Tetrahedron: *Asymmetry* xxx (2017) xxx–xxx

Contents lists available at ScienceDirect

Tetrahedron: *Asymmetry*

journal homepage: www.elsevier.com/locate/tetasy

Highly enantioselective asymmetric reactions involving zinc ions promoted by chiral aziridine alcohols

Szymon Jarzyński, Greta Utecht, Stanisław Leśniak, Michał Rachwalski *

Department of Organic and Applied Chemistry, University of Łódź, Tamka 12, 91-403 Łódź, Poland

ARTICLE INFO

Article history:
Received 16 September 2017
Accepted 9 October 2017
Available online xxxx

ABSTRACT

Enantiomerically pure, chiral secondary and tertiary aziridine alcohols (including the aziridine analogue of ProPhenol—AziPhenol) have proven to be highly effective catalysts for enantioselective asymmetric reactions in the presence of zinc ions, including arylation of aromatic aldehydes, epoxidation of chalcone and addition of diethylzinc to aldehydes, leading to the desired chiral products in high chemical yields (up to 90%) and with *ee*'s up to 90%. A higher catalytic activity of Prophenol-type bis(aziridine alcohol) in the aforementioned asymmetric transformations has been demonstrated.

1. Introduction

The enantioselective formation of carbon–carbon bonds is one of the most essential strategies in modern organic chemistry.[1,2] Among them, asymmetric reactions involving zinc cations constitute current and extensively studied topics in synthetic organic chemistry. Typical examples of zinc(II)-mediated stereocontrolled transformations are asymmetric arylation of aldehydes[3] and epoxidation of α,β-unsaturated carbonyl compounds (e.g. chalcone).[4] Both processes are of great importance due to a high significance of potential applications of their chiral products. Thus, diarylmethanols can act as precursors of many biologically and pharmacologically relevant compounds,[3] e.g. (*R*)-orphenadrine and (*R*)-neobenodine,[5] (*S*)-cetirizine[6] or (*S*)-BMS 184394[7,8] (Fig. 1).

In turn, chiral epoxides are a structural part of many natural products, fragrances (epoxides of carvone), pheromones ((+)-disparlure) or alkaloids (scopolamine). Moreover, chiral epoxides find applications in the synthesis of other natural products such as (−)-Cleistenolide[9] and cascarillic acid,[10] and in many other asymmetric transformations.[11] Chiral epoxides are also present in many biologically relevant molecules such as Neocarzinostatin,[12] Ovalicin[13] or Epothilones (Fig. 2).[14,15]

ProPhenol dinuclear ligands containing proline[16–19] or azetidine[20] can form complexes with zinc ions facilitating various asymmetric transformations such as direct aldol reactions,[16,19] 1,4-additions of diethyl phosphite to enones,[17] nitroaldol (Henry) reactions,[18] or Friedel–Crafts alkylations of pyrrole with chalcones.[20]

In order to continue our studies on stereocontrolled reactions including the formation of carbon–carbon bonds,[21–25] having advantageous results in the asymmetric arylation of aldehydes using secondary and tertiary aziridine alcohols[3] we decided to synthesize a novel ProPhenol-type ligand (AziPhenol) containing chiral aziridine moieties and to compare its catalytic activity with those exhibiting by the aforementioned simple aziridine alcohols. Similar studies have been conducted using azetidine analogues of ProPhenol ligands in Friedel–Crafts reaction.[20] These analogues exhibited much higher catalytic activity in comparison with those found with simple chiral azetidine alcohols.[20] First of all, we decided to check the catalytic activity of all designed ligands in the asymmetric epoxidation of chalcone.

2. Results and discussion

2.1. Synthesis of the ligands

Chiral aziridine alcohols **L1**–**L4** (Fig. 3) were synthesized as described previously.[26,27] AziPhenol ligand **L5** was prepared *via* a three-step synthetic route (Scheme 1).

In the first step, *p*-cresol was treated with formaldehyde in aqueous NaOH solution affording the corresponding diol **2**.[18] To the above diol, HBr in acetic acid was added at room temperature, which gave compound **3** in 80% yield.[18] Dibromide **3** was reacted with aziridine alcohol **4**[26] in dry DMF, in the presence of potassium carbonate at 0 °C. Pure ligand **L5** was obtained after purification *via* column chromatography in 86% yield.

* Corresponding author.
E-mail address: mrach14@wp.pl (M. Rachwalski).

https://doi.org/10.1016/j.tetasy.2017.10.007
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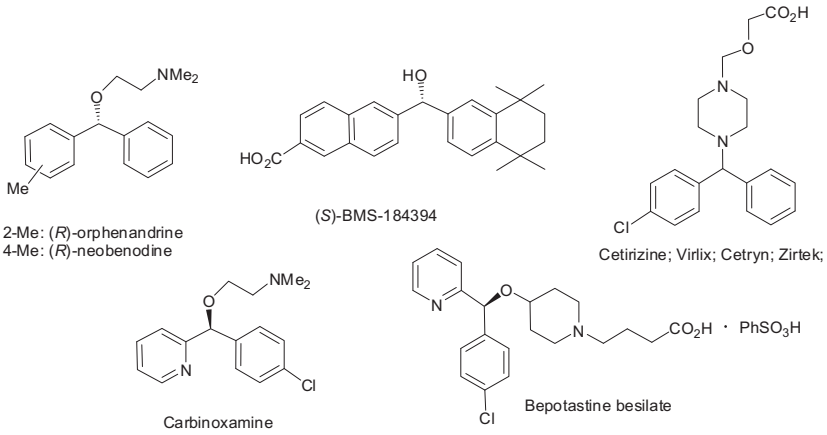


Figure 1. Diarylmethane-based drugs.

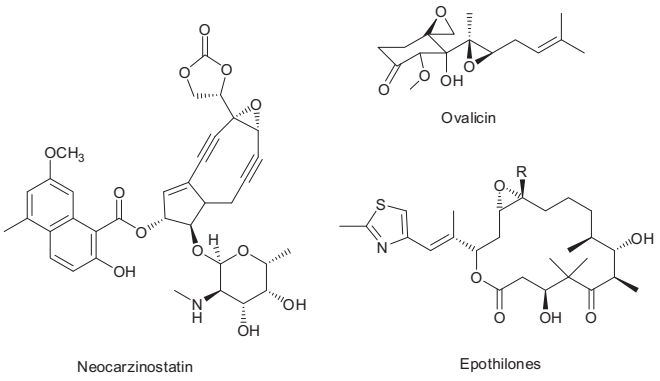
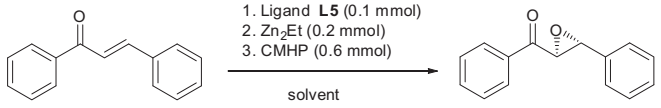


Figure 2. Drugs containing a chiral epoxide.

2.2. Asymmetric epoxidation of chalcone—screening of the conditions and ligands

The designed aziridine alcohol **L5** was tested as a chiral ligand in the asymmetric epoxidation of chalcone following the procedure described by Ulukanli et al.[4] (Scheme 2).[17] The reactions were carried out in various solvents and at 0 °C or 20 °C, respectively, in the



Scheme 2. Asymmetric epoxidation of chalcone catalyzed by ligand **L5** under various conditions.

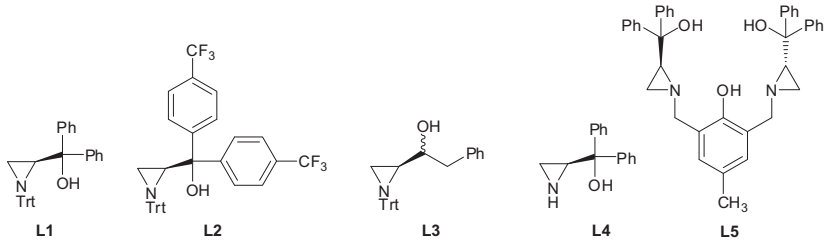
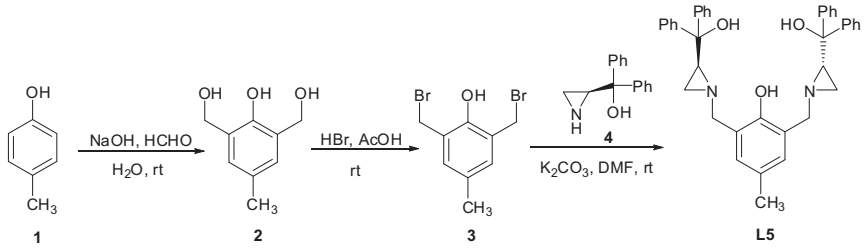


Figure 3. Chiral aziridine alcohols **L1**–**L5**.

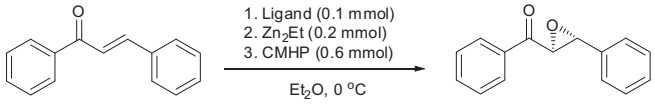


Scheme 1. Synthesis of ProPhenol ligand **L5**.

Table 1
Asymmetric epoxidation of chalcone promoted by an AziPhenol system **L5**, under various conditions

Entry	Solvent	*T* [°C]	Yield [%]	*ee* [%][a]
1	THF	20	73	50
2	CH_2Cl_2	20	81	27
3	Toluene	20	78	60
4	Et_2O	20	88	76
5	Et_2O	0	93	97

[a] Determined by chiral HPLC using Chiralcel OD-H column.



Scheme 3. Asymmetric epoxidation of chalcone catalyzed by aziridine alcohols **L1**–**L5**.

presence of diethylzinc and using cumene hydroperoxide (CMHP) as the epoxidising agent (Table 1).

Inspection of Table 1 clearly shows that the best solvent of this asymmetric transformation is diethyl ether. Enantioselective epoxidation of chalcone carried out in Et_2O at 0 °C led to the formation of the representative epoxide in 93% chemical yield and with very high enantioselectivity (97% *ee*) (Table 1, entry 5).

2.3. Asymmetric epoxidation of chalcone—screening of the ligands

Having established the best results in terms of solvent and temperature, further aziridine alcohols **L1**–**L4** were investigated in the same asymmetric process (Scheme 3). All the results (including those for ligand **L5**) are collected in Table 2.

Analysis of the results from Table 2 clearly evidences that all the aziridine alcohols **L1**–**L5** are efficient catalysts for the enantioselective epoxidation of chalcone, leading to the desired product in high chemical yields and with high enantiomeric excess. The highest enantioselectivity was achieved using an AziPhenol system **L5**.

2.4. Asymmetric addition of arylzinc systems generated from phenylboronic acid and diethylzinc to *p*-tolualdehyde catalyzed by an AziPhenol system L5

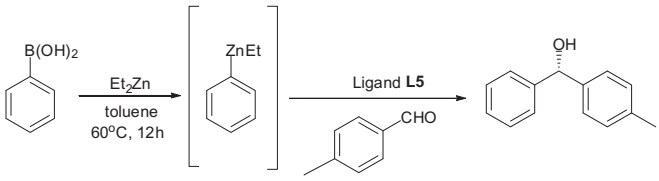
Previously, we reported a successful asymmetric arylation of aromatic aldehydes using aziridine alcohols **L2**–**L4** (**L1** had been tested earlier by others).[3] In the most efficient reaction, the corresponding diarylmethanol was obtained in 89% yield and with 93% of *ee*.[3]

In order to compare the catalytic activity of AziPhenol **L5** with those exhibited by the aforementioned ligands **L1**–**L4**, asymmetric arylation using *p*-tolualdehyde, phenylboronic acid and diethylzinc was performed in the presence of ligand **L5** (Scheme 4).

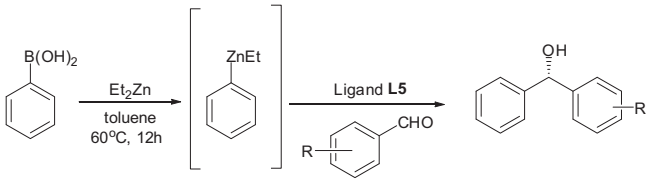
Analyzing the results of the above transformation, the desired diarylmethanol was obtained in 95% chemical yield and with 97% enantiomeric excess. In the light of these values, it should be concluded, that AziPhenol ligand **L5** was the most active catalyst for the asymmetric arylation of *p*-tolualdehyde using phenylboronic acid and Et_2Zn.

2.5. Asymmetric addition of arylzinc systems generated from phenylboronic acid and diethylzinc to aromatic aldehydes in the presence of catalyst L5

Having the most active system **L5** in hands, we decided to perform an enantioselective arylation reactions under the same conditions, using other aromatic aldehydes as starting materials (Scheme 5). The results are summarized in Table 3.



Scheme 4. Asymmetric arylation of *p*-tolualdehyde catalyzed by **L1**–**L5**.



Scheme 5. Addition of arylzinc system to aromatic aldehydes in the presence of ligand **L5**.

Table 2
Asymmetric epoxidation of chalcone—screening of the ligands

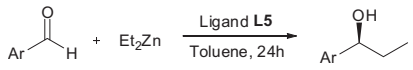
Entry	Ligand	Product			
		Yield [%]	$[\alpha]_D$[a]	*ee* [%][b]	Abs. config.[c]
1	**L1**	88	−176.4	86	(2*R*,3*S*)
2	**L2**	94	−173.9	82	(2*R*,3*S*)
3	**L3**	85	−172.8	80	(2*R*,3*S*)
4	**L4**	90	−178.7	91	(2*R*,3*S*)
5	**L5**	93	−179.2	97	(2*R*,3*S*)

[a] In chloroform (*c* 0.3).
[b] Determined by chiral HPLC using Chiralcel OD-H column.
[c] According to literature data.[4]

Table 3
Addition of arylzinc species to aromatic aldehydes promoted by ligand **L5**

Entry	R	Products			
		Yield [%]	$[\alpha]_D$[a]	*ee* [%][b]	Abs. config.[c]
1	4-Me	95	−4.4	97	(*S*)
2	4-OMe	89	−12.3	92	(*S*)
3	4-NO_2	85	+48.4	91	(*S*)
4	4-CF_3	87	+28.8	93	(*S*)
5	4-Br	91	+17.1	91	(*S*)
6	4-Cl	90	+8.8	98	(*S*)
7	2-Me	94	−2.7	93	(*S*)

[a] In chloroform (*c* 0.3).
[b] Determined by chiral HPLC using Chiralcel OD, OD-H or AD-H columns.
[c] Taken from the literature[28,29] (on the basis of specific rotation signs and retention times in HPLC chromatograms).



Scheme 6. Enantioselective addition of diethylzinc to aromatic aldehydes catalyzed by **L5**.

The results clearly indicate that aziridine alcohol **L5** is a very efficient chiral catalyst for the enantioselective addition of arylzinc system to aromatic aldehydes, leading to the corresponding products in high chemical yields and also with high enantiomeric excess values.

2.6. Asymmetric addition of diethylzinc to aromatic aldehydes in the presence of ligand L5

In our previous work, the enantioselective diethylzinc addition to aromatic aldehydes in the presence of ligands of type **L3**, was described.[27] Using the most efficient system, the corresponding chiral alcohol was formed in 97% yield and with 93% of *ee*.[27]

As in the case of the previous asymmetric reaction described herein, we decided to confirm the catalytic activity of newly synthesized AziPhenol ligand **L5** with those exhibited by aziridine alcohols reported previously.[27] Moreover, anticipating its high catalytic capacity, we also performed the asymmetric diethylzinc addition to other aromatic aldehydes (Scheme 6). All the results are collected in Table 4.

Inspection of Table 4 clearly evidences that aziridine alcohol **L5** constructed on the AziPhenol platform exhibited the highest catalytic activity in the asymmetric addition of diethylzinc to aromatic aldehydes, in comparison with previously studied chiral aziridine alcohols.

3. Conclusions

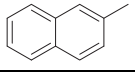
The newly synthesized bis(aziridine alcohol) **L5** introduced into a phenol system has proven to be very effective and versatile ligand catalyzing asymmetric transformations involving zinc ions, such as the enantioselective epoxidation of chalcone, arylation of aromatic aldehydes using phenylboronic acid and diethylzinc addition to aromatic aldehydes. As described earlier, ProPhenol-type ligands can spontaneously form chiral dinuclear metal complexes with e.g. $ZnEt_2$. Such formed complex posses both a Lewis acidic site to activate an electrophile and a Brønsted basic site to deprotonate a pronucleophile.[19] Moreover, this catalytic system exhibited higher catalytic activity in comparison with those showed by aziridine alcohols described earlier.[3,27]

4. Experimental

4.1. General

Toluene, tetrahydrofuran and diethyl ether were distilled from sodium benzophenone ketyl radical. Dichloromethane was distilled over calcium chloride. ^1H, ^{13}C and ^{19}F NMR spectra were recorded on a Bruker instrument at 600 MHz and 150 MHz, respectively, with $CDCl_3$ as solvent and TMS as internal standard. Data are reported as s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet, br. s = broad singlet. Optical rotations were measured on a Anton Paar MCP500 polarimeter with a sodium lamp at room temperature (*c* 0.3). Column chromatography was carried out using Merck 60 silica gel. TLC was performed on Merck 60 F_{254} silica gel plates. Visualization was accomplished with UV light (254 nm) or using iodine vapors. The enantiomeric excess (*ee*) values were determined by chiral HPLC (Chiralcel AD-H, OD or OD-H columns).

Table 4
Diethylzinc addition to aromatic aldehydes in the presence of ligand **L5**

Entry	Ar	Product			
		Yield [%]	$[\alpha]_D$[a]	*ee* [%][b]	Abs. config.[c]
1	Ph	97	−14.8	98	(*S*)
2	4-MePh	92	−13.7	97	(*S*)
3	4-MeOPh	91	−5.3	97	(*S*)
4	4-NCPh	86	−3.9	94	(*S*)
5		93	−4.6	98	(*S*)

[a] In chloroform (*c* 0.3).
[b] Determined by chiral HPLC using Chiralcel OD, OD-H or AD-H columns.
[c] According to literature data.[30,31]

4.2. Synthesis of the ligands

Aziridine alcohols **L1**–**L4** were synthesized according to the protocols described previously.[26,27] Compounds **2** and **3** were obtained according to literature data.[18]

4.2.1. Synthesis of AziPhenol ligand L5

In a round-bottomed flask, aziridine alcohol **4**[26] (2 mmol), K_2CO_3 (8 mmol) and dry DMF (5 mL) were mixed at 0 °C. The mixture was stirred for 15 min under argon followed by the addition of dibromide **3** (1 mmol). The ice bath was removed after the addition and the resulting solution was allowed to stir at room temperature for 12 h. Water was added in order to quench the reaction, which was extracted with Et_2O (3 × 10 mL) and the combined organic layers were dried over anhydrous $MgSO_4$, filtered and the solvents were evaporated *in vacuo*. The crude mixture was purified by column chromatography (silica gel, hexane/ethyl acetate in gradient) to afford the corresponding product **L5**.

4.2.1.1. ((2*S*,2′*S*)-1,1′-((2-Hydroxy-5-methyl-1,3-phenylene)bis(methylene))bis(aziridine-2,1-diyl))bis(diphenylmethanol) L5.

Yield 86%, Colorless solid; m.p. 141–143 °C, $[\alpha]_D^{23}$ = +19.6 (*c* 0.3, $CHCl_3$); ^{1}H NMR (600 MHz, $CDCl_3$) δ: 1.47 (br s, 1H), 1.47 (d, 2H, $J_{H,H}$ = 6.0 Hz), 2.01 (d, 2H, $J_{H,H}$ = 3.0 Hz), 2.12 (s, 3H), 2.48 (dd, 2H, $J_{H,H}$ = 3.0 Hz, $J_{H,H}$ = 6.0 Hz), 3.09 (s, 2H), 3.37 (d, 2H, $J_{H,H}$ = 13.5 Hz), 3.63 (d, 2H, $J_{H,H}$ = 13.5 Hz), 6.72 (s. 2H), 7.07–7.18 (m, 9H), 7.21–7.23 (m, 4H), 7.27–7.31 (m, 7H); ^{13}C NMR (151 MHz, $CDCl_3$): δ 20.5 (CH_3), 30.7 (*C*H azir), 46.8 (*C*H azir), 59.8 (CH_2), 74.8 (C_q), 123.8 ($C_{q\ ar}$), 126.3 (C_{ar}), 126.4 (C_{ar}), 127.0 (C_{ar}), 127.1 (C_{ar}), 128.0 (C_{ar}), 128.0 (C_{ar}), 128.2 ($C_{q\ ar}$) 128.6 (C_{ar}), 144.8 (C_{qar}), 146.7 ($C_{q\ ar}$), 152.0 ($C_{q\ ar}$) ppm; HRMS (ESI-TOF): *m*/*z* $[M + H]^+$ calcd for $C_{39}H_{39}N_2O_3$: 583.2968, found 583.2961. Copies of NMR spectra of compound **L5** are included in the Supporting Information.

4.3. Enantioselective epoxidation of chalcone—general procedure[4]

Aziridine alcohol **L1**–**L5** (0.1 mmol) and anhydrous Et_2O (4 mL) were placed in a round-bottomed flask under nitrogen atmosphere. After cooling to 0 °C, Et_2Zn (0.2 mmol, 1 M solution in toluene) was added whilst stirring. Chalcone (0.5 mmol) and CMHP (0.6 mmol, 80% solution in cumene) were added and the mixture was stirred at 0 °C for 12 h. The reaction was quenched with aqueous saturated $NaHCO_3$ and extracted with Et_2O. The organic layer was washed with aqueous Na_2CO_3 and brine. The combined organic layers were dried over $MgSO_4$, and the solvent was evaporated *in vacuo*. The residue was purified by column chromatography (silica gel, hexane with ethyl acetate in gradient) to afford the appropriate chiral epoxide. Its spectroscopic data are in full agreement with literature.[4] Chemical yield, optical rotation and enantiomeric excess values are collected in Tables 1 and 2. HPLC conditions along with copies of HPLC chromatograms for the epoxidation products are included in the Supporting Information.

4.4. Asymmetric arylation of aldehydes with phenylboronic acid in the presence of diethyl zinc—general procedure

Diethylzinc (2.5 mmol, 1 M solution in toluene) was added to a solution of phenylboronic acid (0.5 mmol) in toluene (1.5 mL) under nitrogen atmosphere. After stirring at 60 °C for 15 min, the mixture was cooled to ambient temperature and the solution of ligand **L5** (10 mol%) in toluene (1 mL) was added. Reaction was cooled to 0 °C and aldehyde (0.5 mmol) was added. After stirring for 24 h, the mixture was quenched by 15 mL of saturated, aqueous solution of NH_4Cl and the aqueous phase was extracted with diethyl ether (3 × 10 mL). The combined organic layers were dried over anhydrous Na_2SO_4, filtered and the solvents were evaporated *in vacuo*. The crude mixture was purified by column chromatography (silica gel, hexane with ethyl acetate in gradient) to afford the corresponding diarylmethanols. Their spectroscopic data are in full agreement with literature.[3,28,29] Chemical yield, specific rotation and enantiomeric excess values are collected in Table 3. HPLC conditions along with copies of HPLC chromatograms for the arylation products are included in the Supporting Information.

4.5. Asymmetric addition of diethylzinc to aldehydes—general procedure

Chiral ligand **L5** (0.1 mmol) was placed in a dried Schlenk tube under a nitrogen atmosphere. Freshly distilled toluene (10 mL) was then added followed by Et_2Zn solution (1.0 M in hexane, 3 mmol). The resulting solution was cooled to 0 °C and stirred for approximately 15 min. The aldehyde (1 mmol) was added and the mixture was stirred at room temperature overnight. The reaction was quenched with saturated ammonium chloride solution, layers were separated and the aqueous phase was extracted with diethyl ether (4 × 10 mL). The combined organic layers were washed with brine (10 mL) and dried over anhydrous $MgSO_4$ and the solvents were evaporated in vacuo. The crude compound was purified by flash chromatography (hexane with ethyl acetate in gradient) to give the final products. Their spectroscopic data are in full agreement with literature.[27,30,31] Chemical yield, specific rotation and enantiomeric excess values are collected in Table 4. HPLC conditions along with copies of HPLC chromatograms for the Et_2Zn addition products are included in the Supporting Information.

Acknowledgement

Financial support by the National Science Centre (NCN), Poland, Grant Preludium No. 2014/15/N/ST5/02897 for S. J. is gratefully acknowledged.

A. Supplementary data

Supplementary data associated with this article can be found, in the online version, at https://doi.org/10.1016/j.tetasy.2017.10.007.

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Please cite this article in press as: Jarzyński, S.; et al. *Tetrahedron: Asymmetry* (2017), https://doi.org/10.1016/j.tetasy.2017.10.007