

Tableau 3. Distances (Å) et angles (°) des liaisons hydrogène

X	Y	X...Y	X-H	H...Y	X-H...Y
O(2)-H(O2)	O(3) ⁱ	2,910 (12)	0,84 (11)	2,26 (10)	134 (5)
O(3)-H(O3)	O(4) ⁱⁱ	2,727 (14)	1,00 (14)	1,92 (13)	135 (5)
O(4)-H(O4)	O(3) ⁱ	2,727 (14)	0,87 (10)	1,93 (10)	152 (5)
O(5)-H(O5)	O(3) ⁱⁱⁱ	2,739 (21)	0,74 (10)	2,42 (9)	108 (6)
O(6)-H(O6)	O(5) ⁱ	2,709 (13)	0,91 (13)	2,09 (12)	125 (5)
N-H(N)	O(1) ^{iv}	2,867 (10)	0,93 (10)	2,09 (10)	140 (4)

Codes de symétrie: (i) $-1 + x, y, z$; (ii) $1 + x, y, z$; (iii) $x, -1 + y, z$; (iv) $x, 1 + y, z$.

hydrogène intermoléculaires (Tableau 3). Elles sont comparables à celles observées dans des carbohydrates (Gatehouse & Poppleton, 1971; Park, Kim & Jeffrey, 1971) et dans les composés déjà cités (Satzke & Mackay, 1975; Sindt & Mackay, 1977; Darbon *et al.*, 1984; Darbon-Meyssonier *et al.*, 1985).

Dans la série des gluconamides le dérivé *N*-isopropyl est légèrement sucré, les dérivés *N*-diéthyl et *N*-cyclohexyl sont amers et le dérivé *N*-benzyl est le plus amer des quatre composés. Cette constatation confirme l'hypothèse selon laquelle l'allongement d'une chaîne hydrophobe serait corrélé à l'apparition de la saveur amère (Brussel, Peer & van der Heijden, 1975; Pautet & Nofre, 1978; Belitz, Chen, Jugel, Treleano, Wieser, Gasteiger & Marsili, 1979; Iwamura, 1981). Ces auteurs montrent une corrélation entre l'intensité de la saveur et la longueur de certaines chaînes latérales ainsi qu'une transition au goût amer lorsque ces chaînes dépassent une longueur optimale ce qui correspond à une augmentation des distances entre les points d'interaction [O(2) et O(3)] et le centre de la partie hydrophobe de la molécule.

Nous tenons à remercier Monsieur Sylvain Lecoq du Centre de Diffractométrie Automatique (Université de

Lyon I, France) pour l'assistance technique qu'il nous a apporté lors de l'enregistrement des intensités diffractées.

Références

- BELITZ, H. D., CHEN, W., JUGEL, H., TRELEANO, R., WIESER, H., GASTEIGER, J. & MARSILI, M. (1979). *Food Taste Chemistry*, pp. 93-131, édité par J. C. BOUDREAU. Washington: American Chemical Society.
- BRUSSEL, L. B. P., PEER, H. G. & VAN DER HEIJDEN, A. (1975). *Z. Lebensm. Unters. Forsch.* **159**, 337-433.
- CROMER, D. T. & MANN, J. B. (1968). *Acta Cryst.* **A24**, 321-324.
- DARBON, N., ODDON, Y., LACOMBE, J. M., DECOSTER, E., PAVIA, A. A. & REBOUL, J. P. (1984). *Acta Cryst.* **C40**, 1105-1107.
- DARBON-MEYSSONNIER, N., ODDON, Y., DECOSTER, E., PAVIA, A. A., PEPE, G. & REBOUL, J. P. (1985). *Acta Cryst.* **C41**, 1324-1327.
- GATEHOUSE, B. M. & POPPLETON, B. J. (1971). *Acta Cryst.* **B27**, 10-20.
- ISHIKAWA, T. (1964). *Nippon Kagaku Zasshi*, **85**, 897-900.
- IWAMURA, H. (1981). *J. Med. Chem.* **24**, 572-583.
- JOHNSON, C. K. (1965). *ORTEP*. Rapport ORNL-3794. Oak Ridge National Laboratory, Tennessee.
- MAIN, P., FISKE, S. J., HULL, S. E., LESSINGER, L., GERMAIN, G., DECLERCQ, J.-P. & WOOLFSON, M. M. (1980). *MULTAN80. A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data*. Univ. de York, Angleterre, et Louvain-la Neuve, Belgique.
- PARK, Y. J., KIM, H. S. & JEFFREY, G. A. (1971). *Acta Cryst.* **B27**, 220-227.
- PAUTET, F. & NOFRE, C. (1978). *Z. Lebensm. Unters. Forsch.* **166**, 167-170.
- SATZKE, L. O. G. & MACKAY, M. F. (1975). *Acta Cryst.* **B31**, 1128-1132.
- SHELDRIK, G. M. (1976). *SHELX*. Programme de détermination de structures cristallines. Univ. de Cambridge, Angleterre.
- SINDT, A. C. & MACKAY, M. F. (1977). *Acta Cryst.* **B33**, 2659-2662.
- STEWART, R. F., DAVIDSON, E. R. & SIMPSON, W. T. (1965). *J. Chem. Phys.* **42**, 3175-3187.

Acta Cryst. (1986). **C42**, 1766-1769

Structure of (±)-*cis*-Pinonic Acid

BY P. A. VANDERHOFF, H. W. THOMPSON AND R. A. LALANCETTE*

Rutgers University, Department of Chemistry, Olson Laboratories, Newark, NJ 07102, USA

(Received 30 January 1986; accepted 28 May 1986)

Abstract. (±)-*cis*-3-Acetyl-2,2-dimethylcyclobutaneacetic acid, C₁₀H₁₆O₃, *M_r* = 184.238, monoclinic, *P*2₁/*n*, *a* = 10.525 (6), *b* = 11.484 (3), *c* = 8.301 (5) Å, β = 97.62 (5)°, *V* = 994.5 (9) Å³, *Z* = 4, *D_m* =

1.22 (1), *D_x* = 1.230 g cm⁻³, λ(Mo Kα) = 0.71069 Å, μ = 0.76 cm⁻¹, *F*(000) = 400, *T* = 252 (1) K, *R* = 0.0466 for 2048 observed reflections. The structure contains C₁₀H₁₆O₃ molecules intermolecularly hydrogen-bonded in helical chains from acid group to ketone carbonyl. The bond distances and angles are

* To whom correspondence should be addressed.

normal and comparable to those in similar structures. The cyclobutane ring is puckered with a C(2)C(3)-C(4)/C(2)C(1)C(4) dihedral angle of $150.2 (1)^\circ$.

Introduction. This structural investigation was undertaken to determine the mode of hydrogen bonding in this keto-carboxylic acid. Normally C=O double-bond stretching vibrations of both carboxylic acid dimers and ketones are observed at $1710\text{--}1715\text{ cm}^{-1}$ in the solid-state IR. However, *cis*-pinonic acid has two carbonyl stretching frequencies in the solid-state IR (KBr pellet): one at 1732.7 cm^{-1} and the other at 1682.6 cm^{-1} . It has been suggested that the displacement from 1710 cm^{-1} for the C=O stretch indicates a strong hydrogen bond (Nakanishi, 1962), and that *intramolecular* hydrogen bonding may be possible if such molecules adopt twisted conformations (Gula & Spencer, 1980).

Experimental. Crystals were grown from commercially available material (Aldrich Chemical Co., Milwaukee, WI) by evaporation from ethanol at 295 K; elongated hexagon, $0.30 \times 0.35 \times 0.80\text{ mm}$, mounted on a glass fiber; density measured by flotation in a mixture of carbon tetrachloride and toluene. Syntex P2, diffractometer; graphite monochromator; 15 high-angle reflections used for orientation matrix and best cell dimensions, $h0l$ with $h+l \neq 2n$, $0k0$ with $k \neq 2n$ absent, space group chosen based on the absent reflections, non-chirality and density; absorption corrections based on Howell's analytical expression for polyhedra, $\min. = 1.022$, $\max. = 1.049$; $2\theta_{\max} = 60^\circ$ ($0 \leq h \leq 15$, $0 \leq k \leq 16$, $-12 \leq l \leq 12$); three standard reflections were recorded every 27 reflections ($\pm 1.5\%$), 3204 reflections measured in θ - 2θ scan mode, 3065 unique, 335 rejected by profile scan (Glick, 1975), 538 unobserved with $I < 3\sigma$, 2048 with $I \geq 3\sigma(I)$; structure solved with *MULTAN80* (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980), all non-H atoms found from best *E* map (CFOM = 3.00); H positions found in difference Fourier maps; H-atom positional parameters refined, *B*'s constant at $B_n + 1$, where *n* is the atom to which the H is bound; all non-H atoms refined with anisotropic thermal parameters; $\sum w|F_o| - |F_c|^2$ minimized, $R = 0.0466$, $wR = 0.0627$, $S = 1.04$, $w = 1/\sigma^2$ with $\sigma = -0.165F_o + 7.100$ for $F_o < 29.9$, $\sigma = 0.038F_o + 1.140$ for $F_o \geq 29.9$; last cycle $\max. \Delta/\sigma = 0.04$, $\text{av.} = 0.01$; final difference Fourier map: $\max. = 0.24$, $\min. = -0.08\text{ e \AA}^{-3}$; atomic scattering factors from *International Tables for X-ray Crystallography* (1974), no anomalous-dispersion corrections. Computer programs used: data reduction - local adaptation of Glick's (1975) Lp correction *SYNCOR* and Hollander's (1975) *ABSOR* [from Templeton & Templeton's (1973) *AGNOST*]; direct methods - *MULTAN80* (Main *et al.*, 1980); refinement and Fourier analysis -

LINUS (Andrews, 1974, from *ORFLS*, Busing, Martin & Levy, 1962); Fourier analysis and difference maps - *FORDAP* (Zalkin, 1962); analysis of variance - *NANOVA* (Lalancette, Cefola, Hamilton & La Placa, 1967); bond-distance and bond-angle error analysis - *ORFFE* (Busing, Martin & Levy, 1964); graphics - *ORTEPII* (Johnson, 1970).

Discussion. Table 1 lists the positional and isotropic thermal parameters.* Fig. 1 shows a view of the molecule indicating the numbering scheme. As can be seen in Fig. 2 (a partial packing diagram), the acid H of one molecule is bonded to the ketone carbonyl of the next molecule, forming an infinite helical chain of hydrogen-bonded molecules. Due to this hydrogen-bonding network, two different carbonyls are seen in the structure: the ketone carbonyl {C(7)-O(1) [1.218 (2) Å]}, which is hydrogen-bonded to O(3) of the next molecule, and the acid carbonyl {C(10)-O(2) [1.201 (2) Å]}, which is free. Table 2 gives the pertinent bond distances and angles; these are all normal and comparable to those in similar structures (Leiserowitz, 1976). The hydrogen-bond length is $2.700 (2)\text{ \AA}$. This is slightly longer than the 2.654 \AA reported in the structure of *cis*-9-carboxy-2-decalone (*cis*-3-oxobicyclo[4.4.0]decane-1-carboxylic acid) (Chadwick & Dunitz, 1979) or the 2.657 \AA observed in the structure of 10-carboxy- $\Delta^{1:9}$ -2-octalone (Vanderhoff, Thompson & Lalancette, 1987); these two keto acids are the only other ones thus far which have been reported to form similar helical chain structures. The cyclobutane ring is puckered with a dihedral angle of $150.2 (1)^\circ$, defined as the angle between the normals to the planes C(2)-C(3)-C(4) and C(2)-C(1)-C(4). This angle is consistent with others found for compounds containing puckered cyclobutane rings. These values range from 145° in cyclobutane itself (Meiboom & Snyder, 1967) to 160° in chlorocyclobutane (Kim & Gwinn, 1966). Planar rings of cyclobutane have been found only in those molecules with a centrosymmetric arrangement of substituents, such as octahydroxycyclobutane (Bock, 1968) and *cis,trans,cis*-1,2,3,4-tetracyanocyclobutane (Greenberg & Post, 1968).

In the solid-state IR (KBr), the ketone carbonyl O(1) is shifted down to 1682.6 cm^{-1} from a normal ketone absorption of 1710 cm^{-1} , due to the hydrogen bonding to O(3) from the adjacent molecule. The solution IR spectrum (CHCl_3), in which this hydrogen bond is broken, shows the free ketone carbonyl absorbing at 1706 cm^{-1} . In the solid state, the acid carbonyl O(3) absorbs 20 cm^{-1} higher than in typical acid dimers and

* Lists of structure factors, anisotropic thermal parameters and hydrogen-bond distances and angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 43181 (13 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. Fractional atomic coordinates ($\times 10^4$) and isotropic thermal parameters

	x	y	z	$B_{eq}^*/B_{iso}(\text{Å}^2)$
O(1)	2526 (1)	4664 (1)	-2044 (1)	3.43 (<1)
O(2)	2295 (1)	271 (1)	2641 (1)	3.66 (1)
O(3)	3594 (1)	861 (1)	4826 (1)	3.91 (1)
C(1)	3588 (1)	1687 (1)	531 (2)	2.18 (<1)
C(2)	4475 (1)	2347 (1)	-519 (2)	2.04 (<1)
C(3)	3207 (1)	2691 (1)	-1649 (2)	2.09 (<1)
C(4)	2449 (1)	2442 (1)	-227 (2)	2.65 (1)
C(5)	5122 (2)	3404 (1)	351 (2)	2.66 (1)
C(6)	5430 (2)	1609 (2)	-1272 (2)	3.34 (1)
C(7)	3166 (1)	3858 (1)	-2448 (2)	2.19 (<1)
C(8)	3976 (2)	4020 (2)	-3786 (2)	3.28 (1)
C(9)	3975 (2)	1655 (1)	2352 (2)	2.75 (1)
C(10)	3182 (1)	853 (1)	3242 (2)	2.43 (<1)
H(C1)	3460 (26)	868 (24)	129 (35)	3.20
H(C3)	3027 (25)	2102 (24)	-2496 (33)	3.10
H(C4)	1624 (30)	2078 (26)	-548 (35)	3.68
H'(C4)	2336 (28)	3158 (26)	430 (35)	3.68
H(C5)	5793 (30)	3169 (25)	1202 (35)	3.64
H'(C5)	5564 (28)	3872 (26)	-399 (36)	3.64
H''(C5)	4504 (29)	3892 (26)	853 (34)	3.64
H(C6)	5850 (31)	2063 (28)	-2031 (38)	4.38
H'(C6)	4991 (31)	952 (30)	-1883 (39)	4.38
H''(C6)	6076 (30)	1335 (29)	-388 (39)	4.38
H(C8)	3594 (30)	4545 (29)	-4583 (39)	4.33
H'(C8)	4847 (32)	4267 (28)	-3348 (38)	4.33
H''(C8)	4113 (30)	3304 (29)	-4250 (37)	4.33
H(C9)	3944 (28)	2384 (27)	2801 (36)	3.75
H'(C9)	4899 (29)	1370 (27)	2649 (34)	3.75
H(O3)	3155 (33)	384 (31)	5315 (42)	4.93

$$* B_{eq} = \frac{8\pi^2}{3} (\beta_{11}a^{*2}a^2 + \beta_{12}a^*b^*abc\cos\gamma + \dots)$$

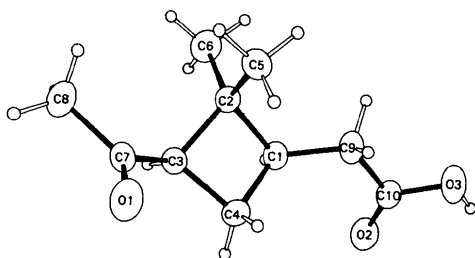


Fig. 1. View of the title compound showing the atom-numbering scheme.

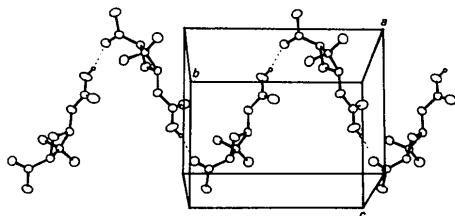


Fig. 2. Partial packing diagram showing the helical intermolecular hydrogen bonding. Only one half of the contents of the unit cell are displayed, for clarity.

Table 2. Bond distances (Å) and angles (°)

Parentheses contain e.s.d.'s in the least-significant digit.

C(2)—C(3)	1.576 (2)	C(7)—O(1)	1.218 (2)
C(1)—C(2)	1.556 (2)	C(7)—C(8)	1.498 (2)
C(2)—C(5)	1.526 (2)	C(9)—C(10)	1.502 (2)
C(2)—C(6)	1.513 (2)	C(10)—O(2)	1.201 (2)
C(3)—C(4)	1.537 (3)	C(10)—O(3)	1.329 (2)
C(3)—C(7)	1.494 (2)	O(1')—H(O3)	1.91 (2)
C(1)—C(4)	1.544 (2)	O(3)—H(O3)	0.85 (4)
C(1)—C(9)	1.513 (2)	O(3)—O(1')	2.700 (2)
C(1)—C(2)—C(3)	86.12 (9)	C(3)—C(7)—O(1)	122.9 (1)
C(3)—C(2)—C(5)	111.9 (1)	C(3)—C(7)—C(8)	117.1 (1)
C(3)—C(2)—C(6)	116.8 (1)	C(8)—C(7)—O(1)	120.0 (1)
C(1)—C(2)—C(5)	112.7 (1)	C(1)—C(9)—C(10)	114.4 (1)
C(1)—C(2)—C(6)	116.0 (1)	O(2)—C(10)—C(9)	126.1 (1)
C(2)—C(3)—C(4)	88.9 (1)	O(2)—C(10)—O(3)	123.2 (1)
C(2)—C(3)—C(7)	117.4 (1)	O(3)—C(10)—C(9)	110.7 (1)
C(4)—C(3)—C(7)	121.4 (1)	C(7)—O(1)—O(3')	152. (1)
C(1)—C(4)—C(3)	87.9 (1)	C(10)—O(3)—H(O3)	110. (2)
C(2)—C(1)—C(9)	118.0 (1)	C(10)—O(3)—O(1')	123.8 (1)
C(2)—C(1)—C(4)	89.4 (1)	O(3)—H(O3)—O(1')	159. (3)
C(4)—C(1)—C(9)	120.8 (1)		

The prime indicates that this atom is from an adjacent molecule.

30 cm^{-1} lower than in gas-phase acid monomers (Conley, 1966; Nakanishi & Solomon, 1977) because it is neither a dimer, nor a monomer; it is not hydrogen-bonded, yet the hydroxyl group to which it is directly attached is involved in hydrogen bonding.

cis-Pinonic acid is not intramolecularly hydrogen-bonded, but is intermolecularly hydrogen-bonded in a helix along the 2_1 screw axis, leaving the acid carbonyl free.

We thank the Center for Computer and Information Services, Rutgers, the State University of New Jersey, New Brunswick and Newark, for funding and assistance. We thank Mr Markian Jaworsky, Rutgers University, Newark, for providing assistance in obtaining the FT-IR results.

References

- ANDREWS, L. (1974). *FLINUS*. Brookhaven National Laboratory, Upton, NY.
- BOCK, C. M. (1968). *J. Am. Chem. Soc.* **90**, 2748–2751.
- BUSING, W. R., MARTIN, K. O. & LEVY, H. A. (1962). *ORFLS*. Report ORNL-TM-305. Oak Ridge National Laboratory, Tennessee.
- BUSING, W. R., MARTIN, K. O. & LEVY, H. A. (1964). *ORFFE*. Report ORNL-TM-306. Oak Ridge National Laboratory, Tennessee.
- CHADWICK, D. J. & DUNITZ, J. D. (1979). *J. Chem. Soc. Perkin Trans. 2*, pp. 276–284.
- CONLEY, R. T. (1966). *Infrared Spectroscopy*, p. 142. Boston, MA: Allyn & Bacon, Inc.
- GLICK, M. (1975). *SYNCOR*. Wayne State Univ., Detroit, MI. Personal communication.
- GREENBERG, B. & POST, B. (1968). *Acta Cryst.* **B24**, 918–924.
- GULA, M. J. & SPENCER, T. A. (1980). *J. Org. Chem.* **45**, 805–809.
- HOLLANDER, F. (1975). *ABSOR*. Brookhaven National Laboratory, Upton, NY.
- International Tables for X-ray Crystallography* (1974). Vol. IV, pp. 72–98. Birmingham: Kynoch Press. (Present distributor D. Reidel, Dordrecht.)

- JOHNSON, C. K. (1970). *ORTEP*. Report ORNL-3794, 2nd revision. Oak Ridge National Laboratory, Tennessee.
- KIM, H. & GWINN, W. D. (1966). *J. Chem. Phys.* **44**, 865–873.
- LALANCETTE, R. A., CEFOLA, M., HAMILTON, W. C. & LA PLACA, S. J. (1967). *Inorg. Chem.* **6**, 2127–2134.
- LEISEROWITZ, L. (1976). *Acta Cryst.* **B32**, 775–802.
- MAIN, P., FISKE, S. J., HULL, S. E., LESSINGER, L., GERMAIN, G., DECLERQ, J.-P. & WOOLFSON, M. M. (1980). *MULTAN80. A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data*. Univs. of York, England, and Louvain, Belgium.
- MEIBOOM, S. & SNYDER, L. C. (1967). *J. Am. Chem. Soc.* **89**, 1038–1039.
- NAKANISHI, K. (1962). *Infrared Absorption Spectroscopy—Practical*, p. 42. San Francisco: Holden-Day, Inc.
- NAKANISHI, K. & SOLOMON, R. (1977). *Infrared Absorption Spectroscopy*, 2nd ed., p. 39. San Francisco: Holden-Day, Inc.
- TEMPLETON, L. K. & TEMPLETON, D. H. (1973). *Am. Crystallogr. Assoc. Program Abstr.*, Ser. 2, Vol. 1, p. 143.
- VANDERHOFF, P. A., THOMPSON, H. W. & LALANCETTE, R. A. (1987). *Acta Cryst.* Submitted.
- ZALKIN, A. (1962). *FORDAP*. Univ. of California, Berkeley, CA.

Acta Cryst. (1986). **C42**, 1769–1771

The Structure of 9-Nitro-7-trifluoromethylpyrido[1,2-*a*]benzimidazole

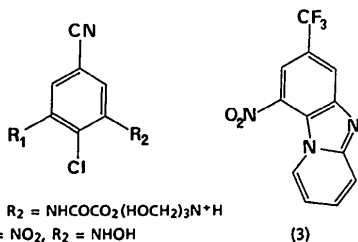
BY CONNIE G. CHIDESTER, DAVID J. ANDERSON AND WILLIAM WATT

Research Laboratories, The Upjohn Company, Kalamazoo, MI 49001, USA

(Received 6 December 1985; accepted 28 May 1986)

Abstract. $C_{12}H_6F_3N_3O_2$, $M_r = 281.2$, triclinic, $P\bar{1}$, $a = 4.994$ (1), $b = 10.713$ (1), $c = 11.172$ (2) Å, $\alpha = 114.31$ (1), $\beta = 92.14$ (2), $\gamma = 95.36$ (1)°, $V = 540.4$ (3) Å³, $Z = 2$, $D_x = 1.73$ g cm⁻³, $\lambda(\text{Cu } K\alpha) = 1.5418$ Å, $\mu(\text{Cu } K\alpha) = 1.2$ cm⁻¹, $F(000) = 284$, $T = 123$ K, $R = 0.072$ for 1765 unique reflections. The structure has an exceptionally large number of close contacts. The close packing prevents rotation of the trifluoromethyl group, hence bond lengths are more accurate than those usually reported for this group. The close contacts have apparently caused this flat molecule to adopt a bowed conformation.

Introduction. The dioxamate (1) (lodoxamide) has been undergoing development in our corporate laboratories as an anti-asthmatic agent. As part of a program to study the metabolism of lodoxamide, an authentic sample of the diaminophenol [derived from (1)] was required. During the chemical synthesis of this potential metabolite, it was found that when the nitrohydroxylamine (2) was treated with acetic anhydride–pyridine in an attempt to acetylate it, the major product was instead a rearrangement which incorporated pyridine (Anderson & Taylor, 1986).



We wished to determine the structure of this interesting rearrangement by X-ray analysis, and since crystals from this reaction were not very good, an analogous reaction was carried out on the nitrohydroxylamine with a trifluoromethyl group substituted for the cyano group. This reaction afforded crystals of the title compound (3), which were suitable for X-ray analysis.

Experimental. Clear prism $0.08 \times 0.13 \times 0.30$ mm, Nicolet $P\bar{1}$ diffractometer controlled by Harris computer, graphite monochromator, $\text{Cu } K\alpha$, $2\theta_{\text{max}} = 138^\circ$, $h \pm 4$, $k \pm 12$, $l \pm 13$, all 1765 unique reflections measured, 1509 intensities $> 2\sigma$, $2^\circ \text{ min}^{-1} \theta/2\theta$ step-scans, scan widths $> 3.4^\circ$, ten reflections periodically monitored showed no trend towards deterioration, $\sigma^2(I)$ was approximated by $\sigma^2(I)$ from counting statistics + $(0.021I)^2$, where the coefficient of I was calculated from the variations in intensities of the monitored reflections, cell parameters by least-squares fit of $\text{Cu } K\alpha_1$ 2θ values [$\lambda(\text{Cu } K\alpha_1) = 1.5402$ Å] for 29 high- 2θ reflections (Duchamp, 1977), L_p correction appropriate for a monochromator with 50% perfect character, no absorption correction. A partial trial solution (8 atoms), obtained by direct methods, using *DIREC* (Duchamp, 1978), was extended using successive Fourier syntheses; hydrogens were found in difference maps very close to positions generated using planar or tetrahedral geometry, generated positions were used. Least-squares refinement included coordinates and anisotropic thermal parameters for non-hydrogen atoms; hydrogen parameters were included in the calculations but were not refined. The function minimized in the refinement was $\sum w(F_o^2 - F_c^2)^2$, where weights w were $1/\sigma^2(F_o^2)$.