Structure of 2,2'-bis(3-allyl-4-cyanatophenyl)isopropylidene

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The structure of the title compound (I) was determined by direct methods using Mo $K\alpha$ diffractometer data, and refined by full-matrix least squares to R = 0.066 for 1536 reflections ($I \ge 3\sigma$ (I)). The structure shows a central tetrahedral carbon atom surrounded by two methyl and two 3-allyl-4-cyanatophenyl groups. The geometry of the cyanato group in this molecule compares well with those in 2,2'-bis(4-cyanatophenyl)isopropylidene **II**) and 4-chloro-3,5-dimethylphenylcyanate (**III**), the only other examples of organic compounds bearing the cyanato moiety in the Cambridge Crystallographic Database (V.3).

Introduction

Cyanate esters (as compounds containing the $-O-C \equiv N$ group are commonly called) may undergo a cyclotrimerization reaction to form, in the case of aromatic compounds, an aryloxy-s-triazine ring structure. This ring formation can occur either thermally or in the presence of transition metal catalysts such as carboxylates of copper, cobalt, or zinc and an active hydrogen donor such as an involatile phenol. The heating of a dior polyfunctional cyanate ester will lead to the formation of a cross-linked, polycyanurate network, and quantitative aspects of this conversion have already been studied (Barton et al., 1991a; Hamerton, 1991a). The combination of the high glass transition temperature (T_{a}) , favorable toughness, low moisture absorption, and low overall cure shrinkage claimed for cyanate ester resins make them of increasing technological importance. Although references concerning aryl cyanate esters date back to the early 1960s (Grigat and Pütter, 1963; Martin, 1964; Jenson and Holm, 1964), it is only in recent years that a satisfactory synthetic procedure has existed to produce materials of sufficient purity for such a structural determination. The improved synthetic procedure

has led to the development of a number of new commercial products (Shimp, 1987) and work continues in this area to find materials with improved physical properties (Barton *et al.*, 1991b; Hamerton, 1991b).

It has already been noted in the literature (Baker and Shulgin, 1958) that analogous o-allylphenols may form intramolecular hydrogen-bonds between the π -system of the allyl group, and the hydroxyl group. Owing to the close proximity of the allyl and cyanato groups in this molecule, it was uncertain whether the possibility of a similar dipole-dipole interaction might exist, perhaps reducing the reactivity of the cyanato moiety. If this were indeed the case then the cyclotrimerization of the monomer to form an s-triazine ring might be adversely affected, thus hindering the formation of a network structure. For this reason a knowledge of the crystal structure of this substituted cyanate ester (in particular the orientation of the allyl substituents) may be of value in aiding clarification of the reaction mechanism, and discerning the effect of substitution on the molecule's subsequent interaction with potential comonomers.

The title compound (I) was synthesized in a pure state, and the structural determination carried out, prior to the independent publication of its preparation in a recent patent (Nishikawa, 1989). The ultimate aim of this work is to copolymerize the title compound with other reactive monomers (e.g., *bis*-maleimides) hopefully resulting in a toughened matrix resin suitable for

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structural composite applications. Aspects of its polymerization behavior, thermal stability, and dynamic mechanical properties are discussed elsewhere (Barton *et al.*, 1991b; Hamerton, 1991a) and work continues to evaluate the ensuing copolymers.

Experimental

All reagents and solvents were reagent grade quality, purchased commercially, and used without further purification unless otherwise noted. Infra-red spectra were recorded with a Perkin-Elmer 1750 Fourier transform infra-red (FT-IR) spectrometer interfaced with a Perkin-Elmer 7300 computer; the samples were presented as molten thin films between KBr plates. The ¹Hand ¹⁵N NMR spectra of (I) were recorded at 298 K using a Bruker AC300 pulse Fourier transform nuclear magnetic resonance (NMR) spectrometer operating at 300 MHz and 30.4 MHz for ¹⁵N. The ¹H NMR spectrum was recorded in D₆-acetone while the sample for ¹⁵N NMR was dissolved in acetone prior to adding chromium(III) acetylacetonate (at an approximate concentration of 15 mg/0.02 mole nitrogen) so as to eliminate nuclear Overhauser effects and reduce relaxation times. The ¹H NMR chemical shifts (δ) are given in parts per million (ppm) and are reported relative to the tetramethylsilane (TMS) internal standard, and the ¹⁵N NMR chemical shifts relative to external nitromethane. Preparative HPLC was carried out using a Waters preparative HPLC apparatus (Model Prep LC 500) equipped with a differential refractometer detector. Analytical HPLC was carried out using a Waters system comprising a pump (Model 510), injector (Model U6K), a differential refractometer (Model R401) and LC spectrophotometer (Model Lambda max 481), coupled to an LDC (Model CI-10) computing integrator. The melting point was determined using a Du Pont (Model 910) differential scanning calorimeter (heating rate 1°C/ minute) interfaced with a Du Pont (Model 9900) computer/thermal analyzer.

2,2'-bis(3-allyl-4-cyanatophenyl)isopropylidene

To prepare 2,2'-bis(3-allyl-4-cyanatophenyl)isopropylidene, a three-neck, 1-liter round-bottom flask equipped with a dropping funnel, overhead stirrer, and thermometer was charged with 2,2'-bis(3-allyl-4hydroxyphenyl)isopropylidene (50 g, 162 mmol), cyanogen bromide (47.67 g, 450 mmol) and freshly distilled acetone (200 cm³). The flask was cooled to -30° C and freshly distilled triethylamine (45.54 g, 450 mmol) was added dropwise over 35 minutes to the vigorously stirred mixture while the temperature was maintained at -25to -30° C by immersion in a dewar flask containing an acetone/liquid nitrogen mixture. The reaction mixture was stirred for a further 1.5 hr while warming to room temperature, and the product isolated by slowly pouring the reaction mixture into ice water (1 liter). After extraction into dichloromethane $(3 \times 50 \text{ cm}^3)$ and concentration on the rotary evaporator, the crude product resembled a mobile amber liquid. Purification by means of preparative HPLC (80/20 dichloromethane/hexane, normal phase silica column) yielded 40.5 g (69.7%) of the desired product, as a clear viscous oil (98% pure). After cooling in the refrigerator and scratching with a glass rod, the product rapidly solidified as a waxy, white crystalline solid; m.p. 48–49°C; FT-IR ν (thin film) 3081 (w), 3063 (w), 3008 (w), 2977 (m), 2936 (m), 2877 (w), 2272 (s), 2211 (m), 1640 (w), 1611 (m), 1594 (m), 1493 (vs), 1453 (m), 1203 (s), 1172 (s), 1135 (vs), 994 (m), 919 (m), and 749 (m) cm⁻¹; ¹H NMR (300 MHz, D₆-acetone, ppm from TMS) 7.38-7.33 (2H, d, J 8.7 Hz, 2 × Ar-H) 7.20-7.13 (2H, d of d, J 6.7 Hz, 2 × Ar-H) 7.12-7.07 (2H, d, J 2.4 Hz, 2 × Ar-H) 5.96-5.80 (2H, c.m., $2 \times -CH=$) 5.14-4.95 (4H, c.m., 2 $\times = CH_2$ 3.40-3.33 (4H, d, J 6.3 Hz, 2 $\times - CH_2$ -) 1.65 (6H, s, $2 \times CH_3$); ¹⁵H NMR (30.4 MHz, acetone, ppm from MeNO₂) -214.72; microanalyses are given in Table 1.

Cell determination and data collection on a crystal of approximate dimensions (0.45, 0.4, 0.025 mm) were carried out on an Enraf Nonius CAD4 four-circle diffractometer using graphite-monochromated Mo K α radiation ($\lambda = 0.71069$ Å). A set of 25 reflections were collected over the angular range 12–14° to determine the unit cell. For the data collection an $\omega - 2\theta$ scan over the range 1° $\leq \theta \leq 25^{\circ}$ was used (maximum scan speed *ca.* 3.3°/minute, a maximum of 90 s for weak reflections) covering the index range 0 $\leq h \leq 11$, 0 $\leq k \leq$ 13, -20 $\leq l \leq 20$. The total time for the data collec-

Table 1. Analytical data (calculated values in parentheses)

Compound	% C	% H	% N
2,2'-bis (3-allyl-4- cyanatophenyl) isopropylidene	77.1 (77.1)	6.0 (6.2)	7.8 (7.6)

tion was 69.2 hr. One reflection (200) was monitored every hour of exposed X-ray time and showed no systematic variation. Mechanical stability of the crystal on the diffractometer was checked by measuring four orientation control reflections every 200 data.

A total of 3933 reflections was collected and, after the removal of systematic absences, it was reduced to 3508 of which 1536 (39%) had $I \ge 3\sigma$ (I). The crystallographic data for compound are given in Table 2.

Structure determination and refinement

The structure was determined by the direct methods program MULTAN (Main *et al.*, 1978). The calculated *E*-map revealed 23 atoms, and a further Fourier synthesis positioned the remaining two nitrogen atoms and a further two carbon atoms. Isotropic refinement of the structure converged at R = 0.141 with hydrogen atoms at calculated positions ($d_{\rm H} = 1.0$ Å). The highest peak in a Fourier difference map was <0.3 e Å⁻³. The pseudo-empirical absorption correction DIFABS (Walker and Stuart, 1983) was applied and a subsequent 4 cycles of anisotropic full matrix least squares resulted in convergence of the *R* factor at R = 0.066, $R_w = 0.083$ incorporating a weighting scheme devised by Killean and Lawrence (1969): $w^{-1} = [\sigma(F)]^2 + [PF]^2 + Q$ with P = 0.04 and Q = 4.0.

All calculations were carried out using the Structure Determination Package (SDP) Plus V1.1a (Frenz, 1983) on a DEC PDP 11/73 computer.

Table 2. Crystal data

Compound (I)							
Molecular formula	C ₂₃ H ₂₂ N ₂ O ₂						
M_r	358.44						
Crystal system	Monoclinic						
Space group	$P2_1/c$						
a	9.921 (4)						
b	11.432 (2)						
С	17.613 (2)						
β	90.73 (4)°						
V	1997.6 $Å^3$ (4)						
Z	4						
D_{\star}	1.192 g cm^{-3}						
F (000)	760						
$\mu(Mo K\alpha)$	0.72 cm^{-1}						

Discussion

The salient features of the FT-IR, ¹H- and ¹⁵N NMR spectra are given in the experimental section and their significance is discussed below. The FT-IR spectral data (Fig. 1) confirms that the parent bis-phenol (upper spectrum) has been successfully converted into the corresponding dicyanate ester (lower spectrum). In particular the appearance of an intense doublet at 2272 cm^{-1} and 2211 cm^{-1} corresponding to the C=N stretch (and characteristic of compounds bearing the cyanato moiety) (Ben-Efraim, 1977; Cozzens et al., 1987), and the disappearance of the intense, broad absorbance at ca. 3300–3600 cm⁻¹ corresponding to the O–H stretch of the bis-phenol (Bellamy, 1975) are of greatest diagnostic use. The single chemical shift in the ¹⁵N NMR spectrum is also consistent with cyanato nitrogens previously reported (Barton et al., 1990a, Hamerton, 1991a), and indicates that the material as prepared contains no partially-converted oligomers (which display a markedly different chemical shift corresponding to the s-triazine nitrogen, Barton et al., 1990a). The ¹H NMR spectrum (Fig. 2) also demonstrates that the allyl group has remained intact during the incorporation of the cyanato group, thus retaining the potential to undergo further reaction via this electron-rich functional group (Stenzenberger, 1986; Zahir et al., 1989). The allylic protons produce a characteristic splitting pattern and are well established in the literature (Liao and Wang, 1989). Furthermore, the appearance of bands at 1640 (w), 994 (m), and 919 (m) cm^{-1} in the FT-IR spectrum are also indicative of the allylic double bond (Bellamy, 1975) and provide additional confirmation of its presence.

The structure of molecule (I) is shown in Fig. 3 and the arrangement of molecules in the unit cell is shown in Fig. 4. The positional and thermal parameters and the bond distances and angles for molecule (I) are given in Tables 3 and 4, respectively.

Figure 3 shows molecule (I) as a central tetrahedral carbon surrounded by two methyl, and two 3-allyl-4cyanatophenyl groups. There are only two other examples of organic molecules containing the cyanato moiety in the Cambridge Crystallographic Database (CCD V.3) (Allen *et al.*, 1979): 2,2'-*bis*(4-cyanatophenyl)isopropylidene (Davies *et al.* 1990) and 4-chloro-3,5-dimethyl-phenyl cyanate which has two molecules in the asymmetric unit (Kutschabsky and Schrauber, 1973). A comparison of the geometry around the cyanato group for the molecule reported here and the molecules



Fig. 1. FT-IR spectra (thin film, KBr plates) of the parent *bis*-phenol (above) and the title compound (below).



Fig. 2. ¹H NMR spectrum of the title compound (300 MHz, D₆-acetone, ppm from TMS).



Fig. 3. Structure of molecule with thermal ellipsoids at 50% probability.



Fig. 4. Stereodiagram of unit-cell.

extracted from CCD, is given in Table 5—the geometries of all three are in good agreement. Table 4 reveals two short distances: C102—C103 (1.068 Å) and C201—C203 (1.143(3) Å). This may be in part due to the poor quality of the original data set (arising from a compound that crystallizes very reluctantly, and is apt to remain liquid once fusion has occurred) and/or to thermal disorder within the molecule (C102, $B_{eq} = 8.4(2)$; C103, $B_{eq} = 21.2(4)$; C202, $B_{eq} = 11.5(3)$; C203, $B_{eq} = 11.0(3)$) where the terminal atoms are undergoing large thermal rotation.

The original rationale for the work (*i.e.*, the determination of the orientation of the substituted allyl group with respect to the cyanato moiety) has been fully satisfied. As the allyl and cyanato substituents are diammetrically opposed (Fig. 3), they are not correctly

orientated for an intramolecular dipole-dipole interaction to occur between them in the solid state. Furthermore, a search of the intermolecular and intramolecular distances reveals no significant interactions between the atoms from which these substituents are composed (a value of 3.25 Å was taken for the sum of the van der Waals' radii for N and C, Bondi, 1964). Subsequent studies involving the successful copolymerization of the title compound with a number of reactive comonomers (Barton et al., 1990b; Hamerton, 1991a) have confirmed these observations. Hence, the cyanato mojety is demonstrably unhindered during copolymerization through the intact allyl group. It is believed that this coreaction with the electron-deficient maleimide C=Cbonds occurs via an 'ene'/Diels-Alder mechanism (Zahir et al. 1989), and work is in progress to confirm this.

		-		-		-				
Atom ^b	x	У	Z	$B_{\rm eq}$	U(1, 1)	U(2, 2)	<i>U</i> (3, 3)	U(1, 2)	U(1, 3)	U(2, 3)
01	0.6642(4)	0.3940(3)	0.5420(2)	5.23(9)	0.094(3)	0.060(2)	0.044(2)	-0.006(2)	-0.003(2)	0.007(2)
O2	0.1716(4)	0.5548(4)	0.0997(2)	6.2(1)	0.062(2)	0.100(3)	0.072(2)	0.004(2)	-0.015(2)	-0.026(2)
N1	0.5894(6)	0.1932(5)	0.5478(3)	7.3(1)	0.141(5)	0.067(3)	0.068(3)	-0.007(4)	0.015(3)	0.011(3)
N2	0.1694(6)	0.4383(7)	-0.0141(4)	9.6(2)	0.098(4)	0.149(5)	0.115(4)	-0.011(4)	-0.017(3)	-0.065(4)
C10	0.6593(5)	0.4551(5)	0.4717(3)	3.7(1)	0.054(3)	0.046(3)	0.041(2)	-0.001(3)	0.003(2)	0.006(3)
C11	0.6176(5)	0.4019(4)	0.4053(3)	3.9(1)	0.056(3)	0.041(3)	0.052(3)	-0.005(3)	-0.004(2)	-0.003(3)
C12	0.6159(5)	0.4686(5)	0.3404(3)	3.6(1)	0.048(3)	0.040(3)	0.047(3)	0.002(3)	-0.003(2)	-0.002(3)
C13	0.6543(5)	0.5856(4)	0.3406(3)	3.3(1)	0.036(2)	0.040(3)	0.050(3)	0.001(2)	0.002(2)	-0.005(3)
C14	0.6947(5)	0.6342(4)	0.4087(3)	3.8(1)	0.045(3)	0.039(3)	0.058(3)	-0.005(3)	0.002(2)	-0.002(3)
C15	0.6980(5)	0.5711(5)	0.4765(3)	3.6(1)	0.047(3)	0.044(3)	0.047(3)	0.002(3)	0.001(2)	-0.008(3)
C16	0.6567(5)	0.6543(5)	0.2652(3)	3.6(1)	0.045(3)	0.049(3)	0.045(3)	-0.002(2)	0.004(2)	0.003(3)
C17	0.7849(5)	0.6178(6)	0.2246(3)	5.1(1)	0.041(3)	0.089(4)	0.065(3)	-0.000(3)	0.009(2)	0.013(3)
C18	0.6634(6)	0.7872(5)	0.2785(3)	5.0(1)	0.072(3)	0.054(3)	0.065(3)	-0.015(3)	-0.008(3)	0.016(3)
C19	0.5301(5)	0.6263(4)	0.2189(3)	3.4(1)	0.047(3)	0.037(3)	0.045(2)	-0.003(2)	0.002(2)	0.008(2)
C20	0.4039(5)	0.6562(5)	0.2464(3)	4.0(1)	0.048(3)	0.057(3)	0.045(3)	0.001(3)	0.007(2)	~0.000(3)
C21	0.2850(5)	0.6336(5)	0.2082(3)	4.3(1)	0.047(3)	0.063(4)	0.052(3)	0.003(3)	0.004(2)	0.000(3)
C22	0.2972(5)	0.5782(5)	0.1386(3)	4.1(1)	0.049(3)	0.052(3)	0.052(3)	0.003(3)	-0.009(2)	0.003(3)
C23	0.4160(5)	0.5467(5)	0.1090(3)	4.2(1)	0.063(3)	0.050(3)	0.048(3)	0.008(3)	0.000(2)	-0.002(3)
C24	0.5334(5)	0.5707(5)	0.1485(3)	3.9(1)	0.052(3)	0.047(3)	0.048(3)	0.007(3)	0.005(2)	-0.001(3)
C25	0.6242(6)	0.2862(5)	0.5426(3)	5.0(1)	0.086(4)	0.057(3)	0.045(3)	-0.000(3)	0.008(3)	0.008(3)
C26	0.1729(6)	0.4926(6)	0.0404(4)	5.9(2)	0.069(4)	0.078(4)	0.076(4)	-0.007(3)	-0.013(3)	-0.012(4)
C101	0.7470(6)	0.6258(5)	0.5500(3)	5.1(1)	0.077(3)	0.072(4)	0.045(3)	-0.003(3)	-0.000(3)	-0.013(3)
C102	0.8914(7)	0.6042(7)	0.5638(4)	8.4(2)	0.097(5)	0.133(6)	0.089(4)	0.011(5)	-0.032(4)	-0.044(5)
C103	0.9831(9)	0.644(1)	0.5832(7)	21.2(4)	0.102(6)	0.37(1)	0.33(1)	0.093(7)	-0.089(6)	-0.262(8)
C201	0.1484(6)	0.6643(7)	0.2382(4)	7.3(2)	0.048(3)	0.143(6)	0.088(4)	0.005(4)	0.007(3)	-0.033(5)
C202	0.1367(7)	0.7471(9)	0.2936(5)	11.5(3)	0.073(4)	0.205(8)	0.158(6)	0.017(5)	0.033(4)	-0.087(6)
C203	0.0879(8)	0.7822(9)	0.3474(5)	11.0(3)	0.091(5)	0.178(8)	0.150(6)	-0.009(6)	0.033(5)	-0.080(6)
H12	0.5863	0.4306	0.2904	5*						
H11	0.5854	0.3166	0.4045	5*						
H14	0.7257	0.7194	0.4097	5*						
H17A	0.7855	0.5297	0.2142	5*						
H17B	0.7936	0.6577	0.1733	5*						
H17C	0.8685	0.6362	0.2552	5*						
H18A	0.5806	0.8152	0.3043	5*						
H18B	0.7448	0.8071	0.3094	5*						
H18C	0.6699	0.8285	0.2275	5*						
H20	0.3967	0.6988	0.2975	5*						
H23	0.4200	0.5047	0.0572	5*						
H24	0.6235	0.5522	0.1263	5*						
H101A	0.6843	0.6017	0.5909	5*						
H101B	0.7394	0.7151	0.5441	5*						
H102	0.9108	0.5077	0.5748	5*						
H103A	1.0674	0.6279	0.5881	5*						
H103B	0.9572	0.7421	0.5695	5*						
H201A	0.0799	0.6639	0.1954	5*						
H201B	0.1168	0.5877	0.2713	5*						
H202	0.1762	0.8334	0.2613	5*						

Table 3. Final positional and thermal parameters with esd's in parentheses and B_{eq} thermal parameters $(Å^2)^a$

^{*a*} The B_{eq} values are calculated according to the equation $4/3\Sigma_i \Sigma_j B_{ij} a_i a_j$.

0.3755

0.3816

0.8609

0.7150

H203A

H203B

0.0925

0.0468

^b The hydrogen atoms were not refined and were allocated fixed temperature factors of B = 5.0 Å². The form of the anisotropic thermal parameter is exp $\left[-2\pi^{2}\{h^{2}a^{*2}U(1, 1) + k^{2}b^{*2}U(2, 2) + l^{2}c^{*2}U(3, 3) + 2hka^{*}b^{*}U(1, 2) + 2hla^{*}c^{*}U(1, 3) + 2klb^{*}c^{*}U(2, 3)\}\right]$.

5*

5*

Atom1	Atom2]	Distance	Atom1	Ator	n2	Distance	Atom1	At	om2	Distance
01	C10	_	1.422(6)	C15	C101 1.513(7)		C201	C202		1.366(12)	
01	C25		1.295(7)	C16	C17	C17 1.525(7)		C202	C	203	1.143(13)
02	C22		1.440(6)	C16	C18	1	1.539(8)				
O2	C26		1.263(8)	C16	C19)	1.522(7)				
N1	C25		1.123(8)	C19	C20)	1.392(7)				
N2	C26		1.143(9)	C19	C24	Ļ	1.394(7)				
C10	C11		1.377(7)	C20	C21		1.375(7)				
C10	C15		1.384(7)	C21	C22		1.386(7)				
C11	C12		1.374(4)	C21	C20)1	1.502(8)				
C12	C13		1.391(7)	C22	C23	,	1.343(7)				
C13	C14		1.376(7)	C23	C24	ļ	1.377(7)				
C13	C16		1.544(7)	C101	C10)2	1.470(9)				
C14	C15		1.395(7)	C102	C10)3	1.068(13)				
Atom 1	Atom2	Atom3	Angle	Atom1	Atom2	Atom3	Angle	Atom1	Atom2	Atom3	Angle
C10	01	C25	117.9(4)	C14	C15	C101	121.5(5)	02	C22	C23	121.6(5)
C22	02	C26	118.7(4)	C13	C16	C17	106.6(4)	C21	C22	C23	123.5(5)
01	C10	C11	122.0(5)	C13	C16	C18	111.8(4)	C22	C23	C24	119.4(5)
01	C10	C15	114.3(4)	C13	C16	C19	109.4(4)	C20	C19	C24	117.0(4)
C11	C10	C15	123.7(5)	C17	C16	C18	107.9(4)	C19	C20	C21	123.6(5)
C10	C11	C12	117.5(5)	C17	C16	C19	112.3(4)	C19	C24	C23	120.7(5)
C11	C12	C13	122.1(5)	C18	C16	C19	108.8(4)	O1	C25	N1	175.9(6)
C12	C13	C14	117.9(4)	C16	C19	C20	120.1(4)	02	C26	N2	177.3(7)
C12	C13	C16	119.6(4)	C16	C19	C24	122.9(4)	C15	C101	C102	112.0(5)
C14	C13	C16	122.5(4)	C20	C21	C22	115.8(5)	C101	C102	C103	144.(1)
C13	C14	C15	122.7(5)	C20	C21	C201	123.8(5)	C21	C201	C202	119.9(6)
C10	C15	C14	116.1(4)	C22	C21	C201	120.5(5)	C201	C202	C203	151.(1)
C10	C15	C101	122.3(5)	O2	C22	C21	114.9(4)				

Table 4. Bond lengths/Å and bond angles/deg with esd's in parentheses

Table 5. Comparison of the cyanato group geometry in 2,2'-bis (3-allyl-4-cyanatophenyl) isopropylidene (I) with that of 2,2'-bis (4-cyanato-
phenyl)isopropylidene (II) and 4-chloro-3,5-dimethylphenyl cyanate (III) (2 molecules in asymmetric unit) (esd's for bond lengths and
angles are not available in the Cambridge database)

$O_{c} - C_{c} \equiv N_{c}$ C_{a}								
$C_a - O_c(Å)$	()	I)	(]	II)	(III)			
	1.422(6)	1.440(6)	1.435(3)	1.423(4)	1.416	1.416		
$O_c - C_c$	1.295(7)	1.263(8)	1.275(4)	1.285(4)	1.272	1.268		
$C_c - N_c$	1.123(8)	1.143(9)	1.119(5)	1.128(5)	1.142	1.131		
$C_a - O_c - C_c(\circ)$	117.9(4)	118.7(4)	117.4(2)	118.6(3)	118.2	118.3		
$O_c - C_c - N_c$	175.9(6)	177.3(7)	176.5(3)	176.3(4)	174.9	173.5		
$C_a = O_c = C_c = N_c$	-167.1()	-155.2()	174.9(6)	159.9(5)	177.4	-137.5		

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Structure factor data have been deposited with the British Library, Boston Spa, Wetherby, West Yorkshire, UK as supplementary publication No. 60755 (9 pages).