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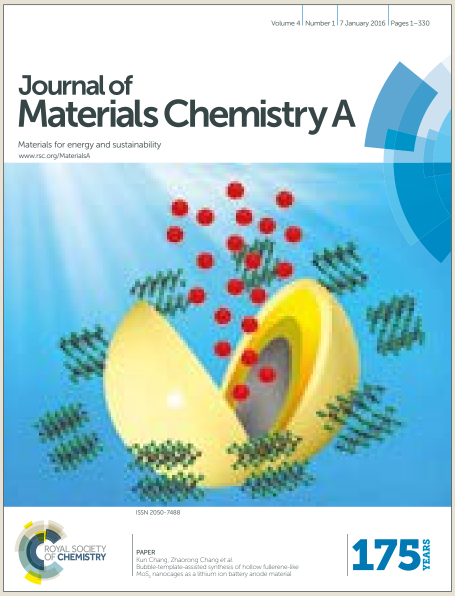

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COMMUNICATION

A Furazan-fused Pyrazole N-Oxide Via Unusual Cyclization

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6-Nitro-pyrazolo[3,4-c]furazanate 5-oxide, a new fused anion with high energy, was designed and synthesized via an unusual intramolecular cyclization reaction of **3** by using a mixture of 100% nitric acid and trifluoroacetic anhydride followed by KI reduction. The potassium (**6**) and nitrogen-rich energetic (**9** – **16**) salts were prepared, and fully characterized by IR and multinuclear NMR spectroscopy, elemental analysis, and single-crystal X-ray structuring, **6**, **9** and **16**. Hydroxylammonium salt (**10**) has excellent detonation performance but high sensitivity, while **13** and **16** have detonation velocities comparable to 1,3,5-trinitro-1,3,5-triazinane (RDX), which suggests they may have potential application as green primary or secondary explosives.

Fused nitrogen-rich compounds are attractive targets in the quest for novel high energy density materials (HEDMs).[1] Interest in such fused nitrogen-rich compounds arises from their coplanar structures and conjugated systems, which can enhance thermal stability and heat of formation.[2] Among the various fused nitrogen-rich compounds reported, several compounds, such as 3,6-dinitropyrazolo[4,3-*c*]pyrazole (A),[2a,3] 1,2,4-triazolo[3,4-*d*]-1,2,4-triazolo[3,4-*f*]-furazano[3,4-*b*]pyrazine (B),[4] 1,2,3-triazolo[4,5,-e]furazano[3,4,-*b*]pyrazine 6-oxide (C),[5] 4*H*,8*H*-bisfurazano[3,4-*b*:3',4'-*e*]pyrazine (D)[6] and their derivatives have been evaluated with relative success as HEDMs (Fig. 1). However, the limited number of fused cyclic compounds for the preparation of different and unusal molecules has long impeded their expansion in the field of energetic materials. As a result, the exploration of new strategies for the construction of fused cyclic backbones and their derivatives is of considerable interest.

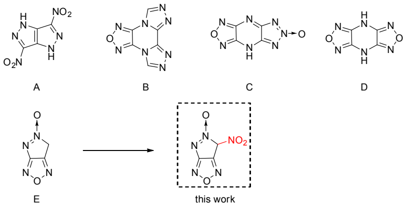

Fig. 1 Molecules with fused nitrogen-rich backbones (A-E).

While many strategies for the synthesis of fused compounds by employing cyclization reactions have been reported,[7] only rarely has the route described here been used to obtain the distinctive pyrazolo[3,4-*c*]furazan N-oxide (E) system. Its derivatives were prepared by treating the corresponding 3-amino-4-(hydroxyiminomethyl)furazan with bromine followed by dibromoisocyanurate (DBI).[8] However, these materials are not energetic molecules since the backbone is functionalized with non-energetic groups (such as methyl, chlorine or bromine). Notably, the C-6 position of pyrazolo[3,4-*c*]furazan N-oxide can be functionalized. In the course of developing new HEDMs, a judicious choice of a substituent to be introduced at this position is the nitro group, which would increase the density and improve the oxygen balance thus enhancing the detonation performance. However, it is a challenge to synthesize such a fused compound.

In this study, we developed an unusual cyclization reaction of **3** by nitration and KI reduction, which gave an interesting nitrogen rich heterocyclic fused system, 6-nitro-6*H*-pyrazolo[3,4-*c*]furazan 5-oxide, integrating the fascinating backbone (E) and the nitro group into one molecule. In addition, several salts (potassium and nitrogen-rich cations) based on 6-nitro-6*H*-pyrazolo[3,4-*c*]furazanate 5-oxide were prepared. These derivatives, **6**, **9** and **16**, are supported by molecular structures which were determined unambiguously by single crystal X-ray crystallography.

Compound **3** was synthesized in several steps from commercially available malonitrile (Scheme 1).[9] Then **3** was treated with a mixture of 100% nitric acid and trifluoroacetic anhydride (TFAA) to form the intermediate compound **5**, which likely arises from cyclization of **4** that has an adjacent nitro-nitramino structural fragment.[5] In the following step, reaction of **5** with a solution of KI in methanol[10] generates the potassium salt **6**, which is converted to **7** (but not isolated) by

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Electronic Supplementary Information (ESI) available: [Crystal refinements, calculation detail, X-ray crystallographic files in CIF format for **6**, **9** and **16** [CCDC number: 1510572, 1510571 and 1527903]. See DOI: 10.1039/x0xx00000x

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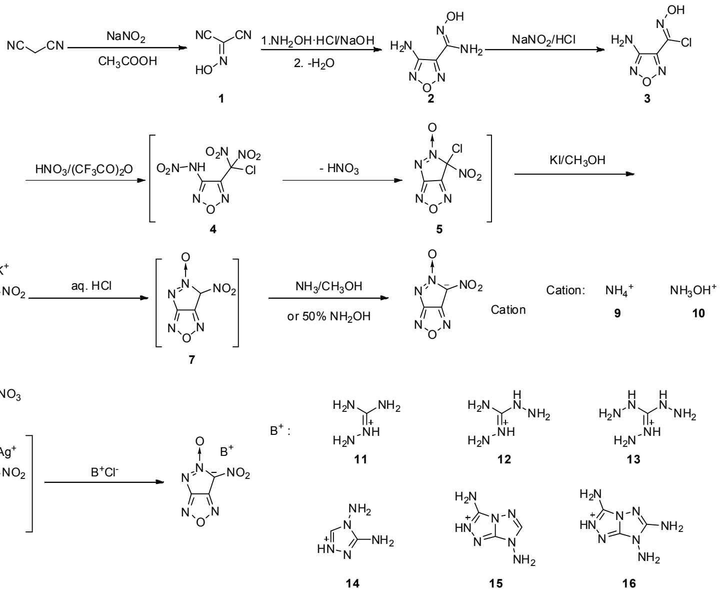
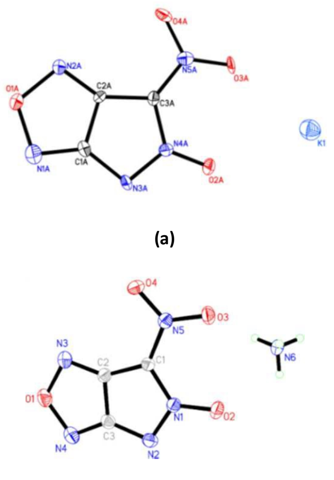


Scheme 1. Synthetic route to energetic salts **6**, **9–16**.

acidifying with dilute HCl. Subsequently, the energetic salts **9** and **10** are obtained by neutralization of **7** with methanolic ammonia or 50% aqueous hydroxylamine. In addition, the energetic salts **11–16** are synthesized by using metathesis reactions of the silver salt (**8**) with the corresponding hydrochloride salts.

The potassium salt **6** crystallizes in the monoclinic space group $P2_1/c$ with a calculated density of 2.124 g cm^{-3} at 173 K (Fig. 2a). The crystal is whole molecule disordered (See Electronic Supporting Information). The bond length of O2A-N4A is 1.223(9) Å, which falls between an N=O double (1.17 Å), and a N-O single bond (1.45 Å). In Fig. 2b the molecular structure of **9** is given. It crystallizes in the orthorhombic space group $P2_12_12_1$ with a unit cell volume of 675.72(3) Å^3. The calculated density is 1.849 g cm^{-3} at 173 K. The atoms in the anion lie approximately in the same plane with torsion angles of O(3)-N(5)-C(1)-N(1) = 0.5(3)^{o} and O(2)-N(1)-N(2)-C(3) = -179.15(18)^{o}. The bond lengths of N1-N2 and C1-N1 are 1.341(3) and 1.393(3) Å, respectively. These results show that deprotonation occurs at C1 rather than that at N2. The ammonium cation is associated with the anion through all the four hydrogen bonds (See Electronic Supporting Information).

Compound **16** crystallizes as a monohydrate in the orthorhombic space group Pccn with a cell volume of 2595.90(7) Å^3 and eight molecules in a unit cell (Fig. 3). As expected, both fused cation and anion form planar ring systems. The parameters for the fused cation are comparable to those in reported studies.[11] Crystal structure analysis also show numerous hydrogen bonds between the amino groups and oxygen atoms of the fused anions and the water molecules; the details can be found in the Electronic Supporting Information.



(a)

(b)

Fig.2 (a) X-ray crystal structure of **6**. Thermal displacement ellipsoids are set at 50% probability; (b) X-ray crystal structure of **9**. Thermal displacement ellipsoids are set at 50% probability.

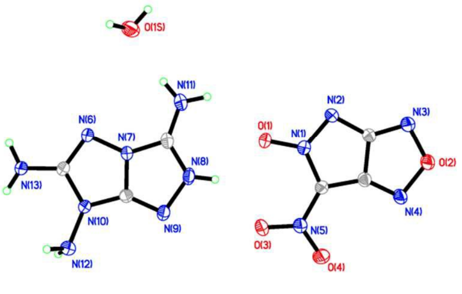


Fig. 3 X-ray crystal structure of **16**. Thermal displacement ellipsoids are set at 50% probability.

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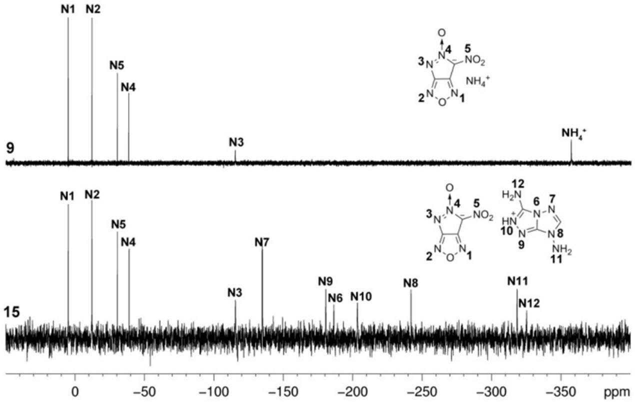

Fig. 4 ^{15}N NMR spectra of **9** and **15**.

The ^{15}N NMR spectra of **9** and **15** were measured and given in Fig. 4. The assignments are based on the literature values,[8] and ^{1}H-^{15}N HMBC corrections as well as comparison with theoretical calculations[12] using B3LYP/6-311+g(2d,p) with IEF-PCM continuum solvation models of the Gaussian 03 program. In the spectrum of **9**, five ^{15}N signals at δ = 5.13 (N1), -12.1 (N2), -30.4 (N5), -38.6 (N4), -115.2 (N3) ppm for the fused nitrogen-rich anions are seen and the ammonium cation is observed at δ = -356.9 ppm. In case of **15**, the signals for the anion are almost the same as those in **9**. For the fused cation in **15**, the correlations were observed for the CH signal at δ_H 8.79 with the ^{15}N signals at δ_N -134.9 (N7), -186.4 (N6) and -242.0 (N8) by ^{1}H-^{15}N HMBC (see Electronic Supporting Information). The signals of the two nitrogen atoms (N11: -318.4 ppm and N12: -325.4 ppm) are identified in the high field.

The physicochemical and energetic properties are summarized in Table 1. The onset decompositiontemperatures were obtained by using differential scanning calorimetry (DSC) measurements at a heating rate of 5 °C min^{-1}. While **12**-**15** melt (T_m, **12**: 106 °C; **13**: 119 °C; **14**: 151 °C; **15**: 105 °C) before decomposition, the other energetic salts (**9**-**11** and **16**) decompose at 131 to 182 °C without melting. The heats of formation were calculated using the Gaussian 03 (Revision E.01) suite of programs.[13] All of them show positive heats of formation falling between 0.76 to 2.49 kJ g^{-1} with **16** exhibiting the highest heat of formation (2.49 kJ g^{-1}) which arises from the high density and highly energetic triamino fused moiety (Table 1) These data, taken together with the measured densities (gas pycnometer), were used to calculate detonation properties with EXPLO5 (version 6.01).[14] The potassium salt (**6**) has a calculated detonation velocity of 7973 m s^{-1} and detonation pressure of 28.6 GPa. The ammonium salt (**9**) has detonation properties comparable with RDX (*vD*: 8777 m s^{-1}; *P*: 33.6 GPa), whereas the detonation properties of the hydroxylammonium salt (**10**) are superior to those of RDX and similar to those of HMX. The 3,6,7-triamino-7H-[1,2,4]triazolo[5,1-c][1,2,4]triazol-2-ium salt (**16**) also shows a detonation velocity similar to RDX; however, detonation pressures are less than that of RDX.

For initial safety testing, the impact and friction sensitivities were measured by using standard Bundesanstalt für Materialforschung (BAM) technology.[15] The salts (**6**, **9** and **10**) are sensitive with impact sensitivities of only 2-3 J and friction sensitivities between 40 N to 60 N. However, the other salts (**11**-**16**) are less impact sensitive (10 J to 20 J) than RDX; they also show lower friction sensitivity than or are comparable to RDX.

Table 1. Physicochemical and energetic properties of the new salts compared with RDX and HMX.

Mat.	T_m[a] [°C]	T_d[b] [°C]	ρ[c] [g cm^3]	Δ_fH[d] [kJ g^{-1}]	vD[e] [m s^{-1}]	P[f] [Gpa]	IS[g] [J]	FS[h] [N]
6	-	175	2.036	0.76	7973	28.6	2	40
9	-	179	1.798	1.52	8777	33.6	3	60
10	-	152	1.869	1.65	9174	39.1	2	40
11	-	163	1.693	1.53	8343	27.2	12	120
12	106	131	1.746	1.84	8741	30.0	10	120
13	119	141	1.758	2.15	8957	31.6	15	240
14	151	153	1.767	1.22	8545	29.2	18	240
15	105	164	1.763	1.92	8540	29.1	15	240
16	-	186	1.810	2.49	8741	30.8	20	360
RDX	-	204	1.800	0.32	8795	34.9	7.4	120
HMX	-	275	1.905	0.25	9144	39.2	7.4	120
$Pb(N_3)_2$	-	315	4.800	1.55	5877	33.4	2.5-4	0.1-1

[a] Melting point. [b] Thermal decomposition temperature (onset) under nitrogen gas (DSC, 5 °C/min). [c] Density was measured by gas pycnometer at room temperature. [d] Calculated heat of formation. [e] Detonation velocity. [f] Detonation pressure. [g] Impact sensitivity. [h] Friction sensitivity.

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In conclusion, the potassium salt of 6-nitro-6*H*-pyrazolo[3,4-*c*]furazan 5-oxide (**6**), containing the fused pyrazolo[3,4-*c*]furazan N-oxide ring, was synthesized by nitration of **3**, followed by reaction with KI in methanol. In addition, several nitrogen-rich energetic salts (**9**-**16**) were prepared by neutralization or metathesis reactions. The structures of **6**, **9** and **16** were confirmed by X-ray single crystal diffraction. The hydroxylammonium salt **10** exhibits excellent detonation properties with a detonation velocity of 9174 m s^{-1} and a detonation pressure of 39.1 Gpa, but is highly impact and friction sensitive. Therefore, **10** is expected to be suitable as a green primary explosive. With respect to the thermal stability, detonation performance and sensitivities, **13** and **16** exhibit promising properties. Overall, the novel synthetic strategy to access a fused pyrazole N-oxide framework by employing nitration of an adjacent amino group and chlorohydroximoyl group followed by KI reduction can be applied widely in various fields of science and technology.

Experimental Section

Caution! Although we have encountered no difficulties during preparation and handling of these compounds, they are potentially energetic materials that can explode under certain conditions such as impact, friction, or electric discharge. Mechanical actions of these energetic materials, involving scratching or scraping, must be avoided. Manipulations must be carried out by using appropriate standard safety precautions.

General methods

Unless otherwise specified, all of the solvents were reagent-grade and used without further purification. All reagents were purchased from AKSci or Alfa Aesar in analytical grade and were used as received. ^{1}H, ^{13}C, and ^{15}N NMR spectra were recorded on a 300 MHz (Bruker AVANCE 300) or 500 MHz (Bruker AVANCE 500) nuclear magnetic resonance spectrometer. Chemical shifts for ^{1}H, ^{13}C, and ^{15}N NMR spectra are reported with respect to external $(CH_3)_4Si$ (^{1}H and ^{13}C) and CH_3NO_2 (^{15}N). [D$_6$]DMSO was used as a locking solvent unless otherwise stated. Infrared (IR) spectra were recorded on an FT-IR spectrometer (Thermo Nicolet AVATAR 370) as thin films using KBr plates. Density was determined at room temperature by employing a Micromeritics AccuPyc II 1340 gas pycnometer. Melting and decomposition (onset) points were recorded on a differential scanning calorimeter (DSC, TA Instruments Q2000) at a scan rate of 5 ^{o}C min^{-1}. Elemental analyses (C, H, N) were performed on a CE-440 Elemental Analyzer. Impact and friction sensitivity measurements were made using a standard BAM Fallhammer and a BAM friction tester.

X-Ray crystallography

Suitable crystals **6** (0.26×0.13×0.03) mm^3, **9** (0.28×0.10×0.05) mm^3 and **16** (0.30×0.11×0.04) mm^3 were selected and mounted on nylon loops with paratone oil on a Bruker APEX-II CCD diffractometer. The crystals were kept at T = 173(2) K during data collection (Table S1).

Data were measured using CuK$_\alpha$ radiation (sealed tube, 40 kV, 30 mA). The total number of runs and images was based on the strategy calculation from the program **COSMO**[16]. Cell parameters were retrieved using the **SAINT**[17] software and refined using **SAINT**[17]. Data reduction was performed using the **SAINT**[17] software which corrects for Lorentz polarization. Using **Olex2**[18], the structure was solved with the XT structure solution program, using the Intrinsic Phasing solution method. The model was refined with version of **XL**[19]using Least Squares minimization. All non-hydrogen atoms were refined anisotropically.

In case of **6**, the crystal is whole molecule disordered, all except the K cation is disordered, but the disorder is in the way that the organic part interacts with the cation. Refinement of the two parts results in those labeled A to be 0.84:0.16 ratio to those atoms labeled B (Figure S1, see ESI).

Syntheses

Potassium 6-nitro-pyrazolo[3,4-*c*]furazanate 5-oxide (6)

Compound **3**[8] (1.0 g, 6.2 mmol) in dichloromethane (20 mL) was added slowly to a cooled and well-stirred mixture of 100% nitric acid (4 mL) and trifluoroacetic acid anhydride (7 mL) at 0 °C. The mixture was allowed to warm to ambient temperature over a period of 1 h. After stirring for 3 h, the green solution was poured onto crushed ice (60 g) and the solution was extracted with dichloromethane (20 mL × 3). The combined organic phases were washed with water and brine, dried over magnesium sulfate and filtered. The filtrate was concentrated in vacuo to give a colorless oil (**5**) that was dissolved in methanol (5 mL), and a solution of potassium iodide (0.01 mol) in methanol (30 mL) was added. The mixture was stirred at ambient temperature for 12 h and the yellow precipitate (**6**) was filtered, washed with cold water (50 mL), methanol (15 mL) and diethyl ether (15 mL) and dried in air.

6: yield: 22%. T_d: 175 ^{o}C. ^{13}C NMR: δ 156.9, 139.8, 123.9 ppm. IR (KBr): $\tilde{\nu}$ 1660, 1541, 1499, 1486, 1424, 1394, 1375, 1300, 1181, 1155, 1105, 1076, 1053, 983, 972, 820, 733, 672, 593 cm^{-1}. Elemental analysis for $C_3KN_5O_4$ (209.16): Calcd C 17.23, H 0, N 33.48 %. Found: C 17.12, H 0.23, N 33.11 %. *IS*: 2 J. *FS*: 40 N.

Ammonium 6-nitro-pyrazolo[3,4-*c*]furazanate 5-oxide (9): Compound **6** (0.42 g, 2.0 mmol) was dissolved in 2 M hydrochloric acid (10 mL). The solution was stirred for 30 minutes and extracted with diethyl ether (15 mL × 3). The organic phase was dried over anhydrous sodium sulfate. After removing sodium sulfate, the filtrate was removed by air. The residue was dissolved in acetonitrile (5 mL), and an equivalent amount of methanolic ammonia was added with vigorous stirring. The solvent was removed under reduced pressure. The crude product was recrystallized from acetonitrile and diethyl ether (2 mL: 2mL), **9** (0.23 g, yield: 61%) was obtained as yellow crystals. T_d: 179 ^{o}C. ^{1}H NMR: δ 7.09 (s, 4H) ppm. ^{13}C NMR: δ 156.9, 139.8, 123.8 ppm. ^{15}N NMR: δ 5.1, -12.1, -30.4, -38.6, -115.2, -356.9 ppm. IR (KBr): $\tilde{\nu}$ 3268, 1649, 1509, 1419, 1390, 1341, 1287, 1185, 1160, 1107, 1077, 1052, 982, 968, 824, 731, 670, 591 cm^{-1}. Elemental analysis for $C_3H_4N_6O_4$

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(188.10): Calcd C 19.16, H 2.14, N 44.68 %. Found: C 19.36, H 2.42, N 42.60 %. *IS*: 3 J. *FS*: 60 N.

Hydroxylammonium 6-nitro-pyrazolo[3,4-*c*]furazanate 5-oxide (10): Compound **6** (0.42 g, 2.0 mmol) was dissolved in 2 M HCl (20 mL) hydrochloric acid. The solution was stirred for 30 minutes and extracted with diethyl ether (15 mL × 3).The organic phase was dried over anhydrous sodium sulfate. After removing sodium sulfate, the filtrate was removed by air. The residue was dissolved in acetonitrile (5 mL), and an equivalent amount of 50% aqueous hydroxylamine was added with vigorous stirring. The solvent was then removed under reduced pressure. The crude product was recrystallized from acetonitrile and diethyl ether (2 mL: 2mL), **10** (0.22 g, yield: 53%) was obtained as yellow crystals. T_d: 152 °C. ^{1}H NMR: δ 7.15 (s, 4H) ppm. ^{13}C NMR: δ 157.0, 140.0, 124.0 ppm. IR (KBr): $\tilde{\nu}$ 3265, 1686, 1650, 1508, 1422, 1391, 1350, 1289, 1185, 1160, 1105, 1077, 982, 968, 840, 968, 840, 823, 801, 750, 731, 692, 669, 591 cm^{-1}. Elemental analysis for $C_3H_4N_6O_5$ (204.10): Calcd C 17.65, H 1.98, N 41.18 %. Found: C 17.64, H 2.18, N 41.19 %. *IS*: 2 J. *FS*: 40 N.

General procedure for preparing 11-16. The potassium salt **5** (0.50 g, 2.39 mmol) was dissolved in water (30 mL); then silver nitrate (0.41 g, 2.39 mmol) was added. The reaction mixture was stirred for 2 h at room temperature. The silver salt **8** was obtained by filtration and washed with water (20 mL); then **8** was suspended in water (40 mL), and a equivalent of the corresponding hydrochloride salt was added. The temperature of the reaction mixture was increased to 60 °C and it was stirred for 4 h. After removing the insoluble solids, the filtration was concentrated to give the final products (**11**-**16**).

Aminoguanidinium 6-nitro-pyrazolo[3,4-*c*]furazanate 5-oxide (11): yellow crystal, yield: 86%. T_d: 163 °C. ^{1}H NMR: δ 8.52 (s, 1H), 7.22 (s, 2H), 6.69 (s, 2H), 4.66 (s, 2H) ppm. ^{13}C NMR: δ 158.7, 156.9, 139.8, 123.8 ppm. IR (KBr): $\tilde{\nu}$ 3455, 3407, 3373, 3227, 1667, 1506, 1438, 1393, 1353, 1296, 1177, 1161, 1072, 979, 942, 821, 799, 729, 671, 593 cm^{-1}. Elemental analysis for $C_4H_7N_9O_4$ (245.16): Calcd C 19.60, H 2.88, N 51.42 %. Found: C 19.60, H 2.71, N 51.47 %. *IS*: 12 J. *FS*: 120 N.

Diaminoguanidinium 6-nitro-pyrazolo[3,4-*c*]furazanate 5-oxide (12): yellow solid, yield: 88%. T_m: 106 °C. T_d: 131 °C. ^{1}H NMR: δ 8.52 (s, 2H), 7.12 (s, 2H), 4.57 (s, 4H) ppm. ^{13}C NMR: δ 159.7, 156.9, 139.8, 123.8 ppm. IR (KBr): $\tilde{\nu}$ 3449, 3383, 3307, 1678, 1642, 1590, 1501, 1424, 1389, 1348, 1283, 1178, 1151, 1102, 1078, 1043, 980, 968, 925, 846, 823, 800, 729, 669, 594 cm^{-1}. Elemental analysis for $C_4H_8N_{10}O_4$ (260.17): Calcd C 18.47, H 3.10, N 53.84 %. Found: C 18.45, H 2.95, N 53.94 %. *IS*: 10 J. *FS*: 120 N.

Triaminoguanidinium 6-nitro-pyrazolo[3,4-*c*]furazanate 5-oxide (13): yellow solid, yield: 87%. T_m: 119 °C. T_d: 141 °C. ^{1}H NMR: δ 8.58 (s, 3H), 4.48 (s, 6H) ppm. ^{13}C NMR: δ 159.0, 156.9, 139.8, 123.8 ppm. IR (KBr): $\tilde{\nu}$ 3360, 3319, 3212, 1684, 1530, 1500, 1432, 1396, 1364, 1293, 1180, 1160, 1132, 1103, 1065, 978, 965, 845, 823, 803, 731, 669, 638, 591 cm^{-1}. Elemental analysis for $C_4H_9N_{11}O_4$ (275.19): Calcd C 17.46, H 3.30, N 55.99 %. Found: C 17.46, H 3.11, N 56.05 %. *IS*: 15 J. *FS*: 240 N.

3,4-Diamino-1,2,4-triazolinium 6-nitro-pyrazolo[3,4-*c*]furazanate 5-oxide monohydrate (14): yellow crystal, yield: 85%. T_m: 151 °C. T_d: 153 °C. ^{1}H NMR: δ 13.78 (br), 8.42 (s, 1H), 8.16 (s, 2H), 6.05 (s, 2H) ppm. ^{13}C NMR: δ 156.9, 150.7, 141.8, 139.8, 123.9 ppm. IR (KBr): $\tilde{\nu}$ 3589, 3482, 3341, 3222, 1694, 1653, 1634, 1568, 1534, 1500, 1433, 1391, 1366, 1292, 1208, 1183, 1157, 1105, 1074, 981, 949, 860, 841, 821, 799, 734, 669, 595, 546 cm^{-1}. Elemental analysis for $C_5H_8N_{10}O_5$ (288.18): Calcd C 20.84, H 2.80, N 48.60 %. Found: C 20.99, H 2.69, N 49.08 %. *IS*: 18 J. *FS*: 240 N.

3,7-diamino-7*H*-[1,2,4]triazolo[4,3-b][1,2,4]triazol-2-ium 6-nitro-pyrazolo[3,4-*c*]furazanate 5-oxide monohydrate (15): brown crystal, yield: 89%. T_m: 105 °C. T_d: 164 °C. ^{1}H NMR: δ 13.97 (br), 8.79 (s, 1H), 8.56 (s, 2H), 6.24 (s, 2H) ppm. ^{13}C NMR: δ 156.9, 152.6, 147.7, 142.2, 139.8, 123.9 ppm. IR (KBr): $\tilde{\nu}$ 3555, 3402, 3263, 1697, 1655, 1619, 1545, 1529, 1499, 1423, 1394, 1353, 1297, 1160, 1104, 1079, 1003, 980, 969, 821, 733, 669 cm^{-1}. Elemental analysis for $C_6H_8N_{12}O_5$ (328.21): Calcd C 21.96, H 2.46, N 51.21 %. Found: C 21.78, H 2.32, N 50.88 %. *IS*: 15 J. *FS*: 240 N.

3,6,7-Triamino-7H-[1,2,4]triazolo[5,1-*c*][1,2,4]triazol-2-ium 6-nitro-pyrazolo[3,4-*c*]furazanate 5-oxide (16): light-yellow crystal, yield: 86%. T_d: 186 °C. ^{1}H NMR: δ 13.29 (br), 8.18 (s, 2H), 7.21 (s, 2H), 5.75 (s, 2H) ppm. ^{13}C NMR: δ 160.1, 156.9, 147.4, 141.1, 139.8, 123.8 ppm. IR (KBr): $\tilde{\nu}$ 3394, 3321, 3256, 3177, 1699, 1653, 1617, 1582, 1449, 1433, 1393, 1376, 1323, 1298, 1162, 1104, 1074, 981, 841, 826, 596 cm^{-1}. Elemental analysis for $C_6H_7N_{13}O_4$ (325.20): Calcd C 22.16, H 2.17, N 55.99 %. Found: C 22.17, H 2.23, N 55.72 %. *IS*: 20 J. *FS*: 360 N.

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