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APPENDIX

The Preparation of *N*-(*o*-Aminophenyl)aminomethanephosphonic Acid

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Chloromethanephosphonic acid was made by the method of Schwarzenbach *et al.* (1949), i.e. by hydrolysing chloromethanephosphonyl dichloride, which was kindly given by Dr. S. G. Warren, and had been made from formaldehyde and phosphorus trichloride. Chloromethanephosphonic acid (5 g) was heated with *o*-phenylenediamine (25 g) at 200°C for 5 min. The mixture was poured into 250 ml of water containing 10 g of NaHCO₃, and this was then heated until all the excess of phenylenediamine had dissolved. The solution was cooled to 0°C, when much of the phenylenediamine crystallized and was filtered off. The solution was extracted with ethyl acetate

(2 × 250 ml) and adjusted to pH 4.1 with 10M-HCl, when the product crystallized. The yield was 5.4 g (70%) (Found: C, 42.3; H, 5.8; N, 12.8; P, 14.5; C₇H₁₁N₂O₃P requires C, 41.6; H, 5.4; N, 13.8; P, 15.3%). The i.r. spectrum of a Nujol mull showed peaks at 3320 (NH), 2100–3000 (broad, NH₃⁺ or NH₂⁺), 1640 and 1535 (NH₃⁺ or NH₂⁺) and 1140 cm⁻¹ (broad, PO₂⁻). The n.m.r. spectrum taken at 100 MHz of a solution in CF₃-CO₂H showed resonance at τ = 2.24 (broad singlet, 4H, aromatic H) and at τ = 5.81 (doublet, 2H, J_{HP} = 13 Hz, P-CH₂-N). Schwarzenbach, G., Ackermann, H. & Ruskstuhl, P. (1949) *Helv. Chim. Acta* **32**, 1175–1186