reaction at 165°C for 4.5 h, a different product characterized as anhydrotris-indene (IV), was obtained. Both III and IV are formed due to intermolecular cyclodehydration of two and three molecules of indan–1–one (II) respectively.

Anhydrobis–indan–1–one (III): β-Phenylpropionic acid (6 g) and PPA (90 g) were kept at 140°C for 3.5 h with constant stirring. The whole mass was then poured into ice-cold water. The organic portion was extracted with ether (3 × 50 ml), washed with 5% aqueous $NaHCO_3$ solution, water and then dried with Na_2SO_4. Removal of the solvent afforded a solid mass which upon recrystallization with ethyl acetate gave shining yellow crystals (1.97 g, 40%), m.p. 141–42°C; M^+ 245; Found C, 87.78%; H, 5.68%; Calcd. for $C_{18}H_{14}O$ (required: C, 87.8%; H, 5.7%).

ν_{max}^{KBr} 1724, 1675, 1600, 1580, 1500, 1480 cm^{-1}

λ_{max}^{MeOH} 395, 240 nm

δ ppm ($CDCl_3$) 3.16 (*t*, Ar–CH_2, –2*H*), 3.53 (*t*, Ar–CH_2–$CH_2C{=}\dot{C}$–, –2*H*) 3.99 (*s*, Ar–CH_2–C=C, –2*H*), 7.41–7.81 (complex, 8*H*-aromatic).

Anhydrotris-indene (IV): β-Phenylpropionic acid (4.5 g) and PPA (100 g) were kept at 165°C for 4.5 h with constant stirring followed by similar treatments as for (III) afforded deep yellow crystals, which did not melt up to 295°C, M^+ 341; C, 94.72%; H, 5.26% Calcd. for $C_{27}H_{18}$ (required C, 94.73%; H, 5.27%).

ν_{max}^{KBr} 3050, 1600, 1450, 750, 700 cm^{-1}

λ_{max}^{MeOH} 440 nm

δ ppm ($CDCl_3$) 3.8 (*s*, Ar–CH_2–, 6*H*); 7.5–7.9 (complex, 12*H* aromatic).

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A SINGLE-STEP SYNTHESIS OF 5- AND 7-SUBSTITUTED 2-(*p*-BROMOBENZOYL)-3-METHYL-4*H*-1, 4-BENZOTHIAZINES

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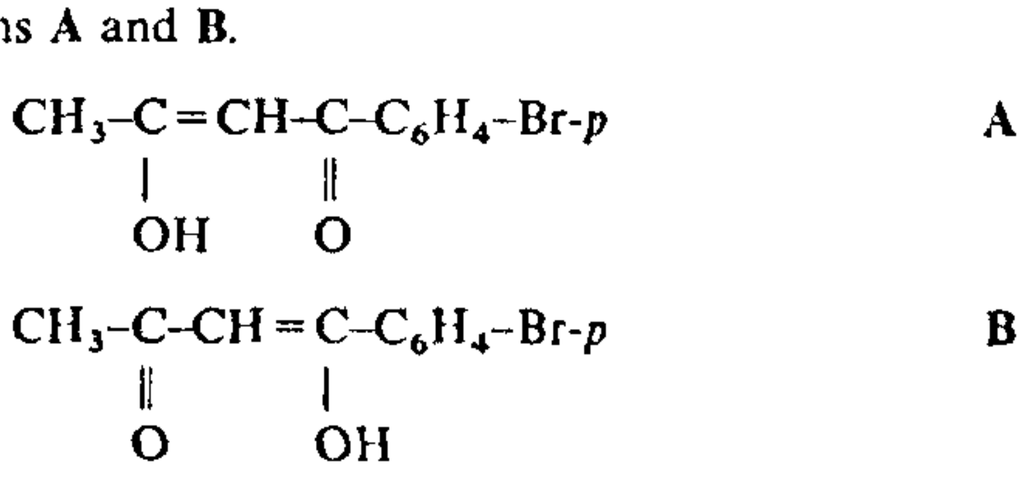
SYNTHESIS of 5- and 7-substituted 2-(*p*-bromobenzoyl)-3-methyl-4*H*-1, 4-benzothiazines by the condensation and oxidative cyclization of 3- and 5-substituted 2-aminobenzenethiols with *p*-bromobenzoylacetone in dimethyl sulphoxide is reported.

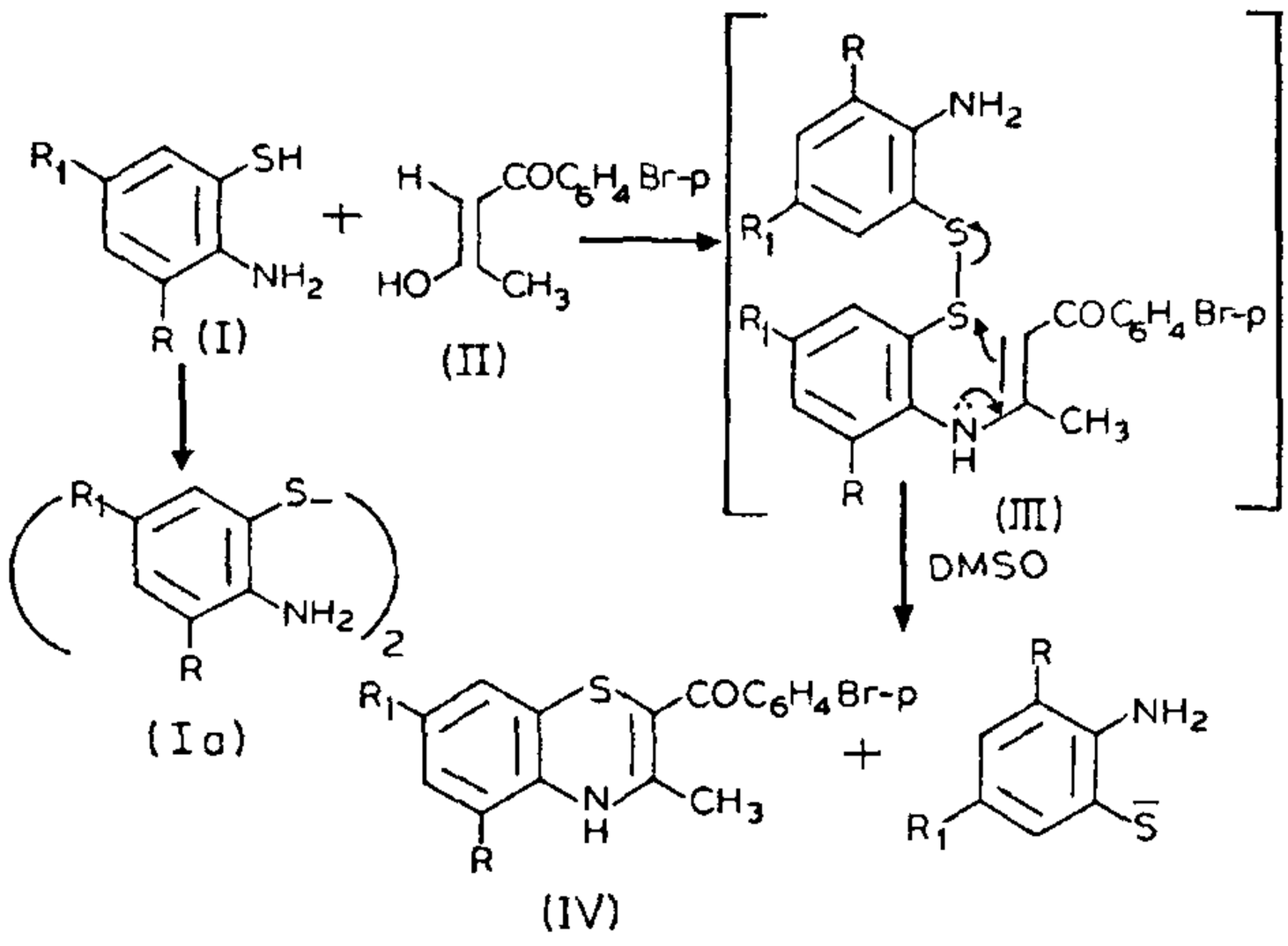
4*H*-1, 4-Benzothiazines resemble phenothiazines in having a fold along nitrogen–sulphur fold, which is the structural feature responsible for biological activity[1]. They constitute an interesting and important class of bioactive molecules. Some derivatives reported have been found to possess anti-inflammatory[2], anthelminthic[3], anti-hypertensive[4], anti-histaminic[5], tranquillizer[6], diuretic[7], lipid regulating[8], spasmolytic[9], anti-bacterial[10], CNS depressant[11] and antiulcer[12] activity.

In continuation of our programme to synthesize novel bio-active molecules[1], the title compounds have been synthesized by one-pot reaction. It involves the condensation of 2-amino-3 and 5-substituted benzenethiols and *p*-bromobenzoylacetone in DMSO which causes oxidative cyclization. The reaction is believed to proceed via the formation of an enaminoketone. 2-Aminobenzenethiols (I) are readily oxidized to disulphides[1,13] (Ia) under the experimental conditions. Disulphides (Ia) undergo condensation with β-diketones[13,14] yielding enaminoketones which cyclize to 1, 4-benzothiazines by scission of the sulphur–sulphur bond[1,14,15] upon attack by the nucleophilic enaminoketone systems as shown in scheme 1.

p-Bromobenzoylacetone can exist in two enolic forms **A** and **B**.



From **A** is likely to predominate due to the electron-pushing nature of the methyl group and electron-withdrawing nature of benzene molecule, hence this form participates in the reaction.



R = H, –Cl, –CH_3, –OCH_3, –OC_2H_5
R_1 = H, –Cl, –Br, –CH_3, –OCH_3, –OC_2H_5

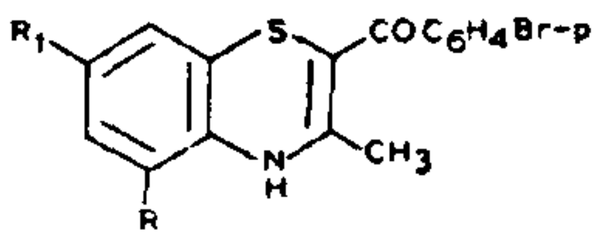
Scheme 1.

All the melting points are uncorrected and the purity of the synthesized compounds checked by TLC.

Preparation of 5- and 7-substituted 2-(p-bromobenzoyl)-3-methyl-4H-1,4-benzothiazines (IV)

A mixture of *p*-bromobenzoylacetone II (0.01 mol), 2-aminobenzenethiol I (0.01 mol) and DMSO (5 ml) was refluxed for 1 h, cooled and filtered. The product was washed with a small quantity of methanol and recrystallized from methanol. The physical and analytical data are included in table 1.

Table 1 *5- And 7-substituted 2-(p-bromobenzoyl)-3-methyl-4H-1, 4-benzothiazines (IV)*



Compound				Yield	Found			Mol.	Calculated		
	R	R_1	M.P.	(%)	C(%)	H(%)	N(%)	formula	C(%)	H(%)	N(%)
a	Cl	H	133	40.5	50.18	2.93	3.63	$C_{16}H_{11}NSOBrCl$	50.45	2.89	3.67
b	OC_2H_5	H	109	45.5	55.26	4.13	3.61	$C_{18}H_{16}NSO_2Br$	55.38	4.10	3.58
c	OCH_3	H	158	46.0	54.16	3.75	3.78	$C_{17}H_{14}NSO_2Br$	54.25	3.72	3.72
d	CH_3	H	141	42.0	56.32	3.92	3.95	$C_{17}H_{14}NSOBr$	56.66	3.88	3 88
e	H	Br	222	68.5	45.38	2.61	3.25	$C_{16}H_{11}NSOBr_2$	45.17	2.58	3.29
f	H	Cl	241	65.0	50.62	2.93	3.71	$C_{16}H_{11}NSOBrCl$	50.45	2.89	3.67
g	H	F	221	63.5	52.32	3.06	3.81	$C_{16}H_{11}NSOBrF$	52.74	3.02	3.84
h	H	OC_2H_5	220	62.5	55.66	4.06	3.55	$C_{18}H_{16}NSO_2Br$	55.38	4.10	3.58
i	H	OCH_3	204	60.5	54.38	3.76	3.68	$C_{17}H_{14}NSO_2Br$	54.25	3.72	3.72
j	H	CH_3	211	70.0	56.28	3.83	3.92	$C_{17}H_{14}NSOBr$	56.66	3.88	3.88
k	H	H	193	55.0	55.28	3.49	4.08	$C_{16}H_{12}NSOBr$	55.49	3.46	4 04

IR (KBr): 3280–3320 cm^{-1} (NH); 1570–1610 cm^{-1} (C=O), 1350–1370 and 1450–1475 cm^{-1} (CH deformation vibrations of $C-CH_3$).

^1H NMR (DMSO-d_6): The spectra of all the synthesized compounds exhibit a signal in the region δ 8.3–10.7 due to the N–H proton, a singlet in the region δ 1.9–2.8 due to methyl protons and a multiplet in the region δ 6.2–8.17 due to aromatic protons.

Mass spectra: Mass spectra were recorded on a JEOL, JMSD-300 mass spectrometer at 70 eV and 100 μA ionizing current. All the compounds, besides the respective molecular ion peaks exhibited a peak at $m/z=184$ due to the $(COC_6H_4Br\text{-}p)^+$. This supports the formation of benzothiazines with benzoyl group rather than acetyl group.

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ISOLATION OF A NEW TRITERPENE FROM *CALOTROPIS PROCERA* LATEX

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A variety of terpenoid alcohols, esters of steam volatile and long chain fatty acids and several cardioactive poisons have earlier been isolated from *Calotropis procera* latex and characterized. In the present communication, we describe the isolation and identification of a new ester of a terpenol.

The pooled frozen latex was thawed a fortnight after collection when the serum separated and the solid part of the latex could easily be removed and dried between filter paper sheets.

The solid was extracted exhaustively with ethanol (95%, v/v) and the alcoholic extract filtered hot and cooled when it deposited a white bulky mass. This was filtered, dried and subjected to column chromatography on a silica gel column dissolved in *n*-hexane and eluted with the same solvent.

The fractions eluted yielded two compounds. While one of them was identified as taraxasteryl acetate by various physical and chemical tests (m.p., m.m.p., IR, NMR, MS and co-TLC) the other appeared to be a new triterpene.

The white crystalline compound melting sharp at 154°C and with 522 as its molecular weight determined by the Rast's method, answered to the molecular formula $C_{36}H_{58}O_2$. (Found: C, 82.7%; H, 11.16%; $C_{36}H_{58}O_2$ requires C, 82.76%; H, 11.11%). The compound responded to all the colour tests for triterpenes.

The IR spectrum exposed strong bands at 1740 cm^{-1} (>C=O group), 1460 $^{-1}$ cm, 1396 cm^{-1}, 1260 cm^{-1} (-O-COR group) and at 880 cm^{-1} (=CH_2 group out of plane bending).

The ^1H NMR spectrum of the compound revealed the presence of an exocyclic methylene group (δ 4.50, 2*H*, C=CH_2). The signals at 0.79, 0.85, 0.92, 0.95