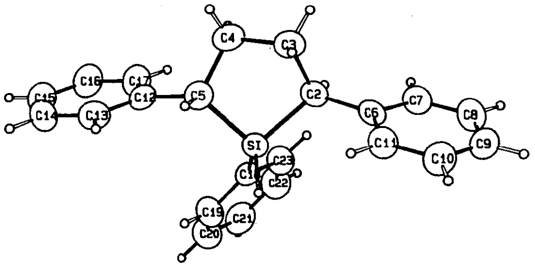

Fig. 1. Perspective view of the molecule with numbering scheme. Thermal ellipsoids are drawn at the 50% probability level.

chromator, ω–2θ scans at 4 to 6° $\min^{-1}$. Data having $2\theta \leq 53°$, $-21 \leq h \leq 21$, $0 \leq k \leq 7$, $-22 \leq l \leq 22$ measured. Three standard reflections 11,0,4, 2,1,11, $9\bar{1}\bar{9}$, ±1·3% maximum variation. 7232 reflections measured, 3618 unique ($R_{\text{int}} = 0{\cdot}03$), 1972 reflections with $I > 3\sigma(I)$ considered observed, corrected for background, Lorentz, polarization, no absorption or extinction correction applied. Solved by direct methods using *MULTAN*11/82 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1982) and Fourier methods. Full-matrix least squares minimized $\sum w(F_o - F_c)^2$ with $w = 4F_o^2[\sigma^2(I) + (0{\cdot}04F_o^2)^2]^{-1}$. Non-H atoms refined with anisotropic thermal parameters, H atoms placed at calculated positions and not refined. Final $R = 0{\cdot}041$, $wR = 0{\cdot}050$, $S = 1{\cdot}34$ for observed data. Max. $\Delta/\sigma = 0{\cdot}01$ in final cycle, max. residual density 0·3 e Å^{-3}. Atomic scattering factors and anomalous-dispersion corrections from *International Tables for X-ray Crystallography* (1974) and programs used were those of Enraf–Nonius (1982) *SDP*. Table 1 gives the atom coordinates and molecular dimensions are given in Table 2.* Fig. 1 (*ORTEP*; Johnson, 1976) shows the molecular structure and numbering scheme.

Related literature. Prior syntheses to related compounds are described in Weyenberg, Toporcer & Bey (1965), Nefedov, Manalcov & Petrov (1961) and Gilman & Atwell (1964).

Acknowledgment is made to the EPSCoR program of the National Science Foundation (CLB) and to the Petroleum Research Foundation, administered by the American Chemical Society (GLL) for partial support of this work.

---

* Lists of H-atom parameters, anisotropic thermal parameters and structure-factor amplitudes have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44804 (40 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

Enraf–Nonius (1982). *Structure Determination Package*. Enraf–Nonius, Delft, The Netherlands.

Gilman, H. & Atwell, W. H. (1964). *J. Am. Chem. Soc.* **86**, 2687–2693.

International Tables for X-ray Crystallography (1974). Vol. IV. Birmingham: Kynoch Press. (Present distributor Kluwer Academic Publishers, Dordrecht.)

Johnson, C. K. (1976). *ORTEP*II. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.

Main, P., Fiske, S. J., Hull, S. E., Lessinger, L., Germain, G., Declercq, J.-P. & Woolfson, M. M. (1982). *MULTAN*11/82. *A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data*. Univs. of York, England, and Louvain, Belgium.

Nefedov, O. M., Manalcov, M. N. & Petrov, A. D. (1961). *Izv. Akad. Nauk SSSR Otd. Khim. Nauk*, p. 1717.

Weyenberg, D. R., Toporcer, L. H. & Bey, A. E. (1965). *J. Org. Chem.* **30**, 4096–4101.

Acta Cryst. (1988). C**44**, 1148–1150

trans-1,1,2,5-Tetraphenylsilacyclopentane

Charles L. Barnes, Gerald L. Larson* and Yuh S. Lee

Department of Chemistry, University of Puerto Rico, Rio Piedras, PR 00931, *USA*

(*Received* 20 *November* 1987; *accepted* 23 *February* 1988)

Abstract. $C_{28}H_{26}Si$, $M_r = 390{\cdot}6$, triclinic, $P\bar{1}$, $a = 12{\cdot}818\,(4)$, $b = 17{\cdot}441\,(4)$, $c = 10{\cdot}8236\,(14)$ Å, $\alpha = 107{\cdot}09\,(6)$, $\beta = 75{\cdot}84\,(9)$, $\gamma = 103{\cdot}32\,(8)°$, $V = 2210\,(2)$ Å^3, $Z = 4$, $D_x = 1{\cdot}174$ g cm^{-3}, λ(Mo $K\bar{\alpha}$) = 0·71073 Å, $\mu = 1{\cdot}12$ cm^{-1}, $F(000) = 832$, $T = 297$ K, $R = 0{\cdot}043$ for 4111 observations (of 6924 unique data). In both independent molecules the five-membered ring assumes a distorted envelope conformation with one of the methylene C atoms displaced from the mean plane of the other four atoms. The degree of distortion from pure envelope conformation and the disposition of the phenyl rings differs between the molecules.

Experimental. Colorless crystal, dimensions 0·30 × 0·35 × 0·45 mm, mounted on a glass fiber, space group from successful refinement of centrosymmetric model, cell dimensions from setting angles for 24 reflections

---

* Current address: Petrarch Systems, Bartram Road, Bristol, PA 19007, USA.

0108-2701/88/061148-03$03.00

Table 1. *Fractional atomic coordinates and equivalent isotropic thermal parameters*

	x	y	z	B_{eq}(Å^2)
SiA	0·14656 (7)	0·08356 (5)	0·73385 (9)	3·63 (2)
C2A	0·0165 (2)	0·0124 (2)	0·7816 (3)	3·85 (8)
C3A	−0·0675 (3)	0·0425 (2)	0·7364 (4)	5·6 (1)
C4A	−0·0489 (3)	0·1350 (2)	0·7854 (4)	6·0 (1)
C5A	0·0734 (3)	0·1686 (2)	0·7381 (3)	4·26 (8)
C6A	0·0147 (2)	−0·0784 (2)	0·7429 (3)	3·61 (8)
C7A	0·0744 (2)	−0·1114 (2)	0·8004 (3)	4·18 (8)
C8A	0·0733 (3)	−0·1945 (2)	0·7693 (3)	4·90 (9)
C9A	0·0107 (3)	−0·2469 (2)	0·6827 (4)	5·4 (1)
C10A	−0·0489 (3)	−0·2156 (2)	0·6259 (4)	5·9 (1)
C11A	−0·0471 (3)	−0·1324 (2)	0·6549 (3)	4·78 (9)
C12A	0·1080 (2)	0·2540 (2)	0·8165 (3)	4·23 (8)
C13A	0·1464 (3)	0·3174 (2)	0·7559 (4)	5·5 (1)
C14A	0·1745 (4)	0·3971 (2)	0·8291 (5)	7·7 (1)
C15A	0·1628 (4)	0·4131 (3)	0·9627 (5)	8·8 (2)
C16A	0·1257 (4)	0·3507 (3)	1·0245 (4)	8·4 (1)
C17A	0·0988 (3)	0·2726 (2)	0·9520 (4)	6·2 (1)
C18A	0·2441 (2)	0·1073 (2)	0·8488 (3)	3·58 (8)
C19A	0·3558 (3)	0·1322 (2)	0·8064 (3)	4·54 (9)
C20A	0·4299 (3)	0·1486 (2)	0·8889 (4)	5·4 (1)
C21A	0·3947 (3)	0·1399 (2)	1·0155 (4)	5·4 (1)
C22A	0·2846 (3)	0·1160 (2)	1·0606 (3)	5·2 (1)
C23A	0·2110 (3)	0·1011 (2)	0·9786 (3)	4·44 (9)
C24A	0·2268 (2)	0·0473 (2)	0·5643 (3)	3·82 (8)
C25A	0·2787 (3)	0·1031 (2)	0·4895 (3)	4·36 (9)
C26A	0·3496 (3)	0·0816 (2)	0·3720 (3)	5·3 (1)
C27A	0·3697 (3)	0·0026 (2)	0·3239 (4)	6·0 (1)
C28A	0·3176 (3)	−0·0537 (2)	0·3939 (4)	6·6 (1)
C29A	0·2473 (3)	−0·0318 (2)	0·5130 (4)	5·4 (1)
SiB	0·62863 (8)	0·32890 (5)	0·4185 (1)	4·36 (2)
C2B	0·5810 (3)	0·2939 (2)	0·5765 (3)	5·3 (1)
C3B	0·4761 (3)	0·3315 (2)	0·6436 (4)	6·5 (1)
C4B	0·4195 (3)	0·3332 (2)	0·5364 (4)	6·5 (1)
C5B	0·5039 (3)	0·3764 (2)	0·4403 (3)	4·82 (9)
C6B	0·6656 (3)	0·3074 (2)	0·6602 (3)	5·5 (1)
C7B	0·6517 (4)	0·3487 (2)	0·7930 (4)	7·1 (1)
C8B	0·7319 (4)	0·3604 (3)	0·8656 (4)	8·3 (1)
C9B	0·8289 (4)	0·3321 (3)	0·8086 (4)	7·9 (1)
C10B	0·8442 (3)	0·2910 (2)	0·6774 (4)	7·0 (1)
C11B	0·7643 (3)	0·2789 (2)	0·6060 (4)	6·1 (1)
C12B	0·4704 (3)	0·3808 (2)	0·3194 (4)	4·90 (9)
C13B	0·3802 (3)	0·3299 (2)	0·2713 (4)	6·9 (1)
C14B	0·3523 (4)	0·3371 (3)	0·1592 (5)	8·4 (1)
C15B	0·4119 (4)	0·3932 (3)	0·0936 (4)	8·3 (1)
C16B	0·5021 (4)	0·4430 (2)	0·1378 (4)	7·2 (1)
C17B	0·5306 (3)	0·4372 (2)	0·2499 (4)	5·8 (1)
C18B	0·7526 (3)	0·4104 (2)	0·4168 (3)	4·31 (8)
C19B	0·7547 (3)	0·4788 (2)	0·5231 (4)	5·6 (1)
C20B	0·8410 (3)	0·5426 (2)	0·5268 (4)	6·7 (1)
C21B	0·9283 (3)	0·5402 (2)	0·4242 (4)	7·0 (1)
C22B	0·9297 (3)	0·4736 (2)	0·3174 (4)	6·2 (1)
C23B	0·8424 (3)	0·4091 (2)	0·3141 (3)	5·03 (9)
C24B	0·6448 (2)	0·2413 (2)	0·2690 (3)	4·12 (8)
C25B	0·6364 (3)	0·1616 (2)	0·2776 (3)	4·77 (9)
C26B	0·6516 (3)	0·0982 (2)	0·1646 (4)	5·4 (1)
C27B	0·6770 (3)	0·1132 (2)	0·0432 (4)	5·8 (1)
C28B	0·6860 (3)	0·1911 (2)	0·0304 (4)	5·6 (1)
C29B	0·6691 (3)	0·2543 (2)	0·1432 (4)	5·15 (9)

Anisotropically refined atoms are given in the form of the isotropic equivalent displacement parameter defined as: $\frac{4}{3}[a^2B(1,1) + b^2B(2,2) + c^2B(3,3) + ab(\cos\gamma)B(1,2) + ac(\cos\beta)B(1,3) + bc(\cos\alpha)B(2,3)]$.

Table 2. *Bond lengths* (Å), *angles* (°) *and selected torsion angles* (°) *with e.s.d.'s in parentheses*

SiA–C2A	1·894 (3)	C21A–C22A	1·385 (5)
SiA–C5A	1·914 (4)	C22A–C23A	1·381 (5)
SiA–G18A	1·871 (3)	C24A–C25A	1·396 (5)
SiA–C24A	1·876 (3)	C24A–C29A	1·388 (5)
C2A–C3A	1·531 (6)	C25A–C26A	1·377 (4)
C2A–C6A	1·511 (4)	C26A–C27A	1·383 (5)
C3A–C4A	1·523 (4)	C27A–C28A	1·368 (6)
C4A–C5A	1·556 (4)	C28A–C29A	1·385 (5)
C5A–C12A	1·507 (4)	SiB–C2B	1·897 (4)
C6A–C7A	1·396 (5)	SiB–C5B	1·900 (4)
C6A–C11A	1·385 (4)	SiB–C18B	1·872 (3)
C7A–C8A	1·386 (5)	SiB–C24B	1·877 (3)
C8A–C9A	1·373 (5)	C2B–C3B	1·554 (5)
C9A–C10A	1·370 (6)	C2B–C6B	1·512 (6)
C10A–C11A	1·387 (5)	C3B–C4B	1·521 (7)
C12A–C13A	1·384 (5)	C4B–C5B	1·535 (5)
C12A–C17A	1·388 (5)	C5B–C12B	1·500 (6)
C13A–C14A	1·396 (5)	C6B–C7B	1·394 (5)
C14A–C15A	1·366 (7)	C6B–C11B	1·396 (6)
C15A–C16A	1·377 (7)	C7B–C8B	1·385 (7)
C16A–C17A	1·370 (5)	C8B–C9B	1·378 (7)
C18A–C19A	1·400 (4)	C9B–C10B	1·378 (5)
C18A–C23A	1·394 (5)	C10B–C11B	1·373 (7)
C19A–C20A	1·386 (5)	C12B–C13B	1·394 (5)
C20A–C21A	1·375 (5)	C12B–C17B	1·389 (5)
C13B–C14B	1·392 (8)	C21B–C22B	1·377 (5)
C14B–C15B	1·359 (7)	C22B–C23B	1·390 (4)
C15B–C16B	1·372 (6)	C24B–C25B	1·398 (5)
C16B–C17B	1·387 (7)	C24B–C29B	1·395 (5)
C18B–C19B	1·394 (4)	C25B–C26B	1·395 (4)
C18B–C23B	1·390 (4)	C26B–C27B	1·362 (6)
C19B–C20B	1·375 (5)	C27B–C28B	1·381 (6)
C20B–C21B	1·368 (5)	C28B–C29B	1·394 (4)

C2A–SiA–C5A	94·7 (1)	C12A–C13A–C14A	120·9 (4)
C2A–SiA–C18A	113·2 (1)	C13A–C14A–C15A	119·8 (4)
C2A–SiA–C24A	115·3 (1)	C14A–C15A–C16A	120·1 (4)
C5A–SiA–C18A	117·6 (1)	C15A–C16A–C17A	119·8 (4)
C5A–SiA–C24A	110·1 (2)	C12A–C17A–C16A	121·8 (4)
C18A–SiA–C24A	106·0 (1)	SiA–C18A–C19A	120·5 (2)
SiA–C2A–C3A	101·8 (2)	SiA–C18A–C23A	122·8 (2)
SiA–C2A–C6A	120·0 (2)	C19A–C18A–C23A	116·7 (3)
C3A–C2A–C6A	116·8 (2)	C18A–C19A–C20A	121·6 (3)
C2A–C3A–C4A	107·0 (3)	C19A–C20A–C21A	120·3 (3)
C3A–C4A–C5A	108·7 (3)	C20A–C21A–C22A	119·5 (4)
SiA–C5A–C4A	103·2 (2)	C21A–C22A–C23A	120·0 (3)
SiA–C5A–C12A	119·5 (3)	C18A–C23A–C22A	122·0 (3)
C4A–C5A–C12A	112·2 (2)	SiA–C24A–C25A	119·5 (2)
C2A–C6A–C7A	119·7 (3)	SiA–C24A–C29A	123·1 (3)
C2A–C6A–C11A	123·1 (3)	C25A–C24A–C29A	117·0 (3)
C7A–C6A–C11A	117·2 (3)	C24A–C25A–C26A	121·7 (3)
C6A–C7A–C8A	121·5 (3)	C25A–C26A–C27A	120·1 (3)
C7A–C8A–C9A	120·2 (4)	C26A–C27A–C28A	119·2 (3)
C8A–C9A–C10A	119·0 (3)	C27A–C28A–C29A	120·7 (3)
C9A–C10A–C11A	121·1 (3)	C24A–C29A–C28A	121·3 (3)
C6A–C11A–C10A	120·9 (4)	C2B–SiB–C5B	95·0 (2)
C5A–C12A–C13A	120·9 (3)	C2B–SiB–C18B	114·9 (2)
C5A–C12A–C17A	121·6 (3)	C2B–SiB–C24B	112·1 (1)
C13A–C12A–C17A	117·5 (3)	C5B–SiB–C18B	108·7 (1)
C5B–SiB–C24B	113·7 (2)	C12B–C13B–C14B	120·6 (4)
C18B–SiB–C24B	111·4 (1)	C13B–C14B–C15B	121·2 (4)
SiB–C2B–C3B	103·6 (3)	C14B–C15B–C16B	119·3 (5)
SiB–C2B–C6B	116·8 (2)	C15B–C16B–C17B	120·4 (4)
C3B–C2B–C6B	117·0 (3)	C12B–C17B–C16B	121·4 (3)
C2B–C3B–C4B	108·3 (3)	SiB–C18B–C19B	118·5 (2)
C3B–C4B–C5B	107·6 (3)	SiB–C18B–C23B	124·6 (2)
SiB–C5B–C4B	102·0 (3)	C19B–C18B–C23B	116·9 (3)
SiB–C5B–C12B	118·3 (2)	C18B–C19B–C20B	121·9 (3)
C4B–C5B–C12B	118·2 (3)	C19B–C20B–C21B	120·2 (3)
C2B–C6B–C7B	123·4 (4)	C20B–C21B–C22B	119·8 (3)
C2B–C6B–C11B	120·7 (3)	C21B C22B–C23B	119·9 (3)
C7B–C6B–C11B	116·0 (4)	C18B–C23B–C22B	121·3 (3)
C6B–C7B–C8B	121·4 (4)	SiB–C24B–C25B	122·4 (3)
C7B–C8B–C9B	121·1 (4)	SiB–C24B–C29B	120·4 (2)
C8B–C9B–C10B	118·4 (5)	C25B–C24B–C29B	117·2 (3)
C9B–C10B–C11B	120·4 (4)	C24B–C25B–C26B	121·1 (3)
C6B–C11B–C10B	122·7 (3)	C25B–C26B–C27B	120·3 (3)
C5B–C12B–C13B	123·0 (3)	C26B–C27B–C28B	120·5 (3)
C5B–C12B–C17B	119·9 (3)	C27B–C28B–C29B	119·3 (4)
C13B–C12B–C21B	117·2 (4)	C24B–C29B–C28B	121·7 (3)

C5A–SiA–C2A–C3A	24·4 (2)	C5B–SiB–C2B–C3B	−6·5 (3)
C2A–SiA–C5A–C4A	3·2 (2)	C2B–SiB–C5B–C4B	−20·9 (3)
SiA–C2A–C3A–C4A	−46·8 (3)	SiB–C2B–C3B–C4B	33·5 (3)
C2A–C3A–C4A–C5A	53·4 (4)	C2B–C3B–C4B–C5B	−53·1 (4)
C3A–C4A–C5A–SiA	−31·1 (3)	C3B–C4B–C5B–SiB	44·2 (3)

having $12 \leq \theta \leq 16°$. Data collection on Enraf–Nonius CAD-4 diffractometer, Mo $K\alpha$ radiation, graphite monochromator, ω–2θ scans at 2·4 to 16·5° min^{-1}. Data having $2\theta \leq 50°$, $-15 \leq h \leq 15$, $-20 \leq k \leq 20$, $0 \leq l \leq 12$ measured. Three standard reflections, $3\bar{2}\bar{9}$, $4\bar{2}7$, $86\bar{8}$, measured every 3600 s, maximum variation 3·1%. 6924 unique data, 4111 reflections with $I > 3\sigma(I)$ considered observed, corrected for background, Lorentz, polarization, no absorption or extinction correction applied. Solved by direct methods using *MULTAN*11/82 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1982) and Fourier methods. Full-matrix least-squares methods minimized $\sum w(F_o - F_c)^2$ with $w = 4F_o^2[\sigma^2(I) + (0{\cdot}04F_o^2)^2]^{-1}$. Non-H atoms refined with anisotropic thermal parameters, H atoms placed at calculated positions and not refined. Final $R = 0{\cdot}043$, $wR = 0{\cdot}059$, $S = 1{\cdot}94$, for observed data. Max. $\Delta/\sigma = 0{\cdot}02$ in final cycle, max. residual density $0{\cdot}2$ e Å^{-3}. Atomic scattering factors and anomalous-dispersion corrections from *International Tables for X-ray Crystallography* (1974) and programs used were those of Enraf–Nonius (1982) *SDP*. Table 1 gives the atom coordinates and molecular dimensions are given in Table 2.* Fig. 1 (*ORTEP*; Johnson, 1976) shows the molecular structure and numbering scheme.

Related literature. Prior syntheses to related compounds are described in Weyenberg, Toporcer & Bey (1965), Nefedov, Manalcov & Petrov (1961) and Gilman & Atwell (1964).

Acknowledgment is made to the EPSCoR program of the National Science Foundation (CLB) and to the Petroleum Research Foundation, administered by the American Chemical Society (GLL) for partial support of this work.

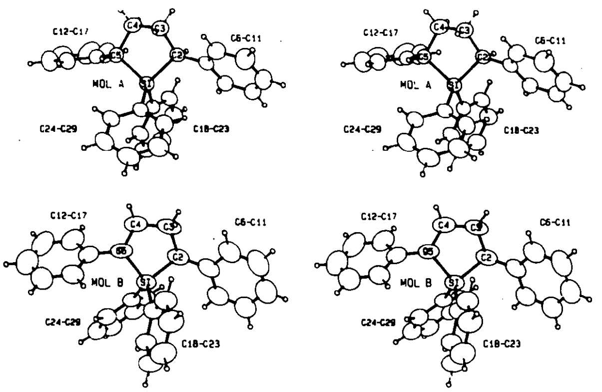

Fig. 1. Stereoviews of the two independent molecules. Thermal ellipsoids are drawn at the 50% probability level.

References

Enraf–Nonius (1982). *Structure Determination Package*. Enraf–Nonius, Delft, The Netherlands.

Gilman, H. & Atwell, W. H. (1964). *J. Am. Chem. Soc.* **86**, 2687–2693.

International Tables for X-ray Crystallography (1974). Vol. IV. Birmingham: Kynoch Press. (Present distributor Kluwer Academic Publishers, Dordrecht.)

Johnson, C. K. (1976). *ORTEP*II. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.

Main, P., Fiske, S. J., Hull, S. E., Lessinger, L., Germain, G., Declercq, J.-P. & Woolfson, M. M. (1982). *MULTAN*11/82. *A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data*. Univs. of York, England, and Louvain, Belgium.

Nefedov, O. M., Manalcov, M. N. & Petrov, A. D. (1961). *Izv. Akad. Nauk SSSR Otd. Khim. Nauk*, p. 1717.

Weyenberg, D. R., Toporcer, L. H. & Bey, A. E. (1965). *J. Org. Chem.* **30**, 4096–4101.

---

* Lists of H-atom parameters, anisotropic thermal parameters and structure-factor amplitudes have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44805 (75 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Acta Cryst. (1988). C**44**, 1150–1152

Ethyl 1-Trifluoromethylindolizine-3-carboxylate

By R. G. Pritchard

Department of Chemistry, University of Manchester Institute of Science and Technology, PO Box 88, Manchester M60 1QD, England

(*Received* 18 *December* 1987; *accepted* 17 *February* 1988)

Abstract. $C_{12}H_{10}F_3NO_2$, $M_r = 257{\cdot}2$, triclinic, $P\bar{1}$, $a = 4{\cdot}530\,(2)$, $b = 8{\cdot}834\,(4)$, $c = 14{\cdot}320\,(5)$ Å, $\alpha = 84{\cdot}85\,(2)$, $\beta = 87{\cdot}17\,(2)$, $\gamma = 84{\cdot}66\,(2)°$, $V = 567{\cdot}8$ Å^3, $Z = 2$, $D_x = 1{\cdot}50$ Mg m^{-3}, $F(000) = 264$, λ(Mo $K\alpha$) = 0·71069 Å, $\mu = 0{\cdot}091$ mm^{-1}, $T = 293$ K, $R = 0{\cdot}074$ for 1434 unique reflexions $[F > 3\sigma(F)]$. The molecules are stacked within the crystal so that neighbouring five- and six-membered rings overlap $[C(2)\cdots C(8^{i})$ 3·47(1) Å; (i) $1+x, y, z]$. A short C—C bond [1·437 (5) Å] linking the indolizine ring and the coplanar carboxy substituent suggests that the title compound has a dipole involving the ring N and carboxy O.

Experimental. The title compound was prepared by a 1,3-dipolar cycloaddition reaction of pyridinium ethoxycarbonylmethylide with 3,3,3-trifluoropropyne and recrystallized from a mixture of ethanol and methylene chloride.

Crystal dimensions 0·5 × 0·3 × 0·3 mm, Enraf–Nonius CAD-4 diffractometer, graphite-monochromatized Mo $K\alpha$ radiation, unit-cell dimensions from

0108-2701/88/061150-03$03.00