and $R_{Si(Ge)}$ are the atomic radii of T and Si(Ge) respectively) increases. This increase helps the formation of compounds whose structures have smaller coordination numbers.

It is characteristic of all phases with the Ru_2Ge_3 type that their a/c ratio is 2. If this ratio were exactly 2, the transition element partial structure would be strictly tetragonal and of the β-tin type. In this respect the Ru_2Ge_3 type structures belong to the chimney-ladder structures although it seems as yet impossible to explain the deviations of the B element sites from those normally found in chimney-ladder structures.

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Structural Investigations of Ylides. V.* The Crystal and Molecular Structure of 3-Ethyl-1,2,5-trimethyl-3*H*-benzo[*c*][1,2,5]triazepino[1,2-*a*]cinnoline-1,2,3,5-tetracarboxylate

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Although not itself an ylide, the title compound is formed directly from an ylide by 1,5-dipolar cycloaddition of dimethyl acetylenedicarboxylate. The crystals are triclinic, space group $A\bar{1}$, with $Z=4$, $a=15\cdot245$, $b=10\cdot212$, $c=17\cdot612$ Å, $\alpha=89\cdot81$, $\beta=117\cdot65$, $\gamma=90\cdot47°$. The structure was solved by direct methods from 2747 independent reflexions collected on a diffractometer, and has been refined by least-squares calculations to a final R of 0·060. The analysis confirms that the ylide behaves as a 6π-electron 1,5-dipole in its reactions with esters of acetylenedicarboxylic acid.
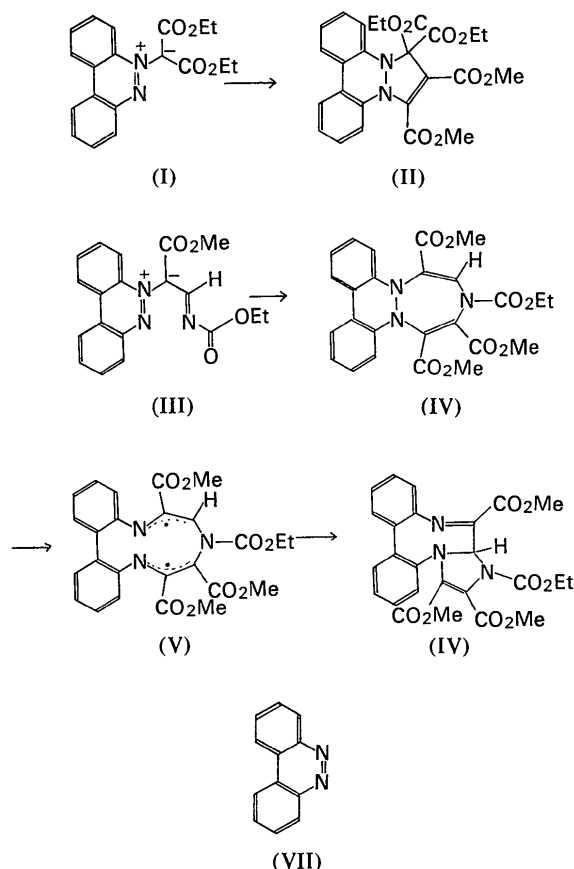
Introduction

1,3-Dipolar cycloaddition of azomethine imines is well known (Huisgen, 1963). Thus the benzocinnolinium ylide (I) (Gait, Rance, Rees & Storr, 1972a) yields the adduct (II) when treated with dimethyl acetylenedicar-

boxylate in dimethylformamide at 70°C for 30 min. However, under identical reaction conditions the derivative (III) does not give analogous products, yielding instead a compound to which the structure (VI) has been tentatively assigned (Gait, Rance, Rees & Storr, 1972b). Moreover, when the reaction of (III) is carried out at room temperature over 24 h, a second isomeric adduct, assigned the structure (IV), is isolated together with (VI). The adduct (IV) rearranges under

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* Part IV: Cameron, Hair & Morris (1974). *Acta Cryst.* B**30**, 221–225.

mild conditions to yield (VI), and it is postulated that this rearrangement takes place *via* the diradical intermediate (V).

(I)

(II)

(III)

(IV)

(V)

(IV)

(VII)

In order to confirm that the ylide (III) functions as a 6π-electron 1,5-dipole in its reactions with dimethyl acetylenedicarboxylate, we have undertaken a crystal-structure analysis of (IV). Further attempts to investigate the reaction sequence in more detail by solving the crystal structure of (VI) have proved unsuccessful.

Experimental

Crystal data

$C_{25}H_{23}N_3O_8$, $M = 493\cdot5$, triclinic,
$a = 15\cdot245$ (2), $b = 10\cdot212$ (1), $c = 17\cdot612$ (3) Å,
$\alpha = 89\cdot81$ (4), $\beta = 117\cdot65$ (3), $\gamma = 90\cdot47$ (6)°,
$U = 2430\cdot1$ Å³, $D_m = 1\cdot41$ g cm⁻³ (by flotation),
$Z = 4$, $D_c = 1\cdot35$ g cm⁻³. $F(000) = 968\cdot0$.
Space group $A\bar{1}$. Mo $K\alpha$ radiation, $\lambda = 0\cdot7107$ Å,
μ(Mo $K\alpha$) = $1\cdot10$ cm⁻¹.

Crystallographic measurements

The cell parameters were initially determined from oscillation and Weissenberg photographs taken with Cu $K\alpha$ ($\lambda = 1\cdot5418$ Å) radiation and from precession photographs taken with Mo $K\alpha$ ($\lambda = 0\cdot7107$ Å) radia-

tion, these values being refined by least-squares techniques prior to the diffractometer data collection. Systematic absences indicated the possible space groups $A\bar{1}$ and $A1$, the former being chosen on the bases of the racemic nature of the compound and intensity statistics. This choice was subsequently vindicated by successful refinement. To avoid working with a more oblique cell, the conversion to the corresponding primitive lattice was not made.

Intensity measurements were made on a Hilger & Watts Y290 diffractometer with Mo $K\alpha$ radiation. For a small crystal ($0\cdot4 \times 0\cdot3 \times 0\cdot1$ mm), 2747 independent reflexions ($I > 2\sigma_I$, $\sigma_I = \sqrt{I + B_1 + B_2}$) were recorded with the θ, ω scan technique in the range $2\theta = 0 \rightarrow 50°$. The intensities were corrected for Lorentz and polarization factors, but absorption effects were considered negligible and were ignored.

Structure determination and refinement

The structure was solved by the symbolic-addition method, an initial E map based on 276 reflexions with $|E| > 1\cdot60$ revealing the complete structure.

The refinement of positional, vibrational and overall scale parameters by least-squares calculations converged after 11 cycles when R was $0\cdot060$. In cycle 6,

Table 1. *Atomic parameters*

(a) Fractional coordinates

	x/a	y/b	z/c
C(1)	0·26280 (37)	0·77184 (47)	−0·06570 (33)
C(2)	0·23418 (38)	0·75243 (48)	−0·14844 (32)
C(4)	0·14444 (39)	0·53781 (50)	−0·17400 (32)
C(5)	0·18678 (38)	0·49008 (48)	−0·09406 (32)
C(6a)	0·33640 (38)	0·53177 (49)	0·04322 (33)
C(7)	0·39422 (44)	0·44215 (55)	0·02811 (40)
C(8)	0·49107 (48)	0·41734 (71)	0·09405 (45)
C(9)	0·52691 (49)	0·48295 (65)	0·17164 (47)
C(10)	0·46775 (46)	0·57286 (61)	0·18627 (39)
C(10a)	0·37070 (41)	0·59798 (52)	0·12209 (34)
C(11a)	0·30502 (42)	0·69225 (54)	0·13133 (34)
C(11)	0·31367 (50)	0·73245 (67)	0·21077 (39)
C(12)	0·24815 (56)	0·82664 (74)	0·21192 (45)
C(13)	0·17534 (55)	0·88299 (71)	0·13686 (47)
C(14)	0·16372 (47)	0·84160 (59)	0·05669 (42)
C(14a)	0·30502 (42)	0·69225 (54)	0·13133 (34)
C(16)	0·34974 (42)	0·85921 (53)	−0·01065 (36)
C(17)	0·40029 (65)	1·07047 (74)	0·04837 (60)
C(18)	0·29004 (42)	0·81180 (57)	−0·19051 (36)
C(19)	0·33539 (59)	0·79224 (80)	−0·30214 (48)
C(20)	0·09976 (64)	0·14555 (63)	−0·14184 (49)
C(21)	0·04951 (45)	0·87321 (58)	−0·38296 (35)
C(22)	0·07982 (57)	0·01854 (68)	−0·37692 (45)
C(23)	0·16488 (41)	0·35331 (53)	−0·07850 (34)
N(3)	0·15535 (31)	0·66518 (41)	−0·19884 (26)
N(6)	0·23850 (32)	0·56199 (38)	−0·01841 (26)
N(15)	0·21627 (31)	0·69949 (39)	−0·02533 (26)
O(1)	0·43301 (30)	0·82217 (42)	0·02382 (29)
O(2)	0·31998 (33)	0·97756 (39)	−0·00367 (30)
O(3)	0·33689 (37)	0·91147 (46)	−0·16672 (29)
O(4)	0·28522 (30)	0·73812 (39)	−0·25424 (25)
O(5)	0·03002 (30)	0·62717 (38)	−0·33461 (24)
O(6)	0·09620 (27)	0·82839 (35)	−0·29351 (22)
O(7)	0·17685 (35)	0·31280 (39)	−0·01083 (26)
O(8)	0·12990 (34)	0·28124 (37)	−0·14992 (25)

Table 1 (cont.)

(b) Anisotropic temperature factors (Å²)

	U_{11}	U_{22}	U_{33}	$2U_{23}$	$2U_{31}$	$2U_{12}$
C(1)	0·0343	0·0290	0·0421	−0·0016	0·0179	0·0014
C(2)	0·0367	0·0324	0·0384	−0·0037	0·0162	0·0025
C(3)	0·0424	0·0343	0·0385	0·0004	0·0219	0·0030
C(5)	0·0426	0·0300	0·0383	−0·0034	0·0216	−0·0028
C(6a)	0·0377	0·0377	0·0394	−0·0049	0·0150	0·0040
C(7)	0·0506	0·0392	0·0639	0·0029	0·0297	0·0061
C(8)	0·0538	0·0466	0·0803	0·0023	0·0270	0·0086
C(9)	0·0522	0·0540	0·0785	0·0011	0·0133	0·0101
C(10)	0·0512	0·0529	0·0529	−0·0075	0·0106	0·0074
C(10a)	0·0458	0·0385	0·0407	−0·0054	0·0176	0·0017
C(11a)	0·0481	0·0453	0·0405	−0·0139	0·0226	−0·0070
C(11)	0·0689	0·0718	0·0479	−0·0220	0·0315	−0·0186
C(12)	0·0832	0·0837	0·0676	−0·0239	0·0480	−0·0354
C(13)	0·0761	0·0726	0·0789	−0·0116	0·0481	−0·0290
C(14)	0·0586	0·0496	0·0718	−0·0076	0·0416	−0·0190
C(14a)	0·0481	0·0453	0·0405	−0·0014	0·0226	−0·0070
C(16)	0·0509	0·0387	0·0448	−0·0056	0·0258	0·0004
C(17)	0·1035	0·0613	0·1295	−0·0484	0·0600	−0·0527
C(18)	0·0458	0·0501	0·0431	−0·0005	0·0160	0·0108
C(19)	0·0885	0·1021	0·0767	0·0029	0·0631	0·0250
C(20)	0·1157	0·0335	0·0788	−0·0283	0·0264	−0·0056
C(21)	0·0578	0·0495	0·0378	0·0030	0·0152	0·0160
C(22)	0·0858	0·0596	0·0632	−0·0077	0·0203	0·0224
C(23)	0·0473	0·0402	0·0393	−0·0054	0·0182	−0·0015
N(3)	0·0394	0·0380	0·0309	−0·0040	0·0142	0·0019
N(6)	0·0436	0·0277	0·0343	−0·0012	0·0156	−0·0010
N(15)	0·0408	0·0284	0·0363	−0·0009	0·0189	−0·0014
O(1)	0·0423	0·0570	0·0773	−0·0063	0·0152	0·0026
O(2)	0·0655	0·0449	0·0853	−0·0123	0·0397	−0·0177
O(3)	0·0876	0·0743	0·0664	−0·0386	0·0418	−0·0041
O(4)	0·0607	0·0580	0·0494	0·0043	0·0352	0·0083
O(5)	0·0585	0·0502	0·0385	−0·0093	0·0082	−0·0008
O(6)	0·0486	0·0385	0·0361	−0·0008	0·0134	0·0055
O(7)	0·0906	0·0475	0·0464	−0·0168	0·0282	0·0029
O(8)	0·0884	0·0374	0·0458	−0·0146	0·0235	−0·0063

Average estimated standard deviations (Å²)

C	0·0040	0·0037	0·0039	0·0030	0·0033	0·0030
O	0·0025	0·0024	0·0023	0·0019	0·0020	0·0018
N	0·0028	0·0025	0·0026	0·0021	0·0022	0·0020

Table 1 (cont.)

(c) Calculated hydrogen atom positions

	x/a	y/b	z/c
H(4)	0·1019	0·4777	−0·2198
H(7)	0·3687	0·3966	−0·0271
H(8)	0·5329	0·3537	0·0847
H(9)	0·5944	0·4656	0·2166
H(10)	0·4940	0·6183	0·2414
H(11)	0·3647	0·6953	0·2644
H(12)	0·2549	0·8543	0·2677
H(13)	0·1316	0·9493	0·1443
H(14)	0·1124	0·8791	0·0031
H(21¹)	0·0740	0·8237	−0·4167
H(21²)	−0·0227	0·8636	−0·4093

anisotropic thermal parameters were introduced, and in this and all subsequent cycles, the block-diagonal approximation was used. Positions were calculated for all non-methyl hydrogen atoms, and their contributions were included, but not refined, in cycle 6 and later refinement cycles. Throughout the refinement it was not found necessary to apply weights other than unity.*

On convergence of the refinement, an electron-density distribution and difference synthesis were evaluated. Neither revealed any error in the structure, and the refinement was considered complete. Final fractional coordinates and thermal parameters are given in Table 1. Table 2 contains all bond lengths, interbond angles, and pertinent intra- and intermolecular non-bonded distances. The estimated standard deviations quoted in parentheses in Tables 1 and 2 are derived from the inverse of the least-squares normal-equation matrix, and are best regarded as minimum values. Selected torsion angles are presented in Table 3, and details of least-squares planes in Table 4.

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* A list of structure factors has been deposited with the British Library Lending Division as Supplementary Publication No. SUP 30565 (25 pp., 1 microfiche). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.

A view of one molecule is shown in Fig. 1; Fig. 2 shows the molecular packing viewed down **b**.

Discussion

The aromatic rings of (IV) constitute a bridged biphenyl system in which the least-squares planes of the two phenyl rings are inclined at 23·3°. While this value results largely from twisting about the C(10a)–C(11a) bond [see Fig. 1], there are other minor factors involved. Thus the non-collinearity of atoms C(8), C(10a), C(11a) and C(13) is evidenced by the value of

124·4 (5)° for the valency angles C(10)C(10a)C(11a) and C(10a)C(11a)C(11), and by the value of 117·9 (5)° for the angles C(6a)C(10a)C(11a) and C(10a)C(11a)–C(14a). The C(10)···C(11) distance [3·08 Å] is also significantly longer than the C(6a)···C(14a) distance [2·82 Å]. In the case of (VII) (van der Meer, 1972), although the phenyl rings are almost coplanar, the valency angles associated with the biphenyl linking bond are almost identical with those observed for the present molecule, thus suggesting similar degrees of non-collinearity for the phenyl rings in the two molecules. The conformation of the six-membered ring containing N(6) and N(15), which completes the bridged biphenyl system, is best described in terms of the torsion angles for the ring bonds. In particular, the torsion angles N(6)C(6a)C(10a)C(11a) and N(15)–C(14a)C(11a)C(10a) have values of 2·3 and 5·7° respectively, while the angle C(6a)N(6)N(15)C(14a) is −60·7°.

The conformation of the seven-membered ring is also best described in terms of the torsion angles associated with the ring bonds. An examination of the torsion angles for the two double bonds C(1)=C(2) and C(4)=C(5) reveals that neither double-bond system is ideally planar. This factor may well reflect both the cyclic environment and bulky substitution of both bonds. It is also worthy of mention that whereas the geometries of N(6) and N(15) are those of slightly flattened tetrahedra, the group of atoms C(2), N(3), C(4) and C(20) is almost planar, while the torsion angle C(4)N(3)C(20)O(5) has a value of −8·3°.

With the exception of those dimensions already discussed, bond distances and interbond angles lie within

Table 2. *Interatomic distances* (Å) *and angles* (°) *with estimated standard deviations in parentheses*

(a) Bonded distances

C(1)—C(2)	1·328 (5)	C(12)—C(13)	1·384 (9)
C(1)—C(16)	1·515 (7)	C(13)—C(14)	1·385 (11)
C(1)—N(15)	1·416 (8)	C(14)—C(14a)	1·388 (8)
C(2)—N(3)	1·423 (7)	C(14a)–N(15)	1·442 (7)
C(2)—C(18)	1·489 (8)	N(6)—N(15)	1·439 (5)
C(4)—N(3)	1·404 (6)	N(3)—C(20)	1·403 (5)
C(4)—C(5)	1·340 (5)	C(16)—O(1)	1·187 (7)
C(5)—N(6)	1·402 (6)	C(16)—O(2)	1·321 (7)
C(5)—C(23)	1·482 (7)	C(17)—O(2)	1·482 (8)
C(6a)—N(6)	1·415 (5)	C(18)—O(3)	1·197 (8)
C(6a)—C(7)	1·378 (8)	C(18)—O(4)	1·328 (9)
C(6a)—C(10a)	1·404 (7)	C(19)—O(4)	1·480 (8)
C(7)—C(8)	1·392 (7)	C(20)—O(5)	1·199 (4)
C(8)—C(9)	1·369 (9)	C(20)—O(6)	1·324 (5)
C(9)—C(10)	1·384 (10)	C(21)—O(6)	1·469 (7)
C(10)—C(10a)	1·399 (7)	C(21)—C(22)	1·540 (8)
C(10a)–C(11a)	1·465 (8)	C(23)—O(7)	1·192 (7)
C(11a)–C(11)	1·398 (9)	C(23)—O(8)	1·341 (6)
C(11a)–C(14a)	1·404 (6)	C(24)—O(8)	1·482 (8)
C(11)—C(12)	1·383 (11)		

Table 2 (*cont.*)

(b) Interbond angles

C(2)C(1)N(15)	119·2 (3)	C(11)C(11a)C(14)a	117·7 (5)
C(2)C(1)C(16)	122·3 (5)	C(11a)C(11)C(12)	119·6 (5)
C(16)C(1)N(15)	118·2 (4)	C(11)C(12)C(13)	121·7(7)
C(1)C(2)N(3)	122·0 (4)	C(12)C(13)C(14)	120·0 (7)
C(1)C(2)C(18)	120·9 (5)	C(13)C(14)C(14a)	118·3 (5)
N(3)C(2)C(18)	116·9 (5)	C(11a)C(14a)C(14)	122·7 (5)
C(2)N(3)C(4)	124·6 (5)	C(11a)C(14a)N(15)	118·5 (5)
C(2)N(3)C(20)	119·2 (5)	C(14)C(14a)N(15)	118·9 (4)
C(4)N(3)C(20)	116·0 (5)	C(1)N(15)N(6)	113·2 (4)
N(3)C(4)C(5)	126·7 (4)	C(1)N(15)C(14a)	118·0 (4)
C(4)C(5)N(6)	126·2 (4)	N(6)N(15)C(14a)	108·6 (4)
C(4)C(5)C(23)	120·1 (4)	C(1)C(16)O(1)	123·3 (5)
N(6)C(5)C(23)	113·0 (4)	C(1)C(16)O(2)	110·8 (5)
C(5)N(6)C(6a)	123·1 (4)	O(1)C(16)O(2)	125·9 (5)
C(5)N(6)N(15)	115·0 (3)	C(16)O(2)C(17)	114·6 (5)
C(6a)N(6)N(15)	113·9 (3)	C(2)C(18)O(3)	123·6 (5)
N(6)C(6a)C(7)	123·3 (4)	C(2)C(18)O(4)	111·8 (5)
N(6)C(6a)C(10a)	115·4 (5)	O(3)C(18)O(4)	124·6 (5)
C(7)C(6a)C(10a)	121·4 (4)	C(18)O(4)C(19)	115·6 (4)
C(6a)C(7)C(8)	119·2 (5)	N(3)C(20)O(5)	123·9 (4)
C(7)C(8)C(9)	120·6 (6)	N(3)C(20)O(6)	109·8 (3)
C(8)C(9)C(10)	120·2 (5)	O(5)C(20)O(6)	126·3 (4)
C(9)C(10)C(10a)	120·7 (5)	C(20)O(6)C(21)	115·5 (5)
C(6a)C(10a)C(10)	117·9 (5)	O(6)C(21)C(22)	104·2 (5)
C(6a)C(10a)C(11a)	117·9 (4)	C(5)C(23)O(7)	124·5 (5)
C(10)C(10a)C(11a)	124·2 (5)	C(5)C(23)O(8)	111·5 (5)
C(10a)C(11a)C(11)	124·4 (4)	O(7)C(23)O(8)	124·0 (5)
C(10a)C(11a)C(14a)	117·9 (5)	C(23)O(8)C(24)	115·2 (5)

Table 2 (cont.)

(c) Intramolecular non-bonded distances < 3.40 Å

C(1)$\cdots$C(4)	3·06	O(3)$\cdots$O(2)	3·08
C(1)$\cdots$C(5)	3·05	O(4)$\cdots$C(20)	2·84
C(2)$\cdots$C(5)	3·05	O(4)$\cdots$N(3)	2·68
C(6a)$\cdots$C(14a)	2·82	O(6)$\cdots$C(15)	2·57
C(10)$\cdots$C(11)	3·08	O(6)$\cdots$C(18)	2·66
O(1)$\cdots$C(2)	3·22	O(6)$\cdots$O(3)	3·40
O(1)$\cdots$C(6a)	3·38	O(6)$\cdots$O(4)	2·80
O(1)$\cdots$C(10a)	3·21	O(5)$\cdots$C(4)	2·70
O(1)$\cdots$C(18)	3·37	O(7)$\cdots$C(6a)	3·10
O(1)$\cdots$N(15)	3·24	O(7)$\cdots$C(7)	3·33
O(2)$\cdots$C(2)	3·22	O(7)$\cdots$N(6)	2·72
O(2)$\cdots$C(14)	3·32	O(8)$\cdots$C(4)	2·68
O(2)$\cdots$C(14a)	3·16	N(3)$\cdots$N(6)	3·01
O(2)$\cdots$N(15)	3·17	N(3)$\cdots$N(15)	2·77
O(3)$\cdots$C(1)	2·88	N(15)$\cdots$C(10a)	2·82
O(3)$\cdots$C(16)	2·72	N(6)$\cdots$C(11a)	2·70
O(3)$\cdots$O(1)	3·11		

(d) Intermolecular distances ≤ 3.65 Å

C(8)$\cdots$O(4^i)	3·65
C(9)$\cdots$O(4^i)	3·42
C(7)$\cdots$C(8^i)	3·65
C(19)$\cdots$C(9^i)	3·64
C(19)$\cdots$O(7^{ii})	3·34
C(19)$\cdots$C(10^{ii})	3·55
C(19)$\cdots$C(10a^{ii})	3·55
C(23)$\cdots$O(7^{ii})	3·64
C(13)$\cdots$O(5^{iii})	3·55
O(6)$\cdots$O(5^{iv})	3·61
C(20)$\cdots$C(20^{iv})	3·51
C(20)$\cdots$O(6^{iv})	3·63
C(21)$\cdots$C(4^{iv})	3·65
C(24)$\cdots$O(5^v)	3·32
C(11)$\cdots$C(10^{vi})	3·57

Roman numerals as superscripts refer to the following equivalent positions which should be applied to the coordinates of the second atom:

i	$1-x, 1-y, \quad -z$
ii	$x, \frac{1}{2}+y, -\frac{1}{2}+z$
iii	$x, \frac{1}{2}+y, \quad \frac{1}{2}+z$
iv	$-x, \frac{3}{2}-y, -\frac{1}{2}-z$
v	$-x, \frac{1}{2}-y, -\frac{1}{2}-z$
vi	$1-x, \frac{3}{2}-y, \quad \frac{1}{2}-z$

Table 3. Selected torsion angles (°)

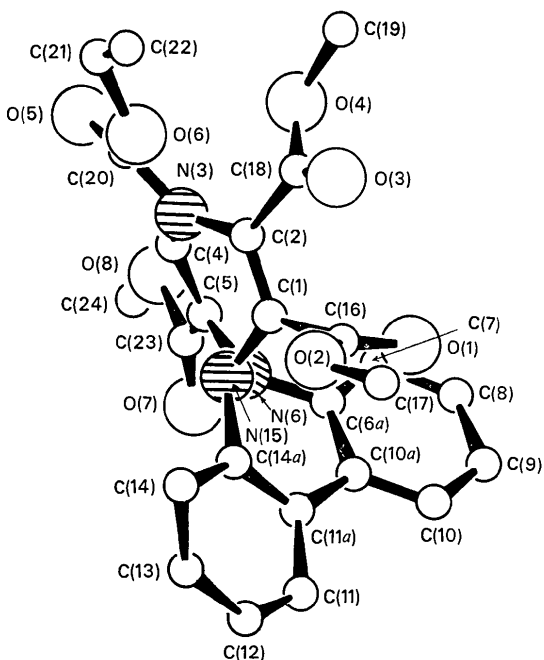|	
C(6a)C(10a)C(11a)C(14a)	−26·2
C(10)C(10a)C(11a)C(11)	−18·6
C(10a)C(11a)C(14a)N(15)	5·7
C(11a)C(14a)N(15)N(6)	35·9
C(14a)N(15)N(6)C(6a)	−60·7
N(15)N(6)C(6a)C(10a)	41·8
N(6)C(6a)C(10a)C(11a)	2·3
C(1)N(15)N(6)C(13)	−77·8
N(15)N(6)C(5)C(4)	22·8
N(6)C(5)C(4)N(3)	8·6
C(23)C(5)C(4)N(3)	178·4
C(5)C(4)N(3)C(2)	20·1
C(4)N(3)C(2)C(1)	−46·8
C(4)N(3)C(2)C(18)	127·6
N(3)C(2)C(1)N(15)	2·8
N(3)C(2)C(1)C(16)	176·8
C(18)C(2)C(1)C(16)	2·6
C(18)C(2)C(1)N(15)	−171·4
C(2)C(1)N(15)N(6)	67·9
C(4)N(3)C(20)O(5)	−8·3

Fig. 1. A view of one molecule showing the atomic numbering. Hydrogen atoms are numbered as the atoms to which they are bonded.
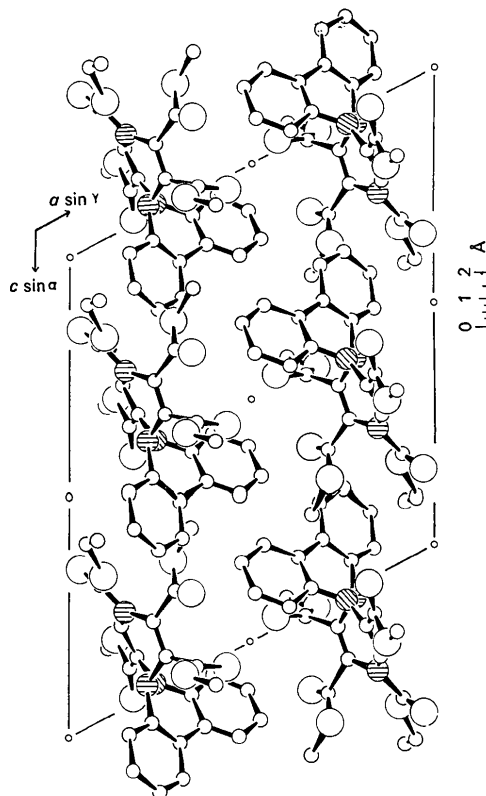

Fig. 2. The molecular packing viewed down b.

Table 4. *Least-squares planes given in the form* $lX' + mY' + nZ' = d$ *where* X', Y' *and* Z' *are coordinates in* Å *with respect to an orthogonalized axial system*

(a) Plane equations

Plane (1):	$0.588X' + 0.725Y' - 0.357Z' = 6.494$
Plane (2):	$-0.692X' - 0.722Y' - 0.033Z' = -7.583$
Plane (3):	$-0.520X' - 0.273Y' + 0.810Z' = -5.288$
Plane (4):	$-0.613X' + 0.498Y' - 0.614Z' = 2.310$
Plane (5):	$-0.953X' + 0.270Y' + 0.139Z' = -2.026$
Plane (6):	$-0.926X' - 0.304Y' + 0.224Z' = 1.534$
Plane (7):	$-0.850X' + 0.376Y' + 0.369Z' = -1.970$

(b) Deviations of atoms (Å) from planes (starred atoms not used to define planes)

Plane (1):	C(6a)	−0·011	C(7)	0·003	C(8)	0·001
	C(9)	0·003	C(10)	−0·012	C(10a)	0·016
	C(11a)*	−0·031	N(6)*	−0·027	N(15)*	0·858
	C(14*)a	0·457				
Plane (2):	C(11a)	−0·016	C(11)	0·004	C(12)	0·011
	C(13)	−0·014	C(14)	0·001	C(14a)	0·013
	C(10a)*	−0·131	N(15)*	0·058	N(6)*	0·865
	C(6a)*	0·370				
Plane (3):	O(1)	0·008	O(2)	0·013	C(16)	−0·016
	C(17)	−0·006				
Plane (4):	O(3)	−0·012	O(4)	−0·019	C(18)	0·022
	C(19)	0·009				
Plane (5):	O(5)	0·037	O(6)	0·056	C(20)	−0·067
	C(21)	−0·029	C(22)	0·003		
Plane (6):	O(7)	0·009	O(8)	0·013	C(23)	−0·016
	C(24)	−0·006				
Plane (7):	C(4)	−0·010	C(2)	−0·011	N(3)	0·031
	C(20)	−0·010				

the ranges which would be expected for similar bonding environments. There are no abnormally short intermolecular contacts.

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Structure Cristalline du Trimétaphosphate de Sodium-Potassium: Na₂KP₃O₉

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Sodium potassium trimetaphosphate, Na₂KP₃O₉, is triclinic with $a = 6.886$ (2), $b = 9.494$ (3), $c = 6.797$ (2) Å, $\alpha = 110.07$ (2), $\beta = 104.69$ (2), $\gamma = 86.68$ (2)°, and $Z = 2$. The space group is $P\bar{1}$. The crystal structure has been solved by a direct method using only 1600 reflexions from the 3706 collected. For these 1600 reflexions the R value is 0.018. Calculated for all the reflexions the R value is 0.032.

Introduction

La préparation chimique et les principales caractéristiques cristallographiques du trimétaphosphate de sodium-potassium, Na₂KP₃O₉, ont déjà été décrites par Cavero-Ghersi & Durif (1974). Ce sel cristallise avec une maille triclinique de paramètres $a = 6,886$ (2), $b = 9,494$ (3), $c = 6,797$ (2) Å, $\alpha = 110,07$ (2), $\beta = $ 104,69 (2), $\gamma = 86,68$ (2)°, renfermant deux unités formulaires. L'étude structurale montre que le groupe spatial est $P\bar{1}$.

Techniques expérimentales

Le cristal utilisé était un fragment de prisme de forme approximativement cubique. La dimension des arêtes