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Structure of *N,N′,N″*-Triphenylbiuret

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Abstract. $C_{20}H_{17}N_3O_2$, $M_r = 331.37$, monoclinic, $P2_1/n$, $a = 12.619$ (1), $b = 8.285$ (1), $c = 16.469$ (1) Å, $\beta = 94.39$ (6)°, $V = 1716.7$ (3) Å^3, $Z = 4$, $D_x = 1.28$ g cm^{-3}, Cu $K\alpha$ radiation, $\lambda = 1.54184$ Å, $\mu = 0.647$ cm^{-1}, $F(000) = 696$, $T = 293$ (2) K, final $R = 0.038$ and $wR = 0.049$ for 2664 observed reflections and 294 refined parameters. The title molecule consists of three phenyl rings connected to the three N atoms of the biuret. Both intra- and intermolecular hydrogen bonds stabilize the molecular and crystal structure.

Introduction. Interest in biuret (NH_2—CO—NH—CO—NH_2) and biuret derivatives arose long ago (Wiedemann, 1848) because of the well known violet color (the 'biuret color') shown by alkaline solutions containing Cu^{2+} ions and biuret. In more modern times, this class of compounds has been investigated mainly by coordination chemists and biochemists because biuret is one of the simplest compounds mimicking a polypeptide chain (Siegel & Martin, 1982; Veersai & Rode, 1981). The biuret system may be viewed as made up of two amidic arms attached to a common N atom. Such a unique arrangement allows interaction between the two arms which directly affects the conformation of the biuret system. Studies addressing such problems are of relevance owing to the following points: (i) amide-based hydrogen bonding is a topic of ever-growing interest in molecular recognition (Jorgensen, 1991); (ii) several compounds related to biuret show a potent activity which may well depend on their conformation (Barton, Paluchowska, Mokrosz & Szneler, 1987; Al Sabbagh, Calmon & Calmon,

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Table 1. *Coordinates and equivalent isotropic thermal parameters* (Å²)

Anisotropically refined atoms are given in the form of the equivalent isotropic displacement parameter defined as (4/3) $[a^2B(1,1) + b^2B(2,2) + c^2B(3,3) + ab\cos(\gamma)B(1,2) + ac\cos(\beta)B(1,3) + bc\cos(\alpha)B(2,3)]$.

	x	*y*	*z*	B_{eq}
O1	0.97996 (7)	−0.0128 (1)	0.13144 (6)	5.68 (2)
O2	0.86787 (7)	0.2547 (1)	0.31462 (6)	5.39 (2)
N1	0.81177 (8)	−0.1076 (1)	0.10740 (7)	4.39 (2)
N2	1.01588 (8)	0.1747 (2)	0.25610 (7)	5.10 (3)
N3	0.84596 (8)	0.0979 (1)	0.20114 (7)	4.27 (2)
C1	0.7346 (1)	0.1416 (2)	0.19502 (8)	4.43 (3)
C2	0.6684 (1)	0.0958 (2)	0.25329 (9)	5.36 (3)
C3	0.5620 (1)	0.1423 (2)	0.2452 (1)	6.82 (4)
C4	0.5243 (1)	0.2333 (3)	0.1796 (1)	7.71 (5)
C5	0.5903 (2)	0.2793 (3)	0.1223 (1)	7.80 (5)
C6	0.6968 (1)	0.2347 (2)	0.1294 (1)	6.00 (4)
C7	0.88615 (9)	−0.0098 (2)	0.14455 (8)	4.13 (3)
C8	0.8286 (1)	−0.2224 (2)	0.04542 (8)	4.41 (3)
C9	0.7427 (1)	−0.3188 (2)	0.0201 (1)	6.10 (4)
C10	0.7519 (2)	−0.4310 (2)	−0.0416 (1)	7.69 (5)
C11	0.8451 (2)	−0.4462 (2)	−0.0785 (1)	7.41 (5)
C12	0.9297 (2)	−0.3514 (2)	−0.0533 (1)	6.88 (4)
C13	0.9229 (1)	−0.2395 (2)	0.0083 (1)	5.55 (3)
C14	0.9107 (1)	0.1820 (2)	0.26218 (8)	4.21 (3)
C15	1.0959 (1)	0.2559 (2)	0.30564 (8)	4.44 (3)
C16	1.1965 (1)	0.2562 (2)	0.27750 (9)	5.10 (3)
C17	1.2794 (1)	0.3339 (2)	0.3216 (1)	5.72 (4)
C18	1.2633 (1)	0.4118 (2)	0.3928 (1)	5.79 (4)
C19	1.1636 (1)	0.4104 (2)	0.4208 (1)	5.98 (4)
C20	1.0793 (1)	0.3323 (2)	0.37802 (9)	5.52 (3)

Table 2. *Bond distances* (Å) *and angles* (°)

O1—C7	1.220 (2)	C5—C6	1.390 (2)
O2—C14	1.213 (2)	C8—C9	1.385 (2)
N1—C7	1.350 (2)	C8—C13	1.385 (2)
N1—C8	1.423 (2)	C9—C10	1.388 (3)
N2—C14	1.340 (2)	C10—C11	1.371 (3)
N2—C15	1.418 (2)	C11—C12	1.363 (3)
N3—C1	1.447 (2)	C12—C13	1.383 (2)
N3—C7	1.412 (2)	C15—C16	1.384 (2)
N3—C14	1.427 (2)	C15—C20	1.379 (2)
C1—C2	1.373 (2)	C16—C17	1.386 (2)
C1—C6	1.383 (2)	C17—C18	1.368 (2)
C2—C3	1.394 (2)	C18—C19	1.372 (2)
C3—C4	1.373 (3)	C19—C20	1.390 (2)
C4—C5	1.360 (3)		

C7—N1—C8	126.1 (1)	C9—C8—C13	119.1 (1)
C14—N2—C15	126.9 (1)	C8—C9—C10	120.0 (2)
C1—N3—C7	120.5 (1)	C9—C10—C11	120.6 (2)
C1—N3—C14	115.3 (1)	C10—C11—C12	119.4 (2)
C7—N3—C14	123.9 (1)	C11—C12—C13	121.2 (2)
N3—C1—C2	121.4 (1)	C8—C13—C12	119.8 (1)
N3—C1—C6	118.0 (1)	O2—C14—N2	125.0 (1)
C2—C1—C6	120.6 (1)	O2—C14—N3	118.8 (1)
C1—C2—C3	119.4 (1)	N2—C14—N3	116.2 (1)
C2—C3—C4	120.1 (2)	N2—C15—C16	116.0 (1)
C3—C4—C5	120.4 (2)	N2—C15—C20	124.5 (1)
C4—C5—C6	120.5 (2)	C16—C15—C20	119.5 (1)
C1—C6—C5	119.2 (2)	C15—C16—C17	120.1 (1)
O1—C7—N1	123.9 (1)	C16—C17—C18	120.8 (1)
O1—C7—N3	122.2 (1)	C17—C18—C19	119.0 (1)
N1—C7—N3	113.9 (1)	C18—C19—C20	121.3 (2)
N1—C8—C9	116.4 (1)	C15—C20—C19	119.4 (1)
N1—C8—C13	124.4 (1)		

1983). In spite of these arguments, little structural information is available for the biuret system (Gstrein & Rode, 1978; Veersai & Rode, 1981). In the present paper, we describe the structure of *N,N′,N″*-triphenylbiuret (Phbiuret).

Experimental. Phbiuret was obtained as described by Tada & Okawara (1970). Crystals were grown in methanol solution.

Unit-cell parameters and intensity data were obtained on an Enraf–Nonius CAD-4 diffractometer with graphite-monochromated Cu $K\alpha$ radiation.

Crystal size: 0.30 × 0.35 × 0.24 mm. Cell dimensions refined by least-squares fitting of 25 centered reflections monitored in the range $25 < \theta < 45°$. Space group from systematic extinctions and intensity statistics. Data collection parameters: ω–2θ scan, scan speed 3.3° min^{-1}, scan width $(0.7 + 0.14\tan\theta)°$, aperture $(1.6 + 0.5\tan\theta)$ mm; 3636 reflections measured to $(\sin\theta/\lambda)_{max} = 0.6095$ Å^{-1}, $0 < h < 15$, $0 < k < 10$, $-20 < l < 20$. Orientation control monitored after every 500 reflections; standard reflections ($1\bar{3}\bar{4}$, $\bar{1}3\bar{4}$, $3\bar{1}3$, 316), measured every 7200 s of scanning time, did not show any significant change in intensity (−1.3%). Lp and empirical absorption corrections (North, Phillips & Mathews, 1968) applied (transmission minimum = 60.48, maximum = 99.78%). Among 3328 unique reflections, 2664 were considered observed $[I > 3\sigma(I)]$. After merging for $P2_1/n$ space group, R_{int} was 0.026.

The structure was solved by direct methods (*MULTAN*80; Main *et al.*, 1980). Positions of all non-H atoms were obtained from the *E* map with the highest CFOM (3.00). Refinement by full-matrix least squares. Non-H atoms refined anisotropically. Positions of H atoms were obtained from a difference Fourier map and refined with isotropic temperature factors. Weighting function $1/\sigma(F)^2$ kept $\sum w(\Delta F)^2$ uniform over ranges of $\sin\theta/\lambda$ and $|F_o|$. Refinement converged to $R = 0.038$ and $wR = 0.049$ for 294 parameters and 2664 observed reflections. $(\text{Shift/e.s.d.})_{max} = 0.09$, $S = 0.80$, excursions in final difference Fourier map within 0.15 and −0.15 e Å^{-3}.

Atomic scattering factors with anomalous-dispersion coefficients from *International Tables for X-ray Crystallography* (1974, Vol. IV, pp. 99–101, 149–150). Calculations performed with *SDP* (B. A. Frenz & Associates, Inc., 1985) on the MicroVAX 3100 computer at CGS–Pavia.

Atomic coordinates, and bond distances and angles are given in Tables 1 and 2.* A view of the

* Lists of structure factors, anisotropic thermal parameters, H-atom parameters, torsion angles, least-squares planes and intermolecular contacts (< 3.5 Å) have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55340 (34 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: GE0305]

asymmetric unit is presented in Fig. 1 and the hydrogen-bonding scheme in Fig. 2.

Discussion. *N,N′,N″*-triphenylbiuret consists of three phenyl rings (C1–C6, C8–C13 and C15–C20) connected to a biuret moiety [N1—C7(—O1)—N3—C14(—O2)—N2] which forms an intramolecular hydrogen bond (N2—HN2···O1).

The three phenyl rings do not present any anomalous features in their planarities, bond distances (Allen *et al.*, 1987) and aromaticity indices (Gdaniec, Turowska-Tyrk & Krygowski, 1989; Bird, 1985, 1986).

Within the biuret moiety, while the individual —NH—CO—N— groups are planar within the standard error, the overall —NH—CO—N—CO—NH— fragment is not planar, the angle between the two —NH—CO—N— groups being 12.8 (2)°. This value is not far from those (5–10°) observed in $[Cd(biuret)_2]Cl_2$ (Cavalca, Nardelli & Fava, 1960), biuret.$0.6H_2O$ (Hughes, Yakel & Freeman, 1961), $K_2[Cu(biuret)_2].4H_2O$ (Freeman, Smith & Taylor, 1961), $[Zn(biuret)_2]Cl_2$ (Nardelli, Fava & Giraldi, 1963), $[Cu(biuret)_2]Cl_2$ (Freeman & Smith, 1966) and $[Sr(biuret)_4](ClO_4)_2$ (Haddad & Gentile, 1975), but very different from that (43°) observed in [Ni(Phbiuret)$(Et_3P)_2$] (Hoberg, Oster, Krüger & Tsay, 1983) where complexation to the $[Ni(Et_3P)_2]^{2+}$ fragment brings about strong deformations in the Phbiuret ligand. However, the sums of the angles around N1, N3 and N2 are 358.3 (1.9), 359.7 (1.9) and 359.1 (3)°, respectively, and it is therefore reasonable to consider all the N atoms as sp^2 hybridized. Consequently, a certain electronic delocalization exists throughout the biuret fragment. The bond distances and angles within that fragment are comparable with those reported for the above compounds. The conformation of the biuret moiety is stabilized by the intramolecular hydrogen bond N2—HN2···O1 [N2—HN2 = 0.93 (2), O1···HN2 = 1.796 (2), N2···O1 = 2.587 (2) Å, N2—HN2···O1 = 141 (1)°]. A six-membered ring is thus formed according to the general rule that 1,6 intramolecular hydrogen bonding is preferred to the alternative intermolecular interaction (Etter, 1990). It is worth noting that a similar hydrogen bond is present in $[Cd(biuret)_2]Cl_2$ and biuret.$0.6H_2O$, and it has been spectroscopically (^{1}H NMR and IR) observed in other related diamide systems (Pirkle, Robertson & Hyun, 1984; Pirkle & Simmons, 1983).

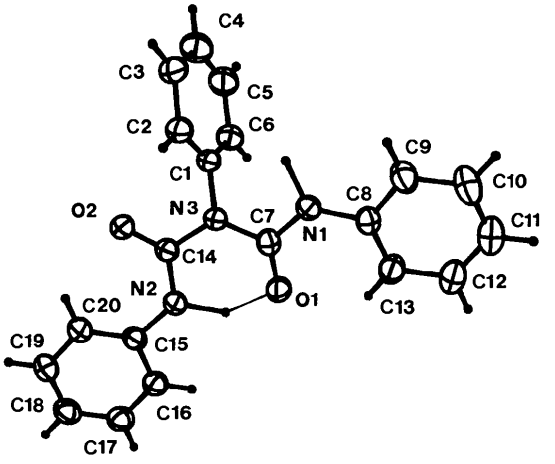

Fig. 1. Numbering scheme and thermal ellipsoids drawn at the 30% probability level.

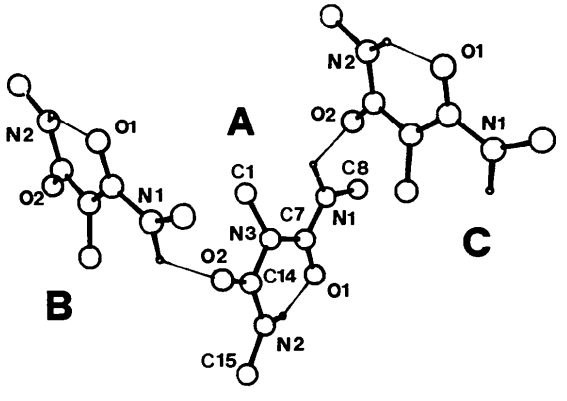

Fig. 2. Hydrogen-bonding scheme (*A*: x, y, z; *B*: $\frac{3}{2}-x, \frac{1}{2}+y, \frac{1}{2}-z$; *C*: $\frac{3}{2}-x, -\frac{1}{2}+y, \frac{1}{2}-z$).

The C1—N3 bond is significantly longer [1.447 (2) Å] than the C8—N1 and C15—N2 bonds [1.423 (2) and 1.418 (2) Å]. While the phenyl rings C8–C13 and C15–C20 are roughly coplanar with the biuret moiety [they form dihedral angles of 11.6 (2) and 10.9 (2)° with it], the ring C1–C6 is folded 107.07 (4)° out of this plane. The reasons for avoiding a completely planar molecule are probably steric. The C1–C6 ring cannot be coplanar with the biuret moiety mainly because of the steric hindrance of HN1. It is worth noting that the orientation of the C8–C13 and C15–C20 phenyl rings is stabilized by C—H···O hydrogen bonds; in fact, both the O1···HC13 and the O2···HC20 bonds [2.16 (1) and 2.27 (1) Å] are shorter than the 2.4 Å limit set by Taylor & Kennard (1982) for C—H···O interaction.

There are few short non-bonding intermolecular contacts and no evident stacking of the phenyl rings. The main intermolecular feature is the chain of N1—HN1···O2^i hydrogen bonds parallel to 2_1 [N1—HN1 = 0.97 (1), O2···HN1 = 2.02 (1), N1···O2 = 2.022 (2) Å, N1—HN1···O2 = 153 (1)°, symmetry code: (i) $\frac{3}{2}-x, y-\frac{1}{2}, \frac{1}{2}-z$]. Since these are weaker than the intramolecular hydrogen bond N2—HN2···O1, the C14—O2 bond [1.213 (2) Å] is slightly shorter than the C7—O1 bond [1.220 (2) Å]. The presence of intermolecular hydrogen bonds obeys the

rule that the best proton donors and acceptors remaining after intramolecular hydrogen-bond formation form intermolecular bonds to one another (Etter, 1990). Analogous intermolecular hydrogen bonds were observed in $[Cd(biuret)_2]Cl_2$ and biuret.$0.6H_2O$, suggesting that the overall hydrogen-bonding scheme (both inter- and intramolecular) in biuret derivatives is hardly influenced by the surroundings (for example, substituents on the N atoms or complexation to metals). It is also worth noting that the N1—HN1 and N2—HN2 bonds, which adopt an *anti* relationship with respect to their neighboring carbonyl groups, behave as in the studied diaryl ureas in which no intramolecular interaction is observed between the O atom and the N-bonded H atoms (Etter & Panunto, 1988).

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Structural Aspects of the 6,11-Dihydro-11-oxodibenz[*b*,*e*]oxepin Skeleton

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Abstract. (I) 6,11-Dihydro-11-oxodibenz[*b*,*e*]oxepin-2-acetic acid, $C_{16}H_{12}O_4$, $M_r = 268.27$, monoclinic, $P2_1/n$, $a = 11.004$ (1), $b = 12.6209$ (9), $c = 9.6920$ (8) Å, $\beta = 108.224$ (7)°, $V = 1278.5$ (3) Å^3, $Z = 4$, $D_m = 1.39$, $D_x = 1.39$ g cm^{-3}, Cu $K\alpha$, $\lambda = 1.54184$ Å, $\mu = 7.9$ cm^{-1}, $F(000) = 560$, $T = 295$ K, $R = 0.041$ for 2422 observed reflections. (II) Methyl 6,11-dihydro-11-oxodibenz[*b*,*e*]oxepin-2-acetate, $C_{17}H_{14}O_4$, $M_r = 282.30$, monoclinic, $P2_1/c$, $a = 13.644$ (1), $b = 8.878$ (1), $c = 13.000$ (2) Å, $\beta =$

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