water. No depression of the melting point was observed on mixing the sample with an authentic specimen.

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The Molecular Structure of Perfluorobutyne-2. Critical Marks for the Description of Electron Diffraction Curves

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A complete structure determination, rather highly precise because it is based mainly on microwave spectroscopic data and only partly on electron diffraction, has been achieved for trifluoromethylacetylene.[2] The present electron diffraction study has led to almost identical bond angle and bond distance values for perfluorobutyne-2: see Table I. The sample[3] was generously supplied by Professor Henne and the detailed techniques were those currently used in these laboratories.[4]

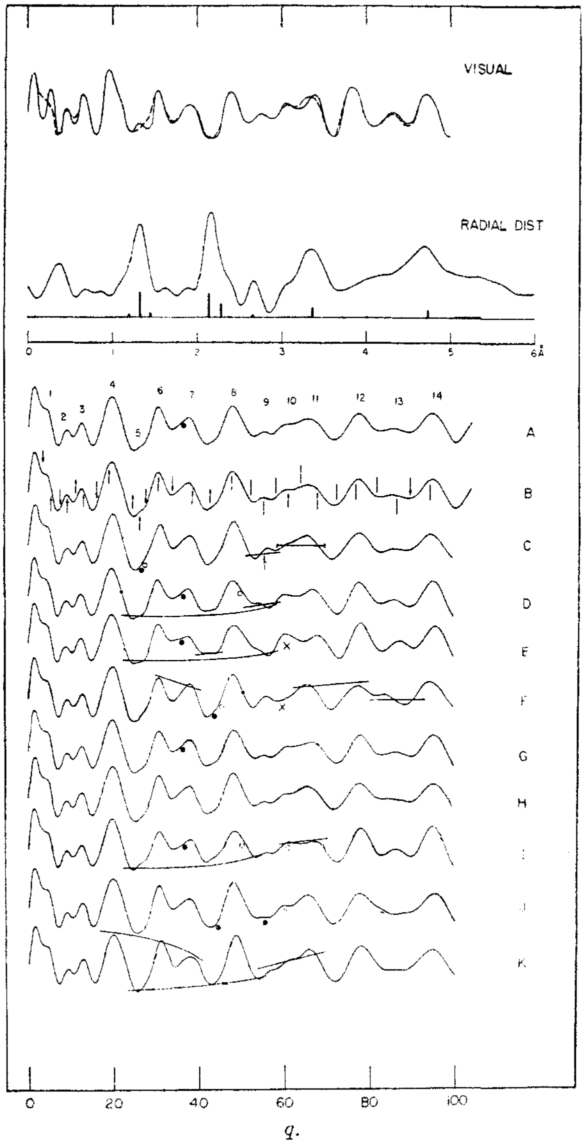

Fig. 1.—Electron diffraction curves for $F_3C—C'\equiv C''—C'''F_3$. Visual: Solid, W.S.; as modified by dashed additions, V.S. Both drawn with the help of differential comparisons with the best theoretical curve and photographs of trifluoromethylacetylene.[1] Radial distribution: Based on *solid* visual curve. Theoretical: Final curves including all interactions, for the following models, illustrating the indicated total range of models studied.

Curve	F—F	C—C'	C'≡C''	$q_{calcd.}/q_{obsd.}$[e]
A[a]	2.15	1.46	1.21	1.007
B[a,b]	2.15	1.46	1.21	1.006
C[c]	2.15	1.43	1.20	1.009
D[d]	2.15	1.49	1.20	1.008
E[d]	2.13	1.47	1.20	1.013
F[d]	2.17	1.47	1.20	1.000
G[a]	2.15	1.47	1.20	1.008
H[a]	2.15	1.45	1.23	1.004
I[c]	2.15	1.49	1.14	1.011
J[c]	2.15	1.43	1.29	1.008
K[d]	2.13	1.37	1.26	1.013
Range	2.11–2.17	1.35–1.51	1.14–1.29	(C—F = 1.33)

[a] Satisfactory curve. [b] Account taken of the effective slight shortening of C···C''' and C···F' expected to result from C—C≡C bendings. [c] Curve near the limit of acceptability, either within or without. [d] Completely unacceptable curve. [e] For 8 best (reasonably symmetrical and well-located) features; average deviation, Model B, 0.006 (for 22 best features, 0.019). Measurements by W. S.

TABLE I

	$F_3C—C\equiv C—H$[1] Value	$F_3C—C\equiv C—H$[1] Limit of error	$F_3C—C\equiv C—CF_3$ Value	$F_3C—C\equiv C—CF_3$ Limit of error
C—F, Å.	1.335	0.01	1.340	0.020[a]
C—C, Å.	1.464	.02	1.465	.055[a,b]
C≡C, Å.	1.201	.002	1.22	.09[a]
C—H, Å.	1.056	.005	.....	.....
∠F—C—F	107.5°	1.0°	107.5°	1.0°

[a] Including 1.0% estimated limit of scale error. [b] If C≡C were assumed to be precisely 1.22 Å., the limit of error for C–C would be reduced to 0.040 Å. but the other limits would be essentially unchanged.

About one hundred seventy-five theoretical intensity curves were calculated in order to cover the rigid-model shape-parameter problem completely and the temperature factor problem, with its considerable and uncertain dependence on possible instrumental effects as well as the low frequencies[2] and large amplitudes of C—C≡C bendings (estimated root-mean-square δ∠ C—C≡C, 9.5°), in some detail. All the models were given D_3 symmetry, and when the long F···F terms were finally included for some of the curves, free internal rotation was assumed. The significant points regarding the theoretical curves illustrated in Fig. 1 are indicated by marks in the figure itself rather than by the usual tedious verbal description.

Such *critical marks* can be generally self-explanatory (Table II seems almost unnecessary, except by way of introduction), and they are convenient, terse, highly legible (aiding, not distracting, the eye), and reasonably precise (more so than ordinary verbal description, especially since they aren't affected by limitations of space); their use should

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(1) U. S. Rubber Co. Predoctoral Fellow, 1950–1951. Present address: Shell Development Company, Emeryville, California.

(2) J. N. Shoolery, R. G. Shulman, W. F. Sheehan, Jr., Verner Schomaker and Don M. Yost, *J. Chem. Phys.*, **19**, 1364 (1951).

(3) A. L. Henne and W. G. Finnegan, THIS JOURNAL, **71**, 298 (1949).

(4) See K. Hedberg and A. J. Stosick, *ibid.*, **74**, 954 (1952).

significantly simplify and shorten electron diffraction articles, while improving readability. These marks are also very helpful, we have found, in the course of the work itself.

TABLE II

EXPLANATION OF CRITICAL MARKS OF FIG. 1

Curve	Mark	Meaning
A	Dot	Creation operator: inner slope should be more convex upwards
B	Arrows	Positions (q_{obsd}) of rings as measured on photographs, adjusted by multiplication by $q_{\text{calcd}}/q_{\text{obsd}}$. Best curve
C	Dot circle	Circle is destruction operator: 5 max. too far up to the right
C	Line	Indicates desired levels: 9 min. too deep; 10 min. too shallow
C	Arrow	Duplicated from best curve: 9 max. significantly misplaced relative to adjacent features
C	Terminated line	Indicates desired width: feature too narrow
D	Curved line	Indicates desired levels: 8 min. too shallow *re* 5 min. and 9 min.
E	Line (short)	Feature, bottom of 8 min., too symmetrical; should tip, and lie closer to 8 max. than to 7 max.
E	Cross	Completely unacceptable feature; *cf.* standards (best curve and visual)
F	Lines	7 max. too high *re* 6 max.; 11 max. too high *re* 12 max.; 13 max. should lie between minima of almost equal depth
K	Curved lines	6 max. too high *re* 4 max. and 7 max.; depth of 8 min. about right *re* 5 min. and 9 min.
K	Line	Region should slope upwards more gently

A more complete presentation of the structure determination is available.[5]

(5) W. F. Sheehan, Jr., Thesis, California Institute of Technology (1952).

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X-Ray Diffraction Patterns of Cuprous Acetate and Cupric Oxyacetate

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In the course of some experiments on catalytic hydrogenation, it became desirable to prepare and to obtain the X-ray diffraction patterns of cuprous acetate, $CuOOCCH_3$, and anhydrous cupric oxyacetate, $Cu_2O(OOCCH_3)_2$. Cuprous acetate was prepared by Calvin's procedure[1]; the method involves solution of cuprous oxide in acetic acid–acetic anhydride, filtration of the hot solution, cooling, and filtration of the separated cuprous acetate, all in the absence of air or moisture. The cuprous acetate was pure white; it contained 50.9% Cu (theor. for $CuOOCCH_3$, 51.8% Cu).

THE *d/n* VALUES AND INTENSITIES OBTAINED FROM DEBYE-SCHERRER X-RAY PHOTOGRAPHS OF THIS MATERIAL

d/n	*I*	*d/n*	*I*	*d/n*	*I*
11.0	vw	2.48	s	1.67	w
10.0	vvs	2.42	s	1.63	vw
5.4	vvw	2.31	s	1.57	w
5.0	vw	2.25	vw	1.55	w
4.05	w	2.15	w	1.50	vw
3.69	vw	2.00	w	1.465	vw
3.50	vw	1.95	w	1.405	vw
3.15	vvs	1.89	vw	1.32	vvw
3.01	vs	1.81	w		
2.62	s	1.72	w		

The unit cell parameters obtained by Hull[2] for a compound listed as "cuprous acetate monohydrate" apply to cupric acetate monohydrate; calculated interplanar spacings, d_{hkl}, from Hull's data agreed within experimental error with the *d/n* values obtained from the diffraction pattern of the cupric salt.

Attempts to prepare cupric oxyacetate by oxidation of a quinoline solution of cuprous acetate failed because of a catalyzed oxidation of the quinoline. It was found possible, however, to prepare the anhydrous oxyacetate by oxidation of dry cuprous acetate; at 100°, the oxygen absorption is stoichiometric. On prolonged heating at higher temperatures (150–200°), the oxyacetate decomposes, with the production of metallic copper. Cupric oxyacetate is dark bluish-green in color. Microscopic examination of the samples prepared showed the crystals to be acicular or prismatic and possibly of equidimensional cross-section. The crystals were too small for interference observations, but the presence of birefringence indicates that the material is not cubic. Bjorstrum charts for tetragonal and hexagonal crystals were prepared for comparison with the observed X-ray diffraction lines obtained from the oxyacetate. As the following table indicates, a possible fit was found in the tetragonal system with $c/a = 1.35$, $c = 16.26$ Å., $a = 12.04$ Å.

I, obsd.	*d/n*, obsd.	*d*, calcd.	*hkl*
vvs	12.0	12.04	100
s	8.0	8.13	002
s	7.4	7.55	111
ms	6.0	6.02	200
vvw	5.4	5.42	003
vvw	5.0	5.10	211
s	3.75	3.78	222
vvvw	3.35	3.37	204
vvvw	3.00	3.01	400
vvw	2.71	2.71	006
m	2.46	2.47	206
w	2.14	2.14	335
w	2.09	2.10	424
vvw	1.89	1.89	444
vvw	1.85	1.85	622
vvw	1.80	1.80	604
vvvw	1.505	1.505	800
vvvw	1.280		

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(1) M. Calvin, THIS JOURNAL, **61**, 2230 (1939).

(2) R. B. Hull, University of Pittsburgh Bulletin, **35**, 142 (1938).