
C_3–C_4	1.38(2)	C_{10}–C_{11}	1.39(2)
C_4–C_5	1.40(2)	C_{11}–C_{12}	1.39(2)
C_4–C_7	1.55(2)	C_{11}–C_{14}	1.55(2)
C_5–C_6	1.39(2)	C_{12}–C_{13}	1.39(2)
C_{15}–C_{16}	1.55(2)	C_{17}–C_{18}	1.53(3)
F_1–C_{16}	1.33(2)	F_4–C_{18}	1.33(2)
F_2–C_{16}	1.30(2)	F_5–C_{18}	1.30(2)
F_3–C_{16}	1.33(2)	F_6–C_{18}	1.32(2)

significantly greater than those of the bridging I–O bonds and than the above standard length of the covalent I–O bond. The lengthening of the outer I–O bonds (with acid substituents) found in molecules I, II [2], and III [3] may be explained by the increased ionicity of these bonds, i.e., by the small contributions of three possible iodonium structures — B, C, and D — to covalent molecular structure A:



(A)

(B)

(C)

(D)

In molecules IV [4] and V [5], the I–O valence bonds are also (but to a lesser extent) longer than the standard length of 2.00 Å (average I–O 2.162(4) Å in IV and 2.139(7) Å in V).

The bond angle at the bridging oxygen atom I_1–O_1–I_2 in molecule I [118.1(5)°] is slightly smaller than that in molecules II [120.8(4)°] [2] and III (123.7(6)°) [3]. The $I_1 \cdots I_2$ intramolecular distance in I [3.446(2) Å] is 0.06-0.09 Å lesser than that in molecules II [3.505(1) Å] and III [3.536(4) Å] and much lesser than the doubled van der Waals radius of the iodine atom [3.96 Å] [9].

The two benzene rings in molecule I are planar within ±0.02(2) Å for the C atoms and have normal bond lengths and angles within the experimental error. Their bonds with the Me substituents C_4–C_7 and C_{11}–C_{14} are slightly longer than the mean statistical length of the C(Ar)–CH_3 single bond (1.506(11) Å [10]). In molecule I, the I_1 and I_2 atoms significantly deviate from the rms planes of the benzene rings bonded to them (by 0.112(1) and 0.135(1) Å, respectively, toward the O_1 bridging atom). Due to the crystal packing effect in structure I, the rotation angles of the two benzene rings around the I_1–C_1 and I_2–C_8 bonds differ, on the average, by 44° (torsion angles $O_1I_1C_1C_2$, $O_1I_2C_8C_9$, etc., Table 4).

TABLE 3. Bond Angles ω (deg) in Molecule I

Angle	φ	Angle	φ	Angle	φ	Angle	φ
$O_1I_1O_2$	167.3(3)	$C_5C_4C_7$	122(2)	$O_1I_2O_4$	170.4(4)	$C_{12}C_{11}C_{14}$	122(2)
$O_1I_1C_1$	87.4(4)	$C_4C_5C_6$	120(2)	$O_1I_2C_8$	89.2(5)	$C_{11}C_{12}C_{13}$	121(1)
$O_2I_1C_1$	80.0(4)	$C_1C_6C_5$	119(1)	$O_4I_2C_8$	86.0(5)	$C_8C_{13}C_{12}$	118(1)
$I_1O_1I_2$	118.1(5)	$O_2C_{15}O_3$	126(1)	–	–	$O_4C_{17}O_5$	126(2)
$I_1O_2C_{15}$	112.5(9)	$O_2C_{15}C_{16}$	112(1)	$I_2O_4C_{17}$	126(1)	$O_4C_{17}C_{18}$	111(2)
$I_1C_1C_2$	117(1)	$O_3C_{15}C_{16}$	121(1)	$I_2C_8C_9$	121(1)	$O_5C_{17}C_{18}$	123(2)
$I_1C_1C_6$	122(1)	$F_1C_{16}F_2$	108(1)	$I_2C_8C_{13}$	117(1)	$F_4C_{18}F_5$	109(1)
$C_2C_1C_6$	121(1)	$F_1C_{16}F_3$	107(1)	$C_9C_8C_{13}$	121(1)	$F_4C_{18}F_6$	104(1)
$C_1C_2C_3$	120(1)	$F_2C_{16}F_3$	107(1)	$C_8C_9C_{10}$	121(2)	$F_5C_{18}F_6$	107(1)
$C_2C_3C_4$	119(2)	$F_1C_{16}C_{15}$	111(2)	$C_9C_{10}C_{11}$	119(2)	$F_4C_{18}C_{17}$	111(2)
$C_3C_4C_5$	120(2)	$F_2C_{16}C_{15}$	113(2)	$C_{10}C_{11}C_{12}$	120(2)	$F_5C_{18}C_{17}$	114(2)
$C_3C_4C_7$	118(2)	$F_3C_{16}C_{15}$	111(2)	$C_{10}C_{11}C_{14}$	118(2)	$F_6C_{18}C_{17}$	111(2)

TABLE 4. Main Torsion Angles τ (deg) in Molecule I

Angle	τ	Angle	τ	Angle	τ	Angle	τ
$O_2I_1O_1I_2$	120.8	$I_1O_2C_{15}O_3$	-5.3	$O_4I_2O_1I_1$	-165.3	$I_2O_4C_{17}O_5$	-10.4
$C_1I_1O_1I_2$	113.7	$I_1O_2C_{15}C_{16}$	177.0	$C_8I_2O_1I_1$	-105.5	$I_2O_4C_{17}C_{18}$	167.5
$O_1I_1O_2C_{15}$	166.0	$F_1C_{16}C_{15}O_2$	69	$O_1I_2O_4C_{17}$	175.8	$F_4C_{18}C_{17}O_4$	89
$C_1I_1O_2C_{15}$	173.2	$F_2C_{16}C_{15}O_2$	-53	$C_8I_2O_4C_{17}$	115.8	$F_5C_{18}C_{17}O_4$	-34
$O_1I_1C_1C_2$	-82.8	$F_3C_{16}C_{15}O_2$	-172	$O_1I_2C_8C_9$	127.5	$F_6C_{18}C_{17}O_4$	-156
$O_1I_1C_1C_6$	96.7	$F_1C_{16}C_{15}O_3$	-109	$O_1I_2C_8C_{13}$	-46.4	$F_4C_{18}C_{17}O_5$	-93
$O_2I_1C_1C_2$	98.7	$F_2C_{16}C_{15}O_3$	129	$O_4I_2C_8C_9$	-60.8	$F_5C_{18}C_{17}O_5$	144
$O_2I_1C_1C_6$	-81.7	$F_3C_{16}C_{15}O_3$	10	$O_4I_2C_8C_{13}$	125.3	$F_6C_{18}C_{17}O_5$	22

The two trifluoroacetate fragments of molecule I have planar (within ±0.01(2) Å) frameworks of O_2, O_3, C_{15}, C_{16} and O_4, O_5, C_{17}, C_{18} atoms. The bond lengths and angles of these trifluoroacetate fragments are close, within the experimental error, to those found for molecules II [2], IV [4], and V [5], which contain the same fragments. Note that the O_2–C_{15} and O_4–C_{17} bond lengths in molecule I (as in molecules II, IV, and V) are appreciably smaller (1.28(1) Å) than the mean statistical (for the C*–O–C(=O)–C* fragments) length of the O–C(sp^2) bond, which equals 1.336(14) Å [10]. Probably, these bonds are shortened because molecular structure I has small contributions from the above-mentioned iodonium structures B, C, and D, which contain the CF_3COO^- anion with equal lengths of the two formal $C\overset{\cdots}{-}O$ sesqui-bonds.

The two trifluoroacetate fragments of molecule I have different conformations and orientations. The rotation angles of the CF_3 groups around the C_{15}–C_{16} and C_{17}–C_{18} bonds differ, on the average, by 16° (F–C–C–O torsion angles, Table 4). Possibly, both CF_3 groups in compound I are disordered due to the reorientation, as indicated by the high thermal vibration parameters of the F atoms of these groups (B_{eq}, Table 1). The plane of the O_2, O_3, C_{15}, and C_{16} atoms of the first trifluoroacetate fragment in molecule I makes a small angle of 8.5° with the O_1, O_2, C_1 coordination plane of the neighboring I_1 atom, and the plane of the O_4, O_5, C_{17}, and C_{18} atoms of the second trifluoroacetate fragment makes a large angle of 75.7° with the O_1, O_4, C_8 coordination plane of the neighboring I_2 atom. A similar difference between the angles of these planes is observed for molecule II.

It should be noted specially that in their crystal structures related molecules I, II [2], and III [3] have very different conformations and roughly approximate symmetries. The greatest conformational difference is orientation of the two aryl substituents relative to the averaged plane of the central molecular fragment O–I–O–I–O and relative to the $I_1 - - - I_2$ intramolecular line. The conformation relative to the latter is characterized best by the C_{Ar}–$I_1 - - - I_2$–C_{Ar} pseudotorsion angle (C_1–$I_1 - - - I_2$–C_8 in compound I), which is 8.1, 51.6, and 101.5° in molecules I, II, and III, respectively. As we can see, both aryl substituents in molecule I are directed to approximately the same side of the averaged plane of the O–I–O–I–O fragment. The same takes place in molecule II, but here the aryl substituents have very different orientations. In molecule III, the aryl substituents lie on different sides of the averaged plane of the O–I–O–I–O fragment. If we neglect noticeable differences between the rotation angles of substituents around the I_1–C_1 and I_2–C_8 bonds, on the one hand, and the I_1–O_2 and I_2–O_4 bonds, on the other, molecule I in a crystal has a roughly approximate symmetry plane C_s passing through the O_1 bridging atom perpendicular to the $I_1 - - - I_2$ line, and molecule III [3] has an approximate twofold symmetry axis C_2 also passing through the bridging oxygen atom. Molecule II [2] in a crystal is asymmetric even in a rough approximation. These facts suggest high conformational flexibility of O-bridged iodoorganic compounds.

As other previously examined crystal structures of trivalent iodine (II-V [2-5] and others), structure I has substantially shortened $I \cdots O$ intra- and intermolecular contacts, which are usually interpreted as secondary bonds or specific nonvalent interactions. In structure I, the I_1 atom has an intramolecular $I_1 \cdots O_3$ contact of 3.061(9) Å and an intermolecular $I_1 \cdots O_5'$ contact of 3.217(9) Å, and the I_2 atom has one shortened intermolecular $I_2 \cdots O_5'$ contact of

3.08(1) Å; symmetric transformation of the O_5' atom is $O_5' = O_5(1/2-x, 1/2+y, z)$. These contacts are smaller than the sum of the van der Waals radii of the I and O atoms (3.50 Å) [9] by 0.28-0.44 Å. The interatomic angles involving the above shortened contacts in compound I are: $O_2-I_1\cdots O_3$ 46.2(3), $O_1-I_1\cdots O_5'$ 68.1(3), $O_2-I_1\cdots O_5'$ 124.6(3), $O_3\cdots I_1\cdots O_5'$ 80.1(3), $O_1-I_2\cdots O_5'$ 71.3(3), $O_4-I_2\cdots O_5'$ 115.3(3), and $I_1\cdots O_5'\cdots I_2$ 66.3(4)°. In crystal structure I, the $I_1\cdots O_5'$ and $I_2\cdots O_5'$ intermolecular secondary bonds connect molecules I bonded by the glide plane *b* into infinite chains (along the *y* axis). Due to the participation of the O_5 atom in the $I_1\cdots O_5'$ and $I_2\cdots O_5'$ intermolecular interactions and the crystal packing effect in molecule I, the second trifluoroacetate substituent markedly deviates from the I_2 atom, thus increasing the $I_2\cdots O_5$ intramolecular distance to 3.45(1) Å. Due to this, the $I_2O_4C_{17}$ bond angle is much larger (by 13.5°) than the other bond angle, $I_1O_2C_{15}$ (Table 3), and the possible intramolecular secondary bond (nonvalent interaction) $I_2\cdots O_5$ in I is absent.

Except for the shortened $I\cdots O$ intermolecular contacts mentioned above, all short intermolecular contacts in crystal structure I are close to the sums of the corresponding van der Waals radii of atoms.

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