Drastic conformational changes occur in the diphenyl phosphate monoanion as it forms the copper complex. The torsion angles ω and ω'^* of $61 \cdot 4$ (4) and $71 \cdot 9$ (4)° in the monoanion are $-179 \cdot 8$ (2) and $77 \cdot 0$ (2)° upon coordination in the title compound.

Uncharged, discrete [Cu(ImH)$_4$(DPhP)$_2$] complex molecules are connected via hydrogen bonds from the pyrrole N atoms [N(2), N(4)] in the imidazole rings to the diphenyl phosphate O(2) atoms of neighbouring molecules, forming a two-dimensional network. Lengths and angles are $2 \cdot 837$ (4) Å, $163°$, and $2 \cdot 756$ (3) Å, $170°$, for the $N(2)[H(2)]\cdots O(2)(x-1, \ y, \ z)$ and $N(4)[H(6)]\cdots O(2)(-x+1, \ -y, \ -z+1)$ hydrogen bonds, respectively. A view of the unit-cell contents along **b**, with the hydrogen-bonding scheme, is shown in Fig. 2.

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* Torsion angles O(3)—P—O(4)—C(21) and O(4)—P—O(3)—C(11) in the title compound.

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Crystal Structures of Polyiodide Salts and Molecular Complexes. IX.* Nonasodium Octa(p-toluenesulphonate) Triiodide Dihydrate, Na$_9$(C$_7$H$_7$O$_3$S)$_8$I$_3$.2H$_2$O

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Abstract. $M_r = 1993 \cdot 24$, triclinic, $P\bar{1}$, Dirichlet reduced cell used in structure analysis has $a = 19 \cdot 214$ (7), $b = 18 \cdot 526$ (7), $c = 10 \cdot 946$ (5) Å, $\alpha = 88 \cdot 69$ (8), $\beta = 81 \cdot 52$ (8), $\gamma = 87 \cdot 36$ (8)° [Delaunay reduced cell has $a = 18 \cdot 526$ (7), $b = 19 \cdot 214$ (7), $c = 10 \cdot 946$ (5) Å, $\alpha = 98 \cdot 48$ (8), $\beta = 91 \cdot 31$ (8), $\gamma = 92 \cdot 64$ (8)°], $V = 3849$ (1) Å^3, $Z = 2$, $D_m = 1 \cdot 69$, $D_x = 1 \cdot 72$ g cm^{-3}, Mo $K\alpha$, $\lambda = 0 \cdot 71069$ Å, $\mu = 14 \cdot 3$ cm^{-1}, $F(000) = 1980$, $T = 298$ K, $R_F = 0 \cdot 089$ for 7975 measured reflections with $F_o > 1 \cdot 5\sigma(F_o)$. There are alternating cationic (Na$^+$, p-toluenesulphonates, waters) and anionic (non-interacting triiodides) sheets parallel to (010).

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The methyl groups of the p-toluenesulphonate layers point away from the Na, O of the cationic layers, forming hydrophilic and hydrophobic layers. The overall arrangement of moieties in the crystal provides for local neutralization of charge. The compound is classified as a triiodide salt.

Introduction. Kastle & Hill (1894) prepared sodium and potassium salts containing both benzenesulphonate and triiodide anions. These are possible analogues to the various compounds of the herapathite series (Herapath, 1857; Jorgensen, 1877), which have quinine as cation and both sulphate (or selenate) and polyiodide anions. The structure of herapathite is not known although a preliminary crystallographic study has been reported (West, 1937).

We have repeated some of the preparations of Kastle & Hill and extended them by using p-toluenesulphonic acid in addition to benzenesulphonic acid. One of the sodium p-toluenesulphonate triiodides (formula in the title) gave particularly large crystals of acceptable diffraction quality and we have determined its structure.

Experimental. Crystals were prepared as follows: saturated aqueous solutions of $NaI+I_2$ (1:1) and p-toluenesulphonic acid were mixed, with immediate precipitation of small dark crystals. These were filtered, dissolved in a minimum quantity of water and the solution left to stand in air. Flat reddish-brown plates were obtained, the largest being $\sim 20 \times 2 \times 0.5$ mm. Thermal decomposition prevented determination of the melting point. Chemical analysis gave (found, calculated for $C_{56}H_{60}I_3Na_9O_{26}S_8$): C 34·3, 33·74; H 2·95, 3·03; O 20·67, 20·87; S 13·64, 12·87; I 18·06, 19·10; Na 9·88, 10·38. Density by flotation in a mixture of toluene and 1,1,2,2-tetrabromoethane. Small cube, 0·2 mm edge, sealed into capillary; cell dimensions from 24 reflections in θ range 2·6–7·5°; intensities of 11 617 reflections measured $(-22 \le h \le 22;$ $-21 \le k \le 21;$ $0 \le l \le 9)$, Philips PW 1100/20 four-circle diffractometer, graphite-monochromated Mo $K\alpha$ radiation, $\omega/2\theta$ measurement mode, no absorption correction, scan range $5 < 2\theta < 50°$, scan speed $3°\ \omega\ \text{min}^{-1}$, scan width $1·3°\ \omega$, $\sim 5\%$ variation of standard reflections ($\bar{2}1\bar{2}$; 307; $\bar{2}30$) during course of measurements ascribed to crystal movement, no evidence of radiation damage; $\sigma(F) = \sigma(F^2)/2F$ for $F^2 > \sigma(F^2)$ or $[1/2\sigma(F^2)]^{1/2}$ if $\sigma(F^2) > F^2 > -\sigma(F^2)$; structure solved by Patterson methods and refined (on F) using *SHELX77* (Sheldrick, 1977) including atomic scattering factors provided (*International Tables for X-ray Crystallography*, 1974), convergence attained at $R_F = 0·089$ after anisotropic refinement of all non-hydrogen atoms (hydrogens not included at any stage). Block-matrix refinement and unit weights used during final least-squares cycles because of large number of variables (922); 7975 reflections with $F_o > 1·5\ \sigma(F_o)$ used in last cycles, av. Δ/σ about 0·3 in final cycle, final difference synthesis showed $\Delta\rho$ excursions $-1·3$ to $+1·7$ e Å^{-3}, mostly in the vicinity of the I atoms. Atomic parameters are given in Table 1.*

Discussion. The overall crystal structure (Fig. 1) can be described in terms of alternating cationic and anionic layers parallel to (010). The repeat unit of the cationic layers contains nine Na$^+$ cations, eight p-toluene-

Table 1. *Atomic positional parameters* $(\times 10^5$ *for* I, $\times 10^4$ *for the rest) and equivalent isotropic thermal parameters* $(\text{Å}^2 \times 10^4$ *for* I, $\text{Å}^2 \times 10^3$ *for the rest)*

$$U_{eq} = \tfrac{1}{3}\textstyle\sum_i\sum_j U_{ij}a_i^*a_j^*(\mathbf{a}_i.\mathbf{a}_j).$$

	x	y	z	U_{eq}
I(1)	50000	50000	0	839(5)
I(2)	50496(8)	34513(9)	6543(14)	1076(5)
I(3)	50000	0	50000	843(5)
I(4)	43700(10)	1194(11)	27299(17)	1355(6)
Na(1)	1016(2)	832(2)	3534(4)	36(1)
Na(2)	807(2)	835(2)	-1385(4)	38(1)
Na(3)	-146(2)	1140(2)	1199(4)	34(1)
Na(4)	227(2)	2677(2)	3587(4)	35(1)
Na(5)	137(3)	2685(3)	-1454(4)	49(1)
Na(6)	233(2)	3953(2)	1148(4)	36(1)
Na(7)	932(3)	5830(2)	976(3)	39(1)
Na(8)	-170(2)	5922(2)	4196(4)	35(1)
Na(9)	172(2)	8901(2)	3935(4)	35(1)
OW(1)	2264(5)	826(5)	3174(10)	68(3)
OW(2)	2204(6)	5957(5)	775(10)	74(3)
S(1)A	1082(2)	2123(2)	5955(5)	29(1)
O(1)A	783(4)	1857(4)	4908(7)	34(2)
O(2)A	917(5)	1651(5)	7026(8)	50(3)
O(3)A	867(5)	2866(4)	6229(9)	49(2)
C(1)A	2009(7)	2079(7)	5526(13)	36(3)
C(2)A	2442(8)	1656(8)	6211(15)	61(5)
C(3)A	3182(10)	1654(10)	5814(18)	75(6)
C(4)A	3439(10)	2064(11)	4781(19)	82(7)
C(5)A	2992(9)	2466(10)	4105(17)	73(6)
C(6)A	2276(8)	2474(8)	4488(15)	54(4)
C(7)A	4262(14)	2067(14)	4373(24)	121(9)
S(1)B	1047(1)	4495(2)	3293(3)	28(1)
O(1)B	989(4)	4705(4)	2029(7)	35(2)
O(2)B	756(5)	3793(4)	3615(9)	48(2)
O(3)B	737(4)	5022(5)	4197(8)	39(2)
C(1)B	1947(6)	4418(6)	3402(12)	30(3)
C(2)B	2149(6)	4430(7)	4607(13)	40(3)
C(3)B	2861(7)	4378(7)	4715(14)	48(4)
C(4)B	3363(8)	4337(8)	3683(16)	55(4)
C(5)B	3167(8)	4332(8)	2502(16)	59(5)
C(6)B	2461(7)	4367(8)	2367(14)	50(4)
C(7)B	4167(10)	4293(10)	3842(18)	81(6)
S(1)C	9157(1)	443(1)	3741(3)	26(1)
O(1)C	9661(4)	989(4)	3889(8)	36(2)
O(2)C	9216(4)	239(4)	2445(7)	31(2)
O(3)C	9227(4)	-183(4)	4538(7)	36(2)
C(1)C	8310(8)	835(6)	4150(11)	27(3)
C(2)C	7734(7)	384(7)	4288(13)	50(4)
C(3)C	7055(8)	704(8)	4528(15)	61(5)
C(4)C	6932(8)	1448(8)	4656(15)	56(4)
C(5)C	7530(7)	1886(7)	4513(13)	48(4)
C(6)C	8210(6)	1582(7)	4261(12)	39(3)
C(7)C	6203(10)	1785(10)	4897(18)	90(6)
S(1)D	9105(1)	410(1)	-1230(3)	25(1)
O(1)D	9552(4)	973(4)	-923(8)	34(2)
O(2)D	9176(4)	298(4)	2538(7)	32(2)
O(3)D	9223(4)	-261(4)	-537(7)	33(2)
C(1)D	8221(6)	681(6)	-727(12)	31(3)
C(2)D	7998(7)	799(7)	502(13)	42(3)
C(3)D	7288(8)	980(8)	895(15)	59(4)
C(4)D	6799(8)	1014(8)	43(16)	59(5)
C(5)D	7042(8)	902(8)	-1191(16)	59(4)
C(6)D	7738(7)	718(7)	1594(13)	44(4)
C(7)D	6036(11)	1217(11)	516(19)	97(7)
S(1)E	9043(1)	2811(1)	1422(3)	28(1)
O(1)E	9231(4)	3090(4)	183(7)	37(2)
O(2)E	9494(4)	3091(5)	2246(8)	43(3)
O(3)E	9032(4)	2032(4)	1471(9)	48(2)
C(1)E	8191(6)	3151(6)	1952(11)	31(3)
C(2)E	7613(7)	2843(7)	1575(13)	49(4)
C(3)E	6933(8)	3122(8)	2012(15)	56(4)
C(4)E	6837(8)	3690(8)	2828(14)	53(4)
C(5)E	7425(7)	4014(8)	3172(14)	53(4)
C(6)E	8098(7)	3729(7)	2760(13)	43(3)
C(7)E	6091(10)	3989(10)	3301(18)	86(6)
S(1)F	9117(1)	5465(1)	1590(3)	28(1)
O(1)F	9223(4)	5310(4)	2851(7)	36(2)
O(2)F	9559(4)	6050(4)	1049(8)	37(2)
O(3)F	9220(4)	4827(4)	823(7)	37(2)
C(1)F	8230(5)	5771(6)	1646(11)	28(3)
C(2)F	7751(7)	5373(7)	1141(13)	51(4)
C(3)F	7041(7)	5598(8)	1289(14)	52(4)
C(4)F	6798(7)	6202(7)	1957(13)	46(4)
C(5)F	7293(8)	6615(9)	2427(15)	68(5)
C(6)F	7999(7)	6396(7)	2273(13)	52(4)
C(7)F	6003(9)	6409(10)	2233(17)	78(6)

Table 1 (cont.)

	x	y	z	U_{eq}
S(1)G	1105 (1)	2163 (1)	880 (3)	26 (1)
O(1)G	852 (4)	1774 (4)	2028 (7)	35 (2)
O(2)G	766 (4)	1904 (4)	−135 (7)	31 (2)
O(3)G	1020 (4)	2938 (4)	1026 (9)	46 (2)
C(1)G	2011 (5)	1959 (6)	512 (11)	29 (3)
C(2)G	2490 (8)	2485 (8)	626 (14)	60 (4)
C(3)G	3206 (10)	2320 (10)	383 (18)	82 (6)
C(4)G	3454 (9)	1637 (9)	−48 (16)	72 (5)
C(5)G	2964 (8)	1112 (8)	−144 (15)	65 (5)
C(6)G	2252 (7)	1271 (7)	148 (13)	51 (4)
C(7)G	4241 (12)	1457 (12)	−415 (21)	112 (8)
S(1)H	9106 (1)	2874 (1)	−3481 (3)	29 (1)
O(1)H	9359 (4)	3039 (4)	−4753 (7)	37 (2)
O(2)H	9368 (4)	3383 (4)	−2659 (7)	35 (2)
O(3)H	9244 (4)	2146 (4)	−3082 (8)	40 (2)
C(1)H	8187 (6)	3026 (6)	−3275 (11)	30 (3)
C(2)H	7754 (7)	2566 (7)	−2528 (14)	53 (4)
C(3)H	7037 (8)	2696 (9)	−2337 (15)	66 (5)
C(4)H	6733 (7)	3287 (8)	−2960 (14)	54 (4)
C(5)H	7168 (7)	3743 (8)	−3694 (14)	52 (4)
C(6)H	7890 (7)	3616 (7)	−3864 (13)	45 (3)
C(7)H	5926 (10)	3437 (10)	−2793 (17)	85 (6)

sulphonate anions and two water molecules, and thus has a net positive charge of one. The oxygens of the anions and waters coordinate the cations in a somewhat irregular fashion (see below). The methyl groups of the p-toluenesulphonates point outwards in both directions, away from the sodium–oxygen interior of the cationic layers; thus these layers have a hydrophilic interior enclosed within two hydrophobic outer surfaces. The triiodides of the interleaving anionic layer are located at independent centres of symmetry at $\frac{1}{2},0,\frac{1}{2}$ and $\frac{1}{2},\frac{1}{2},0$; there is no direct interaction between them as the closest pair of iodines is separated by 7·88 Å.

The structural principle governing this arrangement is 'local neutralization of charge', as set out by Powell & Wait (1958) in their description of (succinimide)₂–tetramethylammonium bromide. The same principle applies in the structures of (xanthotoxin)₂KI₃ and (glycine)₄KI₃ (Herbstein & Kapon, 1980). A table of the sodium–oxygen coordination has been deposited. Coordination numbers 5 for Na(2), Na(4), Na(6), 6 for Na(1), Na(3), Na(5), Na(7), Na(9) and 7 for Na(8) are found when distances in the range 2·2–2·8 Å are included. Sixfold coordination is most nearly octahedral when the ligating atoms all come from different moieties, and appreciable distortion occurs when two oxygens are supplied by one p-toluenesulphonate anion, as is found for five of the nine Na⁺ ions. There is no example of all three oxygens of a sulphonate group coordinating with a particular sodium ion. The present results are in agreement with Na⁺···O coordination arrangements found in some other structures studied recently, e.g. {Fe₄S₄[S(CH₂)₂COO]₄}⁶⁻·[Na₅N(C₄H₉)₄]⁶⁺·5C₅H₉NO (Carrell, Glusker, Job & Bruice, 1977), Na₆{C₆(SO₃)₆}·8H₂O (Chetkina & Sobolev, 1977). However, a coordination number of eight is also sometimes found, as in NaCH₃SO₃ (Wei & Hingerty, 1981).

Mean dimensions for the p-toluenesulphonate anion have been obtained by averaging over the eight independent anions, with the additional simplifying assumptions (whose applicability has been checked) that the several independent S–O and C–C distances cannot be distinguished. We find: $d(\text{S–O}) = 1\cdot454\ (8,10,2)$; $d(\text{C–S}) = 1\cdot758\ (11,8,3)$; $d(\text{C–C}) = 1\cdot393\ (22,15,3)$; $d(\text{C–CH}_3) = 1\cdot540\ (27,28,10)$ Å; $\text{O–S–O} = 111\cdot6\ (5,10,2)$; $\text{C–S–O} = 107\cdot1\ (5,9,2)°$. The values in brackets are, respectively, a typical experimental e.s.d., the sample e.s.d. and the e.s.d. of the mean. These values agree well with those found in related compounds; for example, we note that $d(\text{S–O}) = 1\cdot458\ (1)$ Å and $d(\text{S–CH}_3) = 1\cdot752\ (2)$ Å in NaCH₃SO₃ and $d(\text{C–S}) = 1\cdot769\ (2)$ Å in calcium naphthionate octahydrate (Brown, Ehrenberg & Yadav, 1984). The length of S^{VI}–C(ring) in 120 compounds containing the (aryl)SO₂NXX' fragment has a grand mean of 1·761 Å and is independent of aryl-group rotation about the S–C bond (Kálmán, Czugler & Argay, 1981); this bond length is in agreement with the value of 1·754 Å suggested by Kálmán (1973) for the S^{VI}–C(sp^2) single bond. In Na₆{C₆(SO₃)₆}·8H₂O (Chetkina & Sobolev, 1977) $d(\text{C–S}) = 1\cdot835$ Å (averaged) and this considerably longer value has been ascribed to the tilting of the C–S bonds out of the benzene-ring plane because of the intramolecular overcrowding.

The independent triiodide ions are both centrosymmetric and the values of $d(\text{I–I})$ [2·942 (2) and 2·920 (2) Å] fall into the accepted range (Tebbe, 1977). The thermal vibration factors of the outer iodines are appreciably larger than those of the centre iodines (Table 1; U_{ij} deposited), and indeed all the iodine U_{ij} values are appreciably larger than those of the other atoms in the crystal.
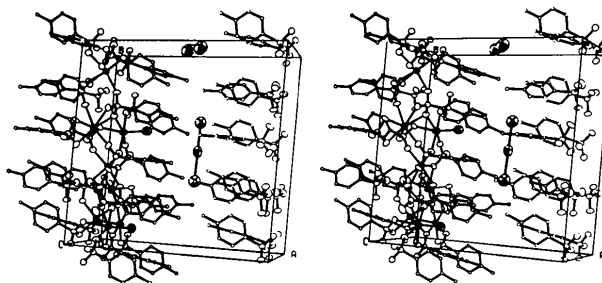
Fig. 1. ORTEP (Johnson, 1965) stereodiagram of the crystal structure. The vibration ellipsoids are 50% probability ellipsoids. Na···O links are shown for $d(\text{Na–O}) < 2\cdot8$ Å.

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The One-Dimensional Chain Structure of (1,2-Benzoquinone dioxime)diiodoplatinum(II), $[PtI_2(C_6H_6N_2O_2)]$

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Abstract. $M_r = 587.02$, triclinic, $P\bar{1}$, $a = 7.325$ (4), $b = 7.658$ (3), $c = 10.722$ (5) Å, $\alpha = 70.06$ (3), $\beta = 79.62$ (4), $\gamma = 76.12$ (4)°, $V = 545.7$ Å³, $Z = 2$, $D_x = 3.57$ g cm⁻³, Mo $K\alpha$, $\lambda = 0.71069$ Å, $\mu = 176$ cm⁻¹, room temperature, $F(000) = 512$, $R = 0.079$ for 2023 observed independent reflections. The planar complex molecules are stacked in a columnar fashion along **a**. Adjacent complexes within a stack are rotated by 180° along the stacking axis. An infinite Pt—Pt—Pt zigzag chain results, with Pt—Pt separations of 3.673 (2) and 3.707 (2) Å and a Pt—Pt—Pt angle of 166.0 (1)°.

Introduction. 1,2-Benzenedione dioxime (1,2-benzoquinone dioxime, H₂bqd) is known as an excellent ligand which readily forms bis-chelated transition-metal complexes, especially with the dipositive metal ions of the Ni triad (Mégnamisi-Bélombé, 1978). In the special case of the Pt complexes, this ligand was observed to occur exclusively as the monoanion Hbqd⁻. Though

adducts of the form $CuX_2.(H_2bqd)$ ($X = Cl^-, Br^-$) have been reported (Mégnamisi-Bélombé, 1979), no analogues of Pt halides are known. This paper describes the synthesis and structure of $PtI_2(H_2bqd)$. Its molecular structure closely resembles those of materials such as $PtCl_2(bpy)$ (bpy = 2,2′-bipyridyl) which are suspected to be potential antitumour agents, based on the similarity of their structural characteristics to those of the anticancer drugs cis-dichloro(1,2-diaminoethane)-platinum(II) and cis-diamminedichloroplatinum(II) (Keller, 1982).

Experimental. Compound obtained as the product of a redox reaction whose mechanism is not clear. Excess molecular iodine (~2 g) dissolved in 400 ml acetone, 270 mg Pt(Hbqd)₂I₂ (Mégnamisi-Bélombé, 1978; Pritzkow, 1976) added. Solution of 320 mg LiTCNQ† in 230 ml acetone filtered into this mixture. Mixture refluxed for 5 h, filtered hot, red-brown filtrate evaporated slowly over 10 d at room temperature.

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† TCNQ = 7,7,8,8-tetracyano-*p*-quinodimethane = 2,2′-(2,5-cyclohexadiene-1,4-diylidene)bis(propanedinitrile).