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CYCLOADDITION REACTIONS OF 1,3-BENZOTHIAZINES - 6.[1] REACTIONS OF 1,3-BENZOTHIAZINE DERIVATIVES WITH SUBSTITUTED ACETYL CHLORIDES

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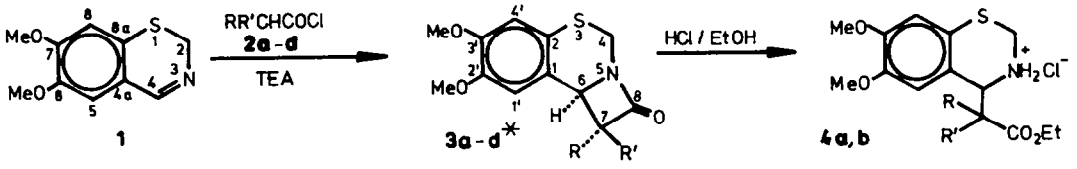
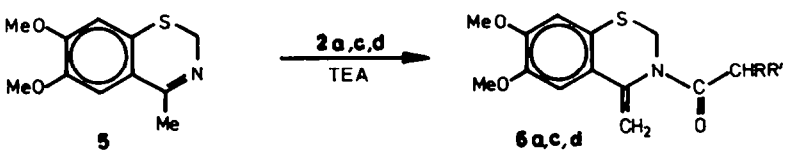
Abstract – 6,7-Dimethoxy-2H-1,3-benzothiazine derivatives (1, 8) react with substituted acetyl chlorides to give angularly condensed β-lactams (3a-d, 10, 11). The cis compound 11 was epimerised to the trans derivative 12. From the interaction of 2-phenyl-6,7-dimethoxy-4H-1,3-benzothiazine (7) and α-chloro-phenylacetyl chloride two stereoisomeric β-lactam derivatives (9a, b) were isolated, whereas in the other cases studied the reactions leading to β-lactams proved to be stereospecific. Analogous reactions of 4-methyl-6,7-dimethoxy-2H-1,3-benzothiazine (5) furnished the enamides 6a, c, d. Structures of the new compounds and configurations of the diastereomers were elucidated by IR and NMR spectroscopy.

In an earlier paper[2] we reported on the cycloaddition reactions of 2-phenyl-4H- and 4-phenyl-2H-1,3-benzothiazines (7, 8) with substituted acetyl chlorides, which gave stereohomogeneous linearly and angularly condensed β-lactam derivatives. In a continuation of this work, in the present paper we describe similar reactions of the benzothiazine derivatives 1 and 5. When 6,7-dimethoxy-2H-1,3-benzothiazine (1) was reacted with the acid chlorides 2a-d in the presence of triethylamine, the products were the β-lactam derivatives 3a-d. The heating of β-lactams 3a, b with hydrogen chloride in ethanol led to cleavage of the β-lactam ring, to yield compounds of β-amino-ester type, 4a, b.



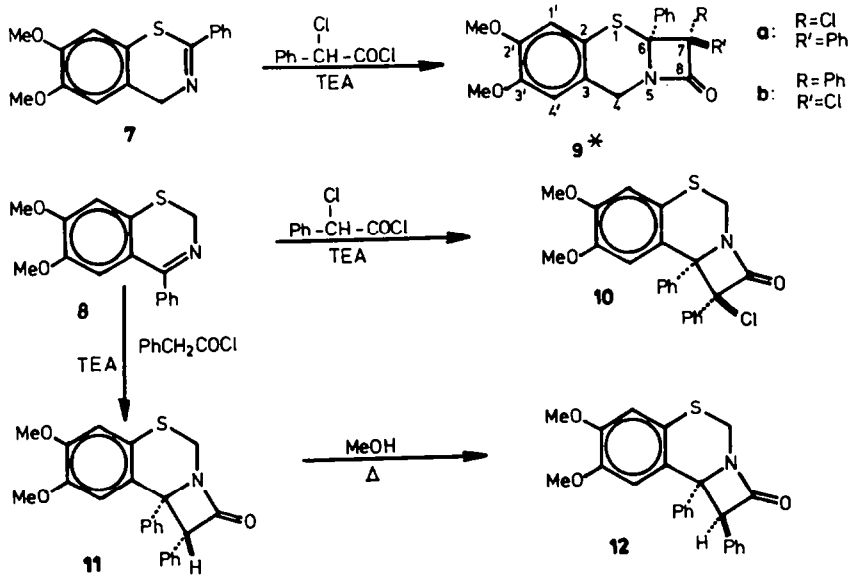
a: R=Ph, R'=H ; b: R=OPh, R'=H ; c: R=Cl, R'=H ; d: R=R'=Cl

The reactions of 4-methyl-6,7-dimethoxy-2H-1,3-benzothiazine (5) and the acid chlorides 2a, c, d gave, under similar conditions, the enamides 6a, c, d in good yield.



* The numbering of the azetidinones in the Scheme follows the nomenclature reported by Bose[3] which is not identical with that used in the text and the Tables (IUPAC numbering of 1); this is to facilitate comparison of spectroscopically analogous atoms.

The cycloaddition reactions of 7 and 8 with 2a–d were stereospecific in the cases investigated,[2] i e only the kinetically-controlled diastereomeric β-lactams were formed. For this reason these reactions have now been further studied using α-chlorophenylacetyl chloride, when the formation of both diastereomeric β-lactams can be expected. Indeed, in the reaction of 7 both isomers (9a, b) could be isolated, whereas compound 8 gave only the stereohomogeneous product 10. The other diastereomer could not even be detected in the reaction mixture.



In the case of the cis-β-lactam[2] 11, prepared from 8 with 2a epimerisation was observed; the cis-diphenyl compound formed by cycloaddition was converted by refluxing in metanol into the thermodynamically more stable trans derivative 12.

IR, ^{1}H and ^{13}C NMR spectra supporting the structures proposed are listed in Tables 1 and 2. The structures of the β-lactams 3a–d, 9a, b and 10–12 are proved by the presence of the high-frequency IR carbonyl band[4] (1753–1790 cm^{-1}) characteristic of the azetidinone ring; in the ^{1}H NMR spectra, the methylene, methoxy and two aromatic proton signals of the benzothiazine skeleton complete the evidence. Further, in the spectra of 3a–c the two doublets of the protons of the azetidine ring can also be identified, as can the one-proton azetidine signal in the spectra of 3d, 11 and 12.

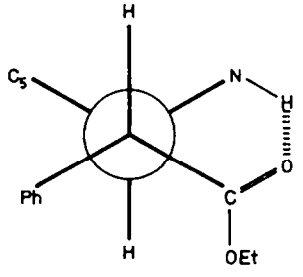


Fig. 1.

The IR spectra of compounds 4a, b contain νNH bands, as well as the νC=O, ν_{as}C–O and ν_sC–O bands characteristic of esters, in the ^{1}H NMR spectra, in addition to those of the hydrogens of the benzothiazine skeleton, the methyl and methylene signals of the carbethoxy groups can be assigned.

It is interesting to note the considerable diamagnetic shift of the signals of one of the skeletal protons and of the methoxy groups in 4a. This observation, together with the rather large coupling constants (~8 Hz) of the methine hydrogens, suggest the predominance of the conformation with the phenyl group close to the benzothiazine ring (c.f. Fig. 1). This means the R, S configuration of the C–4 and C_α atoms. Besides steric factors, the conformation is probably also stabilised by an intramolecular hydrogen-bond between the ester carbonyl and the NH groups.

Table 1. IR[a] and ^{1}H NMR[b] data on compounds 3a–d, 4a,b, 6a,c,d, 9a,b and 10–12

Compound	νC=O IR band	H–4	$C_\alpha H$[c]	CH_2 (2H)[d] (heteroring)	OCH_3 2xs (2x3H)	ArH–5, 8 2xs (2x1H)	Other signals
3a	1765	4.65[e]	4.10[e]	4.25, 4.85	3.60, 3.65	6.60, 6.65	ArH: 7.35 ~ s (5H)
3b	1770	4.78[e]	5.00[e]	4.24, 4.85	3.75, 3.80	6.63[f]	ArH: 6.95–7.45 m (5H)
3c	1775	4.70[e]	4.50[e]	4.24, 4.80	3.80, 3.85	6.60, 6.65	–
3d	1790	5.20[g]	–	4.30, 4.80	3.85, 3.90	6.75, 6.80	–
4a	1725	4.50[e]	4.30[e]	4.10, 4.80	3.25[h], 3.80	5.60[h], 6.50	CH_3: 1.25 t[i], CH_2: 4.30 qa[i] ArH: 7.3 ~ s (5H), NH: 2.00 s (1H)
4b	1725	4.30[e]	4.80[e]	4.35, 4.55	3.75[f]	6.50, 6.95	CH_3: 1.20 t[i], CH_2: 4.20 qa[i] ArH: 6.8–7.3 m (5H), NH: 1.85 s (1H)
6a	1670	–	5.05[e,f] 5.65[e]	5.05[f]	3.80, 3.85[j]	6.50, 6.95	CH_2: 3.85[j], s (2H), ArH: 7.15 s (5H)
6c	1670	–	5.20[e] 5.70[e]	5.00	3.80, 3.85	6.50, 7.10	CH_2: 4.25, s (2H)
6d	1675	–	5.30[e] 5.95[e]	5.00	3.75, 3.80	6.65, 7.25	CH: 6.90 s (1H)
9a	1782	–	–	4.28, 4.95	3.55[h], 3.71	6.23[h], 6.61	ArH: 7.3–7.45 m (6H) ArH: 7.5–7.7 m (4H)[k]
9b	1780	–	–	4.24, 5.00	3.78, 3.80	6.68, 6.76	ArH: 7.0–7.1 m (6H) ArH: 7.2–7.25 m (4H)[k]
10	1771	–	–	4.33, 5.05	3.86, 4.01	6.73, 7.30	ArH: 7.05–7.2 m (8H)
11	1753	–	4.98[g]	4.40, 4.99	3.82, 3.99	6.80, 7.28	ArH: ~ 7.3 m (2H)[k] ArH: 7.0–7.15 m (10H)
12	1761	–	5.10[g]	4.33, 5.02	3.17[h], 3.80	5.80[h], 6.62	ArH: ~ 6.95[k], ~ 7.5[k] 2xm (2x2H) ~ 7.15, ~ 7.4 2xm (2x3H)

[a] In KBr, cm^{-1}, spectrometer: Perkin–Elmer 577 (3a,b, 4b, 6a,c,d), Perkin–Elmer 325 (3c,d, 4a) or Bruker IFS-113v-FT (9a,b, 10–12). In all IR spectra there are bands characteristic of methoxy and aromatic groups (in the cases of 3a,b, 4a,b, 6a and 10–12 also the $\gamma C_{Ar}H$ and $\gamma C_{Ar}C_{Ar}$ bands of the phenyl substitutents at about 750 and 700 cm^{-1}, resp). Other IR bands: νNH: 3330 (4a), 3338 (4b), νC–O (ester): 1235 and 1050 (4a), 1260 and 1060 (4b), νC = C (conj.): 1625 (6a,d) and 1630 (6c). – [b] In $CDCl_3$ at room temperature on a JEOL 60-HL (3a,b, 4b, 6a,c,d) or Varian EM-360 (3c,d, 4a) spectrometer at 60 MHz, and on a Bruker WM-250 FT-spectrometer (9a,b, 10–12) at 250 MHz (δ_{TMS} = 0 ppm). – [c] Signal of protons on C_α attached to C–4 of the benzothiazine skeleton. – [d] A and B parts of an AB multiplet; J(AB) = 13 (3a–d, 4a,b), 16.4 (9a), 16 (9b), 12.1 (10), 11 (11) and 12.4 Hz (12) or singlet (6a,c,d). [e] Doublet, J: 2.5 (3a–c), 8 (4a,b) and 2 Hz (6a,c,d). – [f,j] Overlapping signals. – [g] Singlet (1H). – [h] One of the ArH atoms and of the methoxy groups of the thiazine ring are shielded significantly by the anisotropy of the phenyl substituent. – [i] J ≈ 7 Hz. – [k] Ortho protons of the phenyl rings.

No analogous effect can be observed in compound 4b. The explanation is that the oxygen atom inserted between the phenyl group and the C_α atom cuts off the phenyl substituent from the close approach of the benzo-

Table 2. ^{13}C NMR data on compounds 9a, b and 10 – 12 in $CDCl_3$ at 20.14 MHz (δ_{TMS} = 0 ppm).

Compound	C=O C_α[a]	C-2/4[b] C-4/2[c]	C-4a, 8a	C-5, 8	C-6, 7	OCH_3	C_s(Ph)	C_o(Ph)[d]	C_m(Ph)[d]	C_p(Ph)[d]
9a	166.5	80.8[d]	121.4	112.8	148 0	55.7	134.6	126.5	128.1	128.5
	78.9[d]	42.6	123.3	113.3	148.5	55.8	138 1	127.6	128.2	128.9
9b	166.4	83.9[d]	122.2	111.9	148.3	56.2[e]	134 7	126.4	127.8[f]	128.5
	80.4[d]	43.2	123.8	113.3	149.2		138.0	127.8[f]		128.9
10	163.5	71.1	122.3	110 7	146.1	55.8	135.3	127.8[e]	127.9[f]	127.9[f]
	83.5	36.6	123.1	116.8	149.6	56.7	137.0		128.1	128.3
11	167.6	66.9	122.7	112.5	148 0	56.0	132.5[d]	127.1	127.5	128.6[e]
	68.6	39.8	132.0[d]	112.8	149.4	56.7	137.1	128.1	127.3	
12	168.4	64.4	122.1	109.9	145.9	55.6	133.4	126.9	128.6	127.6
	68.6	36.7	123.5	115.0	148.7	55.7	141.9	128.1	129.7	128.0

[a] Carbon atom of the azetidinone ring attached to C–2 (9a, b) or C–4 (10–12) of the benzothiazine skeleton. – [b] Methine carbon C–2 (9a, b) or C–4 (10–12) of the benzothiazine skeleton – [c] Methylene carbon C–4 (9a, b) or C–2 (10–12) of the benzothiazine skeleton – [d] Reversed assignment is also possible. – [e, f] Overlapping lines.

thiazine ring.

Primary evidence for the structure of compounds 6a, c and d is given by the IR amide-I band (1670–1675 cm^{-1}) and the ^1H NMR signals of the vinylidene protons (two doublets, split by ~2 Hz).[5a] In the latter spectrum, of course, the signals due to the thiazine skeletal and side-chain protons can also be identified. Among the doublets of the olefinic protons with a significant chemical shift difference (0.5–0.65 ppm), the one shifted paramagnetically, is assigned to the hydrogen syn to the benzothiazine skeleton; the anisotropy of the benzene ring decreases the shielding of the coplanar olefinic proton.[5b] This effect is mutual: the olefinic double bond causes a paramagnetic shift of the coplanar benzene hydrogen (H–5). (In compounds 3a-d and 9a, b, where there are no nearby substituents, the H–5 shifts are in the range 6.6–6.8 ppm; the shifts for 6a, c and d are between 6 95 and 7 25 ppm.) The hindrance of rotation generally observable in amides (manifested in doubled signals, corresponding to the rotamers)[5c] cannot be detected in the case of 6a, c and d; this is obviously due to the effect of the electrophilic olefinic substituents attached to the nitrogen, which decrease the probability of the $N^+=C-O^-$ limiting structure.

Determination of the configurations of the β-lactams was greatly facilitated by the fact that the data on the pairs of diastereomers 9a–9b, and 11–12, were already available. Hence, only the ^1H and ^{13}C NMR shifts were needed. (In our earlier investigations,[2] the configurations of analogues had to be determined without a knowledge of their counterparts.) The determination of configuration can be based upon the different anisotropic shielding of the phenyl substituents in the isomers or upon the field effect (steric compression shift)[6] due to the different steric structures in the isomers. The former effect gives rise to considerable differences in the ^1H chemical shifts of the isomeric pairs, and the latter to differences in the ^{13}C shifts.

In one of the linearly fused isomers 9a, b the phenyl groups are in the cis position, and their mutual anisotropic effect result in increased shielding of the ring protons. Structure 9b can therefore be assigned to the compound melting at 180–181° C, whose ^1H NMR spectrum has the phenyl multiplets at 7.0–7.25 ppm; for

the lower-melting isomer this range is 7.3–7.7 ppm (see Table 1). In the spectrum of the latter compound, having structure 9a, considerable diamagnetic shifts of a proton signal of the benzothiazine skeleton and of a methoxy signal can also be observed; the shift differences compared with the other isomer are 0.53 and 0.25 ppm, respectively. The explanation is that, depending on the conformation of the thiazine ring, H–8 and the 7-methoxy group lie above the plane of one or the other phenyl ring, and the anisotropic effect of the latter gives rise to increased shielding.

This effect appears in isomer 9b only in one of the conformers of the thiazine ring, and – owing to the greater distance between the interacting groups – it is smaller. The observed facts thus indicated rapid inversion of the thiazine ring at room temperature, in accordance with expectation.

The closeness of the benzothiazine skeleton and the phenyl groups results in a sterically more compressed structure of isomer 9a, which is also reflected by the ^{13}C NMR data. Steric hindrance causes diamagnetic shifts (increased shielding) of the signals of the carbon atoms carrying hindered groups. This s.c. field effect can be observed in compounds of the most various types, such as α-and β-anomeric carbohydrates,[7] cis- and trans-anellated condensed alicycles,[8] cis- and trans-substituted cyclic compounds,[9,10] etc. The significantly smaller ^{13}C NMR chemical shifts for compound 9a, in comparison with 9b, accordingly provide further, independent evidence for the configuration.

Investigation of the configurations of the angularly condensed azetidinones 3a–d and 10–12 is similarly appropriately began by studying the isomer pair 11 and 12. As in the case of the analogous compounds 9a, b, these isomers differ considerably in the shielding of the phenyl hydrogens. The higher-melting compound 11, which has been reported previously,[2] has a spectrum in which, due to the mutual anisotropic effect of the cis phenyl groups, the ring protons are more shielded (7.0–7.15 ppm) than in isomer 12, where the aromatic multiplets cover a much wider range (6.9–7.6 ppm). Though two protons are more shielded in 12, the signals of the others are shifted paramagnetically. It follows that, in agreement with our earlier results,[2] 11 is the compound containing the phenyl groups in the cis position, and 12 is the corresponding trans isomer. This latter is sterically highly compressed – much more than 9a – and the H–5 atom and the 6-methoxy group are flanked by the two phenyl groups. For this reason the increase in shielding of the signals, analogous to that observed in 9a, is here significantly higher: $\Delta\delta^{c,t}$ H–5 = 1.00, $\Delta\delta^{c,t}$ OCH_3(6) = 0.65 ppm. The rings, of course, also mutually interact, and the vicinity of the benzothiazine skeleton causes the higher shielding of the two phenyl protons (the ortho hydrogens of the C_α substituent).

It is reasonable to conclude that compounds 3a, b and 10, having no counterparts, should have the sterically favoured configuration analogous to isomer 11, where the phenyl (in the case of 3b the phenoxy) substituents and the H–4 atom (in the case of 10 the other phenyl ring on C–4) are in the cis position. This is evidenced by the absence of the anomally shielded H–5 and OCH_3(6) signals.

There is no direct proof of the configuration of 3c, but an analogous structure is indicated by the H–5 shift in 3d; although compound 3d has no diastereomers and only optical isomers are possible, the nearby R′ = Cl substituent gives rise to a well-recognisable paramagnetic shift of the H–5 signal. As no similar effect is observed for 3c, it seems certain that H–4 and the chlorine atom are in the cis position, i.e. the configuration is R = Cl and R′ = H.

Although there are also strong steric hindrances in 11 due to the angularly fused skeleton, the increasing compression in isomer 12 results in the stronger shielding of most carbon atoms. Higher field effects are measured in the cases of atoms C–2, 4, 4a, 5 and 6 (2.5, 3.1, 8.5, 2.1 and 2.6 ppm, respectively). Higher strain is evident in the fused skeleton as compared with the linearly condensed compounds 9a, b, e.g. from a comparison of the C_α chemical shifts of similar character. In the analogue of 9b where the substituents were identical (the substituents at C_α being H and Ph), the C_α and C–2/4 shifts were 2.9 and 4.2 ppm higher (71.5

and 71.1 ppm, respectively)[2] than for compound 11.

EXPERIMENTAL

All m.p.s. are uncorrected. The IR spectra were recorded on different spectrometers (see Table 1) in KBr discs. The ^{1}H NMR spectra were measured in $CDCl_3$ solution in 5 mm tubes at room temperature at 60 or 250 MHz on different instruments (see Table 1), using TMS as internal reference and the ^{2}H resonance of the solvent as the lock signal. The ^{13}C NMR spectra were run under similar conditions on a Bruker WP-80-SY FT-spectrometer at 20.14 MHz. The most important measuring parameters are as follows: sweep width: 5 kHz, pulse width: 3.5 μs, acquisition time: 1.64 s, relaxation delay: 1 s. Complete proton noise decoupling (~1 W) and Lorentzian exponential multiplication for signal-to-noise enhancement (line width 0.5 Hz) were used. Computer memory: 16 K, number of scans: 4 – 25 K.

Table 3. Physical and analytical data on compounds 3a–d, 4a, b, 6a, c, d, 9a, b, 10 and 12[a]

Compound	Yield %	M.p. °C	Formula	Molecular weight	Analysis % Calculated	Analysis % Found		
					C	H	N	S
3a	48	179 – 180	$C_{18}H_{17}NO_3S$	327.39	66.03	5.24	4.28	9.79
					66.01	5.30	4.21	9.68
3b	41	149 – 150	$C_{18}H_{17}NO_4S$	343.39	62.95	4.99	4.08	9.34
					63.02	5.04	4.13	9.22
3c	19	126 – 127	$C_{12}H_{12}ClNO_3S$	285.75	50.44	4.23	4.90	11.22
					50.58	4.32	4.78	11.47
3d	23	226 – 228	$C_{12}H_{11}Cl_2NO_3S$	320.19	45.02	3.46	4.38	10.01
					44.87	3.39	4.29	9.88
4a[b]	96	180 – 183	$C_{20}H_{24}ClNO_4S$	409.90	58.60	5.90	3.42	7.82
					58.49	5.82	3.31	7.93
4a	82	121 – 122	$C_{20}H_{23}NO_4S$	373.45	64.32	6.21	3.75	8.59
					64.20	6.15	3.68	8.67
4b[b]	93	170 – 171	$C_{20}H_{24}ClNO_5S$	425.92	56.40	5.68	3.29	7.53
					56.52	5.76	3.21	7.44
4b	90	117 – 118	$C_{20}H_{23}NO_5S$	389.45	61.68	5.95	3.60	8.23
					61.57	5.89	3.67	8.12
6a	76	105 – 106	$C_{19}H_{19}NO_3S$	341.43	66.83	5.61	4.10	9.39
					66.71	5.57	3.99	9.28
6c	61	157 – 158	$C_{13}H_{14}ClNO_3S$	299.78	52.08	4.71	4.67	10.70
					52.15	4.62	4.56	10.84
6d	67	178 – 179	$C_{13}H_{13}Cl_2NO_3S$	334.22	46.71	3.92	4.19	9.59
					46.64	3.85	4.26	9.65
9a	84	165 – 166	$C_{24}H_{20}ClNO_3S$	437.93	65.82	4.60	3.20	7.32
					65.94	4.66	3.28	7.19
9b	81	180 – 181	$C_{24}H_{20}ClNO_3S$	437.93	65.82	4.60	3.20	7.32
					65.71	4.49	3.27	7.44
10	89	187 – 189	$C_{24}H_{20}ClNO_3S$	437.93	65.82	4.60	3.20	7.32
					65.86	4.68	3.12	7.18
12	79	158 – 160	$C_{24}H_{21}NO_3S$	403.50	71.43	5.25	3.47	7.95
					71.31	5.18	3.39	8.07

[a] Recrystallising solvent: ethanol, except for 3d (benzene) and 12 (methanol). – [b] HCl salt.

Preparation of 3a–d

0.01 Mol of 1 was dissolved in dry dichloromethane (50 ml), the appropriate acetyl chloride (2a–d) (0.01 mol)

and triethylamine (TEA, 0.01 mol) were added, and the mixture was allowed to stand at 5° overnight. It was then extracted with water (2x20 ml) and separated, the organic layer was dried (Na_2SO_4) and evaporated off, and the residue was crystallised.

Preparation of 4a,b

0.01 Mol of 3 was refluxed for 3 h with HCl in ethanol (15 ml); on cooling the product crystallised. After isolation and drying, the TEA.HCl salt was dissolved in water (20 ml), extracted with benzene (3x20 ml), and the combined organic phase was dried, concentrated and crystallised to yield 4a,b.

Preparation of 6a–c

0.01 Mol of 5 was dissolved in dry benzene (30 ml), the acetyl chloride derivative (2a, c, d) (0.01 mol) was added, the mixture refluxed, and a solution of TEA (0.01 mol) in benzene (30 ml) was added over a period of 1 h. After cooling, the TEA.HCl was removed by filtration, the benzene was evaporated off, and the residual brown oil was crystallised.

Preparation of 9a,b

0.02 Mol of 7 was dissolved in dry benzene (70 ml). α-Chlorophenylacetyl chloride (0.02 mol) was added, followed by a solution of TEA (0.02 mol) in dry benzene (50 ml) under reflux conditions over 1 h. The TEA.HCl was removed, the benzene was evaporated off, and the residue was crystallised from ethanol (30 ml). The crystals which separated out (9a) were filtered off, and the mother liquor was concentrated to 5 ml, whereupon crystals of 9b were obtained.

Preparation of 10

0.01 Mol of 8, dissolved in dry benzene (50 ml), was mixed with α-chlorophenylacetyl chloride (0.01 mol). The mixture was refluxed and during 1 h a solution of TEA (0.01 mol) in benzene (30 ml) was added dropwise. The amine salt was removed, the solvent was evaporated off, and the residue was crystallised from ethanol (20 ml).

Preparation of 12

0.005 Mol of 11 was refluxed for 10 h in methanol (20 ml). After concentration of the solution to 5 ml the product was crystallised.

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