

Acta Cryst. (1974). B30, 1631**2,4,4-Triphenyl-1,2-diazetidone-1-carboxylic Acid Ethyl Ester***

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Abstract. $C_6H_5NN(COOC_2H_5)C(C_6H_5)_2CO$, triclinic, $P\bar{1}$, $a=9.877$ (5), $b=14.037$ (5), $c=8.420$ (5) Å, $\alpha=94.45$ (3), $\beta=108.97$ (3), $\gamma=112.67$ (3)°; $Z=2$, $D_x=1.24$ g cm⁻³. Bond distances in the slightly puckered nearly planar four-membered diazetidine ring are N–N 1.450 (2), C–C 1.535 (2), N–C (ketone) 1.362 (2), and N–C 1.511 (2) Å.

Experimental. The title compound was given to us by Dr Robert Kerber (Kerber & Ryan, 1970) and recrystallized from ethanol. A yellow crystal with approximate dimensions 0.1 × 0.2 × 0.4 mm was glued to a Pyrex fiber and used for the measurement of 6599 data, including standards, with a Picker FACS-I automatic diffractometer system. Graphite-monochromated Cu $K\alpha$ ($\lambda=1.54051$ Å for $K\alpha_1$) X-rays were used in the collection of data by a θ - 2θ scan technique using a scintillation counter. No absorption correction was deemed necessary; $\mu=6.8$ cm⁻¹. All of the reflections within the sphere of reflection out to a 2θ angle of 106° were collected, yielding 3279 unique reflections of which 2688 were greater than their standard deviation. Stan-

dard deviations were estimated as described by St. Clair, Zalkin & Templeton (1971), with $(0.04I)^2$ as the additional term in $[\sigma(I)]^2$. The positions of all the non-hydrogen atoms were obtained from an E map phased

Table 1. *Positional parameters*Values are × 10³ for hydrogen atoms, × 10⁴ for the others.

	<i>x</i>	<i>y</i>	<i>z</i>
N(1)	-929 (1)	7749.8 (9)	1585 (2)
N(2)	-380 (1)	7074.1 (9)	2566 (1)
O(1)	1023 (1)	9441.8 (8)	1858 (1)
O(2)	-2555 (1)	6538 (1)	3334 (2)
O(3)	-452 (1)	6229.3 (9)	4667 (2)
C(1)	530 (2)	8595 (1)	2205 (2)
C(2)	1246 (2)	8001 (1)	3421 (2)
C(3)	1713 (2)	8438 (1)	5315 (2)
C(4)	861 (2)	8901 (1)	5837 (2)
C(5)	1250 (2)	9280 (2)	7564 (2)
C(6)	2488 (2)	9203 (2)	8766 (2)
C(7)	3332 (2)	8747 (2)	8266 (2)
C(8)	2953 (2)	8362 (1)	6543 (2)
C(9)	2495 (2)	7814 (1)	2944 (2)
C(10)	3887 (2)	8681 (2)	3188 (2)
C(11)	5068 (2)	8545 (2)	2801 (3)
C(12)	4870 (3)	7558 (2)	2152 (3)
C(13)	3491 (3)	6700 (2)	1892 (3)
C(14)	2290 (3)	6825 (2)	2278 (2)
C(15)	-2137 (2)	7378 (1)	-62 (2)
C(16)	-3165 (2)	6321 (1)	-648 (2)
C(17)	-4310 (2)	5987 (2)	-2284 (3)
C(18)	-4411 (3)	6693 (2)	-3305 (3)
C(19)	-3397 (2)	7740 (2)	-2697 (3)
C(20)	-2256 (2)	8099 (2)	-1072 (2)
C(21)	-1263 (2)	6617 (1)	3556 (2)
C(22)	-1151 (3)	5748 (3)	5874 (4)
C(23)	20 (5)	6080 (3)	7567 (4)
H(1)	-5 (2)	894 (1)	499 (2)
H(2)	62 (2)	960 (1)	791 (2)
H(3)	281 (2)	948 (2)	1001 (3)
H(4)	426 (3)	867 (2)	917 (3)
H(5)	356 (2)	802 (1)	622 (2)
H(6)	399 (2)	942 (2)	366 (2)
H(7)	607 (3)	923 (2)	299 (3)
H(8)	568 (3)	745 (2)	184 (3)
H(9)	322 (3)	601 (2)	138 (3)
H(10)	135 (2)	622 (1)	206 (2)
H(11)	-310 (2)	583 (1)	13 (2)
H(12)	-502 (2)	524 (2)	-263 (3)
H(13)	-523 (3)	643 (2)	-442 (3)
H(14)	-344 (2)	827 (2)	-341 (3)
H(15)	-151 (2)	885 (2)	-65 (2)
H(16)	-196 (5)	516 (4)	546 (6)
H(17)	-180 (5)	610 (3)	604 (5)
H(18)	85 (5)	581 (4)	728 (6)
H(19)	71 (4)	691 (3)	799 (5)
H(20)	-40 (3)	575 (2)	840 (3)

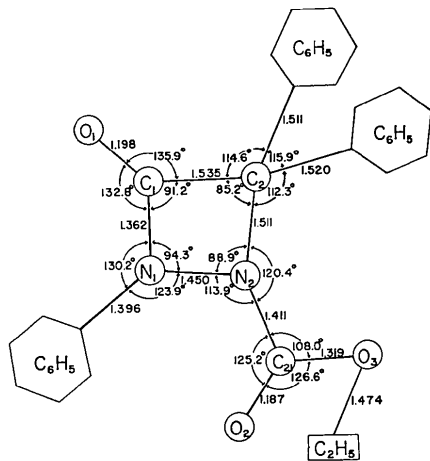


Fig. 1. Schematic drawing showing distances in Å and angles in the ring.

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Table 2. Anisotropic thermal parameters (\AA^2)

Anisotropic temperature factors have the form: $\exp \{-\frac{1}{4} \sum_i \sum_j (a_i^* a_j^* h_i h_j B_{ij})\}$ where a_i^* is a reciprocal cell edge and h_i is one of the Miller indices.

	B_{11}	B_{22}	B_{33}	B_{12}	B_{13}	B_{23}
N(1)	3.39 (5)	3.52 (5)	3.39 (5)	1.46 (4)	0.73 (4)	1.34 (4)
N(2)	3.27 (5)	3.41 (5)	3.47 (5)	1.45 (4)	1.06 (4)	1.40 (4)
O(1)	4.43 (5)	3.50 (5)	4.54 (5)	1.68 (4)	1.69 (4)	1.61 (4)
O(2)	3.86 (5)	7.00 (7)	6.08 (7)	2.47 (5)	2.27 (5)	2.89 (5)
O(3)	4.93 (6)	6.15 (6)	5.86 (6)	2.76 (5)	2.47 (5)	3.96 (5)
C(1)	3.39 (6)	3.33 (6)	3.21 (6)	1.63 (5)	1.33 (5)	0.88 (5)
C(2)	3.06 (6)	3.33 (6)	3.00 (6)	1.44 (5)	1.16 (5)	0.98 (5)
C(3)	3.38 (6)	3.95 (6)	2.98 (6)	1.56 (5)	1.39 (5)	0.95 (5)
C(4)	4.17 (7)	4.97 (8)	3.97 (8)	2.16 (6)	1.77 (6)	0.81 (6)
C(5)	5.51 (9)	5.80 (9)	4.77 (9)	2.29 (8)	2.94 (8)	0.49 (7)
C(6)	6.0 (1)	6.6 (1)	3.31 (8)	1.63 (8)	2.14 (7)	0.53 (7)
C(7)	5.13 (9)	7.1 (1)	3.10 (8)	2.21 (8)	1.13 (7)	1.25 (7)
C(8)	4.24 (7)	5.80 (9)	3.31 (7)	2.52 (7)	1.39 (6)	1.34 (6)
C(9)	3.82 (6)	4.60 (7)	2.75 (6)	2.45 (6)	1.38 (5)	1.37 (5)
C(10)	3.66 (7)	5.83 (9)	4.25 (8)	1.99 (7)	1.77 (6)	1.49 (7)
C(11)	4.52 (9)	9.0 (1)	5.6 (1)	3.6 (1)	2.75 (8)	3.35 (9)
C(12)	7.0 (1)	11.5 (2)	6.5 (1)	6.8 (1)	4.3 (1)	4.6 (1)
C(13)	9.1 (2)	7.6 (1)	7.5 (1)	6.1 (1)	4.5 (1)	2.5 (1)
C(14)	6.0 (1)	5.01 (9)	5.39 (9)	3.37 (8)	2.78 (8)	1.71 (7)
C(15)	3.06 (6)	4.30 (7)	3.30 (6)	1.72 (5)	0.95 (5)	0.98 (5)
C(16)	4.28 (7)	4.41 (8)	4.33 (8)	1.33 (7)	0.76 (6)	0.94 (6)
C(17)	4.84 (9)	5.8 (1)	5.0 (1)	0.89 (8)	0.15 (7)	0.29 (8)
C(18)	5.0 (1)	8.4 (1)	4.6 (1)	2.3 (1)	-0.34 (8)	1.26 (9)
C(19)	4.93 (9)	7.3 (1)	4.73 (9)	2.63 (9)	0.54 (7)	2.49 (9)
C(20)	4.14 (7)	5.12 (9)	4.23 (8)	2.16 (7)	1.04 (6)	1.85 (7)
C(21)	3.87 (7)	3.91 (7)	4.09 (7)	1.66 (6)	1.46 (6)	1.62 (6)
C(22)	6.6 (1)	11.1 (2)	8.1 (2)	3.2 (1)	3.6 (1)	7.3 (2)
C(23)	12.4 (2)	8.5 (2)	5.1 (1)	0.1 (2)	3.6 (1)	2.2 (1)

by the application of 'direct' methods. Hydrogen positions were obtained from a difference Fourier map calculated after a least-squares refinement of the heavy atoms with anisotropic thermal parameters was performed. Full-matrix least-squares refinements were then performed on all of the positional, anisotropic thermal (for the heavy atoms), and isotropic thermal (hydrogen atoms) parameters. The maximum final shift was less than 28% of its standard deviation. The weights were $1/\sigma^2(F)$, or zero if $F < \sigma(F)$. An empirical correction for extinction was applied, $F_o' = F_o(1 + 4 \times 10^{-8}I)$, which increased F_o by about 15% for the strongest reflection ($\bar{2}21$). The final R value, $R = \sum |\Delta F| / \sum |F_o|$, for all of the reflections was 0.045, and for the 2688 non-zero weighted reflections, 0.036.

The weighted R value, $R_w = [\sum w(\Delta F)^2 / \sum w F_o^2]^{1/2}$, was 0.046. The goodness-of-fit was 1.68. Atomic form

Table 3. Isotropic thermal parameters (\AA^2)

The temperature factor has the form: $\exp(-B \sin^2 \theta / \lambda^2)$.

	B		B
H(1)	4.7 (4)	H(11)	4.9 (4)
H(2)	5.7 (4)	H(12)	6.1 (5)
H(3)	6.1 (5)	H(13)	8.4 (6)
H(4)	7.7 (5)	H(14)	6.8 (5)
H(5)	4.4 (4)	H(15)	5.4 (4)
H(6)	6.4 (5)	H(16)	16.0 (15)
H(7)	8.4 (6)	H(17)	13.5 (12)
H(8)	7.1 (5)	H(18)	17.4 (16)
H(9)	8.3 (6)	H(19)	14.2 (11)
H(10)	4.7 (4)	H(20)	9.3 (7)

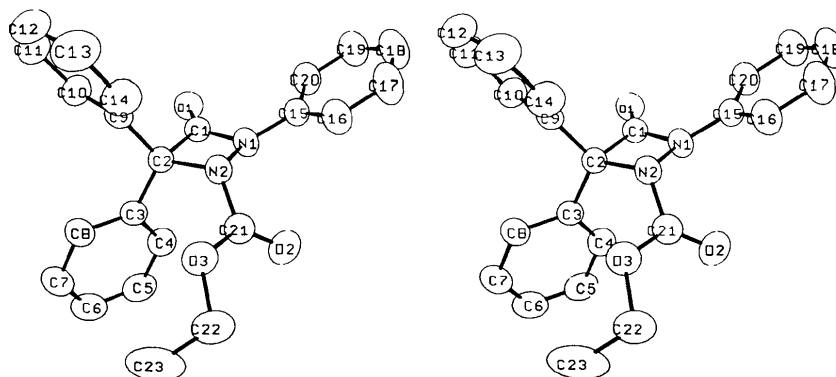


Fig. 2. Stereoscopic view of the molecule.

Table 4. *Interatomic distances* (Å)

N(1)—C(1)	1.362 (2)	C(5)—H(2)	1.00 (2)	C(15)—C(16)	1.370 (2)
N(1)—C(15)	1.396 (2)	C(6)—C(7)	1.372 (3)	C(15)—C(20)	1.386 (2)
N(1)—N(2)	1.450 (2)	C(6)—H(3)	0.98 (2)	C(16)—C(17)	1.369 (3)
N(2)—C(21)	1.411 (2)	C(7)—C(8)	1.380 (2)	C(16)—H(11)	0.99 (2)
N(2)—C(2)	1.511 (2)	C(7)—H(4)	1.03 (2)	C(17)—C(18)	1.372 (3)
O(1)—C(1)	1.198 (2)	C(8)—H(5)	0.99 (2)	C(17)—H(12)	0.97 (2)
O(2)—C(21)	1.187 (2)	C(9)—C(14)	1.368 (2)	C(18)—C(19)	1.357 (3)
O(3)—C(21)	1.319 (2)	C(9)—C(10)	1.379 (2)	C(18)—H(13)	0.95 (3)
O(3)—C(22)	1.474 (3)	C(10)—C(11)	1.382 (3)	C(19)—C(20)	1.360 (3)
C(1)—C(2)	1.535 (2)	C(10)—H(6)	1.03 (2)	C(19)—H(14)	1.00 (2)
C(2)—C(3)	1.511 (2)	C(11)—C(12)	1.364 (3)	C(20)—H(15)	0.98 (2)
C(2)—C(9)	1.520 (2)	C(11)—H(7)	1.04 (3)	C(22)—C(23)	1.413 (4)
C(3)—C(8)	1.369 (2)	C(12)—C(13)	1.362 (3)	C(22)—H(16)	0.84 (5)
C(3)—C(4)	1.394 (2)	C(12)—H(8)	0.97 (2)	C(22)—H(17)	0.99 (4)
C(4)—C(5)	1.380 (3)	C(13)—C(14)	1.394 (3)	C(23)—H(18)	1.11 (5)
C(4)—H(1)	0.97 (2)	C(13)—H(9)	0.92 (2)	C(23)—H(19)	1.06 (4)
C(5)—C(6)	1.358 (3)	C(14)—H(10)	0.93 (2)	C(23)—H(20)	0.99 (3)

Table 5. *Selected bond angles* (°)

N(2)—N(1)—C(1)	94.3 (1)	C(3)—C(2)—C(9)	115.9 (1)	C(10)—C(9)—C(14)	119.0 (2)
N(2)—N(1)—C(15)	123.9 (1)	N(2)—C(2)—C(1)	85.2 (1)	C(9)—C(10)—C(11)	120.3 (2)
C(1)—N(1)—C(15)	130.2 (1)	N(2)—C(21)—O(2)	125.2 (1)	C(10)—C(11)—C(12)	120.8 (2)
N(1)—N(2)—C(2)	88.9 (1)	N(2)—C(21)—O(3)	108.0 (1)	C(11)—C(12)—C(13)	119.2 (2)
N(1)—N(2)—C(21)	113.9 (1)	O(2)—C(21)—O(3)	126.6 (1)	C(12)—C(13)—C(14)	120.7 (2)
C(2)—N(2)—C(21)	120.4 (1)	C(21)—O(3)—C(22)	116.5 (1)	C(13)—C(14)—C(9)	120.1 (2)
N(1)—C(1)—C(2)	91.2 (1)	O(3)—C(22)—C(23)	110.7 (1)	C(16)—C(15)—C(20)	121.5 (2)
N(1)—C(1)—O(1)	132.8 (1)	C(4)—C(3)—C(8)	119.0 (2)	C(15)—C(16)—C(17)	118.0 (2)
C(2)—C(1)—O(1)	135.9 (1)	C(3)—C(4)—C(5)	121.0 (2)	C(16)—C(17)—C(18)	120.8 (2)
C(1)—C(2)—C(3)	114.6 (1)	C(4)—C(5)—C(6)	119.3 (2)	C(17)—C(18)—C(19)	120.5 (2)
C(1)—C(2)—C(9)	110.7 (1)	C(5)—C(6)—C(7)	120.2 (2)	C(18)—C(19)—C(20)	120.1 (2)
N(2)—C(2)—C(9)	112.3 (1)	C(6)—C(7)—C(8)	121.1 (2)	C(19)—C(20)—C(15)	119.0 (2)
N(2)—C(2)—C(3)	114.3 (1)	C(7)—C(8)—C(3)	119.4 (2)		

factors for spherical hydrogen were those of Stewart, Davidson & Simpson (1965) and for the other atoms those of Cromer & Waber (1965).

The final atomic parameters are given in Tables 1, 2 and 3.*

Discussion. The structure determination of this material was undertaken to verify the postulated structure of the four-membered ring, the dimensions of which are shown in Fig. 1. A least-squares plane calculated through the four atoms of the ring shows deviations of 0.028, -0.025, -0.026 and 0.023 for atoms N(1), N(2), C(1) and C(2) respectively. The above results indicate a slight puckering of the ring from planarity. With the exception of two bonds in the ring, the interatomic distances listed in Table 4 are similar to those tabulated for bonds with comparable chemical arrangements (Sutton, 1965). The N(1)—C(1) bond is 1.362 Å *versus* 1.333 Å in amides and the N(2)—C(2) bond is 1.511 Å *versus* 1.472 Å for an N—C bond in paraffinic and saturated heterocyclic compounds. The C—C and N—N bonds are both within 0.002 Å of the average values

reported for C—C and N—N single bonds. A list of bond angles is given in Table 5.

The relative orientation to the ring of the three phenyl rings, the ethyl ester and the oxygen, O(1), can be seen in the stereogram shown in Fig. 2. The oxygen, O(1), is -0.12 Å from the mean plane of the ring, and C(15), the bonding carbon atom of the phenyl group to the four-membered ring at N(1) is -0.55 Å from this plane. The remaining atoms are all more than 1.0 Å away from the plane of the ring. It can be noted that the three bonds about N(1) are somewhat more planar than those about N(2).

Some of the features of this molecule are found in another heterocycle studied by Fritchie & Wells (1968).

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* A table of calculated and observed structure factors has been deposited with the British Library Lending Division as Supplementary Publication No. SUP 30388 (13 pp., 1 microfiche). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.