Fig. 3. The torsion angles (°). Asymmetry parameters (Duax & Norton, 1975) are: $\Delta C_s^P = 0.9$ (2), $\Delta C_s^{C(6)} = 5.3$ (2), $\Delta C_s^{O(1)} = 6.3$ (3), $\Delta C_2^{C(5),C(6)} = 5.4$ (4), $\Delta C_2^{P,O(1)} = 5.8$ (4), $C_2^{O(1),C(6)} = 6.3$ (4)°.

In terms of stereoelectronic and steric effects being considered as the major factors which govern the spatial orientation in these and related cyclic systems (Kirby, 1983) it is most interesting to compare the structural features of the title compound (1) with those obtained for compound (2), where the anilino group occupies an axial position.

The axial P—N bond |1·599 (3) Å| in compound (2) is longer than the equatorial one in (1), and the length of the P=Se bond shows the same tendency: the axial P=Se bond in compound (1) |2·081 (1) Å| is slightly longer than the equatorial one in (2) |2·068 (1) Å|. However, it is also worth pointing out that the N—C bond in (1) is longer |1·456 (5) Å| than the N—C bond in (2) |1·422 (6) Å|. This comparison may indicate a substantial $p_\pi-p_\pi$ interaction between the sp^2-hybridized N lone pair and the phenyl-ring π system in compound (2). Thus repulsive interactions of the filled p orbital on an axial N atom bearing a phenyl substituent, as in (2),

with the endocyclic P—O bonding orbitals are weaker than the corresponding interaction of the axially oriented P=Se bond orbitals, and this accounts for the axial orientation of the anilino group in (2). However, if the exocyclic N atom bearing the ethyl group were to occupy an axial position, the repulsive interactions of its p orbital with the endocyclic P—O bonding orbital could be stronger than those between the endocyclic P—O bonding orbitals and the P=Se bond orbital and for this reason the ethylamino group prefers to occupy the equatorial position as found in (1).

All other bonds and angles have typical values for this class of compounds and will not be discussed in detail.

References

BARTCZAK, T. J., GALDECKI, Z., TRZEŹWIŃSKA, H. B. & WOLF, W. M. (1983). Acta Cryst. C39, 731–732.
BARTCZAK, T. J. & WOLF, W. M. (1983). Acta Cryst. C39, 224–227.
DUAX, W. L. & NORTON, D. A. (1975). Atlas of Steroid Structure, p. 16. New York: Plenum.
GRAND, A. & ROBERT, J. B. (1978). Acta Cryst. B34, 199–204.
International Tables for X-ray Crystallography (1974). Vol. IV. Birmingham: Kynoch Press. (Present distributor D. Reidel, Dordrecht.)
KIRBY, A. T. (1983). The Anomeric Effect and Related Stereoelectronic Effects at Oxygen. p. 4. Berlin: Springer-Verlag.
LESIAK, K., LEŚNIKOWSKI, Z. J., STEC, W. J. & ZIELIŃSKA, B. (1979). Pol. J. Chem. 53, 2041–2045.
SHELDRICK, G. M. (1976). SHELX76. Program for crystal structure determination. Univ. of Cambridge, England.

Dimethylammonium Picrate: Geometry and Interactions of the Picrate Ion

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Abstract. $C_2H_8N^+C_6H_2N_3O_7^-$, $M_r = 274.2$, orthorhombic, $Pb2_1a$ (non-standard setting of $Pca2_1$), $a = 10.001$ (1), $b = 11.087$ (4), $c = 21.332$ (2) Å, $V = 2365.3$ Å^3, $Z = 8$, $D_x = 1.54$ g cm^{-3}, λ(Mo $K\alpha$) = 0.71069 Å, $\mu = 1.28$ cm^{-1}, $F(000) = 1136$, $T = 293$ K. Final $R = 0.049$ from 1494 observed reflections. The structure is composed of alternating sheets of cations and anions. There are two picrate ions in the asymmetric unit and adjacent anion sheets are related by a pseudo crystallographic twofold axis. Aromatic bond lengths in the picrate ring range between 1·45 and 1·36 Å and can be explained by a resonance description of the picrate ion.

Introduction. The picrate ion is particularly useful in forming organic crystalline salts, and over 60 such X-ray structures are in the literature. It is a strong electron acceptor and can also form specific hydrogen bonds. A series of structures including seratonin picrate (Thewalt & Bugg, 1972) and tryptophan picrate (Gartland, Freeman & Bugg, 1974) have been studied to examine the π-acceptor properties of the picrate ion. Structures of salts with small cations like ammonium picrate and potassium picrate (Maartman-Moe, 1969) showed for the first time the effect of the substituent groups on the geometry of the aromatic ring. This structure determination was undertaken to study the geometry of the picrate ion in an attempt to explain the significant differences between bond lengths in the aromatic ring, and also to examine the hydrogen-bonded interaction between cation and anion.

Experimental. Pale-yellow crystal $0.35 \times 0.3 \times 0.45$ mm; Nonius CAD-4 diffractometer with graphite monochromator, $2\theta/\omega$ scan; 20 reflections used for measuring lattice parameters, $10 \le \theta \le 15°$; no correction for absorption; intensities of 2190 unique reflections measured out to $\theta = 25°$, range of hkl: $h\,0\rightarrow11$, $k\,0\rightarrow13$, $l\,0\rightarrow25$, 1494 reflections with $I > 2.5\sigma(I)$ used in refinement; no variation in intensity of standard reflection. Structure solved by direct methods (*SHELX*84, Sheldrick, 1984) and refined by blocked-matrix least squares on F. There are two crystallographically unrelated dimethylammonium picrate ion pairs in the asymmetric unit and both picrate ions were refined in one block with the cations in another block. All non-H atoms were refined anisotropically. The methyl-group H atoms were refined as rigid groups with idealized geometries and isotropic thermal parameters for the three H atoms on each of the four methyl groups were constrained to be equal (Sheldrick, 1976). All other H atoms were located and refined with individual isotropic thermal parameters. Average Δ/σ in final refinement cycle $= 0.08$ with a maximum value of 0.5. Maximum and minimum peak heights in final difference map 0.28 and $0.22\,e\,\text{Å}^{-3}$ respectively. Weighting scheme $w = 1/[\sigma^2(F) + 0.005\,F^2]$ gave final $R = 0.049$, $wR = 0.068$. No correction for primary extinction. Atomic scattering factors from *SHELX*76 (Sheldrick, 1976). Computer: ICL 2972.

Discussion. Table 1 contains positional and mean thermal parameters for all atoms.* Bond lengths, angles and selected torsion angles are given in Table 2. Labelled drawings of the picrate ions are given in Fig. 1, and a packing diagram is given in Fig. 2.

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* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 42535 (11 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. *Fractional coordinates of atoms with standard deviations and isotropic thermal parameters* (Å^2)

$$U_{eq} = (U_{11} + U_{22} + U_{33})/3.$$

|	x	y	z	U_{eq}/U_{iso}
C(11)	0.0942 (6)	0.5590 (6)	0.39955 (24)	0.0338
C(12)	−0.0054 (5)	0.6448 (7)	0.42306 (24)	0.0346
C(13)	−0.0249 (7)	0.6708 (7)	0.4857 (3)	0.0395
C(14)	0.0512 (7)	0.6164 (7)	0.5296 (3)	0.0394
C(15)	0.1508 (7)	0.5333 (7)	0.51274 (24)	0.0409
C(16)	0.1687 (6)	0.5072 (6)	0.4495 (3)	0.0348
O(11)	0.1076 (4)	0.53290 (<1)	0.34282 (16)	0.0477
N(12)	−0.0834 (6)	0.7157 (6)	0.37987 (25)	0.0439
O(122)	−0.0747 (5)	0.7018 (6)	0.32368 (20)	0.0686
O(121)	−0.1520 (7)	0.7954 (7)	0.39975 (23)	0.1022
N(14)	0.0356 (7)	0.6450 (7)	0.59576 (25)	0.0545
O(141)	−0.0530 (7)	0.7136 (7)	0.60982 (22)	0.0835
O(142)	0.1110 (5)	0.6026 (6)	0.63347 (20)	0.0684
N(16)	0.2776 (6)	0.4224 (6)	0.43694 (22)	0.0468
O(161)	0.3187 (7)	0.3614 (8)	0.4802 (3)	0.1232
O(162)	0.3258 (5)	0.4199 (8)	0.38624 (20)	0.0852
C(21)	0.0849 (6)	0.4014 (7)	0.1049 (3)	0.0360
C(22)	−0.0081 (6)	0.3158 (7)	0.0810 (3)	0.0401
C(23)	−0.0226 (7)	0.2894 (8)	0.0183 (3)	0.0428
C(24)	0.0584 (7)	0.3510 (7)	−0.02568 (25)	0.0445
C(25)	0.1511 (7)	0.4336 (8)	−0.0064 (3)	0.0461
C(26)	0.1665 (7)	0.4553 (7)	0.0554 (3)	0.0471
O(21)	0.0961 (4)	0.4308 (4)	0.16122 (18)	0.0502
N(22)	−0.887 (7)	0.2408 (7)	0.12269 (24)	0.0522
O(221)	−0.1691 (7)	0.1727 (8)	0.09916 (22)	0.1049
O(222)	−0.0747 (6)	0.2513 (6)	0.17891 (18)	0.0803
N(24)	0.0420 (6)	0.3201 (8)	−0.09076 (25)	0.0610
O(241)	0.1226 (7)	0.3662 (7)	−0.12800 (23)	0.0928
O(242)	−0.0478 (7)	0.2486 (8)	−0.10674 (22)	0.1013
N(26)	0.2665 (6)	0.5419 (8)	0.07192 (22)	0.0601
O(261)	0.3003 (7)	0.6148 (8)	0.0332 (3)	0.1178
O(262)	0.3191 (6)	0.5431 (8)	0.12434 (25)	0.0967
N(4)	0.2893 (4)	0.4229 (4)	0.25459 (17)	0.0457
C(41)	0.4289 (5)	0.4663 (7)	0.2535 (3)	0.0645
C(42)	0.2752 (7)	0.2903 (6)	0.2565 (3)	0.0749
N(3)	0.4057 (3)	0.4805 (5)	0.74825 (23)	0.0400
C(31)	0.3306 (6)	0.3960 (7)	0.7080 (3)	0.0668
C(32)	0.3213 (7)	0.5599 (6)	0.7864 (3)	0.0707
H(13)	−0.098 (4)	0.737 (4)	0.5044 (20)	0.0195
H(15)	0.215 (6)	0.489 (5)	0.5387 (24)	0.0387
H(23)	0.091 (6)	0.244 (5)	0.014 (3)	0.0374
H(25)	0.164 (9)	0.491 (9)	−0.036 (4)	0.0995
H(41)	0.251 (5)	0.425 (5)	0.2218 (18)	0.0390
H(42)	0.253 (9)	0.452 (7)	0.289 (3)	0.0936
H(411)	0.4884 (5)	0.4514 (7)	0.2120 (3)	0.1073
H(412)	0.4684 (5)	0.4131 (7)	0.2916 (3)	0.1073
H(413)	0.4321 (5)	0.5606 (7)	0.2659 (3)	0.1073
H(421)	0.3310 (7)	0.2574 (6)	0.2964 (3)	0.1113
H(422)	0.3270 (7)	0.2666 (6)	0.2139 (3)	0.1113
H(423)	0.1769 (7)	0.2501 (6)	0.2564 (3)	0.1113
H(31)	0.477 (6)	0.532 (6)	0.7204 (23)	0.0693
H(32)	0.454 (5)	0.450 (5)	0.7727 (19)	0.0274
H(311)	0.3938 (6)	0.3418 (7)	0.6781 (3)	0.0954
H(312)	0.2766 (6)	0.4607 (7)	0.6798 (3)	0.0954
H(313)	0.2604 (6)	0.3385 (7)	0.7322 (3)	0.0954
H(321)	0.3880 (7)	0.6226 (6)	0.8092 (3)	0.0943
H(322)	0.2555 (7)	0.5200 (6)	0.8207 (3)	0.0943
H(323)	0.2628 (7)	0.6071 (6)	0.7516 (3)	0.0943
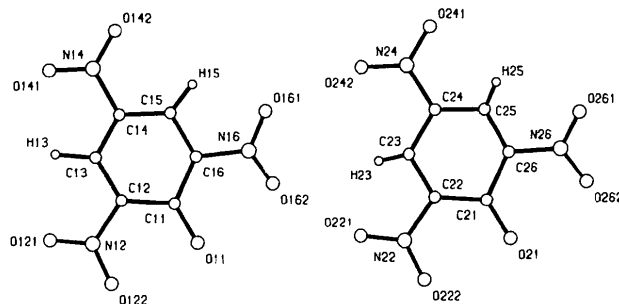
Fig. 1. Labelled drawings of the two independent picrate ions.

Table 2. *Bond lengths* (Å), *angles* (°) *and selected torsion angles* (°)

These are compared with average bond lengths and angles calculated from 31 structures from the CSD. Chemically equivalent parameters have been merged and, when duplicated, are shown in parentheses. Estimated standard deviations $= |\sum_{i=1}^{n}(\bar{x}-x_i)^2/n-1|^{1/2}$ are also given for the average parameters.

	Ion (1) (n = 1)	Ion (2) (n = 2)	Database
C(n1)–C(n2)	1·465 (9)	1·422 (9)	1·452 (9)
C(n1)–C(n6)	1·422 (9)	1·462 (10)	(1·452)
C(n1)–O(n1)	1·252 (7)	1·251 (8)	1·241 (11)
C(n2)–C(n3)	1·382 (9)	1·376 (10)	1·367 (10)
C(n2)–N(n2)	1·441 (9)	1·461 (9)	1·459 (9)
C(n3)–C(n4)	1·350 (9)	1·415 (10)	1·379 (10)
C(n4)–C(n5)	1·404 (9)	1·367 (10)	(1·379)
C(n4)–N(n4)	1·454 (9)	1·439 (10)	1·451 (18)
C(n5)–C(n6)	1·391 (9)	1·348 (10)	(1·367)
C(n6)–N(n6)	1·463 (9)	1·431 (10)	(1·459)
N(n2)–O(n22)	1·212 (8)	1·213 (8)	1·215 (8)
N(n2)–O(n21)	1·196 (9)	1·212 (10)	(1·215)
N(n4)–O(n41)	1·205 (9)	1·241 (10)	1·225 (8)
N(n4)–O(n42)	1·199 (9)	1·246 (10)	(1·225)
N(n6)–O(n61)	1·215 (9)	1·205 (10)	(1·215)
N(n6)–O(n62)	1·184 (9)	1·236 (10)	(1·215)
N(4)–C(41)	1·477 (8)		
N(4)–C(42)	1·477 (8)		
N(3)–C(31)	1·476 (8)		
N(3)–C(32)	1·466 (8)		
C(n2)–C(n1)–C(n6)	111·2 (5)	112·2 (6)	111·1 (10)
C(n2)–C(n1)–O(n1)	123·7 (5)	125·3 (6)	124·9 (11)
C(n6)–C(n1)–O(n1)	125·1 (5)	122·5 (6)	123·9 (14)
C(n1)–C(n2)–C(n3)	124·3 (6)	124·0 (6)	124·6 (8)
C(n1)–C(n2)–N(n2)	120·2 (5)	121·4 (6)	119·0 (14)
C(n3)–C(n2)–N(n2)	115·3 (6)	114·4 (6)	116·4 (8)
C(n2)–C(n3)–C(n4)	119·9 (6)	118·8 (6)	118·9 (7)
C(n3)–C(n4)–C(n5)	121·0 (6)	120·8 (6)	121·6 (8)
C(n3)–C(n4)–N(n4)	121·0 (6)	117·4 (6)	119·2 (7)
C(n5)–C(n4)–N(n4)	117·9 (6)	121·8 (6)	119·1 (7)
C(n4)–C(n5)–C(n6)	118·5 (6)	119·4 (7)	(118·9)
C(n1)–C(n6)–C(n5)	125·1 (6)	124·7 (7)	(124·6)
C(n1)–C(n6)–N(n6)	120·8 (5)	119·1 (6)	(119·0)
C(n5)–C(n6)–N(n6)	114·0 (5)	116·1 (7)	(116·4)
C(n2)–N(n2)–O(n22)	121·6 (6)	119·0 (6)	119·6 (25)
C(n2)–N(n2)–O(n21)	119·1 (6)	117·9 (6)	118·2 (16)
O(n22)–N(n2)–O(n21)	119·1 (6)	123·1 (7)	122·0 (21)
C(n4)–N(n4)–O(n41)	117·3 (6)	116·5 (7)	118·1 (8)
C(n4)–N(n4)–O(n42)	119·8 (6)	119·8 (7)	(118·1)
O(n41)–N(n4)–O(n42)	122·9 (7)	123·7 (7)	123·7 (10)
C(n6)–N(n6)–O(n61)	118·1 (6)	118·5 (7)	118·7 (12)
C(n6)–N(n6)–O(n62)	119·0 (6)	121·9 (7)	118·8 (12)
O(n61)–N(n6)–O(n62)	122·8 (7)	119·6 (7)	122·5 (16)
C(41)–N(4)–C(42)	114·5 (4)		
C(31)–N(3)–C(32)	114·2 (5)		
O(n22)–N(n2)–C(n2)–C(n1)	4·3 (9)	–0·7 (10)	
O(n21)–N(n2)–C(n2)–C(n3)	4·4 (9)	–7·4 (10)	
O(n41)–N(n4)–C(n4)–C(n3)	4·3 (10)	–173·2 (7)	
O(n42)–N(n4)–C(n4)–C(n5)	3·6 (10)	–176·7 (7)	
O(n62)–N(n6)–C(n6)–C(n1)	18·9 (9)	25·8 (11)	
O(n61)–N(n6)–C(n6)–C(n5)	17·8 (9)	22·4 (10)	
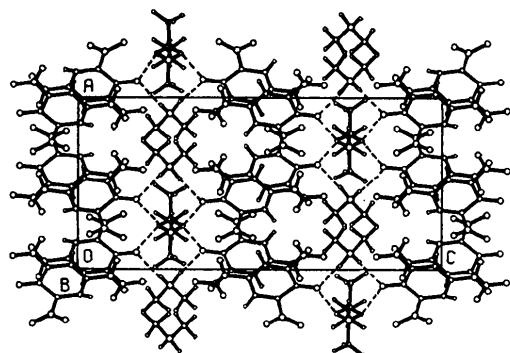
Fig. 2. A projection of the unit cell viewed along the *b* axis.

Picrate-ion geometry

A Cambridge Structural Database (CSD) search (Allen *et al.*, 1979) identified 65 references to structures containing the picrate ion. Structures showing disorder or with an *R* factor greater than 0·09 were removed and the 31 remaining structures were used to provide the average bond lengths and angles given in Table 2. Chemically equivalent parameters in the average structure have been merged.

There are no significant differences in bond lengths or angles between the picrate ions in this structure and the database averaged parameters. The geometry of the picrate ring with C(1)–C(2) = 1·452 (9), C(2)–C(3) = 1·367 (10) and C(3)–C(4) = 1·379 (10) Å (see Fig. 3 for basic numbering scheme) shows significant distortions from the standard benzene ring with C–C bonds of 1·395 Å. There are also significant differences between the intra-ring angles: C(6)–C(1)–C(2) = 111·1 (10), C(1)–C(2)–C(3) = 124·6 (8) and C(2)–C(3)–C(4) = 118·9 (7)°. These differences in bond lengths and angles fit with a simple resonance scheme shown in Fig. 3 in which the negative charge is partially delocalized round the three nitro groups. Bond lengths now correlate with the degree of double-bond character: the shortest bond, C(2)–C(3), has the most double-bond character while the longest, C(1)–C(2), has the least. Despite large ($\pm5°$) distortions from 120°, all C and N atoms in this structure show planar coordination, with the three angles round each atom in both picrate ions summing to $360\pm0\cdot2°$.

The survey of all picrate structures shows that the *ortho*-nitro groups tend to twist significantly out of the plane of the benzene ring within the range $\pm50°$. The average value for the torsion angle O(22)–N(2)–C(2)–C(1) = |24°|. In contrast, the *para*-nitro substituent is much more strictly coplanar with the benzene ring and shows an average twist about the N(4)–C(4) bond of 2° |O(41)–N(4)–C(4)–C(3) = |2°||.

Space group and packing

Systematic absences (0*kl*, *k* odd and *hk*0, *h* odd) define the space group to be either *Pb2₁a* or *Pbma*. Refinement shows *Pb2₁a* to be correct. There are, however, a set of significantly weak reflections (*h0l*, *h*+*l* odd) and out of a total of 125 reflections of this class only 40 have $I > 3\sigma(I)$. This implies a pseudo higher-
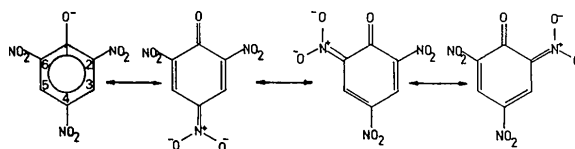

Fig. 3. The likely resonance forms of the picrate ion explaining the differences in C–C bond lengths. C atoms are numbered on the left-hand drawing.

symmetry space group *Pbna* (alternative setting for *Pbcn*) with a twofold rotation axis at $x, 0, \frac{1}{4}$. Inspection of the packing diagram in Fig. 2 shows that this is indeed the case and all atom positions in picrate ion (1) are closely approximated to atom positions of ion (2) by the pseudo-operator $(x, -y, \frac{1}{2}-z)$. The position and orientation of the dimethylammonium ions break the pseudosymmetry. N(3) and N(4) of the dimethylammonium ions are well defined (U_{eq} 0·04 Å^2) and lie 0·2 and 0·8 Å respectively off the pseudo twofold axis.

The structure is clearly laminar with sheets of anions hydrogen-bonded to sheets of cations. Picrate ions are only related to each other by symmetry within the sheets; adjacent sheets are only related by the pseudo twofold axis. The strongest interactions are hydrogen bonds between the deprotonated O on the picrate ions and the ammonium H atoms. Each O acts as a hydrogen-bond acceptor for two H atoms providing four crystallographically unrelated hydrogen bonds as given in Table 3 and Fig. 2. H(42) and H(31) may also form bifurcated hydrogen bonds to O(162) and O(122) respectively.

All other non-bonded contacts (Table 3) are within expected ranges for van der Waals interactions, though the short contacts involving the picrate aromatic H atoms to O(n21) and O(n61) ranging between 2·4 and 2·7 Å suggest a genuine electrostatic interaction.

Table 3. *Intermolecular interactions*

(i) Hydrogen bonding

N(3)···O(21)	2·767 Å	H(32)···O(21)	2·013 Å
N(4)···O(21)	2·777	H(41)···O(21)	2·019
N(3)···O(11)	2·861	H(31)···O(11)	1·876
N(4)···O(11)	2·887	H(42)···O(11)	2·064
N(4)···O(162)	2·832	H(42)···O(162)	2·231
N(4)···O(262)	3·096	H(41)···O(262)	2·551
N(3)···O(122)	2·900	H(31)···O(122)	2·163
N(3)···O(222)	2·985	H(32)···O(222)	2·445

(ii) van der Waals contacts

C(11)···H(31)	2·829 Å	H(25)···O(221)	2·422 Å
C(21)···H(32)	2·971	H(15)···O(121)	2·595
C(13)···O(161)	2·955	H(23)···O(261)	2·731
C(23)···O(261)	2·965	H(13)···O(161)	2·623

References

ALLEN, F. H., BELLARD, S., BRICE, M. D., CARTWRIGHT, B. A., DOUBLEDAY, A., HIGGS, H., HUMMELINK, T., HUMMELINK-PETERS, B. G., KENNARD, O., MOTHERWELL, W. D. S., RODGERS, J. R. & WATSON, D. G. (1979). *Acta Cryst.* B35, 2331–2339.
GARTLAND, G. L., FREEMAN, G. R. & BUGG, C. E. (1974). *Acta Cryst.* B30, 1841–1849.
MAARTMAN-MOE, K. (1969). *Acta Cryst.* B25, 1452–1460.
SHELDRICK, G. M. (1976). *SHELX76.* A program for crystal structure determination. Univ. of Cambridge, England.
SHELDRICK, G. M. (1984). *SHELX84.* A program for crystal structure determination. Univ. of Göttingen, Federal Republic of Germany.
THEWALT, U. & BUGG, C. E. (1972). *Acta Cryst.* 28, 82–92.

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Lead Indium Bismuth Chalcogenides. II. Structure of Pb$_4$In$_3$Bi$_7$S$_{18}$

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Abstract. $M_r = 3213\cdot23$, monoclinic, $P2_1/m$, $a = 21\cdot021$ (5), $b = 4\cdot014$ (2), $c = 18\cdot898$ (5) Å, $\beta = 97\cdot07(2)°$, $V = 1582\cdot46$ Å^3, $Z = 2$, $D_x = 6\cdot744$ g cm^{-3}, $\lambda(Ag\,K\alpha) = 0\cdot56083$ Å, $\mu = 341\cdot8$ cm^{-1}, $F(000) = 2688$, room temperature, final $R = 0\cdot042$ for 3972 independent observed reflections. The structure consists of In—S octahedra [In—S 2·583 (12)–2·853 (8) Å], in which In^{3+} is partially substituted by Bi^{3+} as indicated by the corresponding site-occupancy factor, mono- and bicapped trigonal Pb—S prisms |Pb—S 2·811 (8)–3·464 (13) Å|, and Bi—S octahedra |Bi—S 2·625 (10)–3·287 (13) Å| as well as Bi—S$_5$ pyramids |Bi—S 2·735 (7)–3·018 (8) Å| with two additional S |Bi—S 2·817 (10), 3·398 (12) and 2·840 (9), 3·509 (12) Å| below the basal plane; all polyhedra are distorted and form chains along *y*. The In—S chains are mutually