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13. ABSTRACT (Maximum 200 words)

The synthesis of the energetic compound 4-[(4-nitro-1,2,5-oxadiazol-3-yl)-*NNO*-azoxyl]-1,2,5-oxadiazol-3-amine (**3**) was achieved in two steps from diaminofurazan (**1**). Compound **3** was characterized by X-ray diffraction. From the X-ray structure a bifrucated intramolecular *H*-bond between *O(2)– H(2)– N(4)* was observed. In addition, intramolecular *H*-bonding was observed between *H(1)* and *N(7)'* of an adjacent molecule. One molecule of ethanol and one-half molecule of water per molecule of **3** was observed in the crystal lattice. However, no *H*-bonding was observed between the solvent molecules and **3** in the crystal lattice. Despite the presence of solvent in the crystal lattice, **3** was found to have a high crystal density (d = 1.856 g/cm^3).

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Technical Report No. 1

SYNTHESIS AND STRUCTURE OF 4-[(4-NITRO-1,2,5-OXADIAZOL-3-YL)-*NNO-*

AZOXYL]-1,2,5-OXADIAZOL-3-AMINE

by

Alexander K. Zelenin, Edwin D. Stevens and Mark L. Trudell

Prepared for Publication

in

Structural Chemistry

University of New Orleans
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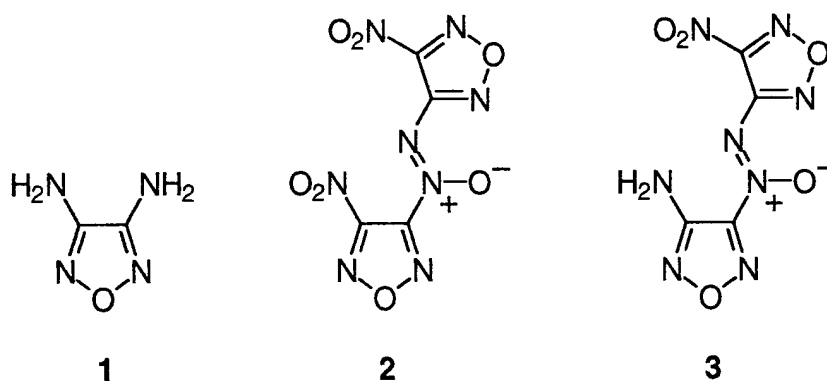New Orleans, LA

October 30, 1996

Synthesis and Structure of 4-[(4-Nitro-1,2,5-oxadiazol-3-yl)-*NNO*-azoxyl]-

1,2,5-oxadiazol-3-amine

Alexander K. Zelenin[1], Edwin D. Stevens[1] and Mark L. Trudell[1,2]

INTRODUCTION

Diaminofurazan (**1**) when used as a ballistic modifier has been shown to suppress the burn-rate and lower the pressure exponent of ammonium perchlorate composite propellants [1]. This is achieved during the combustion of the material in which a thermally-stable melon-like polymer is formed on the burning surface of the propellant [1–3]. The net result is stabilization of the combustion process.

Recently the energetic material dinitroazoxyfurazan (**2**) was prepared in these laboratories [4]. The compound **2** was found to exhibit excellent energetic and thermal properties desired of a castable material (Table I). However, the sensitivity of **2** toward impact was found to be too great to allow for the safe large-scale synthesis of this compound [5]. Impact stability studies demonstrated that **2** was nearly twofold more sensitive to detonation by impact than pentaerythrol tetranitrate (PETN) [6].

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As part of a program aimed at the development of new energetic materials to stabilize rocket motor combustion, the 4-[(4-nitro-1,2,5-oxadiazol-3-yl)-*NNO*-azoxyl]-1,2,5-oxadiazol-3-amine (**3**) [7] was envisaged as a less sensitive energetic material. It was believed that the potential for intramolecular and intermolecular hydrogen-bonding by the amino group would desensitize the material toward detonation by impact and increase the thermal stability in similar fashion to that observed for triaminotrinitrobenzene (TATB) [6,9]. Preliminary computational measurements of the energetic properties of **3** revealed that the replacement of a single nitro group in **2** with an amino group to give **3** resulted in only slightly diminished density and energetic properties (Table I) [10]. Moreover, the aminofurazan moiety present in **3** made it an attractive synthetic target for development as a new high density ballistic modifier to enhance the stability of rocket motor combustion.

Table I. Physical Properties of **2** and **3**.

	2	**3**
Formula	$C_4N_8O_7$	$C_4H_2N_8O_5$
Density (g/cm^3)	1.91 (cryst.)	1.856 (cryst. solvated)
		1.87 (calc.)
mp (°C)	111 – 112	129 – 131
Impact Sensitivity	7.8[a]	
$H_{50\%}$(cm @ 2.5 kg wt)		
Friction Sensitivity (kg)	6.4[b]	
Electrostatic Sensitivity (J)	0.45[c]	
D (mm/μsec)	9.8 (calc.)	9.4 (calc.)
P_{CJ} (kbar)	456 (calc.)	420 (calc.)

[a]Impact sensitivity for PETN, 13 cm. [b]Friction sensitivity for PETN, 6.4 kg.
[c]Electrostatic sensitivity for PETN, 1.25 J.

RESULTS AND DISCUSSION

As illustrated in Scheme 1, the synthesis of **3** was completed in two steps from readily available diaminofurazan (**1**) [11]. Oxidative dimerization of **1** by hydrogen

peroxide/ammonium persulfate in sulfuric acid at 30 °C gave the diaminoazoxyfurazan (**4**) [12]

in 30% yield. Further oxidation of one of the amino groups of **4** was attempted using published

procedures [7,12]. However, these results could not be reproduced in satisfactory yield. The

oxidation of **4** was successfully achieved in the same oxidation media employed to prepare **4**.

Treatment of **4** with hydrogen peroxide/ammonium persulfate in sulfuric acid at 50 °C afforded

a 40% yield of **3** which was obtained as a yellow crystalline material with a well-defined melting

point (Table I). The material was found to be impact sensitive under the conditions of the

simple hammer/anvil test.

Scheme 1

$$1 \xrightarrow[\substack{H_2SO_4 \\ 30-35\ °C}]{H_2O_2,\ (NH_4)_2S_2O_8} \qquad\qquad \xrightarrow[\substack{H_2SO_4 \\ 50-55\ °C}]{H_2O_2,\ (NH_4)_2S_2O_8} 3$$

4

Microcrystalline material could be obtained by recrystallization from

dichloromethane/pentane. This afforded microanalytically pure material without solvent present

in the crystal lattice; however, the crystals were too small for X-ray diffraction. X-ray quality

crystals were obtained by slow evaporation of an ethanol/water solution. From the X-ray

structure of **3** (Fig. 1) the *H(2)* of the amine moiety was observed to be *H*-bonded by both an

oxygen atom *O(2)* of the nitro group and the basic nitrogen atom *N(4)* of the azoxy linkage. In

addition to the bifrucated *H*-bond, intermolecular *H*-bonding was also observed between *H(1)*

and *N(7)'* of adjacent molecules. As seen in the packing diagram of **3** (Fig. 2) one molecule of

ethanol and one-half molecule of water were found to cocrystallize per molecule of **3**. The

solvent molecules were *H*-bonded to each other; however, no intermolecular *H*-bonding was

Table II. Crystal, Intensity Measurement and Refinement Data for Compound **3**.

|	
Empirical Formula	$C_4N_8O_5H_2 \bullet C_2H_5OH \bullet \frac{1}{2}H_2O$
Color, Habit	Yellow Needle
Crystal Dimensions, mm	$0.20 \times 0.36 \times 0.05$
Space Group	$P2_1/c$
Unit Cell Dimensions	
a, Å	10.449(4)
b, Å	5.391(2)
c, Å	18.888(6)
α, deg	90.00
β, deg	91.23(2)
γ, deg	90.00
V, Å^3	1063.7(12)
Temperature, K	110(3)
Density (calc), g/cm^3	1.856
μ (MoKα), cm^{-1}	1.56
Diffractometer	Enraf–Nonius CAD4
Monochromator	Highly Ordered Graphite
Absorption Correction	Empirical, Psi Scans
Scan Type	$\theta - 2\theta$
2θ Range, deg	2.0 - 40.0
Total Number of Reflections Measured	2104
Number of Unique Reflections	1663
Internal Agreement Factor R(F^2)	0.021
Number of Least-Squares Variables	174
Agreement Factors	
R (all data)	0.108
R$_W$ (all data)	0.222
R (F > 3σ)	0.074
R$_W$ (F > 3σ)	0.128
Goodness of Fit	3.45
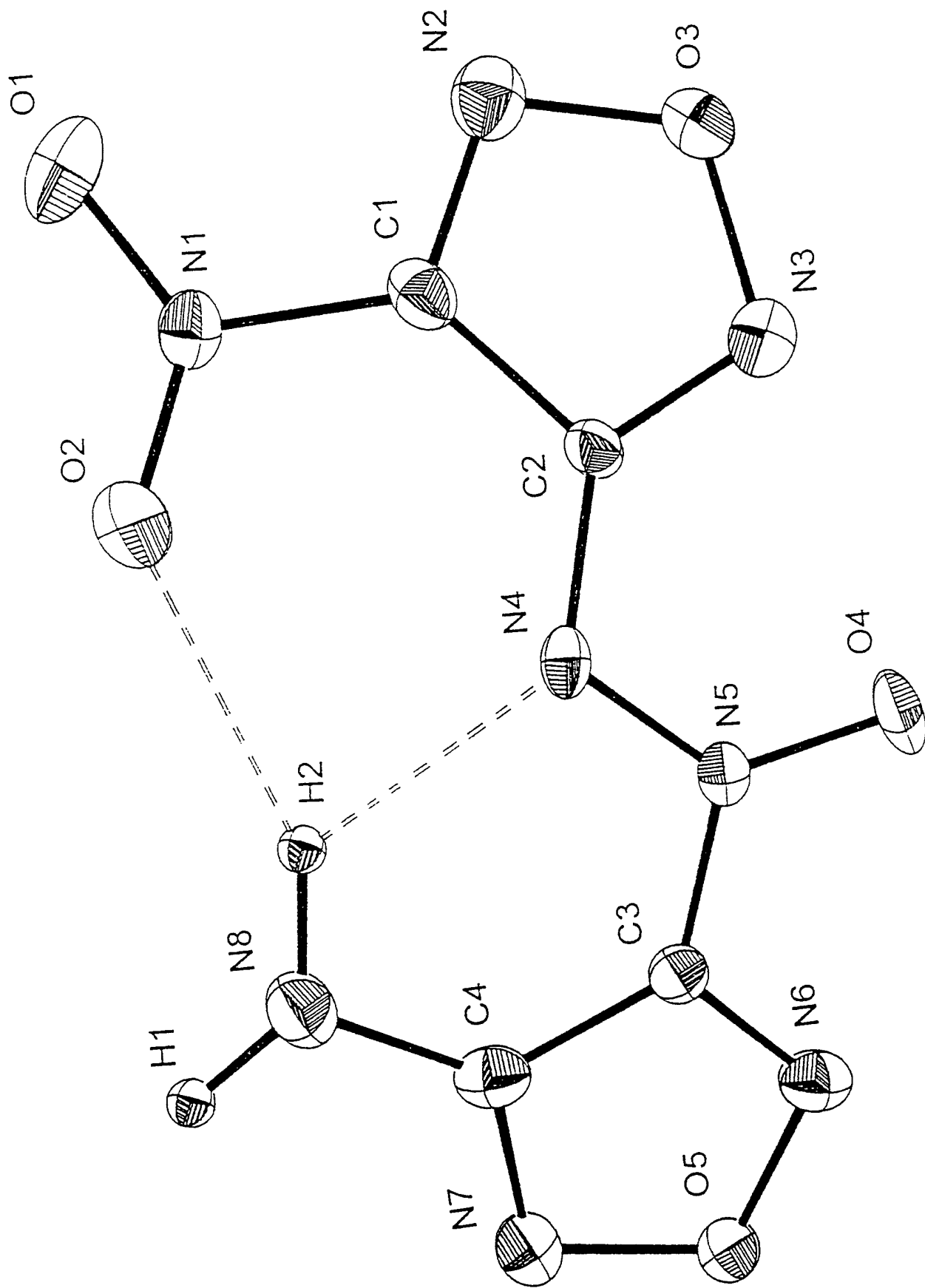
Fig. 1. Thermal ellipsoid drawing of compound **3**. Atoms are drawn at the 50% probability level.
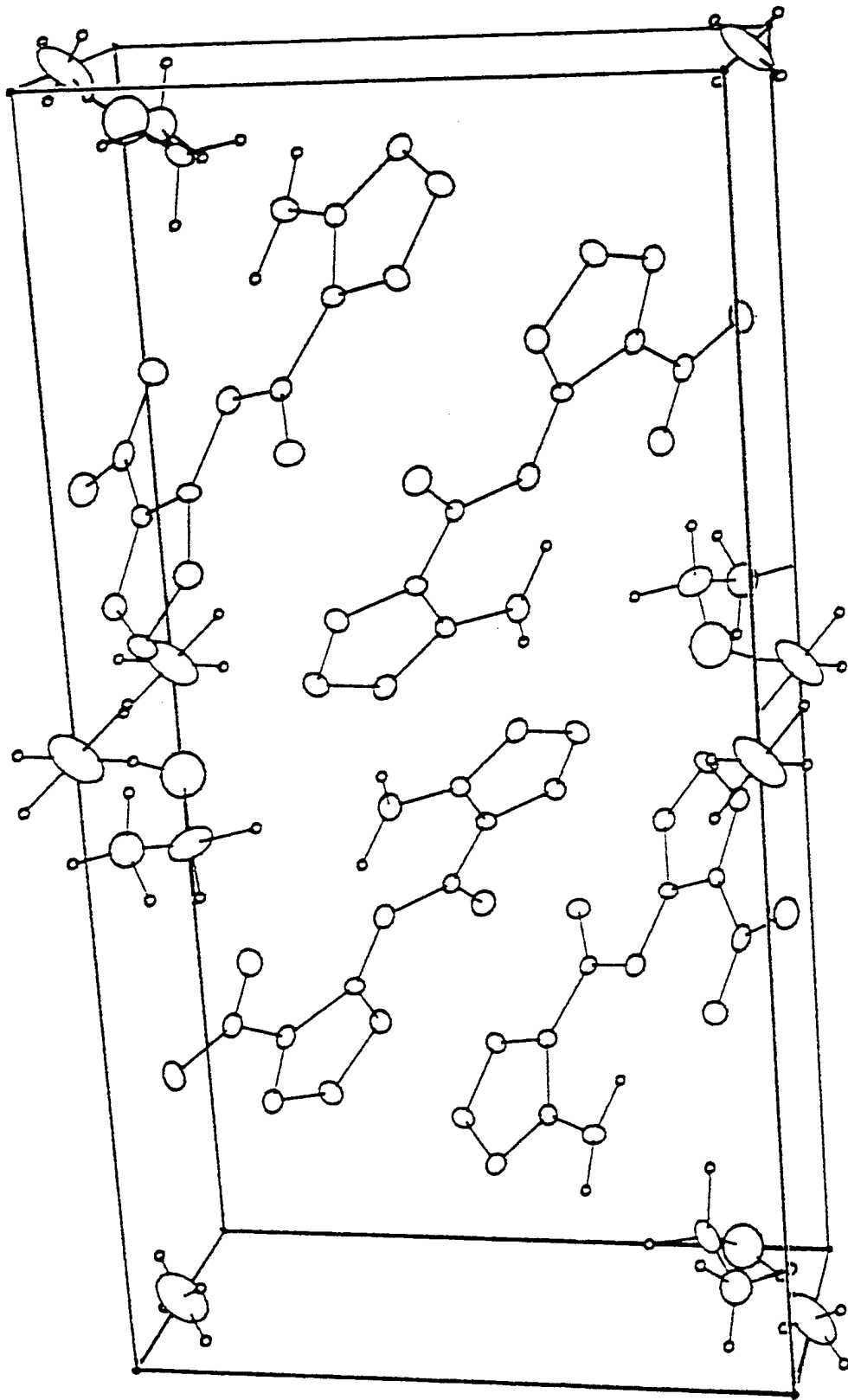
Fig. 2. Packing diagram for **3**•EtOH•½H$_2$O.

observed between **3** and the solvent molecules. Despite the presence of solvent in the crystal lattice, compound **3** was measured to have a high crystal density (d = 1.856 g/cm^3). Unfortunately, attempts to desolvate the material by drying at elevated temperatures (60 °C) under high vacuum (0.1 mm Hg) destroyed the crystallinity of the material.

SUMMARY

These results suggest that **3** is a dense material which when crystalline possesses voids in the crystal lattice which are easily occupied by extraneous solvent molecules. From these results it is possible to speculate that **3** may prove to be an excellent candidate for the formulation of energetic composite materials. Small molecules (such as ammonium perchlorate or ammonium dinitramide) or organic explosives (such as RDX or 1,3,3-trinitroazetidine) may incorporate easily into the crystal lattice voids to give a homogeneous composite material.

EXPERIMENTAL

Diaminoazoxyfurazan (4). To a mixture of hydrogen peroxide (30%, 30 g) and ammonium persulfate (25 g, 0.11 mol) at 20 °C with stirring we added a solution of **1** [11] (2.0 g, 0.023 mol) in sulfuric acid (96%, 18 g) over 20 min. The mixture was kept at 30–35 °C and stirred for 2 h. A yellow precipitate was filtered off and washed with cold water (30 mL) and with dichloromethane (30 mL). The precipitate was then recrystallized from dioxane/water (1:1) to give diaminoazoxyfurazan **7** (1.27 g, 30%), mp 245–247 °C (Ref. [12], mp 249 °C). ^{1}H NMR (DMSO–d$_6$): δ 6.95, 6.69. ^{13}C NMR (DMSO–d$_6$): δ 153.5, 151.9, 150.6, 147.7.

4-[(4-Nitro-1,2,5-oxadiazol-3-yl)-*NNO*-azoxyl]-1,2,5-oxadiazol-3-amine (3). A solution of **4** (1 g, 4.7 mmol) in concentrated sulfuric acid (12 mL) was added to a stirred mixture of hydrogen peroxide (30%, 14 mL) and ammonium persulfate (11.8 g) at room temperature. The resulting mixture was stirred at 50 °C for 22 h and added to ice-cold water (100 mL). A yellow precipitate (unreacted **4**) was filtered off and washed with dichloromethane (100 mL). The

water filtrate was extracted with dichloromethane (50 mL). The combined organic fractions were washed with water (50 mL) and dried (Na_2SO_4). The solvent was removed under vacuum and the residue was subjected to column chromatography (silica gel, pentane/dichloromethane, 1:2) to give **3** as yellow needles (0.27 g, 24%; 40% yield based on recovered starting material), mp 129–131 °C (Ref. [7], mp 131 °C). ^{1}H NMR (DMSO–d_6): δ 7.13 (br, s). ^{13}C NMR (DMSO–d_6): δ 156.5, 151.9, 150.8, 148.3.

Dinitroazoxyfurazan **2** (0.1 g, 8%), mp 111–112 °C (Ref. [4], mp 110–112 °C), was also obtained from this reaction.

Crystallographic Studies

Yellow transparent crystals suitable for data collection were obtained by slow evaporation from an ethanol/water (1:1) mixture. Selected crystals were mounted on an Enraf–Nonius CAD4 diffractometer and data was collected by using MoKα radiation at low temperature under a stream of cold nitrogen gas generated by a locally modified Enraf–Nonius low-temperature device. Cell dimensions were determined by least-squares refinement of the measured setting angles of 25 reflections with $20° < 2θ < 50°$. The structure was solved by direct methods and refined by full-matrix least-squares techniques. The nitrogen and oxygen atoms were identified by inspection of the temperature factors from a refinement in which all other atoms were assumed to be carbon atoms. The hydrogen atoms were assigned to calculated positions and included as fixed contributions in subsequent refinements. Crystal data for **3** is summarized in Table II. In addition, bond distances and angles are listed in Tables III, IV and V. The positional parameters and isotropic thermal parameters are listed in Table VI and anisotropic thermal parameters are listed in Table VII [13]. All software used in structure determination were from the SPD package [14].

Table III. Intramolecular Bond Distances (Å) for Compound 3•EtOH•½H$_2$O.

Atom 1	Atom 2	Distance[a]
O1	N1	1.195(5)
O2	N1	1.237(4)
O3	N2	1.370(5)
O3	N3	1.412(4)
O4	N5	1.241(4)
O5	N6	1.366(4)
O5	N7	1.425(5)
N1	C1	1.444(6)
N2	C1	1.294(6)
N3	C2	1.314(5)
N4	N5	1.305(5)
N4	C2	1.362(5)
N5	C3	1.405(5)
N6	C3	1.297(5)
N7	C4	1.308(5)
N8	C4	1.341(5)
C1	C2	1.446(6)
C3	C4	1.441(6)
N8	H1	0.951(3)
N8	H2	1.045(3)
C5	C6	1.40(2)
C6	O6	1.48(2)
C5[b]	H3[b]	0.94(1)
C5[b]	H4[b]	0.92(1)
C5[b]	H5[b]	0.90(1)
C6[b]	H6[b]	1.019(7)
C6[b]	H7[b]	1.013(8)
O6[b]	H8[b]	0.82(1)
H3[b]	H4[b]	1.551(5)
H3[b]	H5[b]	1.551(5)
H4[b]	H5[b]	1.551(5)
H4[b]	H9[c]	1.039(5)
H6[b]	H7[b]	1.551(5)

[a]Numbers in parentheses are estimated standard deviations in the least significant digits. [b]Atom of ethanol molecule. [c]Atom of water molecule.

Table IV. Intramolecular Bond Angles (degrees) for Compound $3 \cdot Et_3OH \cdot \frac{1}{2}H_2O$.

Atom 1	Atom 2	Atom 3	Angle (deg)[a]
N2	O3	N3	112.1(3)
N6	O5	N7	110.2(3)
O1	N1	O2	125.4(4)
O1	N1	C1	117.9(4)
O2	N1	C1	116.8(4)
O3	N2	C1	104.5(3)
O3	N3	C2	105.0(3)
N5	N4	C2	116.8(3)
O4	N5	N4	125.9(3)
O4	N5	C3	120.4(3)
N4	N5	C3	113.8(3)
O5	N6	C3	106.2(3)
O5	N7	C4	105.6(3)
N1	C1	N2	119.1(4)
N1	C1	C2	129.5(4)
N2	C1	C2	111.4(4)
N3	C2	N4	130.3(4)
N3	C2	C1	107.2(3)
N4	C2	C1	122.7(4)
N5	C3	N6	119.4(4)
N5	C3	C4	130.2(4)
N6	C3	C4	110.3(3)
N7	C4	N8	124.8(5)
N7	C4	C3	107.7(4)
N8	C4	C3	127.6(4)
C5	C6	O6	122.2(8)[b]

[a]Numbers in parentheses are estimated standard deviations in the least significant digits. [b]Atom of ethanol molecule.

Table V. Hydrogen Bond Angles (degrees) of **3•EtOH•½H₂O**.

Atom 1	Atom 2	Atom 3	Angle
C4	N8	H1	115.
C4	N8	H2	112.
H1	N8	H2	130.
C6	C5	H3	101.[a]
C6	C5	H4	104.[a]
C6	C5	H5`	105.[a]
H3	C5	H4	114.[a]
H3	C5	H5	115.[a]
H4	C5	H5	116.[a]
C5	C6	H6	120.[a]
C5	C6	H7	120.[a]
O6	C6	H6	95.[a]
O6	C6	H7	94.[a]
H6	C6	H7	100.[a]
C6	O6	H8	90.[a]

[a]Atoms of ethanol molecule.

Table VI. Atomic Positional Parameters and Isotropic Thermal Parameters for Compound 3•EtOH•½H$_2$O.[a,b]

Atom	x	y	z	B(Å^2)
O1	0.346(5)	-0.086(1)	0.7016(3)	2.8(1)
O2	0.1598(5)	-0.033(1)	0.7928(3)	2.4(1)
O3	0.1948(5)	0.521(1)	0.6246(3)	2.2(1)
O4	0.4454(5)	0.724(1)	0.7751(3)	2.2(1)
O5	0.6434(5)	0.473(1)	0.9445(2)	2.0(1)
N1	0.1155(6)	0.025(1)	0.7339(3)	1.8(1)
N2	0.1280(6)	0.308(1)	0.6390(3)	2.0(1)
N3	0.2768(6)	0.590(1)	0.6815(3)	2.1(1)
N4	0.3223(6)	0.385(1)	0.7936(3)	1.8(1)
N5	0.4162(5)	0.536(1)	0.8091(3)	1.3(1)
N6	0.5902(6)	0.591(1)	0.8870(3)	1.9(1)
N7	0.5687(6)	0.263(1)	0.9625(3)	1.9(1)
N8	0.3817(6)	0.084(1)	0.9138(3)	2.2(1)
C1	0.1671(7)	0.246(1)	0.7020(4)	1.6(1)
C2	0.2619(7)	0.418(1)	0.7296(3)	1.3(1)
C3	0.4881(6)	0.467(1)	0.8696(3)	1.4(1)
C4	0.4735(7)	0.258(1)	0.9163(4)	1.7(1)
C5[c,d]	0.902(2)	0.723(5)	0.052(1)	4.5(4)
C6[c,d]	0.844(1)	0.496(4)	0.0669(5)	9.7(5)
O6[c,d]	0.881(2)	0.263(4)	0.032(1)	8.0(5)
O7[c,e]	0.000	0.500	0.000	13.7(3)

[a]Numbers in parentheses are estimated standard deviations in the least significant digits.

[b]Anisotropically refined atoms are given in the form of the isotropic equivalent displacement parameter defined as: $B_{ISO} = \dfrac{8\pi^2}{3}\sum_{ij} u_{ij} a_i^* a_j^* \mathbf{a}_i \bullet \mathbf{a}_j$

[c]Atoms were refined isotropically.

[d]Atom of ethanol molecule.

[e]Atom of water molecule.

Table VII. Anisotropic Thermal Parameters – U's for **3•EtOH•½H₂O**.[a,b]

Atom	U(1,1)	U(2,2)	U(3,3)	U(1,2)	U(1,3)	U(2,3)
O1	0.032(3)	0.027(3)	0.047(3)	-0.014(3)	-0.001(3)	-0.008(3)
O2	0.035(3)	0.025(3)	0.033(3)	-0.005(3)	-0.001(2)	0.007(3)
O3	0.034(3)	0.025(3)	0.025(2)	-0.001(3)	-0.008(2)	0.005(2)
O4	0.038(3)	0.016(3)	0.029(3)	-0.001(3)	0.004(2)	0.011(2)
O5	0.032(3)	0.019(3)	0.023(2)	-0.002(3)	-0.006(2)	0.001(2)
N1	0.021(3)	0.015(3)	0.034(3)	-0.001(3)	0.007(3)	-0.006(3)
N2	0.023(3)	0.026(3)	0.027(3)	-0.001(3)	0.001(3)	0.000(3)
N3	0.029(3)	0.024(4)	0.028(3)	-0.007(3)	0.000(3)	0.000(3)
N4	0.024(3)	0.015(3)	0.028(3)	-0.001(3)	0.005(3)	0.002(3)
N5	0.017(3)	0.014(3)	0.017(2)	-0.002(3)	0.003(2)	0.000(3)
N6	0.025(3)	0.026(3)	0.021(3)	0.002(3)	-0.004(3)	0.000(3)
N7	0.025(3)	0.022(3)	0.025(3)	0.002(3)	0.005(3)	0.001(3)
N8	0.031(3)	0.025(3)	0.027(3)	-0.007(3)	-0.000(3)	0.013(3)
C1	0.017(3)	0.021(4)	0.021(3)	-0.000(3)	0.002(3)	0.007(3)
C2	0.023(3)	0.015(4)	0.012(3)	-0.000(3)	0.000(3)	0.001(3)
C3	0.023(4)	0.015(4)	0.015(3)	-0.000(3)	0.000(3)	-0.001(3)
C4	0.020(3)	0.028(4)	0.017(3)	0.001(4)	-0.002(3)	-0.003(3)
C6[c]	0.048(6)	0.30(2)	0.023(5)	0.064(9)	-0.009(4)	0.016(9)
O7[d]	0.128(9)	0	0.092(7)	0	-0.090(6)	0

[a]The form of the anisotropic displacement parameter is: $\exp[-2\pi^2\{h^2a^2U(1,1) + k^2b^2U(2,2) + l^2c^2U(3,3) + 2hkabU(1,2) + 2hlacU(1,3) + 2klbcU(2,3)\}]$

[b]Numbers in parentheses are estimated standard deviations in the least significant digits.

[c]Atom of ethanol molecule.

[d]Atom of water molecule.

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13. The authors have deposited the crystallographic data for **3** at the Cambridge Crystallographic Data Centre, University Chemical Laboratory, Lensfield Road, Cambridge, CB2 1EW, United Kingdom.

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