The compounds give fairly stable, brownish-red colors with sodium nitroprusside and alcoholic alkali, in contrast to the transient purple given by the salts of thiocyanoacetic acid.

Alkaline lead solutions give yellow precipitates upon gentle warming[10]; in case of compounds with acidic substituents, the precipitate appears upon acidification.

Selenium oxychloride in concentrated sulfuric acid gives intense colorations, identical with those of the corresponding mercapto-compounds, except for transient differing shades sometimes developing in the first few seconds (see the following paper for details).

The individual compounds and their properties are listed in Table I. The A procedure was in general followed in their preparation; where this was not the case, this is indicated by a footnote. The yields in every case are those obtained upon interaction of stoichiometric quantities of the reactants. Higher yields may be attained by use of excess thiocyanoacetate. The recrystallization was in general carried out in water, acidified with a few drops of approximately 0.1 *N* hydrochloric acid. Where other solvents were employed, these are indicated by footnotes.

Samples of the three aminophenol derivatives were first prepared by Mr. M. J. Lewenstein, of these laboratories, who is applying for U. S. patents. The author, who continued the work on them is much indebted to Mr. Lewenstein for his permission to include those compounds in this report.

Reaction of Semicarbazide with Sodium Thiocyanoacetate.—One-tenth molar quantities of semicarbazide hydrochloride (11.1 g.) and sodium thiocyanoacetate (15.7 g.) were dissolved together in 200 cc. of water. The solution was adjusted to a *p*H of approximately 3 and seeded. The product was worked up after four days, as described in A. Yield was 14.3 g., m. p. 185–186° (dec.),[13] 187° (dec.), after crystallization from 0.1 *N* hydrochloric acid. The nitroprusside reaction was purple, and the color rather more stable than the one given by salts of thiocyanoacetic acid. Selenium oxychloride in concentrated sulfuric acid gives a cloudy, yellowish solution with gas developing.

(13) All m. p.'s are corrected.

Anal. Calcd. for $C_4H_5N_4O_3S$: N, 33.31. Found: N, 15.05.[14]

Salts of Thiocyanoacetic Acid. A.—Three and five-tenths grams of α-aminopyridine hydrochloride and 4.2 g. of sodium thiocyanoacetate were dissolved together in 25 cc. of water at 45°. The solution was filtered, and chilled in ice–salt mixture. The salt crystallizes within a short time in well-formed flat needles.

Yield was 3.0 g.; m. p. 112° dec. With sodium nitroprusside it gives an intense, transient purple color, with a solution of cupric chloride a deep purplish black precipitate[9] is formed after about fifteen minutes. Alkali liberates α-amino-pyridine. Corresponding salts of cyclohexylamine (m. p. 110.5–111.5°) and α-aminothiazole (m. p. 127–128° dec.) were obtained in a similar way.

B. 2,6-Dimethylaniline (2.42 g., b. p. 212–216°) was suspended in 20 cc. of water, and brought into solution by addition of the minimum quantity of dilute hydrochloric acid (1:1). A solution of 3.14 g. of sodium thiocyanoacetate in 20 cc. of water was added. Crystallization of platelets started within a few minutes. The product was filtered within five minutes, by suction, washed with some ice-cold water, and dried *in vacuo* over phosphorus pentoxide. It melts incompletely about 85°, becoming clear at about 140°. No cyanuric acid appears upon further heating. It is moderately soluble in water, very soluble in methanol, chloroform, ether, benzene and pyridine. (Other reactions as described under A.)

Acknowledgment.—The author is much indebted to Drs. S. M. Gordon and N. Weiner for their interest, to Mr. S. Sokol for assistance in part of the experiments.

Summary

A number of N-aryl-α-carbamylmercaptoacetamides has been prepared from the corresponding amines and sodium thiocyanoacetate in aqueous solution at room temperature.

(14) Analyses by Dr. I. A. Kaye, Brooklyn College; method of Pepkowitz and Shive, *Ind. Eng. Chem., Anal. Ed.*, **14**, 280 (1942); *cf.* Kaye and Weiner, *ibid.*, **17**, 397 (1945).

RICHMOND HILL, N. Y. RECEIVED JANUARY 31, 1947

[CONTRIBUTION FROM THE RESEARCH LABORATORY OF ENDO PRODUCTS, INC.]

N-Arylamides of Mercaptoacetic Acid. II. Analogs of α-Mercaptoacetanilide and Corresponding Gold Mercaptides

BY ULRICH WEISS

N-Arylamides of mercaptoacetic acid, I, were prepared by treatment of their carbamyl derivatives, II,[1] with hot dilute ammonia,[2] other methods used occasionally for this reaction being less favorable.[3]

$$\mathrm{Ar{-}N(R){-}CO{-}CH_2{-}S{-}X}$$

I, X = —H
II, X = —$CONH_2$
III, X = —Au

Purification of the compounds obtained (checked by iodometric assay) was achieved with some difficulty in many instances, owing to ease of autoxidation; in a few cases, no entirely satisfactory preparations were obtained.

The compounds were further converted to the gold mercaptides by the method already de-

(1) Paper I, Weiss, THIS JOURNAL, **69**, 2682 (1947).

(2) Beckurts and Frerichs, *J. prakt. Chem.*, [2] **66**, 181 (1902), and subsequent papers by Frerichs, *et al.*

(3) (a) Thermal decomposition: ref. 2; Rheinboldt, Tappermann, Kleu, *J. prakt. Chem.*, [2] **133**, 69 (1939); (b) boiling water: Rizzo, *Gazz. chim. ital.*, **28**, 1, 360 (1898); (c) alcoholic alkali, Frerichs and Foerster, *Ann.*, **371**, 293 (1909); the claim that the grouping—$CONH_2$—is converted into cyanate rather than carbamate was confirmed by identification of NaCNO from II, Ar—N(R=) C_6H_5—NH—. Related cases of formation of cyanates: Arth, *Bull. Soc. Chim.* [2] **41**, 334 (1884), *Ann. Chim.*, [6] **8**, 430 (1886); Mulder, *Rec. trav. chim.*, **6**, 170 (1887); *cf.* also Dox and Yoder, THIS JOURNAL, **45**, 726 (1923).

scribed[4] and also oxidized with iodine to the corresponding disulfides.

The constitutions of the three isomeric N-α-mercaptoacetylaminophenols (I, Ar—N(R) = OH—C_6H_4—NH—)[5] were proved by desulfuration with Raney nickel.[6] The respective N-acetylaminophenols were obtained in good yields, and identified by comparison with authentic samples.

Experimental

Preparation of Mercaptoacetyl Compounds. I. General Procedure.—The corresponding carbamyl compound II is refluxed with a mixture of 3 volumes of concentrated aqueous ammonia and 3 volumes of water for five to ten minutes or until nothing more goes into solution. Charcoal is added, if necessary, and the solution is filtered by suction, without any delay, into a flask containing excess ice. The filtrate is acidified immediately with dilute hydrochloric acid (1:1). The precipitate is collected on a Büchner funnel, and washed well with water acidified with hydrochloric acid. A sample of the filter-cake is tested for presence of disulfide by shaking with dilute ammonia in a test-tube. If the resulting solution is more than slightly cloudy, the whole quantity is dissolved in dilute ammonia at room temperature, filtered again upon ice, the filtrate acidified immediately, and the product worked up as before. The product is finally dried in vacuo, over phosphorus pentoxide. Yields were usually 80–90%.

For purification it is mainly necessary to remove the disulfide formed through autoxidation. As this autoxidation appears to be autocatalytic[7] and is diminished in acidic medium, it is essential to have the crude material as free as possible from disulfide and to recrystallize from acidified solution. Ethanol or methanol acidified with a few drops of hydrochloric acid and particularly water brought to a *p*H of approx. 3 with hydrochloric acid are appropriate. Where the disulfide is soluble in the ammoniacal reaction-mixture because of acidic substituents (—COOH, —SO_2—NH—R) in the aromatic ring, and therefore not eliminated by filtration, preparation of satisfactory[8] samples of the —SH— compound by crystallization was impossible. Sublimation in high-vacuum effected a clear-cut separation of the desired compound from the disulfide in several cases where recrystallization was insufficient, but the mercaptoacetyl derivatives of *p*-aminophenylacetic acid and of the sulfa drugs decomposed upon attempted sublimation, and no sufficiently pure samples have been obtained.[9] The compounds were characterized as the gold mercaptides.

The properties of the individual compounds are listed in Table I.

α-Mercaptoacetyl-N-methylaniline which is liquid at room temperature[4a] was prepared as described above, isolated by extraction into chloroform and evaporation of the solvent under reduced pressure. The compound was converted to the gold salt without further purification.

α-Mercaptoacetyl-*p*-aminobenzoic Acid, prepared as described above, forms a nearly unfilterable suspension. This difficulty can be overcome by carrying out the reaction by fifteen minutes of refluxing with 15 volumes of a mixture of methanol and concentrated ammonia 2:1, chilling the stoppered flask in ice, and acidifying with acetic acid.[10] The carbamyl compounds derived from 3,4-dimethylaniline, *m*- and particularly *p*-nitraniline dissolve incompletely in hot dilute ammonia (at least in the last two cases, this seems to be due to interaction of the nitro- and mercapto- groups). This makes the purification of the reaction-products difficult, especially in the case of the *p*-nitraniline-derivative, where the insoluble contaminant is hard to remove. In this case, even high-vacuum sublimation was not very satisfactory. α-Mercaptoacetyl-*p*-aminoacetophenone, prepared as described above, was similarly purified with difficulty, for unknown reasons. Even crystallization from ethanol, followed by high-vacuum sublimation, failed to give an entirely satisfactory product.

A few other carbamyl compounds (those derived from *o*-amino-diphenyl, β-naphthylamine, ethyl-*p*-aminobenzoate) dissolve likewise incompletely in the hot ammonia, but no difficulties were experienced in purifying the resulting compounds.

Preparation of Disulfides, (Ar—NH—CO—CH_2—S)$_2$.

General Procedure.—Five tenths of a gram of the mercapto compound is dissolved, at room temperature, in ethanol, and a slight excess of aqueous 0.1 *N* iodine solution is added from a buret. The excess is removed by addition of a few drops of aqueous sodium thiosulfate solution. Dilution with water precipitates the disulfide in nearly quantitative yield. It is recrystallized from methanol, ethanol or acetic acid. Several of the compounds are only very slightly soluble. The crude disulfides derived from sulfapyridine and sulfathiazole seem to contain water of crystallization, but separate in anhydrous form from boiling methanol.

The properties of the individual compounds are summarized in Table I.

Preparation of Gold Mercaptides, Ar—N(R)—CO—CH_2—S—Au

These compounds were prepared by the method already described for the aniline derivative.[4] They are obtained, in nearly quantitative yields, as off-white to yellow powders, which are not visibly crystalline, and are insoluble in the common organic solvents. They decompose at fairly reproducible temperatures, if the melting point capillary is immersed into a preheated bath at about 10° below the expected decomposition point.

In a few cases, gold values were not very satisfactory, because the disulfide, a nearly unavoidable contaminant, was too insoluble to be completely removed by repeated washing with ethanol. In two such cases, destruction of the disulfide was attempted by treatment of the gold mercaptide with alkali: to the original suspension of the *m*-nitraniline derivative, enough aqueous sodium hydroxide was added to give a concentration of 5%. After five hours of standing at room temperature, the product was worked up in the usual way. A marked improvement of the analytical value (from 47.0 to 47.6% Au, calcd. 48.3%) resulted. In the case of the derivative of *p*-aminobenzoic acid, solution in 2% alkali, and reprecipitation with dilute hydrochloric acid after five hours resulted likewise in a better gold value (from 47.0 to 47.6%, calcd. 48.4%), but with concomitant change of color from nearly white to yellow.

The properties of the individual compounds are listed in Table I.[11]

Conversion of $HOC_6H_4NHCOCH_2SH$ to $HOC_6H_4NHCOCH_3$.—Three grams of α-mercaptoacetyl-*o*-aminophenol, 80 cc. of methanol, 7 cc. of an alcoholic suspension of Raney nickel and 10 cc. of water were refluxed in a water-bath for five hours, when a sample gave only a

(4) Weiss, THIS JOURNAL, **67**, 1424 (1945).

(5) Samples of these three compounds, and of their gold mercaptides, were first prepared by Mr. M. J. Lewenstein, who is applying for U. S. patents. The author, who continued the work on them, is much indebted to Mr. Lewenstein for his permission to include those compounds in this report.

(6) Bouveault, Cattelain and Chabrier, *Compt. rend.*, **208**, 647 (1939).

(7) *Cf.* the analogous case of mercaptoacetic acid, Harrison, *Biochem. J.*, **21**, 1404 (1927).

(8) Samples were considered satisfactory, if the per cent. of —SH agreed within ±0.2% with the calculated one. Titration in alcoholic solution with 0.1 *N* iodine to the first yellow tinge gives very sharp and reproducible values.

(9) No attempts at purification through reduction of the contaminating disulfide have been made.

(10) *Cf.* ref. 3a, p. 71.

(11) All melting points are corrected.

TABLE I[12]

Ar—N(R)—CO—CH_2—S—X

Ar—N(R)—	X = H: Yield, %	M. p., °C.[11]	Purified[b]	% SH[a] Calcd.	% SH[a] Found
C_6H_5—N(CH_3)—	86	Oil[3a]	..	...	...
o-CH_3—C_6H_4—NH—	82	91[d]	w	18.25	18.15
p-CH_3—C_6H_4—NH—	86	126[f]	w	18.25	18.05
3,4-$(CH_3)_2C_6H_3$—NH—	73	109	s	16.94	16.94
2,6-$(CH_3)_2C_6H_3$—NH—	92	129	w	16.94	16.78
o-C_6H_5—C_6H_4—NH—	92	73	m	13.60	13.43
α-$C_{10}H_7$—NH—	80	132[i]	w	15.23	15.12
β-$C_{10}H_7$—NH—	72	114[k]	e	15.23	15.14
p-CH_3—CO—C_6H_4—NH—	93	140–144	w + s	15.81	15.43
m-NO_2—C_6H_4—NH—	53	90	w	15.58	15.45
p-NO_2—C_6H_4—NH—	51	Unsharp. 180[m]	e + s	15.58	15.69
o-OH—C_6H_4—NH—	78	134[5]	w	18.06	18.07
m-OH—C_6H_4—NH—	90	154[5]	m	18.06	18.07
p-OH—C_6H_4—NH—	83	136[5]	w	18.06	18.00
o—CH_3—O—C_6H_4—NH—	91	68	s	16.78	16.84
p-CH_3—O—C_6H_4—NH—	92	119[p]	w	16.78	16.67
o-COOH—C_6H_4—NH—	89	160	s	15.66	15.59
p-COOH—C_6H_4—NH—	74	230	s	15.66	15.49
p-C_2H_5—OCO—C_6H_4—NH—	82	114	e	13.83	13.76
p-COOH—$CH_2C_6H_4$—NH—	75	149–151	w	14.69	14.27
p-NH_2—SO_2—C_6H_4—NH—	81	203	e	13.43	13.06
p-C_5H_4N—NH—SO_2—C_6H_4—NH—(sulfapyridine)	80	198	m	10.23	9.80
p-C_3H_2NS—NH—SO_2—C_6H_5—NH—(sulfathiazole)	[r]	202–204	e	10.04	9.62
p-$C_4H_3N_2$—NH—C_6H_4—NH—(sulfadiazine)	[r]	208	m	10.20	9.03

[a] Analyses by Dr. I. A. Kaye, Brooklyn College; methods: Pepkowitz and Shive, *Ind. Eng. Chem., Anal. Ed.*, **14**, 280 (1942); Kaye and Weiner, *ibid.*, **17**, 397 (1945). [b] Abbreviations: m, methanol, e, ethanol, w, water, s = high-vacuum sublimation. Solvents acidified with hydrochloric acid for compounds X = H. [c] Ref. 3a, p. 70: 81°. [d] Beckurts, Frerichs and Beyer, *J. prakt. Chem.*, [2] **74**, 39 (1906): 84–85°. [e] *Ibid.*, 164–165°. [f] *Ibid.*, p. 47; Hellstroem and Lauritzson, *Ber.*, **69**, 405 (1935): m. p. 125–126°. [g] Ref. d, p. 47: 180–182°. [h] Dec. point taken the usual way; if capillary is inserted into preheated bath, brown discoloration occurs at 200°, without further change up to 250°. [i] Ref. 3a, p. 71: 127–128.5°. [j] Frerichs, Wildt, *Ann.*, **360**, 115 (1908): m. p. 205–206°; ref. 3a, p. 71: m. p. 211–212°. [k] Ref. 3a, p. 74: m. p. 111–112°; ref. 17, p. 405: m. p. 113–113.5°. [l] Ref. 3a, p. 73: 198–199°. Ref. j, p. 117: 204–205°. [m] Product is light yellow; all attempts to obtain colorless material unsuccessful. [n] Alkali-treated material. [o] No reason for low value apparent. Microanalysis by Mr. W. Saschek, Columbia Univ.; C, calcd. 45.47; found, 45.27; H, calcd., 3.34; found, 3.30. [p] Beckurts and Frerichs, *Arch. Pharm.*, **253**, 137 (1915), m. p. 116°. [q] Ref. p, p. 138: m. p. 185°.

yellow color with sodium nitroprusside and alkali. The warm suspension was filtered by suction, the catalyst washed several times with warm methanol, and the combined filtrates were treated with charcoal and evaporated to dryness. The residue, after drying at 105° weighed 1.8 g., 73%, the melting point[11] after crystallization from water was 209°; mixed m. p. with an authentic sample of N-acetyl-*o*-aminophenol was 208.5°.

The *m*- and *p*-isomers, when treated in the same manner, gave similar yields of the respective N-acetylamino phenols: *m*-isomer, m. p. and mixed m. p. 146.5–147.5°, *p*-isomer, m. p. and mixed m. p. 167.5–168.5°.

Isolation of Sodium Cyanate from α-Carbamylmercaptoacetanilide.—Four and two tenths grams (0.02 mole) of the compound was dissolved in 50 cc. of warm ethanol, and 2 equivalents of *N* alcoholic sodium hydroxide was added. The white precipitate was filtered by suction after three hours, dried *in vacuo* over phosphorus pentoxide, and identified as sodium cyanate by analysis (Na, calcd. 35.38%; found 35.09%), evolution of cyanic acid (odor!) upon acidification of the aqueous solution, and characteristic blue color reaction with cobaltous salts.

It was observed that all compounds of type I tested gave intense, stable colors with a 5% solution of selenium oxychloride in concentrated sulfuric acid. In general, all compounds with the same aryl radical gave the same colors,[13] although some of the carbamyl compounds[1] and some of the disulfides give transient initial colors which change to the characteristic ones in a few seconds. X in formula I may be —H, —Au, —CN or any organic radical, *e. g.*, —$COCH_3$, —CH_3, —$CH_2C_6H_5$. Also $(C_6H_5NH\text{-}COCH_2)_2S$[14] and $(CH_3)_2C(SCH_2\text{—}CO\text{—}NH\text{—}C_6H_5)_2$[14] gave the reaction, while $C_6H_5NHCOCH_2OH$[15] gave only a light yellow color.

Although the use of a 0.5% solution of selenious acid in concentrated sulfuric acid as a color-reagent has been reported,[16] no similar use of selenium oxychloride seems to be recorded.[17] The former reagent was found to give, with the mercapto compounds, colors identical with those

(12) ADDED IN PROOF.—Since submission of this paper U. S. Patent 2,418,947, April 15, 1947, granted to W. A. Lott, *et al.*, has appeared in which are described the preparation, of mercaptoacetylsulfanilamide, m. p. 215–217° with softening at 205°, corresponding gold mercaptide, no m. p. given, mercapto-acetylsulfapyridine, no m. p. given, mercaptoacetylsulfathiazole, m. p. 196°, and its gold mercaptide, m. p. 186–192°. The mercaptoacetyl compounds were here prepared by direct interaction of thioglycolic acid with the sulfa drug.

(13) The compound Ar = *o*-$CH_3OC_6H_4$—, R = H, X = Au; seems to be an exception: brown color, instead of the deep blue of the other *o*-anisidine derivatives of the series.

(14) Ref. 2, p. 187, 188.

(15) Bischoff and Walden, *Ann.*, **279**, 49 (1894).

(16) Dewey and Gelman, *Ind. Eng. Chem., Anal. Ed.*, **14**, 361 (1942); contains bibliography.

(17) Morgan and Burstall, *J. Chem. Soc.*, 3260 ff. (1928), mention colors obtained upon treatment of reaction products of phenols and selenium oxychloride with concentrated sulfuric acid.

TABLE I (*Continued*)

Ar—N(R)—CO—CH_2—S—X

X = Au			(R—NH—CO—CH_2—S)$_2$		%N[a]		
Dec., °C.[11]	Calcd.	Found	M. p., °C.[11]	Purified[b]	Calcd.	Found	Color with $SeOCl_2/H_2SO_4$
208–210	52.3	52.2	82[c]	dil. e	...	...	Red
231	52.3	51.9	171[e]	m	...	...	Red
245	52.3	52.3	186[g]	m	...	...	Brownish red
251	50.4	50.2	185	e	7.21	7.41	Brown
259	50.4	50.2	235	e	7.21	7.19	Yellow
202[h]	44.9	44.7	150	m	5.78	5.73	Reddish purple
246	47.7	47.3	213[j]	m	...	...	Emerald green
260	47.7	47.4	209[j]	e	...	...	Greenish blue
268	48.7	47.9	184	e	...	...	Yellow
256–258[n]	48.3	47.6	176[m]	e	13.26	13.19	Yellow
...	..	..	198[m]	e	13.26	9.11[o]	Yellow
215[5]	51.9	51.1	213	e	7.68	7.67	Blue
238[5]	51.9	50.8	166	dil. e	7.68	7.84	Blue
240[5]	51.9	51.1	202	dil. e	7.68	7.61	Green
202[h,12]	50.1	50.0	126	e	7.14	6.96	Purplish blue
249	50.1	49.9	191[q]	m	...	...	Emerald green
243	48.4	47.7	220–222 (dec.)	AcOH	6.66	6.80	Yellow
284[n]	48.4	47.6	281 (dec.)	AcOH	6.66	6.60	Yellow
274	45.3	44.6	146	e	5.87	5.82	Yellow
276	46.8	45.3	218.5–220.5	AcOH	6.24	6.10	Yellow
268	44.5	43.5	246 (dec.)	m	11.42	11.25	Yellow
231	37.9	37.9	226–228 (dec.)	m	13.03	12.93	Yellow
265	37.5	36.5	236 (dec.)	m	12.79	12.56	Yellow
...	..	..	.....	....	...	...	[s]

Crude preparations used for further work without drying, only samples prepared for analysis. [s] Reactions of these carbamyl compounds (see preceding paper), whose corresponding —SH compounds have not been prepared; Ar—N(R)=C_8H_5NH—NH— purplish red turning brown quickly; $C_6H_5N(CH_2C_6H_5)$— purplish red; $As_3OH_2C_5H_4NH$— yellow 2-OH,5—$COOCH_3C_6H_4NH$— yellowish brown.

obtained with selenium oxychloride, but to react only slowly with carbamyl compounds and disulfides, and not at all with the gold salts.[18]

Acknowledgment.—The author is much indebted to Drs. S. M. Gordon and N. Weiner for their interest, and to Mr. Saul Sokol for technical assistance.

(18) This color reaction might be of interest in connection with the use of α-mercaptoacetyl-β-naphthylamine, "thionalide," as analytical reagent for heavy metals: Berg and Roebling, *Ber.*, **68**, 403 (1935), and subsequent papers.

Summary

A number of N-arylmercaptoacetamides, the corresponding disulfides and gold-mercaptides have been prepared.

RICHMOND HILL, N. Y. RECEIVED JANUARY 31, 1947

[CONTRIBUTION FROM THE RESEARCH LABORATORY, GENERAL ELECTRIC COMPANY]

Alkylation of Hydrochlorosilanes

BY C. A. BURKHARD AND R. H. KRIEBLE

The peroxide and ultraviolet light catalyzed addition of trichlorosilane to 1-octene was announced recently by Sommer, *et al.*[1] We had concluded a similar investigation when this communication appeared and wish to describe further results at this time concerning the addition of the silicon–hydrogen compounds to olefins.

Kharasch, *et al.*,[2] have reported similar reactions in which carbon tetrachloride, carbon tetrabromide, bromoform, and chloroform add to olefins in the presence of peroxide or white light. Unlike the reactions described by Kharasch, the attempted preparation of β-halogeno-organohalogenosilanes from the interaction of chloro- and bromosilanes with olefins has been unsuccessful.[3]

In contrast to this, trichlorosilane is found to add with ease to olefinic double bonds in the presence of peroxides or ultraviolet light. The reac-

(1) Sommer, Pietrusza and Whitmore, THIS JOURNAL, **69**, 188 (1947).

(2) Kharasch, Jensen and Urry, (*a*) *Science*, **102**, 128 (1945); (b) THIS JOURNAL, **67**, 1864 (1945); (c) *ibid.*, **68**, 154 (1946); (d) Kharasch, Urry and Jensen, *ibid.*, **67**, 1626 (1945).

(3) Scott (U. S. Patent 2,407,181 (Sept. 3, 1946)) reports that a *telomeric* product is obtained in the reaction of ethylene with silicon tetrachloride in the presence of peroxides.