Acta Cryst. (1986). C**42**, 1656–1657

(*E*)-*O*-Methyl-*p*-nitrobenzohydroximoyl Cyanide

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(*Received* 12 *March* 1986; *accepted* 3 *June* 1986)

Abstract. $C_9H_7N_3O_3$, $M_r = 205{\cdot}17$, monoclinic, $P2_1/c$, $a = 4{\cdot}036$ (1), $b = 11{\cdot}049$ (2), $c = 21{\cdot}745$ (3) Å, $\beta = 90{\cdot}63$ (1)°, $V = 969{\cdot}6$ (3) Å^3, $Z = 4$, $D_x = 1{\cdot}405$ g cm^{-3}, λ(Mo $K\alpha$) = 0·71069 Å, μ(Mo $K\alpha$) = 1·02 cm^{-1}, $F(000) = 424$, $T = 295$ K, final $R = 0{\cdot}035$ for 837 observed reflections. The *O*-methylhydroximoyl cyanide group is approximately planar with none of its atoms deviating by more than 0·012 (3) Å. The angle between this plane and the phenyl group is 9·1 (1)°.

Experimental. Colorless needles, unit-cell parameters by least-squares fit of 15 reflections in the range $16 < 2\theta < 24°$, $P2_1/c$ from systematic absences. Crystal 0·61 × 0·18 × 0·18 mm, automatic Syntex $P2_1$ diffractometer, graphite-monochromated Mo $K\alpha$ radiation, $\theta/2\theta$ scan mode, 1283 independent reflections in the range $3 < 2\theta < 45°$, *hkl* range, h −4→4, k 0→11, l 0→23, 837 observed with $I > 3\sigma(I)$, $\sigma(I)$ from counting statistics; two standard reflections (100, 012) remeasured after every 100 reflections did not show any significant change in intensity; Lorentz–polarization correction, no absorption or extinction correction; direct methods, *MULTAN*78 (Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978), refinement by full-matrix least squares using *SHELX*76 (Sheldrick, 1976), anisotropic, H located in difference Fourier map, H isotropic, $w = 1/(\sigma_F^2 + 0{\cdot}00214F^2)$, $\sum w(|F_o| - |F_c|)^2$ minimized, $R = 0{\cdot}035$, $wR = 0{\cdot}041$, $S = 0{\cdot}96$; $(\Delta/\sigma)_{max} = 0{\cdot}17$, $\Delta\rho_{max} = 0{\cdot}10$ e Å^{-3} in final difference Fourier map. Atomic scattering factors for C, H, N and O used were those stored in *SHELX*76. The final atomic parameters are given in Table 1.† Bond lengths, bond angles and torsion angles are listed in Table 2. Fig. 1 shows the molecule and the numbering scheme, Fig. 2 the packing of the molecules in the cell.

Table 1. *Fractional atomic coordinates with equivalent isotropic thermal parameters for the non-*H *and isotropic for* H *atoms (e.s.d.'s in parentheses)*

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

	x	y	z	U_{eq}/U(Å^2)
C(1)	0·2923 (5)	0·2294 (2)	0·5677 (1)	0·0562 (5)
C(2)	0·4304 (5)	0·3237 (2)	0·6083 (1)	0·0507 (5)
C(3)	0·3996 (6)	0·4438 (2)	0·5909 (1)	0·0602 (6)
C(4)	0·5301 (6)	0·5356 (2)	0·6259 (1)	0·0640 (6)
C(5)	0·6916 (6)	0·5064 (2)	0·6797 (1)	0·0585 (6)
C(6)	0·7245 (6)	0·3891 (2)	0·6992 (1)	0·0649 (6)
C(7)	0·5934 (6)	0·2972 (2)	0·6635 (1)	0·0620 (5)
N(5)	0·8413 (5)	0·6035 (2)	0·7167 (1)	0·0770 (5)
O(51)	0·9971 (6)	0·5754 (2)	0·7627 (1)	0·1054 (6)
O(52)	0·8050 (7)	0·7072 (2)	0·6997 (1)	0·1138 (8)
C(8)	0·0938 (7)	0·2684 (2)	0·5155 (1)	0·0642 (6)
N(8)	−0·0635 (6)	0·3018 (2)	0·4750 (1)	0·0852 (6)
N(9)	0·3133 (5)	0·1129 (2)	0·5670 (1)	0·0679 (5)
O(10)	0·5014 (5)	0·0684 (1)	0·6145 (1)	0·0812 (5)
C(11)	0·5260 (17)	−0·0616 (3)	0·6092 (3)	0·1201 (7)
H(3)	0·294 (5)	0·466 (2)	0·554 (1)	0·063 (6)
H(4)	0·517 (5)	0·612 (2)	0·614 (1)	0·073 (6)
H(6)	0·839 (6)	0·372 (2)	0·734 (1)	0·083 (7)
H(7)	0·621 (5)	0·212 (2)	0·676 (1)	0·069 (6)
H(111)	0·655 (10)	−0·089 (4)	0·643 (2)	0·146 (16)
H(112)	0·645 (12)	−0·076 (4)	0·576 (2)	0·164 (24)
H(113)	0·320 (13)	−0·096 (5)	0·598 (2)	0·193 (24)

Table 2. *Bond lengths* (Å), *bond angles* (°) *and selected torsion angles* (°) *with e.s.d.'s in parentheses*

C(1)–C(2)	1·471 (3)	C(5)–N(5)	1·467 (3)
C(1)–C(8)	1·448 (3)	C(6)–C(7)	1·380 (3)
C(1)–N(9)	1·290 (3)	N(5)–O(51)	1·216 (3)
C(2)–C(3)	1·385 (3)	N(5)–O(52)	1·212 (3)
C(2)–C(7)	1·394 (3)	C(8)–N(8)	1·141 (3)
C(3)–C(4)	1·370 (3)	N(9)–O(10)	1·367 (3)
C(4)–C(5)	1·371 (3)	O(10)–C(11)	1·444 (6)
C(5)–C(6)	1·370 (3)		

C(2)–C(1)–C(8)	117·5 (2)	C(6)–C(5)–N(5)	119·0 (2)
C(2)–C(1)–N(9)	133·6 (2)	C(5)–C(6)–C(7)	119·1 (2)
C(8)–C(1)–N(9)	108·9 (2)	C(2)–C(7)–C(6)	120·3 (2)
C(1)–C(2)–C(3)	118·8 (2)	C(5)–N(5)–O(51)	118·1 (2)
C(1)–C(2)–C(7)	122·7 (2)	C(5)–N(5)–O(52)	118·4 (2)
C(3)–C(2)–C(7)	118·5 (2)	O(51)–N(5)–O(52)	123·5 (2)
C(2)–C(3)–C(4)	121·7 (2)	C(1)–C(8)–N(8)	178·4 (3)
C(3)–C(4)–C(5)	118·4 (2)	C(1)–N(9)–O(10)	112·7 (2)
C(4)–C(5)–C(6)	122·0 (2)	N(9)–O(10)–C(11)	109·6 (3)
C(4)–C(5)–N(5)	119·0 (2)		

C(3)–C(2)–C(1)–C(8)	−7·8 (3)	C(7)–C(2)–C(1)–N(9)	−9·1 (4)
C(7)–C(2)–C(1)–C(8)	172·6 (2)	C(2)–C(1)–N(9)–O(10)	0·7 (4)
C(3)–C(2)–C(1)–N(9)	170·5 (2)	C(1)–N(9)–O(10)–C(11)	−177·5 (3)

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† Lists of structure factors, anisotropic thermal parameters and least-squares planes have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 43132 (10 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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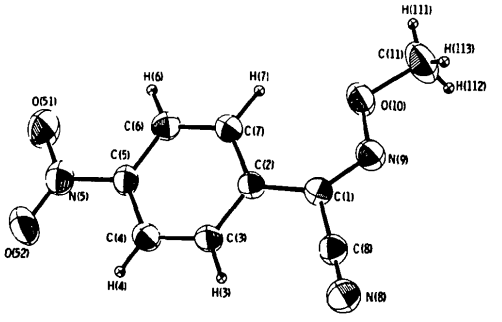

Fig. 1. *ORTEP* (Johnson, 1965) drawing of the molecule.

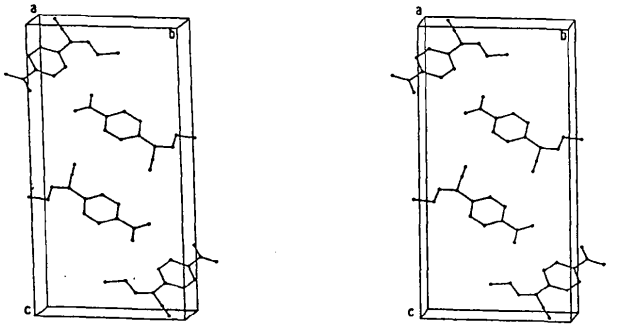

Fig. 2. Stereoscopic view of the packing of the molecules in the unit cell.

Related literature. This compound is one of a series of benzohydroximic acid derivatives that have been prepared for kinetic and stereochemical studies on nucleophilic substitution at the carbon–nitrogen double bond. Previous structures on related compounds include the *Z* and *E* isomers of *O*-methyl-*p*-nitrobenzohydroximoyl chloride, *p*-(NO_2)-$C_6H_4C(Cl)$-$NOCH_3$ (Bertolasi, Sacerdoti & Tassi, 1977; Johnson, Ghafouripour, Haug, Cordes, Pennington & Exner, 1985) and the *Z* and *E* isomers of ethylbenzohydroximate, $C_6H_5C(OC_2H_5)NOH$ (Larsen, 1971).

This work was supported by the Robert A. Welch Foundation (SSCC), the Petroleum Research Fund and the Texas Woman's University Institutional Research Grants Program (JEJ).

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Acta Cryst. (1986). C**42**, 1657–1658

2,2′-Azinodi-2-ethanenitrile

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(*Received* 5 *March* 1986; *accepted* 3 *June* 1986)

Abstract. $C_{16}H_{10}N_4$, $M_r = 258{\cdot}28$, triclinic, $P\bar{1}$, $a = 10{\cdot}617$ (1), $b = 8{\cdot}264$ (1), $c = 3{\cdot}938$ (1) Å, $\alpha = 92{\cdot}41$ (1), $\beta = 84{\cdot}37$ (1), $\gamma = 106{\cdot}29$ (1)°, $V = 330{\cdot}0$ (1) Å^3, $Z = 1$, $D_x = 1{\cdot}300$ g cm^{-3}, λ(Mo $K\alpha$) = 0·71069 Å, μ(Mo $K\alpha$) = 0·76 cm^{-1}, $F(000) = 134$, $T = 295$ K. Final $R = 0{\cdot}038$ for 870 observed reflections. The structure shows that the compound is the *Z,Z* isomer. The bond distances N–N, N=C and C–C(phenyl) are 1·396 (2), 1·289 (2) and 1·465 (2) Å, respectively.

Experimental. Crystals of the title compound are orange prisms. Unit-cell parameters by least-squares fit of 15 reflections in the range $15 < 2\theta < 24°$, space group $P\bar{1}$; crystal 0·48 × 0·26 × 0·13 mm, automatic Syntex $P2_1$ diffractometer, graphite-monochromated Mo $K\alpha$ radiation, $\theta/2\theta$ scan mode, 1156 independent reflections in range $3 < 2\theta < 50°$, *hkl* range,

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0108-2701/86/111657-02$01.50