$y \sim \pm 0.15$ and ± 0.35. It is to be noticed that the planes of the inorganic groups are perpendicular to those of the organic molecules.

Geometrical details of the hydrogen-bond scheme are reported in Table 4. Two points should be noted: there is no intermolecular hydrogen bond between glycine molecules; and the bonding oxygen atoms (OL) of the P_3O_9 ring are not involved in hydrogen bonds, while all the external ones are. This fact is to be compared with an almost systematic observation made in inorganic condensed phosphate chemistry, where OL bonding oxygen atoms do not take part in cation coordination polyhedra. Each glycine molecule is connected by hydrogen bonds to four different phosphoric groups.

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Structure of 5-Nitro-2-{[(S)-1-phenylethyl]amino}pyridine

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Abstract. $C_{13}H_{13}N_3O_2$, $M_r = 243.26$, monoclinic, $P2_1$, $a = 17.945$ (4), $b = 6.372$ (1), $c = 5.401$ (2) Å, $\beta = 94.70$ (2)°, $V = 615.5$ (3) Å^3, $Z = 2$, $D_m = 1.31$, $D_x = 1.313$ g cm^{-3}, λ(Mo $K\alpha$) = 0.71069 Å, $\mu = 0.854$ cm^{-1}, $F(000) = 256$, $T = 295$ K, $R = 0.065$ for 770 unique observed reflections. The twist angle of the nitro group out of the pyridine ring and that of the amino group are 2.8 and $-8.5°$, respectively. The crystal packing is dominated by hydrogen bonds which link the molecules into infinite chains. The title compound proved to have a suitable molecular and crystal structure for nonlinear optical materials.

Introduction. In recent years, conjugated organic molecular crystals have attracted considerable attention as preferable materials in nonlinear optics, such as second harmonic generation (SHG) and optical bistability, owing to their large optical nonlinearities. Among a number of organic compounds examined, the title compound was found to have very high SHG efficiency in the powder form and to have an absorption cut-off in the relatively short-wavelength region (Twieg, Azema, Jain & Cheng, 1982). These properties make this compound highly attractive for application to frequency doubling of the light produced by semi-

conductor lasers. The present X-ray analysis was undertaken to elucidate the relation between the crystal structure and its large optical nonlinearity.

Experimental. The title compound was synthesized by the method reported previously (Bettoni, Catsiotis, Perrone & Tortorella, 1977). Yellow crystal, thin rectangular plate, recrystallized by slow evaporation from a mixture of hexane–methylene chloride, with dimensions $0.6 \times 0.4 \times 0.2$ mm. Density measured by flotation in a $ZnCl_2$ solution. Rigaku AFC-5 four-circle diffractometer, graphite-monochromatized Mo $K\alpha$. Cell dimensions determined with 20 reflections ($20 < 2\theta < 30°$) by least-squares method. Systematic absences $0k0$ for k odd. Intensity data $2 < 2\theta < 30°$, ω–2θ scan, $30 < 2\theta < 60°$, θ–2θ scan, scan speed 4° min^{-1}. Three standard reflections (410, 112, 040) monitored every 100 reflections showed only random variations within 3% in intensity. 1643 unique reflections ($h = -22$ to 22, $k = 0$ to 8, $l = 0$ to 6) measured, 770 reflections with $|F_o| > 3\sigma(|F_o|)$ observed. No correction for absorption. Structure solved by direct methods with *MULTAN* (Main, Woolfson & Germain, 1971). 12 non-H atoms were obtained from the initial E map and the remaining non-H atoms were located from sub-

sequent Fourier syntheses. H atoms were placed at idealized positions. Block-diagonal least-squares refinement on F_o with anisotropic thermal parameters for non-H atoms, isotropic for H. The absolute configuration was determined using the known asymmetric carbon atom [C(6)] as an internal reference for the asymmetric center. Atomic scattering factors from *International Tables for X-ray Crystallography* (1974), and f', f'' values included from Cromer's (1965) tabulation. Unit weights on all reflections. $R = 0.065$, $wR = 0.065$, $S = 0.769$. $\Delta\rho(\text{max}) = 0.24$ e Å^{-3}. $\Delta/\sigma <$ 0.1. All computations were performed on a HITAC M-682H computer at the Computer Center of the University of Tokyo with *UNICS*III (Sakurai & Kobayashi, 1979).

Discussion. Final atomic parameters are given in Table 1, and bond lengths and angles in Table 2.*

An *ORTEP* (Johnson, 1965) drawing of the molecule with the numbering scheme for the atoms is shown in Fig. 1. All bond lengths and angles are normal. The pyridine and the benzene rings are planar with maximum deviations of 0.022 and 0.021 Å, respectively. The mean planes of the two rings make a dihedral angle of 96.5°.

The dihedral angle between the mean plane of the nitro group [O(1), O(2), N(1), C(1)] which acts as an acceptor and that of the pyridine ring is 2.8°, and that between the amino group [N(3), C(5), C(6), H(N3)] which acts as a donor and the pyridine ring is −8.5°. Despite the introduction of the bulky phenylethyl group on N(3), these twist angles are still small. This relatively slight twisted conformation suggests that strong intramolecular charge transfer occurs from the amino group to the nitro group *via* the pyridine ring. This is the reason why this molecule possesses large second-order hyperpolarizabilities.

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* Lists of structure factors, anisotropic thermal parameters, H-atom parameters and least-squares-planes data have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44295 (10 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.
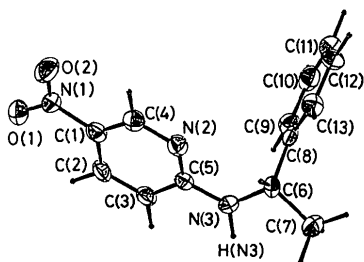
Table 1. *Fractional atomic coordinates* ($\times 10^4$) *and equivalent isotropic thermal parameters* (Å^2) *with e.s.d.'s in parentheses*

$$B_{eq} = \tfrac{4}{3}\sum_i\sum_j B_{ij}(\mathbf{a}_i\cdot\mathbf{a}_j).$$

	x	y	z	B_{eq}
O(1)	323 (3)	7818 (13)	3637 (12)	6.4
O(2)	1120 (4)	7539 (13)	6805 (12)	7.0
N(1)	835 (4)	6907 (12)	4812 (13)	4.7
N(2)	1988 (3)	2175 (13)	4237 (12)	4.4
N(3)	2006 (4)	−312 (12)	1139 (13)	4.7
C(1)	1101 (4)	4967 (16)	3802 (15)	4.1
C(2)	771 (4)	4137 (17)	1588 (16)	4.5
C(3)	1067 (4)	2336 (16)	710 (14)	4.2
C(4)	1693 (5)	3956 (16)	5098 (15)	4.4
C(5)	1693 (4)	1423 (15)	2060 (15)	3.8
C(6)	2720 (4)	−1178 (15)	2201 (15)	4.0
C(7)	2833 (5)	−3280 (16)	856 (19)	5.7
C(8)	3374 (4)	280 (14)	1866 (15)	3.7
C(9)	3419 (5)	1471 (16)	−283 (16)	4.6
C(10)	4060 (5)	2653 (19)	−493 (17)	5.4
C(11)	4639 (5)	2662 (20)	1275 (18)	5.8
C(12)	4611 (5)	1433 (19)	3366 (17)	5.7
C(13)	3968 (5)	223 (17)	3620 (15)	4.8

Table 2. *Bond lengths* (Å) *and angles* (°) *for non-*H *atoms with e.s.d.'s in parentheses*

O(1)—N(1)	1.221 (9)	O(2)—N(1)	1.222 (10)
N(1)—C(1)	1.447 (12)	N(2)—C(4)	1.351 (12)
N(2)—C(5)	1.338 (10)	N(3)—C(5)	1.353 (12)
N(3)—C(6)	1.468 (10)	C(1)—C(2)	1.393 (12)
C(1)—C(4)	1.384 (12)	C(2)—C(3)	1.366 (14)
C(3)—C(5)	1.414 (11)	C(6)—C(7)	1.546 (14)
C(6)—C(8)	1.519 (12)	C(8)—C(9)	1.395 (12)
C(8)—C(13)	1.367 (11)	C(9)—C(10)	1.386 (14)
C(10)—C(11)	1.353 (13)	C(11)—C(12)	1.379 (15)
C(12)—C(13)	1.403 (14)		

O(1)—N(1)—O(2)	123.0 (8)	O(1)—N(1)—C(1)	117.9 (7)
O(2)—N(1)—C(1)	119.2 (7)	C(4)—N(2)—C(5)	117.8 (7)
C(5)—N(3)—C(6)	122.6 (7)	N(1)—C(1)—C(2)	121.2 (8)
N(1)—C(1)—C(4)	118.0 (7)	C(2)—C(1)—C(4)	120.8 (9)
C(1)—C(2)—C(3)	117.9 (8)	C(2)—C(3)—C(5)	118.8 (8)
N(2)—C(4)—C(1)	121.6 (8)	N(2)—C(5)—N(3)	118.0 (7)
N(2)—C(5)—C(3)	123.1 (8)	N(3)—C(5)—C(3)	118.9 (7)
N(3)—C(6)—C(7)	106.6 (7)	N(3)—C(6)—C(8)	112.5 (7)
C(7)—C(6)—C(8)	110.0 (7)	C(6)—C(8)—C(9)	122.0 (7)
C(6)—C(8)—C(13)	117.9 (8)	C(9)—C(8)—C(13)	119.7 (8)
C(8)—C(9)—C(10)	118.0 (8)	C(9)—C(10)—C(11)	122.6 (10)
C(10)—C(11)—C(12)	119.8 (10)	C(11)—C(12)—C(13)	118.6 (8)
C(8)—C(13)—C(12)	121.2 (9)		

Fig. 1. A perspective view of the molecule, with the numbering scheme.
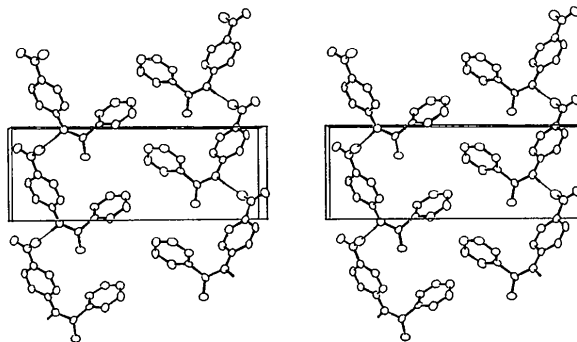

Fig. 2. Stereoscopic view of the molecular packing (viewed down the *c* axis). The hydrogen bonds are represented by fine lines.

Packing of the molecules in the crystal is shown in Fig. 2. The molecules are linked into infinite chains by N(3) $(x, y+1, z+1)$–H(N3) $(x, y+1, z+1)\cdots$O(2) (x, y, z) hydrogen bonds [N(3)$\cdots$O(2) 3·04 (1), N(3)—H(N3) 1·09 (7), H(N3)$\cdots$O(2) 1·97 (7) Å, N(3)—H(N3)$\cdots$ O(2) 170 (5)°]. The chains are parallel to the [011] direction and the symmetry-equivalent [01$\bar{1}$] direction in the layer perpendicular to the a axis. On the other hand, in the direction parallel to the a axis, packing of molecules is governed only by relatively weak van der Waals contacts. As is expected from these results, the easy cleavage plane is the (100) plane.

It is noticeable that the charge-transfer axis in the pyridine ring [N(3)—C(5)—C(1)—N(1)] of each molecule makes a relatively small angle with the twofold screw axis (b axis). For this reason, single crystals of this compound may be expected to have very large second-order nonlinear optical susceptibility.

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Structure of a Potential Cross-Linking Reagent: 2,2'-Bis(methoxymethylene)-2,2'-sulfonyldiacetonitrile (MMSBA)

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Abstract. The title compound (I) was prepared as a potential cross-linking reagent for nucleic acids and/or proteins. The compound is a stable, but reactive, crystalline solid which can be stored indefinitely upon adequate protection. The reagent reacts with amine nucleophiles – primary, secondary as well as heterocyclic – to afford bis-enamines. $C_8H_8N_2O_4S$, $M_r = 228·23$, monoclinic, $C2/c$, $a = 18·031$ (5), $b = 9·372$ (3), $c = 13·455$ (6) Å, $\beta = 108·08$ (5)°, $V = 2161$ (1) Å^3, $Z = 8$, $D_x = 1·40$ g cm^{-3}, $\lambda(\text{Mo } K\alpha) = 0·71069$ Å, $\mu = 2·81$ cm^{-1}, $F(000) = 944$, $T = 295$ K. Final $R = 0·041$ for 1033 observed reflections. The bond distances in (I) are: S—O, 1·424 (3); S—C, 1·737 (4); C—CN, 1·417 (5); C=C, 1·342 (5); =C(H)—O, 1·295 (4); and C≡N, 1·138 (5) Å. The diagonal distance, α–α', between the two *trans* C atoms is 4·976 Å.

Introduction. The study of the synthesis, structure and chemistry of novel bifunctional organic reagents is

important in view of their potential to cross link biomolecules such as nucleic acids and proteins. Cross-linking agents have long been instrumental in investigations relating to structure and function of both nucleic acids and proteins. The recent isolation and structural characterization of a covalent cross-link adduct between the antibiotic mitomycin C and the interstrand guanine residues of DNA have provided direct proof for the long-suspected mode of action of this natural anticancer drug (Tomasz, Lipman, Chowdary, Pawlak, Verdine & Nakanishi, 1987). The above finding has spurred renewed interest in the design and synthesis of synthetic cross-linking reagents. Psoraleins, an important class of photochemical cross-linking reagents, have been extensively employed in probing both static and dynamic secondary structural features of DNA/RNA in viral, bacterial and mammalian systems as well as for studying functional properties such as the role of small nuclear RNA's in processing heteronuclear RNA (Cimino, Gamper, Isaacs & Hearst, 1985). Bifunctional reagents such as glutaraldehyde (Hopwood, Allen & McCabe, 1970), diaspirins