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Key indicators

Single-crystal X-ray study
 $T = 293\text{ K}$
Mean $\sigma(\text{C}-\text{C}) = 0.009\text{ \AA}$
 R factor = 0.046
 wR factor = 0.166
Data-to-parameter ratio = 8.8For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.**(1*E*,3*E*)-4-(Tetra-*O*-acetyl-*D*-arabino-tetritol-1-yl)-
1-(4-tolyl)-1,2-diaza-1,3-butadiene**

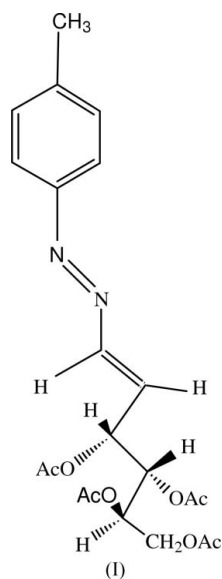
In the title compound, $\text{C}_{21}\text{H}_{26}\text{N}_2\text{O}_8$, the configurations of the three chiral centres are known from the synthesis, corresponding to a *D*-arabino configuration. Both double bonds show the *E* configuration in the solid state. Packing of the molecules is governed by normal van der Waals contacts.

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Comment

Following current work on asymmetric synthesis from carbohydrates, Avalos *et al.* (1995) developed a general synthesis of sugar 1,2-diaza-1,3-butadienes. The title compound, (I), was prepared by treatment of *D*-mannose with aryl hydrazines, and the corresponding aryl hydrazones were readily obtained. Further conventional acetylation and thermal 1,4-elimination gave 1-aryl-1,2-diaza-1,3-butadienes. In all cases, aryl diaza-butadienes were coloured crystalline compounds and could be stored for long periods without appreciable decomposition.



An X-ray investigation of compound (I) was carried out in order to elucidate unequivocally the molecular conformation of (I) in the solid state. A perspective view of (I) with the atom-numbering scheme is shown in Fig. 1.

Principial geometric parameters are given in Table 1. Both double bonds exist in the *E* configuration in the solid state.

The *arabino* chain (C4/C41/C42/C43) is planar, with the terminal atom C44 having a maximum deviation from the least-squares plane of 0.017 (5) Å. The configurations of the chiral centres C41, C42 and C43 are *R*, *S* and *R*, respectively. The packing of the molecules is governed by normal van der Waals contacts.

Experimental

The title compound was synthesized from D-mannose (4-methylphenyl) hydrazone, according to the procedure of Avalos *et al.* (1995). Compound (I) was recrystallized from ethanol–water.

Crystal data

| | |
|--------------------------------|---|
| $C_{21}H_{26}N_2O_8$ | $Z = 4$ |
| $M_r = 434.44$ | $D_x = 1.205 \text{ Mg m}^{-3}$ |
| Orthorhombic, $P2_12_12_1$ | Mo $K\alpha$ radiation |
| $a = 14.065 (1) \text{ \AA}$ | $\mu = 0.09 \text{ mm}^{-1}$ |
| $b = 30.189 (2) \text{ \AA}$ | $T = 293 (2) \text{ K}$ |
| $c = 5.6414 (3) \text{ \AA}$ | Prism, light brown |
| $V = 2395.4 (3) \text{ \AA}^3$ | $0.32 \times 0.28 \times 0.16 \text{ mm}$ |

Data collection

| | |
|-----------------------------------|--|
| Enraf–Nonius CAD-4 diffractometer | 1196 reflections with $I > 2\sigma(I)$ |
| $\omega/2\theta$ scans | $R_{\text{int}} = 0.027$ |
| Absorption correction: none | $\theta_{\text{max}} = 25^\circ$ |
| 5210 measured reflections | 3 standard reflections |
| 2458 independent reflections | frequency: 60 min |
| | intensity decay: 6% |

Refinement

| | |
|---------------------------------|--|
| Refinement on F^2 | $w = 1/[\sigma^2(F_o^2) + (0.0855P)^2 + 0.3134P]$ |
| $R[F^2 > 2\sigma(F^2)] = 0.046$ | where $P = (F_o^2 + 2F_c^2)/3$ |
| $wR(F^2) = 0.166$ | $(\Delta/\sigma)_{\text{max}} = 0.009$ |
| $S = 0.97$ | $\Delta\rho_{\text{max}} = 0.22 \text{ e \AA}^{-3}$ |
| 2458 reflections | $\Delta\rho_{\text{min}} = -0.17 \text{ e \AA}^{-3}$ |
| 280 parameters | |

Table 1

Selected geometric parameters (\AA , $^\circ$).

| | | | |
|-----------|-----------|-----------|-----------|
| N2–N1 | 1.254 (6) | C41–C4 | 1.493 (7) |
| N2–C3 | 1.404 (7) | C4–C3 | 1.318 (8) |
| N1–C11 | 1.427 (7) | | |
| N1–N2–C3 | 112.2 (5) | C3–C4–C41 | 123.8 (5) |
| N2–N1–C11 | 113.1 (5) | C4–C3–N2 | 119.3 (5) |

H atoms were refined using a riding model, with C–H distances in the range 0.93–0.98 \AA and with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ or $1.5U_{\text{eq}}(\text{C}_{\text{methyl}})$. In the absence of significant anomalous scatterers, Friedel pairs were merged. The absolute configuration was assigned by reference to the known chirality of the starting material used in the synthesis.

Data collection: *CAD-4 EXPRESS* (Enraf–Nonius, 1994); cell refinement: *CAD-4 EXPRESS*; data reduction: *XCAD4* (Harms & Wocadlo, 1995); program(s) used to solve structure: *SIR2002* (Burla *et al.*, 2003); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEPII* (Johnson, 1976); software

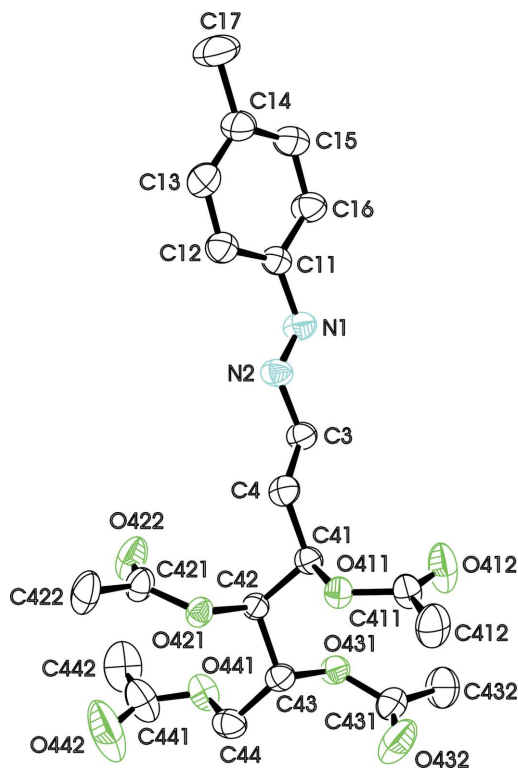


Figure 1

A view of the molecular structure of (I), showing the atomic numbering scheme. Displacement ellipsoids are drawn at the 30% probability level. For clarity, only the most important H atoms are shown.

used to prepare material for publication: *SHELXL97* and *PARST* (Nardelli, 1995).

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