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Hydrogen-bonding patterns in three substituted *N*-benzyl-*N*-(3-*tert*-butyl-1-phenyl-1*H*-pyrazol-5-yl)acetamides

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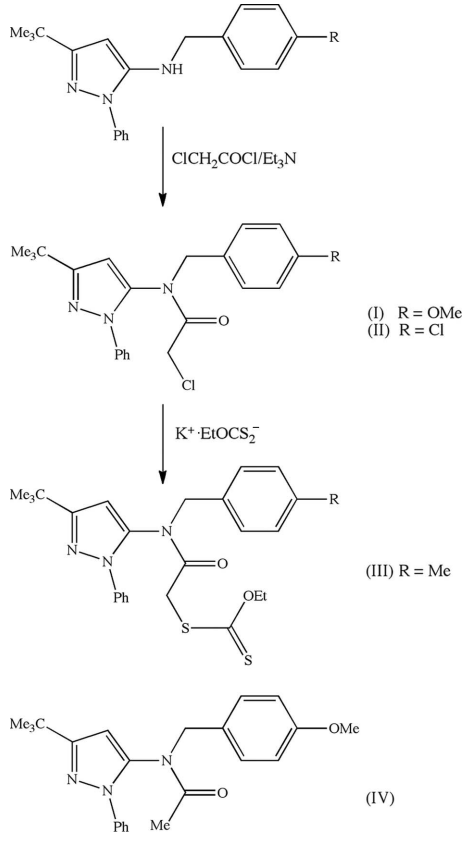
The molecules of *N*-(3-*tert*-butyl-1-phenyl-1*H*-pyrazol-5-yl)-2-chloro-*N*-(4-methoxybenzyl)acetamide, $C_{23}H_{26}ClN_3O_2$, are linked into a chain of edge-fused centrosymmetric rings by a combination of one C—H···O hydrogen bond and one C—H···π(arene) hydrogen bond. In *N*-(3-*tert*-butyl-1-phenyl-1*H*-pyrazol-5-yl)-2-chloro-*N*-(4-chlorobenzyl)acetamide, $C_{22}H_{23}Cl_2N_3O$, a combination of one C—H···O hydrogen bond and two C—H···π(arene) hydrogen bonds, which utilize different aryl rings as the acceptors, link the molecules into sheets. The molecules of *S*-[*N*-(3-*tert*-butyl-1-phenyl-1*H*-pyrazol-5-yl)-*N*-(4-methylbenzyl)carbamoyl]methyl *O*-ethyl carbonodithioate, $C_{26}H_{31}N_3O_2S_2$, are also linked into sheets, now by a combination of two C—H···O hydrogen bonds, both of which utilize the amide O atom as the acceptor, and two C—H···π(arene) hydrogen bonds, which utilize different aryl groups as the acceptors.

Comment

Fused pyrazole derivatives have a wide range of potential applications (Elguero, 1984, 1996) and, in connection with a synthetic study of new routes to such compounds, we report here the structure of three new compounds, (I)–(III) (Figs. 1–3), prepared as intermediates in a synthetic pathway designed to produce fused pyrazole compounds from alkyl xanthate derivatives *via* free-radical cyclization processes (Binot *et al.*, 2003; Bacqué *et al.*, 2004; Ibarra-Rivera *et al.*, 2007). In this synthetic sequence (see scheme), benzyl-substituted *N*-benzyl-*N*-(3-*tert*-butyl-1-phenyl-1*H*-pyrazol-5-yl)-2-chloroacetamides are prepared by reaction of the corresponding 5-benzylamino-3-*tert*-butyl-1-phenyl-1*H*-pyrazoles with chloroacetyl chloride, followed by reaction with potassium *O*-ethyl carbonodithioate to introduce the alkyl xanthate moiety. We report here the molecular and supramolecular structures of two chloroacetamide intermediates, namely *N*-(3-*tert*-butyl-1-phenyl-1*H*-pyrazol-5-yl)-2-chloro-*N*-(4-methoxybenzyl)acetamide, (I), and *N*-(3-*tert*-butyl-1-phenyl-1*H*-pyrazol-5-yl)-2-chloro-*N*-(4-chlorobenzyl)acetamide, (II), and one carbonodithioate intermediate, *S*-[*N*-(3-*tert*-butyl-1-phenyl-1*H*-pyrazol-5-yl)-*N*-(4-methylbenzyl)carbamoyl]methyl *O*-ethyl carbonodithioate, (III), all of which have interesting patterns of hydrogen bonding in their crystal structures. We compare the molecular structures, particularly the molecular conformations, of compounds (I)–(III) with that of the simple acetamide *N*-(3-*tert*-butyl-1-phenyl-1*H*-pyrazol-5-yl)-*N*-(4-methoxybenzyl)acetamide, (IV), whose structure was reported recently (Castillo *et al.*, 2010), and we also compare the hydrogen bonding in (I) with that in (IV).



Compounds (I) and (II) differ only in the identity of the 4-substituent in the benzyl unit, *viz.* methoxy in compound (I) and chloro in compound (II), but despite this rather small change of substituent, these two compounds crystallize in different crystal systems, *viz.* triclinic for (I) and monoclinic for (II). Similarly, the pattern of the intermolecular hydrogen bonds is different in (I) and (II) leading to different forms of supramolecular aggregation, as discussed below, despite the fact that in neither compound does the substituent in question play any direct role in the intermolecular interactions. Compounds (I) and (IV) (Castillo *et al.*, 2010) are even closer in similarity, differing only by the replacement of one of the acetyl H atoms in (IV) by a Cl atom in (I); these two compounds crystallize in the same space group, $P\overline{1}$, with unit-cell volumes which differ by less than 1%, while the corre-

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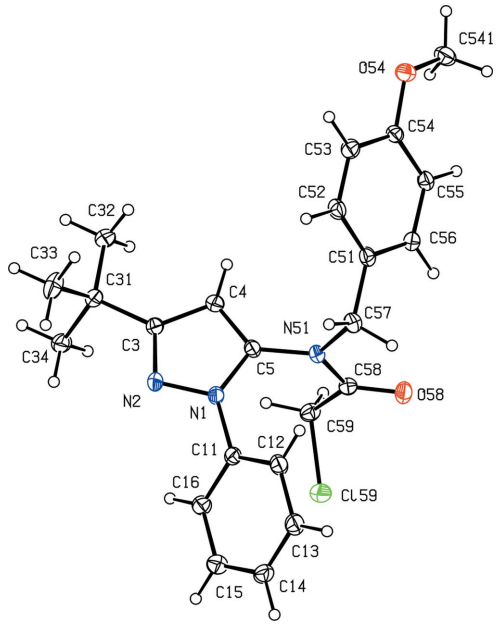

Figure 1
The molecular structure of compound (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level.

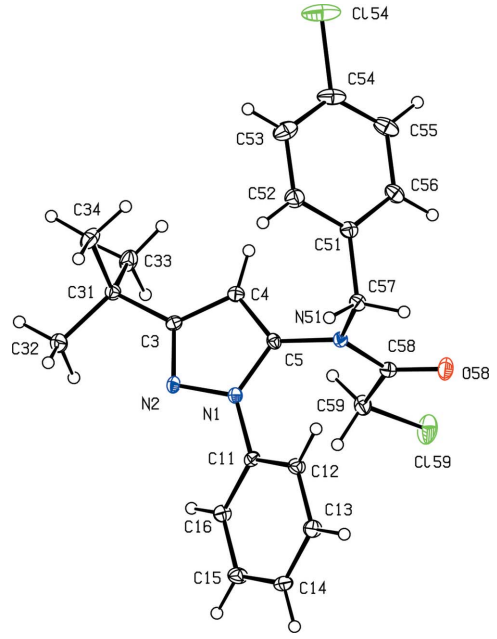

Figure 2
The molecular structure of compound (II), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level.

sponding unit-cell angles differ by less than 2°. However, unit-cell vectors *a* and *b* are larger for (I) by *ca* 2.5 and 6.6%, while the unit-cell vector *c* is smaller for (I) by *ca* 7.1%. This degree of similarity raises the question of whether (I) and (IV) can be regarded as isomorphous or isostructural; examination of the atom coordinates for the two compounds shows that the coordinates of corresponding atoms are approximately related to one another by the noncrystallographic transformation $(1-y, 1-x, \frac{1}{2}-z)$, but, as discussed below, the two reference molecules are selected to be the same conformational

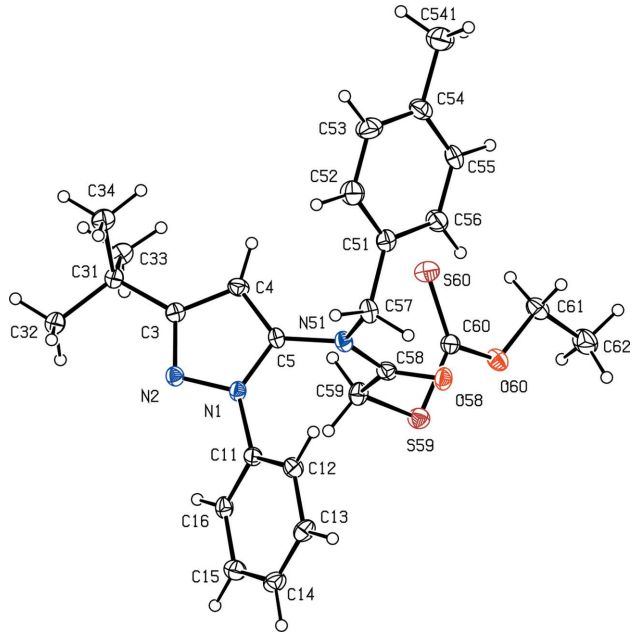

Figure 3
The molecular structure of compound (III), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level.

enantiomer and the hydrogen-bonded structures of (I) and (IV) are significantly different.

The molecular conformations of compounds (I)–(III) can, apart from the carbonodithioate portion of compound (III), be defined in terms of a rather small number of torsion angles, and Table 1 lists the values of these angles along with the corresponding values for compound (IV) (Castillo *et al.*, 2010). It is immediately evident that all four compounds adopt very similar conformations for their common fragments, with the exception of the orientation of the *tert*-butyl groups. In each of (I)–(IV), one methyl C atom of the *tert*-butyl group, denoted C32 in each case, lies close to, but not in the plane of, the adjacent pyrazole ring. However, while in compounds (I) and (IV) atom C32 lies remote from the ring atom N2, in compounds (II) and (III) this atom lies close to N2 (Figs. 1–3 and Table 1). The *tert*-butyl group has local threefold rotational symmetry, and in compounds (I)–(IV) it is bonded directly to a planar pyrazole ring; hence the rotational barriers about the bonds C3—C31 approximate to sixfold barriers and, as such, they are expected to be very low, providing essentially no intramolecular hindrance to rotation about this bond. In addition, there are no direction-specific intermolecular interactions present which might provide intermolecular hindrance to such a rotation. Indeed this type of rapid rotation of *tert*-butyl groups has been observed in a number of systems using solid-state NMR spectroscopy (Riddell & Rogerson, 1996, 1997). Thus, it is possible that the observed orientations of the *tert*-butyl groups are determined primarily by the space left available by the supramolecular aggregation. None of the molecules of (I)–(III) exhibits any internal symmetry and so all are conformationally chiral, but in every case the space group accommodates equal numbers of the two conformational enantiomers.

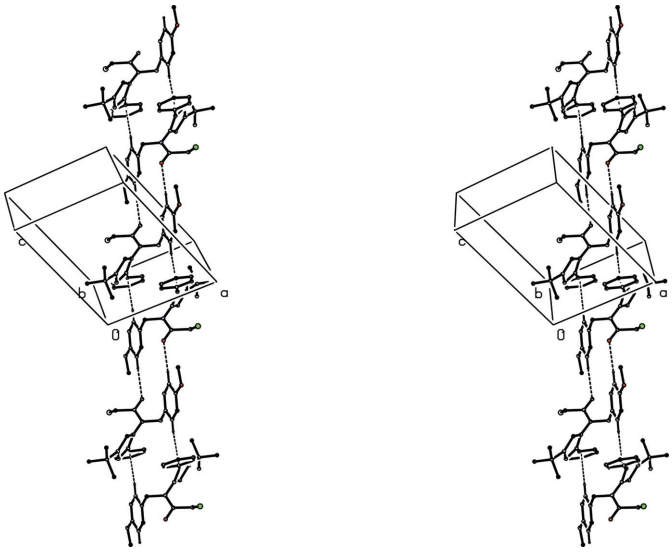

Figure 4
A stereoview of part of the crystal structure of compound (I), showing the formation of a hydrogen-bonded chain of edge-fused centrosymmetric rings running parallel to [111]. For the sake of clarity, H atoms not involved in the motifs shown have been omitted.

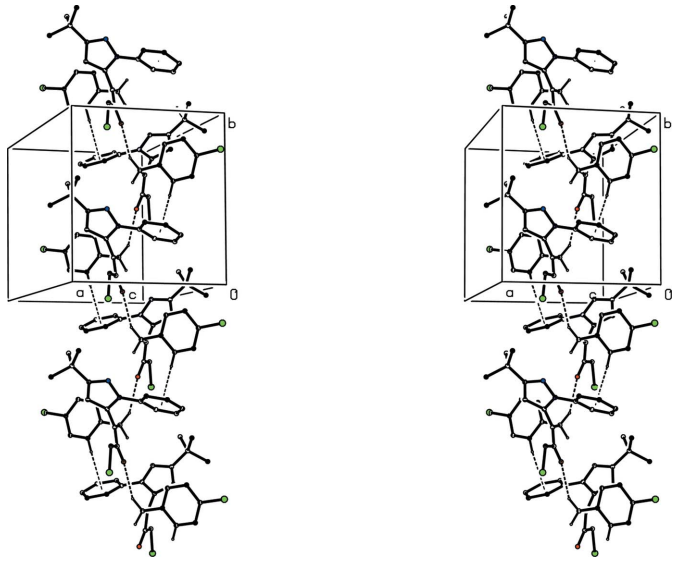

Figure 5
A stereoview of part of the crystal structure of compound (II), showing the formation of a hydrogen-bonded chain of edge-fused centrosymmetric rings running parallel to [010]. For the sake of clarity, H atoms not involved in the motifs shown have been omitted.

The supramolecular aggregation in compounds (I)–(III) is controlled by a combination of C—H···O and C—H···π(arene) hydrogen bonds (Table 2 where, for each compound, the C—H···O hydrogen bonds are listed first). However, C—H···N hydrogen bonds and aromatic π–π stacking interactions are absent from the structures of all three compounds. While amide atom O58 acts as a hydrogen-bond acceptor in each of (I)–(III), as indeed it does in compound (IV) also (Castillo *et al.*, 2010), different hydrogen-bond donors to O58 are active in each of (I)–(III). In compound (I) only the unsubstituted aryl ring acts as an acceptor of a C—H···π(arene) hydrogen bond, but in compounds (II) and (III) both aryl rings accept hydrogen bonds. The hydrogen-bonded structure of compound (I) is one-dimensional and thus fairly simple, whereas the hydrogen-bonded structures of compounds (II) and (III) are both two-dimensional and considerably more complex than that of compound (I).

In compound (I), the two independent hydrogen bonds (Table 2) generate a chain of edge-fused centrosymmetric rings running parallel to the [111] direction, with $R_2^2(16)$ (Bernstein *et al.*, 1995) rings built from pairs of C—H···O hydrogen bonds centred at $(n, n, n-\frac{1}{2})$, where n represents an integer, and with the rings built from pairs of C—H···π(arene) hydrogen bonds centred at $(n+\frac{1}{2}, n+\frac{1}{2}, n)$, where n again represents an integer (Fig. 4).

The hydrogen-bonded structure of compound (II) takes the form of a sheet, whose formation is readily analysed using the substructure approach (Ferguson *et al.*, 1998*a*,*b*; Gregson *et al.*, 2000). Two independent one-dimensional substructures can be identified in the crystal structure of compound (II). In the first of these, the combined actions of the C—H···O hydrogen bond and the C—H···π(arene) hydrogen bond which utilizes the unsubstituted aryl ring as the acceptor link molecules related by the 2_1 screw axis along $(\frac{1}{2}, y, \frac{1}{4})$ into a chain of

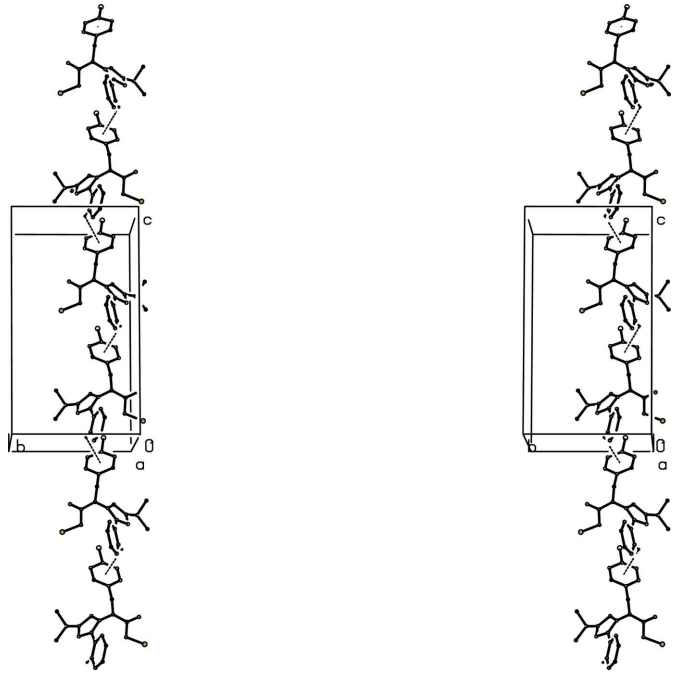

Figure 6
A stereoview of part of the crystal structure of compound (II), showing the formation of a hydrogen-bonded chain running parallel to [001]. For the sake of clarity, H atoms not involved in the motif shown have been omitted.

symmetry-related rings running parallel to the [010] direction, and within which the C—H···O interaction forms a $C(5)$ chain (Fig. 5). In the second, and simpler, substructure, a single C—H···π(arene) hydrogen bond using the substituted aryl ring as the acceptor links molecules related by the c-glide plane at $y=\frac{1}{4}$ into a chain running parallel to the [001] direction (Fig. 6).

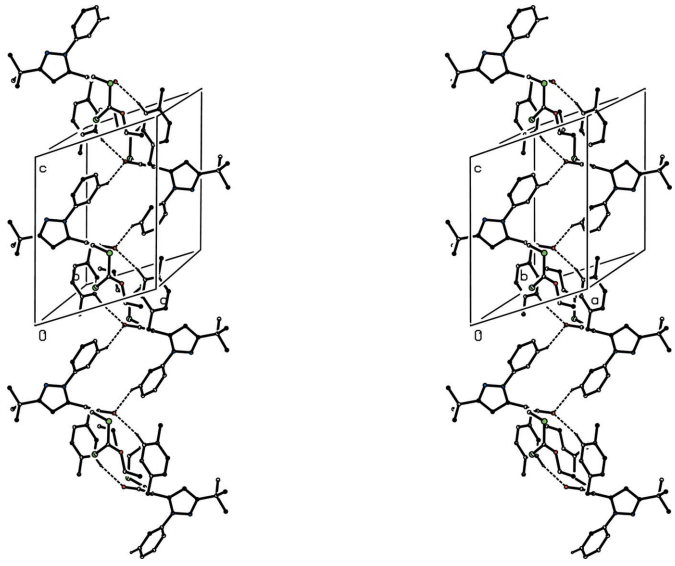

Figure 7
A stereoview of part of the crystal structure of compound (III), showing the formation of a hydrogen-bonded chain of edge-fused centrosymmetric rings running parallel to [001]. For the sake of clarity, H atoms not involved in the motifs shown have been omitted.

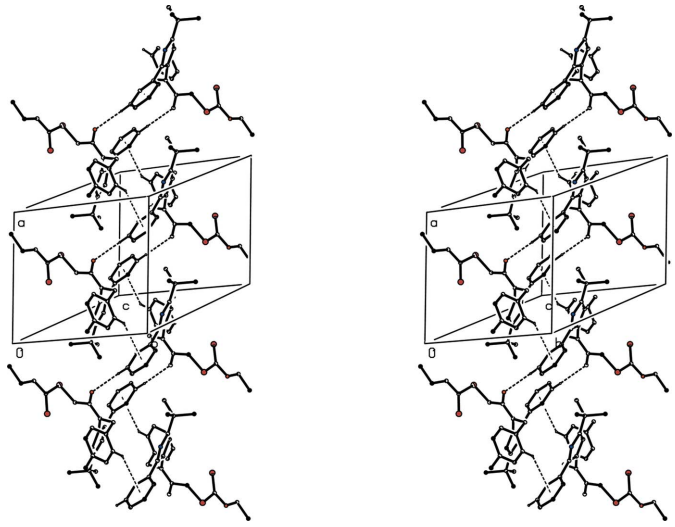

Figure 8
A stereoview of part of the crystal structure of compound (III), showing the formation of a hydrogen-bonded chain of edge-fused centrosymmetric rings running parallel to [100]. For the sake of clarity, H atoms not involved in the motifs shown have been omitted.

Although compound (III) crystallizes in the space group $P\overline{1}$ with only one symmetry operator other than translation available, the occurrence in the structure of four independent hydrogen bonds, each of them linking pairs of molecules related to one another by inversion (Table 2), permits the development of a two-dimensional hydrogen-bonded structure. As with the structure of compound (II), it is convenient to consider the formation of the sheet structure in compound (III) in terms of two one-dimensional substructures. The two C—H···O hydrogen bonds in the structure of (III) both utilize the same atom, O58, as the acceptor and, in combination, these two interactions generate a chain of edge-fused centrosymmetric rings running parallel to the [001] direction. Within this chain, $R_2^2(16)$ rings centred at $(\frac{1}{2}, \frac{1}{2}, n)$, where n represents an integer, alternate with $R_2^2(18)$ rings centred at $(\frac{1}{2}, \frac{1}{2}, n + \frac{1}{2})$, where n again represents an integer (Fig. 7). The combination of the $R_2^2(18)$ ring with the centrosymmetric ring generated by the C—H···π(arene) hydrogen bond involving the unsubstituted aryl group produces a second chain of edge-fused centrosymmetric rings, this time running parallel to the [100] direction. The rings built from pairs of C—H···O hydrogen bonds are centred at $(n + \frac{1}{2}, \frac{1}{2}, \frac{1}{2})$, and those built from paired C—H···π(arene) hydrogen bonds are centred at $(n, \frac{1}{2}, \frac{1}{2})$, where in each case n represents an integer (Fig. 8). The formation of two chains of rings parallel to the [100] and [001] directions, respectively, is sufficient to generate a sheet of considerable complexity lying parallel to (010). The second C—H···π(arene) hydrogen bond, in which the substituted aryl ring acts as the acceptor, lies within this sheet and it can be regarded as a modest reinforcement of the chain parallel to [001].

It is of interest briefly to compare the hydrogen-bonded structures of compounds (I) and (IV), particularly in view of the similarity in their unit-cell dimensions. In compound (IV) (Castillo *et al.*, 2010), a chain of edge-fused centrosymmetric rings is generated by the combination of one C—H···O hydrogen bond and one C—H···π(arene) hydrogen bond, rather as in compound (I). However, the rings formed by the C—H···O hydrogen bond are of $R_2^2(20)$ type in (IV) as opposed to $R_2^2(16)$ type in (I), and the chain runs parallel to [100] in (IV), *i.e.* parallel to the shortest unit-cell edge, whereas in (I) the chain runs parallel to [111], *i.e.* parallel to a body-diagonal of the unit cell.

Experimental

For the synthesis of compounds (I) and (II), mixtures of chloroacetyl chloride (0.1 ml, 1.25 mmol), dichloromethane (8 ml) and triethylamine (0.3 ml, 2.15 mmol) were cooled in an ice-water bath under an argon atmosphere. To each mixture, a solution (0.6 mmol) of the appropriate 5-benzylamino-3-*tert*-butyl-1-phenyl-1*H*-pyrazole [3-*tert*-butyl-5-(4-methoxybenzylamino)-1-phenyl-1*H*-pyrazole for (I) or 3-*tert*-butyl-5-(4-chlorobenzylamino)-1-phenyl-1*H*-pyrazole for (II)] in dichloromethane (2 ml) was added and each mixture was then left at room temperature for 10 h. In each case, the solvent was removed under reduced pressure and the resulting solid product was purified by column chromatography on silica gel (ethyl acetate/dichloromethane gradient) to obtain compound (I) in 86% yield and compound (II) in 82% yield. Crystals of (I) and (II) suitable for single-crystal X-ray diffraction were grown by slow evaporation, at ambient temperature and in air, of solutions in ethanol. For (I): yellow crystals, m.p. 353 K; MS (70 eV) *m*/*z* (%): 411/413 (30/10) [M^+], 226 (43), 121 (100); elemental analysis found: C 67.3, H 6.4, N 9.8%; $C_{23}H_{26}ClN_3O_2$ requires: C 67.1, H 6.4, N 10.2%. For (II): colourless crystals, m.p. 432 K; MS (70 eV) *m*/*z* (%): 419/417/415 (6/34/51) [M^+], 368/366 (23/63) [M−49], 340/338 (19/52), [M−Ph], 127/125 (29/100) [C_7H_6Cl], 77 (40) [Ph]; HRMS found: 415.1213, required for $C_{22}H_{23}Cl_2N_3O$: 415.1218.

For the synthesis of compound (III), a solution of *N*-(3-*tert*-butyl-1-phenyl-1*H*-pyrazol-5-yl)-2-chloro-*N*-(4-methylbenzyl)acetamide

(0.49 mmol), prepared as for compounds (I) and (II), and potassium *O*-ethyl carbonodithioate (0.73 mmol) in acetonitrile (6 ml) was stirred at room temperature in the absence of light for 2.5 h. The solvent was removed under reduced pressure and the residue purified by column chromatography on silica gel (ethyl acetate/dichloromethane gradient) to obtain compound (III) in 94% yield. Yellow crystals suitable for single-crystal X-ray diffraction were grown by slow evaporation, at ambient temperature and in air, of a solution in ethanol (m.p. 359 K). MS (70 eV) *m*/*z* (%): 481 (2) [M^+], 319 (40), 163 (30), 105 (100); elemental analysis found: C 64.8, H 6.4, N 8.7%, $C_{26}H_{31}N_3O_2S_2$ requires: C 64.8, H 6.5, N 8.7%.

Compound (I)

Crystal data

$C_{23}H_{26}ClN_3O_2$
M_r = 411.92
Triclinic, $P\overline{1}$
a = 10.0761 (12) Å
b = 10.5208 (8) Å
c = 11.1868 (19) Å
α = 96.546 (7)°
β = 95.790 (9)°
γ = 116.035 (9)°
V = 1043.4 (2) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.21 mm^{-1}
T = 120 K
0.50 × 0.26 × 0.22 mm

Data collection

Bruker–Nonius KappaCCD diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2003)
T_{min} = 0.914, T_{max} = 0.956
27179 measured reflections
4801 independent reflections
2893 reflections with $I > 2\sigma(I)$
R_{int} = 0.068

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.055
$wR(F^2)$ = 0.154
S = 1.05
4801 reflections
266 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.37 e Å^{-3}
$\Delta\rho_{min}$ = −0.31 e Å^{-3}

Compound (II)

Crystal data

$C_{22}H_{23}Cl_2N_3O$
M_r = 416.33
Monoclinic, $P2_1/c$
a = 10.120 (2) Å
b = 10.7732 (18) Å
c = 19.2751 (14) Å
β = 95.662 (9)°
V = 2091.2 (6) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.33 mm^{-1}
T = 120 K
0.35 × 0.12 × 0.06 mm

Table 1
Selected torsion angles (Å) for compounds (I)–(IV).

	(I)	(II)	(III)	(IV)[a]
N2—N1—C11—C12	−140.8 (2)	−148.0 (2)	−136.1 (4)	−133.97 (14)
N2—C3—C31—C32	−173.3 (2)	−8.9 (3)	6.5 (5)	172.69 (13)
N1—C5—N51—C57	−102.5 (3)	−104.7 (2)	−108.5 (4)	−100.84 (15)
C5—N51—C57—C51	−85.6 (3)	−69.1 (2)	−87.5 (4)	−80.52 (15)
N51—C57—C51—C52	106.8 (3)	102.4 (2)	114.0 (4)	118.10 (14)
N1—C5—N51—C58	78.3 (3)	76.1 (3)	73.1 (4)	83.16 (17)
C5—N51—C58—O58	−177.9 (2)	−179.8 (2)	−173.4 (3)	176.99 (13)
N51—C58—C59—S59			169.8 (2)	
C58—C59—S59—C60			−75.7 (3)	
C59—S59—C60—O60			178.7 (2)	
S59—C60—O60—C61			−175.4 (3)	
C60—O60—C61—C62			−180.0 (3)	
C53—C54—O54—C541	−172.3 (2)			−177.94 (13)

Note: (*a*) data for compound (IV) are taken from Castillo *et al.* (2010).

Table 2
Hydrogen-bond parameters (Å, °) for compounds (I)–(III).

*Cg*1 represents the centroid of the C11–C16 ring and *Cg*2 represents the centroid of the C51–C56 ring.

Compound	*D*—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
(I)	C55—H55···O58^{i}	0.95	2.50	3.449 (3)	178
	C52—H52···*Cg*1^{ii}	0.95	2.70	3.562 (3)	152
(II)	C57—H57*B*···O58^{iii}	0.99	2.43	3.307 (3)	148
	C16—H16···*Cg*2^{iv}	0.95	2.91	3.526 (2)	124
	C56—H56···*Cg*1^{v}	0.95	2.77	3.514 (2)	136
(III)	C13—H13···O58^{vi}	0.95	2.39	3.244 (5)	149
	C55—H55···O58^{ii}	0.95	2.39	3.277 (5)	155
	C52—H52···*Cg*1^{vii}	0.95	2.91	3.632 (5)	133
	C61—H61*A*···*Cg*2^{ii}	0.99	2.82	3.610 (5)	137

Symmetry codes: (i) $2-x, 2-y, 1-z$; (ii) $1-x, 1-y, -z$; (iii) $1-x, \frac{1}{2}+y, \frac{1}{2}-z$; (iv) $x, \frac{1}{2}-y, -\frac{1}{2}+z$; (v) $1-x, -\frac{1}{2}+y, \frac{1}{2}-z$; (vi) $1-x, 1-y, 1-z$; (vii) $-x, 1-y, 1-z$.

Data collection

Bruker–Nonius KappaCCD diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2003)
T_{min} = 0.899, T_{max} = 0.981
33228 measured reflections
4792 independent reflections
3328 reflections with $I > 2\sigma(I)$
R_{int} = 0.063

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.049
$wR(F^2)$ = 0.116
S = 1.04
4792 reflections
256 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.70 e Å^{-3}
$\Delta\rho_{min}$ = −0.83 e Å^{-3}

Compound (III)

Crystal data

$C_{26}H_{31}N_3O_2S_2$
M_r = 481.68
Triclinic, $P\overline{1}$
a = 9.6847 (14) Å
b = 11.3127 (9) Å
c = 12.3425 (12) Å
α = 77.040 (8)°
β = 72.789 (9)°
γ = 84.977 (9)°
V = 1258.5 (2) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.24 mm^{-1}
T = 120 K
0.42 × 0.35 × 0.23 mm

Data collection

Bruker–Nonius KappaCCD diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2003)
T_{min} = 0.901, T_{max} = 0.947
24648 measured reflections
4471 independent reflections
2251 reflections with $I > 2\sigma(I)$
R_{int} = 0.148

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.056
$wR(F^2)$ = 0.172
S = 1.00
4471 reflections
303 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.43 e Å^{-3}
$\Delta\rho_{min}$ = −0.35 e Å^{-3}

All H atoms were located in difference maps and then treated as riding atoms in geometrically idealized positions, with C—H distances of 0.95 (aromatic and pyrazole), 0.98 (CH_3) or 0.99 Å (CH_2), and with U_{iso}(H) = kU_{eq}(C), where k = 1.5 for the methyl groups, which were permitted to rotate but not to tilt, and 1.2 for all other H atoms. The quality of the crystals for compound (III) was consistently rather low, as indicated by the high value of the merging index, 0.148, and the lower precision of the geometric parameters as compared with those for compounds (I) and (II). For each of (I)–

(III), the reference molecule was selected to have the same configuration at atom N51 and the same orientation of the aryl ring (C11—C16) as found in compound (IV) (Castillo *et al.*, 2010). The atom-labelling schemes for (I)–(III) are based on that employed in (IV).

For all compounds, data collection: *COLLECT* (Hooft, 1999); cell refinement: *DIRAX*/*LSQ* (Duisenberg *et al.*, 2000); data reduction: *EVALCCD* (Duisenberg *et al.*, 2003); program(s) used to solve structure: *SIR2004* (Burla *et al.*, 2005); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *PLATON* (Spek, 2009); software used to prepare material for publication: *SHELXL97* and *PLATON*.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: SK3367). Services for accessing these data are described at the back of the journal.

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