& Trueblood, 1965). The preferred mode γ in pyrimidine nucleoside crystal structures is *gauche–gauche* (Saenger, 1984) and in accord with this $\gamma = 50{\cdot}4\ (4)°$.

The pyrimidine ring is approximately planar, with the greatest deviation from the six-atom least-squares plane being 0·05 Å. The molecular packing is characterized by herringbone stacking of the uridine bases with an interplanar spacing of 3·1 Å. There are three hydrogen bonds and they are listed with their corresponding symmetry operations in Table 3. The O(5′) donates a hydrogen bond to the deoxyribose ether oxygen and the pyrimidine H(6) is also situated in an ideal geometry to donate a hydrogen bond to O(5′) although C(6)–H(6) is not a classical hydrogen-bond donor. The C(6)–O(5′) contact distance is 3·28 Å and the C–H···O angle is 172°.

Both bromine atoms exhibit large U_{eq}'s, presumably due to the inability of the applied absorption correction adequately to account for the high absorption coefficient of bromine. The Br–C bond distance is 1·904 Å, giving a bromine covalent radius of 1·1 Å and a bromine van der Waals radius of 1·8 Å. Indeed, the closest Br–Br contact observed has a contact distance of 3·62 Å, twice the calculated van der Waals radius for bromine. In addition, note the 2·89 Å contact between Br(92) and O(4) which is 0·3 Å shorter than the sum of the van der Waals radii of bromine and oxygen.

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Structure of Droperidol–Ethanol (1/1)

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Abstract. 1-{1-[4-(4-Fluorophenyl)-4-oxobutyl]-1,2,-3,6-tetrahydro-4-pyridyl}-1,3-dihydro-2*H*-benzimidazol-2-one ethanol solvate, $C_{22}H_{22}FN_3O_2.C_2H_6O$, $M_r = 426{\cdot}3$, triclinic, $P\bar{1}$, $a = 6{\cdot}083\ (3)$, $b = 10{\cdot}296\ (1)$, $c = 16{\cdot}018\ (2)$ Å, $\alpha = 100{\cdot}93\ (1)$, $\beta = 92{\cdot}72\ (2)$, $\gamma = 96{\cdot}27\ (2)°$, $V = 976{\cdot}7$ Å^3, $Z = 2$, $D_x = 1{\cdot}45$ g cm^{-3}, Mo $K\bar{\alpha}$, $\lambda = 0{\cdot}71073$ Å, $\mu = 0{\cdot}97$ cm^{-1}, $F(000) = 452$, $T = 90\ (2)$ K, final $R = 0{\cdot}046$ for 2261

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observed reflections. The title compound crystallizes with the droperidol skeleton extended and a solvated ethanol molecule disordered about an inversion center. The ethanol molecule forms a hydrogen bond to the droperidol skeleton at ketone atom O(1) on the imidazole ring.

Introduction. Droperidol is a potent neuroleptic which is used as a tranquilizer and to reduce the incidence of nausea and vomiting during surgical procedures. The structure of droperidol dihydrate was reported previously (Blanton, Peeters & De Ranter, 1980). In an effort better to understand how the structure of droperidol relates to its pharmacological activity and the conformation of droperidol in different crystalline environments, we report here the structure of droperidol ethanol solvate.

Experimental. Colorless crystals, obtained from McNeil Pharmaceutical and recrystallized from ethanol, decayed at room temperature, apparently from solvent evaporation. Enraf–Nonius CAD-4 diffractometer with graphite-crystal-monochromatized Mo $K\alpha$ radiation and a locally modified Enraf–Nonius nitrogen-gas-flow system. Crystal $0{\cdot}2 \times 0{\cdot}2 \times 0{\cdot}2$ mm. Unit-cell dimensions and successful determination of the structure confirmed the space group as $P\bar{1}$. Lattice constants determined by least-squares fit of 25 reflections with $26 < 2\theta < 40°$ measured on the diffractometer. Three-dimensional intensity data collected in $\omega{:}2\theta$ scan mode with scan speeds variable between 5·0 and 0·6° min^{-1}. Total of 4256 independent reflections, 2261 observed with $I > 3\sigma(I)$: $2 < 2\theta < 54°$; $-7 \le h \le 7$, $-13 \le k \le 13$ and $0 \le l \le 20$. Equivalent reflections were averaged, $R_{int} = 2{\cdot}6\%$. Data corrected for Lorentz and polarization effects. Three standard reflections measured every 2 h during data collection ($17\bar{4}$, 006 and 400) showed no significant change in intensity. An intermittent instrument malfunction caused the occasional incorrect positioning of φ resulting in a few reflections with unobserved F_o and large F_c. These reflections were not included in the refinement. No absorption correction was applied to the data. Structure solved by direct methods using *MULTAN*11/82 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1982). Disordered ethanol molecule located near an inversion center with an occupancy of 0·50. H-atom positions for ethanol not located on Fourier difference map or calculated because there is more than one way to connect the atoms to make a reasonable ethanol molecule. Positions of droperidol skeleton H atoms located on Fourier difference map. Full-matrix least-squares refinement of 280 variables with coordinates and anisotropic temperature factors for droperidol and ethanol non-H atoms refined on F led to a final $R = 0{\cdot}046$, $wR = 0{\cdot}061$, where $w = 1/\sigma(F)^2$ and $\sigma(F)^2 = \sigma(F)^2_{cs} + (0{\cdot}04)^2(F^2)^2$, $S = 2{\cdot}00$. Isotropic temperature factors for the H atoms were held fixed at $B = 2{\cdot}0$ Å^2. Maximum and minimum peaks in Fourier difference map were 0·35 and 0·21 e Å^{-3}, respectively. $\Delta/\sigma_{max} = 0{\cdot}0$. Scattering factors were taken from *International Tables for X-ray Crystallography* (1974). *CAD*-4 *SDP* (Frenz, 1978) programs used.*

Discussion. Final fractional coordinates for the non-H atoms are given in Table 1. The numbering system for the molecule is shown in Fig. 1. Bond lengths and angles can be found in Table 2. The title compound crystallizes in an extended conformation with a structure similar to that of droperidol in droperidol dihydrate (Blaton *et al.*, 1980). An overlay diagram (Smith, 1984) of the two droperidol structures, Fig. 2, shows the similarity of the two molecules. The only apparent difference is in the orientation of the tetrahydropyridyl ring, although there are some small differences in the torsion angles of the oxobutyl side chain. Benperidol, a potent neuroleptic with a structure similar to that of droperidol (it has a piperidyl ring instead of the tetrahydropyridyl ring found in droperidol), also crystallizes with the oxobutyl side chain extended (Declercq, Germain & Koch, 1973). The torsion angles of the oxobutyl side chains for droperidol–ethanol, droperidol dihydrate and benperidol are listed in Table 3. Because the oxobutyl side chain consists of predominantly sp^3–sp^3 C–C bonds, it is not unusual to find small variations in the torsion angles of the extended chain. Perhaps what is more unusual is that the droperidol and benperidol molecules crystallize with an extended side chain in different crystallographic environments.

The benzimidazole group in the title compound is planar and makes an angle of 52·8° with the mean plane of the tetrahydropyridyl ring (48·1° in droperidol dihydrate).

The ethanol solvent molecule is located next to, and disordered about, an inversion center. The ethanol O atom is within hydrogen-bonding distance of the ketone oxygen {O(1) on the benzimidazole group [O(30)$\cdots$ O(1), 2·802 (7) Å]}. There are no other unusual intermolecular contacts. A stereoscopic packing diagram is given as Fig. 3.

We thank Dr Edwin D. Stevens and the University of New Orleans for allowing us to collect data on their instrument and McNeil Pharmaceutical for providing the sample. We also thank the NIH-MARC program for partial support of this work.

* Lists of structure factors, anisotropic temperature factors and H-atom positions have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51553 (47 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. *Positional parameters and their e.s.d.'s*

	x	y	z	B^*(Å²)
F28	0·2491 (4)	1·0124 (2)	0·9323 (1)	2·36 (4)
O1	1·0026 (4)	−0·3207 (2)	0·5584 (2)	1·64 (4)
O21	0·2283 (4)	0·4007 (2)	0·9288 (2)	2·17 (5)
O30	0·1706 (8)	0·8758 (5)	0·4710 (3)	2·1 (1)
N3	0·6881 (4)	−0·3281 (3)	0·6359 (2)	1·42 (5)
N8	0·7707 (5)	−0·5150 (3)	0·5600 (2)	1·59 (5)
N14	0·7149 (4)	0·0673 (3)	0·7743 (2)	1·27 (5)
C2	0·8380 (5)	−0·3828 (3)	0·5818 (2)	1·39 (6)
C4	0·3334 (5)	−0·4234 (3)	0·6914 (2)	1·61 (6)
C5	0·2042 (6)	−0·5441 (4)	0·6885 (2)	1·85 (7)
C6	0·2589 (6)	−0·6635 (4)	0·6412 (2)	2·04 (7)
C7	0·4471 (6)	−0·6663 (3)	0·5951 (2)	1·87 (7)
C9	0·5225 (5)	−0·4273 (3)	0·6459 (2)	1·37 (6)
C10	0·5759 (5)	−0·5474 (3)	0·5982 (2)	1·57 (6)
C11	0·7042 (5)	−0·1883 (3)	0·6744 (2)	1·26 (6)
C12	0·9088 (5)	−0·1306 (3)	0·7293 (2)	1·46 (6)
C13	0·9245 (5)	0·0220 (3)	0·7468 (2)	1·48 (6)
C15	0·5393 (5)	0·0231 (3)	0·7055 (2)	1·63 (6)
C16	0·5374 (5)	−0·1197 (3)	0·6631 (2)	1·48 (6)
C17	0·7404 (5)	0·2122 (3)	0·8005 (2)	1·50 (6)
C18	0·5406 (6)	0·2662 (3)	0·8428 (2)	1·52 (6)
C19	0·5681 (5)	0·4183 (3)	0·8619 (2)	1·48 (6)
C20	0·3728 (5)	0·4749 (3)	0·9036 (2)	1·43 (6)
C22	0·3505 (5)	0·6193 (3)	0·9138 (2)	1·39 (6)
C23	0·5105 (6)	0·7097 (3)	0·8889 (2)	1·54 (6)
C24	0·4783 (6)	0·8427 (3)	0·8953 (2)	1·77 (6)
C25	0·2844 (6)	0·8819 (3)	0·9277 (2)	1·69 (6)
C26	0·1250 (6)	0·7986 (3)	0·9553 (2)	1·72 (6)
C27	0·1586 (6)	0·6649 (3)	0·9473 (2)	1·66 (6)
C29	0·1116 (9)	0·9872 (6)	0·5024 (3)	1·0 (1)
C31	−0·144 (1)	1·000 (1)	0·4888 (8)	4·6 (2)

* $(4/3)[\beta_{11}a^2 + \beta_{22}b^2 + \cdots + \beta_{23}bc\cos\alpha]$.

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

Numbers in parentheses are e.s.d.'s in the least significant digit.

F28	C25	1·372 (2)	C9	C10	1·405 (3)	
O1	C2	1·241 (3)	C11	C12	1·487 (3)	
O21	C20	1·233 (3)	C11	C16	1·322 (3)	
O30	C29	1·26 (1)	C12	C13	1·534 (3)	
N3	C2	1·379 (3)	C15	C16	1·496 (3)	
N3	C9	1·391 (3)	C17	C18	1·526 (3)	
N3	C11	1·445 (3)	C18	C19	1·526 (3)	
N8	C2	1·352 (3)	C19	C20	1·509 (3)	
N8	C10	1·396 (3)	C20	C22	1·487 (3)	
N14	C13	1·464 (3)	C22	C23	1·397 (3)	
N14	C15	1·468 (3)	C22	C27	1·399 (3)	
N14	C17	1·460 (3)	C23	C24	1·390 (3)	
C4	C5	1·387 (3)	C24	C25	1·381 (3)	
C4	C9	1·390 (3)	C25	C26	1·369 (3)	
C5	C6	1·399 (3)	C26	C27	1·397 (3)	
C6	C7	1·391 (4)	C29	C31	1·584 (8)	
C7	C10	1·370 (3)				

C2	N3	C9	109·5 (2)	C12	C11	C16	123·2 (2)
C2	N3	C11	123·9 (2)	C11	C12	C13	109·6 (2)
C9	N3	C11	126·5 (2)	N14	C13	C12	111·0 (2)
C2	N8	C10	110·5 (2)	N14	C15	C16	112·9 (2)
C13	N14	C15	110·3 (2)	C11	C16	C15	122·3 (2)
C13	N14	C17	110·3 (2)	N14	C17	C18	113·8 (2)
C15	N14	C17	110·4 (2)	C17	C18	C19	111·5 (2)
O1	C2	N3	125·7 (2)	C18	C19	C20	112·8 (2)
O1	C2	N8	127·3 (2)	O21	C20	C19	119·9 (2)
N3	C2	N8	107·0 (2)	O21	C20	C22	119·4 (2)
C5	C4	C9	116·6 (2)	C19	C20	C22	120·7 (2)
C4	C5	C6	121·9 (2)	C20	C22	C23	122·5 (2)
C5	C6	C7	121·1 (2)	C20	C22	C27	118·3 (2)
C6	C7	C10	117·2 (2)	C23	C22	C27	119·1 (2)
N3	C9	C4	132·0 (2)	C22	C23	C24	120·8 (2)
N3	C9	C10	106·8 (2)	C23	C24	C25	117·6 (2)
C4	C9	C10	121·2 (2)	F28	C25	C24	117·9 (2)
N8	C10	C7	131·9 (2)	F28	C25	C26	118·0 (2)
N8	C10	C9	106·2 (2)	C24	C25	C26	124·0 (2)
C7	C10	C9	121·9 (2)	C25	C26	C27	117·6 (2)
N3	C11	C12	116·7 (2)	C22	C27	C26	120·8 (2)
N3	C11	C16	120·0 (2)	O30	C29	C31	115·6 (6)

Table 3. *Selected torsion angles* (°) *that describe the conformation of the oxobutyl side chain*

	Droperidol–ethanol	Droperidol dihydrate*	Benperidol†
C(21)–C(20)–C(19)–C(18)	9·9 (4)	1·0 (6)	5·6
C(20)–C(19)–C(18)–C(17)	180·0 (2)	−169·5 (4)	−166·2
C(19)–C(18)–C(17)–C(14)	−175·4 (2)	−167·3 (4)	−172·8
C(18)–C(17)–N(14)–C(13)	−171·2 (2)	−59·3 (4)	178·5
C(18)–C(17)–N(14)–C(15)	66·8 (2)	179·7 (4)	−60·7

* From Blaton, Peeters & De Ranter (1980).
† From Declercq, Germain & Koch (1973).

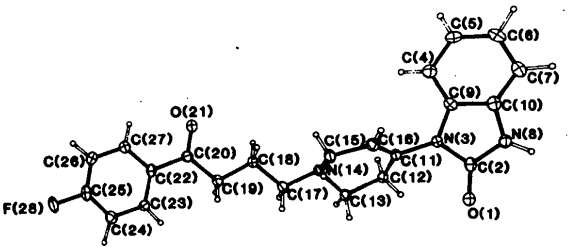

Fig. 1. Molecular structure and labeling system for the droperidol molecule in the title compound. The thermal ellipsoids are drawn at 50% probability levels.

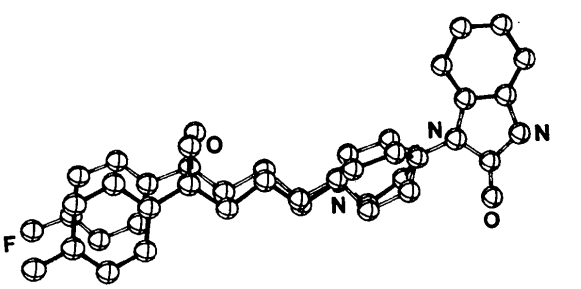

Fig. 2. Overlay diagram of droperidol in the title compound (open bonds) and droperidol in the dihydrate (filled bonds) showing the molecular fit of the two molecules after minimizing the distances between the O and N atoms in the benzimidazolinone group.

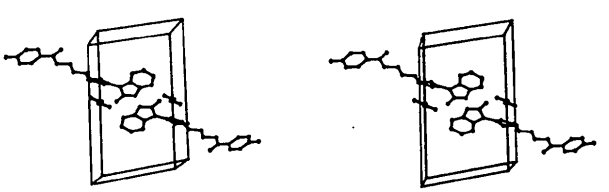

Fig. 3. Stereoscopic diagram showing the packing arrangement of the droperidol and the ethanol molecules viewed down the *c* axis.

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The Synthesis of *N*-Benzoyl-1,2,4-triazole Oximes. Structures of Two Isomers, $C_{16}H_{14}N_4O$: 5-Methyl-3-phenyl-1,2,4-triazol-1-yl Phenyl Ketone Oxime (1) and 3-Methyl-5-phenyl-1,2,4-triazol-4-yl Phenyl Ketone Oxime (3)

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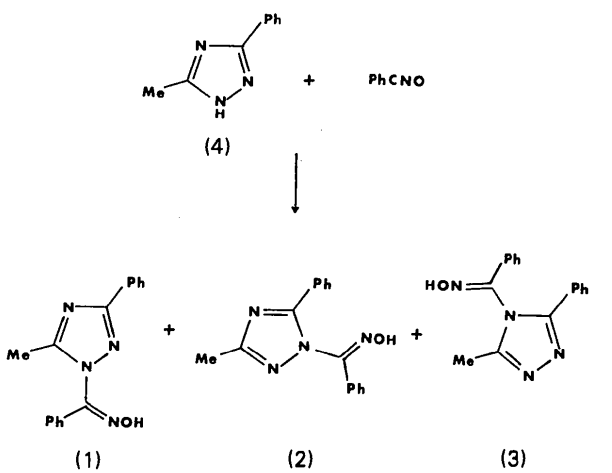
Abstract. (1): $C_{16}H_{14}N_4O$, $M_r = 278{\cdot}3$, orthorhombic, *Pbca*, $a = 23{\cdot}001$ (3), $b = 13{\cdot}155$ (2), $c = 9{\cdot}526$ (1) Å, $V = 2882{\cdot}4$ (6) Å^3, $Z = 8$, $D_x = 1{\cdot}28$ Mg m^{-3}, λ(Mo $K\alpha$) = 0·71069 Å, $\mu = 0{\cdot}78$ cm^{-1}, $F(000) = 1168$, $T = 293$ K, $R = 0{\cdot}054$ for 823 independent reflections. (3): $C_{16}H_{14}N_4O$, $M_r = 278{\cdot}3$, monoclinic, $P2_1/n$, $a = 9{\cdot}869$ (1), $b = 12{\cdot}656$ (2), $c = 12{\cdot}939$ (2) Å, $\beta = 108{\cdot}8$ (1)°, $V = 1530$ (1) Å^3, $Z = 4$, $D_x = 1{\cdot}21$ Mg m^{-3}, λ(Mo $K\alpha$) = 0·71069 Å, $\mu = 0{\cdot}74$ cm^{-1}, $F(000) = 584$, $T = 293$ K, $R = 0{\cdot}047$ for 1040 independent reflections. The reaction of benzonitrile oxide with N-unsubstituted 1,2,4-triazoles yields *N*-benzoyl oximes by nucleophilic addition at the dipole. In (1) and in (3) the heterocyclic ring is planar. The dihedral angles between the mean plane of the benzene ring and that of the triazole ring are 7·6 (2) and 35·9 (2)° for (1) and (3), respectively. The oxime fragment in both isomers is nearly perpendicular with respect to the triazole ring and has *Z* configuration.

Introduction. The reaction between N-unsubstituted 1,2,4-triazoles and benzonitrile oxide occurs by the 1,3-addition mechanism leading to the formation of *N*-benzoyl oximes (Foti, Grassi, Caruso, Risitano & Bruno, 1987). Depending on the symmetrical or unsymmetrical C-substitution, 1,2,4-triazoles would be expected to yield two or three isomeric oximes, respectively. In order to test this, 5-methyl-3-phenyl-1,2,4-triazole (4) was selected as a model substance and was reacted with benzonitrile oxide (scheme below).



Three products were isolated, the elemental compositions of which, $C_{16}H_{14}N_4O$, were identical. The chemical nature of these isomers (1), (2) and (3) was established from spectroscopic data (Table 1) and the X-ray analysis was carried out to confirm the structures of the 1- (1) and the 4-substituted (3) derivatives. In agreement with previous results (Uda, Hisazumi, Sato & Kubota, 1976), the nucleophilic addition

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