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1,3,5-Tris(4-chlorophenyl)-1,3,5-triazinane-2,4,6-trione acetone solvate

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Key indicators

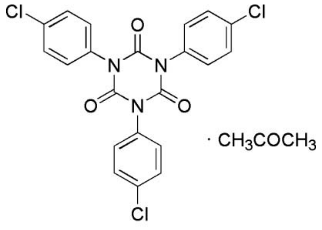
Single-crystal X-ray study
T = 292 K
Mean σ(C–C) = 0.006 Å
Disorder in solvent or counterion
R factor = 0.049
wR factor = 0.112
Data-to-parameter ratio = 11.3

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

In the title compound, $C_{21}H_{12}Cl_3N_3O_3 \cdot C_4H_9O$, the six-membered heterocyclic ring lies on a threefold rotation axis and adopts a planar conformation. The molecule exhibits a propeller-like shape. The acetone solvent molecule is disordered.

Comment

Trimerization of aromatic isocyanates has been known to improve the properties of polyurethanes or coating materials, for example, giving enhanced thermal resistance, flame retardation, chemical resistance, and film-forming characteristics. During the synthesis of tetrazolinones we obtained 1,3,5-tris-(4-chlorophenyl)-1,3,5-triazinane-2,4,6-trione. The title compound, (I), was synthesized by the reaction of 4-chlorophenylisocyanate with sodium azide in *N*,*N*-dimethyl formamide (Tochigi *et al.*, 1996).



(I)

The six-membered heterocyclic ring, lying on a threefold rotation axis, adopts a planar conformation. The dihedral angle between each benzene ring and the heterocyclic ring is 78.2 (1)°. The acetone molecule is disordered; atoms C9 and O2 occupy special position 6*a* of space group *R*3*c*, on a threefold axis, whereas C8 is disordered to simulate threefold symmetry. The crystal packing involves van der Waals interactions only.

Experimental

Anhydrous aluminium chloride (1.5 g) was added to *N*,*N*-dimethylformamide (50 ml) under ice and the resulting mixture was stirred for 15 minutes. Sodium azide (2.6 g) was added to the mixture, which was stirred for 15 more minutes. Then 4-chlorophenylisocyanate (6.0 g) in *N*,*N*-dimethmylformamide (8 ml) was added with continous stirring and heating at 348 K for 3 h and then at 363 K for a further 3 h. After cooling, the reaction mixture was added, with stirring, to a mixture of ice (200 g), water (200 g) and 40% sodium hydroxide (50 ml). The resulting precipitate was collected by suction filtration and recrystallized from acetone to obtain white crystals of 1,3,5-tris-(4-chlorophenyl)-1,3,5-triazinane-2,4,6-trione (yield: 36%), m.p. 588–590 K.

Crystal data

$C_{21}H_{12}Cl_3N_3O_3\cdot C_4H_9O$
$M_r = 533.80$
Trigonal, $R3c$
$a = 12.7911$ (9) Å
$c = 26.403$ (4) Å
$V = 3741.1$ (6) Å^3
$Z = 6$
$D_x = 1.422$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 2224 reflections
$\theta = 2.3$–22.5°
$\mu = 0.41$ mm^{-1}
$T = 292$ (2) K
Block, colourless
0.30 × 0.20 × 0.10 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: none
4820 measured reflections
1261 independent reflections
966 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.062$
$\theta_{\rm max} = 27.0°$
$h = -15 \rightarrow 16$
$k = -16 \rightarrow 16$
$l = -19 \rightarrow 33$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.049$
$wR(F^2) = 0.112$
$S = 0.95$
1261 reflections
112 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0512P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} < 0.001$
$\Delta\rho_{\rm max} = 0.16$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.16$ e Å^{-3}
Absolute structure: Flack (1983), 345 Friedel pairs
Flack parameter: −0.08 (11)

All H atoms were initially located in a difference Fourier map. The aromatic H atoms were then constrained to an ideal geometry, with C—H distances of 0.93 Å and $U_{\rm iso}({\rm H}) = 1.2U_{\rm eq}({\rm C})$. Other H atoms were placed in ideal positions, with C—H distances of 0.96 Å and $U_{\rm iso}({\rm H}) = 1.5U_{\rm eq}({\rm C})$.

Data collection: *SMART* (Bruker, 2003); cell refinement: *SAINT* (Bruker, 2003); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 2001); software used to prepare material for publication: *SHELXTL*.

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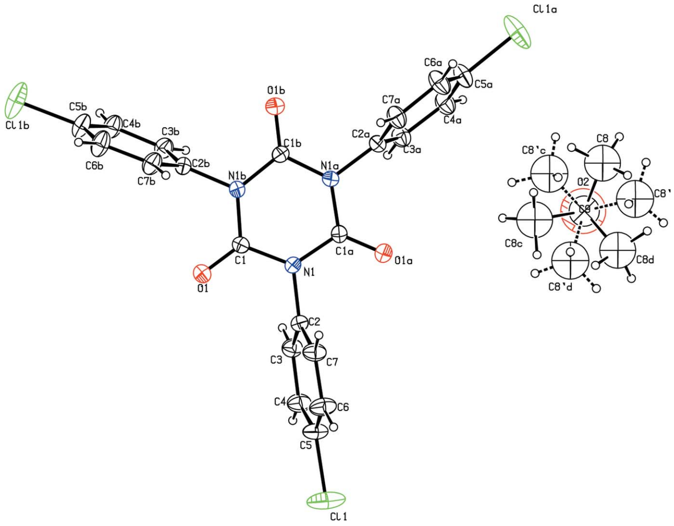

Figure 1
View of the molecule of (I). showing the atom labelling. with displacement ellipsoids drawn at the 50% probability level. The acetone solvent molecule is disordered.

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