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A phthaloyl-linked benzotriazole corrosion inhibitor: 1,1′-(phthaloyl)bis(benzotriazole)

Graham Smith,* Steven E. Bottle, Damien A. Reid, D. Paul Schweinsberg and Raymond C. Bott

Centre for Instrumental and Developmental Chemistry, Queensland University of Technology, GPO Box 2434, Brisbane 4001, Australia

Correspondence e-mail: g.smith@qut.edu.au

Key indicators

Single-crystal X-ray study
T = 295 K
Mean σ(C–C) = 0.007 Å
R factor = 0.037
wR factor = 0.138
Data-to-parameter ratio = 8.8

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

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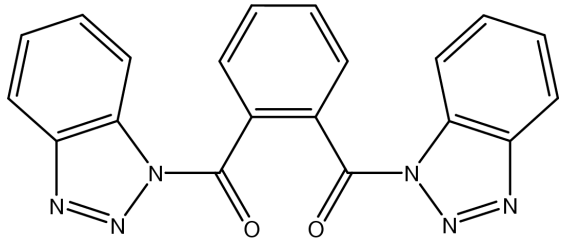
The crystal structure of the phthaloyl-linked benzotriazole 1,1′-(phthaloyl)bis(benzotriazole), $C_{20}H_{12}N_6O_2$, having potential as a corrosion inhibitor for copper, confirms that the *ortho*-related carboxamide substituents in the benzene parent ring adopt a *syn–syn* conformation with respect to one another.

Comment

Benzotriazole (BTAH) has long been used and remains one of the most effective inhibitors of copper corrosion in neutral and alkaline conditions (Cotton, 1963; Cotton & Scholes, 1967; Poling, 1970; Mansfeld *et al.*, 1971). Since the mechanism of its inhibition effect is largely attributed to the formation of a polymeric BTA–Cu–BTA–Cu film (Rubim *et al.*, 1993; Ashour *et al.*, 1996), the BTAH derivatives, therefore, may offer higher inhibition efficiencies than the parent molecule, with the extended molecule giving better interaction with the metal surface. With this goal in mind and in an attempt to further understand the mechanism of corrosion inhibition of the carboxybenzotriazoles, a new dicarboxamide, 1,1′-(phthaloyl)bis(benzotriazole), (I), was synthesized (Reid, 1996) and its structure determined using single-crystal X-ray methods.



(I)

The analysis shows (Fig. 1) that the *ortho*-substituted carboxybenzotriazole side chains adopt a *syn–syn* conformation with respect to one another [torsion angles: C1—C2—C21—O21 = −127.0 (5)° and C6—C1—C11—O11 = −135.2 (5)°]. These chains are slightly distorted from planarity [torsion angles: C1—C11—N11—N12 = 16.3 (6)° and C2—C21—N21—N22 = −9.5 (6)°]. There are no intramolecular associations between the chains and, in addition, the packing in the unit cell shows no significant intermolecular interactions.

Experimental

The title compound was synthesized by the slow addition of 20 ml of a solution of phthaloyl chloride (4.3 ml, 60 mmol) in tetrahydrofuran to

100 ml of a solution containing benzotriazole (7.14 g, 60 mmol) and triethylamine (8.4 ml, 60 mmol) (also in tetrahydrofuran), maintained at 273–278 K. After stirring for *ca* 12 h, the mixture was poured on to ice and the resulting precipitate washed with water and recrystallized from chloroform from which data-quality crystals were obtained.

Crystal data

$C_{20}H_{12}N_6O_2$
M_r = 368.36
Orthorhombic, $P2_12_12_1$
a = 10.744 (5) Å
b = 22.193 (4) Å
c = 7.079 (5) Å
V = 1687.9 (14) Å^3
Z = 4
D_x = 1.450 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 25 reflections
θ = 8.4–14.7°
μ = 0.10 mm^{-1}
T = 295 (2) K
Plate, colourless
0.38 × 0.25 × 0.20 mm

Data collection

Rigaku AFC-7*R* diffractometer
ω–2θ scans
2231 measured reflections
2231 independent reflections
992 reflections with $I > 2\sigma(I)$'
θ_{max} = 27.5°
$h = 0 \rightarrow 13$
$k = 0 \rightarrow 28$
$l = 0 \rightarrow 9$
3 standard reflections
every 150 reflections
intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.037
$wR(F^2)$ = 0.138
S = 0.89
2231 reflections
254 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0638P)^2 + 0.2455P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.18 e Å^{-3}
$\Delta\rho_{min}$ = −0.18 e Å^{-3}
Extinction correction: *SHELXL*97 (Sheldrick, 1997)
Extinction coefficient: 0.014 (2)

The positional and isotropic displacement parameters of the H atoms were fixed in the refinement.

Data collection: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1999*a*); cell refinement: *MSC/AFC Diffractometer Control Software*; data reduction: *TEXSAN for Windows* (Molecular Structure Corporation, 1999*b*); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *PLATON for Windows* (Spek, 1999); software used to prepare material for publication: *TEXSAN for Windows*.

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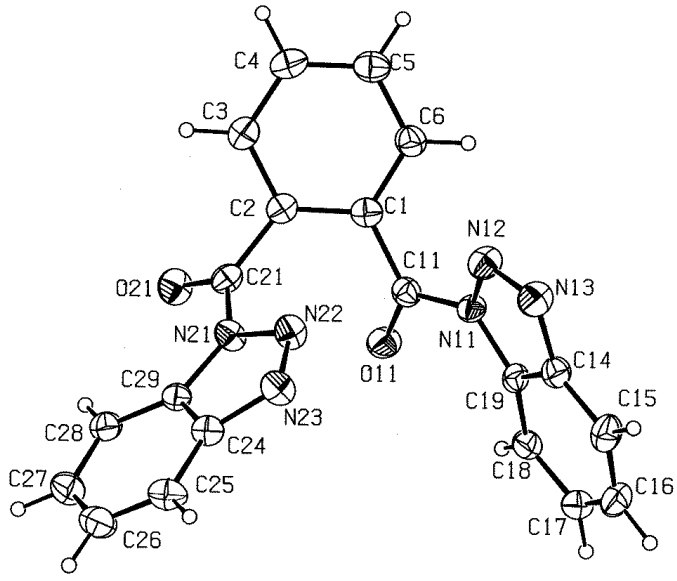

Figure 1
The molecular configuration and atom-numbering scheme for (I). Displacement ellipsoids are shown as the 30% probability level.

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