Table 3. *Selected bond lengths* (Å) *and angles* (°) *with e.s.d.'s of the two independent complex anions in* $[Bu_4N][ReO(bdt)_2]$

Re1—O1	1.70 (2)	Re1—S1	2.316 (5)
Re1—S2	2.311 (6)	Re1—S3	2.304 (5)
Re1—S4	2.307 (6)	S1—C11	1.77 (2)
S2—C12	1.72 (2)	S3—C21	1.77 (2)
S4—C22	1.77 (2)		
O1—Re1—S1	113.5 (6)	O1—Re1—S2	106.7 (6)
O1—Re1—S3	104.6 (6)	O1—Re1—S4	107.9 (6)
S1—Re1—S2	84.9 (3)	S3—Re1—S4	85.4 (2)
S1—Re1—S3	142.0 (2)	S2—Re1—S4	145.3 (2)
S1—Re1—S4	83.9 (3)	S2—Re1—S3	83.5 (2)
Re1—S1—C11	104.8 (7)	Re1—S2—C12	105.6 (8)
Re1—S3—C21	105.4 (7)	Re1—S4—C22	105.1 (7)
Re2—O2	1.72 (2)	Re2—S5	2.321 (6)
Re2—S6	2.306 (5)	Re2—S7	2.314 (6)
Re2—S8	2.309 (6)	S5—C31	1.71 (2)
S6—C32	1.76 (2)	S7—C41	1.78 (2)
S8—C42	1.77 (2)		
O2—Re2—S5	108.3 (7)	O2—Re2—S6	110.7 (6)
O2—Re2—S7	108.9 (7)	O2—Re2—S8	108.6 (6)
S5—Re2—S6	84.3 (2)	S7—Re2—S8	84.7 (2)
S5—Re2—S7	142.9 (3)	S6—Re2—S8	140.7 (3)
S5—Re2—S8	82.5 (3)	S6—Re2—S7	83.9 (2)
Re2—S5—C31	104.8 (8)	Re2—S6—C32	105.4 (7)
Re2—S7—C41	106.2 (9)	Re2—S8—C42	105.7 (7)

only between 108.2 (2) and 108.7 (2)°. The benzene-1,2-dithiolato ligands exhibit an almost ideal planarity in the latter complex.

The structure of the tetrabutylammonium cations corresponds fully to the bonding feature which is observed in other structures. The geometry about the N atom is only slightly distorted from the regular tetrahedral [a mean value of 109.5 (25)° was observed for the C—N—C angles]. The mean C—N bond length is 1.49 (4) Å.

We thank Professor J. Strähle, University of Tübingen, for providing us with facilities and the Deutsche Forschungsgemeinschaft and the Fonds der Chemischen Industrie for financial support.

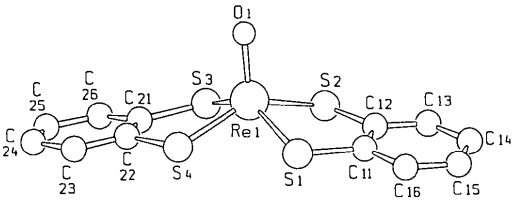

Fig. 1. Plot of the complex anion along with the atomic numbering scheme.

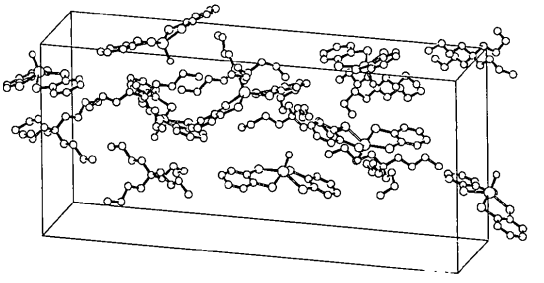

Fig. 2. Unit-cell packing; projection parallel to **a**.

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Structure of *N*-(2-Aminophenyl)-*N'*-methylurea

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Abstract. $C_8H_{11}N_3O$, $M_r = 165.19$, monoclinic, $P2_1/n$, $a = 10.3997$ (14), $b = 4.6395$ (12), $c = 18.2211$ (11) Å, $\beta = 100.994$ (7)°, $V = 868.0$ (2) Å^3, $Z = 4$, $D_x = 1.2271$ Mg m^{-3}, λ(Cu $K\alpha$) = 1.54178 Å (graphite monochromator), $\mu = 0.682$ mm^{-1}, $F(000) = 352$, $T = 293$ K, 1471 unique reflections, $R = 0.054$, $wR = 0.045$ for 887 reflections with $I > 2\sigma(I)$. The ureido moiety is approximately planar and makes a dihedral angle of 58.92 (16)° with the least-squares plane of the aromatic ring. The carbonyl

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Table 1. *Final fractional coordinates and isotropic or equivalent isotropic thermal parameters* (Å^2)

For non-H atoms $B_{eq} = (8\pi^2/3)\sum_i\sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j$.

	x	y	z	B_{iso}/B_{eq}
O(2′)	0.8990 (3)	−0.1400 (5)	0.1151 (1)	4.0 (1)
N(1)	0.9407 (4)	0.2684 (8)	0.1832 (2)	3.7 (2)
N(3)	0.8749 (5)	0.2888 (9)	0.0558 (2)	4.3 (2)
N(4)	1.1501 (4)	−0.1270 (9)	0.2253 (2)	3.9 (2)
C(1)	0.9630 (4)	0.1345 (9)	0.2541 (2)	3.2 (2)
C(2)	1.0652 (4)	−0.0620 (8)	0.2740 (2)	3.2 (2)
C(3)	1.0865 (5)	−0.180 (1)	0.3450 (2)	4.2 (2)
C(4)	1.0102 (5)	−0.101 (2)	0.3963 (3)	5.0 (2)
C(5)	0.9106 (5)	0.094 (1)	0.3768 (3)	4.8 (2)
C(6)	0.8861 (4)	0.213 (1)	0.3058 (2)	4.0 (2)
C(2′)	0.9037 (4)	0.1259 (8)	0.1176 (2)	3.2 (2)
C(4′)	0.8219 (5)	0.172 (1)	−0.0161 (3)	5.3 (3)
H(1)	1.108 (4)	−0.15 (1)	0.177 (2)	5 (1)
H(2)	1.202 (3)	−0.269 (8)	0.240 (2)	3 (1)
H(3)	1.158 (4)	−0.328 (8)	0.360 (2)	5 (1)
H(4)	1.032 (3)	−0.198 (8)	0.445 (2)	4 (1)
H(5)	0.849 (4)	0.159 (9)	0.409 (2)	5 (1)
H(6)	0.801 (3)	0.344 (8)	0.284 (2)	3.8 (9)
H(7)	0.930 (4)	0.413 (7)	0.184 (2)	2 (1)
H(8)	0.873 (4)	0.437 (8)	0.059 (2)	2 (1)
H(9)	0.721 (3)	0.09 (1)	−0.022 (2)	9 (1)
H(10)	0.794 (4)	0.324 (7)	−0.060 (2)	9 (1)
H(11)	0.865 (3)	0.04 (1)	−0.030 (3)	9 (1)

Table 2. *Bond distances* (Å) *and angles* (°) *with e.s.d.'s in parentheses*

O(2′)—C(2′)	1.235 (5)	C(1)—C(2)	1.394 (5)
N(1)—C(1)	1.414 (5)	C(1)—C(6)	1.395 (6)
N(1)—C(2′)	1.356 (6)	C(2)—C(3)	1.384 (6)
N(3)—C(2′)	1.341 (6)	C(3)—C(4)	1.387 (7)
N(3)—C(4′)	1.429 (6)	C(4)—C(5)	1.369 (7)
N(4)—C(2)	1.398 (6)	C(5)—C(6)	1.384 (6)

C(1)—N(1)—C(2′)	124.2 (4)	C(2)—C(3)—C(4)	121.0 (4)
C(2′)—N(3)—C(4′)	122.7 (4)	C(3)—C(4)—C(5)	120.1 (4)
N(1)—C(1)—C(2)	120.6 (4)	C(4)—C(5)—C(6)	120.1 (5)
N(1)—C(1)—C(6)	119.2 (4)	C(1)—C(6)—C(5)	119.9 (4)
C(2)—C(1)—C(6)	120.2 (4)	O(2′)—C(2′)—N(1)	121.5 (4)
N(4)—C(2)—C(1)	121.3 (4)	O(2′)—C(2′)—N(3)	122.1 (4)
N(4)—C(2)—C(3)	120.0 (4)	N(1)—C(2′)—N(3)	116.4 (4)
C(1)—C(2)—C(3)	118.7 (4)		

group is oriented toward the amino group and the distance between the ureido O(2) atom and the amino N(4) atom is 2.975 (4) Å.

Introduction. An NMR study on the conformation of *N*-(*o*-aminophenyl)-*N*′-alkylureas has stimulated interest in determining the geometry of the title compound in the solid state. NOE (nuclear Overhauser enhancement) difference-spectroscopy results (Jean-Claude & Just, 1992) in dimethyl sulfoxide (Me_2SO) confirmed the existence of conformations in which the two protons of the ureido moiety are oriented *trans–trans* to the carbonyl group. This paper deals with the crystal structure of one of the members of this class of aromatic ureas, *N*-(2-aminophenyl)-*N*′-methylurea.

Experimental. The title compound was prepared according to the method described by Jean-Claude & Just (1991). Its recrystallization by slow evaporation of methanol gave colorless needles, m.p. 443 K, one of which (0.50 × 0.10 × 0.14 mm) was used for data collection at 293 K using the $\omega/2\theta$ scanning mode. Data were collected on a Rigaku AFC-6*S* diffractometer with graphite-monochromated Cu $K\alpha$ radiation; 1561 reflections ($+h$, min. 0, max. 10; $+k$, min. 0, max. 5; $\pm l$, min. -20, max. $+20$) were collected with $3 < 2\theta < 120°$. Three standard reflections indicated crystal and electronic stability; 25 centered reflections ($61.6 < 2\theta < 80°$) were used to determine the unit cell. The data were reduced to 1471 unique reflections ($R_{int} = 0.097$) with Lorentz–polarization and absorption (azimuthal scan: transmission range 0.93–1.00) corrections applied; 887 reflections were considered observed [$I > 2\sigma(I)$]. The space group was determined to be $P2_1/n$.

The structure was solved by direct methods. The program *SIR*88 (Burla, Camalli, Cascarano, Giacovazzo, Polidori, Spagna & Viterbo, 1989) revealed all non-H atoms, which were refined anisotropically using *TEXSAN* (Molecule Structure Corporation, 1985). H atoms were found from a difference map and refined isotropically. The final cycle of full-matrix least-squares refinement, mini-

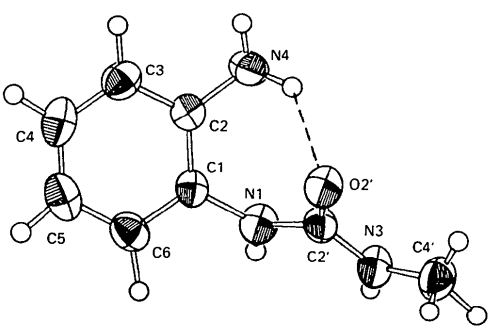

Fig. 1. *ORTEP* plot (Johnson, 1976) of *N*-(2-aminophenyl)-*N*′-methylurea with 50% probability ellipsoids.

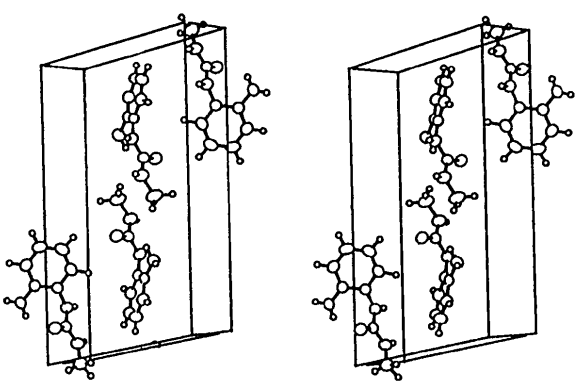

Fig. 2. Stereo *ORTEP* plot (Johnson, 1976) of the unit cell viewed down the *b* axis with the *a* axis parallel to the bottom of the page.

mizing $\sum w(F_o - F_c)^2$ with a 6:1 reflection to parameter ratio, showed a max. shift/e.s.d. of 0.11. Max. peak on final difference map was 0.19 e Å^{-3}, $R = 0.054$, $wR = 0.045$ and $S = 2.40$ with weights based on counting statistics. Scattering factors were taken from Cromer & Waber (1974) and anomalous-dispersion corrections for the non-H atoms were from Cromer (1974).

Discussion. The final atomic coordinates and equivalent isotropic temperature factors are given in Table 1.* The bond distances and angles are listed in Table 2. The structure and labeling of the title compound are shown in Fig. 1 and a stereo packing diagram is given in Fig. 2.

The ureido moiety is planar ($\chi^2 = 3.696$). The plane of the aromatic ring is defined by atoms C(1), C(2), C(3), C(4), C(5), C(6) and makes a dihedral angle of 58.92 (16)° with the plane of the ureido system. This geometry suggests that the lone pair on N(1) is delocalized mainly through the N(1)—C(2′) bond of the ureido group. It would consequently be less conjugated to the aromatic ring [N(1)—C(1) 1.414 (5), N(1)—C(2′) 1.356 (6) Å].

The distance between N(4) and C(2′) is 2.975 (4) Å and the bond angles around N(4) are C(2)—N(4)—H 113.19 and H(1)—N(4)—H 111.90°. These values indicate a pyramidal geometry for N(4).

In the crystal lattice (Fig. 2), the ureido moiety of one molecule is oriented antiparallel to that of another molecule, and the intermolecular distances O(2′)—N(3) and O(2′)—N(1) are 3.009 (5) and 2.855 (5) Å, respectively.

The geometry of the title compound in the solid is similar to that of one of its major conformations in solution, as confirmed by NMR spectroscopy results (Jean-Claude & Just, 1992).

We wish to thank the Natural Research Council of Canada for financial support. We are also grateful to Dr Rosemary Hynes for discussions.

* Lists of structure factors, anisotropic thermal parameters, bond angles involving hydrogen, intermolecular contacts, torsion angles and least-squares-plane data have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55810 (16 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: BR1006]

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Structure of Tris(2,4,6-trimethoxyphenyl)phosphine Oxide Hydrate

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(*Received* 21 *September* 1992; *accepted* 7 *January* 1993)

Abstract. $C_{27}H_{33}O_{10}P.H_2O$, $M_r = 566.54$, triclinic, $P\bar{1}$, $a = 11.429$ (1), $b = 14.390$ (4), $c = 19.459$ (3) Å, $\alpha = 70.49$ (2), $\beta = 78.14$ (1), $\gamma = 69.69$ (2)°, $V = 2814.5$ Å^3, $Z = 4$, $D_x = 1.337$ g cm^{-3}, λ(Mo $K\alpha$) = 0.71069 Å, $\mu = 1.49$ cm^{-1}, $F(000) = 1200$, $T = 293$ K, $R = 0.068$ and $wR = 0.088$ for 6333 observed reflections with $|F^2| > 2\sigma(F^2)$. The asymmetric unit contains two molecules, differing in the orientation of one methoxy group. Water molecules in the crystal are hydrogen bonded to each other and to the O atom of the P═O group [P═O 1.467(3), 1.475 (3) Å, for molecules *A* and *B*, respectively].

Introduction. There has been considerable interest in the use of the basic, hindered phosphine, tris-(2,4,6-trimethoxyphenyl)phosphine (TMPP) (Wada

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