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Antimicrobial activity of new synthesized aza -beta lactam and tetrazole derivatives bearing imidazo[2,1-b]benzothiazole moiety

Kh. T. A. Al-Sultani*, N. Al-Lami
Department of Chemistry, College of sciences, Baghdad University, Baghdad, Iraq

Abstract

This research, included prepare of some new aza-beta lactam and 1,2,3, 4-tetrazole derivatives from 2-aminobenzothiazole. The first step includes formation of imidazo[2,1-*b*]benzothiazoles (1) by the condensation of 2-aminobenzathiazole and ethyl-2- chloro acetoacetate in acetone, then compound (1) reaction with hydrazine hydrate 80% to form hydrazone derivative (2). Schiff bases (3-6) were prepared from condensation of hydrazone(2) with various aromatic aldehyde with little drops of glacial acetic acid. Phenyl isocyanate and sodium azide were used for the cyclocyclization of new Schiff bases to form diazetidine (7-10) and tetrazole (11-14) derivatives. Moreover, Newly prepared derivatives were measured by Fourier-transform infrared and some of them by ^{1}H & ^{13}C-NMR. Furthermore, some new derivatives were evaluated as antibacterial.

Keywords: imidazo[2,1-b] Benzthiazole,Schiff base, Diazetidine, 1, 2, 3, 4-Tetrazole,antibacterial.

Introduction

Imidazo benzothiazoles have been shown to be important heterocycles as a [2,1-*b*] result of their pharmacological activities [1] such as Antiproliferative [2], anticancer [3], antifungal [4], anti-Alzheimer's disease [5], antibacterial [6], immunological activites [7] and antimicrobial [8]. These compounds have been prepared from various precursors by adopting different methods [9]. Schiff's bases is a branch of organic chemistry with a very high importance[10]. It was described for the first time by the German chemist Hugo Schiff in (1864). Schiff bases was made up by the condensation between primary amines and compounds containing carbonyl group, such as aldehyde, ketone in absolute alcohol and a few drops of glacial acetic acid [11,12]. Azetidinones are the carbonyl derivatives of azetidines, these are also known as 2-azetidinones or more commonly aza-*β*-1actams[13], Cyclocondensation of Schiff's bases with phenyl isocyanate yields 1,3-diazetidin -2-one[14]. Tetrazole consists of five member ring of four nitrogen and one carbon atom [15]. The (2+3) cyclo addition method between nitriles and azides to be described common method to prepare the tetrazoles [16]. Tetrazole and their derivatives have different biological activities such as antifungal and antimicrobial [17]. Finally, this paper aims to synthesis and characterize some new azetidinones and tetrazole derivatives.

Experimental section

1. The FT-IR 8300 infrared spectrophotometer made up by SHIMADZU Company as a KBr disc was used as template in Science College, Baghdad University.
2. ^{1}H-NMR &^{13}C-NMR spectral chemical shifts were measured on Bruker Mega Hertz by using DMSO-d_6 as solvent.
3. Melting point (M.P.) was recorded by using Gallen Kamp melting point apparatus.
4. Antibacterial detection by Biology Department, Sciences College, University of Baghdad.

Synthesis of ethyl 2-methyl imidazo[2,1-b] benzthiazole-3-carboxylate [1] [18]

To solution of 2-chloro ethyl acetoacetate (3.8mL, 0.028mol) under dry conditions, 2-aminobenzothiazole (2.635 g, 0.028 mol) in acetone was added. To previous mixture, K_2CO_3 (3g) was added, and the resulting mixture was heated under reflux for 9hrs. The end of reaction was checked by TLC. Acetone was evaporated by vacuum distillation and the residue treated with ethyl acetate and

*Corresponding author e-mail: khitam.t@sc.uobaghdad.edu.iq
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petroleum ether. The solid was filtered, dried then purified by recrystallization from ethanol to give compounds [1]. The Physical properties of compound [1] are listed in Table (1).

Synthesis of 2-methylimidazo [2,1-b] benzothiazole-3-carbohydrazide [2][19]

Compound [1] (0.01mol) was dissolved in (15mL) abs. EtOH, and heated under reflux with hydrazine hydrate 80% (10mL) for 7hrs. Then cooled, filtered and purified by recrystallization from chloroform to give compounds [2]. The Physical properties of compound [2] are listed in Table (2).

Synthesis of, (*E*)- N'-(4- substituted benzylidene)-2-methyl imidazo[2,1-b] benzthiazole-3-carbohydrazide[3-6][11]

Series of Schiff bases compounds were prepared from the reaction of compound [3-6] (0.003mol) in (25 mL) abs. EtOH, different aromatic aldehydes (0.003mol) were added with little drops of glacial AcOH. The resulting mixture was heated under refluxed for (6-7) hrs. Then, cold water was added, and the solid was obtained, filtered and purified by recrystallization from different appropriate solvents. The Physical properties of compound [3-6] are listed in Table (3).

Table 1. The Physical properties of compound (1)

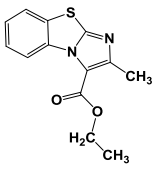
Comp. No.	Physical properties				
	Structures	M.P. C°	Yield%	Color	Res.
1		160 Decom.	90	Light brown	Ethanol

Table 2. The Physical properties of compound (2)

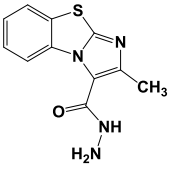
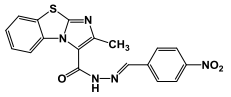
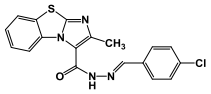
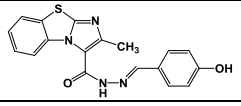
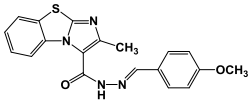
Comp. No.	Physical properties				
	Structures	M.P. C°	Yield%	Colour	Res.
2		160 Decom.	90	Light brown	Ethanol

Table 3. The Physical properties of compounds (3-6)

Comp. No.	Physical properties				
	Structures	M.P. C°	Yield %	Colour	Res.
3		126-128	40	Dark yellow	Acetone
4		186-188	65	Pale yellow	Methanol
5		175-178	30	Brown	Ethanol
6		210-213	43	Off white	Methanol

Synthesis of 2-methyl-N-(2-(4-substitutedphenyl)-4-oxo-3-phenyl-1,3-diazetidin-1-yl) imidazo[2,1-b] benzthiazole-3-carboxamide[7-10][20].

A mixture of compounds [3-6] (0.003mol) and phenylisocyanate (0.003 mol) in chloroform (20mL) was refluxed for 6 hrs. Evaporation of solvent then the residue was treated with ethyl acetate: petroleum ether (1:1) as mixture. The solid was filtered, dried then purified by recrystallized from different appropriate solvents. The Physical properties of compound [7-10] are listed in Table (4).

Synthesis of N-(5-(4-substitutedphenyl)-1H-tetrazol-1-yl)-2-methylimidazo[2,1-b] benzthiazole-3-carboxamide[11-14][20]

A mixture of compounds [3-6] (0.003mol) and NaN_3 (0.03g, 0.003 mol) in (20mL) of (THF) was added. The brevious mixture was heated under refluxe for (12-14) hrs. The solid was filtered, dried then purified by recrystallized from different appropriate solvents. The Physical properties of compound [11-14] are listed in Table (5).

Table 4. The Physical properties of compounds (7-10)

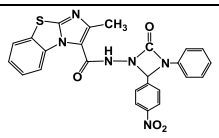
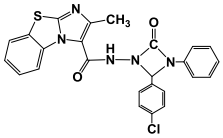
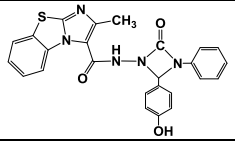
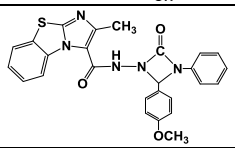
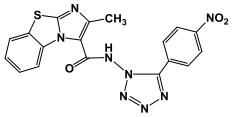
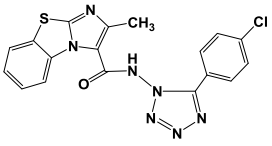
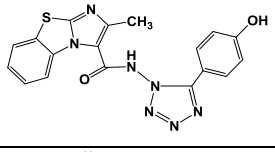
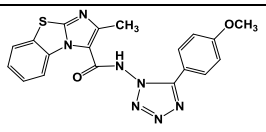
Comp. No.	Physical properties				
	Structures	M.P. C°	Yield %	Colour	Res.
7		238-240	62	Yellow	Ethanol
8		217-219	80	Off white	Ethanol
9		147-150	60	Pale yellow	Ethanol
10		145-148	54	Yellow	Ether

Table 5. The Physical properties of compounds (11-14)

Comp. No.	Physical properties				
	Structures	M.P. C°	Yield%	Colour	Res.
11		243-245	44	Dark yellow	Ethanol
12		165-167	90	yellow	Ethanol
13		186 Decom.	60	Brown	Ethanol
14		225-228	75	Dark brown	dioxane

Results and Discussion

Synthesis of new derivatives of diazetidine and 1,2,3,4-Tetrazole of 2-aminobenzothiazole found in scheme (1).

Reaction of 2-amino benzothiazole with ethyl-2-chloroaceto acetate in dry acetone as solvent yield compound [1]. Ester test (hydroxamic acid) was gave positive indication of formation ester[21]. FT-IR spectrum of compound [1] exhibited of vanishing of ν(NH2) starching band at 3430 asym. cm^{-1},3184 sym. cm^{-1} and revealing of feature bands at 2983cm^{-1} belong υ (C-H) aliphatic, 1724, 1267, and 1126 cm^{-1} which owing to of carbonyl,. Asym. and sym. ν (C-O) of ester respectively [21]. Other bands exhibited at 1641 cm^{-1} and 1565 cm^{-1} owing to ν(C=N) and ν(C=C) aromatic [22]. The FTIR data of compound [1] are listed in Table (6). The ^{1}H & ^{13}C NMR signal date for compound [1] found in Tables (11) &(12) respectively.

Hydrazide [2] was formed by reaction of ester [1] with hydrazine hydrate, This reaction occurred by nucleophilic attack of amino group on carbonyl group, after that elimination of ethanol. FT-IR spectrum of prepared compound [2] showed emergence of absorptions bands at 3350 cm^{-1} and at 3245 cm^{-1} sym of NH_2 asym. and sym. Respectively, 3170 cm^{-1} of NH group, Also it showed shift in the νC=O band from 1724 cm^{-1} of carbonyl of ester to 1649 cm^{-1} of amide. The FTIR data of compound [2] are listed in Table(7). The ^{1}H-NMR signal date for compound [2] found in Tables (11).

Schiff's bases derivatives [3-6] were synthesized from the reaction of hydrazide [2] and a number of substituted aromatic aldehydes. The FTIR of compounds [2-7] includes the disappearance of ν (N-H2) absorption band and appearance of new bands at (1664-1610) cm^{-1} because of the formation of imine group (C=N). In addition to appearance of ν (CH) aromatic bands at (3066-3056) cm^{-1}, ν (C=O) amide absorption bands at 1697-1685 cm^{-1} and ν (C=C) aromatic bands at (1512-1444) cm^{-1}. The FTIR data of compound [3-6] are listed in Table (8). The ^{1}H & ^{13}C NMR signal date for compound [6] found in Tables (11) & (12) respectively.

Two different reagents were used in closing Schiff bases derivatives. The first method is when the Schiff bases reaction with phenyl isocyanate via [2+2] cycloaddition reaction producing Aza-β-lactam compounds of [7-10].

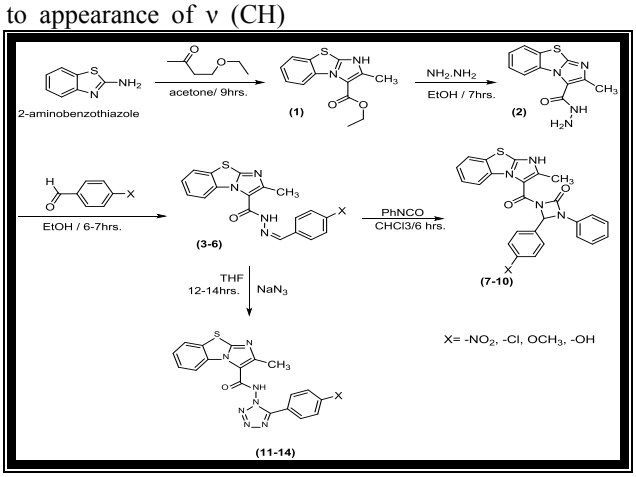
The FT-IR spectra showed the absence of the imine group (CH=N-) absorption band at (1664-1610) cm^{-1} and the emergence new absorption band, of carbonyl group (C=O aza-β-lactam) at (1764-1712) cm-1, these results gave a good evidence for the formation of the aza-β-lactam derivatives. The FTIR data of compound [7-10] are listed in Table (9). The ^{1}H & ^{13}C NMR signal date for compound [7-10] found in Tables (11) & (12) respectively.

Imine derivatives [3-6] reaction with NaN_3 in second method to yield derivatives [11-14].

This reaction takes place according to [3+2] cyclo addition of unsaturated systems to 1,3-dipoles to yield five-member ring [23].

FT-IR of compounds [11-14] showed peaks at (1539-15052) were owing (N=N) tetrazole ring. As well as, the FT-IR for these compounds emerge other bands at (1701-1666) cm^{-1}, (1683-1604) $cm^{-1}$and (1610-1556) cm^{-1} due to carbonyl amide, υ(C=N) group and υ(C=C) aromatic respectively[24]. The FTIR data of compound [11-14] are listed in Table (10). ^{1}H-NMR and ^{13}C-NMR spectrum of compound [11-14] listed in Table (11) and (12) respectively.

The antibacterial activity achieved by disk diffusion method[25]. The results of antibacterial activity are listed in Table (13).



Scheme (3.1)

Table 6. The FTIR spectral data of compound (1)

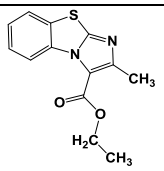
Comp. No.	Structures	Major FTIR Absorption Cm^{-1}				
		νC-H arom. aliph.	νC=O ester	νC=N	νC=C arom.	Others
1		3068 2983	1724	1641	1565	ν(C-O) Ester 1267 1126

Table 7. The FTIR spectral data of compound (2)

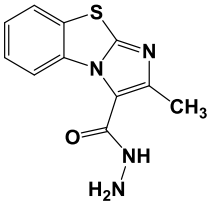
Comp. No.	Structures	Major FTIR Absorption Cm^{-1}					
		ν(N-H_2) asym. Sym.	ν(N-H)	νC-H arom. aliph.	νC=O Amid	νC=N	νC=C arom.
2		3350 3245	3170	3066 2964	1649	1577	1560

Table 8. The FTIR spectral data of compounds (3-6)

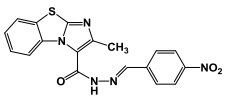
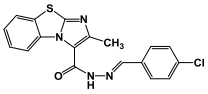
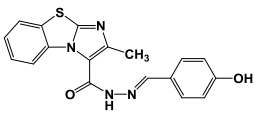
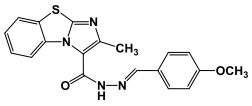
Comp. No.	Structures	Major FTIR Absorption Cm^{-1}					
		νN-H	νC-H arom. aliph.	νC=O Amid	νC=N Imine imidazo	νC=C arom.	Others
3		3139	3064 2977	1697	1623 1596	1444	ν(NO_2) Asym 1521 sym1346
4		3473	3066 2941	1685	1625 1593	1490	ν (C-Cl) 1091
5		3182	3056 2956	1695	1664 1604	1512	ν(C-OH) 3342
6		3434	3060 2997	1695	1610 1573	1502	ν (C-O-C) 1271,1157

Table 9. The FTIR spectral data of compounds (7-10)

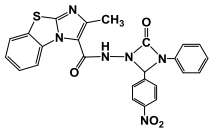
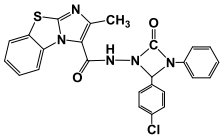
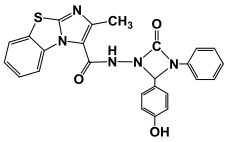
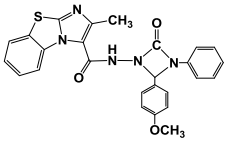
Comp. No.	Structures	Major FTIR Absorption Cm^{-1}					
		νN-H	νC-H arom. aliph	νC=O β-lactam amid	νC=N	νC=C arom.	Others
7		3190	3099 2947	1735 1693	1591	1566	ν(NO_2) asym1566 sym1371 ν (C-N) 1328
8		3164	3083 2991	1764 1699	1591	1527	ν (C-Cl)1081 ν (C-N)1313
9		3290	3058 2987	1712 1649	1596	1552	ν(C-OH) 3328 ν (C-N)1313
10		3211	3090 2971	1712 1652	1590	1554	ν(C-O-C) 1240,1158 ν (C-N) 1340

Table 10. The FTIR spectral data of compounds (11-14)

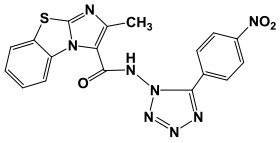
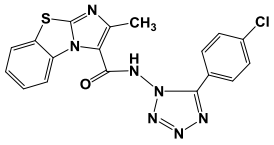
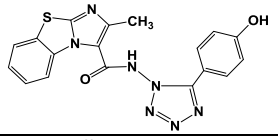
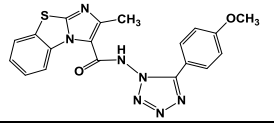
Comp. No.	Structures	Major FTIR Absorption Cm^{-1}					
		νN-H	νC-H arom. aliph	νC=O	νC=N	νC=C arom.	Others
11		3132	3047 2974	1681	1681	1595	νN=N 1519 νNO_2 Asym 1519 sym1346
12		3450	3099 2987	1685	1623	1593	νN=N 1539 νC-Cl 1089
13		3174	3024 2923	1666	1604	1556	ν(C-OH) 3303 ν(N=N) 1514
14		3438	3020 2975	1701	1683	1610	νN=N 1502 νC-O-C 1271,1157

Table 11. ^{1}H-NMR spectral data (δ ppm) of some prepared compounds

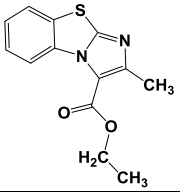
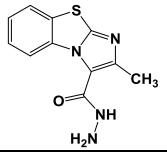
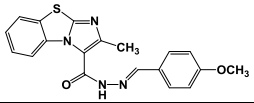
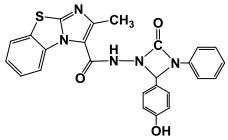
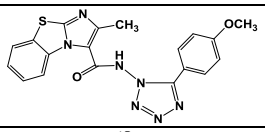
No.	Structures	^{1}HNMR Spectral data($^{\delta}$ppm)
1		1.05(t,3H,-CH$_2$-CH$_3$); 2.20 (s,3H, CH$_3$); 4.11 (q,2H, -O-CH$_2$); 6.97-7.90 (m,4H,Ar-H)
2		1.34 (s,3H,CH$_3$); 4.70 (s,2H,NH$_2$); 6.97-7.84 (m,4H,Ar-H); 8.56 (s,1H,-CO-N-H) 9.05 (s,1H,N-H)
6		2.21 (s,3H,CH$_3$); 3.81 (s,3H,OCH$_3$); 6.98 (s,1H, N=C-H); 7.00-7.96 (m,7H,Ar-H); 8.82 (s,1H, CO-N-H)
9		1.23 (s,3H, CH$_3$); 4.14(s,1H, OCH$_3$);6.62(s,1H, CH aza-β-lactam); 6.88-7.47 (m,14H,Ar-H) ; 8.69 (s, 1H, -(CO)-NH-) ;9.59 (s,1H, OH)
14		1.22 (s,3H,CH$_3$); 3.60 (s,3H,OCH$_3$); 6.62-7.91 (m,8H,Ar-H); 8.82 (s,1H, NH)

Table 12. ^{13}C-NMR spectral data (δ ppm) of some prepared compounds

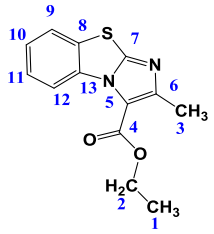
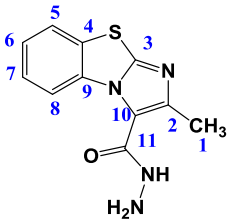
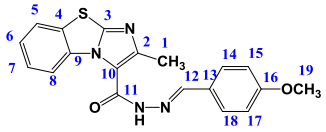
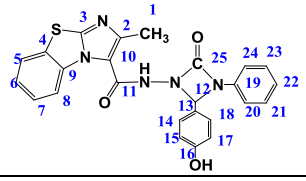
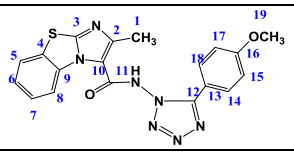
No.	Structure	^{13}CNMR spectral data (δ ppm)
1		23.82(C1); 25.06(C3); 61.67(C2);118.16-132.08 (C-5,6 ,8,9, 10, 11, 12, 13); 153.29 (C7),174.37(C4).
2		18.11(C1);118.21-131.41 (C-2,4,5,6 ,7,8,9,10) 153.29 (C3),166.94(C11).
6		22.70(C458-9); 55.84(C1); 117.68-131.40 (C-3,4,5,6 ,7, 10, 12, 13,14,15,16,17,18); 148,49(C8);152.76 (C11),157.88(C19) 166.37(C2).
9		20.09(C1); 54.73(C9) 118.65-129.24 (C-2,4,5,6 ,7,8,9,10 ,12, 13,14,15,16,17,18,19,20,21,22,23,24); 140.18(C3);153.01(C-11,25).
14		16.77(C1); 56.35(C19); 107.02-128.18 (C-2,4,5,6 ,7,8,9, 10, 13,14,15,16,17,18); 155.97(C8);160.62 (C12),163.84(C11)

Table 13. Anti-bacterial activity for some prepared compounds

Comp. No.	*Staphylococcus aureus+ve*	*Streptococcus +ve*	*Klepsilla –ve*	*Escherichia coli–ve*
4	11	-	26	32
5	30	28	38	28
7	13	15	11	13
8	14	13	-	12
12	26	26	28	30
14	26	26	30	32

Conclusion

Anew derivatives of imidazo(2,1-b)benzthiazole were evaluated against different types of strain cells of bacteria, and antibacterial tests showed promising results regarding inhibition activity of these types, where some of these derivatives exhibited strong activity, others showed moderate. Moreover, these results confirmed antimicrobial activities of imidazo/benzthiazole derivatives, which were reported in literatures. These derivatives were synthesised in five sequence steps, starting from 2-amino benzthiazole and ended with introduced new five heterocycles of tetrazole and aza-beta lactam . Most of new derivatives were confirmed their structures precisely by FT-IR, ^{1}H-NMR, ^{13}C-NMR spectroscopy. These results encourage us to plan a new metholodgy of synthesis a new derivatives of imidazo/benzthiazole with study their pharmacoligal activities**.**

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الفعالية المضادة للمايكروبات لمشتقات جديدة من ازو – بيتالاكتام وتترازول محضرة من جزيئة اميدازو[2,1- ب]بنزوثايازول

ختام طارق احمد، نعيمة جبار عويد

قسم الكيمياء، كلية العلوم، جامعة بغداد، بغداد، العراق

الخلاصة

تضمن هذا البحث تخليق بعض مشتقات ازو- بيتا لاكتام و 4-1,2,3 تترازول الجديدة من 2- امينوبنزوثايازول. تتضمن الخطوة الأولى تكوين اميدازو[2,1- ب]بنزوثايازول (1) بواسطة تكاثف 2- امينوبنزوثايازول واثيل -2- كلورو اسيتواسيتيت بوجود الاسيتون , ثم المركب (1) يتفاعل مع الهيدرازين المائي80% لتكوين مشتق الهيدرازون (2) . تم تحضير قواعد شيف(3-6) من تكاثف الهيدرازون(2) مع الديهايدات اروماتية مختلفة مع قطرات قليلة من حامض الخليك الثلجي.تم استخدام فنيل ازوسيانيد وازيد الصوديوم للغلق الحلقي لقواعد شيف لتكوين مشتقات داي ازيدتين (7-10) وتترازول (11-14). المشتقات المحضرة الجديدة تم تشخيصها بواسطة الأشعة تحت الحمراء وبعضها بواسطة الرنين النووي المغناطيسي .تم تقييم بعض المشتقات المحضرة كمضادات للبكتريا

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