Crystal structure of 1-[(*E*)-2-[4-(trifluoromethyl)phenyl]-1-ethenyl]-naphthalene, C₁₉H₁₃F₃

M. Jevric, D. K. Taylor and E. R. T. Tiekink^{*.†}

The University of Adelaide, Department of Chemistry, Australia 5005

Received May 24, 2001, CCDC-No. 1267/671

Abstract

C₁₉H₁₃F₃, orthorhombic, *P*2₁2₁2 (No. 18), *a* = 14.983(5) Å, *b* = 26.740(6) Å, *c* = 7.120(3) Å, *V* = 2852.6 Å³, *Z* = 8, *R*_{gt}(*F*) = 0.054, *wR*_{ref}(*F*²) = 0.173, *T* = 173 K.

Source of material

To a suspension of (4-trifluoromethylbenzyl)triphenylphosphonium bromide (13.5 g, 0.026 mol) in anhydrous ether (70 mL) at 278 K was added potassium butoxide (3.0 g, 0.027 mol) under a nitrogen atmosphere. The mixture was then allowed to attain ambient temperature over 30 min, after which time 1-naphthaldehyde (4 mL, 0.028 mol) was added dropwise over 20 min. Stirring of the mixture was continued for a further 24 h after which time the suspension was allowed to settle and the solvent decanted. Warm hexane (70 mL) was added to the residue with stirring and decanted after being allowed to cool to ambient temperature and the organics combined. The volatiles were removed in vacuo and the residue purified by squat chromatography (hexane). Removal of the volatile in vacuo afforded a mixture (6.3 g, 81%) of the *E*

and *Z* isomers of the title compound. Upon standing colourless crystals of the title compound formed (1.1 g) and were filtered off and recrystallised from an evaporating solution of hexane. mp 399.5 K – 400.5 K. Spectroscopic data are included in the deposited CIF.

Experimental details

The C-bound H atoms were placed in their geometrically calculated positions and included in the final refinement in the riding model approximation. Despite the experiment being conducted at 173 K, large thermal motion is noted for the F atoms comprising the CF₃ group. No evidence for multiple sites were found. The absolute structure was not determined.

Discussion

Two independent molecules of the title compound comprise the crystallographic asymmetric unit – there are only minor conformational differences between them. The structure analysis shows that the conformation about the C1=C2 double bond is *E*. With the exception of the CF₃ group, the molecule is essentially planar. The crystal structure is stabilized by a number of C–H... π interactions with the closest such contact of 2.70 occurring between C26–H and the ring centroid of C14a–C18a with an angle of 140° subtended at H (symmetry operation: 1–*x*, 1–*y*, *z*).

Table 1. Data collection and handling.

Crystal:	colourless plate, size 0.06 × 0.37 × 0.48 mm
Wavelength:	Mo <i>K</i> α radiation (0.71069 Å)
μ :	1.07 cm ^{−1}
Diffractometer, scan mode:	Rigaku AFC7R, $\omega/2\theta$
2 $\theta_{\max}$:	55°
<i>N</i> (<i>hkl</i>) _{measured} , <i>N</i> (<i>hkl</i>) _{unique} :	3706, 3706
Criterion for <i>I</i> _{obs} , <i>N</i> (<i>hkl</i>) _{gt} :	<i>I</i> _{obs} > 2 σ (<i>I</i> _{obs}), 1878
<i>N</i> (<i>param</i>) _{refined} :	398
Programs:	SIR92 [1], SHELXL-97 [2], teXsan [3], DIFABS [4], PLATON [5]

Table 2. Atomic coordinates and displacement parameters (in Å²).

Atom	Site	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso}
H(1)	4c	0.6869	0.4978	0.9131	0.044(3)
H(2)	4c	0.5545	0.4447	0.7307	0.044
H(12)	4c	0.6026	0.4794	0.4647	0.044
H(13)	4c	0.5776	0.5375	0.2243	0.044
H(14)	4c	0.5915	0.6214	0.2829	0.044
H(15)	4c	0.6280	0.6911	0.5000	0.044
H(16)	4c	0.6729	0.7188	0.7881	0.044
H(17)	4c	0.7013	0.6623	1.0300	0.044

^{*} Correspondence author (e-mail: chmtert@nus.edu.sg)

[†] Current address: National University of Singapore,
Department of Chemistry, Singapore 117543

Table 2. Continued.

Atom	Site	x	y	z	U _{iso}
H(18)	4c	0.6880	0.5773	0.9805	0.044
H(01)	4c	0.8142	0.5107	1.3627	0.044
H(02)	4c	0.9322	0.4472	1.1798	0.044
H(012)	4c	0.8948	0.4855	0.9120	0.044
H(013)	4c	0.9338	0.5405	0.6720	0.044
H(014)	4c	0.9447	0.6249	0.7301	0.044
H(015)	4c	0.9245	0.6967	0.9432	0.044
H(016)	4c	0.8843	0.7286	1.2313	0.044
H(017)	4c	0.8355	0.6753	1.4707	0.044

Table 2. Continued.

Atom	Site	x	y	z	U _{iso}
H(018)	4c	0.8321	0.5898	1.4250	0.044
H(22)	4c	0.6885	0.4441	1.1534	0.044
H(23)	4c	0.6907	0.3808	1.3761	0.044
H(25)	4c	0.5226	0.2988	1.0567	0.044
H(26)	4c	0.5161	0.3637	0.8379	0.044
H(022)	4c	0.8063	0.4596	1.6097	0.044
H(023)	4c	0.7822	0.3968	1.8273	0.044
H(025)	4c	0.9181	0.2987	1.4981	0.044
H(026)	4c	0.9409	0.3622	1.2787	0.044

Table 3. Atomic coordinates and displacement parameters (in Å²).

Atom	Site	x	y	z	U ₁₁	U ₂₂	U ₃₃	U ₁₂	U ₁₃	U ₂₃
F(1)	4c	0.6057(4)	0.3104(1)	1.5531(5)	0.208(5)	0.058(2)	0.039(2)	0.004(3)	-0.003(3)	0.006(2)
F(2)	4c	0.6861(3)	0.2681(2)	1.3731(7)	0.092(3)	0.088(3)	0.144(4)	0.046(2)	0.034(3)	0.067(3)
F(3)	4c	0.5471(3)	0.2599(1)	1.3641(6)	0.107(3)	0.082(3)	0.086(3)	-0.046(2)	-0.030(3)	0.037(2)
F(01)	4c	0.8848(4)	0.3030(2)	1.9601(8)	0.211(6)	0.204(6)	0.155(5)	-0.146(5)	-0.121(5)	0.142(5)
F(02)	4c	0.7548(3)	0.3019(2)	1.8994(8)	0.101(3)	0.119(4)	0.166(5)	0.031(3)	0.070(3)	0.090(4)
F(03)	4c	0.8380(5)	0.2573(1)	1.7593(7)	0.345(9)	0.042(2)	0.105(4)	0.016(3)	0.107(5)	0.014(2)
C(1)	4c	0.6443(3)	0.4915(2)	0.8171(7)	0.036(2)	0.043(3)	0.033(3)	0.006(2)	-0.002(2)	-0.002(2)
C(11)	4c	0.6356(3)	0.5287(2)	0.6669(7)	0.032(2)	0.043(3)	0.036(3)	0.005(2)	0.005(2)	0.005(2)
C(2)	4c	0.5968(3)	0.4498(2)	0.8281(7)	0.034(2)	0.043(3)	0.038(3)	0.007(2)	-0.003(2)	-0.002(2)
C(12)	4c	0.6100(3)	0.5140(2)	0.4900(7)	0.043(3)	0.049(3)	0.039(3)	0.002(2)	0.007(3)	-0.002(2)
C(13)	4c	0.5945(4)	0.5487(2)	0.3458(7)	0.055(3)	0.074(4)	0.028(3)	0.005(3)	0.001(3)	0.000(3)
C(14)	4c	0.6036(4)	0.5981(2)	0.3805(7)	0.057(3)	0.063(4)	0.036(3)	0.008(3)	0.003(3)	0.017(3)
C(14A)	4c	0.6303(3)	0.6159(2)	0.5571(7)	0.039(3)	0.049(3)	0.037(3)	0.001(2)	0.011(2)	0.016(2)
C(15)	4c	0.6401(4)	0.6675(2)	0.5962(9)	0.060(3)	0.046(3)	0.057(4)	0.003(3)	0.023(3)	0.020(3)
C(16)	4c	0.6663(4)	0.6839(2)	0.7664(9)	0.057(3)	0.038(3)	0.066(4)	-0.004(2)	0.013(3)	0.001(3)
C(17)	4c	0.6836(3)	0.6502(2)	0.9102(9)	0.043(3)	0.049(3)	0.053(3)	-0.009(2)	0.002(3)	0.001(3)
C(18)	4c	0.6753(3)	0.5999(2)	0.8810(7)	0.036(2)	0.040(3)	0.045(3)	0.000(2)	-0.003(2)	0.006(2)
C(18A)	4c	0.6481(3)	0.5811(2)	0.7039(7)	0.027(2)	0.046(3)	0.034(3)	0.005(2)	0.005(2)	0.005(2)
C(01)	4c	0.8549(3)	0.5010(2)	1.2670(6)	0.033(2)	0.038(2)	0.031(2)	-0.005(2)	0.002(2)	-0.007(2)
C(011)	4c	0.8746(3)	0.5371(2)	1.1148(7)	0.030(2)	0.035(2)	0.037(3)	0.001(2)	-0.001(2)	0.000(2)
C(02)	4c	0.8913(3)	0.4557(2)	1.2766(7)	0.036(2)	0.037(2)	0.037(3)	-0.002(2)	0.002(2)	-0.002(2)
C(212)	4c	0.8964(3)	0.5204(2)	0.9372(7)	0.046(3)	0.040(3)	0.040(3)	0.006(2)	-0.005(3)	-0.003(2)
C(013)	4c	0.9206(3)	0.5532(2)	0.7936(7)	0.052(3)	0.058(3)	0.032(3)	0.006(3)	-0.003(2)	-0.004(3)
C(014)	4c	0.9254(3)	0.6030(2)	0.8272(7)	0.048(3)	0.052(3)	0.038(3)	0.002(2)	-0.003(2)	0.008(3)
C(04A)	4c	0.9020(3)	0.6230(2)	1.0051(7)	0.037(3)	0.044(3)	0.039(3)	0.003(2)	-0.008(2)	0.004(2)
C(015)	4c	0.9053(3)	0.6747(2)	1.0399(8)	0.046(3)	0.040(3)	0.053(3)	0.003(2)	-0.007(3)	0.011(3)
C(016)	4c	0.8816(3)	0.6936(2)	1.2099(8)	0.052(3)	0.032(2)	0.060(4)	0.001(2)	-0.011(3)	-0.001(3)
C(017)	4c	0.8532(3)	0.6617(2)	1.3532(8)	0.050(3)	0.044(3)	0.044(3)	0.008(2)	-0.011(3)	-0.014(3)
C(018)	4c	0.8507(3)	0.6111(2)	1.3256(7)	0.043(3)	0.036(2)	0.041(3)	0.002(2)	-0.002(2)	0.004(2)
C(08A)	4c	0.8753(3)	0.5901(2)	1.1513(7)	0.030(2)	0.032(2)	0.036(3)	-0.001(2)	-0.005(2)	0.000(2)
C(21)	4c	0.6014(3)	0.4107(2)	0.9709(6)	0.033(2)	0.031(2)	0.035(2)	0.004(2)	0.002(2)	-0.002(2)
C(22)	4c	0.6539(3)	0.4149(2)	1.1338(7)	0.042(2)	0.038(2)	0.042(3)	-0.002(2)	-0.005(2)	-0.003(2)
C(23)	4c	0.6558(3)	0.3771(2)	1.2656(7)	0.047(3)	0.040(3)	0.033(3)	-0.002(2)	-0.012(2)	-0.004(2)
C(24)	4c	0.6068(3)	0.3340(2)	1.2368(6)	0.037(2)	0.033(2)	0.033(2)	0.003(2)	-0.002(2)	-0.004(2)
C(27)	4c	0.6117(4)	0.2937(2)	1.3780(8)	0.052(3)	0.039(3)	0.049(3)	0.003(2)	0.005(3)	-0.004(3)
C(25)	4c	0.5556(3)	0.3287(2)	1.0772(7)	0.044(3)	0.029(2)	0.042(3)	0.001(2)	0.003(2)	-0.004(2)
C(26)	4c	0.5525(3)	0.3672(2)	0.9464(7)	0.030(2)	0.036(2)	0.037(3)	0.001(2)	-0.005(2)	-0.007(2)
C(021)	4c	0.8752(3)	0.4176(2)	1.4197(7)	0.030(2)	0.033(2)	0.040(3)	-0.001(2)	0.000(2)	-0.004(2)
C(022)	4c	0.8285(3)	0.4270(2)	1.5859(7)	0.043(3)	0.031(2)	0.045(3)	-0.001(2)	0.005(2)	-0.007(2)
C(023)	4c	0.8142(3)	0.3897(2)	1.7152(7)	0.046(3)	0.037(2)	0.036(3)	0.002(2)	0.008(2)	-0.009(2)
C(024)	4c	0.8468(3)	0.3412(2)	1.6828(7)	0.036(2)	0.037(2)	0.040(3)	-0.004(2)	0.004(2)	-0.001(2)
C(027)	4c	0.8314(3)	0.3023(2)	1.8240(8)	0.046(3)	0.045(3)	0.045(3)	-0.009(2)	0.004(3)	0.005(3)
C(025)	4c	0.8948(3)	0.3312(2)	1.5202(7)	0.041(3)	0.036(3)	0.050(3)	0.005(2)	0.009(3)	-0.006(2)
C(026)	4c	0.9084(3)	0.3692(2)	1.3902(8)	0.039(2)	0.039(2)	0.040(3)	0.005(2)	0.010(2)	-0.003(2)

Acknowledgment. The Australian Research Council is thanked for support.

References

- Altomare, G.; Cascarano, G.; Giacovazzo, C.; Guagliardi, A.; Burla, M. C.; Polidori, G.; Camalli, M.: SIR92 - a program for automatic solution of crystal structures by direct methods. *J. Appl. Crystallogr.* **27** (1994) 435.
- Sheldrick, G. M.: SHELXL-97. Program for crystal structure refinement. University of Göttingen, Germany 1997.
- teXsan: Single Crystal Structure Analysis Software. Version 1.04. Molecular Structure Corporation. The Woodlands, TX, USA 1997.
- Walker, N.; Stuart, D.: An empirical method for correcting diffractometer data for absorption effects. *Acta Crystallogr. A* **39** (1983) 159-166.
- Spek, T.: PLATON, A Multipurpose Crystallographic Tool, Utrecht University, Utrecht, The Netherlands 2000.