

**SECTION OF ANALYTICAL CHEMISTRY**  
**COMMISSION ON MICROCHEMICAL TECHNIQUES\***

**RECOMMENDED TEST SUBSTANCES FOR THE  
MICRODETERMINATION OF HALOGENS AND  
SULPHUR IN ORGANIC COMPOUNDS**

A number of compounds are recommended for use as reference substances for the microdetermination of halogens and sulphur in organic compounds. All of these substances, or a proper selection from the list, may be used to determine the universal applicability of a given method, either already described or one which might be developed in the future.

The compounds selected are stable over long periods of time and are non-hygroscopic (any exceptions to the latter are so noted). The substances are either commercially available in a sufficiently pure state to be used for test purposes based on the accuracy of present-day methods or may be purified or prepared by conventional laboratory means to meet these standards.

The compounds selected include the following:

- (1) compounds representing a variety of structural types;
- (2) compounds containing elements which may cause interferences in the halogen and/or sulphur determination.

These recommendations may be changed or supplemented as the need arises.

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List of substances

Compound	Empirical formula	Molecular weight	Hal (%)	C (%)	H (%)	O (%)	N (%)
<i>p</i> -Fluorobenzoic acid	$C_7H_5O_2F$	140.113	13.56	60.01	3.60	22.84	
Trifluoroacetanilide	$C_8H_6ONF_3$	189.136	30.13	50.80	3.20	8.46	7.41
Perfluorodicyclohexylethane	$C_{12}F_{26}$	662.102	74.60	23.40			
Teflon	$(CF_2 \cdot CF_2)_n$	(100.014) <sub>n</sub>	75.98	24.02			
Histidine hydrochloride monohydrate	$C_6H_{13}O_3N_3Cl$	209.633	16.91	34.38	5.77	22.90	20.05
1-Chloro-2,4-dinitrobenzene	$C_6H_3O_4N_2Cl$	202.553	17.50	35.58	1.49	31.59	13.83
Chloroacetamide	$C_2H_4ONCl$	93.513	37.91	25.69	4.31	17.11	14.98
Hexachlorocyclohexane	$C_6H_6Cl_6$	290.832	73.14	24.78	2.08		
Hexachlorobenzene	$C_6Cl_6$	284.784	74.69	25.31			
<i>p</i> -Bromoacetanilide	$C_8H_8ONBr$	214.067	37.33	44.89	3.77	7.47	6.54
5,7-Dibromo-8-hydroxyquinoline	$C_8H_5ONBr_2$	302.963	52.75	33.68	1.66	5.28	4.62
2,4,6-Tribromophenol	$C_6H_3ONBr_3$	330.816	72.47	21.78	0.91	4.84	
<i>o</i> -Iodobenzoic acid	$C_7H_5O_2I$	248.019	51.17	33.90	2.03	12.90	
2,4,5,7-Tetraiodofluorescein	$C_{20}H_8O_5I_4$	835.895	60.73	28.74	0.96	9.57	
Compound	Empirical formula	Molecular weight	S (%)	C (%)	H (%)	O (%)	N (%)
Sulphanilic acid (anhydrous)	$C_6H_7O_3NS$	173.190	18.51	41.61	4.07	27.71	8.09
Sulphanilamide	$C_6H_8O_3N_2S$	172.206	18.62	41.85	4.68	18.58	16.27
Sulphonal	$C_7H_{10}O_4S_2$	228.329	28.09	36.82	7.06	28.03	
Thiourea	$CH_4N_2S$	76.121	42.12	15.78	5.30		36.80

## Notes

For the calculation of molecular weights and percentages, the atomic weights used are those proposed by the Commission on Atomic Weights of the I.U.P.A.C., 1961.

The percentage figures are given to the second decimal. Where the third decimal is less than 0.005, the last figure has been disregarded. Where the last figure has been rounded off, it has been underlined ( $0.375 = 0.38$ ).

All of the compounds, with the exceptions of Teflon and histidine hydrochloride monohydrate, have been included in the two previous lists of recommended test substances, "Recommended Test Substances for the Microdetermination of Carbon and Hydrogen", *Pure and Appl. Chem.* **1**, 143 (1960), and "Recommended Test Substances for the Microdetermination of Nitrogen in Organic Compounds", *Pure and Appl. Chem.* **3**, 513 (1961).