

Table 2. Relevant bond distances (Å) and angles (°)

	A	B		A	B		A	B
Br—C(21)	1.89 (1)	1.91 (1)	C(5)—C(6)	1.50 (2)	1.50 (1)	C(13)—C(14)	1.49 (1)	1.53 (1)
O(1)—C(3)	1.44 (2)	1.40 (1)	C(6)—C(7)	1.50 (2)	1.50 (1)	C(13)—C(17)	1.52 (2)	1.53 (1)
O(1)—C(19)	1.33 (2)	1.41 (1)	C(7)—C(8)	1.53 (1)	1.53 (1)	C(13)—C(18)	1.54 (2)	1.53 (1)
C(1)—C(2)	1.42 (2)	1.40 (2)	C(8)—C(9)	1.53 (1)	1.54 (1)	C(14)—C(15)	1.49 (2)	1.53 (1)
C(1)—C(10)	1.35 (2)	1.40 (1)	C(8)—C(14)	1.53 (1)	1.50 (1)	C(15)—C(16)	1.56 (2)	1.57 (2)
C(2)—C(3)	1.38 (2)	1.35 (1)	C(9)—C(10)	1.53 (1)	1.51 (1)	C(16)—C(17)	1.53 (2)	1.49 (1)
C(3)—C(4)	1.34 (2)	1.36 (1)	C(9)—C(11)	1.53 (1)	1.54 (1)	C(17)—C(20)	1.33 (2)	1.30 (1)
C(4)—C(5)	1.35 (2)	1.37 (1)	C(11)—C(12)	1.56 (1)	1.54 (1)	C(20)—C(21)	1.32 (2)	1.32 (1)
C(5)—C(10)	1.44 (1)	1.43 (1)	C(12)—C(13)	1.51 (1)	1.52 (1)			
C(3)—O(1)—C(19)	118 (1)	116.2 (8)	C(9)—C(8)—C(14)	107.6 (8)	107.7 (7)	C(14)—C(13)—C(17)	100.1 (9)	99.9 (7)
C(2)—C(1)—C(10)	120 (1)	121.5 (9)	C(7)—C(8)—C(14)	111.6 (8)	113.3 (7)	C(14)—C(13)—C(18)	112.6 (9)	113.1 (8)
C(1)—C(2)—C(3)	117 (1)	119 (1)	C(8)—C(9)—C(10)	111.7 (8)	113.4 (7)	C(17)—C(13)—C(18)	107.6 (9)	105.6 (8)
O(1)—C(3)—C(4)	114 (1)	114.8 (9)	C(8)—C(9)—C(11)	111.7 (8)	109.7 (8)	C(8)—C(14)—C(15)	119.5 (9)	122.4 (8)
C(2)—C(3)—C(4)	124 (1)	121 (1)	C(10)—C(9)—C(11)	113.5 (8)	115.9 (7)	C(13)—C(14)—C(15)	106 (1)	103.9 (7)
O(1)—C(3)—C(2)	122 (1)	124.3 (9)	C(1)—C(10)—C(5)	119 (1)	117.3 (8)	C(8)—C(14)—C(13)	114.7 (9)	112.8 (7)
C(3)—C(4)—C(5)	119 (1)	122.7 (9)	C(5)—C(10)—C(9)	118.5 (9)	121.6 (7)	C(14)—C(15)—C(16)	104 (1)	103.5 (8)
C(4)—C(5)—C(10)	121 (1)	118.4 (8)	C(1)—C(10)—C(9)	122.8 (9)	120.6 (8)	C(15)—C(16)—C(17)	102 (1)	105.0 (9)
C(6)—C(5)—C(10)	122 (1)	119.8 (8)	C(9)—C(11)—C(12)	112.9 (9)	113.2 (7)	C(13)—C(17)—C(20)	128 (1)	125.2 (8)
C(4)—C(5)—C(6)	118 (1)	121.8 (8)	C(11)—C(12)—C(13)	109.9 (8)	110.2 (8)	C(13)—C(17)—C(16)	109.8 (9)	109.1 (8)
C(5)—C(6)—C(7)	116 (1)	114.8 (8)	C(12)—C(13)—C(14)	110.0 (9)	110.6 (8)	C(16)—C(17)—C(20)	122 (1)	125.7 (9)
C(6)—C(7)—C(8)	111.0 (9)	109.8 (7)	C(12)—C(13)—C(17)	116.9 (9)	116.7 (8)	C(17)—C(20)—C(21)	178 (1)	180 (1)
C(7)—C(8)—C(9)	108.8 (8)	110.3 (8)	C(12)—C(13)—C(18)	109.5 (8)	110.5 (8)	Br—C(21)—C(20)	121.0 (9)	122.2 (8)

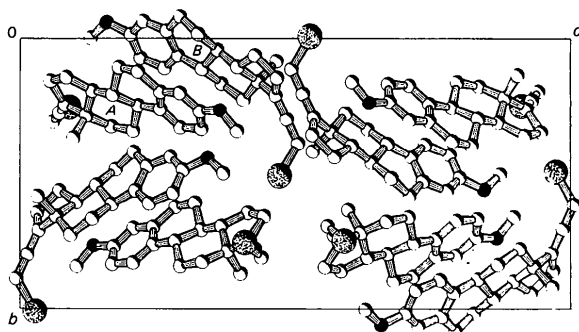


Fig. 2. Projection of the unit-cell contents down the *a* axis. The two independent molecules are indicated by *A* and *B* respectively.

pairs and are related by a pseudo-twofold axis running parallel to their medium axis of inertia.

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Structures of *tert*-Butyl 3-Nitroperbenzoate (I) and *tert*-Butyl 4-Nitroperbenzoate (II), $C_{11}H_{13}NO_5$

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Abstract. $M_r = 239.2$. (I): monoclinic, $P2_1/c$, $a = 8.462$ (4), $b = 17.408$ (10), $c = 8.279$ (4) Å, $\beta = 101.56$ (4)°, $V = 1194.8$ Å³, $Z = 4$, $D_m = 1.30$ (5), $D_x = 1.330$ Mg m⁻³, $\lambda(\text{Mo K}\alpha) = 0.71069$ Å, $\mu =$

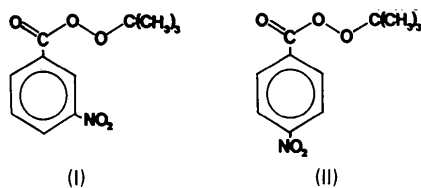
0.114 mm⁻¹, $F(000) = 504$, $T = 293$ (1) K, $R = 0.114$ for 1069 unique observed reflexions. (II): triclinic, $P\bar{1}$, $a = 6.892$ (3), $b = 8.077$ (4), $c = 11.970$ (6) Å, $\alpha = 106.94$ (4), $\beta = 102.11$ (4), $\gamma = 93.37$ (4)°, $V =$

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618.1 \AA^3 , $Z = 2$, $D_m = 1.27 (5)$, $D_x = 1.285 \text{ Mg m}^{-3}$, $\lambda(\text{Mo } K\alpha) = 0.71069 \text{ \AA}$, $\mu = 0.110 \text{ mm}^{-1}$, $F(000) = 252$, $T = 293 (1) \text{ K}$, $R = 0.096$ for 535 unique observed reflexions. The moderate accuracy of the structure determinations is due to considerable decay of the crystals during the data collection. The peroxycarboxyl groups are essentially planar with a torsion angle about the O—O bond of $129 (1)^\circ$ for (I) and $120 (2)^\circ$ for (II).

Introduction. Examples of crystal structure determinations of organic peroxy acids are few. The structure of the aliphatic peroxyelargonic acid was determined by Belitskus & Jeffrey (1965), and the aromatic 2-nitro- and 4-nitroperbenzoic acids were studied by Sax, Beurskens & Chu (1965) and Kim, Chu & Jeffrey (1970), respectively. The main reason for the lack of structural information on peroxy acids and their derivatives is their thermal and chemical instability and the decay of the crystals when exposed to X-rays. The crystals of the *tert*-butyl esters of 3-nitro- and of 4-nitroperbenzoic acid seemed to be sufficiently stable at room temperature to be used for X-ray study. However, in the course of the data collection considerable decomposition was observed, which affected the quality of the final results. The principal reason for determining the crystal structures of peroxy esters was to determine the geometry of the peroxy group and to make a comparison with the free peroxy acids. The crystals for analysis were kindly supplied by Professor B. Plesničar, Chemistry Department, E. Kardelj University, Ljubljana.



Experimental. Transparent plate crystals, covered by silicon grease and sealed in glass capillaries, $0.4 \times 0.4 \times 0.3$ (I) and $0.3 \times 0.3 \times 0.25$ mm (II). D_m by flotation in $\text{CCl}_4/\text{C}_6\text{H}_5\text{Cl}$. Enraf-Nonius CAD-4 automated four-circle diffractometer, graphite monochromator. Cell parameters by least squares from the 2θ values of 45 independent reflexions ($8 < \theta < 12^\circ$; $\text{Mo } K\alpha_1$, $\lambda = 0.70926 \text{ \AA}$). Monoclinic (I) and triclinic (II) systems derived from initial oscillation and Weissenberg photographs; space groups $P2_1/c$ (I) and $P\bar{1}$ (II) chosen after examination of systematic absences and distributions of $|E|$ values. $2\theta \leq 50^\circ$, $\omega/2\theta$ scans, variable scan rate (min. 2.5 , max. $20.1^\circ \text{ min}^{-1}$), maximum scan time 40 s, scan width (2θ) $(0.8 + 0.2 \tan\theta)^\circ$, aperture $(2.5 + 0.9 \tan\theta) \text{ mm}$, background taken as $\frac{1}{4}$ of the scan time at each of the scan limits, $\text{Mo } K\alpha$ radiation at 40 kV and 20 mA. Three reference reflexions monitored at intervals of 120 reflexions

revealed intensity deteriorations of 29% (I) and 43% (II); data corrected for intensity variation and Lp effects, but absorption ignored. 1845 unique reflexions for (I) and 1735 for (II), 1069 (index range $h-10/9$, $k0/20$, $l0/9$) (I) and 535 (index range $h-7/6$, $k-9/8$, $l0/12$) (II) considered observed with $I > 2\sigma(I)$ and retained for use in the structure analyses; $\sigma(I)$ based on counting statistics. Structures solved by *MULTAN*80 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980). Each structure refined isotropically and anisotropically by full-matrix least squares using *F* with *SHELX*76 (Sheldrick, 1976); at convergence $R = 0.114$ for (I), $R = 0.096$ for (II). Unit weights. H atoms included in the refinement at calculated positions [$d(\text{C}-\text{H}) = 1.09 \text{ \AA}$]; fixed isotropic temperature factors (*U*) of 0.09 \AA^2 for H of the *tert*-butyl group and

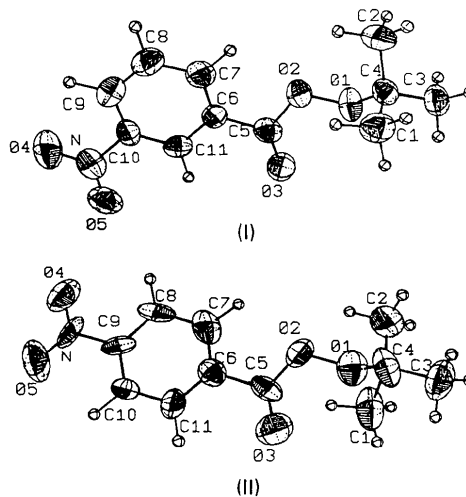


Fig. 1. ORTEP (Johnson, 1965) views of the molecules with the labelling of the atoms. Thermal ellipsoids are drawn at the 50% probability level.

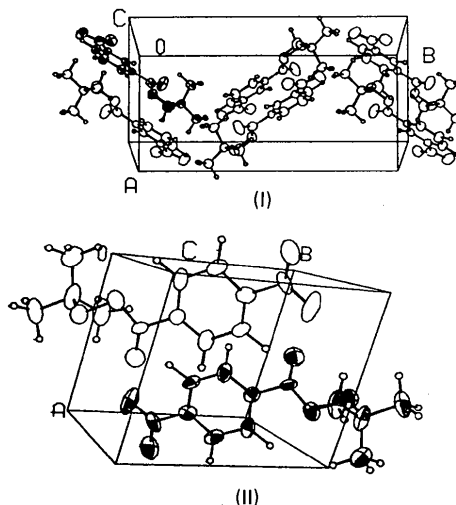


Fig. 2. Packing of the molecules in the unit cells.

0.08 Å² for aromatic H atoms. No features greater than 0.3 (I) and 0.1 e Å⁻³ (II) in the final $\Delta\rho$ map. $(\Delta/\sigma)_{\max}$ 1.10 (I), 0.29 (II). Scattering factors and anomalous-dispersion corrections from *International Tables for X-ray Crystallography* (1974). All calculations performed on the DEC 1091 computer at RCU Ljubljana.

Table 1. *Final fractional coordinates* ($\times 10^4$) *and equivalent isotropic temperature factors* ($\times 10^3$) (Hamilton, 1959)

	x	y	z	$U_{eq}(\text{Å}^2)$
Compound (I)				
O(1)	4956 (7)	1249 (3)	2106 (7)	69 (4)
O(2)	3913 (7)	595 (3)	2231 (7)	69 (4)
O(3)	3765 (10)	1048 (4)	4713 (8)	109 (6)
O(4)	385 (9)	-1810 (5)	7237 (10)	105 (6)
O(5)	1251 (9)	-706 (4)	8169 (9)	92 (6)
N	940 (9)	-1171 (5)	7063 (11)	70 (6)
C(1)	2722 (11)	2018 (5)	676 (12)	75 (7)
C(2)	4146 (14)	1084 (5)	-897 (12)	93 (8)
C(3)	5628 (11)	2227 (5)	551 (12)	73 (6)
C(4)	4311 (10)	1636 (4)	564 (9)	52 (5)
C(5)	3417 (11)	577 (5)	3685 (11)	56 (6)
C(6)	2439 (9)	-126 (4)	3777 (9)	46 (5)
C(7)	1864 (11)	-582 (5)	2431 (10)	62 (6)
C(8)	1022 (12)	-1243 (5)	2634 (12)	76 (7)
C(9)	707 (11)	-1448 (5)	4132 (13)	68 (7)
C(10)	1280 (10)	-975 (5)	5434 (10)	52 (6)
C(11)	2120 (9)	-311 (4)	5319 (9)	47 (5)
Compound (II)				
O(1)	6594 (22)	11487 (17)	7218 (12)	82 (10)
O(2)	7639 (22)	10130 (17)	6526 (12)	81 (10)
O(3)	4543 (25)	8660 (18)	5738 (14)	100 (11)
O(4)	11469 (27)	4055 (18)	2596 (15)	121 (12)
O(5)	8687 (31)	2529 (22)	1928 (15)	142 (13)
N	9775 (31)	3829 (24)	2622 (17)	87 (12)
C(1)	6589 (41)	10038 (26)	8743 (19)	121 (15)
C(2)	9668 (35)	12145 (29)	8925 (20)	105 (15)
C(3)	6314 (40)	13200 (29)	9083 (22)	131 (15)
C(4)	7401 (46)	11718 (27)	8508 (21)	99 (15)
C(5)	6261 (34)	8830 (27)	5807 (19)	54 (12)
C(6)	7180 (35)	7522 (26)	4926 (18)	59 (13)
C(7)	9247 (38)	7838 (25)	5031 (19)	79 (14)
C(8)	10156 (32)	6596 (25)	4199 (19)	75 (13)
C(9)	8867 (32)	5146 (25)	3408 (19)	67 (13)
C(10)	6949 (29)	4864 (25)	3392 (18)	59 (12)
C(11)	6082 (35)	6080 (25)	4161 (19)	84 (13)

Discussion. The final atomic parameters are in Table 1.* Views of the separate molecules and molecular packing are shown in Figs. 1 and 2. Interatomic distances and angles and selected torsion angles are listed in Table 2. In general the bond lengths and valency angles in molecules (I) and (II) agree well and are within normal ranges for aromatic nitro compounds (Dhaneshwar, Tavale & Pant, 1974; Colapietro & Domenicano, 1977). The differences between comparable bond lengths are not statistically significant. However, the bonds C(7)–C(8), C(6)–C(11) and C(9)–C(10) in (II) are rather unusual for an aromatic system, but could be attributed to the considerable decay of the crystals during the data collection. The same reason would explain to some extent the high thermal parameters of O(3) and O(4) in (I) and C(1), C(3), O(4) and O(5) in (II) (Table 1). Because of the presence of a bulky *tert*-butyl group, the molecules exist in elongated form with C(3) and O(2) *trans*, whilst O(1) and O(3), and O(3) and C(11) are in *cis* positions. The torsion angle about O(1)–O(2) is virtually the same in both molecules: 129 (1) in (I) and 120 (2)° in (II). These values are similar to that of 133° in peroxy-pelargonic acid (Belitskus & Jeffrey, 1965), but differ considerably from the 146° in 2-nitroperbenzoic acid (Sax, Beurskens & Chu, 1965) and from the 170° in 4-nitroperbenzoic acid (Kim, Chu & Jeffrey, 1970). The first and third of these values were, however, based on the assumption that a linear hydrogen bond linked the adjacent molecules, as H atoms were not located.

* Lists of structure factors, anisotropic thermal parameters H-atom coordinates and results of mean-plane calculations have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 39015 (14 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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

C(1)–C(4)	1.52 (1)	1.56 (4)	C(5)–O(3)	1.18 (1)	1.17 (3)	C(8)–C(9)	1.37 (2)	1.39 (2)
C(2)–C(4)	1.53 (1)	1.52 (4)	C(5)–C(6)	1.49 (1)	1.53 (3)	C(9)–C(10)	1.37 (1)	1.32 (3)
C(3)–C(4)	1.52 (1)	1.52 (4)	C(6)–C(7)	1.38 (1)	1.40 (4)	C(10)–C(11)	1.37 (1)	1.39 (3)
C(4)–O(1)	1.45 (1)	1.48 (3)	C(6)–C(11)	1.39 (1)	1.32 (3)	N–O(4)	1.23 (1)	1.18 (3)
O(1)–O(2)	1.46 (1)	1.49 (2)	C(7)–C(8)	1.38 (1)	1.46 (3)	N–O(5)	1.21 (1)	1.22 (2)
O(2)–C(5)	1.35 (1)	1.33 (2)						
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C(1)–C(4)–C(2)	112 (1)	113 (2)	O(1)–O(2)–C(5)	112 (1)	108 (2)	C(7)–C(6)–C(11)	120 (1)	122 (2)
C(1)–C(4)–C(3)	111 (1)	108 (2)	O(2)–C(5)–O(3)	123 (1)	129 (2)	C(6)–C(7)–C(8)	119 (1)	118 (2)
C(1)–C(4)–O(1)	109 (1)	107 (2)	O(2)–C(5)–C(6)	110 (1)	111 (2)	C(7)–C(8)–C(9)	122 (1)	115 (2)
C(2)–C(4)–C(3)	112 (1)	113 (2)	O(3)–C(5)–C(6)	127 (1)	120 (2)	C(8)–C(9)–C(10)	117 (1)	124 (2)
C(2)–C(4)–O(1)	111 (1)	114 (2)	C(5)–C(6)–C(7)	123 (1)	118 (2)	C(9)–C(10)–C(11)	124 (1)	121 (2)
C(3)–C(4)–O(1)	100 (1)	101 (2)	C(5)–C(6)–C(11)	117 (1)	121 (2)	C(10)–C(11)–C(6)	117 (1)	120 (2)
C(4)–O(1)–O(2)	107 (1)	107 (2)				O(4)–N–O(5)	123 (1)	120 (2)
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Compound (I)			Compound (II)					
C(10)–N	1.47 (1)	C(10)–N–O(4)	118 (1)	C(9)–N	1.47 (3)	C(9)–N–O(4)	122 (2)	
C(9)–C(10)–N	119 (1)	C(10)–N–O(5)	119 (1)	C(8)–C(9)–N	116 (2)	C(9)–N–O(5)	118 (2)	
C(11)–C(10)–N	117 (1)			C(10)–C(9)–N	120 (2)			

Torsion angles (sign convention of Klyne & Prelog, 1960)

C(4)–O(1)–O(2)–C(5)	129 (1)	120 (2)	O(1)–O(2)–C(5)–O(3)	-2 (1)	-4 (3)	O(5)–N–C(9)–C(10)	—	3 (3)
O(2)–O(1)–C(4)–C(1)	-69 (1)	-71 (2)	O(1)–O(2)–C(5)–C(6)	177 (1)	171 (2)	O(5)–N–C(10)–C(11)	-8 (1)	—
O(2)–O(1)–C(4)–C(2)	55 (1)	55 (2)	O(4)–N–C(9)–C(8)	—	-6 (3)	O(2)–C(5)–C(6)–C(7)	13 (1)	2 (3)
O(2)–O(1)–C(4)–C(3)	174 (1)	176 (2)	O(4)–N–C(10)–C(9)	-11 (1)	—	O(3)–C(5)–C(6)–C(11)	11 (1)	-8 (3)

The distance O(1)—O(2) of 1.46 (1) in (I) and 1.49 (2) Å in (II) falls in the range typical for organic peroxy compounds (Pedersen, 1972; Glidewell, Liles, Walton & Sheldrick, 1979). The nitro, benzene and peroxy-carboxyl parts of the molecules are essentially planar. Full details have been deposited.

There are no unusually short contacts between the molecules.

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Structure of the Adduct between 7,8-Diacetoxycyclo[4.2.0]octa-2,4-diene and 2,3-Dihydronaphthazarin:* C₂₂H₂₀O₈

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Abstract. $M_r = 412.4$, monoclinic, $P2_1/c$, $a = 7.744$ (1), $b = 27.419$ (2), $c = 9.449$ (1) Å, $\beta = 113.16$ (2)°, $V = 1844.6$ Å³, $Z = 4$, $D_x = 1.485$, $D_m = 1.50$ Mg m⁻³, $\mu(\text{Cu K}\alpha) = 0.91264$ mm⁻¹, $\lambda = 1.5418$ Å, room temperature, $F(000) = 864$, final $R = 0.052$ for 2944 observed reflections. The structure was solved by direct methods and refined by least-squares procedure. It was verified that there is no charge-transfer interaction in spite of the presence of a 2,3-dihydronaphthazarin nucleus in the molecule.

Introduction. The present compound is the result of the Diels–Alder reaction between 7,8-diacetoxycyclo[4.2.0]octa-2,4-diene and naphthazarin. Generally the naphthazarin nucleus forms a charge-transfer self-complex (Rodriguez, Florencio, Smith-Verdier & García-Blanco, 1983). The quinonoid ring in the

naphthazarin acts as a dienophile in the Diels–Alder reaction. In the title compound there is a dihydronaphthazarin ring which has less acceptor character than the naphthazarin ring from the point of view of the formation of a charge-transfer complex. Verification of this and elucidation of the stereochemistry of the molecule required the resolution of the crystal structure.

Experimental. D_m measured by flotation. Orange-red plate crystals $0.2 \times 0.15 \times 0.05$ mm. Nonius CAD-4 automatic diffractometer, Mo K α . 3016 independent reflections ($-8 \leq h \leq 8$, $0 \leq k \leq 32$, $0 \leq l \leq 10$), $\theta < 30^\circ$, 2944 with $I > 2\sigma(I)$. Absorption ignored. 30 reflections used for measuring lattice parameters. Direct methods (*MULTAN80*; Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980). Anisotropic full matrix; H (from ΔF synthesis) isotropic; $\sum w(\Delta F)^2$ minimized; $w = k/\sigma^2$ (Martinez-Ripoll & Cano, 1975) with $\sigma = a + b(F_o)$ and $k = 0.6032$ ($a = 0.4141$, $b = 0.0143$ for $F_o < 12.500$ and $a = 0.0815$, $b = 0.0373$

* 2,3-Dihydronaphthazarin is 2,3-dihydro-5,8-dihydroxy-1,4-naphthoquinone.