STRUCTURAL SCIENCE
CRYSTAL ENGINEERING
MATERIALS

Volume 77 (2021)

Supporting information for article:

The crystal structure and thermal decomposition kinetics of cis-hexanitrostilbene

Yunzhang Liu, Lizhen Chen, Jianlong Wang, Jun Chen, Jingqi Wang and Hongxia Pan

S1. FT-IR and NMR Spectra

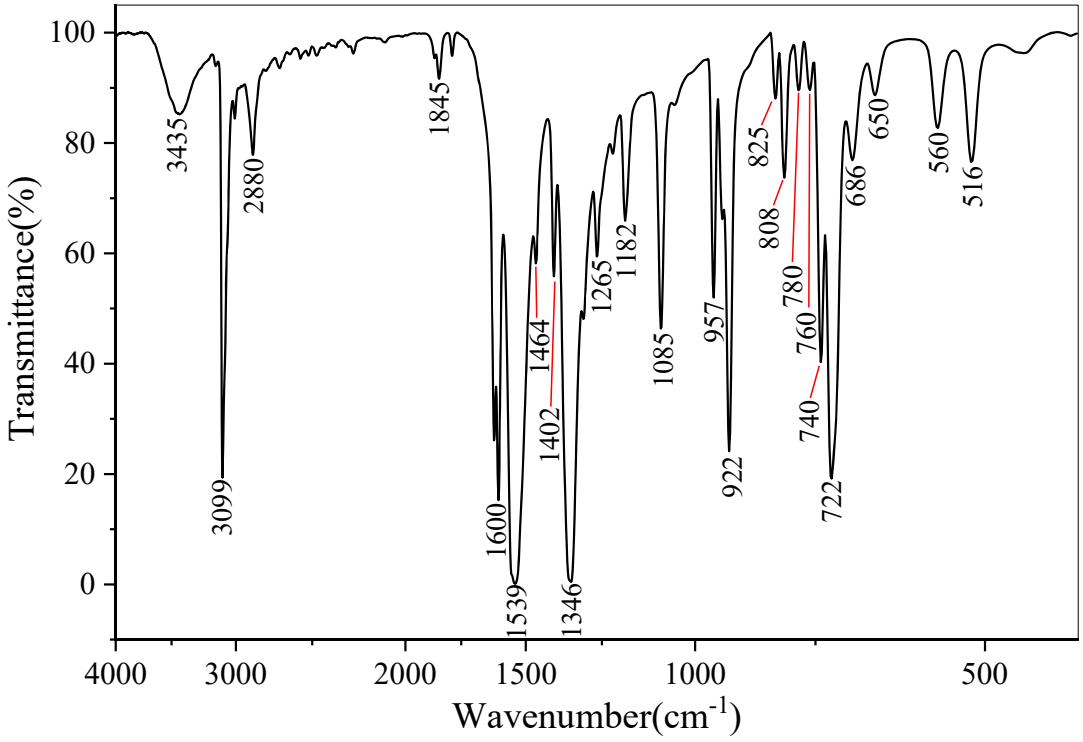

Figure S1 FT-IR spectra for **Substance 1** (Trans-HNS)

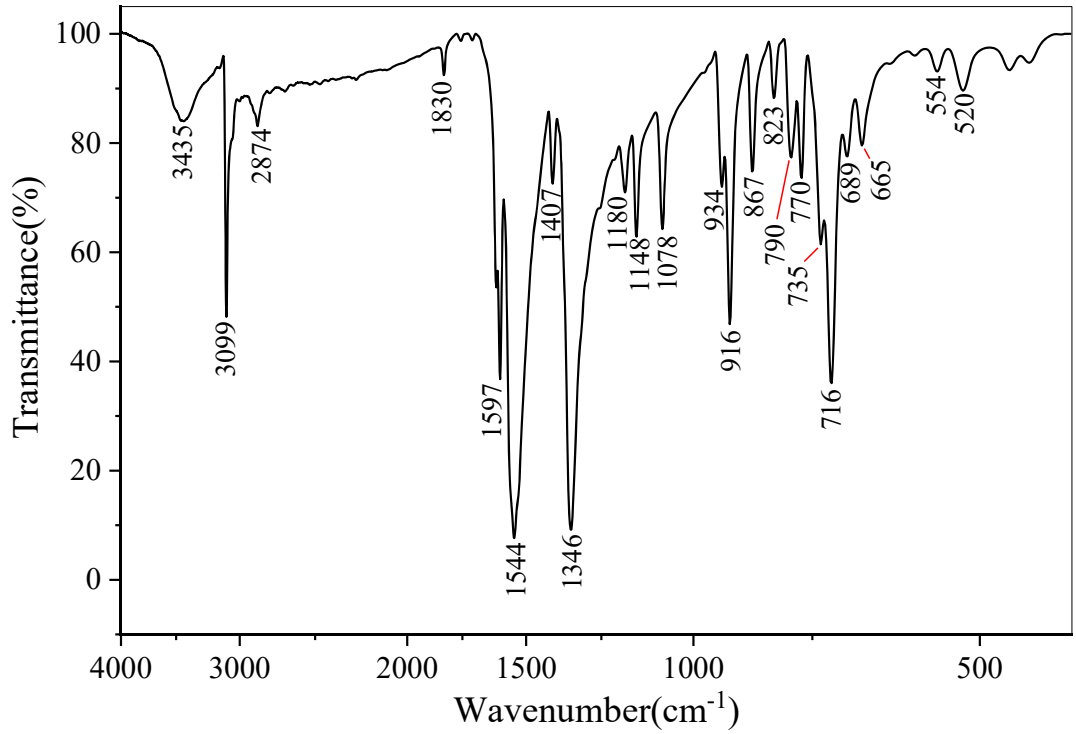

Figure S2 FT-IR spectra for **Substance 2** (Cis-HNS)

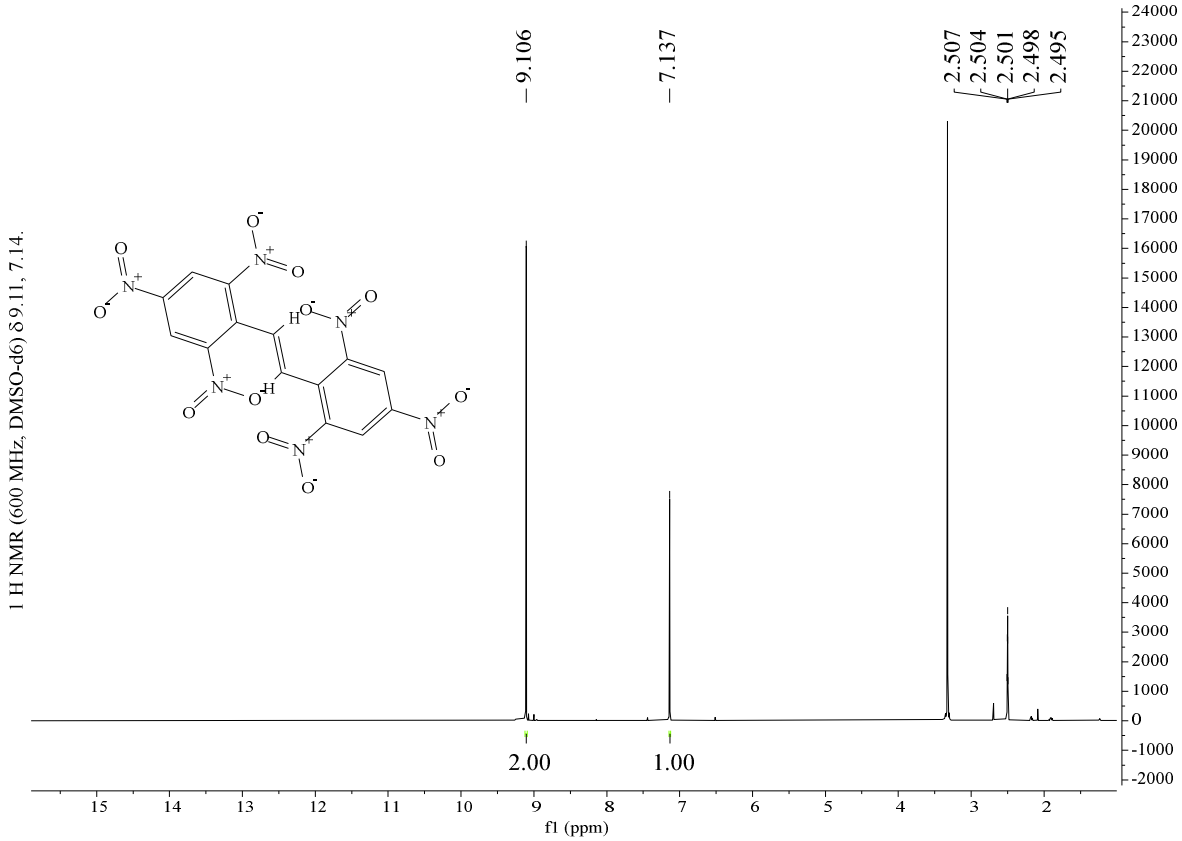

Figure S3 ^{1}H NMR spectra for **Substance 1** (Trans-HNS)

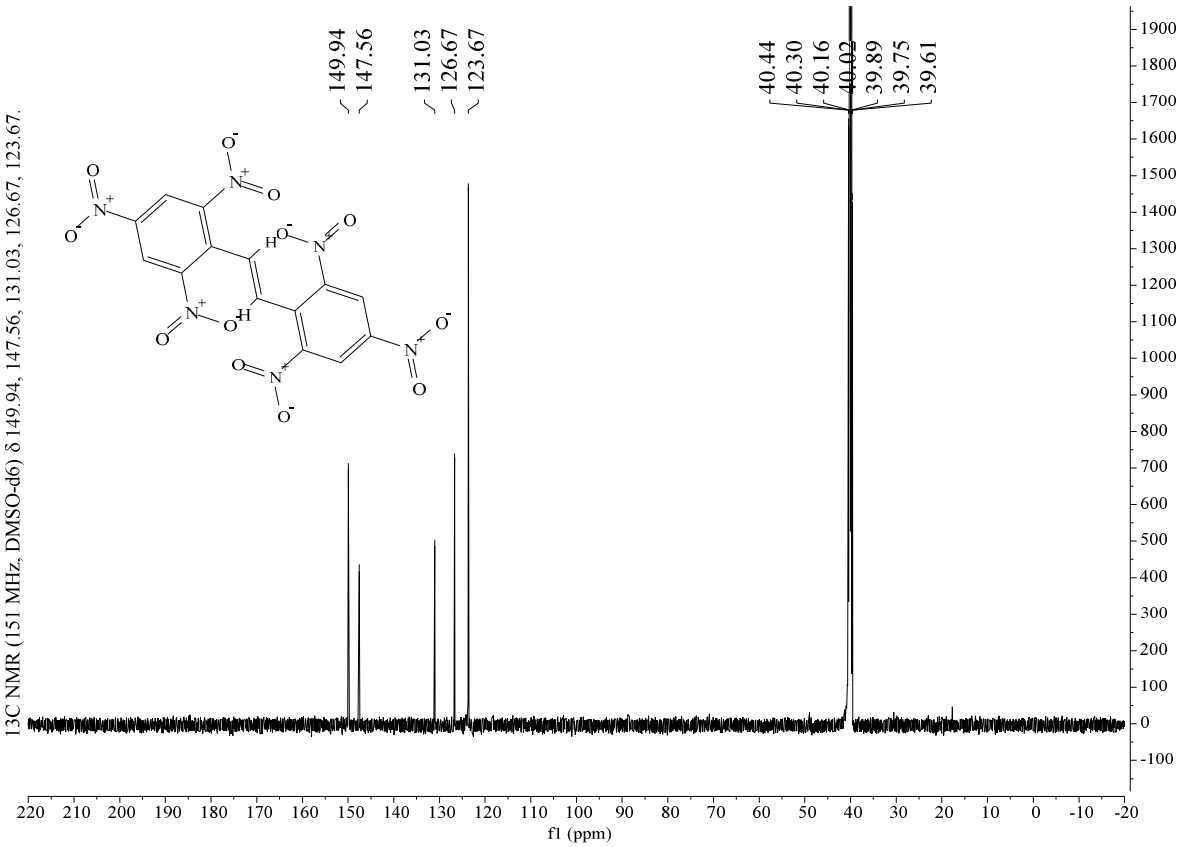

Figure S4 ^{13}C NMR spectra for **Substance 1** (Trans-HNS)

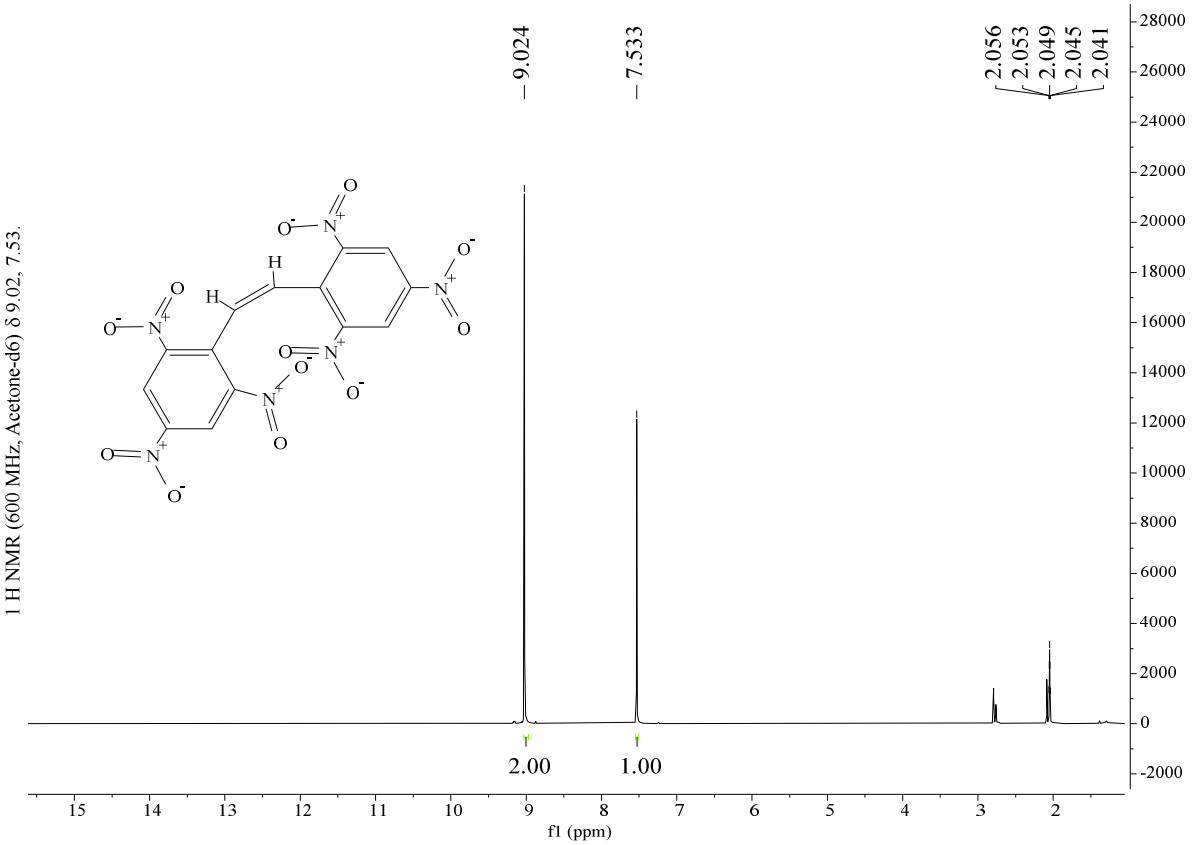

Figure S5 1H NMR spectra for **Substance 2** (Cis-HNS)

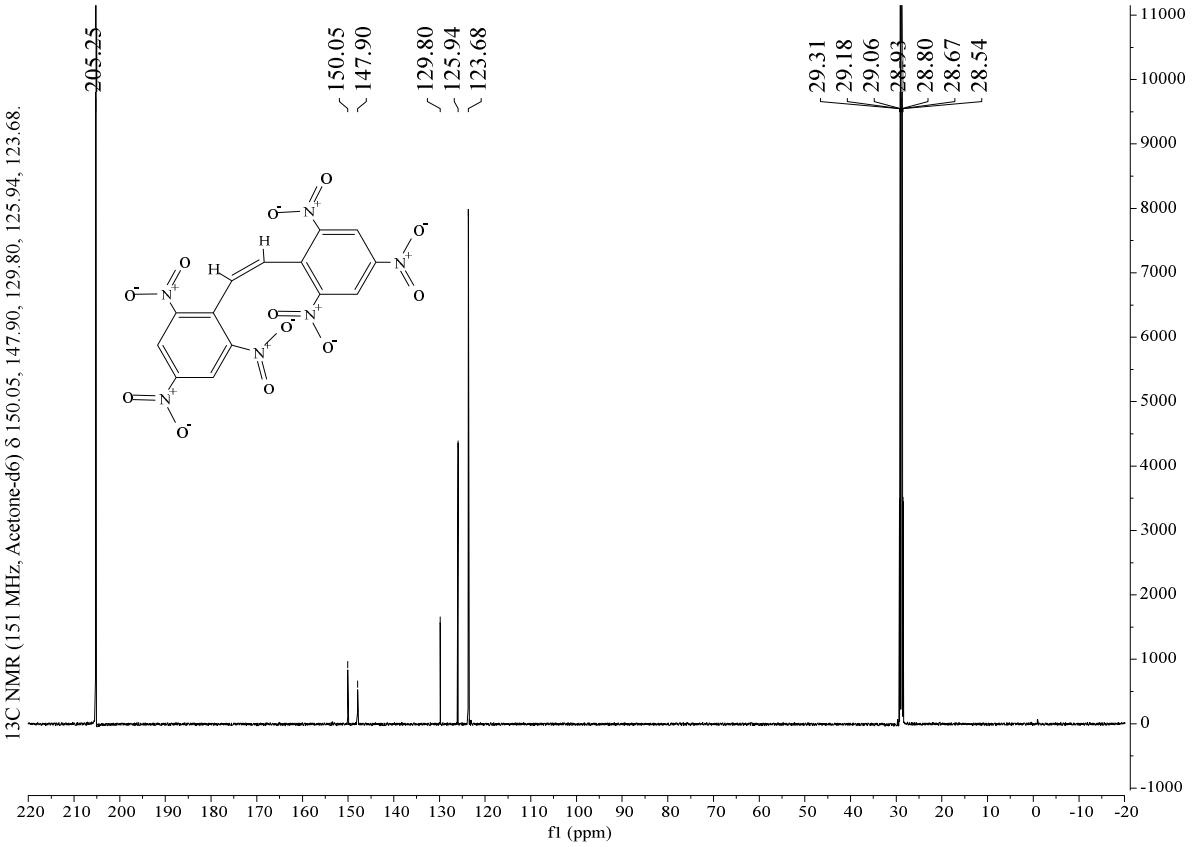

Figure S6 ^{13}C NMR spectra for **Substance 2** (Cis-HNS).

S2. HPLC Supplementary Information

Raw materials and **Substance 1** and **2** were analysed by High performance liquid chromatography (HPLC) using Agress1100 HPLC (Elite, Dalian, Liaoning, China). Performed on a Hypersil ODS2 column (200 mm×4.6 mm, 5 μm), using acetonitrile and water as mobile phases, with a ratio of 56:44, a flow rate of 0.7 mL·min^{-1}, and a UV-visible detector with a detection wavelength of 226 nm, column temperature 25 ℃, injection volume 10 μL.

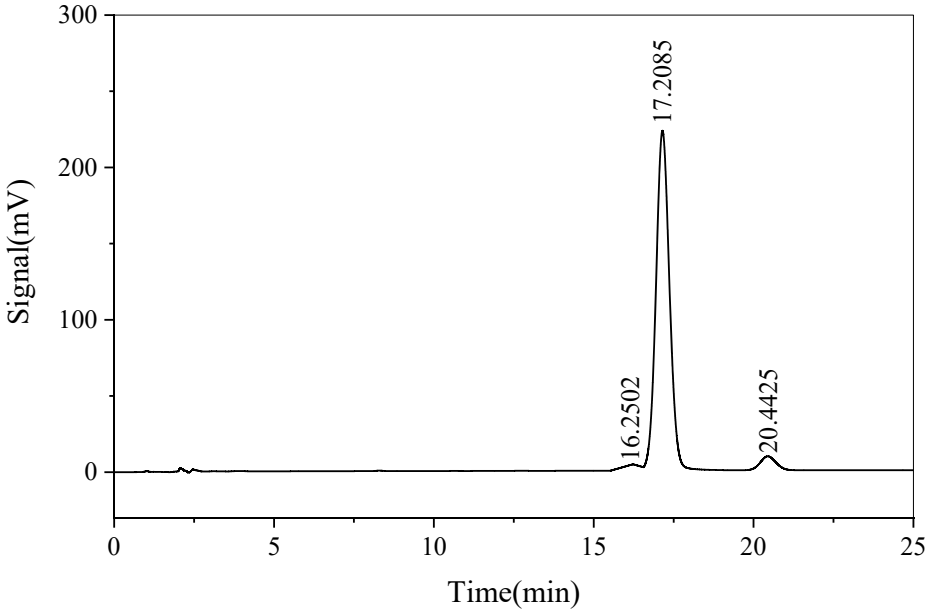

Figure S7 HPLC of Raw material. There are 3 obvious UV absorption peaks, Retention Times : 16.2502 min, 17.2085 min, 20.4425 min.

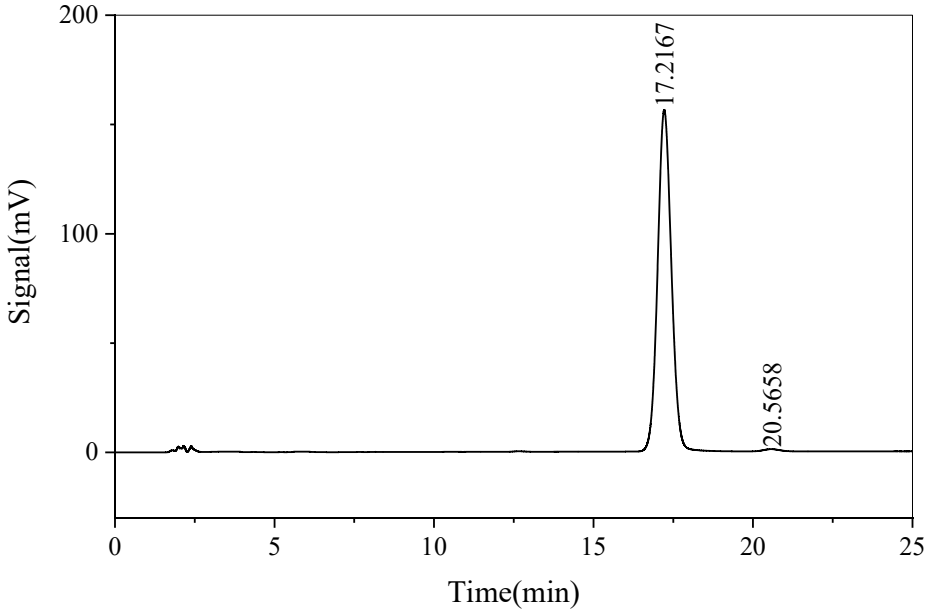

Figure S8 HPLC of **Substance 1** (Trans-HNS). There is an obvious UV absorption peak and a less obvious UV absorption peak, Retention Time: 17.2167 min, 20.5658 min.

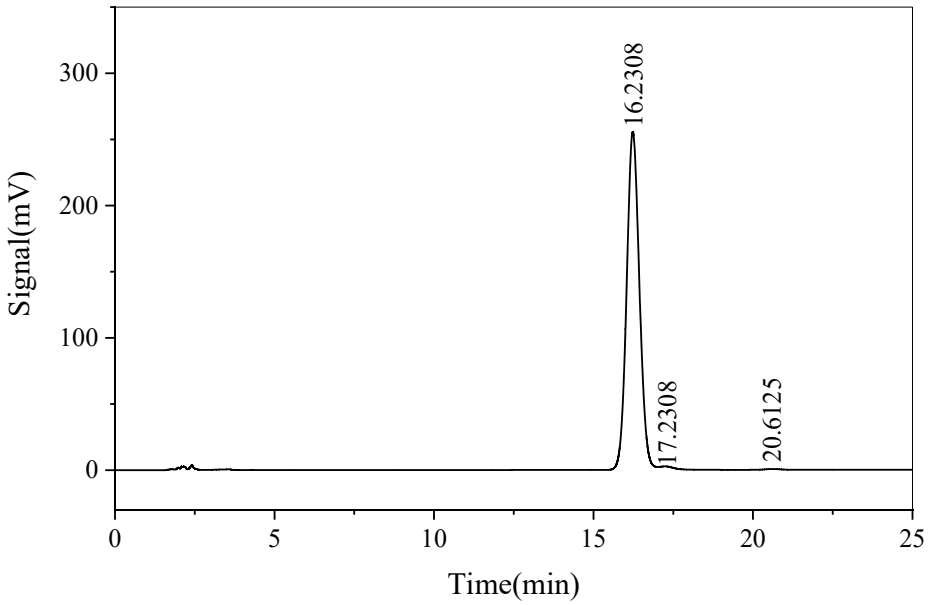

Figure S9 HPLC of **Substance 2** (Cis-HNS). There is an obvious UV absorption peak and two less obvious UV absorption peaks, Retention Time: 16.2308 min, 17.2308 min, 20.6125 min.

Firstly, the sample was prepared into a solution with a concentration of 0.1 mg/mL with acetonitrile, and then tested with Agress1100 HPLC. Finally, compared with the literature, under the current experimental conditions, the retention times of Cis-HNS, Trans-HNS and HNBB are about 16.24 min, 17.22 min and 20.57min.

According to the area normalization method, the contents of Cis-HNS, Trans-HNS and HNBB in the raw materials are about 1.12%, 95.02% and 3.86%; the contents of Trans-HNS and HNBB in **Substance 1** are about 99.24% and 0.76%; The content of Cis-HNS, Trans-HNS and HNBB in **Substance 2** are about 99.39%, 0.32% and 0.29%. That is, the purity of **Substances 1** and **2** are all above 99%.

S3. Experiment to verify the crystal transition phenomenon of Cis-HNS

When using a melting point meter to test the melting point of Cis-HNS, the temperature range is set at 205~350℃, and the heating rate is set at 10℃·min^{-1}. It can be seen from Figure 1 that the Cis-HNS was all solid at the beginning of the test, and the melting point meter showed that the test tube was all bright white.

Figure S10 The state of Cis-HNS at 205°C

It can be seen from Figure 2 that Cis-HNS melted into a liquid at about 232°C, and the melting point meter showed that the bright white color in the test tube disappeared, and the Cis-HNS is in a liquid state afterwards.

Figure S11 The state of Cis-HNS at 232℃

It can be seen from Figure 3 that during the temperature rise from 270°C to 317°C, bright white can be observed in the test tube displayed by the melting point meter, indicating that solid matter is produced during this process. In other words, the liquid Cis-HNS transforms into liquid Trans-HNS during the heating process, and then solidifies into solid Trans-HNS. After the temperature exceeds 317°C, it can be clearly seen that the substance in the test tube has decomposed and the bright white color has disappeared again. Because the temperature has reached the melting point of Trans-HNS at this time, and Trans-HNS will decompose immediately after melting.

Figure S12 The state of Cis-HNS at 270℃~317℃

S4. Kinetic calculation related graph

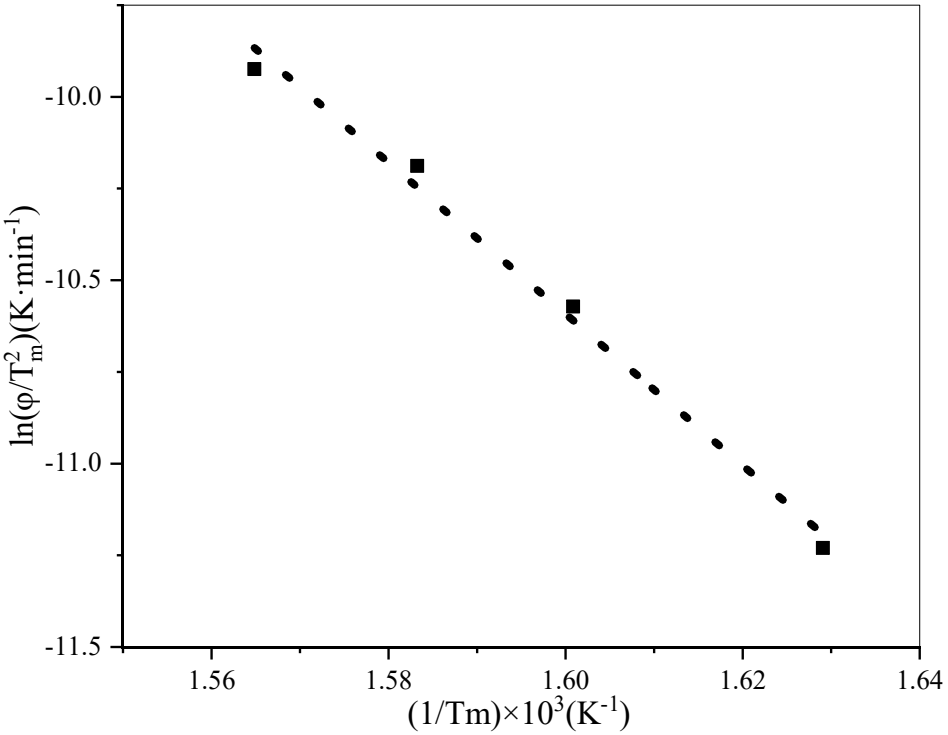

Figure S13 The curve of ln φ/T_m^2~1/T_m of Trans-HNS

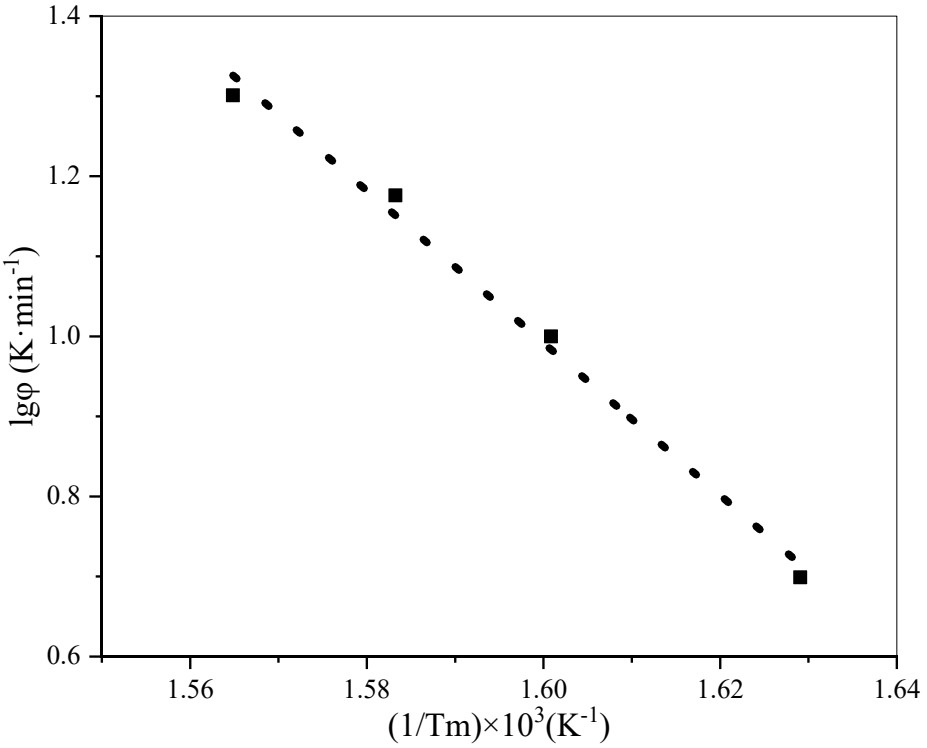

Figure S14 The curve of lg φ~1/T_m of Trans-HNS

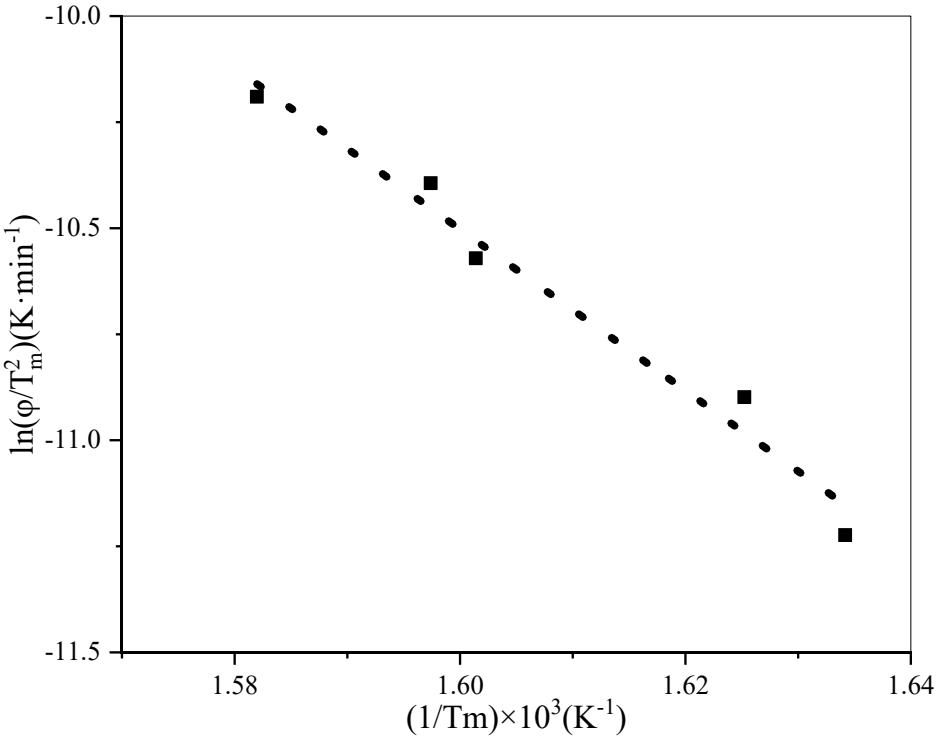

Figure S15 The curve of ln φ/T_m^2~1/Tm of Cis-HNS

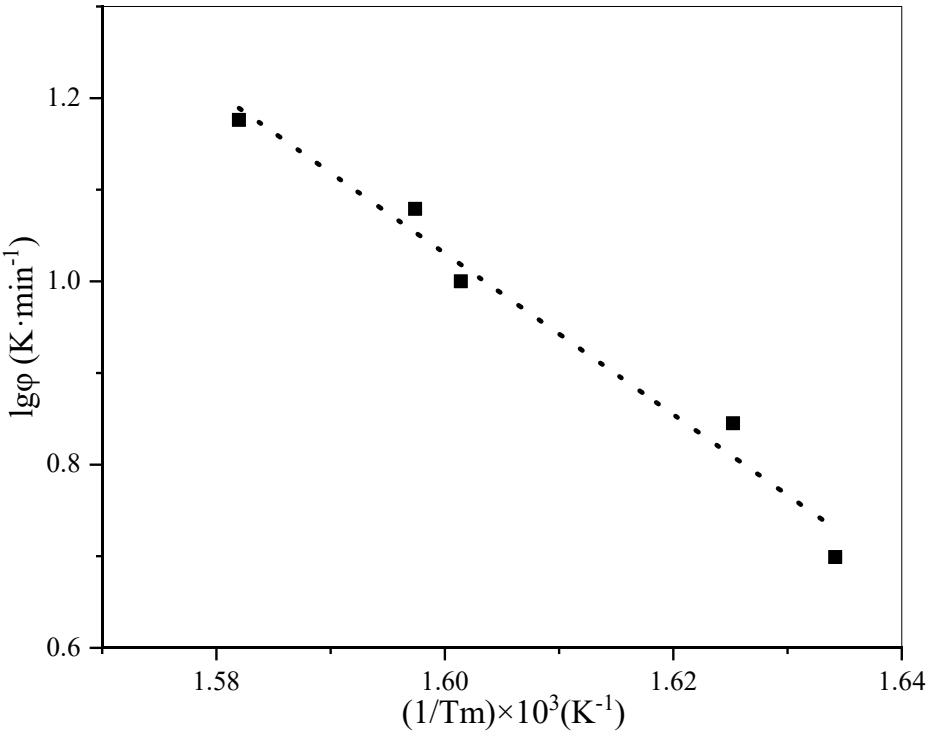

Figure S16 The curve of ln φ/T_m^2~1/T_m of Cis-HNS

S5. Supplementary Tables

Table S1 The bond length between all atoms in the Cis-HNS molecule (Å)

C1—C2	1.382 (3)	C13—H13	0.9500
C1—C6	1.405 (3)	N1—C3	1.469 (3)
C2—H2	0.9500	N2—C1	1.481 (3)
C3—C2	1.379 (3)	N2—O3	1.215 (2)
C4—C3	1.377 (3)	N3—C10	1.469 (2)
C4—C5	1.380 (3)	N4—C12	1.467 (3)
C4—H4	0.9500	N5—C14	1.475 (2)
C6—C5	1.392 (3)	N6—C5	1.475 (2)
C7—C6	1.486 (3)	O1—N1	1.223 (3)
C7—H7	0.9500	O2—N1	1.224 (3)
C8—C7	1.329 (3)	O4—N2	1.208 (2)
C8—H8	0.9500	O5—N3	1.222 (2)
C9—C8	1.484 (3)	O6—N3	1.222 (2)
C9—C10	1.400 (3)	O7—N6	1.217 (2)
C9—C14	1.406 (3)	O8—N6	1.225 (2)
C11—C10	1.384 (3)	O9—N5	1.226 (2)
C11—C12	1.375 (3)	O10—N5	1.218 (2)
C11—H11	0.9500	O11—N4	1.218 (2)
C12—C13	1.383 (3)	O12—N4	1.231 (2)
C13—C14	1.373)		

Table S2 Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters of Cis-HNS(Å2).

Atom	x	y	z	U_{iso}*/U_{eq}
O6	0.75658(10)	0.74827(14)	0.40820(10)	0.0323(4)
O4	0.93227(10)	0.79656(14)	0.53961(10)	0.0316(4)
O7	0.91740(10)	0.33295(17)	0.39913(10)	0.0365(4)
O11	0.46780(9)	0.41964(16)	0.14685(11)	0.0352(4)
O10	0.72632(10)	0.53472(15)	0.09454(10)	0.0332(4)
O12	0.47955(9)	0.37462(16)	0.04637(10)	0.0361(4)
O8	0.79226(10)	0.29174(15)	0.33999(10)	0.0340(4)
O9	0.82603(9)	0.45613(17)	0.22319(10)	0.0361(4)

O1	0.83127(12)	0.21223(18)	0.62517(13)	0.0463(5)
O2	0.92087(12)	0.33397(19)	0.73719(12)	0.0473(5)
O5	0.70941(11)	0.58491(18)	0.43372(11)	0.0442(5)
N4	0.50522(10)	0.41998(16)	0.11853(11)	0.0246(4)
N6	0.85906(10)	0.33945(16)	0.39724(11)	0.0242(4)
N5	0.75777(11)	0.50232(16)	0.17034(11)	0.0241(4)
N2	0.93301(11)	0.72499(17)	0.58937(11)	0.0284(4)
N3	0.72233(11)	0.64347(17)	0.38900(11)	0.0248(4)
N1	0.87752(13)	0.30497(19)	0.65844(14)	0.0343(5)
C11	0.61739(12)	0.52983(18)	0.25464(12)	0.0205(4)
H11	0.585946	0.532206	0.273966	0.025*
C12	0.58705(12)	0.47622(18)	0.17419(12)	0.0206(4)
C13	0.63156(12)	0.47008(18)	0.14454(12)	0.0210(4)
H13	0.609201	0.435269	0.087985	0.025*
C9	0.74599(12)	0.57439(17)	0.28372(12)	0.0180(4)
O3	0.95094(16)	0.7602(2)	0.65949(12)	0.0695(7)
C8	0.82971(12)	0.62658(18)	0.33614(12)	0.0197(4)
H8	0.840355	0.676738	0.303435	0.024*
C4	0.86183(12)	0.3299(2)	0.52343(13)	0.0240(4)
H4	0.843208	0.242844	0.507721	0.029*
C1	0.91004(12)	0.58706(19)	0.56484(12)	0.0214(4)
C7	0.89200(12)	0.61364(18)	0.42201(12)	0.0201(4)
H7	0.941014	0.656852	0.444008	0.024*
C6	0.89143(11)	0.53725(19)	0.48651(12)	0.0196(4)
C14	0.70958(12)	0.51667(18)	0.20071(12)	0.0192(4)
C10	0.69526(12)	0.58021(18)	0.30653(12)	0.0192(4)
C5	0.87002(12)	0.40666(19)	0.47125(12)	0.0212(4)
C3	0.88202(13)	0.3859(2)	0.59941(14)	0.0259(4)
C2	0.90707(13)	0.5131(2)	0.62214(13)	0.0255(4)
H2	0.921868	0.548913	0.675649	0.031*