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Reactions of cyclic anhydrides. Part XXII†. Direct synthesis of N-aryl-α-(2,3,4,5-tetrahydro-2-imino-4-oxo-1,3-thiazole-5-yl) acetamides

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Abstract. Maleimides (2a–j) react with thiourea in refluxing ethanol to yield the corresponding N-aryl α-(2,3,4,5-tetrahydro-4-oxo-1,3-thiazole-5-yl) acetamides (6a–j) in 72–85%. The spectral and analytical data are consistent with the assigned structures for 6a–j. Under similar conditions, isomalemides (3a–b and 3d–f) furnish the corresponding 6a–b and 6d–f in 76–86%. While maleanilic acid (4a) itself failed to react with thiourea, 4b–e, 4i and 4j yielded a mixture of the corresponding 6b–e, 6i and 6j in 2–50% and fumaranilic acids (7b–e, 7i and 7j) in 50–80%. Methyl maleanilates (5b, 5d–f and 5i) gave 6b, 6d–f and 6i in 78–85%. The reaction course of Raney nickel desulphurisation of 6a–b, 6d, 6f and mass spectral fragmentation of 6b support the assigned structures.

Keywords. Thiourea; thiazolidines; mass spectral fragmentation; fumaranilic acids; Raney nickel desulphurisation.

1. Introduction

Recently we have reported the synthesis of α-(2,3,4,5-tetrahydro-2-imino-4-oxo-1, 3-thiazole-5-yl)acetic acid (1) and its derivatives by reaction of thiourea (TU) with maleic anhydride and related substrates (Balasubramaniyan *et al* 1990). As a sequel to this work, we examined the reactions of TU with maleimides (2), isomaleimides (3), maleanilic acids (4) and methyl maleanilates (5). Only scanty and incomplete studies are available on these reactions; however, none of them presents satisfactory structural characterisation of the products. A definitive study on structural details of the products of these conversions appears desirable. The present study is focussed on these aspects and will also extend our earlier observations on the role of electronic effects in Michael additions of heteroatom nucleophiles with maleic anhydride derivatives (Balasubramaniyan *et al* 1986, 1990).

2. Results and discussion

The reaction of maleimides (2) with TU in refluxing ethanol is reported to furnish N-aryl-α-(2,3,4,5-tetrahydro-2-imino-4-oxo-1,3-thiazole-5-yl) acetamides, 6 (Marrian

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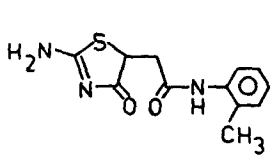
Table 1. Formation of N-aryl-α-(2,3,4,5-tetrahydro-2-imino-4-oxo-1,3-thiazole-5-yl)acetamides (6a–j) from reaction of maleimides (2a–j) with TU.

2/6	X	% yield of 6	m.p. °C (decomp.)
a	H	78	221[a]
b	*o*-Me	81	224
c	*o*-OMe	72	214
d	*o*-Cl	85	235
e	*o*-NO_2	82	208
f	*p*-Me	74	230[b]
g	*p*-OMe	75	240[c]
h	*p*-Cl	76	240[d]
i	*p*-NO_2	84	255
j	*m*-NO_2	78	236

[a]Lit. m.p. 220°C (Marrian 1949; Bethell and Maitland 1961); [b]Lit. m.p. 228°C (Augustin *et al* 1974); [c]Lit. m.p. 245°C (Augustin *et al* 1974); [d]Lit. m.p. 242°C (Augustin *et al* 1974); decomp.: decomposition.

1949; Arakelian *et al* 1960; Bethell and Maitland 1961; Augustin *et al* 1974; Augustin and Muller 1985); however, adequate structural evidence is lacking. We reexamined the reactions of TU with several maleimides (2a–j) in refluxing ethanol; in each case, the reaction furnished N-aryl thiazolidineacetamides (6a–j, table 1). The UV absorption spectrum of 6b featured a shoulder (245 nm, log ε 4·26) and two maxima (225 nm, log ε 4·34; 200 nm, log ε 4·42) in agreement with earlier values (Arakelian *et al* 1960). In its IR spectrum 6b displayed characteristic bands at 3250 (*b*, =NH and –NHCO– overlapped), 1680 (acyclic –NHCO–), 1645 (*sh*, >C=N–) and 1600 cm^{-1} (aromatic >C=C<). The PMR spectrum of 6b displayed a characteristic ABX pattern, with coupling constants $J_{AB} = 16$, $J_{AX} = 8$ and $J_{BX} = 4$ Hz (Nagase 1973; Balasubramaniyan *et al* 1990). The 1-H singlets at 8·8 δ (>C=NH) and 8·9 δ (cyclic –NHCO–) lend further support to formulate this compound as N-(*o*-tolyl) α-(2,3,4,5-tetrahydro-2-imino-4-oxo-1,3,-thiazole-5-yl) acetamide (6b) rather than N-(*o*-tolyl) α-(4,5-dihydro-2-amino-4-oxo-1,3-thiazole-5-yl) acetamide (9). The proton-decoupled CMR spectrum has absorptions, among others, at 189·3 δ (acyclic –NHCO–), 182·5δ (cyclic –NHCO–) and 168·9 δ (>C=NH) further corroborating the above assignment (Levy and Nelson 1972). The mass spectral analysis of 6b revealed the formation of a fragment 10 (*m/z* 149, 1·28%) suggestive of the presence of an *o*-$CH_3C_6H_4NHCOCH_2$ side chain in this molecule (Balasubramaniyan *et al* 1990). A chemical correlation of the product was also made by alkaline hydrolysis of N-(*p*-nitro) α-(2,3,4,5-tetrahydro-2-imino-4-oxo-1,3-thiazole-5-yl) acetamide (6i) to the known 2-imino-thiazolidine-acetic acid, 1 (Balasubramaniyan *et al* 1990).



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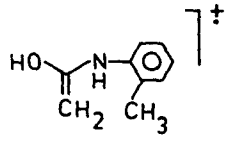

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Table 2. Formation of N-aryl α-(2,3,4,5-tetrahydro-2-imino-4-oxo-1,3-thiazole-5-yl)acetamides (6a–b, 6d–f) from reaction of isomaleimides (3a–b, 3d–f) with TU.

3/6	X	% yield of 6	m.p. °C (decomp.)
a	H	82	221
b	*o*-Me	85	224
d	*o*-Cl	80	235
e	*o*-NO_2	86	208
f	*p*-Me	76	230

decomp.: decomposition

Table 3. Formation of N-aryl α-(2,3,4,5-tetrahydro-2-imino-4-oxo-1,3-thiazole-5-yl)acetamides (6b–e, 6i, 6j) from reaction of maleanilic acids (4b–e, 4i, 4j) with TU.

4/6	X	% yield of 6	m.p. °C (decomp.)
b	*o*-Me	12	224
c	*o*-OMe	14	214
d	*o*-Cl	50	235
e	*o*-NO_2	2·5	208
i	*p*-NO_2	2	255
j	*m*-NO_2	2·6	236

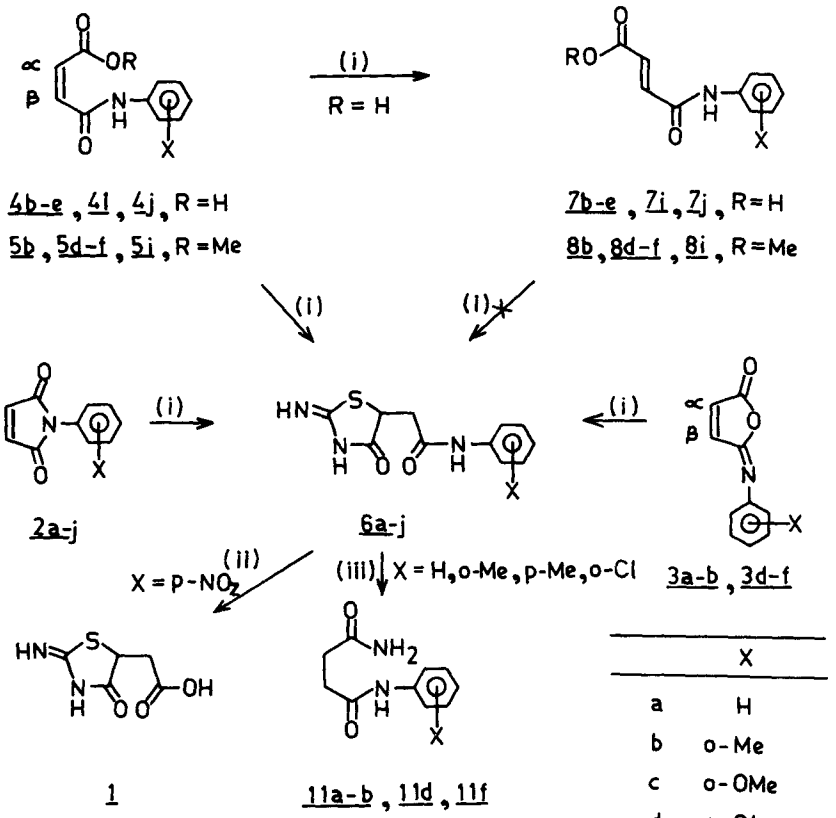
decomp.: decomposition

We next examined the reactions of TU with isomaleimides (3), maleanilic acids (4) and methyl maleanilates (5) in refluxing ethanol. The reaction of TU with isomaleimides (3a–b, 3d–f) furnished the corresponding N-aryl thiazolidineacetamides (6a–b, 6d–f; table 2). Curiously, the reaction of the parent maleanilic acid (4a) with TU in refluxing ethanol failed to occur under a variety of conditions tried by us (see also Augustin *et al* 1974). However, under comparable conditions, several substituted maleanilic acids (4b–e, 4i and 4j) afforded a mixture of N-aryl thiazolidine-acetamides (6b–e, 6i and 6j) in 2–50% yield (table 3) and the corresponding fumaranilic acids (7b–e, 7i and 7j) in 50–80% yields. The vinylic protons in *o*-methylfumaranilic acid (7b) appeared as doublets (6·6 δ and 7·2 δ, $J = 16$ Hz) confirming the *trans* geometry of the olefinic system in the product. The respective values for the vinylic protons in a related *cis* compound, methyl *o*-nitromaleanilate (4e) are 6·3 δ and 6·56 δ with $J = 13$ Hz (Balasubramaniyan *et al* 1983). The reaction of TU with methyl maleanilates (5b, 5d–f, 5i) exclusively furnished the corresponding N-aryl thiazolidineacetamides (6b, 6d–f, 6i) in 78–85% yields (table 4). In contrast, reactions of TU with fumaranilic acids (7b, 7d) and their methyl esters (8b, 8d) under comparable conditions were negative. The failure of the system 8 to react with TU is in contrast to its well-studied reactions with other nucleophilies such as thiophenols, hydrazines (Augustin and Kohler 1976) and amines such as piperidine, morpholine and benzylamine (Augustin and Kohler 1976; Bhatia 1984).

Table 4. Formation of N-aryl α-(2,3,4,5-tetrahydro-2-imino-4-oxo-1,3-thiazole-5-yl)acetamides (6b, 6d–f, 6i) from reaction of methyl maleanilates (5b, 5d–f, 5i) with TU.

5/6	X	% yield of 6	m.p. °C (decomp.)
b	*o*-Me	85	224
d	*o*-Cl	80	235
e	*o*-NO_2	78	208
f	*p*-Me	76	230
i	*p*-NO_2	78	255

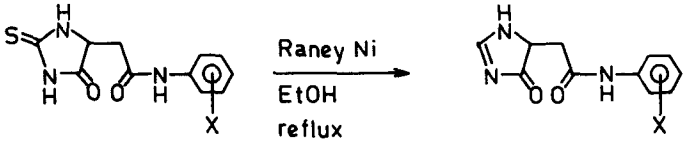
decomp.: decomposition



	X
a	H
b	o-Me
c	o-OMe
d	o-Cl
e	o-NO_2
f	p-Me
g	p-OMe
h	p-Cl
i	p-NO_2
j	m-NO_2

Scheme 1.

Desulphurisation of N-phenyl thiazolidineacetamide (6a) with Raney nickel in absolute ethanol on prolonged refluxing is reported to furnish N-phenyl succindiamide, 11a (Marrian 1949), sulphur being ejected as HCNS. We exploited this reaction as an aid to confirming the position of sulphur in the products obtained in this study. In all the cases, N-aryl succindiamides (11) were obtained (scheme 1). The IR spectrum of 11a displayed characteristic bands at 3410 (PhNHCO–), 3324 and 3210 (–$CONH_2$), 1710 (PhNHCO–), 1650 (–$CONH_2$) and 1600 cm^{-1} (aromatic >C=C<). The PMR spectrum of 11a displayed two overlapping triplets at 2·2 and 2·3 δ for methylene protons, a broad 2-H singlet at 5·5 δ for the amide proton (–$CONH_2$) and a 1-H singlet at 9·1 δ for the anilide proton (PhNHCO–). These observations establish that sulphur is part of the ring in these products. An alternate formulation for the product with an exo sulphur as in N-aryl α-(2,3,4,5-tetrahydro-2-thioxo-4-oxo-1,3-imidazole-5-yl) acetamide (12) should cause formation of N-aryl α-(4,5-dihydro-4-oxo-1,3-imidazole-5-yl) acetamide (13) on desulphurisation. A precedent for this observation has been recorded in the desulphurisation of hexahydropyrimidine-2-thione (Kashima *et al* 1986).



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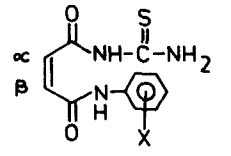
We have earlier commented on the mechanistic implications of the reactions of *o*-aminothiophenol with 2, 3, 4 and 5 (Balasubramaniyan *et al* 1986). The mechanistic features noticed in reactions of TU with the substrates of our current investigation conform to our earlier observations. TU being an ambident nucleophile can cause the reaction to occur via initial ring opening in imide (2) or isoimide (3) leading to a diamide (14) through nitrogen or a thiolactone (15) through sulphur. Both these



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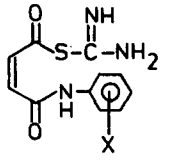

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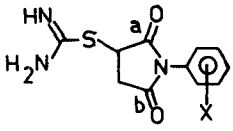

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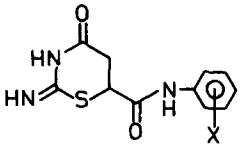

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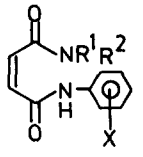

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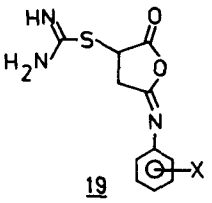

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$R^1, R^2 = -(CH_2)_5-$

$R^1, R^2 = -(CH_2)_2O(CH_2)_2-$

$R^1 = H$, $R^2 = CH_2Ph$

possibilities can be discounted from the structure of the product and non-isolation of any diamide intermediate (14). A scrutiny of the available literature has also failed to reveal any case for ring opening via sulphur in similar reactions of TU.

The regiochemistry of thia-Michael addition as the initial step of the reaction with these substrates poses some interesting questions. The course of addition of TU to the symmetrical substrate, maleimides (2) could lead to the intermediate 16 which in turn could then aminolyse at carbonyl a to yield the isolated product. The alternate site for aminolysis at b does not appear to be preferred since no thiazine (17) is isolated in the reaction#. We have not yet been successful in isolating the above addition intermediate (16). It may be of interest to note that such intermediates have been isolated in the addition of phenylmercaptans to maleimides (Mustafa *et al* 1961). The reaction path in this case appears to follow a kinetically controlled initial Michael step followed by intramolecular ring closure–ring opening to yield 6. The high yields and the exclusive formation of thiazolidineacetamides (6) without any isomers conform to such an interpretation of these results*.

A careful consideration of the results in respect of the reaction of TU with the unsymmetrical substrate isomaleimide (3) enlightens the reaction pathway. Nucleophilic attack on isomaleimides (3) under neutral conditions is established as occurring at the carbonyl (Boyd 1969). Furthermore, the reaction of amines such as morpholine, piperidine and benzylamine with isomaleimides (3) had led to isolation of the corresponding diamides 18 (Bhatia 1984). Our failure to isolate any such intermediate 14 in the current studies rules out isoimide ring opening as the initial step. As a result, a Michael addition at C_α has to be the initial setp to yield 19 followed by aminolysis at carbonyl (preferred to aminolysis at the imino function) to furnish the observed product 6. Again it is of interest to note that the regiochemistry of addition of TU to isomaleimides (3) also displays an apparent reversal of activating effects of the substituents in Michael additions. The structure of the product formed from TU and isomaleimide (3) suggests that the activation order is $\rangle C{=}N- > \rangle C{=}O$ whereas the order of reactivity normally associated with them is the reverse (Sauers and Relles 1973).

The reactions of TU with maleanilic acids (4) and their methyl esters (5) also lead to thiazolidineacetamides (6, scheme 1). Again, an analysis of the mechanistic options in these cases follows similar arguments as above. It can therefore be concluded that in each of the above cases, the reaction appears to occur by an initial thia-Michael addition of TU at C_α to these substrates to give adducts 19/20 followed by aminolysis at the COOH/COOMe function.

In summary, we have demonstrated that (i) the reactions of TU with maleimides (2), isomaleimides (3), maleanilic acids (4) and methyl maleanilates (5) lead to N-aryl 2-imino-thiazolidineacetamides (6); (ii) these reactions appear to proceed predominantly via initial thia-Michael addition (activation order: -CONH > -COOMe > COOH;

The thiazine structure, 17, for the product in these reactions is less likely due to non-formation of fragment ion 10 from 17. However, it is quite possible that thiazine, 17, if formed at all, may rearrange to thiazolidine (6) under electron impact, which may in turn give rise to fragment ion 10. We are examining this possibility.

* We are grateful to the referees for highlighting these observations.

>C=N– > >C=O) followed by aminolysis (propensity for aminolysis: –COOMe > –COOH > –CONH; >C=O > >C=N–).

3. Experimental

The melting points reported are uncorrected. The UV absorption spectrum was recorded in water on a Toshniwal Spectrophotometer Type RL 02. IR spectra were recorded on a Hitachi 270–30 (as KBr pellet) on Perkin-Elmer R 37 (as nujol mull) spectrophotometer (ν max in cm^{-1}). PMR spectra were recorded in DMSO-d_6 on Varian FT-80A or Gemini-200 using TMS as internal standard (chemical shifts in δ ppm). CMR spectrum were recorded on a Jeol FX-90 in DMSO-d_6 (chemical shifts in δ ppm). The mass spectrum was recorded on CEC-2-110 B double focussing mass spectrometer at 70 eV.

Maleimides, 2 (Searle 1948; Cava *et al* 1973), isomaleimides, 3 (Roderick and Bhatia 1963; Pyriadi and Harwood 1971), maleanilic acids, 4 (Patel and Balasubramaniyan 1977; Wagh *et al* 1982), methyl maleanilates, 5 and methyl fumaranilates, 8 (Patel and Balasubramaniyan 1981; Wagh *et al* 1982; Balasubramaniyan *et al* 1983), were prepared by known methods.

3.1 *General procedure for formation of N-aryl α-(2,3,4,5-tetrahydro-2-imino-4-oxo-1, 3-thiazole-5-yl)acetamides (6a–j) and fumaranilic acids (7b–e, 7i, 7j)*

An equimolar mixture of TU (0·76 g, 0·01 mol) with the required substrate (2b, 1·87 g; 3b, 1·87 g; 4b, 2·05 g; 5b, 2·17 g; 0·01 mol) in absolute ethanol (30 ml) was refluxed for 3 h. After aqueous work-up the solid obtained was filtered off, washed with water, dried and recrystallised from DMSO-EtOH (4:6, v/v) (table 1–4). Analytical and spectral data of thiazolidineacetamides (6a–j) thus prepared are given in table 5.

In the reaction of *o*-methylmaleanilic acid (4b) with TU, the solid obtained after aqueous work-up was extracted with saturated $NaHCO_3$ solution (20 ml). The bicarbonate insoluble N-(*o*-tolyl) α-(2,3,4,5-tetrahydro-4-oxo-1,3-thiazole-5-yl) acetamide (6b) was filtered off, washed with water and dried. Yield 0·32 g (12%) m.p. 224°C(d) (DMSO-EtOH, 4:6, v/v). The sodium bicarbonate extract on acidification (conc. HCl) furnished *o*-methylfumaranilic acid (7b); it was filtered, washed with water and dried. Yield of 7b was 1·62 g (82%), m.p. 240°C(abs. EtOH). Compound, substituent (X), % yield and m.p. (°C) are given: 7c, *o*-OMe, 75, 201; 7d, *o*-Cl, 40, 220; 7e, *o*-NO_2, 70, 232 (Lit. Wagh *et al* 1982, m.p. 199–201°C); 7i, *p*-NO_2, 85, 223 and 7j, *m*-NO_2, 85, 247. Analytical and spectral data of fumaranilic acids (7b–e, 7i, 7j) thus prepared are given in table 5.

3.2 *Hydrolysis of N-(p-nitro) 2-iminothiazolidineacetamide (6i) to 2-iminothiazolidine-acetic acid (1)*

A slurry of 6i (6·24 g, 0·02 mol) in ethanol (20 ml) was stirred with aqueous NaOH (10 ml, 2M) at room temperature for 1 h. The reaction mixture was then filtered, the filtrate acidified (AcOH–HCl) and kept in an ice-chest for two days. The solid product

Table 5. Analytical and spectral data of the compounds.

Compound	Molecular Formula	Analyses (%) Found C	Found H	Found N	Calculated C	Calculated H	Calculated N	IR absorption peaks of characteristic groups (cm^{-1})
1	$C_5H_6N_2O_3S$	34·75	3·67	16·04	34·5	3·45	16·1	3260, 3160–3150, 2400(*b*) 1680, 1650, 1640
6a	$C_{11}H_{11}N_3O_2S$	53·36	5·11	16·18	53·01	4·97	15·9	3300, 3250, 3150, 1700(*sh*) 1660(*sh*), 1650
6b	$C_{12}H_{13}N_3O_2S$	55·09	5·0	15·6	54·75	4·94	15·7	3250(*b*), 3000(*b*), 1680, 1660, 1645(*sh*)
6c	$C_{12}H_{13}N_3O_3S$	51·84	4·66	15·27	51·61	4·73	15·05	3260, 3100, 1680, 1660, 1645(*sh*)
6d	$C_{11}H_{10}N_3O_2SCl$	46·56	3·55	15·74	46·65	3·53	15·44	3500–3400, 3280, 1680, 1650–1630
6e	$C_{11}H_{10}N_4O_4S$	44·59	3·52	19·04	44·89	3·4	19·0	3380, 3360, 1690,(*sh*), 1680, 1650, 1610
6f	$C_{12}H_{13}N_3O_2S$	54·96	5·07	16·7	54·75	4·94	15·96	3250, 1680(*sh*), 1660–1650, 1590
6g	$C_{12}H_{13}N_3O_3S$	51·92	4·77	14·92	51·61	4·66	15·05	3250–3150, 1720(*sh*), 1680–1640, 1600
6h	$C_{11}H_{10}N_3O_2SCl$	46·42	3·58	15·57	46·65	3·53	15·44	3250–3110, 1710(*sh*), 1700–1660, 1650
6i	$C_{11}H_{10}N_4O_4S$	44·79	3·62	18·85	44·89	3·4	19·0	3220, 3120, 1660–1640, 1600
6j	$C_{11}H_{10}N_4O_4S$	44·77	3·58	19·24	44·89	3·4	19·0	3240, 3140–3120, 1680(*sh*), 1640–1620
7b	$C_{11}H_{11}NO_3$	64·39	5·28		64·39	5·36		3300, 3190, 1720, 1630, 1590
7c	$C_{11}H_{11}NO_4$	60·09	5·15		59·72	4·97		3250, 3170, 1700, 1620
7d	$C_{10}H_8NO_3Cl$	53·15	3·55		53·09	3·53		3260, 3200, 1720, 1650, 1630
7e	$C_{10}H_8N_2O_5$	50·67	3·44		50·84	3·38		3250, 3100, 1720, 1670, 1630
7i	$C_{10}H_8N_2O_5$	50·92	3·54		50·84	3·38		3300, 3100, 1700, 1680, 1600
7j	$C_{10}H_8N_2O_5$	50·98	3·43		50·84	3·38		3300, 3100, 1700, 1670, 1600
15a	$C_{10}H_{12}N_2O_2$	62·5	6·5		62·46	6·52		3412, 3324, 3210, 1709, 1653
15b	$C_{11}H_{14}N_2O_2$	64·07	6·79		64·35	6·84		3376, 3266, 3040, 1710, 1650
15d	$C_{10}H_{11}N_2O_2Cl$	53·26	4·92		53·09	4·86		3380, 3270–3290, 1710, 1660
15f	$C_{11}H_{14}N_2O_2$	64·13	6·73		64·35	6·84		3370, 3260, 3040, 1710, 1650

6a: UV: 245, 225, 200 nm (log ε 4·26, 4·34, 4·42);
PMR: 2·2(*s*, 3H), 2·76(*dd*, 1H, $J = 8$, 4 Hz), 3·2(*dd*, 1H, $J = 16$, 8 Hz), 3·6(*dd*, 1H, $J = 16$, 4 Hz), 7·25(*m*, 4H), 8·75(*s*, 1H), 8·9(*bs*, 1H), 9·5(*s*, 1H);
CMR: 189·25(C–7), 182·53(C–4), 168·94(C–2), 136·21(C–1′), 131·99(C–6′), 130·54(C–4′), 126·17(C–2′), 125·58(C–5′), 125·25(C–3′), 52·23(C–5), 39·7(C–6), 18·03(*o*–CH_3);
MS: *m/z* 263($M^{\cdot+}$, 8·15), 204($M - 59$, 0·2), 187($M - 76$, 46·44), 149($M - 114$, 1·28), 133($M - 130$, 21·93), 130($M - 133$, 62·35), 91($M - 172$, 43·86), 65($M - 198$, 40·42).

7b: PMR: 2·14(*s*, 3H), 6·58(*d*, 1H, $J = 16$ Hz), 7·22(*d*, 1H, $J = 16$ Hz), 7·0–7·42(*m*, 4H), 7·5(*bs*, 1H).

15a: PMR: 2·2 and 2·3(overlapping *t*, 4H), 5·5(*bs*, 2H), 6·1–7·1(*m*, 5H), 9·1(*bs*, 1H).

3.52 g (50%), m.p. 255–7°C(d) (DMSO-EtOH, 4:6, v/v) (Lit. Balasubramaniyan *et al* 1990, m.p. 256–8°C(d). Analytical and spectral data are given in table 5.

3.3 *Desulphurisation of N-aryl 2-iminothiazolidineacetamides (6a–b, 6d, 6f) with Raney nickel to the corresponding N-aryl succindiamides (11a–b, 11d, 11f).*

This reaction was carried out by adopting the reported procedure (Marrian 1949). Compound, substituent (X), % yield and m.p. (°C) are given: 11a, H, 30, 176 (Lit. Marrian 1949, m.p. 175–6°C); 11b, *o*-Me, 26, 184; 11d, *o*-Cl, 32, 182 and 11f, *p*-Me, 28, 194. Analytical and spectral data are given in table 5.

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References

Augustin M and Kohler M 1976 *Tetrahedron* **32** 2141
Augustin M and Muller W 1985 *J. Prakt. Chem.* 789
Augustin M, Rudorf W D and Pashe R 1974 *Z. Chem.* **14** 434
Arakelian A N, Dunn H Jr., Grieshammer L L and Coleman L E 1960 *J. Org. Chem.* **25** 465
Balasubramaniyan V, Balasubramaniyan P and Shaikh A S 1986 *Tetrahedron* **42** 2731
Balasubramaniyan V, Balasubramaniyan P and Wani M J 1990 *Indian J. Chem.* **B29** 1092
Balasubramaniyan V, Bhatia V G and Wagh S B 1983 *Tetrahedron* **39** 1475
Bethell J R and Maitland J 1961 *J. Chem. Soc.* 5217
Bhatia V G 1984 *Preparation of anilic acid esters using novel sulphur and phosphorus reagents and a study of their reactions*, Ph D. thesis, University of Pune
Boyd G V 1969 *J. Chem. Soc., Chem. Commun.* 1147
Cava M P, Deanna A D, Muth K and Mitchell M J 1973 *Org. Synth. Coll. Vol.* **5** 944
Kashima C, Schimizu M, Eto T and Omote Y 1986 *Bull. Chem. Soc. Jpn.* **59** 3317
Levy G C and Nelson G L 1972 *Carbon-13 nuclear magnetic resonance for organic chemists* (New York: Wiley)
Marrian D H 1949 *J. Chem. Soc.* 1797
Mustafa A, Asker W, Khattab S and Zayed S M A D 1961 *J. Org. Chem.* **26** 787
Nagase H 1973 *Chem. Pharm. Bull.* **21** 270
Patel M V and Balasubramaniyan V 1977 *Indian J. Chem.* **B15** 1142
Patel M V and Balasubramaniyan V 1981 *Indian J. Chem.* **B20** 804
Pyriadi T M and Harwood H J 1971 *J. Org. Chem.* **36** 821
Roderick W R and Bhatia P L 1963 *J. Org. Chem.* **36** 821
Sauers C K and Relles H M 1973 *J. Am. Chem. Soc.* **95** 7731
Searle N E 1948 US Patent 2 444 536; 1948 *Chem. Abstr.* **42** 7340c
Wagh S B, Balasubramaniyan P and Balasubramaniyan V 1982 *Indian J. Chem.* **B21** 1071