$V = 1155.1\ (1)$ Å^3
$Z = 4$

0.30 × 0.15 × 0.15 mm
Colourless

Data collection

Enraf-Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: none
2196 measured reflections
2021 independent reflections
588 observed reflections $[I_{\text{net}} > 2.0\sigma(I_{\text{net}})]$

$R_{\text{int}} = 0.01$
$\theta_{\max} = 25.0°$
$h = -7 \rightarrow 7$
$k = 0 \rightarrow 11$
$l = 0 \rightarrow 20$
3 standard reflections
frequency: 60 min
intensity variation: 33% decay during data collection

Refinement

Refinement on F
$R = 0.050$
$wR = 0.042$
$S = 1.30$
588 reflections
119 parameters
H atoms refined as riding, C—H 0.95 Å
$w = 1/[\sigma^2(F) + 0.0004F^2]$
$(\Delta/\sigma)_{\max} < 0.001$

$\Delta\rho_{\max} = 0.15$ e Å^{-3}
$\Delta\rho_{\min} = -0.17$ e Å^{-3}
Extinction correction: Larson (1970)
Extinction coefficient: 9798 (1598)
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV, Table 2.2B)

Table 1. *Fractional atomic coordinates and equivalent isotropic thermal parameters* (Å^2)

$$U_{\text{eq}} = \tfrac{1}{3}\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	U_{eq}
N1	0.1872 (8)	0.5964 (6)	0.1899 (3)	0.094 (4)
N9	0.1592 (9)	0.2151 (7)	0.0299 (4)	0.111 (5)
C2	0.0185 (12)	0.5934 (7)	0.1958 (3)	0.061 (5)
C3	−0.2004 (9)	0.5938 (5)	0.2030 (3)	0.051 (4)
C31	−0.2694 (9)	0.7393 (5)	0.2181 (3)	0.087 (4)
C32	−0.2564 (9)	0.5017 (5)	0.2673 (3)	0.078 (4)
C4	−0.2957 (8)	0.5463 (5)	0.1283 (3)	0.059 (4)
O5	−0.2236 (5)	0.4149 (3)	0.1124 (2)	0.058 (2)
C6	−0.3034 (9)	0.3607 (5)	0.0441 (3)	0.060 (4)
C7	−0.2303 (9)	0.2171 (5)	0.0347 (3)	0.047 (4)
C71	−0.3152 (9)	0.1600 (5)	−0.03982 (24)	0.077 (4)
C72	−0.2950 (9)	0.1295 (5)	0.1009 (3)	0.077 (4)
C8	−0.0100 (12)	0.2177 (6)	0.0320 (4)	0.066 (5)

The crystal decomposed by 33% during data collection and this was allowed for in the data reduction by using a five-point smoothing correction in the scaling of the data. The H atoms, visible in the difference maps at an intermediate stage of refinement, were positioned geometrically and included as riding atoms in the structure-factor calculations. Data collection: *CAD-4 Software* (Enraf-Nonius, 1989). Cell refinement: *CAD-4 Software*. Data reduction: *NRCVAX DATRD2* (Gabe, Le Page, Charland, Lee & White, 1989). Program(s) used to solve structure: *NRCVAX SOLVER*. Program(s) used to refine structure: *NRCVAX LSTSQ*. Molecular graphics: *NRCVAX*; *ORTEP*II (Johnson, 1976). Software used to prepare material for publication: *NRCVAX TABLES*.

GF thanks NSERC, Canada, for research grants.

Lists of structure factors, anisotropic thermal parameters, H-atom coordinates, torsion angles and complete geometry have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71475 (8 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: MU1064]

References

Enraf-Nonius (1989). *CAD-4 Software*. Version 5.0. Enraf-Nonius, Delft, The Netherlands.

Ferguson, G., McAlees, A. J., McCrindle, R., Restivo, R. J. & Roberts, P. J. (1977). *J. Am. Chem. Soc.* pp. 3170-3171.

Gabe, E. J., Le Page, Y., Charland, J.-P., Lee, F. L. & White, P. S. (1989). *J. Appl. Cryst.* **22**, 384-387.

Johnson, C. K. (1976). *ORTEP*II. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.

Larson, A. C. (1970). *Crystallographic Computing*, edited by F. R. Ahmed, S. R. Hall & C. P. Huber, p. 293. Copenhagen: Munksgaard.

McAlees, A. J. & McCrindle, R. (1981). *J. Chem. Soc. Perkin Trans.* 1, pp. 741-745.

McCrindle, R., Ferguson, G., McAlees, A. J., Parvez, M., Ruhl, B., Stephenson, D. K. & Wieckowski, T. (1986). *J. Chem. Soc. Dalton Trans.* pp. 2351-2359.

McCrindle, R., Ferguson, G., McAlees, A. J., Parvez, M. & Stephenson, D. K. (1982). *J. Chem. Soc. Dalton Trans.* pp. 1291-1296.

McCrindle, R., Ferguson, G., McAlees, A. J., Parvez, M. & Stephenson, D. K. (1983). *J. Chem. Soc. Dalton Trans.* pp. 1865-1868.

McCrindle, R., Ferguson, G., McAlees, A. J. & Roberts, P. J. (1981). *J. Chem. Soc. Dalton Trans.* pp. 1406-1414.

McCrindle, R., Ferguson, G. & Parvez, M. (1984). *Acta Cryst.* C**40**, 354-356.

Acta Cryst. (1994). C**50**, 78-81

Structures of 4-Hydroxyimino-4-phenylbutanoic Acid, $C_{10}H_{11}NO_3$ (I), and 5-Hydroxyimino-5-phenylpentanoic Acid, $C_{11}H_{13}NO_3$ (II), at 223 K

JAN K. MAURIN

Institute of Atomic Energy, 05-400 Otwock-Świerk, Poland

IAIN C. PAUL AND DAVID Y. CURTIN

Department of Chemistry, University of Illinois, Urbana, Illinois 61801, USA

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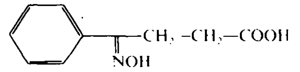
Abstract

Infinite polar chains of molecules with strong intermolecular hydrogen bonds (O—H···N and O—H···O) are observed between carboxyl and oxime

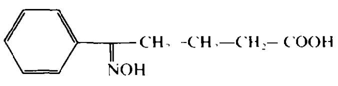
groups [N···O 2.697 (1) and O···O 2.784 (1) Å for (I), and 2.690 (2) and 2.744 (2) Å for (II), respectively]. There are no carboxyl–carboxyl or oxime–oxime interactions.

Comment

The crystal structures of (I) and (II) were studied as part of a program to examine the packing characteristics of compounds containing the carboxyl and oxime groups (Maurin, Paul & Curtin, 1992*a*,*b*; Padmanabhan, Paul & Curtin, 1989). The final atomic parameters are given in Table 1 and Table 2 contains the bond lengths and angles involving the non-H atoms. Figs. 1 and 2 show the structures of molecules (I) and (II), respectively, and the numbering of atoms, while Figs. 3 and 4 illustrate their crystal packing, with the inclusion of only those H atoms involved in hydrogen bonding.



(I)



(II)

In both structures, there are two strong intermolecular hydrogen bonds between the carboxyl and oxime groups (marked as thin lines on Figs. 3 and 4). As found in the crystals of *p*-acetylbenzoic acid oxime (Maurin, Paul & Curtin, 1992*a*) and 3-benzoylbenzoic acid oxime (Maurin, Paul & Curtin, 1992*b*) the molecules form two anti-parallel polar chains. The O—H···N hydrogen bond is much shorter than those observed for several oximes [2.853 (3), 2.784 (Bachechi & Zambonelli, 1973, 1972), 2.83 (Brehm & Watson, 1972), 2.81 (1) (Gieren, Huebner & Ruiz-Perez, 1986), 2.776 (5) (Grattan & Whitehouse, 1990), 2.830 (2) Å (Hooft, van der Sluis, Kanters & Kroon, 1990)], while the O—H···O bond length is somewhat longer than those observed for carboxylic acid dimers [2.623 (6) Å (*e.g.* Patil, Curtin & Paul, 1985)]. No close contacts other than the hydrogen bonds described above were observed.

The ease with which both hydroxyl H atoms were found from the difference Fourier maps and the lack of any peaks in the neighbourhood of either the N1 or O2 atoms is strong evidence against any H exchange or other type of disorder. No carboxyl–carboxyl or oxime–oxime interactions, typical of those found in molecules containing either functional group alone, were observed.

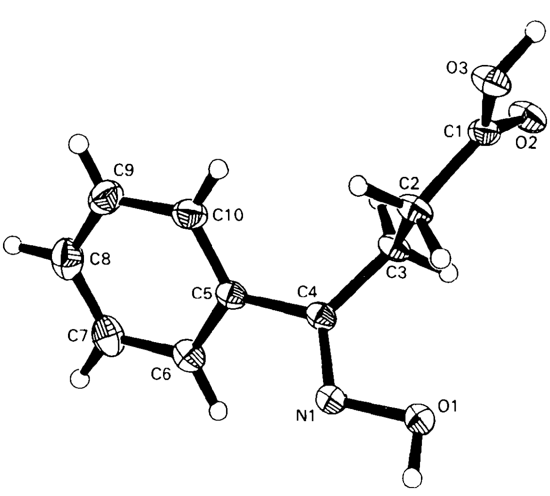


Fig. 1. Molecular geometry of (I) and atomic numbering.

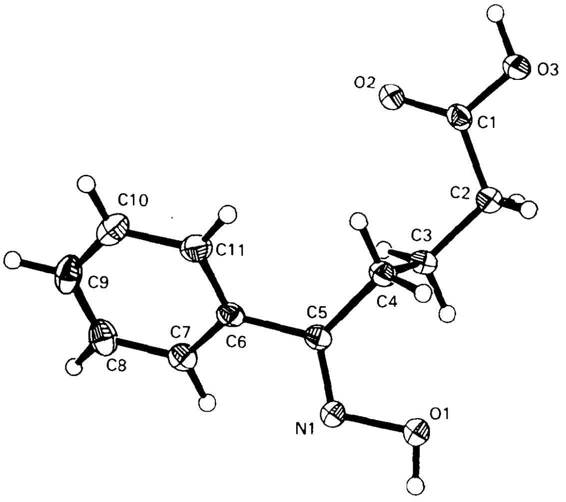


Fig. 2. Molecular geometry of (II) and atomic numbering.

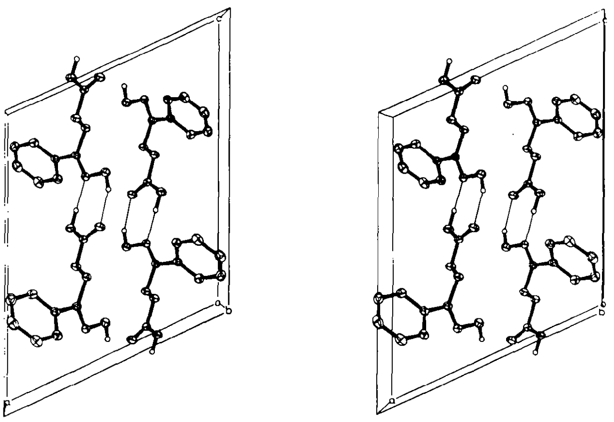


Fig. 3. Stereoview of the cell contents of (I) along the *b* axis. Only H atoms involved in hydrogen-bond formation are shown.

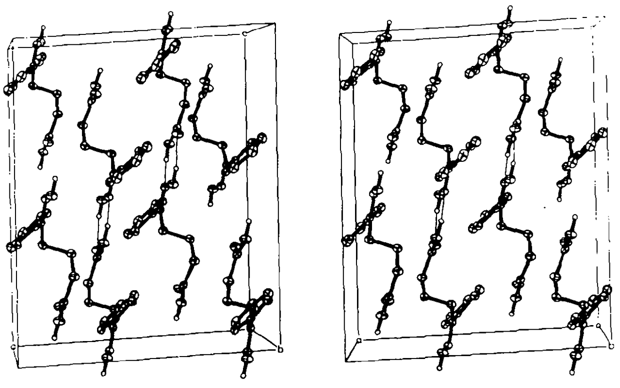


Fig. 4. Stereoview of the cell contents of (II) along the *b* axis.

Experimental

Compound (I)

Crystal data

$C_{10}H_{11}NO_3$
$M_r = 193.20$
Monoclinic
$P2_1/c$
$a = 12.242$ (3) Å
$b = 5.869$ (2) Å
$c = 14.812$ (2) Å
$\beta = 114.38$ (1)°
$V = 969.32$ Å^3
$Z = 4$

$D_x = 1.324$ Mg m^{-3}
Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 24 reflections
$\theta = 8$-14°
$\mu = 0.092$ mm^{-1}
$T = 223$ (1) K
Prismatic
$0.42 \times 0.25 \times 0.1$ mm
Colourless

Data collection

Enraf-Nonius CAD-4 diffractometer
ω-θ scans
Absorption correction: none
3274 measured reflections
2825 independent reflections
1978 observed reflections [$F > 3.92\sigma(F)$]

$R_{\rm int} = 0.0145$
$\theta_{\rm max} = 30$°
$h = 0 \rightarrow 17$
$k = -8 \rightarrow 0$
$l = -20 \rightarrow 20$
3 standard reflections monitored every 200 reflections
intensity variation: none

Refinement

Refinement on F
$R = 0.040$
$wR = 0.041$
$S = 1.471$
1978 reflections
171 parameters
All H-atom parameters refined
$w = 2.013/[\sigma^2(F) + 0.00035(F)^2]$

$(\Delta/\sigma)_{\rm max} = 0.002$
$\Delta\rho_{\rm max} = 0.24$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.18$ e Å^{-3}
Extinction correction: none
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

Table 1. *Fractional atomic coordinates and equivalent isotropic thermal parameters* (Å^2) *for compound* (I)

$$U_{\rm eq} = \tfrac{1}{3}\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	$U_{\rm eq}$
C1	0.3477 (1)	0.2954 (2)	0.54291 (8)	0.0270 (6)
C2	0.3127 (1)	0.2418 (3)	0.6267 (1)	0.0321 (7)
C3	0.3587 (1)	0.0091 (2)	0.6719 (1)	0.0298 (7)
C4	0.3164 (1)	−0.0582 (2)	0.75032 (8)	0.0258 (6)
C5	0.2225 (1)	−0.2343 (2)	0.73020 (9)	0.0278 (6)
C6	0.2320 (1)	−0.3950 (3)	0.8025 (1)	0.0328 (6)
C7	0.1436 (2)	−0.5573 (3)	0.7839 (1)	0.0430 (9)
C8	0.0442 (2)	−0.5600 (3)	0.6948 (1)	0.0468 (9)
C9	0.0341 (1)	−0.4032 (3)	0.6226 (1)	0.0423 (9)
C10	0.1233 (1)	−0.2423 (3)	0.6389 (1)	0.0355 (7)
N1	0.3577 (1)	0.0268 (2)	0.83798 (7)	0.0276 (5)
O1	0.44852 (8)	0.1870 (2)	0.85498 (7)	0.0348 (5)
O2	0.42099 (8)	0.1855 (2)	0.52540 (7)	0.0373 (6)
O3	0.28983 (9)	0.4729 (2)	0.48961 (7)	0.0348 (5)
HO1	0.460 (1)	0.238 (3)	0.915 (1)	0.072 (6)
HO3	0.315 (2)	0.489 (4)	0.437 (1)	0.053 (5)

Table 2. *Geometric parameters* (Å, °) *for compound* (I)

C1—C2	1.503 (2)	C8—C9	1.376 (2)
C2—C3	1.523 (2)	C5—C10	1.396 (2)
C3—C4	1.506 (2)	C9—C10	1.388 (2)
C4—C5	1.482 (2)	C1—O2	1.217 (2)
C5—C6	1.396 (2)	C1—O3	1.323 (2)
C6—C7	1.382 (2)	C4—N1	1.284 (1)
C7—C8	1.378 (2)	O1—N1	1.398 (1)
C2—C1—O2	123.5 (1)	C4—C5—C10	120.7 (1)
C2—C1—O3	113.1 (1)	C6—C5—C10	118.9 (1)
O2—C1—O3	123.4 (1)	C5—C6—C7	120.2 (1)
C1—C2—C3	111.8 (1)	C6—C7—C8	120.5 (2)
C2—C3—C4	113.1 (1)	C7—C8—C9	119.8 (2)
C3—C4—C5	120.8 (1)	C8—C9—C10	120.5 (1)
C3—C4—N1	124.1 (1)	C5—C10—C9	119.9 (1)
C5—C4—N1	115.0 (1)	C4—N1—O1	113.3 (1)
C4—C5—C6	120.3 (1)		

Compound (II)

Crystal data

$C_{11}H_{13}NO_3$
$M_r = 207.23$
Monoclinic
$C2/c$
$a = 13.700$ (6) Å
$b = 9.111$ (1) Å
$c = 16.779$ (5) Å
$\beta = 92.95$°
$V = 2091.59$ Å^3
$Z = 8$

$D_x = 1.316$ Mg m^{-3}
Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 10$-13.7°
$\mu = 0.090$ mm^{-1}
$T = 223$ (1) K
Columnar
$0.55 \times 0.26 \times 0.23$ mm
Colourless

Data collection

Enraf-Nonius CAD-4 diffractometer
ω-θ scans
Absorption correction: none
2811 measured reflections
2512 independent reflections
1613 observed reflections [$F > 3.92\sigma(F)$]

$R_{\rm int} = 0.0177$
$\theta_{\rm max} = 28$°
$h = -18 \rightarrow 18$
$k = -12 \rightarrow 0$
$l = -22 \rightarrow 0$
3 standard reflections monitored every 200 reflections
intensity variation: none

Refinement

Refinement on F
$R = 0.041$
$wR = 0.042$
$S = 1.225$
1613 reflections
188 parameters
All H-atom parameters refined
$w = 1.155/[\sigma^2(F) + 0.0006(F)^2]$
$(\Delta/\sigma)_{\max} = 0.02$
$\Delta\rho_{\max} = 0.21$ e Å^{-3}
$\Delta\rho_{\min} = -0.21$ e Å^{-3}
Extinction correction: none
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

Table 3. *Fractional atomic coordinates and equivalent isotropic thermal parameters* (Å^2) *for compound* (II)

$$U_{\rm eq} = \tfrac{1}{3}\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	$U_{\rm eq}$
N1	0.8810 (1)	0.0794 (2)	1.00709 (9)	0.0295 (8)
O1	0.8616 (1)	−0.0704 (1)	1.01849 (8)	0.0372 (8)
O2	0.8365 (1)	0.0300 (1)	0.67821 (7)	0.0396 (8)
O3	0.8495 (1)	−0.2073 (1)	0.64794 (8)	0.0366 (8)
C1	0.8279 (1)	−0.0985 (2)	0.6960 (1)	0.029 (1)
C2	0.7895 (2)	−0.1507 (2)	0.7730 (1)	0.036 (1)
C3	0.7941 (1)	−0.0359 (2)	0.8393 (1)	0.032 (1)
C4	0.8985 (1)	−0.0021 (2)	0.8696 (1)	0.032 (1)
C5	0.8997 (1)	0.1110 (2)	0.9348 (1)	0.0273 (9)
C6	0.9185 (1)	0.2682 (2)	0.9185 (1)	0.0276 (9)
C7	0.8671 (1)	0.3782 (2)	0.9557 (1)	0.032 (1)
C8	0.8871 (2)	0.5244 (2)	0.9417 (1)	0.041 (1)
C9	0.9592 (2)	0.5621 (2)	0.8910 (1)	0.044 (1)
C10	1.0103 (2)	0.4548 (2)	0.8534 (1)	0.042 (1)
C11	0.9897 (2)	0.3082 (2)	0.8660 (1)	0.036 (1)
HO3	0.868 (2)	−0.168 (3)	0.598 (2)	0.072 (8)
HO1	0.848 (2)	−0.070 (2)	1.070 (2)	0.057 (7)

Table 4. *Geometric parameters* (Å, °) *for compound* (II)

O1—N1	1.405 (2)	C5—C6	1.484 (2)
O2—C1	1.215 (2)	C6—C7	1.391 (3)
O3—C1	1.321 (2)	C7—C8	1.382 (3)
C1—C2	1.498 (3)	C8—C9	1.380 (3)
C2—C3	1.525 (3)	C9—C10	1.375 (3)
C3—C4	1.525 (3)	C6—C11	1.396 (3)
N1—C5	1.285 (2)	C10—C11	1.383 (3)
C4—C5	1.503 (3)		
O1—N1—C5	113.2 (1)	C4—C5—C6	121.5 (1)
O2—C1—O3	123.1 (2)	C5—C6—C7	121.1 (2)
O2—C1—C2	124.1 (2)	C5—C6—C11	120.2 (2)
O3—C1—C2	112.8 (2)	C7—C6—C11	118.8 (2)
C1—C2—C3	114.1 (2)	C6—C7—C8	120.6 (2)
C2—C3—C4	112.6 (2)	C7—C8—C9	120.0 (2)
C3—C4—C5	110.9 (2)	C8—C9—C10	120.2 (2)
N1—C5—C4	122.6 (2)	C9—C10—C11	120.3 (2)
N1—C5—C6	115.9 (2)	C6—C11—C10	120.2 (2)

(I) was obtained by the reaction of 3-benzoylpropionic acid with hydroxylamine hydrochloride in alkaline solution and purified by recrystallization. (II) was obtained by the reaction of 4-benzoylbutyric acid with hydroxylamine hydrochloride in alkaline solution and also purified by recrystallization. Crystals of both (I) and (II) were grown from ethanol/water solution by slow evaporation. Melting points were found to be 400.5-401.5 and 379-380.2 K for (I) and (II), respectively.

Both structures were solved by use of *SHELXS*86 (Sheldrick, 1990) and refined by application of *SHELX*76 (Sheldrick, 1976). Most of the H atoms were positioned according to geometric criteria but the two hydroxyl H atoms were located from a difference Fourier map.

Lists of structure factors, anisotropic thermal parameters, H-atom coordinates and complete geometry have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71394 (21 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: AB1067]

References

Bachechi, F. & Zambonelli, L. (1972). *Acta Cryst.* **B28**, 2489–2494.
Bachechi, F. & Zambonelli, L. (1973). *Acta Cryst.* **B29**, 2598–2600.
Brehm, L. & Watson, K. J. (1972). *Acta Cryst.* **B28**, 3646–3652.
Gieren, A., Huebner, T. & Ruiz-Perez, C. (1986). *Chem. Ztg.* **110**, 73–78.
Gratten, T. J. & Whitehouse, J. S. (1990). *J. Chem. Soc. Perkin Trans.* 1, pp. 11–18.
Hooft, R. W. W., van der Sluis, P., Kanters, J. A. & Kroon, J. (1990). *Acta Cryst.* **C46**, 1133–1135.
Maurin, J. K., Paul, I. C. & Curtin, D. Y. (1992*a*). *Acta Cryst.* **C48**, 2163–2165.
Maurin, J. K., Paul, I. C. & Curtin, D. Y. (1992*b*). *Acta Cryst.* **C48**, 2165–2167.
Padmanabhan, K., Paul, I. C. & Curtin, D. Y. (1989). *Acta Cryst.* **B45**, 411–416.
Patil, A., Curtin, D. Y. & Paul, I. C. (1985). *Isr. J. Chem.* **25**, 320–325.
Sheldrick, G. M. (1976). *SHELX76. Program for Crystal Structure Determination.* Univ. of Cambridge, England.
Sheldrick, G. M. (1990). *Acta Cryst.* **A46**, 467–473.

Acta Cryst. (1994). **C50**, 81–83

1-Chloro-1,2,2-triphenylethene, $C_{20}H_{15}Cl$

EBERHARDT HERDTWECK, WOLFGANG ZICHE
AND NORBERT AUNER

Anorganisch-chemisches Institut der Technischen Universität München, Lichtenbergstrasse 4, D-85748 Garching, Germany

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Abstract

All phenyl rings are twisted out of the plane of the ethene framework. The molecular geometry shows no significant difference to that expected.

Comment

The title compound was synthesized by a published method (Crenshaw & Zimmer, 1983) as part of our efforts to obtain vinyl-chlorosilanes (Ziche, Auner & Behm, 1992). It has also found interest as a nonsteroidal anti-

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