Acta Cryst. (1986). C**42**, 1273–1274

The Structure of 3,5,5-Trimethyl-3-pyrazoline *N,N'*-Dioxide*

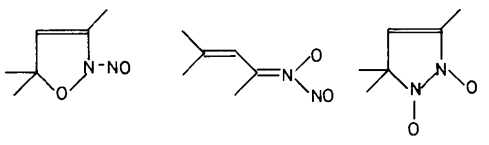
BY P. JØRGENSEN, R. KOKSBANG† AND P. LINDHARDT

Department of Inorganic Chemistry, Chemical Institute, University of Aarhus, DK-8000 Aarhus C, Denmark

(*Received* 10 *January* 1986; *accepted* 23 *May* 1986)

Abstract. $C_6H_{10}N_2O_2$, $M_r = 142{\cdot}16$, triclinic, $P\bar{1}$, $a = 6{\cdot}618$ (4), $b = 6{\cdot}320$ (3), $c = 8{\cdot}872$ (6) Å, $\alpha = 89{\cdot}86$ (5), $\beta = 95{\cdot}76$ (6), $\gamma = 90{\cdot}76$ (5)°, $V = 369{\cdot}2$ (4) Å^3, $Z = 2$, $D_x = 1{\cdot}279$ (1) Mg m^{-3}, λ(Cu $K\alpha$) = 1·5418 Å, $\mu = 0{\cdot}775$ mm^{-1}, $F(000) = 152$, room temperature, $R = 0{\cdot}043$ for 992 reflections. The molecules are nearly planar, and stacked on the *bc* plane. Greatest distance from a least-squares plane through the heavy atoms, except methyl carbons, is 0·007 Å for C(3), and 0·016 Å with C(4) included in the plane. There are no unusually short intermolecular contacts.

Experimental. Nitrosation of mesityl oxide oxime with a nitrite ester in acetic acid leads to a white solid, for which different structures (1), (2) and (3) have been proposed:



(1) (2) (3)

The structure (3) suggested on the basis of spectra and chemical reactions (Freeman, 1962) is confirmed in this work. The compound (3) is probably a result of ring formation of an intermediate with structure (2). Density not measured. Reflections from a monoclinic shaped crystal approximately 0·4 × 0·4 × 0·7 mm collected on a Picker FACS-1 diffractometer using Ni-filtered Cu $K\alpha$ radiation.

Cell dimensions calculated from setting angles of 20 reflections with $35 < 2\theta < 89°$. 2455 reflections with $2\theta \in [3°; 120°]$ measured between the limits: $-6 \le h \le 7$, $-7 \le k \le 7$ and $-9 \le l \le 9$. Two standard reflections ($1\bar{1}3$, $\bar{1}\bar{1}3$) varied ±4%, linear-drift correction applied. Intensities measured with a scan width of $(3{\cdot}0 + 0{\cdot}36\tan\theta)°$, step length 0·04° and a counting time of 1 s step^{-1}, using the ω–2θ scan technique. 26 reflections discarded due to obviously wrong background making 1087 independent reflections, of which 992 had $I > 3\sigma(I)$. No absorption corrections made. Structure solved by direct methods using *MULTAN*80 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980). The structure could be solved when scaling of the parity groups was omitted, but not when they were scaled separately. Use of $\Delta\rho$ maps together with geometrical calculations gave the positions of the H atoms. Least-squares refinement with anisotropic temperature factors for non-hydrogen atoms carried out with the program *LINEX* [a 1971 version of *ORFLS* (Busing, Martin & Levy 1971)]. Scattering factors those of Cromer & Mann (1968) for C, N and O, and of Stewart, Davidson & Simpson (1965) for H. Refinement on $|F|$ including extinction gave a final R value of 0·043 ($wR = 0{\cdot}047$ with unit weights), $g = 0{\cdot}0053$, $S = 1{\cdot}8008$ and $(\Delta/\sigma)_{max} = 0{\cdot}242$. The final $\Delta\rho$ gave $(\Delta\rho)_{max} = 0{\cdot}13$ and $(\Delta\rho)_{min} = -0{\cdot}20$ e Å^{-3}. Position and thermal parameters are given in Table 1,‡ bond lengths and angles in Table 2. Carbon–hydrogen bond distances are between 0·87 (3) and 1·10 (3) Å. An *ORTEP* (Johnson, 1965) drawing of the molecule is shown in Fig. 1, and a stereographic drawing of the unit cell in Fig. 2.

Related literature. The N—O and N—N bond distances are not different from those observed in other compounds with unstrained *N,N'*-dioxide groups (Prout, Stothard & Watkin, 1978).

‡ Lists of structure factors, anisotropic thermal parameters, H-atom parameters and least-squares-planes data have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 43095 (8 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. *Fractional coordinates* (×10^4) *and thermal parameters* (×10^4)

$U_{eq} = \frac{1}{3}(U_{11} + U_{22} + U_{33})$.

	x	y	z	U_{eq}(Å^2)
C(1)	7447 (4)	−2493 (4)	10044 (3)	471 (14)
C(2)	7708 (4)	−3722 (4)	11235 (3)	537 (16)
C(3)	8048 (4)	−2465 (4)	12659 (3)	507 (15)
C(4)	7104 (6)	−2845 (5)	8395 (4)	647 (21)
C(5)	10178 (5)	−2708 (5)	13460 (4)	632 (19)
C(6)	6406 (6)	−2784 (6)	13727 (5)	749 (24)
N(1)	7892 (3)	−276 (3)	12035 (2)	458 (12)
N(2)	7547 (3)	−324 (3)	10554 (2)	444 (12)
O(1)	8055 (3)	1428 (2)	12785 (2)	607 (12)
O(2)	7374 (3)	1317 (2)	9748 (2)	603 (12)

* Part of this work has been presented at the 18th Danish Crystallographic Meeting 1982.

† Present address: Energy Research Laboratory, Niels Bohrs Allé 25, DK-5230 Odense M, Denmark.

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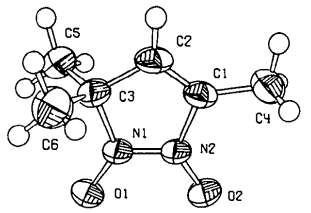

Fig. 1. View of the molecule showing atomic numbering and thermal ellipsoids scaled to indicate 50% probability. Hydrogen atoms are shown as spheres of arbitrary size.

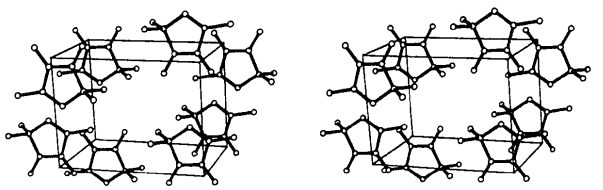

Fig. 2. Stereoscopic view of the unit cell. The *a* axis is pointing inwards, the *b* axis upwards and the *c* axis from left to right.

We wish to thank Professor K. Torssell of this Institute for suggesting the problem and providing the crystal, and R. G. Hazell and A. C. Hazell of this Institute for crystallographic advice.

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

C(2)–C(1)	1·308 (4)	N(1)–C(3)	1·490 (3)
C(3)–C(2)	1·491 (4)	N(2)–C(1)	1·443 (3)
C(4)–C(1)	1·475 (4)	N(2)–N(1)	1·311 (3)
C(5)–C(3)	1·523 (4)	O(1)–N(1)	1·265 (2)
C(6)–C(3)	1·523 (4)	O(2)–N(2)	1·258 (2)
C(4)–C(1)–C(2)	134·9 (2)	C(5)–C(3)–N(1)	107·7 (2)
C(4)–C(1)–N(2)	116·9 (2)	C(6)–C(3)–N(1)	108·5 (2)
C(2)–C(1)–N(2)	108·2 (2)	C(3)–N(1)–N(2)	110·5 (2)
C(1)–C(2)–C(3)	111·4 (2)	C(3)–N(1)–O(1)	126·6 (2)
C(2)–C(3)–C(5)	112·9 (2)	N(2)–N(1)–O(1)	122·9 (2)
C(2)–C(3)–C(6)	114·0 (3)	C(1)–N(2)–N(1)	109·5 (2)
C(2)–C(3)–N(1)	100·4 (2)	C(1)–N(2)–O(2)	127·3 (2)
C(5)–C(3)–C(6)	112·4 (3)	N(1)–N(2)–O(2)	123·2 (2)

References

BUSING, W. R., MARTIN, K. O. & LEVY, H. A. (1962). *ORFLS*. Report ORNL-TM-305. Oak Ridge National Laboratory, Tennessee.

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

FREEMAN, J. P. (1962). *J. Org. Chem.* **27**, 1309–1314.

JOHNSON, C. K. (1965). *ORTEP*. Report 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). *MULTAN*80. *A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data*. Univs. of York, England, and Louvain, Belgium.

PROUT, K., STOTHARD, V. P. & WATKIN, D. J. (1978). *Acta Cryst.* B**34**, 2602–2605.

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

SHORT COMMUNICATION

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Acta Cryst. (1986). C**42**, 1274

X-ray structure analysis of 1,4-dioxane, phase I at 279 K and phase II at 153 K: erratum.

By JÜRGEN BUSCHMANN, EVELINE MÜLLER and PETER LUGER, *Institut für Kristallographie, Fachbereich Chemie, Freie Universität Berlin, Takustrasse* 6, *D*-1000 *Berlin* 33, *Federal Republic of Germany*

(*Received* 7 *July* 1986)

Abstract

In the *Abstract* of the paper by Buschmann, Müller & Luger [*Acta Cryst.* (1986), C**42**, 873–876], incorrect values for the linear absorption coefficients are given. The correct values are $\mu = 0{\cdot}899$ cm^{-1} for I and $\mu = 0{\cdot}962$ cm^{-1} for II.

All relevant information is given in the *Abstract*.

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