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# INVESTIGATION OF THE ACID-MEDIATED CYCLISATION OF AMIDE ACETAL FOR THE SYNTHESIS OF BENZAZEPINONE

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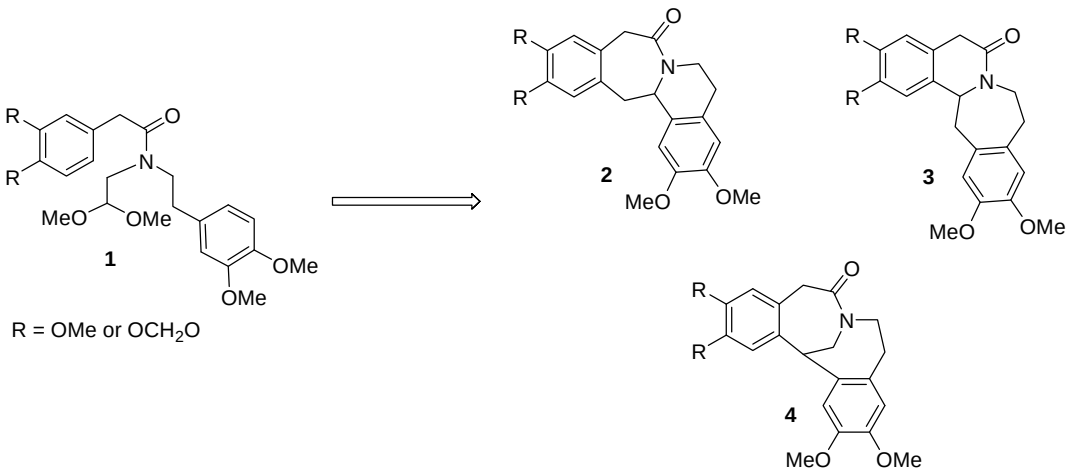
**Abstract** – On treatment with acids, the open-chained amide acetals (**1**) gave the 7,7 membered ring fused benzazepinones (**4**) *via* the bis-cyclisation.

## INTRODUCTION

New approaches for the synthesis of heterocyclic and carbocyclic molecules by cyclisation through various means of carbon-carbon bond formation are important in organic chemistry because they can be potentially applied for the syntheses of many biologically active compounds having heterocyclic and carbocylic structures.[1] An attractive strategy to form heterocyclic molecules is the acid-mediated ring closure of acetal,[2] some examples are known for intramolecular acid-mediated cyclisation of acetals with direct participation of aromatic moiety as a nucleophile. Takayama *et al*.[3] reported the acid-mediated cyclisation of dibenzylaminoacetaldehyde dimethyl acetal using hydrochloric acid in acetone for the synthesis of (-)-cherylline. Recently, Chrzanowska *et al*.[4] reported acid-mediated cyclisation of isoquinolone acetal, derived from the condensation of acyclic imine acetal with (*S*)-(-)-*O*-toluamide under basic condition, to furnish a protoberberine alkaloid (*S*)-(-)-*O*-methylbharatamine. Alternatively, Domínguez *et al*.[5] reported acid-mediated cyclisation of sulfonamide acetal to form chromeno-3-benzazepine. To the best of our knowledge, the acid-mediated *bis*-cyclisation reactions of amide acetal have not been studied previously. This paper explores the acid-mediated cyclisation of amide acetals **1** using mixtures of HCl/AcOH, $H_2SO_4$/AcOH, and refluxing $HCO_2H$ aiming at a possible synthesis of the tetracyclic benzazepinone. Theoretically, three possible cyclisation products **2-4** could be formed by the

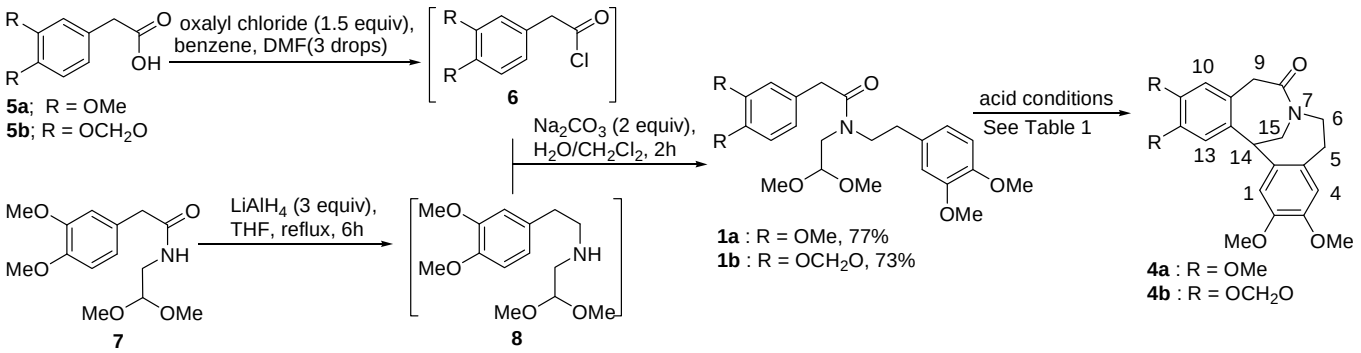
treatment of the open chain amide acetals with acids as shown in Scheme 1. The results of the investigation are reported herein.




Scheme 1

## RESULTS AND DISCUSSION

The amide acetals **1a** and **1b** could be prepared by the reaction of amine acetal **8** with either homoveratroyl chloride **6a** or 3, 4-methylenedioxyphenylacetyl chloride **6b** respectively. The required amide acetals **1a** and **1b** were obtained in 77 and 73% yield respectively. The amine acetal **8** was conveniently prepared by reduction of the corresponding amide **7** using lithium aluminium hydride. The acid chlorides **6** were obtained by reaction of the corresponding acids **5a** and **5b** with oxalyl chloride as shown in Scheme 2. Interestingly, $^{1}$H NMR analyses of both amide acetals (**1a** and **1b**) were complicated by the presence of amide rotamers.[6]




Scheme 2

When cyclisation of amide acetal **1a** was performed under acidic conditions including HCl/AcOH, $H_2SO_4$/AcOH, and refluxing $HCO_2H$, one main product was obtained from the reaction as shown in Table 1. High resolution MS showed MW of 384.1811 ($M^+$+1) for the reaction product of **1a**, corresponding to

our expected products (**2a-4a)**. Note that compounds **2a-4a** are isomers of one another (formula: $C_{22}H_{25}NO_5$ expected MW 384.1805 ($M^+$+1)). The NMR spectrum of the product clearly indicated the presence of only four aromatic protons, suggesting that the double cyclisation indeed occurred. Comparing the structures of compounds **2a-4a**, there are two different types of connectivity of the fused rings, namely $Ar_2CH_2CHNCO$ (**2a** or **3a**) or $Ar_2CHCH_2NCO$ (**4a**). The benzazepinone core structure was confirmed using HMQC spectral data. It was shown that one of the methylene protons at C-15 showed a doublet at 3.59 ppm (1H, *J* = 15.2 Hz) and another proton appeared as a doublet of doublet at 4.61 (1H, *J* = 15.2, 3.4 Hz). It was also shown that one benzylic proton at C-14 displayed a doublet at 4.04 (1H, *J* = 3.4 Hz). The relatively downfield chemical shift of one proton attached to C-15 at 4.61 ppm was apparently due to the anisotropic effect of the carbonyl group at C-8. The NMR data are sufficient to confirm the structure of 2,3,11,12-tetramethoxy-5,8,9,14-tetrahydro-6*H*-7,14-methanodibenz[*d*,*g*]azecin-8-one (**4a**). By the same structural and NMR data analysis for compound **4a**, it can be concluded that product of the acid-mediated reaction of **1b** is **4b**.

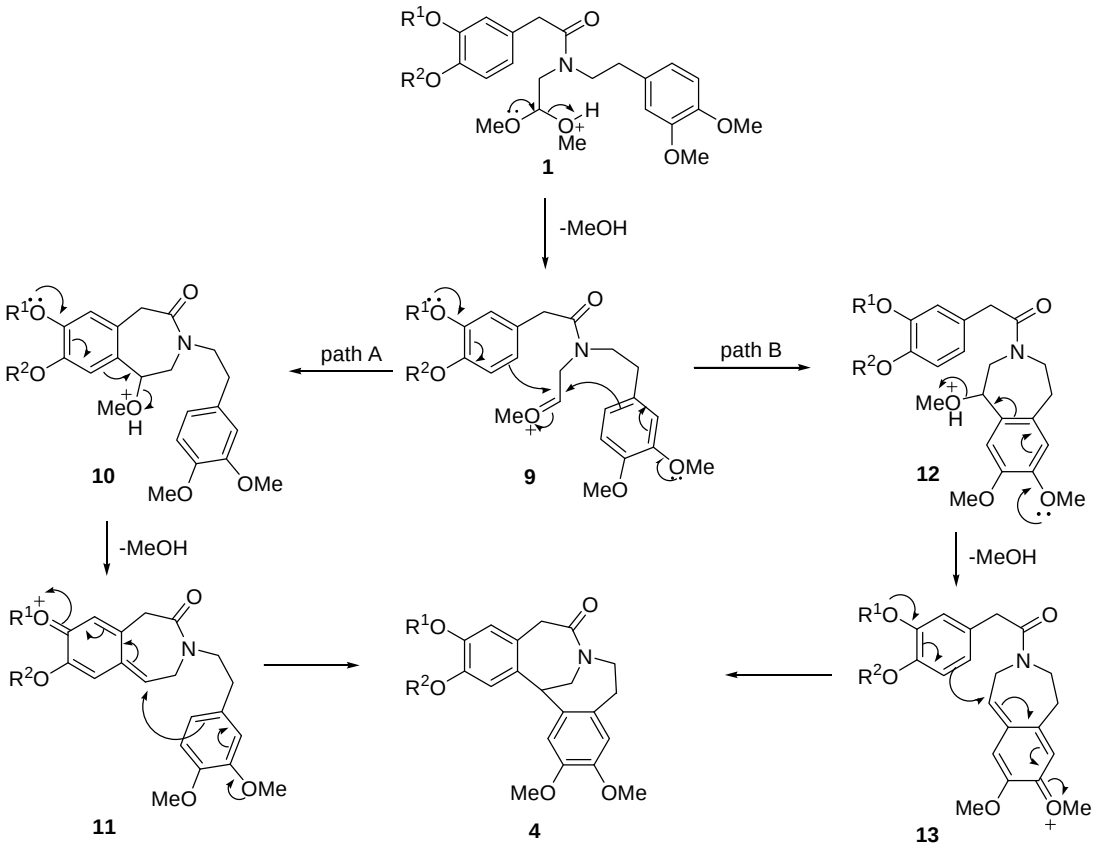
The acid-mediated cyclisation converting the amide acetals **1** to tetracyclic benzazepinones **4** was studied. Table 1 summerizes the results of our investigation when different acidic conditions were employed. The results show that changing HCl to $H_2SO_4$ in the mixed acid system with AcOH resulted in 31- 41% yields of the products. However, formic acid, a weaker acid, could be employed in the above transformation furnishing the desired benzazepinones **4a** and **4b** in 36-38% yields. The maximum yield was obtained within 4 h. Longer reaction time did not result in improved yield, as the starting amide acetals were depleted by that time.

**Table 1.** Tetracyclic products **4** from acid-mediated cyclisation

| Amide Acetals | Acids | Conditions | Time (h) | Yield[c] (**4a**) | Yield[c] **(4b)** |
|---|---|---|---|---|---|
| **1a** | HCl/AcOH[a] | 0 °C to rt | 17 | 35 | |
| **1a** | $H_2SO_4$/AcOH[b] | 0 °C to rt | 17 | 41 | |
| **1a** | $HCO_2H$ | reflux | 4 | 36 | |
| **1b** | HCl/AcOH[a] | 0 °C to rt | 20 | | 31 |
| **1b** | $H_2SO_4$/AcOH[b] | 0 °C to rt | 20 | | 33 |
| **1b** | $HCO_2H$ | reflux | 4 | | 38 |

[a]All reactions were performed with HCl/AcOH (1:1). [b]All reactions were performed with $H_2SO_4$/AcOH (1:5). [c]Isolated yield after chromatography on silica gel

A possible mechanism of acid-mediated cyclisation of amide acetals **1** to the benzazepinone products **4** is shown in Scheme 3.




Scheme 3

The reaction was initiated by the formation of oxonium ion **9** from acetal **1** which was followed by further intramolecular cyclisation reaction of one of the aromatic rings with oxonium ion to form the seven-membered rings of compounds **10** or **12** (path A or path B). Loss of methanol from **10** and **12** facilitated by the lone pair electrons on oxygen could lead to the reactive quinone methide intermediates **11** and **13** which could be trapped by the electron rich aromatic ring to give the *bis*-cyclisation product **4**. In conclusion the new benzazepinone core structures,[7] 2,3,11,12-tetramethoxy-5,8,9,14-tetrahydro-6*H*-7,14-methanodibenz[*d*,*g*]azecin-8-one **4a** and 2,3-dimethoxy-11,12-methylenedioxy-5,8,9,14-tetrahydro-6*H*-7,14-methanodibenz[*d*,*g*]azecin-8-one **4b**, could be conveniently obtained from acid-mediated cyclisation of amide acetals using different acidic conditions.

## EXPERIMENTAL

All common reagents were purchased from Merck, Fluka, and Labscan Asia Co., Ltd., Bangkok Thailand. Melting points were determined on a Electrothermal 9100 apparatus and are uncorrected. $^{1}H$ and $^{13}C$ NMR spectra were recorded on a Bruker DPX-300 using deuterated chloroform ($CDCl_3$) as the solvent. IR spectra were collected on a Perkin Elmer system 2000 FT-IR and JASCO A-302 spectromerters. Mass spectra were recorded on a Finnigan INCOS.50 and Bruker Daltonics (microTOF). Elemental analyses were performed on a Perkin Elmer Elemental Analyzer 2400 CHN.

**General procedure for the preparation of *N*-(3,4-dimethoxyphenethyl)-2,2-dimethoxyethanamine (8)** Amide **7** (2 mmol) dissolved in dry THF was slowly added into a stirred solution of lithium aluminium hydride (6 mmol) in dry THF at 0 $^{o}$C. The reaction was allowed to stir at rt and then refluxed for 6 h. Excess lithium aluminium hydride was decomposed with saturated aqueous sodium sulfate. The inorganic material was removed by filtration and washed with EtOAc. The aqueous layer was extracted twice with EtOAc. The combined organic layer was washed with water, dried (anh. $Na_2SO_4$), filtered, and evaporated *in vacuo* to give the crude *N*-(3,4-dimethoxyphenethyl)-2,2-dimethoxyethanamine **8** which was used in the next step without further purification.

**General procedure for the synthesis of amide acetals (1)** Oxalyl chloride (1.5 mmol) was added slowly to the stirred solution either of homoveratric acid **5a** (1 mmol) or 3,4-methylenedioxyphenylacetic acid **5b** and DMF (2 drops) in benzene (5 mL). The reaction was stirred at rt for 1 h, then concentrated under reduced pressure to give the crude acid chloride **6**. To the mixture of amine **8** (2 mmol), prepared *in situ* from $LiAlH_4$ reduction, in $CH_2Cl_2$ and sodium carbonate (4 mmol) in water, was added the solution of acid chloride in $CH_2Cl_2$. The reaction was stirred at rt for 2 h. Water was added and the two phases were separated. The aqueous layer was extracted with $CH_2Cl_2$ (3 x 10 mL). The combined organic extracts were washed with water, dried (anh. $Na_2SO_4$), filtered, and evaporated to give the crude amide acetals which were purified by PTLC using 50% EtOAc in hexane to give amide acetals **1** as a mixture of rotational isomers.

***N*-(2,2-Dimethoxyethyl)-*N*-(3,4-dimethoxyphenethyl)-2-(3,4-dimethoxyphenyl)acetamide (1a)** From 0.1962 g of homoveratric acid **5a,** (1 mmol), *N*-(2,2-dimethoxyethyl)-*N*-(3,4-dimethoxyphenethyl)-2-(3,4-dimethoxyphenyl)acetamide **1a** was obtained in 77% yield (0.3465 g) using the general procedure for the synthesis of amide acetal as described above. FTIR (UATR): $\nu_{max}$ 1636, 1513, 1451, 1417, 1025 $cm^{-1}$; $^1$H NMR ($CDCl_3$, 300 MHz) δ (ppm) major isomer 2.72 (t, *J* = 7.3 Hz, 2H), 3.40 (s, $OCH_3$, 6H), 3.42 (s, 2H), 3.45 (d, *J* = 5.3 Hz, 2H), 3.59 (t, *J* = 7.3 Hz, 2H), 3.85, 3.86, 3.87 (3s, Ar-OC*H*$_3$, 12H), 4.57 (t, *J* = 5.3 Hz, 1H), 6.61-6.84 (m, Ar*H*, 6H); minor isomer 2.79 (t, *J* = 7.3 Hz, 2H), 3.28 (d, *J* = 5.3 Hz, 2H), 3.37 (s, $OCH_3$, 6H), 3.59 (t, *J* = 7.3 Hz, 2H), 3.74 (s, 2H)**,** 3.85, 3.86, 3.87, 3.88 (4s, Ar-OC*H*$_3$, 12H), 4.25 (t, *J* = 5.3 Hz, 1H), 6.61-6.84 (m, Ar*H*, 6H); $^{13}$C NMR ($CDCl_3$, 75 MHz): δ (ppm) major isomer 34.5, 40.0, 48.4, 51.2, 55.3, 55.84, 55.86, 103.4, 111.1, 111.4, 111.92, 111.95, 120.7, 120.8, 127.5, 130.7, 147.79, 147.83, 148.97, 149.01, 171.7; minor isomer 33.3, 40.4, 51.0, 48.4, 55.1, 55.76, 55.81, 103.9, 111.1, 111.2, 111.9, 111.95, 120.6, 120.9, 127.5, 130.7, 147.4, 147.8, 148.8, 149.0, 171.8; LRMS (EI) 448 ($M^+$+1, 4), 447 ($M^+$, 11), 283 (28), 264 (35), 251 (46), 166 (41), 165 (100), 151 (35); HRMS (TOF). Calcd. for $C_{24}H_{34}NO_7[M+H]^+$: 448.2330. Found: 448.2336.

***N*-(2,2-Dimethoxyethyl)-*N*-(3,4-dimethoxyphenethyl)-2-(3,4-methylenedioxyphenyl)acetamide (1b)** From 0.1802 g of 3,4-methylenedioxyphenylacetic acid **5b** (1 mmol), *N*-(2,2-dimethoxyethyl)-

*N*-(3,4-dimethoxyphenethyl)-2-(3,4dimethoxyphenyl)acetamide **1b** was obtained in 73% yield (0.3158 g) using the general procedure for the synthesis of amide acetal as described above. FTIR (UATR): $\nu_{max}$ 1639, 1514, 1442, 1237, 1020 $cm^{-1}$; $^1H$ NMR ($CDCl_3$, 300 MHz) δ (ppm) major isomer 2.72 (t, *J* = 7.2 Hz, 2H), 3.41 (s, 6H, $OCH_3$), 3.43 (s, 2H), 3.44 (d, *J* = 5.3 Hz, 2H), 3.58 (t, *J* = 7.2 Hz, 2H), 3.87 (s, Ar-OC*H*$_3$, 6H), 4.57 (t, *J* = 5.3 Hz, 1H), 5.92 (s, 2H), 6.56-6.83 (m, 6H, Ar*H*); minor isomer 2.79 (t, *J* = 7.3 Hz, 2H), 3.29 (d, *J* = 5.2 Hz, 2H), 3.38 (s, $OCH_3$, 6H), 3.58 (t, *J* = 7.3 Hz, 2H), 3.69 (s, 2H), 3.82, 3.85, (2s, Ar-OC*H*$_3$, 6H), 4.29 (t, *J* = 5.2 Hz, 1H), 5.93 (s, 2H), 6.56-6.83 (m, Ar*H*, 6H); $^{13}C$ NMR ($CDCl_3$, 75 MHz): δ (ppm) major isomer 34.5, 40.0, 48.5, 51.2, 55.4, 55.87, 100.9, 103.4, 1082, 109.2, 111.4, 111.9, 120.8, 121.7 128.7, 130.7, 146.4, 147.77, 147.82, 149.0, 171.5; minor isomer 33.3, 40.3, 48.4, 50.9, 55.2, 55.75, 55.81, 100.9, 103.9, 108.3, 109.4, 111.1, 112.0, 120.7, 121.9, 128.8, 131.6, 146.4, 147.4, 147.8, 148.8, 171.6; LRMS (EI) 432 ($M^+$+1, 2), 431 ($M^+$, 6), 248 (13), 165 (20), 164 (100); HRMS (TOF). Calcd. for $C_{23}H_{30}NO_7[M+H]^+$: 432.2017. Found: 432.2019.

**Synthesis of 2,3,11,12-tetramethoxy-5,8,9,14-tetrahydro-6*H*-7,14-methanodibenz[*d,g*]azecin-8-one (4a)** *Reaction with HCl/AcOH*; Amide acetal **1a** (0.10 g, 0.22 mmol) was dissolved in AcOH (2.50 mL). Then, concentrated HCl (2.50 mL) was slowly added drop-wise at 0 °C and subsequently stirred at rt for 17 h. The reaction was then quenched with water, neutralized with 25% $NH_4OH$, and extracted with $CH_2Cl_2$ (3 x 20 mL). The combined organic layers were washed with water and brine, dried (anh. $Na_2SO_4$), filtered, and evaporated under reduced pressure to give crude product which was purified by PTLC using 60% EtOAc in hexane as a mobile phase to give the required benzazepinone **4a** in 35% yield (0.0302 g). Mp 243-244 °C; FTIR (KBr): $\nu_{max}$ 1656, 1633, 1607, 1516, 1464 $cm^{-1}$; $^1H$ NMR ($CDCl_3$, 300 MHz) δ (ppm) 2.46 (dd, *J* = 13.6, 4.1 Hz, 1H), 2.61 (t, *J* = 13.6, Hz, 1H), 2.81 (t, *J* = 13.6 Hz, 1H), 3.35 (d, *J* = 14.2 Hz, 1H), 3.59 (d, *J* = 15.2 Hz, 1H), 3.60, 3.86, 3.87, 3.95 (4s, 12H), 4.04 (d, *J* = 3.4 Hz, 1H), 4.51 (d, *J* = 14.2 Hz, 1H), 4.61 (dd, *J* = 15.2, 3.4 Hz, 1H), 4.63 (dd, *J* = 13.6, 4.1 Hz, 1H), 6.51 (s, Ar*H*, 1H), 6.66 (s, Ar*H*, 1H), 6.67 (s, Ar*H*, 1H), 6.85 (s, Ar*H*, 1H); $^{13}C$ NMR (75 MHz, $CDCl_3$) δ 36.6, 43.2, 47.6, 49.0, 52.1, 55.6, 55.78, 55.82, 56.3, 113.9, 114.0, 115.4, 115.5, 123.6, 129.1, 132.1, 136.6, 146.4, 147.2, 147.5, 147.9, 172.8; LRMS (EI) 384 ($M^+$+1, 22), 383 ($M^+$, 100), 382 (25), 355 (15), 354 (59), 340 (21), 311 (20), 296 (15), 295 (38), 165 (15); HRMS (TOF). Calcd. for $C_{22}H_{26}NO_5[M+H]^+$: 384.1805. Found: 384.1811; Anal. calcd for $C_{22}H_{25}NO_5$: C, 68.91; H, 6.57; N, 3.65. Found: C, 68.84; H, 6.74; N, 3.79.

*Reaction with $H_2SO_4$/AcOH*; Amide acetal **1a** (0.10 g, 0.22 mmol) was dissolved in AcOH (5.00 mL). Then conc. $H_2SO_4$ (1.00 mL) was slowly added drop-wise at 0 °C and subsequently stirred at rt for 17 h. Similar work-up and purification gave the required benzazepinone **4a** in 41% yield (0.0351 g).

*Reaction with refluxing $HCO_2H$*; Amide acetal **1a** (0.10 g, 0.22 mmol) was refluxed in $HCO_2H$ (10 mL) for 4 h. Similar work-up and purification gave the required benzazepinone **4a** in 36% yield (0.0310 g).

**Synthesis of 2,3-dimethoxy-11,12-methylendioxy-5,8,9,14-tetrahydro-6*H*-7,14-methanodibenz[*d*,*g*]-azecin-8-one (4b)** *Reaction with HCl/AcOH*; Amide acetal **1b** (0.10 g, 0.23 mmol) was dissolved in AcOH (2.50 mL). Then, conc. HCl (2.50 mL) was slowly added dropwise at 0 $^{o}$C and subsequently stirred at rt for 20h. Similar work-up and purification gave benzazepinone **4b** in 31% yield (0.0267 g). Mp 257.0-257.5 °C; FTIR (KBr) $\nu_{max}$ 1659, 1606, 1517, 1482, 1459, 1445 cm$^{-1}$; $^{1}$H NMR (300 MHz, $CDCl_3$) δ (ppm) 2.46 (dd, *J* = 13.9, 4.3 Hz, 1H), 2.62 (t, *J* = 13.9 Hz, 1H), 2.81 (t, *J* = 13.9 Hz, 1H), 3.31 (d, *J* = 14.2 Hz, 1H), 3.57 (d, *J* = 15.5 Hz, 1H), 3.87, 3.95 (2s, 6H), 4.01 (d, *J* = 4.0 Hz, 1H), 4.49 (d, *J* = 14.2 Hz, 1H), 4.60 (dd, *J* = 15.5, 4.0 Hz, 1H), 4.64 (dd, *J* = 13.9, 4.3 Hz, 1H), 5.84 (d, *J* = 1.3 Hz, 1H), 5.89 (d, *J* = 1.3 Hz, 1H), 6.50 (s, ArH, 1H), 6.65 (s, ArH, 1H), 6.68 (s, ArH, 1H), 6.81 (s, ArH, 1H); $^{13}$C NMR (75 MHz, $CDCl_3$) δ 36.5, 43.3, 47.5, 49.4, 52.0, 55.9, 56.2, 101.1, 110.6, 111.0, 115.4, 115.5, 124.5, 130.6, 131.8, 136.7, 146.3, 146.4, 146.9, 147.2, 172.6; LRMS (EI) 368 ($M^+$+1, 22), 367 ($M^+$, 100), 338 (51), 279 (39), 178 (47); HRMS (TOF). Calcd. for $C_{21}H_{22}NO_5[M+H]^+$: 368.1492 Found: 368.1500. Anal. calcd. for $C_{21}H_{21}NO_5$: C, 68.65; H, 5.76; N, 3.81. Found: C, 68.50; H, 5.54; N, 3.55.

*Reaction with $H_2SO_4$/AcOH*; Amide acetal **1b** (0.10 g, 0.23 mmol) was dissolved in AcOH (5.00 mL) then conc. $H_2SO_4$ (1.00 mL) was slowly added drop-wise at 0 $^{o}$C and subsequently stirred at rt for 20 h. Similar work-up and purification gave benzazepinone **4b** in 33% yield (0.0283 g).

*Reaction with refluxing $HCO_2H$;* Amide acetal **1b** (0.10 g, 0.23 mmol) was refluxed in $HCO_2H$ (10 mL) for 4 h. Similar work-up and purification gave benzazepinone **4b** in 38% yield (0.0325 g).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