



# Elucidation of inhibitors in the dimerization of acrylonitrile using $[\text{RuCl}_2(\text{DMSO})_4]/\text{CH}_3\text{CH}_2\text{COONa}/\text{DMSO}/o\text{-benzoylbenzoic acid}$ as catalyst

Kohichi Kashiwagi<sup>a,b,\*</sup>, Ryoji Sugise<sup>a</sup>, Toshihiro Shimakawa<sup>a</sup>,  
Tunao Matuura<sup>a</sup>, Masashi Shirai<sup>a</sup>

<sup>a</sup> UBE INDUSTRIES, LTD., 1978-5 Kogushi, Ube, Yamaguchi 755-8633, Japan

<sup>b</sup> Department of Chemistry, Graduate School of Science, Hiroshima University, 1-3-1 Kagamiyama, Higashi-hiroshima 739-8526, Japan

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## ABSTRACT

A study of inhibitors in the dimerization of acrylonitrile using the novel catalytic system of  $[\text{RuCl}_2(\text{DMSO})_4]/\text{CH}_3\text{CH}_2\text{COONa}/\text{DMSO}/o\text{-benzoylbenzoic acid}$  was carried out by examining the effect of reaction products on the catalytic activity. 1,4-Dicyanobuta-1,3-diene (**3**), one of the linear dimer products of acrylonitrile, inhibited the Ru-catalyzed reaction. Methylthiomethyl *o*-benzoylbenzoate (**8**), a by-product which was produced from carboxylic acid and DMSO under the reaction conditions, also acted as an inhibitor.

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## 1. Introduction

C6 dinitriles are important intermediates for the commercial production of hexamethylenediamine, one of the monomer components of Nylon-6,6 [1–7]. As an alternative to previous industrial methods, the tail-to-tail dimerization of acrylonitrile (AN) using ruthenium-based catalysts has emerged as an attractive and significant route to C6 dinitriles [8–18]. Previously, we reported on the selective tail-to-tail dimerization of AN by a new catalytic system  $[\text{RuCl}_2(\text{DMSO})_4]/\text{CH}_3\text{CH}_2\text{COONa}/\text{DMSO}/\text{carboxylic acid}$  that does not require the use of hydrogen [19]. In this catalytic system, which utilizes carboxylic acid in place of hydrogen, the production of propionitrile (PN, **5**) which is an inevitable undesired by-product in the reaction under a hydrogen atmosphere was remarkably suppressed. Thus, the conversion of AN into the linear dimers was effected with the highest selectivity (85.8%) reported thus far, providing a breakthrough for overcoming the intrinsic limit of 66.6% in the dimerization procedure requiring hydrogen pressure. However, we were faced with the formidable problem of the deactivation of the catalyst in our new type catalytic system [20]. In order to

maximize reaction efficiency it would be important to reveal the negative factors. In this paper, we wish to report on the examination of potential inhibitors to the dimerization of AN using the catalytic system  $[\text{RuCl}_2(\text{DMSO})_4]/\text{CH}_3\text{CH}_2\text{COONa}/\text{DMSO}/\text{carboxylic acid}$ .

## 2. Experimental

All reagents were purchased from Wako Pure Chemical Industries, Tokyo Kasei Kogyo, Aldrich or Ishizu Seiyaku.  $\text{RuCl}_2(\text{DMSO})_4$  [21] and *o*-benzoylbenzoic acid anhydride (**6**) [22] were prepared according to the literature. Other reagents were used without purification.

### 2.1. 1,4-Dicyanobuta-1,3-diene (DCBD) (**3**)

A mixture of  $\text{RuCl}_3 \cdot 3\text{H}_2\text{O}$  (4.93 g, 0.0185 mol), AN (200 g, 3.77 mol), and triethylamine (40.00 g, 0.395 mol) was stirred for 1.5 h at 150 °C under argon. After the solution was concentrated, the residual oil was distilled under reduced pressure to afford 2.40 g (0.6% based on AN) of a colorless oil mixture of 1,4-dicyanobuta-1,3-diene (**3**) and 1,4-dicyanobut-1-ene **1** (bp 97–105 °C, 1 mmHg). The composition of the mixture was determined by gas chromatography: *cis,cis*-**3**:*cis,trans*-**3**:*trans,trans*-**3**:1,4-dicyanobut-1-ene **1** = 28:49:20:3 (molar ratio).

\* Corresponding author at: UBE INDUSTRIES, LTD., 1978-5 Kogushi, Ube, Yamaguchi 755-8633, Japan. Tel.: +81 836 31 6319; fax: +81 836 31 7644.

E-mail address: [25988u@ube-ind.co.jp](mailto:25988u@ube-ind.co.jp) (K. Kashiwagi).

## 2.2. *o*-Benzoylbenzoic acid anhydride (**6**)

Mp 109–110 °C. IR (KBr, cm<sup>-1</sup>) 1798, 1734, 1666, 1595, 1579, 1448, 1316, 1279, 1211, 1103, 1043, 1012, 928, 775, 710. <sup>1</sup>H NMR (CDCl<sub>3</sub>) δ 7.4 (m, 6H), 7.55 (m, 4H), 7.65 (m, 6H), 7.95 (d, *J* = 6 Hz, 2H). Anal. Calcd. for C<sub>28</sub>H<sub>18</sub>O<sub>5</sub>: C, 77.41; H, 4.18. Found: C, 77.43; H, 4.25.

## 2.3. 3-(2'-Benzoylbenzoyloxy)-3-phenyl-1(3H)-isobenzofuranone (pseudoanhydride) (**7**)

A mixture of *o*-benzoylbenzoic acid anhydride (**6**, 1.00 g, 2.30 mmol), DMSO (0.36 g, 46 mmol), and toluene (10 mL) was stirred for 7 h at 90–100 °C. Then Na<sub>2</sub>CO<sub>3</sub> (1 g) was added and the resulting mixture was stirred at room temperature for 3 h. After filtration of the precipitate, the filtrate was evaporated. Recrystallization of the residue from diethyl ether–hexane in the refrigerator gave 0.6134 g (yield 61%) of 3-(2'-benzoylbenzoyloxy)-3-phenyl-1(3H)-isobenzofuranone (**7**) as a colorless crystal. Mp 139–142 °C (lit. mp 140 °C) [23]. IR (KBr, cm<sup>-1</sup>) 1793, 1743, 1665, 1595, 1449, 1316, 1273, 1233, 1112, 1079, 995, 945, 773, 720, 701, 687, 637. <sup>1</sup>H NMR (CDCl<sub>3</sub>) δ 7.2–7.4 (m, 9H), 7.4–7.7 (m, 7H), 7.80 (d, *J* = 5 Hz, 1H), 8.03 (d, *J* = 6 Hz, 1H). Anal. Calcd. for C<sub>28</sub>H<sub>18</sub>O<sub>5</sub>: C, 77.41; H, 4.18. Found: C, 77.61; H, 4.23.

## 2.4. Methylthiomethyl *o*-benzoylbenzoate (**8**)

A mixture of *o*-benzoylbenzoic acid anhydride (**6**, 0.54 g, 1.20 mmol) and DMSO (6.50 g, 83.2 mmol) was stirred for 0.5 h at 130 °C. Removal of DMSO by distillation under reduced pressure gave a residue, to which toluene (15 mL) and 10% aqueous Na<sub>2</sub>CO<sub>3</sub> were added. The toluene extract was dried over Na<sub>2</sub>SO<sub>4</sub> and the solvent was removed to afford a crude product. Purification by column chromatography on silica gel gave 0.30 g (yield 88%) of **8** as a pale yellow oil. IR (neat, cm<sup>-1</sup>) 1724, 1673, 1597, 1581, 1449, 1338, 1316, 1272, 1152, 1130, 1114, 1068, 935, 767, 733. <sup>1</sup>H NMR (CDCl<sub>3</sub>) δ 2.03 (s, 3H), 5.11 (s, 2H), 7.2–7.5 (m, 3H), 7.5–7.6 (m, 2H), 7.6–7.7 (m, 1H), 7.8 (m, 2H), 8.1 (m, 1H). HRMS 210.0669 (M–CH<sub>3</sub>SCHO C<sub>14</sub>H<sub>10</sub>O<sub>2</sub> requires 210.0681), 209.0602 (M–CH<sub>3</sub>SCH<sub>2</sub>O C<sub>14</sub>H<sub>9</sub>O<sub>2</sub> requires 209.0603).

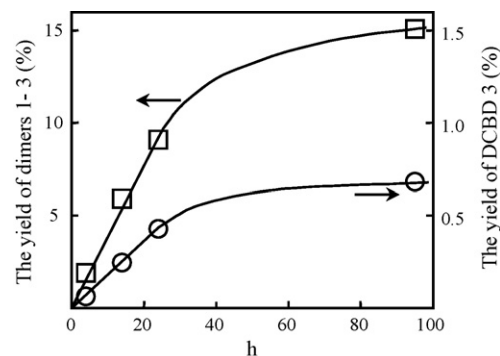
## 2.5. Dimerization of AN at 150 °C (Table 1)

A mixture of AN (15.00 g, 282.7 mmol), anisole (internal standard for gas chromatography; 2.00 g, 18.5 mmol), DMSO (0.50 g, 6.4 mmol), RuCl<sub>2</sub>(DMSO)<sub>4</sub> (18.4 mg, 0.0381 mmol; Ru/AN = 1/7400), sodium propionate (29.4 mg, 0.306 mmol), and *o*-benzoylbenzoic acid (1.29 g, 5.70 mmol) was stirred at room temperature under argon for 30 min. The resulting yellow solution (1.2 mL) was heated under argon to 150 °C for 6 h. The yield of the dimers is defined as follows: yield of dimer = 100 × [2 × (mols of dimer)]/[mols of charged acrylonitrile]. The selectivity for dimers **1–3** is defined as follows: selectivity for dimers **1–3** = 100 × [2 × (combined mols of dimers **1–3**)]/[mols of converted acrylonitrile]. Turnover number (TON) for dimers **1–3** is defined as follows: TON = [2 × (combined mols of dimers **1–3**)]/[mols of Ru].

The run with a 1/10,000 molar ratio of Ru/AN was carried out by reducing only the amount of RuCl<sub>2</sub>(DMSO)<sub>4</sub>.

## 2.6. Dimerization of AN at 120 °C (Fig. 1)

A mixture of AN (7.96 g, 150 mmol), anisole (internal standard for gas chromatography; 1.07 g, 9.9 mmol), DMSO (0.26 g,



**Fig. 1.** Time profile of the combined yield of dimer **1–3** (□) and the yield of DCBD **3** (○) at 120 °C. Reaction conditions: AN 150 mmol, anisole 9.9 mmol, DMSO 3.3 mmol, RuCl<sub>2</sub>(DMSO)<sub>4</sub> 0.0206 mmol, sodium propionate 0.161 mmol, *o*-benzoylbenzoic acid 3.0 mmol.

3.3 mmol), RuCl<sub>2</sub>(DMSO)<sub>4</sub> (10.0 mg, 0.0206 mmol), sodium propionate (15.5 mg, 0.161 mmol), and *o*-benzoylbenzoic acid (0.68 g, 3.0 mmol) was stirred at room temperature under argon for 30 min. The resulting yellow solution (1.2 mL) was heated under argon to 120 °C.

## 2.7. DCBD (**3**) as an additive

To a mixture of AN (7.50 g, 141 mmol), anisole (internal standard for gas chromatography) (1.00 g, 9.25 mmol), DMSO (0.25 g, 3.2 mmol), RuCl<sub>2</sub>(DMSO)<sub>4</sub> (0.0068 g, 0.014 mmol, Ru/AN = 1/10,000 (molar ratio)), sodium propionate (0.0135 g, 0.141 mmol), and *o*-benzoylbenzoic acid [0.64 g, 2.8 mmol, AN/acid = 50 (molar ratio)] was added DCBD [**3**, 72.9 mg, 0.700 mmol, additive/Ru = 50 (molar ratio)] prepared in Section 2.1. The mixture (1.2 mL) was heated for 9 h to 150 °C under argon. The yield of the products **1–3** was determined as follows: combined yield of produced dimers **1–3** = 100 × [2 × {(mols of detected dimers **1–3**) – (mols of added DCBD **3**)}] / [mols of charged AN].

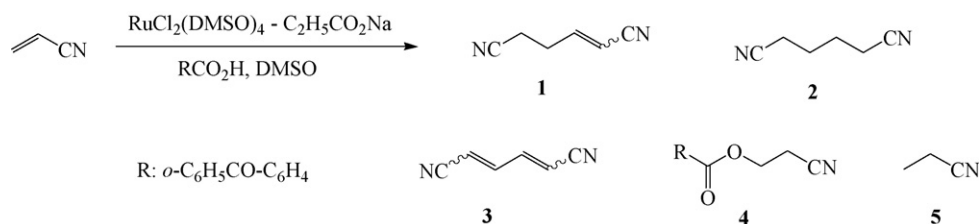
In the run with the molar ratio of additive/Ru = 250, 364.4 mg of DCBD (**3**, 3.500 mmol) was added.

## 2.8. Quantitative analysis of methylthiomethyl *o*-benzoylbenzoate (**8**) in the dimerization of AN

A mixture of AN (7.50 g, 141 mmol), anisole (internal standard for gas chromatography; 1.00 g, 9.25 mmol), DMSO (0.25 g, 3.2 mmol), RuCl<sub>2</sub>(DMSO)<sub>4</sub> (0.0093 g, 0.019 mmol), sodium propionate (0.0148 g, 0.154 mmol), and *o*-benzoylbenzoic acid (1.08 g, 4.77 mmol) was stirred at 150 °C for 6 h under argon. After 16.6% conversion of AN, the yield of PN was 0.6%. The combined yield of the dimers **1–3** was 14.5%. The conversion of *o*-benzoylbenzoic acid was 46.4%. The yields of 2-cyanoethyl *o*-benzoylbenzoate (**4**), and methylthiomethyl *o*-benzoylbenzoate (**8**) were 33.8% and 3.8% (about 10 times the mols of the ruthenium complex), respectively, based on *o*-benzoylbenzoic acid. *o*-Benzoylbenzoic acid, 2-cyanoethyl *o*-benzoylbenzoate (**4**) and methylthiomethyl *o*-benzoylbenzoate (**8**) were analyzed by gas chromatography on a OV-17/Chromosorb W(AW-DMCS) column. The structure of methylthiomethyl *o*-benzoylbenzoate (**8**) was confirmed by GC-MS (CI) 287 [M + 1]<sup>+</sup> and a synthesized authentic sample.

## 2.9. Acid anhydride **6**, pseudoanhydride **7**, and ester **8** as additives

To a mixture of AN (15.00 g, 282.7 mmol), anisole (internal standard for gas chromatography; 2.00 g, 18.5 mmol), DMSO (0.50 g,



Scheme 1.

6.4 mmol), RuCl<sub>2</sub>(DMSO)<sub>4</sub> (0.0185 g, 0.0382 mmol), sodium propionate (0.011 g, 0.11 mmol), and *o*-benzoylbenzoic acid (1.72 g, 7.61 mmol) was added 1.9 mmol of either *o*-benzoylbenzoic acid anhydride (**6**, 0.83 g), pseudoanhydride **7** (0.83 g) or methylthiomethyl *o*-benzoylbenzoate (**8**, 0.53 g) as an additive (molar ratio of additive/Ru = 50). For each reaction, the mixture (1.2 mL) was heated for 6 h to 150 °C under argon.

### 3. Results and discussion

#### 3.1. Deactivation of the catalyst at 150 °C

As previously reported, the tail-to-tail dimerization of AN by the catalyst consisting of Ru complexes and *o*-benzoylbenzoic acid yielded a mixture of 1,4-dicyanobut-1-ene (**1**, *cis*- and *trans*-DCB), adiponitrile (**2**, ADN), 1,4-dicyanobuta-1,3-diene (**3**, *cis,cis*-, *cis,trans*- and *trans,trans*-DCBD), 2-cyanoethyl *o*-benzoylbenzoate (**4**), and propionitrile (**5**) as shown in Scheme 1.

When the dimerization reaction catalyzed by RuCl<sub>2</sub>(DMSO)<sub>4</sub> (0.0382 mmol) and DMSO (6.4 mmol) was performed at 150 °C for 6 h in the presence of sodium propionate (0.306 mmol) and *o*-benzoylbenzoic acid (5.70 mmol) in AN (282.7 mmol), the conversion of AN was 15.5% [20]. The combined yield of dimers **1–3** was 13.0% (TON 960, selectivity 83.9%) (Table 1). The yield of the dimers was essentially the same even when the reaction time was elongated from 6 to 9 h. Therefore, it could be assumed that the catalyst was completely deactivated in 6 h under these reaction conditions.

#### 3.2. Decrease in catalytic activity at lower reaction temperature (120 °C)

Previously, we reported that the reaction rates of the catalytic dimerization of AN using several carboxylic acids at 120 °C were constant for the first 24 h [24]. In order to determine the fate of the reaction beyond this time, the reaction was extended over a few days. As a result, a gradual decrease in catalytic activity was found to occur beyond the reaction time of 24 h. Fig. 1 shows an example of the time profile of the dimer yield at 120 °C using *o*-benzoylbenzoic acid as the carboxylic acid. The yield of the dimers (**1–3**) was 9.1% at 24 h and was only up to 15.1% at 95 h. Thus, lowering the reaction

temperature led to a slower decay of the catalyst; however, it was not possible to completely suppress the deactivation.

#### 3.3. Possible factors for the decrease in catalytic activity

##### 3.3.1. DCBD (**3**)

It has been reported previously that strongly coordinating compounds such as pyridine and triphenylphosphine inhibit the catalytic dimerization of AN by blocking the coordination of AN to ruthenium [13,14]. Since 1,3-butadiene ligands are known to be good ligands for ruthenium complexes [25–27], it is reasonable to imagine that DCBD (**3**), which has a 1,3-butadiene substructure and is one of the dimeric products (ca. 4% content of the dimers) of the reaction, might inhibit dimer production in a similar way. Thus, we examined the additive effect of DCBD (**3**) on the dimerization of AN (Table 1).

The TON for the dimers was 1030 in the absence of additional DCBD (**3**). However, the TON decreased to 830 by adding 50 equivalents of DCBD (**3**) to the reaction mixture. In the presence of 250 equivalents of DCBD (**3**), the TON dropped to 200. These results are in good agreement with the assumption that DCBD (**3**) and AN compete to coordinate to ruthenium. However, DCBD (**3**) was not able to deactivate the catalyst completely. An examination of other products, such as DCB (**1**), ADN (**2**), PN (**5**), and 2-cyanoethyl *o*-benzoylbenzoate (**4**), demonstrated that they did not exhibit such an inhibition effect.

##### 3.3.2. Other inhibitors

In the reaction using *o*-benzoylbenzoic acid and DMSO, methylthiomethyl *o*-benzoylbenzoate (**8**), a Pummerer rearrangement product, was found to form as a by-product in 3.7% yield (based on the corresponding carboxylic acid), which corresponds to about 10 times the molar amount of the ruthenium catalyst (see Section 2.8) [28]. The structure of ester **8** was confirmed by a comparison of its GC-MS (CI) profile (287 [M+1]) and retention time in gas chromatography with those of a synthesized authentic sample. Heating acid anhydride **6** with DMSO at 100 °C in toluene did not afford the corresponding ester **8** but instead yielded 3-(2'-benzoylbenzoyloxy)-3-phenyl-1(3H)-isobenzofuranone (**7**) (pseudoanhydride), which is an isomer of

Table 1

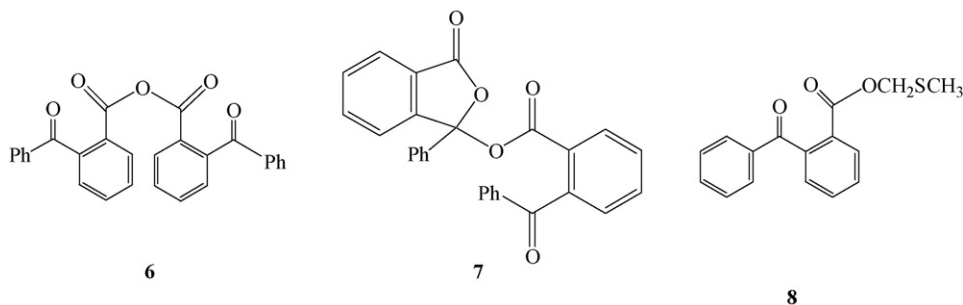
Additive effect of DCBD **3**, acid anhydride **6**, pseudoanhydride **7**, and ester **8** on the dimerization of AN at 150 °C<sup>a</sup>

Additive	Additive/Ru	Ru/AN <sup>b</sup>	Conversion of AN (%)	Combined yield of dimers <b>1–3</b> (%)	TON
–	–	1/7400	15.5	13.0	960
–	–	1/10000	13.1	10.3	1030
DCBD <b>3</b>	50	1/10000	10.2	8.3 <sup>c</sup>	830 <sup>c</sup>
DCBD <b>3</b>	250	1/10000	23.0	2.0 <sup>c</sup>	200 <sup>c</sup>
Acid anhydride <b>6</b>	50	1/7400	29.6	9.2	680
Pseudoanhydride <b>7</b>	50	1/7400	26.4	9.4	694
Ester <b>8</b>	50	1/7400	5.0	4.6	340

<sup>a</sup> Reaction conditions: see Section 2.7 and Section 2.9.

<sup>b</sup> Molar ratio.

<sup>c</sup> Yield and TON are based on dimers **1–3** produced by the dimerization reaction.



Scheme 2.

acid anhydride **6** [21]. However, the reaction of the acid anhydride with DMSO at temperatures over 130 °C gave rise to the rearrangement product **8**. The formation of ester **8** suggests the formation of acid anhydride **6** and/or pseudoanhydride **7** during the dimerization of AN. Since these compounds had the possibility of being inhibitors, an examination of the influence of acid anhydride **6**, pseudoanhydride **7**, as well as ester **8** on the catalytic activity was carried out. Table 1 shows the results. All of these compounds were found to diminish the catalytic activity in the dimerization of AN. Especially, ester **8** had a significant adverse impact, and the TON for the dimers decreased from 960 to 340. In the examinations with additional acid anhydride **6** or pseudoanhydride **7**, polymerization was promoted. In the catalytic dimerization of AN, however, such polymer formation was usually not observed, and furthermore, neither acid anhydride **6** nor pseudoanhydride **7** could be detected in the reaction mixture by gas chromatography. Thus, it seems that neither acid anhydride **6** nor pseudoanhydride **7** is accumulating during the reaction, probably because of their rapid transformations to ester **8** (Scheme 2).

#### 4. Conclusion

In order to elucidate possible factors in the deactivation of our Ru-catalyzed dimerization of AN, an examination of temperature and potential inhibitors was carried out. Whereas deactivation was significant just after 6 h at 150 °C, the same activity was maintained over 24 h at 120 °C, after which a gradual deterioration of catalyst was observed. DCBD (**3**), which is one of the linear dimer products of AN, and methylthiomethyl ester (**8**), which is formed from carboxylic acid and DMSO during the reaction, were found to decrease the catalytic activity, presumably by inhibiting the coordination of AN to ruthenium. Although these two substances do not account for all of the deactivation, we believe that they are the major causes.

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