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Diethyl *trans*-1,2-dicyanoethene-1,2-dicarboxylate at *ca* 150 K

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In the title planar centrosymmetric molecule, $C_{10}H_{10}N_2O_4$, multiple-bond character remains essentially localized: C—O/N = 1.202 (3) and 1.143 (3) Å, and central C═C 1.344 (4) Å.

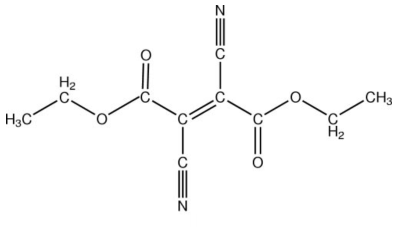
Key indicators

Single-crystal X-ray study
T = 150 K
Mean σ(C–C) = 0.004 Å
R factor = 0.085
wR factor = 0.142
Data-to-parameter ratio = 13.8

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

Comment

Tetracyanoethylene (TCNE) is widely involved as a dienophile acceptor in the formation of diverse charge-transfer adducts, with numerous structural studies of such species available, while various derivative species such as the title compound, (I), are employed in similar or precursive roles. The dimethyl radical anion analogue of (I) has been structurally characterized in its decamethylchromocenium 1:1 adduct (Kaul *et al.*, 2000), and also as the free ester (Hall *et al.*, 1982). [Coordinates for the latter have not been deposited as yet in the Cambridge Structural Database (Version 5.25; Allen, 2002).] In order to provide a modern precise determination of the uncomplexed species as a baseline comparator for a series of organometallic adducts currently under study by us, we saw fit to determine the structure of (I), used as the precursor in our syntheses.



(I)

One half of the centrosymmetric molecule (Fig. 1), necessarily '*trans*' about the inversion centre at the mid-point of the central bond, comprises the asymmetric unit of the structure of (I) in space group $P2_1/c$. Bond lengths and angles are generally in agreement with those of the dimethyl analogue (Hall *et al.*, 1982), albeit more precise, confirming the comparisons made with the radical anion form of the latter (Kaul *et al.*, 2000). The non-H-atom skeleton of the molecule is close to planar [χ^2 9.3 × 10^3; atom deviations (Å): C1 −0.011 (3), C11 0.008 (3), C2 −0.031 (3), C3 −0.023 (3), C4 0.196 (4), N11 0.007 (3), O2 0.023 (2) and O3 −0.078 (2)], despite the highly localized multiple bonds in the cyanide, carbonyl and olefin components. Bond distances and angles are generally as expected, the asymmetries in the latter about the trigonal C1 and C2 generally reflecting the electron-pair repulsion effects of the associated double bonds. The planar molecules pack staggered in a herringbone fashion in projection down a (Fig. 2).

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Experimental

The title compound, (I), was synthesized according to a literature method and identified by comparison with reported IR and NMR data (Ireland *et al.*, 1976; Ireland & Pizey, 1972). Suitable crystals were obtained from a dichloromethane–hexane mixture.

Crystal data

$C_{10}H_{10}N_2O_4$
$M_r = 222.2$
Monoclinic, $P2_1/c$
$a = 10.503\ (7)$ Å
$b = 4.436\ (3)$ Å
$c = 12.571\ (8)$ Å
$\beta = 111.161\ (8)°$
$V = 546.2\ (6)$ Å^3
$Z = 2$
$D_x = 1.351$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 1359 reflections
$\theta = 2.8–27.0°$
$\mu = 0.11$ mm^{-1}
$T = 150\ (2)$ K
Lath, colourless
$0.65 \times 0.15 \times 0.03$ mm

Data collection

Bruker SMART CCD diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{\min} = 0.814$, $T_{\max} = 0.999$
4840 measured reflections
1281 independent reflections
913 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.043$
$\theta_{\max} = 28.5°$
$h = -13 \rightarrow 13$
$k = -5 \rightarrow 5$
$l = -16 \rightarrow 15$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.055$
$wR(F^2) = 0.142$
$S = 1.10$
1281 reflections
93 parameters
All H-atom parameters refined
$w = 1/[\sigma^2(F^2) + 1.2F^2 + 0.0005F^4]$
$(\Delta/\sigma)_{\max} = 0.011$
$\Delta\rho_{\max} = 0.43$ e Å^{-3}
$\Delta\rho_{\min} = -0.33$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

C1—C11	1.447 (3)	C11—N11	1.143 (3)
C1—C2	1.523 (4)	C2—O2	1.202 (3)
C1—C1$^{\rm i}$	1.344 (4)	C2—O3	1.318 (3)
C11—C1—C2	115.5 (2)	C2—O3—C3	115.88 (19)
C11—C1—C1$^{\rm i}$	122.3 (2)	O3—C3—C4	106.8 (2)
C2—C1—C1$^{\rm i}$	122.2 (2)		

Symmetry code: (i) $-x, 1-y, 1-z$.

H atoms were located in difference Fourier maps and placed at idealized positions (C—H = 0.95 Å). Their positional and displacement parameters were then refined.

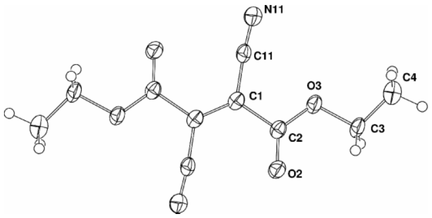

Figure 1
The molecular structure of (I), showing the atom-numbering scheme. and 50% probability displacement ellipsoids for the non-H atoms.

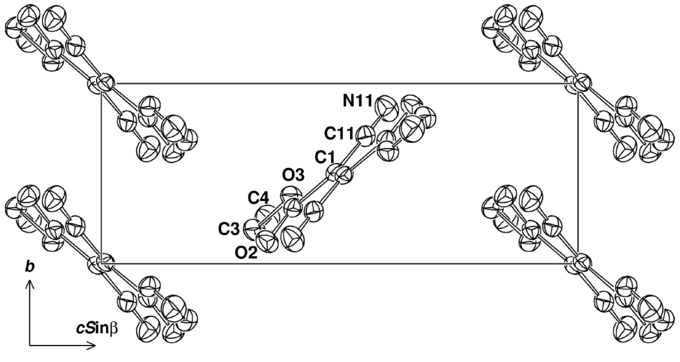

Figure 2
The unit-cell contents, projected down *a*. H atoms have been omitted.

Data collection: *SMART* (Siemens, 1995); cell refinement: *SAINT* (Siemens, 1995); data reduction: *SAINT*; program(s) used to solve structure: *Xtal*3.5 (Hall *et al.*, 1995); program(s) used to refine structure: *Xtal*3.5 *CRYLSQ*; molecular graphics: *Xtal*3.5; software used to prepare material for publication: *Xtal*3.5 *CIFIO*.

References

Allen, F. H. (2002). *Acta Cryst.* B**58**, 380–388.
Hall, H. K. Jr, Reineke, K. E., Ried, J. H., Sentman, R. C. & Miller, D. (1982). *J. Polym. Sci. Polym. Chem. Ed.* **20**, 361–372.
Hall, S. R., King, G. S. D. & Stewart, J. M. (1995). Editors. *Xtal*3.5 *Users Manual.* University of Western Australia, Perth: Lamb.
Ireland, C. J., Jones, K., Pizey, J. S. & Johnson, S. (1976). *Synth. Commun.* **6**, 185–191.
Ireland, C. J. & Pizey, J. S. (1972). *J. Chem. Soc. Chem. Commun.* pp. 4–5.
Kaul, B. B., Sommer, R. D., Noll, B. C. & Yee, G. T. (2000). *Inorg. Chem.* **37**, 865–868.
Sheldrick, G M. (1996). *SADABS*. University of Göttingen, Germany.
Siemens (1995). *SMART* and *SAINT*. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.

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