2,4,6-Tris(2,4,6-tribromophenoxy)-1,3,5-triazine

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Key indicators

Single-crystal X-ray study
$T = 293$ K
Mean σ(C–C) = 0.010 Å
R factor = 0.044
wR factor = 0.105
Data-to-parameter ratio = 16.4

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

In the title compound, $C_{21}H_6Br_9N_3O_3$, the triazine ring is almost perpendicular to the three benzene rings. In the crystal structure, molecules are linked into a three-dimensional framework by C—H···O interactions.

Comment

Triazine derivatives are known to be versatile and selective coupling reagents for amide synthesis (Masala & Taddei, 1999) and high-energy explosives with numerous military applications (Williams *et al.*, 2005). Recently, we have prepared an excellent flame-retardant additive to synthetic resins with cyanuric chloride as the skeleton. Here we report the synthesis and crystal structure of the title compound, (I).

(I)
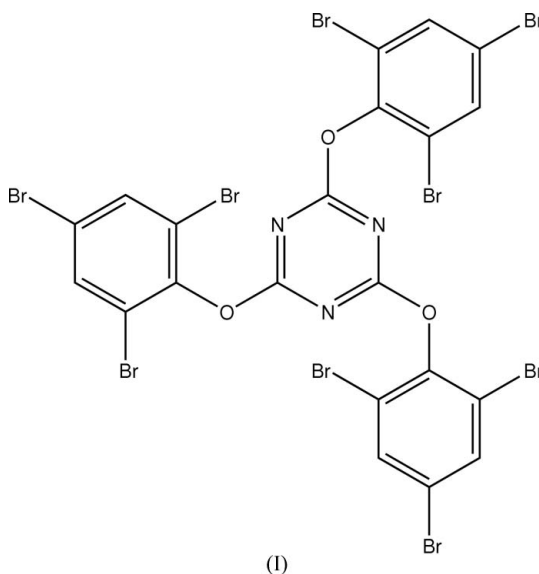
The bond lengths and angles in (I) are within normal ranges (Allen *et al.*, 1987). The triazine ring is almost perpendicular to the three benzene rings, the triazine ring making dihedral angles of 81.7 (3)°, 85.7 (3)° and 81.2 (3)° with the C4–C9, C10–C15 and C16–C21 benzene rings, respectively. The three benzene rings make dihedral angles of 53.7 (3)° (rings C4–C9 and C10–C15), 64.8 (3)° (C4–C9 and C16–C21) and 61.7 (3)° (C10–C15 and C16–C21) with each other. In the crystal structure, molecules are linked into a three-dimensional framework (Fig. 2) by a C18—H18A···O3[i] interaction (symmetry code as in Table 21).

Experimental

Sodium hydroxide (34.2 g, 0.86 mol) and sodium sulfite (0.14 g) were dissolved in water (96 ml) in a 500 ml-capacity reactor. To the solu-

tion dichloromethane (130 g, 1.53 mol) was added 2,4,6-tribromo-phenol (272 g, 0.82 mol). After cooling to room temperature, methylene chloride (150 g, 1.76 mol) was further added and then cyanuric chloride (50 g, 0.27 mol) was added at 273–280 K. The resulting mixture was refluxed for 3 h. The solvent was evaporated at atmospheric pressure and the precipitate was filtered. The resulting white powder was washed with methanol and then filtered. Colourless single crystals suitable for X-ray diffraction were obtained by slow evaporation of an ethyl acetate solution over a period of 3 d.

Crystal data

$C_{21}H_6Br_9N_3O_3$
$M_r = 1067.48$
Trigonal, $R\bar{3}$
$a = 42.524 (3)$ Å
$c = 8.4270 (13)$ Å
$V = 13197 (2)$ Å^3
$Z = 18$

$D_x = 2.418$ Mg m^{-3}
Mo $K\alpha$ radiation
$\mu = 12.33$ mm^{-1}
$T = 293 (2)$ K
Column, colourless
$0.25 \times 0.19 \times 0.10$ mm

Data collection

Siemens SMART 1000 CCD area
    detector diffractometer
ω scans
Absorption correction: multi-scan
    (*SADABS*; Sheldrick, 1996)
    $T_{min} = 0.065$, $T_{max} = 0.291$

23460 measured reflections
5342 independent reflections
3182 reflections with $I > 2\sigma(I)$
$R_{int} = 0.055$
$\theta_{max} = 25.3°$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.044$
$wR(F^2) = 0.105$
$S = 1.03$
5342 reflections
325 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0486P)^2$
    $+ 1.6465P]$
    where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.002$
$\Delta\rho_{max} = 0.79$ e Å^{-3}
$\Delta\rho_{min} = -0.51$ e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

$D—H\cdots A$	$D—H$	$H\cdots A$	$D\cdots A$	$D—H\cdots A$
C18—H18A$\cdots$O3^i	0.93	2.46	3.357 (9)	163

Symmetry code: (i) $-x + y - \frac{1}{3}, -x + \frac{4}{3}, z + \frac{1}{3}$.

All H atoms were located in a difference Fourier map and constrained to ride on their parent atoms, with C—H = 0.93 Å, and with $U_{iso}(H) = 1.2U_{eq}(C)$.

Data collection: *SMART* (Siemens, 1996); cell refinement: *SAINT* (Siemens, 1996); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 1997); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*, *PARST* (Nardelli, 1995) and *PLATON* (Spek, 2003).
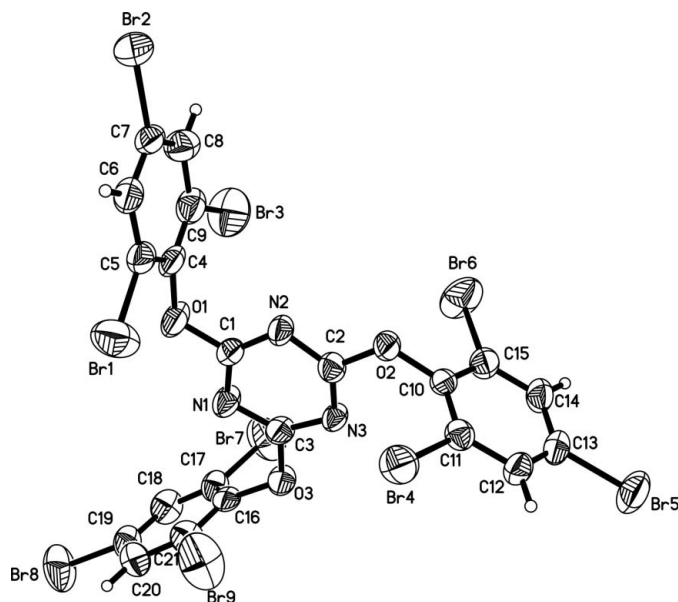
Figure 1
The molecular structure of (I), showing 50% probability displacement ellipsoids and the atom-numbering scheme.
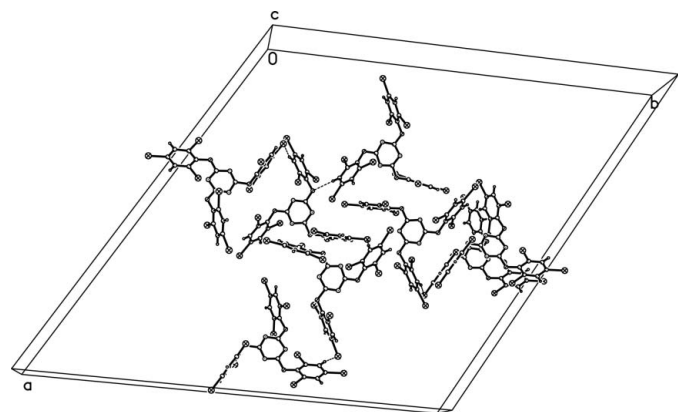

Figure 2
The packing, viewd down the *c* axis. C—H$\cdots$O hydrogen bonds are indicated by dashed lines.

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