diiodoformamide (Pritzkow, 1974). The various parameters are given in Table 3. Of the two short O···I contacts in *N*,*N*-diiodoformamide, one is very similar to that in *N*-iodosuccinimide and the other one weaker. The crystal structures of *N*-chloro- and *N*-bromosuccinimide display similar features. Intermolecular O···Cl and O···Br distances (2·88 and 2·80 Å) in these structures are shorter than the van der Waals sums of 3·20 and 3·35 Å respectively (Brown, 1961; Jabay, Pritzkow & Jander, 1977).*

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* A referee has noted that C—H···O contacts might be as important (Desiraju, 1987; Murray-Rust & Glusker, 1984) as the I···O contacts discussed here. Thus there is a short O(2)···H(2)—C(2) distance of 2·45 Å between an oxygen of one molecule and a methylene group of the molecule produced by the 4_1 operation. The C—O···H angle is 155°.

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The Structures of the Isostructural Adducts Triphenylphosphine Oxide–Trifluoroborane and Triphenylarsine Oxide–Trifluoroborane

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Abstract. $C_{18}H_{15}OP.BF_3$, $M_r = 346{\cdot}09$, monoclinic, $P2_1/n$, $a = 9{\cdot}427\ (3)$, $b = 18{\cdot}043\ (2)$, $c = 10{\cdot}089\ (2)$ Å, $\beta = 104{\cdot}24\ (2)°$, $V = 1663{\cdot}3$ Å^3, $Z = 4$, $D_x = 1{\cdot}382$ Mg m^{-3}, λ(Mo $K\alpha$) = 0·70926 Å, $\mu = 0{\cdot}205$ mm^{-1}, $F(000) = 712$, $T = 293$ K, $R = 0{\cdot}035$ for 1185 significant reflections. $C_{18}H_{15}AsO.BF_3$, $M_r = 390{\cdot}0$, monoclinic, $P2_1/n$, $a = 9{\cdot}520\ (2)$, $b = 18{\cdot}231\ (3)$, $c = 10{\cdot}142\ (2)$ Å, $\beta = 104{\cdot}21\ (2)°$, $V = 1706{\cdot}3$ Å^3, $Z = 4$, $D_x = 1{\cdot}518$ Mg m^{-3}, λ(Mo $K\alpha$) = 0·70926 Å, $\mu = 2{\cdot}14$ mm^{-1}, $F(000) = 784$, $T = 293$ K, $R = 0{\cdot}0281$ for 1982 reflections. The structures consist of discrete molecular units and are isostructural. The angles at oxygen [P—O—B = 134·5 (2), As—O—B = 125·7 (2)°] and the E—O (E = P, As) bond lengths [P—O = 1·522 (3) Å, As—O = 1·690 (3) Å] are typical for related adducts and allow a useful comparison of the adduct effect on the E—O π character.

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Introduction. Lewis-acid adducts of pnictogen oxides are well known and have been extensively spectroscopically characterized. However, structural information is limited. Here we report the crystal structures of the BF_3 adducts of Ph_3PO and Ph_3AsO, which are isostructural, adopting the usual bent geometry. The compounds provide a useful E—O (E = P, As) bond-length comparison in terms of the relative effect of the acid on the π character of the E—O bond.

Experimental. The title compounds were obtained in high yield from reactions of Ph_3E (E = P, As) with $NOBF_4$ in CH_2Cl_2. White crystals were obtained by slow evaporation of CH_2Cl_2. Enraf–Nonius CAD-4 diffractometer; graphite-monochromated Mo $K\alpha$ radiation; lattice constants from 25 reflections with 2θ = 22–28°; $\omega/2\theta$ scan mode, ω-scan width (1·0 + 0·35 tanθ)° at 0·7–4·0° min^{-1} extended 25% on each side for background measurement; three standard reflections monitored every hour, no significant deviations in intensity, intensities reduced to a standard scale (Cameron & Cordes, 1979); Lp corrections applied. Anisotropic refinement (on F) of the non-H atoms and refinement of individual isotropic temperature factors on the H atoms employed a 3-block-matrix least-squares method (Sheldrick, 1976) minimizing $\sum w(\Delta F)^2$, where $w = 1/[\sigma^2(F) + 0{\cdot}004 F^2]$, σ from counting statistics. Scattering factors for neutral atoms were taken from *International Tables for X-ray Crystallography* (1974) and corrected for the real part of anomalous dispersion.

$Ph_3PO.BF_3$ (P): Crystal 0·075 × 0·25 × 0·50 mm; intensities for $2\theta < 46°$; *hkl*: −10 to 10, 0 to 19, −11 to 11, after equivalent reflections sorted and merged: −10 to 10, 0 to 19, 0 to 11; 2906 reflections measured, 2306 unique (R_{int} = 0·029), 1185 considered observed [$I > 2\sigma(I)$]. No absorption corrections applied. Direct methods (*MULTAN*80; Main *et al.*, 1980) revealed a partial structure, all remaining atoms, including the H atoms, were located from a subsequent Fourier synthesis. H atoms were constrained to ride on the atoms to which they were bonded. The final cycles of refinement of 232 parameters converged to R = 0·031 and wR = 0·0360, $(\Delta/\sigma)_{max}$ = 0·5. A final difference Fourier map showed no significant features and had a maximum of 0·20 e $Å^{-3}$ and a minimum of −0·22 e $Å^{-3}$. No correction for extinction.

Table 1. *Fractional positional and equivalent isotropic thermal parameters* ($Å^2$) *of* $Ph_3PO.BF_3$ *with e.s.d.'s in parentheses*

$U_{eq} = (U_1U_2U_3)^{1/3}$.

	x	y	z	U_{eq}
P	0·89243 (11)	0·15141 (6)	0·53300 (10)	0·0324
C11	0·7959 (4)	0·2352 (2)	0·5450 (3)	0·0357
C12	0·8072 (4)	0·2656 (2)	0·6731 (4)	0·0436
C13	0·7449 (4)	0·3333 (2)	0·6860 (4)	0·0503
C14	0·6724 (5)	0·3710 (2)	0·5721 (4)	0·0516
C15	0·6594 (5)	0·3410 (2)	0·4436 (4)	0·0518
C16	0·7222 (4)	0·2733 (2)	0·4296 (3)	0·0447
C21	1·0780 (4)	0·1754 (2)	0·5389 (3)	0·0362
C22	1·1164 (4)	0·2457 (2)	0·5058 (4)	0·0510
C23	1·2617 (5)	0·2643 (2)	0·5205 (4)	0·0593
C24	1·3681 (5)	0·2115 (2)	0·5665 (5)	0·0598
C25	1·3315 (4)	0·1417 (2)	0·5976 (4)	0·0572
C26	1·1868 (4)	0·1232 (2)	0·5847 (4)	0·0466
C31	0·8085 (4)	0·1058 (2)	0·3763 (3)	0·0363
C32	0·8894 (4)	0·0828 (2)	0·2862 (4)	0·0443
C33	0·8239 (5)	0·0451 (2)	0·1697 (4)	0·0557
C34	0·6771 (5)	0·0296 (2)	0·1421 (4)	0·0621
C35	0·5947 (5)	0·0517 (3)	0·2296 (5)	0·0638
C36	0·6594 (4)	0·0893 (2)	0·3475 (4)	0·0537
B	0·7884 (5)	0·0621 (2)	0·7158 (5)	0·0446
F1	0·8509 (3)	0·0550 (1)	0·8515 (2)	0·0809
F2	0·7657 (3)	−0·0060 (1)	0·6582 (3)	0·0846
F3	0·6657 (3)	0·1021 (1)	0·6911 (3)	0·0902
O	0·9006 (2)	0·1017 (1)	0·6565 (2)	0·0391

Table 2. *Fractional positional and equivalent isotropic thermal parameters* ($Å^2$) *for* $Ph_3AsO.BF_3$ *with e.s.d.'s in parentheses*

$U_{eq} = (U_1U_2U_3)^{1/3}$.

	x	y	z	U_{eq}
As	0·89775 (3)	0·15178 (2)	0·53270 (4)	0·0307
C11	0·7973 (3)	0·2399 (1)	0·5504 (3)	0·0322
C12	0·8105 (3)	0·2686 (2)	0·6780 (3)	0·0413
C13	0·7472 (4)	0·3352 (2)	0·6917 (4)	0·0485
C14	0·6722 (4)	0·3723 (2)	0·5790 (4)	0·0490
C15	0·6598 (4)	0·3436 (2)	0·4518 (4)	0·0509
C16	0·7226 (4)	0·2771 (2)	0·4357 (3)	0·0448
C21	1·0945 (3)	0·1761 (1)	0·5410 (3)	0·0354
C22	1·1304 (4)	0·2459 (2)	0·5096 (4)	0·0510
C23	1·2752 (4)	0·2642 (2)	0·5261 (5)	0·0665
C24	1·3802 (4)	0·2134 (2)	0·5722 (4)	0·0635
C25	1·3440 (4)	0·1430 (2)	0·6014 (4)	0·0613
C26	1·1999 (3)	0·1242 (2)	0·5856 (4)	0·0484
C31	0·8059 (3)	0·1021 (1)	0·3687 (3)	0·0357
C32	0·8887 (3)	0·0777 (2)	0·2843 (3)	0·0444
C33	0·8250 (4)	0·0406 (2)	0·1667 (4)	0·0572
C34	0·6784 (4)	0·0281 (2)	0·1358 (4)	0·0653
C35	0·5951 (4)	0·0513 (3)	0·2216 (5)	0·0708
C36	0·6596 (4)	0·0885 (2)	0·3402 (4)	0·0587
O	0·9140 (2)	0·0982 (1)	0·6715 (2)	0·0398
B	0·7928 (4)	0·0619 (2)	0·7147 (4)	0·0454
F1	0·8355 (3)	0·0553 (1)	0·8516 (2)	0·0862
F2	0·7721 (3)	−0·0063 (1)	0·6583 (3)	0·0771
F3	0·6712 (2)	0·1024 (1)	0·6712 (3)	0·0866

$Ph_3AsO.BF_3$ (As): Crystal 0·5 × 0·25 × 0·25 mm; intensities for $2\theta < 50°$; *hkl*: −11 to 11, −1 to 21, −1 to 12, 3865 reflections measured, 2998 unique (R_{int} = 0·024), 1982 considered observed [$I > 2\sigma(I)$]. Absorption corrections applied (Walker & Stuart, 1983), maximum and minimum absorption coefficients 1·093, 0·867. Non-H atomic positions from P were used successfully in initial refinement. The H atoms were located in a difference Fourier synthesis and were constrained to ride on the atoms to which they were bonded. The final cycles of refinement of 232 parameters converged to R = 0·0281 and wR = 0·0292, $(\Delta/\sigma)_{max}$ = 1·8 [F(3), U_{33}], 0·8 otherwise. A final difference Fourier map showed no significant features and had a maximum of 0·69 e $Å^{-3}$ and a

Table 3. *Bond lengths* (Å) *for* $Ph_3EO.BF_3$ *with e.s.d.'s in parentheses*

		$E = P$	$E = As$			$E = P$	$E = As$
E	C11	1·784 (4)	1·900 (3)	C23	C24	1·379 (7)	1·360 (6)
E	C21	1·788 (4)	1·907 (3)	C24	C25	1·362 (8)	1·379 (7)
E	C31	1·785 (4)	1·907 (3)	C25	C26	1·379 (6)	1·384 (5)
E	O	1·522 (3)	1·690 (3)	C31	C32	1·387 (6)	1·373 (6)
C11	C12	1·384 (6)	1·373 (5)	C31	C36	1·396 (6)	1·373 (5)
C11	C16	1·382 (5)	1·383 (5)	C32	C33	1·367 (6)	1·376 (5)
C12	C13	1·376 (6)	1·377 (6)	C33	C34	1·371 (7)	1·372 (6)
C13	C14	1·366 (6)	1·369 (5)	C34	C35	1·371 (8)	1·379 (8)
C14	C15	1·382 (6)	1·370 (6)	C35	C36	1·375 (7)	1·387 (7)
C15	C16	1·381 (6)	1·379 (6)	B	F1	1·357 (5)	1·354 (5)
C21	C22	1·383 (6)	1·374 (5)	B	F2	1·353 (6)	1·362 (5)
C21	C26	1·385 (5)	1·373 (5)	B	F3	1·334 (6)	1·352 (5)
C22	C23	1·383 (7)	1·388 (6)	B	O	1·516 (6)	1·486 (5)

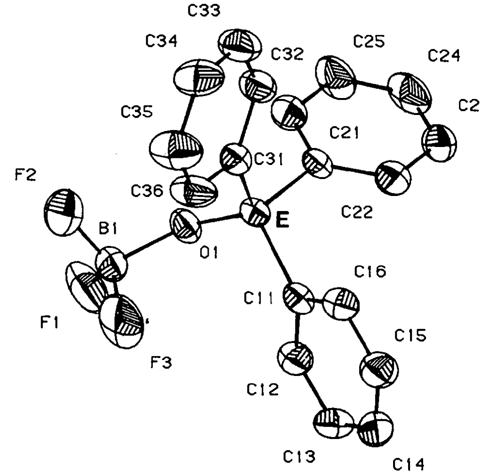

Fig. 1 A view of *P* and *As* (Davies, 1983). H atoms have been omitted for clarity.

minimum of $-0{\cdot}28\,e\,\text{Å}^{-3}$. No correction for extinction.

Discussion. Tables 1 and 2 list refined fractional coordinates of $Ph_3PO.BF_3$ and $Ph_3AsO.BF_3$, respectively, and Tables 3 and 4 provide selected bond lengths and angles.* Views of the molecule and of the unit-cell contents are displayed in Figs. 1 and 2, respectively. The crystal structures of $Ph_3PO.BF_3$ (*P*) and $Ph_3AsO.BF_3$ (*As*) are isostructural and consist of discrete molecular units. The Ph_3E and BF_3 moieties have slightly distorted tetrahedral local geometries. Both molecules adopt a bent geometry at the oxygen center [*P*, 134·5 (2); *As*, 125·7 (2)°], typical for related adducts [143·7° in $Me_3PO.SbCl_5$ (Branden & Lindqvist, 1963), 135·5 (1·4)° av. in $2Ph_3AsO.HgCl_2$ (Branden, 1963)]. The *E*—O bonds [1·522 (3) Å in *P* and 1·690 (3) Å in *As*] are significantly longer than in the parent oxides [1·46 (1) Å in Ph_3PO (Bandoli, Bortolozzo, Clemente, Croatto & Panattoni, 1970), 1·644 (7) Å in $Ph_3AsO.H_2O$ (Ferguson & Macaulay, 1969)], and are comparable to those in related adducts [*e.g.* 1·56 (4) Å in $Me_3PO.SbCl_5$ (Branden & Lindqvist, 1963), 1·69 (3) Å in $2Ph_3AsO.HgCl_2$ (Branden, 1963)]. The long P—O bond is consistent with the low-energy PO stretching frequency

Table 4. *Bond angles* (°) *for* $Ph_3EO.BF_3$ *with e.s.d.'s in parentheses*

			$E = P$	$E = As$
C11	*E*	C21	107·7 (2)	108·1 (1)
C11	*E*	C31	109·2 (2)	110·5 (1)
C11	*E*	O	111·6 (2)	110·8 (1)
C21	*E*	C31	111·0 (2)	112·6 (2)
C21	*E*	O	105·3 (2)	102·1 (1)
C31	*E*	O	112·1 (2)	112·4 (1)
E	C11	C12	118·4 (3)	118·7 (2)
E	C11	C16	121·5 (3)	120·2 (3)
C12	C11	C16	119·8 (4)	120·9 (3)
C11	C12	C13	120·3 (4)	119·4 (3)
C12	C13	C14	119·9 (4)	120·2 (4)
C13	C14	C15	120·4 (4)	120·3 (4)
C14	C15	C16	120·0 (4)	120·5 (4)
C11	C16	C15	119·6 (4)	118·7 (4)
E	C21	C22	121·7 (3)	120·2 (3)
E	C21	C26	119·0 (3)	119·0 (3)
C22	C21	C26	119·2 (4)	120·7 (3)
C21	C22	C23	120·5 (4)	119·4 (4)
C22	C23	C24	119·2 (4)	120·1 (4)
C23	C24	C25	120·8 (4)	120·4 (4)
C24	C25	C26	120·1 (4)	119·9 (4)
C21	C26	C25	120·1 (4)	119·4 (4)
E	C31	C32	121·6 (3)	119·1 (2)
E	C31	C36	119·2 (3)	119·6 (3)
C32	C31	C36	119·1 (4)	121·2 (3)
C31	C32	C33	120·5 (4)	120·1 (3)
C32	C33	C34	119·7 (5)	119·2 (4)
C33	C34	C35	120·9 (4)	121·0 (4)
C34	C35	C36	120·0 (4)	119·8 (4)
C31	C36	C35	119·7 (5)	118·8 (4)
F1	B	F2	109·3 (4)	109·0 (4)
F1	B	F3	112·4 (4)	113·6 (4)
F1	B	O	105·7 (3)	106·4 (3)
F2	B	F3	111·9 (4)	109·7 (3)
F2	B	O	108·1 (4)	109·0 (4)
F3	B	O	109·2 (4)	109·0 (3)
E	O	B	134·5 (2)	125·7 (2)

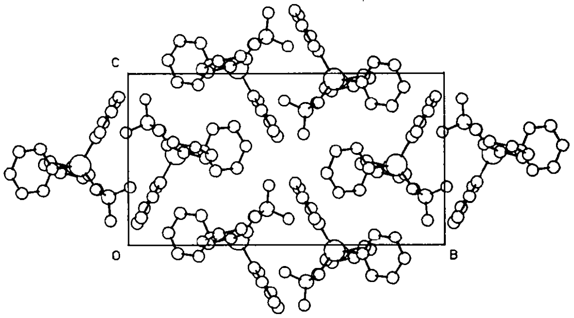

Fig. 2. A projection of the unit-cell packing of *P* and *As* down the *a* axis.

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* Lists of structure factors, anisotropic thermal parameters and H-atom positions, bond lengths and angles involving H atoms and torsion angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52171 (43 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

observed in the infrared spectrum (Linder, Lehner & Scheer, 1967). The degree of *E*—O lengthening is significantly greater in *P* than in *As* with respect to the corresponding bond in the free base, demonstrating a more dramatic disruption of the π character of the *E*—O bond. The B—O bond lengths in the two structures are indistinguishable and are typical of a single B—O bond (Greenwood & Earnshaw, 1984).

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Structure of Bis(cyclohexylammonium) D-Glucose 6-Phosphate Trihydrate

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Abstract. $C_6H_{11}O_9P^{2-}.2C_6H_{14}N^+.3H_2O$, M_r = 512·54, triclinic, $P1$, a = 9·486 (7), b = 12·696 (8), c = 11·736 (7) Å, α = 99·93 (5), β = 102·66 (5), γ = 104·90 (5)°, V = 1293 (2) Å^3, Z = 2, D_m = 1·30 (2), D_x = 1·317 (2) Mg m^{-3}, λ(Mo $K\alpha$) = 0·71069 Å, μ = 0·173 mm^{-1}, F(000) = 556, T = 301 (2) K, final R = 0·0442 for 4760 observed reflections. Two crystallographically independent sugar moieties have different configurations at C(1). The first dianion exists as β-D-glucopyranose. The second is about 65% α-glucopyranose and about 35% β-glucopyranose forms. The orientation of the phosphate group with respect to the sugar system is similar in the two anions. The lengths of the phosphate ester bonds are 1·610 (4) and 1·625 (4) Å.

Introduction. Depending on pH, glucose 6-phosphate may exist in water solutions as free acid, monoanion and dianion. All these forms are difficult to obtain in the crystalline state. The only known crystal structures are of the Ba salt of the dianion (Katti, Seshadri & Viswamitra, 1982; Lis, 1985) and the Na salt of the monoanion (Lis, 1985; Narendra & Viswamitra, 1985). Since our attempts to isolate the crystalline Na and K salts of the dianion failed, it was decided to obtain some other crystalline salts of the D-glucose 6-phosphate dianion with different organic cations. Here the structure of D-glucose 6-phosphate dianion as the cyclohexylammonium salt is reported.

Experimental. Bis(cyclohexylammonium) D-glucose 6-phosphate trihydrate was obtained from the reaction between barium D-glucose 6-phosphate heptahydrate and bis(cyclohexylammonium) sulfate in water. $BaSO_4$ was filtered off and the title compound was grown from water solution as large many-faced colorless crystals. D_m by flotation in a C_6H_5Cl/CH_2Cl_2 mixture. Preliminary examination by rotation and Weissenberg photographs. A specimen 0·6 × 0·5 × 0·6 mm was cut from a large crystal. Syntex $P2_1$ diffractometer, Mo $K\alpha$ radiation for lattice parameters (15 reflections in the range 21 < 2θ < 26°) and intensity measurements; θ–2θ scan technique; 5110 reflections measured up to 2θ = 50°, h 0→11, k −15→14, l −13→13 (some Friedel opposites measured and not averaged); two standards measured after every 50 reflections, variation ∓7%. Absorption and extinction ignored. Structure solved

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