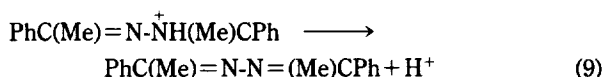
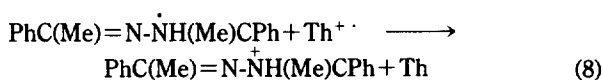
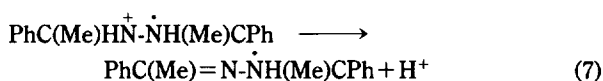
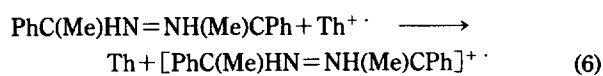
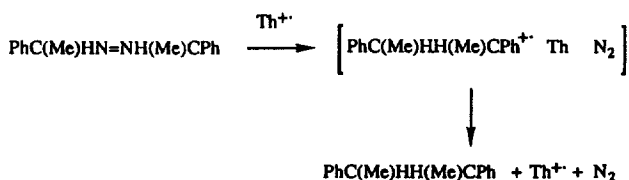


pound. A possible mechanism for the formation of the azine is shown in Eq. (6)-(9).



The formation of the thianthrene 5-oxide (ThO) can be rationalized by hydrolysis of either some of  $\text{Th}^{+\cdot}$  by incompletely dried solvent during the course of reaction, or unused  $\text{Th}^{+\cdot}$  during workup.<sup>10</sup>

No evidence for ethylbenzene was found from the  $\alpha$ -phenylethyl radical disproportionation reaction or from hydrogen abstraction from MeCN. When the reaction of  $\text{Th}^{+\cdot}$  with *meso*-ABPE was carried out in the presence of  $\text{BrCCl}_3$  in MeCN, as we did earlier with AA, the formation of DBP was not stopped. Therefore, we can conclude that DPB is probably not formed in the oxidative reactions by dimerization of  $\alpha$ -phenylethyl radical. Generally, radical is destined to abstract hydrogen atom from MeCN rather than to dimerize in the absence of a competing reaction.<sup>11</sup> The mechanism for the formation of DPB is rationalized in Scheme 2, which is very similar to that of formation of AdAd by  $\text{Th}^{+\cdot}$ .<sup>11</sup> DPB may have arisen from the DPB cation radical  $[\text{PhC(Me)HH}(\text{Me})\text{CPh}]^{+\cdot}$ , formed by a cage recombination between  $\alpha$ -phenylethyl cation and  $\alpha$ -phenylethyl radical, rather than the coupling between two  $\alpha$ -phenylethyl radicals. Conversion of DPB cation radical into DPB would have to occur by electron-transfer reaction from  $\text{Th}^{+\cdot}$  within solvent cage. In that case,  $\text{Th}^{+\cdot}$  would have served as a catalyst for the formation of DPB from *meso*-ABPE. It is interesting to compare the yield of AdAd and DPB from oxidative decomposition of corresponding azoalkane  $\text{Th}^{+\cdot}$ . Whereas 2.5% of AdAd was obtained from oxidation of AA, 23.6% of DPB was formed in the oxidative of *meso*-ABPE by  $\text{Th}^{+\cdot}$ . In the oxidation of *meso*-ABPE with  $\text{Th}^{+\cdot}$ ,  $\alpha$ -phenylethyl radical would not survive so long enough to be reduced to cation as tertiary adamantyl radical. Therefore, relatively lots of  $\alpha$ -phenylethyl radical would recombine with  $\alpha$ -phenylethyl cation to form a DPB without further oxidation in the solvent cage. *Meso*-ABPE gave 21.8% of *meso* and 1.8% of non-*meso*-DPB, indicating that some changes in orientations (by out-of-plane rotation) of the cations and radicals are occurring in these original cages prior to combination between  $\alpha$ -phenylethylcation and



**Scheme 2.** Possible reaction pathways for the formation of 2,3-diphenylbutane.

$\alpha$ -phenylethyl radical.

In conclusion, the reaction of  $\text{Th}^{+\cdot}$  with *meso*-ABPE, possessing one  $\alpha$  hydrogen, in acetonitrile follows not only the carbocationic route but also undergoes tautomerization to its hydrazone, and no oxidative cycloaddition was observed.

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- Caution:**  $\text{Th}^{+\cdot}\text{ClO}_4^-$  is explosive. It should be prepared in small quantities only and used soon after preparation. Sintered glass should not be used for filtration.
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## The Crystal Structure and Magnetic Properties of Triethylenediaminenickel(II)-Bis(maleonitriledithiolato)nickelate(II); $[\text{Ni}(\text{C}_2\text{H}_5\text{N}_2)_3]\text{Ni}(\text{C}_4\text{N}_2\text{S}_2)_2$

Chulmin Keum, Chonhan Kim, Chulsung Kim,  
Hyontae Kwak, Moonhee Kwon, and Hae Namgung†

Department of Chemical Education, Kookmin University,  
Seoul 136-702

†Department of Physic Education, Kookmin University,  
136-702

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Bidentate dithiolate ligands form very well square planar complexes with Ni-triad ions of different oxidation states,

**Table 1.** Experiment Data for the X-ray Diffraction Study

$a=8.719(3)$ Å	Crystal	= Red, Needle
$b=9.556(3)$ Å	Formula	= $\text{Ni}_2\text{C}_{14}\text{H}_{24}\text{N}_{10}\text{S}_4$
$c=16.279(4)$ Å	Space Group	= $\text{P}\bar{1}$ (No=2)
$\alpha=85.74(2)^\circ$	$Z$	= 2
$\beta=99.38(2)^\circ$	Mol. Wt.	= 578.09
$\gamma=117.14(2)^\circ$	$D_c$	= $1.612 \text{ g cm}^{-3}$
$V=1190.8 \text{ cm}^3$	$\mu$	= $19.5 \text{ cm}^{-1}$
	$F_{(000)}$	= 596
Radiation		= Mo-K $\alpha$ , 0.7107 Å
Monochromator		= Incident beam, Graphite
Mode		= $\theta/2\theta$
$2\theta$ range ( $^\circ$ )		= $2.50^\circ$
HKL ranges		= H -10 to 10 K 0 to 10 L -19 to 19
Correction		= Lorentz, Polarisation, Linear decay (averaging 1.00163 on $I$ )
Reflection		= 4344 total = 4187 unique = 3418 used with $I > 3.0 \sigma(I)$
Parameter refined		= 376
R, wR, R (all)		= 0.046, 0.065, 0.071
Maximum shift e.s.d		= 0.03
Scale factor (final)		= 0.297
Goodness of fit		= 1.76
$\Delta\rho$		= $0.611 \text{ e}\text{\AA}^{-3}$

whose solid state properties exhibit widespread electrical behavior from insulating through semiconducting to metallic.<sup>1</sup> The high metallic conductive complex  $\text{Li}_{0.75}[\text{Pt}(\text{mnt})_2] \cdot 2\text{H}_2\text{O}$  ( $\text{mnt}=1,2$ -Dicyanoethylene-1,2-dithiolate) has an one-dimensional columnar structure similar to those of the partially oxidized tetracyanoplatinate and bis (oxalato)-platinate complexes.<sup>2</sup> The semiconducting complex of  $(\text{C}_2\text{H}_5)_4\text{N} \cdot [\text{Ni}(\text{mnt})_2]$  also shows the one-dimensional stacks of coplanar overlapped diadic anion unit.<sup>3</sup> The electrically insulating complex of  $[(\text{C}_4\text{H}_9)_4\text{N}]_2 \cdot [\text{Ni}(\text{mnt})_2]$  is isomorphous with  $\text{Co}^{2+}$  and  $\text{Cu}^{2+}$  com-

**Table 2.** Atomic Coordinates and equivalent Isotropic Thermal Parameters of Nonhydrogen Atoms

Atom	X	Y	Z	B ( $\text{\AA}^2$ )
Ni-1	0.000	0.000	0.000	4.10(3)
S1	-0.0841(3)	0.1288(2)	-0.0975(1)	4.82(5)
S2	0.1940(3)	0.2146(2)	0.0627(1)	5.08(5)
C1	0.0472(9)	0.3233(8)	-0.0674(5)	4.3(2)
C2	0.1667(9)	0.3583(8)	0.0015(5)	4.4(2)
C3	0.019(1)	0.4414(9)	-0.1179(5)	5.0(2)
C4	0.278(1)	0.5172(9)	0.0269(5)	4.9(2)
N1	-0.006(1)	0.5345(8)	-0.1582(5)	6.9(2)
N2	0.369(1)	0.6436(8)	0.0492(5)	6.4(2)
Ni-2	0.500	0.000	0.500	4.11(3)
S3	0.7128(3)	0.1288(2)	0.5973(1)	4.79(5)
S4	0.5207(3)	0.2146(2)	0.4372(1)	5.06(5)
C5	0.7767(9)	0.3230(8)	0.5676(5)	4.2(2)
C6	0.6917(9)	0.3585(8)	0.4982(5)	4.3(2)
C7	0.922(1)	0.4414(8)	0.6177(5)	4.9(2)
C8	0.740(1)	0.5177(8)	0.4731(5)	4.8(2)
N3	1.040(1)	0.5352(9)	0.6582(5)	6.8(2)
N4	0.775(1)	0.6442(7)	0.4510(5)	6.4(2)
Ni-3	0.7229(1)	-0.05429(9)	0.25003(6)	3.44(2)
N5	0.6945(8)	-0.0377(7)	0.1199(4)	5.1(2)
N6	0.6367(9)	0.1217(7)	0.2368(5)	6.2(2)
N7	0.7673(8)	-0.0378(7)	0.3793(4)	5.1(2)
N8	0.9846(9)	0.1212(8)	0.2631(5)	6.2(2)
N9	0.7870(8)	-0.2431(7)	0.2542(4)	5.2(2)
N10	0.4694(8)	-0.2436(8)	0.2459(4)	5.3(2)
C9	0.676(2)	0.099(1)	0.0950(7)	8.6(3)
C10	0.585(1)	0.137(1)	0.1473(7)	7.8(3)
C11	0.922(2)	0.099(1)	0.4047(7)	8.7(4)
C12	1.053(1)	0.138(1)	0.3512(7)	7.9(3)
C13	0.627(1)	-0.3892(9)	0.2311(7)	6.9(3)
C14	0.484(1)	-0.389(1)	0.2695(7)	6.9(3)

Anisotropically refined atoms are given in the form of the isotropic equivalent displacement parameter defined as:  $(4/3) \times [a^2 \times B(1, 1) + b^2 \times B(2, 2) + c^2 \times B(3, 3) + ab(\cos \gamma) \times B(1, 2) + ac(\cos \beta) \times B(1, 3) + bc(\cos \alpha) \times B(2, 3)]$