

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71626 (16 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: OH1047]

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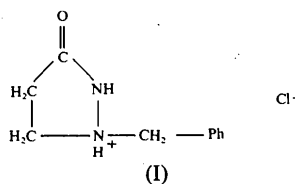
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of the cation has an approximate half-chair conformation. Atom N(1) is *sp*<sup>3</sup> hybridized while atom N(2) is *sp*<sup>2</sup> hybridized. The C(3)—N(2) bond distance of 1.373 (4) Å is a partial double bond. Both H atoms connected to N atoms are involved in hydrogen bonding of the N—H...Cl type.

## Comment

γ-Aminobutyrate (GABA) is known to be a depressant neurotransmitter. Therefore, an increase in the GABA content of the brain might be correlated with anticonvulsant activity. Since 3-pyrazolidones were reported to be a potent inhibitor of GABA-transaminase (White *et al.*, 1982), these kinds of compounds have been expected to be effective in elevating GABA levels in the brain and correspondingly to have anticonvulsant activity. Pharmacological tests have proved that expectation (Ling, Li, Liu & Pei, 1986; Du, Ling, Liu, Ku & Liu, 1989; Lei, Luan, Ling, Li & Pei, 1990; Lei, Zhang, Ling & Ku, 1990). The X-ray analysis of the title compound was undertaken as part of a search for common conformational features of 3-pyrazolidones, which may help in understanding the mechanism of action of such compounds at the molecular level, as well as providing a reliable molecular model for 3D-QSAR comparative molecular-field analysis.

According to the results of structural analysis, the crystal structure of the title compound (I) consists of cations of protonated 1-benzyl-3-pyrazolidone and chloride anions. This indicates that the lone pair of electrons on atom N(1) is clearly protophilic. Fig. 1 provides a view of the structural features of the cation.



The conformation of the five-membered ring is approximately a half-chair; atoms N(1) and C(5) are displaced on opposite sides of the plane through atoms N(2), C(3), C(4) and O (planar within 0.006 Å) with displacements of 0.264 and 0.220 Å, respectively. Examination of the intramolecular bond angles associated with atoms N(1) and N(2) suggests that N(1) is *sp*<sup>3</sup> hybridized, displaying bond angles N(2)—N(1)—C(5) 103.2 (2), N(2)—N(1)—C(6) 111.4 (2), C(5)—N(1)—C(6) 116.0 (2) and N(2)—N(1)—H(N1) 106 (2)°, while N(2) is approximately *sp*<sup>2</sup> hybridized. The sum of the bond angles about atom N(2) is 351.0°. The C(3)—N(2) bond distance

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## 1-Benzyl-3-pyrazolidone Hydrochloride

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### Abstract

The crystal of the title compound, C<sub>10</sub>H<sub>13</sub>N<sub>2</sub>O<sup>+</sup>.Cl<sup>-</sup>, consists of 1-benzyl-3-oxo-1*H*<sup>+</sup>-pyrazolidinium cations and chloride anions. The five-membered ring

of 1.373 (4) Å is slightly longer than the corresponding C—N length of 1.362 (3) Å found in 2-phenylpyrazolidin-3-one by Ege *et al.* (1983) and longer still than that of 1.348 (2) Å found in 1-phenylpyrazolidin-3-one (Sbit, Dupont, Dideberg, Liegeois & Delarge, 1987). In contrast, the C(3)—O bond distance of 1.210 (3) Å is shorter than the C(3)—O bond lengths of 1.223 (3) and 1.228 (2) Å presented in the aforementioned compounds, respectively. Substituting groups with electron-releasing capabilities at the 1 or 2 positions might have an effect on the C—O and C—N bond lengths of the conjugated N(2)—C(3)—O system. However, the N(1)—N(2) bond distance of 1.436 (2) Å is similar to the N—N single bonds in 2-phenylpyrazolidin-3-one and 1-phenylpyrazolidin-3-one [1.445 (3) and 1.434 (2) Å, respectively]. The packing of molecules in the crystal is shown in Fig. 2. The Cl ion forms two hydrogen bonds with atoms N(1) and N(2) of the nearest neighbouring molecules [N(1)—H(N1) 0.99 (4), N(1)⋯Cl 3.029 Å, N(1)—H(N1)⋯Cl 167.7°; N(2)—H(N2) 0.90 (4), N(2)⋯Cl<sup>i</sup> 3.119 Å, N(2)—H(N2)⋯Cl<sup>i</sup> 167.9°, symmetry code: (i)  $\frac{1}{2} - x, -\frac{1}{2} + y, \frac{1}{2} - z$ ].

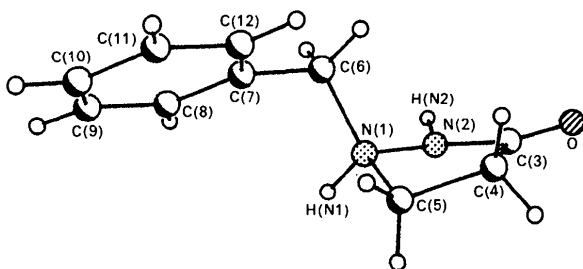


Fig. 1. Structure of the cation showing the atom-numbering scheme.

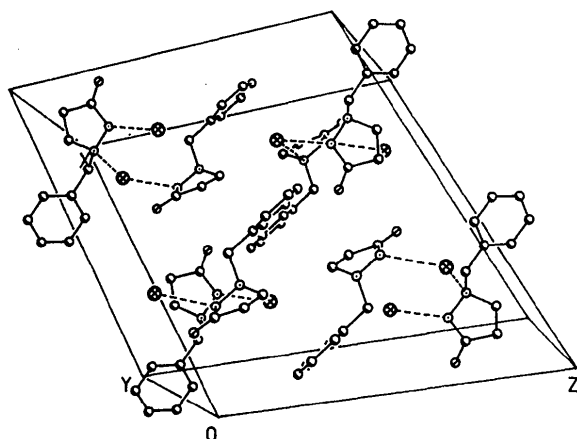


Fig. 2. Packing of 1-benzyl-3-pyrazolidone within the unit cell.

## Experimental

### Crystal data

C<sub>10</sub>H<sub>13</sub>N<sub>2</sub>O<sup>+</sup>.Cl<sup>-</sup>  
*M<sub>r</sub>* = 212.7  
 Monoclinic  
 C2/c  
*a* = 17.970 (4) Å  
*b* = 7.418 (2) Å  
*c* = 17.634 (6) Å  
 $\beta$  = 116.84 (2)°  
*V* = 2097 (1) Å<sup>3</sup>  
*Z* = 8  
*D<sub>x</sub>* = 1.347 Mg m<sup>-3</sup>

### Mo K $\alpha$ radiation

$\lambda$  = 0.71073 Å  
 Cell parameters from 25 reflections  
 $\theta$  = 8.5–13.5°  
 $\mu$  = 0.331 mm<sup>-1</sup>  
*T* = 296 K  
 Prismatic  
 0.40 × 0.35 × 0.15 mm  
 Colorless  
 Crystal source: crystallized from water/acetone solution

### Data collection

Siemens R3m/V diffractometer  
 2 $\theta$ – $\theta$  scans  
 Absorption correction: none  
 2088 measured reflections  
 1861 independent reflections  
 1429 observed reflections [*F* > 4.0 $\sigma$ (*F*)]

*R*<sub>int</sub> = 0.0153  
 $\theta_{\max}$  = 25.0°  
*h* = –21 → 0  
*k* = 0 → 8  
*l* = –18 → 20  
 2 standard reflections monitored every 100 reflections  
 intensity variation: 4%

### Refinement

Refinement on *I*  
*R* = 0.0405  
*wR* = 0.0464  
*S* = 1.74  
 1429 reflections  
 133 parameters  
 $w = 1/[\sigma^2(F) + 0.0002F^2]$   
 $(\Delta/\sigma)_{\max} = 0.540$

$\Delta\rho_{\max} = 0.22 \text{ e \AA}^{-3}$   
 $\Delta\rho_{\min} = -0.25 \text{ e \AA}^{-3}$   
 Extinction correction: none  
 Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

Table 1. Fractional atomic coordinates and equivalent isotropic displacement parameters (Å<sup>2</sup>)

$$U_{\text{eq}} = (1/3)\sum_i\sum_j U_{ij}a_i^*a_j^*a_i\cdot a_j$$

	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> <sub>eq</sub>
Cl	0.2446 (1)	0.0411 (1)	0.6322 (1)	0.049 (1)
O	0.4186 (1)	–0.6195 (3)	0.7920 (1)	0.055 (1)
N(1)	0.2560 (1)	–0.3663 (3)	0.6433 (1)	0.029 (1)
N(2)	0.3155 (1)	–0.4125 (3)	0.7284 (1)	0.034 (1)
C(3)	0.3707 (2)	–0.5406 (3)	0.7286 (2)	0.035 (1)
C(4)	0.3606 (2)	–0.5587 (3)	0.6395 (2)	0.036 (1)
C(5)	0.3026 (2)	–0.4065 (4)	0.5916 (2)	0.035 (1)
C(6)	0.1745 (1)	–0.4659 (3)	0.6170 (1)	0.032 (1)
C(7)	0.1119 (1)	–0.4223 (3)	0.5272 (1)	0.028 (1)
C(8)	0.0651 (2)	–0.2660 (4)	0.5088 (2)	0.039 (1)
C(9)	0.0065 (2)	–0.2307 (4)	0.4254 (2)	0.046 (1)
C(10)	–0.0047 (2)	–0.3482 (4)	0.3611 (2)	0.044 (1)
C(11)	0.0413 (2)	–0.5040 (4)	0.3791 (2)	0.047 (1)
C(12)	0.0995 (2)	–0.5416 (4)	0.4625 (2)	0.038 (1)
H(N1)	0.2472 (19)	–0.2346 (53)	0.6440 (20)	0.080
H(N2)	0.2982 (20)	–0.4076 (49)	0.7687 (21)	0.080

Table 2. Selected geometric parameters (Å, °)

O—C(3)	1.210 (3)	N(1)—N(2)	1.436 (2)
N(1)—C(5)	1.521 (4)	N(1)—C(6)	1.514 (3)
N(1)—H(N1)	0.991 (39)	N(2)—C(3)	1.373 (4)

N(2)—H(N2)	0.897 (44)	C(3)—C(4)	1.504 (4)
C(4)—C(5)	1.510 (3)	C(6)—C(7)	1.506 (3)
C(7)—C(8)	1.383 (4)	C(7)—C(12)	1.380 (4)
C(8)—C(9)	1.393 (3)	C(9)—C(10)	1.372 (4)
C(10)—C(11)	1.371 (4)	C(11)—C(12)	1.395 (3)
N(2)—N(1)—C(5)	103.2 (2)	N(2)—N(1)—C(6)	111.4 (2)
C(5)—N(1)—C(6)	116.0 (2)	N(2)—N(1)—H(N1)	105.7 (16)
C(5)—N(1)—H(N1)	109.6 (24)	C(6)—N(1)—H(N1)	110.3 (20)
N(1)—N(2)—C(3)	111.4 (2)	N(1)—N(2)—H(N2)	117.3 (18)
C(3)—N(2)—H(N2)	122.3 (21)	O—C(3)—N(2)	123.3 (3)
O—C(3)—C(4)	128.5 (3)	N(2)—C(3)—C(4)	108.2 (2)
C(3)—C(4)—C(5)	104.2 (2)	N(1)—C(5)—C(4)	103.8 (2)
N(1)—C(6)—C(7)	112.2 (2)	C(6)—C(7)—C(8)	120.9 (2)
C(6)—C(7)—C(12)	119.8 (2)	C(8)—C(7)—C(12)	119.3 (2)
C(7)—C(8)—C(9)	119.7 (3)	C(8)—C(9)—C(10)	120.8 (3)
C(9)—C(10)—C(11)	119.7 (2)	C(10)—C(11)—C(12)	119.9 (3)
C(7)—C(12)—C(11)	120.6 (2)		

The data were collected with a variable scan speed of  $5.45\text{--}29.30^\circ \text{ min}^{-1}$  in  $\omega$  and a scan range of  $0.60^\circ + K\alpha$  separation. Background measurements were made with a stationary crystal and a stationary counter at the beginning and end of each scan, each for 25.0% of the total scan time. The structure was solved by direct methods and refined by full-matrix least squares using *SHELXTL-Plus* (Sheldrick, 1987). The H atoms bonded to C atoms were positioned theoretically with a geometrically fixed C—H distance of 0.96 Å, those bonded to N atoms were located from a difference Fourier map. The coordinates of the H atoms at N(1) and N(2) were refined. All H atoms were given a fixed isotropic displacement parameter of  $0.08 \text{ \AA}^2$  during refinement.

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Lists of structure factors, anisotropic displacement parameters and H-atom coordinates have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71615 (10 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: LI1063]

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## 1-(3-Quinoly)-4-(2-thienyl)-1,3-butadiyne, $C_{17}H_9NS$

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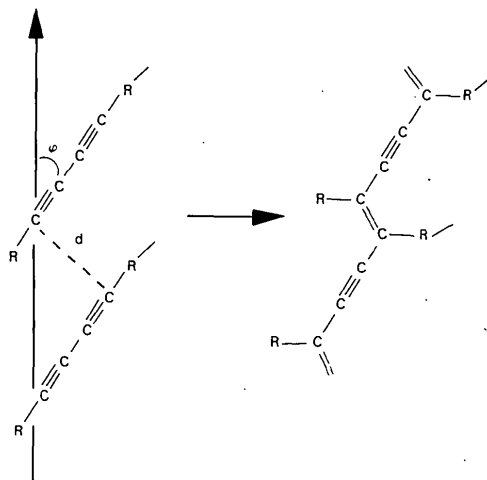
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### Abstract

The S atom in the thiophene ring is disordered between positions 1 and 3. The molecular packing would not afford any facile mechanism for crystal-phase polymerization. The thiophene ring is rotated by  $55^\circ$  with respect to the plane of the quinoline system.

### Comment

Diacetylenes ( $R-C\equiv C-C\equiv C-R'$ ) may undergo solid-state *trans*-1,4 polymerization upon thermal, photochemical or mechanical stimulation.



The reactivity of a diacetylene in the crystalline state is governed by the molecular packing of the monomer. Only those diacetylenes which have suitable packing conditions (stacking angle  $\varphi$  and contact distance  $d$ ) will undergo facile polymerization (Baughman, 1974; Wegner, 1977; Bloor, 1982). The side groups,  $R$  and  $R'$ , in diacetylenes influence the crystal packing through steric and electronic effects and hence determine the reactivity. Our present