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# Structure of *N*-Methyl Benzaldehyde Nitrone


BY R. B. BEDFORD, P. A. CHALONER* AND P. B. HITCHCOCK

*School of Chemistry and Molecular Sciences, University of Sussex, Falmer, Brighton, England*


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**Abstract.** *N*-(Benzylidene)methylamine *N*-oxide, $C_8H_9NO$, $M_r = 135{\cdot}2$, orthorhombic, *Pbca*, $a = 9{\cdot}665\ (2)$, $b = 7{\cdot}981\ (1)$, $c = 19{\cdot}071\ (2)$ Å, $U = 1471{\cdot}0$ Å$^3$, $Z = 8$, $D_x = 1{\cdot}22$ g cm$^{-3}$, $\lambda$(Mo $K\alpha$) = 0·71069 Å, $\mu = 0{\cdot}8$ cm$^{-1}$, $F(000) = 576$, $T = 298$ K, $R = 0{\cdot}043$ and $wR = 0{\cdot}050$ for 661 observed reflections with $|F^2| > 3\sigma(F^2)$. The geometry about the carbon–nitrogen double bond is *Z*, and there is little deviation from planarity.


**Experimental.** The compound was prepared by reaction of *N*-methylhydroxylamine with benzaldehyde, and crystals were obtained by recrystallization from benzene/light petroleum. A crystal of dimensions 0·35 × 0·35 × 0·08 mm, cut from a larger plate crystal, was used for data collection. Unit-cell parameters by least squares fit of 25 reflections in the range $15 < 2\theta < 22°$, space group *Pbca* from systematic absences of 0*kl*, *k* odd; *h*0*l*, *l* odd; *hk*0, *h* odd; Enraf–Nonius CAD-4 diffractometer, graphite-monochromated Mo $K\alpha$ radiation, $\theta$–$2\theta$ scan, $\Delta\theta = (0{\cdot}8 + 0{\cdot}35\tan\theta)°$, max. scan time 1 min, 1538 measured unique reflections for $2 < \theta < 25°$ and $h\ 0 \rightarrow 9$, $k\ 0 \rightarrow 11$, $l\ 0 \rightarrow 22$, 661 reflections for $|F^2| > 3\sigma(F^2)$, $\sigma(F^2) = [\sigma^2(I) + (0{\cdot}04I)^2]^{1/2}$/Lp. Two standard reflections measured every hour showed 11·3% decay and a correction was applied to the data, Lorentz and polarization corrections, no absorption or extinction corrections. The structure was solved by direct methods using *SHELXS*86 (Sheldrick, 1985), refinement by full-matrix least squares with anisotropic thermal parameters. H atoms were located from the difference map and refined with isotropic thermal parameters. With a weighting scheme of $w = 1/\sigma^2(F)$, $\sum w(|F_o| - |F_c|)^2$ minimized, the final residuals were $R = 0{\cdot}043$, $wR = 0{\cdot}050$ for 661 observed reflections, 127 variables, $S = 1{\cdot}5$, $(\Delta/\sigma)_{max} = 0{\cdot}01$, $(\Delta\rho)_{max,min} = +0{\cdot}16$, $-0{\cdot}18$ e Å$^{-3}$ on a final difference map. Programs from *SDP-Plus Structure Determination Package* (B. A. Frenz & Associates, Inc., 1984) were run on a MicroVAX II computer. Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV). Atomic parameters are given in Table 1† and selected bond distances and angles are presented in Table 2. Fig. 1 shows the molecular structure and the numbering scheme.

**Related literature.** *Z*-Nitrones are generally the major products from this preparative route (Breuer, Aurich & Nielsen, 1988). The structure of the 4-chlorobenzaldehyde derivative has been determined (Folting, Lipscomb & Jerslev, 1964). The N—O bond is shorter and the C=N bond longer than in our case. In the derivative of 4-chloro-2,6-dimethylbenzaldehyde there is considerable deviation from planarity due to steric crowding (Jensen & Jerslev, 1969). Deviations from planarity are also noted in other highly substituted nitrones (Falshaw, Hashi &

* To whom correspondence should be addressed.

† Lists of structure factors, anisotropic temperature factors, bond lengths and angles involving H atoms, H-atom parameters, torsion angles and a packing diagram have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54360 (9 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Table 1. *Fractional atomic coordinates* ($\times 10^4$ for O, N, C; $\times 10^3$ *for* H) *and equivalent isotropic thermal parameters* ($Å^2 \times 10^3$)

| | $x$ | $y$ | $z$ | $U_{iso}/U_{eq}$ |
|---|---|---|---|---|
| O | 2539 (2) | 1193 (3) | 4605 (1) | 65 (1)* |
| N | 3712 (2) | 1962 (3) | 4659 (1) | 50 (1)* |
| C1 | 4147 (3) | 2879 (4) | 4026 (1) | 64 (2)* |
| C2 | 4478 (3) | 1985 (4) | 5218 (1) | 49 (1)* |
| C3 | 4168 (3) | 1246 (3) | 5888 (1) | 46 (1)* |
| C4 | 3035 (3) | 220 (3) | 6036 (1) | 52 (1)* |
| C5 | 2812 (3) | −361 (4) | 6710 (1) | 61 (2)* |
| C6 | 3716 (4) | 47 (4) | 7245 (1) | 65 (2)* |
| C7 | 4850 (3) | 1027 (4) | 7104 (1) | 64 (2)* |
| C8 | 5093 (3) | 1612 (4) | 6436 (1) | 54 (2)* |
| H1*a* | 344 (3) | 385 (4) | 394 (1) | 53 (9) |
| H1*b* | 403 (3) | 216 (4) | 365 (1) | 41 (8) |
| H1*c* | 506 (4) | 334 (5) | 405 (2) | 78 (12) |
| H2 | 530 (3) | 261 (3) | 516 (1) | 20 (7) |
| H4 | 245 (3) | −6 (3) | 567 (1) | 23 (7) |
| H5 | 209 (3) | −115 (4) | 682 (1) | 39 (9) |
| H6 | 354 (3) | −35 (3) | 772 (1) | 36 (8) |
| H7 | 551 (3) | 129 (4) | 747 (2) | 56 (9) |
| H8 | 588 (3) | 234 (3) | 633 (1) | 27 (7) |

* $U_{eq}$ is defined as one third of the trace of the orthogonalized $U_{ij}$ tensor.

Table 2. *Intramolecular distances* (Å) *and angles* (°) *with e.s.d.'s in parentheses*

| | | | |
|---|---|---|---|
| O—N | 1·293 (3) | N—C1 | 1·473 (4) |
| N—C2 | 1·298 (3) | C2—C3 | 1·438 (4) |
| C3—C4 | 1·395 (4) | C3—C8 | 1·406 (4) |
| C4—C5 | 1·383 (4) | C5—C6 | 1·383 (4) |
| C6—C7 | 1·373 (5) | C7—C8 | 1·378 (4) |
| O—N—C1 | 114·8 (2) | O—N—C2 | 124·9 (2) |
| C1—N—C2 | 120·3 (2) | N—C2—C3 | 127·2 (2) |
| C2—C3—C4 | 125·7 (2) | C2—C3—C8 | 116·2 (2) |
| C4—C3—C8 | 118·1 (2) | C3—C4—C5 | 120·4 (2) |
| C4—C5—C6 | 120·6 (3) | C5—C6—C7 | 119·6 (3) |
| C6—C7—C8 | 120·7 (3) | C3—C8—C7 | 120·5 (3) |

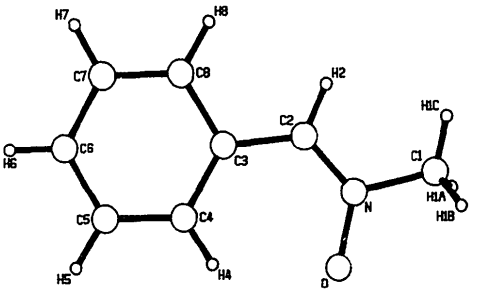


Fig. 1. Molecular structure and numbering scheme for PhCH=N(Me)O.

Taylor, 1985; Pritchard, Banks, DuBoisson & Tipping, 1991).

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# Mesogenic 4′-(4-Hydroxy-1-butoxy)biphenyl-4-carbonitrile and Non-Mesogenic 4-(4′-Cyano-4-biphenyloxy)-1-butyl Acrylate


By Stefan Gehring, Udo Quotschalla,* Helmut Paulus† and Wolfgang Haase‡

*Institut für Physikalische Chemie, Technische Hochschule Darmstadt, Petersenstr.* 20, *D*-6100 *Darmstadt, Germany*


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**Abstract.** (*A*) $C_{17}H_{17}NO_2$, $M_r = 267{\cdot}3$, triclinic, $P\bar{1}$, $a = 14{\cdot}418\ (6)$, $b = 9{\cdot}236\ (4)$, $c = 5{\cdot}651\ (3)$ Å, $\alpha = 89{\cdot}91\ (2)$, $\beta = 85{\cdot}37\ (2)$, $\gamma = 72{\cdot}93\ (2)°$, $V = 716{\cdot}8$ Å$^3$, $Z = 2$, $D_x = 1{\cdot}238$ g cm$^{-3}$, $\lambda$(Mo $K\alpha$) = 0·71069 Å, $\mu = 0{\cdot}46$ cm$^{-1}$, $F(000) = 284$, $T = 295$ K, final $R = 0{\cdot}0820$ for 1942 unique reflections with $F_o > 3\sigma(F_o)$. (*B*) $C_{20}H_{19}NO_3$, $M_r - 321{\cdot}4$, triclinic, $P\bar{1}$, $a = 12{\cdot}978\ (4)$, $b = 9{\cdot}941\ (3)$, $c = 6{\cdot}971\ (2)$ Å, $\alpha = 95{\cdot}81\ (1)$, $\beta = 100{\cdot}63\ (1)$, $\gamma = 102{\cdot}62\ (1)°$, $V = 853{\cdot}1$ Å$^3$, $Z = 2$, $D_x = 1{\cdot}251$ g cm$^{-3}$, $\lambda$(Mo $K\alpha$) = 0·71069 Å, $\mu = 0{\cdot}48$ cm$^{-1}$, $F(000) = 340$, $T = 297$ K,



* Present address: CIBA GEIGY Marienberg GmbH, Bensheim, Germany.

† Fachbereich Materialwissenschaft, Fachgebiet Strukturforschung, TH Darmstadt, Germany.

‡ To whom correspondence should be addressed.


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