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Acta Cryst. (1991). C**47**, 2164–2167

Structure of Ammonium Hydrogen Bis(phosphoenolpyruvate)

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(*Received* 3 *December* 1990; *accepted* 2 *April* 1991)

Abstract. Ammonium hydrogen bis[2-(phosphonooxy)propenoate], $NH_4^+.C_3H_4O_6P^-.C_3H_5O_6P$, M_r = 353·12, monoclinic, $P2_1$, a = 7·618 (4), b = 22·321 (10), c = 8·311 (3) Å, β = 90·14 (4)°, V = 1413·2 (11) Å^3, Z = 4, D_m = 1·668, D_x = 1·660 (2) g cm^{-3}, Mo $K\alpha$, λ = 0·71069 Å, μ = 3·78 cm^{-1}, $F(000)$ = 728, T = 298 (2) K, final R = 0·0354 for 4625 observed reflections. The asymmetric unit contains two phosphoenolpyruvic acid molecules, two phosphoenolpyruvate monoanions and two ammonium cations. The P—O$_{\text{ester}}$ bond lengths are 1·590 (3) and 1·594 (3) Å in the acid molecules and 1·614 (2) and 1·609 (2) Å in the monoanions. All OH and NH groups are involved in intermolecular hydrogen bonds.

Introduction. This investigation was undertaken as part of a study of the structure of the phosphoenolpyruvate (PEP) moiety which may exist in four different forms: the tribasic acid, monoanion, dianion and trianion. All these forms have been characterized in the crystalline state (Weichsel & Lis, 1991). Recently, we prepared new PEP salts by saturating aqueous or alcoholic solutions of PEP acid with gaseous ammonia. This procedure usually produces monoammonium and triammonium salts (Weichsel & Lis, 1991). Here we report the structure of an acid ammonium PEP salt of formula NH_4^+.PEP$^-$.PEP which can also be obtained by this procedure.

Experimental. Almost parallelepipedal platy crystals of the title compound were obtained at ~277 K by slow concentration of an aqueous solution of PEP acid (Weichsel, Lis & Kuczek, 1989) and ammonium hydroxide in a molar ratio of 5:1. Slightly higher ammonium concentrations (*e.g.* a molar ratio of 3:1) result in orthorhombic crystals of the monoammonium PEP salt (Weichsel & Lis, 1991). The crystals were grown also by extremely slow diffusion of ammonia through the vapour phase into concentrated solution of PEP acid at ~277 K. The density of the crystals was measured by flotation in $CHCl_3$/$C_2H_4Br_2$; preliminary examination by oscillation and Weissenberg photographs. A fragment (0·35 × 0·25 × 0·5 mm) was cut from a larger crystal and covered with protective lacquer. A Syntex $P2_1$ diffractometer, Mo $K\alpha$ radiation and graphite monochromator were used for measurements of lattice parameters (15 reflections in the range $20 < 2\theta < 25°$) and intensities. An almost full Ewald sphere up to $2\theta = 56°$ was collected operating in the ω–2θ scan mode. After each group of 50 reflections two standards were measured, variation ± 8%. The intensities were corrected for Lorentz and polarization factors only. Of 7918 measured reflections 6589 had $I > 3\sigma(I)$. Merging these in point group 2 yielded 4625 reflections with h −10–10, k −29–29, l −10–10 (including 2187 Friedel pairs) which were used for the structure analysis; R_{merge} was 0·0287.

The structure was solved by the use of direct methods (Sheldrick, 1986). Refinement was by full-matrix least-squares methods (Sheldrick, 1976), minimizing the function $\sum w(|F_o| - |F_c|)^2$, with $w = 1/\sigma^2(F_o)$, where $\sigma(F_o)$ was based on counting statistics. The H atoms were found from difference Fou-

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Table 1. *Final atomic coordinates and temperature factors* (Å^2) *with e.s.d.'s in parentheses*

$$U_{eq} = (1/3)\Sigma_i \Sigma_j U_{ij} a_i^* a_j^* \mathbf{a}_i . \mathbf{a}_j .$$

	x	y	z	U_{iso}/U_{eq}
P(1)	0·69870 (12)	0·15976 (7)	0·19507 (12)	0·0284 (3)
P(2)	0·86151 (13)	0·97450 (7)	0·94275 (12)	0·0288 (3)
P(3)	0·88333 (11)	0·16920 (5)	0·65554 (12)	0·0239 (3)
P(4)	0·67743 (11)	0·964 (fixed)	0·48273 (11)	0·0237 (3)
O(11)	0·82995 (42)	0·21086 (16)	0·23195 (38)	0·0386 (7)
O(12)	0·68672 (35)	0·15585 (16)	0·01274 (33)	0·0399 (7)
O(13)	0·73974 (38)	0·10408 (14)	0·28228 (36)	0·0397 (7)
O(14)	0·50854 (34)	0·18109 (14)	0·24886 (32)	0·0371 (7)
O(15)	0·25169 (42)	0·14369 (16)	0·05102 (41)	0·0495 (7)
O(16)	0·19581 (40)	0·23708 (17)	−0·03252 (42)	0·0524 (8)
O(21)	0·72889 (39)	0·92346 (16)	0·90461 (37)	0·0402 (7)
O(22)	0·87474 (35)	0·97724 (16)	1·12644 (31)	0·0382 (7)
O(23)	0·82075 (38)	1·03027 (14)	0·85657 (36)	0·0397 (7)
O(24)	1·05180 (35)	0·95294 (13)	0·88825 (31)	0·0346 (6)
O(25)	1·30972 (41)	0·99151 (16)	1·08272 (40)	0·0490 (7)
O(26)	1·36510 (41)	0·89908 (17)	1·16804 (41)	0·0526 (7)
O(31)	0·91774 (35)	0·19343 (14)	0·82084 (34)	0·0357 (7)
O(32)	0·93620 (33)	0·10345 (14)	0·63761 (35)	0·0342 (6)
O(33)	0·96121 (32)	0·20427 (13)	0·51914 (33)	0·0287 (6)
O(34)	0·67327 (28)	0·16608 (13)	0·63146 (30)	0·0259 (6)
O(35)	0·33469 (34)	0·14637 (14)	0·58911 (40)	0·0417 (8)
O(36)	0·28075 (32)	0·24319 (14)	0·55212 (38)	0·0401 (8)
O(41)	0·64263 (35)	0·93967 (14)	0·31796 (34)	0·0356 (7)
O(42)	0·62738 (34)	1·03087 (14)	0·49988 (35)	0·0322 (6)
O(43)	0·59765 (35)	0·92972 (15)	0·61680 (36)	0·0302 (6)
O(44)	0·88697 (28)	0·96601 (13)	0·50723 (31)	0·0272 (6)
O(45)	1·22659 (34)	0·98478 (14)	0·54287 (40)	0·0451 (8)
O(46)	1·27923 (32)	0·88821 (14)	0·58632 (37)	0·0390 (7)
N(1)	0·44187 (44)	0·05765 (18)	0·80627 (43)	0·0456 (8)
N(2)	0·11777 (43)	0·07689 (18)	0·33098 (41)	0·0431 (8)
C(11)	0·28014 (53)	0·19695 (22)	0·05433 (52)	0·0355 (8)
C(12)	0·41799 (54)	0·22393 (21)	0·15882 (50)	0·0333 (8)
C(13)	0·45053 (71)	0·28111 (24)	0·17201 (72)	0·0640 (10)
C(21)	1·27987 (53)	0·93838 (21)	1·08128 (51)	0·0343 (8)
C(22)	1·14280 (53)	0·91055 (21)	0·97799 (51)	0·0340 (8)
C(23)	1·11576 (69)	0·85238 (24)	0·96386 (65)	0·0570 (9)
C(31)	0·38327 (46)	0·19778 (19)	0·58757 (51)	0·0288 (8)
C(32)	0·56680 (45)	0·21645 (18)	0·62517 (47)	0·0254 (7)
C(33)	0·61356 (54)	0·27178 (21)	0·65290 (59)	0·0437 (9)
C(41)	1·17716 (44)	0·93367 (20)	0·55007 (48)	0·0280 (8)
C(42)	0·99091 (50)	0·91613 (19)	0·51863 (51)	0·0293 (8)
C(43)	0·94160 (57)	0·86044 (23)	0·50400 (77)	0·0643 (11)

Table 2. *Principal bond lengths* (Å), *bond angles* (°) *and torsion angles* (°) *for phosphoenolpyruvic acid molecules* (1) *and* (2) *and phosphoenolpyruvate monoanions* (3) *and* (4)

	(1) $n = 1$	(2) $n = 2$	(3) $n = 3$	(4) $n = 4$
P(*n*)—O(*n*1)	1·547 (4)	1·555 (4)	1·499 (3)	1·496 (3)
P(*n*)—O(*n*2)	1·520 (3)	1·531 (3)	1·529 (3)	1·547 (3)
P(*n*)—O(*n*3)	1·472 (3)	1·469 (3)	1·501 (3)	1·483 (3)
P(*n*)—O(*n*4)	1·590 (3)	1·594 (3)	1·614 (2)	1·609 (2)
O(*n*4)—C(*n*2)	1·396 (5)	1·389 (5)	1·387 (5)	1·369 (5)
C(*n*1)—C(*n*2)	1·488 (6)	1·486 (6)	1·491 (5)	1·494 (5)
C(*n*2)—C(*n*3)	1·305 (7)	1·320 (7)	1·306 (6)	1·304 (7)
C(*n*1)—O(*n*5)	1·209 (6)	1·208 (6)	1·206 (5)	1·203 (5)
C(*n*1)—O(*n*6)	1·317 (6)	1·307 (6)	1·313 (5)	1·313 (5)
O(*n*1)—P(*n*)—O(*n*2)	106·1 (2)	105·9 (2)	113·0 (2)	113·0 (2)
O(*n*1)—P(*n*)—O(*n*3)	112·9 (2)	112·6 (2)	115·8 (2)	115·4 (2)
O(*n*1)—P(*n*)—O(*n*4)	108·2 (2)	108·2 (2)	107·5 (2)	107·4 (2)
O(*n*2)—P(*n*)—O(*n*3)	117·0 (2)	117·7 (2)	108·8 (2)	109·1 (2)
O(*n*2)—P(*n*)—O(*n*4)	104·2 (2)	103·7 (2)	102·0 (2)	101·9 (2)
O(*n*3)—P(*n*)—O(*n*4)	107·9 (2)	108·0 (2)	108·8 (2)	109·1 (2)
P(*n*)—O(*n*4)—C(*n*2)	120·3 (3)	120·4 (3)	123·3 (3)	124·7 (3)
O(*n*4)—C(*n*2)—C(*n*1)	112·5 (4)	112·0 (4)	109·2 (4)	110·3 (4)
O(*n*4)—C(*n*2)—C(*n*3)	122·1 (5)	123·0 (5)	126·9 (4)	127·0 (5)
C(*n*1)—C(*n*2)—C(*n*3)	125·3 (5)	124·9 (5)	123·8 (4)	122·6 (5)
C(*n*2)—C(*n*1)—O(*n*5)	122·5 (5)	123·2 (5)	123·5 (4)	122·5 (4)
C(*n*2)—C(*n*1)—O(*n*6)	112·8 (4)	112·7 (4)	112·8 (4)	113·5 (4)
O(*n*5)—C(*n*1)—O(*n*6)	124·7 (5)	124·1 (5)	123·7 (4)	124·0 (3)
P(*n*)—O(*n*2)—H(*n*2)	125·5 (16)	112·8 (12)	117·5 (13)	114·9 (11)
C(*n*1)—O(*n*6)—H(*n*6)	110·9 (15)	112·7 (15)	109·0 (16)	105·6 (15)
P(*n*)—O(*n*1)—H(*n*1)	110·8 (15)	121·1 (18)		
C(*n*3)—C(*n*2)—C(*n*1)—O(*n*5)	−177·4 (6)	174·4 (10)	−166·9 (7)	170·1 (10)
C(*n*3)—C(*n*2)—C(*n*1)—O(*n*6)	3·0 (7)	−4·8 (11)	13·4 (6)	−10·1 (9)
C(*n*3)—C(*n*2)—O(*n*4)—P(*n*)	−80·2 (6)	82·4 (9)	−7·3 (7)	3·6 (9)
C(*n*1)—C(*n*2)—O(*n*4)—P(*n*)	101·9 (5)	−101·4 (9)	175·0 (5)	−176·8 (6)
C(*n*2)—O(*n*4)—P(*n*)—O(*n*1)	73·1 (5)	−74·1 (10)	66·2 (6)	−67·9 (9)
C(*n*2)—O(*n*4)—P(*n*)—O(*n*2)	−39·4 (5)	38·0 (9)	−174·8 (6)	173·1 (6)
C(*n*2)—O(*n*4)—P(*n*)—O(*n*3)	−164·5 (5)	163·7 (9)	−59·9 (6)	57·9 (9)
O(*n*5)—C(*n*1)—C(*n*2)—O(*n*4)	0·5 (6)	−1·6 (10)	10·8 (6)	−9·4 (9)
O(*n*6)—C(*n*1)—C(*n*2)—O(*n*4)	−179·1 (6)	179·2 (9)	−168·9 (5)	170·3 (8)
O(*n*4)—P(*n*)—O(*n*2)—H(*n*2)	141 (3)	−114 (2)	−68 (2)	71 (1)
C(*n*2)—C(*n*1)—O(*n*6)—H(*n*6)	−176 (2)	166 (2)	−154 (2)	161 (2)
O(*n*4)—P(*n*)—O(*n*1)—H(*n*1)	86 (2)	−96 (2)		

rier syntheses. The positional parameters of H atoms were refined with constraints d(C—H) = 1·08, d(N—H) = 1·01 and d(O—H) = 0·97 Å. The refinement yielded final R and wR values of 0·0354 and 0·0375. (The refinement of the inverted structure gave R = 0·0359 and wR = 0·0379.) In the final cycle of refinement no parameter shifted more than 0·4 of its standard deviation. The final difference Fourier synthesis showed max. and min. electron densities of 0·70 and −0·45 e Å^{-3}, respectively. Neutral atomic scattering factors were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV). Anomalous dispersion was included for P, O, N and C atoms. The atomic positional parameters and equivalent temperature factors are listed in Table 1.*

Discussion. The crystals contain crystallographically independent pairs of (i) phosphoenolpyruvic acid molecules, (ii) phosphoenolpyruvate monoanions and (iii) ammonium cations; the members of each pair are related by a pseudo-inversion centre at $a \simeq 0·780$, $b \simeq 0·567$, $z \simeq 0·569$. The molecular structures and the numbering schemes for the acid molecules [(1) and (2)] are illustrated in Fig. 1 and those of the monoanions [(3) and (4)] are shown in Fig. 2. Principal interatomic distances and angles are given in Table 2.

The acid molecules [(1) and (2)] have very similar structures. The enolpyruvate systems are not exactly planar. The angles between the carboxyl planes and those formed by C(12), C(13), O(14) in (1), and C(22), C(23), O(24) in (2) are 2·2 (8) and 5·0 (8)°, respectively. The phosphate groups have similar orientations relative to the enolpyruvate systems. The torsion angles P(1)—O(14)—C(12)—C(11) and P(2)—O(24)—C(22)—C(21) are 101·9 (5) and −101·4 (9)° in (1) and (2), respectively. The phosphate ester bonds in (1) [1·590 (3)] and (2) [1·594 (3) Å] are a little longer than those found earlier in crystals of phosphoenolpyruvic acid [1·578 (3) and 1·583 (3) Å; Weichsel, Lis & Kuczek, 1989]. The unprotonated O atom in the dihydrogen

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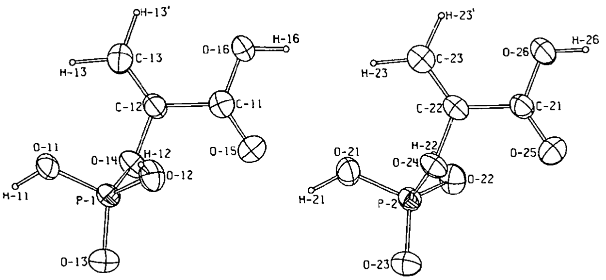
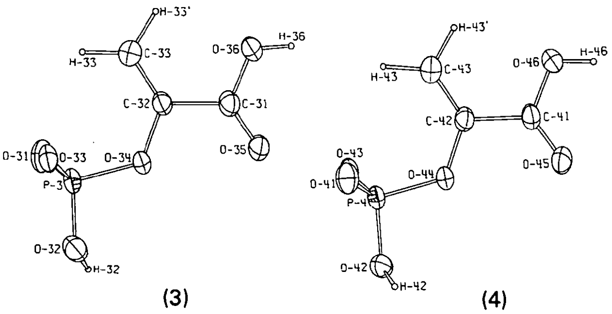
* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54128 (31 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

phosphate groups is *trans* to the ester C atom. In contrast, the corresponding O atom in crystalline phosphoenolpyruvic acid is *gauche* to the ester C atom (Weichsel, Lis & Kuczek, 1989). The enolic C—O bond lengths in (1) and (2) [1·396 (5) and 1·389 (5) Å, respectively] are comparable with those observed in the triclinic form of PEP acid.



(1) (2)

Fig. 1. The molecular structure of two crystallographically independent molecules [(1) and (2)] of phosphoenolpyruvic acid.



(3) (4)

Fig. 2. The molecular structure of two crystallographically independent molecules [(3) and (4)] of phosphoenolpyruvate monoanions.

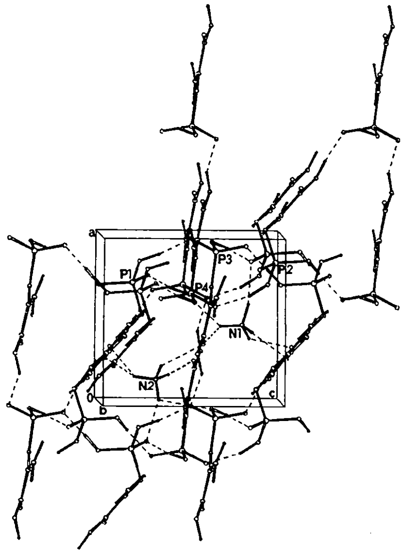


Fig. 3. The packing in the crystal of ammonium hydrogen bis(phosphoenolpyruvate).

Table 3. *Hydrogen-bond distances* (Å) *and angles* (°)

X—H···O	*X*···O	H···O	*X*—H···O
O(11)—H(11)···O(33)	2·590 (4)	1·62 (2)	174 (3)
O(12)—H(12)···O(31^{i})	2·522 (4)	1·56 (3)	174 (3)
O(21)—H(21)···O(43)	2·594 (4)	1·65 (2)	164 (3)
O(22)—H(22)···O(41^{ii})	2·525 (4)	1·68 (3)	143 (2)
O(32)—H(32)···O(23^{iii})	2·600 (4)	1·63 (3)	175 (2)
O(42)—H(42)···O(13^{iv})	2·585 (4)	1·62 (2)	171 (2)
O(16)—H(16)···O(31^{v})	2·629 (4)	1·67 (3)	171 (3)
O(26)—H(26)···O(41^{vi})	2·614 (4)	1·65 (3)	173 (3)
O(36)—H(36)···O(33^{vii})	2·598 (4)	1·71 (2)	150 (3)
O(46)—H(46)···O(43^{viii})	2·608 (4)	1·69 (2)	156 (3)
N(1)—H(1)···O(23^{iii})	2·979 (4)	1·98 (2)	168 (2)
N(1)—H(2)···O(25^{ix})	2·913 (5)	2·15 (2)	131 (2)
N(1)—H(3)···O(35)	2·800 (5)	2·21 (2)	116 (2)
N(1)—H(3)···O(42^{iii})	2·976 (5)	2·23 (2)	129 (2)
N(1)—H(4)···O(15^{ii})	3·153 (5)	2·25 (2)	148 (2)
N(2)—H(5)···O(13^{vii})	2·970 (4)	2·03 (2)	155 (2)
N(2)—H(5)···O(32^{vii})	2·962 (4)	2·46 (2)	110 (2)
N(2)—H(6)···O(15)	2·948 (5)	2·38 (2)	114 (2)
N(2)—H(6)···O(25^{x})	3·169 (5)	2·39 (2)	133 (2)
N(2)—H(7)···O(45^{ix})	2·830 (5)	1·83 (2)	170 (2)
N(2)—H(8)···O(35)	3·118 (5)	2·31 (2)	136 (2)

Symmetry codes: (i) $x, y, z-1$; (ii) $x, y, z+1$; (iii) $x, y-1, z$; (iv) $x, y+1, z$; (v) $x-1, y, z-1$; (vi) $x+1, y, z+1$; (vii) $x-1$, y, z; (viii) $x+1, y, z$; (ix) $x-1, y-1, z$; (x) $x-1, y-1, z-1$.

The phosphoenolpyruvate monoanions (3) and (4) also have similar conformations. The enolpyruvate moiety is non-planar in both anions; the angle between the carboxyl group and the plane through O(34), C(32), C(33) [O(44), C(42), C(43)] is 11·9 (8) [9·7 (8)°]. The orientation of the phosphate group is *trans* with respect to the carboxylic group and similar in both anions [the P(3)—O(34)—C(32)—C(31) and P(4)—O(44)—C(42)—C(41) torsion angles are 175·0 (5) and −176·8 (6)°, respectively]. The P—O_{ester} bond lengths [1·614 (2) and 1·609 (2) Å] are comparable to those found in the cyclohexylammonium [1·610 (2) and 1·604 (2) Å; Weichsel & Lis, 1989], potassium [1·622 (2) Å; Lis, 1987], sodium [1·618 (3) and 1·606 (3) Å; Lis, 1991] and ammonium [1·623 (2) Å; Weichsel & Lis, 1991] salts. There are no noticeable differences among corresponding C—C and C—O bond lengths in Table 2.

The carboxylic hydroxyl groups in all four PEP moieties are *trans* to O_{ester} atoms. The same situation has been observed in other PEP structures which were crystallized from aqueous solutions.

The crystal packing and hydrogen-bonding schemes are shown in Fig. 3. The hydrogen-bond distances and angles are listed in Table 3. All OH groups participate in relatively strong hydrogen bonds forming a three-dimensional network. The carboxylic groups (as donors) are utilized in hydrogen bonds with phosphate O atoms, although carboxylic groups usually prefer to form hydrogen bonds with one another.

Each ammonium cation is located in a cavity defined by nine nearest non-H-atom neighbours at distances of 2·80 (1)–3·47 (1) Å. Some of these contacts may be interpreted as partially bifurcated hydrogen bonds (Table 3).

This work was financially supported in part by the Polish Ministry of Education, project R.P.II.10.

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Structures of 1,3-Di(9-carbazolyl)propane and 1,5-Di(9-carbazolyl)pentane. Polyvinylcarbazole Model Compounds

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(*Received* 8 *November* 1990; *accepted* 3 *April* 1991)

Abstract. 9,9′-Propylenedicarbazole, $C_{27}H_{22}N_2$, $M_r = 374{\cdot}49$, monoclinic, $P2_1/c$, $a = 9{\cdot}303\,(3)$, $b = 17{\cdot}697\,(3)$, $c = 12{\cdot}477\,(3)$ Å, $\beta = 98{\cdot}30\,(3)°$, $V = 2033\,(2)$ Å^3, $Z = 4$, $D_m = 1{\cdot}221$, $D_x = 1{\cdot}224$ g cm^{-3}, $\lambda(\text{Mo } K\alpha) = 0{\cdot}71073$ Å, $\mu = 0{\cdot}7$ cm^{-1}, $F(000) = 792$, $T = 302$ K, $R = 0{\cdot}075$, $wR = 0{\cdot}095$ for 1650 independent reflections with $I \geq 3\sigma(I)$ and 262 variables. 9,9′-Pentylenedicarbazole, $C_{29}H_{26}N_2$, $M_r = 402{\cdot}54$, monoclinic, $P2_1/c$, $a = 9{\cdot}158\,(3)$, $b = 28{\cdot}204\,(8)$, $c = 8{\cdot}717\,(3)$ Å, $\beta = 100{\cdot}97\,(3)°$, $V = 2210\,(2)$ Å^3, $Z = 4$, $D_m = 1{\cdot}21$, $D_x = 1{\cdot}210$ g cm^{-3}, $\lambda(\text{Mo } K\alpha) = 0{\cdot}71073$ Å, $\mu = 0{\cdot}7$ cm^{-1}, $F(000) = 856$, $T = 302$ K, $R = 0{\cdot}054$, $wR = 0{\cdot}052$ for 2115 independent reflections with $I \geq 2\sigma(I)$ and 280 variables. The stereochemistry of the carbazole substituents is *gauche*, *anti* for 1,3-di(9-carbazolyl)propane and *gauche*, *gauche* for 1,5-di(9-carbazolyl)pentane, two polyvinylcarbazole model compounds. Although no intramolecular interactions between carbazole rings are evident, one carbazole of each molecule associates in a shifted parallel planar orientation with that of another molecule.

Introduction. Poly(9-vinylcarbazole) (PVC) is one of the most useful conducting organic polymers (Masuhara, Ohwada, Mataga, Itaya, Okamoto & Kusabayashi, 1978). Overlap between the carbazole groups is believed to be responsible for the electronic transport properties (Turner & Pai, 1979). Structural studies of PVC have been conducted in order to establish the role of polymer structure in photoconductivity (Tsuchihashi, Hatano & Sohna, 1976; Limburg & Williams, 1973).

1,3-Di(9-carbazolyl)propane (I) and related compounds which contain two neighboring carbazole substituents have been employed to model the interactions of the pendant carbazole groups in PVC (Masuhara *et al.*, 1978; Itaya, Okamoto & Kusabayashi, 1979; Masuhara, Tamai, Mataga, De Schryver & Vandendriessche, 1983; Klöpffer, 1969). We found that spatial relationships in such model systems affect the electronic interactions between the carbazole groups (Masnovi, Krafcik, Baker & Towns, 1990). Therefore, we have undertaken single-crystal X-ray structure determinations of (I) and of a higher analog, 1,5-di(9-carbazolyl)pentane (II), in order to establish the preferred relative orientation of the carbazole groups in these compounds. The structures and properties of molecular complexes of PVC model compounds with electron-accepting dopants are also of interest (Masnovi, Baker, Towns & Chen, 1991) and will be reported subsequently.

Experimental. The title compounds were prepared by reaction of the potassium salt of carbazole and 1,3-dibromopropane and 1,5-dibromopentane in dimethyl sulfoxide (Johnson, 1974). The reaction mixtures were poured into water and the crude products collected by filtration. Purification was accomplished by chromatography on silica gel, eluting fractionally using petroleum ether and dichloromethane.

Colorless crystals of (I) and (II) were attached to the end of glass capillaries using quick-drying epoxy cement at room temperature. An Enraf–Nonius CAD-4 X-ray diffractometer equipped with a graphite monochromator (Mo $K\alpha$ radiation) was used for data collection. Cell parameters were determined

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0108-2701/91/102167-04$03.00