carboxamide (VII) (5 per cent), m.p. 348°, and 27 per cent of compound (III). When (VI) was similarly treated with aqueous-ethanolic ammonia at 120°, 16 per cent of (III) was obtained, together with 63 per cent of (VII).

T. HARGREAVES
A. T. PETERS

Colour Chemistry Department,
University of Leeds.

Structure of Azide Complexes

AZIDES of many metals, for example, silver, mercury, lead, copper and zinc, are extremely explosive. It has been found, however, that amine complexes of these azides are much more stable. Investigations of the crystal structure of such compounds are being carried out at this Institute.

The compound $\text{Zn}(\text{N}_3)_2(\text{NH}_3)_2$ has been found to have an orthorhombic unit cell, and to belong to the space group $Pna2_1$, the axial ratios being $a = 9.52 \text{ \AA}$, $b = 19.03 \text{ \AA}$, $c = 7.20 \text{ \AA}$. The cell contains eight formula units. The structure is under refinement.

Table 1. SPACE GROUP $Pnma$

Atom	Position	No.	x	y	z
Cu	c	4	0.226	0.250	0.359
N	c	4	0.351	0.250	0.211
N	c	4	0.233	0.250	0.141
N	c	4	0.122	0.250	0.071
N	c	4	0.119	0.250	0.511
N	c	4	0.240	0.250	0.584
N	c	4	0.346	0.250	0.656
NH ₃	d	8	0.231	0.517	0.362

The structure of $\text{Cu}(\text{N}_3)_2(\text{NH}_3)_2$ has been fully determined by the usual X-ray methods. The reliability index R is calculated to be 0.080. The compound has orthorhombic symmetry and belongs to the space group $Pnma$, the dimensions of the unit cell being $a = 6.38 \text{ \AA}$, $b = 7.43 \text{ \AA}$, $c = 12.78 \text{ \AA}$. The cell contains four formula units. Parameter values for all atoms with the exception of the hydrogen atoms are given in Table 1.

Fig. 1. Configuration of $\text{Cu}(\text{N}_3)_2(\text{NH}_3)_2$. Black, copper; white, nitrogen (in N_3 group); shaded, NH_3 . Distances in $\text{\AA}$. The atoms lie in the same plane, except for the NH_3 groups, which are placed above and below copper.

The configuration of the compound can be seen in Fig. 1.

CYRILL BROSSET
INGELA AGRELL

Division of Inorganic Chemistry,
Chalmers University of Technology,
Gothenburg, Sweden.

Isolation of Tryptophan from Air-cured Tobacco by Gel-filtration

TRYPTOPHAN is generally regarded as one of the less-prominent amino-acids of *Nicotiana tabacum*, but has been found in both free^{1,2} and bound³ forms. Detection of tryptophan, in most instances, has been accomplished by conventional paper chromatographic resolution of mixtures of amino-acids contained in selective extracts or hydrolysates. In the continuation of an investigation on polymeric browning reaction pigments of aged 'Burley' tobacco³ (U.S. type 31), an air-cured type, a small amount of free tryptophan was isolated by gel-filtration of concentrated dialysates of aqueous extracts applied to a column of 'Sephadex G-25' (Pharmacia, medium grade), a cross-linked dextran. Tryptophan was strongly adsorbed on the gel matrix. Separation of a thymine fraction, to be reported in detail elsewhere, was feasible since the pyrimidine derivative was less strongly adsorbed than tryptophan. These observations are in conformity with those previously noted by Gelotte⁴ and Porath⁵ regarding the behaviour of aromatic amino-acids and heterocyclic substances on 'Sephadex'. In regard to the resolution of complex mixtures of tobacco leaf constituents in aqueous media, the gel-filtration technique provides a useful approach in which both the molecular sieve and adsorption effects can be utilized to advantage. It has been of particular value in the resolution of mixtures of complex pigment polymers, comprised of phenolic and proteinaceous moieties, in which heterocyclic and other nitrogenous substances of a monomeric nature occur as contaminants, for example, the type of mixture described herein from which tryptophan was isolated.

Samples (15 g) of the lamina portion of the leaf of aged 'Burley' tobacco, ground (1 mm particle size) and pre-extracted in a Soxhlet apparatus successively with pentane, ether, chloroform, acetone, and ethanol, were next extracted with de-ionized water in a Waring blender and filtered by suction through a pad of powdered cellulose (Whatman, standard grade). Concentrated filtrates were dialysed ('Visking' dialysis tubing) against de-ionized water in suitable glass cylinders. Dialysate fractions collected during the first 20 h, representing six changes of water and containing, *inter alia*, considerable amounts of calcium and potassium salts of citric, malic, and glycolic acids, were concentrated *in vacuo* at 37° C and run through a chromatographic column (40 × 600 mm) containing a bed of 100 g of 'Sephadex G-25' (ref. 6). Tryptophan, in a comparatively pure form, was eluted from the column with de-ionized water after the polymeric brown and yellow pigments and a small well-separated fraction of thymine had passed through. The amino-acid was detected in eluate fractions by ultra-violet spectroscopy (Beckman DK-2 ratio recording spectrophotometer).

The ultra-violet absorption spectrum of tryptophan isolated from the tobacco, identical with that of the authentic compound, exhibited maxima at 220.5, 273, 280, and 289.5 mμ in ethanol. Additional results concerning the identification of tryptophan were provided by two-dimensional paper chromatography. The following single phase solvent systems, prepared on a v/v basis, with corresponding R_F values, were used: *n*-butanol-acetic acid-water (12 : 3 : 5) R_F 0.54, followed in the second direction by *n*-butanol-methyl ethyl ketone-ammonium hydroxide-water (5 : 3 : 1 : 1) run twice R_F 0.44; and *sec*-butanol-formic acid-water (15 : 3 : 2) R_F 0.54, followed in the second direction by *n*-butanol-pyridine-water (1 : 1 : 1) R_F 0.66. Chromatograms (Whatman No. 1 paper) were run by the ascending technique for a period of 20 h. Tryptophan, detected on chromatograms by a ninhydrin spray⁷, was examined by co-chromatography with the authentic compound in all solvent systems.

The small amount of free tryptophan so isolated, estimated to be in the order of 0.003 per cent, represents only that fraction extractable with water subsequent to