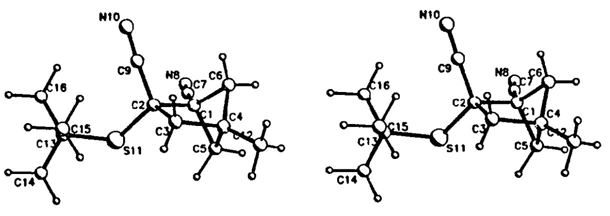

Fig. 1. Stereoscopic view of the molecule (program *PLUTO*; Motherwell & Clegg, 1978).

= 0·046 for 891 observed reflections. Final maximum shift to e.s.d. = 0·28. $S = 0{\cdot}86$. Maximum and minimum heights in final difference Fourier synthesis = 0·16 and −0·18 e Å^{-3}. Atomic scattering factors were from *International Tables for X-ray Crystallography* (1974).

Atomic parameters are given in Table 1, bond distances and angles in Table 2.* Fig. 1 is a stereoscopic view of the molecule.

Related literature. The structure of (1′*R*,2′*R*,4′*R*)-2-(1′-methyl-5′-oxo-2′-bicyclo[2.1.1]hexyl)ethyl 4-bromobenzoate has been reported recently (Rettig & Trotter, 1987).

The geometry of *cd*-substituted ethane and cyclopropane derivatives has been discussed (Parfonry, Declercq, Tinant, Van Meerssche & Schweiss, 1988; Tinant, Wu, Declercq, Van Meerssche, Masamba, De Mesmaeker & Viehe, 1988).

We thank Dr V. Gallez and Professor H. G. Viehe, Laboratoire de Chimie Organique, Université de Louvain, for providing the crystals.

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* Lists of structure factors, anisotropic thermal parameters, H-atom parameters and bond distances and angles involving H atoms have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52464 (8 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Acta Cryst. (1990). C**46**, 905–907

Dimethyl 5-Amino-3-methyl-4-triisopropylsilyl-1,2-benzenedicarboxylate

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(*Received* 10 *October* 1989; *accepted* 22 *November* 1989)

Abstract. $C_{20}H_{33}NO_4Si$, $M_r = 379{\cdot}57$, monoclinic, $P2_1/c$, $a = 17{\cdot}287\,(5)$, $b = 8{\cdot}895\,(2)$, $c = 15{\cdot}049\,(4)$ Å, $\beta = 109{\cdot}53\,(2)°$, $V = 2181\,(1)$ Å^3, $Z = 4$, $D_x = 1{\cdot}16$ g cm^{-3}, λ(Mo $K\alpha$) = 0·71069 Å, μ = 1·35 cm^{-1}, $F(000) = 824$, $T = 291$ K, $R = 0{\cdot}050$ for 3013 observed reflections. Severe steric strain in this pentasubstituted benzene derivative leads to: (*a*) rotation of the methoxycarbonyls at C1, C2 out of the benzene plane by 29·5 (3) and 99·7 (3)° respectively. (*b*) Significant lengthening of the C3—C4 and C4—C5 bonds to 1·418 (3) and 1·429 (3) Å and a stretching of the Si—C(ar.) bond to 1·930 (2) compared with an expected value of 1·868 Å [Allen, Kennard, Watson, Brammer, Orpen & Taylor (1987). *J. Chem.*

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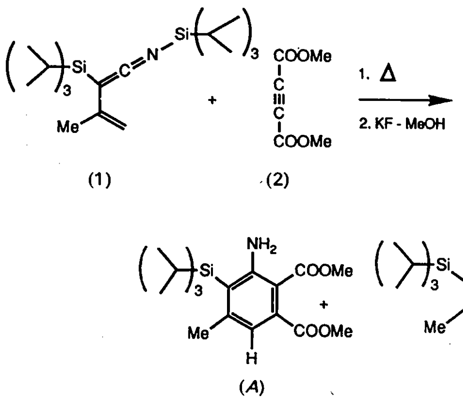
Soc. Perkin Trans. 2, pp. S1–S19]. (*c*) Significant deviation of the aromatic ring from planarity: the endocyclic torsion angles are $-8·2$, 1·3, 10·5, $-15·8$, 9·7 and 2·7° ($\sigma_{av} = 0·6°$). (*d*) Out-of-plane bending of the atoms C15, Si16 and N26 which deviate by $-0·12$, $+0·58$ and $-0·30$ Å from the mean plane through the aromatic carbons. Hydrogen bonds with —NH_2 as donor and the two carbonyl oxygens as acceptors are observed: O8···N26 = 3·077 (3), O8···H26*A* = 2·24 (4) Å, O—H—N = 154° (N26: $1-x$, $y-0·5$, $2·5-z$); O12···N26 = 3·131 (3), O12···H26*B* = 2·53 (4) Å, O—H—N = 129° (N26: x, $0·5-y$, $z+0·5$).

Experimental. The cycloaddition of dimethyl acetylenedicarboxylate (2) to the diene (1) yields two isomers *A* and *B* which were difficult to assign by spectroscopic methods (Differding, Vandevelde, Roekens, Van & Ghosez, 1987). One of the compounds was a yellow oil, the other a white crystalline solid.



The X-ray analysis was undertaken and confirms that structure *B* is the solid product.

Table 1. *Fractional atomic coordinates and equivalent isotropic temperature factors*

$B_{eq} = (8\pi^2/3)\sum_i\sum_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$

	x	y	z	B_{eq}(Å^2)
C1	0·3992 (1)	0·1708 (3)	1·2302 (2)	3·20 (4)
C2	0·3410 (1)	0·0557 (3)	1·2017 (2)	3·10 (4)
C3	0·2782 (1)	0·0662 (3)	1·1148 (2)	3·19 (4)
C4	0·2721 (1)	0·1892 (3)	1·0528 (2)	3·17 (4)
C5	0·3415 (1)	0·2874 (3)	1·0772 (2)	3·56 (4)
C6	0·4000 (2)	0·2824 (3)	1·1677 (2)	3·54 (4)
C7	0·4618 (2)	0·1698 (3)	1·3258 (2)	3·73 (5)
O8	0·4868 (1)	0·0589 (3)	1·3716 (1)	5·86 (4)
O9	0·4863 (1)	0·3075 (2)	1·3558 (1)	5·33 (4)
C10	0·5510 (2)	0·3169 (5)	1·4466 (2)	7·48 (8)
C11	0·3433 (2)	–0·0750 (3)	1·2647 (2)	3·56 (5)
O12	0·3183 (1)	–0·0748 (2)	1·3298 (2)	5·85 (5)
O13	0·3741 (1)	–0·1969 (2)	1·2371 (1)	4·33 (4)
C14	0·3742 (2)	–0·3353 (4)	1·2875 (3)	6·17 (7)
C15	0·2172 (2)	–0·0621 (3)	1·0898 (2)	5·17 (6)
Si16	0·1729 (1)	0·2338 (1)	0·9497 (1)	3·00 (1)
C17	0·0782 (2)	0·1758 (4)	0·9790 (2)	4·21 (5)
C18	–0·0021 (2)	0·2193 (5)	0·9026 (3)	6·96 (8)
C19	0·0770 (2)	0·2271 (5)	1·0757 (3)	6·88 (8)
C20	0·1678 (2)	0·4431 (3)	0·9233 (2)	4·63 (6)
C21	0·0996 (2)	0·4932 (4)	0·8341 (3)	7·16 (8)
C22	0·1687 (3)	0·5428 (4)	1·0054 (3)	8·67 (10)
C23	0·1713 (2)	0·1327 (3)	0·8383 (2)	3·97 (5)
C24	0·2476 (2)	0·1707 (4)	0·8124 (2)	5·39 (7)
C25	0·1583 (3)	–0·0372 (4)	0·8359 (3)	6·51 (8)
N26	0·3547 (2)	0·3913 (4)	1·0168 (2)	6·17 (6)

Table 2. *Bond distances* (Å) *and angles* (°)

C2—C1	1·398 (3)	C6—C1	1·370 (4)
C7—C1	1·483 (3)	C3—C2	1·397 (3)
C11—C2	1·492 (3)	C4—C3	1·418 (3)
C15—C3	1·514 (3)	C5—C4	1·429 (3)
Si16—C4	1·930 (2)	C6—C5	1·399 (3)
N26—C5	1·368 (3)	O8—C7	1·198 (3)
O9—C7	1·325 (3)	C10—O9	1·450 (3)
O12—C11	1·195 (3)	O13—C11	1·334 (3)
C14—O13	1·445 (3)	C17—Si16	1·903 (3)
C20—Si16	1·899 (3)	C23—Si16	1·894 (3)
C18—C17	1·528 (4)	C19—C17	1·533 (4)
C21—C20	1·527 (4)	C22—C20	1·517 (5)
C24—C23	1·533 (4)	C25—C23	1·526 (4)

C6—C1—C2	119·4 (2)	C7—C1—C2	120·6 (2)
C7—C1—C6	119·9 (2)	C3—C2—C1	119·5 (2)
C11—C2—C1	120·8 (2)	C11—C2—C3	119·6 (2)
C4—C3—C2	122·1 (2)	C15—C3—C2	116·2 (2)
C15—C3—C4	121·7 (2)	C5—C4—C3	115·5 (2)
Si16—C4—C3	122·6 (2)	Si16—C4—C5	121·6 (2)
C6—C5—C4	120·1 (2)	N26—C5—C4	123·7 (2)
N26—C5—C6	116·2 (2)	C5—C6—C1	121·4 (2)
O8—C7—C1	124·7 (3)	O9—C7—C1	111·8 (2)
O9—C7—O8	123·5 (2)	C10—O9—C7	115·6 (2)
O12—C11—C2	125·4 (2)	O13—C11—C2	111·4 (2)
O13—C11—O12	123·2 (3)	C14—O13—C11	117·4 (2)
C17—Si16—C4	111·1 (1)	C20—Si16—C4	109·7 (1)
C20—Si16—C17	109·3 (1)	C23—Si16—C4	111·1 (1)
C23—Si16—C17	108·4 (1)	C23—Si16—C20	107·1 (1)
C18—C17—Si16	113·1 (2)	C19—C17—Si16	115·2 (2)
C19—C17—C18	109·9 (3)	C21—C20—Si16	116·0 (2)
C22—C20—Si16	114·5 (2)	C22—C20—C21	109·9 (3)
C24—C23—Si16	111·1 (2)	C25—C23—Si16	116·7 (2)
C25—C23—C24	110·1 (3)		

Crystals obtained by evaporation from dichloromethane. D_m not measured. Parallelepiped crystal with dimensions 0·35 × 0·32 × 0·13 mm. Lattice parameters refined using 15 reflections in the range 5° $\leq 2\theta \leq$ 25°. Syntex $P2_1$, graphite-monochromatized Mo $K\alpha$ radiation. 4284 $hk\pm l$ independent reflections with $(\sin\theta)/\lambda \leq 0·617$ Å^{-1}; $0 \leq h \leq 21$, $0 \leq k \leq 10$, $-18 \leq l \leq 16$, 3013 with $I \geq 2·5\sigma(I)$. Standard reflection (144) checked every 50 reflections: no significant intensity variation. Structure solved by *SHELXS*86 (Sheldrick, 1985); six H atoms from difference Fourier synthesis; those of the methyl groups in idealized positions (C—H = 1·08 Å, H—C—H = 109·5°). Anisotropic least-squares refinement (*SHELX*76; Sheldrick, 1976) on *F*; H isotropic with common refined temperature factor. $w = 1/(\sigma^2 + 0·00053F^2)$, $R = 0·050$, $wR = 0·054$ for 3013 observed reflections. Final maximum shift/e.s.d. = 0·26 (U_{23} of atom C6); $S = 1·78$. Max. and min. heights in final difference Fourier synthesis 0·26 and $-0·26$ e Å^{-3}. Atomic scattering factors from *International Tables for X-ray Crystallography* (1974). The

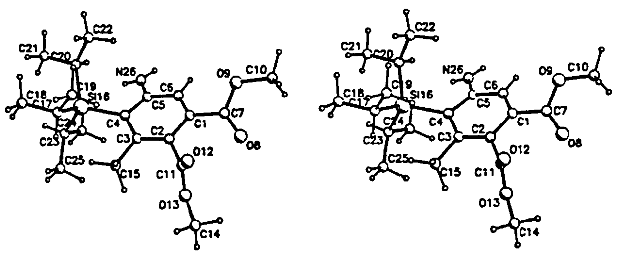


Fig. 1. Stereoscopic view of the molecule (*PLUTO*; Motherwell & Clegg, 1978) showing the atomic numbering scheme.

atomic parameters are given in Table 1.* The bond lengths and the bond angles are listed in Table 2. Fig. 1 shows a stereoscopic view of the molecule.

Related literature. Strong deviations from planarity of non-condensed, non-bridged benzene or pyridine derivatives have been reported for *tert*-butyl 1,2,3-tris(*tert*-butyl)-4,6-bis(methoxycarbonyl)-5-benzoate (I) and 2,3,4-tris(*tert*-butyl)-5-*tert*-butoxycarbonyl-6-ethoxycarbonylpyridine (II) (Maas, Fink, Wingert, Blatter & Regitz, 1987). In those cases the steric overcrowding is caused by *vic*-tri-*tert*-butyl substitution of the aromatic ring which results in unsymmetrical boat conformations with bow and stern angles as high as 30·1 and 11·6° (I) and 25·5 and 12·9° (II).

For silyl aromatic compounds with both *ortho* positions substituted, endocyclic torsion angles similar to those reported here have been observed in the tri-*tert*-butylphenyl moiety of 1,1-difluoro-2,2-di-mesityl-1-(2,4,6-tri-*tert*-butylphenyl)disilane (Weidenbruch, Kramer, Pohl & Saak, 1986).

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* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52465 (21 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Acta Cryst. (1990). C**46**, 907–909

Structure of Ethyl *p*-Nitrophenylsulfonylacetate

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(*Received* 28 *September* 1989; *accepted* 10 *November* 1989)

Abstract. $C_{10}H_{11}NO_6S$, $M_r = 273{\cdot}26$, monoclinic, $P2_1/c$, $a = 11{\cdot}440\ (1)$, $b = 13{\cdot}239\ (2)$, $c = 8{\cdot}131\ (1)$ Å, $\beta = 92{\cdot}69\ (1)°$, $V = 1230$ Å^3, $Z = 4$, $D_x = 1{\cdot}475$ g cm^{-3}, $\lambda(\mathrm{Cu}\ K\alpha) = 1{\cdot}54178$ Å, $\mu = 25{\cdot}04$ cm^{-1}, $F(000) = 568$, $T = 293$ K, $R = 0{\cdot}060$, $wR = 0{\cdot}058$ for 1302 reflections. All bond lengths and angles lie within normal ranges. The ethyl acetate substituent is almost planar [maximum deviation of 0·14 (1) Å for the terminal —CH_2CH_3 group] and makes a dihedral angle of 93·5 (1)° with the plane of the phenyl ring. The NO_2 group lies nearly in the plane of the phenyl ring [max. deviation of

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