$$2N_2H_5^+ \cdot C_2H_2N_4O_4^{2-}$$

All H-atom parameters refined

$w = 1/[\sigma^2(F_o^2) + (0.0609P)^2 + 0.0403P]$

where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max} < 0.001$

Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters ($\mathring{A}^2$)*

$$U_{eq} = (1/3)\Sigma_i\Sigma_j U_{ij}a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	U_{eq}
C1	0.61351 (14)	0.0622 (2)	0.48545 (3)	0.0210 (2)
N1	0.53110 (13)	0.1687 (2)	0.42285 (3)	0.0224 (2)
N2	0.70608 (12)	0.2697 (2)	0.39243 (3)	0.0202 (2)
O1	0.65279 (12)	0.3642 (2)	0.33602 (3)	0.0320 (2)
O2	0.93140 (11)	0.2820 (2)	0.41425 (3)	0.0318 (2)
N4	0.4160 (2)	0.4111 (3)	0.21075 (4)	0.0286 (2)
N3	0.29882 (14)	0.2325 (2)	0.15746 (4)	0.0241 (2)
H1	0.737 (3)	−0.123 (4)	0.4865 (6)	0.033 (3)
H2	0.694 (3)	0.263 (4)	0.5103 (7)	0.032 (3)
H3	0.165 (3)	0.110 (5)	0.1693 (7)	0.042 (4)
H4	0.407 (3)	0.090 (4)	0.1386 (6)	0.035 (3)
H5	0.231 (3)	0.389 (5)	0.1287 (8)	0.055 (5)
H6	0.560 (3)	0.483 (5)	0.2009 (7)	0.051 (4)
H7	0.449 (3)	0.240 (5)	0.2352 (9)	0.057 (5)

Table 2. *Selected geometric parameters ($\mathring{A}$, °)*

C1—N1	1.459 (1)	N4—N3	1.443 (1)
C1—H1	0.97 (1)	N4—H6	0.87 (2)
C1—H2	1.01 (1)	N4—H7	0.85 (2)
C1—C1^i	1.518 (1)	N3—H3	0.92 (2)
N1—N2	1.273 (1)	N3—H4	0.92 (1)
N2—O1	1.294 (1)	N3—H5	0.91 (2)
N2—O2	1.271 (1)		
N1—C1—H1	111.6 (8)	N3—N4—H6	106 (1)
N1—C1—H2	112.3 (8)	N3—N4—H7	102 (1)
N1—C1—C1^i	107.16 (6)	H6—N4—H7	105 (2)
H1—C1—H2	106 (1)	N4—N3—H3	107.6 (9)
H1—C1—C1^i	110.9 (9)	N4—N3—H4	112.8 (8)
H2—C1—C1^i	109.4 (9)	N4—N3—H5	112 (1)
C1—N1—N2	113.64 (6)	H3—N3—H4	113 (1)
N1—N2—O1	118.48 (7)	H3—N3—H5	104 (1)
N1—N2—O2	123.65 (7)	H4—N3—H5	107 (1)
O1—N2—O2	117.85 (7)		
H1—C1—N1—N2	−56.4 (9)	H6—N4—N3—H4	42 (2)
H2—C1—N1—N2	61.9 (9)	H6—N4—N3—H5	−79 (2)
C1^i—C1—N1—N2	−178.00 (7)	H7—N4—N3—H3	58 (2)
C1—N1—N2—O1	178.90 (7)	H7—N4—N3—H4	−68 (2)
C1—N1—N2—O2	−0.9 (1)	H7—N4—N3—H5	172 (2)
H6—N4—N3—H3	167 (2)		

Symmetry code: (i) $1 - x, -y, 1 - z$.

Table 3. *Contact distances ($\mathring{A}$)*

N1$\cdots$H5^i	2.03 (2)	O1$\cdots$H7	2.42 (2)
O1$\cdots$H3^{ii}	1.95 (2)	O1$\cdots$H4^{iv}	2.55 (1)
O1$\cdots$H6^{iii}	2.32 (2)	O2$\cdots$H4^{iv}	1.91 (1)

Symmetry codes: (i) $\frac{1}{2} - x, y - \frac{1}{2}, \frac{1}{2} - z$; (ii) $\frac{1}{2} - x, \frac{1}{2} + y, \frac{1}{2} - z$; (iii) $\frac{1}{2} - x, y - \frac{1}{2}, \frac{1}{2} - z$; (iv) $\frac{1}{2} - x, \frac{1}{2} + y, \frac{1}{2} - z$.

Data collection: *CAD-4 Software* (Enraf–Nonius, 1989). Cell refinement: *CAD-4 Software*. Data reduction: *SHELXTL/PC* (Sheldrick, 1990). Program(s) used to solve structure: *SHELXTL/PC*. Program(s) used to refine structure: *SHELXL93* (Sheldrick, 1993). Molecular graphics: *SHELXTL/PC*. Software used to prepare material for publication: *Xtal3.2 BONDLA* (Dreissig, Doherty, Stewart & Hall, 1992).

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(4,6-Dichloro-1,3,5-triazin-2-ylethynyl)-dimethylamine

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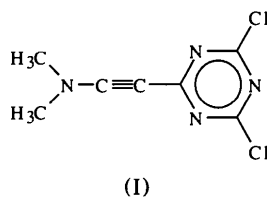
Abstract

The title compound, $C_7H_6Cl_2N_4$, is a push–pull acetylene. The minimum distance between the centers of triple bonds of 5.27 (3) $\mathring{A}$ is unsuitably long for solid-state

polymerization. The molecule shows the expected delocalization of the π electrons. The molecular geometry deviates from the chemical mirror plane perpendicular to the molecular plane.

Comment

Acetylenes containing both an electron-donor and an electron-acceptor group are called push–pull acetylenes. Their polymers are known to show conductive properties (Himbert & Brunn, 1985; Himbert, 1979; Himbert, Feustel & Jungs, 1981). The crystal structure of the title compound, (I), was determined in order to investigate its suitability for solid-state polymerization.

Fig. 1. The molecular structure of the title compound showing the atomic numbering and 50% probability displacement ellipsoids.

(I)

The crystal packing shows intermolecular interactions in two distinct directions, firstly between the Cl1 and N5 atoms of neighboring molecules related by the c glide and secondly between the Cl2 atoms of two molecules linked *via* the inversion center at $\frac{1}{2},\frac{1}{2},\frac{1}{2}$. The corresponding interatomic distances [Cl1···N5 3.127 (2) and Cl2···Cl2 3.370 (1) Å] are about 0.2 Å shorter than the sum of the respective van der Waals radii and may represent weak donor–acceptor interactions. The minimum distance between the centers of triple bonds is 5.27 (3) Å, which is unsuitably long for a polymerization reaction (Wegner, 1974; Galli, Neuenschwander & Engel, 1989).

The aromatic ring is nearly planar and the maximum deviation from the best plane defined by the six ring atoms is 0.009 (1) Å. The angle between the former and the best plane through the aminoethyne moiety (C7–Cl1) is 9.59 (8)°. The angles at the three N atoms in the aromatic ring are smaller than those at the three C atoms. This can be explained by the fact that an N-atom lone pair needs more space than a C—Cl bonding electron pair (Gillespie & Hargittai, 1991). The angles at the triple bond differ from the expected value of 180°. The C4—C7 single bond is about 0.02 Å longer and the C7≡C8 triple bond is about 0.02 Å shorter than expected values (Watson, Brammer, Orpen & Taylor, 1992). This is evidence for the expected delocalization of the π electrons in this push–pull acetylene. The molecular geometry deviates from the chemical mirror plane perpendicular to the molecular plane; the difference in bond length between Cl1—C2 and Cl2—C6 is 0.008 (2) Å and between N9—C10 and N9—Cl1 is 0.012 (3) Å, while the N1—C2—N3 angle is 128.55 (13)°, whereas the N1—C6—N5 angle is 129.4 (2)°.
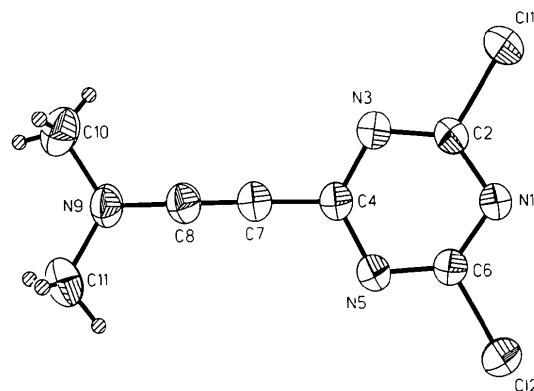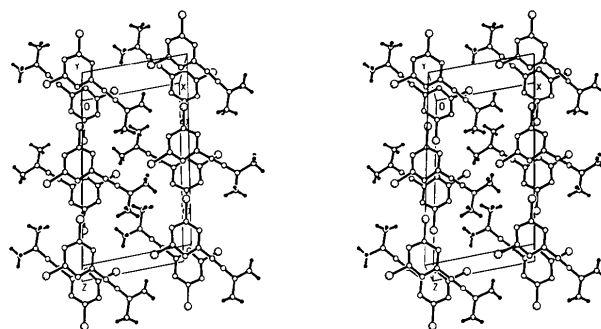
Fig. 2. The packing diagram of the title compound.

Experimental

The title compound was synthesized by reacting one equivalent of trichloro(dimethylamino)ethene with two equivalents of *n*-butyllithium. The resulting lithium dimethylaminoacetylide was immediately reacted with tributyltin chloride to form 1-tributylstannyl-2-dimethylaminoethyne. After reaction of the aminoethyne with cyanuric chloride, the title compound was obtained in 54% yield. Crystals were obtained by evaporation of a methylene chloride/hexane solution (*v/v* 1:1) at room temperature excluding moisture and light.

Crystal data

$C_7H_6Cl_2N_4$	Mo $K\alpha$ radiation
$M_r = 217.06$	$\lambda = 0.71069$ Å
Monoclinic	Cell parameters from 14
$P2_1/c$	reflections
$a = 8.481$ (2) Å	$\theta = 10.78–21.53°$
$b = 7.584$ (2) Å	$\mu = 0.644$ mm^{-1}
$c = 14.917$ (3) Å	$T = 293$ (2) K
$\beta = 100.57$ (18)°	Elongated prism
$V = 943.2$ (3) Å^3	$0.36 \times 0.18 \times 0.18$ mm
$Z = 4$	Dark yellow
$D_x = 1.529$ Mg m^{-3}	
$D_m = 1.51$ (2) Mg m^{-3}	
D_m measured by flotation	

Data collection

Enraf–Nonius CAD-4	1657 observed reflections
diffractometer	$[I > 2\sigma(I)]$

ω scans
Absorption correction:
  ψ scan (SDP; Enraf–Nonius, 1985)
  $T_{min} = 0.986$, $T_{max} = 1.000$
3410 measured reflections
2509 independent reflections

$R_{int} = 0.0130$
$\theta_{max} = 28.96°$
$h = -1 \rightarrow 11$
$k = -1 \rightarrow 10$
$l = -20 \rightarrow 20$
3 standard reflections
  frequency: 60 min
  intensity decay: 2.4%

Refinement

Refinement on F^2
$R(F) = 0.0308$
$wR(F^2) = 0.0876$
$S = 1.014$
2509 reflections
143 parameters
All H-atom parameters refined
$w = 1/[\sigma^2(F_o^2) + (0.0487P)^2 + 0.0159P]$
  where $P = \max[0, (F_o^2 + 2F_c^2)/3]$

$(\Delta/\sigma)_{max} = 0.081$
$\Delta\rho_{max} = 0.278$ e Å^{-3}
$\Delta\rho_{min} = -0.225$ e Å^{-3}
Extinction correction: SHELXL93
Extinction coefficient: 0.019 (2)
Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters (Å²)*

$$U_{eq} = (1/3)\Sigma_i\Sigma_j U_{ij}a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	U_{eq}
Cl1	0.02022 (5)	0.28215 (7)	−0.24307 (2)	0.0571 (2)
Cl2	0.33418 (5)	0.37513 (8)	−0.50554 (3)	0.0632 (2)
N1	0.16555 (14)	0.3256 (2)	−0.37948 (7)	0.0417 (3)
C2	0.0269 (2)	0.2767 (2)	−0.35773 (9)	0.0405 (3)
N3	−0.10331 (15)	0.2220 (2)	−0.41167 (8)	0.0469 (3)
C4	−0.0909 (2)	0.2142 (2)	−0.50074 (9)	0.0432 (3)
N5	0.0427 (2)	0.2626 (2)	−0.53269 (8)	0.0446 (3)
C6	0.1598 (2)	0.3141 (2)	−0.46908 (9)	0.0401 (3)
C7	−0.2214 (2)	0.1523 (3)	−0.56372 (11)	0.0558 (5)
C8	−0.3283 (2)	0.0871 (2)	−0.61796 (10)	0.0442 (4)
N9	−0.44441 (15)	0.0197 (2)	−0.67764 (9)	0.0465 (3)
C10	−0.5912 (2)	−0.0418 (3)	−0.6495 (2)	0.0601 (5)
C11	−0.4189 (3)	−0.0252 (3)	−0.76840 (12)	0.0590 (5)

Table 2. *Selected geometric parameters (Å, °)*

Cl1—C2	1.722 (2)	C4—C7	1.395 (3)
Cl2—C6	1.730 (2)	N5—C6	1.301 (3)
N1—C2	1.329 (2)	C8—C7	1.205 (3)
N1—C6	1.331 (2)	C8—N9	1.305 (3)
C2—N3	1.309 (3)	N9—C11	1.450 (2)
N3—C4	1.353 (2)	N9—C10	1.462 (3)
C4—N5	1.357 (2)		
C2—N1—C6	110.8 (2)	N5—C6—N1	129.4 (2)
N3—C2—N1	128.55 (13)	N5—C6—Cl2	115.67 (12)
N3—C2—Cl1	116.1 (2)	N1—C6—Cl2	114.9 (2)
N1—C2—Cl1	115.3 (2)	C8—C7—C4	175.1 (2)
C2—N3—C4	114.1 (2)	C7—C8—N9	178.8 (2)
N3—C4—N5	123.6 (2)	C8—N9—C11	120.4 (2)
N3—C4—C7	118.7 (2)	C8—N9—C10	120.3 (2)
N5—C4—C7	117.69 (14)	C11—N9—C10	118.3 (2)
C6—N5—C4	113.54 (13)		

Data collection: *CAD-4 Software* (Enraf–Nonius, 1989). Cell refinement: *CAD-4 Software*. Data reduction: *SDP* (Enraf–Nonius, 1985). Program(s) used to solve structure: *SHELXS86* (Sheldrick, 1985). Program(s) used to refine structure: *SHELXL93* (Sheldrick, 1993). Molecular graphics: *SHELXTL* (Sheldrick, 1990). Software used to prepare material for publication: *SHELXL93*.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates, complete geometry and torsion angles have been deposited with the IUCr (Reference: KA1174). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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1-(4-Amino-6-methyl[1,3]oxazolo[5,4-d]pyrimidin-2-yl)-1-deoxy-D-ribose 5-Benzoate Dihydrate

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Abstract

In the crystal structure of the title compound, $C_{18}H_{18}N_4O_6.2H_2O$, the [1,3]oxazolo[5,4-d]pyrimidine ring is planar and seems to possess a π-stacking interaction with the phenyl ring of the ester group.

Comment

Although the crystal structures of 2,4-dimethyl-5-(4-nitrophenyl)oxazole (Pompa, Albano, Bellon & Scatturin, 1965), 2-methyl-4-(bromomethyl)-5-(4-nitrophenyl)oxazole (Scatturin & Zannetti, 1959), 2-methyl-