ted only to the sulphate ions by hydrogen bonds and the water molecule lies between the sulphate ions and the hydroxyl group of the 1,3-diammonio-2-propanol cation. The role of the hydrogen bonds has been found to be similar in the bis(oxalato)cuprate(II) complex.

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Structure of 3-[4,5-Bis(methoxycarbonyl)isoxazol-3-yl]-4,6-dioxo-*syn-cis-syn*-1,3,5-triphenylperhydrothieno[3,4-*c*]pyrrole-1-carbonitrile

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Abstract. $C_{32}H_{23}N_3O_7S$, monoclinic, $P2_1/c$, $Z = 4$, a = 12·764 (6), b = 22·90 (1), c = 10·647 (4) Å, β = 110·41 (3)°, D_m = 1·351, D_x = 1·334 Mg m^{-3}. The structure was solved by the symbolic-addition procedure; block-diagonal least-squares refinement led to a final R of 0·0425 for 3471 observed reflexions. Molecules are held together by van der Waals interactions. The condensed perhydrothiophene–pyrrolidine ring has a chair conformation. A partial selective ester-exchange reaction occurred during the recrystallization from ethanol at the more protruding methoxycarbonyl group.

Introduction. In contrast to the adduct of 1,3-diphenylthieno[3,4-*c*][1,2,5]thiadiazole and *N*-phenylmaleimide (Tsuge, Takata & Ueda, 1979), neither the *endo* nor the *exo* adduct of 1,3-diphenylthieno[3,4-*c*]-[1,2,5]oxadiazole and the imide underwent a thermal retro-cycloaddition reaction, but reacted with dimethyl acetylenedicarboxylate (DMAD) to yield the corresponding 1:1 adduct (Tsuge, Takata & Noguchi, 1977). The product was assumed from spectroscopy to be a 1,3-cycloadduct of the nitrile oxide generated *in situ* from a ring opening of the oxadiazole moiety of the initial reactant, and DMAD (Tsuge, Takata & Ueda, 1979). Therefore, the X-ray analysis of the title compound was carried out to provide conclusive evidence for the chemical structure of the product obtained from the reaction of the *endo* adduct with DMAD.

The title compound (Fig. 1) was recrystallized at 313 K from an ethanol solution as colorless prisms. Preliminary Weissenberg photographs showed the space group to be $P2_1/c$. The unit-cell parameters were refined by a least-squares procedure using the 2θ values of 15 reflexions measured on a Syntex $P\bar{1}$ four-circle diffractometer. The density was measured by flotation in an aqueous KI solution. A cylindrical crystal (length 0·45 mm, diameter 0·39 mm) was used for the data collection. The intensities of 5746 reflexions within $2\theta \leq 50°$ were measured by the θ–2θ scan technique (monochromated Mo $K\alpha$ radiation), of which 3471 independent reflexions with $I > 2{\cdot}33\sigma(I)$ were used for the analysis. Corrections for Lorentz and polarization effects and for fluctuation of the monitored intensity were applied, but not for absorption (μ = 0·166 mm^{-1} for Mo $K\alpha$).

The structure was solved by the direct method using the program *MULTAN* (Germain, Main & Woolfson, 1971) and refined by a block-matrix least-squares

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Table 1. *Final atomic coordinates with their e.s.d.'s in parentheses*

	x	y	z		x	y	z
S	0.35082(6)	0.36563(3)	0.06438(7)	N(1)	0.0193(2)	0.3155(1)	0.1148(2)
C(1)	0.3209(2)	0.3225(1)	0.1942(3)	N(2)	0.3225(3)	0.3924(1)	0.3891(3)
C(2)	0.2019(2)	0.2984(1)	0.1189(3)	N(3)	0.0987(2)	0.4365(1)	0.1117(3)
C(3)	0.1240(2)	0.2964(1)	0.1981(3)	O(1)	0.1469(2)	0.2797(1)	0.3118(2)
C(4)	0.0208(2)	0.3383(1)	-0.0068(3)	O(2)	-0.0594(2)	0.3554(1)	-0.0965(2)
C(5)	0.1396(2)	0.3330(1)	-0.0075(3)	O(3)	0.1072(2)	0.4834(1)	0.1972(2)
C(6)	0.2046(2)	0.3906(1)	-0.0126(3)	O(4)	0.3730(2)	0.5200(1)	0.0220(3)
C(7)	0.4120(2)	0.2760(1)	0.2458(3)	O(5)	0.4447(2)	0.5018(1)	0.2406(3)
C(8)	0.4161(3)	0.2294(2)	0.1650(3)	O(6)	0.3027(2)	0.5974(1)	0.3019(3)
C(9)	0.4976(3)	0.1871(2)	0.2109(4)	O(7)	0.1642(2)	0.5677(1)	0.3696(2)
C(10)	0.5760(3)	0.1911(2)	0.3365(4)	H(C2)	0.208(2)	0.257(1)	0.095(3)
C(11)	0.5746(3)	0.2381(2)	0.4161(4)	H(C5)	0.133(2)	0.312(1)	-0.089(3)
C(12)	0.4927(3)	0.2807(2)	0.3716(3)	H(C8)	0.358(3)	0.226(2)	0.071(4)
C(13)	0.3213(3)	0.3619(1)	0.3038(3)	H(C9)	0.498(3)	0.151(2)	0.150(4)
C(14)	-0.0771(2)	0.3170(1)	0.1549(3)	H(C10)	0.636(3)	0.158(2)	0.372(4)
C(15)	-0.1423(3)	0.3666(1)	0.1325(3)	H(C11)	0.634(4)	0.243(2)	0.510(4)
C(16)	-0.2353(3)	0.3668(2)	0.1725(4)	H(C12)	0.491(3)	0.317(2)	0.430(4)
C(17)	-0.2607(3)	0.3184(2)	0.2329(3)	H(C15)	-0.121(3)	0.402(2)	0.086(3)
C(18)	-0.1948(3)	0.2694(2)	0.2548(4)	H(C16)	-0.284(3)	0.404(2)	0.154(4)
C(19)	-0.1024(3)	0.2682(1)	0.2150(3)	H(C17)	-0.328(3)	0.319(2)	0.265(4)
C(20)	0.1846(2)	0.4100(1)	-0.1557(3)	H(C18)	-0.214(3)	0.233(2)	0.300(4)
C(21)	0.1334(3)	0.4628(2)	-0.2033(3)	H(C19)	-0.054(3)	0.231(2)	0.226(3)
C(22)	0.1156(3)	0.4793(2)	-0.3349(4)	H(C21)	0.108(3)	0.488(2)	-0.140(4)
C(23)	0.1473(3)	0.4437(2)	-0.4192(3)	H(C22)	0.079(3)	0.517(2)	-0.368(4)
C(24)	0.1979(4)	0.3912(2)	-0.3721(4)	H(C23)	0.134(3)	0.455(2)	-0.517(4)
C(25)	0.2170(3)	0.3745(2)	-0.2409(3)	H(C24)	0.221(3)	0.364(2)	-0.435(4)
C(26)	0.1863(2)	0.4387(1)	0.0751(3)	H(C25)	0.260(3)	0.337(2)	-0.202(4)
C(27)	0.2553(2)	0.4870(1)	0.1354(3)	H(1C30)	0.597(5)	0.533(2)	0.334(5)
C(28)	0.2019(3)	0.5128(1)	0.2097(3)	H(2C30)	0.546(4)	0.555(2)	0.178(5)
C(29)	0.3638(3)	0.5051(1)	0.1245(3)	H(3C30)	0.588(5)	0.490(2)	0.210(5)
C(30)	0.5549(4)	0.5205(3)	0.2443(6)	H(1C32)	0.200(4)	0.651(2)	0.415(5)
C(31)	0.2300(3)	0.5642(1)	0.2986(3)	H(2C32)	0.246(4)	0.607(2)	0.543(5)
C(32)	0.1802(5)	0.6177(2)	0.4576(5)				
C(33)*	0.0783(15)	0.6391(8)	0.4601(17)				

* The occupancy is 0.3.

method. An E map calculated with a phase set having the highest figure of merit (1·40) for 351 reflexions ($E \geq 1·7$) revealed all 43 non-hydrogen atom positions, which coincided with the proposed molecular structure. In the course of the refinement, a difference Fourier map showed an abnormally high peak at the H atom [H(3C32)] corresponding to the most protruding C atom of the methoxycarbonyl moieties. The NMR spectra of the crystals used in this analysis showed that one of the methoxycarbonyl groups was partially replaced with ethoxycarbonyl in ethanol solution by ester-exchange reactions during recrystallization. The disordered structure was taken into account during the refinement. For the final three cycles of refinement, the weighting scheme was changed from unit weight to $w = a_1 + b_1|F_o|$ for $|F_o| < F_1$, $w = c_2$ for $F_1 \leq |F_o| \leq F_2$ and $w = d_3/(a_3 + b_3|F_o| + c_3|F_o|^2)$ for $|F_o| > F_2$ where the constants were selected as follows: $F_1 = 18·90$, $F_2 = 36·0$, $a_1 = 0·8031$, $b_1 = 0·0177$, $c_2 = 1·0$, $a_3 = 2·6052$, $b_3 = -0·0786$, $c_3 = 0·0011$ and $d_3 = 0·9799$. The final R value was 0·0425 for the observed reflexions. A final difference Fourier map had no spurious peaks greater than 0·25 e Å^{-3}. The final atomic coordinates are listed in Table 1.*

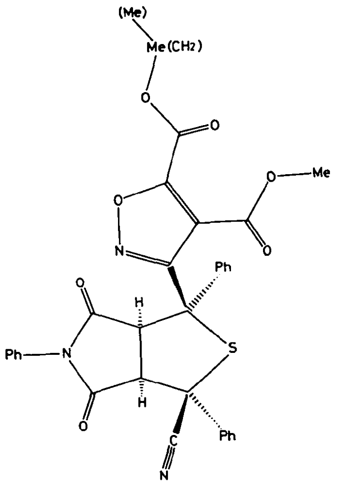

Fig. 1. The structural formula.

The atomic scattering factors and the anomalous-dispersion factors were taken from *International Tables for X-ray Crystallography* (1974). All calculations were performed on a FACOM M-190 com-

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* Lists of structure factors and thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 34923 (37 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

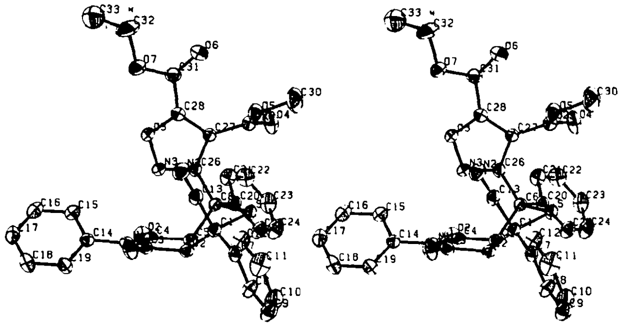

Fig. 2. A stereoview of the molecular conformation.

puter at the Computer Center of Kyushu University with the *UNICS* II program system (Sakurai, Iwasaki, Watanabe, Kobayashi, Bando & Nakamichi, 1974).

Discussion. Fig. 1 shows the structural formula for one of the racemic molecules. Fig. 2 shows a stereoview of the molecular conformation for the non-hydrogen atoms together with the atom numbering, drawn with the program *ORTEP* (Johnson, 1965). The racemic molecules are held together by van der Waals forces.

All the bond distances, given in Table 2, are very close to the expected values (*International Tables for X-ray Crystallography*, 1962). The bond angles are also normal (Table 3). The molecule consists of a condensed perhydrothiophene–pyrrolidine ring, an isoxazole ring with ethoxy- and methoxycarbonyl groups, a cyano group and three benzene rings. The condensed ring has a chair conformation as shown in Fig. 2. The other four rings are planar. The angles made by the three benzene rings with respect to the pyrrolidine ring are successively 43, 51 and 98° with increasing atom number. These different tilting angles may cause the small differences of the chemical shifts between the H(C2) and H(C5) atoms observed in the NMR spectrum. If we compare the difference between the methoxy- and ethoxycarbonyl moieties, the latter is more protruded from the molecule, and the bond distance C(29)–O(5) is slightly shorter than that of C(31)–O(7). The selective ester-exchange reaction which occurred during the recrystallization may be affected by these factors.

Table 2. *Interatomic distances*

	Å		Å
S -C(1)	1.843(3)	C(16)-H(C16)	1.03(4)
S -C(6)	1.848(3)	C(17)-C(18)	1.372(5)
C(1) -C(2)	1.551(4)	C(17)-H(C17)	1.03(4)
C(1) -C(7)	1.532(4)	C(18)-C(19)	1.385(5)
C(1) -C(13)	1.473(4)	C(18)-H(C18)	1.04(4)
C(2) -C(3)	1.512(4)	C(19)-H(C19)	1.03(4)
C(2) -C(5)	1.524(4)	C(20)-C(21)	1.383(5)
C(2) -H(C2)	1.00(3)	C(20)-C(25)	1.384(5)
C(3) -N(1)	1.392(4)	C(21)-C(22)	1.391(5)
C(3) -O(1)	1.204(4)	C(21)-H(C21)	1.02(4)
C(4) -C(5)	1.524(4)	C(22)-C(23)	1.373(6)
C(4) -N(1)	1.402(4)	C(22)-H(C22)	0.99(4)
C(4) -O(2)	1.197(4)	C(23)-C(24)	1.374(6)
C(5) -C(6)	1.571(4)	C(23)-H(C23)	1.03(4)
C(5) -H(C5)	0.98(3)	C(24)-C(25)	1.386(6)
C(6) -C(20)	1.521(4)	C(24)-H(C24)	1.03(4)
C(6) -C(26)	1.513(4)	C(25)-H(C25)	1.03(4)
C(7) -C(8)	1.384(5)	C(26)-C(27)	1.421(4)
C(7) -C(12)	1.380(5)	C(26)-N(3)	1.306(4)
C(8) -C(9)	1.380(5)	C(27)-C(28)	1.348(4)
C(8) -H(C8)	1.02(4)	C(27)-C(29)	1.488(5)
C(9) -C(10)	1.365(6)	C(28)-C(31)	1.474(5)
C(9) -H(C9)	1.06(4)	C(28)-O(3)	1.350(4)
C(10)-C(11)	1.373(6)	C(29)-O(4)	1.188(5)
C(10)-H(C10)	1.05(4)	C(29)-O(5)	1.308(5)
C(11)-C(12)	1.388(6)	C(30)-O(5)	1.458(7)
C(11)-H(C11)	1.03(5)	C(30)-H(1C30)	0.96(6)
C(12)-H(C12)	1.04(4)	C(30)-H(2C30)	1.04(6)
C(13)-N(2)	1.142(5)	C(30)-H(3C30)	0.96(6)
C(14)-C(15)	1.379(5)	C(31)-O(6)	1.190(5)
C(14)-C(19)	1.381(5)	C(31)-O(7)	1.313(4)
C(14)-N(1)	1.437(4)	C(32)-O(7)	1.447(7)
C(15)-C(16)	1.393(5)	C(32)-C(33)	1.40(2)
C(15)-H(C15)	1.03(4)	C(32)-H1(1C32)	0.97(5)
C(16)-C(17)	1.377(5)	C(32)-H2(2C32)	1.03(5)
		N(3) -O(3)	1.388(4)

Table 3. *Bond angles*

C(1) -S -C(6)	93.3(1)°	C(17) -C(18)-H(C18)	121(2)°
C(2) -C(1) -C(7)	114.9(2)	C(19) -C(18)-H(C18)	119(2)
C(2) -C(1) -C(13)	111.1(3)	C(14) -C(19)-C(18)	119.2(3)
C(7) -C(1) -C(13)	110.4(3)	C(14) -C(19)-H(C19)	120(2)
C(1) -C(2) -C(3)	116.0(3)	C(18) -C(19)-H(C19)	121(2)
C(1) -C(2) -C(5)	112.7(3)	C(6) -C(20)-C(21)	121.0(3)
C(1) -C(2) -H(C2)	109(2)	C(6) -C(20)-C(25)	120.1(3)
C(3) -C(2) -C(5)	105.5(3)	C(21) -C(20)-C(25)	118.9(3)
C(3) -C(2) -H(C2)	104(2)	C(20) -C(21)-C(22)	119.8(3)
C(5) -C(2) -H(C2)	110(2)	C(20) -C(21)-H(C21)	118(2)
C(2) -C(3) -N(1)	107.9(3)	C(22) -C(21)-H(C21)	122(2)
C(2) -C(3) -O(1)	126.5(3)	C(21) -C(22)-C(23)	121.0(4)
N(1) -C(3) -O(1)	125.6(3)	C(21) -C(22)-H(C22)	119(2)
C(5) -C(4) -N(1)	108.0(2)	C(23) -C(22)-H(C22)	120(2)
C(5) -C(4) -O(2)	126.7(3)	C(22) -C(23)-C(24)	119.3(4)
N(1) -C(4) -O(2)	125.2(3)	C(22) -C(23)-H(C23)	122(2)
C(2) -C(5) -C(4)	104.5(2)	C(24) -C(23)-H(C23)	119(2)
C(2) -C(5) -C(6)	110.4(3)	C(23) -C(24)-C(25)	120.2(4)
C(2) -C(5) -H(C5)	112(2)	C(23) -C(24)-H(C24)	120(2)
C(4) -C(5) -C(6)	118.1(3)	C(25) -C(24)-H(C24)	120(2)
C(4) -C(5) -H(C5)	106(2)	C(20) -C(25)-C(24)	120.8(4)
C(6) -C(5) -H(C5)	106(2)	C(20) -C(25)-H(C25)	118(2)
C(5) -C(6) -C(20)	111.9(2)	C(24) -C(25)-H(C25)	121(2)
C(5) -C(6) -C(26)	113.2(2)	C(6) -C(26)-C(27)	129.8(3)
C(20)-C(6) -C(26)	113.4(3)	C(6) -C(26)-N(3)	119.2(3)
C(1) -C(7) -C(8)	120.2(3)	C(27) -C(26)-N(3)	110.8(3)
C(1) -C(7) -C(12)	120.6(3)	C(26) -C(27)-C(28)	104.3(3)
C(8) -C(7) -C(12)	119.2(3)	C(26) -C(27)-C(29)	128.9(3)
C(7) -C(8) -C(9)	120.5(3)	C(28) -C(27)-C(29)	126.8(3)
C(7) -C(8) -H(C8)	120(2)	C(27) -C(28)-C(31)	131.9(3)
C(9) -C(8) -H(C8)	120(2)	C(27) -C(28)-O(3)	110.0(3)
C(8) -C(9) -C(10)	120.3(4)	C(31) -C(28)-O(3)	118.1(3)
C(8) -C(9) -H(C9)	120(2)	C(27) -C(29)-O(4)	123.0(3)
C(10)-C(9) -H(C9)	120(2)	C(27) -C(29)-O(5)	111.1(3)
C(9) -C(10)-C(11)	119.6(4)	O(4) -C(29)-O(5)	125.9(4)
C(9) -C(10)-H(C10)	120(2)	O(5) -C(30)-H(1C30)	109(4)
C(11)-C(10)-H(C10)	120(2)	O(5) -C(30)-H(2C30)	109(3)
C(10)-C(11)-C(12)	120.8(4)	O(5) -C(30)-H(3C30)	108(4)
C(10)-C(11)-H(C11)	121(3)	H(1C30)-C(30)-H(2C30)	110(5)
C(12)-C(11)-H(C11)	118(3)	H(1C30)-C(30)-H(3C30)	115(5)
C(7) -C(12)-C(11)	119.6(4)	H(2C30)-C(30)-H(3C30)	105(5)
C(7) -C(12)-H(C12)	119(2)	C(28) -C(31)-O(6)	122.7(3)
C(11)-C(12)-H(C12)	122(2)	C(28) -C(31)-O(7)	111.0(3)
C(1) -C(13)-N(2)	179.5(4)	O(6) -C(31)-O(7)	126.3(3)
C(15)-C(14)-C(14)	121.5(3)	O(7) -C(32)-C(33)	112(1)
C(15)-C(14)-N(1)	119.8(3)	O(7) -C(32)-H(1C32)	109(3)
C(19)-C(14)-N(1)	118.8(3)	O(7) -C(32)-H(2C32)	106(3)
C(14)-C(15)-C(16)	118.4(3)	C(33) -C(32)-H(1C32)	97(3)
C(14)-C(15)-H(C15)	119(2)	C(33) -C(32)-H(2C32)	123(3)
C(16)-C(15)-H(C15)	123(2)	H(1C32)-C(32)-H(2C32)	110(4)
C(15)-C(16)-C(17)	120.4(3)	C(3) -N(1) -C(4)	112.7(2)
C(15)-C(16)-H(C16)	117(2)	C(3) -N(1) -C(14)	123.4(2)
C(17)-C(16)-H(C16)	122(2)	C(4) -N(1) -C(14)	123.7(2)
C(16)-C(17)-C(18)	120.5(4)	C(26) -N(3) -O(3)	106.7(2)
C(16)-C(17)-H(C17)	120(2)	C(28) -O(3) -N(3)	108.3(2)
C(18)-C(17)-H(C17)	119(2)	C(29) -O(5) -C(30)	116.4(4)
C(17)-C(18)-C(19)	120.1(4)	C(31) -O(7) -C(32)	116.6(3)

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3-Aminopropylphosphonic Acid

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Abstract. $C_3H_{10}NO_3P$, $M_r = 139{\cdot}01$, orthorhombic, $Pna2_1$, $a = 9{\cdot}495$ (2), $b = 7{\cdot}925$ (1), $c = 8{\cdot}017$ (1) Å, $Z = 4$, $D_m = 1{\cdot}53$ (by flotation), $D_x = 1{\cdot}53$ Mg m^{-3}; final $R = 0{\cdot}062$. The molecule exists as the zwitterion $H_3^+N{-}(CH_2)_3{-}PO_3H^-$. There are four independent hydrogen bonds in the structure; three are of the type N—H···O with lengths 2·811, 2·812 and 2·831 Å, and one is of the type O—H···O with length 2·522 Å.

Introduction. 3-Aminopropylphosphonic acid was prepared by a method developed by Dr R. Tyka at the Institute of Organic and Physical Chemistry, Technical University of Wrocław. Crystals suitable for X-ray analysis were colourless prisms. The space group and cell constants were obtained initially from Weissenberg photographs. The cell parameters were determined by least-squares refinement from the setting angles of 15 reflexions given by the automatic centring program [λ(Cu $K\alpha$) = 1·5418 Å]. All measurements for a crystal 0·08 × 0·15 × 0·15 mm were made on a Syntex $P2_1$ computer-controlled four-circle diffractometer equipped with a scintillation counter and graphite monochromator. 475 independent reflexions were measured up to $2\theta = 114{\cdot}0°$ with the variable θ–2θ scan technique. The scan rate varied from 3·0 to 20·0° min^{-1}, depending on the intensity. After each group of 15 reflexions the intensity of a standard was measured; no significant change was observed. The intensities were corrected for Lorentz and polarization factors, but not for absorption [μ(Cu $K\alpha$) = 3·44 mm^{-1}].

The structure was solved by the heavy-atom technique and refined anisotropically by full-matrix least squares. A difference synthesis revealed the positions of nine H atoms. The H atom of the OH group was not located. The coordinates of the H atoms and their isotropic thermal factor ($B = 3{\cdot}8$ Å^2) were not refined. The final $R = 0{\cdot}062$ and $R_w = 0{\cdot}081$ for 436 observed reflexions for which $F > 3{\cdot}92\sigma(F)$. For all 475 reflexions R and R_w are 0·066 and 0·081. The function minimized was $\sum w(|F_o| - |F_c|)^2$ with $w = 1/\sigma^2(F)$. Scattering factors for neutral atoms were taken from *International Tables for X-ray Crystallography* (1974). All calculations were performed with the Syntex *XTL* structure determination system on a Nova 1200

Table 1. *Positional parameters* (×10^4; *for* H ×10^3) *with e.s.d.'s in parentheses*

The atomic parameters of H were not refined; $B_{\rm iso} = 3{\cdot}8$ Å^2.

	x	y	z
P	1559 (3)	2270 (1)	3500 (1)
O(1)	2769 (8)	3176 (11)	4570 (12)
O(2)	1809 (8)	2422 (11)	1653 (12)
O(3)	187 (8)	3063 (11)	4134 (13)
N	667 (10)	−3548 (12)	5087 (15)
C(1)	1665 (13)	65 (14)	4066 (18)
C(2)	465 (14)	−949 (13)	3220 (18)
C(3)	697 (15)	−2884 (14)	3303 (21)
H(1)	157	−7	530
H(2)	259	−42	370
H(3)	42	−59	200
H(4)	−44	−67	377
H(5)	166	−316	279
H(6)	−4	−347	262
H(7)	72	−483	498
H(8)	−23	−322	578
H(9)	149	−312	575

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