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SYNTHESIS OF N-MALTOSYLATED THIOCARBAMIDES, THIOCARBAMATES AND BENZOTHIAZOLYL THIOCARBAMIDES

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ABSTRACT

Several 1-hepta-O-acetyl-β-D-maltosyl-3-aryl thiocarbamides **(IIa-g)**, N-hepta-O-acetyl-β-D-maltosyl-O-alkyl thiocarbamates **(IIIa-f)** and 1-hepta-O-acetyl-β-D-maltosyl-3-(2)-substituted benzothiazolyl thiocarbamides **(IVa-g)** have been prepared by the interaction of hepta-O-acetyl-β-D-maltosyl isothiocyanate **(I)** with various aryl amines, alcohols and 2-aminobenzothiazole/ substituted benzothiazoles. The identities of these newly synthesized compounds were established on the basis of elemental analysis, I. R., ^{1}H NMR and Mass spectral analysis.

Key words: Maltosyl isothiocyanate, Aryl amines, Thiocarbamates, Substituted benzothiazoles.

INTRODUCITON

Sugar isothiocyanates[1,2] are versatile synthetic intermediates that have been used in the field of synthetic carbohydrate chemistry. In the same way, maltosyl isothiocyanate[3-5] is used as versatile intermediate in carbohydrate chemistry.

The N-maltosylated compounds show great potential in biological processes and medicinal chemistry. They act as diuretic agents and bacteriostatic agents[6], which is also used as an antifungal[7], antitumor[8] and antidibetic agents. Carbamides and their derivatives shows strong antibacterial activities[9]. Benzothiazoles are bicyclic ring system with multiple applications. They have broad spectrum of biological activity[10]. Bis-substituted benzothiazole acts as the potential anti-HIV agent[11].

In view of these applications, We wish to report the synthesis of new series of N-maltosylated title compounds by the interaction of hepta-O-acetyl-β-D-maltosyl

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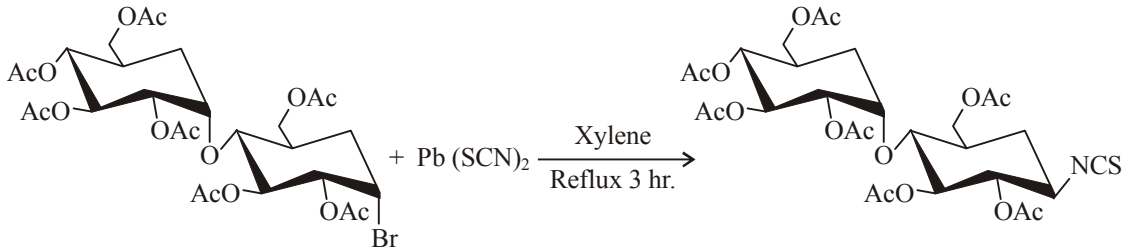
isothiocyanate **(I)** with various aryl amines, alcohols and 2-aminobenzothialzole/ substituted benzothiazoles.

EXPERIMENTAL

Melting points were taken in open capillary tubes and are found uncorrected. Optical rotations $[\alpha]_D$ were measured on Equip-Tronics EQ 800 Digital polarimeter in $CHCl_3$. I.R. spectra were recorded on Perkin-Elmer RXI (4000-450 cm^{-1}) FTIR spectrometer[12,13]. ^1H NMR were obtained on a Bruker DRX-300 NMR spectrometer[14,15]. The samples were prepared in $CDCl_3$ with TMS as an internal reference. The mass spectra were recorded on SX-102 FAB mass spectrometer[16,17].



Hepta-O-acetyl-α-D-maltosyl bromide — Lead thiocyanate — Hepta-O-acetyl-β-D-maltosyl isothiocyanate (I)

Scheme 1

General procedure

Hepta-O-acetyl-β-D-maltosyl-3-aryl thiocarbamide **(IIa-g)** and hepta-O-acetyl-β-D-maltosyl-3-(2)-substituted benzothiazolyl thiocarbamides **(IVa-g)** were prepared by the interaction of hepta-O-acetyl-β-D-maltosyl isthiocyanate **(I)** and various amines and 2-aminobenzothiazole/substituted benzothiazoles in a benzene medium for 3 hr. and 4 hr., respectively. After condensation, the solvent was distilled off and sticky residue was obtained, which was triturated with petroleum ether (b.p. 60-80^oC) to afford a white solid. It was purified by water-ethanol.

N-Hepta-O-acetyl-β-D-maltosyl-O-alkyl thiocarbamates **(IIIa-f)** were prepared by the reaction of hepta-O-acetyl-β-D-maltosyl isothiocyanate **(I)** and various alcohols by refluxing for 3 hr. After condensation, the reaction mixture was poured in ice cold water with vigorous stirring where white granular solid separated out.

The required amines and alcohols were commercially obtained, while 2-amino-benzothiazole/ substituted benzothiazoles were prepared by oxidative cyclisation of aryl

thiocarbamides with help of molecular bromine. 1-Aryl thiocarbamides have been prepared by the interaction of ammonium thiocyanate with aryl amines hydrochlorides. The structures of title compounds were confirmed by elemental analysis, IR, ^{1}H NMR and Mass spectra.

Hepta-O-acetyl-β –D-maltosyl isothiocyanate (I)

Hepta-O-acetyl-β-D-maltosyl isothiocyanate **(I)** has been prepared by condensation reaction of hepta-O-acetyl-α-D-maltosyl bromide (0.005 M, 4 g) with lead thiocyanate (0.004 m, 1.98 g) in boiling xylene for 3 hr. After completion of reaction, the xylene solution is filtered and the excess xylene has been removed by distillation. The sticky residue obtained was triturated several times with petroleum ether (b.p. 60-80^{o}C) to afford a solid. It was then purified from $CHCl_3$. The purity of product was checked by TLC.

1-Hepta-O-acetyl-β-D-maltosyl-3-aryl thiocarbamides (IIa-g) (Scheme 2) (Table 1)

1-Hepta-O-acetyl-β-D-maltosyl-3-aryl thiocarbamides **(IIa-g)** were prepared by the condensation of Hepta-O-acetyl-β-D-maltosyl isothiocyanate **(I)**, (0.005 M, 3.5 g) with aryl amines (0.005 M) in boiling benzene for 3 hr. After completion of reaction, the excess of benzene was distilled off and sticky residue obtained was triturated with petroleum ether (b.p. 60-80^{o}C) to afford a solid. All products **(IIa-g)** were purified from ethanol-water and purity of compounds was checked by TLC. The products were found desulphurisable when boiled with plumbite solution and also show positive charring test when heated with conc. H_2SO_4. The % yield, M.P., optical rotation, R_f value and elemental analysis are shown in Table 1.

Table 1: 1-Hepta-*O*-acetyl-β –D-maltosyl–3-aryl thiocarbamides (IIa-g)

Reactants : (1) Hepta-*O*-acetyl-β-D-maltosyl isothiocyanate **(I)** (0.005 M, 3.5 g)
(2) Aryl amines (0.005 M)

Products (IIa-g)	Amines (g)	Yield (%)	M.P. (°C)	$[\alpha]_D^{32}$ (c,$CHCl_3$)	Analysis		R_f (CCl_4 : EtOAc)
					Found (%)	Required (%)	
IIa	Aniline (0.48)	75.78	110	+ 87.66° (c, 1.0266)	N, 3.50 S, 4.02	N, 3.63 S, 4.15	0.86 (3 : 2)
IIb	*o*-Cl-Aniline (0.65)	62.18	222	+ 180.01° (c, 0.3333)	N, 3.40 S, 4.00	N, 3.48 S, 3.97	0.83 (3 : 2)
IIc	m-Cl-Aniline (0.65)	58.00	112	+ 153.09° (c, 0.3266)	N, 3.46 S, 3.89	N, 3.48 S, 3.97	0.84 (3 : 2)

Cont…

Products (IIa-g)	Amines (g)	Yield (%)	M.P. (°C)	$[\alpha]_D^{32}$ ($c,CHCl_3$)	Analysis		R_f (CCl_4 : EtOAc)
					Found (%)	Required (%)	
IId	p-Cl-Aniline (0.65)	62.88	161	+ 111.11° (c, 0.3600)	N, 3.45 S, 4.10	N, 3.48 S, 3.97	0.83 (3 : 2)
IIe	*o*-Toluidine (0.55)	63.56	202	+ 235.29° (c, 0.3400)	N, 3.53 S, 4.08	N, 3.57 S, 4.08	0.81 (3 : 2)
IIf	m-Toluidine (0.55)	65.79	230	+ 294.11° (c, 0.3400)	N, 3.39 S, 4.06	N, 3.57 S, 4.08	0.78 (3 : 2)
IIg	p-Toluidine (0.55)	58.00	134	+ 91.85° (c, 0.3266)	N, 3.52 S, 4.17	N, 3.57 S, 4.10	0.90 (3 : 2)

Satisfactory C & H analysis was found in all cases.

N-Hepta-O-acetyl-β-D-maltosyl-O-alkyl thiocarbamates (IIIa-f) (Scheme 2) (Table 2)

N-Hepta-O-acetyl-β-D-maltosyl-O-alkyl thiocarbamates **(IIIa-f)** were prepared by the condensation of hepta-O-acetyl-β-D-maltosyl isothiocyanate **(I)** (0.005 M.3.5 g) with various alcohols (20 mL) for 3 hr. After completion of reaction, the reaction mixture was poured in ice cold water with vigorous stirring to obtain products **(III a-f)**. All products were purified form ethanol-water and purity of compounds was checked by TLC. The % yield, M.P., optical rotation, R_f value and elemental analysis are shown in Table 2.

Table 2: N-hepta-*O*-acetyl-β-D-maltosyl–*O*-alkyl thiocarbamates (IIIa-f)

Reactants : (1) Hepta-*O*-acetyl-β-D-maltosyl isothiocyanate (3.5 g, 0.005 M)

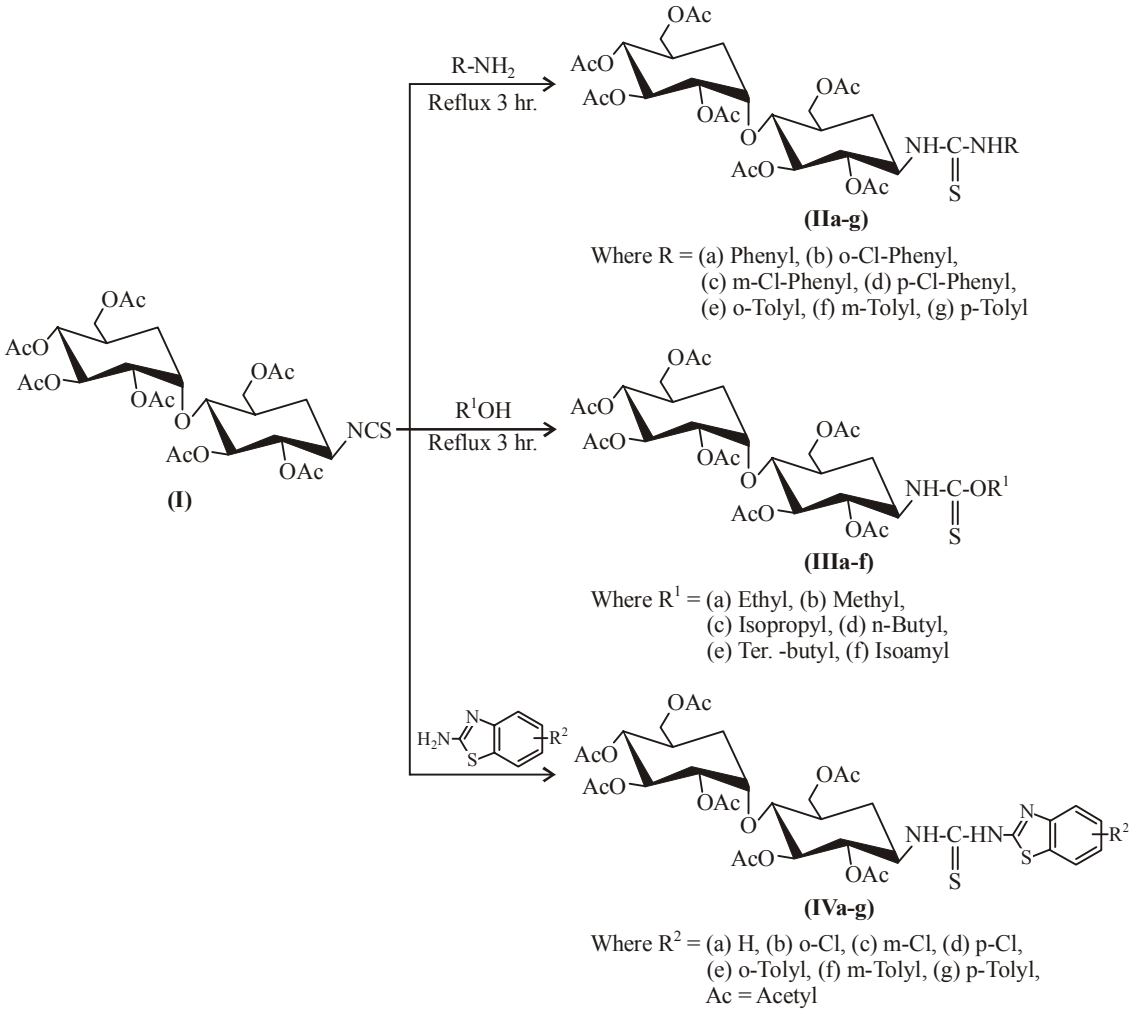
(2) Alcohols

Products (IIIa-f)	Alcohols	Yield (%)	M.P. (°C)	$[\alpha]_D^{36}$ ($c,CHCl_3$)	Analysis		R_f (CCl_4 : EtOAc)
					Found (%)	Required (%)	
III a	Ethyl	75	136	+ 20.20° (c,0.9933)	N, 1.91 S, 4.71	N, 1.92 S, 4.42	0.84 (3 : 2)
III b	Methyl	88	218	+ 98..03° (c, 1.02)	N, 1.86 S, 4.48	N, 1.97 S, 4.51	0.84 (3 : 2)
III c	Isopropyl	65	204-206	-40° (c, 1.00)	N, 1.93 S, 4.41	N, 1.89 S, 4.34	0.69 (3 : 2)

Cont…

Products (IIIa-f)	Alcohols	Yield (%)	M.P. (°C)	$[\alpha]_D^{36}$ (c,$CHCl_3$)	Analysis		R_f (CCl_4 : EtOAc)
					Found (%)	Required (%)	
IIId	n-Butyl	74	170-174	+ 39.21° (c, 1.02)	N, 1.85 S, 4.36	N, 1.86 S, 4.26	0.52 (3 : 2)
IIIe	t-Butyl	65	225	-20° (c, 1.00)	N, 1.91 S, 4.28	N, 1.86 S, 4.26	0.94 (3 : 2)
IIIf	Isoamyl	68	113	+ 133.33° (c, 1.02)	N, 1.80 S, 4.23	N, 1.82 S, 4.18	0.90 (3 : 2)

Satisfactory C & H analysis was found in all cases



Scheme 2

1-Hepta-O-acetyl-β-D-maltosyl-3-(2)-substituted benzothiazolylthiocarbamides (IVa-g), (Scheme 2) (Table 3)

1-Hepta-O-acetyl-β-D-maltosyl-3-(2)-substituted benzothiazolyl thiocarbamides **(IVa-g)** were prepared by the condensation of hepta-O-acetyl-β-D-maltosyl isthiocyanate **(I)**, (0.005 M, 3.5 g) with 2-aminobenzothiazole and substituted benzothiazoles (0.005M) in boiling benzene for 4 hr. After completion of reaction, the excess of solvent was distilled off and sticky residue obtained was triturated several times with petroleum ether (b.p. 60-80^{o}C) to afford a solid. All products **(IVa-g)** were purified by ethanol-water and the purity of compound was checked by TLC. The % yield, M.P., optical rotation, R_f value and elemental analysis are shown in Table 3.

Table 3: 1-Hepta-*O*-acetyl-β-D-maltosyl-3-(2)-substituted benzothiazolyl thiocarbamides (IVa-g)

Reactants : (1) Hepta-*O*-acetyl-β-D-maltosyl isothiocyanate **(I)** (0.005 M, 3.5 g)

(2) 2-Aminobenzothiazole/Substituted benzothiazloes (0.005 M)

Product (IV a-g)	Substituted benzothiazoles (g)	Yield (%)	M.P. (°C)	$[\alpha]_D^{38}$ (c,$CHCl_3$)	Analysis		R_f (CCl_4 : EtOAc)
					Found (%)	Required (%)	
IVa	2-Amino- (0.75)	92.85	138	+ 70° (c, 1.00)	N, 4.98 S, 7.72	N, 5.07 S, 7.73	0.6 (3 : 2)
IVb	-*o*-Cl- (0.92)	77.27	118-120	+ 144.23° (c, 1.04)	N, 4.92 S, 7.40	N, 4.87 S, 7.43	0.91 (3 : 2)
IVc	-m-Cl- (0.92)	72.07	135	+ 166° (c, 1.2)	N, 4.92 S, 7.40	N, 4.87 S, 7.43	0.54 (3 : 2)
IVd	-p-Cl- (0.92)	65.31	145-147	+ 37.73° (c, 1.06)	N, 4.90 S, 7.41	N, 4.87 S, 7.43	0.71 (3 : 2)
IVe	-*o*-Tolyl- (0.82)	64.51	105-108	+ 58.82° (c, 1.02)	N, 4.98 S, 7.63	N, 4.99 S, 7.60	0.38 (3 : 2)
IVf	-m-Tolyl- (0.82)	71.42	155-158	+ 27.07° (c, 1.1)	N, 5.0 S, 7.64	N, 4.99 S, 7.60	0.61 (3 : 2)
IVg	-p-Tolyl- (0.82)	70	116-117	+ 103.77° (c, 1.06)	N, 5.09 S, 7.75	N, 4.99 S, 7.60	0.79 (3 : 2)

Satisfactory C & H analysis was found in all cases

RESULTS AND DISCUSSION

Hepta-O-acetyl-β-D-maltosyl isothiocyanate (I)

Yield 72%, M.P. 118-120°C, $[\alpha]_D^{32}$ + 30^0 (c, 0.3333 mol, chloroform), R_f-0.76 (CCl_4: EtOAc, 3:2), IR (KBr); *v* 2962 (ali. C-H), 2032 (N=C=S), 1753 (C=O), 1375 (C-N), 1231 (C-O), 1039.7 and 938.9 (characteristic of maltose), 770 cm^{-1} (C-S); ^1H NMR ($CDCl_3$):δ 5.4-3.7 (m, 14H, maltosyl ring protons); 2.2-2.0 (m, 21H, 7-$COCH_3$); Mass (m/z): 677 (M^+), 619, 559, 457, 331, 169, 109. Anal. Calcd. for $C_{27}H_{35}O_{17}SN$, C,47.85; H,5.16, N, 3.63; S,4.15 found C,47.80; H,5.20; N,3.50; S, 4.02%

1-Hepta-O-acetyl-β-D-maltosyl-3-phenyl thiocarbamides (IIa)

IR (KBr)**:** *v* 3430 (N-H), 2817 (Ar-H), 1750 (C=O), 1352 (C-N), 1236 (C-O), 1043.3 and 899.6 (Cha. maltose ring), 766.2 (benzene ring), 669.6 cm^{-1} (C-S); ^1H NMR ($CDCl_3$) δ 8.2-7.17 (m, 5H, Ar-H); 6.54-6.51 (m, 1H, maltosyl NH); 5.86-3.8 (m, 14H, maltosyl rings protons); 2.15-1.9 (m, 21 H, acetyl protons); Mass (m/z): 770 (M+), 771, 669, 651, 619, 559, 331, 169, 109. Anal. Calcd. For $C_{33}H_{42}O_{17}N_2S$; C, 51.42; H, 5.45; N, 3.63; S, 4.15 found C, 51.40; H, 5.39; N; 3.50; S,4.02%.

1-Hepta-O-acetyl-β-D-maltosyl-3-p-Cl-phenyl thiocarbamides (IId)

IR (KBr): *v* 3430 (N-H), 2817 (Ar-H), 1750 (C=O), 1352 (C-N), 1236 (C-O), 1043 and 899 (Cha. maltose ring) 766.2 (monosubstituted benzene ring), 602 cm^{-1} (C-S); ^1H NMR ($CDCl_3$): δ 8.17 (d, 1H,-NH); 7.36-7.1 (d, 4H, Ar-H); 6.56-6.54 (t, 1H, maltosyl-NH); 5.7-3.8 (m, 21H, acetylene proton 7-OCH_3). Mass (m/z): 806 (M^++2), 771, 711, 619, 559, 331,169, 109. Anal. Calcd. For $C_{33}H_{41}O_{17}N_2SCl$; C, 49.25; H, 5.09; N, 3.48; S, 3.97 found C, 49.30; H, 5.07; N, 3.45; S, 4.10%.

1-Hepta-O-acetyl-β-D-maltosyl-3-o-tolyl thiocarbamides (IIe)

IR (KBr): *v* 3435 (N-H), 2818 (Ar-H), 1748 (C=O), 1379 (C-N), 1257 (C-O), 1044 and 942 (Cha. maltose ring), 765.4 (disubstituted ring), 602.9 cm^{-1} (C-S); ^1H NMR ($CDCl_3$): δ 7.678, (S, 1H, Ar-NH); 7.32-7.12 (m,4H,Ar-H); 6.15-6.12 (d,1H,maltosyl N-H); 5.84-3.82 (m, 14H, maltosyl ring protons); 2.32-1.97 (m, 21H, aeetyl protons); 1.62 (3H, tolyl); Mass (m/z): 785 (M+1), 619, 559, 457, 331, 169, 109, Anal. Calcd. for $C_{33}H_{44}0_{17}N_2S$: C,52.04; H,5.61; N, 3.57; S, 4.08 found C, 50.47; H, 5.63; N, 3.53; S, 4.08%

N-Hepta-O-acetyl-β-D-maltosyl-O-ethyl thiocarbamates (IIIa)

IR (KBr): *v* 3459 (N-H), 2966 (Ali. C-H), 1751 (C=O) 1375 (C-N), 1233 (C-O),

1042 & 902 (Cha. maltose ring), 604.1 cm^{-1} (C-S); ^1H NMR ($CDCl_3$) : δ 6.7-6.68 (1H,-NH); 5.64-3.85 (m, 16H, maltosyl ring protons, -OCH_2); 1.38 -1.29 (m,3H, -CH_3); 2.22-2.0 (m, 21H, 7-$COCH_3$) 1.3-1.2 (m, 3H, CH_3); Mass (m/z); 724 (M+1), 664, 619, 559, 457, 331, 169, 109. Anal. Calcd. for $C_{29}H_{41}O_{18}NS$: C, 48.13; H,5.67; N, 1.92; S, 4.42 found C, 48.09; H, 5.71; N, 1.91; S, 4.71%.

N-Hepta-O-acetyl-β-D-maltosyl-O-isopropyl thiocarbamates (IIIc)

IR (KBr) **:** *v* 3469 (N-H), 2972 (Ali. C-H), 1752 (C=O), 1375 (C-N), 1233 (C-O), 1041 and 900 cm^{-1} (Cha.maltose ring); ^1H NMR ($CDCl_3$): δ 6.61-6.59 (d,1H,-NH); 5.64-3.85 (m, 15H, maltosyl ring protons and –CH); 2.11 -2.0 (m, 21 H, 7-$COCH_3$); 1.37-1.27 (m,6H, -$(CH_3)_2$); Mass (m/z): 738 (M+1), 619, 559, 457, 331, 169, 109. Anal. Calcd. for $C_{30}H_{43}O_{18}NS$: C, 48.84; H, 5.83; N, 1.89; S, 4.34 found C, 48.79; H, 5-80; N, 1.93; S,4.41%

N-Hepta-O-acetyl-β-D-maltosyl-O-isoamyl thiocarbamates (IIIf)

IR (KBr): *v* 3466 (N-H), 2962 (Ali., C-H), 1752 (C=O); 1372 (C-N); 1235 (C-O); 1037 and 900 cm^{-1} (Cha. maltose ring); ^1H NMR ($CDCl_3$): δ 6.69-6.67 (d, 1H, -NH); 5.81-3.93 (m, 19 H, maltosyl ring proton and CH_2-CH_2-CH group); 2.22 -2.0 (m, 21 H, 7 –$COCH_3$); 1.65 -0.95 (m, 6H, 2 –CH_3); Mass (m/z) :765 (M+) 619, 559, 457, 331, 169, 109. Anal. Calcd. for C_{32} H_{47} O_{18} NS : C, 50.19; H, 6.14; N, 1.89; S, 4.18 found C, 50.11; H, 6.19; N, 1.82; S, 4.23%

1-Hepta-O-acetyl-β-D-maltosyl-3-(2)- amino benzothiazolyl thiocarbamides (IVa)

IR (KBr) **:** *v* 3444 (N-H), 2960 (Ar-H), 1752 (C=O), 1541 (C=N), 1371 (C-N), 1231 (C-O) 1038 and 899.3 (Cha maltose ring); 753 cm^{-1} (C-S); ^1H NMR ($CDCl_3$): δ7.68-7.13 (m, 5H, NH and Ar-H); 5.09 (d, 1H, -NH); 5.76 -3.96 (m, 14H, maltosyl ring protons); 2.22 -1.94 (m, 21 H, 7-$COCH_3$); Mass (m/z): 828 (M^++1), 768, 708, 619, 559, 457, 331, 169, 109. Anal. Calcd. For C_{34} H_{41} N_3 O_{17} S_2; C, 49.33; H, 4.95; N.5.07; S, 7.73 found C, 49.49; H, 4.80; N, 4.98; S, 7.72%

1-Hepta-O-acetyl-β-D-maltosyl-3-(2)-o-Cl-phenyl benzothiazolyl thiocarbamides (IVb)

IR(KBr) **:** *v* 3483 (N-H), 2962 (Ar-H), 1751 (C=O), 1614 (C = N), 1372 (C-N), 1231 (C-O), 1038 and 899.4 (Cha. maltose ring), 603 cm^{-1} (C-C1), ^1H NMR ($CDCl_3$): δ 7.26-7.25 (Ar-H); 5.07 (d,14,-NH); 5.06-3.93 (m, 14H, maltosyl ring protons); 2.2-1.4 (m, 21H, 7-$COCH_3$); Mass (m/z): 862 (M+1), 830, 724, 619, 559, 457, 331, 169, 109. Anal. Calcd. For C_{34} H_{40} O_{17} N_2S_2Cl : C, 47.38; H, 4.64; N, 4.87; S, 7.43 found C, 47.40; H, 4.60; N, 4.92; S, 7.40%

1-Hepta-O-acetyl-β-D-maltosyl-3-(2)- m-tolyl benzothiazolyl thiocarbamides (IVf)

IR (KBr) **:** *v* 3482 (N-H), 2961 (Ar-H), 1750 (C=0), 1542 (C=N), 1373 (C-N), 1232 (C-O), 1039 and 899.5 cm^{-1} (Cha. maltose ring); ^1H NMR ($CDCl_3$): δ 7.27-7.26 (m, 5H, N-H & Ar-H); 5.07 (d, 1H, NH), 5.06-3.93 (m, 14H; maltosyl ring protons); 2.2-1.4 (m, 21H, 7-OCH_3); Mass (m/z): 841 (M^+), 826, 782, 695, 619, 559, 457, 31, 169, 109 Anal. Calcd. For C_{35} H_{43} O_{17} N_3 S_2; C, 49.94; H, 5.11; N, 4.99; S, 7.60 found C,50.10; H, 4.97; N, 4.98; S, 7.63%

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