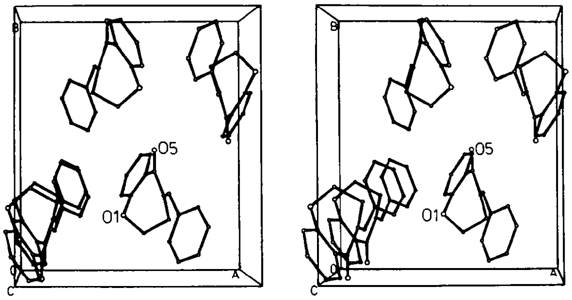

Fig. 2. A stereoscopic view of the unit-cell contents. H atoms are omitted for clarity.

observed in torsion angles O(5)—C(5)—C(11)—C(6) and O(5)—C(5)—C(4)—C(12) (Table 2) – the corresponding torsion angles in 3-benzylidene-4-chromanone are −0·7 (2) and −21·8 (2)° respectively. These two torsion angles are part of the tetrahydro-oxepin ring and presumably the strains imposed on this fragment of the 1,3-enone bridge by the seven-membered ring are responsible for their different magnitudes.

A stereoscopic view of the unit-cell contents (Motherwell & Clegg, 1978) is shown in Fig. 2. Only van der Waals intermolecular contacts are observed for this crystal.

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Structures of *N*-Trityl-L-aspartate Dibenzyl Ester and *N*-Trityl-L-leucine Benzyl Ester

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Abstract. $C_{37}H_{33}NO_4$ (I), $M_r = 555{\cdot}7$, monoclinic, $P2_1$, $a = 11{\cdot}211$ (7), $b = 7{\cdot}983$ (4), $c = 17{\cdot}582$ (14) Å, $\beta = 103{\cdot}88$ (6)°, $V = 1527$ (2) Å^3, $Z = 2$, $D_x = 1{\cdot}208$ (1) g cm^{-3}, λ(Mo $K\alpha$) = 0·71069 Å, $\mu = 0{\cdot}844$ cm^{-1}, $F(000) = 588$, room temperature, $R = 0{\cdot}0479$ for 1544 unique observed reflections. $C_{32}H_{33}NO_2$ (II), $M_r = 463{\cdot}7$, monoclinic, $C2$, $a = 28{\cdot}531$ (12), $b = 11{\cdot}730$ (3), $c = 16{\cdot}885$ (10) Å, $\beta = 106{\cdot}39$ (4)°, $V = 5421$ (4) Å^3, $Z = 8$, $D_x = 1{\cdot}136$ (1) g cm^{-3}, λ(Mo $K\alpha$) = 0·71069 Å, $\mu = 0{\cdot}756$ cm^{-1}, $F(000) = 1984$, room temperature, $R = 0{\cdot}0958$ with 2573 observed reflections. The structure of (I) clearly demonstrates that the α-benzyl ester approaches the bulky trityl group and this could explain

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the difficulty in the hydrolysis of this ester bond. A weak interaction also exists between the N—H and the two carbonyl groups [H(5)···O(2) = 2·393 (75), H(5)···O(4) = 2·436 (75) Å]. The two molecules of the asymmetric unit of compound (II) are interconnected by two hydrogen bonds [H(20*A*)···O(23*B*) = 2·383 (115), H(20*B*)···O(23*A*) = 2·728 (140) Å].

Introduction. The trityl (Trt) group is an important α-amino protecting group in peptide synthesis (Zervas & Theodoropoulos, 1956). For the introduction of the trityl group *via* trityl chloride, amino acids are alkylated as diethylammonium salts or esters (Stelakatos, Theodoropoulos & Zervas, 1959). The latter have to be saponified by strong alkali. Racemization has not been found during this step. In addition, Trt groups can be removed selectively in the presence of other protecting groups.

Recently, it has been established that the preparation of *N*-trityl-amino acids can be effected by 'one pot' synthesis *via* their silyl esters in high yields (Barlos, Papaioannou & Theodoropoulos, 1982). On the other hand, due to the steric hindrance of the Trt group, the alkaline hydrolysis of the *N*-Trt-L-aspartate dibenzyl ester proceeds through selective removal of the β-benzyl group, whereas in the presence of other N^{α}-protecting groups (benzyloxycarbonyl-, *tert*-butyloxycarbonyl-, *etc.*) the β-benzyl-L-aspartate derivative is obtained (Cordopatis & Theodoropoulos, 1975). The *N*-trityl-α-benzyl-L-aspartate, upon coupling with other amino acids or peptides by the dicyclohexylcarbodiimide/1-hydroxybenzotriazole (DCC/HOBt) method (König & Geiger, 1970), gives derivatives containing a β-aspartyl bond.

We report here the crystal and molecular structure of the title compounds for the purpose of comparison. The molecular conformation may be of use in comparative and theoretical studies of peptide conformation.

Experimental. Compound (I) was prepared from L-aspartate dibenzyl ester *p*-toluenesulfonate (Mazur, 1962) and trityl chloride in the presence of triethylamine according to the general procedure without racemization (Zervas & Theodoropoulos, 1956). Crystals after recrystallization from ethanol have m.p. 376–377 K and $[\alpha]_D^{25.0\,^{\circ}\mathrm{C}} = +12.8^{\circ}(\mathrm{CH_2Cl_2}$, 2·0 g dm^{-3}). Crystal 0·06 × 0·13 × 0·21 mm, Syntex $P2_1$ diffractometer, ω–2θ scanning technique to $2\theta = 47^{\circ}$, $-12 \leq h \leq 11$, $0 \leq k \leq 8$, $0 \leq l \leq 19$, variable scan speed, graphite-monochromatized Mo $K\alpha$. 15 reflections ($2\theta < 25^{\circ}$) for lattice parameters, one standard reflection every 50 without deviation. Absorption ignored. 2453 independent unique reflections of which 1544 with $I > 2{\cdot}5\sigma(I)$ remained for the refinement. The structure was solved by the version *YZARC* (Declercq, Germain & Woolfson, 1979) of the direct-methods program *MULTAN*, and refined by full-matrix least-squares techniques. All non-H atoms were located in a $\Delta\rho$ map and refined anisotropically. H atoms were calculated except H(5) which was located and refined with isotropic thermal parameters. Final $R = 0{\cdot}0479$, $wR = 0{\cdot}0504$, $\sum w(\Delta F)^2$ minimized, $w = k/[\sigma^2(F) + 0{\cdot}001199F^2]$, max. Δ/σ (for non-H atoms) = 0·08, max. and min. in final $\Delta\rho$ map 0·15 and −0·07 e Å^{-3}.

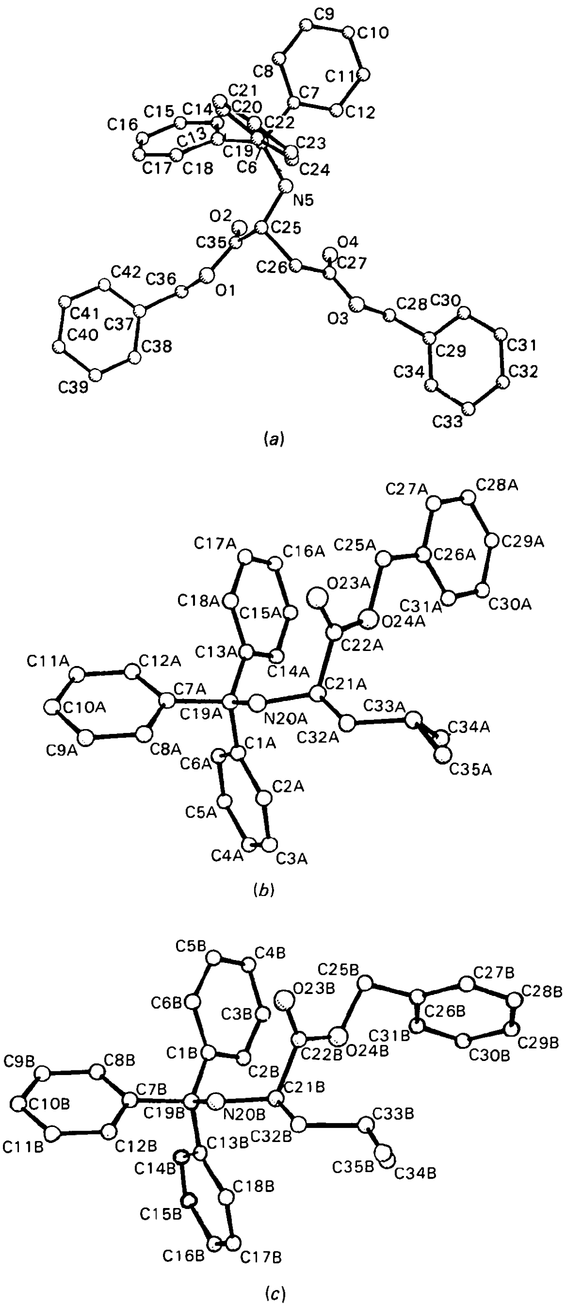

Fig. 1. Perspective view of (*a*) compound (I), (*b*) compound (II), molecule *A* and (*c*) compound (II), molecule *B*, showing atom numbering.

Table 1. *Positional* ($\times 10^4$) *parameters with their e.s.d.'s in parentheses and estimated isotropic thermal parameters* ($\times 10^2$)

$$B_{eq} = \tfrac{4}{3}\textstyle\sum_i\sum_j\beta_{ij}\mathbf{a}_i.\mathbf{a}_j.$$

	x	*y*	*z*	B_{eq}/B(Å^2)
Compound (I)				
O(1)	1390 (4)	950	8287 (2)	601
O(2)	3028 (4)	2467 (11)	8822 (3)	738
O(3)	650 (4)	5841 (9)	6822 (3)	522
O(4)	2259 (6)	5463 (10)	7771 (4)	1054
N(5)	3674 (4)	2688 (10)	7360 (3)	382
C(6)	4660 (5)	1446 (11)	7337 (3)	396
C(7)	5821 (5)	2452 (11)	7288 (3)	369
C(8)	6922 (5)	1632 (11)	7351 (4)	489
C(9)	7953 (6)	2489 (14)	7288 (4)	589
C(10)	7933 (7)	4182 (13)	7152 (5)	625
C(11)	6840 (7)	4993 (12)	7077 (5)	656
C(12)	5780 (6)	4156 (12)	7149 (4)	536
C(13)	4908 (5)	328 (11)	8066 (3)	362
C(14)	5716 (6)	841 (12)	8756 (4)	485
C(15)	5879 (7)	−91 (14)	9439 (4)	597
C(16)	5263 (7)	−1571 (13)	9442 (4)	597
C(17)	4457 (6)	−2114 (13)	8772 (4)	569
C(18)	4277 (5)	−1164 (11)	8087 (4)	446
C(19)	4293 (5)	474 (11)	6549 (3)	357
C(20)	4760 (6)	−1101 (11)	6463 (4)	470
C(21)	4513 (7)	−1905 (13)	5740 (4)	581
C(22)	3798 (7)	−1118 (13)	5097 (4)	555
C(23)	3335 (6)	443 (13)	5160 (4)	516
C(24)	3589 (5)	1247 (11)	5876 (4)	425
C(25)	2491 (5)	2022 (10)	7408 (3)	356
C(26)	1449 (5)	3146 (10)	6942 (4)	373
C(27)	1533 (6)	4919 (11)	7215 (4)	458
C(28)	588 (7)	7580 (11)	7062 (4)	580
C(29)	−132 (6)	8552 (10)	6371 (4)	419
C(30)	456 (6)	9192 (12)	5829 (4)	529
C(31)	−203 (9)	10071 (13)	5204 (5)	714
C(32)	−1435 (9)	10287 (13)	5100 (5)	667
C(33)	−2036 (8)	9687 (13)	5629 (5)	701
C(34)	−1373 (7)	8780 (11)	6276 (4)	566
C(35)	2365 (6)	1863 (12)	8259 (4)	469
C(36)	1166 (9)	727 (15)	9071 (4)	825
C(37)	469 (7)	−862 (14)	9053 (4)	558
C(38)	−811 (8)	−903 (15)	8916 (4)	650
C(39)	−1403 (9)	−2394 (21)	8894 (5)	845
C(40)	−728 (18)	−3856 (18)	9014 (6)	993
C(41)	482 (17)	−3785 (20)	9172 (7)	1027
C(42)	1094 (9)	−2332 (19)	9194 (5)	793
H(5)	3927 (57)	3484 (89)	7806 (37)	582
Compound (II)				
C(1*A*)	6094 (4)	443 (20)	3471 (8)	603
C(2*A*)	6026 (7)	−662 (20)	3794 (12)	884
C(3*A*)	5602 (8)	−726 (20)	4151 (12)	1004
C(4*A*)	5329 (8)	332 (24)	4123 (13)	1054
C(5*A*)	5409 (8)	1372 (23)	3823 (14)	1091
C(6*A*)	5805 (8)	1369 (18)	3569 (12)	906
C(7*A*)	6901 (4)	1396 (12)	3807 (7)	457
C(8*A*)	7178 (4)	842 (13)	4526 (7)	523
C(9*A*)	7488 (6)	1506 (16)	5133 (8)	663
C(10*A*)	7514 (6)	2687 (16)	5081 (9)	747
C(11*A*)	7215 (8)	3186 (15)	4409 (12)	975
C(12*A*)	6908 (6)	2547 (14)	3739 (10)	849
C(13*A*)	6374 (5)	1269 (11)	2305 (8)	561
C(14*A*)	5888 (6)	1343 (14)	1847 (9)	811
C(15*A*)	5783 (8)	1896 (23)	1044 (13)	1060
C(16*A*)	6162 (13)	2355 (19)	787 (13)	1278
C(17*A*)	6661 (7)	2285 (15)	1252 (10)	902
C(18*A*)	6754 (5)	1720 (13)	2000 (8)	612
C(19*A*)	6526 (4)	627	3137 (6)	442
N(20*A*)	6773 (4)	−435 (9)	3081 (5)	401
C(21*A*)	6549 (4)	−1161 (10)	2375 (6)	410
C(22*A*)	6703 (4)	−898 (11)	1611 (6)	367
O(23*A*)	7117 (3)	−788 (9)	1597 (5)	603
O(24*A*)	6322 (2)	−802 (8)	933 (4)	462
C(25*A*)	6443 (4)	−540 (13)	168 (6)	571
C(26*A*)	5977 (5)	−562 (13)	−525 (7)	523
C(27*A*)	6018 (7)	−157 (31)	−1256 (11)	1547
C(28*A*)	5576 (9)	−238 (34)	−1962 (11)	1680
C(29*A*)	5174 (7)	−595 (23)	−1853 (12)	1098
C(30*A*)	5164 (8)	−1089 (32)	−1138 (13)	2004
C(31*A*)	5563 (7)	−1004 (26)	−494 (10)	1452
C(32*A*)	6704 (5)	−2394 (12)	2612 (8)	575
C(33*A*)	6515 (6)	−3319 (13)	1974 (9)	750
C(34*A*)	5930 (5)	−3332 (16)	1634 (11)	885
C(35*A*)	6714 (7)	−4499 (16)	2330 (12)	936
C(1*B*)	8483 (5)	1303 (10)	2005 (8)	573
C(2*B*)	8930 (5)	1646 (14)	2525 (10)	797
C(3*B*)	8961 (8)	2552 (20)	3090 (13)	1125
C(4*B*)	8526 (10)	3023 (17)	3194 (11)	1095
C(5*B*)	8083 (7)	2713 (14)	2680 (11)	859
C(6*B*)	8043 (5)	1813 (12)	2084 (8)	636
C(7*B*)	8053 (3)	435 (11)	589 (6)	414
C(8*B*)	7960 (4)	1527 (12)	276 (8)	587
C(9*B*)	7634 (5)	1608 (12)	−556 (9)	648
C(10*B*)	7414 (5)	695 (15)	−993 (8)	674
C(11*B*)	7535 (5)	−401 (12)	−648 (7)	595
C(12*B*)	7853 (4)	−534 (11)	150 (7)	506
C(13*B*)	8912 (4)	−86 (12)	1303 (6)	484
C(14*B*)	9173 (5)	760 (17)	1004 (8)	790
C(15*B*)	9623 (6)	415 (26)	855 (11)	969
C(16*B*)	9809 (6)	−653 (23)	968 (9)	872
C(17*B*)	9547 (5)	−1472 (15)	1262 (7)	693
C(18*B*)	9090 (4)	−1164 (13)	1401 (7)	523
C(19*B*)	8428 (4)	212	1468 (6)	438
N(20*B*)	8229 (3)	−725 (8)	1840 (5)	369
C(21*B*)	8477 (4)	−1034 (10)	2696 (6)	405
C(22*B*)	8331 (4)	−340 (10)	3335 (7)	407
O(23*B*)	7914 (3)	−149 (8)	3315 (4)	557
O(24*B*)	8701 (3)	−38 (7)	3945 (4)	551
C(25*B*)	8598 (4)	501 (14)	4656 (6)	621
C(26*B*)	9042 (5)	376 (16)	5386 (9)	622
C(27*B*)	9058 (10)	−231 (25)	6017 (22)	1381
C(28*B*)	9447 (12)	−497 (22)	6646 (16)	1260
C(29*B*)	9812 (13)	45 (27)	6765 (18)	1301
C(30*B*)	9933 (16)	172 (40)	6089 (31)	1909
C(31*B*)	9481 (16)	593 (33)	5301 (23)	1801
C(32*B*)	8369 (4)	−2281 (10)	2812 (6)	474
C(33*B*)	8616 (5)	−2813 (11)	3674 (7)	655
C(34*B*)	9184 (5)	−2708 (19)	3883 (10)	1056
C(35*B*)	8450 (8)	−4024 (14)	3702 (11)	997
H(20*A*)	7049 (41)	−260 (101)	3028 (64)	237
H(20*B*)	8058 (34)	−989 (90)	1535 (60)	277

Very small crystals of (II) were prepared from L-leucine benzyl ester *p*-toluenesulfonate and trityl chloride by the above procedure. The crystalline product after recrystallization from methanol has m.p. 373–374 K. Crystal 0·05 × 0·12 × 0·20 mm, Syntex $P2_1$ diffractometer, $2\theta = 47°$ with ω–2θ scan, Mo*Kα*, 4238 unique independent reflections of which 2573 with $I > 2{\cdot}5\sigma(I)$ used for the refinement, $-31 \leq h \leq 28$, $0 \leq k \leq 12$, $0 \leq l \leq 18$. 15 reflections ($2\theta < 27°$) for lattice parameters, one standard reflection every 50 without deviation. Absorption ignored. The asymmetric unit contains two molecules with 70 independent non-H atoms. The structure was solved by the version *MULTAN*11/84 (Main, Germain & Woolfson, 1984). The *E* map revealed the positions of all 70 independent non-H atoms which were refined with anisotropic thermal parameters. H atoms omitted except H(20*A*) and H(20*B*) which were located and refined isotropically. Final $R = 0{\cdot}0922$, $wR = 0{\cdot}0955$ for the 2573 remaining reflexions with *SHELX76* (Sheldrick, 1976). $\sum w(\Delta F)^2$ minimized, $w = k/[\sigma^2(F) + 0{\cdot}00076F^2]$, max. $\Delta/\sigma < 0{\cdot}5$ [except C(1*A*)–C(6*A*), C(26*B*)–C(31*B*) (large thermal vibration)], max. and min. in final $\Delta\rho$ map 0·54 and −0·32 e Å^{-3}. Atomic scattering factors from *SHELX76*.

Discussion. Fig. 1 shows the two compounds with atomic numbering. Positional parameters and B_{eq}

Table 2. *Important bond lengths* (Å) *and angles* (°) *with e.s.d.'s*

Compound (I)

C(6)—C(7)	1·549 (8)	C(25)—C(35)	1·542 (9)
C(6)—C(13)	1·532 (8)	C(35)—O(2)	1·188 (7)
C(6)—C(19)	1·554 (8)	C(35)—O(1)	1·324 (8)
N(5)—H(5)	0·996 (68)	O(1)—C(36)	1·469 (8)
C(6)—N(5)	1·493 (8)	C(27)—O(4)	1·194 (7)
C(25)—N(5)	1·450 (7)	C(27)—O(3)	1·292 (7)
C(25)—C(26)	1·543 (8)	O(3)—C(28)	1·458 (8)
C(25)—N(5)—H(5)	106·2 (37)	C(26)—C(25)—N(5)	110·0 (5)
C(6)—N(5)—C(25)	116·8 (5)	C(35)—C(25)—N(5)	112·6 (5)
C(6)—N(5)—H(5)	111·9 (36)	C(35)—C(25)—C(26)	109·2 (5)

Compound (II)

	Molecule *A*	Molecule *B*
C(19)—C(1)	1·507 (19)	1·551 (14)
C(19)—C(7)	1·598 (14)	1·586 (12)
C(19)—C(13)	1·545 (15)	1·525 (17)
C(19)—N(20)	1·447 (12)	1·458 (12)
C(21)—N(20)	1·458 (14)	1·463 (12)
H(20)—N(20)	0·842 (124)	0·677 (90)
C(21)—C(22)	1·507 (17)	1·501 (17)
C(21)—C(32)	1·532 (18)	1·518 (17)
C(22)—O(23)	1·193 (15)	1·202 (13)
C(22)—O(24)	1·342 (11)	1·299 (11)
O(24)—C(25)	1·460 (14)	1·458 (15)
C(19)—N(20)—C(21)	116·6 (8)	118·3 (8)
H(20)—N(20)—C(19)	106·5 (81)	107·5 (92)
H(20)—N(20)—C(21)	105·1 (75)	133·0 (90)
N(20)—C(21)—C(22)	114·5 (10)	114·9 (9)
N(20)—C(21)—C(32)	108·1 (9)	107·9 (8)
C(22)—C(21)—C(32)	106·6 (11)	108·9 (10)

values are given in Table 1. Important bond lengths and angles are listed in Table 2.*

The structure of (I) clearly demonstrates that the α-benzyl ester group approaches the bulky trityl moiety, whereas the *β*-ester is pointing away. This can explain the difficulty in the alkaline hydrolysis of the α-benzyl ester of this compound. Moreover, the bonds observed between the N—H and the two carbonyl groups [H(5)···O(2) = 2·393 (75), H(5)···O(4) = 2·436 (75) Å] show a weak interaction between the two ester chains and the bulky N-terminal trityl group.

The low precision of the refinement of (II) is due to the poor quality of the crystals and to large thermal vibration of the phenyl rings (two independent molecules with 70 non-H atoms in the asymmetric unit). We have refined the structure with the phenyl rings constrained to be regular hexagons. Also, we have tried to include two different orientations of the rings and refined occupation factors, without obtaining better convergence. In the final *Δρ* map we were able to localize the positions of H(20*A*) and H(20*B*). The two molecules of the asymmetric unit are interconnected by two hydrogen bonds involving, as in compound (I), the N-terminal N—H moiety and the ester carbonyl group [H(20*A*)···O(23*B*) = 2·383 (115), H(20*B*)···O(23*A*) = 2·728 (140) Å].

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* Lists of structure factors, anisotropic thermal parameters, H-atom parameters, a complete list of bond lengths and angles, torsion angles and equations of planes have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 43427 (65 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Structure of 1,3,4-Thiadiazole-2,5-diamine

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Abstract. $C_2H_4N_4S$, $M_r = 116{\cdot}1$, monoclinic, $P2_1/n$, $a = 11{\cdot}099\ (2)$, $b = 10{\cdot}328\ (3)$, $c = 8{\cdot}971\ (2)$ Å, $\beta = 112{\cdot}58\ (1)°$, $V = 949{\cdot}6\ (4)$ Å^3; $Z = 8$, $D_m = 1{\cdot}65$ (by flotation), $D_x = 1{\cdot}63$ Mg m^{-3}, λ(Mo $K\alpha$) = 0·71069 Å, $\mu = 0{\cdot}322$ mm^{-1}, $F(000) = 480$, $T = 293$ K, final $R = 0{\cdot}029$ for 1436 significant reflections. The asymmetric unit contains two crystallographically unique molecules which are essentially planar. Both molecules have almost complete C_{2v} symmetry and are linked by N(amino)—H···N(ring) hydrogen bonds in the crystal.

Introduction. Recently we reported the formation of heterocyclic ring compounds by the reactions between substituted thioureas and iron(II or III) chloride

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