organic compounds

1,3,5-Tris(4-methoxyphenyl)-1,3,5-triazinane-2,4,6-trione

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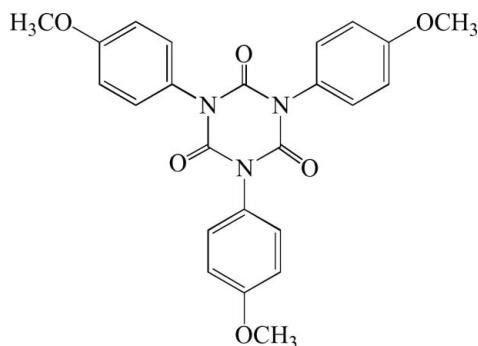Correspondence e-mail: fangli@sxu.edu.cn

The complete molecule of the title compound, $C_{24}H_{21}N_3O_6$, is generated by the application of threefold rotation symmetry about an axis perpendicular to the central ring. The molecule exhibits a propeller-like shape. The dihedral angle between each benzene ring and the heterocyclic ring is 74.0 (1)°. The molecules pack with no specific intermolecular interactions between them. The *SQUEEZE* procedure in *PLATON* [Spek (2009). *Acta Cryst.* D**65**, 148–155] was used to model disordered solvent molecules, presumed to be acetone; the calculated unit-cell data do not take into account the presence of these.

Related literature

For general background to trimerization of aromatic iso-cyanates, see: Raders & Verkade (2010); Duong *et al.* (2004); Tang *et al.* (1994); Zhitinkina *et al.* (1985); Nawata *et al.* (1975); Nicholas & Gmitter (1965).

Experimental

Crystal data

$C_{24}H_{21}N_3O_6$	$Z = 6$
$M_r = 447.44$	Mo $K\alpha$ radiation
Trigonal, $R3c$	$\mu = 0.08$ mm^{-1}
$a = 13.2008$ (14) Å	$T = 200$ K
$c = 26.695$ (3) Å	$0.51 \times 0.49 \times 0.04$ mm
$V = 4028.7$ (8) Å^3	

Data collection

Bruker SMART CCD area-detector diffractometer	6995 measured reflections
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)	1577 independent reflections
$T_{min} = 0.960$, $T_{max} = 0.997$	1142 reflections with $I > 2\sigma(I)$
	$R_{int} = 0.031$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.044$	1 restraint
$wR(F^2) = 0.122$	H-atom parameters constrained
$S = 1.05$	$\Delta\rho_{max} = 0.16$ e Å^{-3}
1577 reflections	$\Delta\rho_{min} = -0.12$ e Å^{-3}
101 parameters	

Data collection: *SMART* (Bruker, 2007); cell refinement: *SAINT* (Bruker, 2007); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL* and *PLATON* (Spek, 2009).

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supporting information

1,3,5-Tris(4-methoxyphenyl)-1,3,5-triazinane-2,4,6-trione

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S1. Experimental

S1.1. Synthesis and crystallization

To a stirred solution of lithium dibenzylamide (0.04 g) in diethyl ether was added 4-methoxyphenyl isothiocyanate (29.8 g). After stirring for 2 min, the mixture afforded a white precipitate. The resulting precipitate was collected by suction filtration and recrystallized from acetone to obtain white crystals of 1,3,5-tris-(4-methoxyphenyl)-1,3,5-triazinane-2,4,6-trione (yield: 90%), m.p. 530–531 K.
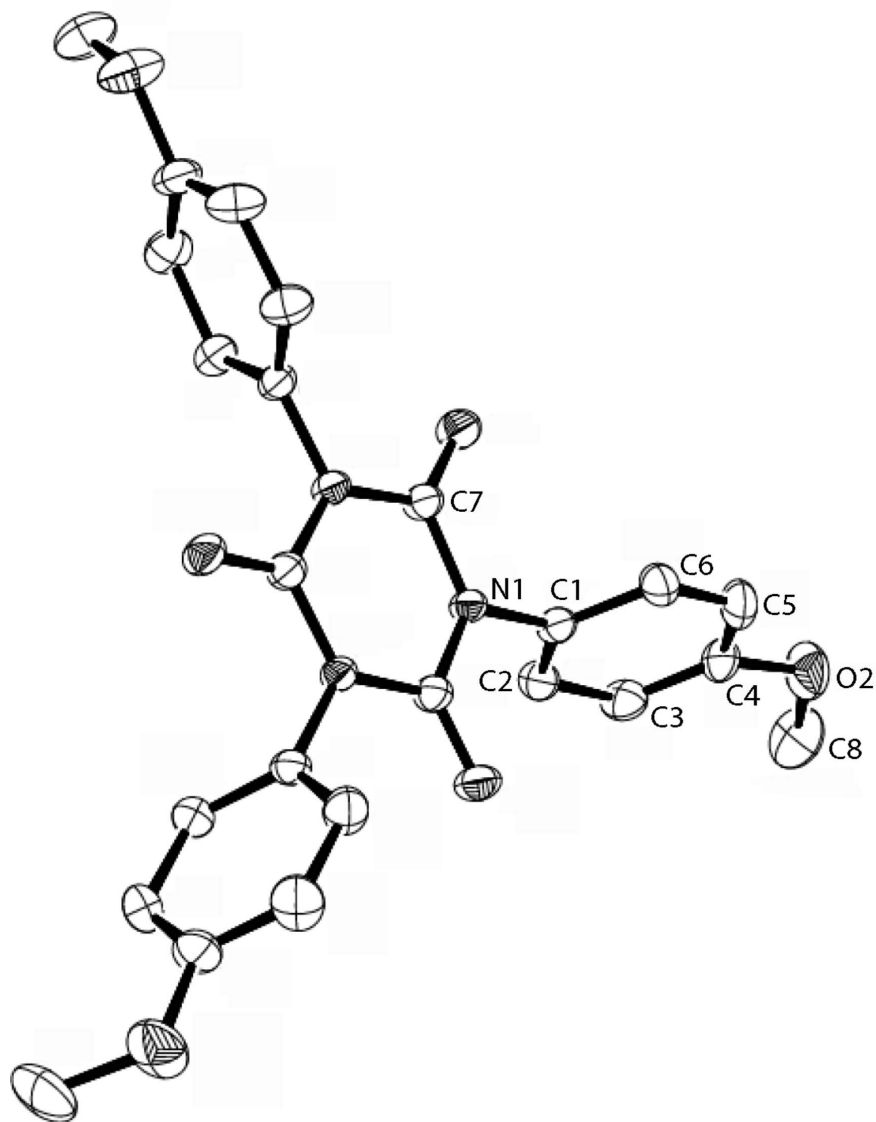
S1.2. Refinement

The H atoms were placed in their idealised positions with C—H = 0.95–0.98 Å, and refined as riding with $U_{iso}(H) = 1.2$–$1.5U_{eq}$. The structure contains solvent accessible voids of 153 A^3. The *SQUEEZE* procedure in *PLATON* (Spek, 2009) was used to model the disordered solvent molecules, presumed to be 4–6 acetone molecules per unit cell.

S2. Results and discussion

Trimerization of aromatic isocyanates, manufactured by cyclotrimerizing corresponding isocyanates, has been known to enhance the properties of polyurethanes or coating materials (Raders & Verkade, 2010; Duong *et al.*, 2004; Tang *et al.*, 1994; Zhitinkina *et al.*, 1985; Nawata *et al.*, 1975; Nicholas & Gmitter, 1965). Polymers, such as polyurethanes incorporated with isocyanurates, have enhanced thermal resistance, flame retardation, chemical resistance and film-forming characteristics. Isocyanurates are also used in the synthesis of co-polymer resins to improve their water-resistance, transparency and impact resistance. During research on the properties of lithium dibenzylamide, a simple and efficient catalyst for isocyanate cyclotrimerization to isocyanurate, we obtained crystals of 1,3,5-tris-(4-methoxy-phenyl)-1,3,5-triazinane-2,4,6-trione.

In the title compound, $C_{24}H_{21}N_3O_6$, the six-membered heterocyclic ring lies on a threefold rotation axis and adopts a planar conformation. The molecule exhibits a propeller-like shape. The dihedral angle between each benzene ring and the heterocyclic ring is 74.0 (1).

Figure 1
Molecular structure of I showing 30% probability displacement ellipsoids. The hydrogen atoms are omitted for clarity.

1,3,5-Tris(4-methoxyphenyl)-1,3,5-triazinane-2,4,6-trione

Crystal data

$C_{24}H_{21}N_3O_6$	$D_x = 1.107$ Mg m^{-3}
$M_r = 447.44$	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Trigonal, $R3c$	Cell parameters from 1247 reflections
Hall symbol: R 3 -2"c	$\theta = 2.3$–$20.4°$
$a = 13.2008\ (14)$ Å	$\mu = 0.08$ mm^{-1}
$c = 26.695\ (3)$ Å	$T = 200$ K
$V = 4028.7\ (8)$ Å^3	Block, colourless
$Z = 6$	$0.51 \times 0.49 \times 0.04$ mm
$F(000) = 1404$	

Data collection

Bruker SMART CCD area-detector diffractometer	6995 measured reflections
Radiation source: fine-focus sealed tube	1577 independent reflections
Graphite monochromator	1142 reflections with $I > 2\sigma(I)$
phi and ω scans	$R_{int} = 0.031$
Absorption correction: multi-scan ($SADABS$; Sheldrick, 1996)	$\theta_{max} = 25.0°, \theta_{min} = 2.4°$
$T_{min} = 0.960, T_{max} = 0.997$	$h = -9\rightarrow15$
	$k = -15\rightarrow14$
	$l = -31\rightarrow31$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	
$R[F^2 > 2\sigma(F^2)] = 0.044$	Hydrogen site location: inferred from neighbouring sites
$wR(F^2) = 0.122$	
$S = 1.05$	H-atom parameters constrained
1577 reflections	$w = 1/[\sigma^2(F_o^2) + (0.0687P)^2]$
101 parameters	where $P = (F_o^2 + 2F_c^2)/3$
1 restraint	$(\Delta/\sigma)_{max} = 0.001$
Primary atom site location: structure-invariant direct methods	$\Delta\rho_{max} = 0.16$ e Å^{-3}
	$\Delta\rho_{min} = -0.12$ e Å^{-3}

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R-factor wR and goodness of fit S are based on F^2, conventional R-factors R are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating R-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R-factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	y	z	U_{iso}*/U_{eq}
N1	0.24317 (15)	0.68853 (16)	0.06336 (8)	0.0507 (6)
O1	0.37628 (17)	0.88360 (16)	0.06161 (9)	0.0710 (6)
O2	−0.1275 (2)	0.7774 (3)	0.06263 (12)	0.1146 (9)
C1	0.1470 (2)	0.7108 (2)	0.06541 (11)	0.0552 (7)
C2	0.0864 (2)	0.6942 (2)	0.10907 (12)	0.0652 (7)
H2	0.1090	0.6709	0.1387	0.078*
C3	−0.0104 (3)	0.7124 (3)	0.10944 (13)	0.0768 (9)
H3	−0.0565	0.6970	0.1388	0.092*
C4	−0.0369 (2)	0.7525 (3)	0.06691 (14)	0.0724 (8)
C5	0.0253 (3)	0.7708 (3)	0.02514 (15)	0.0878 (10)
H5	0.0050	0.7983	−0.0039	0.105*
C6	0.1168 (3)	0.7514 (3)	0.02308 (12)	0.0729 (8)
H6	0.1600	0.7654	−0.0071	0.088*
C7	0.3566 (2)	0.7838 (2)	0.06225 (11)	0.0566 (7)
C8	−0.2060 (4)	0.7446 (5)	0.1028 (2)	0.1312 (17)
H8A	−0.2414	0.6604	0.1087	0.197*

H8B	−0.2674	0.7631	0.0948	0.197*
H8C	−0.1641	0.7874	0.1329	0.197*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0390 (12)	0.0406 (12)	0.0734 (15)	0.0206 (10)	0.0029 (11)	0.0033 (11)
O1	0.0571 (12)	0.0488 (12)	0.1124 (15)	0.0306 (10)	−0.0006 (11)	−0.0036 (10)
O2	0.0943 (19)	0.158 (3)	0.127 (2)	0.0896 (19)	0.0056 (17)	0.0234 (18)
C1	0.0452 (16)	0.0456 (15)	0.0770 (18)	0.0242 (14)	0.0011 (15)	−0.0026 (14)
C2	0.0608 (17)	0.0636 (18)	0.0762 (19)	0.0350 (14)	0.0076 (14)	0.0051 (15)
C3	0.0591 (18)	0.080 (2)	0.089 (2)	0.0331 (16)	0.0207 (15)	0.0075 (17)
C4	0.0538 (16)	0.078 (2)	0.098 (2)	0.0429 (16)	−0.0027 (18)	0.0036 (17)
C5	0.071 (2)	0.111 (3)	0.093 (3)	0.054 (2)	−0.008 (2)	0.0187 (19)
C6	0.0654 (19)	0.079 (2)	0.081 (2)	0.0410 (16)	0.0038 (16)	0.0179 (16)
C7	0.0465 (16)	0.0472 (16)	0.0751 (19)	0.0227 (11)	0.0012 (14)	−0.0021 (14)
C8	0.085 (3)	0.174 (4)	0.166 (4)	0.088 (3)	0.016 (3)	−0.011 (4)

Geometric parameters (Å, º)

N1—C7[i]	1.384 (3)	C3—C4	1.370 (5)
N1—C7	1.393 (3)	C3—H3	0.9500
N1—C1	1.440 (3)	C4—C5	1.333 (4)
O1—C7	1.209 (3)	C5—C6	1.356 (4)
O2—C4	1.395 (4)	C5—H5	0.9500
O2—C8	1.400 (5)	C6—H6	0.9500
C1—C2	1.368 (4)	C7—N1[ii]	1.384 (3)
C1—C6	1.392 (4)	C8—H8A	0.9800
C2—C3	1.413 (4)	C8—H8B	0.9800
C2—H2	0.9500	C8—H8C	0.9800
C7[i]—N1—C7	124.2 (3)	C4—C5—C6	121.7 (3)
C7[i]—N1—C1	117.35 (19)	C4—C5—H5	119.2
C7—N1—C1	118.43 (19)	C6—C5—H5	119.2
C4—O2—C8	117.0 (3)	C5—C6—C1	119.6 (3)
C2—C1—C6	119.7 (2)	C5—C6—H6	120.2
C2—C1—N1	120.3 (3)	C1—C6—H6	120.2
C6—C1—N1	120.0 (2)	O1—C7—N1[ii]	122.2 (2)
C1—C2—C3	119.1 (3)	O1—C7—N1	122.1 (2)
C1—C2—H2	120.5	N1[ii]—C7—N1	115.7 (3)
C3—C2—H2	120.5	O2—C8—H8A	109.5
C4—C3—C2	119.1 (3)	O2—C8—H8B	109.5
C4—C3—H3	120.4	H8A—C8—H8B	109.5
C2—C3—H3	120.4	O2—C8—H8C	109.5
C5—C4—C3	120.7 (3)	H8A—C8—H8C	109.5

| C5—C4—O2 | 114.3 (3) | H8B—C8—H8C | 109.5 |
| C3—C4—O2 | 125.0 (3) | | |

Symmetry codes: (i) $-y+1$, $x-y+1$, z; (ii) $-x+y$, $-x+1$, z.