NOTES.

A New Gravimetric Method for Palladium Determination. By (MISS) R. SCHÖNTAL.

THE author has already reported (*Mikrochem.*, 1938, **24**, 20) that a solution of *p*-aminoacetophenone is a specific and very sensitive reagent for palladium, forming in dilute neutral or slightly acid solution a voluminous yellow precipitate of the complex $(NH_2\cdot C_6H_4\cdot CO\cdot CH_3)_2,PdCl_2$; this reaction detects 5×10^{-6} g. of palladium chloride per c.c.

The precipitate, insoluble in cold water, dilute acids, alcohols, ether, acetone, and chloroform, is readily filtered, is non-hygroscopic, and can be used for quantitative determination of palladium. The only interfering metals are platinum and cerium : the former is not inimical if the solution is largely diluted, but cerium must be removed.

Procedure.—To the cold palladium solution, 0·1N with respect to hydrochloric acid, is added an excess of the reagent solution (1 g. in 100 c.c. of water containing 2 c.c. of concentrated hydrochloric acid), and after being shaken for a few minutes, the yellow precipitate is collected on a sintered-glass filter (G.1) or a tared double filter-paper, and washed with cold water until the filtrate ceases to give a reaction for the reagent. (For this purpose, a solution of 0·2 g. of cerous sulphate and 5 c.c. of concentrated sulphuric acid in 100 c.c. of water is used; 2×10^{-6} g. of *p*-aminoacetophenone in 1 c.c. give a violet coloration, and larger concentrations afford a violet precipitate.)

The precipitated complex salt is dried to constant weight at 80° :

$$(NH_2\cdot C_6H_4\cdot CO\cdot CH_3)_2,PdCl_2 \times 0{\cdot}2384 = Pd.$$

Alternatively, the precipitate may be ignited to metallic palladium.

The following table gives specimen analyses of solutions of palladium chloride either alone or in presence of other salts.

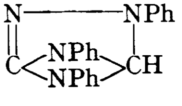
0·2% $PdCl_2$, c.c.	Added salt.	Vol. of soltn., c.c.	Wt. of ppt., g. Calc.	Wt. of ppt., g. Found.	Diff., g.	Error, %.
5	—	20	0·0252	0·0250	−0·0002	− 0·8
10	—	50	0·0504	0·0497	−0·0007	− 1·4
10	—	50	0·0504	0·0505	+0·0001	+ 0·2
20	—	50	0·1008	0·0995	−0·0013	− 1·3
10	5 c.c. NaCl, satd. 5 ,, KCl, ,,	50	0·0504	0·0498	−0·0006	− 1·2
10	5 ,, $MgCl_2$, 10% 5 ,, $CaCl_2$, 10% 5 ,, $BaCl_2$, 10% 5 ,, $Sr(NO_3)_2$, 10%	50	,,	0·0505	+0·0001	+ 0·2
10	10 ,, $FeCl_3$, 10%	50	,,	0·0501	−0·0003	− 0·6
10	5 ,, $Co(NO_3)_2$, 10% 5 ,, $NiCl_2$, 20%	50	,,	0·0499	−0·0005	− 1·0
10	1 ,, $AuCl_3$, 2%	50	,,	0·0498	−0·0006	− 1·2
10	2 ,, $PtCl_4$, 3%	50	,,	0·0500	−0·0004	− 0·8
10	5 ,, $RhCl_3$, 0·2% 5 ,, $RuCl_3$, 0·2% 5 ,, $IrCl_3$, 0·2%	50	,,	0·0505	+0·0001	+ 0·2
10	$Ce_2(SO_4)_3$, trace	50	,,	0·0510	+0·0006	+ 1·2
10	10 c.c. $Ce_2(SO_4)_3$	50	,,	0·0856	+0·0352	+70·4

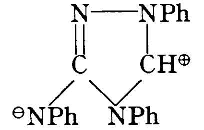
It is evident that only cerium salts interfere, and they should be removed before the precipitation. The excess of the reagent required in presence of other salts depends on their nature and quantity and on the volume of the solution.—INSTITUTE OF FORENSIC MEDICINE, JAGELL UNIVERSITY OF CRACOW. [*Received, March 15th,* 1938.]

Dipole Moments and Molecular Structure. The Dipole Moment of Nitron.—By FRANK L. WARREN.

SCHÖNBERG has suggested (private communication; see also this vol., p.824) that *endo*triazoles do not exist as such but as zwitter-ions. These compounds are practically insoluble in all non-polar solvents, and an attempt to effect an approximate measurement of the dipole moment of 1 : 4-diphenyl-5-methyl-3 : 4-*endo*thiotriazole and of nitron in chloroform (compare Hassel and Uhl, *Z. physikal. Chem.*, 1930, *B*, **8**, 157) was frustrated by the large damping effect of such solutions when placed in the dielectric cell.

Amongst the compounds of this type, however, nitron was found to be slightly soluble in benzene, and measurement in this solvent gave a dipole moment of 7·2 D.; this high value is indicative of a zwitter-ion structure, which might be represented as a resonance hybrid between formulæ (II), (III), and (IV) (compare Ingold, J., 1933, 1120; Sidgwick, J., 1937, 694), rather than of Busch's structure (I) (*Ber.*, 1905, **38**, 858).


(I.)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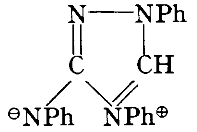
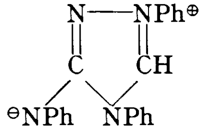

(III.)


(IV.)

Nitron, obtained from Schering–Kahlbaum, and twice crystallised from alcohol and benzene and washed with benzene, had m. p. 188—189° (Busch, *loc. cit.*). The measurements were made as described previously (J., 1937, 1858), and their ability to afford accurate results with such small dilutions was tested with *p*-nitrosodimethylaniline, for which, at $f_2 = 0{\cdot}000546$, P_2 was found equal to 1030 (Le Fèvre and Smith, J., 1932, 2239, give $P_2^\infty = 1044$ c.c. at 25°).

Nitron in benzene.

f_2.	$\epsilon^{30°}$.	$d_{4°}^{30°}$.	$P_{1,2}$.	P_2.
0	2·2621	0·86808	26·6280	—
0·0003574	2·2854	0·86849	26·9883	1035
0·0004129	2·2892	0·86856	27·0465	1040
0·0004306	2·2902	0·86861	27·0610	1032
0·0006719	2·3046	0·86881	27·2849	1004

Nitron in chloroform.

f_2.	$n_D^{30°}$.	$d_{4°}^{30°}$.	${}_EP_{1,2}$.	${}_EP_2$.
0	1·4401	1·46921	21·4189	—
0·003702	1·4433	1·46695	21·7164	101·8
0·003873	1·4435	1·46681	21·7330	102·6

$$P_2^\infty = 1036 \text{ c.c.};\quad P_E = 102{\cdot}2 \text{ c.c.};\quad \mu = 7{\cdot}2 \times 10^{-18} \text{ e.s.u.}$$

Thanks are due to Prof. Schönberg for his kind interest in the work.—EGYPTIAN UNIVERSITY, ABBASSIA, CAIRO. [*Received, May 23rd,* 1938.]