Table 1

Substance	Molecular weight	Value of optical density 200 μgm. of sulphonate	Optical density × molecular weight	
n-Tetradecylbenzene-sulphonate	376	0·935	352	Average value 347
n-Dodecylbenzene-sulphonate	348	1·010	351	
n-Decylbenzene-sulphonate	320	1·075	344	
n-Octylbenzene-sulphonate	292	1·166	340	
n-Hexylbenzene-sulphonate	264	1·138	300	86% of av.
n-Butylbenzene-sulphonate	236	0·106	22	6% ,,
α-Dimethyldecyl-benzenesulphonate	346	0·926	320	93% ,,
Alkylbenzenesulphonate of commerce	356	0·926	330	96% ,,
'Manoxol O.T.'	444	0·780	346	100% ,,

density multiplied by the molecular weight is approximately a constant for the members of the series from *n*-octylbenzene upwards. This is a useful check on the molecular weight of an unknown compound of similar composition. Figures for α-dimethyldecylbenzene sodium sulphonate, the alkylbenzene sodium sulphonate of commerce and for manoxol (sodium-dioctylsulphosuccinate) are included for purposes of comparison. The sodium sulphonates below butyl benzene in the series give no colour under the conditions of the Longwell–Maniece test.

Paper chromatography of some alkyl-aryl sodium sulphonates. Commercial detergents of the alkyl-aryl sulphonate type are difficult to study by paper chromatography, and special methods such as that of F. Franks[2] have been devised for their examination. The difficulties appear to be due to their detergent properties which cause them to remove other substances, including other detergents, from the paper and to carry them forward with their own movement. These remarks also apply to α-dimethyldecylbenzene-sodium sulphonate and to detergents of the 'Teepol' type. With the straight-chain alkyl-aryl sulphonates there are no such effects and chromatography proceeds normally. Values of R_F for four such compounds in various mixtures of alcohol and water are given in Table 2. Whatman No. 4 paper was used and the spots were shown up with a solution of rhodamine 6*B* used as a spray at a concentration of 0·05 per cent w/v.

Table 2. R_F VALUES

Substance	Per cent alcohol/water by volume					
	10	30	35	40	50	100
n-Octylbenzene-sulphonate	0·67	0·81	0·82	0·83	0·87	0·89
n-Decylbenzene-sulphonate	0·03	0·54	0·74	0·75	0·84	0·89
n-Dodecylbenzene-sulphonate	Nil	0·22	0·23	0·23	0·76	0·89
n-Tetradecylbenzene-sulphonate	Nil	0·06	0·06	0·10	0·20	0·89

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[1] Longwell, J., and Maniece, W. D., *Analyst*, **80**, 167 (1955).
Franks, F., *Nature*, **176**, 693 (1955).

Labelling of Radiographic Contrast Media with Iodine-131

THE rapid development and general acceptance of contrast media represents one of the outstanding successes of diagnostic röntgenology. Roentgen contrast media are generally di- or tri-iodo derivatives of benzene or pyridine, containing further substituents influencing solubility. The most widely used contrast compounds are 3,5-diiodo-4-pyridon-N-acetic acid as its diethanolamine salt ('Iodopyracet', 'Diodrast', 'Diodone', 'Ioduron', etc.), and N-methyl-3,5-diiodo-chelidamate as its disodium salt ('Neo-Iopax', 'Iodoxyl', 'Uropac', etc.). A new compound of the urotropic class is sodium-3-acetyl-amino-2,4,6-triiodobenzoate known as 'Triopac', 'Urokon', 'Triumbren' and diacetylamino-2,4,6-triiodobenzoic acid, the methylglucamate of which is known as 'Urografin'. The hepatotropic contrast media are represented by N,N′-adipyl-*bis*(3-amino-2,4,6-triiodo)-benzoic acid as methylglucamate ('Biligrafin' or 'Chولografin') and 2-ethyl-3-(3′-amino-2,4,6-triiodophenyl)-propionic acid ('Cistobil', 'Telepaque').

The application of these contrast media for diagnostic purposes gives only a static picture. Pathological changes are diagnosed on the basis of permanent differences in the morphological picture. It is not generally possible to gain information on the functional state of the organs by these means.

The labelling of contrast compounds with iodine-131 opens the possibility of investigating the functional state of the organs by measuring the amount of the compound applied and excreted and its concentration. Indicator diagnosis with the aid of labelled contrast media allows the registration and analysis of the dynamics of processes, and therefore combines both possibilities.

In this communication a method for labelling the more important contrast media with iodine-131 is presented; these are 3,5-diiodo-4-pyridon-N-acetic acid ('Ioduron', 'Diodon'), 3-acetylamino-2,4,6-triiodobenzoic acid ('Triopac', 'Triumbren'), N,N′-adipyl-*bis*(3-amino-2,4,6-triiodo)-benzoic acid ('Biligrafin') and 3,5-diacetylamino-2,4,6-triiodobenzoic acid ('Urografin'). An exchange reaction method was used. The use of labelled contrast media ('Triopac') for determining the vitality of bone tissue will be described elsewhere.

Sodium-3-acetylamino-2,4,6-triiodobenzoate-^{131}I, the preparation used, was 'Triopac-400' (Cilag A.G., Schaffhausen, Switzerland) or 'Triumbren' (Spofa, Prague); 2 ml. of the solution contains 1·292 gm. of the sodium salt.

To 2 ml. of 'Triopac-400' solution were added 8 ml. acetate buffer solution *p*H 5, 1 ml. potassium iodide carrier solution (20 mgm. in 100 ml. water) and 4 ml. potassium iodide-131 solution with a radioactivity of 9 mc. The mixture was heated under reflux on the water-bath for 12 hr. The solution was then allowed to cool to room temperature and free 3-acetylamino-2,4,6-triiodobenzoic acid precipitated by addition of concentrated phosphoric acid to *p*H 1. The precipitate was filtered by suction and washed with cold water until the volume of the filtrate was exactly 32 ml. The loss of radioactivity amounted to 1·5 per cent. The precipitate was dissolved in a slight excess of ammonia and the precipitation repeated twice (yield, 1·12 gm. of the free acid (89 per cent) and 90 per cent of the original radioactivity). The purity of the 3-acetylamino-

2,4,6-triiodobenzoic acid was controlled by chromatography in butanol/ammonia/water (50 : 5 : 45) on Whatman No. 3 paper. The acid was detected by radioactivity measurement, and the potassium iodide by oxidation to iodine with 0·1 *N* ferrous nitrate solution containing 3 per cent of hydrogen peroxide and starch. The recrystallized products were not contaminated by potassium iodide.

3,5-Diacetylamino-2,4,6-triiodobenzoic acid-^{131}I, 'Urografin' solution 76 per cent (Schering A.G., Berlin), containing 2 gm. of the sodium and 13·2 gm of the methyl-glucamine salt in 20 ml. (2 ml. of the 'Urografin' solution correspond to 1·193 gm. of 3,5-diacetylamino-2,4,6-triiodobenzoic acid) were iodinated by the exchange reaction described above. The yield was 0·775 gm. (65 per cent) of the acid and 66·4 per cent of the original radioactivity. The product was chromatographically pure.

3,5-Diiodo-4-pyridon-N-acetic acid-^{131}I, 70 per cent 'Ioduron' (Cilag A.G.) or 'Diodon' (Spofa) was employed as the diethanolamine salt. 2 ml. 70 per cent 'Ioduron', corresponding to 1·11 gm. of the free acid, was labelled as described above. The yield was 67 per cent of the acid and 70 per cent of the original radioactivity. The product was chromatographically pure.

5 ml. of a 30 per cent solution of the methyl-glucamine salt of 3,3′-(adipyl-diimino)*bis*-2,4,5-triiodobenzoic acid 'Biligrafin' (Schering A.G., Berlin) containing 1·125 gm. of the free acid was labelled as above. The yield was 45 per cent of the acid and 46·5 per cent of the original radioactivity. The product was chromatographically pure. Identical yields were obtained with the corresponding Spofa product.

The radioactivity of the samples was determined in liquid samples with the aid of a scintillation counter.

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Infra-red and the Near Ultra-violet Absorption Spectra of Polyphenyl Derivatives of the Elements of Groups IV*b* and V*b*

WE have been investigating the infra-red and the near ultra-violet absorption spectra of several polyphenyl derivatives of the elements of groups IV*b* and V*b*. In the case of the derivatives of the V*b* elements, we have studied the spectra of derivatives where the central atoms possess and do not possess unshared *p*-electrons (derivatives of the atoms in their 'trivalent' and 'pentavalent' states respectively). In addition, we have been studying the spectra of *o*-, *m*- and *p*-substituted tritylbenzenes (trityl = triphenylmethyl). The ultra-violet spectra were recorded in alcohol solutions, and the infra-red spectra in mulls and also in solutions.

Our results on the ultra-violet spectra of the triphenyl derivatives of the V*b* elements ($M\,Ph_3$ where M = V*b* element) agree well with those of Jaffe[1] and show trends similar to the derivatives of the type $Ph\,M\,Me_2$ studied by Bowden and Braude[2]. The ultra-violet spectra of the triphenyl and tetraphenyl halides ($Ph_n M\,X_m$ where $X = \mathrm{Cl}$ or Br and $n + m = 5$) are similar to those of the other derivatives of the pentavalent atoms[1–4]. In general, the phenyl derivatives of the V*b* elements in the trivalent state exhibit only one strong absorption band ($n \rightarrow \pi^*$) while the pentavalent atom compounds show vibrational structure ($\pi \rightarrow \pi^*$) similar to the secondary band (*B*-band at 2500 A.) of benzene itself. This indicates that the unshared *p*-electrons in the trivalent atom derivatives participate in resonance interaction with the π-electrons of the benzene system. This is further demonstrated by the progressive bathochromic shifts observed with the increase in the number of phenyl groups on the central atom. Such variations in spectra are considered to be proportional to the magnitude of the resonance interaction of the phenyl groups with the central atom[5]. The positions of the secondary bands of aniline, diphenylamine and triphenylamine are found to be 2800, 2850 and 2970 A. respectively. The primary band (*E*-band) seems to vary little, having the values of 2300 and 2280 A. for aniline and triphenylamine. Jaffe[1] did not seem to observe such variations in the secondary bands in the series $Ph\mathrm{N}Me_2$, $Ph_2\mathrm{N}Me$ and $\mathrm{N}Ph_3$ because of the bathochromic shifts due to the methyl groups. He, however, obtained a progressive increase in the primary band wave-length with the number of the methyl groups. It is interesting to see that in spite of the bathochromic shift caused by the methyl groups, $\mathrm{P}\,Ph_3$ absorbs around 2610 A. compared with 2510 A. in the case of $Ph\,\mathrm{P}\,Me_2$. The intensity is also considerably greater in the former compound. The arsenic and antimony derivatives also show similar variations.

Among the $M\,Ph_3$ derivatives of the V*b* elements, $\mathrm{N}\,Ph_3$ shows the largest hyperchromic effect, which seems reasonable, nitrogen being a first-row element. There is a marked decrease in the wave-length and the intensity in the case of $\mathrm{P}\,Ph_3$ and a further decrease in the wave-length in $\mathrm{As}\,Ph_3$. While in phosphorus the 3*d* shell is vacant, in arsenic it is completely filled. But while going from $\mathrm{As}\,Ph_3$ to $\mathrm{Sb}\,Ph_3$ and $\mathrm{Bi}\,Ph_3$ there are progressive shifts to longer wave-lengths (2480, 2560 and 2800 A.). This is considered to be due to the predominating influence of the decreasing ionization potential of the central atom. The importance of the ionization potentials in determining such bathochromic shifts has been recognized[6,7]. $\mathrm{Bi}\,Ph_3$ also shows another shoulder around 2500 A., which may represent dissociation.

The presence of the vibrational structure of benzene in the derivatives of the pentavalent V*b* atoms indicates negligible or no resonance interaction of the central atoms with the π-electrons of the benzene system. The spectra of these derivatives show little variation with the number of phenyl groups attached to the atom. For example, both tetraphenylstiboniumchloride and triphenylstiboniumdichloride show the absorption maxima at 2700, 2630, 2580 and 2530 A. The derivatives of IV*b* elements behave similarly and show almost no variation in their spectra with the number of phenyl groups. Our results on the phenyl derivatives of IV*b* elements agree with those of Milazzo[8] and of Bowden and Braude[2] on derivatives of the type $PhMR_3$ (R = alkyl group). Among IV*b* derivatives, the silicon