hybridization of the C atoms concerned, by the junction, as indicated by the bond lengths and the bond angles around each.

Views of the unit cells are presented in Fig. 2. Short O4···O5 intermolecular distances of 2·726 (4) Å in (1) and 2·756 (4) Å in (2) are observed, indicating hydrogen bonding between the solvent molecules and the dimers.

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Structures of 2,2′,2″-Trimethoxytriphenylamine and 2,2′,2″-Trihydroxytriphenylammonium Benzenesulfonate

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Abstract. 2,2′,2″-Trimethoxytriphenylamine, (I): $C_{21}H_{21}NO_3$, $M_r = 335{\cdot}43$, cubic, $Pa3$, $a = 15{\cdot}4265\,(10)$ Å, $V = 3671{\cdot}2$ Å^3, $Z = 8$, $D_x = 1{\cdot}214$ g cm^{-3}, $\lambda(\mathrm{Mo}\;K\alpha) = 0{\cdot}71069$ Å, $\mu = 0{\cdot}756$ cm^{-1}, $F(000) = 1424$, $T = 295$ K, $R = 0{\cdot}037$ for 582 observed reflections ($> \sigma_F$) and 104 parameters. 2,2′,2″-Trihydroxytriphenylammonium benzenesulfonate, (II): $C_{18}H_{16}NO_3^+.C_6H_5O_3S^-$, $M_r = 451{\cdot}497$, monoclinic, $P2_1/c$, $a = 9{\cdot}654\,(3)$, $b = 10{\cdot}686\,(3)$, $c = 21{\cdot}019\,(5)$ Å, $\beta = 95{\cdot}18\,(1)^\circ$, $V = 2159{\cdot}5$ Å^3, $Z = 4$, $D_x = 1{\cdot}389$ g cm^{-3}, $\lambda(\mathrm{Mo}\;K\alpha) = 0{\cdot}71069$ Å, $\mu = 1{\cdot}820$ cm^{-1}, $F(000) = 944$, $T = 295$ K, $R = 0{\cdot}058$ for 2082 observed reflections ($> \sigma_F$) and 374 parameters. Both compounds adopt a propellerlike conformation with a crystallographic and local C_3 axis respectively. The C—O distances in (I) and (II), as well as the C—N distances in (I), are shorter than the sum of the corresponding covalent single-bond radii, due to conjugation of the O and N lone pairs with the aromatic rings. The environment of the N atom in (I) is almost planar. Protonation pyramidalizes the geometry of the N atom [in (II)], and the C—N distances elongate towards the corresponding single-bond values.

Introduction. The structure determinations of the title compounds were part of a larger study on complexes of 2,2′,2″-trihydroxytriphenylamine (= 2,2′,2″-nitrilotriphenole = H_3NTPH) (Müller, 1982; Müller & Bürgi, 1987). The structures presented here serve as reference data for the interpretation of observed structural variations within the NTPH skeleton upon formation of complexes.

Experimental. 2,2′,2″-Trimethoxytriphenylamine [(I), Me_3NTPH] and 2,2′,2″-trihydroxytriphenylamine (H_3NTPH) were obtained as described by Frye, Vincent & Hauschildt (1966). 2,2′,2″-Trihydroxytriphenylammonium benzenesulfonate (II) was obtained from H_3NTPH and benzenesulfonic acid in

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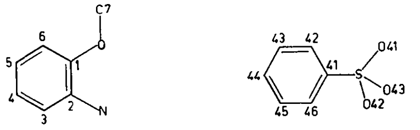
chloroform. Crystals were grown by slow evaporation of the solvent. Crystals of (I) were obtained by thermal recrystallization from toluene.



Atom numbering of the C atoms in Me₃NTPH and of the benzenesulfonate anion

Scheme 1

(I): Crystal of octahedral shape, 0·2 mm edge length, mounted on top of a glass fibre, Enraf–Nonius CAD-4 diffractometer, graphite-monochromatized Mo $K\alpha$ radiation, lattice parameters from 16 reflections with $8 < \theta < 12°$. Data collected in bisecting geometry, integrating ω-scan technique (1·5° reflection^{-1}, 90 s), with periodic checks for orientation and intensity on two standard reflections. No intensity decay noted during data collection. First and last sixth of each scan used for background correction. 582 unique reflections were retained with $F > \sigma_F$ and $0 \leq h \leq 17$, $0 \leq k \leq h$, $k \leq l \leq 17$, in the range of $0 \leq \theta \leq 25°$. No equivalent reflections measured, no absorption correction. Data reduction, Lorentz and polarization corrections, structure solution (*MULTAN77*; Main, Lessinger, Woolfson, Germain & Declercq, 1977) and refinement of parameters (on F) by least-squares methods were performed with the Enraf–Nonius *Structure Determination Package* (Frenz, 1983). Weighting system $w = 1/\sigma_F^2$; final $wR = 0{\cdot}032$ for 582 observations ($> \sigma_F$) and 104 parameters (non-H atoms with anisotropic, H atoms with isotropic displacement). No anomalous features were detected in the residual electron map. Atom N occupies a special position (x,x,x) on a threefold axis of the space group. Scattering factors were from *International Tables for X-ray Crystallography* (1974). Max. shift to e.s.d. <0·5.

(II): Crystal 0·1 × 0·1 × 0·2 mm, mounted in a Lindemann capillary, Enraf–Nonius CAD-4 diffractometer, graphite-monochromatized Mo $K\alpha$ radiation, lattice parameters from 25 reflections with $8 < \theta < 12°$. Data collected in bisecting geometry, integrating ω-scan technique (1·5° reflection^{-1}, 90 s), with periodic checks for orientation and intensity on two standard reflections. No intensity decay detected during data collection. First and last sixth of each scan used for background correction. 2994 independent reflections collected with $-11 \leq h \leq 11$, $0 \leq k \leq 12$, $0 \leq l \leq 24$, in the range $0 \leq \theta \leq 25°$. Lorentz and polarization correction of raw data in *XRAY72* (Stewart, Kruger, Ammon, Dickinson & Hall, 1972). No absorption correction. Structure solution by direct methods (*MULTAN77*; Main *et al.*, 1977) gave the positions of all non-H atoms. Least-squares refinements (on F) in *XRAY72* (Stewart *et al.*, 1972) of all parameters (positional and anisotropic displacement for non-H atoms, positional and isotropic displacement for H atoms) converged at a final weighted R value of $wR = 0{\cdot}073$ (2082 observations $> \sigma_F$ and 374 parameters). No anomalous features were detected in the residual electron density map. The weighting scheme was that of Dunitz & Seiler (1973) with $w = 1/\sigma_F \times \exp[20(\sin\theta/\lambda)^2]$. Scattering factors were taken from *International Tables for X-ray Crystallography* (1974). Max. shift to e.s.d. <0·5.

Table 1. *Fractional coordinates and equivalent isotropic displacement parameters for* Me₃NTPH, *with e.s.d.'s in parentheses*

Anisotropically refined atoms are given in the form of the isotropic equivalent thermal parameter defined as: $\frac{4}{3}[a^2B(1,1) + b^2B(2,2) + c^2B(3,3) + ab(\cos\gamma)B(1,2) + ac(\cos\beta)B(1,3) + bc(\cos\alpha)B(2,3)]$.

	x	y	z	B(Å^2)
N	0·1087 (1)	0·1087	0·1087	3·19 (2)
O	0·06306 (9)	0·27159 (8)	0·1519 (1)	4·83 (4)
C1	0·0034 (1)	0·2089 (1)	0·1704 (1)	3·74 (4)
C2	0·0274 (1)	0·1245 (1)	0·1492 (1)	3·21 (4)
C3	−0·0285 (1)	0·0575 (1)	0·1691 (2)	4·61 (5)
C4	−0·1069 (1)	0·0738 (2)	0·2098 (2)	6·14 (6)
C5	−0·1304 (1)	0·1565 (2)	0·2291 (2)	6·13 (7)
C6	−0·0763 (2)	0·2244 (2)	0·2096 (1)	4·99 (6)
C7	0·0378 (2)	0·3600 (2)	0·1571 (2)	7·03 (7)
H3	−0·011 (1)	−0·000 (1)	0·152 (1)	4·6 (4)*
H4	−0·141 (1)	0·026 (1)	0·225 (1)	6·9 (6)*
H5	−0·182 (1)	0·171 (1)	0·260 (1)	7·7 (6)*
H6	−0·092 (1)	0·284 (1)	0·222 (1)	5·1 (5)*
H71	0·024 (2)	0·373 (2)	0·218 (2)	11·3 (8)*
H72	0·086 (2)	0·396 (2)	0·131 (2)	11·3 (8)*
H73	−0·013 (2)	0·368 (1)	0·118 (1)	9·7 (7)*

* Starred atoms were refined isotropically.

Tables 1 and 2 give positional and equivalent isotropic displacement parameters for the two compounds. Table 3 contains a juxtaposition of important bond lengths and angles of Me₃NTPH and [H₄NTPH]$^+$. Complete geometric data and anisotropic displacement parameters are given in Müller (1982).* Figs. 1 and 2 are *ORTEP* stereoviews.

Discussion. Both Me₃NTPH and [H₄NTPH]$^+$ are found in a propeller-shaped conformation with a crystallographic or local C_3 axis respectively; the planes of the phenyl rings make a mean angle of 45·6 and 35·5° respectively with this axis. The methyl groups of Me₃NTPH are coplanar with the aromatic rings, enabling conjugation of the oxygen electron

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* Lists of structure factors, r.m.s. amplitudes of thermal motion and least-squares planes, for Me₃NTPH, and anisotropic thermal parameters, bond distances, angles and torsional angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51911 (12 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 2. *Fractional coordinates and equivalent isotropic displacement parameters* ($Å^2 \times 10^2$) *for* H_4NTPH^+ *benzenesulfonate*

	x	*y*	*z*	*U*
N1	0·7072 (3)	−0·0038 (4)	0·8470 (2)	3·9 (2)
O1	0·9011 (4)	−0·1678 (4)	0·8851 (2)	6·6 (2)
C11	0·8392 (4)	−0·1157 (5)	0·9333 (2)	5·0 (3)
C12	0·7352 (4)	−0·0287 (5)	0·9167 (2)	4·4 (2)
C13	0·6656 (5)	0·0337 (6)	0·9608 (3)	6·0 (3)
C14	0·7019 (6)	0·0113 (8)	1·0251 (3)	7·4 (4)
C15	0·8040 (6)	−0·0765 (8)	1·0425 (3)	7·6 (4)
C16	0·8728 (6)	−0·1403 (6)	0·9974 (3)	6·5 (3)
O2	0·5309 (3)	−0·1955 (3)	0·8448 (2)	5·8 (2)
C21	0·4731 (4)	−0·0866 (4)	0·8257 (2)	4·0 (2)
C22	0·5571 (4)	0·0183 (4)	0·8256 (2)	3·8 (2)
C23	0·5092 (4)	0·1331 (5)	0·8070 (3)	5·2 (3)
C24	0·3668 (5)	0·1468 (5)	0·7878 (3)	6·1 (3)
C25	0·2817 (4)	0·0437 (6)	0·7870 (3)	5·7 (3)
C26	0·3317 (4)	−0·0722 (6)	0·8049 (3)	5·4 (3)
O3	0·7750 (3)	−0·0211 (3)	0·7283 (2)	5·0 (2)
C31	0·8341 (4)	0·0764 (5)	0·7607 (2)	4·3 (3)
C32	0·8021 (4)	0·0924 (4)	0·8233 (2)	4·4 (2)
C33	0·8541 (5)	0·1881 (5)	0·8614 (3)	5·5 (3)
C34	0·9423 (5)	0·2735 (6)	0·8353 (3)	6·6 (4)
C35	0·9762 (5)	0·2573 (6)	0·7742 (3)	6·1 (4)
C36	0·9248 (4)	0·1599 (6)	0·7370 (3)	5·6 (3)
S1	1·21778 (11)	−0·38679 (13)	0·92539 (6)	4·8 (6)
O41	1·1867 (4)	−0·5058 (4)	0·8949 (2)	6·5 (2)
O42	1·3128 (4)	−0·3148 (5)	0·8923 (2)	7·4 (3)
O43	1·0928 (4)	−0·3208 (6)	0·9366 (2)	9·3 (3)
C41	1·2974 (3)	−0·4194 (5)	1·0029 (3)	5·1 (3)
C42	1·2312 (6)	−0·4999 (6)	1·0418 (3)	6·7 (3)
C43	1·2926 (8)	−0·5225 (8)	1·1024 (3)	8·7 (5)
C44	1·4154 (8)	−0·4703 (8)	1·1237 (4)	9·0 (5)
C45	1·4814 (6)	−0·3916 (7)	1·0846 (3)	7·7 (4)
C46	1·4225 (5)	−0·3649 (6)	1·0230 (3)	5·8 (3)
H13	0·592 (5)	0·098 (6)	0·946 (3)	6·0 (15)*
H14	0·646 (7)	0·073 (8)	1·056 (4)	9·1 (22)*
H15	0·836 (5)	−0·092 (6)	1·088 (3)	6·7 (15)*
H16	0·939 (7)	−0·216 (8)	1·011 (4)	10·0 (22)*
H23	0·578 (5)	0·205 (5)	0·810 (3)	5·1 (14)*
H24	0·340 (6)	0·223 (7)	0·774 (3)	7·4 (18)*
H25	0·179 (6)	0·040 (7)	0·769 (4)	8·0 (19)*
H26	0·268 (6)	−0·147 (7)	0·804 (4)	9·5 (20)*
H33	0·823 (5)	0·196 (6)	0·903 (3)	6·6 (16)*
H34	0·975 (6)	0·348 (7)	0·864 (3)	8·2 (18)*
H35	0·033 (4)	0·318 (5)	0·760 (2)	4·2 (12)*
H36	0·952 (5)	0·153 (5)	0·697 (3)	6·0 (14)*
H1	0·975 (10)	−0·222 (13)	0·894 (6)	16·5 (41)*
H2	0·471 (8)	−0·248 (10)	0·846 (5)	9·2 (28)*
H3	0·798 (6)	−0·013 (6)	0·689 (3)	5·4 (16)*
H4	0·726 (5)	−0·075 (6)	0·826 (3)	6·1 (15)*
H42	1·143 (6)	−0·558 (7)	1·022 (3)	6·8 (17)*
H43	1·254 (9)	−0·582 (11)	1·135 (5)	13·3 (32)*
H44	1·472 (9)	−0·490 (9)	1·166 (5)	14·5 (30)*
H45	1·561 (6)	−0·346 (7)	1·095 (3)	7·4 (18)*
H46	1·471 (4)	−0·307 (4)	0·996 (2)	3·3 (10)*

*Starred atoms were refined isotropically.

lone pairs into the aromatic π systems. This is clearly demonstrated by the O—C1 bond distance (1·356 Å), which is shorter than the distance O—C7 (1·421 Å); the latter approximately equals a C—O single-bond length. This corresponds well to the reference values for a phenyl methyl ether of 1·370 (11) and 1·424 (12) Å (Allen, Kennard, Watson, Brammer, Orpen & Taylor, 1987). An analogous effect is found for the C2—N bond distance (1·423 Å), which is also shorter than the C—N bond distances of protonated $[H_4NTPH]^+$ (1·493 Å). These values are, however, clearly larger than the reference values for *N*,*N*-disubstituted anilines which are 1·371 (16) Å for a tricoordinate planar N atom and 1·426 (11) Å for a tetracoordinate pyramidal one (Allen *et al.*, 1987).

Table 3. *Important bond distances* (Å) *and angles* (°) *of the trimethylated and the protonated* NTPH *skeleton and hydrogen bridges* (Å) *to the benzenesulfonate anion*

	Me_3NTPH	H_4NTPH^+
N—H4		0·91 (6)
O···H4 (mean)		2·27 (6)
O—H		0·87 (9)
O—C7	1·421 (3)	
O—C1	1·365 (2)	1·341 (6)
C1—C2	1·393 (3)	1·387 (7)
C2—N	1·423 (2)	1·493 (6)
C1—O—H		111·0 (40)
C1—O—C7	118·9 (2)	
O—C1—C2	115·7 (2)	117·4 (4)
C1—C2—N	119·8 (2)	115·3 (4)
C—N—C	117·6 (1)	113·5 (3)
C7—O—C1—C2	168·9	
O—C1—C2—N	−1·7	0·3
O1—H1···O43		2·629 (6)
O2—H2···O42		2·726 (6)
O3—H3···O41		2·654 (5)

Values were averaged with respect to the threefold molecular axis, if appropriate.

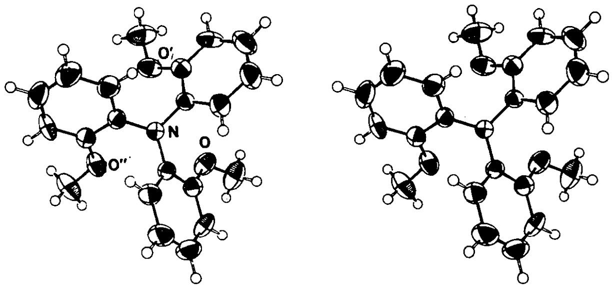

Fig. 1. *ORTEP* (Johnson, 1976) drawing of Me_3NTPH, viewed down the molecular C_3 axis.

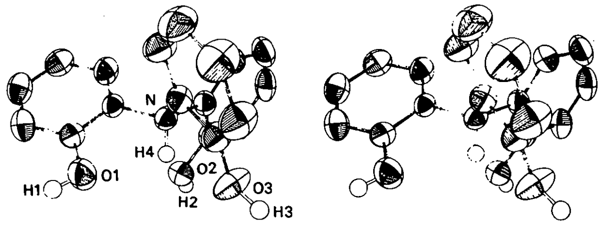

Fig. 2. *ORTEP* drawing of the $[H_4NTPH]^+$ cation, viewed perpendicular to the molecular C_3 axis. (Numbering of the atoms: the first digit after the element symbol refers to the ring number; the second digit specifies the C atom within the six-membered ring, according to Scheme 1.)

The environment of the N atom in Me_3NTPH is almost planar (C—N—C angles of 117·6°), a further sign of conjugation of the nitrogen electron lone pair with the three phenyl rings. Upon protonation, it is pyramidalized, as shown by the decreased C—N—C angles of $[H_4NTPH]^+$ (113·5°).

None of the benzene ring distances and angles show anomalous values; the latter lie within 1 and 5°, respectively, of the 120° expected for trigonal planar coordinated atoms. The relatively small angle O—C1—C2 (115·7°) in Me$_3$NTPH, if compared to its homologue in [H$_4$NTPH]$^+$ (117·4°), can be explained in terms of the enhanced steric requirements of a methyl group (C7) compared to those of a proton. The small angle C1—C2—N (115·3°) in H$_4$NTPH$^+$ could be due to an attractive interaction of the proton H4 with the phenolic O atoms (O1, O2, O3; mean contact distance H4···O of 2·27 Å). Such an interaction is absent in Me$_3$NTPH (angle C1—C2—N of 119·8°). Finally we mention the hydrogen bridges between the phenolic O atoms of H$_4$NTPH$^+$ and the SO$_3$ group of the benzenesulfonate anion, mediated by H1, H2 and H3.

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First Unsymmetrical Donor Which Gives Organic Superconductors: DMET

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Abstract. 2-(4,5-Dimethyldiselenol-2-ylidene)dithiolo[4,5-*b*]dihydrodithiin, $C_{10}H_{10}S_4Se_2$, $M_r = 416{\cdot}34$, monoclinic, $P2_1/c$, $a = 6{\cdot}724\,(1)$, $b = 12{\cdot}144\,(3)$, $c = 18{\cdot}228\,(3)$ Å, $\beta = 111{\cdot}21\,(1)°$, $V = 1387{\cdot}7\,(4)$ Å^3, $Z = 4$, $D_x = 1{\cdot}99$ Mg m^{-3}, λ(Mo $K\alpha$) $= 0{\cdot}71068$ Å, $\mu = 5{\cdot}84$ mm^{-1}, $F(000) = 808$, $T = 297$ K, $R = 0{\cdot}058$ for 1816 reflections. The molecule is bent at the inner and the outer S atoms in contrast to the planar conformation in the partially oxidized states in organic conductors (DMET)$_2X$ (X = monovalent anion).

Introduction. The title compound DMET is an unsymmetrical hybrid donor between TMTSF and BEDT–TTF, and forms radical salts of the type (DMET)$_2X$ with various monovalent anions X. The electrical properties of (DMET)$_2X$ have been investigated for a few years by the present authors (Kikuchi, Ikemoto & Kobayashi, 1987; Murata *et al.*, 1988; Kikuchi, Saito, Ikemoto, Murata, Ishiguro & Kobayashi, 1988). In the course of the study, some superconductors were discovered in 1987 (Kikuchi, Kikuchi *et al.*, 1987; Kikuchi, Murata, Honda, Namiki, Saito, Kobayashi, Ishiguro & Ikemoto, 1987; Kikuchi, Murata, Honda, Namiki, Saito, Ishiguro, Kobayashi & Ikemoto, 1987; Kikuchi, Murata, Honda, Namiki, Saito, Anzai, Kobayashi, Ishiguro & Ikemoto, 1987; Kikuchi, Honda *et al.*, 1988). Thus, (DMET)$_2X$ is the first group of organic superconductors based on an unsymmetrical donor. From

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