Table 3. *Comparison of some molecular features of capnoidine and bicuculline*

| | Capnoidine | Bicuculline |
|---|---|---|
| Dihedral angle between planes *A* and *D* (°) | 42·3 (2) | 14·3 |
| Torsion angle N—C5—C11—O5 (°) | 64·5 (4) | 164·1 |
| Conformation of asymmetric centers C5, C11 | *R*, *R* | *R*, *S* |
| Ring *B* | Almost screw-boat | Half-chair |

In the five-membered rings containing methylenedioxy bridges, there are two types of C—O single bonds: the shorter type in the range 1·360 (5)–1·389 (6) Å involve the C atom of an aromatic ring, whereas the longer type in the range 1·427 (6)–1·439 (7) Å involve the C atom of the $CH_2$ groups. The mean value of the three C—N—C angles is 113·0 (4)° indicating an $sp^3$ hybridization of the N atom.

In accordance with the puckering parameters of Cremer & Pople (1975) ring *B* [$Q$ = 0·509 (5) Å, $\varphi$ = 101·9 (6), $\theta$ = 68·7 (5)°] has almost a screw-boat shape, while in bicuculline the same ring possesses a half-chair conformation. A comparison of some molecular features of capnoidine and bicuculline is given in Table 3.

The interaction between the molecules, which lie parallel to the *c* axis (Fig. 2), occurs only through van der Waals contacts.

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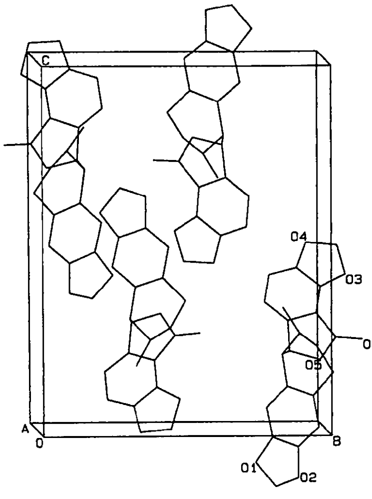

Fig. 2. Crystal packing diagram.

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# Structures of Two 1,2,3-Triazine *N*-Oxides

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**Abstract.** (I) 6-Methyl-4-phenyl-1,2,3-triazine 1-oxide, $C_{10}H_9N_3O$, $M_r$ = 187·2, monoclinic, $Pc$, $a$ = 10·333 (1), $b$ = 6·153 (1), $c$ = 7·058 (2) Å, $\beta$ = 92·48 (2)°, $V$ = 448·2 (2) Å$^3$, $Z$ = 2, $D_x$ = 1·387 Mg m$^{-3}$, $\lambda$(Cu $K\alpha_1$) = 1·5405 Å, $\mu$ = 0·782 mm$^{-1}$, $F(000)$ = 196, $T$ = 295 K, final $R$ = 0·036 for 656 observed reflections. (II) 4,5,6-Tris(*p*-tolyl)-1,2,3-triazine 2-oxide, $C_{24}H_{21}N_3O$, $M_r$ = 367·4, monoclinic, $P1$, $a$ = 9·330 (1), $b$ = 10·073 (1), $c$ = 5·903 (1) Å, $\alpha$ = 94·32 (1), $\beta$ = 98·62 (1), $\gamma$ = 113·01 (1)°, $V$ = 499·4 (1) Å$^3$, $Z$ = 1, $D_x$ = 1·222 Mg m$^{-3}$, $\lambda$(Cu $K\alpha_1$) = 1·5405 Å, $\mu$ = 0·612 mm$^{-1}$, $F(000)$ = 194, $T$ = 295 K, final $R$ = 0·039 for 1453 observed reflections. The bond distances in the *N*-oxides ($N^+$—$O^-$) are 1·264 (6) (I) and 1·243 (4) (II) Å. The dihedral angles between the triazine and phenyl rings are 22·7 (4) in (I) and 34·2 (5), 80·1 (5) and 34·5 (6)° in (II).

Table 1. *Details of data collection and structure refinement*

| | (I) | (II) |
|---|---|---|
| Data collection | | |
| Crystal size (mm) | 0·25 × 0·20 × 0·05 | 0·30 × 0·20 × 0·45 |
| Cell-parameter determination: number, $\theta$ range (°) | 20, 56–61 | 20, 57–60 |
| Max. $(\sin\theta)/\lambda$ (Å$^{-1}$) | 0·56 | 0·56 |
| Range $h$ | −11–11 | −10–10 |
| $k$ | 0–6 | −11–11 |
| $l$ | 0–7 | 0–6 |
| Standard reflexions: number; interval (ref.) | 3; 150 | 3; 150 |
| Number of reflexions | 818 | 1697 |
| independent | 664 | 1488 |
| with $F > 3\sigma(F)$ | 656 | 1453 |
| Merging $R$ for equivalent | 0·012 | 0·008 |
| reflexions | 65 | 172 |
| Refinement | | |
| $R$ | 0·036 | 0·039 |
| $wR$ | 0·039 | 0·039 |
| $G$* | 2·813 | 1·919 |
| $(\Delta/\sigma)_{max}$ | 0·18 | 0·08 |
| $\Delta\rho_{max}/\Delta\rho_{min}$ (e Å$^{-3}$) | 0·11/−0·21 | 0·24/−0·28 |

* $G = \sum w\{[|(|F_o|)^2 - (|F_c|)^2|]^2/(N_r - N_v)\}^{1/2}$.

Table 2. *Fractional atomic coordinates and equivalent isotropic thermal parameters*

$$B_{eq} = (1/3)\sum_i\sum_j B_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

| | $x$ | $y$ | $z$ | $B_{eq}$ (Å$^2$) |
|---|---|---|---|---|
| (I) | | | | |
| N(1) | 0·8056 (4) | 0·9128 (4) | 0·9266 (6) | 3·52 (7) |
| N(2) | 0·6940 (4) | 0·8325 (5) | 0·8635 (7) | 3·92 (8) |
| N(3) | 0·5993 (5) | 0·9703 (5) | 0·8059 (7) | 3·85 (8) |
| C(4) | 0·6118 (4) | 1·1921 (6) | 0·8159 (7) | 3·44 (8) |
| C(5) | 0·7281 (5) | 1·2710 (6) | 0·8820 (8) | 3·41 (8) |
| C(6) | 0·8276 (5) | 1·1264 (6) | 0·9359 (7) | 2·91 (8) |
| O(7) | 0·4948 | 0·8866 (5) | 0·7406 | 5·56 (9) |
| C(8) | 0·4981 (5) | 1·3221 (7) | 0·7479 (9) | 4·68 (12) |
| C(9) | 0·9594 (4) | 1·1915 (6) | 1·0021 (7) | 2·98 (8) |
| C(10) | 1·0620 (5) | 1·0487 (6) | 0·9868 (7) | 3·52 (9) |
| C(11) | 1·1864 (4) | 1·1083 (7) | 1·0464 (7) | 4·15 (10) |
| C(12) | 1·2084 (5) | 1·3112 (8) | 1·1273 (7) | 4·29 (11) |
| C(13) | 1·1073 (5) | 1·4559 (7) | 1·1421 (7) | 3·83 (10) |
| C(14) | 0·9830 (5) | 1·3977 (6) | 1·0792 (7) | 3·46 (9) |
| (II) | | | | |
| N(1) | 0·2535 (4) | 0·3151 (4) | 0·9953 (7) | 4·19 (8) |
| N(2) | 0·2307 (4) | 0·2077 (4) | 0·8304 (7) | 4·02 (8) |
| N(3) | 0·3405 (4) | 0·1967 (4) | 0·7202 (7) | 3·93 (8) |
| C(4) | 0·4870 (4) | 0·3042 (4) | 0·7796 (7) | 3·53 (9) |
| C(5) | 0·5257 (4) | 0·4231 (4) | 0·9508 (7) | 3·50 (9) |
| C(6) | 0·4013 (4) | 0·4226 (4) | 1·0555 (7) | 3·83 (10) |
| O(7) | 0·0951 | 0·1084 | 0·7742 | 6·01 (9) |
| C(8) | 0·5972 (4) | 0·2766 (4) | 0·6441 (7) | 3·76 (9) |
| C(9) | 0·5381 (5) | 0·2070 (4) | 0·4147 (7) | 3·93 (9) |
| C(10) | 0·6324 (5) | 0·1686 (5) | 0·2895 (8) | 4·55 (10) |
| C(11) | 0·7871 (5) | 0·1937 (5) | 0·3844 (8) | 5·03 (11) |
| C(12) | 0·8458 (5) | 0·2647 (5) | 0·6140 (9) | 5·38 (12) |
| C(13) | 0·7542 (5) | 0·3054 (5) | 0·7414 (8) | 4·52 (10) |
| C(14) | 0·8867 (8) | 0·1447 (8) | 0·2528 (11) | 7·58 (19) |
| C(15) | 0·6872 (4) | 0·5430 (4) | 1·0120 (7) | 3·66 (9) |
| C(16) | 0·7431 (5) | 0·6406 (4) | 0·8591 (8) | 4·19 (9) |
| C(17) | 0·8895 (5) | 0·7578 (4) | 0·9226 (7) | 4·56 (11) |
| C(18) | 0·9857 (5) | 0·7784 (5) | 1·1366 (8) | 4·45 (10) |
| C(19) | 0·9298 (5) | 0·6786 (4) | 1·2877 (8) | 4·55 (10) |
| C(20) | 0·7836 (5) | 0·5626 (5) | 1·2273 (8) | 4·29 (10) |
| C(21) | 1·1424 (6) | 0·9069 (6) | 1·2078 (11) | 5·96 (15) |
| C(22) | 0·4096 (5) | 0·5357 (5) | 1·2416 (8) | 4·26 (10) |
| C(23) | 0·5053 (6) | 0·6830 (5) | 1·2535 (9) | 5·33 (13) |
| C(24) | 0·5039 (7) | 0·7846 (5) | 1·4253 (9) | 6·08 (14) |
| C(25) | 0·4054 (6) | 0·7438 (5) | 1·5828 (8) | 6·20 (15) |
| C(26) | 0·3126 (6) | 0·5979 (6) | 1·5723 (9) | 6·11 (14) |
| C(27) | 0·3134 (5) | 0·4948 (5) | 1·4051 (9) | 5·02 (12) |
| C(28) | 0·4045 (13) | 0·8573 (8) | 1·7628 (11) | 9·45 (29) |

**Introduction.** In the course of our work on chemistry of 1,2,3-triazines, various derivatives have been synthesized including 1,2,3-triazine *N*-oxides (Ohsawa, Arai, Ohnishi & Igeta, 1981; Ohsawa, Arai, Ohnishi, Kaihoh, Yamaguchi, Igeta & Iitaka, 1986; Ohsawa, Kaihoh, Itoh, Okada, Kawabata, Yamaguchi & Igeta, 1988). A focus of our interest is the existence of a back donation effect of an unshared pair of electrons on the electron-deficient nitrogen in 1,2,3-triazine *N*-oxides. Previous analysis revealed the existence of this effect in several N(2)-oxides and an N(2)-imine (Yamaguchi, Ohsawa & Itoh, 1990). This paper presents the X-ray crystallographic analysis of 1,2,3-triazine 1- and 2-oxide in order to investigate the structural properties of the 1,2,3-triazine ring system including electronic features.

**Experimental.** Preparation and physical properties of (I) are discussed by Ohsawa *et al.* (1986). (II) was obtained by the oxidation of 4,5,6-triphenyl-1,2,3-triazine (Neunhoeffer, Clausen, Votter, Ohl, Kruger & Angermund, 1985) with *m*-chloroperbenzoic acid (*m*-CPBA) in $CH_2Cl_2$. Details of data collection and refinement are listed in Table 1. Intensity data were collected with a Rigaku AFC-5 four-circle diffractometer, $\omega$–$2\theta$ scan method, $\omega$-scan width $(1{\cdot}3 + 0{\cdot}41\tan\theta)^\circ$ and scan speed 16° min$^{-1}$. Intensity variation was less than 3% for both crystals. Intensities corrected for Lorentz and polarization factors, but absorption correction not applied. Structure solved using the *SAPI*85 (Yao, Zheng, Qian, Han, Gu & Fan, 1985) version of *MULTAN*80 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980). The refinement was carried out by the full-matrix least-squares method with anisotropic temperature factors for non-H atoms. The function minimized was $\sum[|(|F_o|)^2 - (|F_c|)^2|]^2$ with $w = 1/[\sigma^2(F_o) + 0{\cdot}02(F_o)^2]$, $\sigma(F_o)$ being determined from counting statistics. All H atoms located from a difference Fourier map and theoretical calculations were refined, and initial thermal parameters were set at the equivalent isotropic thermal parameter of each bonded atom. All major computations performed on a PANAFACOM computer with the *RCRYSTAN* (Rigaku Corporation, 1985) X-ray analysis system. The atomic scattering factors were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV).

**Discussion.** Final atomic parameters are listed in Table 2.[*] The bond lengths and angles in the triazine rings are listed in Table 3.

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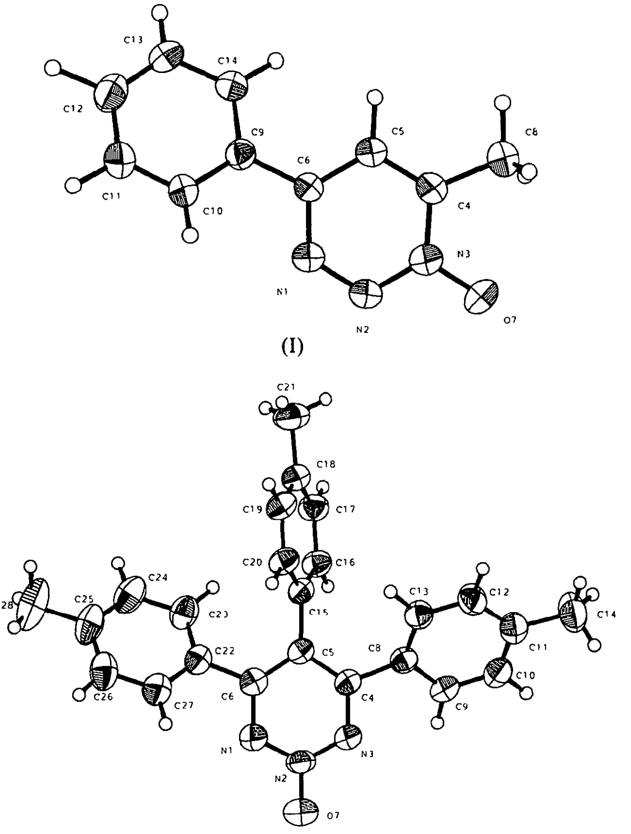
[*] Lists of structure amplitudes, further bond lengths and angles, anisotropic thermal parameters, H-atom parameters, and least-squares-planes data have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54131 (13 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 3. *Bond lengths* (Å) *and angles* (°) *in the triazine rings*

| | (I) | (II) |
|---|---|---|
| N(1)—N(2) | 1·314 (6) | 1·328 (6) |
| N(2)—N(3) | 1·345 (6) | 1·328 (7) |
| N(2)—O(7) | | 1·243 (4) |
| N(3)—O(7) | 1·264 (6) | |
| N(3)—C(4) | 1·373 (5) | 1·343 (5) |
| C(4)—C(5) | 1·360 (7) | 1·399 (6) |
| C(5)—C(6) | 1·400 (7) | 1·393 (7) |
| C(6)—N(1) | 1·335 (5) | 1·353 (5) |
| | | |
| C(6)—N(1)—N(2) | 122·0 (4) | 117·0 (4) |
| N(1)—N(2)—N(3) | 118·8 (3) | 125·6 (3) |
| N(2)—N(3)—C(4) | 123·0 (4) | 117·1 (4) |
| N(3)—C(4)—C(5) | 116·9 (4) | 122·5 (4) |
| C(4)—C(5)—C(6) | 119·6 (3) | 115·2 (3) |
| C(5)—C(6)—N(1) | 119·4 (4) | 122·3 (4) |
| N(1)—N(2)—O(7) | | 117·4 (4) |
| O(7)—N(2)—N(3) | | 116·9 (4) |
| N(2)—N(3)—O(7) | 116·9 (3) | |
| O(7)—N(3)—C(4) | 120·0 (4) | |



(I)

(II)

Fig. 1. *ORTEP* drawings of compounds (I) and (II). Ellipsoids are drawn at the 50% probability level while isotropic hydrogen thermal parameters are represented by spheres of arbitrary size.

Fig. 1 shows *ORTEP* drawings (Johnson, 1965) of the molecules. The triazine ring is almost planar, with maximum displacement from the least-squares plane of 0·015 (5) Å for N(3) (I) and 0·003 (5) Å for N(3) (II). This planarity and the endocyclic bond distances indicate an extensive delocalization of $\pi$-electron density for both (I) and (II). The respective bond lengths are close to each other. However, differences in N(3)—C(4) [1·373 (5) (I), 1·343 (5) Å (II)] and C(4)—C(5) [1·360 (7) (I), 1·399 (6) Å (II)] are found. The opposite tendency for (I) and (II) is also found in the angles N(1)—N(2)—N(3) [118·8 (3) (I), 125·6 (3)° (II)] and N(2)—N(3)—C(4) [123·0 (4) (I), 117·6 (3)° (II)]. These differences may be ascribed to the different site of the *N*-oxide moiety. The *N*-oxide bond distances [1·264 (6) (I) and 1·243 (4) Å (II)] indicate that the contribution of double-bond character to these bonds is greater than that found in pyridine *N*-oxides [1·33 and 1·37 Å (Ülkü, Huddle & Morrow, 1971)]. In the 2-oxide (II), lengthened N—N bonds and widened ∠N(2) in comparison with the *N*-free compounds including unsubstituted 1,2,3-triazine (Yamaguchi, Ohsawa, Arai, Ohnishi, Igeta & Iitaka, 1983) are consistent with the situations found in 4-methyl-6-phenyl-1,2,3-triazine 2-oxide and 4,6-dimethyl-1,2,3-triazine 2-oxide (Yamaguchi *et al.*, 1990). This feature may point to an effect of back donation of the unshared pair of electrons on the electron-deficient nitrogen as observed in pyridine *N*-oxides (*Comprehensive Heterocyclic Chemistry*, 1984). Furthermore, we can also recognize this effect in the case of the 1-oxide (I) because the same structural feature mentioned above is observed in (I). The semiempirical molecular orbital calculation using *MNDO-PM*3 methods* (Stewart, 1989) also suggests the back donation effect. The calculated net atomic charges on O(7), N(3) and C(6) are −0·54, 0·93 and −0·11 e, respectively in (I), and on O(7), N(2) and C(5) are −0·50, 0·99 and −0·21 e, respectively in (II). The dihedral angle between the triazine and phenyl rings in (I) is 22·7(4)°. In (II) two tolyl phenyl rings are rotated in opposite directions by almost the same angle [34·2 (5) and 34·5 (6)°] and the torsion in the central tolyl group is close to perpendicular to the triazine ring [80·1 (5)°].

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* Used with standard parameters as implemented in *MOPAC*, Quantum Chemistry Program Exchange, No. 455. Indiana Univ., USA.

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*Acta Cryst.* (1991). C**47**, 2196–2198

## Electrostatic Potential for O—H—O in Tetragonal Ammonium Dihydrogenphosphate

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**Abstract.** $NH_4H_2PO_4$, $M_r = 115{\cdot}03$, tetragonal, $I\bar{4}2d$, $a = 7{\cdot}500\,(1)$, $c = 7{\cdot}550\,(2)$ Å, $V = 424{\cdot}7$ Å$^3$, $Z = 4$, $D_x = 1{\cdot}799$ g cm$^{-3}$, $\lambda(\mathrm{Cu}\,K\alpha) = 1{\cdot}5418$ Å, $\mu = 49{\cdot}08$ cm$^{-1}$, $F(000) = 240$, $T = 298$ K, $R = 0{\cdot}021$ for 99 independent reflections. The O—H—O system with O···O distance 2·493 (3) Å is described in terms of the H atom in a single site undergoing anisotropic thermal vibrations. Then the O···H distance is 1·31 (9) Å, the O—H—O angle is 145 (8)° and the maximum mean-square amplitude of vibration for H is 1·1 (7) Å$^2$. However, the probability distribution function for the proton could well have a double peak as reported in isomorphous $KH_2PO_4$ from high-resolution neutron diffraction. The electrostatic potential well in which the H occurs has a minimum of $-1{\cdot}13$ e Å$^{-1}$.

**Experimental.** Colorless crystals were grown from water. Crystal size 0·13 × 0·13 × 0·66 mm. CAD-4 diffractometer, cell dimensions from 19 centered reflections ($16 < 2\theta < 76°$), Cu $K\alpha$ radiation, Ni-filtered, intensities measured to $2\theta = 120°$, $\omega$–$2\theta$ scans, with $\omega$-scan width $(0{\cdot}7 + 0{\cdot}15\tan\theta)°$, scan speed 0·91–2·50° min$^{-1}$, $-8 \le h \le 8$, $-8 < k \le 8$, $0 \le l \le 8$, $h + k + l =$ odd not collected, 680 reflections measured. Three standard reflections ($\bar{2}20$, $\bar{1}$, $\bar{1}\bar{1}2$) were measured every 6000 s and varied in intensity by <3% during the data collection. An absorption correction was applied (Busing & Levy, 1957). Correction factors were in the range 0·412 to 0·580. Averaging ($R_{\mathrm{int}} = 0{\cdot}027$) gave 99 independent reflections, all with $F_o > 3\sigma(F_o)$.

After placing the P and N atoms according to Ueda (1948) and refining isotropic temperature factors, the O atom was located in a difference map. The positional and anisotropic temperature factors of the O atom were refined by full-matrix least-squares refinement on $F_o$, including all 99 independent reflections, using the computer program *POP* (Craven, Weber & He, 1977). The $H_N$ atom was located in a difference map. The $H_O$ was placed in a special position ($x$, 0·25, 0·125), with $x = 0{\cdot}146$ as starting value as assumed by Tenzer, Frazer & Pepinsky (1958). The positional parameters and isotropic temperature factor of the $H_N$ atom and the $x$ coordinate and the anisotropic temperature factors of the $H_O$ atom were refined. The atomic scattering factors were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV). A type I Lorentzian extinction correction was assumed (Becker & Coppens, 1974), giving $g = 0{\cdot}082\,(14)$ rad$^{-1}$ × 10$^{-4}$. Final $R = 0{\cdot}021$, $wR =$

0108-2701/91/102196-03$03.00