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209. The Crystal and Molecular Structure of 3,5,7-Triphenyl-4*H*-1,2-Diazepine Picrate

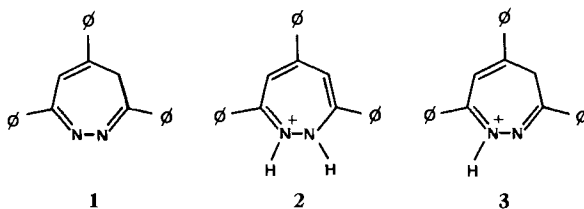
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Summary. The crystal structure of 3,5,7-triphenyl-4*H*-1,2-diazepine (monoclinic, $a = 8.132$, $b = 13.180$, $c = 24.204$ Å, $\beta = 104.15^\circ$, space group $P2_1/c$, $Z = 4$) has been determined by X-ray analysis. The 1,2-diazepine ring adopts a boat conformation with a methylene group at the prow. The phenoxide group of the picrate moiety is bonded to one of the diazepine nitrogen atoms through an hydrogen bond.

The N-unsubstituted configuration **1** was assigned to 3,5,7-triphenyl-1,2-diazepine which was formed by the condensation of 2,4,6-triarylthiapyrylium salts with hydrazine [1]. The protonation of **1** was formerly thought to produce the 'planar' cation **2**. However a recent NMR. investigation [2] has demonstrated the presence of a methylene group in the protonated form of **1** thus suggesting the more credible 'non-planar' structure **3**.



It was therefore interesting to study the crystal structure of an available crystalline derivative of the protonated form of **1** (picrate salt) to ascertain the position of the extra proton and to find out how the formal positive charge would influence the conformation of the diazepine ring.

Several valid indications about the conformation of the neutral ring are offered by recent crystal structure determinations [3] of substituted 1,2-diazepines.

Experimental Part. – The X-ray data were collected by Dr. *E. Klingsberg* at the crystallographic centre of the *Lederle Laboratories*¹⁾, and kindly submitted to us for the present structure analysis.

¹⁾ A division of the American Cyanamid Company, Pearl River, New York, USA.

The lattice of 3,5,7-triphenyl-4*H*-1,2-diazepine picrate ($C_{29}N_5O_7H_{21}$) is monoclinic with: $a = 8.132 \text{ \AA}$, $b = 13.180 \text{ \AA}$, $c = 24.204 \text{ \AA}$, $\beta = 104.15^\circ$, $U = 2516 \text{ \AA}^3$, $Z = 4$, space group $P2_1/c$.

Intensity data were measured on a *General Electric* XRD-6 diffractometer by the stationary crystal-stationary counter method with balanced filter and $CuK\alpha$ radiation, at a fixed count time of 10 seconds. A total of 3406 independent reflexions were measured to a 2θ maximum of 110° ; 1339 reflexions were in the observable range.

Structure Analysis and Refinement. - Some essential features about the structure were obtained from a three dimensional E-synthesis calculated on the basis of 224 probable phases arrived at by direct methods. Most of the atoms of the picrate ion appeared well resolved; however, the shape of the diazepine molecule was very much obscured by a large number of spurious peaks. A three-dimensional array of the E-synthesis plotted on stacked plastic sheets allowed the assignment of more peaks on the basis of simple steric requirements associated with a postulated orientation of the diazepine ring with its nitrogen atoms near the picrate ion. A preliminary set of 23 'atoms' was finally chosen as a phasing model. After a few F_o - and difference-synthesis more atoms were located. The process was repeated till a chemically sound structure with 41 atoms was obtained; a R factor calculated at this stage was 0.28. Then a first cycle of full-matrix least-squares analysis gave an R value = 0.176. Several more cycles of anisotropic refinement (hydrogen atoms were included in the

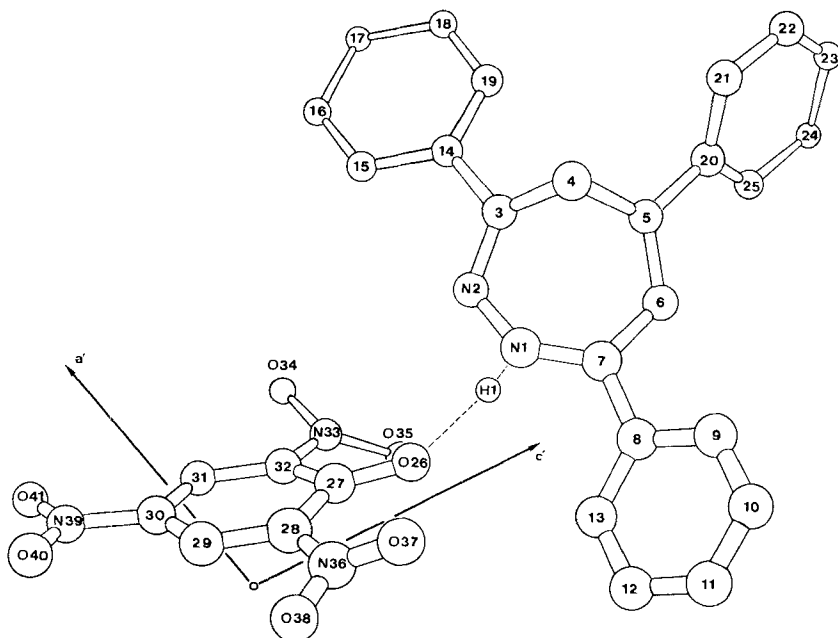


Fig. 1. *Projection drawing of the molecular packing of 3,5,7-triphenyl-4H-1,2-diazepine with picric acid in the crystal lattice*

The atomic positions are projected onto the best plane through N(2), C(3), C(5), C(6). This best plane lies almost parallel to the crystallographic (001)-plane and therefore the projected a' and c' directions (as shown with the origin) are almost coincident with the true crystallographic a and c axis. The hydrogen bond, with the intervening hydrogen atom H1, is pictured by the dotted line between O(26) and N(1).

latter stages) yielded a final *R* value of 0.066, based on 1334 included reflexions. The estimated standard deviations in the atomic positions obtained by inversion of the normal-equations matrix are in the range 0.005–0.010 Å, the larger values being associated with the atoms of the phenyl rings.

Results. – A projective view of the mutual orientation of both molecular components of 3,5,7-triphenyl-1,2-diazepine picrate in the crystal, as well as their conformation, is shown in Fig. 1, together with the adopted atom-numbering system.

Table 1. *Final positional parameters* (standard deviation $\times 10^4$)

Atom	x	y	z
N(1)	.1843 (8)	.1605 (5)	.2241 (2)
N(2)	.3323 (8)	.1058 (5)	.2344 (2)
C(3)	.4471 (11)	.1254 (6)	.2805 (3)
C(4)	.4361 (10)	.2116 (6)	.3198 (3)
C(5)	.3051 (9)	.1773 (6)	.3501 (3)
C(6)	.1414 (10)	.1643 (6)	.3207 (3)
C(7)	.0885 (10)	.1803 (6)	.2598 (3)
C(8)	–.0835 (9)	.2192 (6)	.2358 (3)
C(9)	–.1466 (11)	.2934 (6)	.2665 (3)
C(10)	–.3056 (12)	.3350 (7)	.2454 (3)
C(11)	–.4066 (11)	.2996 (8)	.1936 (4)
C(12)	–.3460 (11)	.2237 (7)	.1632 (3)
C(13)	–.1854 (9)	.1840 (6)	.1838 (3)
C(14)	.6024 (11)	.0573 (6)	.2934 (3)
C(15)	.6502 (11)	.0069 (7)	.2497 (4)
C(16)	.7935 (10)	–.0551 (6)	.2635 (3)
C(17)	.8831 (10)	–.0698 (6)	.3204 (3)
C(18)	.8318 (9)	–.0184 (6)	.3632 (4)
C(19)	.6919 (11)	.0449 (6)	.3500 (3)
C(20)	.3547 (10)	.1572 (6)	.4132 (3)
C(21)	.4817 (10)	.2151 (7)	.4489 (3)
C(22)	.5165 (12)	.2004 (7)	.5076 (3)
C(23)	.4302 (12)	.1275 (7)	.5303 (3)
C(24)	.3071 (12)	.0673 (8)	.4953 (3)
C(25)	.2714 (10)	.0826 (6)	.4366 (3)
O(26)	.0783 (7)	.1868 (4)	.1089 (2)
C(27)	.1074 (11)	.1493 (7)	.0650 (3)
C(28)	.0652 (11)	.1972 (7)	.0100 (3)
C(29)	.1199 (11)	.1604 (6)	–.0368 (3)
C(30)	.2130 (10)	.0719 (6)	–.0316 (3)
C(31)	.2438 (10)	.0154 (6)	.0179 (3)
C(32)	.1878 (10)	.0486 (6)	.0631 (3)
N(33)	.2075 (9)	–.0175 (5)	.1125 (3)
O(34)	.3247 (9)	–.0796 (5)	.1200 (3)
O(35)	.1016 (7)	–.0131 (4)	.1412 (2)
N(36)	–.0363 (9)	.2897 (5)	–.0002 (3)
O(37)	–.0567 (9)	.3379 (6)	.0398 (3)
O(38)	–.0999 (8)	.3141 (5)	–.0491 (2)
N(39)	.2788 (9)	.0369 (6)	–.0785 (3)
O(40)	.2535 (9)	.0906 (5)	–.1215 (2)
O(41)	.3553 (9)	–.0435 (5)	–.0729 (3)

The atomic positions are listed in Table 1 (with their estimated standard deviations), and the thermal parameters of the anisotropic temperature factors in the form $\exp - \{b_{11}h^2 + b_{22}k^2 + b_{33}l^2 + b_{12}hk + b_{13}hl + b_{23}kl\}$ are listed in Table 2. Their average standard deviations ($\sigma \cdot 10^4$) are: b_{11} (18), b_{22} (6), b_{33} (2), b_{12} (16), b_{13} (9), b_{23} (5).

The positions of the hydrogen atoms were calculated (with the exception of H(1)) on the basis of the final distribution of the non-hydrogen atoms and were not refined. The co-ordinates shown for the hydrogen atom H(1) involved in the hydrogen bond were obtained from a three-dimensional difference-synthesis calculated at a latter stage of the refinement.

Table 2. *Final anisotropic thermal parameters* ($\times 10^4$)

Atom	b_{11}	b_{22}	b_{33}	b_{12}	b_{13}	b_{23}
N(1)	148	47	15	25	17	8
N(2)	170	50	13	21	12	0
C(3)	133	42	16	- 1	20	1
C(4)	161	37	17	11	35	- 9
C(5)	158	32	15	3	30	- 11
C(6)	163	49	12	- 1	14	0
C(7)	162	33	18	- 50	32	- 7
C(8)	140	45	15	3	20	4
C(9)	171	45	17	24	26	1
C(10)	183	53	19	32	33	12
C(11)	208	75	17	55	11	18
C(12)	160	74	16	22	23	0
C(13)	153	48	15	- 23	24	- 8
C(14)	173	33	19	- 28	32	- 3
C(15)	172	42	17	- 1	18	- 8
C(16)	177	37	21	1	34	- 6
C(17)	164	48	22	- 23	7	- 7
C(18)	109	49	24	18	0	3
C(19)	206	41	17	- 32	3	- 7
C(20)	146	47	14	26	14	- 10
C(21)	192	53	16	33	20	- 9
C(22)	212	91	14	- 4	1	- 15
C(23)	264	91	16	2	44	5
C(24)	222	63	18	10	30	- 1
C(25)	163	57	13	- 26	17	- 5
O(26)	279	59	15	79	40	13
C(27)	177	58	14	- 13	27	12
C(28)	161	43	16	- 20	19	11
C(29)	184	46	19	- 76	26	3
C(30)	171	48	16	- 51	18	- 6
C(31)	162	46	16	- 9	10	- 8
C(32)	162	42	14	- 13	- 11	10
N(33)	218	35	19	3	- 15	0
O(34)	339	80	28	142	45	34
O(35)	273	58	17	- 33	34	5
N(36)	200	55	20	1	6	7
O(37)	361	63	27	112	5	- 4
O(38)	317	97	25	168	14	42
N(39)	191	77	21	- 53	31	- 5
O(40)	325	97	18	- 59	66	- 10
O(41)	316	93	29	44	60	- 27

Table 3. *Calculated coordinates^{a)} of hydrogen atoms*
 Assuming C–H distance 1.09 Å and local C_{2v}-symmetry of methylene groups

Atom	x	y	z
H(1)	.1400	.1770	.1861
H _A (4)	.3958	.2810	.2959
H _B (4)	.5579	.2241	.3500
H(6)	.0475	.1412	.3436
H(9)	–.0692	.3187	.3076
H(10)	–.3518	.3948	.2688
H(11)	–.5329	.3312	.1770
H(12)	–.4256	.1960	.1231
H(13)	–.1379	.1256	.1599
H(15)	.5780	.0154	.2057
H(16)	.8367	–.0929	.2296
H(17)	.9915	–.1211	.3308
H(18)	.9017	–.0281	.4075
H(19)	.6518	.0852	.3838
H(21)	.5526	.2711	.4310
H(22)	.6125	.2468	.5358
H(23)	.4591	.1170	.5763
H(24)	.2400	.0094	.5135
H(25)	.1766	.0353	.4086
H(29)	.0890	.2015	–.0769
H(31)	.3133	–.0557	.0207

^{a)} The coordinates of H(1) are *observed* values taken from a three dimensional difference synthesis.

Table 4. *Molecular parameters derived from coordinates of Table 1*
 (For atom-numbering system, see Fig. 1)

<i>Bond distances (Å)</i>		<i>Bond distances (Å)</i>	
N(1)–N(2)	1.372	C(20)–C(21)	1.400
N(1)–C(7)	1.324	C(20)–C(25)	1.392
N(2)–C(3)	1.293	C(21)–C(22)	1.381
C(3)–C(4)	1.499	C(23)–C(24)	1.391
C(3)–C(14)	1.519	C(24)–C(25)	1.394
C(4)–C(5)	1.503	O(26)–C(27)	1.245
C(5)–C(6)	1.359	C(27)–C(28)	1.436
C(5)–C(20)	1.503	C(27)–C(32)	1.485
C(6)–C(7)	1.447	C(28)–C(29)	1.402
C(7)–C(8)	1.471	C(28)–N(36)	1.458
C(8)–C(9)	1.399	C(29)–C(30)	1.379
C(8)–C(13)	1.404	C(30)–C(31)	1.384
C(9)–C(10)	1.383	C(30)–N(39)	1.442
C(10)–C(11)	1.398	C(31)–C(32)	1.356
C(11)–C(12)	1.402	C(32)–N(33)	1.458
C(12)–C(13)	1.382	N(33)–O(34)	1.234
C(14)–C(15)	1.381	N(33)–O(35)	1.233
C(14)–C(19)	1.395	N(36)–O(37)	1.203
C(15)–C(16)	1.397	N(36)–O(38)	1.216
C(16)–C(17)	1.405	N(39)–O(40)	1.236
C(17)–C(18)	1.384	N(39)–O(41)	1.222
C(18)–C(19)	1.385		

Table 4. (continued)

<i>Bond angles</i>		<i>Bond angles</i>	
C(7)–N(1)–N(2)	127.87°	C(21)–C(20)–C(25)	119.49°
N(1)–N(2)–C(3)	117.95°	C(20)–C(21)–C(22)	119.31°
N(2)–C(3)–C(4)	123.82°	C(21)–C(22)–C(23)	120.46°
N(2)–C(3)–C(14)	116.62°	C(22)–C(23)–C(24)	121.02°
C(4)–C(3)–C(14)	119.55°	C(23)–C(24)–C(25)	118.47°
C(3)–C(4)–C(5)	103.99°	C(24)–C(25)–C(20)	121.20°
C(4)–C(5)–C(6)	120.20°		
C(4)–C(5)–C(20)	120.13°	O(26)–C(27)–C(28)	124.30°
C(6)–C(5)–C(20)	119.67°	O(26)–C(27)–C(32)	123.90°
C(5)–C(6)–C(7)	121.63°	C(28)–C(27)–C(32)	111.80°
C(6)–C(7)–C(1)	124.18°	C(27)–C(28)–C(29)	123.06°
C(6)–C(7)–C(8)	118.07°	C(27)–C(28)–N(36)	121.46°
N(1)–C(7)–C(8)	117.75°	C(29)–C(28)–N(36)	115.48°
C(7)–C(8)–C(9)	118.06°	C(28)–C(29)–C(30)	119.80°
C(7)–C(8)–C(13)	122.31°	C(29)–C(30)–C(31)	120.72°
C(9)–C(8)–C(13)	119.64°	C(29)–C(30)–N(39)	120.15°
C(8)–C(9)–C(10)	120.76°	C(31)–C(30)–N(39)	119.14°
C(9)–C(10)–C(11)	119.37°	C(30)–C(31)–C(32)	120.09°
C(10)–C(11)–C(12)	120.21°	C(31)–C(32)–C(27)	123.44°
C(11)–C(12)–C(13)	120.20°	C(31)–C(32)–N(33)	118.43°
C(12)–C(13)–C(8)	119.79°	C(27)–C(32)–N(33)	118.13°
C(3)–C(14)–C(15)	120.17°	C(32)–N(33)–O(34)	116.34°
C(3)–C(14)–C(19)	118.62°	C(32)–N(33)–O(35)	118.71°
C(15)–C(14)–C(19)	121.19°	O(34)–N(33)–O(35)	124.76°
C(14)–C(15)–C(16)	118.27°	C(28)–N(36)–O(37)	119.26°
C(15)–C(16)–C(17)	121.15°	C(28)–N(36)–O(38)	118.35°
C(16)–C(17)–C(18)	119.19°	O(37)–N(36)–O(38)	122.37°
C(17)–C(18)–C(19)	120.15°	C(30)–N(39)–O(40)	117.57°
C(18)–C(19)–C(14)	120.00°	C(30)–N(39)–O(41)	118.08°
C(5)–C(20)–C(21)	120.36°	O(40)–N(39)–O(41)	124.35°
C(5)–C(20)–C(25)	120.13°		
<i>Torsion angles</i>			
C(7)–N(1)–N(2)–C(3)	– 47.20°		
N(1)–N(2)–C(3)–C(4)	– 6.88°		
N(2)–C(3)–C(4)–C(5)	73.95°		
C(3)–C(4)–C(5)–C(6)	– 65.55°		
C(4)–C(5)–C(6)–C(7)	1.54°		
C(5)–C(6)–C(7)–N(1)	33.83°		
C(6)–C(7)–N(1)–N(2)	13.09°		

Parameters for the intramolecular geometry are given in Table 4 and are not corrected for thermal vibrations. The average standard deviation was estimated to be 0.71% in the bond distances and 0.65% in the bond angles. An independent estimation for the standard deviations is provided by the dispersion of the values of the bond lengths and angles for the three phenyl rings, whose average dimensions are accurately known [4]. Our observed values, averaged over 18 parameters, are 1.392 Å for the bond distances ($\sigma = 0.008$) and 119.99° for the bond angles ($\sigma = 0.89^\circ$). The

phenyl rings are planar within the error limits and the mean deviation of the ring atoms from the best plane, averaged over the three rings, is 0.007 Å.

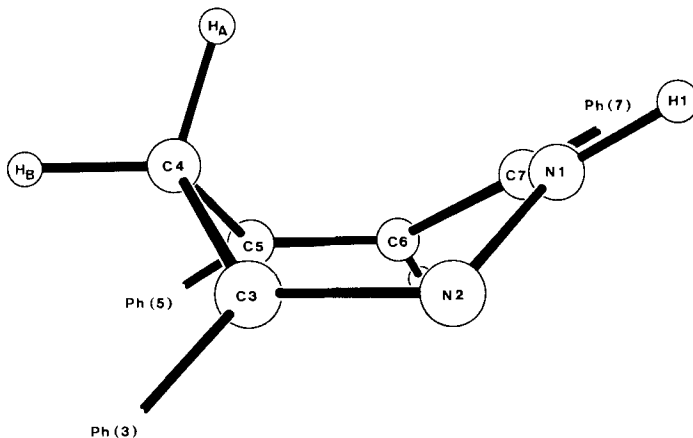
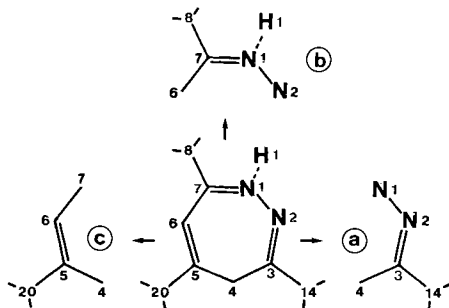


Fig. 2. Perspective view of the molecular conformation of the protonated 1,2-diazepine ring

Discussion. – a) *Conformation of the substituted 1,2-diazepine molecule.* Like its unprotonated homologue [3a], the N-protonated 1,2-diazepine ring exists in a ‘boat’ conformation (see Fig. 2). Inspection of the ring bond distances points to a strong localization of the double bonds; the same situation is also found in other diazepine crystal structures investigated so far.

The boat conformation of the 1,2-diazepine ring can be visualized as the assembly of three unsaturated systems, each one preserving its planarity. This approach is supported by the small observed deviations from the best planes (least squares) through atoms selected as follows (deviations in Å are given in parentheses):

- a. N(1) (0.04), N(2) (0.05), C(3) (0.00), C(4) (0.01), C(14) (0.02)
- b. N(1) (0.06), N(2) (0.10), H(1) (0.08), C(6) (0.05), C(7) (0.02), C(8) (0.09)
- c. C(4) (0.00), C(5) (0.00), C(6) (0.01), C(7) (0.01), C(20) (0.00)



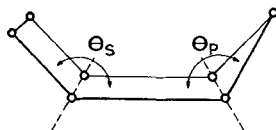
Sets **a** and **b** are assembled through the common single bond N(1)–N(2); sets **b** and **c** through the common bond C(6)–C(7); and the sets **a** and **c** are connected across the methylene group C(4). This simple treatment makes clear how the whole system minimizes the torsional strain across the double bonds. As is easily seen the chair

conformation would imply a large out-of-plane deformation of the double bonds N(2)–C(3) and C(5)–C(6) (torsional angles between 55° and 75° are measured on molecular models). These twists bring about overall torsional strains of 50–75 kcal/mole²) and the ensuing destabilization undoubtedly favors the boat conformation.

None of the phenyl groups is coplanar with its adjacent double bond; the out-of-plane torsion angles with the unsaturated systems **a**, **b** and **c** are, respectively, 38.2° , 33.0° and 38.9° . One of the rings Ph(3)³ and Ph(5) is rotated clockwise and the other anti-clockwise around the bond to the central ring. There is some non-bonded interaction between the equatorial hydrogen atom H_B(4) at the prow of the boat and the hydrogen atoms H(19) on Ph(3), and H(21) on Ph(5). Both the calculated distances H_B(4)–H(19) and H_B(4)–H(21) are 2.07 Å. The interaction of Ph(5) and Ph(7) with H(6) of the central ring is less severe and the calculated distances are 2.59 Å from H(9), and 2.17 Å from H(25).

The positive charge induced on N(1) by protonation shortens the adjacent bonds N(1)–N(2) and N(1)–C(7) by about 0.03 Å compared to the bond lengths observed on the unprotonated ring [3a]. In the rhodium complex of 3,5,7-triphenyl-1,2-diazepine [3b] the metal atom is co-ordinated to the nitrogen atom N(1), and the adjacent bonds to this atom have lengths very close to the values observed in the present study. This would support *Carty's* assumption that the diazepine ligand contributes mainly to the bond with the central atom.

The boat conformation of the cycloheptatriene ring implies C_s symmetry, and the ring of the 'unsymmetrically' substituted 7,7-dimethyl-cycloheptatriene-3-carboxylic acid [5] still very nearly conforms to this symmetry. The 1,2-diazepine ring, however, suffers some distortion which is reflected by the torsion angle across the N(2)–C(3) bond at the stern of the boat. In the present structure the dihedral angle between the best planes through atoms 3–4–5 and 2–3–5–6 is 131.1° (θ_P); the dihedral angle between the planes 2–1–7–6 and 2–3–5–6 is 144.8° (θ_S). The angles θ_P and θ_S (as illustrated below) afford an additional measure of comparison with the other already



mentioned structures. These angles are decreased by about 14° (the boat is more folded) when a carbon atom is substituted by a nitrogen atom at one of the folds (either stern or prow) of the boat (in the crystalline cycloheptatriene derivative [5]: $\theta_P = 132.1^\circ$ and $\theta_S = 155.6^\circ$). In the 1-tosyl-1,2-diazepine structure [3c] the nitrogen atoms are located at the prow of the boat and θ_P decreases to 118.2° . Protonation seems to impede the folding to some extent as is suggested by comparison with the structure of 3,7-bis(*p*-iodophenyl)-4,5,6-triphenyl-4*H*-1,2-diazepine [3a] where: $\theta_P = 128.7^\circ$ and $\theta_S = 139.7^\circ$.

²) These values are estimated from the twisting force constant of ethylene given by *R. L. Arnott & B. L. Crawford* [8].

³) Phenyl groups are labelled according to the number associated with the carbon atom to which they are bonded.

b) *Structure of the picrate anion and packing with the 1,2-diazepine molecule.* – The picrate ion is bonded to the diazepine ring through the hydrogen bond O(26)...H(1)...N(1), the length of which is 2.73 Å. This value compares well with the hydrogen bond distances found in the literature [6]. The angle between the N(1)–H(1) bond and a line along the direction N(1)–O(26) is 7.8°; the angle N(1)–N(1)–N(2) at N(1) is 114.5°. There is a strong intermolecular interaction, at a distance of 2.83 Å, between the diazepine atom N(2) and the atom O(35) of one of the *o*-nitro groups. The packing forces are large enough to bend the bulky nitro group ‘downward’ (see Fig. 1) out of the plane of the benzene ring, at an angle of 29.6°. This twisting is not due to the proximity of the central atom O(26) which otherwise, but to a lesser extent, prevents the coplanarity of the *o*-nitro groups with the benzene ring. The intramolecular strain is apparent in the ‘upward’ twist of the second *o*-nitro group which is bent at an angle of 18.7° out of the plane of the benzene ring. The short O(26)–O(37) distance amounts to 2.66, whereas the O(26)–O(35) distance, associated with the largest distortion, is 2.74 Å. The *p*-nitro group plane makes an angle of only 4.3° with the benzene ring. The magnitude of the distortions having an intramolecular origin compares well with other observations made on a picric acid complex [7].

There is an out-of-plane bending of the C(27)–O(26) bond and the departure of O(26) from the best plane through the benzene ring is 0.22 Å. The average deviation of the six ring atoms from the plane is only 0.036 Å, with a maximum deviation of 0.052 Å. The C(27)–O(26) bond distance (1.245 Å) lies close to the average value for the C–O distance in carboxylate ions (1.26 ± 0.01 Å) [4], whereas the C–OH bond distance in the undissociated picric acid is 1.33 Å [7].

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