Abstract. C₉H₇N₃O₃, Mᵣ = 205.17, monoclinic, P₂₁/c, a = 4.036(1), b = 11.049(2), c = 21.745(3) Å, β = 90.63(1)°. V = 969.6(3) Å³, Z = 4, Dᵣ = 1.405 g cm⁻³. λ(Mo Kα) = 0.71069 Å, μ(Mo Kα) = 1.02 cm⁻¹. F(000) = 424, T = 295 K, final R = 0.035 for 837 observed reflections. The O-methyl-
hydroximoyl 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 < 2θ < 24°, P₂₁/c from systematic absences. Crystal 0.61 x 0.18 x 0.18 mm, automatic Syntex P2, diffractometer, graphite-monochromated Mo Kα radiation, θ/2θ scan mode, 1283 independent reflections in the range 3 < 2θ < 45°, hkl, range, h = -4 to 4, k = 0 to 11, l = 0 to 23, 837 observed with I > 3 or (I), σ(λ) from counting statistics; two standard reflections (100, 012) measuring statistics; two standard reflections (100, 012) corrected, no absorption or extinction correction; Lorentz-polarization correction, isotropic for H atoms (e.s.d.'s in parentheses); direct methods, MULTAN78 (Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978), refinement by full-matrix least squares using SHELX76 (Sheldrick, 1976), anisotropic, H located in difference Fourier map, H isotropic, w = 1/(σ² + 0.00214F²), w = 1/(σ² + 0.00214F²), minimized, R = 0.035, wR = 0.041, S = 0.966; (Δσ) max = 0.17, Δρ max = 0.10 e Å⁻³ in final difference Fourier map. Atomic scattering factors for C, H, N and O used were those stored in SHELX76. The final atomic parameters are given in Table 1.† Bond lengths, bond angles and torsion angles are listed in Table 2. 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)

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

* To whom correspondence should be addressed.
† 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.

© 1986 International Union of Crystallography
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$_6$H$_4$C(Cl)-NOCH$_3$ (Bertolasi, Sacerdoti & Tassi, 1977; Johnson, GhaforiPou, Haug, Cordes, Pennington & Exner, 1985) and the Z and E isomers of ethylbenzohydroximate, C$_6$H$_5$C(OCH$_2$H$_2$)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).

References


2,2’-Azinodi-2-ethanenitrile

BY PATRICE DE MEESTER AND SHIRLEY S. C. CHU*

School of Engineering and Applied Science, Southern Methodist University, Dallas, Texas 75275, USA

AND JAMES ELVER JOHNSON

Department of Chemistry, Texas Woman’s University, Denton, Texas 76204, USA

(Received 5 March 1986; accepted 3 June 1986)

Abstract. C$_{16}$H$_{10}$N$_4$, $M_r$ = 258.28, triclinic, $\bar{P}1$, $a = 10.617$ (1), $b = 8.264$ (1), $c = 3.938$ (1) Å, $\alpha = 92.41$ (1), $\beta = 84.37$ (1), $\gamma = 106.29$ (1)$^\circ$, $V = 330.0$ (1) Å$^3$, $Z = 1$, $D_x = 1.300$ g cm$^{-3}$, $\lambda$(Mo $K\alpha$) = 0.71069 Å, $\mu$(Mo $K\alpha$) = 0.76 cm$^{-1}$, $F(000) = 134$, $T = 295$ K. Final $R = 0.038$ 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$^\circ$, space group $P\bar{1}$; crystal 0.48 x 0.26 x 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$^\circ$, $hkl$ range,

* To whom correspondence should be addressed.

© 1986 International Union of Crystallography