observation of slightly elongated C—S single bonds in planar or near-planar systems (Andreetti, Bocelli & Sgarabotto, 1980; Ammon, Watts & Stewart, 1970). The above distances compare well to those found, for example, in 5,6-dihydrothiazolo[2,3-*c*][1,2,4]thiadiazol-3-one, 6*β*-(1-ethyl-1-hydroxypropyl)-5α-phenyl-2,3,5,6-tetrahydroimidazo[2,1-*b*]thiazole and 5,5-diphenyl-2,3-dihydroimidazo[2,1-*b*]thiazol-6(5*H*)-one (Cameron, Cameron & Duncanson, 1981). There are no intermolecular distances shorter than the van der Waals separations. The shortest intermolecular contact is C(14)···O(5) $(-1+x, y, z)$ of 3·230 (5) Å.

The financial support of the Research Community of Slovenia is gratefully acknowledged. We thank Professor B. Stanovnik for stimulating discussions.

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Acta Cryst. (1987). C**43**, 1818–1821

Structure of Isoquinolinium Nitrate and a Redetermination of the Corresponding Chloride

BY JOHN J. DALY

ZFE, F. Hoffmann–La Roche AG, 4002 *Basel, Switzerland*

A. ALAN PINKERTON

Department of Chemistry, The University of Toledo, Toledo, OH 43606, *USA*

AND DIETER SCHWARZENBACH

Institut de Cristallographie, BSP, 1015 *Lausanne–Dorigny, Switzerland*

(*Received* 9 *October* 1986; *accepted* 8 *May* 1987)

Abstract. $C_9H_8N^+.NO_3^-$, $M_r = 192·17$, triclinic, $P\bar{1}$, $a = 5·114$ (1), $b = 9·304$ (2), $c = 9·973$ (3) Å, $\alpha = 100·85$ (2), $\beta = 104·39$ (2), $\gamma = 92·14$ (2)°, $V = 449·6$ (2) Å^3, $Z = 2$, $D_x = 1·42$ g cm^{-3}, λ(Mo $K\alpha$) = 0·71069 Å, $\mu = 1·2$ cm^{-1}, $F(000) = 200$, $T = 295$ K, $R = 0·042$ for 809 unique observed reflections. $C_9H_8N^+.Cl^-$, $M_r = 165·6$, monoclinic, $P2_1/a$, $a = 9·192$ (5), $b = 17·486$ (6), $c = 5·161$ (3) Å, $\beta = 100·45$ (4)°, $V = 815·8$ (7) Å^3, $Z = 4$, $D_x = 1·35$ g cm^{-3}, λ(Mo $K\alpha$) = 0·71069 Å, $\mu = 4·0$ cm^{-1}, $F(000) = 344$, $T = 295$ K, $R = 0·044$ for 748 unique observed reflections. The isoquinolinium ion is planar and hydrogen-bonded to the counter ion. The variation in skeletal bond lengths is similar to that observed for isoelectronic naphthalene. The smaller size of N is manifested in shorter 1,2 and 2,3 bond lengths and an opening of the 4,10,5 angle (IUPAC atom numbering). Shorter C—C bonds in the heterocycle are attributed to *π*-electron transfer from the carbocycle due to the formal positive charge on N.

Introduction. As part of an NMR study we had need of accurate structural parameters for the isoquinolinium ion, $C_9H_8N^+$. A survey of the literature revealed the chloride (both anhydrous and the monohydrate) as the only simple salt to have been structurally characterized (Genet, 1965). The structures were determined from photographic data and refined to $R = 0·15$ and $R = 0·18$ respectively. In both cases no e.s.d.'s were quoted for the bond lengths and angles of the molecular skeleton, thus no discussion of the variation of these parameters is possible. The positions of the H atoms

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Table 1. *Summary of intensity measurements, structure solution and refinement for isoquinolinium nitrate and chloride*

Formula	$C_9H_8N^+.NO_3^-$	$C_9H_8N^+.Cl^-$
Crystal dimensions (mm)	0·33 × 0·19 × 0·15	0·30 × 0·27 × 0·10
Transmission	0·97–0·99	0·94–0·97
Scan method	θ–2θ	
$[(\sin\theta)/\lambda_{\max}$ (Å^{-1})	0·60	0·65
Reflections measured	1857	2181
Range of *hkl*	0→6, −10→11, −11→11	0→11, 0→22, −6→6
No. of unique reflections	1566	1883
No. of reflections with $I < 3\sigma(I)$	757	1135
$R_{\rm int}$	0·012	0·023
Variation in standards (three)	$17\sigma(I)$	$12\sigma(I)$
Structure solution	Direct methods	Refinement only
Refinement method*	Full-matrix least squares	
Function minimized	$\sum w(\lvert F_o\rvert - \lvert F_c\rvert)^2$	
w	$1/\sigma(F)^2$	
No. of variables	159	132
R	0·042	0·044
wR	0·040	0·037
S	1·84	1·45
Final $(\Delta/\sigma)_{\max}$	0·01	0·41
Final $\Delta\rho_{\max}$(e Å^{-3})	0·20	0·22

* Only reflections of $I > 3\sigma(I)$, or of $I < 3\sigma(I)$ with $\lvert F_c\rvert > \lvert F_o\rvert$ were included.

were not determined in either case. For the purposes of our NMR project it is necessary to have both the H positions and accurate (to a few thousandths Å) positions for the C and N atoms. We have thus redetermined the structure of the anhydrous chloride and determined that of the nitrate analogue.

Experimental. The salts were prepared from the free base and the appropriate acid in aqueous solution. The neutral solution was evaporated and the product crystallized from acetonitrile as colorless needles in both cases. The chloride was mounted under nitrogen in a sealed capillary to avoid hydration. The nitrate is stable in air.

Determination of lattice parameters (25 reflections, $15 < \theta < 18°$) and X-ray intensity measurements were carried out with a Syntex $P2_1$ automatic diffractometer with Nb-filtered Mo $K\alpha$ radiation and corrected for absorption as before (Pinkerton & Schwarzenbach, 1980). Backgrounds were obtained from analysis of the scan profile (Blessing, Coppens & Becker, 1972; Schwarzenbach, 1971). Measurement methods, structure determination and refinement are summarized in Table 1. Scattering factors and anomalous-scattering coefficients were taken from the literature (Cromer & Mann, 1968; Stewart, Davidson & Simpson, 1965; Cromer & Liberman, 1970).

For the chloride, the starting coordinates for the non-H atoms were taken from the literature (Genet, 1965). The initial solution for the nitrate was obtained by direct methods using *SHELXTL* (Sheldrick, 1983). In both cases structure completion and refinement were carried out with *XRAY*72 (Stewart, Kundell & Baldwin, 1972). In both cases the position of the N was unambiguous (temperature factors, bond lengths, hydrogen bonding). The figures were prepared with *ORTEP* (Johnson, 1971).

In the final cycles of full-matrix least squares, all of the non-H atoms were refined with anisotropic thermal parameters and the H atoms with isotropic ones. Final atomic coordinates are given in Tables 2 and 3.* Derived bond lengths and angles are reported in Table 4.

Table 2. *Atomic coordinates and isotropic thermal parameters for* $C_9H_8N^+.NO_3^-$

$$U_{\rm eq} = \tfrac{1}{3}(U_{11} + U_{22} + U_{33}).$$

	x	y	z	$U_{\rm eq}/U$(Å^2)
C(1)	0·3652 (5)	0·3570 (3)	0·3364 (3)	0·051 (1)
C(3)	0·3316 (6)	0·1220 (3)	0·3863 (3)	0·065 (1)
C(4)	0·4811 (6)	0·0701 (3)	0·2979 (3)	0·062 (1)
C(5)	0·7369 (6)	0·1142 (4)	0·1260 (3)	0·067 (1)
C(6)	0·8160 (6)	0·2082 (5)	0·0530 (3)	0·081 (2)
C(7)	0·7547 (6)	0·3551 (4)	0·0722 (3)	0·081 (1)
C(8)	0·6121 (6)	0·4058 (4)	0·1665 (3)	0·067 (1)
C(9)	0·5212 (5)	0·3102 (3)	0·2425 (2)	0·0458 (9)
C(10)	0·5812 (5)	0·1619 (3)	0·2221 (2)	0·050 (1)
N(1)	−0·0637 (5)	0·2810 (2)	0·6578 (3)	0·065 (1)
N(2)	0·2775 (4)	0·2656 (2)	0·4052 (2)	0·0574 (9)
O(1)	−0·0598 (4)	0·3607 (2)	0·5692 (2)	0·0737 (8)
O(2)	0·0766 (5)	0·1776 (2)	0·6610 (3)	0·128 (1)
O(3)	−0·2127 (5)	0·3064 (2)	0·7360 (2)	0·124 (1)
H(1)	0·311 (5)	0·459 (3)	0·356 (2)	0·074 (8)
H(2)	0·157 (6)	0·311 (3)	0·482 (3)	0·12 (1)
H(3)	0·252 (5)	0·060 (3)	0·435 (2)	0·071 (8)
H(4)	0·520 (5)	−0·029 (3)	0·288 (2)	0·066 (8)
H(5)	0·760 (5)	0·017 (3)	0·111 (3)	0·07 (1)
H(6)	0·921 (5)	0·181 (3)	−0·007 (3)	0·09 (1)
H(7)	0·816 (5)	0·424 (3)	0·019 (3)	0·10 (1)
H(8)	0·563 (5)	0·498 (3)	0·179 (2)	0·069 (9)

Table 3. *Atomic coordinates and isotropic thermal parameters for* $C_9H_8N^+.Cl^-$

$$U_{\rm eq} = \tfrac{1}{3}(U_{11} + U_{22} + U_{33}).$$

	x	y	z	$U_{\rm eq}/U$(Å^2)
Cl	0·65756 (9)	0·37540 (6)	0·3468 (2)	0·0567 (3)
C(1)	0·3797 (5)	0·4179 (3)	0·7925 (8)	0·047 (1)
C(3)	0·3746 (6)	0·2861 (3)	0·7174 (9)	0·057 (2)
C(4)	0·2761 (5)	0·2751 (3)	0·8785 (8)	0·050 (2)
C(5)	0·1179 (5)	0·3302 (3)	1·1700 (7)	0·050 (2)
C(6)	0·0731 (5)	0·3933 (3)	1·2855 (8)	0·062 (2)
C(7)	0·1291 (6)	0·4656 (3)	1·2473 (9)	0·058 (2)
C(8)	0·2298 (4)	0·4759 (3)	1·0886 (8)	0·052 (1)
C(9)	0·2779 (4)	0·4103 (3)	0·9626 (7)	0·037 (1)
C(10)	0·2242 (4)	0·3365 (4)	1·0056 (6)	0·039 (1)
N	0·4254 (3)	0·3570 (2)	0·6765 (6)	0·049 (2)
H(1)	0·414 (4)	0·465 (2)	0·754 (6)	0·03 (1)
H(2)	0·509 (3)	0·357 (2)	0·554 (6)	0·08 (1)
H(3)	0·409 (4)	0·247 (2)	0·626 (6)	0·05 (1)
H(4)	0·244 (3)	0·229 (3)	0·915 (8)	0·05 (1)
H(5)	0·085 (4)	0·271 (3)	1·207 (7)	0·06 (1)
H(6)	0·000 (3)	0·378 (3)	1·386 (5)	0·07 (1)
H(7)	0·100 (4)	0·510 (2)	1·323 (7)	0·07 (1)
H(8)	0·269 (4)	0·528 (3)	1·053 (8)	0·09 (2)

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* Tables of observed and calculated structure factors and anisotropic thermal parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44037 (18 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 4. *Bond lengths* (Å) *and angles* (°) *for isoquinolinium nitrate and chloride with e.s.d.'s in parentheses*

	NO_3^-	Cl^-		NO_3^-	Cl^-		NO_3^-	Cl^-
C(1)–N(2)	1·319 (4)	1·326 (6)	C(6)–C(7)	1·402 (6)	1·392 (8)	C(3)–H(3)	0·96 (3)	0·92 (4)
C(1)–C(9)	1·395 (4)	1·401 (6)	C(7)–C(8)	1·356 (5)	1·354 (7)	C(4)–H(4)	0·94 (2)	0·88 (4)
N(2)–C(3)	1·363 (4)	1·354 (7)	C(8)–C(9)	1·407 (5)	1·428 (7)	C(5)–H(5)	0·90 (3)	1·11 (4)
C(3)–C(4)	1·340 (5)	1·350 (7)	C(9)–C(10)	1·412 (4)	1·413 (8)	C(6)–H(6)	0·90 (3)	0·96 (3)
C(4)–C(10)	1·402 (4)	1·387 (7)	C(1)–H(1)	1·00 (2)	0·92 (4)	C(7)–H(7)	0·99 (3)	0·93 (4)
C(5)–C(10)	1·413 (4)	1·410 (6)	N(2)–H(2)	1·13 (3)	1·08 (3)	C(8)–H(8)	0·89 (3)	1·01 (5)
C(5)–C(6)	1·348 (6)	1·353 (7)						
C(9)–C(1)–N(2)	120·9 (3)	120·6 (5)	C(3)–C(4)–H(4)	119 (2)	123 (3)	C(8)–C(7)–H(7)	119 (2)	115 (3)
C(9)–C(1)–H(1)	123 (2)	121 (2)	C(10)–C(4)–H(4)	120 (2)	116 (3)	C(7)–C(8)–C(9)	119·9 (3)	118·1 (5)
N(2)–C(1)–H(1)	116 (2)	118 (2)	C(10)–C(5)–C(6)	119·9 (3)	120·1 (5)	C(7)–C(8)–H(8)	122 (2)	122 (3)
C(1)–N(2)–C(3)	121·7 (3)	121·2 (4)	C(10)–C(5)–H(5)	116 (2)	114 (2)	C(9)–C(8)–H(8)	118 (2)	119 (3)
C(1)–N(2)–H(2)	118 (2)	125 (2)	C(6)–C(5)–H(5)	124 (2)	125 (2)	C(8)–C(9)–C(1)	121·5 (3)	120·5 (5)
C(3)–N(2)–H(2)	121 (2)	113 (2)	C(5)–C(6)–C(7)	121·9 (3)	121·9 (4)	C(8)–C(9)–C(10)	120·2 (3)	121 ·1 (4)
N(2)–C(3)–C(4)	120·3 (3)	121·0 (5)	C(5)–C(6)–H(6)	121 (2)	108 (3)	C(1)–C(9)–C(10)	118·3 (3)	118·5 (4)
N(2)–C(3)–H(3)	117 (1)	116 (2)	C(7)–C(6)–H(6)	117 (2)	130 (3)	C(9)–C(10)–C(4)	118·1 (2)	118·2 (4)
C(4)–C(3)–H(3)	122 (1)	123 (2)	C(6)–C(7)–C(8)	119·8 (4)	121·1 (5)	C(9)–C(10)–C(5)	118·3 (3)	117·7 (5)
C(3)–C(4)–C(10)	120·7 (3)	120·5 (5)	C(6)–C(7)–H(7)	121 (2)	124 (3)	C(4)–C(10)–C(5)	123·7 (3)	124 ·1 (5)

Hydrogen bonds: O(1)···H(2), 1·60 (3); Cl···H(2) 1·91 (3); N(2)–H(2)···O(1), 170 (2); N(2)–H(2)···Cl, 171 (3). Nitrate groups: N–O, 1·259 (3), 1·222 (3), 1·218 (4); O–N–O, 119·3 (3), 119·2 (2), 121·5 (3).

Perspective drawings of the nitrate (Fig. 1) and chloride (Fig. 2) show the hydrogen bonding and indicate the atom numbering.

Discussion. In both structures the isoquinolinium ion is planar as expected. The maximum deviation from the least-squares plane is 0·024 Å [C(7)] for the nitrate and 0·017 Å [C(6)] for the chloride.

It is instructive to compare the variations in the bond lengths observed here (Fig. 3) with those observed for the isoelectronic naphthalene molecule (Brock & Dunitz, 1982). The carbocycle – C(5), C(6), C(7), C(8), C(9), C(10) – is directly comparable. The same variations of long and short bonds are observed. The apparent overall shortening observed here (0·01–0·02 Å) is due to the current values being uncorrected for thermal motion. In the heterocycle – C(1), N(2), C(3), C(4), C(9), C(10) – the same overall variations in bond lengths are observed with the additional shortening due to the smaller size of N superimposed, *e.g.* C(1)–N(2) = 1·323, C(3)–N(2) = 1·359 Å. There is also an overall shortening of the C–C bonds in this ring compared to equivalent ones in the carbocycle. We believe this to be not only due to the size of the N shrinking this ring, but also due to a drift in π-electron density into this ring due to the formal positive charge on N. The smaller size of the heterocycle is also manifested by the opening of the C(4)–C(10)–C(5) angle.

We have observed (Buranda, Newman & Pinkerton, 1987) significantly different NMR behavior for C(1) *vs* C(3) and H(1) *vs* H(3) for the free base in the presence of lanthanide shift reagents. We may now interpret this as largely due to the asymmetry of the bonding at N.

As previously deduced without access to H-atom positions (Genet, 1965), a hydrogen bond exists between the chloride ion and N(2). Not surprisingly, we also observe hydrogen bonding in the nitrate analogue. It is noteworthy that the nitrate N–O bond involved in hydrogen bonding is lengthened (0·04 Å) compared to the other two. This may be partly due to the difference in thermal parameters of the O atoms; however, the opening of the O(2)–N(1)–O(3) angle suggests that the lengthening is real. This type of behavior is in the same direction as observed for monodentate binding of a nitrate group to a metal ion (Addison, Logan, Wallwork & Garner, 1971).

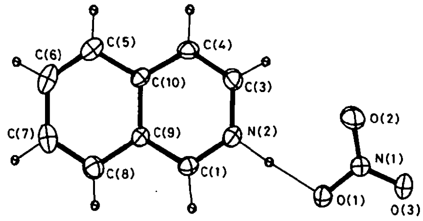

Fig. 1. *ORTEP* plot of $C_9H_8N^+.NO_3^-$ – 50% probability ellipsoids for C, N, O.

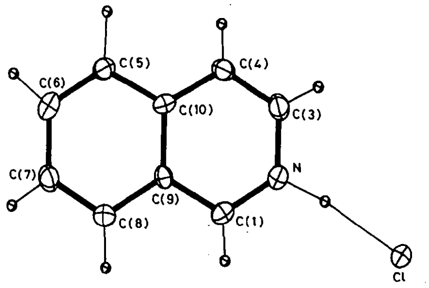

Fig. 2. *ORTEP* plot of $C_9H_8N^+.Cl^-$ – 50% probability ellipsoids for C, N, Cl.

We thank the Swiss National Science Foundation and the College of Arts and Sciences of The University of Toledo for financial support.

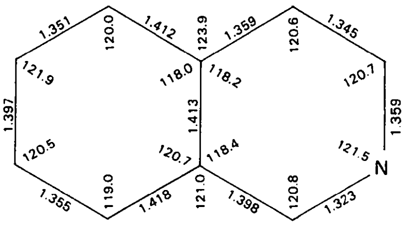

Fig. 3. Average bond distances (Å) and angles (°) in $C_9H_8N^+$.

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Acta Cryst. (1987). C**43**, 1821–1822

Structure of a Substituted Butene

BY GENE B. CARPENTER

Department of Chemistry, Brown University, Providence, Rhode Island 02912, *USA*

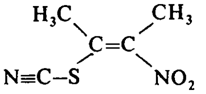
AND KYONG PAE PARK

Department of Chemistry, KAIST, Dong Dae Mun, PO Box No. 131, *Seoul, Korea*

(*Received* 30 *June* 1986; *accepted* 17 *November* 1986)

Abstract. *cis*-3-Nitro-2-thiocyanato-2-butene, $C_5H_6N_2O_2S$, $M_r = 158{\cdot}2$, monoclinic, $P2_1/c$, $a = 14{\cdot}410$ (5), $b = 6{\cdot}828$ (1), $c = 7{\cdot}581$ (2) Å, $\beta = 102{\cdot}63$ (2)°, $V = 727{\cdot}89$ Å^3, $Z = 4$, $D_x = 1{\cdot}44$ g cm^{-3}, λ(Mo $K\alpha$) = 0·71069 Å, $\mu = 3{\cdot}8$ cm^{-1}, $F(000) = 328$, $T = 298$ K, $R = 0{\cdot}040$ for 1171 unique observed reflections with $I > \sigma(I)$. The C atoms of the butene portion, together with the S of the thiocyanate group and the N of the nitro group, form a nearly planar backbone (no atom more than 0·0055 Å from the mean plane). The nitro group is twisted 8·9° and the thiocyanate group 6·5° from the mean plane of the backbone.

Introduction. *cis*-3-Nitro-2-thiocyanato-2-butene (I) is one of a series of multiply substituted butenes prepared and investigated by KPP for biological activity. The structure was determined to confirm the stereochemistry and to examine the consequences of the steric crowding. The chemistry of the title compound will be described elsewhere.



(I)

Experimental. Pale-yellow nearly cubic single crystal, 0·40 × 0·41 × 0·42 mm, cut from a large block and wedged in a thin-walled glass capillary because epoxy cement caused decomposition of the crystal. Nicolet *R*3*m* diffractometer, graphite monochromator, unit-cell parameters by least-squares refinement of 25 reflections ($25 \le 2\theta \le 35°$), θ–2θ scans at variable rates 4·9–29·3° min^{-1}, $2\theta_{\max} = 50°$ for the range $-17 \le h \le 16$, $0 \le k \le 8$, $0 \le l \le 9$, three reflections monitored every 97 reflections with a 4% decrease in intensity over the course of data collection for which a linear correction was applied, 1571 measured intensities, empirical ellipsoidal absorption correction based on ψ scans of six strong reflections (maximum and minimum transmission factors 0·847, 0·898), 1448 unique data (not including space-group absences), $R_{\rm int} = 0{\cdot}014$, 1171 data with $I > \sigma(I)$ used for refinement. Structure by direct methods, all H atoms found on difference map; for refinement, all C—H bond lengths fixed at 0·96 Å and methyl groups refined as rigid groups with ideal geometry; anisotropic thermal parameters for all non-H atoms and fixed isotropic parameters for H atoms (20% greater than that of carrying C atom). Refined by cascade block-diagonal least squares on F with maximum $(\sin\theta)/\lambda = 0{\cdot}59$ Å^{-1}; refinement of 97 parameters

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