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## Nonstereospecific 1,3-Dipolar Cycloaddition Reactions of Azomethine Ylides: Proof of the Structures for Four Diastereoisomeric [3+2] Cycloadducts

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### Abstract

Racemic azomethine ylides [(I) or (IV)] have been found to undergo cycloaddition to a *trans*-enamine [(II)] in a nonstereospecific way to yield mixtures of racemic diastereoisomeric 1:1 adducts: 5-dimethylamino-1,1,6-trimethyl-2,6a-di-*p*-tolyl-1,1a,5,6,6a,6b-hexahydro-4*H*-3,3a-diazacyclopropa[*c*]indene-4,4-dicarbonitrile, C<sub>29</sub>H<sub>33</sub>N<sub>5</sub> [(IIIa) and (IIIb)], or 2-dimethylamino-1,5,5-trimethyl-6-(4-methylbenzoyl)-7a-*p*-tolyl-2,3,5,7a-tetrahydro-1*H*-pyrrolizine-3,3-dicarbonitrile (pyrrolizine is pyrrolo[1,2-*a*]pyrrole), C<sub>29</sub>H<sub>32</sub>N<sub>4</sub>O [(Va) and (Vb)]. Crystal structure analyses prove the structures and configurations for the three diastereoisomeric adducts (IIIa), (IIIb) and (Vb) unequivocally. In all three cases, the dimethylamino group is *endo* to the five-membered ring formed in the cycloaddition step. In (*cis*-1a-*cis*-4a-*transoid*-8a,8b-*transoid*-8b,8c)-1,1-dimethyl-4a-(pyrrolidin-1-yl)-2,8b-di-*p*-tolyl-1,1a,4a,5,6,7,8,8a,8b,8c-

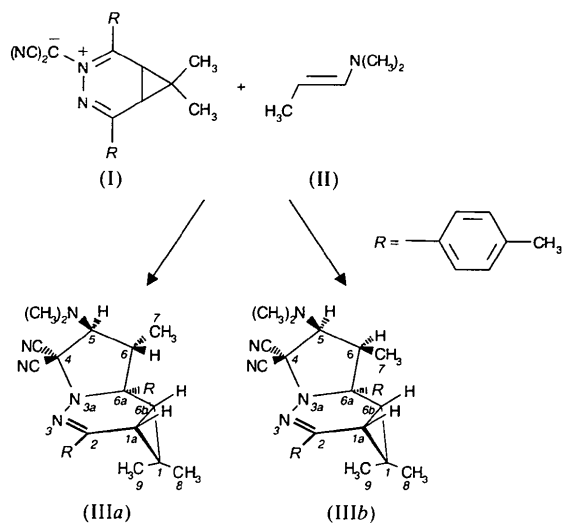
decahydro-4*H*-3,3a-diazacyclopropa[*c*]fluorene-4,4-dicarbonitrile, C<sub>34</sub>H<sub>39</sub>N<sub>5</sub>, (VII), the cycloadduct formed using an azomethine ylide [(I)] and a cyclic enamine [(VI)], the amino function is again found in an *endo* position.

### Comment

1,3-Dipolar cycloadditions (Huisgen, 1984) are 'symmetry-allowed' reactions (Woodward & Hoffmann, 1969) and occur in a stereospecific manner as suprafacial-suprafacial additions. Only recently have a few exceptions been reported for nonstereospecific 1,3-cycloaddition reactions (Huisgen, 1988). Thiocarbonyl ylides, as electron-rich 1,3-dipoles, react with 2,3-dicyanofumarate (an electron-poor dipolarophile) in a nonstereospecific way, *via* a dipolar intermediate.

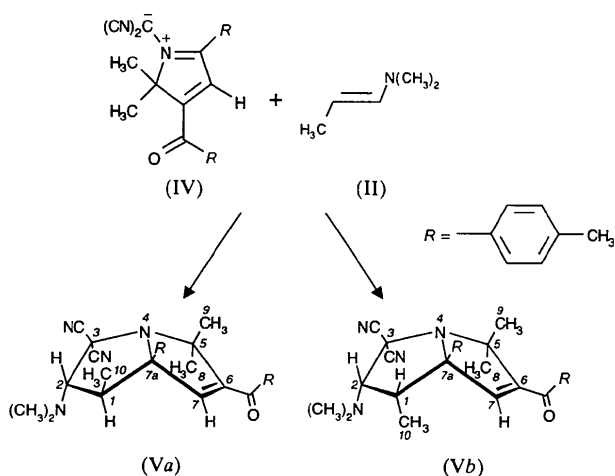
Recently, we reported the synthesis of two new classes of coloured stable azomethine ylides [(I) and (IV)], which are electron-poor 1,3-dipoles (Riebel, Weber, Troll, Sauer & Breu, 1996; Riebel, Weber, Troll, Sauer, Breu & Nöth, 1996; Breu *et al.*, 1996). These azomethine ylides preferentially combine with electron-rich dipolarophiles.

In the following, we describe [3+2] cycloaddition reactions of (I) and (IV) with the *trans*-enamine (II), all of which do not obey the Woodward–Hoffmann rules but occur in a nonstereospecific way yielding mixtures of diastereomeric adducts. Since the starting dipoles were racemates, all products were, of course, also isolated as racemates.



When the azomethine ylide (I) was combined with 2.1 equivalents of the enamine (II) at ambient temperature in acetonitrile, the addition occurred immediately with rapid decolourization to yield a mixture of 1:1 adducts in almost quantitative yield (Weber, 1997; Weber & Sauer, 1998). Flash column chromatography separated

38% *trans* adduct (**IIIa**) (m.p. 425–426 K) and 42% *cis* adduct (**IIIb**) (m.p. 415–426 K) as analytically pure compounds. It was impossible to elucidate the structures and configurations of these adducts by NMR techniques alone because coupling constants of protons at five-membered rings do not distinguish between *cis* and *trans* isomers. Usually, because of the great conformational flexibility of cyclopentane derivatives, the dihedral angles for the coupling protons cannot be predicted.



In a similar way, a roughly 1:1 mixture of the diastereoisomeric 1,3-dipolar cycloadducts (**Va**) (m.p. 481–482 K) and (**Vb**) (m.p. 488–489 K) was obtained in a rapid reaction when the azomethine ylide (**IV**) was combined with 1.2 equivalents of the enamine (**II**) in acetonitrile at room temperature. Separation of (**Va**) and (**Vb**) was again achieved by flash column chromatography. As in the case of (**IIIa**) and (**IIIb**), NMR techniques alone were not able to elucidate the configurations of the diastereoisomers (**Va**) and (**Vb**).

Crystal structure analyses of (**IIIa**), (**IIIb**) and (**Vb**) finally allowed unequivocal proof of the structures and configurations of these three diastereoisomers. The *trans* adduct (**IIIa**) shows a dihedral angle of 178.3° for H5–H6 (see scheme above for numbering) and, according to the Karplus equation (Hesse *et al.*, 1995), it has a larger coupling constant compared with the *cis* isomer (**IIIb**), with a dihedral angle of 44.3° for H5–H6 ( $^3J_{\text{trans}}$ : calculated 9.21, found 12.4 Hz;  $^3J_{\text{cis}}$ : calculated 4.07, found 4.7 Hz).

The six-membered heterocycle (C11–C16) (see Figs. 1 and 2 for numbering) is planar in both (**IIIa**) and (**IIIb**), with maximum deviations from the least-squares planes of 0.040 (1) Å for C16 in (**IIIa**) and –0.041 (3) Å for C14 in (**IIIb**). The most appropriate puckering description for the pyrrolidine ring (N13–C14) is ‘twisted about C43–C42’ for (**IIIa**) and ‘envelope on C42’ for (**IIIb**). The Cremer & Pople (1975) puckering param-

eters are  $Q = 0.435$  (1) Å,  $\Phi = 270.1$  (2)° for (**IIIa**) and  $Q = 0.444$  (3) Å,  $\Phi = 283.0$  (4)° for (**IIIb**). The dihedral angle between the cyclopropane and the six-membered ring is 65.3 (1)° in (**IIIa**) and 68.1 (2)° in (**IIIb**), and that between the pyrrolidine and the six-membered ring is 42.3 (7)° in (**IIIa**) and 43.0 (1)° in (**IIIb**).

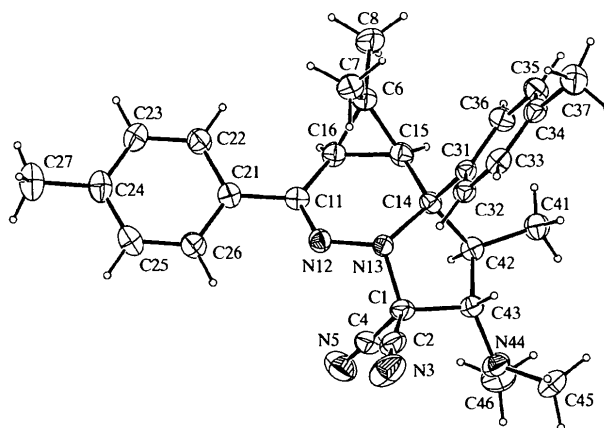


Fig. 1. The molecular structure of compound (**IIIa**) showing the labelling of the non-H atoms. Displacement ellipsoids are shown at 50% probability levels; H atoms are drawn as small circles of arbitrary radii.

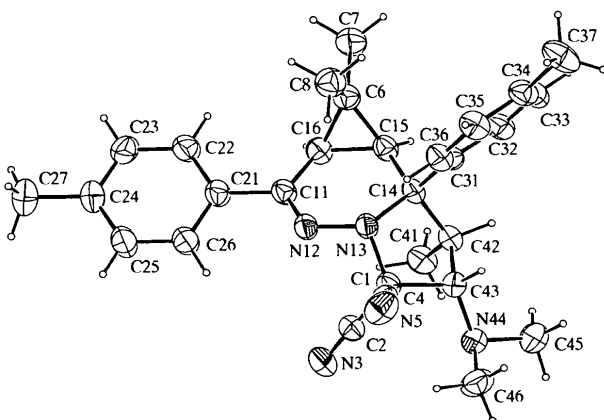


Fig. 2. The molecular structure of compound (**IIIb**) showing the labelling of the non-H atoms. Displacement ellipsoids are shown at 50% probability levels; H atoms are drawn as small circles of arbitrary radii.

Cycloadduct (**Vb**), the product of the addition of the enamine (**II**) to the azomethine ylide (**IV**), shows, according to crystal structure analysis, a *cis* arrangement for H1 and H2 (see scheme above for numbering); the dihedral angle of 44.4° is in accord with the coupling constant  $^3J_{\text{cis}} = 5.4$  Hz (4.06 Hz calculated according to the Karplus equation). Consequently, the second stereoisomer (**Va**), showing  $^3J = 12.3$  Hz for H1–H2, must be the cycloadduct with the *trans* configuration.

The dihedral angle between the five-membered rings in (Vb) is  $33.98(9)^\circ$ . While the N1–C5 ring (see Fig. 3 for numbering) is planar with a maximum deviation from the least-squares plane of  $0.086(1) \text{ \AA}$  for N1, the N1–C8 ring is twisted about C6–C7 [ $Q = 0.445(2) \text{ \AA}$ ,  $\Phi = 267.1(2)^\circ$ ].

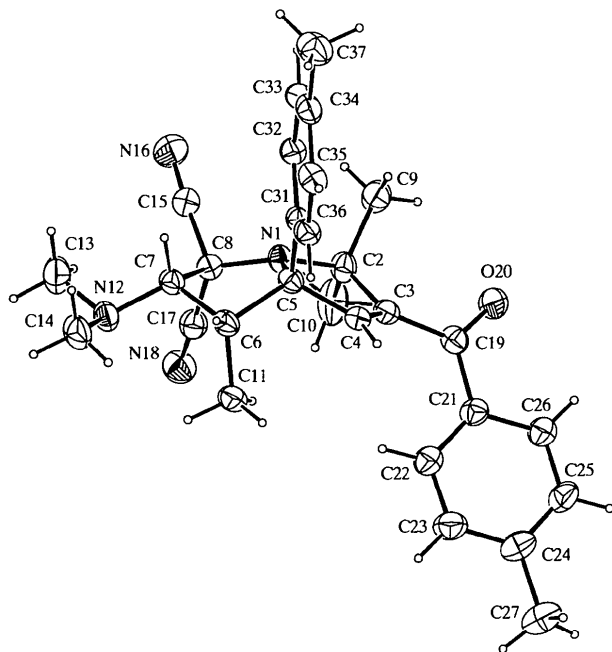
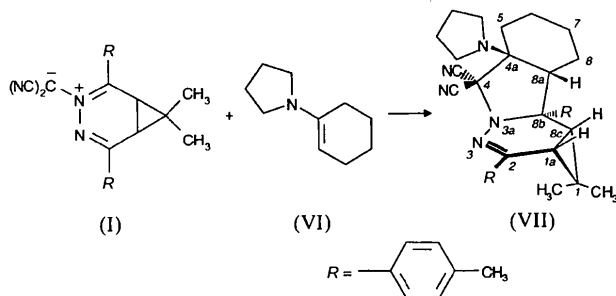


Fig. 3. The molecular structure of compound (Vb) showing the labelling of the non-H atoms. Displacement ellipsoids are shown at 50% probability levels; H atoms are drawn as small circles of arbitrary radii.

A comparison of the configurations of all the diastereoisomeric cycloadducts, (IIIa) and (IIIb), or (Va) and (Vb), reveals that the dimethylamino group of the dienophile (II) adds to the dipoles exclusively in an *endo* fashion. This is confirmed for the combination of the enamine (VI) with the azomethine ylide (I). Crystal structure analysis proves the *endo* configuration for the pyrrolidin-1-yl substituent in the cycloadduct (VII). (VII) was obtained from acetonitrile as solvent at room temperature [m.p. 448 K (dec.)] in 56% yield and in analytically pure form.



In the tetracyclic adduct (VII), the six-membered heterocycle (N1, N4, C7, C8, C9, C1) (numbering as in Fig. 4) deviates significantly from planarity, with a maximum deviation from the least-squares plane of  $-0.128(2) \text{ \AA}$  for N1. The most appropriate puckering description for the N1–C2 ring is an 'envelope on C3' [ $Q = 0.375(2) \text{ \AA}$ ,  $\Phi = 291.6(3)^\circ$ ]. The dihedral angle between this five-membered heterocycle and the six-membered heterocycle is  $49.04(8)^\circ$  and that between the cyclopropane and the six-membered heterocycle is  $62.35(14)^\circ$ .

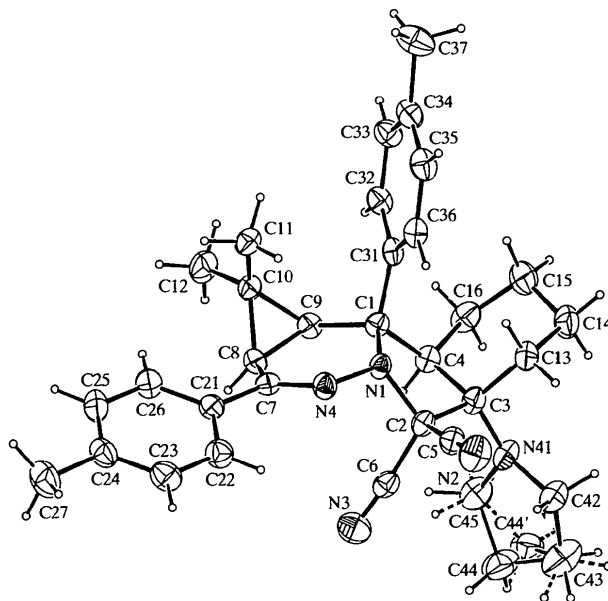


Fig. 4. The molecular structure of compound (VII) showing the labelling of the non-H atoms. Displacement ellipsoids are shown at 50% probability levels; H atoms are drawn as small circles of arbitrary radii. The minor disorder component is indicated by broken bonds.

In all reported cycloadducts, (IIIa), (IIIb), (Vb) and (VII), the bond lengths and angles are normal and therefore will not be discussed.

The *trans*-enamine (II) is the thermodynamically more stable enamine isomer; a *trans*–*cis* isomerization prior to the cycloaddition step can be excluded. Therefore, both 1,3-dipolar cycloadditions of the azomethine ylides (I) and (IV) with the *trans*-enamine (II) occurred in a nonstereospecific way, possibly *via* a zwitterionic intermediate.

## Experimental

Compounds (IIIa), (IIIb), (Vb) and (VII) were prepared as described and illustrated in the *Comment*.

### Compound (IIIa)

#### Crystal data

$C_{29}H_{33}N_5$   
 $M_r = 451.61$

Mo  $K\alpha$  radiation  
 $\lambda = 0.71069 \text{ \AA}$

**Monoclinic**

$P2_1/c$   
 $a = 14.3132 (10) \text{ \AA}$   
 $b = 10.5769 (15) \text{ \AA}$   
 $c = 17.9455 (13) \text{ \AA}$   
 $\beta = 107.351 (6)^\circ$   
 $V = 2593.1 (5) \text{ \AA}^3$   
 $Z = 4$   
 $D_x = 1.157 \text{ Mg m}^{-3}$   
 $D_m$  not measured

**Data collection**

Stoe IPDS diffractometer  
 Rotation scans  
 Absorption correction: none  
 8101 measured reflections  
 4113 independent reflections  
 3114 reflections with  
 $I > 2\sigma(I)$

**Refinement**

Refinement on  $F^2$   
 $R(F) = 0.034$   
 $wR(F^2) = 0.087$   
 $S = 1.191$   
 4113 reflections  
 314 parameters  
 H atoms constrained  
 $w = 1/[\sigma^2(F_o^2) + (0.0554P)^2]$   
 where  $P = (F_o^2 + 2F_c^2)/3$

**Compound (IIIb)****Crystal data**

C<sub>29</sub>H<sub>33</sub>N<sub>5</sub>  
 $M_r = 451.61$   
 Monoclinic  
 $P2_1/c$   
 $a = 16.246 (3) \text{ \AA}$   
 $b = 10.8936 (8) \text{ \AA}$   
 $c = 20.173 (3) \text{ \AA}$   
 $\beta = 134.282 (9)^\circ$   
 $V = 2555.9 (7) \text{ \AA}^3$   
 $Z = 4$   
 $D_x = 1.174 \text{ Mg m}^{-3}$   
 $D_m$  not measured

**Data collection**

Stoe IPDS diffractometer  
 Rotation scans  
 Absorption correction: none  
 6868 measured reflections  
 3167 independent reflections  
 2311 reflections with  
 $I > 2\sigma(I)$

**Refinement**

Refinement on  $F^2$   
 $R(F) = 0.048$   
 $wR(F^2) = 0.125$   
 $S = 1.07$

**Cell parameters from 2000 reflections**

$\theta = 2.26\text{--}24.64^\circ$   
 $\mu = 0.07 \text{ mm}^{-1}$   
 $T = 170.0 \text{ K}$   
 Monoclinic sphenoid  
 $0.58 \times 0.43 \times 0.30 \text{ mm}$   
 Transparent

$R_{\text{int}} = 0.022$   
 $\theta_{\text{max}} = 24.64^\circ$   
 $h = -16 \rightarrow 15$   
 $k = -10 \rightarrow 12$   
 $l = -20 \rightarrow 19$

$(\Delta/\sigma)_{\text{max}} < 0.001$   
 $\Delta\rho_{\text{max}} = 0.155 \text{ e \AA}^{-3}$   
 $\Delta\rho_{\text{min}} = -0.127 \text{ e \AA}^{-3}$   
 Extinction correction: none  
 Scattering factors from  
*International Tables for Crystallography* (Vol. C)

3167 reflections  
 314 parameters  
 H atoms constrained  
 $w = 1/[\sigma^2(F_o^2) + (0.0944P)^2]$   
 where  $P = (F_o^2 + 2F_c^2)/3$

**Compound (Vb)****Crystal data**

C<sub>29</sub>H<sub>32</sub>N<sub>4</sub>O  
 $M_r = 452.60$   
 Monoclinic  
 $P2_1/c$   
 $a = 12.156 (2) \text{ \AA}$   
 $b = 14.5370 (11) \text{ \AA}$   
 $c = 14.4990 (14) \text{ \AA}$   
 $\beta = 100.352 (11)^\circ$   
 $V = 2520.4 (5) \text{ \AA}^3$   
 $Z = 4$   
 $D_x = 1.193 \text{ Mg m}^{-3}$   
 $D_m$  not measured

**Data collection**

Stoe IPDS diffractometer  
 Rotation scans  
 Absorption correction: none  
 7756 measured reflections  
 3968 independent reflections  
 2961 reflections with  
 $I > 2\sigma(I)$

**Refinement**

Refinement on  $F^2$   
 $R(F) = 0.039$   
 $wR(F^2) = 0.102$   
 $S = 1.159$   
 3968 reflections  
 314 parameters  
 H atoms constrained  
 $w = 1/[\sigma^2(F_o^2) + (0.0689P)^2]$   
 where  $P = (F_o^2 + 2F_c^2)/3$

**Compound (VII)****Crystal data**

C<sub>34</sub>H<sub>39</sub>N<sub>5</sub>  
 $M_r = 517.72$   
 Monoclinic  
 $P2_1/n$   
 $a = 9.2212 (4) \text{ \AA}$   
 $b = 18.0246 (10) \text{ \AA}$   
 $c = 17.4099 (8) \text{ \AA}$   
 $\beta = 101.016 (5)^\circ$   
 $V = 2840.4 (2) \text{ \AA}^3$   
 $Z = 4$   
 $D_x = 1.211 \text{ Mg m}^{-3}$   
 $D_m$  not measured

**Data collection**

Stoe IPDS diffractometer  
 Rotation scans

Scattering factors from  
*International Tables for Crystallography* (Vol. C)

Mo  $K\alpha$  radiation  
 $\lambda = 0.71069 \text{ \AA}$   
 Cell parameters from 2000 reflections  
 $\theta = 2.00\text{--}24.64^\circ$   
 $\mu = 0.07 \text{ mm}^{-1}$   
 $T = 150 \text{ K}$   
 Lath  
 $0.34 \times 0.30 \times 0.15 \text{ mm}$   
 Transparent

$R_{\text{int}} = 0.024$   
 $\theta_{\text{max}} = 24.64^\circ$   
 $h = -10 \rightarrow 14$   
 $k = -16 \rightarrow 16$   
 $l = -15 \rightarrow 16$

$(\Delta/\sigma)_{\text{max}} = 0.001$   
 $\Delta\rho_{\text{max}} = 0.184 \text{ e \AA}^{-3}$   
 $\Delta\rho_{\text{min}} = -0.142 \text{ e \AA}^{-3}$   
 Extinction correction: none  
 Scattering factors from  
*International Tables for Crystallography* (Vol. C)

Mo  $K\alpha$  radiation  
 $\lambda = 0.71069 \text{ \AA}$   
 Cell parameters from 4615 reflections  
 $\theta = 2.38\text{--}24.75^\circ$   
 $\mu = 0.07 \text{ mm}^{-1}$   
 $T = 170 \text{ K}$   
 Lath  
 $0.29 \times 0.28 \times 0.09 \text{ mm}$   
 Transparent

$R_{\text{int}} = 0.043$   
 $\theta_{\text{max}} = 24.81^\circ$

Absorption correction: none  $h = -10 \rightarrow 7$   
 9439 measured reflections  $k = -21 \rightarrow 16$   
 4535 independent reflections  $l = -20 \rightarrow 20$   
 2319 reflections with  
 $I > 2\sigma(I)$

#### Refinement

Refinement on  $F^2$   $(\Delta/\sigma)_{\max} = -0.001$   
 $R(F) = 0.034$   $\Delta\rho_{\max} = 0.139 \text{ e } \text{\AA}^{-3}$   
 $wR(F^2) = 0.050$   $\Delta\rho_{\min} = -0.111 \text{ e } \text{\AA}^{-3}$   
 $S = 0.950$  Extinction correction: none  
 4535 reflections Scattering factors from  
 366 parameters *International Tables for*  
*Crystallography* (Vol. C)  
 H atoms constrained  
 $w = 1/[\sigma^2(F_o^2) + (0.0141P)^2]$   
 where  $P = (F_o^2 + 2F_c^2)/3$

Data were collected applying an imaging-plate system with the following measurement parameters yielding data completeness of typically 0.9: detector distance 75–80 mm; irradiation/exposure 12–25 min;  $0 < \varphi < 94^\circ$ . Methyl H atoms were placed in idealized positions based on difference electron synthesis and torsion angles were refined with fixed isotropic displacement parameters of  $1.5U_{\text{eq}}$ (parent C). All other H atoms were placed in idealized positions and refined with fixed isotropic displacement parameters of  $1.2U_{\text{eq}}$ (parent C). Atom C44 of compound (VII) is disordered and was refined using a split model.

For all compounds, program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1993); program(s) used to refine structure: *SHELXL93* (Sheldrick, 1993); molecular graphics: *PLATON* (Spek, 1990); software used to prepare material for publication: *PLATON*.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: JZ1241). Services for accessing these data are described at the back of the journal.

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## Pyridine *N*-Oxide and 4-Nitrophenol (1:1 Complex)

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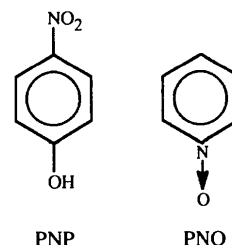
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#### Abstract

In the title hydrogen-bonded complex,  $\text{C}_5\text{H}_5\text{NO} \cdot \text{C}_6\text{H}_5\text{NO}_3$ , the two components are linked by an  $\text{O}—\text{H} \cdots \text{O}$  hydrogen bond between the phenol hydroxyl group and the *N*-oxide O atom [ $\text{O} \cdots \text{O}$  2.553(2) Å]. The interplanar angle between the aromatic rings in the two components is 41.29(6)°.

#### Comment

The present work is part of a structural study of molecular complexes of *N*-oxide systems with hydrogen-bond donors and we report here on the structure of the 1:1 complex between pyridine *N*-oxide (PNO) and 4-nitrophenol (PNP). A similar series of molecular complexes formed from 4-nitropyridine *N*-oxide and diverse hydrogen-bond donors, which observes the possibility of application in non-linear optics, has been reported previously (Moreno-Fuquen *et al.*, 1996, and references therein).



The crystallographic data of the free PNO molecule (Ülkü *et al.*, 1971) can be used as a comparison for the parameters obtained for the title complex. A *ZORTEP* (Zsolnai, 1995) view of the hydrogen-bonded PNO.PNP complex and its numbering scheme are shown in Fig. 1. The molecular complex owes its formation to a hydrogen bond between the O4 atom of the *N*-oxide group of PNO and the O1 atom of the  $\text{O}—\text{H}$  group of PNP; the dimensions are  $\text{O1} \cdots \text{O4}$  2.553(2),  $\text{O1}—$