Crystal data

$C_6H_7NO.C_8H_8O_2$
$M_r = 245.27$
Monoclinic
$P2_1/c$
$a = 13.6819\,(9)$ Å
$b = 7.2379\,(3)$ Å
$c = 14.0407\,(7)$ Å
$\beta = 111.00\,(1)°$
$V = 1298.11\,(12)$ Å^3
$Z = 4$
$D_x = 1.255$ Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 9.57–18.16°$
$\mu = 0.089$ mm^{-1}
$T = 295$ K
Transparent prism
0.18 × 0.12 × 0.10 mm
Colourless

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: none
2740 measured reflections
2629 independent reflections
1844 reflections with $I > 2\sigma(I)$

$R_{\rm int} = 0.015$
$\theta_{\rm max} = 26.29°$
$h = -17 \rightarrow 0$
$k = 0 \rightarrow 9$
$l = -16 \rightarrow 17$
3 standard reflections
frequency: 120 min
intensity decay: 1.13%

Refinement

Refinement on F^2
$R(F) = 0.046$
$wR(F^2) = 0.132$
$S = 1.067$
1844 reflections
169 parameters
H-atom parameters not refined, except for HO2
$w = 1/[\sigma^2(F_o^2) + (0.0813P)^2 + 0.2843P]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\rm max} = 0.005$
$\Delta\rho_{\rm max} = 0.197$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.244$ e Å^{-3}
Extinction correction: *SHELXL*93 (Sheldrick, 1993)
Extinction coefficient: 0.015 (3)
Scattering factors from *International Tables for X-ray Crystallography* (Vol. IV)

Table 1. *Selected geometric parameters (Å, °)*

C1—N1	1.346 (2)	C7—O2	1.316 (3)
C2—C6	1.494 (3)	C7—C8	1.486 (3)
N1—O1	1.328 (2)	C11—C14	1.512 (3)
C7—O3	1.206 (2)	O2—H2	0.90 (3)
C3—C2—C6	122.5 (2)	C9—C8—C7	122.2 (2)
C1—C2—C6	119.6 (2)	C13—C8—C7	119.2 (2)
O1—N1—C5	118.8 (2)	C12—C11—C14	121.2 (2)
O3—C7—C8	123.7 (2)	C10—C11—C14	121.1 (2)
O2—C7—C8	113.6 (2)		

The ring H and methyl H atoms were included at geometrically idealized positions. The H2 atom was located from a Fourier difference map and its coordinates were refined. An isotropic displacement parameter of 6.0 Å^2 was assigned to all H atoms.

Data collection: *CAD*-4 *Software* (Enraf–Nonius, 1989). Cell refinement: *CAD*-4 *Software*. Data reduction: *VAXSDP* (Frenz, 1978). Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1985). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *ORTEP*II. Software used to prepare material for publication: *VAXSDP*.

One of the authors (RMF) wants to thank the Brazilian Agency FAPESP for a Postdoctoral Research Fellowship, and FINEP and Universidad del Valle (Colombia) for partial financial support.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: FG1302). Services for accessing these data are described at the back of the journal.

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Acta Cryst. (1997). C**53**, 1635–1637

The 1:1 Complex Formed by 2-Picoline *N*-Oxide and 3-Chlorobenzoic Acid

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Abstract

The title co-crystal, $C_6H_7NO.C_7H_5ClO_2$, belongs to a series of molecular complexes based on the N—O group and was studied in order to establish its possible non-linear optical behaviour. The 2-picoline *N*-oxide and 3-chlorobenzoic acid moieties are held together by an intermolecular hydrogen bond between the N—O and O—H groups. This system shows a transparency in the visible region of spectra.

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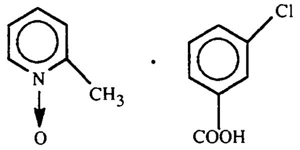
Comment

Organic molecular compounds are being recognized as excellent systems to maximize non-linear responses (Prasad & Williams, 1991). We are exploring the possibilities of obtaining different organic molecular systems with possible non-linear optical properties. The present structure determination is part of a series studying molecular complexes based on interactions of the N—O group with different hydrogen-bond donors (Moreno-Fuquen, De Almeida Santos & Lechat, 1996) and was obtained to establish its possible non-linear optical behaviour. The 3-chlorobenzoic acid used in this work as a hydrogen-bond donor has a centre of symmetry in its crystal structure and is a dimer (Gougoutas & Lessinger, 1975). To remove this centre of symmetry from the crystal, the formation of the 2-picoline *N*-oxide (OPNO) and 3-chlorobenzoic acid (MCBA) molecular complex has been undertaken.



OPNO MCBA

A perspective view of the OPNO.MCBA molecular complex showing the atomic numbering scheme is given in Fig. 1. The complex is held together by an intermolecular hydrogen bond between the O2 atom of MCBA and the O3 atom of the N—O group of OPNO with an O2···O3 distance equal to 2.528 (3) Å. There are no other intermolecular hydrogen bonds and the distances of the Cl atom to other atoms are greater than 3.0 Å. The N—O bond length of 1.322 (3) Å in this structure is very close to the value of 1.324 (2) Å found in the adduct formed by the OPNO and 4-nitrophenol molecules (Moreno-Fuquen, Gambardella & De Almeida Santos, 1996). Due to the formation of the intermolecular hydrogen bond in the dimer, MCBA shows a C7—O1 distance of 1.222 (6) Å. This distance changes to 1.206 (4) Å when the intermolecular hydrogen bond of the dimer breaks forming the molecular complex. The Cl—C1 bond length and other internal parameters of the MCBA dimer are comparable with those reported previously (Moreno-Fuquen, Gambardella & De Almeida Santos, 1996). The dihedral angle between the mean planes formed by the rings of the OPNO and MCBA molecules is 50.30 (8)°. Physicochemical and crystallographic characteristics, crystal growth and transparency in the visible region of spectra make this system an excellent candidate for the study of non-linear optical behaviour.

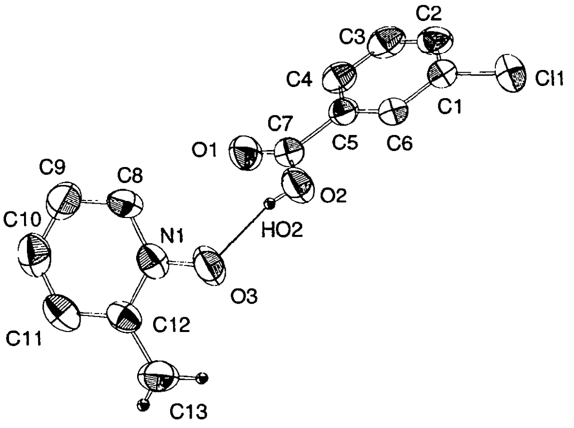


Fig. 1. A perspective view of the title molecular complex with the atomic numbering scheme. The displacement ellipsoids are plotted at the 50% probability level. Some H atoms are omitted for clarity; others are shown as small spheres.

Experimental

Good single crystals suitable for X-ray analysis were obtained by slow evaporation from an equimolecular solution of 2-picoline *N*-oxide and 3-chlorobenzoic acid in acetonitrile. Colourless prisms of good quality were obtained with a melting point of 335 (2) K.

Crystal data

$C_6H_7NO.C_7H_5ClO_2$
M_r = 265.69
Monoclinic
Pn
a = 3.9241 (3) Å
b = 13.0404 (8) Å
c = 12.4181 (5) Å
β = 92.21 (1)°
V = 634.98 (7) Å^3
Z = 2
D_x = 1.390 Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
λ = 0.71073 Å
Cell parameters from 25 reflections
θ = 10.92–18.49°
μ = 0.30 mm^{-1}
T = 295 K
Transparent prism
0.15 × 0.10 × 0.10 mm
Colourless

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: none
1471 measured reflections
1471 independent reflections
1352 reflections with $I > 2\sigma(I)$

$\theta_{\max}$ = 26.28°
$h = 0 \rightarrow 4$
$k = 0 \rightarrow 16$
$l = -15 \rightarrow 15$
3 standard reflections
frequency: 120 min
intensity decay: 0.74%

Refinement

Refinement on F^2
$R(F)$ = 0.029
$wR(F^2)$ = 0.106
S = 1.006
1471 reflections
167 parameters

$\Delta\rho_{\max}$ = 0.184 e Å^{-3}
$\Delta\rho_{\min}$ = −0.115 e Å^{-3}
Extinction correction: *SHELXL*93
Extinction coefficient: 0.058 (12)

H atoms: see below
$w = 1/[\sigma^2(F_o^2) + (0.1P)^2]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$

Scattering factors from *International Tables for X-ray Crystallography* (Vol. IV)

Table 1. *Selected geometric parameters (Å, °)*

Cl1—C1	1.743 (3)	C7—O2	1.313 (4)
C1—C6	1.376 (4)	O3—N1	1.322 (3)
C1—C2	1.382 (5)	N1—C8	1.350 (4)
C2—C3	1.386 (6)	N1—C12	1.357 (3)
C3—C4	1.383 (5)	C8—C9	1.358 (5)
C4—C5	1.392 (4)	C9—C10	1.374 (6)
C5—C6	1.396 (4)	C10—C11	1.382 (6)
C5—C7	1.485 (4)	C11—C12	1.387 (5)
C7—O1	1.206 (4)	C12—C13	1.492 (5)
C6—C1—C2	121.9 (3)	O2—C7—C5	112.9 (2)
C6—C1—Cl1	118.0 (2)	O3—N1—C8	120.2 (3)
C2—C1—Cl1	120.1 (2)	O3—N1—C12	118.1 (2)
C1—C2—C3	118.5 (3)	C8—N1—C12	121.6 (3)
C4—C3—C2	120.8 (3)	N1—C8—C9	121.2 (3)
C3—C4—C5	120.0 (3)	C8—C9—C10	119.4 (3)
C6—C5—C4	119.5 (3)	C11—C10—C9	118.9 (3)
C6—C5—C7	121.0 (2)	C10—C11—C12	121.3 (3)
C4—C5—C7	119.5 (2)	N1—C12—C11	117.6 (3)
C1—C6—C5	119.3 (2)	N1—C12—C13	117.6 (3)
O1—C7—O2	123.9 (3)	C11—C12—C13	124.8 (3)
O1—C7—C5	123.1 (3)		

The ring and methyl H atoms were included at geometrically idealized positions. The HO2 atom was located from a difference map and its coordinates were refined. An isotropic displacement parameter of 6.0 Å^2 was assigned to all H atoms.

Data collection: *CAD*-4 *Software* (Enraf–Nonius, 1989). Cell refinement: *CAD*-4 *Software*. Data reduction: *VAXSDP* (Frenz, 1978). Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1985). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *ORTEP*II (Johnson, 1976). Software used to prepare material for publication: *VAXSDP*.

The authors want to thank FINEP, CNPq and FAPESP (Brazilian agencies), and Universidad del Valle (Colombia) for financial support.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: BS1020). Services for accessing these data are described at the back of the journal.

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Steric Inhibition of Molecular Weaving: Non-Woven Nets of $R_6^6(40)$ and $R_6^6(60)$ Rings in 1,1,3-Tris(2-methyl-4-hydroxy-5-*tert*-butylphenyl)butane–Hexamethylenetetramine (1/1)

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Abstract

In the 1:1 adduct formed by 1,1,3-tris(2-methyl-4-hydroxy-5-*tert*-butylphenyl)butane and hexamethylenetetramine (HMTA; alternative name: 1,3,5,7-tetraazatricyclo[3.3.1.1^{3,7}]decane), $C_{37}H_{52}O_3.C_6H_{12}N_4$, the tris-phenol [alternative name: 4,4′,4″-(butane-1,1,3-triyl)-tris(2-*tert*-butyl-5-methylphenol)] acts as a triple donor and a single acceptor of hydrogen bonds, while the HMTA acts as a double acceptor. The tris-phenol and HMTA units are linked into $C_2^2(16)$ chains along [101] by means of O—H···N hydrogen bonds, with O···N distances of 2.759 (4) and 2.828 (4) Å; the tris-phenol units are themselves linked into *C*(14) chains along [010] by O—H···O hydrogen bonds, each having an O···O distance of 2.828 (4) Å. These two types of chain intersect to generate two-dimensional nets built from alternating $R_6^6(40)$ and $R_6^6(60)$ rings; despite the size of these rings, interweaving of the nets is prevented by the presence of the *tert*-butyl groups in the tris-phenol.

Comment

The successful construction of microporous molecular solids requires the production of two- or three-dimensional supramolecular networks containing continuous voids with dimensions that exceed those of small molecules, but the interweaving of such networks is a common occurrence. For example, in 4,4′-sulfonyldiphenol, $O_2S(C_6H_4OH)_2$ (Glidewell & Ferguson, 1996), there is pairwise interweaving of square nets