Les groupements organiques $[NH_3(CH_2)_3NH_3]^{2+}$, présents sous forme de zwittérion, se lient aux chaines précédentes par des liaisons par pont hydrogène du type N—H···O, lesquelles assurent la cohésion du réseau tridimensionel.

Les distances et les angles décrivant les liaisons hydrogènes sont donnés dans le Tableau 2. L'ensemble des distances O(N)···O varie entre 2,602 (3) et 2,922 (3) Å. La force de la liaison hydrogène peut être interprétée selon les critères portant sur les distances O(N)···O: $d_{O(N)···O} > 2,73$ Å faible; $d_{O(N)···O} < 2,73$ Å forte (Brown, 1976; Blessing, 1986). Ainsi parmi les neufs liaisons par pont hydrogène appartenant aux trois types N—H···O, O(W)—H···O et O—H···O, quatre sont considérées fortes (cf. Tableau 2). Les groupements ammoniums (—NH_3^+) engagent chacun une liaison hydrogène forte N(1)···O(3) 2,964 (3) et N(2)···O(4) 2,725 (3) Å et deux liaisons faibles N···O > 2,73 Å. L'une des liaisons par pont hydrogène engagée par la molécule d'eau O(W)···O(1) 2,708 (4) Å constitue la troisième liaison hydrogène forte. La quatrième liaison forte concerne le troisième type de liaison P—O(H)···O(P) [O(2)···

O(1) 2,602 (3) Å], liant les groupements acides $[HPO_4^{2-}]$.

La structure cristalline contient le même nombre de donneurs et d'accepteurs engagés dans les liaisons par pont hydrogène: six (N)H, deux O(W)H, un O(H) comme donneurs et huit O et un O(W) comme accepteurs. La molécule d'eau joue à la fois le rôle de donneur et d'accepteur.

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Structure of N-(o-Carboxyphenyl)iminodiacetic Acid

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Abstract. $C_{11}H_{11}NO_6$, $M_r = 253\cdot0$, monoclinic, $C2/c$, $a = 28\cdot146$ (8), $b = 6\cdot063$ (5), $c = 13\cdot820$ (4) Å, $\beta = 99\cdot51$ (2)°, $V = 2325\cdot61$ Å³, $Z = 8$, $D_x = 1\cdot445$, D_m (flotation) $= 1\cdot442$ g cm⁻³, λ(Mo $K\alpha$) $= 0\cdot7107$ Å, $\mu = 1\cdot24$ cm⁻¹, $F(000) = 1056$, $T = 295$ K, $R = 0\cdot0397$ with unit weights for 1609 unique reflections. The phenyl group, two glycinato arms and one H atom are attached to the N atom in a zwitterionic configuration. The N—H group and the hydroxyl groups of the glycinato arms are involved in hydrogen bonding.

Introduction. N-(o-Carboxyphenyl)iminodiacetic acid (cpida), below, is an amino polycarboxylic acid which forms many metal complexes. The structure

analysis of the title compound was undertaken in order to determine the geometry of the ligand and compare it with its metal complexes.
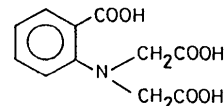
Experimental. The compound was synthesized by the literature method (Young & Sweet, 1958; Tomita, Kyuno & Tsuchia, 1969) and recrystallized, from hot water in which activated charcoal had been added, to obtain transparent crystals. A crystal of dimensions $0\cdot40 \times 0\cdot28 \times 0\cdot38$ mm was chosen for data collection on an Enraf-Nonius CAD-4 diffractometer, using graphite-monochromated Mo $K\alpha$ radiation, ω–2θ

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scan. Unit-cell parameters were determined by the least-squares method using 12 reflections in the range $3 < \theta < 12°$. Intensity of one standard reflection collected every hour showed no significant changes. 4493 reflections were collected for a triclinic cell in the range $2 < \theta < 25°$ ($0 \le h \le 7$, $-16 \le k \le 16$, $-17 \le l \le 17$, resolution $d = 0.84$ Å), transformed to monoclinic symmetry and symmetry-equivalent reflections were averaged. 1752 unique reflections were obtained and used for structure solution, $R_{int} = 0.086$. The structure was solved by direct methods using *SHELXS*86 (Sheldrick, 1985). H atoms were located from difference Fourier maps. The structure was refined by the full-matrix least-squares method based on *F* values using anisotropic thermal parameters for non-H atoms and isotropic thermal parameters for H atoms. When all the atoms were located and refined ($R = 0.0847$), the difference map showed a peak with $\Delta\rho = 2.33$ e Å^{-3}. It was assumed that this peak corresponded to randomly distributed water molecules, statistically averaged. Thermogravimetric and differential thermal analyses indicated that there was about 1.7% weight loss corresponding to 0.25 of a water molecule. As the peak corresponded to a special position, site occupation was taken as 0.5 and half of the real part of the scattering factors of oxygen were taken and refined anisotropically with constraints on U_{12} and U_{23} (Peterse & Palm, 1966). The parameters for the unique H atom of the water molecule were fixed. The *R* value decreased by about 4% and there was no significant residue in the difference map. In the final calculation reflections with $|F_o| < 5\sigma(|F_o|)$ and those for which $(\Delta F)/\sigma(|F_o|) > 4.0$ were omitted. 212 parameters were refined. $(\Delta/\sigma)_{max} = 0.147$, $(\Delta\rho)_{max} = 0.19$, $(\Delta\rho)_{min} = -0.16$ e Å^{-3}, $R = 0.0397$ with unit weights for 1609 observed unique reflections. *SHELX*76 (Sheldrick, 1976) program was used for refinement and the source for atomic scattering factors. A CYBER 180 computer was used for all calculations. C—H, O—H distances are in the range 0.92 (4)–1.14 (3) and 0.98 (4)–1.09 (3) Å respectively. The O—H distance in the water molecule is 0.67 (4) Å.

Discussion. The atomic coordinates and equivalent isotropic temperature factors of non-H atoms are given in Table 1 (Swaminathan, 1989) and important bond lengths, bond angles and torsion angles are given in Table 2.* Fig. 1 is a *PLUTO* (Motherwell & Clegg, 1978) diagram of the molecule. It is seen that the molecule exists as a zwitterion, as found for one

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* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53120 (12 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. *Final atomic coordinates* ($\times 10^4$) *and equivalent isotropic temperature factors* (Å$^2 \times 10^3$) *with e.s.d.'s in parentheses*

$$U_{eq} = (1/3)\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	*y*	*z*	U_{eq}
N	3986 (1)	5297 (3)	3303 (1)	32 (2)
C(1)	3487 (1)	5482 (4)	3502 (2)	33 (2)
C(2)	3177 (1)	6986 (4)	2991 (2)	46 (3)
C(3)	2716 (1)	7204 (5)	3216 (2)	56 (3)
C(4)	2573 (1)	5959 (5)	3944 (2)	55 (3)
C(5)	2890 (1)	4475 (5)	4449 (2)	48 (3)
C(6)	3353 (1)	4181 (4)	4241 (2)	34 (2)
C(7)	3677 (1)	2522 (4)	4822 (2)	38 (2)
C(8)	4280 (1)	7227 (4)	3739 (2)	39 (2)
C(9)	4806 (1)	6694 (4)	3914 (2)	38 (2)
C(10)	4025 (1)	4955 (4)	2250 (2)	39 (2)
C(11)	3840 (1)	2711 (4)	1911 (2)	38 (2)
O(1)	3506 (1)	1368 (3)	5416 (1)	61 (2)
O(2)	4110 (1)	2427 (3)	4685 (1)	45 (2)
O(3)	4968 (1)	4906 (3)	3786 (1)	52 (2)
O(4)	5054 (1)	8426 (3)	4254 (2)	62 (2)
O(5)	3710 (1)	1393 (3)	2461 (1)	64 (2)
O(6)	3843 (1)	2399 (3)	988 (1)	55 (2)
O*W*	5000 (0)	705 (12)	2500 (0)	129 (13)

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

C(1)—N	1.481 (3)	C(7)—C(6)	1.500 (3)
C(8)—N	1.501 (3)	O(1)—C(7)	1.236 (3)
C(10)—N	1.492 (3)	O(2)—C(7)	1.265 (3)
C(2)—C(1)	1.373 (3)	C(9)—C(8)	1.496 (3)
C(6)—C(1)	1.390 (3)	O(3)—C(9)	1.200 (3)
C(3)—C(2)	1.390 (4)	O(4)—C(9)	1.303 (3)
C(4)—C(3)	1.369 (4)	C(11)—C(10)	1.504 (3)
C(5)—C(4)	1.373 (4)	O(5)—C(11)	1.200 (3)
C(6)—C(5)	1.392 (3)	O(6)—C(11)	1.290 (3)

C(8)—N—C(1)	110.1 (2)	C(7)—C(6)—C(5)	118.9 (2)
C(10)—N—C(1)	114.7 (2)	O(1)—C(7)—C(6)	117.7 (2)
C(10)—N—C(8)	112.1 (2)	O(2)—C(7)—C(6)	117.8 (2)
C(2)—C(1)—N	119.7 (2)	O(2)—C(7)—O(1)	124.5 (2)
C(6)—C(1)—N	118.3 (2)	C(9)—C(8)—N	111.2 (2)
C(6)—C(1)—C(2)	121.9 (2)	O(3)—C(9)—C(8)	124.1 (2)
C(3)—C(2)—C(1)	119.0 (3)	O(4)—C(9)—C(8)	109.8 (2)
C(4)—C(3)—C(2)	120.7 (3)	O(4)—C(9)—O(3)	126.0 (2)
C(5)—C(4)—C(3)	119.3 (3)	C(11)—C(10)—N	110.6 (2)
C(6)—C(5)—C(4)	122.0 (3)	O(5)—C(11)—C(10)	122.1 (2)
C(5)—C(6)—C(1)	117.1 (2)	O(6)—C(11)—C(10)	112.5 (2)
C(7)—C(6)—C(1)	124.0 (2)	O(6)—C(11)—O(5)	125.5 (2)

N—C(1)—C(2)—C(3)	−177.3 (2)
N—C(1)—C(6)—C(5)	176.6 (2)
C(4)—C(5)—C(6)—C(7)	−179.7 (3)
C(1)—C(6)—C(7)—O(2)	5.9 (4)
N—C(8)—C(9)—O(4)	177.3 (2)
N—C(10)—C(11)—O(6)	176.4 (2)

molecule of anthranilic acid (Brown & Ehrenberg, 1985). Structures of Cr(bipy)(cpida) (Swaminathan, Sinha, Chatterjee, Patel & Padmanabhan, 1988) and Cr(H$_2$O)$_2$(cpida) (Swaminathan, Sinha, Chatterjee, Yadava & Padmanabhan, 1989) have been reported. The values given below in parentheses are those for Cr(bipy)(cpida) and Cr(H$_2$O)$_2$(cpida), respectively. The distance C(7)—O(2) = 1.265 (3) Å is shorter than its corresponding values [1.292 (4), 1.292 (6) Å] in these complexes whereas the C(7)—O(1) distance [O(1) does not take part in coordination] is almost
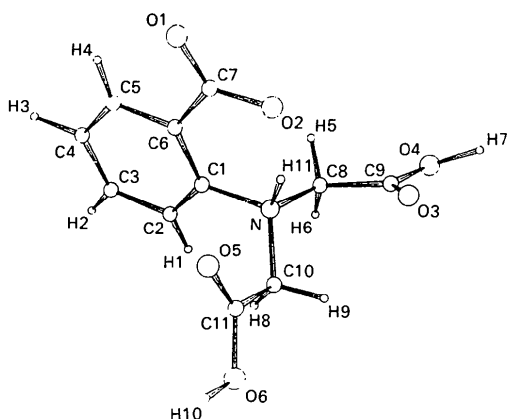
Fig. 1. General view of the molecule.

equal. Other bond lengths in this ligand almost match the corresponding values in the other metal complexes. The phenyl ring is planar with an average C—C distance of 1·381 (4) Å. The angle O(2)—C(7)—C(6) is = 117·8 (2)° [119·5 (3), 120·0 (4)°] whereas other O—C—C angles involving O(4) and O(6) are 109·8 (2)° [114·3 (3), 117·0 (4)°] and 112·5 (2)° [117·1 (3), 117·3 (4)°] respectively. The interplanar angle between the phenyl ring and the carboxylate group is 3·2 (3)° [29·6 (2), 18·9 (3)°]. The corresponding angle in anthranilic acid is 1·71 (5)°. The torsion angle C(1)—C(6)—C(7)—O(2) is = 5·9 (4)° [29·9 (3), 27·7 (6)°]. These values indicate that the carboxylate group is almost coplanar with the phenyl ring in free cpida. The torsion angles

N—C(8)—C(9)—O(4) and N—C(10)—C(11)—O(6) with values −2·7 (2) [16·3 (3), 22·9 (6)] and −3·6 (2)° [−17·6 (5), −24·1 (6)°] indicate that the glycinato arms are almost planar. In general, the strain in the glycinato arms increases when they are coordinated. The N—H(11) group is involved in intramolecular hydrogen bonding with O(2). The distance N···O(2) is 2·564 (3) Å. The hydroxyl groups are involved in intermolecular hydrogen bonding with values O(4)···O(2^{iii}) 2·610 (4) and O(6)···O(1^{iv}) 2·548 (3) Å where iii, iv represent the symmetry codes $-x + 1$, $-y + 1$, $-z + 1$ and x, $-y$, $z + \frac{1}{2} - 1$, respectively.

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Structure of the
3-(Methoxycarbonylamino)phenyl Ester of N-(3-Methylphenyl)carbamate

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Abstract. Methyl N-{3-[N-(3-methylphenyl)carbamoyloxy]phenyl}carbamate, $C_{16}H_{16}N_2O_4$, $M_r = 300·3$, triclinic, $P\bar{1}$, $a = 11·528$ (1), $b = 12·885$ (2), $c = 5·086$ (2) Å, $\alpha = 90·47$ (3), $\beta = 101·62$ (3), $\gamma = 94·97$ (1)°, $V = 736·93$ (7) Å^3, $Z = 2$, $D_x = 1·352$ g cm^{-3}, $\lambda(\text{Cu } K\alpha) = 1·5418$ Å, $\mu = 7·26$ cm^{-1}, $F(000) = 316$, $T = 293$ K, final $R = 0·077$ for 2611 observed independent diffractometer-measured intensity data. Conformations around the carbamate groups and benzene rings are similar to those found