

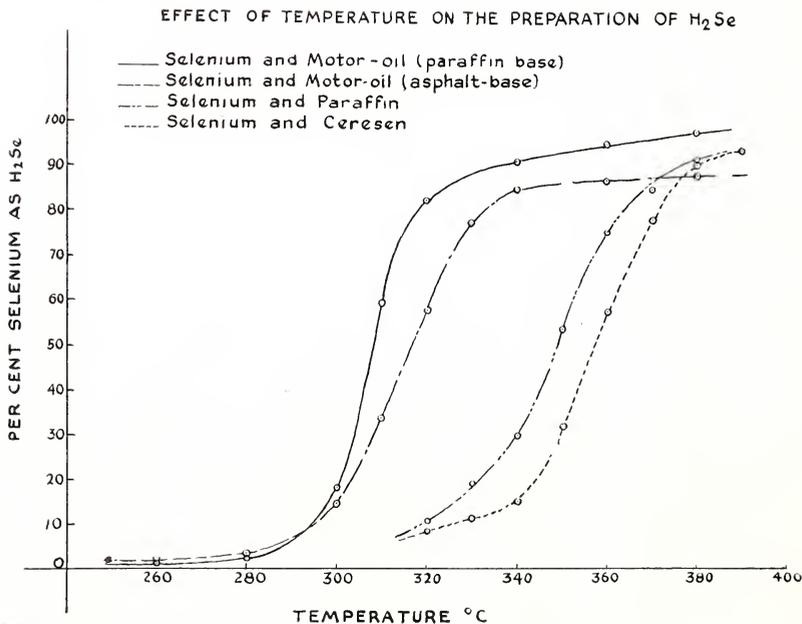
THE PREPARATION OF HYDROGEN SELENIDE BY THE INTERACTION OF SELENIUM AND HYDROCARBONS

CLARENCE GREEN¹ and W. E. BRADT,² State College of Washington

Graefe³ and Siebeneck⁴ reported the formation of hydrogen selenide when selenium was heated with lubricating oil (3) and paraffin (4). E. D. Scudder and R. E. Lyons⁵ completed a systematic study of the formation of hydrogen sulfide by a similar reaction in which they obtained a maximum yield of 70 per cent H₂S.

The authors have investigated some of the factors affecting the preparation of hydrogen selenide.

Procedure. In each experiment one gram of powdered selenium was placed with 50 grams of hydrocarbon in a 150 cc. Pyrex flask. The flask was connected to a source of hydrogen and to an absorption apparatus containing aqueous NaOH. Temperature was controlled by means of an insulating jacket and was observed by means of a copper-nichrome thermocouple.



¹ Graduate student in chemistry.

² Assistant Professor of Chemistry.

³ Z. angew. Chem. 34:509 (1921).

⁴ Petroleum Zeit. 18:281 (1922).

⁵ Proc. Ind. Acad. Sci. 40:185 (1930).

Prior to beginning each experiment all air was washed out of the apparatus and solutions by the passage of hydrogen for 15 minutes. At the completion of each heating period (one hour) all H_2Se was washed into the absorbing solution by the same process.

At this point the absorbing solution was diluted, treated with sodium peroxide, acidified with HCl , and saturated with sulfur dioxide. The precipitated selenium was then determined gravimetrically.

Experimental Data. The following table is self explanatory:

TABLE I

Heating period: 60 minutes
Selenium: 1.000 g. powdered
Yield: Based on selenium

Hydrocarbon (50 g.)	Temperature (° C.)	Yield H_2Se (%)
Paraffin (<i>a</i>)	320	10.5
"	330	18.9
"	340	29.7
"	350	53.0
"	360	74.6
"	370	83.7
"	380	90.2
Ceresin (<i>b</i>)	320	8.7
"	330	11.8
"	340	15.1
"	350	31.5
"	360	57.0
"	370	77.6
"	380	90.1
"	390	92.5
Motor Lubricant (<i>c</i>)	260	1.9
" "	280	3.5
" "	300	14.6
" "	310	33.4
" "	320	57.8
" "	330	76.5
" "	340	84.0
" "	360	86.0
" "	380	87.1
Motor Lubricant (<i>d</i>)	260	1.7
" "	280	2.7
" "	300	18.0
" "	310	59.0
" "	320	81.6
" "	330	87.1
" "	340	90.5
" "	360	94.2
" "	380	96.8

Motor Lubricant (e)	320	46.4
“ “	340	79.6
Motor Lubricant (f)	340	31.5
“ “	350	67.2

(a) M. P. = 60° C.

(b) Technical (White) Eimer and Amend.

(c) Asphalt Base (Zerolene No. 5).

(d) Paraffin Base (Standard Aviation Oil).

(e) Same as (d), previously heated for 1 hr. with 1 g. of selenium.

(f) Same as (d), previously heated for two 1 hr. periods with 1 g. of selenium each period.

Summary. Hydrogen selenide is obtained by heating powdered selenium with high boiling hydrocarbons. A maximum yield of 96.8 per cent H_2Se (based on the selenium used) was obtained by heating 50 g. of a paraffin base motor oil with 1 g. of selenium for one hour at 380° C.

Heating the same 50-gram portions of a paraffin oil with repeated additions of selenium (1 g. each) reduced the formation of H_2Se to approximately that obtained by the use of paraffin at corresponding temperatures.

In contrast with the results obtained by E. D. Scudder and R. E. Lyons with hydrogen sulfide the yield of hydrogen selenide increases with increased temperatures. Only negligible yields were obtained below 300° C.

ANALYSIS OF GROUP III

RALPH W. HUFFERD, Greencastle

Since reporting a paper under the same title to this group eleven years ago several changes have been introduced into the scheme. In conversation with instructors and students in other laboratories the writer still hears the common complaint that cobalt and zinc are prone to give trouble. It has been our experience that the careful student has trouble with neither of these.

As reported before (1) the main departure from the generally accepted scheme of analysis lies in the very strenuous treatment of the group precipitate at the time of separating it into divisions. Detection of cobalt has been made certain and easy by a modification of the Morrel-Vogel test (2). Since the difficulty with detection of zinc lies in the confirmatory test, unless it has been left in the Iron Division precipitate, it has been found expedient, especially for the beginning student, to employ two confirmatory tests. One of these, the ferrocyanide test, has been modified to meet all conditions.

The scheme has been worked out with both speed and accuracy in mind. Almost all filtrations are carried out by suction. Solutions are