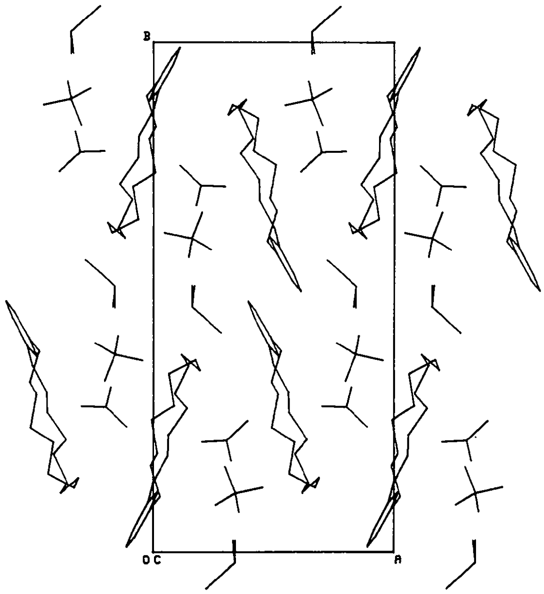

Fig. 2. Unit-cell contents; H atoms omitted for clarity.

torsion angles in the macrocycle and selected non-bonded distances and angles are in Table 2. Atom numbering in the macrocycle is shown in Fig. 1(*a*); guanidinium [C(22)–N(25)], perchlorate [Cl(26)–O(30)] and deuterochloroform [C(31)–Cl(34)] are not shown. A stereoscopic view of the complex, showing hydrogen bonds, is in Fig. 1(*b*). Unit-cell contents are in Fig. 2.

Related literature. In the 1:2 complex of 18-crown-6 with guanidinium nitrate (Bandy, Truter, Wingfield & Lamb, 1981) each guanidinium cation interacts with one crown molecule and one nitrate anion. Larger macrocycles are able to encapsulate the guanidinium cation completely, as shown for 27- and 30-membered benzo- (de Boer, Uiterwijk, Geevers, Harkema & Reinhoudt, 1983) and 2,6-pyrido-crown ethers (Uiterwijk *et al.*, 1986).

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Structure of 3-Amino-1*H*-indazole–Di-*tert*-butyl Fumarate Adduct

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Abstract. $C_{19}H_{25}N_3O_4$, $M_r = 359{\cdot}4$, monoclinic, $P2_1/n$, $a = 11{\cdot}197\ (2)$, $b = 11{\cdot}257\ (2)$, $c = 16{\cdot}407\ (3)$ Å, $\beta = 100{\cdot}59\ (2)°$, $V = 2033\ (1)$ Å^3, $Z = 4$, $D_m = 1{\cdot}20\ (5)$, $D_x = 1{\cdot}174$ g cm^{-3}, $\lambda(\text{Mo }K\alpha) = 0{\cdot}71069$ Å, $\mu = 0{\cdot}897$ cm^{-1}, $F(000) = 768$, $T = 293\ (1)$ K, final $R = 0{\cdot}049$ for 1560 observed reflexions. The adduct crystallizes as di-*tert*-butyl 2-(1*H*-indazol-3-yl)aminofumarate. The indazole part of the molecule is planar to within 0·016 (4) Å. The H atom of the amino group is involved in an intramolecular N—H···O hydrogen bond of 2·685 (5) Å, thus keeping part of the molecule planar. The bond lengths and angles agree with expected values.

Experimental. Single crystals grown from ethanol solution of a mixture of 3-aminoindazole and di-*tert*-butyl fumarate. Density measured by flotation in chlorobenzene/CCl_4; transparent plates, 0·20 × 0·10 × 0·05 mm; Enraf–Nonius CAD-4 diffractometer, Mo $K\alpha$ radiation (graphite monochromator), lattice parameters from 25 reflexions ($6 < \theta < 10°$); $\omega/2\theta$ scans, variable scan rate, max. scan time 60 s,

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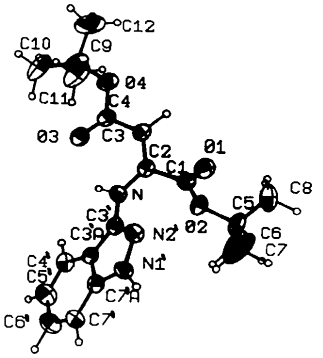
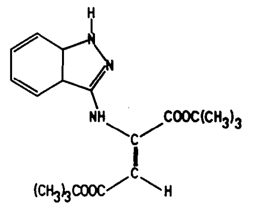


Fig. 1. *ORTEP* (Johnson, 1965) view of the molecule with the atom-numbering scheme. Thermal ellipsoids are drawn at the 50% probability level.

scan width (2θ) $(0{\cdot}8 + 0{\cdot}3\tan\theta)^\circ$, aperture $(2{\cdot}5 + 0{\cdot}9\tan\theta)$ mm, background measured for 1/4 of scan time at each scan limit, max. $(\sin\theta)/\lambda$ in intensity measurements: 0·60 Å^{-1}. Three reflexions monitored at intervals of 350 reflexions, no significant intensity decline (−1·0%); data corrected for intensity variation and Lp effects, but absorption ignored; 7671 ($\pm h$, $+k$, $\pm l$) measured reflexions, 3285 unique [$R_{\rm int}(I) = 0{\cdot}015$ for 7192 contributing reflexions], 1560 observed (h −13/12, k 0/12, l 0/19) with $I > 2\sigma(I)$, $\sigma(I)$ based on counting statistics. Structure solution by direct methods with *SHELXS*86 (Sheldrick, 1985), full-matrix least-squares refinement minimizing $\sum w(\Delta F)^2$ with *SHELX*76 (Sheldrick, 1976); final R and wR values: 0·049 and 0·048 with $w = 0{\cdot}896/[\sigma^2(F_o) + 0{\cdot}0011F_o^2]$; H atoms found from a difference synthesis and included in the refinement at calculated positions [d(C—H) = 1·08 Å], a common isotropic temperature factor U for H atoms was fixed at 0·12 Å^2. $\Delta\rho$ within 0·09 and −0·09 e Å^{-3} in final difference map. At convergence, max. and mean values of Δ/σ: 0·13 and 0·02. Scattering factors for O, N and C and anomalous-dispersion corrections from *International Tables for X-ray Crystallography* (1974), for H from Stewart, Davidson & Simpson (1965). All calculations performed on the DEC 1091 computer at University Computer Centre, Ljubljana. *XRAY*76 (Stewart, Machin, Dickinson, Ammon, Heck & Flack, 1976) was used for data reduction and interpretation.



The molecular structure with atom-numbering scheme is shown in Fig. 1. Atomic coordinates are

Table 1. *Fractional atomic coordinates* (×10^4) *and equivalent isotropic temperature factors* (Å^2 × 10^3) (*Hamilton*, 1959)

	x	y	z	$U_{\rm eq}$
O(1)	2747 (3)	3446 (3)	282 (2)	78 (2)
O(2)	1792 (3)	2227 (3)	1046 (2)	61 (2)
O(3)	811 (4)	90 (3)	−1614 (2)	78 (3)
O(4)	2748 (3)	108 (3)	−1819 (2)	76 (2)
N	308 (4)	1622 (3)	−469 (2)	55 (2)
N(1′)	−1259 (3)	4140 (3)	025 (2)	60 (3)
N(2′)	−216 (3)	3581 (3)	−152 (2)	58 (3)
C(1)	2087 (4)	2630 (4)	361 (3)	53 (3)
C(2)	1524 (4)	1829 (4)	−354 (3)	49 (3)
C(3)	2292 (4)	1299 (4)	−782 (3)	53 (3)
C(4)	1852 (5)	436 (4)	−1439 (3)	57 (3)
C(5)	2186 (6)	2793 (5)	1868 (3)	78 (4)
C(6)	1639 (8)	1989 (7)	2433 (4)	153 (7)
C(7)	1639 (8)	3983 (6)	1861 (4)	155 (7)
C(8)	3503 (7)	2809 (10)	2085 (4)	190 (9)
C(9)	2557 (7)	−800 (5)	−2492 (4)	91 (4)
C(10)	1564 (7)	−432 (5)	−3193 (3)	118 (5)
C(11)	2282 (7)	−1963 (5)	−2121 (4)	128 (6)
C(12)	3766 (7)	−801 (8)	−2754 (5)	187 (9)
C(3′)	−534 (4)	2463 (4)	−307 (3)	50 (3)
C(3′*A*)	−1775 (4)	2270 (4)	−312 (3)	49 (3)
C(4′)	−2579 (5)	1296 (4)	−451 (3)	65 (3)
C(5′)	−3781 (5)	1480 (5)	−416 (4)	85 (4)
C(6′)	−4217 (5)	2624 (6)	−243 (4)	85 (4)
C(7′)	−3451 (4)	3586 (5)	−111 (3)	67 (4)
C(7′*A*)	−2229 (4)	3393 (4)	−139 (3)	53 (3)

Table 2. *Bond distances* (Å) *and angles* (°) *with e.s.d.'s in parentheses*

O(1)—C(1)	1·201 (5)	O(2)—C(1)	1·309 (6)
O(2)—C(5)	1·483 (6)	O(3)—C(4)	1·212 (7)
O(4)—C(9)	1·491 (7)	O(4)—C(4)	1·327 (7)
N—C(2)	1·360 (6)	N—C(3)	1·396 (6)
C(1)—C(2)	1·521 (6)	C(2)—C(3)	1·346 (7)
C(3)—C(4)	1·467 (7)	C(5)—C(6)	1·504 (10)
C(5)—C(7)	1·472 (9)	C(5)—C(8)	1·452 (10)
C(9)—C(10)	1·504 (9)	C(9)—C(11)	1·499 (9)
C(9)—C(12)	1·494 (12)	N(1′)—N(2′)	1·376 (5)
N(1′)—C(7′*A*)	1·359 (6)	N(2′)—C(3′)	1·320 (6)
C(3′)—C(3′*A*)	1·405 (7)	C(3′*A*)—C(4′)	1·410 (7)
C(3′*A*)—C(7′*A*)	1·411 (7)	C(4′)—C(5′)	1·373 (8)
C(5′)—C(6′)	1·424 (9)	C(6′)—C(7′)	1·374 (8)
C(7′)—C(7′*A*)	1·395 (7)		
C(1)—O(2)—C(5)	123·5 (4)	C(4)—O(4)—C(9)	121·4 (4)
C(2)—N—C(3)	123·5 (3)	O(1)—C(1)—O(2)	127·9 (4)
O(1)—C(1)—C(2)	123·1 (5)	O(2)—C(1)—C(2)	108·8 (4)
N—C(2)—C(1)	118·1 (4)	N—C(2)—C(3)	124·8 (4)
C(1)—C(2)—C(3)	116·8 (4)	C(2)—C(3)—C(4)	121·1 (4)
O(4)—C(4)—O(3)	125·2 (4)	O(4)—C(4)—C(3)	110·4 (4)
O(3)—C(4)—C(3)	124·4 (5)	O(2)—C(5)—C(6)	102·4 (5)
O(2)—C(5)—C(7)	109·3 (4)	O(2)—C(5)—C(8)	110·3 (5)
C(6)—C(5)—C(7)	109·7 (6)	C(6)—C(5)—C(8)	111·5 (6)
C(7)—C(5)—C(8)	113·1 (7)	O(4)—C(9)—C(10)	111·3 (5)
O(4)—C(9)—C(11)	108·1 (5)	O(4)—C(9)—C(12)	101·5 (5)
C(10)—C(9)—C(11)	111·9 (6)	C(10)—C(9)—C(12)	111·2 (6)
C(11)—C(9)—C(12)	112·4 (6)	N(2′)—N(1′)—C(7′*A*)	112·0 (3)
N(1′)—N(2′)—C(3′)	105·0 (3)	N—C(3′)—N(2′)	121·0 (4)
N—C(3′)—C(3′*A*)	126·6 (4)	N(2′)—C(3′)—C(3′*A*)	112·3 (4)
C(3′)—C(3′*A*)—C(4′)	136·3 (5)	C(3′)—C(3′*A*)—C(7′*A*)	104·6 (4)
C(4′)—C(3′*A*)—C(7′*A*)	119·1 (4)	C(3′*A*)—C(4′)—C(5′)	118·5 (5)
C(4′)—C(5′)—C(6′)	121·3 (5)	C(5′)—C(6′)—C(7′)	121·2 (5)
C(6′)—C(7′)—C(7′*A*)	117·4 (5)	N(1′)—C(7′*A*)—C(3′*A*)	105·9 (4)
N(1′)—C(7′*A*)—C(7′)	131·5 (4)	C(3′*A*)—C(7′*A*)—C(7′)	122·6 (4)
Hydrogen bond			
O(3)···N	2·685 (5)	O(3)—H(1)—N	114·3 (4)
H(1)···O(3)	2·054 (6)		
H(1)—N	1·079 (5)		

listed in Table 1,* and bond lengths and angles are in Table 2.

Related literature. The crystal structure analysis was undertaken to assist in the characterization of the products of the reactions of dialkyl fumarates with amino heterocycles, in which the amino group is attached at the C atom adjacent to a ring N atom (Acheson & Elmore, 1978; Polanc & Colnar, 1984).

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* Lists of structure factors, anisotropic thermal parameters, H-atom coordinates, selected torsion angles and the results of mean-plane calculations have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44240 (14 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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A Product Predicted with the Felkin–Anh Model

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Abstract. (*Z*)-2-*O*-Benzyl-5,6-*O*-isopropylidene-*N*-methyl-3-*S*-phenyl-3-thio-D-*erythro*-hex-2-enonamide, $C_{23}H_{27}NO_5S$, $M_r = 429{\cdot}54$, monoclinic, $P2_1$, $a = 12{\cdot}524\,(4)$, $b = 7{\cdot}938\,(2)$, $c = 11{\cdot}933\,(2)$ Å, $\beta = 108{\cdot}82\,(2)°$, $V = 1122{\cdot}90\,(96)$ Å^3, $Z = 2$, $D_m = 1{\cdot}28$, $D_x = 1{\cdot}27$ Mg m^{-3}, $\lambda(\text{Mo}\,K\alpha) = 0{\cdot}71073$ Å, $\mu = 1{\cdot}795$ mm^{-1}, $F(000) = 456$, $T = 295$ K, $R = 0{\cdot}039$ for 1379 observed unique reflections. The stereochemistry of the title compound is that predicted with the Felkin–Anh model [Anh (1980). *Top. Curr. Chem.* **88**, 145–166]. Its absolute configuration is inferred from the known configuration of the glyceraldehyde moiety that is introduced into the molecule. Adjacent molecules unite by an intermolecular hydrogen bond between an alcohol function and a carbonyl group, the O···O distance being 2·785 (5) Å.

Experimental. Colorless crystal of dimensions 0·2 × 0·2 × 0·6 mm, D_m by flotation in hexane/methylene chloride. Syntex $P\bar{1}$ diffractometer with incident-beam monochromator, 15 centered reflections within $12{\cdot}5 \leq 2\theta \leq 25°$ used for determining lattice parameters. Absorption ignored; $(\sin\theta/\lambda)_{\max} = 0{\cdot}557$ Å^{-1}, range of *hkl*: $-13 \leq h \leq 13$, $0 \leq k \leq 8$, $-13 \leq l \leq 13$. Six standard reflections monitored every 100 reflections with random variation of 2·2% over data collection, θ–2θ scans of 1° min^{-1}, 3719 measured reflections, 1758 unique reflections, $R_{\text{int}} = 0{\cdot}039$, 1379 observed $[F_o > 3\sigma(F_o)]$.

Structure solved by direct methods with *MITHRIL* (Gilmore, 1983), *DIRDIF* (Beurskens, 1984) and Fourier procedures. H atoms located in difference maps; constrained to idealized positions with isotropic $B = 1{\cdot}2 \times B$ of bonded atom. H atoms of N and O(1)

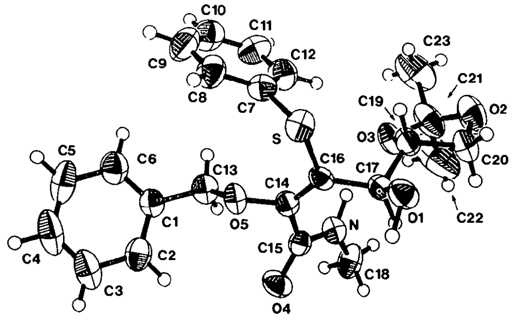

Fig. 1. A drawing of the molecule with thermal ellipsoids scaled at the 50% probability level.