References

CROMER, D. T. & LIBERMAN, D. (1970). *J. Chem. Phys.* **53**, 1891–1898.

CROMER, D. T. & MANN, J. B. (1968). *Acta Cryst.* A**24**, 321–324.

FLIPPEN, J. L., KARLE, J. & KARLE, I. L. (1970). *J. Am. Chem. Soc.* **92**, 3749–3754.

JOHNSON, C. K. (1965). *ORTEP*. Report ORNL-3794. Oak Ridge National Laboratory, Tennessee, USA.

RAO, B. H. L., SESHADRI, T. P. & RAO, L. M. (1987). *Acta Cryst.* C**43**, 495–497.

SHELDRICK, G. M. (1976). *SHELX*76. Program for crystal structure determination. Univ. of Cambridge, England.

SPEK, A. L., KOJIĆ-PRODIĆ, B. & LABADIE, R. P. (1984). *Acta Cryst.* C**40**, 2068–2071.

STEWART, R. F., DAVIDSON, E. R. & SIMPSON, W. T. (1965). *J. Chem. Phys.* **42**, 3175–3187.

VALENTE, E. J., EGGLESTON, D. S. & SCHOMAKER, V. (1987). *Acta Cryst.* C**43**, 533–536.

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Structure of Phenazine

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Abstract. $C_{12}H_8N_2$, $M_r = 180{\cdot}21$, monoclinic, $P2_1/n$, $a = 7{\cdot}083$ (1), $b = 5{\cdot}072$ (1), $c = 12{\cdot}794$ (8) Å, $\beta = 102{\cdot}34$ (2)°, $V = 449{\cdot}01$ (3) Å^3, $Z = 2$, $D_x = 1{\cdot}333$ g cm^{-3}, λ(Mo $K\alpha$) = 0·71069 Å, $\mu = 0{\cdot}756$ cm^{-1}, $F(000) = 188$, $T = 291$ K, $R = 0{\cdot}0532$ for 2005 observed reflections. The structure consists of phenazine molecules oriented about a centre of symmetry. The molecule is planar within experimental error, and a pseudo C_{2v} axis is observed in the molecule.

Experimental. Crystals of phenazine were crystallized from acetonitrile. An Enraf–Nonius CAD-4 diffractometer was used with graphite-monochromatized Mo $K\alpha$ radiation. Crystal size was 0·25 × 0·30 × 0·35 mm. Unit-cell parameters were obtained by least-squares fit of the setting angles of 25 reflections in the range $3 \le 2\theta \le 18°$. The intensities of 2702 reflections were measured ($\sin\theta/\lambda \le 0{\cdot}704$ Å^{-1}, $-9 \le h \le 9$, $0 \le k \le 7$, $0 \le l \le 18$, ω–2θ scan mode). No significant variation (< 3%) was found in the intensities of the intensity control reflections $3\bar{1}\bar{1}$ and 103. The data were corrected for Lorentz and polarization effects but no absorption correction was applied. 2005 reflections with $|F| \ge 34\sigma(F)$ were used in the calculations. The structure was solved with multisolution direct methods (*SHELXS*86; Sheldrick, 1990) and refined using full-matrix least-squares refinement (*SHELX*76; Sheldrick, 1976), minimizing $\sum w(|F_o| - |F_c|)^2$, $w = \{2{\cdot}7955/[\sigma^2(F) + 0{\cdot}000553F^2]\}$. The C and N atoms were refined with anisotropic, and H atoms with isotropic temperature factors, 81 parameters were varied. The refinement converged to $R = 0{\cdot}0532$, $wR = 0{\cdot}061$, $(\Delta/\sigma)_{max} = 0{\cdot}002$, $(\Delta/\sigma)_{mean} = 0{\cdot}001$, $\Delta\rho_{max} = 0{\cdot}52$, $\Delta\rho_{min} = -0{\cdot}35$ e Å^{-3}. The

Table 1. *Fractional coordinates* (× 10^4) *and equivalent isotropic temperature coefficients* (× 10^4) *for non*-H *atoms*

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

	x	y	z	U_{eq}(Å^2)
N1	8035 (1)	550 (1)	9523 (1)	461 (3)
C2	9491 (1)	1851 (2)	9233 (1)	423 (2)
C3	8523 (1)	−1293 (2)	10284 (1)	415 (2)
C4	7050 (1)	−2758 (2)	10629 (1)	522 (3)
C5	9077 (1)	3834 (2)	8429 (1)	551 (3)
C6	7519 (1)	−4624 (2)	11395 (1)	572 (4)
C7	10525 (2)	5155 (2)	8124 (1)	590 (4)

Table 2. *Bond lengths* (Å) *and bond angles* (°)

C2—N1	1·342 (1)	C4—C3	1·426 (1)
C3—N1	1·341 (1)	C6—C4	1·352 (2)
C3—C2^i	1·438 (1)	C7—C5	1·351 (2)
C5—C2	1·424 (2)	C7—C6	1·416 (1)

N1—C2—C3^i	121·56 (10)	C3^i—C2—C5	118·75 (9)
N1—C2—C5	119·70 (7)	C2—C5—C7	120·49 (8)
C2—N1—C3	116·72 (7)	C3—C4—C6	120·44 (7)
N1—C3—C2^i	121·73 (9)	C4—C6—C7^i	120·94 (9)
N1—C3—C4	119·74 (7)	C5—C7—C6^i	120·85 (10)
C2^i—C3—C4	118·53 (10)		

Symmetry code: (i) $2-x, -y, 2-z$.

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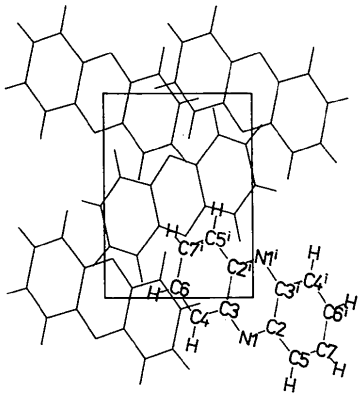

Fig. 1. A projection of the unit-cell content on the *ab* plane with the labelling of atoms.

largest residual electron density maximum is midway between C2 and C3^{i}. The second largest residual electron density maximum is less than 0·3 e Å^{-3}. Final fractional coordinates and equivalent isotropic temperature coefficients for non-H atoms are given in Table 1.* The atomic scattering factors used were those given in *SHELX*76. The molecular geometry is given in Table 2 with atom labelling shown in Fig. 1. The packing of the molecules in the unit cell is also given in Fig. 1. The figure was drawn with *PLUTO* (Motherwell & Clegg, 1978).

Related literature. The structure of phenazine was previously determined from photographic data (Glazer, 1970) with rather low accuracy ($R = 0{\cdot}10$) and not very precisely refined. Some of its TCNQ (7,7′,8,8′-tetracyano-*p*-quinodimethanide) derivatives are organic semiconductors (Fritchie, 1966; Morosin, 1975; Morosin, Plastas, Coleman & Stewart, 1978; Gundel, Sixl, Metzger, Heimer, Harms, Keller, Nöthe & Wehe, 1983; Endres, Keller, Moroni & Nöthe, 1980).

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* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53678 (13 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

ENDRES, H., KELLER, H. J., MORONI, W. & NÖTHE, D. (1980). *Acta Cryst.* B**36**, 1435–1440.
FRITCHIE, C. J. (1966). *Acta Cryst.* **20**, 892–898.
GLAZER, A. M. (1970). *Philos. Trans. R. Soc. London*, A**266**, 593–622.
GUNDEL, D., SIXL, H., METZGER, R. M., HEIMER, N. E., HARMS, R. H., KELLER, H. J., NÖTHE, D. & WEHE, D. (1983). *J. Chem. Phys.* **79**, 3678–3688.
MOROSIN, B. (1975). *Phys. Lett. A*, **53**, 455–456.
MOROSIN, B., PLASTAS, H. J., COLEMAN, L. B. & STEWART, J. M. (1978). *Acta Cryst.* B**34**, 540–543.
MOTHERWELL, W. D. S. & CLEGG, W. (1978). *PLUTO*. Program for plotting molecular and crystal structures. Univ. of Cambridge, England.
SHELDRICK, G. M. (1976). *SHELX*76. Program for crystal structure determination. Univ. of Cambridge, England.
SHELDRICK, G. M. (1990). *Acta Cryst.* A**46**, 467–473.

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Structure of a 7,6-Lactone*

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Abstract. 9-Ethynyl-8,8-dimethyl-10-oxatricyclo-[7.2.2.0^{1,7}]tridec-6-en-11-one, $C_{16}H_{20}O_2$, $M_r = 244{\cdot}34$, orthorhombic, $P2_12_12_1$, $a = 6{\cdot}207$ (2), $b = 13{\cdot}199$ (1), $c = 16{\cdot}865$ (3) Å, $V = 1381{\cdot}9$ (7) Å^3, $Z = 4$, $D_m = 1{\cdot}16$ (3), $D_x = 1{\cdot}174$ Mg m^{-3}, λ(Cu $K\alpha$) = 1·5418 Å, $\mu = 0{\cdot}563$ mm^{-1}, $F(000) = 528$, $T = 298$ K, final $R = 0{\cdot}052$, $wR = 0{\cdot}057$ for 1134 reflections with $I > 2\sigma(I)$. All the three fused ring systems are in the boat conformation and the packing of the molecules is stabilized by van der Waals interactions.

Experimental. Needle shaped crystals from benzene, 0·30 × 0·40 × 0·20 mm, D_m measured by flotation. Enraf–Nonius CAD-4 single-crystal diffractometer, graphite monochromator, $\omega/2\theta$ scan, cell dimensions from 20 centred reflections, $30 \le 2\theta \le 60°$. Three check reflections for every 100 reflections, no significant change in the intensity of standard reflections. $0 \le h \le 6$, $0 \le k \le 14$, $0 \le l \le 18$, 1134 reflections

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